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New approaches in QuEChERS sample preparation for pesticide and pollutant analysis – expanding the scope for matrix and sample size

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aus

Freyung

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Erklärung

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Eidesstattliche Versicherung

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Table of contents

1. Introduction

1.1. Pesticides in agriculture – a blessing and a curse

For many decades, it has been impossible to imagine our agriculture without agrochemicals. This heterogeneous group of mostly synthetic compounds includes fertilizers, soil conditioners, and pesticides.[1] However, their use and impacts – both positive and negative – have since then been, and still are, a matter of debate. The most important field of agrochemical usage certainly is plant protection with pesticides.

Undeniably, pesticides have many advantages: Most importantly, they secure harvests by protecting them from certain pests, which on the one hand secures the farmers economically as they do not have to fear major crop failures, and on the other hand minimizes the risk of food shortage or even famines, especially in poorer countries. Particularly in view of the steadily growing world population and the associated increased demand for foodstuffs, pesticides have become an indispensable part of modern agriculture.[2] By now, the compounds used for crop protection are further developed than in the past. Due to a higher potency, the amount of active ingredient applied could be reduced to 10% compared to the 1960s.[2] Many of them are highly selective and can be used specifically against the respective pest, for example pirimicarb, a “soft insecticide”, which is used against aphids but does not negatively affect beneficial insects.[3, 4] Furthermore, it is possible to apply the pesticides at all stages of plant growth, depending on the occurrence of the disease or pest: from seed treatment (*e.g.* tolclofos-methyl against black scurf (*Rhizoctonia solani*) in potatoes) and post-emergence treatment (*e.g.* fluazifop-p-butyl against grass weeds in crops) to application on the harvested fruit (for example imazalil and thiabendazole as antifungal agents on citrus fruits).[3, 5, 6] This application procedure helps to control the plant diseases in the most effective way.

Due to the globally high demand of comestibles, it is also very important to use the available arable land in the best possible way. However, producing on the same area through ecological agriculture brings between 20 and 50% lower yields at the harvest, which also leads to higher food prices.[7] Hence, pesticides can contribute to a sufficient and affordable food supply worldwide. Especially the abovementioned post-harvest treatment can also help to prevent storage rots and the food is thus preserved longer.[3]

In spite of these important advantages, the use of agrochemicals also has major drawbacks. Most importantly, the compounds pose a high risk to health and the environment, which is further discussed in Chapter 1.3. Apart from the health aspect, they can also negatively affect the cultivation. For many crops, like for example maize or rice, the main cultivation method in modern agriculture is monocultural farming. This kind of cultivation is advantageous for farmers because they always need the same type of agricultural machinery, which eases sowing,

harvesting and processing of the plants.[8] Farmers also frequently use high yielding variety (HYV) seeds or hybrid (F_1) plants, as these promise better harvest results and grow more synchronized on the field.[9] However, monocultural farming makes the plants more vulnerable to pests, because one pest can destroy a whole crop very easily. Thus, extensive pesticide use is inevitable in order to avoid crop failures. In turn, this can eventually lead to a development of resistance of the respective pest. Additionally, monocultures leach soils much faster, which leads to a one-sided nutrient depletion and eventually also results in more vulnerable plants.[8] Another possible disadvantage is the unintentional elimination of a natural predator of a certain pest, caused by broad-spectrum pesticides such as organophosphates or pyrethroids (**Fig. 9, 10**).[10] All scenarios ultimately lead to the same outcome: an even more increased pesticide use. **Fig. 1** shows the resulting *circulus vitiosus* of chemical agriculture.[11]

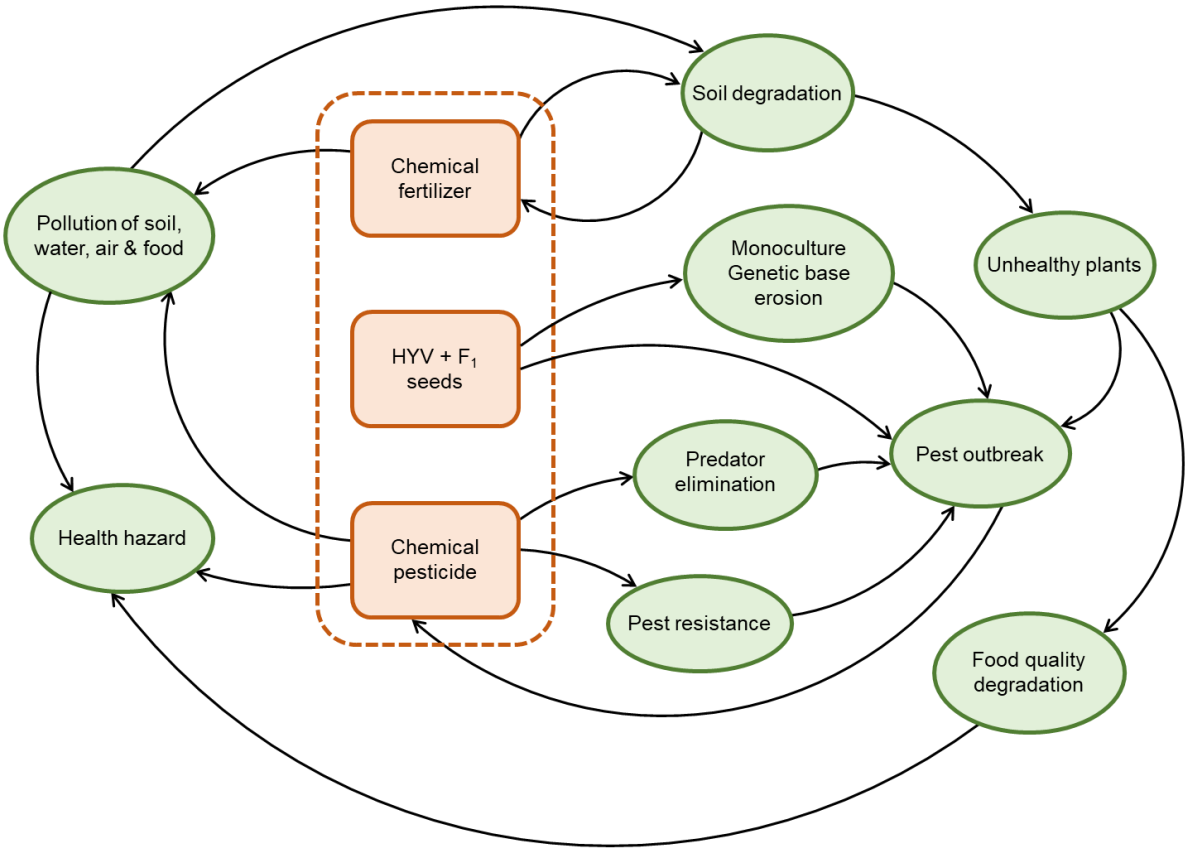


Figure 1 The vicious cycle of chemical agriculture, adapted from Murakami.[11] The chart shows how the use of agrochemicals, like fertilizers and pesticides, and the possible consequences for humans and the environment are interrelated. HYV: high yielding variety; F_1: hybrid seeds.

1.2. Classifications of pesticides and pollutants

In order to get a better overview of the vast amount of different plant protection products – solely in Germany, 288 different active ingredients were approved for application in 2019[12] – the most important classes and their respective representatives are listed in the following chapter. Additionally, the most widespread pollutants are introduced, as they bear a deep environmental impact as well.

1.2.1. Fungicides

A fungicide, antifungal or antimycotic is "any toxic substance used to kill or inhibit the growth of fungi. Fungicides are generally used to control parasitic fungi that either cause economic damage to crop or ornamental plants or endanger the health of domestic animals or humans."[13] There are multiple modes of action that the different fungicide classes take advantage of. Two of the most important targets are presented below: the biosynthesis of ergosterol, a sterol solely produced in fungi, and the mitochondrial respiration.

The most frequently used fungicides for agricultural purposes are azole antifungals. They intervene in ergosterol biosynthesis predominantly by inhibiting the CYP51 dependent enzyme sterol C14-demethylase.[14, 15] The common functional group is an azole ring; modern azole antifungals all contain a triazole ring, whereas the early substances were imidazoles. All azoles are broad-spectrum fungistatics, *i.e.* they inhibit the fungal growth but do not kill the fungus. Due to their frequent use, they are prone to resistances. There are different resistance mechanisms: on the one hand, the target enzyme CYP51 can mutate or be overexpressed, which both results in an azole tolerance. On the other hand, an overexpression of efflux transporters can reduce the intracellular concentration of the azoles.[15] **Fig. 2** shows two important representatives of triazole antifungals.

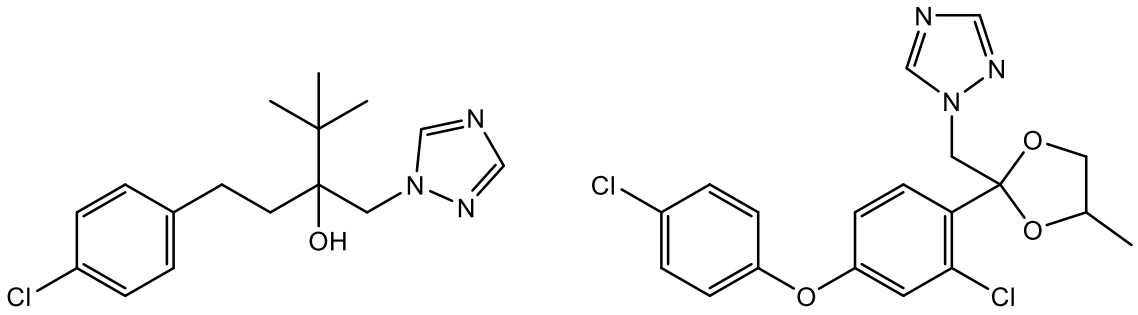


Figure 2 Triazole antifungals currently approved for use in the EU.[3, 16] Left: tebuconazole. Right: difenoconazole.

Morpholine antifungals interfere with ergosterol biosynthesis as well. This group of fungicides is able to inhibit two enzymes: sterol C14-reductase and sterol C8-isomerase.[14] As already indicated in the name, the functional group responsible for the mode of action is a morpholine ring. The first marketed substance was fenpropimorph, which is not approved for application

anymore. One example for an approved morpholine in 2022 is dodemorph.[16] Both are shown in **Fig. 3**.

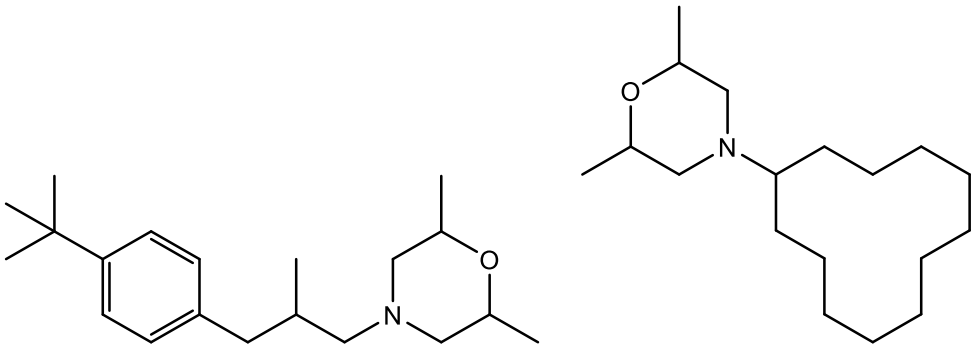


Figure 3 Morpholine fungicides. Left: the first morpholine fenpropimorph. Right: the currently approved fungicide dodemorph.

The strobilurins represent another important fungicide class. These synthetic compounds originate in strobilurin A, an antimycotic compound produced in the fungus *Strobilurus tenacellus*.[17] The characteristic functional group of strobilurins is the methyl β-methoxyacrylate group, which is also found in the first synthetic compound azoxystrobin. However, in other compounds like pyraclostrobin, this key group was replaced by a methyl N-methoxycarbamate (**Fig. 4**).[18, 19] Their target is the Q_0 site of cytochrome bc1, an enzyme complex in the mitochondrial respiration. An inhibition of the enzyme stops the electron transfer and thereby adenosine triphosphate (ATP) production, and ultimately leads to cell death of the fungus.[18] Most strobilurins are broad-spectrum fungicides, and as they are site specific inhibitors, they are considered to be especially prone to resistances.[20] Known resistances originate from an amino acid shift in the targeted cytochrome b protein.[19]

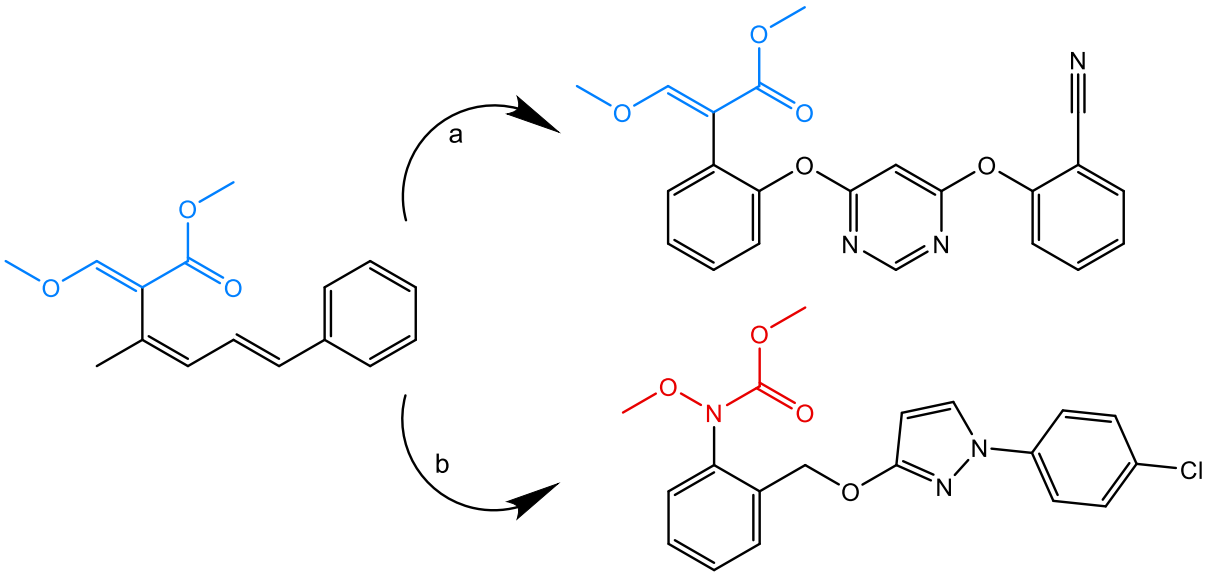


Figure 4 Different toxophores in strobilurins. The left structure shows strobilurin A, a natural compound produced by *Strobilurus tenacellus*. Modification **a** leads to azoxystrobin, the first synthetic strobilurin. Modification **b** leads to pyraclostrobin, a more recent compound with a modified functional group. Blue partial structure shows methyl β-methoxyacrylate group, red partial structure shows methyl N-methoxycarbamate.

1.2.2. Herbicides

A herbicide is “an agent, usually chemical, for killing or inhibiting the growth of unwanted plants, such as residential or agricultural weeds and invasive species.”[21]

By far the best known broad-spectrum compound surely is glyphosate, a systemic broad-spectrum herbicide with a phosphonic acid group (**Fig. 5**). This herbicide, originally traded under the name Roundup® by Monsanto, is known to be the most used herbicide worldwide.[22] Especially since the introduction of glyphosate-resistant crops, the popularity of glyphosate has increased rapidly, as it could be applied at all plant stages without harming the crop.[23, 24] Before that, glyphosate could only be applied before emergence of the crop or when contact to crop foliage could be avoided, as it is taken up by foliar plant parts, but has no soil activity.[24] The compound acts by inhibition of the enzyme enolpyruvylshikimate-3-phosphate synthase (EPSPS), which is part of the shikimate pathway in plants.[22, 25] Consequently, the endogenous synthesis of aromatic amino acids is stopped, which supposedly causes the herbicidal effect.[24]

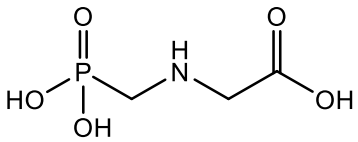


Figure 5 The broad-spectrum herbicide glyphosate.

Among the oldest synthetic herbicides are 2,4-D (2,4-dichlorophenoxyacetic acid) and MCPA (2-methyl-4-chlorophenoxyacetic acid), which were both invented during or shortly after World War II, and are still approved for use in parts of the European Union today.[3] Both belong to the auxin herbicides. The most important natural auxin is indole-3-acetic acid (IAA), which is known as a phytohormone and is responsible for many plant growth processes. Auxin herbicides are able to mimic IAA and overstimulate auxin processes, leading to an uncontrolled growth of dicot weeds and ultimately, plant death.[26] Auxin herbicides are a chemically heterogeneous group, including phenoxy carboxylic acids (*e.g.* 2,4-D, MCPA, MCPB), benzoic acids (*e.g.* dicamba) and pyridine carboxylic acids (*e.g.* triclopyr).[27] Many substances are also available as salts or ester compounds (*e.g.* triclopyr-butotyl).[3] **Fig. 6** shows the natural auxin IAA and 2,4-D.

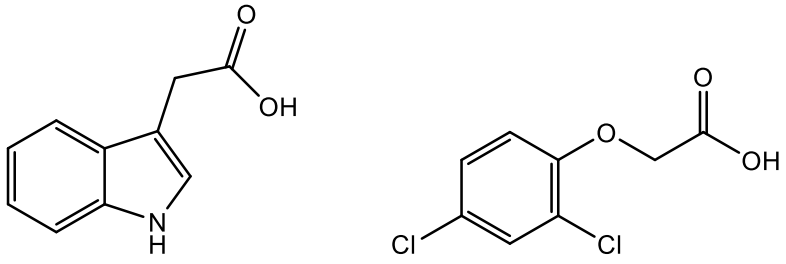


Figure 6 Auxin active substances. Left: the natural phytohormone IAA. Right: the phenoxy acetic acid 2,4-D.

Another mode of action of herbicides is the inhibition of acetyl-CoA carboxylase (ACCase). This enzyme catalyses the carboxylation of acetyl-CoA in order to build malonyl-CoA, which is crucial for fatty acid biosynthesis. The lack of fatty acids negatively impacts plant cell membranes and also surfaces, like wax coverings.[28] Just like the auxin herbicides, this group is chemically heterogeneous. Important substances are the aryloxyphenoxypropionates, also called “fops”, with diclofop-methyl being the first ACCase inhibitor on the market in 1978, and the cyclohexanediones (“dims”, *e.g.* tralkoxydim; **Fig. 7**).[28, 29]

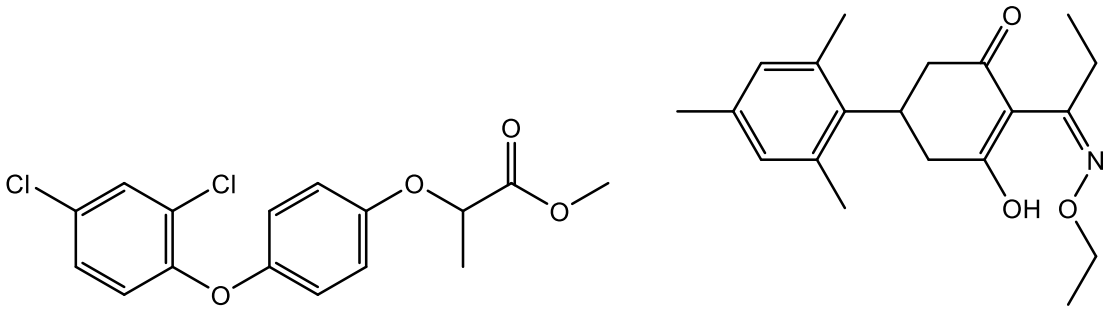


Figure 7 ACCase inhibitors. Left: the aryloxyphenoxypropionate diclofop-methyl. Right: the cyclohexanedione tralkoxydim.

An inhibition of photosynthetic processes is the mechanism of action in phenylurea herbicides. These compounds bind to a protein complex of photosystem II and thus block the electron transport during photosynthesis. As a consequence, the plant suffers from a lack of ATP, along with oxidative stress.[30] Apart from phenylurea compounds, also uracil derivatives work with the same mode of action. **Fig. 8** shows the phenylurea herbicide chlorotoluron and the uracil herbicide lenacil, which are both approved in EU countries.[3, 16]

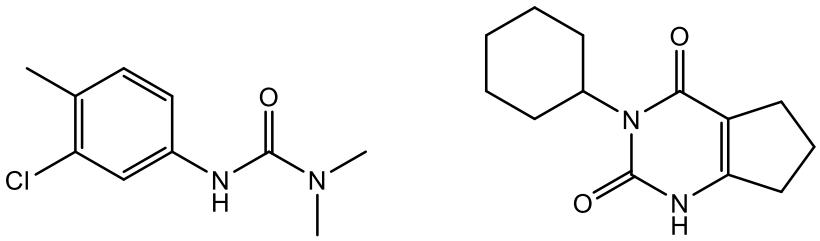


Figure 8 Herbicides inhibiting photosystem II. Left: the phenylurea derivative chlorotoluron. Right: the uracil derivative lenacil.

1.2.3. Insecticides

An insecticide is “any toxic substance that is used to kill insects. Such substances are used primarily to control pests that infest cultivated plants or to eliminate disease-carrying insects in specific areas.”[31] Insecticides are an important tool in agriculture, as there are multiple insect pests like beetles, aphids, flies or caterpillars that need to be controlled in order to avoid harvest failures.

The first ever synthetically produced insecticides were the organochlorines (OCs), a substance class consisting of chlorinated hydrocarbons. Their extensive production and use began during World War II, but after becoming aware of their adverse effects, their use were drastically reduced in the 1970s, and they were ultimately forbidden after the Stockholm Convention in 2001.[3, 32] Important substances belonging to that insecticide group are dichlorodiphenyltrichloroethane (DDT) with its metabolite dichlorodiphenyldichloroethylene (DDE, **Fig. 9a**), and lindane (γ-hexachlorocyclohexane (γ-HCH), **Fig. 9b**). The mode of action is an overexcitation of motoneurons by preventing the closure of a voltage dependent Na^+ channel, causing convulsions and ultimately, death of the insect.[33] Some organochlorines can block γ-aminobutyric acid (GABA) receptors, which causes a hyperexcitation in the central nervous system (CNS), also resulting in convulsions.[34]

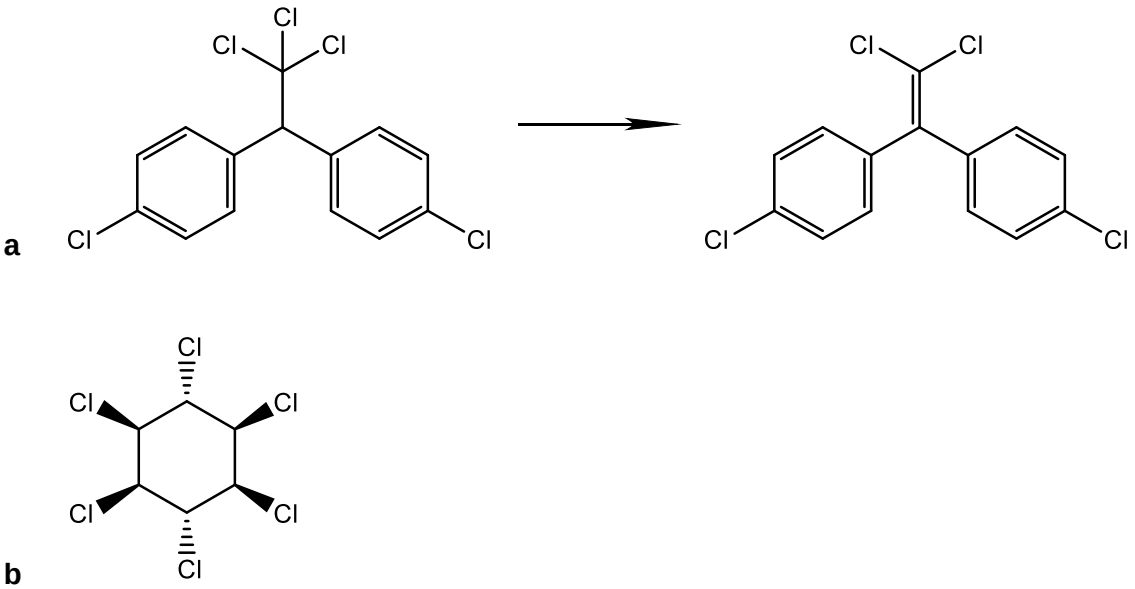


Figure 9 a: Conversion from *p,p'*-DDT (left) to *p,p'*-DDE (right) through an elimination of HCl, **b:** the organochlorine insecticide lindane.

Though structurally not related to OCs, the systemic insecticide fipronil **(Fig. 10**) also blocks GABA receptors.[35] This phenylpyrazole compound was introduced in the 1990s and was frequently used for plant protection until its expiration of approval in 2017.[16] It is still used today in veterinary medicine, predominantly as spot-on applications for flea control in cats and dogs.[36]

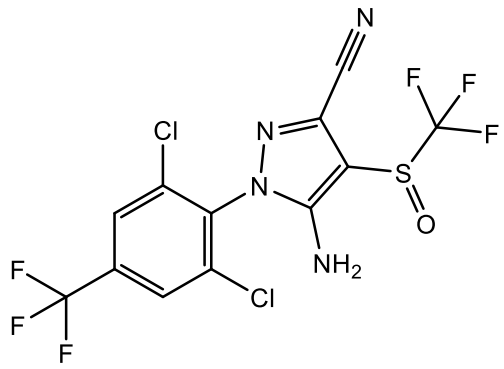


Figure 10 The phenylpyrazole insecticide fipronil.

Pyrethroids act on the same Na^+ channel as organochlorine insecticides, both on peripheral nerves and in the CNS, and they are still used in recent days.[3, 34] These synthetic substances originate from the pyrethrins, that naturally occur in plants of *Chrysanthemum* spp.[33] The synthetic pyrethroids are improving the drawbacks of the natural products, such as photoinstability and a rather low potency. Type I and II pyrethroids differ in a nitrile group at the α-carbon of the alcohol moiety, which again increases the molecule's activity, as the nitrile compounds are able to block the ion channel for a longer period of time.[33] **Fig. 11** shows examples of a natural pyrethrin and both types of pyrethroids.

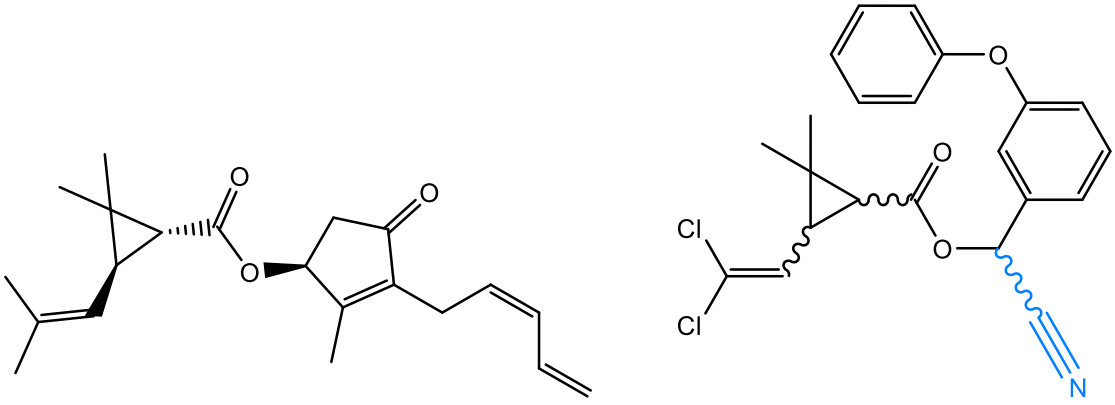


Figure 11 Left: The natural compound pyrethrin I. Right: black structure shows type I pyrethroid permethrin (without defined stereochemistry), addition of a cyano group (blue partial structure) gives type II pyrethroid cypermethrin.

Another mode of action of insecticides is the inhibition of acetylcholinesterase (AChE), which is made use of in organophosphates. Due to this enzyme inhibition, the neurotransmitter acetylcholine (ACh) is accumulated and causes an overstimulation of acetylcholine receptors located at the neuromuscular junctions, leading to muscle cramps and ultimately, paralysis.[34] As acetylcholine is an important neurotransmitter not only in insects, but also mammals, the toxicity for humans and animals is rather high; the most famous substance is parathion, better known as E605®, which used to cause frequent poisonings before its banning in 2001.[16, 37]

Parathion can be regarded as a prodrug – the active form is paraoxon, which is formed in the course of metabolism in the liver (**Fig. 12 a**).[38] A more recent and frequently used organophosphate is chlorpyrifos, which was approved for use in the EU until 2020 (**Fig. 12 b**).[16]

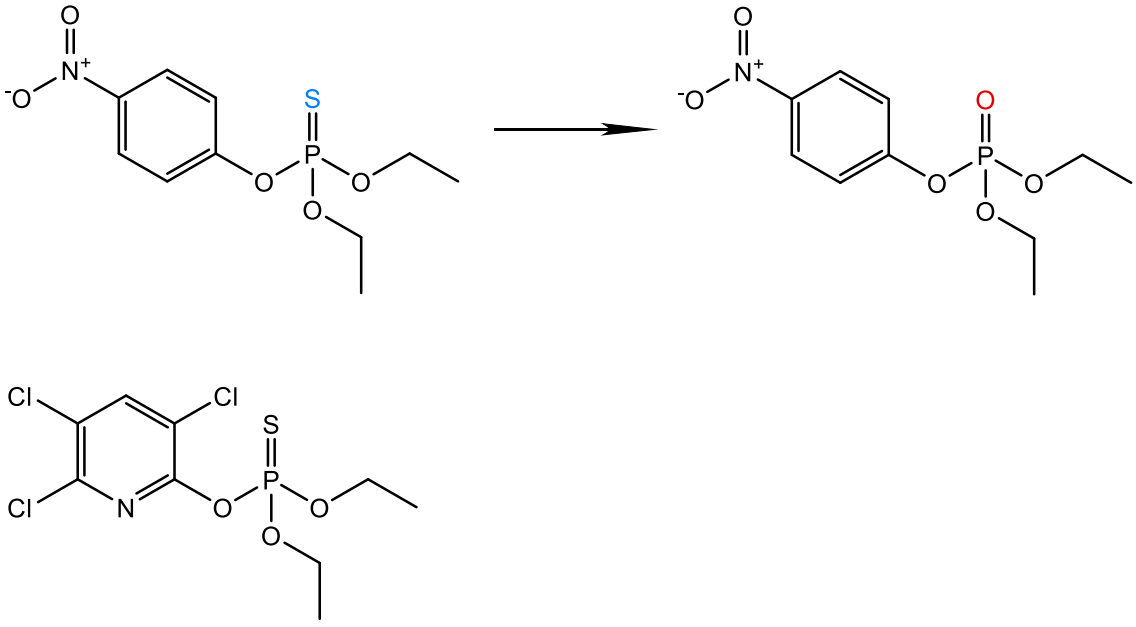


Figure 12 a: Conversion of parathion (E605®, left) to paraoxon (right), the highly toxic active form, by replacing the sulfur with an oxygen, **b:** the organophosphate chlorpyrifos.

Today, the most important insecticides are the neonicotinoids. They act as agonists of the nicotinic acetylcholine receptor (nAChR) in the CNS of insects by mimicking the natural neurotransmitter ACh, ultimately leading to an overexcitation, associated with paralysis and insect death.[39, 40] Thus, the neonicotinoids' mode of action also targets the acetylcholine pathway, similar to the organophosphates. As the nAChR of mammals and insects differs in certain receptor subunits, the substances are considered only moderately toxic to mammals.[40] **Fig. 13** shows the alkaloid nicotine, a nAChR activator, and imidacloprid, a neonicotinoid.

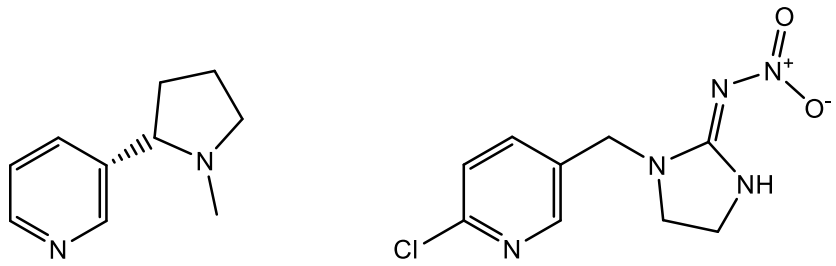


Figure 13 Structures of nAChR active substances. Left: the plant alkaloid nicotine. Right: the neonicotinoid imidacloprid.

1.2.4. Rodenticides

A rodenticide is “any substance that is used to kill rats, mice, and other rodent pests.”[41] For many decades, the application of anticoagulants used to be a common method to kill rodents, until the last substance of this group, bromadiolone, was prohibited in May 2021.[16] The mode of action of anticoagulants is the inhibition of vitamin K epoxide reductase complex, thus preventing the formation of coagulation factors.[42, 43]

There are two chemical types of anticoagulant rodenticides. Anticoagulants of the coumarin type are also well-known from human medicine, where they are used to prevent thromboembolic diseases.[43, 44] They can be divided into 1st (warfarin) and 2nd (bromadiolone) generation coumarins, depending on their potency.[45] The second type of anticoagulant rodenticides are indanedione derivatives, like chlorophacinone.

In recent days, the most common rodenticides are phosphides. They build phosphine after contact with air moisture or digestion in the stomach, thereby creating a toxic gas (PH_3) that inhibits oxidative respiration in mitochondria.[3, 46] **Fig. 14** shows the different classes of anticoagulant rodenticides.

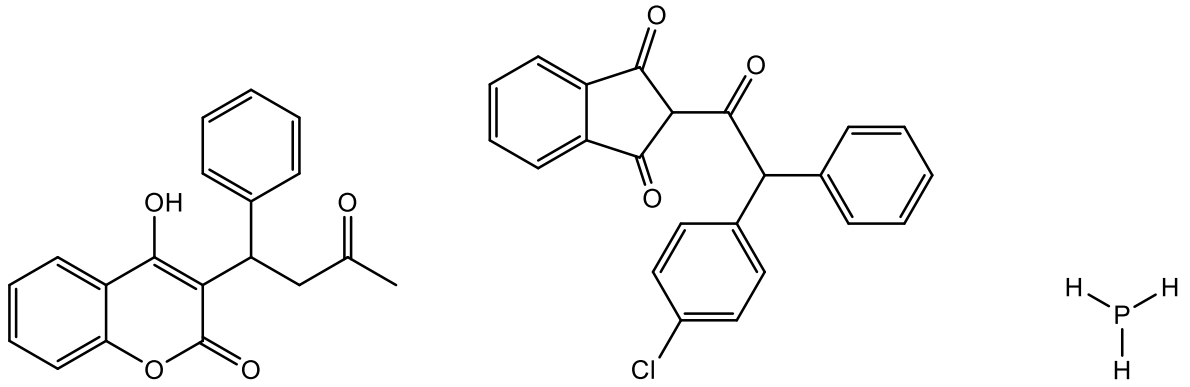


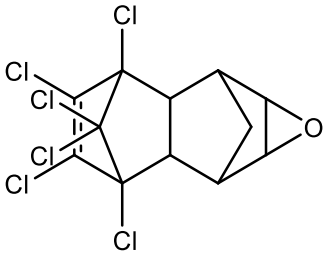
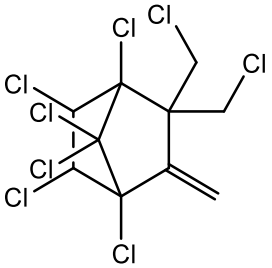
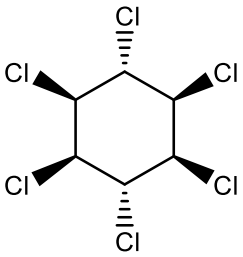
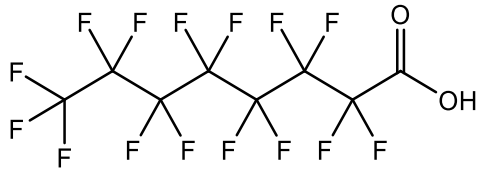
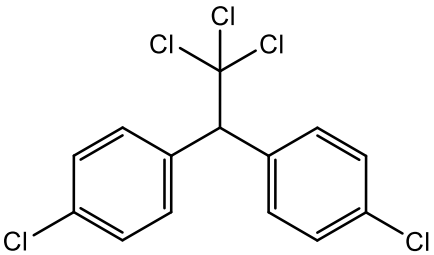
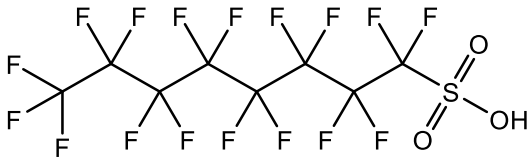
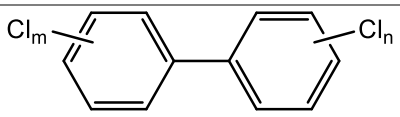
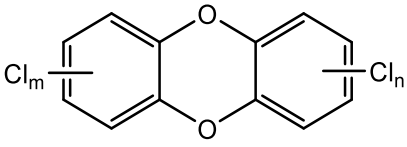
Figure 14 Rodenticides. From left to right: the coumarin derivative warfarin, the indanedione derivative chlorophacinone, and the toxic gas phosphine.

1.2.5. Pollutants

“A pollutant is a substance that is present in concentrations that may harm organisms (humans, plants and animals) or exceed an environmental quality standard.”[47] Thus, a pollutant is not necessarily spread in the environment intentionally – unlike the abovementioned plant protection products –, but can also be a by-product of industrial production, for example.[32] Pollutants can settle in soil, water or air and cause various adverse effects to human health, such as an exacerbation of asthma or COPD (chronic obstructive pulmonary disease) symptoms through air polluting agents like ozone or nitrogen dioxide.[48]

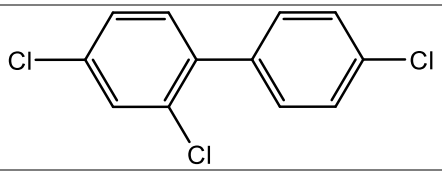
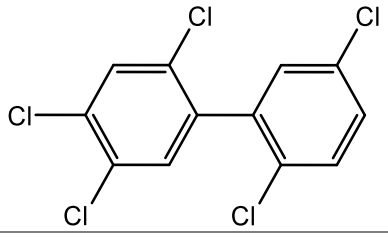
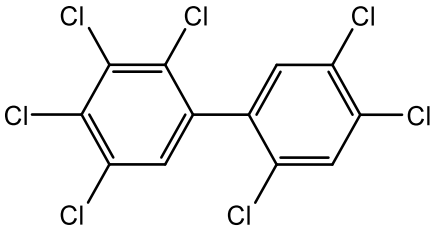
However, the greatest health and environmental concern is caused by persistent organic pollutants (POPs). This term was coined by the Stockholm Convention on Persistent Organic Pollutants, which was initiated by the UN Environment Programme in 2001.[32] POPs are defined as organic compounds that “remain intact for exceptionally long periods of time, and become widely distributed throughout the environment as a result of natural processes.” Furthermore, they “accumulate in the fatty tissue of living organisms including humans” and “are toxic to both humans and wildlife.”[32] Additional to this definition, the POPs were classified in different categories, depending on their potential toxicity. Annex A of the Stockholm Convention lists substances that are aimed to be completely eliminated from production, whereas the compounds in Annex B are under a severe restriction. In Annex C, one can find substances that bear a health risk but are inevitably built as by-products. The aim is to reduce the release of Annex C substances into our environment *via* industrial production to a minimum.[32] **Table 1** shows a summary of the most important POPs.

Table 1 Classification of persistent organic pollutants (Annex A-C) according to the Stockholm Convention.[32] PFOA: perfluorooctanoic acid, PFOS: perfluorooctanesulfonic acid, PCB: polychlorinated biphenyl, PCDD: polychlorinated dibenzodioxin.

Annex	Type of limitation	Examples	
A	Elimination	Dieldrin	
		Toxaphene	
		Lindane	
		PFOA	
B	Restriction	DDT	
		PFOS	
C	Unintentional production	PCB	 m, n = 1,2,3,4, or 5
		PCDD	 m, n = 1,2,3, or 4

Hence, also pesticides, more precisely insecticides of the organochlorine type, were later classified as POPs and therefore banned from agricultural use. Taking a closer look at the chemical structures reveals that, even though the POP compounds have different carbon backbones and various physicochemical properties, there is one common feature: all of the compounds are polyhalogenated. The degree of halogenation closely correlates with the environmental persistence, as the carbon-chlorine or carbon-fluorine bonds in these substances are less reactive than a carbon-hydrogen bond, which makes them less prone to environmental degradation.[49] Regarding the polychlorinated biphenyls (PCBs), the substance half-life dramatically increases along with the chlorination degree, which is shown in **Table 2**.[50]

Table 2 Structures and half-lives in soil of polychlorinated biphenyls with different chlorination levels.[50]

PCB congener	Structure	Cl atoms	Half-life in soil [years]
PCB 28		3	1
PCB 101		5	11
PCB 180		7	114

1.3. Adverse effects of pesticides and pollutants

In spite of all the benefits of pesticides, there are also massive impacts on the environment and health of humans and animals, and their far-reaching consequences were underestimated in the past century. In Chapter 1.2.5., it was already outlined that especially organochlorines and halogenated industrial chemicals have adverse health effects. All of them were classified as POPs and banned in 2001, but had been released in the environment for several decades before.[32] DDT, the most prominent organochlorine compound, was introduced as an insecticide in the 1940s. It was effectively used to prevent malaria or typhus outbreaks during and after World War II, which until today is considered the most positive aspect of DDT, and the reason for its listing in Annex B of the Stockholm Convention.[32] Nevertheless, the majority

of DDT was used for agricultural purposes after 1945, and in the 1960s, approximately 400,000 tons of the compound were applied per year on farmlands worldwide.[51] However, during this time, voices began to rise about the adverse effects of DDT and other organochlorine compounds like dieldrin. Finally, public awareness fully rose after the publication of the book *Silent Spring* by marine biologist and conservationist Rachel Carson in 1962.[52] The book title refers to the massive decrease in bird populations in the USA after repeated sprayings with organochlorine compounds in order to fight insect pests. Especially DDT and its main metabolite DDE (**Fig. 9a**) weaken the eggshells of birds, which causes a higher mortality of the offspring. Furthermore, beneficial insects were eradicated and aquatic animals also suffered from reproduction failures.[51] Carson's statements in *Silent Spring* caused an environmental movement and eventually lead to the prohibition of DDT by the United States Environmental Protection Agency (US EPA) in 1972.[53] But due to its persistence, the consequences are still perceptible today, and even in very rural places where DDT has never been used.[54] Apart from the threats to wildlife, POPs also pose a risk to human health. OCs and PCBs are possible endocrine disruptors, and their estrogenic properties can contribute to premature births and a reduced birth weight of the child.[51, 55] Additionally, a prenatal exposure or uptake through breast milk can negatively affect the child's immune system.[51] Furthermore, many of the compounds are considered to be carcinogens, *e.g.* DDT is suspected to cause mammary cancer and was proven to cause liver tumours in rats.[51]

Another substance associated with an enormous cancer risk is 2,3,7,8-tetrachloro-dibenzodioxin (TCDD). It was a by-product of Agent Orange, a herbicide mixture containing 2,4-D and 2,4,5-T (2,4,5-trichlorophenoxyacetic acid), that was widely spread by the US army during Vietnam War in the 1960s.[56] The consequence was a higher occurrence of Non-Hodgkin lymphoma, chronic lymphocytic leukaemia (CLL) and Hodgkin disease in the exposed persons, and cases of acute myeloid leukaemia (AML) in their children.[56]

Apart from insecticides, also fungicides, especially azoles, can pose a risk to human health. The problem is not caused directly by the properties of azole antifungals, as this chemical class of antifungals is also used in human medicine,[14] but indirectly through their proneness to fungal resistances, which can be caused by their excessive use for agricultural purposes.[15] Azole medication is the most common therapy for fungal infections in clinic patients. However, even patients who had not received azoles before, no longer responded to the treatment.[57] Supposedly, pathogenic fungi, such as *Aspergillus fumigatus*, one of the main representatives of *Aspergillus* spp., have developed azole-resistant strains through the abovementioned resistance mechanisms (see Chapter 1.2.1.) because of their enduring contact with azole antifungals in nature. This effect exacerbates appropriate clinical therapy for patients with pathogenic fungal infections.[57, 58]

In general, pesticides can also be accumulated through the food chain. Especially lipophilic pesticides are hazardous, as they are likely to be absorbed in adipose tissue, and can be found in many different sources of animal origin.[59] Hence, humans, being at the top of the food chain, are prone to pesticide exposure through food, which in turn can lead to the abovementioned adverse health effects. Therefore, an appropriate monitoring of pesticide residues in comestibles is inevitable.

1.4. Monitoring of pesticides and POPs

In order to safely prevent possible health risks through pesticide contaminated food, various international guidelines have been brought to life. Especially in the light of international food trade, such regulations are essential, and the establishment of maximum residue limits (MRLs) was a milestone in achieving the goal of preserving international food standards. For the European Union, the European Commission created the EU Pesticides database,[16] whereas the Codex Alimentarius, founded by the Joint FAO/ WHO (Food and Agriculture Organization of the United Nations/ World Health Organization) Food Standards Programme, aims to secure the food trade globally.[60] MRL data are specified for every pesticide and every commodity separately, *e.g.* the EU MRL for the fungicide tebuconazole is 0.3 mg kg^{-1} in apples, but 40 mg kg^{-1} in hops.[16] Overall, there are 66 different MRLs for tebuconazole in the Codex Alimentarius.[60] Besides MRLs of approved pesticides, also banned chemicals like the POPs DDT and lindane are still monitored and have a maximum residue limit.[16]

As important as the MRL itself is the compliance of the food products with the given limit, which needs to be confirmed by appropriate analytical methods. Due to the vast amount of different active ingredients in plant protection products and their varying physicochemical properties, this can be a highly challenging task. In the 1960s, it was sufficient to find a method that covered a rather small spectrum of different pesticides, especially organochlorines. The first residue method that found broad application was the Mills method,[61] which initially extracted the target compounds from the matrix with acetonitrile, followed by a liquid-liquid-extraction (LLE) with petroleum ether, a clean-up with a Florisil® solid phase extraction (SPE) column and analysis *via* gas chromatography (GC) and electron capture detection (ECD). As the method covered 21 organochlorine pesticides, it is considered the first multiresidue method. However, with the introduction of multiple other pesticide classes, “multiresidue analysis” took on a new meaning. Thus, the Mills method was further improved in order to meet the increased requirements. In 1975, Luke *et al*.[62] (adapted by the Association of Official Analytical Chemists (AOAC) as AOAC official method 985.22) established an initial extraction with acetone instead of acetonitrile. As a second step, partitioning was performed with a mixture of petroleum ether and dichloromethane, which was assisted by the addition of sodium chloride. Additionally, for

the analysis of organophosphate and organonitrogen pesticides, the Florisil® clean-up was skipped, and a nitrogen-phosphorus detector (NPD) was used instead of an ECD in order to increase sensitivity. In the 1980s, the Specht method[63] (adapted by the German Research Foundation (Deutsche Forschungsgemeinschaft, DFG) as the DFG S 19 method) introduced further improvements to the dichloromethane extract clean-up by performing a gel permeation chromatography (GPC) prior to GC analysis. Furthermore, Specht introduced mass spectrometry (MS) as a detection method in pesticide multiresidue analysis.[64] Both the Luke and the Specht method paved the way for the parallel analysis of more than 70 different pesticide residues at once. However, there were still major drawbacks. The sample preparations required large amounts of sample material (100 g), were time-consuming and labour-intensive, and involved the usage of large amounts of hazardous solvents like dichloromethane, cyclohexane, or petroleum ether. Some steps also required additional instruments like GPC, and there were several different detection methods. Thus, there was a need for various instruments, and the samples needed to be analysed more than once. In order to eliminate the hazardous solvents, the DFG S 19 method was further modified, replacing the whole partitioning step with dichloromethane by a mixed SPE clean-up using C_{18} and cyano material.[65] This step abandoned the toxicological concerns caused by the solvent, but further increased the costs. Additionally, even though SPE is an efficient clean-up method, it did not exclude the necessity of using GPC for challenging matrices.

In 2003, a new milestone was set for multiresidue analysis with the introduction of the QuEChERS approach by Anastassiades *et al*.[66] The acronym stands for “Quick, Easy, Cheap, Effective, Rugged, and Safe” and describes the key characteristics of this sample preparation method. In comparison with the abovementioned methods, QuEChERS introduced a row of novelties and modifications: the method includes a salt-assisted liquid-liquid extraction (SALLE) solely using acetonitrile as a solvent, followed by a dispersive SPE (dSPE) of the raw extract with magnesium sulfate ($MgSO_4$) and primary secondary amine (PSA) sorbent, prior to GC-MS analysis. Hence, the sample preparation essentially consists of two steps only, thus allowing a high sample throughput. Furthermore, only 10 g of sample material are required for this new approach, which helps reducing the amount of solvent and consumables used per sample. Also, with QuEChERS, mass selective detection became fully established in pesticide multiresidue analysis. Even though the original method uses only gas chromatography, it was quickly applied to liquid chromatography (LC) systems, too, making the method even more changeable.[67] For all these reasons, QuEChERS-based sample preparations are the basis for most regulatory measures used today, *e.g.* the DIN EN 15662 guideline[68] in Europe, and the AOAC official method 2007.01[69] in the US.

2. Objective

Since its publication in 2003, the QuEChERS approach has proven to be a stable, reproducible, and convertible sample preparation method.[66] Even though originally developed for matrices with high water content such as fruit and vegetables, the method was rapidly extended to other matrices like food of animal origin or dry commodities like raisins.[70, 71] Due to its variability, there are many modifications considering the extraction solvent,[72] sample clean-up,[73] and analytical instrumentations.[74] Over the years, the method has been adapted by many laboratories, not only for food analysis, but also for environmental, clinical, or forensic matters, thus expanding the range of matrices, but also of target compounds. Hence, it is now possible to analyse human matrices like blood,[75] urine,[76] or breast milk,[77] and also to screen for xenobiotics[78] and drugs of abuse.[79, 80] The state-of-the-art detection method is tandem mass spectrometry (MS/MS), usually a triple quadrupole (QQQ) MS, which has a dramatically increased selectivity and sensitivity compared to a single quadrupole MS, and can hide matrix effects up to a certain degree due to specific transitions for each analyte.

However, even when combining the QuEChERS approach with this powerful detection method, there are still limitations considering the sample size and also the clean-up. Especially complex matrices, for example with high fat content (*e.g.* avocado, salmon) or highly pigmented samples (*e.g.* herbs, spices), can bear a problem to the analytical instrument without a proper clean-up, as the system gets contaminated and requires a more frequent maintenance compared to analysis with cleaner samples. Furthermore, as the QuEChERS method originally was designed for multiresidue analysis in foodstuffs, the sampling was rather unproblematic because there is always a sufficient amount of material. But, as soon as an individual analysis must be performed on small sample sizes, major modifications must be made to the sample processing.

The focus of this work was to address these limitations for the purpose of environmental multiresidue analysis with GC-MS/MS.

Firstly, the pesticide and pollutant exposure of wildlife animals was investigated. A special focus was set on the exposure of bats, as all bat species in Germany are under conservation.[81] Therefore, liver samples of different animal species (hedgehogs, various bat species) were provided by Dr. Egbert Kröner from the wildlife animal rehabilitation center (Wildvogel-Pflegestation Kirchwald e.V.) in Kirchwald, Germany, and the group around Prof. Dr. Michael Veith from the Chair of Biogeography at Trier University, Germany. For the investigation of pesticide residues in wildlife animals, liver was the matrix of choice, as it is easily obtainable from deceased animals by dissection and is a main organ for contaminant accumulation. These properties also makes the liver a popular matrix in forensic analysis.[79, 80] Additionally, liver tissue is more suitable for the analysis of compounds with various polarities compared to

adipose tissue, where mostly very lipophilic compounds accumulate.[82] Also, liver contains only the target analyte or phase I metabolites, which are GC-amenable without further modification, whereas for example, phase II metabolites would need a deconjugation of glucuronides or sulfates.[83, 84] Deconjugation should be avoided as it would complicate the sample preparation and, in most cases, exacerbate the recoveries. Furthermore, as some pesticides, *e.g.* cypermethrin (**Fig. 11**), are hepatotoxic,[85] a significant concentration within liver tissue is especially alarming.

As the goal was to perform individual analysis of all animals, a miniaturization of the QuEChERS approach was necessary, because for the bat species, less than 1 g of sample material was available. Additionally, liver is a dense tissue and requires a suitable sample preparation to ensure an efficient analyte extraction. Furthermore, liver has a rather high fat content, so an efficient removal of lipids had to be implemented. The sample preparation procedure therefore needed optimizations considering comminution, extraction, clean-up, and also suitable consumables, in order to deliver an equally precise result than a conventional QuEChERS sample preparation.

Besides method development, also method application was a part of this work. Therefore, almost 400 bats of different species were analysed with the developed micro QuEChERS approach, and the results were eventually compiled into a dataset.

Secondly, apart from non-target animals, also non-agricultural plants can be affected by pesticides and pollutants and were therefore part of this work. Besides agriculture, forestry is another sector with a frequent pesticide use.[59] Additionally, depending on the polarity of an individual compound, it can undergo an environmental drift through air, ground water, or soil drainage.[54, 59] Thus, even with a certain distance to agricultural use, flora and fauna can be affected by agrochemicals and pollutants.

Jun.-Prof. Dr. Henrik Krehenwinkel from the Department of Biogeography of Trier University, Germany, is investigating insect biodiversity, supported by the German Environment Specimen Bank (Umweltprobenbank) of the Umweltbundesamt (UBA). With the help of DNA analysis, his group wants to find out more about trends in insect populations, especially leaf-associated arthropod communities. Particularly in the light of decreasing species diversity, it is crucial to find out more about what causes the insect decline. To this end, method development was conducted for the multiresidue analysis of beech leaves and spruce needles, which represent the habitat of many insects. For the sample preparation of the leaf and needle samples, several obstacles hat to be overcome. Especially, the intense coloration of plant parts with chlorophyll is challenging for sample preparation, as the pigment has to be removed from the extract to avoid system contamination as well as interferences on the analyte transitions.

All these challenges were overcome in the respective QuEChERS-based sample preparations, and the results are presented in the following Chapters.

3. Development of a miniaturized QuEChERS approach for limited sample sizes

Schanzer, S.; Kröner, E.; Wibbelt, G.; Koch, M.; Kiefer, A.; Bracher, F.; Müller, C., Miniaturized multiresidue method for the analysis of pesticides and persistent organic pollutants in non-target wildlife animal liver tissues using GC-MS/MS. *Chemosphere* **2021,** *279*, 130434.

3.1. Topic

The monitoring of pesticide residues is highly important, not only in food analysis, but also for environmental matters and health aspects. Due to the frequent use of plant protection products in agriculture, it is probable for non-target flora and fauna to suffer from a pesticide contamination, too.[59] Therefore, the QuEChERS approach is a suitable sample preparation method not only for foodstuffs, but also to investigate the pesticide load of non-target animals. Dr. Egbert Kröner is a veterinarian at the wildlife animal rehabilitation center (Wildvogel-Pflegestation Kirchwald e.V.) in Kirchwald, Germany. Annually, the center takes care of up to 3,000 animals in morbid conditions, predominantly birds and hedgehogs. As the decline in bird populations has been associated with the extensive use of arable land for years now,[86] pesticide analysis came into focus of the group around Dr. Kröner. Unfortunately, apart from unintentional and indirect bird poisoning through rodenticides, even deliberate poisoning incidents, such as with the banned insecticide carbofuran, have occurred in recent years.[87, 88] Apart from birds, also hedgehogs can be prone to unintentional pesticide exposure. They often live near residential areas and may be exposed to pesticides in home gardens, for example. Furthermore, they have been exposed to rodenticides in the past.[45] Hence, the risk of contamination with other pesticides and pollutants is obvious.

The conservation status of different bat species in Germany and the contribution to species conservation is the central issue of Dr. Andreas Kiefer, Martin Koch and Prof. Dr. Michael Veith from the Chair of Biogeography at Trier University, Germany. Therefore, they are developing novel monitoring methods in order to track bats' movements and roosting behaviours. Investigating the pesticide exposure of bats came into their focus for different reasons. On the one hand, by now, it is well known that bats often are highly exposed to different classes of pesticides, leading to population declines and vulnerability to certain diseases such as the white nose syndrome.[89, 90] On the other hand, the neonicotinoid insecticide imidacloprid was proven to affect the spatial memory of echolocation bats, which can also lead to a change in their roosting behaviour.[91] Hence, the ability to detect multiple residues of pesticides and pollutants in the protected bat species is vital for further investigation.

For the multiresidue analysis of animal liver samples, the QuEChERS approach was the sample preparation method of choice as it allows a fast and simultaneous analysis of a large number of compounds at once.[66] This method works with 5-10 g of sample material, which was sufficient for the analysis of hedgehog liver samples. However, some bats only weigh a few grams in total, so the liver weight was too low for a conventional QuEChERS sample preparation. Thus, it was necessary to find a way to downscale the sample preparation method. Usually, sample size does not bear a problem during a QuEChERS sample preparation since fruits, vegetables, or animal products (*e.g.* apples, spinach or beef) are available in sufficient amounts. Even if one fruit or cereal is too small for an individual analysis, it is possible to pool them, for example grapes from a vineyard or oats. For food analysis, pooling is unproblematic, or even an advantage, because picking fruits or cereals from several plants is more representative for a whole cultivation area. However, this is not applicable for individual wildlife animals as they can move freely and have different habitats or hunting grounds. Apart from the direct contact to pesticides in their individual habitats, insectivorous animals can have a pesticide intake through their prey, and the insects also can be loaded differently with those compounds. Thus, it is inevitable to perform an individual analysis for each respective animal. **Fig. 15** shows the graphical abstract of the article, which gives a brief overview of the pesticide residue analysis of bat and hedgehog samples.[92]

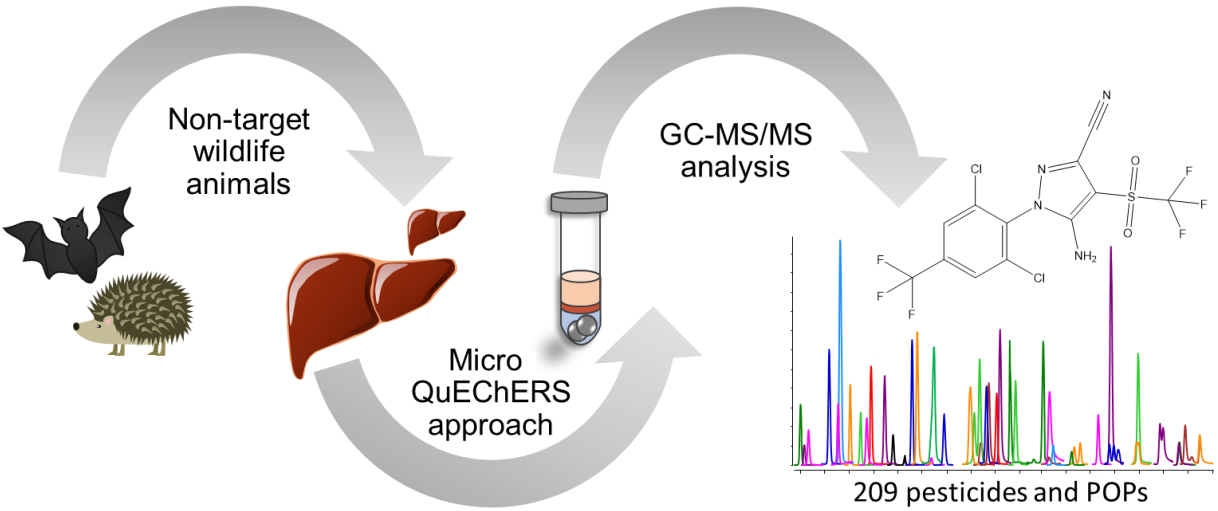


Figure 15 Graphical abstract of the article. Animal livers were processed with a micro QuEChERS approach and analysed for 209 different pesticides and POPs with GC-MS/MS.[92]

The QuEChERS-based sample preparation was first optimized using 5 g liver samples. For the optimization, parameters like solvent acidification, salt combinations for the SALLE, and sorbents for the dSPE step were considered. Particularly, the high fat content in liver required special attention during method development. The optimized sample preparation method was validated according to SANTE/12682/2019,[93] regarding LOQs, linearity, recoveries and matrix

effects. Afterwards, the fully validated method was downscaled in order to analyse 100 mg liver samples. Single working steps were adapted to the limited sample size, and the miniaturized method was again fully validated, regarding the same criteria. For proof of concept, hedgehog and bat livers were analysed with the miniaturized QuEChERS sample preparation method.

All samples were analysed with a GC-MS/MS system. The tandem mass spectrometer was operated in dynamic multiple reaction monitoring (dMRM) mode, which allows to blank out signals of matrix components that remain in the final extract after sample clean-up. Thus, the sensitivity of detection is very high, with limits of quantification in the low µg kg^{-1} range. Hence, the combination of the novel micro QuEChERS approach with the GC-MS/MS system contributes to investigating the pesticide and pollutant exposure of small wildlife animals.

3.2. Personal contribution

My contribution to this publication was the previous research as well as the performing of preliminary tests. Development, optimization, and validation of the analytical methodologies were all part of my contribution. All experiments during method optimization and validation were performed by me. I furthermore optimized the GC-MS/MS method, obtained all data, and performed the formal analysis of the datasets. Finally, I wrote the original draft, including the visualization of the article, and incorporated the changes recommended by the co-authors.

Dr. Andreas Kiefer, Dr. Gudrun Wibbelt and Dr. Egbert Kröner were all involved in the acquisition of funding and provided resources (liver samples) for the project. Furthermore, they contributed to reviewing and editing the original draft.

Martin Koch was also involved in providing resources (liver samples) for this project and furthermore contributed to reviewing and editing the original draft.

Prof. Dr. Franz Bracher contributed to funding acquisition and providing resources. Additionally, he supervised the project and furthermore reviewed and edited the original draft.

Dr. Christoph Müller's contribution was the conceptualization as well as the supervision of the whole project. Furthermore, he aided with the methodology and formal analysis of the curated data. Finally, he reviewed and edited the original draft and additionally supported the visualization.

3.3. Article

The article is printed in its original wording as published in Chemosphere. The formatting may vary slightly compared to the journal article.

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Miniaturized multiresidue method for the analysis of pesticides and persistent organic pollutants in non-target wildlife animal liver tissues using GC-MS/MS

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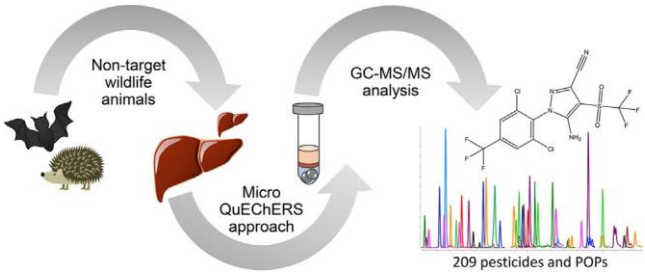
HIGHLIGHTS

- Multiresidue method for 100 mg of liver tissues of different wildlife animals.
- Micro QuEChERS method equally robust than the conventional QuEChERS analysis.
- *p,p'*-DDE and fipronil sulfone found in most wildlife animal samples.

GRAPHICAL ABSTRACT



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ABSTRACT

In order to gain a better insight into pesticide and pollutant exposure of small (non-target) wildlife animals, a QuEChERS sample preparation method was first developed for 5 g liver tissues (*e.g.* hedgehog samples) and then downscaled for the analysis of 100 mg liver tissues (*e.g.* bat samples). The optimized (micro) QuEChERS methods used 1% acetic acid in acetonitrile as organic solvent for liquid-liquid extraction (LLE) and salting out was performed with anhydrous magnesium sulfate and sodium acetate (4:1). After a freezing-out step, sample clean-up was carried out with anhydrous magnesium sulfate, PSA, C_{18}, and GCB (150:25:20:5). Overall, 209 pesticides and persistent organic pollutants (POPs) can be analysed within each sample with gas chromatography coupled to tandem mass spectrometry (GC-MS/MS). Both methods were validated with representative analytes according to the European Commission guideline SANTE/12682/2019. Limits of quantification were between 1 and 20 μg kg^{-1}, and the methods proved to be linear up to 400 μg kg^{-1}. Additionally, the analytes delivered satisfactory results regarding recovery and precision. As proof of concept, samples of six hedgehog livers were analysed with both methods to prove the accuracy of the micro QuEChERS method. Additionally, six livers of different bat species were analysed with the downscaled method. The newly developed micro QuEChERS method for multiresidue analysis requires only minute amounts of biomaterial and represents a sophisticated novel technique for determining the exposure of small wildlife animals to different contaminants.

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1 Introduction

As the global demand for comestibles is constantly growing, use of pesticides has increased continuously around the world over the past decades. During the last 50 years, the application of pesticides on corn fields in the USA has increased sevenfold (Fernandez-Cornejo et al., 2014). In Germany, a total of 30,000 tons of 285 different legally approved pesticides was applied in 2018 (German Federal Office of Consumer Protection and Food Safety, 2020). Apart from the approved pesticides, many persistent organic pollutants (POPs) like lindane or polychlorinated biphenyls (PCBs) are still present in the environment (Ashraf, 2017), even though their production and use has been prohibited since the Stockholm Convention in 2001 (UN Environment Programme, 2019). Furthermore, the approval status of pesticides is being reviewed on a regular basis, so there is a need for monitoring residues of pesticides that are not approved for application anymore. Overall, residues of several hundreds of compounds need to be analysed in order to get a comprehensive overview of the ecological burden. Such a large number of substances with different physicochemical properties is challenging for analysis as standard sample preparation methods often cover only a part of the analytes of interest, are time-consuming, and use hazardous solvents (Holden and Marsden, 1969; Snyder et al., 1993; Tadeo et al., 1996). In 2003, a major breakthrough in multiresidue analysis was achieved with the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) approach, allowing the simultaneous determination of many pesticides of different polarities (Anastassiades et al., 2003a). Since then, this sample preparation method has been modified and applied on different analytes and matrices like veterinary drugs in animal tissues or pesticides in human milk (Stubbings and Bigwood, 2009; Du et al., 2017). The broad applicability of this method also encouraged method development for analysis of human matrices like whole blood or urine (Plößl et al., 2006; Correia-Sá et al., 2018). However, the main focus was mostly on the pesticide analysis of fruits, vegetables or animal tissues meant for human consumption. Many pesticides are known to be hazardous to human health because they are carcinogens or endocrine disruptors (Smeds and Saukko, 2001; Sabarwal et al., 2018). Nonetheless, not only humans, but also non-target wildlife animals (both vertebrates and invertebrates) can be affected by pesticide application. The uptake may be direct *via* agricultural pesticide application or indirect through contaminated food (Stöckelhuber et al., 2017). For instance, neonicotinoids like clothianidin play an important role in bee colony collapses as they supposedly have adverse effects on the bees' immune system (Di Prisco et al., 2013).As insecticides can not only have an impact on pests, but also non-target or beneficial insects, Kiljanek et al. (2016) investigated bee poisonings with a modified QuEChERS multiresidue method in 2016. Also odonate nymphs have been the subject of a multiresidue study as they function as biomonitors for aquatic ecosystems (Jesús et al., 2018). However, not only insects can be affected, but also other species in the water ecosystem like fish are sensitive towards organophosphorus insecticides such as chlorpyrifos (Sunanda et al.,2016). In addition to insects and fish, blood samples of different bird species have been investigated recently for multiple residues (Rial-Berriel et al., 2020). In order to protect wildlife animals, it is crucial to learn more about possible interactions of pesticides and POPs with flora and fauna (Löbbert et al., 2021). In turn, this affords the availability of very sensitive multiresidue analyses for identification and quantification of the xenobiotics.

