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POLICY BRIEF

Using environmental monitoring data from apex predators for chemicals management: towards harmonised sampling and processing of archived wildlife samples to increase the regulatory uptake of monitoring data in chemicals management

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Abstract

Monitoring data from apex predators were key drivers in the development of early chemicals legislations due to the population declines of many species during the twentieth century, which was linked to certain persistent organic pollutants (POPs). Besides triggering the development of global treaties (e.g. the Stockholm Convention), chemical monitoring data from apex predators have been particularly important for identifying compounds with bioaccumulative properties under field conditions. Many apex predators are protected species and only a few environmental specimen banks (ESBs) regularly collect samples as many ESBs were established during the 1980–1990s when apex predators were scarce. Today, many POPs have been banned, which contributed to the recovery of many apex predator populations. As a consequence, apex predator samples are now available in research collections (RCs) and natural history museums (NHMs). These samples can be used for routine analysis as well as for screening studies using novel analytical techniques and advanced data treatment workflows, such as suspect and non-target screening. The LIFE APEX project has demonstrated how these samples can be used in a cost-efficient way to generate data on legacy compounds and contaminants of emerging concern. Furthermore, it has described quality assurance/control measures to ensure high quality and comparable data, with a view to uses in chemicals risk assessment and management. To increase the visibility of available sample collections and monitoring data from apex predators we developed accessible online database systems. Additionally, the acquired high-resolution mass spectrometric data were stored in a digital sample freezing platform that allows retrospective suspect screening in previously analysed samples for substances that may be of concern/under assessment in the future. These databases provide open access to a wide range

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of chemical data, for use by regulators, researchers, industry and the general public, and contribute to a stronger link between science and policy.

Keywords: Apex predators, Quality assurance, Archived samples, Digital sample freezing platform, Suspect screening, Chemicals regulations

Introduction

The objectives of chemical monitoring are related to screening environmental samples for contaminants of emerging concern, assessing the effectiveness of management measures, comparing data against quality criteria in status assessments and collecting information for risk assessments, including the bioaccumulation of substances in food webs and their transfer across environmental media. Monitoring data from wildlife taxa, including apex predators, can be used as evidence for the presence of contaminants in the environment to support chemical legislation. In addition, they can provide information on bioaccumulation and biomagnification of a given chemical. Currently, large-scale contaminant monitoring programmes under European chemicals and environmental legislation mainly focus on fish and molluscs, providing only indirect information on risks to higher trophic level taxa, with a focus on the aquatic environment.

The LIFE APEX project (LIFE17 ENV/SK/000355) aimed to improve the systematic use of chemical monitoring data from apex predators and their prey in chemicals management to ultimately protect human health and the environment. The project demonstrated how valuable but underused existing sample collections across Europe can be used in modern environmental monitoring to support chemicals management. The project applied novel analytical techniques, such as (wide scope) target, suspect and non-target screening, to investigate the presence of several thousands of chemicals in European wildlife. Specifically, the selected samples included marine mammals, Eurasian otter (*Lutra lutra*) and common buzzards (*Buteo buteo*) representing the marine, freshwater and terrestrial environment. The project started with a 'Tier 1' screening exercise, in which recently collected samples from the United Kingdom, Sweden, the Netherlands and Germany were analysed. 'Tier 2' retrospectively analysed time series of archived fish, harbour seal (*Phoca vitulina)*, otter and buzzard samples from the United Kingdom and Germany (2000–2018) to establish temporal trends for contaminants. 'Tier 3' analysed samples from 19 European countries to demonstrate the replicability of the LIFE APEX screening approach at a pan-European scale. Comprehensive analytical methodologies together with systematic quality assurance and quality control measures for collecting, transporting, processing and archiving of samples were established to ensure high quality monitoring data on legacy pollutants, contaminants of emerging concern as well as on chemical mixtures. A parallel study under LIFE APEX analysed common buzzard samples from 12 European countries to assess the statistical power of using pooled samples to detect trends in contaminant concentrations following the introduction of risk management measures. The data from these studies have a range of applications in support of environmental management (Fig. 1), such as the EU's Zero Pollution Ambition [1], and are highly pertinent to innovative European research initiatives in the field of safe use of chemicals, such as the European Partnership for the Assessment of Risks from Chemicals (PARC) [2].

To contribute to the implementation of the LIFE APEX outcomes, we provide two policy briefs that address challenges, recommendations, requirements and achievements for the use of chemical monitoring data from biota, in particular apex predators, in chemicals risk assessment.

- The first policy brief (this article) addresses initiatives that enable better use of apex predator