As representative wildlife species, the European hedgehog (*Erinaceus europaeus*) and three bat species native in Germany (*Myotis myotis, Pipistrellus nathusii* and *Plecotus auritus*) were chosen for pesticide and POP analysis. They all represent different ecotypes: the omnivorous hedgehogs are bound to the ground and are found in parks and gardens, while the insectivorous bats choose different roosts in attics or tree hollows and their spatial behaviour ranges from local, *e.g.* parks (*Plecotus auritus*), to regional, *e.g.* open terrain like meadows and open forests, but also human settlements (*Myotis myotis*), to long distance migration in landscapes rich in forests and water bodies (*Pipistrellus nathusii*). In hedgehogs, accumulation of anticoagulant rodenticides like difenacoum was already observed (Dowding et al., 2010). Nonetheless, being also an insectivore, *E. europaeus* might as well be contaminated with other pesticides through its invertebrate prey. Poisonings of bats with organochlorine insecticides were repeatedly reported through the years (Eidels et al., 2007; Buchweitz et al., 2018). Additionally, risks and adverse effects of currently used pesticides on bats have been investigated (Eidels et al., 2016; Hsiao et al., 2016; Mineau and Callaghan, 2018). As all bat species are considered endangered and therefore need to be protected, multiresidue analysis might give further information on their pesticide and POP exposure (Bayat et al., 2014; UNEP/EUROBATS, 2020).

Gas chromatography coupled to tandem mass spectrometry (GC-MS/MS) is a robust and sensitive method which is able to identify and quantify analytes in the ultra-trace range. Hence, GC-MS/MS is routinely used in pesticide and POP analysis (Payá et al., 2007; Shabeer et al., 2018). Additionally, in recent years, miniaturization is a popular trend in analytical chemistry as it requires less sample material, consumes less solvents and chemicals, and might save time and costs (Kristenson et al., 2001; Correia-Sá et al., 2018; Jesús et al., 2018; Rial-Berriel et al., 2020). The original QuEChERS method (Anastassiades et al., 2003a) uses 10 g of sample material. As European hedgehogs weigh approximately 700−1000 g, with a liver weight of about 5% of the body weight, our sample preparation worked with a standard scale QuEChERS method optimized for fatty matrix using 5 g of liver. However, as bats have a much lower body weight (3−40 g), only liver samples in the milligram range were available. In consequence of this reduced sample size, the powerful GC-MS/MS system was combined with a newly developed micro QuEChERS approach.

First, a standard scale sample preparation method for medium-sized wildlife animals (*e.g.* badgers, hedgehogs, foxes) was optimized and validated according to SANTE/12682/2019 (European Commission, 2019). The next step was to downscale the method by a factor of 50. This micro scale method (micro QuEChERS approach) for small wildlife species such as bats, songbirds or lizards was also validated and compared to the standard scale method. Both sample preparation methods delivered virtually equivalent results. Hence, the developed (micro) QuEChERS approaches were successfully applied on 5 g and 100 mg hedgehog liver tissues for proof of concept and additionally on 100 mg bat liver tissues.

For the selection of analytes, legally allowed pesticides throughout Europe as well as pesticide spreading plans of local agricultural areas in Germany were taken into consideration. However, also POPs were of a high interest as they emphasize the deep impact that persistent substances can have on living nature. Despite being forbidden for decades, many of the substances are still present up to today.

2 Materials and methods

2.1 Chemicals

All analytical and internal standards (≥98.9%) were obtained from HPC Standards (Cunnersdorf, Germany). Triphenyl phosphate,

chlorpyrifos-D_{10} and azoxystrobin-D_4 were used as internal standards.
Acetonitrile (MeCN) in HPLC grade and acetic acid (HOAc, ≥99%) were purchased from VWR (Darmstadt, Germany). 3-Ethoxy-1,2-propanediol (98%), L-gulonic acid γ-lactone (95%) and D-sorbitol (99%) were purchased from Sigma-Aldrich (Darmstadt, Germany). Shikimic acid (≥98%) was purchased from Carl Roth (Karlsruhe, Germany). SPE bulk sorbents of primary secondary amine (PSA), C_{18} (octadecylsilane, ODS), carbon (graphitized carbon black, GCB) and Enhanced Matrix Removal (EMR)-Lipid were purchased from Agilent Technologies (Santa Clara, CA, USA). Anhydrous magnesium sulfate ($MgSO_4$, ≥98%) and sodium acetate (NaOAc, ≥99%) were obtained from Grüssing (Filsum, Germany). Sodium citrate dihydrate (Na_3citrate·$2H_2O$, ≥99%) was purchased from Th. Geyer (Renningen, Germany). Sodium hydrogencitrate sesquihydrate (Na_2Hcitrate·$1.5H_2O$, ≥99%) was purchased from Sigma Aldrich (Darmstadt, Germany). Sodium chloride (NaCl, p.a.) was obtained from Bernd Kraft (Duisburg, Germany).

2.2 Reagents

Analyte stock solutions and internal standard (ISTD) stock solutions (each 1 mg mL^{-1}) were prepared with 0.1% HOAc in MeCN (*v/v*). Working standard solutions were prepared with 10 µg mL^{-1} and diluted as needed. ISTD stock solutions were diluted to achieve a working concentration of 10 µg mL^{-1} for the 5 g samples and 200 ng mL^{-1} for the 100 mg samples, respectively. All stock solutions and working solutions were stored at -20 °C and were tempered for 1 h and shaken before use. Ultrapure water was freshly generated by an in-house water purification system.
The analytes were extracted with 1% HOAc in MeCN (*v/v*). The salt mixture for phase separation was $MgSO_4$ and NaOAc (4:1). The mixture for the dSPE step was $MgSO_4$, PSA, C_{18} and GCB (150:25:20:5). The analyte protectants (AP) mixture was prepared with 3-ethoxy-1,2-propanediol (200 mg mL^{-1}), L-gulonic acid γ-lactone (10 mg mL^{-1}), shikimic acid (5 mg mL^{-1}) and D-sorbitol (5 mg mL^{-1}) in a mixture of MeCN and water (6:4, (*v/v*)). The AP mixture was stored at 8 °C and was tempered for 15 min and shaken before use.

2.3 Laboratory equipment

Sample comminution for 5 g samples was performed with an IKA Ultra-Turrax® Tube Drive (Staufen, Germany) combined with IKA BMT-20-S tubes. The 100 mg samples were homogenized with a Vortex Genie 2 from Scientific Industries (Bohemia, NY, USA) equipped with a bead tube holder from Macherey-Nagel (Düren, Germany). The Vortex Genie 2 was also used for all other homogenization steps. Centrifugation steps were performed with a Heraeus Megafuge 1.0 R (Hanau, Germany) for 50 mL tubes and a 5415 D centrifuge from Eppendorf (Hamburg, Germany) for all microcentrifuge tubes.

2.4 Analytical instrument

All samples were analysed with an Agilent 7890B gas chromatograph (Santa Clara, CA, USA) coupled with an Agilent 7010B triple quadrupole mass spectrometer with a high efficiency source (HES). Sampling was performed with a PAL3 RSI autosampler from CTC Analytics (Zwingen, Switzerland). The GC system was supplied with an Agilent multimode inlet (MMI) and two Agilent HP-5ms ultra inert columns (each 15 m × 0.25 mm × 0.25 µm). The two columns were connected by a backflush capillary flow technology device (CFT). The carrier gas was helium 5.0 from Air Liquide (Düsseldorf, Germany). The MMI was used in solvent vent mode with an injection volume of 1 µL. The initial temperature was 60 °C with 0.2 min hold time and a vent flow of 100 mL min^{-1}. The temperature ramp was 900 °C min^{-1} until reaching 280 °C. The MMI stayed at 280 °C throughout the whole GC run and was heated to 310 °C during post run.
The oven temperature was initially set at 60 °C with a hold time of 1 min and then ramped with a heat rate of 40 °C min^{-1} to 170 °C, followed by 10 °C min^{-1} up to 310 °C (3 min hold time). Overall GC run time was 20.75 min, post run time with backflush was set at 5 min. Helium flow was set at 1.1 mL min^{-1} on the first and 1.3 mL min^{-1} on the second column during the GC run. During backflush, column flow was -4.0 mL min^{-1} on the first and 4.4 mL min^{-1} on the second column. The 7010B triple quadrupole mass spectrometer was operated in electron ionization mode at 70 eV and all samples were analysed in dynamic multiple reaction monitoring (dMRM) mode. Therefore, the source temperature was set at 230 °C and the quadrupole temperatures at 150 °C. Argon 4.5 from Air Liquide (Düsseldorf, Germany) was used as collision gas. Transfer line temperature was permanently set at 280 °C.
Instrument control was performed with Agilent MassHunter Data Acquisition 10. The Agilent P & EP Enhanced MRM Database A.04.02 was used to generate the dMRM method (Supplementary Table 1). Data analysis was accomplished with Agilent MassHunter QQQ Quantitative Analysis 7.

2.5 Samples

Fresh bovine and chicken livers from local grocery stores were used as blank matrix for sample preparation and method validation experiments. All blank matrices were tested for the analytes of interest before being used for further experiments. None of the pesticides were present in the blank sample matrix.
Livers from *E. europaeus*, *M. myotis, P. auritus* and *P. nathusii* were dissected from deceased animals by veterinarians at the wildlife rehabilitation center in Kirchwald, Germany. The animals were either found dead in natural surroundings or were in a morbid condition and died at the rehabilitation center. All samples were stored at -20 °C before analysis.

2.6 Sample preparation

2.6.1 Standard scale method for medium-sized wildlife animals (5 g liver samples)

For sample preparation, 5.00 (±0.05) g of liver were weighed into an IKA tube containing ten steel beads. The sample was spiked with 10 µL internal standard working solution and was allowed to stand for 5 min. Then 5000 µL of water were added, and the sample was homogenized for 4 min at 6000 rpm at room temperature (RT).
This comminution technique can prevent cross-contamination of samples. The homogenate was transferred to a 50 mL centrifuge tube and 9990 µL of 1% HOAc in MeCN (*v/v*) were added, providing a dilution factor of 2. The sample was shaken vigorously for 1 min. Then 5.00 (±0.05) g of $MgSO_4$:NaOAc (4:1) were added. The tube was closed and shaken immediately to avoid formation of salt agglomerates. Afterwards it was vortexed for 30 s. For phase separation, the sample was centrifuged for 5 min at 3300 *g* at RT (Supplementary Figure 1). The sample was stored at -20 °C overnight in order to freeze and separate the co-extracted fatty components. An aliquot of 750 µL of the upper organic layer was transferred to a 2.0 mL microcentrifuge tube containing 300.0 (±3.0) mg of dSPE mixture ($MgSO_4$, PSA, C_{18} and GCB (150:25:20:5)). The tube was vortexed for 20 s and then centrifuged for 5 min at 12,000 *g* at RT. At last, 500 µL of the supernatant were transferred to an autosampler vial and 15 µL of AP mixture were added before GC-MS/MS analysis.

2.6.2 *Micro scale method (micro QuEChERS approach) for small wildlife animals (100 mg samples)*

Concerning the micro QuEChERS approach, all amounts of reagents were downscaled by a factor of 50. For the homogenization step, 100.0 (±1.0) mg of liver were weighed into a 2.0 mL screw-cap tube containing two steel beads. Then 100 µL of water, 190 µL of 1% HOAc in MeCN (*v/v*) and 10 µL internal standard working solution were added. The tube was vortexed for 15 min at RT. Afterwards, 100.0 (±1.0) mg of $MgSO_4$:NaOAc (4:1) were added and the tube was shaken immediately and then vortexed for 15 min at RT. The tube was centrifuged for 5 min at 12,000 *g* at RT (Supplementary Figure 2) and then stored at -20 °C for 2 h in order to freeze and separate the co-extracted fatty components. An aliquot of 100 µL of the upper organic layer was transferred to a 0.5 mL microcentrifuge tube containing 40.0 (±0.4) mg of the dSPE mixture. The tube was vortexed for 20 s and then centrifuged for 5 min at 12,000 *g* at RT. At last, 50 µL of the supernatant were transferred to an autosampler vial with a 250 µL insert and 1.5 µL AP mix were added prior to GC-MS/MS analysis.

3 Results and discussion

3.1 *Method optimization*

The whole method optimization was performed with the standard scale method using 5 g of bovine and chicken blank liver because no analyte-free sample matrix from wildlife animals was available. The optimized method was then adapted to the micro scale method using only 100 mg of liver. All analytes used for optimization experiments are listed in Table 1.

3.1.1 *Choice of extraction solvent and salts*

In order to achieve the best possible extraction, the three most common QuEChERS approaches which are mentioned below, were investigated. All experiments were performed with 5 g of bovine liver spiked with 43 analytes (100 µg kg^{-1}) from a broad range of chemotypes and 10 µL of the internal standard working solution. The spiked samples were allowed to stand for 5 min before adding 5000 µL of ultrapure water. For the salt-assisted liquid-liquid extraction (SALLE), three solvent and salt combinations were tested (n=6): *(1)* the original method using acetonitrile and $MgSO_4$:NaCl (4:1) (Anastassiades et al., 2003a), *(2)* the AOAC 2007.01 method using 1.0% acetic acid in acetonitrile and $MgSO_4$:NaOAc (4:1) (Lehotay, 2007) and *(3)* the EN 15662 method using 0.5% formic acid in acetonitrile and a buffered mixture of $MgSO_4$:NaCl:Na_3citrate·$2H_2O$:Na_2Hcitrate·$1.5H_2O$ (8:2:2:1) (European Comittee for Standardization, 2018). A modification was made for the addition of formic acid in *(3)*; the acid was added during the extraction step instead of acidifying the final extract. The acidification is necessary to protect base-labile analytes like thiabendazole or imazalil (Lehotay et al., 2005a). The organic phases were then all cleaned up with a mixture of 150 mg $MgSO_4$ and 25 mg PSA and analysed with GC-MS/MS.

The quality of extraction was evaluated by the summed up peak areas of all spiked standards. Fig. 1 shows the relative peak area sums of all extraction procedures. The AOAC and EN extraction methods both showed significantly better results than the original method, which was proven with two-tailed *t*-tests. This indicates that the buffered extraction improves extractability and might stabilize pH-sensitive analytes. The AOAC extraction method did not prove to be significantly different from the EN method, which was also verified by a two-tailed *t*-test. For further experiments, the AOAC 2007.01 extraction was the method of choice. It requires less chemicals than the EN 15662 method and is known to deliver satisfactory results in fatty matrices (Lehotay et al., 2005b).

3.1.2 *Choice of dSPE sorbents for extract clean-up*

Removing co-extracted matrix components is an important step during the QuEChERS sample preparation in order to minimize matrix effects. Co-eluting matrix might overlay the analytes of interest, even after selecting specific MRM transitions for each analyte. Thus, the more efficient the clean-up step, the more sensitive and selective the detection method becomes. Additionally, less effort is needed for GC-MS/MS maintenance when injecting cleaner extracts. In order to find the optimal clean-up for all analytes, ten mixtures of customized dSPE sorbents and one commercially available mixture (Agilent EMR Lipid) were tested. The compositions of the sorbent mixtures are summarized in Table 2. EMR Lipid was tested as well because it is known to remove lipids from fatty matrices without having negative impacts on the analytes of interest (Zhao and Lucas, 2015). Blank matrices of bovine and chicken livers were spiked with three concentrations (10, 50 and 100 µg kg^{-1}) and extracted with the developed SALLE method.

After freezing the sample overnight, an aliquot of 1000 µL extract was used for the dSPE step. Each mixture was tested with both liver tissues in triplicates. Preferably, the cleaned up extracts delivered high peak areas and low relative standard deviations (RSD). Fig. 2 shows the efficient removal of co-extracted cholesterol from the prepared liver tissue extract after using other dSPE sorbents than PSA. While PSA, a weak anion exchanger, cannot remove cholesterol, both C_{18} as a non-polar sorbent and GCB as an unselective adsorbent are able to remove the co-extractive. The overall best results (high peak area sum, low RSD) were obtained with dSPE mixtures 5 and 10 as well as the EMR Lipid clean-up sorbent.

In a second step, those three dSPE mixtures were tested in order to determine the optimum amount of dSPE sorbent per mL extract. Again, three concentrations (10, 50 and 100 µg kg^{1}) were tested both in chicken and bovine livers. The dSPE step was performed with 1000 µL of extract and *(1)* 200 mg dSPE mixture, *(2)* 300 mg dSPE mixture and *(3)* 400 mg dSPE mixture. The overall optimal results, which were a compromise between highest possible peak areas and lowest possible relative standard deviations, were achieved with 400 mg of dSPE mixture 10 per mL of extract.

3.1.3 *Matrix effects*

Matrix effects (MEs) are a common effect in GC and HPLC applications (Rutkowska et al., 2020). These effects exacerbate quantification of analytes as the detector response might be very different in solvent and matrix extracts. MEs were examined by injecting solutions of 26 analytes (Table 1, 25 ng mL^{-1}, corresponding to 50 µg kg^{-1}) in MeCN and in liver matrix extract ten times, respectively. The average area of each analyte was used to calculate the matrix effects. A positive value represents a matrix enhancement effect, a negative value shows a signal suppression in matrix extracts. The following equation was used to calculate the matrix effect for each analyte:

$$ME\ [\%] = \left(\frac{area_{matrix}}{area_{solvent}} - 1\right) \times 100\%$$

According to the SANTE guideline, a matrix effect higher than 20% must be addressed in the calibration (European Commission, 2019). All of the investigated analytes showed matrix effects, of which all were enhancement effects, 85% even showed very high matrix effects (>70%). Thus, matrix-matched standards were used for all experiments and quality control with regard to comparability.

3.1.4 *Usage of analyte protectants*

Analyte protectants (APs) are important in GC-MS analysis. Those highly polar compounds, mostly sugars or sugar derivatives,

Table 1
Summary of all analytes used for method optimization and validation. Results shown for both developed methods. Standard scale method used 5 g of liver; micro scale method used 100 mg. n.d.: not determined,[1] used for solvent optimization,[2] used for dSPE optimization,[3] used for determination of matrix effects,[4] used for analyte protectants optimization, A: Azoxystrobin-D_4, C: Chlorpyrifos-D_{10}, T: Triphenyl phosphate. MRL values show the limits for livers meant for human consumption. *: LOQ > MRL.

Pesticides and properties			GC-MS/MS parameters			Validation results for standard scale method			Validation results for micro scale method		
Name	Pesticide classification	MRL [µg kg^{-1}]	RT [min]	Internal standard	Quantifier transition	Calibration range [µg kg^{-1}]	R^2	Recovery [%]	Calibration range [µg kg^{-1}]	R^2	Recovery [%]
Ametoctradin[1]	Fungicide	30	15.20	A	246.0 → 188.2	4-400	0.998	74.9	20-400	0.989	60.0
Amisulbrom[1]	Fungicide	10	16.14	A	225.9 → 147.0	1-400	0.994	85.1	4-400	0.983	65.0
Azoxystrobin[1-4]	Fungicide	70	18.30	A	344.1 → 171.9	2-400	0.999	114.2	4-400	0.995	108.6
Bifenazate	Acaricide	20	13.87	C	184.1 → 77.0	n.d.	n.d.	n.d.	20-400	0.991	82.1
Bifenthrin[1]	Insecticide	200	13.83	C	181.0 → 115.1	4-400	0.999	95.3	4-200	0.992	88.8
Boscalid[1-4]	Fungicide	50	16.50	A	140.0 → 76.0	1-400	0.999	112.1	1-400	0.993	102.7
Carbetamide*	Herbicide	10	9.84	C	119.1 → 64.1	n.d.	n.d.	n.d.	20-400	0.984	109.7
cis-Chlordane[1-4]	Insecticide	50	11.20	C	372.8 → 265.8	1-400	0.999	91.7	1-400	0.998	91.1
trans-Chlordane[2-4]	Insecticide	50	10.94	C	372.8 → 265.8	1-400	0.999	100.9	1-400	0.997	95.8
Chlorpyrifos[1-4]	Insecticide	10	9.86	C	313.8 → 257.8	1-400	0.999	95.3	1-400	0.998	90.2
Chlorpyrifos-methyl[3,4]	Insectide	10	8.95	C	285.9 → 93.0	n.d.	n.d.	n.d.	1-400	0.995	93.1
Cyflufenamid[1-4]	Fungicide	20	11.88	C	118.1 → 90.0	4-400	0.998	111.1	4-400	0.995	95.9
Cyhalothrin (*gamma* and *lambda*)[1-4]	Insecticide	50	14.60 14.79	T	208.0 → 181.0	2-400	0.999	101.9	4-400	0.998	104.5
Cypermethrin (3 isomers)[1-4]	Insecticide	200	16.39 16.48 16.57	T	162.9 → 127.0	2-400	0.999	99.9	4-400	0.995	100.5
p,p'-DDE[1]	Metabolite	1000	11.52	C	246.1 → 176.2	1-400	0.999	88.8	1-400	0.998	85.9
p,p'-DDT[2-4]	Insecticide	1000	12.91	T	235.0 → 165.2	n.d.	n.d.	n.d.	2-400	0.993	97.1
Deltamethrin[1-4]	Insecticide	30	18.02	A	252.9 → 93.1	2-400	0.998	104.4	4-400	0.993	106.5
Desmedipham	Herbicide	50	7.67	C	181.0 → 109.0	n.d.	n.d.	n.d.	4-400	0.991	92.3
Dieldrin[1-4]	Insecticide	200	11.62	C	277.0 → 241.0	4-400	0.999	101.8	2-400	0.997	96.8
Difenoconazole (2 isomers)[1-4]	Fungicide	200	17.72 17.78	A	322.8 → 264.8	4-400	0.998	97.9	4-400	0.995	99.8
Diflubenzuron	Insecticide	10	5.01	C	141.0 → 63.0	n.d.	n.d.	n.d.	4-400	0.995	85.6
Dimethomorph (2 isomers)[1-4]	Fungicide	10	18.35 18.66	A	300.9 → 165.0	2-400	0.999	109.7	4-400	0.993	104.8
Epoxiconazole[1]	Fungicide	200	13.52	C	192.0 → 138.1	1-400	0.999	105.5	1-400	0.998	98.0
Fenazaquin	Insecticide	10	14.05	C	160.0 → 145.2	n.d.	n.d.	n.d.	2-400	0.998	61.8
Fenhexamid[1-4]	Fungicide	50	12.97	T	177.1 → 113.0	2-400	0.999	99.2	2-400	0.997	89.8
Fenpropidin	Fungicide	200	9.26	C	273.0 → 98.0	n.d.	n.d.	n.d.	4-400	0.998	66.8
Fenpropimorph[1]	Fungicide	700	9.81	C	128.1 → 70.1	2-400	0.999	86.6	4-400	0.991	84.3
Fenvalerate (2 isomers)[1-4]	Insecticide	20	17.31 17.50	C	167.0 → 125.1	2-400	0.996	103.5	2-400	0.996	98.3
Fipronil sulfone[1]	Metabolite	5	11.71	C	382.8 → 254.9	1-400	0.999	110.8	2-400	0.998	103.9
Fluazifop-p-butyl	Herbicide	30	11.80	T	281.9 → 238.0	n.d.	n.d.	n.d.	1-400	0.998	82.2
Fludioxonil[1]	Fungicide	50	11.51	C	248.0 → 127.1	1-400	0.999	104.6	1-400	0.998	91.1
Fluopyram[1]	Fungicide	8000	10.57	C	222.9 → 196.0	1-400	0.999	107.6	1-400	0.998	103.8
Flupyradifurone[1,*]	Insecticide	10	14.87	A	126.0 → 73.0	20-400	0.978	116.8	20-400	0.973	116.3
tau-Fluvalinate (2 isomers)[1]	Insecticide	10	17.48 17.52	T	250.0 → 200.1	4-400	0.998	99.0	4-400	0.996	101.6
gamma-HCH (Lindane)[1-4]	Insecticide	10	8.08	C	216.9 → 181.0	1-400	0.999	106.6	1-400	0.996	100.1
Imidacloprid[1]	Insecticide	300	11.31	C	126.0 → 73.0	20-400	0.990	125.3	20-400	0.972	96.4
Lenacil[1]	Herbicide	100	12.95	T	153.1 → 82.1	2-400	0.999	100.3	2-400	0.993	97.3
Metazachlor[1]	Herbicide	200	10.45	T	209.0 → 132.2	1-400	0.999	100.0	1-400	0.996	109.2
Metrafenone[1-4]	Fungicide	10	15.24	C	394.8 → 364.8	4-400	0.999	100.3	4-400	0.998	91.6
Myclobutanil[1-4]	Fungicide	10	11.68	C	179.0 → 125.1	2-400	0.999	106.8	2-400	0.999	106.2
Oxychlordane	Metabolite	50	10.53	C	184.8 → 121.0	n.d.	n.d.	n.d.	2-400	0.997	93.9
Pentachloro-nitrobenzene[1]	Fungicide, Nematicide	10	8.20	C	141.9 → 106.9	1-400	0.999	82.3	1-400	0.998	73.1
Permethrin (*cis* and *trans*)[1]	Insecticide	50	15.51 15.63	T	162.9 → 127.0	4-400	0.999	91.5	4-400	0.997	94.3
Picolinafen[1-4]	Herbicide	20	13.87	C	376.0 → 238.1	1-400	0.999	70.7	2-400	0.999	60.4
Pirimicarb[1]	Insecticide	50	8.73	T	238.0 → 166.2	4-400	0.996	101.3	1-400	0.995	100.3
Propiconazole (2 isomers)[1-4]	Fungicide	500	12.89 13.00	C	172.9 → 74.0	2-400	0.999	103.2	2-400	0.998	99.6
Pyrimethanil[1-4]	Fungicide	100	8.24	C	198.0 → 118.1	1-400	0.999	44.2	1-400	0.997	34.0
Quinoxyfen[1]	Fungicide	200	12.85	C	237.0 → 208.0	1-400	0.999	65.8	1-400	0.998	49.5
Spirodiclofen[1]	Insecticide	50	15.56	C	312.1 → 108.9	2-400	0.999	98.1	2-400	0.997	98.1
Tebuconazole[1-4]	Fungicide	200	13.22	T	250.0 → 125.0	1-400	0.996	93.5	2-400	0.997	95.8
Terbuthylazine	Herbicide, Microbiocide	50	7.98	C	172.9 → 138.1	n.d.	n.d.	n.d.	1-400	0.995	103.4
Terbuthylazine-desethyl	Herbicide, Microbiocide	50	7.30	C	145.1 → 110.1	n.d.	n.d.	n.d.	1-400	0.997	109.1
Tetraconazole[1-4]	Fungicide	1000	9.99	C	336.0 → 203.8	1-400	0.999	110.7	2-400	0.998	106.1
Thiabendazole[1,3,4]	Fungicide	150	10.73	C	201.9 → 175.0	20-400	0.997	48.7	n.d.	n.d.	n.d.
Tolclofos-methyl[1]	Fungicide	10	9.14	C	267.0 → 252.0	2-400	0.999	99.8	2-400	0.999	99.5
Zoxamide[1-4]	Fungicide	10	13.47	T	257.9 → 187.1	1-400	0.995	101.5	4-400	0.995	105.1

are able to decrease interaction of analytes with active sites in the GC inlet system and thus minimize degradation and peak tailing (Anastassiades et al., 2003b; EURL-SRM, 2013; European Commission, 2019). Active sites are often located in the liner or the GC column and analyte degradation becomes worse over time as non-volatile matrix components settle in the injector parts. In order to evaluate the benefit of APs, a mixture of 26 representative analytes (Table 1, 25 ng mL^{-1}, corresponding to 50 µg kg^{-1}) was injected 50 times in a row in four different settings: *(1)* MeCN without AP mixture, *(2)* MeCN with AP mixture, *(3)* liver extract without AP mixture, *(4)* liver extract with AP mixture (composition of AP mixture see 2.2). For each setting, a new liner was used in order to have a clean system at the beginning of the experiment. The relative increase or decrease of the peak area was investigated for each analyte after 50 injections. Every five injections, the average peak area of the respective analyte was calculated. The first average area was set to 100% and the subsequent averages were calculated in relation to the first one. The quality of each setting was determined by the number of analytes that were within a range of 80-120% of their initial peak area. Regarding *(4)* matrix with APs, only one analyte (*trans*-chlordane, 134%) did not meet the above-mentioned criteria after 50 injections, whereas in *(1)* solvent without APs, all peak areas were between 0 and 60% of the initial area. Both in *(2)* solvent with APs and *(3)* matrix without APs, approximately 50% of all analytes met the criteria. Thus, the best results were obtained when using liver extracts with APs (Supplementary Figure 3). As a consequence, the AP mixture was added to all matrix extracts prior to analysis in order to minimize deviations.

3.2 Method validation

Method validation was performed for both methods according to SANTE/12682/2019 (European Commission, 2019). Either 44 analytes (5 g samples) or 55 analytes (100 mg samples) of various pesticide chemotypes (*e.g.* pyrethroids, azoles, organochlorines) were chosen to perform the method validation, respectively. All of the following validation data are summarized in Table 1.

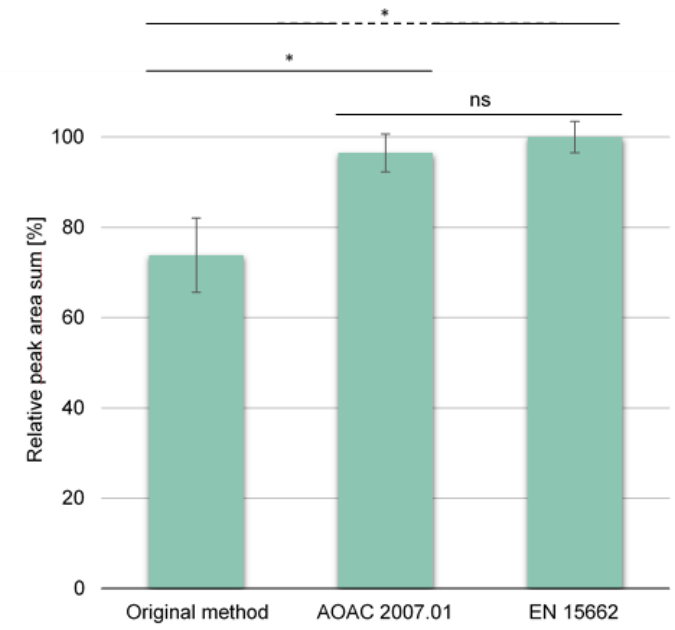

Fig. 1 Average extractability of 43 compounds (Table 1, 100 mg kg^{-1}) with different QuEChERS-based methods. Error bars show standard deviation (n = 6). * significant (t- test, P < 0.05), ns not significant (t-test, P > 0.05)

3.2.1 Linearity and limit of quantification

For linearity evaluation, a minimum of five levels is required. Matrix-matched calibration standards were prepared by spiking blank bovine liver extracts with a freshly prepared standard mixture containing the respective analytes (Table 1). Eight levels (0.5, 1, 2, 10, 50, 100, 150 and 200 ng mL^{-1} corresponding to 1, 2, 4, 20, 100, 200, 300 and 400 µg kg^{-1}) were analysed in order to confirm linearity and to determine the limit of quantification (LOQ).The calibration standards were injected six times each. In total, six levels were picked for the calibration curves. For each analyte, the LOQ concentration was defined as the lowest measured concentration that provided an overall bias and RSD lower than 20%, respectively. With regard to comparability, the aim for the respective LOQ concentration was to remain lower than the maximum residue limit (MRL) of each pesticide in liver tissue (European Commission, 2016). However, as MRLs are only available for products meant for human consumption, analytes whose LOQ exceeded the MRL were not excluded from the method as their presence in wildlife animal tissues is still of a high interest. LOQ concentrations were between 1 and 20 µg kg^{-1}. In case of an LOQ of 20 µg kg^{-1}, five instead of six levels were used for the linearity check.

Regarding the 5 g method, only for the insecticide flupyradifurone, the LOQ (20 µg kg^{-1}) exceeded its MRL (10 µg kg^{-1}). All correlation coefficients (R^2) of the calibration curves were ≥0.990, except for flupyradifurone, which had an R^2 of 0.978. For the 100 mg method, two LOQs (carbetamide and flupyradifurone, both20 µg kg^{-1}) exceeded their corresponding MRLs (both 10 µg kg^{-1}). Correlation coefficients (R^2) were ≥ 0.990 for all analytes, except the fungicides ametoctradin (R^2 0.989) and amisulbrom (R^2 0.983), the herbicide carbetamide (R^2 0.984) and the insecticides flupyradifurone (R^2 0.973) and imidacloprid (R^2 0.972).

3.2.2 Recovery

For recovery evaluation, the guideline demands a minimum of five replicates at the LOQ and at least one other concentration. Blank bovine liver samples were spiked with the analytes of interest (Table 1) and internal standard working solution. The samples were then processed with the developed method. The respective LOQ concentration of each analyte, 20 µg kg^{-1} and 200 µg kg^{-1} were analysed (n = 6), covering a low, medium and high concentration. Matrix-matched solutions with the corresponding concentrations were analysed before and after the spiked samples. The spiked samples were then compared to the matrix-matched solutions and the average recovery was calculated for all concentrations.

For the standard scale method, three of 44 analytes deceeded the demanded range of 70-120% recovery (pyrimethanil (44.2%), quinoxyfen (65.8%) and thiabendazole (48.7%)) and one exceeded

Table 2
Composition of mixtures of customized dSPE sorbents for method optimization.

Mixture	$MgSO_4$ [mg]	PSA [mg]	GCB [mg]	C_{18} [mg]
1	150	50	-	-
2	150	-	50	-
3	150	-	-	50
4	150	25	25	-
5	150	25	-	25
6	150	-	25	25
7	150	25	12.5	12.5
8	150	12.5	25	12.5
9	150	12.5	12.5	25
10	150	25	5	20

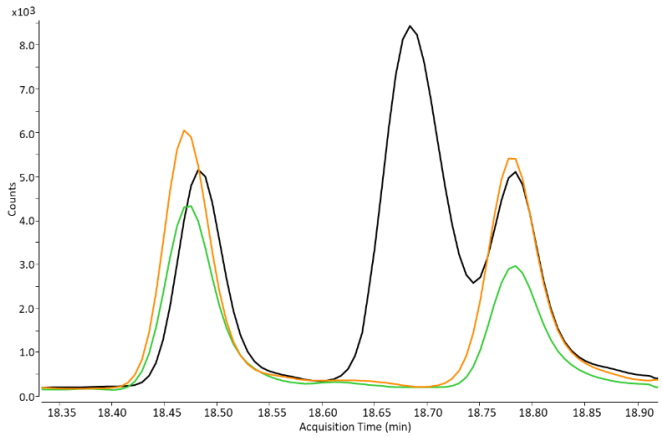

Fig. 2. The dMRM chromatograms show the quantifier transitions of dimethomorph isomers (18.48 and 18.78 min) which were interfered by cholesterol (18.68 min, identified by full scan MS spectrum). Cholesterol was removed after clean-up with different dSPE sorbents: black: 50 mg PSA, orange: 50 mg C_{18}, green: 50 mg GCB. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

this range (imidacloprid (125.3%)).

For the micro QuEChERS approach, seven of 55 analytes showed a recovery outside of the range of 70-120% (ametoctradin (60.0%), amisulbrom (65.0%), fenazaquin (61.8%), fenpropidin (66.8%), picolinafen (60.4%), pyrimethanil (34.0%) and quinoxyfen (49.5%)). Most likely, the poor recoveries (<70%) were caused by the use of GCB, as the decrease of peak areas could be observed for certain analytes when using GCB during the dSPE optimization experiments (data not shown). However, according to SANTE/12682/2019, a recovery of >30% is sufficient for method validation and the respective recovery can be used for calculation.

3.2.3. Method precision

For evaluation of the method precision, six samples were spiked with the representative analytes, respectively (Table 1), at concentrations representing the individual LOQ of each analyte, 20 µg kg^{-1} and 200 µg kg^{-1}, prior to extraction. According to the SANTE guideline, the RSD must be <20% (European Commission, 2019).

Regarding the standard scale method, for the low concentration, the average method precision was 2.2%, reaching from 0.7% (pirimicarb) to 11.4% (boscalid). For the middle concentration, the average method precision was 1.9%, reaching from 0.7% (quinoxyfen) to 7.3% (bifenthrin). For the high concentration, the average method precision was 0.9%, reaching from 0.2% (fenhexamid) to 3.9% (ametoctradin). Hence, the RSD remained notably below the permitted 20% limit.

Regarding the micro QuEChERS method, for the low concentration, the average method precision was 6.0%, reaching from 1.8% (fenpropimorph) to 17.3% (chlorpyrifos). For the middle concentration, the average method precision was 5.4%, reaching from 1.8% (dieldrin) to 19.6% (pyrimethanil). For the high concentration, the average method precision was 3.7%, reaching from 0.8% (tebuconazole) to 13.5% (pyrimethanil). Overall, all values were within the required range.

All RSD data can be found in Supplementary Table 2.

3.3 Comparison of the method validation results for both sample sizes

Regarding the LOQ concentrations, the two investigated methods showed similar results. Of 43 analytes that were used for both validations, 28 analytes (65%) showed the same LOQ concentration for both methods. Linearities were satisfactory for both methods as well, however, correlation coefficients (R^2) were higher for most analytes when using the standard scale method.

Recovery evaluations showed equally good results for all analytes except the fungicides ametoctradin, amisulbrom, metrafenone, pyrimethanil and quinoxyfen, which showed significant differences in recovery (proven with *t*-tests, $P < 0.05$). Those analytes (excluding metrafenone) were also the ones that showed the poorest results for linearity in the micro QuEChERS approach. For metrafenone, despite the significant difference, the recoveries of both methods were within the range of 70-120%. The insecticide imidacloprid was the only one to exceed the maximum recovery of 120% for the standard scale method. Fig. 3 shows the comparison of the recoveries found in 5 g and 100 mg samples.

Regarding the method precision, on average, the 5 g samples showed a better precision compared to the micro QuEChERS approach with 100 mg sample size. Only azoxystrobin, imidacloprid and tebuconazole showed a better precision for the micro QuEChERS approach. However, despite the better results of the standard scale method, the micro QuEChERS approach delivered satisfying results for all of the investigated analytes as well and no analyte exceeded an RSD of 20%.

Overall, the performance of the standard scale method was slightly better than the micro QuEChERS approach. A very likely reason for this is due to the fact that small variations, for example deviations of solvent volume in a pipet, are more severe when working with small sample sizes. Thus, it is plausible that especially linearity and precision were somewhat lower, and furthermore, the micro QuEChERS approach meets all validation criteria demanded by the SANTE guideline (European Commission, 2019).

3.4 Analysis of wildlife animal samples

3.4.1 Hedgehog samples

Following the validation, six samples of *E. europaeus* were analysed with both optimized QuEChERS approaches for proof of concept. For this purpose, the livers were cut in 5 g and 100 mg portions and processed with the standard scale and micro scale sample preparation method, respectively. Additionally, one animal (hedgehog 1) was analysed six times with the micro QuEChERS method to confirm method precision. During qualitative analysis, six different pesticides were identified and quantified: the fungicides fenpropimorph and tebuconazole, the insecticides dieldrin and permethrin as well as the metabolites *p,p'*-DDE (originating from *p,p'*-DDT) and fipronil sulfone (originating from fipronil). The results are shown in Table 3. All samples were quantified with automated standard addition utilizing the PAL autosampler. Standard addition is a safe quantification approach as it eradicates possible errors caused by matrix effects and allows quantification below the LOQ, as long as the spiked concentration exceeds the LOQ(German Institute for Standardization, 2018).

Hedgehog 1 was analysed with the micro scale sample preparation protocol (n = 6). The RSDs were <20% for all identified analytes, which confirms the accuracy of this newly developed micro QuEChERS approach.

All hedgehog samples contained fipronil sulfone. Fipronil and its metabolites (fipronil sulfone and fipronil sulfide) have a very low MRL (5 µg kg^{-1}, only declared for livers meant for human consumption). Four hedgehog livers contained fipronil sulfone residues that exceeded this MRL between two- and almost tenfold. All other analytes were detected in concentrations that stayed below their MRLs.

For proof of concept, the results obtained with both methods

were compared. Fig. 4 shows the absolute concentration of all analytes that were quantified. All pesticides showed comparable results for the two methods. In order to take a closer look at comparability, a matching factor was calculated:

$$Matching\ factor = \frac{analyte\ concentration_{micro\ scale}}{analyte\ concentration_{standard\ scale}}$$

The desired scope of this factor was 0.7-1.2, corresponding to the 70-120% range of SANTE/12682/2019 (European Commission, 2019). The mean matching factor was 0.95, reaching from 0.70 (fipronil sulfone, hedgehog 1) to 1.36 (fenpropimorph, hedgehog 6), the latter one being the only outside of the desired range (Table 3). A possible reason for variations could be that the liver tissues were not homogenized before portioning. Probably the hepatocytes contained varying degrees of lipids depending on the nutritional status of the individual animal. Thus, depending on the lipid content of the liver segment, the concentration of certain analytes might vary due to their individual lipophilicity. Consequently, the 5 g samples are more representative for the whole liver. However, as the micro QuEChERS approach was designed for very small wildlife animal samples, homogenization before weighing is not necessary, and omission of homogenization does not represent a source of error as the whole liver is required for analysis here.

3.4.2 *Bat samples*

Two samples of each chiropteran species (*M. myotis, P. auritus, and P. nathusii*) were worked up with the micro QuEChERS method. After qualitative analysis, the detected analytes were quantified with standard addition: the insecticides *p,p*'-DDT, dieldrin, *gamma*-HCH (lindane) and the metabolites *p,p*'-DDE and fipronil sulfone.

For all analytes that exceeded the maximum of the linear range (400 µg kg^{-1}), the final extract was diluted 1:10 with acetonitrile prior to standard addition. The obtained results are summarized in Table 4.

When comparing the data, it became apparent that animals that were found in the same region and at the same time showed a very similar pesticide exposure. The circumstances of finding are also mentioned in Table 4. Both the *M. myotis* (samples 1 and 2) and both the *P. nathusii* bats (samples 5 and 6) were found under the same circumstances, respectively, whereas the *P. auritus* samples (samples 3 and 4) were from two different regions. The *M. myotis* results were very similar, the *P. nathusii* results were a bit further apart, but still showed comparable composition. In contrast to those samples, the *P. auritus* samples contained different pesticides and *p,p*'-DDE, found in both bats, showed a twentyfold higher concentration in one animal.

All bat samples contained *p,p*'-DDE or *p,p*'-DDE combined with *p,p*'-DDT. Also, lindane could be found in all bats, except sample 3. Especially the *P. nathusii* samples (bats 5 and 6) showed a tremendously high exposure to all of the abovementioned organochlorine insecticides, exceeding the corresponding MRLs (only declared for livers meant for human consumption) more than tenfold. The high concentrations of DDE, DDT and lindane in these particular samples could originate in their habitat - both bats were found in an attic. As DDT and lindane, both being persistent organic pollutants, used to be the ingredients of certain wood preservatives that were frequently used in former German Democratic Republic (GDR, East Germany) until 1989, the high exposure could correlate with the bats' roosting behaviour. However, bats 1 and 2 that also roosted in a building did not show such a high exposure. Most probably, this wood preservative was not used in this church building in the former Federal Republic of Germany (FRG, West Germany).

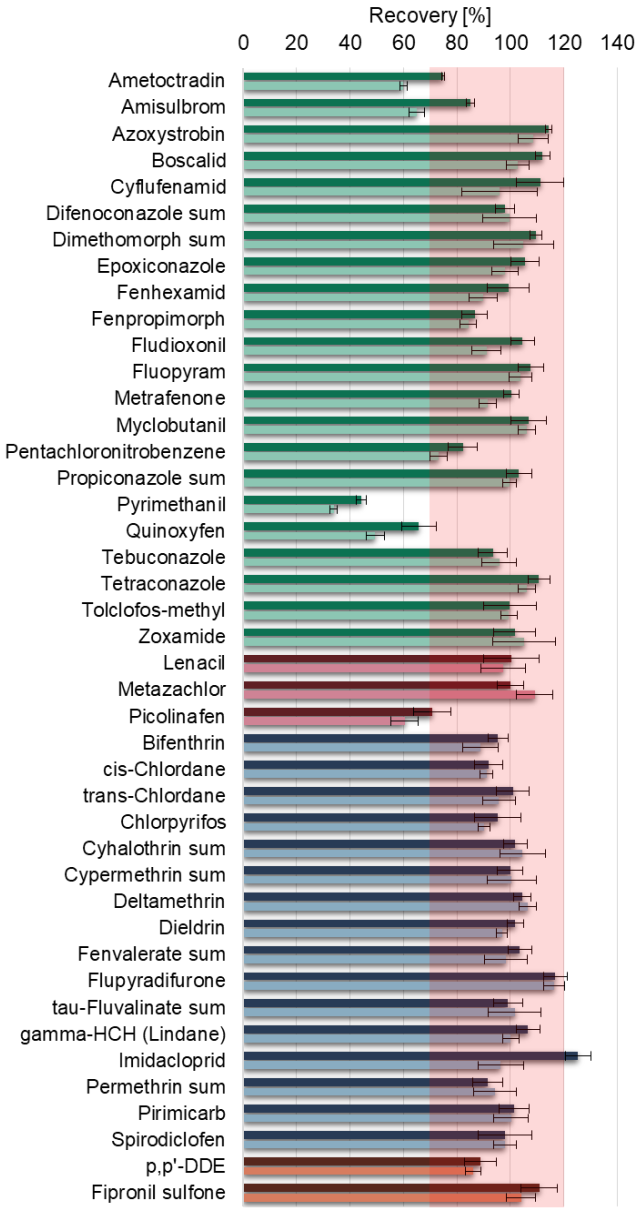

Fig. 3. Comparison of recoveries of 43 compounds (Table 1) that were validated for both sample preparation methods. Error bars show standard deviations. Red background shows 70-120% range, demanded by SANTE/12682/2019. Dark bars show standard scale method, light bars show micro scale method. Green: fungicides, red: herbicides, blue: insecticides, orange: metabolites. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

3.5 *Comparison to previous investigations*

In 2007, Eidels et al., (2007) detected different organochlorine pesticides in several bat samples from Indiana, USA. The bats contained various residues, including dieldrin (40-230 µg kg^{-1}), and *p,p*'-DDE (30-300 µg kg^{-1}). In this study here, the dieldrin concentrations found in the *M. myotis*, *P. auritus* and *P. nathusii* samples were notably lower (5-6 µg kg^{-1}), whereas the concentration range of *p,p*'-DDE was even wider (14-11,000 µg kg^{-1}) compared to the findings of Eidels et al. Another finding of residues of DDT and its metabolites in a North American bat species was published by Buchweitz et al. in 2018 (Buchweitz et al., 2018),

Table 3
Comparison of the pesticide content using both developed methods for liver samples of *E. europaeus*. Standard scale method used 5 g of liver; micro scale method used 100 mg. For hedgehog 1, average concentrations are shown with standard deviations in brackets (n = 6). Matching factor shows correlation between the two methods. n.q. not quantified. n.a. not applicable.

Hedgehog sample	Compound	Standard scale method [µg kg^{-1}]	Micro scale method [µg kg^{-1}]	Matching factor
1	*p,p*'-DDE	2.46	2.17 (±0.16)	0.88
	Fenpropimorph	0.91	0.98 (±0.05)	1.07
	Fipronil sulfone	5.70	3.94 (±0.58)	0.70
2	Fenpropimorph	0.46	0.51	1.11
	Fipronil sulfone	n.q.	0.23	n.a.
	Tebuconazole	0.84	0.65	0.77
3	Fipronil sulfone	10.17	9.49	0.93
4	*p,p*'-DDE	22.23	21.73	0.98
	Dieldrin	3.10	2.49	0.80
	Fipronil sulfone	12.56	10.62	0.85
5	Fenpropimorph	1.27	1.43	1.13
	Fipronil sulfone	10.66	10.03	0.94
6	*p,p*'-DDE	1.19	1.03	0.87
	Fenpropimorph	1.46	2.16	1.36
	Fipronil sulfone	48.60	40.63	0.84
	Permethrin sum	6.91	7.31	1.06
	Tebuconazole	n.q.	0.32	n.a.

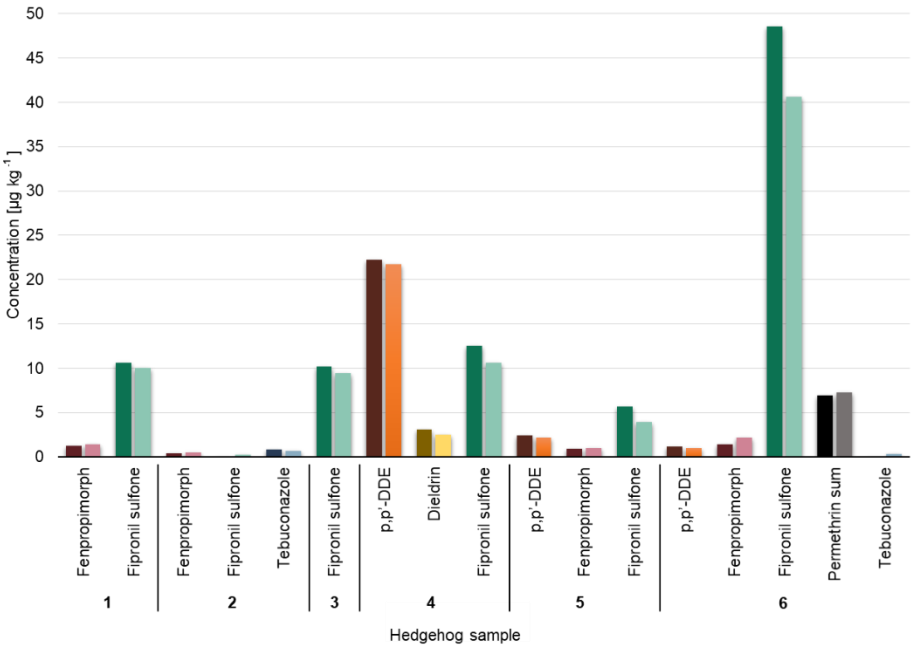

Fig. 4. Absolute concentrations of all pesticides found in six hedgehog livers after standard addition. Dark bars show results of standard QuEChERS method, light bars show resultsof micro-QuEChERS method, respectively.

reporting a DDT concentration of more than 1000 mg kg^{-1} in one bat liver sample. The sample preparation was performed with a modified QuEChERS method there, however, 3 g of sample material were necessary for work-up and only DDT and its metabolites DDE and DDD were covered with the applied GC-MS/MS method. A recent review article dealing with literature from 1951 to 2020, covering studies from America, Europe, Asia and Australia, showed that organochlorine insecticides, such as DDT, DDE, dieldrin and lindane, are the most investigated group of pesticides associated with bat species (Torquetti et al., 2020). Compounds of that group could be found by analysing whole animal carcasses, but also in bat brains, livers, and fat tissue or even in guano and washes from wings or fur. These findings from all over the world are reflected by this study.

Besides exposure to mammals, also pesticide residues in birds are of a high interest. Different studies claim declines in bird populations due to neonicotinoid use (Hallmann et al., 2014; Turaga et al., 2016). Rial-Berriel et al. (2020) investigated more than 300 pesticides, rodenticides and pharmaceuticals in blood samples of different raptor species and found persistent compounds like PCB's in one third of all samples. DDE was found in more than a half of thecollected samples, but the concentration was comparatively low with a maximum content of 4.40 ng mL^{-1}.

4 Conclusion

Within the frame of this work, a QuEChERS-based sample preparation method was optimized and validated for liver samples

Table 4
Pesticide concentrations of six bat liver samples from Germany analysed with the newly developed micro QuEChERS approach and quantified with standard addition. GDR: former German Democratic Republic (East Germany), FRG: former Federal Republic of Germany (West Germany).

Bat sample				Pesticide exposure	
Sample number	Species	Location	Year	Compound	Concentration [µg kg^{-1}]
1	*Myotis myotis*	Church building, FRG	2018	*p,p*'-DDE	21.90
				Dieldrin	4.89
				gamma-HCH	2.46
2				*p,p*'-DDE	24.66
				Dieldrin	4.62
				gamma-HCH	2.93
3	*Plecotus auritus*	Unknown, FRG	2018	*p,p*'-DDE	280.19
				p,p'-DDT	42.12
				Dieldrin	6.42
				Fipronil sulfone	0.39
4		Unknown, FRG	2019	*p,p*'-DDE	14.44
				gamma-HCH	0.65
5	*Pipistrellus nathusii*	House attic, GDR	2017	*p,p*'-DDE	10797.72
				p,p'-DDT	4082.05
				gamma-HCH	232.88
				Fipronil sulfone	2.38
6				*p,p*'-DDE	9704.23
				p,p'-DDT	22908.18
				gamma-HCH	198.45

of medium-sized wildlife animals and successfully miniaturized for the analysis of small wildlife animals. This new micro QuEChERS approach enables the analysis of more than 200 pesticides and persistent organic pollutants (POPs) in liver samples within a single GC-MS/MS run and gets along with 100 mg of matrix. The miniaturized method consumes only minute amounts of solvent and other chemicals while delivering robust and accurate results, which was proven by a full validation according to SANTE/12682/2019. Limits of quantification lay between 1 and 20 µg kg^{-1} and linearity was confirmed up to 400 µg kg^{-1}. Hence, pesticide residues can be analysed and quantified in the low µg kg^{-1} range. Thus, it is now possible to perform multiresidue analysis on small (wildlife) animals that cannot be analysed with a conventional QuEChERS sample preparation protocol. Particularly in the light of biodiversity loss, further knowledge of the exposure of potentially endangered wildlife species like bats to xenobiotics, through such a methodical approach, is urgently needed.

The analysis of six hedgehog and six bat samples revealed their exposure to different classes of pesticides, including organochlorine insecticides and azole fungicides. The most prominent analytes were the metabolites fipronil sulfone, which reached concentrations up to 50 µg kg^{-1} in the hedgehog samples, and *p,p*'-DDE, which could be detected in all bat samples with concentrations between 14 µg kg^{-1} and 11 mg kg^{-1}. These findings emphasize the importance of being able to monitor the pesticide load of small non-target animals, to which this micro QuEChERS approach contributes.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

We are deeply saddened to hear of the death of Dr. Med. vet. Anja Baronetzky-Mercier. Her passing is not only a great loss to our environmental research project but also the living nature has lost a great advocate and fighter. We have lost an outstanding personality with the heart in the right place, who has become, for many of us, a wonderful friend.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2021.130434.

Author statement

SS, Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation; Writing - original draft, Writing - review & editing, Visualization. CM; Conceptualization, Methodology, Formal analysis, Writing - review & editing, Supervision. EK, Resources, Writing - review & editing, Funding acquisition. GW, Resources, Writing - review & editing, Funding acquisition. MK, Resources, Writing - review & editing. AK, Resources, Writing - review & editing, Funding acquisition. FB; Resources, Writing - review & editing, Funding acquisition.

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3.4. Supplementary material

Supplementary Table 1 Pesticide classification, retention time (RT), dMRM transitions and collision energy (CE) of analyzed compounds. Quantifier transitions are marked in bold.

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
2,4-D-ethyl ester	Herbicide	7.49	**247.9 → 185.0** 185.0 → 114.9 175.0 → 111.0	10 25 10
2-Phenylphenol	Microbiocide	6.25	169.1 → 91.0 **141.1 → 63.0** 115.1 → 65.0	35 45 25
8-Hydroxyquinoline	Fungicide, Microbiocide	5.38	145.0 → 63.0 117.0 → 63.0 **117.0 → 39.1**	40 40 40
Acequinocyl	Insecticide	16.77	342.9 → 188.8 **341.9 → 187.9** 187.9 → 131.0	20 15 20
Acetamiprid	Insecticide	13.85	221.0 → 56.1 126.0 → 90.0 **126.0 → 72.9**	15 5 20
Acibenzolar-S-methyl	Fungicide	9.30	182.0 → 167.1 182.0 → 153.1 **182.0 → 135.0**	10 10 15
Aclonifen	Herbicide	12.39	264.1 → 194.2 **194.1 → 167.1** 194.1 → 139.1	15 20 25
Acrinathrin	Insecticide	15.02	**288.9 → 92.8** 207.8 → 152.0 181.0 → 127.0	10 35 30
Aldrin	Insecticide	9.94	262.9 → 192.9 262.9 → 190.9 254.9 → 220.0	35 35 20
Ametoctradin	Fungicide	15.20	275.0 → 246.2 275.0 → 190.3 **246.0 → 188.2**	0 15 25
Amisulbrom	Fungicide	16.14	227.9 → 147.0 **225.9 → 147.0** 214.0 → 160.0	15 15 20
Azoxystrobin	Fungicide	18.30	344.1 → 182.9 **344.1 → 171.9** 344.1 → 155.8	25 40 40
Azoxystrobin-D_4	Internal Standard	18.29	407.0 → 348.0 392.0 → 364.0 **348.0 → 172.1**	5 5 35
Beflubutamid	Herbicide	10.67	192.9 → 145.1 192.9 → 95.0 **176.1 → 79.1**	15 35 25
Benalaxyl	Fungicide	12.87	266.0 → 148.1 **233.9 → 146.0** 206.0 → 162.1	5 20 5

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Bentazone	Herbicide	10.11	**225.0 → 181.9** 198.0 → 92.0 182.0 → 90.0	5 30 15
Benthiavalicarb-isopropyl	Fungicide	14.57	222.0 → 125.9 **180.0 → 127.0** 180.0 → 83.0	40 20 30
Bifenazate	Insecticide	13.94	184.1 → 91.1 **184.1 → 77.0** 168.1 → 140.1	40 40 10
Bifenox	Herbicide	14.21	340.9 → 309.9 **340.9 → 280.9** 189.1 → 126.0	10 15 20
Bifenthrin	Insecticide	13.83	**181.0 → 115.1** 166.0 → 139.1 166.0 → 115.1	45 35 35
Boscalid	Fungicide	16.50	140.0 → 112.0 **140.0 → 76.0** 111.9 → 76.0	10 25 15
Bromoxynil	Herbicide	7.41	276.8 → 88.0 **274.7 → 167.9** 274.7 → 88.0	30 15 30
Bromuconazole (2 isomers)	Fungicide	13.85 14.29	295.0 → 172.9 **293.0 → 172.9** 173.0 → 109.0	10 10 30
Bupirimate	Fungicide	11.80	**315.8 → 207.9** 208.0 → 68.9 193.0 → 109.0	10 30 15

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Buprofezin	Insecticide	11.74	**304.9 → 175.0** 249.1 → 193.0 171.1 → 115.0	10 10 10
Captan	Fungicide	10.73	**263.8 → 79.0** 149.0 → 70.0 116.9 → 82.0	15 15 30
Carbetamide	Herbicide	9.95	120.1 → 92.0 120.1 → 77.0 **119.1 → 64.1**	10 15 25
Carboxin	Fungicide	11.75	**234.9 → 143.0** 234.9 → 87.0 131.9 → 77.0	10 20 20
Carfentrazone-ethyl	Herbicide	12.81	**339.9 → 311.9** 329.9 → 309.9 311.9 → 150.8	10 10 20
cis-Chlordane	Insecticide	11.20	374.8 → 265.8 **372.8 → 265.8** 271.7 → 236.9	15 15 15
trans-Chlordane	Insecticide	10.94	374.8 → 265.8 **372.8 → 265.8** 271.7 → 236.9	15 15 15
Chloridazon (Pyrazon)	Herbicide	13.04	221.0 → 220.2 **220.0 → 193.1** 220.0 → 166.0	5 20 25
Chlorothalonil	Fungicide	8.54	265.9 → 230.9 **265.9 → 133.0** 265.9 → 109.0	20 45 45

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Chlorotoluron	Herbicide	9.70	**212.1 → 166.0** 212.1 → 72.0 167.0 → 132.1	10 15 15
Chlorpropham	Herbicide, Plant growth regulator	7.11	**213.0 → 171.1** 171.0 → 127.1 153.0 → 90.0	5 5 25
Chlorpyrifos	Insecticide	9.86	**313.8 → 257.8** 196.9 → 107.0 196.9 → 98.0	15 40 30
Chlorpyrifos-D_{10}	Internal Standard	9.80	325.9 → 262.1 **323.9 → 260.0** 259.8 → 167.0	10 10 15
Chlorpyrifos-methyl	Insecticide	9.14	287.9 → 92.9 **285.9 → 93.0** 124.9 → 47.0	20 25 15
Clodinafop-propargyl	Plant growth regulator	12.97	348.9 → 265.9 **348.9 → 237.8** 238.0 → 130.0	10 15 15
Clomazone	Herbicide	7.98	**205.1 → 107.1** 127.0 → 101.0 125.0 → 89.0	20 20 15
Cloquintocet-mexyl	Herbicide safener	14.00	220.0 → 191.9 **163.0 → 128.0** 163.0 → 101.0	10 15 30
Cyflufenamid	Fungicide	11.88	188.1 → 88.0 **118.1 → 90.0** 118.1 → 89.0	35 10 25

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Cyfluthrin (3 isomers)	Insecticide	16.17 16.25 16.37	206.0 → 176.9 206.0 → 150.0 **162.9 → 127.0**	25 40 5
Cyhalofop-butyl	Herbicide	14.68	**357.1 → 229.1** 256.2 → 120.1 229.2 → 109.1	15 10 15
Cyhalothrin (*gamma* and *lambda* isomer)	Insecticide	14.79 14.60	**208.0 → 181.0** 208.0 → 152.0 197.0 → 161.1	5 25 5
Cypermethrin (3 isomers)	Insecticide	16.39 16.48 16.57	165.0 → 127.1 165.0 → 91.1 **162.9 → 127.0**	0 10 0
Cyproconazole	Fungicide	11.99	**222.0 → 124.9** 138.9 → 111.0 138.9 → 75.0	25 15 35
Cyprodinil	Fungicide	10.39	225.2 → 224.3 **224.2 → 131.1** 210.0 → 93.0	10 15 20
Cyromazine	Insecticide	7.97	**165.9 → 109.0** 151.0 → 82.0 109.0 → 68.0	20 30 20
Dazomet	Fungicide	7.76	161.9 → 89.0 **89.0 → 46.0** 88.9 → 74.0	25 5 15
o,p'-DDD	Insecticide, Breakdown	11.78	235.0 → 200.1 235.0 → 139.1 **199.1 → 164.1**	10 45 20

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
p,p'-DDD	Insecticide, Breakdown	12.36	237.0 → 200.1 **199.1 → 164.1** 165.1 → 139.0	15 20 35
o,p'-DDE	Breakdown	10.98	317.8 → 248.0 248.0 → 176.2 **246.0 → 176.2**	15 30 30
p,p'-DDE	Breakdown	11.52	317.8 → 246.0 315.8 → 246.0 **246.1 → 176.2**	15 15 30
o,p'-DDT	Insecticide	12.27	237.0 → 199.1 **235.0 → 199.1** 199.0 → 163.1	15 15 35
p,p'-DDT	Insecticide	12.94	237.0 → 165.2 235.0 → 199.2 **235.0 → 165.2**	20 15 20
Deltamethrin	Insecticide	18.02	252.9 → 174.0 **252.9 → 93.1** 251.0 → 172.0	0 15 0
Desmedipham	Herbicide	7.59	181.0 → 122.0 **181.0 → 109.0** 135.0 → 52.0	10 10 25
Diazinon	Insecticide	8.29	276.0 → 137.1 **199.1 → 135.1** 179.1 → 137.1	25 10 20
Dicamba-methyl ester	Herbicide	6.26	234.0 → 173.0 **205.0 → 149.0** 175.0 → 111.0	20 15 20

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Diclofop-methyl	Herbicide	13.26	339.9 → 252.9 **280.8 → 119.9** 253.0 → 162.1	10 10 15
Dieldrin	Insecticide	11.62	**277.0 → 241.0** 262.9 → 193.0 262.9 → 191.0	5 35 35
Diethofencarb	Fungicide	9.76	225.0 → 96.0 **207.0 → 179.1** 207.0 → 151.0	30 5 15
Difenoconazole (2 isomers)	Fungicide	17.72 17.78	324.8 → 266.8 **322.8 → 264.8** 264.9 → 202.0	15 15 20
Diflubenzuron	Insecticide	5.01	141.0 → 113.0 **141.0 → 63.0** 113.0 → 63.0	40 40 40
Diflufenican	Herbicide	13.29	**393.9 → 265.9** 266.0 → 246.1 218.0 → 140.1	10 15 20
Dimethachlor	Herbicide	8.99	209.9 → 134.1 **196.9 → 148.2** 134.1 → 79.1	10 10 20
Dimethenamide-P	Herbicide	9.02	229.9 → 154.0 **229.9 → 111.0** 202.9 → 154.0	10 25 10
Dimethoate	Insecticide	7.79	228.7 → 87.0 157.0 → 93.0 **157.0 → 63.0**	5 10 25

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Dimethomorph (2 isomers)	Fungicide	18.35 18.66	302.9 → 164.9 **300.9 → 165.0** 300.9 → 138.8	10 10 15
Dimoxystrobin	Fungicide	13.85	237.0 → 116.0 **205.0 → 116.0** 174.0 → 115.0	15 10 30
Diuron	Herbicide	10.66	231.7 → 71.8 **186.9 → 124.0** 158.9 → 123.9	15 20 10
Dodemorph (2 isomers)	Fungicide	10.24 10.55	**281.0 → 154.0** 238.1 → 55.1 154.0 → 112.1	10 20 10
Epoxiconazole	Fungicide	13.52	**192.0 → 138.1** 192.0 → 111.0 138.0 → 75.0	10 25 25
Ethofenprox	Insecticide	16.78	183.0 → 168.0 **163.0 → 135.1** 163.0 → 107.1	10 10 20
Ethofumesate	Herbicide	9.61	285.9 → 207.1 **178.9 → 137.1** 178.9 → 105.1	5 0 15
Ethoprophos	Insecticide	7.02	199.9 → 97.0 **157.9 → 97.0** 157.9 → 81.0	20 15 15
Etoxazole	Insecticide	14.07	329.9 → 315.0 299.9 → 284.9 **299.9 → 269.9**	20 10 20

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Etridiazole	Fungicide	5.85	211.1 → 183.0 211.1 → 140.0 **185.0 → 142.0**	10 25 15
Famoxadone	Fungicide	18.44	329.9 → 329.0 **329.9 → 223.9** 223.9 → 196.2	10 10 10
Fenamiphos	Insecticide	11.31	302.9 → 287.9 302.9 → 153.9 **287.9 → 259.7**	10 15 5
Fenazaquin	Insecticide	14.19	**160.0 → 145.2** 160.0 → 117.1 146.0 → 118.1	5 20 10
Fenbuconazole	Fungicide	16.21	197.9 → 129.0 197.9 → 102.0 **125.0 → 89.0**	5 30 20
Fenhexamid	Fungicide	12.97	301.0 → 97.0 179.0 → 115.0 **177.1 → 113.0**	15 15 15
Fenoxaprop-P-ethyl	Herbicide	15.33	360.8 → 287.8 **287.8 → 118.8** 287.8 → 90.9	10 10 20
Fenoxycarb	Insecticide	13.86	256.1 → 187.2 **186.2 → 109.0** 186.2 → 77.1	10 15 20
Fenpropidin	Fungicide	9.45	**273.0 → 98.0** 145.0 → 117.0 145.0 → 91.0	5 10 25

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Fenpropimorph	Fungicide	9.81	128.1 → 110.1 128.1 → 86.1 **128.1 → 70.1**	5 10 10
Fenpyroximate	Insecticide	7.85	212.0 → 185.0 **212.0 → 76.9** 198.1 → 114.0	40 40 35
Fenvalerate (2 isomers)	Insecticide	17.31 17.50	419.1 → 166.8 **167.0 → 125.1** 167.0 → 89.0	10 10 40
Fipronil	Insecticide	10.64	**366.8 → 212.8** 350.8 → 254.8 254.9 → 228.0	25 15 15
Fipronil sulfide	Breakdown	10.50	420.0 → 350.9 **351.0 → 254.9** 254.9 → 156.9	10 20 35
Fipronil sulfone	Breakdown	11.71	384.8 → 256.8 **382.8 → 254.9** 254.9 → 227.9	20 20 15
Fluazifop-P-butyl	Herbicide	11.97	382.9 → 282.0 **281.9 → 238.0** 254.0 → 146.1	10 15 15
Fludioxonil	Fungicide	11.51	248.0 → 182.1 248.0 → 154.1 **248.0 → 127.1**	10 20 30
Flufenacet	Herbicide	9.96	211.0 → 123.0 **211.0 → 96.0** 183.0 → 69.0	5 15 20

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Flumetralin	Herbicide	11.19	403.9 → 156.8 359.9 → 313.9 **157.0 → 109.0**	15 15 25
Flumioxazin	Herbicide	17.43	354.0 → 325.9 354.0 → 175.8 **287.0 → 258.7**	5 15 15
Fluometuron	Herbicide	6.98	232.0 → 72.0 **213.0 → 167.9** 187.0 → 109.0	15 10 20
Fluopyram	Fungicide	10.57	395.9 → 223.1 **222.9 → 196.0** 222.9 → 187.1	5 10 10
Fluorochloridone	Herbicide	10.11	311.0 → 174.1 **311.0 → 102.9** 187.1 → 109.1	15 15 20
Flupyradifurone	Insecticide	14.87	288.0 → 126.1 128.0 → 90.0 **126.0 → 73.0**	15 10 25
Fluquinconazole	Fungicide	15.85	342.0 → 107.8 **340.0 → 298.0** 340.0 → 107.8	40 15 40
Fluroxypyr-meptyl	Herbicide	13.29	237.0 → 209.0 **237.0 → 181.0** 208.9 → 178.9	5 15 20
Flurtamone	Herbicide	14.43	**332.7 → 120.0** 157.0 → 137.1 157.0 → 107.0	15 15 25

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Flutolanil	Herbicide	11.38	322.9 → 281.0 **280.9 → 173.0** 173.0 → 95.0	5 10 30
Flutriafol	Fungicide	11.30	**219.1 → 123.1** 219.1 → 95.0 164.1 → 109.1	15 35 20
tau-Fluvalinate (2 isomers)	Insecticide	17.48 17.52	252.0 → 200.0 **250.0 → 200.1** 250.0 → 198.1	15 15 40
Fluxapyroxad	Fungicide	14.57	**321.1 → 152.9** 222.0 → 152.9 222.0 → 125.9	35 15 40
Fosthiazate (2 isomers)	Nematicide	10.27 10.31	**199.0 → 102.0** 195.0 → 60.0 165.9 → 106.0	5 20 10
Fuberidazole	Fungicide	9.16	184.0 → 155.1 **156.0 → 103.1** 155.0 → 129.1	30 20 10
Haloxyfop-P-methyl	Herbicide	10.93	375.1 → 316.0 **375.1 → 91.1** 288.0 → 180.0	10 35 25
alpha-HCH	Insecticide	7.64	218.9 → 183.0 **216.9 → 181.0** 180.9 → 145.0	5 5 15
beta-HCH	Insecticide	7.99	218.9 → 183.1 **216.9 → 181.1** 181.0 → 145.0	5 5 15

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
gamma-HCH (Lindane)	Insecticide	8.08	218.9 → 183.1 **216.9 → 181.0** 181.0 → 145.0	5 5 15
delta-HCH	Insecticide	8.51	**217.0 → 181.1** 183.1 → 147.1 181.1 → 145.1	5 15 15
epsilon-HCH	Insecticide	8.69	254.0 → 180.9 218.9 → 182.9 **182.9 → 109.0**	10 5 30
Heptachlor	Insecticide	9.34	273.7 → 238.9 273.7 → 236.9 **271.7 → 236.9**	15 15 15
Heptachlor endo-epoxide	Breakdown	10.67	216.9 → 182.0 216.9 → 109.0 **183.0 → 119.0**	20 45 30
Heptachlor exo-epoxide	Breakdown	10.61	**354.8 → 264.9** 352.8 → 262.9 262.9 → 193.0	15 15 35
Hexachloro-benzene	Fungicide	7.70	**283.8 → 213.9** 281.8 → 211.9 248.9 → 179.0	30 30 30
Imazalil	Fungicide	11.48	216.8 → 175.0 174.9 → 147.0 **172.9 → 109.0**	5 15 30
Imidacloprid	Insecticide	11.31	211.0 → 113.0 126.0 → 89.9 **126.0 → 73.0**	15 5 25

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Indoxacarb	Insecticide	18.02	**264.0 → 175.8** 202.9 → 134.0 202.9 → 106.0	15 20 15
Ipconazole	Fungicide	15.00	249.0 → 125.0 167.0 → 125.0 **125.0 → 89.0**	15 5 20
Iprovalicarb (2 isomers)	Fungicide	11.61 11.78	**158.0 → 98.0** 134.1 → 93.0 116.0 → 98.1	10 15 5
Isopyrazam	Fungicide	15.30	359.0 → 159.0 **302.1 → 262.1** 159.0 → 139.0	40 15 10
Isoxaben	Herbicide	15.17	165.0 → 150.0 165.0 → 107.0 **149.9 → 121.9**	15 25 5
Kresoxim-methyl	Fungicide	11.81	206.0 → 131.1 **206.0 → 116.0** 116.0 → 89.0	10 5 15
Lenacil	Herbicide	12.95	233.9 → 153.1 153.1 → 110.1 **153.1 → 82.1**	5 20 20
Lufenuron	Insecticide	5.58	251.6 → 157.8 **202.9 → 75.9** 173.9 → 109.9	15 40 30
Malathion	Insecticide	9.73	172.9 → 117.0 **172.9 → 99.0** 157.8 → 125.0	15 10 5

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
MCPA-methyl ester	Herbicide	6.51	214.1 → 155.1 214.1 → 141.1 **155.1 → 125.1**	10 10 10
MCPB-methyl ester	Herbicide	8.17	211.1 → 155.0 **142.1 → 107.1** 142.1 → 77.1	10 10 30
Mefenpyr-diethyl	Herbicide safener	13.59	**299.0 → 252.9** 253.0 → 190.0 253.0 → 189.0	10 20 30
Mepanipyrim	Fungicide	11.16	222.2 → 158.1 221.2 → 220.2 **207.1 → 179.1**	25 15 25
Metalaxyl	Fungicide	9.33	234.0 → 146.1 **220.0 → 160.1** 206.1 → 162.1	20 10 5
Metamitron	Herbicide	11.83	**202.1 → 186.1** 202.1 → 104.1 173.1 → 132.1	5 15 10
Metazachlor	Herbicide	10.45	209.0 → 133.2 **209.0 → 132.2** 209.0 → 117.1	10 15 35
Metconazole	Fungicide	14.22	153.1 → 125.0 153.1 → 70.0 **125.0 → 89.0**	10 5 20
Methiocarb	Insecticide	9.58	169.0 → 154.1 168.0 → 109.1 **153.0 → 91.1**	10 15 20

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Metobromuron	Herbicide	8.79	258.0 → 61.0 **196.9 → 89.9** 169.9 → 142.9	10 25 20
(S)-Metolachlor	Herbicide	9.89	238.0 → 162.2 **238.0 → 133.2** 162.1 → 133.2	10 30 15
Metrafenone	Fungicide	15.24	**394.8 → 364.8** 376.9 → 346.8 226.9 → 169.0	15 20 10
Metribuzin	Herbicide	9.00	**198.0 → 82.0** 198.0 → 55.0 182.0 → 114.9	15 30 10
Myclobutanil	Fungicide	11.68	**179.0 → 125.1** 179.0 → 90.0 150.0 → 123.0	10 30 15
Napropamide	Herbicide	11.40	**271.0 → 100.1** 271.0 → 72.1 128.0 → 100.1	15 15 10
Oryzalin	Herbicide	15.51	316.8 → 274.9 **275.0 → 217.0** 258.0 → 193.9	5 5 5
Oxadiazon	Herbicide	11.63	**301.8 → 175.0** 257.8 → 112.0 174.9 → 112.0	15 30 15
Oxamyl	Insecticide, Nematicide	6.30	162.0 → 114.9 145.0 → 71.9 **145.0 → 60.9**	10 20 10

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Oxychlordane	Breakdown	10.53	386.8 → 262.8 271.8 → 236.8 **184.8 → 121.0**	15 25 15
Oxyfluorfen	Herbicide	11.71	299.9 → 222.8 252.0 → 196.0 **252.0 → 146.0**	15 20 30
Paclobutrazol	Plant growth regulator	11.09	236.0 → 167.1 **167.1 → 132.1** 125.1 → 89.0	10 10 20
Parathion	Insecticide	9.97	**291.0 → 137.1** 291.0 → 109.0 139.0 → 81.0	5 15 15
Parathion-methyl	Insecticide, Nematicide	9.14	262.9 → 109.0 262.9 → 79.0 **109.0 → 79.0**	10 30 5
PCB 28	Pollutant	9.04	**258.0 → 186.0** 256.0 → 186.0 186.0 → 151.0	25 25 25
PCB 52	Pollutant	9.61	291.9 → 221.9 **289.9 → 219.9** 255.0 → 220.0	25 25 10
PCB 101	Pollutant	11.12	325.9 → 255.9 325.9 → 253.9 **253.9 → 184.0**	35 30 30
PCB 138	Pollutant	13.12	**361.9 → 289.9** 359.9 → 289.9 287.9 → 217.9	30 30 40

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
PCB 153	Pollutant	12.62	361.9 → 289.9 **359.9 → 289.9** 287.9 → 217.9	25 25 40
PCB 180	Pollutant	14.30	395.8 → 325.8 393.8 → 358.8 **393.8 → 323.8**	30 15 30
Penconazole	Fungicide	10.54	250.0 → 194.1 **250.0 → 157.1** 159.0 → 123.0	15 25 20
Pendimethalin	Herbicide	10.52	**251.8 → 162.2** 251.8 → 146.1 161.9 → 147.0	10 20 10
Pentachloro-nitrobenzene	Fungicide, Nematicide	8.20	294.8 → 236.8 248.8 → 213.8 **141.9 → 106.9**	15 15 30
Permethrin (*cis* and *trans* isomer)	Insecticide	15.51 15.63	165.0 → 127.0 **162.9 → 127.0** 162.9 → 91.0	0 0 10
Phenmedipham	Herbicide	7.08	**167.0 → 135.0** 167.0 → 122.0 122.0 → 94.0	15 15 15
Phosmet	Insecticide	13.90	**161.0 → 134.0** 161.0 → 78.0 160.0 → 133.1	10 20 10
Phosmet-oxon	Breakdown	13.00	**301.0 → 191.8** 172.9 → 104.0 160.0 → 133.0	10 15 15

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Picloram-methyl ester	Herbicide	9.55	**198.0 → 163.1** 198.0 → 161.0 196.0 → 181.0	15 15 15
Picolinafen	Herbicide	13.87	376.0 → 239.1 **376.0 → 238.1** 238.1 → 145.1	10 20 25
Picoxystrobin	Fungicide	11.29	**334.9 → 172.9** 302.8 → 156.9 145.0 → 102.1	10 15 25
Pirimicarb	Insecticide	8.73	**238.0 → 166.2** 166.0 → 71.1 152.0 → 123.0	10 25 10
Pirimiphos-methyl	Insecticide	9.58	290.0 → 125.0 **232.9 → 151.0** 232.9 → 125.0	20 5 5
Prochloraz	Fungicide	15.91	310.0 → 69.8 **266.0 → 69.9** 180.0 → 68.9	15 10 15
Propamocarb	Fungicide	5.39	188.0 → 58.0 **143.0 → 99.1** 129.1 → 84.1	10 10 5
Propaquizafop	Herbicide	19.92	298.8 → 254.8 **162.9 → 135.8** 162.9 → 99.9	25 10 20
Propiconazole (2 isomers)	Fungicide	12.89 13.00	258.8 → 172.9 172.9 → 109.0 **172.9 → 74.0**	15 30 45

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Prosulfocarb	Herbicide	9.37	251.0 → 218.3 **251.0 → 128.2** 251.0 → 100.1	10 5 5
Prothioconazole-desthio	Fungicide	11.91	**186.0 → 89.0** 186.0 → 70.0 125.0 → 99.0	10 10 20
Pymetrozine	Insecticide	11.51	132.0 → 105.0 132.0 → 78.0 **113.0 → 98.0**	10 20 5
Pyraclostrobin	Fungicide	17.46	**324.8 → 131.7** 164.0 → 132.1 110.8 → 75.0	15 10 15
Pyraflufen-ethyl	Herbicide	13.03	412.0 → 349.0 349.0 → 307.0 **338.9 → 288.9**	10 15 15
Pyridaben	Insecticide	15.77	**309.0 → 147.1** 147.2 → 132.2 147.2 → 117.1	15 10 20
Pyridalyl	Insecticide	16.73	204.0 → 148.0 164.0 → 146.0 **146.0 → 126.0**	25 15 10
Pyridate	Herbicide	17.26	**205.2 → 141.1** 205.2 → 114.0 205.2 → 102.0	25 35 30
Pyrimethanil	Fungicide	8.24	198.0 → 183.1 198.0 → 158.1 **198.0 → 118.1**	15 20 35

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Pyriproxyfen	Insecticide	14.61	**321.0 → 222.0** 321.0 → 153.0 136.1 → 96.0	10 25 15
Quinoclamine	Herbicide	9.76	209.0 → 172.1 207.0 → 172.1 **172.0 → 89.0**	10 20 20
Quinoxyfen	Fungicide	12.85	306.8 → 237.0 271.9 → 237.1 **237.0 → 208.0**	20 10 30
Spirodiclofen	Insecticide	15.56	312.1 → 259.0 **312.1 → 108.9** 157.0 → 73.0	10 15 25
Spiromesifen	Insecticide	13.71	272.0 → 209.2 **253.8 → 185.1** 231.0 → 157.1	10 15 15
Spiroxamine (2 Isomers)	Fungicide	9.08 9.53	**198.0 → 126.1** 126.0 → 84.0 100.0 → 58.1	5 5 10
Tebuconazole	Fungicide	13.22	**250.0 → 125.0** 125.0 → 99.0 125.0 → 89.0	20 20 15
Tebufenpyrad	Insecticide	14.09	332.9 → 171.0 318.0 → 131.0 **275.9 → 171.1**	15 15 10
Tefluthrin	Insecticide	8.41	199.0 → 161.1 **197.0 → 161.1** 177.1 → 127.1	5 5 15

Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Terbuthylazine	Herbicide, Microbiocide	8.12	228.9 → 138.0 214.0 → 71.0 **172.9 → 138.1**	15 20 5
Terbuthylazine-desethyl	Herbicide, Microbiocide	7.36	186.2 → 104.0 186.2 → 83.1 **145.1 → 110.1**	15 20 10
Tetraconazole	Fungicide	9.99	336.0 → 217.9 **336.0 → 203.8** 170.9 → 136.0	20 30 10
Thiabendazole	Fungicide	10.73	**201.9 → 175.0** 201.0 → 130.0 173.9 → 65.0	15 30 30
Thiacloprid	Insecticide	17.50	126.0 → 99.1 **126.0 → 90.1** 126.0 → 73.0	10 5 20
Tolclofos-methyl	Fungicide	9.14	**267.0 → 252.0** 267.0 → 93.0 267.0 → 63.0	15 30 45
Tralkoxydim	Herbicide	14.75	282.1 → 226.0 268.2 → 143.0 **226.0 → 143.0**	10 40 25
Triadimenol	Fungicide	10.73	**129.9 → 102.0** 129.9 → 65.0 112.0 → 58.0	15 25 10
Triallate	Herbicide	8.57	**270.0 → 228.1** 268.0 → 226.1 268.0 → 184.1	10 10 20

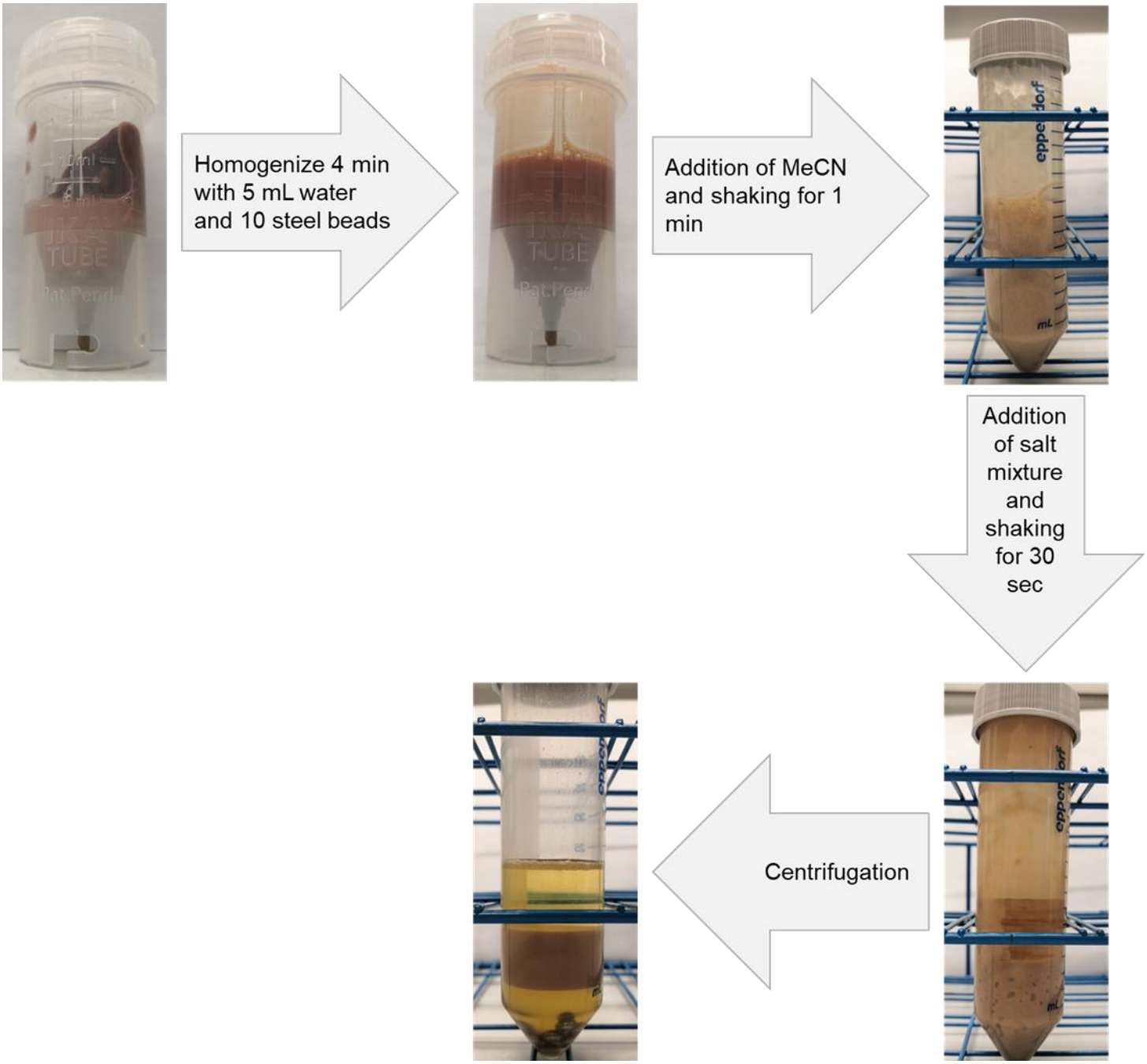
Name	Pesticide classification	RT [min]	dMRM transitions	CE [eV]
Triclopyr-methyl ester	Herbicide	7.51	**209.9 → 145.9** 209.9 → 109.9 145.9 → 110.0	20 35 15
Trifloxystrobin	Fungicide	12.94	186.0 → 145.1 **172.0 → 145.1** 172.0 → 95.0	15 15 30
Triflumizole	Fungicide	10.81	345.0 → 302.0 239.1 → 66.9 **132.0 → 90.0**	10 40 35
Trinexapac-ethyl	Herbicide	9.50	**224.0 → 151.0** 224.0 → 95.0 207.0 → 68.9	5 25 25
Triphenyl phosphate	Internal Standard	13.35	**326.0 → 325.0** 325.0 → 169.1 325.0 → 77.0	5 20 35
Triticonazole	Fungicide	14.51	237.0 → 182.0 237.0 → 167.1 **234.8 → 182.1**	10 25 10
Warfarin	Rodenticide	15.44	308.0 → 187.0 **265.0 → 187.0** 265.0 → 121.0	20 5 15
Zoxamide	Fungicide	13.47	259.9 → 189.0 **257.9 → 187.1** 189.0 → 161.1	10 10 15

Supplementary Table 2 Summary of all method precision data obtained during validation. All RSD values are shown in %. The respective LOQ concentration can be found in **Table 1**. n.d.: not determined. *: LOQ was 20 µg kg^{-1}.

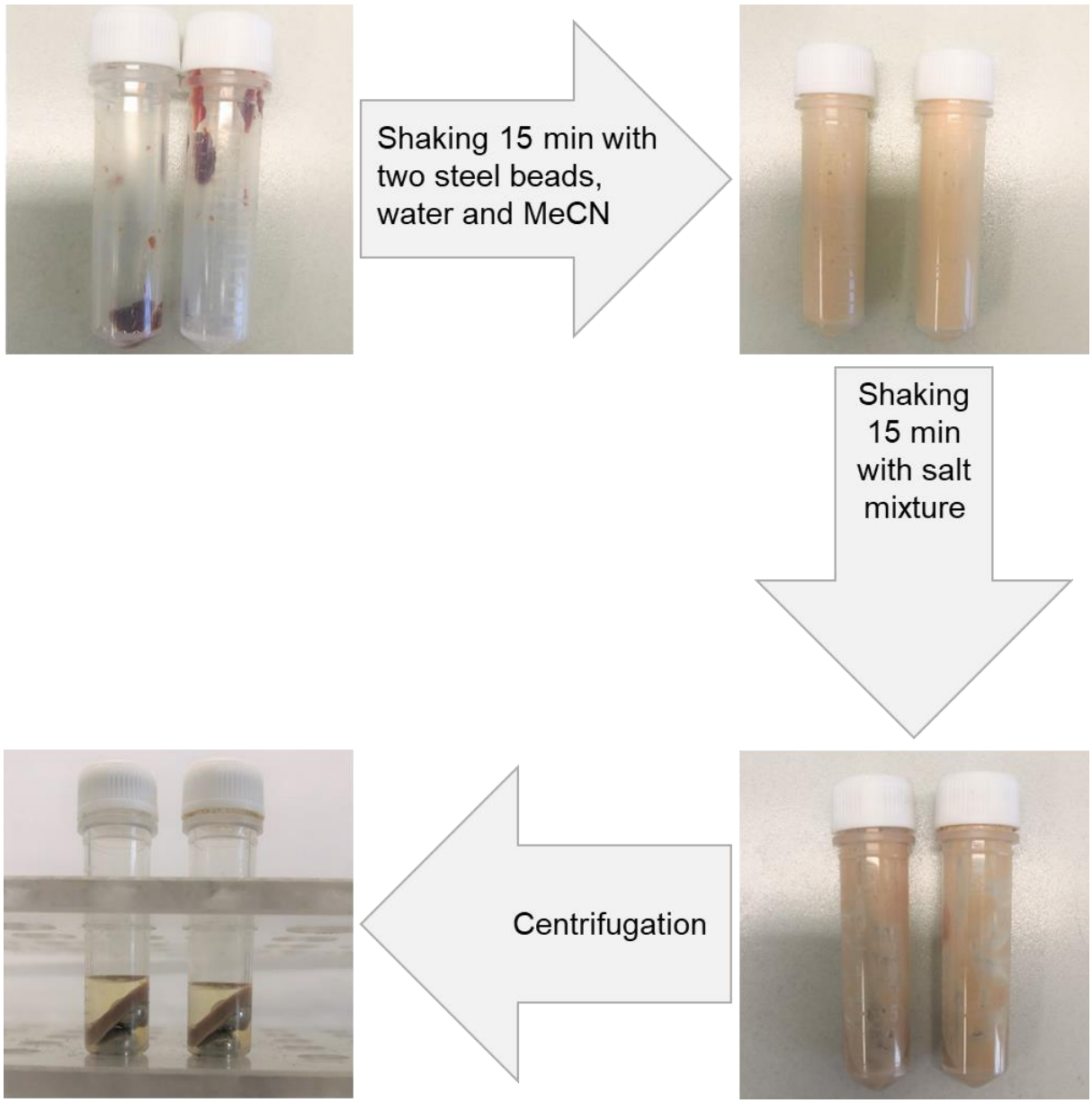
Analyte	Standard scale method			Micro scale method		
	LOQ	20 µg kg^{-1}	200 µg kg^{-1}	LOQ	20 µg kg^{-1}	200 µg kg^{-1}
Ametoctradin	7.9	6.1	3.9	*	14.4	12.4
Amisulbrom	4.4	5.6	3.2	6.3	9.7	5.3
Azoxystrobin	6.0	3.9	2.2	12.6	3.8	1.6
Bifenazate	n.d.	n.d.	n.d.	*	9.1	5.9
Bifenthrin	5.4	7.3	2.6	5.8	5.5	4.1
Boscalid	11.4	5.4	2.6	3.1	2.7	3.8
Carbetamide	n.d.	n.d.	n.d.	*	3.9	1.3
***cis*-Chlordane**	2.3	2.1	0.8	6.9	3.0	3.1
***trans*-Chlordane**	2.1	0.8	0.3	3.4	4.2	4.2
Chlorpyrifos	2.3	1.1	0.3	17.3	2.8	2.1
Chlorpyrifos-methyl	n.d.	n.d.	n.d.	6.7	1.8	1.7
Cyflufenamid	2.2	0.8	0.5	9.4	2.5	1.9
Cyhalothrin (sum)	1.3	1.5	0.8	2.4	3.3	2.4
Cypermethrin (sum)	1.5	1.6	0.9	3.2	4.7	2.7
***p,p'*-DDE**	1.4	1.2	0.2	4.6	3.8	4.5
***p,p'*-DDT**	n.d.	n.d.	n.d.	4.7	4.7	3.8
Deltamethrin	2.0	1.3	1.2	8.7	8.6	1.5
Desmedipham	n.d.	n.d.	n.d.	11.6	2.5	1.4
Dieldrin	1.2	1.7	0.4	8.4	1.8	3.5
Difenoconazole (sum)	1.0	0.9	1.1	8.2	6.5	7.5
Diflubenzuron	n.d.	n.d.	n.d.	3.8	6.0	4.2
Dimethomorph (sum)	1.0	1.0	0.9	4.1	5.3	5.4
Epoxiconazole	0.8	1.4	0.4	3.4	4.1	1.0
Fenazaquin	n.d.	n.d.	n.d.	6.7	15.3	7.4
Fenhexamid	1.7	2.2	0.2	9.0	3.8	2.7
Fenpropidin	n.d.	n.d.	n.d.	4.8	2.8	8.0
Fenpropimorph	1.4	0.8	0.5	1.8	3.2	4.0
Fenvalerate (sum)	1.5	1.7	0.6	3.8	5.3	2.4
Fipronil sulfone	1.5	1.4	0.4	5.1	2.6	1.3
Fluazifop-butyl	n.d.	n.d.	n.d.	7.8	11.6	11.2
Fludioxonil	1.1	0.9	0.5	7.0	2.6	1.4
Fluopyram	1.3	2.0	0.7	5.0	3.1	2.1
Flupyradifurone	n.d.	n.d.	n.d.	*	4.1	4.1
***tau*-Fluvalinate (sum)**	1.7	1.9	1.0	3.9	4.8	3.6
***gamma*-HCH (Lindane)**	0.8	1.1	0.4	4.5	2.0	3.4
Imidacloprid	*	6.7	3.6	*	9.1	3.3
Lenacil	1.9	0.9	0.5	7.3	3.7	2.9
Metazachlor	1.9	1.4	1.0	7.0	3.8	1.4
Metrafenone	1.4	1.3	0.4	2.2	5.0	1.0
Myclobutanil	1.1	1.2	0.4	2.4	1.9	1.9
Oxychlordane	n.d.	n.d.	n.d.	7.8	3.0	3.2
Pentachloronitrobenzene	3.1	1.1	0.3	7.2	4.9	2.0
Permethrin (sum)	1.6	1.5	0.7	2.2	5.0	1.8
Picolinafen	2.7	1.1	0.3	10.4	17.1	6.2
Pirimicarb	0.7	1.1	0.6	3.9	2.3	2.4
Propiconazole (sum)	1.1	1.0	0.3	3.5	2.1	1.7
Pyrimethanil	3.2	1.0	1.1	5.3	19.6	13.5
Quinoxyfen	1.6	0.7	0.5	6.8	11.9	8.2
Spirodiclofen	1.3	1.2	0.8	6.2	3.0	1.2
Tebuconazole	1.1	1.5	0.9	3.7	2.5	0.8
Terbuthylazine	n.d.	n.d.	n.d.	7.0	7.4	5.1
Terbuthylazine-desethyl	n.d.	n.d.	n.d.	7.3	8.9	4.7
Tetraconazole	1.1	1.3	0.5	5.8	2.7	1.7

Supplementary Table 2 (continued)

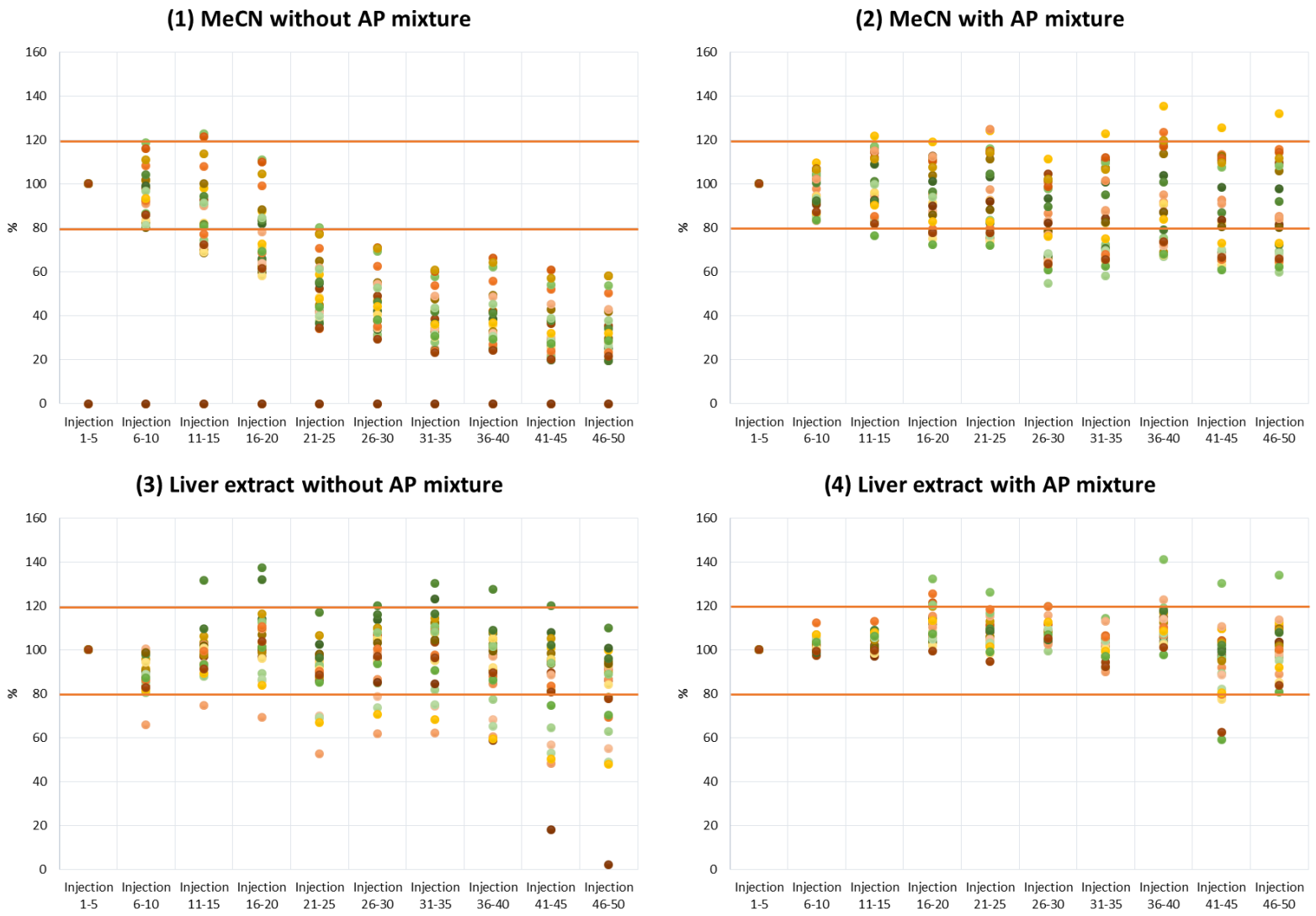
Analyte	Standard scale method			Micro scale method		
	LOQ	20 µg kg^{-1}	200 µg kg^{-1}	LOQ	20 µg kg^{-1}	200 µg kg^{-1}
Thiabendazole	*	2.5	1.2	n.d.	n.d.	n.d.
Tolclofos-methyl	1.3	1.8	0.4	5.7	3.3	1.7
Zoxamide	1.0	1.0	1.0	5.8	3.7	3.7



Supplementary Figure 1 Extraction procedure for 5 g samples.



Supplementary Figure 2 Extraction method for 100 mg samples.



Supplementary Figure 3 Evaluation of analyte protectants. Every experiment setting included 50 injections. The dots show the relative area of each analyte. The experiments clearly show the smallest deviations for experiment setting (4), liver extract with AP mixture.

4. Pesticide and persistent organic pollutant exposure of bats in Germany

Schanzer, S.; Koch, M.; Kiefer, A.; Jentke, T.; Veith, M.; Bracher, F.; Bracher, J.; Müller, C., Pesticide and persistent organic pollutant exposure of bats in Germany.

The article was submitted to Chemosphere as shown here and is currently under revision. Moderate revisions were requested by the reviewers.

4.1. Topic

Many non-target animals with various habitats may be exposed to pesticide contamination, as the active substances are used on arable land, in forestry and horticulture, but also in home gardens.[12] Pesticide exposure and its possible adverse effects are known for a manifold of animals, such as fish,[94] birds,[95] insects,[96] and bats.[90] As all bats native in Germany are protected animals, their pesticide and pollutant load was of a special interest.

As the bat species native in Germany are small animals with a body weight of 3-40 g,[97] the sample size was very limited, which needed to be addressed during sample preparation. Hence, to be able to investigate pesticide residues in bat liver tissue, a micro QuEChERS method, detecting up to 209 pesticides and pollutants, was developed (see Chapter 3.).[92] Nearly 400 specimens of five bat species (*Eptesicus serotinus, Myotis myotis, Nyctalus noctula, Pipistrellus pipistrellus, Plecotus auritus*), that were sampled by cooperation partners all over Germany, were analysed with this multiresidue method. The chosen species differed regarding size, body weight, migratory behaviour, hunting grounds, and prey, so that we could depict as broad a spectrum of the Vespertilionidae family as possible. For example, *M. myotis* prefers woodlands as a foraging area, whereas *P. pipistrellus* hunts on woodlands, farmlands, but also urban areas.[97] It was possible to detect both persistent organic pollutants, that were banned decades ago (PCBs, DDT, dieldrin), and pesticides that are currently approved for application in Germany (such as tetraconazole) or were approved at the time of sampling (*e.g.* chlorpyrifos). The graphical abstract of this article, showing some of the abovementioned compounds, is shown in **Fig. 16**.

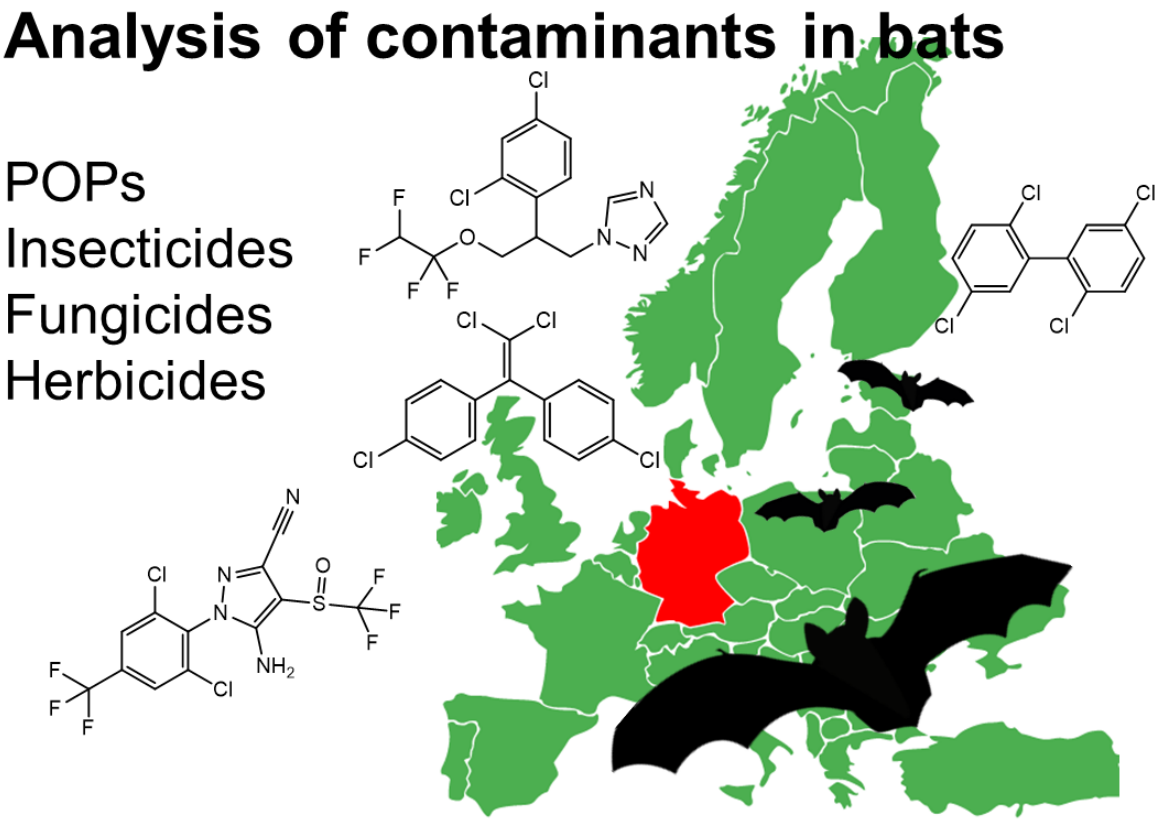


Figure 16 The graphical abstract of the article shows the origin of the samples, the distribution area of the bats (all of them are native in Central Europe), and examples of detected substances.

Wherever possible, data was collected for every animal, including age, sex, place and time of sampling, death circumstances and the bat season (*e.g.* hibernation, migrating/ swarming or mating). The liver samples, that were provided by Martin Koch of the Chair of Biogeography of Trier University who dissected the animals with the help of Thalia Jentke, were screened for pesticides and pollutants with the developed GC/MS-MS method. Subsequently, the investigated pesticide load was correlated to the metadata of each animal. Mainly, correlations between a certain pesticide group and the respective bat species were investigated. In addition to descriptive analyses, also statistical tests (Kruskal-Wallis tests and Wilcoxon tests with Bonferroni correction) were applied. Additionally, possible differences between males and females, juvenile and adult animals, and bat seasons were investigated with the same statistical tests.

In conclusion, the following work presents the hitherto largest dataset on pesticide exposure of bats in Germany and Central Europe, underpinning the importance of pesticide monitoring for environmental protection.

4.2. Personal contribution

My contribution to this article was the conceptualization, including the preliminary development and validation of the analytical method that was presented in Chapter 3. Furthermore, I conducted all sample preparations, instrumental analysis, and data analysis of the bat samples. I wrote the original draft of the manuscript and reviewed and incorporated the changes suggested by the co-authors.

Martin Koch provided the bat livers for sample preparation by collecting and dissecting the animals and collected the metadata for each specimen. He aided with the conceptualization of the project and reviewed the manuscript.

Dr. Andreas Kiefer was involved in the conceptualization and reviewed and edited the original draft. He also helped with the collection of bats.

Thalia Jentke supported the collection and dissection of the bat samples. Furthermore, she reviewed and edited the original draft.

Prof. Dr. Michael Veith provided resources for the project and supported the conceptualization. In addition, he reviewed and edited the original manuscript.

Prof. Dr. Franz Bracher provided resources for this work and reviewed and edited the original draft.

Dr. Johannes Bracher performed all statistical tests and visualized the descriptive analysis shown in the manuscript. He also provided resources and reviewed and edited the original draft of this work.

Dr. Christoph Müller contributed to the conceptualization of this project and was responsible for the supervision. In addition, he aided with the formal analysis of the gathered data. Furthermore, he was involved in the writing of the original manuscript and reviewed and edited the draft at later stages.

4.3. Article

The article is printed in its original wording as submitted to Chemosphere. The formatting and wording may slightly vary compared to the final journal article.

Pesticide and persistent organic pollutant exposure of bats in Germany

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Abstract

Bats are strictly protected throughout Europe. They are a highly diverse order of mammals in terms of body size, body weight, migratory behaviour, trophic niche specialisation and habitat use. The latter ranges from urban areas and arable land to forest. Due to their low reproductive rate, environmental stressors can have a major impact on bat populations. Pesticides in particular are discussed as an important driver of bat population declines. In this work, we analysed nearly 400 animals of five different species (*Eptesicus serotinus, Myotis myotis, Nyctalus noctula, Pipistrellus pipistrellus,* and *Plecotus auritus*) from all over Germany for residues of 209 pesticides and persistent organic pollutants. Residue analysis was conducted with a previously developed method using a miniaturized quick, easy, cheap, effective, rugged and safe (QuEChERS) sample preparation and gas chromatography-tandem mass spectrometry for separation and detection. These analytical data were statistically correlated with the known data on the animals (*e.g.* age, sex, place and time of finding). Of 209 pesticides and pollutants investigated, 28 compounds were detected, the most frequent being

organochlorine insecticides and polychlorinated biphenyls, which have been banned for decades by the Stockholm Convention on Persistent Organic Pollutants. Findings of more recent pesticides that were legally used for the last decade included azole antifungals and the insecticide fipronil. The bats contained between four and 25 different residues. In conclusion, this work provides the largest dataset of pesticide and persistent organic pollutant residues in European bats to date.

Keywords

Bats, environmental contaminants, pesticides, DDT, fipronil, tebuconazole

1. Introduction

In the light of a globally growing need for food, conventional farming relies on the application of pesticides to prevent harvest losses from insects, fungi, and weeds. This use of pesticides has been steadily increased over the last decades (German Federal Office of Consumer Protection and Food Safety, 2020; Sharma *et al.*, 2020b). In Germany, the average use of pesticides per hectare cropland increased by 27.5% from 2.98 kg in 2000 to 3.80 kg in 2019 (FAO, 2021). However, it is well-known that pest control can have a negative impact on our environment (Sharma *et al.*, 2020a).

The most prominent compound from the history of crop protection surely is dichlorodiphenyltrichloroethane (DDT), an organochlorine (OC) insecticide (see 3.1.2.) which can, apart from plant protection, effectively help to prevent outbreaks of malaria by eliminating the disease carrier insect, but also is highly persistent in the environment and has adverse effects on human and animal health (UN Environment Programme, 2019). DDT is considered to be an endocrine disruptor and a possible carcinogen, increasing the risk for mammary cancer, for example (Turusov *et al.*, 2002). Especially birds suffer from a DDT exposure, as this compound and its main metabolite dichlorodiphenyldichloroethylene (DDE) lead to a thinning of their eggshells, which often results in a higher mortality of the hatch (Turusov *et al.*, 2002). In addition, fish may also show reduced reproduction rates associated with DDT (Turusov *et al.*, 2002). Because of its environmental behaviour, DDT was later classified as one of the persistent organic pollutants (POPs).

POPs are a heterogenous group of chemicals, mostly organochlorine insecticides (see 3.1.2.) like DDT or industrial chemicals like polychlorinated biphenyls (PCBs; see 3.1.1.), that have very long half-lives, accumulate in adipose tissue, and are transported through water or in the atmosphere (UN Environment Programme, 2019). Thus, even though DDT was prohibited by

many countries in the 1970s, it can still be found up to today, even in the Arctic, where it was never used as an active substance (Ashraf, 2017; UN Environment Programme, 2019). A more recent compound is imidacloprid, a neonicotinoid insecticide which was first synthesized in 1985. It was approved for agricultural use in the European Union until December 2020 and belongs to the most frequently used insecticides worldwide (Simon-Delso *et al.*, 2015; European Commission, 2021). In Germany, neonicotinoids were the second-to-most used insecticides after carbamates in 2019 (German Federal Office of Consumer Protection and Food Safety, 2020). The public awareness for this compound group dramatically rose after its fatal effects on bees became known (Di Prisco *et al.*, 2013; European Food Safety Authority, 2013; Simon-Delso *et al.*, 2015). Evidently, apart from humans, birds or beneficial insects, many other non-target animals can be affected by pesticides as well (Dowding *et al.*, 2010; Taylor *et al.*, 2020).

Recently, the EFSA (European Food Safety Authority) Panel on Plant Protection Products and their Residues (PPR) assessed the coverage of bats by the current pesticide risk assessment for birds and mammals. The assessment concludes that “bats are not adequately covered by the current risk assessment approach, and that there is a need to develop a bat-specific risk assessment scheme” (EFSA PPR *et al.*, 2019). Bats may be especially sensitive to pesticides due to their high metabolic rate (high food intake) and longevity (risk of bioaccumulation of pesticide components or their residues) (Oliveira *et al.*, 2021). At the same time, a low reproductive rate with only one to two offspring per year make bats vulnerable on a population level, since exposure to pesticides lowers reproductive success or survival (Stahlschmidt *et al.*, 2017).

The adverse effects of pesticides and POPs on bats have already been observed for many decades. For instance, the bats’ immune system is very sensitive towards the presence of persistent organic pollutants, which are associated with a higher metabolic rate and thus, starving bats (Clark Jr and Stafford, 1981). Additionally, exposure to POPs is associated with white nose syndrome, a fungal disease that affects bats mostly during hibernation and increases their mortality (Kannan *et al.*, 2010). However, also more recently used insecticide classes like organophosphates or pyrethroids were detected in bat tissues and guano (O’Shea and Clark Jr, 2002; Eidels *et al.*, 2007). Modern insecticides are also known for their adverse effects on bats: Chlorpyrifos, a typical organophosphate compound, was shown to cause impaired flight, ataxia and spasm in bats even at low concentrations (Eidels *et al.*, 2016), and the neonicotinoid imidacloprid is suspected to cause a spatial memory disorder in echolocation bats (Hsiao *et al.*, 2016). The majority of these studies originate from outside Europe (Hsiao *et al.*, 2016; Kuzukiran *et al.*, 2021), predominantly the USA, and the most investigated species there is the big brown bat (*Eptesicus fuscus*) (Bayat *et al.*, 2014; Torquetti *et al.*, 2020). This insectivorous bat can be found all over North America, and its habitats are both near cities as

well as in rural areas (Agosta, 2002). However, there is little evidence on pesticide exposure of German (or even European) bat species, and available data only refer to organochlorine insecticides (Nagel and Disser, 1990; Streit *et al.*, 1995; Swanepoel *et al.*, 1999; Lüftl *et al.*, 2005); alternatively, pesticide analyses were performed indirectly by investigating the bats' habitats or prey (Stahlschmidt and Brühl, 2012; Brühl *et al.*, 2021). Hence, there is a strong need for a substantial dataset to broadly examine the pesticide and pollutant load of Chiroptera in Central Europe.

Currently, 288 active ingredients are approved for pest control in Germany, and their distribution highly depends on the respective land use (German Federal Office of Consumer Protection and Food Safety, 2020). In order to investigate the exposure to both recently approved pesticides (*e.g.* azoxystrobin, tebuconazole, see 3.1.4.) and forbidden pesticides or pollutants (*e.g.* dieldrin, DDT, PCBs, see 3.1.1. and 3.1.2.), we here examine the pesticide and POP load of five insectivorous bat species in Germany: serotine bat (*Eptesicus serotinus*), greater mouse-eared bat (*Myotis myotis*), common noctule bat (*Nyctalus noctula*), common pipistrelle (*Pipistrellus pipistrellus*), and brown long-eared bat (*Plecotus auritus*). They vary widely in size and habitat use as well as roosting, hibernation, migration behaviour and dietary requirements (see **Table 1**), and thus represent well the Central European bat community. We analysed nearly 400 specimens (see 2.1.) from all over Germany, sampled over the last 35 years. We screened for more than 200 pesticides and POPs (see 2.2.), thus providing the largest study of bats' pesticide and pollutant exposure in Germany to date.

2. Materials and methods

2.1. Sample collection

Bat samples (n = 387) were collected from ten federal states all over Germany. The carcasses were collected by animal caretakers or conservation authorities. Apart from animals found dead, there were also bats that died in care or were put to sleep by veterinarians due to their moribund condition. Depending on their occurrence and chance to be found by humans, the sample numbers highly vary between the different species (**Table 1**).

Table 1 Bat species studied: body mass, predominant diet, migratory category, and predominant foraging habitats (Dietz and Kiefer, 2020).

Code	Species	Body mass	Predominant diet	Migratory category	Predominant foraging habitat
▼	*E. serotinus* (n=74)	large (18-25 g)	beetles, moths, dipterans, hymenopterans	short distance	farmland, grass land, urban areas, forest edges
▲	*M. myotis* (n=17)	large (20-27 g)	beetles	medium distance	woodlands
◆	*N. noctula* (n=49)	large (12-30 g)	various flying insects (*e.g.* dipterans, beetles, moths)	long distance	open space
■	*P. pipistrellus* (n=215)	small (3-7 g)	small flying insects (*e.g.* dipterans)	short distance	woodlands farmland urban areas
●	*P. auritus* (n=32)	medium (6-9 g)	moths, dipterans	stationary	woodlands open landscapes

Data collected for each animal included age (adult n = 143, juvenile n = 37, unknown n = 207), sex (female n = 106, male n = 161, unknown n = 120), bat season (hibernation n = 70, post hibernation n = 45, mating n = 117, migration/ swarming n = 106, unknown n = 49), place, and circumstances of finding (death in care n = 111, found dead n = 80, other n = 48, unknown n = 148; **Figure 1,** for details see **Supporting Table S1)**. The deceased animals were collected and stored at -20 °C until dissection. The livers were used for pesticide and pollutant analysis as described below.

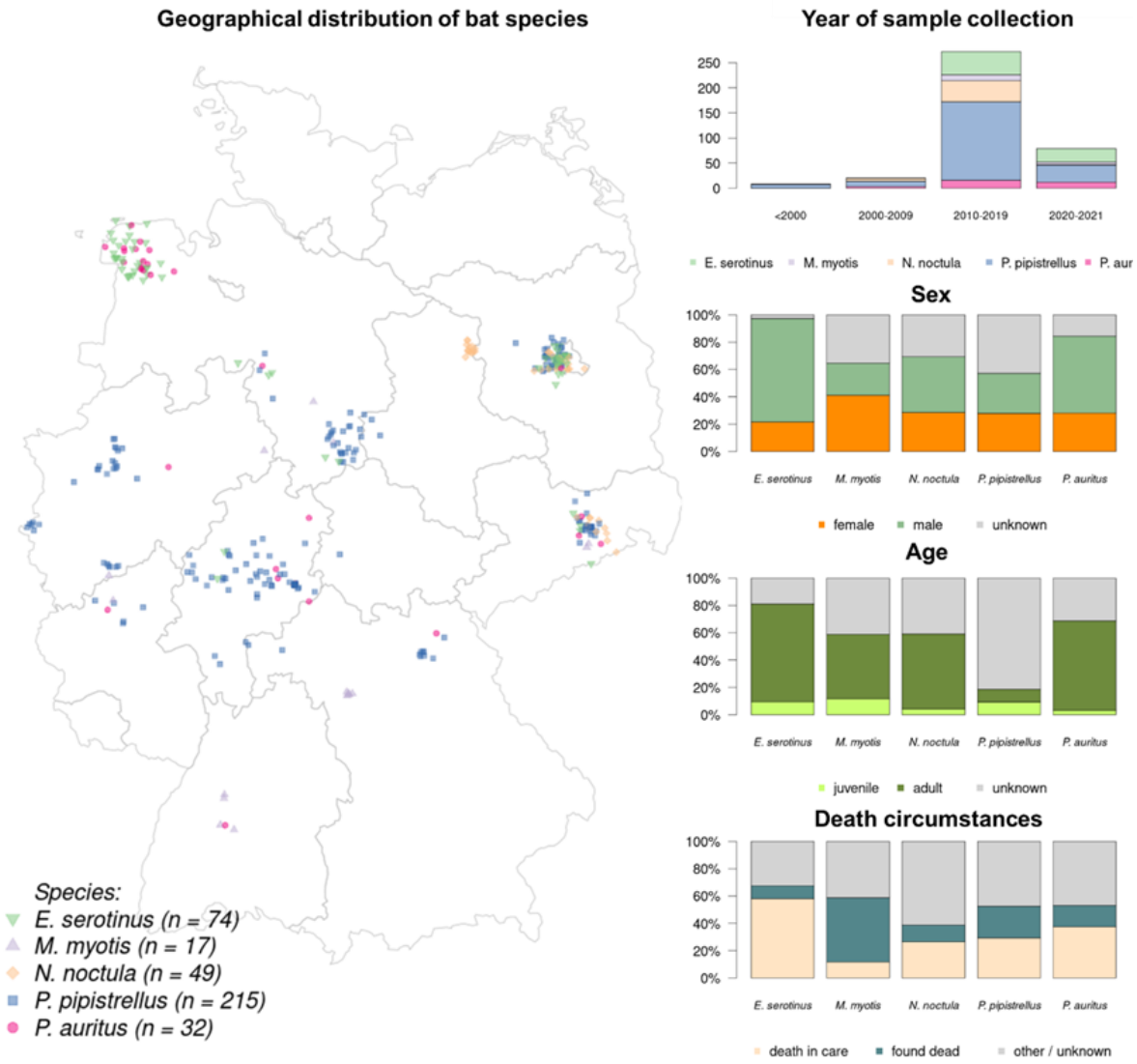


Figure 1 Description of the available samples: Left: Geographical distribution of bats in Germany. Right, from top to bottom: Year of collection, distribution of sex, age and death circumstances for the different species; "other" stands for put to sleep, death or injured from outside influence (*e.g.* wind turbines, cats).

2.2. Pesticide and pollutant analysis

The analysis of all bat samples was conducted with a miniaturized, QuEChERS-based sample preparation method, combined with a GC-MS/MS (gas chromatography-tandem mass spectrometry) system (Schanzer *et al.*, 2021). The micro QuEChERS sample preparation was developed due to the challenging matrix, and especially, the small sample size. To this end, 100 mg of liver were homogenized with 100 µL of water and 200 µL of 1% acetic acid in acetonitrile (*v/v*). After addition of 100 mg of a salt mixture consisting of anhydrous magnesium sulfate and sodium acetate (4:1), the sample was centrifuged for 5 min at 12,000 *g* and stored at -20 °C for 2 h. After this freezing-out step, used to remove fatty components, 100 µL of the supernatant were transferred to a tube containing 40 mg of the dSPE (dispersive solid phase extraction) mixture (anhydrous magnesium sulfate, PSA, C_{18} and GCB (150:25:20:5)) for

sample clean-up. After 20 s of vortexing and 5 min of centrifuging at 12,000 *g*, 50 µL of the final extract were transferred to an autosampler vial and 1.5 µL of analyte protectant mixture (consisting of shikimic acid, sorbitol, ethylglycerol and gulonolacton (EURL-SRM, 2013)) were added prior to GC-MS/MS analysis. A list of all analysed compounds (sum n = 209, among them: polychlorinated biphenyls (PCBs) n = 6, organochlorine (OC) insecticides n = 19, other insecticides n = 51, fungicides n = 66, herbicides n = 60, and others n = 7) can be taken from **Supporting Table S2**.

2.3. Statistical analyses

All statistical analyses were conducted using the R software for statistical computing (R Core Team, 2020). The package rgdal was used to generate maps (Bivand *et al.*, 2021). Colour scales are based on the RColorBrewer package (Neuwirth, 2014).

As the sample set in this work is diverse but cannot be considered a random sample from a well-defined population, the presented statistical analyses are mostly descriptive in nature (see 3.1.1. – 3.1.5.). To complete this study, we applied the Kruskal-Wallis test as implemented in the R-Package psych (Revelle, 2022) and pairwise Wilcoxon tests with Bonferroni correction for multiple testing (see 3.1.7.). However, these statistical tests must be assumed to be influenced by the specific composition of the available dataset, which due to the spatial clustering of the sample cannot be assumed to mirror the entire bat population in Germany.

3. Results and discussion

3.1. Pesticide and POP exposure by chemotypes and species

The investigated bat species were exposed to a variety of different pesticide types (**Figure 2**). In the following, the results are listed for each pesticide group, and compared to findings from other regions (see 3.1.1. – 3.1.5.). All given LOQ (limit of quantification) concentrations are taken from the previously published analytical method (Schanzer *et al.*, 2021). All median concentrations were calculated including the samples that exceeded the LOQ. Detailed information on the exposure of all 387 animals can be found in **Supporting Table S3**. The overall results are compared to a recent multiresidue study (Kuzukiran *et al.,* 2021) and discussed on the basis of statistical hypothesis testing (see 3.1.6. and 3.1.7.).

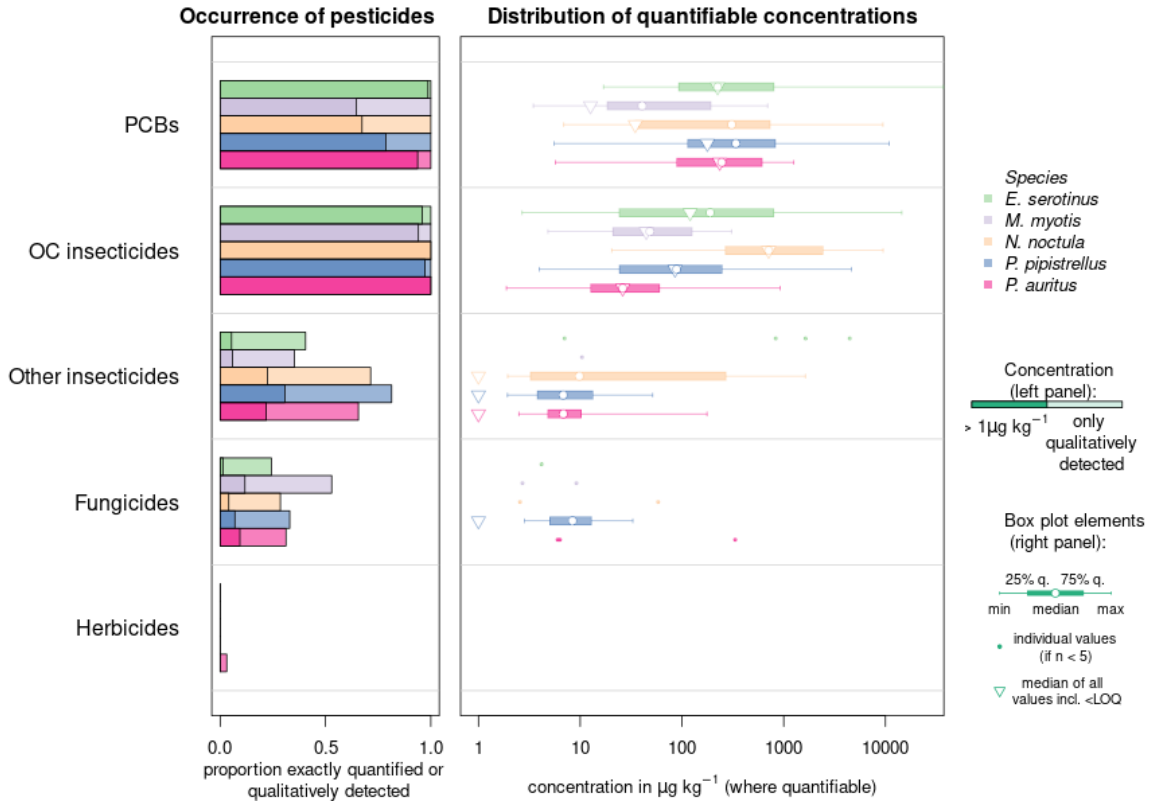


Figure 2 Pesticide concentrations by species. In each sub-figure, the barplots on the left show the proportion of samples in which a quantifiable amount of pesticides from a given group could be detected (dark bars) and the additional proportion in which they were only detected qualitatively (light bars). Boxplots in the right panel show the distribution of the quantified values on a logarithmic scale. The minimum, 25% quantile, median, 75% quantile and maximum are shown. Additionally, the median across all samples, including bats where a pathogen was not or only qualitatively detected, is shown by a triangle. Whenever less than 5 values were available, these are shown as individual dots. A more detailed display for individual pesticide loads is available in **Supporting Figure S1**.

3.1.1. Polychlorinated biphenyls (PCBs)

Polychlorinated biphenyls (PCBs), with the formula $C_{12}H_{10-x}Cl_x$, are a group of halogenated aromatic compounds that belong to the persistent organic pollutants. These compounds are industrial chemicals and were formerly used as heat exchange fluids or paint additives, for example (UN Environment Programme, 2019). PCBs belong to the twelve initial POPs that were prohibited by the Stockholm Convention in 2001, also known as the “dirty dozen” (UN Environment Programme, 2019; Prabhu and Lakshmipraba, 2022). There are more than 200 congeners, mostly occurring as mixtures instead of pure substances, and their number corresponds to the degree of chlorination. PCBs with a low chlorination (1-5 Cl atoms, *e.g.* PCB 28, 52, 101) have shorter half-lives and are indicators for an acute exposure (German Environmental Specimen Bank, 2021a), whereas PCBs with a higher chlorination degree (6-10 Cl atoms, *e.g.* PCB 138, 153, 180) have long half-lives and are indicators for a bioaccumulation, for example through the food chain (German Environmental Specimen Bank, 2021b).

The six abovementioned PCBs (28, 52, 101, 138, 153, and 180, **Fig. 3**) are regarded as representative congeners (German Federal Ministry of Food and Agriculture, 2003) and therefore monitored in the investigated bat species.

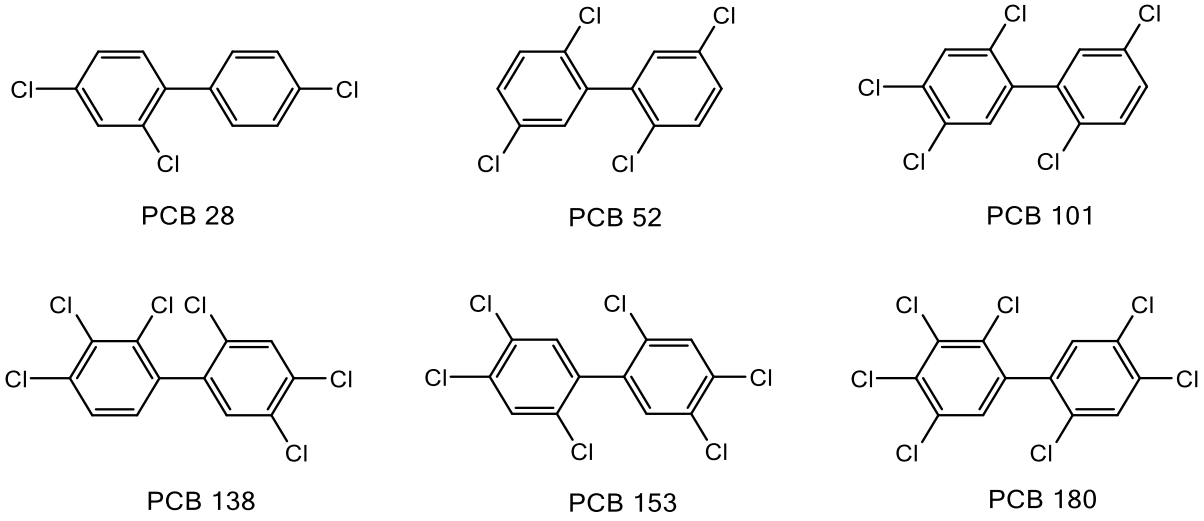


Figure 3 Detected polychlorinated biphenyls (PCBs). The congener number is given below the structure, respectively. Upper row shows low chlorinated compounds (1-5 Cl atoms), lower row shows high chlorinated compounds (6-10 Cl atoms).

Of 387 animals, every single bat was exposed to one or more PCBs (see **Supporting Figure S2**). About 98% of the animals were exposed to the low chlorinated PCBs (**Table 2**), and there was just one animal without an exposure to high chlorinated PCBs (*M. myotis*, No. 3073, see **Supporting Table S1** and **Supporting Table S3**). The congeners 138, 153 and 180 were present in clearly higher concentrations than the congeners 28, 52 and 101, indicating a bioaccumulation of the PCBs. The median concentration of the summed PCBs found in the bat samples was 276 µg kg^{-1} (medians of the single substances: PCB 28 (3 µg kg^{-1}) PCB 52 (4 µg kg^{-1}) PCB 101 (2 µg kg^{-1}) PCB 138 (84 µg kg^{-1}) PCB 153 (118 µg kg^{-1}) PCB 180 (73 µg kg^{-1})), but the investigated bat species were exposed to a highly different degree. The species with the lowest overall PCB exposure was *M. myotis*, a species that feeds predominantly on medium to large ground-dwelling forest beetles, such as Carabidae and Geotrupidae (Dietz and Kiefer, 2020). No animal had an exposure of low chlorinated PCBs that exceeded the quantification limit (1 µg kg^{-1}), and 65% of the investigated bats (**Table 2**) had an exposure to high chlorinated PCBs above the limit of quantification (LOQ). The highest exposure to low chlorinated PCBs was found in *P. auritus*. Here, about 34% of all samples exceeded the LOQ, and also the highest detected concentration of 514 µg kg^{-1} was found in a brown long-eared bat (No. 3559, see **Supporting Table S1** and **Supporting Table S3**). The most frequent exposure to high chlorinated PCBs was found in *E. serotinus*. Notably, 99% of these animals exceeded the LOQ, and the median content of PCBs 138, 153 and 180 was

223 µg kg^{-1}. The maximum content measured in a serotine bat was 37 mg kg^{-1} (No. 3465, see **Supporting Table S1** and **Supporting Table S3**).

A study from Spain in 1993 also revealed high concentrations of PCBs in two bat species, *Miniopterus schreibersii* and *Rhinolophus ferrumequinum*, after a Soxhlet extraction of the lipophilic substances and GC-ECD (gas chromatography-electron capture detector) analysis (Hernández *et al.*, 1993). Seven PCBs were investigated, and the average concentration was 0.6 mg kg^{-1}. However, it was not specified which PCBs were under investigation, which exacerbates a comparison of these values. A study from Austria, using a similar sample preparation method to analyse 149 bats, also investigated seven PCBs: the six PCBs mentioned in this study, and additionally PCB 118, with five chlorine atoms (Lüftl *et al.*, 2005). With median concentrations of 11 µg kg^{-1}, 10 µg kg^{-1}, 103 µg kg^{-1}, 180 µg kg^{-1} and 74 µg kg^{-1} (PCB 28, PCB 52, PCB 138, PCB 153 and PCB 180; PCB 101 was not found in this study) and a maximum of 25 mg kg^{-1} (sum of all seven PCBs), the findings correlate well with the concentrations found in this study. A more recent study from the USA also found a high exposure of PCBs in little brown bats (*Myotis lucifugus*) that were suffering from white nose syndrome (Kannan *et al.*, 2010). The samples were pooled and extracted with a Soxhlet apparatus and analysed with a GC-MS system. More than 100 PCBs were investigated, and the average concentration was 3 mg kg^{-1}. As the extracted matrix here was fat tissue, the main compartment of storage of the lipophilic POPs, it is probable that the concentration in liver tissue would be somewhat lower.

Table 2 Number of detected components in each bat species. n.d.: not detected; n.q: not quantifiable (analyte was not quantified at this point of the study); <LOQ: determined concentration was between LOD (limit of detection) and LOQ (limit of quantification); >LOQ: compound was quantifiable (LOQs see **Supporting Table S2**). Percentages are rounded (sum between 99-101%); qualitative analysis sum of not detected compounds (no) and detected compounds (yes), yes sum of n.q., <LOQ and >LOQ.

Compound			**Bat species**												
			E. serotinus		***M. myotis***		***N. noctula***		***P. pipistrellus***		***P. auritus***				**sum**
Class	**Name**		*(n = 74)*		*(n = 17)*		*(n = 49)*		*(n = 215)*		*(n = 32)*				(n = 387)
			n	[%]	**n**	[%]	**n**	[%]	**n**	[%]	**n**	[%]	**n**	[%]	qualitative [%]
PCBs	low chlorinated PCBs (sum of PCB 28, PCB 52, PCB 101)	n.d.	**0**	0	**1**	6	**1**	2	**5**	2	**0**	0	**7**	2	**no** 2
		n.q.	**0**	0	**6**	35	**14**	29	**43**	20	**2**	6	**65**	17	
		<LOQ	**62**	84	**10**	59	**20**	41	**138**	64	**19**	59	**249**	64	**yes** 98
		>LOQ	**12**	16	**0**	0	**14**	29	**29**	14	**11**	34	**66**	17	
	High chlorinated PCBs (sum of PCB 138, PCB 153, PCB 180)	n.d.	**0**	0	**1**	6	**0**	0	**0**	0	**0**	0	**1**	0	**no** 0
		n.q.	**0**	0	**5**	29	**14**	29	**45**	21	**2**	6	**66**	17	
		<LOQ	**1**	1	**0**	0	**2**	4	**1**	1	**0**	0	**4**	1	**yes** 100
		>LOQ	**73**	99	**11**	65	**33**	67	**169**	79	**30**	94	**316**	82	
OC insecticides	DDT (sum of *p,p'*-DDT and its metabolite *p,p'*-DDE)	n.d.	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**no** 0
		n.q.	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	
		<LOQ	**5**	7	**2**	12	**0**	0	**10**	5	**1**	3	**18**	5	**yes** 100
		>LOQ	**69**	93	**15**	88	**49**	100	**205**	95	**31**	97	**369**	95	
	Dieldrin	n.d.	**29**	39	**7**	41	**36**	74	**109**	51	**8**	25	**189**	49	**no** 49
		n.q.	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	
		<LOQ	**7**	9	**0**	0	**5**	10	**29**	14	**7**	22	**48**	12	**yes** 51
		>LOQ	**38**	51	**10**	59	**8**	16	**77**	36	**17**	53	**150**	39	
	HCH (sum *beta* and *gamma* isomer)	n.d.	**0**	0	**0**	0	**4**	8	**7**	3	**1**	3	**12**	3	**no** 3
		n.q.	**0**	0	**1**	6	**0**	0	**14**	7	**3**	9	**18**	5	
		<LOQ	**14**	19	**6**	35	**9**	18	**36**	17	**13**	41	**78**	20	**yes** 97
		>LOQ	**60**	81	**10**	59	**36**	74	**158**	73	**15**	47	**279**	72	
	Heptachlor	n.d.	**60**	81	**16**	97	**33**	67	**205**	95	**26**	81	**340**	88	**no** 88
		n.q.	**0**	0	**1**	6	**11**	22	**2**	1	**0**	0	**14**	4	
		<LOQ	**14**	19	**0**	0	**4**	8	**7**	3	**5**	16	**30**	8	**yes** 13
		>LOQ	**0**	0	**0**	0	**1**	2	**1**	1	**1**	3	**3**	1	
	Oxychlordane	n.d.	**19**	26	**7**	41	**35**	71	**76**	35	**12**	38	**149**	39	**no** 39
		n.q.	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	
		<LOQ	**17**	23	**2**	12	**7**	14	**56**	26	**8**	25	**90**	23	**yes** 61
		>LOQ	**38**	51	**8**	47	**7**	14	**83**	39	**12**	38	**148**	38	

Table 2 (continued)

Compound			Bat species													
Class	**Name**		***E. serotinus*** *(n = 74)*		***M. myotis*** *(n = 17)*		***N. noctula*** *(n = 49)*		***P. pipistrellus*** *(n = 215)*		***P. auritus*** *(n = 32)*		**sum** (n = 387)			
			n	[%]	**n**	[%]	**n**	[%]	**n**	[%]	**n**	[%]	**n**	[%]	**qualitative**	**[%]**
Other insecticides	Chlorpyrifos	n.d.	**62**	84	**17**	100	**42**	86	**206**	96	**24**	75	**351**	91	**no**	91
		<LOQ	**12**	16	**0**	0	**5**	10	**7**	3	**4**	13	**28**	7	**yes**	9
		>LOQ	**0**	0	**0**	0	**2**	4	**2**	1	**4**	13	**8**	2		
	Deltamethrin	n.d.	**69**	93	**17**	100	**48**	98	**214**	99	**27**	84	**375**	97	**no**	97
		<LOQ	**5**	7	**0**	0	**0**	0	**1**	1	**4**	13	**10**	3	**yes**	4
		>LOQ	**0**	0	**0**	0	**1**	2	**0**	0	**1**	3	**2**	1		
	Fipronil (sum of fipronil and fipronil sulfone)	n.d.	**52**	70	**11**	65	**15**	31	**43**	20	**13**	41	**134**	35	**no**	35
		<LOQ	**20**	27	**5**	29	**26**	53	**111**	52	**15**	47	**177**	46	**yes**	66
		>LOQ	**2**	3	**1**	6	**8**	16	**61**	28	**4**	13	**76**	20		
	Permethrin (sum of *cis* and *trans* isomer)	n.d.	**60**	81	**17**	100	**43**	88	**187**	87	**25**	78	**332**	86	**no**	86
		<LOQ	**12**	16	**0**	0	**2**	4	**19**	9	**6**	6	**39**	10	**yes**	14
		>LOQ	**2**	3	**0**	0	**4**	8	**9**	4	**1**	3	**16**	4		
Fungicides	Azoxystrobin	n.d.	**74**	100	**17**	100	**49**	100	**215**	100	**31**	97	**386**	100	**no**	100
		<LOQ	**0**	0	**0**	0	**0**	0	**0**	0	**1**	3	**1**	0	**yes**	0
		>LOQ	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0		
	Difenoconazole (sum of isomers)	n.d.	**74**	100	**17**	100	**48**	98	**215**	100	**31**	97	**385**	99	**no**	99
		<LOQ	**0**	0	**0**	0	**1**	2	**0**	0	**1**	3	**2**	1	**yes**	1
		>LOQ	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0		
	Dimethomorph (sum of isomers)	n.d.	**74**	100	**17**	100	**48**	98	**214**	99	**31**	97	**384**	99	**no**	99
		<LOQ	**0**	0	**0**	0	**1**	2	**1**	1	**1**	3	**3**	1	**yes**	1
		>LOQ	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0		
	Epoxiconazole	n.d.	**65**	88	**11**	65	**48**	98	**202**	94	**24**	75	**350**	90	**no**	90
		<LOQ	**8**	11	**4**	24	**0**	0	**12**	6	**7**	22	**31**	8	**yes**	10
		>LOQ	**1**	1	**2**	12	**1**	2	**1**	1	**1**	3	**6**	2		
	Fenpropimorph	n.d.	**64**	86	**15**	88	**37**	76	**178**	83	**26**	81	**320**	83	**no**	83
		<LOQ	**10**	14	**2**	12	**11**	22	**35**	16	**5**	16	**63**	16	**yes**	17
		>LOQ	**0**	0	**0**	0	**1**	2	**2**	1	**1**	3	**4**	1		
	Propiconazole (sum of isomers)	n.d.	**74**	100	**17**	100	**49**	100	**208**	97	**31**	97	**379**	98	**no**	98
		<LOQ	**0**	0	**0**	0	**0**	0	**4**	2	**0**	0	**4**	1	**yes**	2
		>LOQ	**0**	0	**0**	0	**0**	0	**3**	1	**1**	3	**4**	1		
	Tebuconazole	n.d.	**64**	86	**16**	94	**46**	94	**197**	92	**25**	78	**348**	90	**no**	90
		<LOQ	**10**	14	**1**	6	**2**	4	**9**	4	**4**	13	**26**	7	**yes**	10
		>LOQ	**0**	0	**0**	0	**1**	2	**9**	4	**3**	9	**13**	3		
	Tetraconazole	n.d.	**58**	78	**13**	76	**45**	92	**182**	85	**22**	69	**320**	83	**no**	83
		<LOQ	**16**	22	**4**	24	**4**	8	**31**	14	**9**	28	**64**	17	**yes**	18
		>LOQ	**0**	0	**0**	0	**0**	0	**2**	1	**1**	3	**3**	1		
Herbicides	Picolinafen	n.d.	**74**	100	**17**	100	**49**	100	**215**	100	**31**	97	**386**	100	**no**	100
		<LOQ	**0**	0	**0**	0	**0**	0	**0**	0	**1**	3	**1**	0	**yes**	0
		>LOQ	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0	**0**	0		

3.1.2. Organochlorine (OC) insecticides

The organochlorines (OCs) have a special position among the insecticides, as they all belong to the POPs as well (UN Environment Programme, 2019). Despite their prohibition decades ago, they are still present in the environment. Just like the PCBs, the OCs could be found in every single animal under investigation (see **Supporting Figure S2**). There were five different analytes of the organochlorine type that could be found (**Fig. 4**): DDT (sum parameter of *p,p'*-DDT and its metabolite *p,p'*-DDE), dieldrin, hexachlorocyclohexane (HCH, sum of *beta* and *gamma* isomer), heptachlor, and oxychlordane (metabolite of chlordane). The most frequently found compound was DDT (or its metabolite DDE), which could be detected within all samples, exceeding the LOQ of 1 µg kg^{-1} in 95% of all samples (**Table 2**). The concentrations highly differed, ranging from 1 µg kg^{-1} to 14 mg kg^{-1}. HCH could be found in 97% of all bats, in 72% of the animals the concentration was above the LOQ of 1 µg kg^{-1}. Dieldrin and oxychlordane were both found approximately in half of the animals, and heptachlor was detected in 12% of all bat samples.

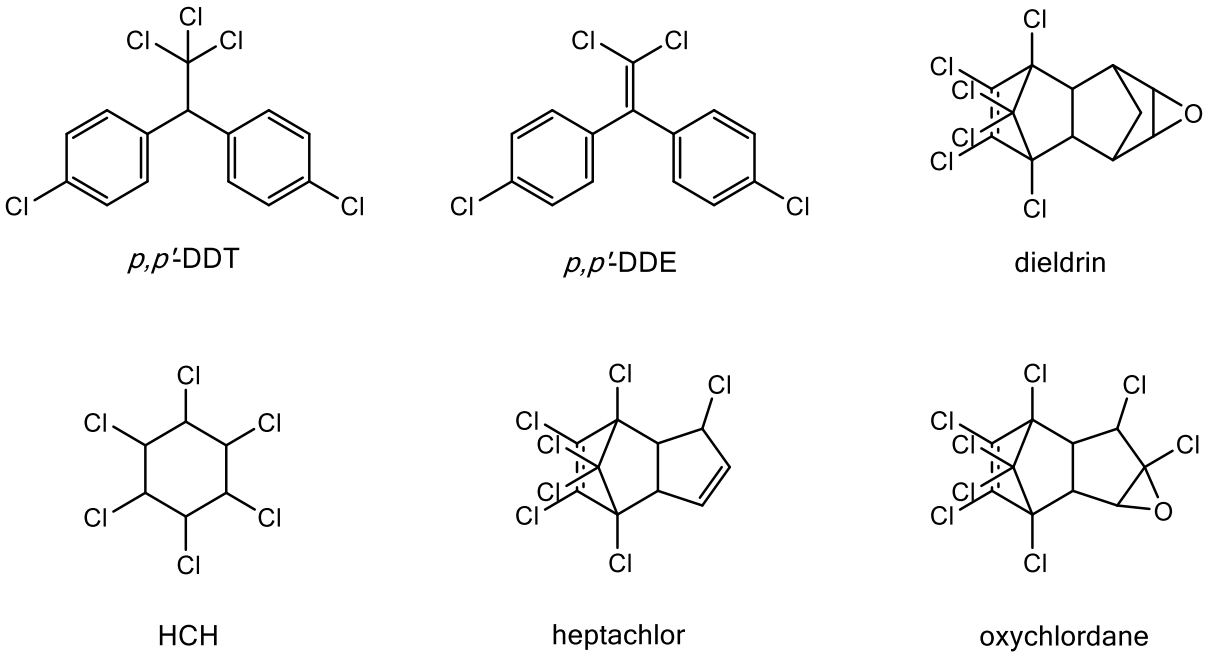


Figure 4 Detected organochlorine insecticides. The names are shown below the respective structure. All structures are shown without specified stereochemistry.

Plecotus auritus and *Myotis myotis* were the species with the lowest overall exposure: *P. auritus* showed the lowest median concentration of the summed up OC insecticides, but *M. myotis* overall had the smallest proportion of exposed animals. Even though a high percentage of *M. myotis* was exposed to dieldrin, the median concentration was rather low (6 µg kg^{-1}). For all other compounds, *M. myotis* had the lowest detected concentrations of all samples. For example, heptachlor could not be detected in noticeable concentrations, and the

highest concentration for HCH was 7 µg kg^{-1}. The highest median concentrations for oxychlordane (12 µg kg^{-1}) and for DDT sum (710 µg kg^{-1}) were found in noctule bats, and the analysis of *N. noctula* samples also revealed a frequent exposure of the species to heptachlor, with one third of all samples containing residues of this compound (**Table 2**). All other species showed a heptachlor exposure in less than 20% of the samples.

Interestingly, most of our *N. noctula* samples come from Eastern Germany (former German Democratic Republic), where DDT was phased out from 1970 on but used until 1988 (Heinisch *et al.*, 1993). In this part of Germany, the background contamination of the environment with DDT (and its metabolite DDE) is still much higher than in Western Germany, as evidenced by the analyses of the German Environmental Species Bank on roe deer (*Capreolus capreolus*), zebra mussels (*Dreissena polymorpha*), bream (*Abramis brama*) and suspended sediment loads in limnic ecosystems (German Environmental Specimen Bank, 2022; see also Abb *et al.,* 2010).

E. serotinus showed the highest percentage of samples containing HCH with 81%. Also, serotine bats contained the maximum detected concentrations for DDT (14 mg kg^{-1}; No. 3272 and 3509), dieldrin (267 µg kg^{-1}; No. 3458) and HCH (566 µg kg^{-1}; No. 3485), respectively (see **Supporting Table S1** and **Supporting Table S3**).

The abovementioned studies (Hernández *et al.*, 1993; Lüftl *et al.*, 2005; Kannan *et al.*, 2010) also investigated organochlorine insecticides. Hernández *et al.* found residues of DDT (sum parameter; average 2.3 mg kg^{-1}), dieldrin (average 66 µg kg^{-1}) and HCH (only the *delta* isomer was found in quantifiable concentrations; average 43 µg kg^{-1}). Lüftl *et al.* found residues of lindane (the samples were not screened for other HCH isomers; median 12 µg kg^{-1}) and DDT (sum with DDE; median 131 µg kg^{-1}). Kannan *et al.* found residues of DDT (sum parameter; average 2.4 mg kg^{-1}), chlordanes (average 340 µg kg^{-1}) and HCH (only *alpha*-HCH could be detected with an average of 1 µg kg^{-1}). Dieldrin could not be detected in their bat samples.

3.1.3. Other insecticides

Apart from the OC insecticides, other insecticide chemotypes were part of the analytical method as well, such as pyrethroids, organophosphates, carbamates or triazines (n = 53, **Supporting Table S2**). Within the bat samples, it was possible to detect the pyrethroids deltamethrin and permethrin, the organophosphate chlorpyrifos and the phenylpyrazole fipronil (sum parameter with the metabolite fipronil sulfone, see **Supporting Figure S2**). The structures can be seen in **Fig. 5**. Most of the non-OC insecticide chemotypes are still legally approved and applied in recent days and therefore of particular interest. Both permethrin and fipronil are used in veterinary medicine (Gupta and Anadón, 2018; Rosumeck *et al.*, 2018),

and even though the approval of chlorpyrifos ended in January 2020 (European Commission, 2021; University of Hertfordshire, 2021), a high percentage of the investigated bats was sampled before that date.

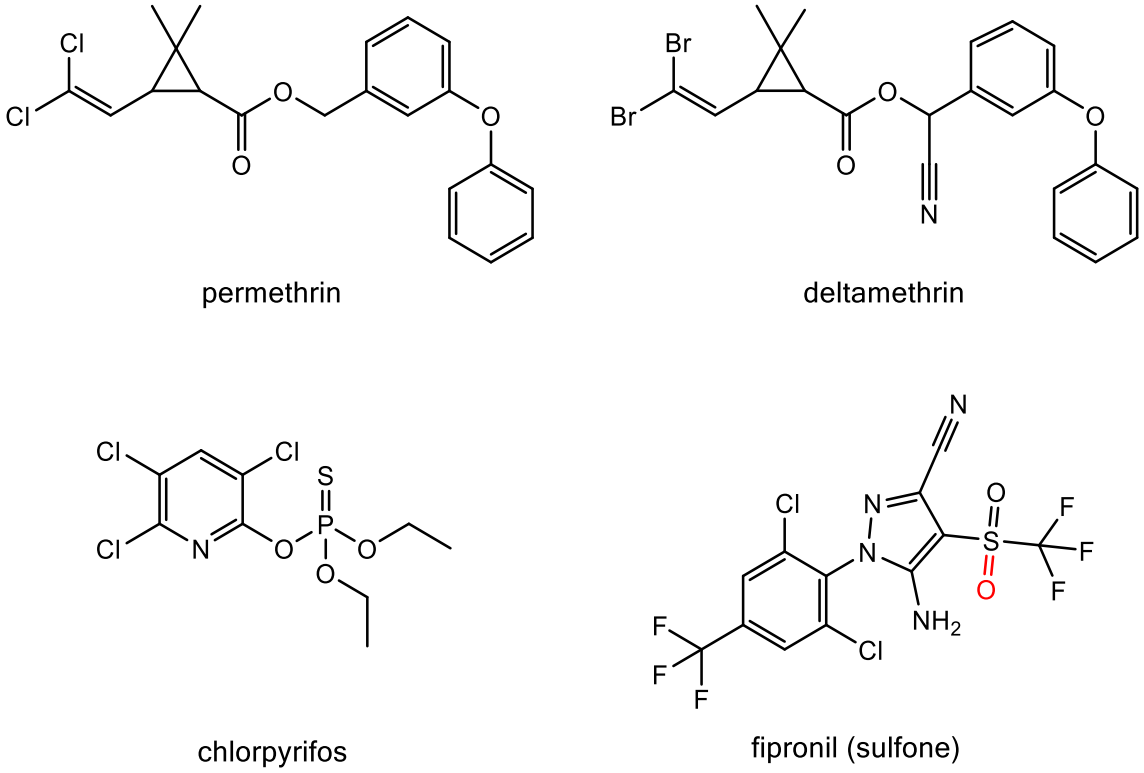


Figure 5 Different classes of detected non-organochlorine insecticides. The names are given under the respective structures. All structures are shown without defined stereochemistry. Addition of the red oxygen shows fipronil sulfone.

Fipronil (sulfone) was the most frequently detected compound, which could be found within 66% of all bat samples (**Table 2**). The common pipistrelles had the highest percentage of exposed animals with 80%, but the maximum concentration, which was more than 4 mg kg^{-1}, was found in a serotine bat (No. 3458, see **Supporting Table S1** and **Supporting Table S3**). The pyrethroids were found much less frequently, around 15% of all sampled bats were exposed to deltamethrin and/or permethrin. One specimen each of *N. noctula* (No. 3486) and *P. auritus* (No. 3300) were exposed to deltamethrin above the LOQ of 4 µg kg^{-1}, respectively, both with a concentration of about 50 µg kg^{-1}. For permethrin, the exposure was higher among the noctule bats, with a median concentration of 485 µg kg^{-1}, and a maximum concentration of 1.6 mg kg^{-1} (*N. noctula,* No. 3123). Again, *M. myotis* was the species with the lowest overall exposure. The only detected pesticide was fipronil sulfone.

None of the sampled animals showed an exposure to a carbamate insecticide, such as pirimicarb, which is frequently used to control aphids (University of Hertfordshire, 2021).

Neither was there a detectable exposure of the bats to the potentially harmful neonicotinoid insecticides, such as imidacloprid or thiacloprid.

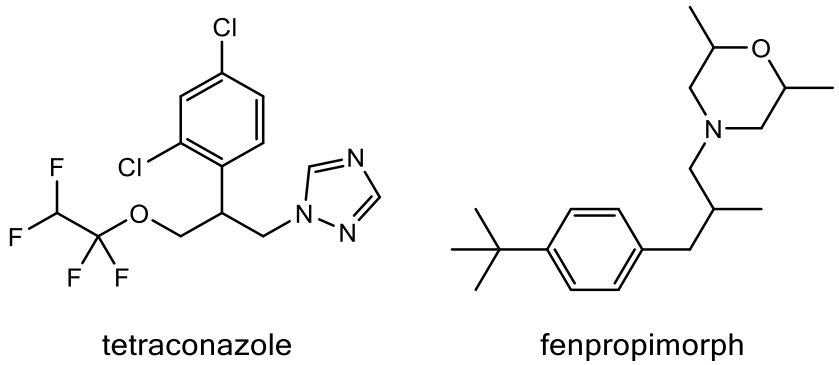
The effects of pyrethroids on *P. pipistrellus* were studied in experiments where the bats were roosting in boxes treated with permethrin, as this compound is frequently used for wood treatment (Racey and Swift, 1986; Shore *et al.*, 1991). Even though the experiments could not prove any harm done to the pipistrelle bats, data show that the LD_{50} (lethal dose 50%) of permethrin is 25 times lower for the little brown bat (*Myotis lucifugus*) than for rats (O'Shea and Clark Jr, 2002), revealing a higher vulnerability of the Chiroptera compared to the Rodentia. In 2017, permethrin residues were found in two Geoffroy's bats (*Myotis emarginatus*) in similar concentrations to specimen No. 3123 (1.6 mg kg^{-1}) in this study. Especially high concentrations of 320 mg kg^{-1} could be found in wood shavings of their roosts in the Netherlands (Janssen *et al.*, 2017).

Thus far, there are no specific studies dealing with the toxicity of fipronil to bats. However, many studies indicate that there is an increased health risk to mammals caused by this insecticide. Observed effects in mammals include decreased reproduction rate, reduced weight gain and also reduced developmental effects of the offspring, which have been studied primarily in rats (Gibbons *et al.*, 2015). Due to the already low reproductive rate of bats, this adverse effect is to be considered especially critical.

3.1.4. Fungicides

Fungicides are among the most used pesticides in agriculture, as many plant diseases originate in fungal infections (Price *et al.*, 2015). The analytical method covered more than 60 different fungicides of various chemotypes, such as azoles, strobilurins, morpholines, or anilides (**Supporting Table S2**).

The investigated bat samples contained eight different fungicides: five azoles (difenoconazole, epoxiconazole, propiconazole, tebuconazole, and tetraconazole), the strobilurin azoxystrobin, the morpholine fenpropimorph, and the anilide dimethomorph (see **Supporting Figure S2**). Tetraconazole and fenpropimorph (**Fig. 6**) were detected in 17% of all bat samples, but mostly below the respective limit of quantification (2 µg kg^{-1} and 4 µg kg^{-1}). Tebuconazole and epoxiconazole were the fungicides that most frequently exceeded their LOQs (2 µg kg^{-1}, 3% of samples and 1 µg kg^{-1}, 2% of samples, respectively). Azoxystrobin, difenoconazole, and dimethomorph could not be detected above the LOQ and occurred in less than 1% of the bat samples.



tetraconazole fenpropimorph

Figure 6 The most frequently detected fungicides: the azole fungicide tetraconazole, and the morpholine fungicide fenpropimorph. All structures are shown without defined stereochemistry.

Proportionally, the most epoxiconazole containing samples were greater mouse-eared bats; 24% of the animals showed traces of the compound (<LOQ) and 12% contained quantifiable, but small concentrations (median 5 µg kg^{-1}). As *M. myotis* predominantly feeds on forest beetles, the bats most likely were exposed to the fungicide directly through their environment. The metadata reveal that similar percentages of the mouse-eared bats were found near arable land and forests, respectively (see **Supporting Figure S3**). The highest concentration of epoxiconazole, and all other fungicides, was found within one brown long-eared bat (No. 3300, see **Supporting Table S1** and **Supporting Table S3**), epoxiconazole with 88 µg kg^{-1} and for example propiconazole with 91 µg kg^{-1}.

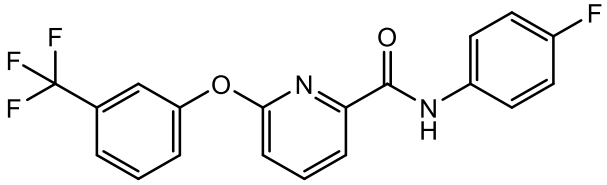
Three of the detected fungicides are by now prohibited in the European Union: epoxiconazole (since April 2020), fenpropimorph (since April 2019) and propiconazole (since December 2018). Also bats that were sampled after these dates showed an exposure to the three compounds, but as the usage period usually ends 1.5 years after the end of the approval, this does not indicate a misuse of the fungicides (Federal Office of Consumer Protection and Food Safety, 2021). However, two *P. pipistrellus* samples were exposed to propiconazole and overall, seven samples were exposed to fenpropimorph after the respective end of the usage period. As propiconazole has a DT_{50} (dissipation time for 50% of the compound in soil) value of 72 days and fenpropimorph of 35 days, both compounds are considered moderately persistent and therefore, the pesticide exposure can also originate from persisting residues (University of Hertfordshire, 2021). In addition, tebuconazole was also found in comparatively high concentrations in some *P. pipistrellus* samples in northern Bavaria. Propiconazole, fenpropimorph and tebuconazole are used for wood preservation, among other things. The common pipistrelle is a pronounced synantropic species, often roosting in crevices containing wood on the exterior façades of houses. This may indicate why this species may be locally exposed to exceptionally high concentrations of wood preservatives, so the contamination of the animals is likely to be roost induced.

There is no previous data about direct or indirect antifungal exposure of bats in Europe. An experimental study from Brazil was conducted with the frugivorous bat *Artibeus lituratus*, where male animals were fed tebuconazole-treated fruit for one week or one month, respectively (Machado-Neves *et al.*, 2018). After 30 days of treatment, histological alterations in the testes were observed, indicating a potential risk of adverse effects on reproduction. However, while this bat species feeds on fruit, which is frequently treated with antifungals, the bats native to Germany are insectivores, so such high exposure to tebuconazole seems unlikely.

Apart from the possible adverse effects of azoles, this group of antifungals has also been tested as a potential treatment against *Pseudogymnoascus destructans*, the fungus that causes white-nose syndrome in bats. Azoles that are used in human medicine were tested in therapeutic doses and showed efficacy against the pathogenic fungus (Chaturvedi *et al.*, 2011).

3.1.5. Herbicides

Only one herbicide of 60 detectable compounds (**Supporting Table S2**) could be found in the investigated bat samples: the pyridine derivative picolinafen (**Fig. 7**). The compound was detected in one *P. auritus* sample (No. 3300, see **Supporting Figure S2**, **Supporting Table S1,** and **Supporting Table S3**), but did not exceed the LOQ of 2 µg kg^{-1}.



picolinafen

Figure 7 The only detected herbicide picolinafen.

3.1.6. Comparison with recent findings

A recently published paper from Turkey reports the first multiresidue analysis performed on bats that also covers a high number of recently approved pesticides (Kuzukiran *et al.*, 2021). Although the work does not originate in Europe, it is very suitable to compare the results. Kuzukiran *et al.* overall investigated 42 animals of two bat species that are also part of this study *(P. pipistrellus* (n = 23), and *M. myotis* (n = 19)). These bats are also native to Asia Minor and are among the most common bat species in Turkey (UNEP/EUROBATS, 2021).

The sample preparation followed a SALLE (salt-assisted liquid-liquid extraction) protocol with a subsequent SPE (solid phase extraction) clean-up. The whole carcass (without head, skin, wings, and intestines), which weighed about 2 g, was used for sample preparation. The final extract was analysed with GC-MS, GC-MS/MS, and LC-MS/MS. The samples were screened for PCBs, PAHs, and all classes of pesticides.

Overall, Kuzukiran *et al.* detected a large number of residues in bat samples. Especially, several organophosphates and pyrethroids were found in nearly every sample, while the *M. myotis* samples from Germany did not contain any of these pesticide classes, and *P. pipistrellus* only contained the OP chlorpyrifos in 4% and permethrin and/or deltamethrin in 13% of all samples. The samples from Turkey did not contain low chlorinated PCBs, while the bats from Germany contained the PCBs 28, 52 and 101 in more than 90% of all investigated samples. Also, the findings of fungicides differed: Kuzukiran *et al.* detected 20 fungicides, while in this work, only eight antifungals were found. Common to both were epoxiconazole and tebuconazole. The variations in detected compounds can originate in the different legal regulations in the European Union and Turkey, and in different land use. However, some findings resemble: *p,p'*-DDE was the most common compound and was found in every sample, and also high chlorinated PCBs were frequently found in both locations. Neonicotinoids were detected neither in samples from Germany nor from Turkey.

3.1.7. Statistical analysis of pesticide and POP exposure by chemotypes and species

As shown in the previous descriptive analyses (see 3.1.1. – 3.1.5.), the median concentrations of individual compounds and compound groups highly differed between the bat species. In order to assess whether the observed differences in median values shown in **Figure 2** could also arise by chance, we applied statistical tests to our dataset. We applied these tests to all values including zero values and values below the limit of quantification (*i.e.,* the values represented by triangles). To compare all groups jointly, we applied the Kruskal-Wallis test. For PCBs and OC insecticides, the results indicate very strong evidence for differences between the bat species (p-values of 0.0003 and below). Pairwise Wilcoxon tests with Bonferroni correction for multiple testing indicate evidence that PCB values for *M. myotis* are below those of *E. serotinus*, *P. auritus* as well as *P. pipistrellus* (p-values of 0.04 and below), and values for *N. noctula* are below those of *E. serotinus* (p-value of 0.0002). For OC insecticides, there is strong evidence that values for *N. noctula* exceed those of all other species (p-values of 0.001 and below), and that those for *P. auritus* are below those of *E. serotinus* and *P. pipistrellus*. We consider these tests helpful to establish that the aforementioned differences between species are unlikely to be due to random fluctuations

only. However, we stress that any conclusions about an underlying population (*e.g.,* all bats in Germany) are problematic as the available sample is not a random sample from a well-defined population.

3.2. Statistical analysis of pesticide and POP distribution by age, sex, circumstances of death, and bat season

Apart from species-specific exposure, also potential differences between male and female, juvenile and adult, the circumstances of death (found dead *versus* died in care) and the bat season, *e.g.* hibernation and migration, were regarded. **Figure 8** shows the box plots of the different chosen criteria, analogous to **Figure 2**. Detailed information on the animals can be found in **Supporting Table S1**.

We applied again the Kruskal-Wallis test to the medians shown in the four panels of **Fig. 8** (as before, we used the medians of all values including zeros and values below the LOQ). In each analysis, we only used samples where the respective data was available (*i.e.,* we ignored samples with unknown sex when testing for differences between male and female bats). For certain combinations of stratification variable and type of pesticide there is evidence for different median levels of concentrations (between male and female bats for PCBs, p-value 0.001; between bats found dead and death in care for PCBs, p-value 0.04).
The joint Kruskal-Wallis test indicates evidence that concentrations of both PCBs and OC insecticides are not equal across all bat seasons (both with p-values of 0.01). Pairwise Wilcoxon tests with Bonferroni correction indicate moderate evidence for the following pairwise differences: The concentration of OC insecticides in samples from the migration/swarming period is lower than for the post-hibernation period (0.02). Moreover, the concentration of PCBs in samples from the post-hibernation period is below those for the hibernation and mating periods (p-values of 0.03 and 0.05, respectively). However, we again stress that the presented results should be read as a description of the available sample, which is not representative of a clearly defined population.

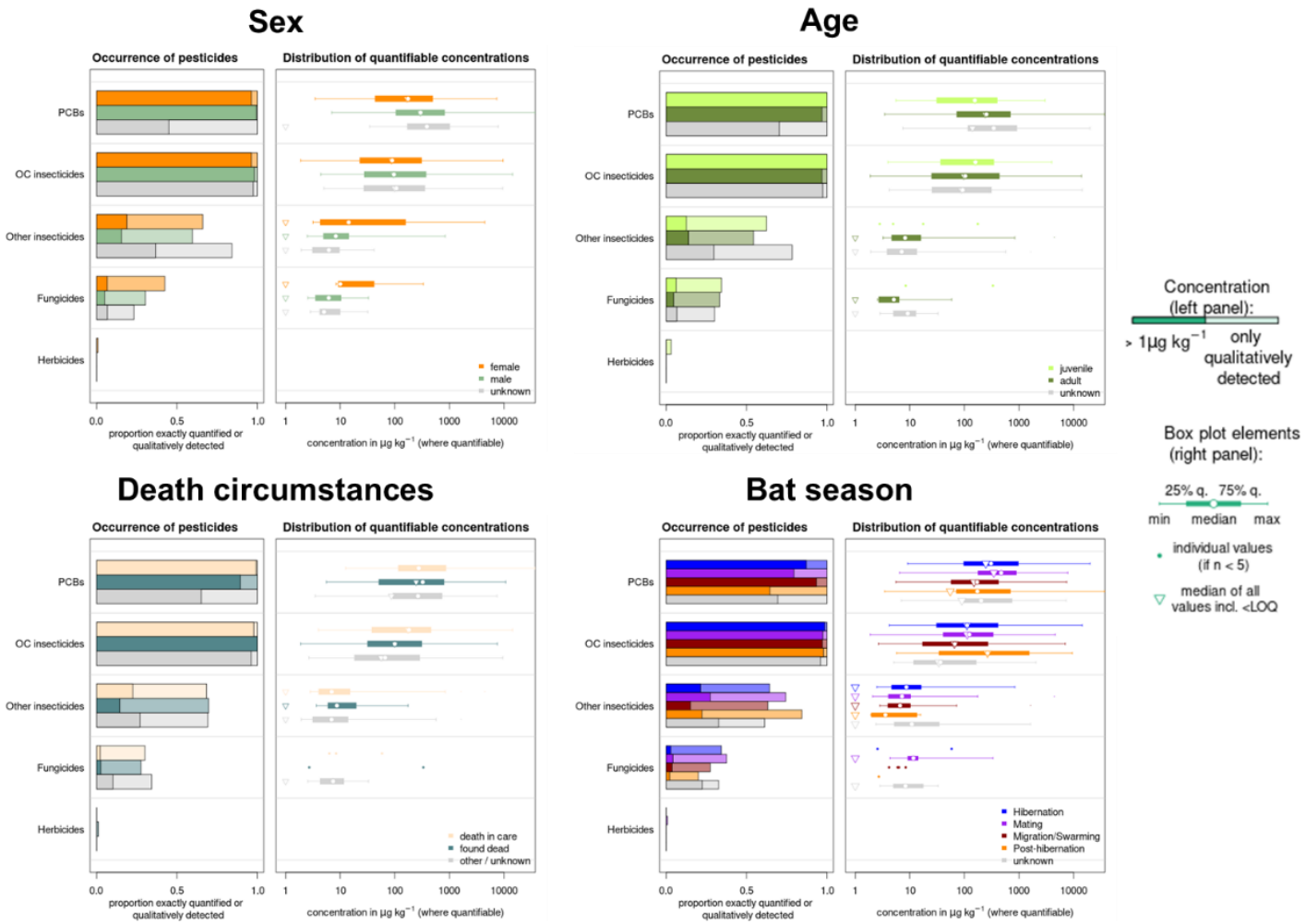


Figure 8 Pesticide concentrations by age, sex, circumstances of death and bat season. In each sub-figure, the barplots on the left show the proportion of samples in which a quantifiable amount of pesticides from a given group could be detected (dark bars) and the additional proportion in which they were only detected qualitatively (light bars). Boxplots in the right panel show the distribution of the quantified values on a logarithmic scale. The minimum, 25% quantile, median, 75% quantile and maximum are shown. Additionally, the median across all samples, including bats where a pathogen was not or only qualitatively detected, is shown by a triangle. Whenever less than 5 values were available, these are shown as individual dots. A more detailed display for individual pesticide loads is available in **Supporting Figure S1**.

4. Conclusion

We investigated the exposure of nearly 400 bats to more than 200 different environmental contaminants with a validated micro QuEChERS-based multiresidue approach using gas chromatography-tandem mass spectrometry. The animals belonged to five different species (*Eptesicus serotinus*, *Myotis myotis*, *Nyctalus noctula*, *Pipistrellus pipistrellus*, and *Plecotus auritus*), wide-spread all over Europe and representing most of the ecological variety of European bats. Hence, this work provides the largest dataset upon pesticide exposure of European bats to date.

The individual pesticide exposure ranged from four compounds (*M. myotis,* No. 3073) to 25 different contaminants (*P. auritus*, No. 3300). The investigated specimens showed an exposure to insecticides (organophosphates, pyrethroids, fipronil) and fungicides (predominantly azoles). Most frequently, organochlorine insecticides were detected along with polychlorinated biphenyls, revealing the ongoing environmental impact of substances that have been banned already decades ago. Statistical analyses including species, date and place of finding, sex, age, circumstances of death and bat season showed that the contamination of bats is more or less homogeneous across Germany and does not substantially depend on any of the parameters studied. Bats as typical apex predators thus reflect the general and broad background contamination of the environment with pesticides. Exceptions in one direction or the other can be explained by specialised trophic niches (*e.g. M. myotis* feeding mainly on epigeic forest beetles) or by locally contaminated roosts (*e.g. P. pipistrellus*).

Our findings highlight the necessity of adequate pesticide monitoring for non-target animals such as bats. Their longevity, which makes long-term accumulation of pesticides likely, and their low reproductive rate make bats particularly vulnerable. With regard to species conservation, an effective risk assessment is indispensable.

Author’s contributions

Sonja Schanzer: Conceptualization, Methodology, Investigation, Formal analysis, Performing analysis, Visualization, Writing – Original Draft, Writing - Review and Editing; Martin Koch: Conceptualization, Methodology, Collection of bats, Formal analysis, Writing - Review and Editing; Andreas Kiefer: Conceptualization, Collection of bats, Writing - Review and Editing; Thalia Jentke: Collection of bats, Writing - Review and Editing; Michael Veith: Methodology, Resources, Writing - Review and Editing; Franz Bracher: Resources, Writing - Review and Editing, Johannes Bracher: Methodology, Investigation, Visualization, Resources, Writing - Review and Editing; Christoph Müller: Conceptualization, Methodology, Investigation, Formal analysis, Visualization, Writing – Original Draft, Writing - Review and Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interest or personal relationships that could have appeared to influence the work reported in this study.

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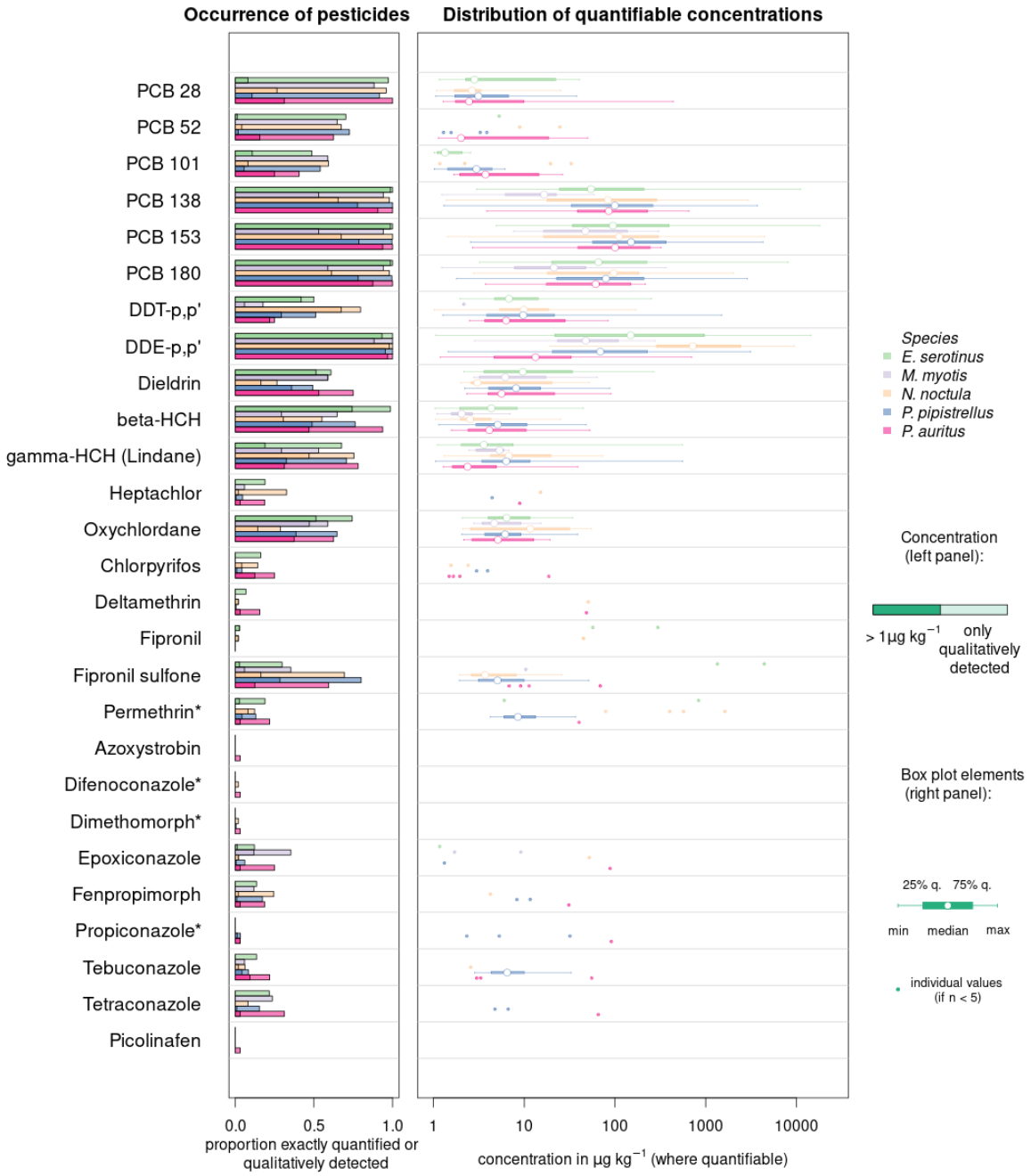
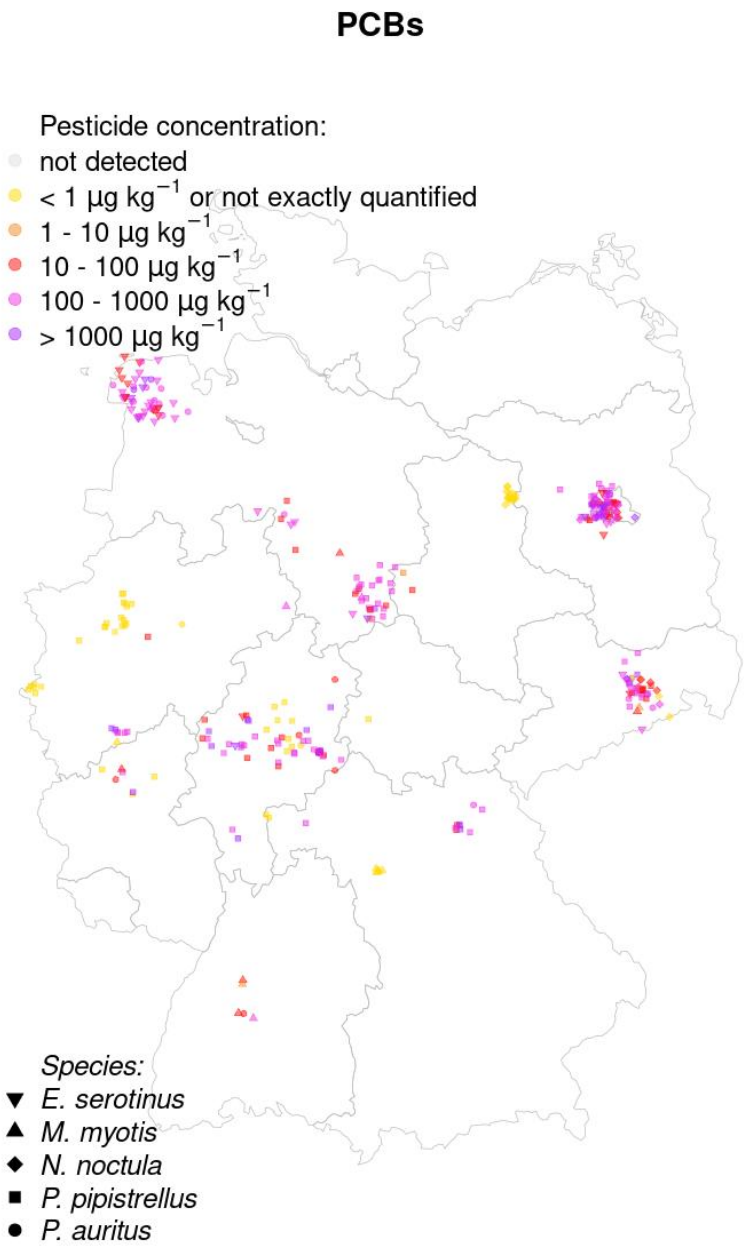
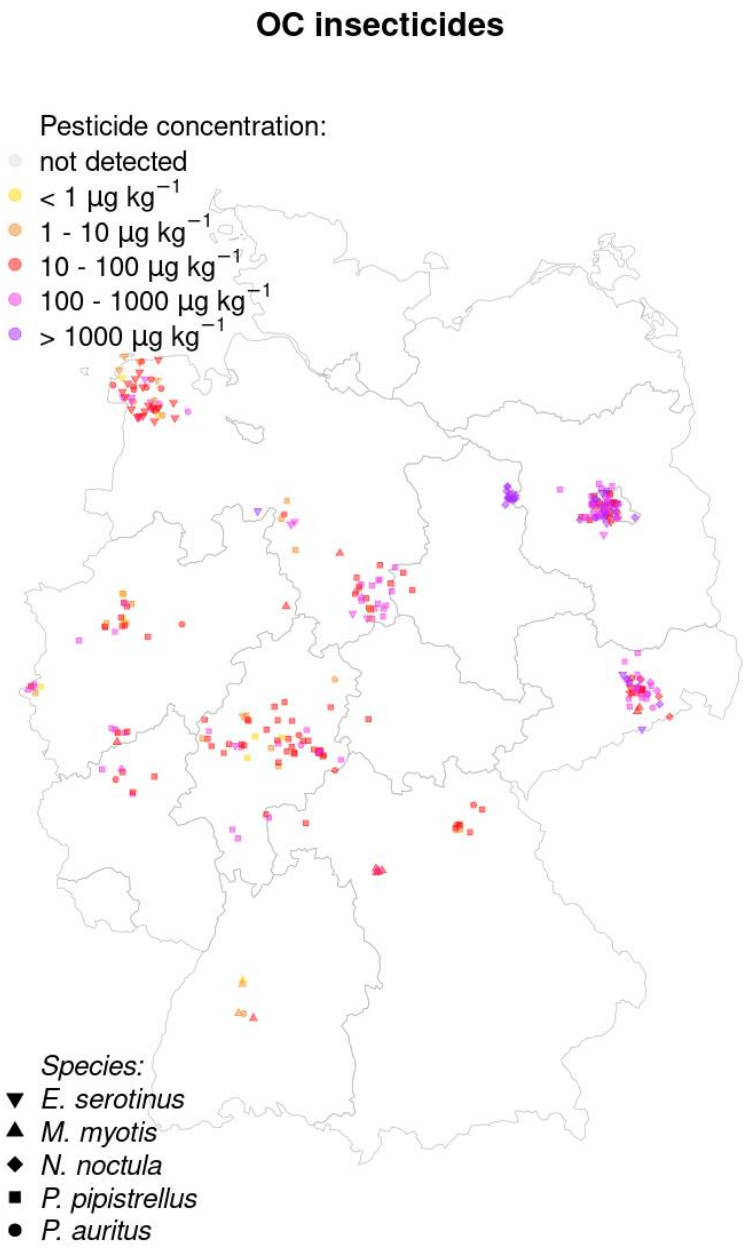
4.4. Supplementary material



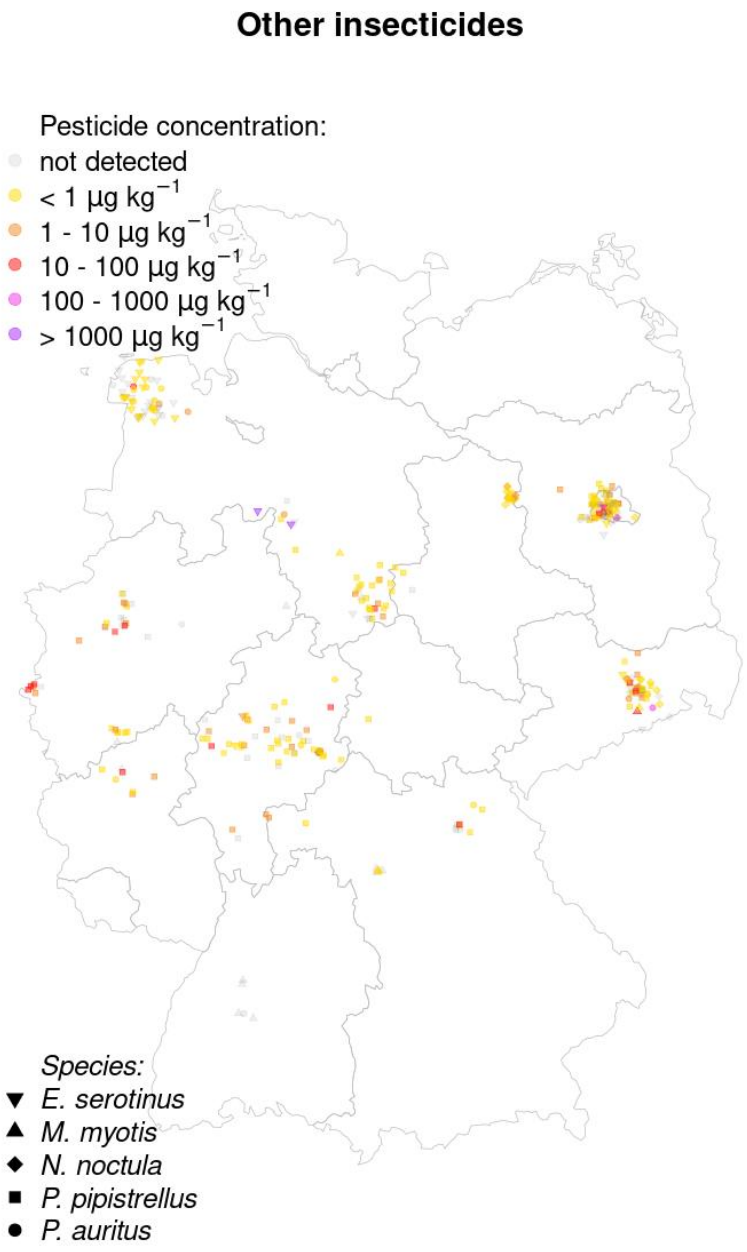
Supporting Figure S1 A more detailed descriptive analysis of pesticide concentrations, stratified by species. Compounds were sorted according to their occurrence in the text. The barplots on the left show the proportion of samples in which a quantifiable amount of pesticides from a given group could be detected (dark bars) and the additional proportion in which they were only detected qualitatively (light bars). Boxplots in the right panel show the distribution of the quantified values on a logarithmic scale. The minimum, 25% quantile, median, 75% quantile and maximum are shown; see also legend in top panel. Whenever less than 5 values were available, these are shown as individual dots. *: sum of isomers.



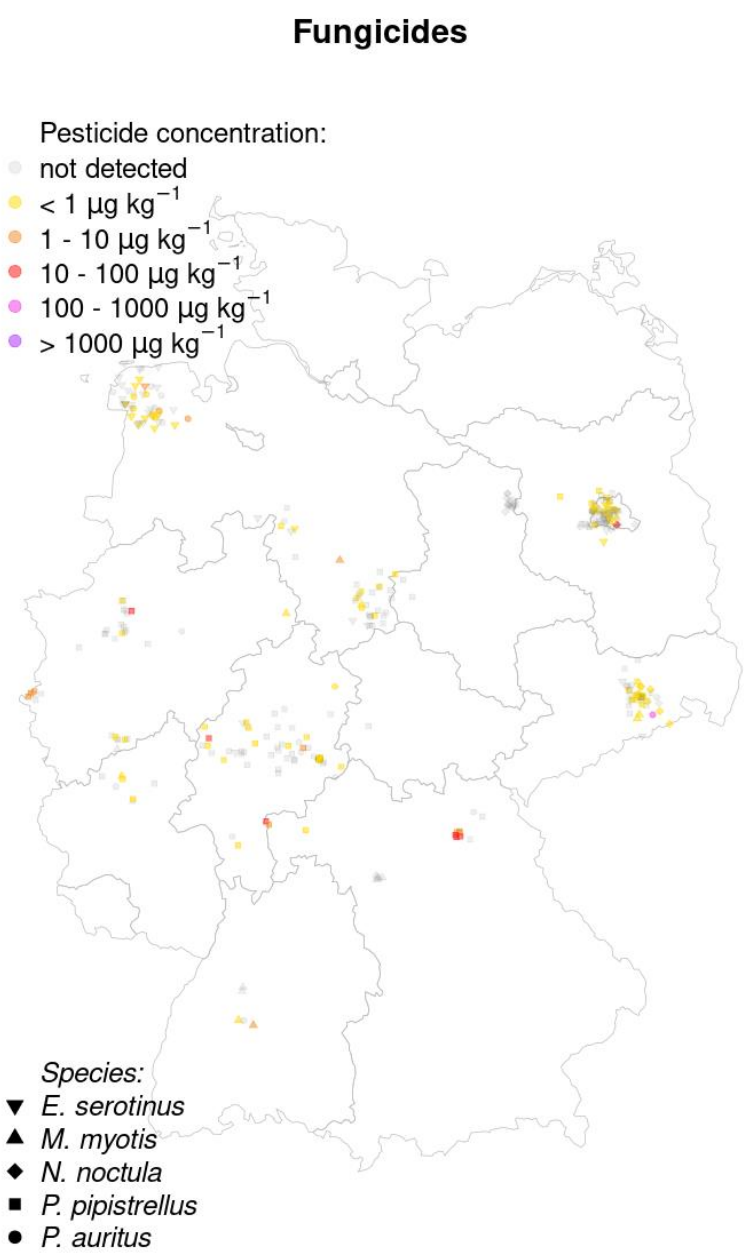
Supporting Figure S2 Geographical distribution of pesticides and pollutants in relation to the bat species and detected concentration.



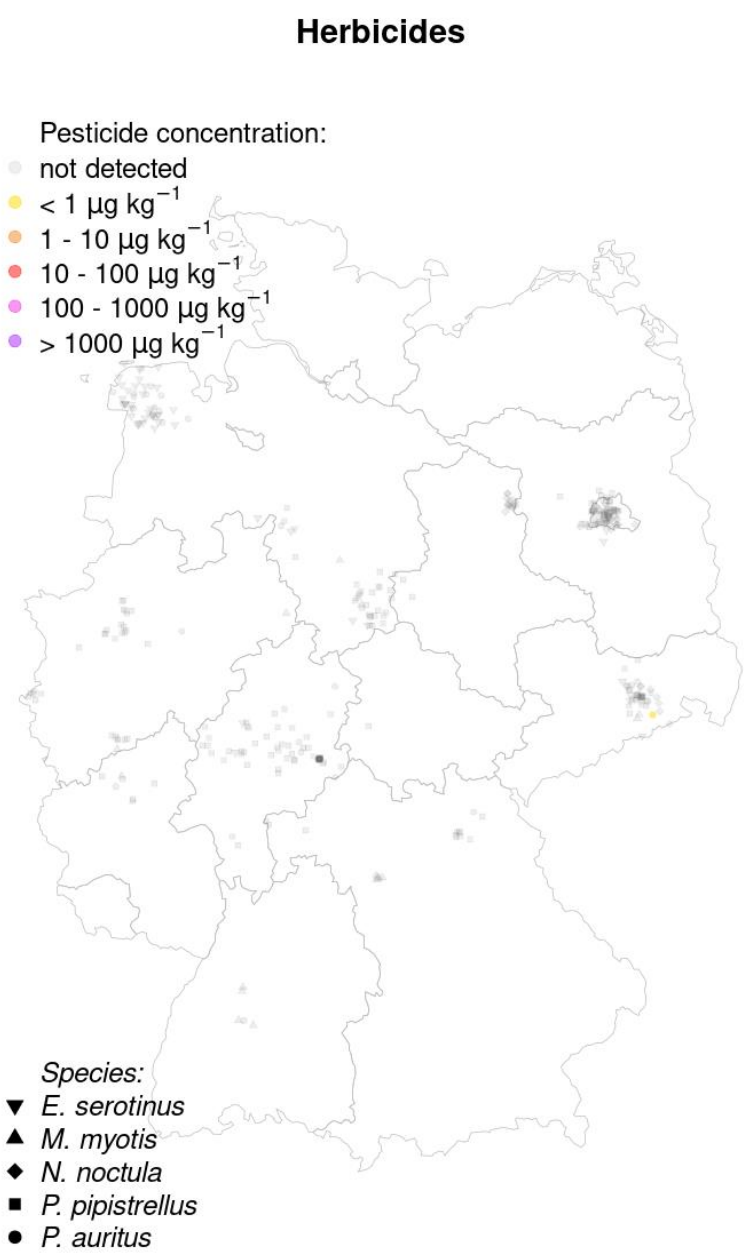
Supporting Figure S2 (continued).



Supporting Figure S2 (continued).



Supporting Figure S2 (continued).



Supporting Figure S2 (continued).

Supporting Table S1 Additional data on bat samples. Used abbreviations: hib (hibernation), ldm (long distance movement), mat (mating), mig/swa (migrating/ swarming), other (put to sleep, death or injured from outside influence (*e.g.* wind turbines, cats)), post hib (post hibernation), sdm (short distance movement). The formatting slightly varies from the original table due to different data types.

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3272	*E. serotinus*	male	adult	11	2016	death in care	30	01773	Altenberg	Saxony	Germany	hib	sdm
3276	*E. serotinus*	female	juvenile	8	2017	other	not applicable	01689	Niederau	Saxony	Germany	mig/swa	sdm
3280	*E. serotinus*	male	adult	unknown	unknown	unknown	unknown	01705	Freital	Saxony	Germany	unknown	sdm
3288	*E. serotinus*	male	adult	9	2019	death in care	13	01156	Cossebaude	Saxony	Germany	mig/swa	sdm
3311	*E. serotinus*	male	unknown	8	2019	found dead	not applicable	35041	Heskem	Hessia	Germany	mig/swa	sdm
3319	*E. serotinus*	male	unknown	4	2019	found dead	not applicable	35444	Biebertal	Hessia	Germany	post hib	sdm
3450	*E. serotinus*	male	adult	3	2021	found dead	not applicable	31559	Hohnhorst	Lower Saxony	Germany	hib	sdm
3451	*E. serotinus*	female	unknown	8	2020	death in care	2	31698	Lindhorst	Lower Saxony	Germany	mig/swa	sdm
3452	*E. serotinus*	male	adult	5	2021	death in care	23	37412	Herzberg am Harz	Lower Saxony	Germany	mat	sdm
3454	*E. serotinus*	female	juvenile	7	2020	found dead	not applicable	31647	Rehburg-Loccum	Lower Saxony	Germany	mat	sdm
3456	*E. serotinus*	female	adult	5	2021	death in care	1	37191	Kaltenburg-Lindau	Lower Saxony	Germany	mat	sdm
3458	*E. serotinus*	female	adult	5	2020	other	not applicable	31600	Uchte	Lower Saxony	Germany	mat	sdm
3463	*E. serotinus*	male	adult	7	2019	death in care	1	14129	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3464	*E. serotinus*	male	adult	5	2019	other	not applicable	14979	Grossbeeren	Berlin/ Brandenburg	Germany	mat	sdm
3465	*E. serotinus*	male	adult	4	2019	other	not applicable	13469	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3467	*E. serotinus*	male	adult	1	2020	other	not applicable	12203	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3468	*E. serotinus*	female	adult	12	2019	death in care	2	14974	Ludwigsfelde	Berlin/ Brandenburg	Germany	hib	sdm
3469	*E. serotinus*	male	adult	9	2019	other	not applicable	12307	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3470	*E. serotinus*	male	adult	10	2019	other	not applicable	10117	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3472	*E. serotinus*	male	adult	5	2019	other	not applicable	14195	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3473	*E. serotinus*	male	adult	3	2019	found dead	not applicable	10825	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3474	*E. serotinus*	male	adult	9	2018	other	not applicable	10555	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3485	*E. serotinus*	male	adult	3	2017	death in care	unknown	unknown	unknown	Berlin/ Brandenburg	Germany	hib	sdm
3488	*E. serotinus*	male	adult	9	2018	other	not applicable	10825	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3494	*E. serotinus*	female	adult	8	2018	unknown	unknown	10965	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3498	*E. serotinus*	male	unknown	8	2019	unknown	unknown	12163	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3499	*E. serotinus*	male	unknown	1	2018	death in care	1	14055	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3500	*E. serotinus*	male	unknown	8	2019	unknown	unknown	13355	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3501	*E. serotinus*	female	unknown	3	2018	death in care	7	13503	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3502	*E. serotinus*	male	adult	3	2019	death in care	4	10407	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3503	*E. serotinus*	female	unknown	4	2019	death in care	50	10407	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3506	*E. serotinus*	male	unknown	8	2019	death in care	1	14469	Potsdam	Berlin/ Brandenburg	Germany	mig/swa	sdm
3509	*E. serotinus*	male	unknown	3	2019	death in care	1	10553	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3510	*E. serotinus*	male	unknown	4	2018	death in care	21	10779	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3511	*E. serotinus*	male	unknown	3	2018	death in care	17	13593	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3512	*E. serotinus*	male	unknown	6	2019	death in care	1	13509	Berlin	Berlin/ Brandenburg	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3513	*E. serotinus*	male	adult	10	2018	other	not applicable	10405	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3514	*E. serotinus*	female	unknown	1	2020	death in care	112	12051	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3515	*E. serotinus*	male	adult	1	2019	death in care	2	14057	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3522	*E. serotinus*	male	adult	8	2019	unknown	unknown	26506	Norden	Lower Saxony	Germany	mig/swa	sdm
3523	*E. serotinus*	male	adult	8	2019	death in care	1	26506	Norden	Lower Saxony	Germany	mig/swa	sdm
3525	*E. serotinus*	male	adult	8	2019	unknown	unknown	26835	Hesel	Lower Saxony	Germany	mig/swa	sdm
3526	*E. serotinus*	male	adult	8	2019	death in care	2	26721	Emden	Lower Saxony	Germany	mig/swa	sdm
3527	*E. serotinus*	male	juvenile	7	2019	unknown	unknown	26835	Hesel	Lower Saxony	Germany	mat	sdm
3529	*E. serotinus*	male	adult	7	2019	death in care	1	26632	Riepe	Lower Saxony	Germany	mat	sdm
3530	*E. serotinus*	unknown	adult	8	2019	unknown	unknown	26607	Aurich	Lower Saxony	Germany	mig/swa	sdm
3531	*E. serotinus*	female	adult	4	2019	unknown	unknown	26624	Südbrookmerland	Lower Saxony	Germany	post hib	sdm
3532	*E. serotinus*	male	adult	8	2019	unknown	unknown	26802	Moormerland	Lower Saxony	Germany	mig/swa	sdm
3533	*E. serotinus*	male	adult	8	2019	unknown	unknown	26553	Dornum	Lower Saxony	Germany	mig/swa	sdm
3534	*E. serotinus*	male	adult	8	2019	unknown	unknown	26689	Apen	Lower Saxony	Germany	mig/swa	sdm
3535	*E. serotinus*	male	adult	3	2019	unknown	unknown	26721	Emden	Lower Saxony	Germany	hib	sdm
3536	*E. serotinus*	male	adult	11	2019	unknown	unknown	26835	Hesel	Lower Saxony	Germany	hib	sdm
3537	*E. serotinus*	male	adult	4	2019	unknown	unknown	26789	Leer	Lower Saxony	Germany	post hib	sdm
3538	*E. serotinus*	male	adult	7	2020	unknown	unknown	26849	Filsum	Lower Saxony	Germany	mat	sdm
3539	*E. serotinus*	male	adult	4	2019	unknown	unknown	26721	Emden	Lower Saxony	Germany	post hib	sdm
3540	*E. serotinus*	male	adult	7	2020	death in care	1	26759	Hinte	Lower Saxony	Germany	mat	sdm
3541	*E. serotinus*	male	adult	3	2021	unknown	unknown	26607	Aurich	Lower Saxony	Germany	hib	sdm
3542	*E. serotinus*	male	adult	4	2020	death in care	1	26842	Ostrhauderfehn	Lower Saxony	Germany	post hib	sdm
3543	*E. serotinus*	female	adult	8	2020	death in care	1	26548	Norderney	Lower Saxony	Germany	mig/swa	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3544	*E. serotinus*	male	adult	8	2020	found dead	not applicable	26670	Uplengen	Lower Saxony	Germany	mig/swa	sdm
3545	*E. serotinus*	male	adult	4	2020	found dead	not applicable	26427	Neuharlingersiel	Lower Saxony	Germany	post hib	sdm
3546	*E. serotinus*	male	adult	7	2021	death in care	1	26532	Großheide	Lower Saxony	Germany	mat	sdm
3556	*E. serotinus*	male	juvenile	7	2020	unknown	unknown	26721	Emden	Lower Saxony	Germany	mat	sdm
3564	*E. serotinus*	male	adult	9	2019	unknown	unknown	26802	Moormerland	Lower Saxony	Germany	mig/swa	sdm
3565	*E. serotinus*	male	adult	5	2021	death in care	1	26789	Leer	Lower Saxony	Germany	mat	sdm
3566	*E. serotinus*	male	adult	8	2021	unknown	unknown	26605	Aurich	Lower Saxony	Germany	mig/swa	sdm
3567	*E. serotinus*	male	juvenile	7	2021	death in care	2	26789	Leer	Lower Saxony	Germany	mat	sdm
3568	*E. serotinus*	male	juvenile	8	2021	death in care	3	26844	Jemgum	Lower Saxony	Germany	mig/swa	sdm
3569	*E. serotinus*	male	adult	9	2020	unknown	unknown	26789	Leer	Lower Saxony	Germany	mig/swa	sdm
3570	*E. serotinus*	female	juvenile	8	2021	death in care	24	26789	Leer	Lower Saxony	Germany	mig/swa	sdm
3571	*E. serotinus*	female	adult	8	2021	death in care	9	unknown	unknown	unknown	Germany	mig/swa	sdm
3572	*E. serotinus*	unknown	adult	8	2021	death in care	4	26529	Wirdum	Lower Saxony	Germany	mig/swa	sdm
3573	*E. serotinus*	male	adult	7	2021	death in care	2	unknown	unknown	Lower Saxony	Germany	mat	sdm
3574	*E. serotinus*	female	adult	8	2021	death in care	5	26835	Hesel	Lower Saxony	Germany	mig/swa	sdm
3010	*M. myotis*	unknown	unknown	6	2018	found dead	not applicable	91484	Sugenheim-Ullstadt	Bavaria	Germany	mat	sdm
3012	*M. myotis*	unknown	unknown	6	2018	found dead	not applicable	91484	Sugenheim-Ullstadt	Bavaria	Germany	mat	sdm
3015	*M. myotis*	unknown	unknown	6	2018	found dead	not applicable	91484	Sugenheim-Ullstadt	Bavaria	Germany	mat	sdm
3016	*M. myotis*	unknown	unknown	6	2018	found dead	not applicable	91484	Sugenheim-Ullstadt	Bavaria	Germany	mat	sdm
3017	*M. myotis*	unknown	unknown	6	2018	found dead	not applicable	91484	Sugenheim-Ullstadt	Bavaria	Germany	mat	sdm
3073	*M. myotis*	unknown	unknown	11	2017	unknown	unknown	53343	Wachtberg	North Rhine-Westphalia	Germany	hib	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3188	*M. myotis*	male	adult	11	2019	unknown	unknown	37671	Höxter	North Rhine-Westphalia	Germany	hib	sdm
3247	*M. myotis*	female	adult	8	2016	death in care	7	01768	Glashütte	Saxony	Germany	mig/swa	sdm
3279	*M. myotis*	female	adult	7	2019	other	not applicable	01768	Glashütte	Saxony	Germany	mat	sdm
3378	*M. myotis*	female	unknown	5	2015	unknown	unknown	31139	Hildesheim	Lower Saxony	Germany	mat	sdm
3457	*M. myotis*	male	adult	3	2021	found dead	not applicable	37539	Bad Grund	Lower Saxony	Germany	hib	sdm
3459	*M. myotis*	female	juvenile	5	2021	found dead	not applicable	37534	Bad Grund	Lower Saxony	Germany	mat	sdm
3462	*M. myotis*	female	adult	5	2021	other	not applicable	56653	Wehr	Lower Saxony	Germany	mat	sdm
3517	*M. myotis*	male	juvenile	7	2019	other	not applicable	72160	Horb am Neckar	Baden-Wuerttemberg	Germany	mat	sdm
3518	*M. myotis*	male	adult	4	2016	found dead	not applicable	72181	Starzach	Baden-Wuerttemberg	Germany	post hib	sdm
3520	*M. myotis*	female	adult	4	2021	other	not applicable	75365	Calw	Baden-Wuerttemberg	Germany	post hib	sdm
3521	*M. myotis*	female	adult	4	2021	other	not applicable	75365	Calw	Baden-Wuerttemberg	Germany	post hib	sdm
3074	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3075	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3076	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3077	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3078	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3079	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3080	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3081	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3082	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3083	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3084	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3085	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3086	*N. noctula*	unknown	adult	4	2011	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	post hib	ldm
3087	*N. noctula*	unknown	juvenile	7	2017	unknown	unknown	39524	Sandau	Saxony-Anhalt	Germany	mat	ldm
3121	*N. noctula*	male	unknown	unknown	unknown	unknown	unknown	unknown	unknown	Hessia	Germany	unknown	ldm
3122	*N. noctula*	female	unknown	unknown	2002	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	ldm
3123	*N. noctula*	female	unknown	unknown	unknown	unknown	unknown	unknown	unknown	Hessia	Germany	unknown	ldm
3197	*N. noctula*	female	unknown	2	2009	unknown	unknown	36123	Eiterfeld	Hessia	Germany	hib	ldm
3202	*N. noctula*	male	unknown	unknown	2005	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	ldm
3203	*N. noctula*	male	unknown	8	2004	unknown	unknown	36037	Fulda	Hessia	Germany	mig/swa	ldm
3245	*N. noctula*	male	adult	10	2019	death in care	16	01328	Dresden	Saxony	Germany	mig/swa	ldm
3248	*N. noctula*	female	adult	12	2019	found dead	not applicable	01824	Rathen	Saxony	Germany	hib	ldm
3252	*N. noctula*	female	juvenile	2	2019	other	not applicable	01067	Dresden	Saxony	Germany	hib	ldm
3254	*N. noctula*	male	adult	9	2018	other	not applicable	01127	Dresden-Albertstadt	Saxony	Germany	mig/swa	ldm
3255	*N. noctula*	female	adult	4	2019	death in care	unknown	01099	Dresden-Albertstadt	Saxony	Germany	post hib	ldm
3260	*N. noctula*	female	adult	10	2016	other	not applicable	01847	Lohmen	Saxony	Germany	mig/swa	ldm
3263	*N. noctula*	male	juvenile	unknown	2019	unknown	unknown	01067	Dresden	Saxony	Germany	unknown	ldm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3275	*N. noctula*	female	adult	10	2015	found dead	not applicable	01109	Dresden-Hellerau	Saxony	Germany	mig/swa	ldm
3277	*N. noctula*	male	adult	8	2018	other	not applicable	01109	Dresden	Saxony	Germany	mig/swa	ldm
3282	*N. noctula*	male	adult	4	2019	death in care	unknown	01640	Coswig	Saxony	Germany	post hib	ldm
3284	*N. noctula*	female	adult	3	2017	death in care	1	01796	Pirna	Saxony	Germany	hib	ldm
3294	*N. noctula*	unknown	adult	1	2018	found dead	not applicable	01279	Dresden	Saxony	Germany	hib	ldm
3453	*N. noctula*	male	adult	11	2020	other	not applicable	31545	Steyerberg	Lower Saxony	Germany	hib	ldm
3466	*N. noctula*	male	adult	5	2019	found dead	not applicable	12203	Berlin	Berlin/ Brandenburg	Germany	mat	ldm
3471	*N. noctula*	male	adult	3	2019	found dead	not applicable	12205	Berlin	Berlin/ Brandenburg	Germany	hib	ldm
3483	*N. noctula*	female	adult	2	2018	found dead	not applicable	14467	Potsdam	Berlin/ Brandenburg	Germany	hib	ldm
3484	*N. noctula*	male	adult	2	2017	found dead	not applicable	12559	Köpenick	Berlin/ Brandenburg	Germany	hib	ldm
3486	*N. noctula*	female	adult	11	2017	other	not applicable	12353	Berlin	Berlin/ Brandenburg	Germany	hib	ldm
3487	*N. noctula*	male	adult	10	2017	other	not applicable	12057	Berlin	Berlin/ Brandenburg	Germany	mig/swa	ldm
3489	*N. noctula*	male	adult	11	2017	other	not applicable	12353	Berlin	Berlin/ Brandenburg	Germany	hib	ldm
3490	*N. noctula*	male	adult	8	2017	death in care	1	10557	Berlin	Berlin/ Brandenburg	Germany	mig/swa	ldm
3492	*N. noctula*	male	adult	8	2018	other	not applicable	14469	Potsdam	Berlin/ Brandenburg	Germany	mig/swa	ldm
3493	*N. noctula*	male	adult	7	2017	death in care	18	14109	Berlin	Berlin/ Brandenburg	Germany	mat	ldm
3495	*N. noctula*	female	adult	11	2017	other	not applicable	13599	Berlin	Berlin/ Brandenburg	Germany	hib	ldm
3496	*N. noctula*	female	adult	11	2017	death in care	1	12353	Berlin	Berlin/ Brandenburg	Germany	hib	ldm
3497	*N. noctula*	female	adult	5	2018	other	not applicable	10967	Berlin	Berlin/ Brandenburg	Germany	mat	ldm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3507	*N. noctula*	male	adult	10	2017	death in care	7	13587	Berlin	Berlin/ Brandenburg	Germany	mig/swa	ldm
3508	*N. noctula*	male	adult	1	2018	death in care	14	13587	Berlin	Berlin/ Brandenburg	Germany	hib	ldm
3516	*N. noctula*	female	adult	4	2019	unknown	unknown	10249	Volkspark	Berlin/ Brandenburg	Germany	post hib	ldm
3008	*P. pipistrellus*	unknown	unknown	1	2019	other	not applicable	56068	Koblenz	Rhineland-Palatinate	Germany	hib	sdm
3009	*P. pipistrellus*	male	unknown	7	2017	other	not applicable	53518	Leimbach	Rhineland-Palatinate	Germany	mat	sdm
3013	*P. pipistrellus*	unknown	unknown	6	2017	found dead	not applicable	56754	Binningen	Rhineland-Palatinate	Germany	mat	sdm
3021	*P. pipistrellus*	unknown	unknown	4	2017	unknown	unknown	35315	Homberg (Ohm)	Bavaria	Germany	post hib	sdm
3023	*P. pipistrellus*	unknown	unknown	7	2018	unknown	unknown	36341	Lauterbach	Hessia	Germany	mat	sdm
3024	*P. pipistrellus*	unknown	unknown	6	2018	unknown	unknown	unknown	unknown	Hessia	Germany	mat	sdm
3025	*P. pipistrellus*	unknown	unknown	1	2018	unknown	unknown	34613	Schwalmstadt	Hessia	Germany	hib	sdm
3026	*P. pipistrellus*	unknown	unknown	1	2018	unknown	unknown	unknown	Schwalmstadt	Hessia	Germany	hib	sdm
3027	*P. pipistrellus*	unknown	unknown	6	2018	unknown	unknown	unknown	unknown	Hessia	Germany	mat	sdm
3028	*P. pipistrellus*	unknown	unknown	7	2018	unknown	unknown	unknown	unknown	Hessia	Germany	mat	sdm
3029	*P. pipistrellus*	unknown	unknown	3	2018	unknown	unknown	unknown	unknown	Hessia	Germany	hib	sdm
3030	*P. pipistrellus*	unknown	unknown	9	2018	unknown	unknown	36369	Lautertal-Engelrodt	Hessia	Germany	mig/swa	sdm
3031	*P. pipistrellus*	unknown	unknown	4	2018	unknown	unknown	34633	Ottrau	Hessia	Germany	post hib	sdm
3033	*P. pipistrellus*	unknown	unknown	5	2018	unknown	unknown	63791	Karlstein	Hessia	Germany	mat	sdm
3034	*P. pipistrellus*	unknown	unknown	7	2018	unknown	unknown	63791	Karlstein	Hessia	Germany	mat	sdm
3036	*P. pipistrellus*	unknown	unknown	7	2018	unknown	unknown	36318	Schwalmtal Hopfgarten	Hessia	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3037	*P. pipistrellus*	unknown	unknown	7	2018	unknown	unknown	36319	Schwalmtal Hopfgarten	Hessia	Germany	mat	sdm
3038	*P. pipistrellus*	unknown	unknown	7	2018	unknown	unknown	36320	Schwalmtal Hopfgarten	Hessia	Germany	mat	sdm
3039	*P. pipistrellus*	unknown	unknown	7	2017	found dead	not applicable	53518	Leimbach	Rhineland-Palatinate	Germany	mat	sdm
3040	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	52525	Heinsberg	North Rhine-Westphalia	Germany	unknown	sdm
3041	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	52525	Heinsberg	North Rhine-Westphalia	Germany	unknown	sdm
3042	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	52525	Heinsberg	North Rhine-Westphalia	Germany	unknown	sdm
3043	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	52525	Heinsberg	North Rhine-Westphalia	Germany	unknown	sdm
3044	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	52525	Heinsberg	North Rhine-Westphalia	Germany	unknown	sdm
3045	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	52525	Heinsberg	North Rhine-Westphalia	Germany	unknown	sdm
3046	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	unknown	sdm
3047	*P. pipistrellus*	unknown	unknown	unknown	2019	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	unknown	sdm
3048	*P. pipistrellus*	unknown	unknown	10	2018	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	mig/swa	sdm
3049	*P. pipistrellus*	unknown	unknown	6	2018	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	mat	sdm
3050	*P. pipistrellus*	unknown	unknown	5	2018	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	mat	sdm
3051	*P. pipistrellus*	unknown	unknown	6	2018	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	mat	sdm
3052	*P. pipistrellus*	unknown	unknown	unknown	2017	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	unknown	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3053	*P. pipistrellus*	unknown	unknown	8	2018	unknown	unknown	47441	Moers	North Rhine-Westphalia	Germany	mig/swa	sdm
3054	*P. pipistrellus*	unknown	unknown	6	2018	unknown	unknown	45721	Haltern am See	North Rhine-Westphalia	Germany	mat	sdm
3055	*P. pipistrellus*	unknown	unknown	6	2018	unknown	unknown	45881	Gelsenkirchen	North Rhine-Westphalia	Germany	mat	sdm
3056	*P. pipistrellus*	unknown	unknown	1	2018	unknown	unknown	45964	Gladbeck	North Rhine-Westphalia	Germany	hib	sdm
3063	*P. pipistrellus*	unknown	unknown	unknown	2018	unknown	unknown	45721	Haltern am See	North Rhine-Westphalia	Germany	unknown	sdm
3064	*P. pipistrellus*	unknown	unknown	unknown	2018	unknown	unknown	45721	Haltern am See	North Rhine-Westphalia	Germany	unknown	sdm
3065	*P. pipistrellus*	unknown	unknown	unknown	2018	unknown	unknown	45721	Haltern am See	North Rhine-Westphalia	Germany	unknown	sdm
3066	*P. pipistrellus*	unknown	unknown	unknown	2018	unknown	unknown	45721	Haltern am See	North Rhine-Westphalia	Germany	unknown	sdm
3067	*P. pipistrellus*	unknown	unknown	unknown	2018	unknown	unknown	45721	Haltern am See	North Rhine-Westphalia	Germany	unknown	sdm
3068	*P. pipistrellus*	unknown	unknown	6	2017	unknown	unknown	45964	Gladbeck	North Rhine-Westphalia	Germany	mat	sdm
3069	*P. pipistrellus*	unknown	unknown	4	2018	unknown	unknown	45657	Recklinghausen	North Rhine-Westphalia	Germany	post hib	sdm
3070	*P. pipistrellus*	unknown	unknown	2	2019	unknown	unknown	45964	Gladbeck	North Rhine-Westphalia	Germany	hib	sdm
3071	*P. pipistrellus*	unknown	unknown	2	2018	unknown	unknown	unknown	unknown	unknown	Germany	hib	sdm
3116	*P. pipistrellus*	male	unknown	9	2018	death in care	2	56753	Mertloch	Rhineland-Palatinate	Germany	mig/swa	sdm
3117	*P. pipistrellus*	male	unknown	9	2017	other	not applicable	56745	Rieden	Rhineland-Palatinate	Germany	mig/swa	sdm
3119	*P. pipistrellus*	male	unknown	6	2008	unknown	unknown	53639	Königswinter	North Rhine-Westphalia	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3126	*P. pipistrellus*	unknown	unknown	unknown	unknown	unknown	unknown	53639	Königswinter	North Rhine-Westphalia	Germany	unknown	sdm
3128	*P. pipistrellus*	male	juvenile	unknown	1990	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3129	*P. pipistrellus*	male	unknown	unknown	1990	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3130	*P. pipistrellus*	female	unknown	unknown	1990	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3132	*P. pipistrellus*	male	juvenile	unknown	1990	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3133	*P. pipistrellus*	male	juvenile	unknown	1990	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3134	*P. pipistrellus*	unknown	unknown	unknown	1990	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3145	*P. pipistrellus*	male	unknown	unknown	unknown	unknown	unknown	53113	Bonn	North Rhine-Westphalia	Germany	unknown	sdm
3147	*P. pipistrellus*	female	unknown	unknown	2020	found dead	not applicable	53119	Bonn	North Rhine-Westphalia	Germany	unknown	sdm
3148	*P. pipistrellus*	male	unknown	unknown	2019	unknown	unknown	95488	Ekersdorf	Bavaria	Germany	unknown	sdm
3149	*P. pipistrellus*	male	unknown	unknown	2019	unknown	unknown	95488	Ekersdorf	Bavaria	Germany	unknown	sdm
3150	*P. pipistrellus*	female	unknown	unknown	2019	unknown	unknown	95488	Ekersdorf	Bavaria	Germany	unknown	sdm
3151	*P. pipistrellus*	female	unknown	unknown	2019	unknown	unknown	95488	Ekersdorf	Bavaria	Germany	unknown	sdm
3152	*P. pipistrellus*	female	unknown	unknown	2019	unknown	unknown	95488	Ekersdorf	Bavaria	Germany	unknown	sdm
3153	*P. pipistrellus*	male	unknown	unknown	2019	unknown	unknown	95488	Ekersdorf	Bavaria	Germany	unknown	sdm
3161	*P. pipistrellus*	female	unknown	2	2019	death in care	30	95482	Gefrees	Bavaria	Germany	hib	sdm
3163	*P. pipistrellus*	male	unknown	9	2019	unknown	unknown	95448	Bayreuth	Bavaria	Germany	mig/swa	sdm
3169	*P. pipistrellus*	male	juvenile	7	2020	found dead	not applicable	53113	Bonn	North Rhine-Westphalia	Germany	mat	sdm
3175	*P. pipistrellus*	female	unknown	4	2002	found dead	not applicable	36115	Dorfborn-Neuhof	Hessia	Germany	post hib	sdm
3176	*P. pipistrellus*	male	unknown	6	2013	death in care	1	35745	Herborn	Hessia	Germany	mat	sdm
3189	*P. pipistrellus*	female	unknown	8	2018	unknown	unknown	53115	Bonn	North Rhine-Westphalia	Germany	mig/swa	sdm
3198	*P. pipistrellus*	female	unknown	2	2009	unknown	unknown	36123	Eiterfeld	Hessia	Germany	hib	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3205	*P. pipistrellus*	female	unknown	1	2003	unknown	unknown	36037	Fulda	Hessia	Germany	hib	sdm
3207	*P. pipistrellus*	male	unknown	unknown	2003	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3208	*P. pipistrellus*	male	unknown	unknown	2003	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3209	*P. pipistrellus*	female	unknown	unknown	2005	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3210	*P. pipistrellus*	female	unknown	unknown	2005	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3211	*P. pipistrellus*	female	unknown	unknown	2005	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3215	*P. pipistrellus*	female	unknown	3	1987	found dead	not applicable	64283	Darmstadt	Hessia	Germany	hib	sdm
3216	*P. pipistrellus*	female	unknown	5	1996	unknown	unknown	36037	Fulda	Hessia	Germany	mat	sdm
3220	*P. pipistrellus*	female	unknown	8	2003	unknown	unknown	36037	Fulda	Hessia	Germany	mig/swa	sdm
3221	*P. pipistrellus*	male	unknown	unknown	2006	unknown	unknown	36037	Fulda	Hessia	Germany	unknown	sdm
3222	*P. pipistrellus*	male	unknown	3	2010	unknown	unknown	36110	Schlitz	Hessia	Germany	hib	sdm
3224	*P. pipistrellus*	male	unknown	7	2013	unknown	unknown	35687	Dillenburg	Hessia	Germany	mat	sdm
3225	*P. pipistrellus*	male	unknown	9	2013	unknown	unknown	35745	Herborn	Hessia	Germany	mig/swa	sdm
3226	*P. pipistrellus*	female	unknown	7	2013	unknown	unknown	35708	Hauger	Hessia	Germany	mat	sdm
3228	*P. pipistrellus*	male	unknown	9	2013	other	not applicable	64331	Weiterstadt	Hessia	Germany	mig/swa	sdm
3229	*P. pipistrellus*	female	juvenile	7	2019	unknown	unknown	01705	Freital	Saxony	Germany	mat	sdm
3230	*P. pipistrellus*	female	juvenile	7	2018	death in care	3	01445	Radebeul	Saxony	Germany	mat	sdm
3233	*P. pipistrellus*	male	adult	6	2015	death in care	3	01326	Dresden	Saxony	Germany	mat	sdm
3235	*P. pipistrellus*	male	adult	9	2018	other	not applicable	01309	Dresden	Saxony	Germany	mig/swa	sdm
3237	*P. pipistrellus*	male	juvenile	8	2019	found dead	not applicable	01307	Dresden	Saxony	Germany	mig/swa	sdm
3240	*P. pipistrellus*	female	adult	5	2019	unknown	unknown	01219	Dresden-Seevorstadt	Saxony	Germany	mat	sdm
3242	*P. pipistrellus*	female	adult	6	2019	death in care	3	01468	Moritzburg	Saxony	Germany	mat	sdm
3244	*P. pipistrellus*	female	juvenile	7	2019	death in care	4	01445	Radebeul	Saxony	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3250	*P. pipistrellus*	male	juvenile	8	2019	found dead	not applicable	01307	Dresden-Johannstadt-Nord	Saxony	Germany	mig/swa	sdm
3253	*P. pipistrellus*	female	adult	unknown	2020	unknown	unknown	01067	Dresden	Saxony	Germany	unknown	sdm
3256	*P. pipistrellus*	female	adult	2	2017	death in care	11	01239	Dresden	Saxony	Germany	hib	sdm
3257	*P. pipistrellus*	female	juvenile	8	2019	found dead	not applicable	01307	Dresden-Johannstadt-Nord	Saxony	Germany	mig/swa	sdm
3258	*P. pipistrellus*	male	juvenile	7	2019	found dead	not applicable	01558	Großenhain	Saxony	Germany	mat	sdm
3259	*P. pipistrellus*	male	juvenile	8	2019	found dead	not applicable	01307	Dresden-Johannstadt-Nord	Saxony	Germany	mig/swa	sdm
3261	*P. pipistrellus*	female	adult	8	2019	found dead	not applicable	01307	Dresden-Johannstadt-Nord	Saxony	Germany	mig/swa	sdm
3264	*P. pipistrellus*	male	adult	unknown	2020	death in care	unknown	01259	Dresden	Saxony	Germany	unknown	sdm
3265	*P. pipistrellus*	male	juvenile	9	2017	death in care	5	01561	Großenhain-Zabeltitz	Saxony	Germany	mig/swa	sdm
3268	*P. pipistrellus*	female	adult	4	2019	found dead	not applicable	01640	Coswig	Saxony	Germany	post hib	sdm
3278	*P. pipistrellus*	male	juvenile	8	2019	found dead	not applicable	01307	Dresden-Johannstadt-Nord	Saxony	Germany	mig/swa	sdm
3281	*P. pipistrellus*	male	juvenile	8	2019	found dead	not applicable	01307	Dresden-Johannstadt-Nord	Saxony	Germany	mig/swa	sdm
3286	*P. pipistrellus*	male	adult	5	2017	death in care	2	01774	Klingenberg-Colmnitz	Saxony	Germany	mat	sdm
3291	*P. pipistrellus*	female	adult	9	2017	found dead	not applicable	01099	Dresden	Saxony	Germany	mig/swa	sdm
3293	*P. pipistrellus*	female	juvenile	6	2019	found dead	not applicable	01187	Dresden-Plauen	Saxony	Germany	mat	sdm
3295	*P. pipistrellus*	male	adult	7	2017	found dead	not applicable	01067	Dresden	Saxony	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3299	*P. pipistrellus*	female	adult	4	2020	death in care	5	01159	Dresden-Plauen	Saxony	Germany	post hib	sdm
3302	*P. pipistrellus*	male	unknown	1	2020	death in care	4	35037	Marburg	Hessia	Germany	hib	sdm
3303	*P. pipistrellus*	female	unknown	8	2019	found dead	not applicable	35041	Marburg	Hessia	Germany	mig/swa	sdm
3304	*P. pipistrellus*	male	unknown	7	2019	found dead	not applicable	35039	Marburg	Hessia	Germany	mat	sdm
3305	*P. pipistrellus*	male	unknown	1	2018	other	not applicable	97848	Rechtenbach	Hessia	Germany	hib	sdm
3306	*P. pipistrellus*	male	unknown	3	2019	found dead	not applicable	35390	Gießen	Hessia	Germany	hib	sdm
3309	*P. pipistrellus*	female	unknown	9	2020	death in care	1	35415	Pohlheim	Hessia	Germany	mig/swa	sdm
3312	*P. pipistrellus*	male	unknown	9	2019	death in care	6	35390	Gießen	Hessia	Germany	mig/swa	sdm
3313	*P. pipistrellus*	male	unknown	6	2019	found dead	not applicable	35578	Wetzlar	Hessia	Germany	mat	sdm
3314	*P. pipistrellus*	female	unknown	10	2019	death in care	13	35466	Rabenau	Hessia	Germany	mig/swa	sdm
3317	*P. pipistrellus*	female	unknown	1	2020	other	not applicable	35584	Wetzlar-Naunheim	Hessia	Germany	hib	sdm
3318	*P. pipistrellus*	unknown	unknown	3	2019	found dead	not applicable	35390	Gießen	Hessia	Germany	hib	sdm
3320	*P. pipistrellus*	female	unknown	4	2019	found dead	not applicable	36358	Herbstern	Hessia	Germany	post hib	sdm
3321	*P. pipistrellus*	male	unknown	12	2018	found dead	not applicable	35325	Mücke-Merlau	Hessia	Germany	hib	sdm
3322	*P. pipistrellus*	male	unknown	1	2019	other	not applicable	35630	Ehringshausen	Hessia	Germany	hib	sdm
3326	*P. pipistrellus*	unknown	unknown	4	2019	other	not applicable	36041	Fulda	Hessia	Germany	post hib	sdm
3327	*P. pipistrellus*	male	juvenile	9	2019	found dead	not applicable	36358	Schadger	Hessia	Germany	mig/swa	sdm
3328	*P. pipistrellus*	female	juvenile	8	2019	death in care	7	36367	Angersbach	Hessia	Germany	mig/swa	sdm
3330	*P. pipistrellus*	male	unknown	6	2019	other	not applicable	36251	Ensrode	Hessia	Germany	mat	sdm
3331	*P. pipistrellus*	female	unknown	5	2019	death in care	4	36329	Romrod	Hessia	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3332	*P. pipistrellus*	female	juvenile	9	2019	found dead	not applicable	34636	Neukirchen	Hessia	Germany	mig/swa	sdm
3333	*P. pipistrellus*	female	juvenile	4	2019	death in care	75	36110	Schlitz	Hessia	Germany	post hib	sdm
3334	*P. pipistrellus*	male	juvenile	10	2019	death in care	74	63679	Schotten	Hessia	Germany	mig/swa	sdm
3335	*P. pipistrellus*	female	unknown	4	2019	death in care	302	36093	Künzell	Hessia	Germany	post hib	sdm
3336	*P. pipistrellus*	male	juvenile	8	2019	death in care	27	36325	Feldatal	Hessia	Germany	mig/swa	sdm
3337	*P. pipistrellus*	female	adult	7	2019	death in care	3	36325	Feldatal	Hessia	Germany	mat	sdm
3338	*P. pipistrellus*	male	juvenile	8	2019	death in care	20	63679	Schotten/ Michelbach	Hessia	Germany	mig/swa	sdm
3339	*P. pipistrellus*	female	unknown	3	2019	other	not applicable	36093	Künzell	Hessia	Germany	hib	sdm
3341	*P. pipistrellus*	female	unknown	7	2020	found dead	not applicable	37534	Bad Grund (Harz)	Lower Saxony	Germany	mat	sdm
3342	*P. pipistrellus*	male	unknown	7	2020	found dead	not applicable	37431	Bad Lauterberg im Harz	Lower Saxony	Germany	mat	sdm
3343	*P. pipistrellus*	male	unknown	7	2020	found dead	not applicable	37412	Herzberg am Harz	Lower Saxony	Germany	mat	sdm
3345	*P. pipistrellus*	female	unknown	8	2020	found dead	not applicable	37520	Osterode am Harz	Lower Saxony	Germany	mig/swa	sdm
3346	*P. pipistrellus*	female	unknown	7	2020	found dead	not applicable	38704	Liebenburg	Lower Saxony	Germany	mat	sdm
3348	*P. pipistrellus*	female	unknown	6	2020	found dead	not applicable	38667	Bad Harzburg	Lower Saxony	Germany	mat	sdm
3350	*P. pipistrellus*	female	unknown	7	2020	found dead	not applicable	38642	Goslar	Lower Saxony	Germany	mat	sdm
3351	*P. pipistrellus*	female	unknown	7	2020	found dead	not applicable	37412	Herzberg am Harz	Lower Saxony	Germany	mat	sdm
3352	*P. pipistrellus*	female	unknown	8	2020	found dead	not applicable	37444	St. Andreasberg	Lower Saxony	Germany	mig/swa	sdm
3353	*P. pipistrellus*	female	unknown	8	2020	found dead	not applicable	37444	St. Andreasberg	Lower Saxony	Germany	mig/swa	sdm
3354	*P. pipistrellus*	female	unknown	8	2020	found dead	not applicable	38642	Goslar	Lower Saxony	Germany	mig/swa	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3355	*P. pipistrellus*	female	unknown	7	2020	found dead	not applicable	38835	Osterwiek	Lower Saxony	Germany	mat	sdm
3356	*P. pipistrellus*	male	unknown	9	2020	found dead	not applicable	37520	Osterode am Harz	Lower Saxony	Germany	mig/swa	sdm
3358	*P. pipistrellus*	male	unknown	7	2020	found dead	not applicable	37520	Osterode am Harz	Lower Saxony	Germany	mat	sdm
3359	*P. pipistrellus*	male	unknown	9	2020	found dead	not applicable	37412	Hörden am Harz	Lower Saxony	Germany	mig/swa	sdm
3360	*P. pipistrellus*	female	unknown	7	2020	found dead	not applicable	37520	Osterode am Harz	Lower Saxony	Germany	mat	sdm
3361	*P. pipistrellus*	male	unknown	5	2020	found dead	not applicable	44267	Dortmund	North Rhine-Westphalia	Germany	mat	sdm
3363	*P. pipistrellus*	female	unknown	8	2019	other	not applicable	38315	Schalden	Lower Saxony	Germany	mig/swa	sdm
3364	*P. pipistrellus*	female	unknown	7	2020	found dead	unknown	38855	Wernigerode	Lower Saxony	Germany	mat	sdm
3365	*P. pipistrellus*	male	unknown	7	2020	found dead	unknown	38871	Ilsenburg (Harz)	Lower Saxony	Germany	mat	sdm
3373	*P. pipistrellus*	female	unknown	7	2020	unknown	unknown	38723	Seesen	Lower Saxony	Germany	mat	sdm
3374	*P. pipistrellus*	female	unknown	12	2016	unknown	unknown	38723	Seesen	Lower Saxony	Germany	hib	sdm
3376	*P. pipistrellus*	male	unknown	8	2019	unknown	unknown	38723	Seesen	Lower Saxony	Germany	mig/swa	sdm
3379	*P. pipistrellus*	male	unknown	7	2020	death in care	unknown	38723	Seesen	Lower Saxony	Germany	mat	sdm
3381	*P. pipistrellus*	male	unknown	9	2020	death in care	unknown	38723	Seesen	Lower Saxony	Germany	mig/swa	sdm
3383	*P. pipistrellus*	male	unknown	7	2019	unknown	unknown	38723	Seesen	Lower Saxony	Germany	mat	sdm
3384	*P. pipistrellus*	male	unknown	7	2019	death in care	unknown	38685	Langelsheim	Lower Saxony	Germany	mat	sdm
3386	*P. pipistrellus*	unknown	unknown	6	2018	found dead	not applicable	16548	Glienicke	Berlin/ Brandenburg	Germany	mat	sdm
3387	*P. pipistrellus*	unknown	unknown	unknown	unknown	unknown	unknown	10711	Berlin	Berlin/ Brandenburg	Germany	unknown	sdm
3388	*P. pipistrellus*	unknown	unknown	unknown	unknown	found dead	not applicable	10777	Berlin	Berlin/ Brandenburg	Germany	unknown	sdm
3389	*P. pipistrellus*	unknown	unknown	unknown	unknown	unknown	unknown	unknown	unknown	unknown	Germany	unknown	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3390	*P. pipistrellus*	unknown	unknown	1	2019	death in care	1	10369	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3391	*P. pipistrellus*	unknown	unknown	5	2018	unknown	unknown	unknown	unknown	Berlin/ Brandenburg	Germany	mat	sdm
3392	*P. pipistrellus*	unknown	unknown	5	2019	death in care	14	12163	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3393	*P. pipistrellus*	unknown	unknown	unknown	2017	unknown	unknown	12157	Berlin	Berlin/ Brandenburg	Germany	unknown	sdm
3394	*P. pipistrellus*	unknown	unknown	unknown	unknown	unknown	unknown	unknown	unknown	unknown	Germany	unknown	sdm
3395	*P. pipistrellus*	unknown	unknown	6	2018	death in care	4	13439	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3396	*P. pipistrellus*	unknown	unknown	8	2019	death in care	4	14089	Berlin-Kladow	Berlin/ Brandenburg	Germany	mig/swa	sdm
3397	*P. pipistrellus*	unknown	unknown	2	2018	death in care	3	10829	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3398	*P. pipistrellus*	unknown	unknown	7	2019	death in care	6	14163	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3399	*P. pipistrellus*	unknown	unknown	5	2019	death in care	2	13597	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3400	*P. pipistrellus*	unknown	unknown	5	2018	death in care	2	13351	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3401	*P. pipistrellus*	unknown	unknown	8	2019	unknown	unknown	13591	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3402	*P. pipistrellus*	unknown	unknown	1	2019	death in care	3	14482	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3405	*P. pipistrellus*	unknown	unknown	6	2018	death in care	2	13591	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3406	*P. pipistrellus*	unknown	unknown	10	2019	death in care	7	16727	Velten	Berlin/ Brandenburg	Germany	mig/swa	sdm
3407	*P. pipistrellus*	unknown	unknown	6	2019	unknown	unknown	unknown	unknown	Berlin/ Brandenburg	Germany	mat	sdm
3408	*P. pipistrellus*	unknown	unknown	8	2019	death in care	1	13591	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3409	*P. pipistrellus*	unknown	unknown	3	2019	found dead	not applicable	unknown	unknown	Berlin/ Brandenburg	Germany	hib	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3410	*P. pipistrellus*	unknown	unknown	4	2018	other	not applicable	13467	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3411	*P. pipistrellus*	unknown	unknown	5	2018	death in care	15	10435	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3413	*P. pipistrellus*	unknown	unknown	4	2018	found dead	not applicable	13595	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3414	*P. pipistrellus*	unknown	unknown	8	2019	death in care	1	13593	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3415	*P. pipistrellus*	unknown	unknown	6	2019	death in care	84	14163	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3417	*P. pipistrellus*	unknown	unknown	10	2018	death in care	1	14469	Potsdam	Berlin/ Brandenburg	Germany	mig/swa	sdm
3419	*P. pipistrellus*	unknown	unknown	9	2019	death in care	11	16567	Mühlenbeck	Berlin/ Brandenburg	Germany	mig/swa	sdm
3420	*P. pipistrellus*	unknown	unknown	5	2019	found dead	not applicable	14621	Pausin	Berlin/ Brandenburg	Germany	mat	sdm
3421	*P. pipistrellus*	unknown	unknown	8	2018	death in care	1	16540	Hohen Neuendorf	Berlin/ Brandenburg	Germany	mig/swa	sdm
3423	*P. pipistrellus*	unknown	unknown	8	2019	death in care	1	13587	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3425	*P. pipistrellus*	unknown	unknown	8	2019	death in care	5	13591	Berlin	Berlin/ Brandenburg	Germany	mig/swa	sdm
3426	*P. pipistrellus*	unknown	unknown	9	2019	unknown	unknown	unknown	unknown	Berlin/ Brandenburg	Germany	mig/swa	sdm
3427	*P. pipistrellus*	unknown	unknown	2	2019	death in care	1	14057	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3428	*P. pipistrellus*	unknown	unknown	6	2018	death in care	2	14199	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3429	*P. pipistrellus*	unknown	unknown	4	2018	found dead	not applicable	10589	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3430	*P. pipistrellus*	unknown	unknown	4	2019	death in care	18	14109	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3433	*P. pipistrellus*	unknown	unknown	5	2019	unknown	unknown	10557	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3434	*P. pipistrellus*	unknown	unknown	7	2018	death in care	2	14641	Paulinenaue	Berlin/ Brandenburg	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3435	*P. pipistrellus*	unknown	unknown	5	2018	death in care	1	12105	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3460	*P. pipistrellus*	female	adult	3	2021	other	not applicable	37520	Osterode am Harz	Lower Saxony	Germany	hib	sdm
3475	*P. pipistrellus*	female	adult	4	2019	other	not applicable	14129	Berlin	Berlin/ Brandenburg	Germany	post hib	sdm
3476	*P. pipistrellus*	male	adult	11	2018	other	not applicable	10437	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3477	*P. pipistrellus*	male	adult	3	2019	found dead	not applicable	10711	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3478	*P. pipistrellus*	male	adult	12	2019	other	not applicable	13469	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3479	*P. pipistrellus*	male	adult	6	2020	death in care	1	31632	Husum	Lower Saxony	Germany	mat	sdm
3480	*P. pipistrellus*	female	adult	5	2020	death in care	2	31556	Wölpinghausen	Lower Saxony	Germany	mat	sdm
3481	*P. pipistrellus*	unknown	unknown	4	2020	death in care	3	37520	Osterode	Lower Saxony	Germany	post hib	sdm
3482	*P. pipistrellus*	unknown	unknown	7	2020	other	not applicable	31840	Hessisch Oldendorf	Lower Saxony	Germany	mat	sdm
3505	*P. pipistrellus*	unknown	unknown	7	2019	death in care	2	13403	Berlin	Berlin/ Brandenburg	Germany	mat	sdm
3032	*P. auritus*	unknown	unknown	10	2018	unknown	unknown	36367	Wartenberg	Hessia	Germany	mig/swa	sdm
3072	*P. auritus*	unknown	juvenile	8	2018	death in care	unknown	59457	Werl	North Rhine-Westphalia	Germany	mig/swa	sdm
3144	*P. auritus*	female	unknown	4	2018	other	not applicable	56729	Ettringen	Rhineland-Palatinate	Germany	post hib	sdm
3154	*P. auritus*	male	unknown	10	2019	other	not applicable	95339	Neuenmarkt	Bavaria	Germany	mig/swa	sdm
3196	*P. auritus*	male	unknown	2	2009	unknown	unknown	36123	Eiterfeld	Hessia	Germany	hib	sdm
3201	*P. auritus*	male	unknown	1	2002	unknown	unknown	36037	Fulda	Hessia	Germany	hib	sdm
3212	*P. auritus*	male	unknown	unknown	2008	unknown	unknown	36129	Gersfeld	Hessia	Germany	unknown	sdm
3267	*P. auritus*	female	adult	8	2019	found dead	not applicable	01468	Moritzburg	Saxony	Germany	mig/swa	sdm
3296	*P. auritus*	male	adult	3	2018	death in care	2	01705	Freital	Saxony	Germany	hib	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3300	*P. auritus*	female	juvenile	7	2017	found dead	not applicable	01819	Bahretal	Saxony	Germany	mat	sdm
3329	*P. auritus*	male	unknown	7	2019	found dead	not applicable	36341	Lauterbach	Hessia	Germany	mat	sdm
3357	*P. auritus*	female	unknown	8	unknown	found dead	not applicable	37284	Waldkappel	Lower Saxony	Germany	mig/swa	sdm
3372	*P. auritus*	unknown	unknown	7	2016	unknown	unknown	unknown	unknown	unknown	Germany	mat	sdm
3461	*P. auritus*	female	adult	10	2020	other	not applicable	31547	Rehburg-Loccum	Lower Saxony	Germany	mig/swa	sdm
3491	*P. auritus*	female	adult	1	2017	death in care	2	12249	Berlin	Berlin/ Brandenburg	Germany	hib	sdm
3519	*P. auritus*	female	adult	5	2020	found dead	not applicable	72184	Eutingen im Gäu	Baden-Wuerttemberg	Germany	mat	sdm
3524	*P. auritus*	male	adult	9	2019	unknown	unknown	26802	Moormerland	Lower Saxony	Germany	mig/swa	sdm
3548	*P. auritus*	male	adult	9	2020	unknown	unknown	unknown	unknown	Lower Saxony	Germany	mig/swa	sdm
3549	*P. auritus*	male	adult	8	2021	death in care	5	26835	Hesel	Lower Saxony	Germany	mig/swa	sdm
3550	*P. auritus*	male	adult	3	2019	death in care	9	26624	Südbrookmer-land	Lower Saxony	Germany	hib	sdm
3551	*P. auritus*	male	adult	7	2021	death in care	2	26624	Südbrookmer-land	Lower Saxony	Germany	mat	sdm
3552	*P. auritus*	unknown	adult	12	2018	death in care	10	26553	Dornum	Lower Saxony	Germany	hib	sdm
3553	*P. auritus*	male	adult	8	2021	unknown	unknown	26629	Großefehn	Lower Saxony	Germany	mig/swa	sdm
3554	*P. auritus*	male	adult	8	2021	unknown	unknown	26835	Hesel	Lower Saxony	Germany	mig/swa	sdm
3555	*P. auritus*	male	adult	8	2021	death in care	3	26655	Westerstede	Lower Saxony	Germany	mig/swa	sdm
3557	*P. auritus*	male	adult	8	2021	unknown	unknown	26835	Holtland	Lower Saxony	Germany	mig/swa	sdm
3558	*P. auritus*	male	adult	5	2019	unknown	unknown	26736	Krummhörn	Lower Saxony	Germany	mat	sdm
3559	*P. auritus*	unknown	adult	10	2019	death in care	1	26607	Aurich	Lower Saxony	Germany	mig/swa	sdm
3560	*P. auritus*	female	adult	5	2021	death in care	1	26670	Uplengen	Lower Saxony	Germany	mat	sdm
3561	*P. auritus*	male	adult	7	2019	unknown	unknown	26639	Wiesmoor	Lower Saxony	Germany	mat	sdm
3562	*P. auritus*	male	adult	5	2021	death in care	1	26835	Holtland	Lower Saxony	Germany	mat	sdm

No.	Species	Sex	Age	Month of sampling	Year of sampling	Circumstances of death	Days in care	Postal code	Town	State	Country	Bat season	Move type
3563	*P. auritus*	female	adult	6	2021	unknown	unknown	26603	Aurich	Lower Saxony	Germany	mat	sdm

Supporting Table S2 List of analysed compounds including name, pesticide type, retention time (RT), dMRM transitions, collision energy (CE) and LOQ (limit of quantification). Quantifier transitions are marked in bold. OC: organochlorine, PCB: polychlorinated biphenyl.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
2,4-D-ethyl ester	Herbicide	7.49	**247.9 → 185.0** 185.0 → 114.9 175.0 → 111.0	10 25 10	n.d.
2-Phenylphenol	Other (Microbiocide)	6.25	169.1 → 91.0 **141.1 → 63.0** 115.1 → 65.0	35 45 25	n.d.
8-Hydroxyquinoline	Fungicide, Microbiocide	5.38	145.0 → 63.0 117.0 → 63.0 **117.0 → 39.1**	40 40 40	n.d.
Acequinocyl	Other Insecticide	16.77	342.9 → 188.8 **341.9 → 187.9** 187.9 → 131.0	20 15 20	n.d.
Acetamiprid	Other Insecticide	13.85	221.0 → 56.1 126.0 → 90.0 **126.0 → 72.9**	15 5 20	n.d.
Acibenzolar-S-methyl	Fungicide	9.30	182.0 → 167.1 182.0 → 153.1 **182.0 → 135.0**	10 10 15	n.d.
Aclonifen	Herbicide	12.39	264.1 → 194.2 **194.1 → 167.1** 194.1 → 139.1	15 20 25	n.d.
Acrinathrin	Other Insecticide	15.02	**288.9 → 92.8** 207.8 → 152.0 181.0 → 127.0	10 35 30	n.d.
Aldrin	OC Insecticide	9.94	262.9 → 192.9 262.9 → 190.9 254.9 → 220.0	35 35 20	n.d.
Ametoctradin	Fungicide	15.20	275.0 → 246.2 275.0 → 190.3 **246.0 → 188.2**	0 15 25	20
Amisulbrom	Fungicide	16.14	227.9 → 147.0 **225.9 → 147.0** 214.0 → 160.0	15 15 20	4
Azoxystrobin	Fungicide	18.30	344.1 → 182.9 **344.1 → 171.9** 344.1 → 155.8	25 40 40	4
Azoxystrobin-D_4	Internal Standard	18.29	407.0 → 348.0 392.0 → 364.0 **348.0 → 172.1**	5 5 35	n.a.
Beflubutamid	Herbicide	10.67	192.9 → 145.1 192.9 → 95.0 **176.1 → 79.1**	15 35 25	n.d.
Benalaxyl	Fungicide	12.87	266.0 → 148.1 **233.9 → 146.0** 206.0 → 162.1	5 20 5	n.d.
Bentazone	Herbicide	10.11	**225.0 → 181.9** 198.0 → 92.0 182.0 → 90.0	5 30 15	n.d.
Benthiavalicarb-isopropyl	Fungicide	14.57	222.0 → 125.9 **180.0 → 127.0** 180.0 → 83.0	40 20 30	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Bifenazate	Other Insecticide	13.94	184.1 → 91.1 **184.1 → 77.0** 168.1 → 140.1	40 40 10	20.
Bifenox	Herbicide	14.21	340.9 → 309.9 **340.9 → 280.9** 189.1 → 126.0	10 15 20	n.d.
Bifenthrin	Other Insecticide	13.83	**181.0 → 115.1** 166.0 → 139.1 166.0 → 115.1	45 35 35	4
Boscalid	Fungicide	16.50	140.0 → 112.0 **140.0 → 76.0** 111.9 → 76.0	10 25 15	1
Bromoxynil	Herbicide	7.41	276.8 → 88.0 **274.7 → 167.9** 274.7 → 88.0	30 15 30	n.d.
Bromuconazole (2 isomers)	Fungicide	13.85 14.29	295.0 → 172.9 **293.0 → 172.9** 173.0 → 109.0	10 10 30	n.d.
Bupirimate	Fungicide	11.80	**315.8 → 207.9** 208.0 → 68.9 193.0 → 109.0	10 30 15	n.d.
Buprofezin	Other Insecticide	11.74	**304.9 → 175.0** 249.1 → 193.0 171.1 → 115.0	10 10 10	n.d.
Captan	Fungicide	10.73	**263.8 → 79.0** 149.0 → 70.0 116.9 → 82.0	15 15 30	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Carbetamide	Herbicide	9.95	120.1 → 92.0 120.1 → 77.0 **119.1 → 64.1**	10 15 25	20
Carboxin	Fungicide	11.75	**234.9 → 143.0** 234.9 → 87.0 131.9 → 77.0	10 20 20	n.d.
Carfentrazone-ethyl	Herbicide	12.81	**339.9 → 311.9** 329.9 → 309.9 311.9 → 150.8	10 10 20	n.d.
***cis*-Chlordane**	OC Insecticide	11.20	374.8 → 265.8 **372.8 → 265.8** 271.7 → 236.9	15 15 15	1
***trans*-Chlordane**	OC Insecticide	10.94	374.8 → 265.8 **372.8 → 265.8** 271.7 → 236.9	15 15 15	1
Chloridazon (Pyrazon)	Herbicide	13.04	221.0 → 220.2 **220.0 → 193.1** 220.0 → 166.0	5 20 25	n.d.
Chlorothalonil	Fungicide	8.54	265.9 → 230.9 **265.9 → 133.0** 265.9 → 109.0	20 45 45	n.d.
Chlorotoluron	Herbicide	9.70	**212.1 → 166.0** 212.1 → 72.0 167.0 → 132.1	10 15 15	n.d.
Chlorpropham	Herbicide, Plant growth regulator	7.11	**213.0 → 171.1** 171.0 → 127.1 153.0 → 90.0	5 5 25	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Chlorpyrifos	Other Insecticide	9.86	**313.8 → 257.8** 196.9 → 107.0 196.9 → 98.0	15 40 30	1
Chlorpyrifos-D_{10}	Internal Standard	9.80	325.9 → 262.1 **323.9 → 260.0** 259.8 → 167.0	10 10 15	n.a.
Chlorpyrifos-methyl	Other Insecticide	9.14	287.9 → 92.9 **285.9 → 93.0** 124.9 → 47.0	20 25 15	1
Clodinafop-propargyl	Other (Plant growth regulator)	12.97	348.9 → 265.9 **348.9 → 237.8** 238.0 → 130.0	10 15 15	n.d.
Clomazone	Herbicide	7.98	**205.1 → 107.1** 127.0 → 101.0 125.0 → 89.0	20 20 15	n.d.
Cloquintocet-mexyl	Other (Herbicide safener)	14.00	220.0 → 191.9 **163.0 → 128.0** 163.0 → 101.0	10 15 30	n.d.
Cyflufenamid	Fungicide	11.88	188.1 → 88.0 **118.1 → 90.0** 118.1 → 89.0	35 10 25	4
Cyfluthrin (3 isomers)	Other Insecticide	16.17 16.25 16.37	206.0 → 176.9 206.0 → 150.0 **162.9 → 127.0**	25 40 5	n.d.
Cyhalofop-butyl	Herbicide	14.68	**357.1 → 229.1** 256.2 → 120.1 229.2 → 109.1	15 10 15	n.d.
Cyhalothrin (*gamma* and *lambda* isomer)	Other Insecticide	14.79 14.60	**208.0 → 181.0** 208.0 → 152.0 197.0 → 161.1	5 25 5	4
Cypermethrin (3 isomers)	Other Insecticide	16.39 16.48 16.57	165.0 → 127.1 165.0 → 91.1 **162.9 → 127.0**	0 10 0	4
Cyproconazole	Fungicide	11.99	**222.0 → 124.9** 138.9 → 111.0 138.9 → 75.0	25 15 35	n.d.
Cyprodinil	Fungicide	10.39	225.2 → 224.3 **224.2 → 131.1** 210.0 → 93.0	10 15 20	n.d.
Cyromazine	Other Insecticide	7.97	**165.9 → 109.0** 151.0 → 82.0 109.0 → 68.0	20 30 20	n.d.
Dazomet	Fungicide	7.76	161.9 → 89.0 **89.0 → 46.0** 88.9 → 74.0	25 5 15	n.d.
***o,p'*-DDD**	OC Insecticide (Breakdown)	11.78	235.0 → 200.1 235.0 → 139.1 **199.1 → 164.1**	10 45 20	n.d.
***p,p'*-DDD**	OC Insecticide (Breakdown)	12.36	237.0 → 200.1 **199.1 → 164.1** 165.1 → 139.0	15 20 35	n.d.
***o,p'*-DDE**	OC Insecticide (Breakdown)	10.98	317.8 → 248.0 248.0 → 176.2 **246.0 → 176.2**	15 30 30	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
***p,p'*-DDE**	OC Insecticide (Breakdown)	11.52	317.8 → 246.0 315.8 → 246.0 **246.1 → 176.2**	15 15 30	1
***o,p'*-DDT**	OC Insecticide	12.27	237.0 → 199.1 **235.0 → 199.1** 199.0 → 163.1	15 15 35	n.d.
***p,p'*-DDT**	OC Insecticide	12.94	237.0 → 165.2 235.0 → 199.2 **235.0 → 165.2**	20 15 20	2
Deltamethrin	Other Insecticide	18.02	252.9 → 174.0 **252.9 → 93.1** 251.0 → 172.0	0 15 0	4
Desmedipham	Herbicide	7.59	181.0 → 122.0 **181.0 → 109.0** 135.0 → 52.0	10 10 25	n.d.
Diazinon	Other Insecticide	8.29	276.0 → 137.1 **199.1 → 135.1** 179.1 → 137.1	25 10 20	n.d.
Dicamba-methyl ester	Herbicide	6.26	234.0 → 173.0 **205.0 → 149.0** 175.0 → 111.0	20 15 20	n.d.
Diclofop-methyl	Herbicide	13.26	339.9 → 252.9 **280.8 → 119.9** 253.0 → 162.1	10 10 15	n.d.
Dieldrin	OC Insecticide	11.62	**277.0 → 241.0** 262.9 → 193.0 262.9 → 191.0	5 35 35	2

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Diethofencarb	Fungicide	9.76	225.0 → 96.0 **207.0 → 179.1** 207.0 → 151.0	30 5 15	n.d.
Difenoconazole (2 isomers)	Fungicide	17.72 17.78	324.8 → 266.8 **322.8 → 264.8** 264.9 → 202.0	15 15 20	4
Diflubenzuron	Other Insecticide	5.01	141.0 → 113.0 **141.0 → 63.0** 113.0 → 63.0	40 40 40	4
Diflufenican	Herbicide	13.29	**393.9 → 265.9** 266.0 → 246.1 218.0 → 140.1	10 15 20	n.d.
Dimethachlor	Herbicide	8.99	209.9 → 134.1 **196.9 → 148.2** 134.1 → 79.1	10 10 20	n.d.
Dimethenamide-P	Herbicide	9.02	229.9 → 154.0 **229.9 → 111.0** 202.9 → 154.0	10 25 10	n.d.
Dimethoate	Other Insecticide	7.79	228.7 → 87.0 157.0 → 93.0 **157.0 → 63.0**	5 10 25	n.d.
Dimethomorph (2 isomers)	Fungicide	18.35 18.66	302.9 → 164.9 **300.9 → 165.0** 300.9 → 138.8	10 10 15	4
Dimoxystrobin	Fungicide	13.85	237.0 → 116.0 **205.0 → 116.0** 174.0 → 115.0	15 10 30	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Diuron	Herbicide	10.66	231.7 → 71.8 **186.9 → 124.0** 158.9 → 123.9	15 20 10	n.d.
Dodemorph (2 isomers)	Fungicide	10.24 10.55	**281.0 → 154.0** 238.1 → 55.1 154.0 → 112.1	10 20 10	n.d.
Epoxiconazole	Fungicide	13.52	**192.0 → 138.1** 192.0 → 111.0 138.0 → 75.0	10 25 25	1
Ethofenprox	Other Insecticide	16.78	183.0 → 168.0 **163.0 → 135.1** 163.0 → 107.1	10 10 20	n.d.
Ethofumesate	Herbicide	9.61	285.9 → 207.1 **178.9 → 137.1** 178.9 → 105.1	5 0 15	n.d.
Ethoprophos	Other Insecticide	7.02	199.9 → 97.0 **157.9 → 97.0** 157.9 → 81.0	20 15 15	n.d.
Etoxazole	Other Insecticide	14.07	329.9 → 315.0 299.9 → 284.9 **299.9 → 269.9**	20 10 20	n.d.
Etridiazole	Fungicide	5.85	211.1 → 183.0 211.1 → 140.0 **185.0 → 142.0**	10 25 15	n.d.
Famoxadone	Fungicide	18.44	329.9 → 329.0 **329.9 → 223.9** 223.9 → 196.2	10 10 10	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Fenamiphos	Other Insecticide	11.31	302.9 → 287.9 302.9 → 153.9 **287.9 → 259.7**	10 15 5	n.d.
Fenazaquin	Other Insecticide	14.19	**160.0 → 145.2** 160.0 → 117.1 146.0 → 118.1	5 20 10	2
Fenbuconazole	Fungicide	16.21	197.9 → 129.0 197.9 → 102.0 **125.0 → 89.0**	5 30 20	n.d.
Fenhexamid	Fungicide	12.97	301.0 → 97.0 179.0 → 115.0 **177.1 → 113.0**	15 15 15	2
Fenoxaprop-P-ethyl	Herbicide	15.33	360.8 → 287.8 **287.8 → 118.8** 287.8 → 90.9	10 10 20	n.d.
Fenoxycarb	Other Insecticide	13.86	256.1 → 187.2 **186.2 → 109.0** 186.2 → 77.1	10 15 20	n.d.
Fenpropidin	Fungicide	9.45	**273.0 → 98.0** 145.0 → 117.0 145.0 → 91.0	5 10 25	4
Fenpropimorph	Fungicide	9.81	128.1 → 110.1 128.1 → 86.1 **128.1 → 70.1**	5 10 10	4
Fenpyroximate	Other Insecticide	7.85	212.0 → 185.0 **212.0 → 76.9** 198.1 → 114.0	40 40 35	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Fenvalerate (2 isomers)	Other Insecticide	17.31 17.50	419.1 → 166.8 **167.0 → 125.1** 167.0 → 89.0	10 10 40	2
Fipronil	Other Insecticide	10.64	**366.8 → 212.8** 350.8 → 254.8 254.9 → 228.0	25 15 15	n.d.
Fipronil sulfide	Other Insecticide (Breakdown)	10.50	420.0 → 350.9 **351.0 → 254.9** 254.9 → 156.9	10 20 35	n.d.
Fipronil sulfone	Other Insecticide (Breakdown)	11.71	384.8 → 256.8 **382.8 → 254.9** 254.9 → 227.9	20 20 15	2
Fluazifop-p-butyl	Herbicide	11.97	382.9 → 282.0 **281.9 → 238.0** 254.0 → 146.1	10 15 15	1
Fludioxonil	Fungicide	11.51	248.0 → 182.1 248.0 → 154.1 **248.0 → 127.1**	10 20 30	1
Flufenacet	Herbicide	9.96	211.0 → 123.0 **211.0 → 96.0** 183.0 → 69.0	5 15 20	n.d.
Flumetralin	Herbicide	11.19	403.9 → 156.8 359.9 → 313.9 **157.0 → 109.0**	15 15 25	n.d.
Flumioxazin	Herbicide	17.43	354.0 → 325.9 354.0 → 175.8 **287.0 → 258.7**	5 15 15	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Fluometuron	Herbicide	6.98	232.0 → 72.0 **213.0 → 167.9** 187.0 → 109.0	15 10 20	n.d.
Fluopyram	Fungicide	10.57	395.9 → 223.1 **222.9 → 196.0** 222.9 → 187.1	5 10 10	1
Fluorochloridone	Herbicide	10.11	311.0 → 174.1 **311.0 → 102.9** 187.1 → 109.1	15 15 20	n.d.
Flupyradifurone	Other Insecticide	14.87	288.0 → 126.1 128.0 → 90.0 **126.0 → 73.0**	15 10 25	20
Fluquinconazole	Fungicide	15.85	342.0 → 107.8 **340.0 → 298.0** 340.0 → 107.8	40 15 40	n.d.
Fluroxypyr-meptyl	Herbicide	13.29	237.0 → 209.0 **237.0 → 181.0** 208.9 → 178.9	5 15 20	n.d.
Flurtamone	Herbicide	14.43	**332.7 → 120.0** 157.0 → 137.1 157.0 → 107.0	15 15 25	n.d.
Flutolanil	Herbicide	11.38	322.9 → 281.0 **280.9 → 173.0** 173.0 → 95.0	5 10 30	n.d.
Flutriafol	Fungicide	11.30	**219.1 → 123.1** 219.1 → 95.0 164.1 → 109.1	15 35 20	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
***tau*-Fluvalinate (2 isomers)**	Other Insecticide	17.48 17.52	252.0 → 200.0 **250.0 → 200.1** 250.0 → 198.1	15 15 40	4
Fluxapyroxad	Fungicide	14.57	**321.1 → 152.9** 222.0 → 152.9 222.0 → 125.9	35 15 40	n.d.
Fosthiazate (2 isomers)	Other (Nematicide)	10.27 10.31	**199.0 → 102.0** 195.0 → 60.0 165.9 → 106.0	5 20 10	n.d.
Fuberidazole	Fungicide	9.16	184.0 → 155.1 **156.0 → 103.1** 155.0 → 129.1	30 20 10	n.d.
Haloxyfop-P-methyl	Herbicide	10.93	375.1 → 316.0 **375.1 → 91.1** 288.0 → 180.0	10 35 25	n.d.
***alpha*-HCH**	OC Insecticide	7.64	218.9 → 183.0 **216.9 → 181.0** 180.9 → 145.0	5 5 15	n.d.
***beta*-HCH**	OC Insecticide	7.99	218.9 → 183.1 **216.9 → 181.1** 181.0 → 145.0	5 5 15	1
***gamma*-HCH (Lindane)**	OC Insecticide	8.08	218.9 → 183.1 **216.9 → 181.0** 181.0 → 145.0	5 5 15	1
***delta*-HCH**	OC Insecticide	8.51	**217.0 → 181.1** 183.1 → 147.1 181.1 → 145.1	5 15 15	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
***epsilon*-HCH**	OC Insecticide	8.69	254.0 → 180.9 218.9 → 182.9 **182.9 → 109.0**	10 5 30	n.d.
Heptachlor	OC Insecticide	9.34	273.7 → 238.9 273.7 → 236.9 **271.7 → 236.9**	15 15 15	1
Heptachlor endo-epoxide	OC Insecticide (Breakdown)	10.67	216.9 → 182.0 216.9 → 109.0 **183.0 → 119.0**	20 45 30	n.d.
Heptachlor exo-epoxide	OC Insecticide (Breakdown)	10.61	**354.8 → 264.9** 352.8 → 262.9 262.9 → 193.0	15 15 35	n.d.
Hexachlorobenzene	Fungicide	7.70	**283.8 → 213.9** 281.8 → 211.9 248.9 → 179.0	30 30 30	n.d.
Imazalil	Fungicide	11.48	216.8 → 175.0 174.9 → 147.0 **172.9 → 109.0**	5 15 30	n.d.
Imidacloprid	Other Insecticide	11.31	211.0 → 113.0 126.0 → 89.9 **126.0 → 73.0**	15 5 25	20
Indoxacarb	Other Insecticide	18.02	**264.0 → 175.8** 202.9 → 134.0 202.9 → 106.0	15 20 15	n.d.
Ipconazole	Fungicide	15.00	249.0 → 125.0 167.0 → 125.0 **125.0 → 89.0**	15 5 20	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Iprovalicarb (2 isomers)	Fungicide	11.61 11.78	**158.0 → 98.0** 134.1 → 93.0 116.0 → 98.1	10 15 5	n.d.
Isopyrazam	Fungicide	15.30	359.0 → 159.0 **302.1 → 262.1** 159.0 → 139.0	40 15 10	n.d.
Isoxaben	Herbicide	15.17	165.0 → 150.0 165.0 → 107.0 **149.9 → 121.9**	15 25 5	n.d.
Kresoxim-methyl	Fungicide	11.81	206.0 → 131.1 **206.0 → 116.0** 116.0 → 89.0	10 5 15	n.d.
Lenacil	Herbicide	12.95	233.9 → 153.1 153.1 → 110.1 **153.1 → 82.1**	5 20 20	2
Lufenuron	Other Insecticide	5.58	251.6 → 157.8 **202.9 → 75.9** 173.9 → 109.9	15 40 30	n.d.
Malathion	Other Insecticide	9.73	172.9 → 117.0 **172.9 → 99.0** 157.8 → 125.0	15 10 5	n.d.
MCPA-methyl ester	Herbicide	6.51	214.1 → 155.1 214.1 → 141.1 **155.1 → 125.1**	10 10 10	n.d.
MCPB-methyl ester	Herbicide	8.17	211.1 → 155.0 **142.1 → 107.1** 142.1 → 77.1	10 10 30	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Mefenpyr-diethyl	Other (Herbicide safener)	13.59	**299.0 → 252.9** 253.0 → 190.0 253.0 → 189.0	10 20 30	n.d.
Mepanipyrim	Fungicide	11.16	222.2 → 158.1 221.2 → 220.2 **207.1 → 179.1**	25 15 25	n.d.
Metalaxyl	Fungicide	9.33	234.0 → 146.1 **220.0 → 160.1** 206.1 → 162.1	20 10 5	n.d.
Metamitron	Herbicide	11.83	**202.1 → 186.1** 202.1 → 104.1 173.1 → 132.1	5 15 10	n.d.
Metazachlor	Herbicide	10.45	209.0 → 133.2 **209.0 → 132.2** 209.0 → 117.1	10 15 35	1
Metconazole	Fungicide	14.22	153.1 → 125.0 153.1 → 70.0 **125.0 → 89.0**	10 5 20	n.d.
Methiocarb	Other Insecticide	9.58	169.0 → 154.1 168.0 → 109.1 **153.0 → 91.1**	10 15 20	n.d.
Metobromuron	Herbicide	8.79	258.0 → 61.0 **196.9 → 89.9** 169.9 → 142.9	10 25 20	n.d.
***(S)*-Metolachlor**	Herbicide	9.89	238.0 → 162.2 **238.0 → 133.2** 162.1 → 133.2	10 30 15	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Metrafenone	Fungicide	15.24	**394.8 → 364.8** 376.9 → 346.8 226.9 → 169.0	15 20 10	4
Metribuzin	Herbicide	9.00	**198.0 → 82.0** 198.0 → 55.0 182.0 → 114.9	15 30 10	n.d.
Myclobutanil	Fungicide	11.68	**179.0 → 125.1** 179.0 → 90.0 150.0 → 123.0	10 30 15	2
Napropamide	Herbicide	11.40	**271.0 → 100.1** 271.0 → 72.1 128.0 → 100.1	15 15 10	n.d.
Oryzalin	Herbicide	15.51	316.8 → 274.9 **275.0 → 217.0** 258.0 → 193.9	5 5 5	n.d.
Oxadiazon	Herbicide	11.63	**301.8 → 175.0** 257.8 → 112.0 174.9 → 112.0	15 30 15	n.d.
Oxamyl	Other Insecticide, Nematicide	6.30	162.0 → 114.9 145.0 → 71.9 **145.0 → 60.9**	10 20 10	n.d.
Oxychlordane	OC Insecticide (Breakdown)	10.53	386.7 → 262.7 236.9 → 142.9 **184.9 → 121.0**	15 25 15	2
Oxyfluorfen	Herbicide	11.71	299.9 → 222.8 252.0 → 196.0 **252.0 → 146.0**	15 20 30	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Paclobutrazol	Other (Plant growth regulator)	11.09	236.0 → 167.1 **167.1 → 132.1** 125.1 → 89.0	10 10 20	n.d.
Parathion	Other Insecticide	9.97	**291.0 → 137.1** 291.0 → 109.0 139.0 → 81.0	5 15 15	n.d.
Parathion-methyl	Insecticide, Nematicide	9.14	262.9 → 109.0 262.9 → 79.0 **109.0 → 79.0**	10 30 5	n.d.
PCB 28	PCB	9.04	**258.0 → 186.0** 256.0 → 186.0 186.0 → 151.0	25 25 25	1
PCB 52	PCB	9.61	291.9 → 221.9 **289.9 → 219.9** 255.0 → 220.0	25 25 10	1
PCB 101	PCB	11.12	325.9 → 255.9 325.9 → 253.9 **253.9 → 184.0**	35 30 30	1
PCB 138	PCB	13.12	**361.9 → 289.9** 359.9 → 289.9 287.9 → 217.9	30 30 40	1
PCB 153	PCB	12.62	361.9 → 289.9 **359.9 → 289.9** 287.9 → 217.9	25 25 40	1
PCB 180	PCB	14.30	395.8 → 325.8 393.8 → 358.8 **393.8 → 323.8**	30 15 30	1

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Penconazole	Fungicide	10.54	250.0 → 194.1 **250.0 → 157.1** 159.0 → 123.0	15 25 20	n.d.
Pendimethalin	Herbicide	10.52	**251.8 → 162.2** 251.8 → 146.1 161.9 → 147.0	10 20 10	n.d.
Pentachloro-nitrobenzene	Fungicide, Nematicide	8.20	294.8 → 236.8 248.8 → 213.8 **141.9 → 106.9**	15 15 30	1
Permethrin (*cis* and *trans* isomer)	Other Insecticide	15.51 15.63	165.0 → 127.0 **162.9 → 127.0** 162.9 → 91.0	0 0 10	4
Phenmedipham	Herbicide	7.08	**167.0 → 135.0** 167.0 → 122.0 122.0 → 94.0	15 15 15	n.d.
Phosmet	Other Insecticide	13.90	**161.0 → 134.0** 161.0 → 78.0 160.0 → 133.1	10 20 10	n.d.
Phosmet-oxon	Other Insecticide (Breakdown)	13.00	**301.0 → 191.8** 172.9 → 104.0 160.0 → 133.0	10 15 15	n.d.
Picloram-methyl ester	Herbicide	9.55	**198.0 → 163.1** 198.0 → 161.0 196.0 → 181.0	15 15 15	n.d.
Picolinafen	Herbicide	13.87	376.0 → 239.1 **376.0 → 238.1** 238.1 → 145.1	10 20 25	2

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Picoxystrobin	Fungicide	11.29	**334.9 → 172.9** 302.8 → 156.9 145.0 → 102.1	10 15 25	n.d.
Pirimicarb	Other Insecticide	8.73	**238.0 → 166.2** 166.0 → 71.1 152.0 → 123.0	10 25 10	1
Pirimiphos-methyl	Other Insecticide	9.58	290.0 → 125.0 **232.9 → 151.0** 232.9 → 125.0	20 5 5	n.d.
Prochloraz	Fungicide	15.91	310.0 → 69.8 **266.0 → 69.9** 180.0 → 68.9	15 10 15	n.d.
Propamocarb	Fungicide	5.39	188.0 → 58.0 **143.0 → 99.1** 129.1 → 84.1	10 10 5	n.d.
Propaquizafop	Herbicide	19.92	298.8 → 254.8 **162.9 → 135.8** 162.9 → 99.9	25 10 20	n.d.
Propiconazole (2 isomers)	Fungicide	12.89 13.00	258.8 → 172.9 172.9 → 109.0 **172.9 → 74.0**	15 30 45	2
Prosulfocarb	Herbicide	9.37	251.0 → 218.3 **251.0 → 128.2** 251.0 → 100.1	10 5 5	n.d.
Prothioconazole-desthio	Fungicide	11.91	**186.0 → 89.0** 186.0 → 70.0 125.0 → 99.0	10 10 20	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Pymetrozine	Other Insecticide	11.51	132.0 → 105.0 132.0 → 78.0 **113.0 → 98.0**	10 20 5	n.d.
Pyraclostrobin	Fungicide	17.46	**324.8 → 131.7** 164.0 → 132.1 110.8 → 75.0	15 10 15	n.d.
Pyraflufen-ethyl	Herbicide	13.03	412.0 → 349.0 349.0 → 307.0 **338.9 → 288.9**	10 15 15	n.d.
Pyridaben	Other Insecticide	15.77	**309.0 → 147.1** 147.2 → 132.2 147.2 → 117.1	15 10 20	n.d.
Pyridalyl	Other Insecticide	16.73	204.0 → 148.0 164.0 → 146.0 **146.0 → 126.0**	25 15 10	n.d.
Pyridate	Herbicide	17.26	**205.2 → 141.1** 205.2 → 114.0 205.2 → 102.0	25 35 30	n.d.
Pyrimethanil	Fungicide	8.24	198.0 → 183.1 198.0 → 158.1 **198.0 → 118.1**	15 20 35	1
Pyriproxyfen	Other Insecticide	14.61	**321.0 → 222.0** 321.0 → 153.0 136.1 → 96.0	10 25 15	n.d.
Quinoclamine	Herbicide	9.76	209.0 → 172.1 207.0 → 172.1 **172.0 → 89.0**	10 20 20	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Quinoxyfen	Fungicide	12.85	306.8 → 237.0 271.9 → 237.1 **237.0 → 208.0**	20 10 30	1
Spirodiclofen	Other Insecticide	15.56	312.1 → 259.0 **312.1 → 108.9** 157.0 → 73.0	10 15 25	2
Spiromesifen	Other Insecticide	13.71	272.0 → 209.2 **253.8 → 185.1** 231.0 → 157.1	10 15 15	n.d.
Spiroxamine (2 Isomers)	Fungicide	9.08 9.53	**198.0 → 126.1** 126.0 → 84.0 100.0 → 58.1	5 5 10	n.d.
Tebuconazole	Fungicide	13.22	**250.0 → 125.0** 125.0 → 99.0 125.0 → 89.0	20 20 15	2
Tebufenpyrad	Other Insecticide	14.09	332.9 → 171.0 318.0 → 131.0 **275.9 → 171.1**	15 15 10	n.d.
Tefluthrin	Other Insecticide	8.41	199.0 → 161.1 **197.0 → 161.1** 177.1 → 127.1	5 5 15	n.d.
Terbuthylazine	Herbicide, Microbiocide	8.12	228.9 → 138.0 214.0 → 71.0 **172.9 → 138.1**	15 20 5	1
Terbuthylazine-desethyl	Herbicide, Microbiocide	7.36	186.2 → 104.0 186.2 → 83.1 **145.1 → 110.1**	15 20 10	1

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Tetraconazole	Fungicide	9.99	336.0 → 217.9 **336.0 → 203.8** 170.9 → 136.0	20 30 10	2
Thiabendazole	Fungicide	10.73	**201.9 → 175.0** 201.0 → 130.0 173.9 → 65.0	15 30 30	n.d.
Thiacloprid	Other Insecticide	17.50	126.0 → 99.1 **126.0 → 90.1** 126.0 → 73.0	10 5 20	n.d.
Tolclofos-methyl	Fungicide	9.14	**267.0 → 252.0** 267.0 → 93.0 267.0 → 63.0	15 30 45	2
Tralkoxydim	Herbicide	14.75	282.1 → 226.0 268.2 → 143.0 **226.0 → 143.0**	10 40 25	n.d.
Triadimenol	Fungicide	10.73	**129.9 → 102.0** 129.9 → 65.0 112.0 → 58.0	15 25 10	n.d.
Triallate	Herbicide	8.57	**270.0 → 228.1** 268.0 → 226.1 268.0 → 184.1	10 10 20	n.d.
Triclopyr-methyl ester	Herbicide	7.51	**209.9 → 145.9** 209.9 → 109.9 145.9 → 110.0	20 35 15	n.d.

Name	Pesticide type	RT [min]	dMRM transitions	CE [eV]	LOQ [µg/ kg]
Trifloxystrobin	Fungicide	12.94	186.0 → 145.1 **172.0 → 145.1** 172.0 → 95.0	15 15 30	n.d.
Triflumizole	Fungicide	10.81	345.0 → 302.0 239.1 → 66.9 **132.0 → 90.0**	10 40 35	n.d.
Trinexapac-ethyl	Herbicide	9.50	**224.0 → 151.0** 224.0 → 95.0 207.0 → 68.9	5 25 25	n.d.
Triphenyl phosphate	Internal Standard	13.35	**326.0 → 325.0** 325.0 → 169.1 325.0 → 77.0	5 20 35	n.a.
Triticonazole	Fungicide	14.51	237.0 → 182.0 237.0 → 167.1 **234.8 → 182.1**	10 25 10	n.d.
Warfarin	Other (Rodenticide)	15.44	308.0 → 187.0 **265.0 → 187.0** 265.0 → 121.0	20 5 15	n.d.
Zoxamide	Fungicide	13.47	259.9 → 189.0 **257.9 → 187.1** 189.0 → 161.1	10 10 15	4

Supporting Table S3 Pesticide analysis data on 387 bat samples. Key: 0 (not detected), <1/ <2/ <4 (below the respective LOQ), n.q. (not quantifiable; analytical standard was not available at this point of the study), * (sum of isomers). Analytes are marked in green; sum parameters are marked in yellow. The formatting slightly varies from the original table due to different data types.

Sheet 1 Polychlorinated biphenyls

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3272	*Eptesicus serotinus*	<1	<1	<1	49	73	29	<1	151
3276	*Eptesicus serotinus*	<1	<1	0	50	73	23	<1	146
3280	*Eptesicus serotinus*	<1	<1	<1	28	38	20	<1	87
3288	*Eptesicus serotinus*	<1	<1	0	41	74	34	<1	149
3311	*Eptesicus serotinus*	<1	<1	<1	8	23	14	<1	45
3319	*Eptesicus serotinus*	<1	<1	<1	353	972	357	<1	1683
3450	*Eptesicus serotinus*	<1	<1	<1	46	132	88	<1	266
3451	*Eptesicus serotinus*	<1	<1	0	170	431	375	<1	976
3452	*Eptesicus serotinus*	<1	<1	0	265	395	416	<1	1075
3454	*Eptesicus serotinus*	<1	<1	<1	335	651	211	<1	1196
3456	*Eptesicus serotinus*	<1	<1	0	133	231	106	<1	470
3458	*Eptesicus serotinus*	<1	0	0	137	188	113	<1	438
3463	*Eptesicus serotinus*	<1	<1	0	112	113	46	<1	271
3464	*Eptesicus serotinus*	<1	<1	0	166	260	156	<1	583
3465	*Eptesicus serotinus*	<1	<1	<1	11133	17990	8089	<1	37211
3467	*Eptesicus serotinus*	<1	<1	<1	17	14	12	<1	42
3468	*Eptesicus serotinus*	<1	<1	1	48	22	20	<1	90
3469	*Eptesicus serotinus*	<1	<1	0	61	31	23	<1	115
3470	*Eptesicus serotinus*	<1	<1	<1	1424	1459	425	<1	3308
3472	*Eptesicus serotinus*	0	0	<1	1246	2338	1459	<1	5043
3473	*Eptesicus serotinus*	<1	<1	0	97	120	307	<1	524
3474	*Eptesicus serotinus*	<1	<1	<1	35	95	121	<1	251
3485	*Eptesicus serotinus*	0	0	<1	175	290	182	<1	646
3488	*Eptesicus serotinus*	41	5	0	8	8	7	46	23
3494	*Eptesicus serotinus*	<1	0	0	<1	<1	<1	<1	<1
3498	*Eptesicus serotinus*	<1	<1	1	96	78	47	1	221
3499	*Eptesicus serotinus*	<1	<1	0	986	1741	732	<1	3458
3500	*Eptesicus serotinus*	2	<1	0	69	86	66	2	220
3501	*Eptesicus serotinus*	<1	0	0	23	34	23	<1	80
3502	*Eptesicus serotinus*	<1	<1	0	331	462	178	<1	971
3503	*Eptesicus serotinus*	<1	<1	<1	9	16	15	<1	41
3506	*Eptesicus serotinus*	22	0	0	293	1011	401	22	1706
3509	*Eptesicus serotinus*	3	<1	1	4153	10820	5000	4	19973
3510	*Eptesicus serotinus*	<1	<1	<1	35	54	34	<1	123
3511	*Eptesicus serotinus*	<1	<1	<1	356	1258	405	<1	2018
3512	*Eptesicus serotinus*	<1	0	<1	40	57	18	<1	114
3513	*Eptesicus serotinus*	<1	0	0	6	9	3	<1	18
3514	*Eptesicus serotinus*	1	<1	0	46	89	35	1	170
3515	*Eptesicus serotinus*	2	0	0	327	1228	469	2	2024

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3522	*Eptesicus serotinus*	<1	0	<1	8	11	7	<1	26
3523	*Eptesicus serotinus*	<1	0	0	8	9	6	<1	22
3525	*Eptesicus serotinus*	<1	0	0	54	186	144	<1	384
3526	*Eptesicus serotinus*	<1	<1	2	11	20	15	2	46
3527	*Eptesicus serotinus*	<1	0	0	210	414	134	<1	758
3529	*Eptesicus serotinus*	<1	0	0	330	524	329	<1	1183
3530	*Eptesicus serotinus*	<1	<1	3	212	738	387	3	1336
3531	*Eptesicus serotinus*	<1	<1	2	37	67	65	2	169
3532	*Eptesicus serotinus*	<1	<1	0	128	374	296	<1	798
3533	*Eptesicus serotinus*	<1	0	0	24	30	18	<1	72
3534	*Eptesicus serotinus*	<1	<1	<1	26	44	40	<1	110
3535	*Eptesicus serotinus*	<1	<1	0	7	10	6	<1	22
3536	*Eptesicus serotinus*	<1	<1	<1	10	18	16	<1	45
3537	*Eptesicus serotinus*	<1	<1	0	74	150	156	<1	380
3538	*Eptesicus serotinus*	<1	0	0	3	5	8	<1	16
3539	*Eptesicus serotinus*	<1	<1	0	12	25	16	<1	53
3540	*Eptesicus serotinus*	<1	0	0	72	98	53	<1	223
3541	*Eptesicus serotinus*	<1	0	0	21	45	34	<1	101
3542	*Eptesicus serotinus*	<1	<1	0	46	90	64	<1	200
3543	*Eptesicus serotinus*	<1	<1	<1	8	8	6	<1	21
3544	*Eptesicus serotinus*	<1	<1	0	22	59	38	<1	119
3545	*Eptesicus serotinus*	<1	<1	0	117	226	155	<1	497
3546	*Eptesicus serotinus*	<1	<1	1	47	89	101	1	237
3556	*Eptesicus serotinus*	<1	0	1	253	262	105	1	621
3564	*Eptesicus serotinus*	<1	0	0	147	288	226	<1	661
3565	*Eptesicus serotinus*	<1	<1	<1	105	252	288	<1	645
3566	*Eptesicus serotinus*	<1	<1	<1	247	797	493	<1	1537
3567	*Eptesicus serotinus*	<1	<1	<1	295	755	184	<1	1235
3568	*Eptesicus serotinus*	<1	<1	<1	54	100	44	<1	199
3569	*Eptesicus serotinus*	<1	0	0	90	143	108	<1	342
3570	*Eptesicus serotinus*	<1	<1	0	49	83	77	<1	208
3571	*Eptesicus serotinus*	<1	<1	<1	21	49	48	<1	118
3572	*Eptesicus serotinus*	<1	<1	<1	31	32	20	<1	82
3573	*Eptesicus serotinus*	<1	0	<1	1050	2321	1177	<1	4548
3574	*Eptesicus serotinus*	<1	<1	<1	3	6	7	<1	16
3010	*Myotis myotis*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3012	*Myotis myotis*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3015	*Myotis myotis*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3016	*Myotis myotis*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3017	*Myotis myotis*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3073	*Myotis myotis*	0	0	n.q.	0	0	0	n.q.	0
3188	*Myotis myotis*	<1	<1	0	40	137	48	<1	225
3247	*Myotis myotis*	<1	<1	<1	<1	8	<1	<1	8
3279	*Myotis myotis*	<1	<1	<1	<1	<1	1	<1	1

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3378	*Myotis myotis*	<1	<1	0	17	47	32	<1	96
3457	*Myotis myotis*	<1	0	0	59	246	118	<1	422
3459	*Myotis myotis*	<1	<1	<1	22	89	43	<1	154
3462	*Myotis myotis*	<1	<1	<1	5	16	8	<1	29
3517	*Myotis myotis*	<1	0	0	9	21	10	<1	40
3518	*Myotis myotis*	<1	0	0	23	304	367	<1	694
3520	*Myotis myotis*	<1	0	0	6	9	8	<1	23
3521	*Myotis myotis*	0	0	0	1	<1	1	0	2
3074	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3075	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3076	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3077	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3078	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3079	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3080	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3081	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3082	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3083	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3084	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3085	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3086	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3087	*Nyctalus noctula*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3121	*Nyctalus noctula*	3	<1	<1	84	117	54	3	255
3122	*Nyctalus noctula*	<1	<1	<1	59	60	38	<1	158
3123	*Nyctalus noctula*	<1	<1	<1	137	195	82	<1	415
3197	*Nyctalus noctula*	1	<1	0	49	137	119	1	306
3202	*Nyctalus noctula*	<1	0	<1	32	34	18	<1	84
3203	*Nyctalus noctula*	3	0	0	191	301	296	3	788
3245	*Nyctalus noctula*	<1	0	<1	5	8	3	<1	16
3248	*Nyctalus noctula*	<1	0	<1	0	<1	0	<1	<1
3252	*Nyctalus noctula*	<1	0	<1	3	4	<1	<1	6
3254	*Nyctalus noctula*	5	0	0	294	483	156	5	933
3255	*Nyctalus noctula*	<1	0	0	9	16	8	<1	34
3260	*Nyctalus noctula*	<1	0	0	<1	<1	<1	<1	<1
3263	*Nyctalus noctula*	<1	0	0	<1	4	<1	<1	4
3275	*Nyctalus noctula*	1	<1	<1	1	1	<1	1	3
3277	*Nyctalus noctula*	<1	<1	<1	7	12	5	<1	23
3282	*Nyctalus noctula*	<1	<1	0	761	1329	784	<1	2874
3284	*Nyctalus noctula*	3	0	0	83	111	111	3	305
3294	*Nyctalus noctula*	<1	<1	0	291	298	103	<1	692
3453	*Nyctalus noctula*	<1	<1	0	153	177	134	<1	463
3466	*Nyctalus noctula*	<1	<1	1	371	310	195	1	876
3471	*Nyctalus noctula*	<1	0	0	2952	4464	2030	<1	9446
3483	*Nyctalus noctula*	<1	0	0	6	2	4	<1	12

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3484	*Nyctalus noctula*	2	<1	0	430	1649	807	2	2886
3486	*Nyctalus noctula*	25	25	33	182	146	139	83	467
3487	*Nyctalus noctula*	0	0	<1	10	11	9	<1	30
3489	*Nyctalus noctula*	<1	<1	<1	25	20	10	<1	56
3490	*Nyctalus noctula*	0	0	0	47	63	40	0	149
3492	*Nyctalus noctula*	<1	0	0	48	50	44	<1	141
3493	*Nyctalus noctula*	3	<1	0	313	649	328	3	1290
3495	*Nyctalus noctula*	<1	<1	0	4	3	5	<1	11
3496	*Nyctalus noctula*	8	9	19	155	105	90	36	351
3497	*Nyctalus noctula*	2	<1	0	142	132	183	2	457
3507	*Nyctalus noctula*	2	<1	2	442	798	396	4	1635
3508	*Nyctalus noctula*	<1	<1	0	48	103	39	<1	190
3516	*Nyctalus noctula*	1	0	0	287	313	132	1	732
3008	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3009	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3013	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3021	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3023	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3024	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3025	*Pipistrellus pipistrellus*	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3026	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3027	*Pipistrellus pipistrellus*	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3028	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3029	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3030	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3031	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3033	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3034	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3036	*Pipistrellus pipistrellus*	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3037	*Pipistrellus pipistrellus*	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3038	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3039	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	0	n.q.	n.q.	n.q.
3040	*Pipistrellus pipistrellus*	0	0	0	n.q.	n.q.	n.q.	0	n.q.
3041	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3042	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3043	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3044	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3045	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3046	*Pipistrellus pipistrellus*	0	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3047	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3048	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3049	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3050	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3051	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3052	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3053	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3054	*Pipistrellus pipistrellus*	0	0	0	n.q.	n.q.	n.q.	0	n.q.
3055	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3056	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3063	*Pipistrellus pipistrellus*	0	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3064	*Pipistrellus pipistrellus*	0	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3065	*Pipistrellus pipistrellus*	0	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3066	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3067	*Pipistrellus pipistrellus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3068	*Pipistrellus pipistrellus*	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3069	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3070	*Pipistrellus pipistrellus*	n.q.	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3071	*Pipistrellus pipistrellus*	0	0	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3116	*Pipistrellus pipistrellus*	<1	<1	0	349	666	641	<1	1655
3117	*Pipistrellus pipistrellus*	<1	0	0	176	349	276	<1	801
3119	*Pipistrellus pipistrellus*	<1	0	0	175	213	145	<1	533
3126	*Pipistrellus pipistrellus*	<1	0	0	80	111	72	<1	264
3128	*Pipistrellus pipistrellus*	<1	0	0	2033	2004	1533	<1	5571
3129	*Pipistrellus pipistrellus*	<1	0	<1	268	428	290	<1	986
3130	*Pipistrellus pipistrellus*	1	<1	0	175	252	112	1	539
3132	*Pipistrellus pipistrellus*	8	0	0	255	301	181	8	738
3133	*Pipistrellus pipistrellus*	38	<1	0	1548	1884	763	38	4195
3134	*Pipistrellus pipistrellus*	<1	0	0	86	123	59	<1	268
3145	*Pipistrellus pipistrellus*	<1	0	0	46	65	38	<1	149
3147	*Pipistrellus pipistrellus*	<1	0	0	442	595	604	<1	1641
3148	*Pipistrellus pipistrellus*	<1	0	0	50	78	22	<1	150
3149	*Pipistrellus pipistrellus*	<1	0	0	13	25	21	<1	59
3150	*Pipistrellus pipistrellus*	<1	<1	0	3	7	8	<1	18
3151	*Pipistrellus pipistrellus*	<1	0	0	689	969	837	<1	2494
3152	*Pipistrellus pipistrellus*	<1	0	0	856	639	119	<1	1613
3153	*Pipistrellus pipistrellus*	<1	0	0	248	394	103	<1	745
3161	*Pipistrellus pipistrellus*	<1	0	0	99	97	112	<1	308
3163	*Pipistrellus pipistrellus*	<1	0	0	37	66	43	<1	146
3169	*Pipistrellus pipistrellus*	<1	<1	0	300	414	180	<1	894
3175	*Pipistrellus pipistrellus*	15	0	0	197	535	184	15	916
3176	*Pipistrellus pipistrellus*	<1	0	<1	27	39	20	<1	86
3189	*Pipistrellus pipistrellus*	<1	<1	<1	435	529	413	<1	1377
3198	*Pipistrellus pipistrellus*	<1	0	0	144	194	288	<1	627
3205	*Pipistrellus pipistrellus*	<1	<1	0	27	18	15	<1	60
3207	*Pipistrellus pipistrellus*	<1	<1	0	249	594	261	<1	1103
3208	*Pipistrellus pipistrellus*	0	0	0	67	192	60	0	319
3209	*Pipistrellus pipistrellus*	<1	0	<1	19	71	17	<1	107
3210	*Pipistrellus pipistrellus*	<1	<1	0	27	41	28	<1	96

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3211	*Pipistrellus pipistrellus*	<1	<1	0	21	61	19	<1	101
3215	*Pipistrellus pipistrellus*	18	<1	0	366	695	230	18	1291
3216	*Pipistrellus pipistrellus*	2	0	0	335	503	282	2	1119
3220	*Pipistrellus pipistrellus*	<1	<1	0	74	303	53	<1	430
3221	*Pipistrellus pipistrellus*	5	0	0	2380	2551	2358	5	7289
3222	*Pipistrellus pipistrellus*	<1	0	0	1202	1447	1351	<1	4000
3224	*Pipistrellus pipistrellus*	4	3	4	236	777	208	11	1222
3225	*Pipistrellus pipistrellus*	0	0	0	77	114	65	0	256
3226	*Pipistrellus pipistrellus*	0	0	<1	16	56	22	<1	94
3228	*Pipistrellus pipistrellus*	0	0	0	41	70	34	0	144
3229	*Pipistrellus pipistrellus*	<1	0	<1	22	27	12	<1	61
3230	*Pipistrellus pipistrellus*	<1	0	<1	181	213	115	<1	508
3233	*Pipistrellus pipistrellus*	<1	<1	<1	2045	2222	1656	<1	5923
3235	*Pipistrellus pipistrellus*	<1	0	<1	22	64	21	<1	107
3237	*Pipistrellus pipistrellus*	<1	<1	<1	7	13	4	<1	24
3240	*Pipistrellus pipistrellus*	<1	0	0	122	602	67	<1	791
3242	*Pipistrellus pipistrellus*	<1	0	<1	716	534	275	<1	1525
3244	*Pipistrellus pipistrellus*	<1	<1	0	1085	1341	576	<1	3002
3250	*Pipistrellus pipistrellus*	<1	<1	<1	6	11	6	<1	23
3253	*Pipistrellus pipistrellus*	<1	0	0	<1	<1	0	<1	<1
3256	*Pipistrellus pipistrellus*	<1	0	<1	36	112	11	<1	160
3257	*Pipistrellus pipistrellus*	<1	<1	0	6	12	5	<1	22
3258	*Pipistrellus pipistrellus*	1	0	1	32	96	9	2	136
3259	*Pipistrellus pipistrellus*	<1	0	0	24	90	8	<1	122
3261	*Pipistrellus pipistrellus*	<1	0	0	<1	3	<1	<1	3
3264	*Pipistrellus pipistrellus*	<1	0	0	15	25	10	<1	50
3265	*Pipistrellus pipistrellus*	<1	0	0	69	67	24	<1	160
3268	*Pipistrellus pipistrellus*	15	2	6	312	449	93	22	853
3278	*Pipistrellus pipistrellus*	<1	<1	<1	12	19	12	<1	42
3281	*Pipistrellus pipistrellus*	<1	<1	<1	7	11	10	<1	27
3286	*Pipistrellus pipistrellus*	<1	<1	0	168	298	225	<1	691
3291	*Pipistrellus pipistrellus*	<1	0	<1	20	24	9	<1	53
3293	*Pipistrellus pipistrellus*	<1	<1	<1	54	80	40	<1	175
3295	*Pipistrellus pipistrellus*	4	<1	0	137	180	83	4	400
3299	*Pipistrellus pipistrellus*	<1	<1	0	208	195	89	<1	491
3302	*Pipistrellus pipistrellus*	<1	<1	0	1683	2505	2210	<1	6399
3303	*Pipistrellus pipistrellus*	<1	<1	<1	16	24	16	<1	57
3304	*Pipistrellus pipistrellus*	<1	<1	0	501	710	445	<1	1656
3305	*Pipistrellus pipistrellus*	<1	<1	0	93	156	114	<1	362
3306	*Pipistrellus pipistrellus*	1	<1	0	906	1239	1424	1	3569
3309	*Pipistrellus pipistrellus*	<1	<1	<1	5	5	6	<1	16
3312	*Pipistrellus pipistrellus*	<1	<1	<1	120	200	168	<1	488
3313	*Pipistrellus pipistrellus*	<1	<1	<1	225	325	210	<1	760
3314	*Pipistrellus pipistrellus*	<1	<1	<1	34	47	42	<1	123

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3317	*Pipistrellus pipistrellus*	0	0	<1	198	266	168	<1	632
3318	*Pipistrellus pipistrellus*	<1	<1	<1	200	316	140	<1	656
3320	*Pipistrellus pipistrellus*	<1	<1	<1	44	56	33	<1	134
3321	*Pipistrellus pipistrellus*	<1	<1	<1	17	25	27	<1	69
3322	*Pipistrellus pipistrellus*	<1	<1	0	325	395	293	<1	1012
3326	*Pipistrellus pipistrellus*	1	<1	<1	116	152	163	1	430
3327	*Pipistrellus pipistrellus*	<1	<1	0	56	95	95	<1	246
3328	*Pipistrellus pipistrellus*	<1	<1	2	62	108	74	2	244
3330	*Pipistrellus pipistrellus*	<1	<1	0	358	426	495	<1	1278
3331	*Pipistrellus pipistrellus*	<1	<1	0	9	27	11	<1	47
3332	*Pipistrellus pipistrellus*	<1	<1	0	6	10	6	<1	22
3333	*Pipistrellus pipistrellus*	<1	<1	0	47	72	33	<1	152
3334	*Pipistrellus pipistrellus*	<1	<1	0	8	13	9	<1	30
3335	*Pipistrellus pipistrellus*	<1	<1	0	9	14	4	<1	28
3336	*Pipistrellus pipistrellus*	<1	<1	0	182	258	173	<1	613
3337	*Pipistrellus pipistrellus*	<1	<1	0	83	118	50	<1	251
3338	*Pipistrellus pipistrellus*	<1	<1	0	76	142	68	<1	286
3339	*Pipistrellus pipistrellus*	<1	<1	0	141	263	122	<1	526
3341	*Pipistrellus pipistrellus*	<1	<1	0	335	468	235	<1	1038
3342	*Pipistrellus pipistrellus*	<1	<1	0	110	151	83	<1	344
3343	*Pipistrellus pipistrellus*	<1	<1	0	306	366	154	<1	826
3345	*Pipistrellus pipistrellus*	<1	<1	0	116	163	80	<1	359
3346	*Pipistrellus pipistrellus*	<1	<1	0	36	56	20	<1	112
3348	*Pipistrellus pipistrellus*	<1	<1	0	118	208	126	<1	452
3350	*Pipistrellus pipistrellus*	<1	4	3	93	147	75	7	314
3351	*Pipistrellus pipistrellus*	<1	<1	0	714	967	320	<1	2001
3352	*Pipistrellus pipistrellus*	<1	<1	0	97	156	61	<1	315
3353	*Pipistrellus pipistrellus*	<1	<1	0	12	18	7	<1	38
3354	*Pipistrellus pipistrellus*	<1	<1	0	97	161	79	<1	337
3355	*Pipistrellus pipistrellus*	<1	<1	0	1	3	2	<1	6
3356	*Pipistrellus pipistrellus*	<1	<1	0	85	137	103	<1	325
3358	*Pipistrellus pipistrellus*	<1	<1	0	91	158	105	<1	355
3359	*Pipistrellus pipistrellus*	<1	<1	0	<1	3	2	<1	4
3360	*Pipistrellus pipistrellus*	<1	<1	0	160	208	105	<1	473
3361	*Pipistrellus pipistrellus*	<1	<1	0	7	10	7	<1	24
3363	*Pipistrellus pipistrellus*	<1	1	2	113	163	113	3	389
3364	*Pipistrellus pipistrellus*	<1	<1	<1	5	4	4	<1	13
3365	*Pipistrellus pipistrellus*	<1	<1	0	63	111	50	<1	224
3373	*Pipistrellus pipistrellus*	<1	<1	0	116	150	78	<1	344
3374	*Pipistrellus pipistrellus*	<1	0	0	3	5	4	<1	11
3376	*Pipistrellus pipistrellus*	<1	<1	0	188	216	130	<1	534
3379	*Pipistrellus pipistrellus*	<1	<1	0	147	175	81	<1	402
3381	*Pipistrellus pipistrellus*	<1	<1	0	26	44	19	<1	88
3383	*Pipistrellus pipistrellus*	<1	<1	0	124	200	90	<1	414

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3384	*Pipistrellus pipistrellus*	<1	<1	0	238	329	136	<1	703
3386	*Pipistrellus pipistrellus*	<1	<1	<1	320	367	137	<1	823
3387	*Pipistrellus pipistrellus*	<1	<1	<1	18	34	18	<1	70
3388	*Pipistrellus pipistrellus*	<1	<1	<1	63	91	88	<1	242
3389	*Pipistrellus pipistrellus*	<1	<1	0	45	60	50	<1	155
3390	*Pipistrellus pipistrellus*	3	<1	<1	100	124	74	3	298
3391	*Pipistrellus pipistrellus*	4	<1	1	271	270	285	5	827
3392	*Pipistrellus pipistrellus*	<1	<1	<1	102	111	48	<1	261
3393	*Pipistrellus pipistrellus*	<1	<1	0	24	34	27	<1	85
3394	*Pipistrellus pipistrellus*	<1	<1	<1	414	910	276	<1	1599
3395	*Pipistrellus pipistrellus*	3	<1	<1	1470	2012	1165	3	4648
3396	*Pipistrellus pipistrellus*	<1	<1	<1	131	248	199	<1	578
3397	*Pipistrellus pipistrellus*	7	<1	<1	878	1630	1265	7	3773
3398	*Pipistrellus pipistrellus*	<1	<1	<1	179	220	69	<1	468
3399	*Pipistrellus pipistrellus*	<1	<1	4	1461	1451	520	4	3431
3400	*Pipistrellus pipistrellus*	<1	<1	<1	2196	3102	2602	<1	7900
3401	*Pipistrellus pipistrellus*	<1	<1	<1	2531	3709	1257	<1	7497
3402	*Pipistrellus pipistrellus*	<1	<1	0	22	33	34	<1	89
3405	*Pipistrellus pipistrellus*	<1	<1	<1	65	68	46	<1	179
3406	*Pipistrellus pipistrellus*	<1	<1	0	243	344	125	<1	712
3407	*Pipistrellus pipistrellus*	<1	<1	0	218	262	219	<1	698
3408	*Pipistrellus pipistrellus*	<1	<1	0	24	43	21	<1	88
3409	*Pipistrellus pipistrellus*	<1	<1	<1	512	1246	333	<1	2091
3410	*Pipistrellus pipistrellus*	2	<1	0	37	56	20	2	114
3411	*Pipistrellus pipistrellus*	<1	<1	0	410	853	1077	<1	2340
3413	*Pipistrellus pipistrellus*	3	<1	<1	53	70	44	3	167
3414	*Pipistrellus pipistrellus*	<1	<1	0	125	207	73	<1	405
3415	*Pipistrellus pipistrellus*	<1	<1	0	961	1465	645	<1	3071
3417	*Pipistrellus pipistrellus*	3	<1	1	61	65	40	4	166
3419	*Pipistrellus pipistrellus*	<1	<1	<1	127	109	43	<1	279
3420	*Pipistrellus pipistrellus*	<1	<1	<1	124	228	115	<1	467
3421	*Pipistrellus pipistrellus*	<1	<1	<1	40	63	24	<1	127
3423	*Pipistrellus pipistrellus*	1	<1	<1	117	143	103	1	363
3425	*Pipistrellus pipistrellus*	<1	<1	<1	72	131	60	<1	264
3426	*Pipistrellus pipistrellus*	<1	<1	<1	163	341	506	<1	1010
3427	*Pipistrellus pipistrellus*	6	<1	5	418	544	293	11	1255
3428	*Pipistrellus pipistrellus*	3	<1	<1	282	291	153	3	726
3429	*Pipistrellus pipistrellus*	<1	<1	<1	8	14	10	<1	32
3430	*Pipistrellus pipistrellus*	<1	<1	<1	69	70	135	<1	273
3433	*Pipistrellus pipistrellus*	<1	<1	<1	46	51	18	<1	114
3434	*Pipistrellus pipistrellus*	<1	<1	<1	43	74	44	<1	161
3435	*Pipistrellus pipistrellus*	<1	<1	0	288	513	272	<1	1073
3460	*Pipistrellus pipistrellus*	<1	<1	<1	75	116	64	<1	254
3475	*Pipistrellus pipistrellus*	0	0	3	2817	3073	1478	3	7367

No	Species	PCB 28 [µg kg^{-1}]	PCB 52 [µg kg^{-1}]	PCB 101 [µg kg^{-1}]	PCB 138 [µg kg^{-1}]	PCB 153 [µg kg^{-1}]	PCB 180 [µg kg^{-1}]	low chlorinated PCBs [µg kg^{-1}]	high chlorinated PCBs [µg kg^{-1}]
3476	*Pipistrellus pipistrellus*	<1	<1	<1	28	23	21	<1	72
3477	*Pipistrellus pipistrellus*	2	<1	5	3717	4281	2876	7	10874
3478	*Pipistrellus pipistrellus*	<1	<1	<1	61	62	55	<1	179
3479	*Pipistrellus pipistrellus*	<1	<1	<1	18	29	43	<1	90
3480	*Pipistrellus pipistrellus*	<1	<1	<1	7	10	7	<1	24
3481	*Pipistrellus pipistrellus*	<1	<1	<1	17	28	23	<1	69
3482	*Pipistrellus pipistrellus*	<1	<1	0	12	11	13	<1	36
3505	*Pipistrellus pipistrellus*	<1	<1	<1	283	303	168	<1	754
3032	*Plecotus auritus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3072	*Plecotus auritus*	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.	n.q.
3144	*Plecotus auritus*	<1	0	0	14	39	19	<1	72
3154	*Plecotus auritus*	<1	0	0	110	122	53	<1	285
3196	*Plecotus auritus*	<1	0	0	37	30	7	<1	73
3201	*Plecotus auritus*	2	1	2	39	59	7	5	105
3212	*Plecotus auritus*	<1	0	0	22	42	7	<1	71
3267	*Plecotus auritus*	<1	0	0	<1	3	<1	<1	3
3296	*Plecotus auritus*	<1	0	0	26	80	17	<1	123
3300	*Plecotus auritus*	11	19	24	130	287	93	54	509
3329	*Plecotus auritus*	<1	0	0	39	38	11	<1	88
3357	*Plecotus auritus*	<1	<1	<1	4	6	<1	<1	9
3372	*Plecotus auritus*	<1	<1	<1	60	191	73	<1	325
3461	*Plecotus auritus*	<1	<1	0	82	106	44	<1	231
3491	*Plecotus auritus*	<1	0	0	48	34	15	<1	96
3519	*Plecotus auritus*	3	0	0	7	7	4	3	17
3524	*Plecotus auritus*	<1	0	<1	41	59	29	<1	129
3548	*Plecotus auritus*	<1	<1	0	131	107	54	<1	292
3549	*Plecotus auritus*	<1	<1	0	116	108	71	<1	296
3550	*Plecotus auritus*	<1	<1	0	232	244	173	<1	649
3551	*Plecotus auritus*	<1	<1	0	653	321	178	<1	1152
3552	*Plecotus auritus*	<1	0	0	85	80	70	<1	236
3553	*Plecotus auritus*	<1	0	0	41	61	48	<1	150
3554	*Plecotus auritus*	1	2	4	295	278	152	7	726
3555	*Plecotus auritus*	3	2	5	229	255	185	10	669
3557	*Plecotus auritus*	<1	<1	2	20	24	18	2	62
3558	*Plecotus auritus*	10	<1	0	272	284	159	10	715
3559	*Plecotus auritus*	438	50	26	268	259	217	514	743
3560	*Plecotus auritus*	<1	<1	0	132	130	115	<1	377
3561	*Plecotus auritus*	2	<1	0	244	220	146	2	610
3562	*Plecotus auritus*	2	<1	4	272	280	192	6	744
3563	*Plecotus auritus*	1	<1	2	87	95	68	3	250

Sheet 2 Organochlorine insecticides

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3272	*Eptesicus serotinus*	13876	<2	13876	17	17	<1	17	0	11
3276	*Eptesicus serotinus*	3918	36	3954	10	30	0	30	0	10
3280	*Eptesicus serotinus*	83	5	88	0	3	<1	3	<1	0
3288	*Eptesicus serotinus*	45	<2	45	0	16	<1	16	0	0
3311	*Eptesicus serotinus*	5	0	5	0	<1	<1	<1	0	<2
3319	*Eptesicus serotinus*	206	<2	206	<2	<1	<1	<1	0	<2
3450	*Eptesicus serotinus*	181	0	181	3	3	<1	3	0	<2
3451	*Eptesicus serotinus*	182	0	182	7	5	<1	5	0	4
3452	*Eptesicus serotinus*	418	15	433	0	7	10	18	0	5
3454	*Eptesicus serotinus*	528	7	535	65	5	<1	5	0	5
3456	*Eptesicus serotinus*	482	5	487	0	2	<1	2	0	<2
3458	*Eptesicus serotinus*	994	7	1001	267	4	<1	4	0	34
3463	*Eptesicus serotinus*	969	9	978	4	<1	0	<1	0	<2
3464	*Eptesicus serotinus*	3715	114	3829	0	25	0	25	0	<2
3465	*Eptesicus serotinus*	7736	9	7745	128	13	0	13	0	21
3467	*Eptesicus serotinus*	61	66	127	0	<1	<1	<1	0	<2
3468	*Eptesicus serotinus*	101	0	101	0	2	<1	2	0	7
3469	*Eptesicus serotinus*	29	0	29	0	1	0	1	0	<2
3470	*Eptesicus serotinus*	2362	4	2367	0	<1	0	<1	0	0
3472	*Eptesicus serotinus*	4326	13	4339	9	11	0	11	0	18
3473	*Eptesicus serotinus*	6046	0	6046	0	16	3	19	0	11
3474	*Eptesicus serotinus*	373	0	373	0	<1	8	8	0	<2
3485	*Eptesicus serotinus*	2556	0	2556	12	14	552	566	0	20
3488	*Eptesicus serotinus*	190	0	190	0	7	0	7	0	0
3494	*Eptesicus serotinus*	1	0	1	0	0	1	1	0	0
3498	*Eptesicus serotinus*	6164	252	6416	12	28	0	28	0	12
3499	*Eptesicus serotinus*	5113	16	5128	4	7	0	7	0	11
3500	*Eptesicus serotinus*	6967	56	7023	10	9	0	9	<1	16
3501	*Eptesicus serotinus*	2616	11	2627	0	1	0	1	0	0
3502	*Eptesicus serotinus*	13007	13	13020	12	8	<1	8	0	18
3503	*Eptesicus serotinus*	1413	0	1413	<2	1	<1	1	0	3
3506	*Eptesicus serotinus*	304	0	304	0	5	<1	5	0	3
3509	*Eptesicus serotinus*	14400	9	14409	2	45	0	45	<1	11
3510	*Eptesicus serotinus*	199	140	339	0	4	<1	4	<1	3
3511	*Eptesicus serotinus*	176	0	176	9	17	0	17	0	5
3512	*Eptesicus serotinus*	248	0	248	0	1	<1	1	0	0
3513	*Eptesicus serotinus*	62	7	69	0	<1	<1	<1	0	0
3514	*Eptesicus serotinus*	9640	5	9645	0	5	0	5	0	5
3515	*Eptesicus serotinus*	412	5	417	0	27	4	31	0	13
3522	*Eptesicus serotinus*	<1	0	<1	0	<1	0	<1	0	0
3523	*Eptesicus serotinus*	6	0	6	2	1	0	1	0	0
3525	*Eptesicus serotinus*	15	0	15	4	3	<1	3	0	2
3526	*Eptesicus serotinus*	1	<2	1	<2	1	<1	1	<1	<2

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3527	*Eptesicus serotinus*	197	0	197	104	<1	<1	<1	0	<2
3529	*Eptesicus serotinus*	122	5	127	50	6	1	7	0	10
3530	*Eptesicus serotinus*	154	6	160	26	4	2	7	<1	11
3531	*Eptesicus serotinus*	10	2	12	2	2	<1	2	<1	<2
3532	*Eptesicus serotinus*	43	3	46	53	1	<1	1	0	4
3533	*Eptesicus serotinus*	8	0	8	0	<1	0	<1	0	0
3534	*Eptesicus serotinus*	3	<2	3	3	4	<1	4	<1	3
3535	*Eptesicus serotinus*	<1	0	<1	0	<1	<1	<1	0	0
3536	*Eptesicus serotinus*	3	2	5	<2	2	<1	2	<1	<2
3537	*Eptesicus serotinus*	10	0	10	4	<1	<1	<1	0	3
3538	*Eptesicus serotinus*	<1	0	<1	0	<1	0	<1	0	0
3539	*Eptesicus serotinus*	8	0	8	<2	1	<1	1	0	0
3540	*Eptesicus serotinus*	22	0	22	3	2	4	6	0	<2
3541	*Eptesicus serotinus*	<1	0	<1	2	2	<1	2	0	<2
3542	*Eptesicus serotinus*	37	<2	37	5	2	0	2	0	5
3543	*Eptesicus serotinus*	<1	0	<1	0	1	6	7	0	0
3544	*Eptesicus serotinus*	23	2	25	24	4	0	4	0	7
3545	*Eptesicus serotinus*	70	0	70	3	2	0	2	0	2
3546	*Eptesicus serotinus*	5	3	7	6	5	<1	5	<1	3
3556	*Eptesicus serotinus*	52	3	55	39	2	1	3	0	6
3564	*Eptesicus serotinus*	36	0	36	16	10	0	10	0	5
3565	*Eptesicus serotinus*	10	0	10	<2	<1	<1	<1	0	<2
3566	*Eptesicus serotinus*	39	0	39	34	3	0	3	0	6
3567	*Eptesicus serotinus*	149	5	154	140	2	2	4	<1	14
3568	*Eptesicus serotinus*	46	0	46	7	2	<1	2	<1	<2
3569	*Eptesicus serotinus*	45	0	45	3	2	<1	2	0	4
3570	*Eptesicus serotinus*	11	0	11	<2	<1	2	2	0	0
3571	*Eptesicus serotinus*	4	0	4	0	<1	<1	<1	0	0
3572	*Eptesicus serotinus*	11	0	11	0	1	<1	1	<1	0
3573	*Eptesicus serotinus*	224	0	224	129	3	<1	3	0	18
3574	*Eptesicus serotinus*	1	0	1	0	<1	8	8	<1	0
3010	*Myotis myotis*	22	0	22	5	0	2	2	0	0
3012	*Myotis myotis*	25	0	25	5	0	3	3	0	0
3015	*Myotis myotis*	51	1	52	13	0	5	5	0	4
3016	*Myotis myotis*	65	0	65	21	0	6	6	0	6
3017	*Myotis myotis*	190	1	191	64	0	7	7	0	15
3073	*Myotis myotis*	7	0	7	3	0	<1	<1	0	0
3188	*Myotis myotis*	77	0	77	3	n.q.	0	n.q.	n.q.	3
3247	*Myotis myotis*	33	0	33	0	3	<1	3	0	0
3279	*Myotis myotis*	8	0	8	3	<1	<1	<1	0	0
3378	*Myotis myotis*	48	0	48	0	<1	0	<1	0	3
3457	*Myotis myotis*	275	0	275	17	2	0	2	0	13
3459	*Myotis myotis*	196	0	196	0	1	0	1	0	5
3462	*Myotis myotis*	141	2	144	7	2	0	2	0	<2

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3517	*Myotis myotis*	3	0	3	0	<1	0	<1	0	<2
3518	*Myotis myotis*	40	0	40	0	<1	0	<1	0	4
3520	*Myotis myotis*	<1	0	<1	0	<1	<1	<1	0	0
3521	*Myotis myotis*	<1	0	<1	0	7	0	7	0	0
3074	*Nyctalus noctula*	1519	6	1525	0	0	6	6	n.q.	0
3075	*Nyctalus noctula*	2070	18	2089	0	0	7	7	n.q.	<2
3076	*Nyctalus noctula*	9462	6	9468	0	0	16	16	0	0
3077	*Nyctalus noctula*	1527	10	1537	<2	0	7	7	n.q.	0
3078	*Nyctalus noctula*	2955	1	2956	<2	0	20	20	n.q.	0
3079	*Nyctalus noctula*	1621	31	1653	<2	0	3	3	n.q.	0
3080	*Nyctalus noctula*	1348	5	1352	<2	0	19	19	n.q.	0
3081	*Nyctalus noctula*	2311	1	2312	3	0	5	5	n.q.	3
3082	*Nyctalus noctula*	7415	19	7434	0	0	49	49	n.q.	<2
3083	*Nyctalus noctula*	3526	1	3527	0	0	6	6	0	0
3084	*Nyctalus noctula*	5460	74	5535	<2	0	3	3	0	2
3085	*Nyctalus noctula*	981	18	999	2	0	2	2	n.q.	<2
3086	*Nyctalus noctula*	1354	0	1354	0	0	42	42	n.q.	0
3087	*Nyctalus noctula*	2413	91	2503	0	0	73	73	n.q.	<2
3121	*Nyctalus noctula*	<1	4	4	0	0	29	29	0	0
3122	*Nyctalus noctula*	147	<2	147	0	0	8	8	0	0
3123	*Nyctalus noctula*	566	13	579	0	n.q.	51	51	0	0
3197	*Nyctalus noctula*	464	<2	464	3	7	<1	7	0	0
3202	*Nyctalus noctula*	33	9	41	35	3	5	8	0	12
3203	*Nyctalus noctula*	315	4	320	52	10	6	15	0	55
3245	*Nyctalus noctula*	590	4	594	0	<1	<1	<1	0	0
3248	*Nyctalus noctula*	53	<2	53	0	<1	<1	<1	0	0
3252	*Nyctalus noctula*	339	15	353	0	<1	<1	<1	0	0
3254	*Nyctalus noctula*	1134	0	1134	0	2	<1	2	0	0
3255	*Nyctalus noctula*	732	8	740	0	<1	<1	<1	0	0
3260	*Nyctalus noctula*	19	0	19	0	<1	0	<1	0	0
3263	*Nyctalus noctula*	196	10	206	0	<1	2	2	0	0
3275	*Nyctalus noctula*	34	0	34	0	1	<1	1	<1	0
3277	*Nyctalus noctula*	466	19	485	3	<1	1	1	0	2
3282	*Nyctalus noctula*	4444	170	4614	0	2	<1	2	0	<2
3284	*Nyctalus noctula*	2505	<2	2505	0	3	<1	3	0	0
3294	*Nyctalus noctula*	680	16	695	0	3	<1	3	<1	0
3453	*Nyctalus noctula*	375	28	403	0	2	0	2	0	0
3466	*Nyctalus noctula*	238	3	241	0	1	<1	1	0	0
3471	*Nyctalus noctula*	7515	3	7518	6	0	0	0	0	49
3483	*Nyctalus noctula*	345	9	354	0	<1	0	<1	0	0
3484	*Nyctalus noctula*	5485	35	5521	0	25	0	25	0	0
3486	*Nyctalus noctula*	119	91	209	2	0	16	16	15	15
3487	*Nyctalus noctula*	37	0	37	0	0	0	0	0	0
3489	*Nyctalus noctula*	118	0	118	0	0	0	0	0	0

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3490	*Nyctalus noctula*	710	0	710	0	<1	0	<1	<1	0
3492	*Nyctalus noctula*	2422	14	2436	0	<1	0	<1	0	0
3493	*Nyctalus noctula*	1309	7	1316	0	<1	<1	<1	0	<2
3495	*Nyctalus noctula*	79	0	79	0	0	0	0	0	0
3496	*Nyctalus noctula*	380	0	380	0	0	4	4	0	0
3497	*Nyctalus noctula*	317	0	317	0	3	0	3	0	0
3507	*Nyctalus noctula*	4814	5	4819	0	5	<1	5	<1	<2
3508	*Nyctalus noctula*	2941	4	2945	0	1	<1	1	0	0
3516	*Nyctalus noctula*	258	7	265	0	2	0	2	0	0
3008	*Pipistrellus pipistrellus*	22	0	22	3	0	6	6	0	0
3009	*Pipistrellus pipistrellus*	78	2	80	28	0	11	11	0	12
3013	*Pipistrellus pipistrellus*	403	1	404	59	0	12	12	0	12
3021	*Pipistrellus pipistrellus*	56	1	57	10	0	12	12	0	6
3023	*Pipistrellus pipistrellus*	56	4	60	6	0	8	8	0	6
3024	*Pipistrellus pipistrellus*	76	4	80	7	0	11	11	0	3
3025	*Pipistrellus pipistrellus*	3	0	3	3	0	6	6	0	<2
3026	*Pipistrellus pipistrellus*	4	0	4	2	0	4	4	0	4
3027	*Pipistrellus pipistrellus*	111	9	120	13	0	21	21	0	9
3028	*Pipistrellus pipistrellus*	49	3	51	3	0	13	13	0	4
3029	*Pipistrellus pipistrellus*	4	0	4	0	0	6	6	0	0
3030	*Pipistrellus pipistrellus*	22	<2	22	0	0	3	3	0	<2
3031	*Pipistrellus pipistrellus*	34	<2	34	<2	0	4	4	0	<2
3033	*Pipistrellus pipistrellus*	34	0	34	0	0	4	4	0	4
3034	*Pipistrellus pipistrellus*	150	0	151	9	0	8	8	0	7
3036	*Pipistrellus pipistrellus*	76	3	79	4	0	6	6	0	3
3037	*Pipistrellus pipistrellus*	48	0	48	6	0	8	8	0	4
3038	*Pipistrellus pipistrellus*	57	2	58	0	0	14	14	0	<2
3039	*Pipistrellus pipistrellus*	40	1	41	35	0	11	11	0	7
3040	*Pipistrellus pipistrellus*	<1	0	<1	0	0	1	1	0	0
3041	*Pipistrellus pipistrellus*	3	0	3	0	0	2	2	0	0
3042	*Pipistrellus pipistrellus*	158	0	158	0	0	5	5	0	10
3043	*Pipistrellus pipistrellus*	49	<2	49	0	0	5	5	0	3
3044	*Pipistrellus pipistrellus*	183	0	183	6	0	27	27	0	7
3045	*Pipistrellus pipistrellus*	301	0	301	39	0	9	9	0	25
3046	*Pipistrellus pipistrellus*	<1	0	<1	0	0	1	1	0	0
3047	*Pipistrellus pipistrellus*	39	2	41	3	0	21	21	0	0
3048	*Pipistrellus pipistrellus*	7	0	7	0	0	3	3	0	0
3049	*Pipistrellus pipistrellus*	134	0	134	12	0	8	8	0	7
3050	*Pipistrellus pipistrellus*	23	0	23	0	0	5	5	0	<2
3051	*Pipistrellus pipistrellus*	3	10	13	0	0	1	1	0	0
3052	*Pipistrellus pipistrellus*	7	0	7	0	0	2	2	0	0
3053	*Pipistrellus pipistrellus*	79	0	79	12	0	25	25	0	8
3054	*Pipistrellus pipistrellus*	<1	0	<1	0	0	0	0	0	0
3055	*Pipistrellus pipistrellus*	98	0	98	9	0	12	12	0	0

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3056	*Pipistrellus pipistrellus*	15	0	15	0	0	2	2	0	0
3063	*Pipistrellus pipistrellus*	5	0	5	0	0	1	1	0	0
3064	*Pipistrellus pipistrellus*	9	0	9	0	0	1	1	0	0
3065	*Pipistrellus pipistrellus*	4	0	4	0	0	1	1	0	0
3066	*Pipistrellus pipistrellus*	59	0	59	11	0	19	19	0	3
3067	*Pipistrellus pipistrellus*	108	0	108	0	0	5	5	0	3
3068	*Pipistrellus pipistrellus*	34	0	34	0	0	0	0	0	0
3069	*Pipistrellus pipistrellus*	20	0	20	0	0	14	14	n.q.	0
3070	*Pipistrellus pipistrellus*	6	0	6	0	0	2	2	0	0
3071	*Pipistrellus pipistrellus*	<1	0	<1	0	0	6	6	0	0
3116	*Pipistrellus pipistrellus*	82	0	82	0	n.q.	0	n.q.	0	6
3117	*Pipistrellus pipistrellus*	9	0	9	3	0	0	0	0	5
3119	*Pipistrellus pipistrellus*	101	0	101	4	0	8	8	0	0
3126	*Pipistrellus pipistrellus*	21	0	21	0	n.q.	0	n.q.	0	<2
3128	*Pipistrellus pipistrellus*	829	0	829	12	n.q.	21	21	0	15
3129	*Pipistrellus pipistrellus*	13	0	13	<2	n.q.	5	5	0	0
3130	*Pipistrellus pipistrellus*	<1	0	<1	0	n.q.	<1	n.q.	0	10
3132	*Pipistrellus pipistrellus*	32	<2	32	0	n.q.	2	2	0	<2
3133	*Pipistrellus pipistrellus*	1363	3	1365	87	n.q.	556	556	0	39
3134	*Pipistrellus pipistrellus*	16	0	16	0	n.q.	<1	n.q.	0	0
3145	*Pipistrellus pipistrellus*	5	<2	5	0	n.q.	<1	n.q.	0	<2
3147	*Pipistrellus pipistrellus*	299	0	299	15	n.q.	0	n.q.	0	<2
3148	*Pipistrellus pipistrellus*	20	1	21	0	n.q.	<1	n.q.	0	<2
3149	*Pipistrellus pipistrellus*	4	0	4	3	n.q.	0	n.q.	0	2
3150	*Pipistrellus pipistrellus*	2	0	2	0	n.q.	<1	n.q.	0	<2
3151	*Pipistrellus pipistrellus*	8	0	8	0	n.q.	0	n.q.	0	0
3152	*Pipistrellus pipistrellus*	55	3	58	8	n.q.	2	2	0	4
3153	*Pipistrellus pipistrellus*	15	0	15	0	n.q.	<1	n.q.	0	3
3161	*Pipistrellus pipistrellus*	27	0	27	0	n.q.	9	9	0	<2
3163	*Pipistrellus pipistrellus*	10	0	10	0	n.q.	1	1	0	2
3169	*Pipistrellus pipistrellus*	61	0	61	14	n.q.	<1	n.q.	0	16
3175	*Pipistrellus pipistrellus*	207	3	209	41	n.q.	0	n.q.	n.q.	13
3176	*Pipistrellus pipistrellus*	6	0	6	0	n.q.	0	n.q.	0	0
3189	*Pipistrellus pipistrellus*	190	0	190	16	14	0	14	0	13
3198	*Pipistrellus pipistrellus*	24	0	24	0	<1	5	5	0	<2
3205	*Pipistrellus pipistrellus*	14	0	14	11	4	<1	4	0	10
3207	*Pipistrellus pipistrellus*	260	0	260	23	7	<1	7	0	19
3208	*Pipistrellus pipistrellus*	65	0	65	0	10	<1	10	0	4
3209	*Pipistrellus pipistrellus*	28	0	28	0	1	<1	1	0	0
3210	*Pipistrellus pipistrellus*	38	0	38	4	4	<1	4	0	0
3211	*Pipistrellus pipistrellus*	8	0	8	0	2	<1	2	0	0
3215	*Pipistrellus pipistrellus*	271	<2	271	32	48	8	56	0	28
3216	*Pipistrellus pipistrellus*	90	0	90	19	2	<1	2	0	<2
3220	*Pipistrellus pipistrellus*	300	28	328	5	7	1	8	0	5

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3221	*Pipistrellus pipistrellus*	473	<2	473	18	18	6	24	0	17
3222	*Pipistrellus pipistrellus*	533	0	533	15	11	<1	11	0	14
3224	*Pipistrellus pipistrellus*	134	4	138	7	14	4	18	4	9
3225	*Pipistrellus pipistrellus*	16	0	16	0	3	<1	3	0	4
3226	*Pipistrellus pipistrellus*	13	0	13	5	2	<1	2	0	3
3228	*Pipistrellus pipistrellus*	135	0	135	2	6	1	7	0	3
3229	*Pipistrellus pipistrellus*	43	4	47	0	<1	<1	<1	0	0
3230	*Pipistrellus pipistrellus*	240	15	255	0	8	26	34	0	0
3233	*Pipistrellus pipistrellus*	1392	21	1413	22	18	24	42	0	0
3235	*Pipistrellus pipistrellus*	441	0	441	11	4	6	10	0	0
3237	*Pipistrellus pipistrellus*	115	8	123	0	<1	<1	<1	0	0
3240	*Pipistrellus pipistrellus*	361	22	383	0	10	7	17	0	0
3242	*Pipistrellus pipistrellus*	546	279	825	0	8	11	18	0	<2
3244	*Pipistrellus pipistrellus*	1269	61	1330	0	16	12	28	0	<2
3250	*Pipistrellus pipistrellus*	231	132	363	0	<1	<1	<1	0	0
3253	*Pipistrellus pipistrellus*	34	0	34	0	<1	<1	<1	0	0
3256	*Pipistrellus pipistrellus*	284	14	298	0	10	<1	10	0	0
3257	*Pipistrellus pipistrellus*	143	73	216	0	<1	<1	<1	0	0
3258	*Pipistrellus pipistrellus*	391	11	402	0	4	0	4	0	2
3259	*Pipistrellus pipistrellus*	331	0	331	0	3	<1	3	0	0
3261	*Pipistrellus pipistrellus*	174	17	191	0	<1	<1	<1	0	0
3264	*Pipistrellus pipistrellus*	195	31	226	<2	5	<1	5	0	4
3265	*Pipistrellus pipistrellus*	461	8	469	<2	3	<1	3	0	4
3268	*Pipistrellus pipistrellus*	741	12	753	<2	5	<1	5	0	0
3278	*Pipistrellus pipistrellus*	56	4	60	0	4	0	4	0	0
3281	*Pipistrellus pipistrellus*	14	0	14	0	2	<1	2	0	<2
3286	*Pipistrellus pipistrellus*	396	10	406	<2	15	4	19	0	<2
3291	*Pipistrellus pipistrellus*	96	<2	96	0	4	<1	4	0	0
3293	*Pipistrellus pipistrellus*	84	<2	84	0	2	<1	2	<1	0
3295	*Pipistrellus pipistrellus*	329	<2	329	0	7	<1	7	0	0
3299	*Pipistrellus pipistrellus*	158	11	169	<2	17	<1	17	0	<2
3302	*Pipistrellus pipistrellus*	48	0	48	0	5	0	5	0	5
3303	*Pipistrellus pipistrellus*	2	0	2	<2	<1	<1	<1	0	<2
3304	*Pipistrellus pipistrellus*	28	0	28	0	11	<1	11	0	<2
3305	*Pipistrellus pipistrellus*	17	0	17	0	0	0	0	0	0
3306	*Pipistrellus pipistrellus*	231	<2	231	0	2	<1	2	0	4
3309	*Pipistrellus pipistrellus*	<1	0	<1	0	0	0	0	0	0
3312	*Pipistrellus pipistrellus*	41	0	41	0	2	<1	2	0	<2
3313	*Pipistrellus pipistrellus*	18	<2	18	<2	7	<1	7	<1	<2
3314	*Pipistrellus pipistrellus*	<1	0	<1	0	<1	0	<1	0	0
3317	*Pipistrellus pipistrellus*	23	0	23	0	0	0	0	0	0
3318	*Pipistrellus pipistrellus*	63	<2	63	<2	13	<1	13	0	<2
3320	*Pipistrellus pipistrellus*	10	0	10	<2	<1	<1	<1	0	3
3321	*Pipistrellus pipistrellus*	<1	0	<1	0	<1	<1	<1	0	5

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3322	*Pipistrellus pipistrellus*	33	0	33	<2	0	0	0	0	12
3326	*Pipistrellus pipistrellus*	5	0	5	0	2	<1	2	0	8
3327	*Pipistrellus pipistrellus*	2	0	2	0	4	<1	4	0	6
3328	*Pipistrellus pipistrellus*	16	<2	16	5	12	<1	12	<1	<2
3330	*Pipistrellus pipistrellus*	24	0	24	<2	3	0	3	0	4
3331	*Pipistrellus pipistrellus*	<1	0	<1	<2	<1	0	<1	0	<2
3332	*Pipistrellus pipistrellus*	1	0	1	0	1	<1	1	0	0
3333	*Pipistrellus pipistrellus*	30	<2	30	0	2	<1	2	0	0
3334	*Pipistrellus pipistrellus*	5	0	5	0	1	<1	1	0	0
3335	*Pipistrellus pipistrellus*	15	0	15	0	<1	<1	<1	0	0
3336	*Pipistrellus pipistrellus*	69	0	69	0	<1	44	44	0	0
3337	*Pipistrellus pipistrellus*	77	<2	77	<2	4	<1	4	0	7
3338	*Pipistrellus pipistrellus*	31	0	31	0	4	0	4	0	<2
3339	*Pipistrellus pipistrellus*	84	0	84	0	<1	<1	<1	0	<2
3341	*Pipistrellus pipistrellus*	303	21	324	32	15	0	15	0	20
3342	*Pipistrellus pipistrellus*	89	0	89	10	<1	<1	<1	0	8
3343	*Pipistrellus pipistrellus*	199	<2	199	10	7	<1	7	0	9
3345	*Pipistrellus pipistrellus*	25	<2	25	<2	<1	<1	<1	0	<2
3346	*Pipistrellus pipistrellus*	84	<2	84	0	<1	<1	<1	0	<2
3348	*Pipistrellus pipistrellus*	45	<2	45	0	<1	0	<1	0	<2
3350	*Pipistrellus pipistrellus*	128	9	137	8	18	7	25	<1	9
3351	*Pipistrellus pipistrellus*	293	34	327	12	18	246	263	0	7
3352	*Pipistrellus pipistrellus*	160	<2	160	<2	11	<1	11	0	<2
3353	*Pipistrellus pipistrellus*	275	<2	275	0	<1	<1	<1	0	0
3354	*Pipistrellus pipistrellus*	148	<2	148	6	13	0	13	0	9
3355	*Pipistrellus pipistrellus*	14	0	14	0	<1	0	<1	0	0
3356	*Pipistrellus pipistrellus*	11	<2	11	0	<1	<1	<1	0	0
3358	*Pipistrellus pipistrellus*	49	<2	49	<2	3	0	3	0	5
3359	*Pipistrellus pipistrellus*	<1	0	<1	0	2	0	2	0	<2
3360	*Pipistrellus pipistrellus*	101	15	116	5	3	2	5	0	5
3361	*Pipistrellus pipistrellus*	9	<2	9	<2	<1	<1	<1	0	<2
3363	*Pipistrellus pipistrellus*	75	<2	75	6	12	3	15	<1	14
3364	*Pipistrellus pipistrellus*	5	2	7	0	7	0	7	0	0
3365	*Pipistrellus pipistrellus*	306	45	350	10	<1	0	<1	0	8
3373	*Pipistrellus pipistrellus*	129	0	129	7	6	0	6	0	13
3374	*Pipistrellus pipistrellus*	3	0	3	0	<1	0	<1	0	0
3376	*Pipistrellus pipistrellus*	51	<2	51	<2	7	0	7	0	<2
3379	*Pipistrellus pipistrellus*	83	0	83	3	3	0	3	0	5
3381	*Pipistrellus pipistrellus*	63	<2	63	0	2	0	2	0	<2
3383	*Pipistrellus pipistrellus*	201	4	205	10	3	<1	3	0	14
3384	*Pipistrellus pipistrellus*	117	<2	117	<2	3	<1	3	0	4
3386	*Pipistrellus pipistrellus*	1082	245	1327	22	20	<1	20	0	<2
3387	*Pipistrellus pipistrellus*	6	0	6	0	3	<1	3	0	0
3388	*Pipistrellus pipistrellus*	15	<2	15	0	5	0	5	0	0

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3389	*Pipistrellus pipistrellus*	80	100	180	0	5	7	12	0	<2
3390	*Pipistrellus pipistrellus*	228	11	238	4	5	0	5	0	<2
3391	*Pipistrellus pipistrellus*	68	2	70	3	3	1	5	0	<2
3392	*Pipistrellus pipistrellus*	301	3	304	0	5	0	5	0	0
3393	*Pipistrellus pipistrellus*	25	<2	25	0	<1	0	<1	0	0
3394	*Pipistrellus pipistrellus*	432	0	432	<2	11	0	11	<1	<2
3395	*Pipistrellus pipistrellus*	98	0	98	4	3	<1	3	0	<2
3396	*Pipistrellus pipistrellus*	159	0	159	16	5	0	5	0	<2
3397	*Pipistrellus pipistrellus*	418	<2	418	8	24	2	26	0	9
3398	*Pipistrellus pipistrellus*	338	<2	338	0	<1	0	<1	0	<2
3399	*Pipistrellus pipistrellus*	250	<2	250	7	6	0	6	0	3
3400	*Pipistrellus pipistrellus*	160	0	160	<2	8	0	8	0	6
3401	*Pipistrellus pipistrellus*	1101	6	1107	0	3	0	3	0	<2
3402	*Pipistrellus pipistrellus*	45	0	45	0	2	<1	2	0	0
3405	*Pipistrellus pipistrellus*	120	9	129	0	<1	<1	<1	0	0
3406	*Pipistrellus pipistrellus*	322	<2	322	4	<1	0	<1	0	3
3407	*Pipistrellus pipistrellus*	344	<2	344	<2	4	3	6	0	<2
3408	*Pipistrellus pipistrellus*	566	43	609	<2	<1	0	<1	0	2
3409	*Pipistrellus pipistrellus*	294	<2	294	9	8	0	8	0	0
3410	*Pipistrellus pipistrellus*	1011	31	1042	5	4	0	4	0	3
3411	*Pipistrellus pipistrellus*	430	3	433	4	14	0	14	0	<2
3413	*Pipistrellus pipistrellus*	38	0	38	0	<1	0	<1	0	0
3414	*Pipistrellus pipistrellus*	165	13	178	3	<1	0	<1	0	<2
3415	*Pipistrellus pipistrellus*	265	<2	265	<2	12	0	12	0	0
3417	*Pipistrellus pipistrellus*	10	4	13	0	3	0	3	0	0
3419	*Pipistrellus pipistrellus*	416	30	446	3	2	<1	2	0	<2
3420	*Pipistrellus pipistrellus*	3119	1496	4615	<2	3	6	9	0	3
3421	*Pipistrellus pipistrellus*	518	18	536	0	<1	0	<1	0	<2
3423	*Pipistrellus pipistrellus*	11	4	15	4	2	<1	2	0	<2
3425	*Pipistrellus pipistrellus*	244	15	259	0	3	0	3	0	<2
3426	*Pipistrellus pipistrellus*	28	0	28	0	2	<1	2	0	<2
3427	*Pipistrellus pipistrellus*	36	<2	36	0	5	<1	5	0	0
3428	*Pipistrellus pipistrellus*	49	<2	49	46	8	<1	8	0	4
3429	*Pipistrellus pipistrellus*	25	<2	25	0	1	<1	1	0	0
3430	*Pipistrellus pipistrellus*	165	<2	165	<2	2	<1	2	0	<2
3433	*Pipistrellus pipistrellus*	539	17	556	<2	2	0	2	0	<2
3434	*Pipistrellus pipistrellus*	189	17	207	4	5	0	5	0	4
3435	*Pipistrellus pipistrellus*	2428	24	2453	4	16	0	16	0	3
3460	*Pipistrellus pipistrellus*	75	7	81	18	13	<1	13	0	8
3475	*Pipistrellus pipistrellus*	404	0	404	4	23	0	23	0	6
3476	*Pipistrellus pipistrellus*	84	9	93	0	3	<1	3	0	0
3477	*Pipistrellus pipistrellus*	390	0	390	0	20	<1	20	0	<2
3478	*Pipistrellus pipistrellus*	57	0	57	0	<1	<1	<1	0	0
3479	*Pipistrellus pipistrellus*	3	0	3	0	<1	<1	<1	0	0

No	Species	DDE-*p,p'* [µg kg^{-1}]	DDT-*p,p'* [µg kg^{-1}]	DDT sum [µg kg^{-1}]	Dieldrin [µg kg^{-1}]	*beta*-HCH [µg kg^{-1}]	*gamma*-HCH (Lindane) [µg kg^{-1}]	HCH sum [µg kg^{-1}]	Heptachlor [µg kg^{-1}]	Oxy-chlordane [µg kg^{-1}]
3480	*Pipistrellus pipistrellus*	3	0	3	0	<1	0	<1	0	0
3481	*Pipistrellus pipistrellus*	5	0	5	0	<1	0	<1	0	0
3482	*Pipistrellus pipistrellus*	4	0	4	0	<1	0	<1	0	0
3505	*Pipistrellus pipistrellus*	210	0	210	23	9	<1	9	<1	6
3032	*Plecotus auritus*	280	42	322	6	n.q.	0	n.q.	0	0
3072	*Plecotus auritus*	14	0	14	0	0	<1	<1	0	0
3144	*Plecotus auritus*	5	0	5	5	n.q.	<1	n.q.	0	3
3154	*Plecotus auritus*	21	0	21	3	n.q.	0	n.q.	0	2
3196	*Plecotus auritus*	12	0	12	6	2	<1	2	0	4
3201	*Plecotus auritus*	1	0	1	0	7	39	46	0	6
3212	*Plecotus auritus*	25	0	25	0	<1	<1	<1	0	0
3267	*Plecotus auritus*	35	0	35	0	<1	<1	<1	0	0
3296	*Plecotus auritus*	110	0	110	0	<1	<1	<1	0	0
3300	*Plecotus auritus*	700	84	784	42	53	15	68	9	19
3329	*Plecotus auritus*	30	0	30	0	<1	<1	<1	0	<2
3357	*Plecotus auritus*	6	0	6	0	<1	<1	<1	0	0
3372	*Plecotus auritus*	11	0	11	<2	<1	<1	<1	0	0
3461	*Plecotus auritus*	65	0	65	<2	<1	0	<1	0	0
3491	*Plecotus auritus*	65	0	65	5	<1	0	<1	0	0
3519	*Plecotus auritus*	2	0	2	0	0	0	0	0	0
3524	*Plecotus auritus*	4	0	4	<2	<1	0	<1	0	0
3548	*Plecotus auritus*	3	0	3	4	<1	<1	<1	0	<2
3549	*Plecotus auritus*	31	<2	31	48	11	3	14	0	10
3550	*Plecotus auritus*	13	0	13	6	<1	<1	<1	0	<2
3551	*Plecotus auritus*	5	0	5	<2	2	<1	2	0	<2
3552	*Plecotus auritus*	9	2	12	2	2	5	7	0	0
3553	*Plecotus auritus*	<1	0	<1	<2	4	1	5	0	<2
3554	*Plecotus auritus*	55	4	58	90	17	2	20	<1	19
3555	*Plecotus auritus*	36	14	50	53	6	4	10	<1	15
3557	*Plecotus auritus*	5	4	8	4	2	1	4	<1	<2
3558	*Plecotus auritus*	20	0	20	<2	10	0	10	0	<2
3559	*Plecotus auritus*	4	0	4	3	<1	<1	<1	0	3
3560	*Plecotus auritus*	5	0	5	<2	2	<1	2	0	<2
3561	*Plecotus auritus*	14	0	14	22	4	<1	4	0	5
3562	*Plecotus auritus*	11	6	17	9	4	2	6	<1	5
3563	*Plecotus auritus*	1	0	1	4	15	2	17	<1	2

Sheet 3 Other insecticides

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3272	*Eptesicus serotinus*	0	0	0	0	0	0
3276	*Eptesicus serotinus*	0	0	0	0	0	<1
3280	*Eptesicus serotinus*	0	0	0	0	0	0
3288	*Eptesicus serotinus*	0	0	0	0	0	0
3311	*Eptesicus serotinus*	0	6	0	<2	<2	0
3319	*Eptesicus serotinus*	0	0	0	<2	<2	0
3450	*Eptesicus serotinus*	0	0	0	0	0	0
3451	*Eptesicus serotinus*	0	0	297	1345	1642	0
3452	*Eptesicus serotinus*	0	0	0	0	0	0
3454	*Eptesicus serotinus*	0	0	0	0	0	0
3456	*Eptesicus serotinus*	0	0	0	0	0	0
3458	*Eptesicus serotinus*	0	0	57	4404	4461	0
3463	*Eptesicus serotinus*	0	0	0	<2	<2	0
3464	*Eptesicus serotinus*	0	0	0	<2	<2	0
3465	*Eptesicus serotinus*	0	0	0	0	0	0
3467	*Eptesicus serotinus*	0	0	0	0	0	0
3468	*Eptesicus serotinus*	0	0	0	0	0	0
3469	*Eptesicus serotinus*	0	0	0	0	0	0
3470	*Eptesicus serotinus*	0	0	0	0	0	0
3472	*Eptesicus serotinus*	0	0	0	0	0	0
3473	*Eptesicus serotinus*	0	0	0	0	0	0
3474	*Eptesicus serotinus*	0	0	0	0	0	0
3485	*Eptesicus serotinus*	0	<4	0	0	0	0
3488	*Eptesicus serotinus*	0	0	0	0	0	0
3494	*Eptesicus serotinus*	0	0	0	0	0	0
3498	*Eptesicus serotinus*	0	0	0	0	0	0
3499	*Eptesicus serotinus*	0	0	0	0	0	0
3500	*Eptesicus serotinus*	0	0	0	0	0	0
3501	*Eptesicus serotinus*	0	0	0	0	0	0
3502	*Eptesicus serotinus*	0	0	0	0	0	<1
3503	*Eptesicus serotinus*	0	0	0	0	0	0
3506	*Eptesicus serotinus*	0	0	0	0	0	0
3509	*Eptesicus serotinus*	0	<4	0	0	0	<1
3510	*Eptesicus serotinus*	0	<4	0	0	0	<1
3511	*Eptesicus serotinus*	0	0	0	0	0	0
3512	*Eptesicus serotinus*	0	0	0	0	0	0
3513	*Eptesicus serotinus*	0	0	0	0	0	0
3514	*Eptesicus serotinus*	0	<4	0	0	0	0
3515	*Eptesicus serotinus*	0	835	0	0	0	0
3522	*Eptesicus serotinus*	0	0	0	0	0	0
3523	*Eptesicus serotinus*	0	0	0	0	0	0
3525	*Eptesicus serotinus*	0	0	0	0	0	0
3526	*Eptesicus serotinus*	0	<4	0	0	0	<1
3527	*Eptesicus serotinus*	0	0	0	0	0	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3529	*Eptesicus serotinus*	0	0	0	0	0	0
3530	*Eptesicus serotinus*	0	<4	0	<2	<2	<1
3531	*Eptesicus serotinus*	0	<4	0	<2	<2	<1
3532	*Eptesicus serotinus*	0	0	0	<2	<2	0
3533	*Eptesicus serotinus*	0	0	0	<2	<2	0
3534	*Eptesicus serotinus*	<4	<4	0	<2	<2	<1
3535	*Eptesicus serotinus*	0	0	0	0	0	0
3536	*Eptesicus serotinus*	<4	<4	0	<2	<2	<1
3537	*Eptesicus serotinus*	0	0	0	0	0	0
3538	*Eptesicus serotinus*	0	0	0	0	0	0
3539	*Eptesicus serotinus*	0	0	0	<2	<2	0
3540	*Eptesicus serotinus*	0	0	0	0	0	0
3541	*Eptesicus serotinus*	0	0	0	0	0	0
3542	*Eptesicus serotinus*	0	0	0	<2	<2	0
3543	*Eptesicus serotinus*	0	0	0	0	0	0
3544	*Eptesicus serotinus*	0	0	0	0	0	0
3545	*Eptesicus serotinus*	0	0	0	<2	<2	0
3546	*Eptesicus serotinus*	<4	<4	0	<2	<2	<1
3556	*Eptesicus serotinus*	0	0	0	<2	<2	0
3564	*Eptesicus serotinus*	0	0	0	0	0	0
3565	*Eptesicus serotinus*	0	0	0	<2	<2	0
3566	*Eptesicus serotinus*	0	0	0	<2	<2	0
3567	*Eptesicus serotinus*	<4	<4	0	<2	<2	<1
3568	*Eptesicus serotinus*	<4	<4	0	<2	<2	<1
3569	*Eptesicus serotinus*	0	0	0	0	0	0
3570	*Eptesicus serotinus*	0	0	0	0	0	0
3571	*Eptesicus serotinus*	0	0	0	0	0	0
3572	*Eptesicus serotinus*	0	0	0	0	0	0
3573	*Eptesicus serotinus*	0	0	0	<2	<2	0
3574	*Eptesicus serotinus*	0	0	0	0	0	0
3010	*Myotis myotis*	0	0	0	0	0	0
3012	*Myotis myotis*	0	0	0	0	0	0
3015	*Myotis myotis*	0	0	0	0	<2	0
3016	*Myotis myotis*	0	0	0	0	0	0
3017	*Myotis myotis*	0	0	0	0	<2	0
3073	*Myotis myotis*	0	0	0	0	0	0
3188	*Myotis myotis*	0	0	0	0	0	0
3247	*Myotis myotis*	0	0	0	10	10	0
3279	*Myotis myotis*	0	0	0	<2	<2	0
3378	*Myotis myotis*	0	0	0	<2	<2	0
3457	*Myotis myotis*	0	0	0	0	0	0
3459	*Myotis myotis*	0	0	0	<2	<2	0
3462	*Myotis myotis*	0	0	0	0	0	0
3517	*Myotis myotis*	0	0	0	0	0	0
3518	*Myotis myotis*	0	0	0	0	0	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3520	*Myotis myotis*	0	0	0	0	0	0
3521	*Myotis myotis*	0	0	0	0	0	0
3074	*Nyctalus noctula*	0	0	0	2	<2	0
3075	*Nyctalus noctula*	0	0	0	1	<2	0
3076	*Nyctalus noctula*	0	0	0	1	<2	0
3077	*Nyctalus noctula*	0	0	0	2	2	0
3078	*Nyctalus noctula*	0	0	0	1	<2	0
3079	*Nyctalus noctula*	0	0	0	0	<2	0
3080	*Nyctalus noctula*	0	0	0	1	<2	0
3081	*Nyctalus noctula*	0	0	0	2	<2	0
3082	*Nyctalus noctula*	0	0	0	4	4	0
3083	*Nyctalus noctula*	0	0	0	2	2	0
3084	*Nyctalus noctula*	0	0	0	1	<2	0
3085	*Nyctalus noctula*	0	0	0	0	<2	0
3086	*Nyctalus noctula*	0	0	0	2	<2	0
3087	*Nyctalus noctula*	0	0	0	10	10	0
3121	*Nyctalus noctula*	0	569	0	<2	<2	<1
3122	*Nyctalus noctula*	0	400	0	<2	<2	<1
3123	*Nyctalus noctula*	0	1631	0	<2	<2	<1
3197	*Nyctalus noctula*	0	0	0	<2	<2	0
3202	*Nyctalus noctula*	0	0	0	0	0	0
3203	*Nyctalus noctula*	0	0	45	26	71	2
3245	*Nyctalus noctula*	0	0	0	<2	<2	0
3248	*Nyctalus noctula*	0	0	0	0	0	0
3252	*Nyctalus noctula*	0	0	0	0	0	0
3254	*Nyctalus noctula*	0	0	0	3	3	0
3255	*Nyctalus noctula*	0	0	0	<2	<2	0
3260	*Nyctalus noctula*	0	0	0	0	0	0
3263	*Nyctalus noctula*	0	0	0	0	0	0
3275	*Nyctalus noctula*	0	0	0	<2	<2	<1
3277	*Nyctalus noctula*	0	0	0	<2	<2	0
3282	*Nyctalus noctula*	0	0	0	3	3	0
3284	*Nyctalus noctula*	0	0	0	<2	<2	0
3294	*Nyctalus noctula*	0	0	0	<2	<2	0
3453	*Nyctalus noctula*	0	0	0	<2	<2	0
3466	*Nyctalus noctula*	0	0	0	0	0	0
3471	*Nyctalus noctula*	0	0	0	0	0	0
3483	*Nyctalus noctula*	0	0	0	0	0	0
3484	*Nyctalus noctula*	0	0	0	<2	<2	0
3486	*Nyctalus noctula*	51	79	0	7	7	2
3487	*Nyctalus noctula*	0	0	0	0	0	0
3489	*Nyctalus noctula*	0	0	0	0	0	0
3490	*Nyctalus noctula*	0	0	0	0	0	0
3492	*Nyctalus noctula*	0	0	0	0	0	0
3493	*Nyctalus noctula*	0	0	0	<2	<2	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3495	*Nyctalus noctula*	0	0	0	0	0	0
3496	*Nyctalus noctula*	0	0	0	0	0	0
3497	*Nyctalus noctula*	0	0	0	<2	<2	0
3507	*Nyctalus noctula*	0	<4	0	0	0	<1
3508	*Nyctalus noctula*	0	0	0	<2	<2	0
3516	*Nyctalus noctula*	0	<4	0	<2	<2	0
3008	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3009	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3013	*Pipistrellus pipistrellus*	0	0	0	9	9	0
3021	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3023	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3024	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3025	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3026	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3027	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3028	*Pipistrellus pipistrellus*	0	0	0	37	37	0
3029	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3030	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3031	*Pipistrellus pipistrellus*	0	0	0	2	2	0
3033	*Pipistrellus pipistrellus*	0	0	0	4	4	0
3034	*Pipistrellus pipistrellus*	0	0	0	7	7	0
3036	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3037	*Pipistrellus pipistrellus*	0	0	0	2	2	0
3038	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3039	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3040	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3041	*Pipistrellus pipistrellus*	0	0	0	5	5	0
3042	*Pipistrellus pipistrellus*	0	0	0	8	8	0
3043	*Pipistrellus pipistrellus*	0	37	0	5	5	0
3044	*Pipistrellus pipistrellus*	0	0	0	23	23	0
3045	*Pipistrellus pipistrellus*	0	0	0	16	16	0
3046	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3047	*Pipistrellus pipistrellus*	0	0	0	2	2	0
3048	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3049	*Pipistrellus pipistrellus*	0	0	0	7	7	0
3050	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3051	*Pipistrellus pipistrellus*	0	0	0	2	2	0
3052	*Pipistrellus pipistrellus*	0	0	0	2	2	0
3053	*Pipistrellus pipistrellus*	0	0	0	6	6	0
3054	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3055	*Pipistrellus pipistrellus*	0	0	0	24	24	0
3056	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3063	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3064	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3065	*Pipistrellus pipistrellus*	0	0	0	0	0	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3066	*Pipistrellus pipistrellus*	0	0	0	8	8	0
3067	*Pipistrellus pipistrellus*	0	0	0	6	6	0
3068	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3069	*Pipistrellus pipistrellus*	0	0	0	14	14	0
3070	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3071	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3116	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3117	*Pipistrellus pipistrellus*	0	0	0	18	18	0
3119	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3126	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3128	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3129	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3130	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3132	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3133	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3134	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3145	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3147	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3148	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3149	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3150	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3151	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3152	*Pipistrellus pipistrellus*	0	26	0	<2	<2	0
3153	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3161	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3163	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3169	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3175	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3176	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3189	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3198	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3205	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3207	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3208	*Pipistrellus pipistrellus*	0	0	0	6	6	0
3209	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3210	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3211	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3215	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3216	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3220	*Pipistrellus pipistrellus*	0	0	0	0	0	<1
3221	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3222	*Pipistrellus pipistrellus*	0	0	0	9	9	0
3224	*Pipistrellus pipistrellus*	<4	<4	0	2	2	4
3225	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3226	*Pipistrellus pipistrellus*	0	0	0	0	0	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3228	*Pipistrellus pipistrellus*	0	<4	0	7	7	0
3229	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3230	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3233	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3235	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3237	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3240	*Pipistrellus pipistrellus*	0	0	0	4	4	0
3242	*Pipistrellus pipistrellus*	0	0	0	5	5	0
3244	*Pipistrellus pipistrellus*	0	0	0	18	18	0
3250	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3253	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3256	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3257	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3258	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3259	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3261	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3264	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3265	*Pipistrellus pipistrellus*	0	0	0	3	3	0
3268	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3278	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3281	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3286	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3291	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3293	*Pipistrellus pipistrellus*	0	0	0	4	4	<1
3295	*Pipistrellus pipistrellus*	0	0	0	7	7	0
3299	*Pipistrellus pipistrellus*	0	0	0	16	16	0
3302	*Pipistrellus pipistrellus*	0	0	0	6	6	0
3303	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3304	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3305	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3306	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3309	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3312	*Pipistrellus pipistrellus*	0	0	0	4	4	0
3313	*Pipistrellus pipistrellus*	0	0	0	<2	<2	<1
3314	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3317	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3318	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3320	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3321	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3322	*Pipistrellus pipistrellus*	0	0	0	14	14	0
3326	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3327	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3328	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	<1
3330	*Pipistrellus pipistrellus*	0	0	0	52	52	0
3331	*Pipistrellus pipistrellus*	0	0	0	0	0	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3332	*Pipistrellus pipistrellus*	0	<4	0	0	0	0
3333	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3334	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3335	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3336	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3337	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3338	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3339	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3341	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3342	*Pipistrellus pipistrellus*	0	8	0	<2	<2	0
3343	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3345	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3346	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3348	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3350	*Pipistrellus pipistrellus*	0	0	0	<2	<2	3
3351	*Pipistrellus pipistrellus*	0	0	0	14	14	0
3352	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3353	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3354	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3355	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3356	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3358	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3359	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3360	*Pipistrellus pipistrellus*	0	0	0	4	4	0
3361	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3363	*Pipistrellus pipistrellus*	0	0	0	<2	<2	<1
3364	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3365	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3373	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3374	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3376	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3379	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3381	*Pipistrellus pipistrellus*	0	0	0	10	10	0
3383	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3384	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3386	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3387	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3388	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3389	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3390	*Pipistrellus pipistrellus*	0	12	0	6	6	0
3391	*Pipistrellus pipistrellus*	0	4	0	3	3	0
3392	*Pipistrellus pipistrellus*	0	<4	0	5	5	0
3393	*Pipistrellus pipistrellus*	0	13	0	0	0	0
3394	*Pipistrellus pipistrellus*	0	0	0	4	4	0
3395	*Pipistrellus pipistrellus*	0	0	0	3	3	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3396	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3397	*Pipistrellus pipistrellus*	0	6	0	3	3	0
3398	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3399	*Pipistrellus pipistrellus*	0	<4	0	4	4	0
3400	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3401	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3402	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3405	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3406	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3407	*Pipistrellus pipistrellus*	0	0	0	11	11	0
3408	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3409	*Pipistrellus pipistrellus*	0	0	0	11	11	0
3410	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3411	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3413	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3414	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3415	*Pipistrellus pipistrellus*	0	4	0	3	3	0
3417	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3419	*Pipistrellus pipistrellus*	0	<4	0	3	3	0
3420	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3421	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3423	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3425	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3426	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3427	*Pipistrellus pipistrellus*	0	<4	0	<2	<2	0
3428	*Pipistrellus pipistrellus*	0	0	0	4	4	0
3429	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3430	*Pipistrellus pipistrellus*	0	<4	0	3	3	0
3433	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3434	*Pipistrellus pipistrellus*	0	0	0	6	6	0
3435	*Pipistrellus pipistrellus*	0	9	0	<2	<2	0
3460	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3475	*Pipistrellus pipistrellus*	0	0	0	14	14	<1
3476	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3477	*Pipistrellus pipistrellus*	0	0	0	26	26	0
3478	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3479	*Pipistrellus pipistrellus*	0	0	0	0	0	0
3480	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3481	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3482	*Pipistrellus pipistrellus*	0	0	0	<2	<2	0
3505	*Pipistrellus pipistrellus*	0	0	0	<2	<2	<1
3032	*Plecotus auritus*	0	0	0	<2	<2	0
3072	*Plecotus auritus*	0	0	0	0	0	0
3144	*Plecotus auritus*	0	0	0	<2	<2	0
3154	*Plecotus auritus*	0	0	0	<2	<2	0

No	Species	Deltamethrin [µg kg^{-1}]	Permethrin* [µg kg^{-1}]	Fipronil [µg kg^{-1}]	Fipronil sulfone [µg kg^{-1}]	Fipronil sum [µg kg^{-1}]	Chlorpyrifos [µg kg^{-1}]
3196	*Plecotus auritus*	0	0	0	0	0	0
3201	*Plecotus auritus*	0	<4	0	0	0	1
3212	*Plecotus auritus*	0	0	0	0	0	0
3267	*Plecotus auritus*	0	0	0	0	0	0
3296	*Plecotus auritus*	0	0	0	7	7	0
3300	*Plecotus auritus*	48	40	0	69	69	19
3329	*Plecotus auritus*	0	0	0	0	0	0
3357	*Plecotus auritus*	0	0	0	<2	<2	<1
3372	*Plecotus auritus*	0	0	0	0	0	0
3461	*Plecotus auritus*	0	0	0	9	9	0
3491	*Plecotus auritus*	0	0	0	<2	<2	0
3519	*Plecotus auritus*	0	0	0	0	0	0
3524	*Plecotus auritus*	0	0	0	<2	<2	0
3548	*Plecotus auritus*	0	0	0	0	0	0
3549	*Plecotus auritus*	0	0	0	<2	<2	0
3550	*Plecotus auritus*	0	0	0	11	11	0
3551	*Plecotus auritus*	0	0	0	<2	<2	0
3552	*Plecotus auritus*	0	0	0	<2	<2	0
3553	*Plecotus auritus*	0	0	0	<2	<2	0
3554	*Plecotus auritus*	<4	<4	0	<2	<2	2
3555	*Plecotus auritus*	<4	<4	0	<2	<2	2
3557	*Plecotus auritus*	0	<4	0	0	0	<1
3558	*Plecotus auritus*	0	0	0	0	0	0
3559	*Plecotus auritus*	0	0	0	0	0	0
3560	*Plecotus auritus*	0	0	0	0	0	0
3561	*Plecotus auritus*	0	0	0	<2	<2	0
3562	*Plecotus auritus*	<4	<4	0	<2	<2	<1
3563	*Plecotus auritus*	<4	<4	0	<2	<2	<1

Sheet 4 Fungicides

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3272	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3276	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3280	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3288	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3311	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3319	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3450	*Eptesicus serotinus*	0	0	0	0	0	<2	0	0
3451	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3452	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3454	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3456	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3458	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3463	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3464	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3465	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3467	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3468	*Eptesicus serotinus*	0	0	0	0	0	0	0	<4
3469	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3470	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3472	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3473	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3474	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3485	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3488	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3494	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3498	*Eptesicus serotinus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3499	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3500	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3501	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3502	*Eptesicus serotinus*	0	0	0	0	0	<2	0	0
3503	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3506	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3509	*Eptesicus serotinus*	0	0	0	0	<2	<2	0	<4
3510	*Eptesicus serotinus*	0	0	0	0	0	<2	0	<4
3511	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3512	*Eptesicus serotinus*	0	0	0	0	0	<2	0	0
3513	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3514	*Eptesicus serotinus*	0	0	0	0	<2	0	0	0
3515	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3522	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3523	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3525	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3526	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	<4
3527	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3529	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3530	*Eptesicus serotinus*	0	0	1	0	<2	<2	0	<4
3531	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	<4
3532	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3533	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3534	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	<4
3535	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3536	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	<4
3537	*Eptesicus serotinus*	0	0	0	0	0	<2	0	0
3538	*Eptesicus serotinus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3539	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3540	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3541	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3542	*Eptesicus serotinus*	0	0	0	0	0	<2	0	0
3543	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3544	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3545	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3546	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	<4
3556	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3564	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3565	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3566	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3567	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	<4
3568	*Eptesicus serotinus*	0	0	<1	0	<2	<2	0	0
3569	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3570	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3571	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3572	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3573	*Eptesicus serotinus*	0	0	0	0	0	0	0	0
3574	*Eptesicus serotinus*	0	0	<1	0	0	<2	0	0
3010	*Myotis myotis*	0	0	0	0	0	0	0	0
3012	*Myotis myotis*	0	0	0	0	0	0	0	0
3015	*Myotis myotis*	0	0	0	0	0	0	0	0
3016	*Myotis myotis*	0	0	0	0	0	0	0	0
3017	*Myotis myotis*	0	0	0	0	0	0	0	0
3073	*Myotis myotis*	0	0	0	0	0	0	0	0
3188	*Myotis myotis*	0	0	<1	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3247	*Myotis myotis*	0	0	0	0	0	0	0	<4
3279	*Myotis myotis*	0	0	<1	0	0	0	0	<4
3378	*Myotis myotis*	0	0	9	0	0	0	0	0
3457	*Myotis myotis*	0	0	0	0	0	<2	0	0
3459	*Myotis myotis*	0	0	<1	0	<2	<2	0	0
3462	*Myotis myotis*	0	0	0	0	0	<2	0	0
3517	*Myotis myotis*	0	0	<1	0	0	0	0	0
3518	*Myotis myotis*	0	0	2	0	0	<2	0	0
3520	*Myotis myotis*	0	0	0	0	0	0	0	0
3521	*Myotis myotis*	0	0	0	0	0	0	0	0
3074	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3075	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3076	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3077	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3078	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3079	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3080	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3081	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3082	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3083	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3084	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3085	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3086	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3087	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3121	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3122	*Nyctalus noctula*	0	0	0	0	0	<2	0	0
3123	*Nyctalus noctula*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3197	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3202	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3203	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3245	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3248	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3252	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3254	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3255	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3260	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3263	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3275	*Nyctalus noctula*	0	0	0	0	<2	<2	0	<4
3277	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3282	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3284	*Nyctalus noctula*	0	0	0	0	0	0	0	<4
3294	*Nyctalus noctula*	0	0	0	0	<2	<2	0	<4
3453	*Nyctalus noctula*	0	0	0	0	3	0	0	0
3466	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3471	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3483	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3484	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3486	*Nyctalus noctula*	0	<4	52	0	0	0	<4	4
3487	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3489	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3490	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3492	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3493	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3495	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3496	*Nyctalus noctula*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3497	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3507	*Nyctalus noctula*	0	0	0	0	0	<2	0	<4
3508	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3516	*Nyctalus noctula*	0	0	0	0	0	0	0	0
3008	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3009	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3013	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3021	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3023	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3024	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3025	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3026	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3027	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3028	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3029	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3030	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3031	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3033	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	12
3034	*Pipistrellus pipistrellus*	0	0	0	2	0	<2	0	<4
3036	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3037	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3038	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3039	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3040	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3041	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3042	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3043	*Pipistrellus pipistrellus*	0	0	0	5	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3044	*Pipistrellus pipistrellus*	0	0	0	0	3	0	0	0
3045	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	8
3046	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3047	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3048	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3049	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3050	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3051	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3052	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3053	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3054	*Pipistrellus pipistrellus*	0	0	0	0	<2	0	0	0
3055	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3056	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3063	*Pipistrellus pipistrellus*	0	0	0	32	0	0	0	0
3064	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3065	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3066	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3067	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3068	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3069	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3070	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3071	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3116	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3117	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3119	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	<4
3126	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3128	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3129	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3130	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3132	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3133	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3134	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3145	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3147	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3148	*Pipistrellus pipistrellus*	0	0	0	0	6	0	0	0
3149	*Pipistrellus pipistrellus*	0	0	0	0	4	0	0	0
3150	*Pipistrellus pipistrellus*	0	0	0	0	10	0	0	0
3151	*Pipistrellus pipistrellus*	0	0	0	0	25	0	0	0
3152	*Pipistrellus pipistrellus*	0	0	0	0	9	0	0	0
3153	*Pipistrellus pipistrellus*	0	0	0	0	33	0	0	0
3161	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3163	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3169	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3175	*Pipistrellus pipistrellus*	0	0	0	0	<2	0	0	0
3176	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3189	*Pipistrellus pipistrellus*	0	0	<1	0	0	0	0	<4
3198	*Pipistrellus pipistrellus*	0	0	<1	0	0	0	0	<4
3205	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3207	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3208	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3209	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3210	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3211	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3215	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3216	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3220	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3221	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3222	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3224	*Pipistrellus pipistrellus*	0	0	1	0	4	7	<4	<4
3225	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3226	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3228	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3229	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3230	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3233	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3235	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3237	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3240	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3242	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	<4
3244	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3250	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3253	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3256	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3257	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3258	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3259	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3261	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3264	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3265	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3268	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3278	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	<4
3281	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3286	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3291	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3293	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3295	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3299	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3302	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3303	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3304	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3305	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3306	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3309	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3312	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3313	*Pipistrellus pipistrellus*	0	0	<1	0	<2	<2	0	<4
3314	*Pipistrellus pipistrellus*	0	0	<1	0	0	0	0	<4
3317	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3318	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3320	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3321	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3322	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3326	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3327	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3328	*Pipistrellus pipistrellus*	0	0	<1	<2	4	<2	0	<4
3330	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3331	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3332	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3333	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3334	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3335	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3336	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3337	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3338	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3339	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3341	*Pipistrellus pipistrellus*	0	0	0	<2	0	0	0	0
3342	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3343	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3345	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3346	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3348	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3350	*Pipistrellus pipistrellus*	0	0	<1	<2	<2	<2	0	<4
3351	*Pipistrellus pipistrellus*	0	0	0	0	<2	<2	0	<4
3352	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3353	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3354	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3355	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3356	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3358	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3359	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3360	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3361	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3363	*Pipistrellus pipistrellus*	0	0	<1	0	0	<2	0	<4
3364	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3365	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3373	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3374	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3376	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3379	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3381	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3383	*Pipistrellus pipistrellus*	0	0	<1	0	<2	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3384	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3386	*Pipistrellus pipistrellus*	0	0	<1	<2	<2	<2	0	0
3387	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3388	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3389	*Pipistrellus pipistrellus*	0	0	0	0	0	5	0	0
3390	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3391	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	<4
3392	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3393	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3394	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3395	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3396	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3397	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3398	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3399	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3400	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3401	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3402	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3405	*Pipistrellus pipistrellus*	0	0	<1	0	0	<2	0	0
3406	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3407	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3408	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3409	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3410	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3411	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3413	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3414	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3415	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3417	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3419	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3420	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3421	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3423	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3425	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3426	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3427	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3428	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3429	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3430	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3433	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3434	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3435	*Pipistrellus pipistrellus*	0	0	0	0	<2	<2	0	0
3460	*Pipistrellus pipistrellus*	0	0	0	0	<2	<2	0	0
3475	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3476	*Pipistrellus pipistrellus*	0	0	0	0	0	<2	0	0
3477	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3478	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3479	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3480	*Pipistrellus pipistrellus*	0	0	<1	0	0	<2	0	<4
3481	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3482	*Pipistrellus pipistrellus*	0	0	0	0	0	0	0	0
3505	*Pipistrellus pipistrellus*	0	0	<1	0	0	<2	0	<4
3032	*Plecotus auritus*	0	0	0	0	0	0	0	0
3072	*Plecotus auritus*	0	0	0	0	0	0	0	0
3144	*Plecotus auritus*	0	0	0	0	0	0	0	0

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3154	*Plecotus auritus*	0	0	0	0	0	0	0	0
3196	*Plecotus auritus*	0	0	0	0	0	0	0	0
3201	*Plecotus auritus*	0	0	<1	0	<2	<2	0	0
3212	*Plecotus auritus*	0	0	0	0	0	0	0	0
3267	*Plecotus auritus*	0	0	0	0	0	0	0	0
3296	*Plecotus auritus*	0	0	0	0	0	0	0	0
3300	*Plecotus auritus*	0	<4	88	91	55	65	<4	31
3329	*Plecotus auritus*	0	0	0	0	0	0	0	0
3357	*Plecotus auritus*	0	0	0	0	0	<2	0	0
3372	*Plecotus auritus*	0	0	0	0	0	0	0	0
3461	*Plecotus auritus*	0	0	0	0	0	0	0	0
3491	*Plecotus auritus*	0	0	0	0	0	0	0	0
3519	*Plecotus auritus*	0	0	0	0	0	0	0	0
3524	*Plecotus auritus*	<4	0	<1	0	0	<2	0	0
3548	*Plecotus auritus*	0	0	0	0	0	0	0	0
3549	*Plecotus auritus*	0	0	0	0	0	0	0	0
3550	*Plecotus auritus*	0	0	0	0	0	0	0	0
3551	*Plecotus auritus*	0	0	0	0	0	<2	0	0
3552	*Plecotus auritus*	0	0	0	0	0	0	0	0
3553	*Plecotus auritus*	0	0	0	0	0	0	0	0
3554	*Plecotus auritus*	0	0	<1	0	3	<2	0	<4
3555	*Plecotus auritus*	0	0	<1	0	3	<2	0	<4
3557	*Plecotus auritus*	0	0	<1	0	<2	<2	0	<4
3558	*Plecotus auritus*	0	0	0	0	0	0	0	0
3559	*Plecotus auritus*	0	0	0	0	0	0	0	0
3560	*Plecotus auritus*	0	0	0	0	0	0	0	0
3561	*Plecotus auritus*	0	0	0	0	0	0	0	0
3562	*Plecotus auritus*	0	0	<1	0	<2	<2	0	<4

No	Species	Azoxy-strobin [µg kg^{-1}]	Difeno-conazole* [µg kg^{-1}]	Epoxi-conazole [µg kg^{-1}]	Propi-conazole* [µg kg^{-1}]	Tebu-conazole [µg kg^{-1}]	Tetra-conazole [µg kg^{-1}]	Dimetho-morph* [µg kg^{-1}]	Fenpropi-morph [µg kg^{-1}]
3563	*Plecotus auritus*	0	0	<1	0	<2	<2	0	<4

Sheet 5 Herbicides

No	Species	Picolinafen [µg kg^{-1}]
3272	*Eptesicus serotinus*	0
3276	*Eptesicus serotinus*	0
3280	*Eptesicus serotinus*	0
3288	*Eptesicus serotinus*	0
3311	*Eptesicus serotinus*	0
3319	*Eptesicus serotinus*	0
3450	*Eptesicus serotinus*	0
3451	*Eptesicus serotinus*	0
3452	*Eptesicus serotinus*	0
3454	*Eptesicus serotinus*	0
3456	*Eptesicus serotinus*	0
3458	*Eptesicus serotinus*	0
3463	*Eptesicus serotinus*	0
3464	*Eptesicus serotinus*	0
3465	*Eptesicus serotinus*	0
3467	*Eptesicus serotinus*	0
3468	*Eptesicus serotinus*	0
3469	*Eptesicus serotinus*	0
3470	*Eptesicus serotinus*	0
3472	*Eptesicus serotinus*	0
3473	*Eptesicus serotinus*	0
3474	*Eptesicus serotinus*	0
3485	*Eptesicus serotinus*	0
3488	*Eptesicus serotinus*	0
3494	*Eptesicus serotinus*	0
3498	*Eptesicus serotinus*	0
3499	*Eptesicus serotinus*	0
3500	*Eptesicus serotinus*	0
3501	*Eptesicus serotinus*	0
3502	*Eptesicus serotinus*	0
3503	*Eptesicus serotinus*	0
3506	*Eptesicus serotinus*	0
3509	*Eptesicus serotinus*	0
3510	*Eptesicus serotinus*	0
3511	*Eptesicus serotinus*	0
3512	*Eptesicus serotinus*	0
3513	*Eptesicus serotinus*	0
3514	*Eptesicus serotinus*	0
3515	*Eptesicus serotinus*	0
3522	*Eptesicus serotinus*	0
3523	*Eptesicus serotinus*	0
3525	*Eptesicus serotinus*	0
3526	*Eptesicus serotinus*	0
3527	*Eptesicus serotinus*	0
3529	*Eptesicus serotinus*	0
3530	*Eptesicus serotinus*	0
3531	*Eptesicus serotinus*	0
3532	*Eptesicus serotinus*	0
3533	*Eptesicus serotinus*	0
3534	*Eptesicus serotinus*	0
3535	*Eptesicus serotinus*	0
3536	*Eptesicus serotinus*	0
3537	*Eptesicus serotinus*	0
3538	*Eptesicus serotinus*	0
3539	*Eptesicus serotinus*	0
3540	*Eptesicus serotinus*	0
3541	*Eptesicus serotinus*	0
3542	*Eptesicus serotinus*	0
3543	*Eptesicus serotinus*	0
3544	*Eptesicus serotinus*	0
3545	*Eptesicus serotinus*	0
3546	*Eptesicus serotinus*	0
3556	*Eptesicus serotinus*	0
3564	*Eptesicus serotinus*	0
3565	*Eptesicus serotinus*	0
3566	*Eptesicus serotinus*	0
3567	*Eptesicus serotinus*	0
3568	*Eptesicus serotinus*	0
3569	*Eptesicus serotinus*	0
3570	*Eptesicus serotinus*	0
3571	*Eptesicus serotinus*	0
3572	*Eptesicus serotinus*	0
3573	*Eptesicus serotinus*	0
3574	*Eptesicus serotinus*	0
3010	*Myotis myotis*	0
3012	*Myotis myotis*	0
3015	*Myotis myotis*	0
3016	*Myotis myotis*	0
3017	*Myotis myotis*	0
3073	*Myotis myotis*	0
3188	*Myotis myotis*	0
3247	*Myotis myotis*	0
3279	*Myotis myotis*	0
3378	*Myotis myotis*	0
3457	*Myotis myotis*	0
3459	*Myotis myotis*	0
3462	*Myotis myotis*	0
3517	*Myotis myotis*	0
3518	*Myotis myotis*	0

No	Species	Picolinafen [µg kg^{-1}]
3520	*Myotis myotis*	0
3521	*Myotis myotis*	0
3074	*Nyctalus noctula*	0
3075	*Nyctalus noctula*	0
3076	*Nyctalus noctula*	0
3077	*Nyctalus noctula*	0
3078	*Nyctalus noctula*	0
3079	*Nyctalus noctula*	0
3080	*Nyctalus noctula*	0
3081	*Nyctalus noctula*	0
3082	*Nyctalus noctula*	0
3083	*Nyctalus noctula*	0
3084	*Nyctalus noctula*	0
3085	*Nyctalus noctula*	0
3086	*Nyctalus noctula*	0
3087	*Nyctalus noctula*	0
3121	*Nyctalus noctula*	0
3122	*Nyctalus noctula*	0
3123	*Nyctalus noctula*	0
3197	*Nyctalus noctula*	0
3202	*Nyctalus noctula*	0
3203	*Nyctalus noctula*	0
3245	*Nyctalus noctula*	0
3248	*Nyctalus noctula*	0
3252	*Nyctalus noctula*	0
3254	*Nyctalus noctula*	0
3255	*Nyctalus noctula*	0
3260	*Nyctalus noctula*	0
3263	*Nyctalus noctula*	0
3275	*Nyctalus noctula*	0
3277	*Nyctalus noctula*	0
3282	*Nyctalus noctula*	0
3284	*Nyctalus noctula*	0
3294	*Nyctalus noctula*	0
3453	*Nyctalus noctula*	0
3466	*Nyctalus noctula*	0
3471	*Nyctalus noctula*	0
3483	*Nyctalus noctula*	0
3484	*Nyctalus noctula*	0
3486	*Nyctalus noctula*	0
3487	*Nyctalus noctula*	0
3489	*Nyctalus noctula*	0
3490	*Nyctalus noctula*	0
3492	*Nyctalus noctula*	0
3493	*Nyctalus noctula*	0

No	Species	Picolinafen [µg kg^{-1}]
3495	*Nyctalus noctula*	0
3496	*Nyctalus noctula*	0
3497	*Nyctalus noctula*	0
3507	*Nyctalus noctula*	0
3508	*Nyctalus noctula*	0
3516	*Nyctalus noctula*	0
3008	*Pipistrellus pipistrellus*	0
3009	*Pipistrellus pipistrellus*	0
3013	*Pipistrellus pipistrellus*	0
3021	*Pipistrellus pipistrellus*	0
3023	*Pipistrellus pipistrellus*	0
3024	*Pipistrellus pipistrellus*	0
3025	*Pipistrellus pipistrellus*	0
3026	*Pipistrellus pipistrellus*	0
3027	*Pipistrellus pipistrellus*	0
3028	*Pipistrellus pipistrellus*	0
3029	*Pipistrellus pipistrellus*	0
3030	*Pipistrellus pipistrellus*	0
3031	*Pipistrellus pipistrellus*	0
3033	*Pipistrellus pipistrellus*	0
3034	*Pipistrellus pipistrellus*	0
3036	*Pipistrellus pipistrellus*	0
3037	*Pipistrellus pipistrellus*	0
3038	*Pipistrellus pipistrellus*	0
3039	*Pipistrellus pipistrellus*	0
3040	*Pipistrellus pipistrellus*	0
3041	*Pipistrellus pipistrellus*	0
3042	*Pipistrellus pipistrellus*	0
3043	*Pipistrellus pipistrellus*	0
3044	*Pipistrellus pipistrellus*	0
3045	*Pipistrellus pipistrellus*	0
3046	*Pipistrellus pipistrellus*	0
3047	*Pipistrellus pipistrellus*	0
3048	*Pipistrellus pipistrellus*	0
3049	*Pipistrellus pipistrellus*	0
3050	*Pipistrellus pipistrellus*	0
3051	*Pipistrellus pipistrellus*	0
3052	*Pipistrellus pipistrellus*	0
3053	*Pipistrellus pipistrellus*	0
3054	*Pipistrellus pipistrellus*	0
3055	*Pipistrellus pipistrellus*	0
3056	*Pipistrellus pipistrellus*	0
3063	*Pipistrellus pipistrellus*	0
3064	*Pipistrellus pipistrellus*	0
3065	*Pipistrellus pipistrellus*	0

No	Species	Picolinafen [µg kg^{-1}]
3066	*Pipistrellus pipistrellus*	0
3067	*Pipistrellus pipistrellus*	0
3068	*Pipistrellus pipistrellus*	0
3069	*Pipistrellus pipistrellus*	0
3070	*Pipistrellus pipistrellus*	0
3071	*Pipistrellus pipistrellus*	0
3116	*Pipistrellus pipistrellus*	0
3117	*Pipistrellus pipistrellus*	0
3119	*Pipistrellus pipistrellus*	0
3126	*Pipistrellus pipistrellus*	0
3128	*Pipistrellus pipistrellus*	0
3129	*Pipistrellus pipistrellus*	0
3130	*Pipistrellus pipistrellus*	0
3132	*Pipistrellus pipistrellus*	0
3133	*Pipistrellus pipistrellus*	0
3134	*Pipistrellus pipistrellus*	0
3145	*Pipistrellus pipistrellus*	0
3147	*Pipistrellus pipistrellus*	0
3148	*Pipistrellus pipistrellus*	0
3149	*Pipistrellus pipistrellus*	0
3150	*Pipistrellus pipistrellus*	0
3151	*Pipistrellus pipistrellus*	0
3152	*Pipistrellus pipistrellus*	0
3153	*Pipistrellus pipistrellus*	0
3161	*Pipistrellus pipistrellus*	0
3163	*Pipistrellus pipistrellus*	0
3169	*Pipistrellus pipistrellus*	0
3175	*Pipistrellus pipistrellus*	0
3176	*Pipistrellus pipistrellus*	0
3189	*Pipistrellus pipistrellus*	0
3198	*Pipistrellus pipistrellus*	0
3205	*Pipistrellus pipistrellus*	0
3207	*Pipistrellus pipistrellus*	0
3208	*Pipistrellus pipistrellus*	0
3209	*Pipistrellus pipistrellus*	0
3210	*Pipistrellus pipistrellus*	0
3211	*Pipistrellus pipistrellus*	0
3215	*Pipistrellus pipistrellus*	0
3216	*Pipistrellus pipistrellus*	0
3220	*Pipistrellus pipistrellus*	0
3221	*Pipistrellus pipistrellus*	0
3222	*Pipistrellus pipistrellus*	0
3224	*Pipistrellus pipistrellus*	0
3225	*Pipistrellus pipistrellus*	0
3226	*Pipistrellus pipistrellus*	0

No	Species	Picolinafen [µg kg^{-1}]
3228	*Pipistrellus pipistrellus*	0
3229	*Pipistrellus pipistrellus*	0
3230	*Pipistrellus pipistrellus*	0
3233	*Pipistrellus pipistrellus*	0
3235	*Pipistrellus pipistrellus*	0
3237	*Pipistrellus pipistrellus*	0
3240	*Pipistrellus pipistrellus*	0
3242	*Pipistrellus pipistrellus*	0
3244	*Pipistrellus pipistrellus*	0
3250	*Pipistrellus pipistrellus*	0
3253	*Pipistrellus pipistrellus*	0
3256	*Pipistrellus pipistrellus*	0
3257	*Pipistrellus pipistrellus*	0
3258	*Pipistrellus pipistrellus*	0
3259	*Pipistrellus pipistrellus*	0
3261	*Pipistrellus pipistrellus*	0
3264	*Pipistrellus pipistrellus*	0
3265	*Pipistrellus pipistrellus*	0
3268	*Pipistrellus pipistrellus*	0
3278	*Pipistrellus pipistrellus*	0
3281	*Pipistrellus pipistrellus*	0
3286	*Pipistrellus pipistrellus*	0
3291	*Pipistrellus pipistrellus*	0
3293	*Pipistrellus pipistrellus*	0
3295	*Pipistrellus pipistrellus*	0
3299	*Pipistrellus pipistrellus*	0
3302	*Pipistrellus pipistrellus*	0
3303	*Pipistrellus pipistrellus*	0
3304	*Pipistrellus pipistrellus*	0
3305	*Pipistrellus pipistrellus*	0
3306	*Pipistrellus pipistrellus*	0
3309	*Pipistrellus pipistrellus*	0
3312	*Pipistrellus pipistrellus*	0
3313	*Pipistrellus pipistrellus*	0
3314	*Pipistrellus pipistrellus*	0
3317	*Pipistrellus pipistrellus*	0
3318	*Pipistrellus pipistrellus*	0
3320	*Pipistrellus pipistrellus*	0
3321	*Pipistrellus pipistrellus*	0
3322	*Pipistrellus pipistrellus*	0
3326	*Pipistrellus pipistrellus*	0
3327	*Pipistrellus pipistrellus*	0
3328	*Pipistrellus pipistrellus*	0
3330	*Pipistrellus pipistrellus*	0
3331	*Pipistrellus pipistrellus*	0

No	Species	Picolinafen [µg kg^{-1}]
3332	*Pipistrellus pipistrellus*	0
3333	*Pipistrellus pipistrellus*	0
3334	*Pipistrellus pipistrellus*	0
3335	*Pipistrellus pipistrellus*	0
3336	*Pipistrellus pipistrellus*	0
3337	*Pipistrellus pipistrellus*	0
3338	*Pipistrellus pipistrellus*	0
3339	*Pipistrellus pipistrellus*	0
3341	*Pipistrellus pipistrellus*	0
3342	*Pipistrellus pipistrellus*	0
3343	*Pipistrellus pipistrellus*	0
3345	*Pipistrellus pipistrellus*	0
3346	*Pipistrellus pipistrellus*	0
3348	*Pipistrellus pipistrellus*	0
3350	*Pipistrellus pipistrellus*	0
3351	*Pipistrellus pipistrellus*	0
3352	*Pipistrellus pipistrellus*	0
3353	*Pipistrellus pipistrellus*	0
3354	*Pipistrellus pipistrellus*	0
3355	*Pipistrellus pipistrellus*	0
3356	*Pipistrellus pipistrellus*	0
3358	*Pipistrellus pipistrellus*	0
3359	*Pipistrellus pipistrellus*	0
3360	*Pipistrellus pipistrellus*	0
3361	*Pipistrellus pipistrellus*	0
3363	*Pipistrellus pipistrellus*	0
3364	*Pipistrellus pipistrellus*	0
3365	*Pipistrellus pipistrellus*	0
3373	*Pipistrellus pipistrellus*	0
3374	*Pipistrellus pipistrellus*	0
3376	*Pipistrellus pipistrellus*	0
3379	*Pipistrellus pipistrellus*	0
3381	*Pipistrellus pipistrellus*	0
3383	*Pipistrellus pipistrellus*	0
3384	*Pipistrellus pipistrellus*	0
3386	*Pipistrellus pipistrellus*	0
3387	*Pipistrellus pipistrellus*	0
3388	*Pipistrellus pipistrellus*	0
3389	*Pipistrellus pipistrellus*	0
3390	*Pipistrellus pipistrellus*	0
3391	*Pipistrellus pipistrellus*	0
3392	*Pipistrellus pipistrellus*	0
3393	*Pipistrellus pipistrellus*	0
3394	*Pipistrellus pipistrellus*	0
3395	*Pipistrellus pipistrellus*	0

No	Species	Picolinafen [µg kg^{-1}]
3396	*Pipistrellus pipistrellus*	0
3397	*Pipistrellus pipistrellus*	0
3398	*Pipistrellus pipistrellus*	0
3399	*Pipistrellus pipistrellus*	0
3400	*Pipistrellus pipistrellus*	0
3401	*Pipistrellus pipistrellus*	0
3402	*Pipistrellus pipistrellus*	0
3405	*Pipistrellus pipistrellus*	0
3406	*Pipistrellus pipistrellus*	0
3407	*Pipistrellus pipistrellus*	0
3408	*Pipistrellus pipistrellus*	0
3409	*Pipistrellus pipistrellus*	0
3410	*Pipistrellus pipistrellus*	0
3411	*Pipistrellus pipistrellus*	0
3413	*Pipistrellus pipistrellus*	0
3414	*Pipistrellus pipistrellus*	0
3415	*Pipistrellus pipistrellus*	0
3417	*Pipistrellus pipistrellus*	0
3419	*Pipistrellus pipistrellus*	0
3420	*Pipistrellus pipistrellus*	0
3421	*Pipistrellus pipistrellus*	0
3423	*Pipistrellus pipistrellus*	0
3425	*Pipistrellus pipistrellus*	0
3426	*Pipistrellus pipistrellus*	0
3427	*Pipistrellus pipistrellus*	0
3428	*Pipistrellus pipistrellus*	0
3429	*Pipistrellus pipistrellus*	0
3430	*Pipistrellus pipistrellus*	0
3433	*Pipistrellus pipistrellus*	0
3434	*Pipistrellus pipistrellus*	0
3435	*Pipistrellus pipistrellus*	0
3460	*Pipistrellus pipistrellus*	0
3475	*Pipistrellus pipistrellus*	0
3476	*Pipistrellus pipistrellus*	0
3477	*Pipistrellus pipistrellus*	0
3478	*Pipistrellus pipistrellus*	0
3479	*Pipistrellus pipistrellus*	0
3480	*Pipistrellus pipistrellus*	0
3481	*Pipistrellus pipistrellus*	0
3482	*Pipistrellus pipistrellus*	0
3505	*Pipistrellus pipistrellus*	0
3032	*Plecotus auritus*	0
3072	*Plecotus auritus*	0
3144	*Plecotus auritus*	0
3154	*Plecotus auritus*	0

No	Species	Picolinafen [µg kg^{-1}]
3196	*Plecotus auritus*	0
3201	*Plecotus auritus*	0
3212	*Plecotus auritus*	0
3267	*Plecotus auritus*	0
3296	*Plecotus auritus*	0
3300	*Plecotus auritus*	<2
3329	*Plecotus auritus*	0
3357	*Plecotus auritus*	0
3372	*Plecotus auritus*	0
3461	*Plecotus auritus*	0
3491	*Plecotus auritus*	0
3519	*Plecotus auritus*	0
3524	*Plecotus auritus*	0
3548	*Plecotus auritus*	0
3549	*Plecotus auritus*	0
3550	*Plecotus auritus*	0
3551	*Plecotus auritus*	0
3552	*Plecotus auritus*	0
3553	*Plecotus auritus*	0
3554	*Plecotus auritus*	0
3555	*Plecotus auritus*	0
3557	*Plecotus auritus*	0
3558	*Plecotus auritus*	0
3559	*Plecotus auritus*	0
3560	*Plecotus auritus*	0
3561	*Plecotus auritus*	0
3562	*Plecotus auritus*	0
3563	*Plecotus auritus*	0

5. Modified QuEChERS approach for leaf and needle samples

Löbbert, A.; **Schanzer, S.**; Krehenwinkel, H.; Bracher, F.; Müller, C., Determination of multi pesticide residues in leaf and needle samples using a modified QuEChERS approach and gas chromatography-tandem mass spectrometry. *Analytical Methods* **2021**, 13, 1138-1146.

5.1. Topic

In order to investigate a possible connection between pesticide use and leaf-associated insect decline, Jun.-Prof. Dr. Henrik Krehenwinkel from the Department of Biogeography of Trier University, Germany provided samples of leaves and needles that were collected by the German Environment Specimen Bank over the last 30 years to screen them for pesticides and pollutants. The samples originated from sites with different land use, from national parks to urban areas.

Especially insects are prone to unintentional killings through pest control. For example, the insecticide KARATE® FORST is approved for bark beetle control in German forests.[98] However, the ingredient *lambda*-cyhalothrin (**Fig. 17**) is a broad-spectrum insecticide and can also kill aphids or caterpillars.[3] Thus, the likelihood of unintended eradication of beneficial insects by the application of such substances is rather high. Consequently, the investigation of pesticide residues on trees, that serve as a habitat for insects, was a vital parameter for the project of Jun.-Prof. Dr. Krehenwinkel.

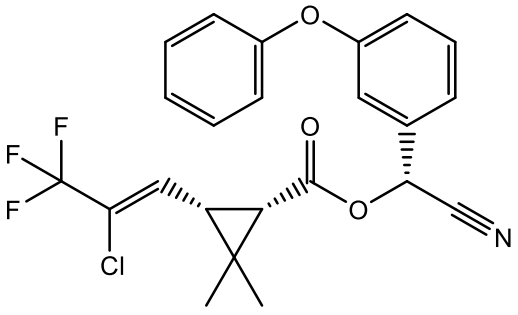


Figure 17 The active ingredient of KARATE® FORST, the pyrethroid insecticide *lambda*-cyhalothrin.

Even though the occurrence of pesticides in forests is very probable, there is no special analytical method for the investigation of these target compounds within vegetation samples. Thus, in order to investigate the pesticide load of forest ecosystems, a novel sample preparation method had to be developed. Leaves and needles present an appropriate matrix, as they are easily obtained without critical damage to the plants. For this project, beech (*Fagus sylvatica*) leaves and spruce (*Picea abies*) needles were chosen as representative matrices

for German forests. Spruce trees are conifers and are the most common trees in Germany, covering 26% of all forest areas; beech trees are deciduous trees and the third most common species with a rate of 16%.[99] Therefore, they are well-suited for a representative study.

From the German Environment Specimen Bank, 2 g of material, which was previously comminuted with a cryo mill, was available for each sample. Therefore, a modified QuEChERS approach with a reduced sample size of 2 g had to be developed and optimized. During method development, water and solvent amount, buffering, different salt combinations and clean-up strategies were tested. Especially the sample clean-up was a crucial step for this approach, as the vegetation samples represented a highly challenging matrix due to their high content of chlorophyll. For the optimized sample preparation, a full validation was performed in accordance with SANTE/12682/2019, regarding LOQs, linearity, recoveries and matrix effects.[93] Additionally, various vegetation samples were analysed with the developed method for proof of concept. All samples were analysed with a GC-MS/MS system, which was operated in dMRM mode in order to maximize specificity and sensitivity. The graphical abstract of the article, which is depicted in **Fig. 18**, shows the partitioned raw extract of the investigated matrix.[100]

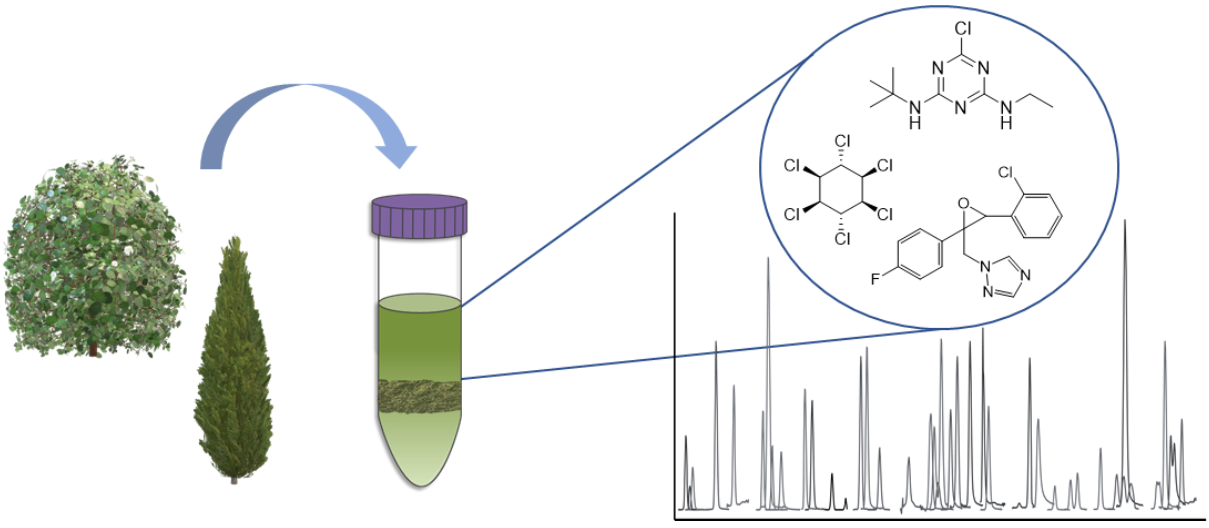


Figure 18 Graphical abstract of the article. Beech leaves and spruce needles were analysed with a modified QuEChERS approach in order to investigate the pesticide load.[100]

5.2. Personal contribution

All method optimization experiments and the validation were carried out under my supervision by Arnelle Löbbert who absolved her master's thesis at our group. She performed the experiments and curated the GC-MS/MS data. Furthermore, she wrote the original draft and incorporated the changes suggested by the other authors.

My contribution to this article was the supervision of the project at all stages as well as the initial conceptualization of the methodology and the formal analysis. Additionally, I reviewed and edited the original draft and supported the visualization.

Jun.-Prof. Dr. Henrik Krehenwinkel provided resources for this project, including the vegetation samples analysed for proof of concept, and helped with the funding acquisition. He also reviewed and edited the original draft.

Prof. Dr. Franz Bracher provided resources for the project and furthermore reviewed and edited the original draft.

Dr. Christoph Müller contributed to the conceptualization and supported the supervision of the project at all stages. Furthermore, he aided with the formal analysis, and reviewed and edited the original draft.

5.3. Article

The article is printed in its original wording as published in Analytical Methods. The formatting may vary slightly compared to the journal article.

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Determination of multi pesticide residues in leaf and needle samples using a modified QuEChERS approach and gas chromatography-tandem mass spectrometry†

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In order to gain a better insight into pesticide and pollutant exposure in forests, a rapid and sensitive gas chromatography-tandem mass spectrometry (GC-MS/MS) method for the determination of 208 pesticide residues in leaves and needles has been established. The modified QuEChERS (quick, easy, cheap, effective, rugged and safe) approach uses 2 g of homogenized sample, acetonitrile and water as extraction agents, combined with citrate buffer for the following salting out step. The limits of quantification (LOQs) were determined to 0.0025–0.05 mg kg^{-1}, respectively. Calibration curves showed a linear range between the respective LOQ and 1.0 mg kg^{-1} with coefficients of determination (R^2) ≥ 0.99 for all analyzed pesticides. The recovery rates ranged from 69.7% to 92.0% with a relative standard deviation below 20%. The analysis of beech leaves, spruce and pine needles (each n = 3) provided a proof of concept for the developed methodology and revealed the presence of six pesticide residues (boscalid, epoxiconazole, fenpropimorph, lindane, terbuthylazine, terbuthylazine-desethyl). The results underline the strong need for systematic surveillance of the uncontrollable exposure of pesticides to nature.

1 Introduction

In the last few decades, public concern regarding the long-term impact of pesticides and pollutants on the environment has increased. An extensive array of chemicals including chlorinated pesticides, dioxins, polychlorinated biphenyls and phthalates are nowadays prohibited or limited in their use as non-controlled dissemination of these substances can exhibit a significant ecotoxicological risk.[1]

Wherever humans intervene in the complex structure of ecosystems, appropriate surveillance is indispensable to discover detrimental effects at an early stage. Therefore, monitoring of chemical pollutants, especially pesticides, has become an important practice. Numerous companies and institutions worldwide are focusing their research on pollutant related topics including regulatory enforcement, risk assessment, method development, and residue analysis.[2,3] In view of the enormous variety of employed pesticides, multiresidue methods are the most common and unquestionably the most efficient approaches as they facilitate the detection of large numbers of pesticides in a single run.[4,5] Considering this trend towards multi class analysis, the task of analyzing a high amount of target compounds with a wide range in physico-chemical properties at the same time becomes extremely challenging.[6,7] In the last two decades, the QuEChERS approach has emerged as a powerful method for multiresidue analysis of pesticides. It was first introduced in 2003 by Anastassiades *et al.*[8] and enables the simultaneous detection of a large variety of multi class pesticides. The method requires only few steps and uses a minimized amount of solvents and glassware. These advantages paved the way for the implementation of a more sustainable and faster sample preparation. The sample preparation method is based on a first step of salting-out assisted liquid–liquid extraction (SALLE), followed by a clean-up step using dispersive solid-phase extraction (dSPE). The QuEChERS concept has been applied successfully worldwide due to its several advantages, all of which are already indicated in the method's name: quick, easy, cheap, effective, rugged and safe. The method can easily be adjusted to various matrices and

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target analytes through modifications of *e.g.* the extraction solvent, sample amount, sample pH, type and amount of salts utilized for phase separation.[9,10] The flexibility of the concept has resulted in numerous modifications which are now rapidly developing beyond its original scope of application. A large variety of comestible goods *e.g.* vegetables,[9] rice[11] and Chinese herbs[12] have been investigated in view of their pesticide exposure based on a modified QuEChERS approach. Besides the analysis of foods, the investigation of environmental matrices like soil[13] and water[14] has been the focus in various studies. Also, the suitability of a QuEChERS-derived technique for medicinal purposes such as the detection of pharmaceuticals in blood samples has been demonstrated.[15]

While plenty of validated methods are available for food and water resources[8,14,16] currently, no tailored method is available to carry out accurate multi pesticide residue analysis on vegetation samples of forests, even though forests represent an extremely complex ecosystem where the use of pesticides has a deep impact. Forests are highly diverse ecosystems harboring the majority of the global terrestrial species. Diverse communities of animals and microorganisms are associated with forest trees, forming complex interaction networks. Direct exposure due to the protection of trees against pests (*e.g.* bark beetles)[17] and indirect exposure due to atmospheric drift or drainage of pesticides during application[18] make their occurrence in woodlands inevitable. Increasing evidence suggests that the indirect effects of pesticides are more common and complex than their direct effects.[19] Declines of insect populations, supposedly caused by a class of insecticides called neonicotinoids, is of particular concern.[20] However, until now, the effects of pesticides on the taxonomic and functional diversity in forest ecosystems remain elusive and are poorly understood.[21]

This study focuses on the development of a suitable QuEChERS-based gas chromatography-tandem mass spectrometry (GC-MS/MS) method which allows the simultaneous detection of 208 pesticide and pollutant residues in leaves (*e.g. Fagus sylvatica*) and needles (*e.g. Picea abies*), aiming at the improvement of analytical techniques for multi pesticide residues in vegetation matrices of forest ecosystems. The developed methodology was validated for 61 pesticides in line with the SANTE/12682/2019 guideline.[22]

2 Experimental

2.1 Chemicals

Pesticide standards and triphenyl phosphate (TPP, internal standard) were obtained from HPC Standards GmbH (Cunnersdorf, Germany). The purities of the standard pesticides were ≥98%. Acetic acid (HOAc, ≥99.9%) was obtained from VWR International GmbH (Darmstadt, Germany). Acetonitrile HPLC grade (MeCN, ≥99.9%) was purchased from Fisher Scientific GmbH (Schwerten, Germany). The primary secondary amine (PSA) and graphitized carbon black (GCB) sorbents were purchased from Agilent Technologies, Inc. (Santa Clara, CA, USA). Throughout the study, ultrapure water prepared by an in-house purification system was used. Analytical-grade sodium acetate (99%) and anhydrous magnesium sulfate ($MgSO_4$, 99%) were obtained from Grüssing GmbH (Filsum, Germany). The salts sodium chloride (NaCl, p.a.) and trisodium citrate dihydrate (≥99.0%) were purchased from Bernd Kraft GmbH (Duisburg, Germany) and Chem-Solutions GmbH (Renningen, Germany). Disodium hydrogencitrate sesquihydrate (99%), 3-ethoxy-1,2-propanediol (98%), L-gulonic acid γ-lactone (95%), and D-sorbitol (99%) were purchased from Sigma-Aldrich GmbH (Darmstadt, Germany). Shikimic acid (≥98%) was obtained from Carl Roth GmbH (Karlsruhe, Germany).

2.2 Preparation of starting materials

Blank samples. For method development, beech leaves and spruce needles were collected from a rural area 80 km east of Munich, Germany (N48.290306°, E12.581893°), in October 2019. The leaves were prechopped and homogenized with an Ultra Turrax at 13 000 rpm for 5 min while stored under liquid nitrogen in a Dewar vessel. The sample stocks were stored at -80 °C after full evaporation of the liquid nitrogen. Spruce needles were prepared accordingly. The water content for either matrix was determined at approximately 55% by using loss on drying[23] and Karl Fischer titration.[24] A sieve analysis was conducted to analyse the particle size distribution of both sample stocks. Particles derived from beech leaves were mainly distributed (40%) within the size range 500–800 µm. The majority of needle particles (70%) ranged between 800–1040 µm. The blank sample matrix was analysed using the newly developed method to determine carry-over effects. None of the pesticides were present in the blank sample matrix.

Stock solutions. Stock solutions of pesticide standards and the internal standard triphenyl phosphate (TPP) were prepared in a concentration of 1.00 mg mL^{-1} in MeCN and stored in an amber glass vial at -20 °C. A pesticide working mixture (standard solution) containing each pesticide standard in a concentration of 0.01 mg mL^{-1} was prepared in a separate vial. The stock standard solutions were tempered at room temperature for 1 h and shaken well before use. A working solution containing TPP only (0.01 mg mL^{-1}) was prepared accordingly. A final concentration of 10 ng mL^{-1} (corresponding to 0.05 mg kg^{-1}) was applied as internal standard. The analyte protectant (AP) mixture was prepared with 3-ethoxy-1,2-propanediol (200 mg mL^{-1}), L-gulonic acid γ-lactone (10 mg mL^{-1}), shikimic acid (5 mg mL^{-1}) and D-sorbitol (5 mg mL^{-1}) in a mixture of MeCN and water 6 : 4 (v/v). The AP mixture was stored at 8 °C.

Buffer salt and dSPE mixture. The buffer salt was prepared by weighing $MgSO_4$, NaCl, trisodium citrate dihydrate and disodium hydrogencitrate sesquihydrate in a ratio of 8 : 2 : 2 : 1. A portion of 6.5 g (±0.2 g) of the salt mixture was used as salting out agent. The dSPE mixture was prepared by weighing $MgSO_4$, PSA and GCB (300 : 50 : 15). A portion of 182.5 mg (±1.0 mg) of the dSPE mixture was used for the clean- up step.

2.3 Instrumentation and apparatus

Sample comminution. An Ultra Turrax T25 basic from IKA Labortechnik (Staufen, Germany) was used for homogenization of leaf and needle blank samples. Samples were shaken with the help of the Vortex-Genie 2 manufactured by Scientific Industries, Inc. (Bohemia, NY, USA). A Megafuge 1.0 R from Heraeus Instruments (Hanau, Germany) was used for centrifugation of 50 mL tubes. Centrifugation of 2 mL microcentrifuge tubes was

conducted using an Eppendorf 5415 D centrifuge (Hamburg, Germany).

Gas chromatography-tandem mass spectrometry (GC-MS/MS). Sample analyses were performed using an Agilent Technologies 7890B gas chromatograph (Santa Clara, CA, USA), equipped with an Agilent Technologies 7010B triple quadrupole mass spectrometer with a high efficiency source (HES). Two Agilent J&W HP-5MS ultra inert capillary columns à 15 m × 250 µm × 0.25 µm coupled to a backflush capillary flow technology device (CFT) were used. The injector comprised an Agilent Technologies multimode inlet (MMI). The injection of 1 µL sample volume was accomplished by using a PAL3 RSI autosampler from CTC Analytics (Zwingen, Switzerland). The conditions of the autosampler and the GC-MS/MS system are summarized in Table 1.

The triple quadrupole was operated in dynamic multiple reaction monitoring (dMRM) mode with a total run time of 20.75 min and a solvent delay of 4.00 min. Post run time with backflush was set to 5.00 min. The mass transitions for quantifier and qualifier ions of the analyzed compounds are listed in ESI Table 1.†

2.4 Final sample preparation

For sample preparation, 2.00 g (±0.02 g) of homogenized sample were weighed into a 50 mL centrifuge tube and were spiked with adequate concentrations of standard solution (0.0025–1.0 mg kg^{-1}) and 10 µL of TPP working solution (0.01 mg mL^{-1}). After 10 min, ultrapure water (10.0 mL) and MeCN (10.0 mL) were added and the sample was vortexed for 1 min and allowed to stand for 15 min. Afterwards, the sample was again vortexed for 1 min. The premixed buffer salt (6.5 g ± 0.2 g) was added to the sample mixture and the centrifuge tube was immediately shaken vigorously for 30 s. The sample was centrifuged for 5 min at 3300*g* at room temperature. For sample clean-up, 1 mL of the organic upper layer was transferred into a 2 mL microcentrifuge tube containing the dSPE mixture (182.5 mg ± 1.0 mg). After shaking vigorously for 30 s, the microcentrifuge tube was centrifuged for 5 min at 12 000*g* at room temperature. Afterwards, 500 µL of the supernatant were transferred into a GC vial and 15 µL of the AP mixture were added. Subsequently, the sample was analyzed with GC-MS/MS.

Table 1 Setting of the GC-MS/MS system

GC-MS parameters	
Carrier gas	Helium 5.0 (Air Liquide, Düsseldorf, Germany)
Flow rate	1.1 mL min^{-1} (1st column) 1.3 mL min^{-1} (2nd column)
Back flush	5 min -4.0 mL min^{-1} (1st column) 4.4 mL min^{-1} (2nd column)
Ion source	EI, 70 eV
Split ratio	Splitless
Oven temperature	60 °C (1 min hold) 60 to 170 °C at 40 °C min^{-1} 170 to 310 °C at 10 °C min^{-1} (3 min hold)
Transfer line temperature	280 °C
Inlet temperature	Solvent vent mode 60 °C (0.2 min hold) 60 to 280 °C at 900 °C min^{-1} (20.75 min hold) 280 to 310 °C (during post run)
Vent flow	100 mL min^{-1}
Ion source temperature	230 °C
Collision gas	Argon 4.5 (Air Liquide, Düsseldorf, Germany)
Quadrupole temperature	150 °C

2.5 Method validation

Validation of the developed method was conducted for 61 pesticides (Table 4) in line with the SANTE/12682/2019 guideline.[22] In this study, the parameters limit of quantification (LOQ), recovery, method precision, system precision, and matrix effects were considered.

3 Results and discussion

3.1 Method optimization

Currently, no standard procedure for the quantification of pesticides in vegetation matrices such as tree leaves and needles is available. Aiming at the development of a powerful analytical technique to address this issue, the EN 15662 [25] guideline was used as a template. This European standard procedure describes a multiresidue method for the determination of pesticide residues with GC- and LC-based analytical techniques for foods of plant origin. Sample preparation was performed with acetonitrile extraction/partitioning, followed by sample clean-up using dispersive SPE (dSPE).

Method optimization was implemented in chronological order, beginning with the evaluation of extraction solvent ratio and salt compositions, followed by dSPE mixtures. Finally, matrix effects and the influence of analyte protectants were investigated. Within method optimization, 20 representative pesticides out of the 61 pesticides used for validation (Table 4, see footnotes), comprising a broad spectrum of functional groups, retention times and polarity, were examined at a concentration level of 0.05 mg kg^{-1}.

Amount of water and extraction solvent. Key characteristics of the extraction solvent are high solubility of target compounds and a good permeability into the matrix. Especially for solid samples, the full penetration of the matrix is of high importance.[26,27] Consistent with the EN 15662 standard protocol, MeCN and water were chosen as liquid–liquid extraction solvents and the ideal ratio of matrix : MeCN : H2O was investigated. Based on the determined water content of approximately 55%, three mixtures were examined (Table 2). The amount of buffer salt containing $MgSO_4$, NaCl, trisodium citrate dihydrate and disodium hydrogencitrate sesquihydrate (8 : 2 : 2 : 1) was adapted to the amount of water in accordance with EN 15662.

Two parameters were brought into the evaluation of the different approaches: the average peak area sums of all compounds of interest, and the standard deviation of those

Table 2 Extraction solvent ratio based on the modules E5, E6 and E7 of EN 15662

EN 15662 Module	Matrix [g]	H_2O [mL]	MeCN [mL]	Salt amount [g]
E5	2.00	4.0	4.0	2.6
E6	2.00	2.4	4.0	1.3
E7	2.00	10.0	10.0	6.5

Table 3 Composition of the dSPE mixtures

Composition no	$MgSO_4$ [mg]	PSA [mg]	GCB [mg]
1	150	25	—
2	150	50	—
3	150	25	7.5
4	150	25	25
5	150	50	25
6	150	50	50

sums. Experiments were performed in triplicates. The best results were obtained by using 10.0 mL MeCN and 10.0 mL water as extraction solvents. Hence, this approach was chosen for liquid–liquid extraction.

Salt composition. The addition of salt represents the second extraction step, combining extraction and liquid–liquid partitioning.[6] The EN 15662[25] advises the user to add 4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate for approximately 10 mL water content. The second commonly applied official method, the AOAC Official Method 2007.01,[28] comprises the addition of a salt mixture containing 1 g NaOAc instead of NaCl and the utilization of 1% HOAc in MeCN (v/v) as organic extraction solvent mixture, creating an acetate buffer system. In the original QuEChERS method, Anastassiades *et al*. (2003)[8] suggest an unbuffered system using $MgSO_4$ and NaCl (4 : 1) as partitioning agent. These three different salt compositions were evaluated with regard to resulting peak areas and standard deviations of the measurements. Both plant matrices were investigated separately. The standard deviation was clearly lowest when using the salt mixture described in the EN method for either matrix. Also, peak areas of the beech leaf samples were the highest in this approach. The highest outcome for spruce needle samples was achieved by using the AOAC approach. However, the obtained results were not significantly higher (*t*- test, $p < 0.05$) when compared to the results of the EN 15662 approach. In the following, the salt mixture according to EN 15662 (4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate) was therefore used as partitioning agent.

Dispersive solid-phase extraction (dSPE). To select the most suitable dSPE sorbents and their appropriate amount for the clean-up step, samples were spiked with pesticide standards (20 out of the 61 representative pesticides (Table 4, see footnotes)) at 0.05, 0.25 or 0.5 mg kg^{-1} prior to extraction. Each experiment was performed in triplicates and GC-MS/MS analyses were evaluated based on the above-mentioned criteria (summed peak areas and relative standard deviations). As experiments were carried out at three different concentration levels, the mean value of the triplicates was averaged across all three concentration levels. Six commonly applied adsorbent mixtures were used (Table 3).[29]

The choice of a suitable dSPE mixture must be based on a multi criteria decision, compromising between the minimal loss of analytes and the maximal removal of matrix components. Consequently, the optimal clean-up procedure of leaf and needle samples was determined to be a 150 mg $MgSO_4$, 25 mg PSA and 7.5 mg GCB dSPE mixture for 1 mL of raw extract.

Matrix effects and analyte protectants. During method development, special attention should be paid to the influence of matrix effects as they are known to occur frequently in both GC and LC analysis.[12,30] Coelution of compounds arising from the matrix with target compounds can result in an influenced ionization efficiency, leading to signal enhancement or suppression.[30] Within this study, matrix effects were evaluated based on the comparison of the peak areas in solvent (MeCN) and matrix-matched solutions, each with a concentration of 50 ng mL^{-1} (corresponding to 0.25 mg kg^{-1}), respectively. A tenfold measurement of analytes (Table 4, see footnotes) in MeCN, leaf or needle matrix with and without the addition of analyte protectants (AP) was conducted. The matrix effect was calculated as follows:

$$((area_{matrix} - area_{solvent}) \div area_{solvent}) \times 100\%$$

Bar charts of matrix effects display an overall high impact of the tested matrices on signal intensities (Fig. 1A).

In needle extract, 96% of analytes revealed a very high matrix effect (>70%). Peak areas measured in beech leaf matrix were not as strongly affected as in needle extract. Nevertheless, also in leaf matrix, only 24% of target compounds showed a weak matrix effect (≤20%).

The addition of analyte protectants to both sample matrices also resulted in strongly differing peak areas when matrix measurements are compared to analytes detected in solvent (Fig. 1B). All pesticide standards revealed a moderate to very high matrix effect in beech leaf extracts. Consequently, results got even worse by adding the supplement. For spruce needle matrix, 92% of analytes showed a moderate to very high matrix effect, slightly lowering the beforehand seen influence of the matrix on analyzed signals. According to the SANTE/12682/2019 guideline,[22] an increase or decrease of peak areas above 20% must be addressed in method development by the utilization of matrix-matched calibrants or the addition of analyte protectants. As the addition of analyte protection did not equalize the matrix influence, a matrix-matched calibration was inevitable for the analysis of multi pesticide residues in leaf and needle matrices.

Two different approaches are commonly applied to compensate matrix effects in GC-analyses: matrix-matched calibration and the use of analyte protectants (AP).[12, 31] Interaction of analytes with active sites such as silanol groups and metal ions present in the liner and column would result in

Table 4 Calibration range, coefficient of determination (R^2) and recovery of the analyzed target compounds

Compound Name	Quantifier transition	Calibration range [mg kg^{-1}]	R^2	Recovery [%]
Acetamiprid	126.0 → 72.9	0.01–1.0	0.997	88.5
Ametoctradin[a,b]	246.0 → 188.2	0.05–1.0	0.998	82.7
Amisulbrom[a]	225.9 → 147.0	0.0025–1.0	0.997	86.8
Azoxystrobin[a,b]	344.1 → 171.9	0.005–1.0	0.998	89.3
Bifenazate	196.0 → 115.1	0.05–1.0	0.994	86.6
Bifenthrin[a]	181.0 → 115.1	0.05–1.0	0.993	76.3
Boscalid[b]	140.0 → 76.0	0.0025–1.0	0.997	86.1
Carbetamide	119.1 → 64.1	0.01–1.0	0.997	92.0
Chlordane (*cis*)	372.8 → 265.8	0.0025–1.0	0.999	77.0
Chlordane (*trans*)[b]	372.8 → 265.8	0.0025–1.0	0.999	78.3
Chlorpyrifos[a]	313.8 → 257.8	0.005–1.0	0.999	83.7
Chlorothalonil	265.9 → 109.0	0.005–1.0	0.997	80.5
Cyflufenamid	188.1 → 88.0	0.01–1.0	0.997	88.3
Cyhalothrin (lambda[a] and gamma)[b]	208.0 → 181.0	0.005–1.0	0.998	84.2
Cypermethrin (2 isomers)[b]	163.0 → 127.0	0.05–1.0	0.992	75.9
p,p′-DDE[a]	246.1 → 176.2	0.005–1.0	0.993	71.7
p,p′-DDT	235.0 → 165.2	0.005–1.0	0.997	74.2
Deltamethrin (2 isomers)[a]	251.0 → 172.0	0.0025–1.0	0.997	82.4
Dieldrin[b]	262.9 → 193.0	0.01–1.0	0.997	81.4
Difenconazole (2 isomers)[b]	322.8 → 264.8	0.0025–1.0	0.994	89.0
Diflubenzuron	141.0 → 63.0	0.01–1.0	0.997	86.9
Dimethomorph[b]	300.9 → 165.0	0.005–1.0	0.996	86.5
Epoxiconazole	192.0 → 138.1	0.005–1.0	0.990	87.8
Fenazaquin	145.0 → 117.1	0.01–1.0	0.991	78.4
Fenhexamid[a,b]	177.1 → 113.0	0.005–1.0	0.998	88.1
Fenpropidin	273.0 → 98.0	0.005–1.0	0.998	87.8
Fenpropimorph[b]	128.1 → 70.1	0.005–1.0	0.998	85.7
Fenvalerate (2 isomers)[b]	167.0 → 125.1	0.05–1.0	0.992	81.4
Fipronil sulfone[b]	382.8 → 254.9	0.0025–1.0	0.992	90.2
Fluazifop-*P*-butyl	281.9 → 91.0	0.0025–1.0	0.998	91.7
Fludioxonil[a,b]	248.0 → 127.1	0.01–1.0	0.994	87.2
Fluopyram	222.9 → 196.0	0.005–1.0	0.999	89.9
Flupyradifurone[a]	126.0 → 73.0	0.01–1.0	0.997	85.9
tau-Fluvalinate (2 isomers)[a]	250.0 → 200.1	0.0025–1.0	0.998	82.1
Folpet	259.8 → 130.1	0.01–1.0	0.996	79.7
gamma-HCH (Lindane)[a,b]	216.9 → 181.0	0.0025–1.0	0.998	86.8
Imidacloprid	126.0 → 73.0	0.05–1.0	0.996	85.9
Isoxaben	165.0 → 107.0	0.05–1.0	0.989	90.0
Lenacil	153.1 → 136.1	0.01–1.0	0.996	86.1
Metamitron	104.1 → 51.0	0.05–1.0	0.991	90.4
Metazachlor[a,b]	209.0 → 132.2	0.0025–1.0	0.999	91.6
Metrafenone	394.8 → 364.8	0.0025–1.0	0.999	86.8
Myclobutanil[a,b]	179.0 → 125.1	0.0025–1.0	0.992	88.8
Pentachloronitrobenzene[a,b]	248.8 → 213.8	0.005–1.0	0.999	78.2
Permethrin (*cis* and *trans*)[b]	163.0 → 127.0	0.005–1.0	0.990	75.7
Picolinafen[a,b]	376.0 → 238.1	0.0025–1.0	0.999	86.2
Pirimicarb	238.0 → 166.2	0.005–1.0	0.999	84.0
Propiconazole (2 isomers)[b]	172.9 → 74.0	0.005–1.0	0.997	84.4
Propyzamide	173.0 → 74.0	0.0025–1.0	0.997	87.9
Pyrimethanil	198.0 → 118.1	0.01–1.0	0.997	86.1
Quinoxyfen[a]	273.0 → 208.1	0.0025–1.0	0.999	76.1
Spirodiclofen	312.1 → 108.9	0.005–1.0	0.998	83.2
Tebuconazole[a,b]	250.0 → 125.0	0.005–1.0	0.999	86.7
Terbuthylazine	214.0 → 71.0	0.0025–1.0	0.999	86.5
Terbuthylazine-desethyl	186.2 → 83.1	0.005–1.0	0.998	87.6
Tetraconazole[b]	336.0 → 203.8	0.0025–1.0	0.999	90.5
Thiabendazole	201.9 → 175.0	0.05–1.0	0.995	69.7
Thiacloprid	126.0 → 73.0	0.05–1.0	0.991	82.3
Tolclofos-methyl[a,b]	267.0 → 252.0	0.01–1.0	0.998	86.4
Warfarin	265.0 → 121.0	0.05–1.0	0.991	91.9
Zoxamide[b]	187.0 → 123.0	0.005–1.0	0.997	90.8

[a] Pesticide standards used for optimizing extraction solvents, partitioning agent and dSPE mixture. [b] Pesticide standards used for the evaluation of matrix effects and AP experiments.

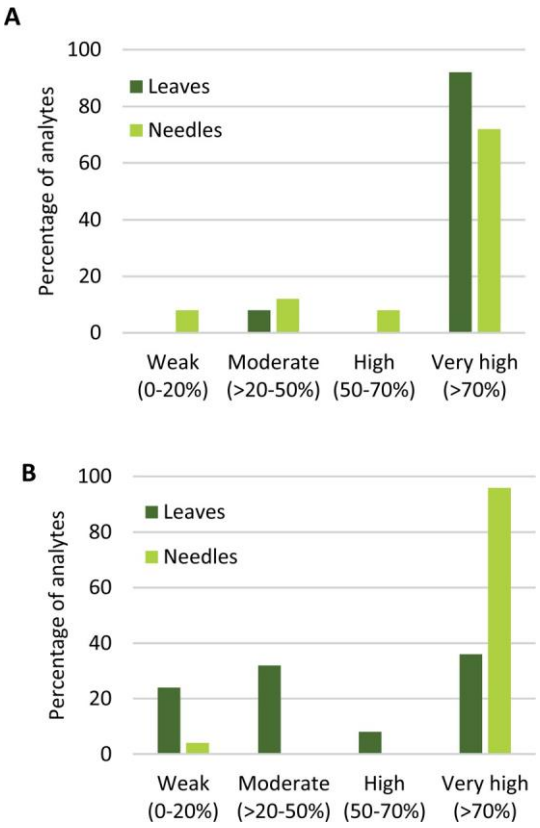

Fig. 1 Matrix induced enhancement or suppression effects of leaf and needle extracts compared to solvent (MeCN). Diagrams depict the results, calculated based on total peak areas, for solvent and plant extracts without analyte protectants (A) and both with analyte protectants (B).

analyte losses and distorted peak shapes.[12] Analyte protectants such as D-sorbitol, L-gulonic acid-γ-lactone and 3-ethoxy-1,2-propanediol competitively cover these active sites, ideally leading to an equalized response of target compounds in solvent calibrants and sample extracts.[12, 25] Matrix-matched calibration was determined to be compulsory based on the beforehand described results (see Matrix effects). Hence, it was investigated whether a simultaneous implementation of both approaches would be beneficial to mitigate matrix effects in the best possible way. The advantages of using analyte protectants in quantitative analysis can include improvement of the shape and intensity of chromatographic peaks,[12,31] which was also evidenced by the obtained data. Furthermore, results revealed a considerably higher response for target compounds measured in needle matrix compared to solvent and leaf matrix (Fig. 2). While the additive doubled the sum of total peak areas in MeCN, the effect was even stronger in leaf matrix. Here, the addition of AP attained a tenfold increase of responses. Consequently, lower detection limits can be achieved. When taking a closer look at the data of analytes in needle extracts, the levelling effect arising from the supplement became visible. Summed peak areas were decreased by the addition of the AP mixture. As a result, the curves of the peak areas of analytes detected in needle and leaf extracts showed a close resemblance. For this reason, the addition of analyte protectants in combination with a matrix-matched calibration was considered as the most accurate determination of target analytes in leaves and needles.

3.2 Method validation

Prior to validation, a pre-screening of leaf and needle samples was conducted using the parameters described in ESI Table 1.† Based on this experiment, 61 pesticide residues (Table 4) were suspected to be present. In the following, this group of pesticides was subjected to validation, which was performed according to SANTE/12682/2019.[22]

Using the optimized methodology, analysis was conducted to determine the linear range for the 61 pesticides. Linearity was proven within the range of 0.0025, 0.005, 0.01, 0.05 and 1.0 mg kg^{-1} depending on the analyte, for all pesticides with an $R^2 \geq 0.990$ using the back-calculated concentrations. Within this

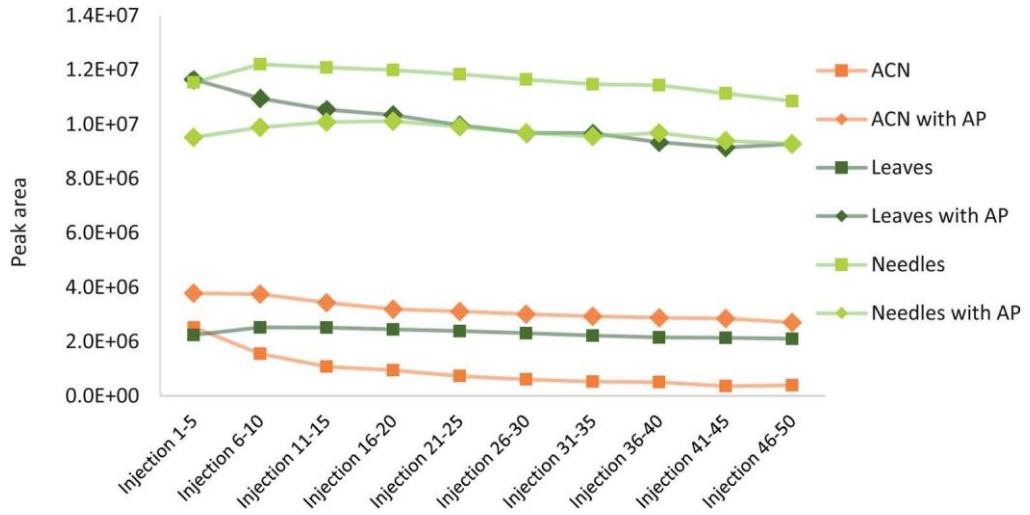

Fig. 2 Impact of analyte protectants on summed up peak areas of analyzed target compounds (50 ng mL^{-1}) in MeCN, beech leaf and spruce needle extract.

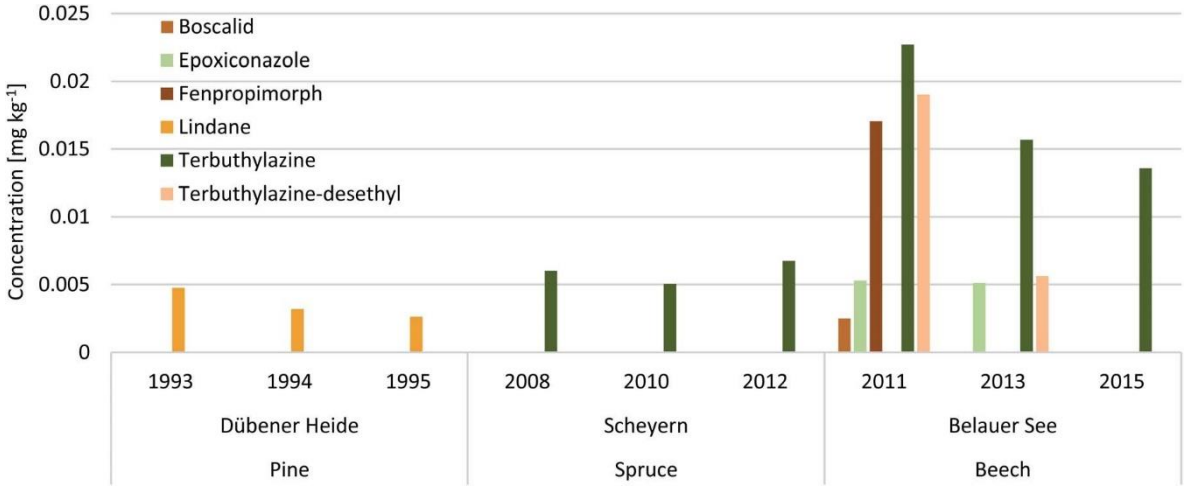

Fig. 3 Results of analyzed forest samples from beech, spruce and pine trees. Samples were collected from the German Federal Environment Agency from three different sites in Germany (Dübener Heide, Scheyern, and Belauer See) over the last 30 years.

study, recoveries for pesticide standards were determined at each level of the calibration curve (n = 5 for LOQ, 0.05 and 0.1 mg kg^{-1}; n = 3 for 0.25, 0.5 and 1.0 mg kg^{-1}). In order to determine a representative value for the recovery across all concentration levels, the actual concentration was plotted against the nominal concentration. The slope of the linear equation reflects the average recovery of all concentration levels. Recovery of all analytes was shown to lie within the accepted range of 70–120%, reaching from 69.7% (thiabendazole) to 92.0% (carbetamide), with a relative standard deviation (RSD) below 20%. Results are summarized in Table 4. For evaluation of the method precision, five samples were spiked in three concentration levels, respectively: individual LOQ of each compound, 0.05 mg kg^{-1} or 0.1 mg kg^{-1}, prior to extraction.

The method precision must not exceed 20%. For the lowest concentration, the average method precision was 14.2%, for the middle concentration it was 8.4%, and for the highest concentration, the average method precision was 9.7%. System precision was evaluated based on multiple measurements (n = 5) from the same sample, spiked with the individual LOQ, 0.05 and 0.1 mg kg^{-1}, respectively. For the individual LOQ, the average system precision was 7.3%. For the middle concentration level, a system precision of 4.2% was obtained, and the highest concentration level delivered a system precision of 4.1%.

3.3 Method application

Following the completion of method validation, the method was applied to nine leaf and needle samples. These samples were collected by the German Environmental Specimen Bank of the German Federal Environment Agency in different sites throughout Germany.[32] The samples were stored at -80 °C before use. Detected pesticide residues were quantified by interpolation against external calibration curves obtained using procedural standards[22] (calibration range: 0.0025–1.0 mg kg^{-1}). The use of procedural standards allows to compensate for matrix effects and mitigates measuring errors caused by low extraction recoveries.[22] Procedural standards were prepared by spiking blank samples prior to extraction with varying amounts of analyte standard solution according to the calibration curve set-up. The standards need to be processed and measured in a bracketing manner within the same batch as the samples to be analyzed. The response factor of bracketing calibrants at each concentration level must not differ by more than 30% and deviation of back-calculated concentrations should not exceed 20%.[22]

The analysis of leaf and needle samples revealed the presence of six pesticides (boscalid, epoxiconazole, fenpropimorph, lindane, terbuthylazine, terbuthylazine-desethyl), providing a proof of concept for the developed methodology (Fig. 3). The herbicide terbuthylazine and its metabolite terbuthylazine-desethyl were detected in the highest concentrations. The herbicide terbuthylazine is becoming one of the most commonly employed pesticides in most EU countries, being mainly used in maize cultivation as a substitute for atrazine.[33] Both compounds are chemicals of arising concern, due to their persistence and toxicity towards aquatic organisms.[34] Also, they are known to have significant endocrine disruption capacity to wildlife and humans.[33,35] Hence, the outcome of this analysis again highlights the need for adequate surveillance of pesticide residues in our environment to discover detrimental effects at an early stage. Beside the effect of single compounds, the presence of a mixture of pesticides, detected in the sample from Belauer See (2011), is a concerning result.

The chromatogram of this sample is shown in ESI Fig. 1.† The sampling site Belauer See is a rural area in northern Germany surrounded by arable land, supporting the plausibility of the obtained results. The environmental effects of such mixtures often remain elusive and therefore require an increased monitoring.

Conclusions

Within this study, a modified QuEChERS-based GC-MS/MS approach, capturing 208 pesticide and pollutant residues, was developed for the novel purpose of analyzing leaves (*e.g.* from *Fagus sylvatica*) and spruce needles (*Picea abies*). Validation was conducted according to SANTE/12682/2019 in terms of linearity, LOQ, recovery, system precision and method precision. High matrix effects were successfully addressed by using matrix- matched calibrants in combination with analyte protectant addition to sample extracts. Within the validation process, a linear range with adequate accuracy and precision for all investigated pesticides was determined. The applied sample preparation resulted in satisfactory recoveries and a high precision, indicating the reliability of this routine analysis of multi pesticide residues in the investigated matrices. This QuEChERS approach will allow for a rapid, routine and parallel monitoring of large numbers of pesticides in environmental samples. This will contribute to a better understanding of the environmental impact of simultaneously present pesticides in forest ecosystems. In parallel to the pesticide monitoring, we are currently exploring a large dataset on leaf associated arthropod communities. This will allow us to draw direct conclusions on factors associating with taxonomic declines. This approach provides a specifically qualified and propulsive analytical method, paving the way for the assessment of risks related to the occurrence of organic pollutants by elevating the analytical technique for pesticide multi residues in vegetation matrices of forests. A proof of concept for the developed methodology was provided by the analysis of nine leaf and needle samples, which revealed the occurrence of six pesticide residues. The obtained results underline the need to regularly conduct systematic surveillance and monitor pollutant residues in order to keep chemical pollution within safe levels.

Author contributions

Conceptualization, SS, CM; methodology, AL, SS, CM; validation, AL; formal analysis, SS, CM; investigation, AL; resources, HK, FB; data curation; AL, writing – original draft, AL; writing - review and editing, SS, HK, FB, CM; visualization, AL; supervision, SS, CM; funding acquisition, HK, FB.

Conflicts of interest

There are no conflicts to declare.

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5.4. Supplementary material

Supplementary Table 1. Pesticide classification, retention time (RT), dynamic multiple reaction monitoring (dMRM) transitions and collision energy (CE) of analyzed target compounds. Quantifier transitions are marked in bold.

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
2,4-D-ethyl ester	Herbicide	7.49	**247.9 → 185.0**	10
			185.0 → 114.9	25
			175.0 → 111.0	10
2-Phenylphenol	Microbiocide	6.25	169.1 → 91.0	35
			141.1 → 63.0	45
			115.1 → 65.0	25
8-Hydroxyquinoline	Fungicide, Microbiocide	5.38	145.0 → 63.0	40
			117.0 → 63.0	40
			117.0 → 39.1	40
Acequinocyl	Insecticide	16.77	342.9 → 188.8	20
			341.9 → 187.9	15
			187.9 → 131.0	20
Acetamiprid	Insecticide	13.85	221.0 → 56.1	15
			126.0 → 90.0	5
			126.0 → 72.9	20
Acibenzolar-S-methyl	Fungicide	9.30	182.0 → 167.1	10
			182.0 → 153.1	10
			182.0 → 135.0	15
Aclonifen	Herbicide	12.39	264.1 → 194.2	15
			194.1 → 167.1	20
			194.1 → 139.1	25
Acrinathrin	Insecticide	15.02	**288.9 → 92.8**	10
			207.8 → 152.0	35
			181.0 → 127.0	30
Aldrin	Insecticide	9.94	**262.9 → 192.9**	35
			262.9 → 190.9	35
			254.9 → 220.0	20
Ametoctradin	Fungicide	15.20	275.0 → 246.2	0
			275.0 → 190.3	15
			246.0 → 188.2	25
Amisulbrom	Fungicide	16.14	227.9 → 147.0	15
			225.9 → 147.0	15
			214.0 → 160.0	20
Azoxystrobin	Fungicide	18.30	344.1 → 182.9	25
			344.1 → 171.9	40
			344.1 → 155.8	40
Beflubutamid	Herbicide	10.67	192.9 → 145.1	15
			192.9 → 95.0	35
			176.1 → 79.1	25
Benalaxyl	Fungicide	12.87	266.0 → 148.1	5
			233.9 → 146.0	20
			206.0 → 162.1	5
Bentazone	Herbicide	10.11	**225.0 → 181.9**	5
			198.0 → 92.0	30
			182.0 → 90.0	15
Benthiavalicarb-isopropyl	Fungicide	14.57	222.0 → 125.9	40
			180.0 → 127.0	20
			180.0 → 83.0	30
Bifenazate	Insecticide	13.94	258.0 → 170.1	20
			199.0 → 77.0	40
			196.0 → 115.1	5

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Bifenox	Herbicide	14.21	340.9 → 309.9 **340.9 → 280.9** 189.1 → 126.0	10 15 20
Bifenthrin	Insecticide	13.83	**181.0 → 115.1** 166.0 → 139.1 166.0 → 115.1	45 35 35
Boscalid	Fungicide	16.50	140.0 → 112.0 **140.0 → 76.0** 111.9 → 76.0	10 25 15
Bromoxynil	Herbicide	7.41	276.8 → 88.0 **274.7 → 167.9** 274.7 → 88.0	30 15 30
Bromuconazole (2 isomers)	Fungicide	13.85 14.29	295.0 → 172.9 **293.0 → 172.9** 173.0 → 109.0	10 10 30
Bupirimate	Fungicide	11.80	**315.8 → 207.9** 208.0 → 68.9 193.0 → 109.0	10 30 15
Buprofezin	Insecticide	11.74	**304.9 → 175.0** 249.1 → 193.0 171.1 → 115.0	10 10 10
Captan	Fungicide	10.73	**263.8 → 79.0** 149.0 → 70.0 116.9 → 82.0	15 15 30
Carbetamide	Herbicide	9.95	120.1 → 77.0 **119.1→ 64.1** 91.0 → 64.1	25 15 10
Carboxin	Fungicide	11.75	**234.9 → 143.0** 234.9 → 87.0 131.9 → 77.0	10 20 20
Carfentrazone-ethyl	Herbicide	12.81	**339.9 → 311.9** 329.9 → 309.9 311.9 → 150.8	10 10 20
cis-Chlordane	Insecticide	11.20	374.8 → 265.8 **372.8 → 265.8** 271.7 → 236.9	15 15 15
trans-Chlordane	Insecticide	10.94	374.8 → 265.8 **372.8 → 265.8** 271.7 → 236.9	15 15 15
Chloridazon (Pyrazon)	Herbicide	13.04	221.0 → 220.2 **220.0 → 193.1** 220.0 → 166.0	5 20 25
Chlorothalonil	Fungicide	8.54	265.9 → 230.9 **265.9 → 133.0** 265.9 → 109.0	20 45 45
Chlorotoluron	Herbicide	9.70	**212.1 → 166.0** 212.1 → 72.0 167.0 → 132.1	10 15 15
Chlorpropham	Herbicide, Plant growth regulator	7.11	**213.0 → 171.1** 171.0 → 127.1 153.0 → 90.0	5 5 25
Chlorpyrifos	Insecticide	9.86	313.8 → 257.8 **196.9 → 107.0** 196.9 → 98.0	15 40 30

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Chlorpyrifos -methyl	Insecticide	9.14	287.9 → 92.9 **285.9 → 93.0** 124.9 → 47.0	20 25 15
Clodinafop-propargyl	Plant growth regulator	12.97	348.9 → 265.9 **348.9 → 237.8** 238.0 → 130.0	10 15 15
Clomazone	Herbicide	7.98	**205.1 → 107.1** 127.0 → 101.0 125.0 → 89.0	20 20 15
Cloquintocet-mexyl	Herbicide safener	14.00	220.0 → 191.9 **163.0 → 128.0** 163.0 → 101.0	10 15 30
Cyflufenamid	Fungicide	11.88	188.1 → 88.0 **118.1 → 90.0** 118.1 → 89.0	35 10 25
Cyfluthrin (3 isomers)	Insecticide	16.17 16.25 16.37	206.0 → 176.9 206.0 → 150.0 **162.9 → 127.0**	25 40 5
Cyhalofop-butyl	Herbicide	14.68	**357.1 → 229.1** 256.2 → 120.1 229.2 → 109.1	15 10 15
Cyhalothrin (*gamma* and *lambda* isomer)	Insecticide	14.79 14.60	**208.0 → 181.0** 208.0 → 152.0 197.0 → 161.1	5 25 5
Cypermethrin (3 isomers)	Insecticide	16.39 16.48 16.57	165.0 → 127.1 165.0 → 91.1 **162.9 → 127.0**	0 10 0

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Cyproconazole	Fungicide	11.99	**222.0 → 124.9** 138.9 → 111.0 138.9 → 75.0	25 15 35
Cyprodinil	Fungicide	10.39	225.2 → 224.3 **224.2 → 131.1** 210.0 → 93.0	10 15 20
Cyromazine	Insecticide	7.97	**165.9 → 109.0** 151.0 → 82.0 109.0 → 68.0	20 30 20
o,p'-DDD	Insecticide, Metabolite	11.78	235.0 → 200.1 235.0 → 139.1 **199.1 → 164.1**	10 45 20
p,p'-DDD	Insecticide, Metabolite	12.36	237.0 → 200.1 **199.1 → 164.1** 165.1 → 139.0	15 20 35
o,p'-DDE	Metabolite	10.98	317.8 → 248.0 248.0 → 176.2 **246.0 → 176.2**	15 30 30
p,p'-DDE	Metabolite	11.52	317.8 → 246.0 315.8 → 246.0 **246.1 → 176.2**	15 15 30
o,p'-DDT	Insecticide	12.27	237.0 → 199.1 **235.0 → 199.1** 199.0 → 163.1	15 15 35
p,p'-DDT	Insecticide	12.94	237.0 → 165.2 235.0 → 199.2 **235.0 → 165.2**	20 15 20

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Deltamethrin	Insecticide	18.02	252.9 → 174.0 **252.9 → 93.1** 251.0 → 172.0	0 15 0
Desmedipham	Herbicide	7.67	**135.0 → 79.0** 122.0 → 94.0 122.0 → 65.0	10 25 10
Diazinon	Insecticide	8.29	276.0 → 137.1 **199.1 → 135.1** 179.1 → 137.1	25 10 20
Dicamba-methyl ester	Herbicide	6.26	234.0 → 173.0 **205.0 → 149.0** 175.0 → 111.0	20 15 20
Diclofop-methyl	Herbicide	13.26	339.9 → 252.9 **280.8 → 119.9** 253.0 → 162.1	10 10 15
Dieldrin	Insecticide	11.62	**262.9 → 193.0** 262.9 → 191.0 247.0 → 241.0	5 35 35
Diethofencarb	Fungicide	9.76	225.0 → 96.0 **207.0 → 179.1** 207.0 → 151.0	30 5 15
Difenoconazole (2 isomers)	Fungicide	17.72 17.78	324.8 → 266.8 **322.8 → 264.8** 264.9 → 202.0	15 15 20
Diflubenzuron	Insecticide	5.00	141.0 → 113.0 **141.0 → 63.0** 113.0 → 63.0	25 25 25

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Diflufenican	Herbicide	13.29	**393.9 → 265.9** 266.0 → 246.1 218.0 → 140.1	10 15 20
Dimethachlor	Herbicide	8.99	209.9 → 134.1 **196.9 → 148.2** 134.1 → 79.1	10 10 20
Dimethenamide-P	Herbicide	9.02	229.9 → 154.0 **229.9 → 111.0** 202.9 → 154.0	10 25 10
Dimethoate	Insecticide	7.79	227.7 → 87.0 **157.0 → 93.0** **157.0** → 63.0	5 10 25
Dimethomorph (2 isomers)	Fungicide	18.35 18.66	302.9 → 164.9 **300.9 → 165.0** 300.9 → 138.8	10 10 15
Dimoxystrobin	Fungicide	13.85	237.0 → 116.0 **205.0 → 116.0** 174.0 → 115.0	15 10 30
Diuron	Herbicide	10.66	231.7 → 71.8 **186.9 → 124.0** 158.9 → 123.9	15 20 10
Dodemorph (2 isomers)	Fungicide	10.24 10.55	**281.0 → 154.0** 238.1 → 55.1 154.0 → 112.1	10 20 10
Epoxiconazole	Fungicide	13.52	**192.0 → 138.1** 192.0 → 111.0 138.0 → 75.0	10 25 25

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Ethofenprox	Insecticide	16.78	183.0 → 168.0 163.0 → 135.1 **163.0 → 107.1**	10 10 20
Ethofumesate	Herbicide	9.61	285.9 → 207.1 **178.9 → 137.1** 178.9 → 105.1	5 0 15
Ethoprophos	Insecticide	7.02	199.9 → 97.0 **157.9 → 97.0** 157.9 → 81.0	20 15 15
Etoxazole	Insecticide	14.07	329.9 → 315.0 299.9 → 284.9 **299.9 → 269.9**	20 10 20
Etridiazole	Fungicide	5.85	211.1 → 183.0 211.1 → 140.0 **185.0 → 142.0**	10 25 15
Famoxadone	Fungicide	18.44	329.9 → 329.0 **329.9 → 223.9** 223.9 → 196.2	10 10 10
Fenamiphos	Insecticide	11.31	302.9 → 287.9 302.9 → 153.9 **287.9 → 259.7**	10 15 5
Fenazaquin	Insecticide	14.05	160.0 → 145.2 146.0 → 118.1 **145.0 → 117.1**	5 10 20
Fenbuconazole	Fungicide	16.21	197.9 → 129.0 197.9 → 102.0 **125.0 → 89.0**	5 30 20

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Fenhexamid	Fungicide	12.97	301.0 → 97.0 179.0 → 115.0 **177.1 → 113.0**	15 15 15
Fenoxaprop-P-ethyl	Herbicide	15.33	360.8 → 287.8 **287.8 → 118.8** 287.8 → 90.9	10 10 20
Fenoxycarb	Insecticide	13.86	256.1 → 187.2 **186.2 → 109.0** 186.2 → 77.1	10 15 20
Fenpropidin	Fungicide	9.45	**273.0 → 98.0** 117.0 → 91.0 98.0 → 55.1	5 25 10
Fenpropimorph	Fungicide	9.81	128.1 → 110.1 128.1 → 86.1 **128.1 → 70.1**	5 10 10
Fenpyroximate	Insecticide	7.85	212.0 → 185.0 **212.0 → 76.9** 198.1 → 114.0	40 40 35
Fenvalerate (2 isomers)	Insecticide	17.31 17.50	419.1 → 166.8 **167.0 → 125.1** 167.0 → 89.0	10 10 40
Fipronil	Insecticide	10.64	**366.8 → 212.8** 350.8 → 254.8 254.9 → 228.0	25 15 15
Fipronil sulfide	Metabolite	10.50	420.0 → 350.9 **351.0 → 254.9** 254.9 → 156.9	10 20 35

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Fipronil sulfone	Metabolite	11.71	384.8 → 256.8 **382.8 → 254.9** 254.9 → 227.9	20 20 15
Fluazifop-P-butyl	Herbicide	11.97	382.9 → 282.0 **281.9 → 238.0** 281.9 → 91.0	10 15 15
Fludioxonil	Fungicide	11.51	248.0 → 182.1 248.0 → 154.1 **248.0 → 127.1**	10 20 30
Flufenacet	Herbicide	9.96	211.0 → 123.0 **211.0 → 96.0** 183.0 → 69.0	5 15 20
Flumetralin	Herbicide	11.19	403.9 → 156.8 359.9 → 313.9 **157.0 → 109.0**	15 15 25
Flumioxazin	Herbicide	17.43	354.0 → 325.9 354.0 → 175.8 **287.0 → 258.7**	5 15 15
Fluometuron	Herbicide	6.98	232.0 → 72.0 **213.0 → 167.9** 187.0 → 109.0	15 10 20
Fluopyram	Fungicide	10.57	395.9 → 223.1 **222.9 → 196.0** 222.9 → 187.1	5 10 10
Fluorochloridone	Herbicide	10.11	311.0 → 174.1 **311.0 → 102.9** 187.1 → 109.1	15 15 20

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Flupyradifurone	Insecticide	14.87	288.0 → 126.1 128.0 → 90.0 **126.0 → 73.0**	15 10 25
Fluquinconazole	Fungicide	15.85	342.0 → 107.8 **340.0 → 298.0** 340.0 → 107.8	40 15 40
Fluroxypyr-meptyl	Herbicide	13.29	237.0 → 209.0 **237.0 → 181.0** 208.9 → 178.9	5 15 20
Flurtamone	Herbicide	14.43	**332.7 → 120.0** 157.0 → 137.1 157.0 → 107.0	15 15 25
Flutolanil	Herbicide	11.38	322.9 → 281.0 **280.9 → 173.0** 173.0 → 95.0	5 10 30
Flutriafol	Fungicide	11.30	**219.1 → 123.1** 219.1 → 95.0 164.1 → 109.1	15 35 20
tau-Fluvalinate (2 isomers)	Insecticide	17.48 17.52	252.0 → 200.0 **250.0 → 200.1** 250.0 → 198.1	15 15 40
Fluxapyroxad	Fungicide	14.57	**321.1 → 152.9** 222.0 → 152.9 222.0 → 125.9	35 15 40
Fosthiazate (2 isomers)	Nematicide	10.27 10.31	**199.0 → 102.0** 195.0 → 60.0 165.9 → 106.0	5 20 10

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Fuberidazole	Fungicide	9.16	184.0 → 155.1 **156.0 → 103.1** 155.0 → 129.1	30 20 10
Haloxyfop-P-methyl	Herbicide	10.93	375.1 → 316.0 **375.1 → 91.1** 288.0 → 180.0	10 35 25
alpha-HCH	Insecticide	7.64	218.9 → 183.0 **216.9 → 181.0** 180.9 → 145.0	5 5 15
beta-HCH	Insecticide	7.99	218.9 → 183.1 **216.9 → 181.1** 181.0 → 145.0	5 5 15
gamma-HCH (Lindane)	Insecticide	8.08	218.9 → 183.1 **216.9 → 181.0** 181.0 → 145.0	5 5 15
delta-HCH	Insecticide	8.51	**217.0 → 181.1** 183.1 → 147.1 181.1 → 145.1	5 15 15
epsilon-HCH	Insecticide	8.69	254.0 → 180.9 218.9 → 182.9 **182.9 → 109.0**	10 5 30
Heptachlor	Insecticide	9.34	273.7 → 238.9 273.7 → 236.9 **271.7 → 236.9**	15 15 15
Heptachlor endo-epoxide	Metabolite	10.67	216.9 → 182.0 216.9 → 109.0 **183.0 → 119.0**	20 45 30
Heptachlor exo-epoxide	Metabolite	10.61	**354.8 → 264.9** 352.8 → 262.9 262.9 → 193.0	15 15 35
Hexachloro-benzene	Fungicide	7.70	**283.8 → 213.9** 281.8 → 211.9 248.9 → 179.0	30 30 30
Imazalil	Fungicide	11.48	216.8 → 175.0 174.9 → 147.0 **172.9 → 109.0**	5 15 30
Imidacloprid	Insecticide	11.31	**211.0 → 113.0** 126.0 → 89.9 126.0 → 73.0	15 5 25
Indoxacarb	Insecticide	18.02	**264.0 → 175.8** 202.9 → 134.0 202.9 → 106.0	15 20 15
Ipconazole	Fungicide	15.00	249.0 → 125.0 167.0 → 125.0 **125.0 → 89.0**	15 5 20
Iprovalicarb (2 isomers)	Fungicide	11.61 11.78	158.0 → 98.0 **143.1 → 93.0** 116.0 → 98.1	10 15 5
Isopyrazam	Fungicide	15.30	359.0 → 159.0 **302.1 → 262.1** 159.0 → 139.0	40 15 10
Isoxaben	Herbicide	15.17	165.0 → 150.0 165.0 → 107.0 **149.9 → 121.9**	15 25 5

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Kresoxim-methyl	Fungicide	11.81	206.0 → 131.1 **206.0 → 116.0** 116.0 → 89.0	10 5 15
Lenacil	Herbicide	12.95	233.9 → 153.1 153.1 → 110.1 **153.1 → 82.1**	5 20 20
Lufenuron	Insecticide	5.58	251.6 → 157.8 **202.9 → 75.9** 173.9 → 109.9	15 40 30
Malathion	Insecticide	9.73	172.9 → 117.0 **172.9 → 99.0** 157.8 → 125.0	15 10 5
MCPA-methyl ester	Herbicide	6.51	214.1 → 155.1 214.1 → 141.1 **155.1 → 125.1**	10 10 10
MCPB-methyl ester	Herbicide	8.17	211.1 → 155.0 142.1 → 107.1 **142.1 → 77.1**	10 10 30
Mefenpyr-diethyl	Herbicide safener	13.59	**299.0 → 252.9** 253.0 → 190.0 253.0 → 189.0	10 20 30
Mepanipyrim	Fungicide	11.16	222.2 → 158.1 221.2 → 220.2 **207.1 → 179.1**	25 15 25
Metalaxyl	Fungicide	9.33	234.0 → 146.1 **220.0 → 160.1** 206.1 → 162.1	20 10 5

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Metamitron	Herbicide	11.83	202.1 → 186.1 **104.1 → 51.0** 104.1 → 77.0	10 15 5
Metazachlor	Herbicide	10.45	209.0 → 133.2 **209.0 → 132.2** 209.0 → 117.1	10 15 35
Metconazole	Fungicide	14.22	153.1 → 125.0 153.1 → 70.0 **125.0 → 89.0**	10 5 20
Methiocarb	Insecticide	9.58	169.0 → 154.1 168.0 → 109.1 **153.0 → 91.1**	10 15 20
Metobromuron	Herbicide	8.79	258.0 → 61.0 **196.9 → 89.9** 169.9 → 142.9	10 25 20
(S)-Metolachlor	Herbicide	9.89	238.0 → 162.2 **238.0 → 133.2** 162.1 → 133.2	10 30 15
Metrafenone	Fungicide	15.24	**394.8 → 364.8** 376.9 → 346.8 226.9 → 169.0	15 20 10
Metribuzin	Herbicide	9.00	**198.0 → 82.0** 198.0 → 55.0 182.0 → 114.9	15 30 10
Myclobutanil	Fungicide	11.68	**179.0 → 125.1** 179.0 → 90.0 150.0 → 123.0	10 30 15

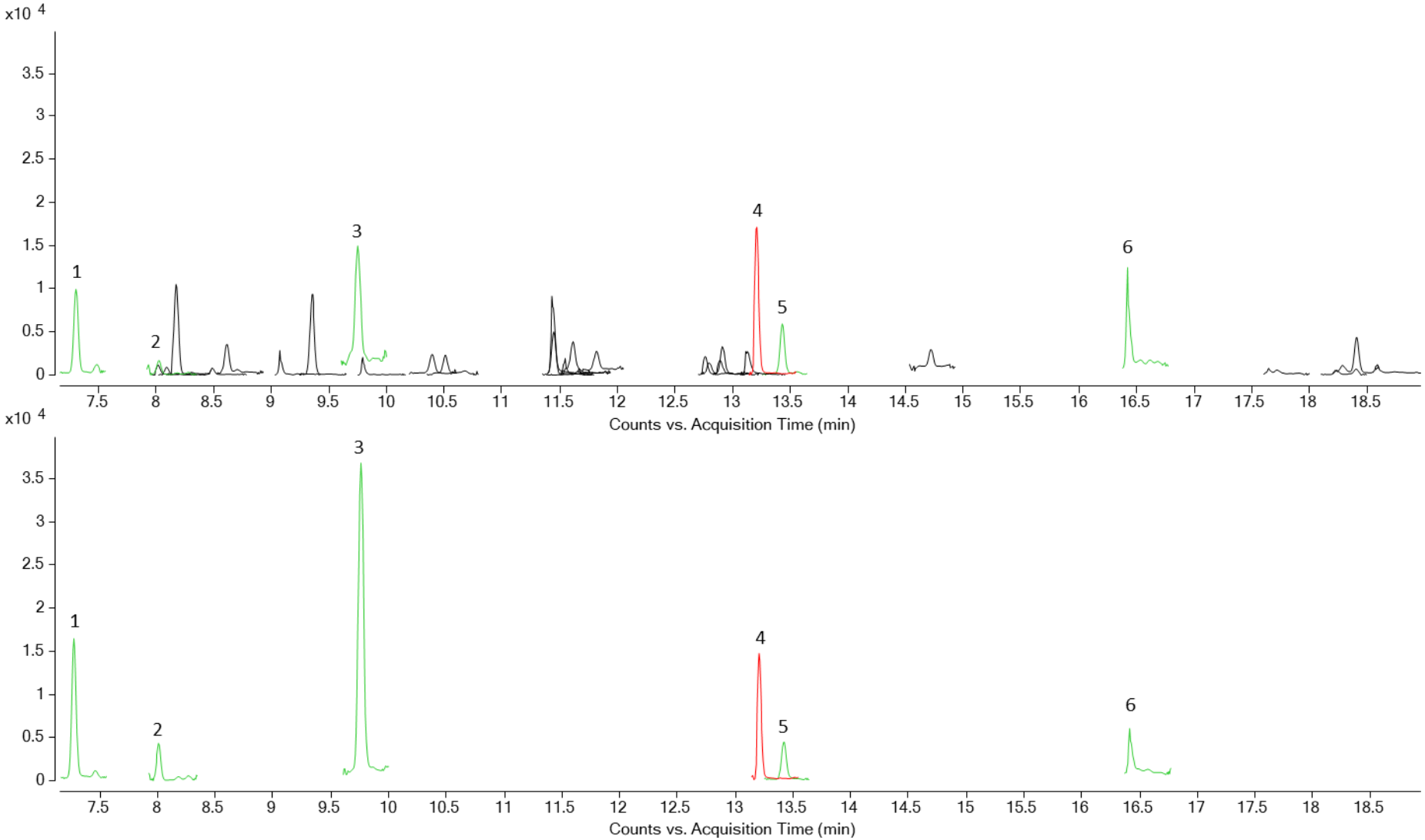
Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Napropamide	Herbicide	11.40	**271.0 → 100.1**	15
			271.0 → 72.1	15
			128.0 → 100.1	10
Oryzalin	Herbicide	15.51	316.8 → 274.9	5
			275.0 → 217.0	5
			258.0 → 193.9	5
Oxadiazon	Herbicide	11.63	**301.8 → 175.0**	15
			257.8 → 112.0	30
			174.9 → 112.0	15
Oxamyl	Insecticide, Nematicide	6.30	162.0 → 114.9	10
			145.0 → 71.9	20
			145.0 → 60.9	10
Oxychlordane	Metabolite	10.53	386.7 → 262.7	15
			236.9 → 142.9	25
			184.9 → 121.0	15
Oxyfluorfen	Herbicide	11.71	299.9 → 222.8	15
			252.0 → 196.0	20
			252.0 → 146.0	30
Paclobutrazol	Plant growth regulator	11.09	236.0 → 167.1	10
			167.1 → 132.1	10
			125.1 → 89.0	20
Parathion	Insecticide	9.97	**291.0 → 137.1**	5
			291.0 → 109.0	15
			139.0 → 81.0	15
Parathion-methyl	Insecticide, Nematicide	9.14	262.9 → 109.0	10
			262.9 → 79.0	30
			109.0 → 79.0	5
PCB 28	Pollutant	9.04	**258.0 → 186.0**	25
			256.0 → 186.0	25
			186.0 → 151.0	25
PCB 52	Pollutant	9.61	291.9 → 221.9	25
			289.9 → 219.9	25
			255.0 → 220.0	10
PCB 101	Pollutant	11.12	325.9 → 255.9	35
			325.9 → 253.9	30
			253.9 → 184.0	30
PCB 138	Pollutant	13.12	**361.9 → 289.9**	30
			359.9 → 289.9	30
			287.9 → 217.9	40
PCB 153	Pollutant	12.62	361.9 → 289.9	25
			359.9 → 289.9	25
			287.9 → 217.9	40
PCB 180	Pollutant	14.30	395.8 → 325.8	30
			393.8 → 358.8	15
			393.8 → 323.8	30
Penconazole	Fungicide	10.54	250.0 → 194.1	15
			250.0 → 157.1	25
			159.0 → 123.0	20
Pendimethalin	Herbicide	10.52	**251.8 → 162.2**	10
			251.8 → 146.1	20
			161.9 → 147.0	10
Pentachloro-nitrobenzene	Fungicide, Nematicide	8.20	294.8 → 236.8	15
			248.8 → 213.8	15
			141.9 → 106.9	30

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Permethrin (*cis* and *trans* isomer)	Insecticide	15.51 15.63	165.0 → 127.0 **162.9 → 127.0** 162.9 → 91.0	0 0 10
Phenmedipham	Herbicide	7.08	**167.0 → 135.0** 167.0 → 122.0 122.0 → 94.0	15 15 15
Phosmet	Insecticide	13.90	301.0 → 191.8 172.9 → 104.0 **160.0 → 133.0**	10 15 15
Phosmet-oxon	Metabolite	13.00	**301.0 → 191.8** 172.9 → 104.0 160.0 → 133.0	10 15 15
Picloram-methyl ester	Herbicide	9.55	**198.0 → 163.1** 198.0 → 161.0 196.0 → 181.0	15 15 15
Picolinafen	Herbicide	13.87	376.0 → 239.1 **376.0 → 238.1** 238.1 → 145.1	10 20 25
Picoxystrobin	Fungicide	11.29	**334.9 → 172.9** 302.8 → 156.9 145.0 → 102.1	10 15 25
Pirimicarb	Insecticide	8.73	**238.0 → 166.2** 166.0 → 71.1 166.0 → 55.1	10 25 10
Pirimiphos-methyl	Insecticide	9.58	290.0 → 125.0 **232.9 → 151.0** 232.9 → 125.0	20 5 5

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Prochloraz	Fungicide	15.91	310.0 → 69.8 **266.0 → 69.9** 180.0 → 68.9	15 10 15
Propamocarb	Fungicide	5.39	188.0 → 58.0 **143.0 → 99.1** 129.1 → 84.1	10 10 5
Propaquizafop	Herbicide	19.92	298.8 → 254.8 **162.9 → 135.8** 162.9 → 99.9	25 10 20
Propiconazole (2 isomers)	Fungicide	12.89 13.00	258.8 → 172.9 172.9 → 109.0 **172.9 → 74.0**	15 30 45
Propyzamide	Herbicide	8.06	173.0 → 145.0 173.0 → 109.0 **173.0 → 74.0**	25 25 25
Prosulfocarb	Herbicide	9.37	251.0 → 218.3 **251.0 → 128.2** 251.0 → 100.1	10 5 5
Prothioconazole-desthio	Fungicide	11.91	**186.0 → 89.0** 186.0 → 70.0 125.0 → 99.0	10 10 20
Pymetrozine	Insecticide	11.51	**132.0 → 105.0** 132.0 → 78.0 113.0 → 98.0	10 20 5
Pyraclostrobin	Fungicide	17.46	**324.8 → 131.7** 164.0 → 132.1 110.8 → 75.0	15 10 15

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Pyraflufen-ethyl	Herbicide	13.03	412.0 → 349.0 349.0 → 307.0 **338.9 → 288.9**	10 15 15
Pyridaben	Insecticide	15.77	**309.0 → 147.1** 147.2 → 132.2 147.2 → 117.1	15 10 20
Pyridalyl	Insecticide	16.73	204.0 → 148.0 164.0 → 146.0 **146.0 → 126.0**	25 15 10
Pyrimethanil	Fungicide	8.24	198.0 → 183.1 198.0 → 158.1 **198.0 → 118.1**	15 20 35
Pyriproxyfen	Insecticide	14.61	**321.0 → 222.0** 321.0 → 153.0 136.1 → 96.0	10 25 15
Quinoclamine	Herbicide	9.76	209.0 → 172.1 207.0 → 172.1 **172.0 → 89.0**	10 20 20
Quinoxyfen	Fungicide	12.85	306.8 → 237.0 271.9 → 237.1 **237.0 → 208.0**	20 10 30
Spirodiclofen	Insecticide	15.56	312.1 → 259.0 **312.1 → 108.9** 157.0 → 73.0	10 15 25
Spiromesifen	Insecticide	13.71	272.0 → 209.2 **253.8 → 185.1** 231.0 → 157.1	10 15 15
Spiroxamine (2 Isomers)	Fungicide	9.08 9.53	**198 → 126.1** 126.0 → 84.0 100.0 → 58.0	5 5 10
Tebuconazole	Fungicide	13.22	**250.0 → 125.0** 125.0 → 99.0 125.0 → 89.0	20 20 15
Tebufenpyrad	Insecticide	14.09	332.9 → 171.0 318.0 → 131.0 **275.9 → 171.1**	15 15 10
Tefluthrin	Insecticide	8.41	199.0 → 161.1 **197.0 → 161.1** 177.1 → 127.1	5 5 15
Terbuthylazine	Herbicide, Microbiocide	8.12	228.9 → 138.0 214.0 → 104.0 **214.0 → 71.0**	15 20 5
Terbuthylazine-desethyl	Herbicide, Microbiocide	7.36	186.2 → 104.0 **186.2 → 83.1** 145.1 → 68.1	15 20 10
Tetraconazole	Fungicide	9.99	336.0 → 217.9 **336.0 → 203.8** 170.9 → 136.0	20 30 10
Thiabendazole	Fungicide	10.73	**201.9 → 175.0** 201.0 → 130.0 173.9 → 65.0	15 30 30
Thiacloprid	Insecticide	17.20	126.0 → 99.1 126.0 → 90.1 **126.0 → 73.0**	10 5 20

Name	Pesticide classification	RT [min]	dMRM Transitions	CE [eV]
Tolclofos-methyl	Fungicide	9.14	**267.0 → 252.0**	15
			267.0 → 93.0	30
			267.0 → 63.0	45
Tralkoxydim	Herbicide	14.75	282.1 → 226.0	10
			268.2 → 143.0	40
			226.0 → 143.0	25
Triadimenol	Fungicide	10.73	**129.9 → 102.0**	15
			129.9 → 65.0	25
			112.0 → 58.0	10
Triallate	Herbicide	8.57	**270.0 → 228.1**	10
			268.0 → 226.1	10
			268.0 → 184.1	20
Triclopyr-methyl ester	Herbicide	7.51	**209.9 → 145.9**	20
			209.9 → 109.9	35
			145.9 → 110.0	15
Trifloxystrobin	Fungicide	12.94	186.0 → 145.1	15
			172.0 → 145.1	15
			172.0 → 95.0	30
Triflumizole	Fungicide	10.81	345.0 → 302.0	10
			239.1 → 66.9	40
			132.0 → 90.0	35
Trinexapac-ethyl	Herbicide	9.50	**224.0 → 151.0**	5
			224.0 → 95.0	25
			207.0 → 68.9	25
Triticonazole	Fungicide	14.51	237.0 → 182.0	10
			237.0 → 167.1	25
			234.8 → 182.1	10
Warfarin	Rodenticide	15.44	308.0 → 187.0	20
			265.0 → 187.0	5
			265.0 → 121.0	15
Zoxamide	Fungicide	13.47	259.9 → 189.0	10
			257.9 → 187.1	10
			189.0 → 161.1	15



Supplementary Figure 1. Chromatograms of a matrix matched calibration standard (LOQ concentration, top) and the Beech sample from Belauer See, 2011 (bottom). 1 = Terbuthylazine-desethyl, 2 = terbuthylazine, 3 = fenpropimorph, 4 = triphenyl phosphate (ISTD), 5 = epoxiconazole, 6 = boscalid.

6. Other projects

6.1. Drivers of leaf-associated insect decline

As already stated in Chapter 5., Jun.-Prof. Dr. Henrik Krehenwinkel is investigating the shift in insect populations *via* DNA analysis. With the help of a quantitative polymerase chain reaction (qPCR) protocol, it was possible to draw conclusions about the species and quantity of leaf-associated insects that were present in the samples. For qPCR, pulverized leaves and needles from different sampling sites in Germany were used. The samples were collected by the UBA from the same locations with different land use in a time series of 30 years.

In order to find possible reasons for insect decline or a shift in their populations, different influences were under evaluation. Besides climate data, one of the parameters was the pesticide and pollutant load in forests. Therefore, the developed multiresidue analysis for vegetation samples was used to analyse more than 250 leaf and needle samples of common beech (*Fagus sylvatica*), black poplar (*Populus nigra*), Norway spruce (*Picea abies*) and Baltic pine (*Pinus sylvestris*). The sampling sites covered all kinds of land use, from national parks over agricultural areas to urban places. The found results were correlated with the DNA analysis data obtained by the group of Jun.-Prof. Dr. Krehenwinkel.

Personal contribution:

Analysis of samples with the developed QuEChERS method for leaf and needle samples,[100] review and editing of the article.

Article:

Krehenwinkel, H.; Weber, S.; Broekmann, R.; Melcher, A.; Hans, J.; Wolf, R.; Hochkirch, A.; Kennedy, S. R.; Koschorrek, J.; Künzel, S.; Müller, C.; Retzlaff, R.; Teubner, D.; **Schanzer, S.**; Klein, R.; Paulus, M.; Udelhoven, T.; Veith, M., Environmental DNA from archived leaves reveals widespread temporal turnover and biotic homogenization in forest arthropod communities. *bioRxiv* **2022**. https://doi.org/10.1101/2022.04.27.489699.

6.2. Pesticide exposure of garden dormice (*Eliomys quercinus*)

The garden dormouse (*Eliomys quercinus*) is a rodent species that is native solely in Europe and thus, also in Germany. The species has been fighting population decreases for decades and is facing extinction in parts of Europe.[101] As the reasons for the species decline are barely

known, Johannes Lang and Eva Marie Famira-Parcsetich, from the Clinic of Birds, Reptiles, Amphibians and Fish (working group for Wildlife Research) of the University of Gießen, Germany, are investigating the possible causes. One field of interest in this context was the pesticide load of the small rodents. To this end, the developed micro QuEChERS approach (see Chapter 3.) was used to analyse the livers of more than 100 garden dormice.
The results of this cooperative work were presented at the 11th International Dormice Conference (09.-13.05.2022) by Eva Marie Famira-Parcsetich.

Personal contribution:

Method application of the developed micro QuEChERS approach[92] for multiresidue analysis of garden dormice; review and editing of the conference abstract.

Conference abstract:

Famira-Parcsetich, E. M.; **Schanzer, S.**; Müller, C.; Schenke, D.; Lierz, M.; Lang, J., Another one bites the dust: pollutants and pesticides in Garden Dormice found dead, *ARPHA Conference Abstracts* **2022**, *5*, e82820. https://doi.org/10.3897/aca.5.e82820.

6.3. Analysis of per- and polyfluoroalkyl substances with LC-MS/MS

Apart from multiresidue analysis of pesticides and pollutants with gas chromatography, a liquid chromatography method for the analysis of three representative per- and polyfluoroalkyl substances (PFAS) was developed and validated. Method development, validation and proof of concept were part the bachelor's thesis of Carolin Ellerbrock, which I supervised.
PFAS are a group of organic compounds with different functional groups, *e.g.* alcohols, carboxylic acids or sulfonic acids. They all have in common that some or all hydrogen atoms of the carbon backbone are substituted by a fluorine atom. Due to the reduced reactivity of the carbon-fluorine bond, PFAS have very long half-lives, which gave them the name "forever chemicals".[102] Because of their persistence and their probability of adverse health effects, they are listed in the Stockholm Convention Annex (see 1.2.5.).[32]
Their physicochemical properties required another analytical approach than gas chromatography-tandem mass spectrometry, as these amphiphilic substances are not GC amenable. Therefore, a QuEChERS approach, followed by liquid chromatography coupled to tandem mass spectrometry, was developed. Again, liver matrix was used for sample preparation. Within a runtime of 6 min, three very common PFAS were detected and quantified with an internal standard: PFOA (perfluorooctanoic acid), PFOS (perfluorooctanesulfonic

acid), and PFHxS (perfluorohexanesulfonic acid) with SDS (sodium dodecyl sulfate). Every substance was identified with three MRM transitions.
After method development and validation, animal liver samples were analysed for proof of concept. The livers of 26 garden dormice, ten bats, ten hedgehogs, ten birds and two deer were worked up with the optimized sample preparation method and analysed with LC-MS/MS. PFOA and PFHxS were hardly found, whereas PFOS was found in a high percentage of the samples. The maximum concentration of PFOS was >400 µg kg^{-1} (exceeding the upper limit of the linear range). Three garden dormice were exposed to this high amount of PFOS.

Personal contribution:

Project supervision at all stages; preliminary experiments.

Article:

A publication on the subject is in preparation.

7. Summary

The “Quick, Easy, Cheap, Effective, Rugged and Safe” (QuEChERS) method was originally developed for pesticide analysis in foodstuffs with high water content. The changeability of this method lead to many adaptions in terms of matrices and analytes. However, the sample preparation still has limits with regard to sample sizes or challenging matrices. Hence, one aim of this work was to optimize the sample preparation so that a minimum of sample can be used. Furthermore, the obstacle of challenging matrices, such as high fat content, low water content, or high pigmentation, had to be overcome.

In Chapter 3., the problem of limited sample sizes was addressed in the first place. Here, the miniaturization of a QuEChERS approach with liver tissue as a matrix was presented. For individual analysis of liver samples, a new comminution technique had to be implemented. Additionally, modifications of the SALLE and of the dSPE were evaluated, respectively, with a special focus on lipid removal. After method optimization with 5 g of liver tissue, the sample preparation was downscaled to 100 mg of sample. Subsequently, both methods were validated according to SANTE/12682/2019. For proof of concept, livers of wildlife animals (hedgehogs and bats) were analysed for pesticide and pollutant residues. Ultimately, it was possible to analyse 209 compounds in concentration ranges between 1 and 400 µg kg^{-1}.

The miniaturization was necessary to be able to analyse a large dataset of bats from all over Germany. As the animals’ body weight ranges from 3 g to 30 g, the liver weight was not high enough to use a conventional QuEChERS approach with up to 10 g material. To this end, the micro QuEChERS method with 100 mg of sample, that was developed within the frame of this work, was applied on nearly 400 samples of five different bat species (Chapter 4.). These results present the largest study on pesticide and pollutant exposure of Chiroptera in Germany as of yet. The pesticide data of the animals were correlated with metadata such as place and time of finding, age, sex, and bat season. Statistical tests revealed that the pesticide exposure is largely homogenous across Germany.

Another limitation of the QuEChERS method is the matrix of use. In Chapter 5., a method for the analysis of vegetation samples (beech leaves and spruce needles) was developed. Due to the high chlorophyll content of these matrices, the sample preparation required modifications different to those of the liver samples. Hence, another QuEChERS-based sample preparation was developed, which paid special attention to the removal of chlorophyll. The analysis of vegetation samples was eventually used for the monitoring of the pesticide load of trees from different sampling sites with a sampling period of 30 years.

In conclusion, this work emphasizes the importance of an appropriate pesticide monitoring, not only in foodstuffs, but also in our environment, with the help of sensitive analytical methods, and helps to assess the existing environmental burden of the German flora and fauna.

8. List of abbreviations

ACCase	acetyl-CoA carboxylase
ACh	acetylcholine
AChE	acetylcholinesterase
AML	acute myeloid leukaemia
AOAC	Association of Official Analytical Chemists
ATP	adenosine triphosphate
CLL	chronic lymphocytic leukaemia
CNS	central nervous system
COPD	chronic obstructive pulmonary disease
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DFG	Deutsche Forschungsgemeinschaft
dMRM	dynamic multiple reaction monitoring
dSPE	dispersive solid phase extraction
EPSPS	enolpyruvylshikimate-3-phosphate synthase
FAO	Food and Agriculture Organization of the United Nations
GABA	γ-aminobutyric acid
GC	gas chromatography
GC-MS	gas chromatography-mass spectrometry
GPC	gel permeation chromatography
HCH	hexachlorocyclohexane
ISTD	internal standard
LC	liquid chromatography
LC-MS	liquid chromatography-mass spectrometry
MeCN	acetonitrile
MRL	maximum residue limit
MS	mass spectrometry
MS/MS	tandem MS
NPD	nitrogen-phosphorus detector
OC	organochlorine (insecticide)
PCB	polychlorinated biphenyl

PCDD	polychlorinated dibenzodioxin
PFAS	per- and polyfuoroalkyl substances
PFHxS	perfluorohexanesulfonic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic acid
POP	persistent organic pollutant
PSA	primary secondary amine (sorbent)
qPCR	quantitative polymerase chain reaction
QQQ	triple quadrupole MS
QuEChERS	quick, easy, cheap, effective, rugged and safe
SALLE	salt-assisted liquid-liquid extraction
SDS	sodium dodecyl sulfate
SPE	solid phase extraction
TCDD	2,3,7,8-tetrachlorodibenzodioxin
UBA	Umweltbundesamt (German Environment Agency)
US EPA	United States Environmental Protection Agency
WHO	World Health Organization

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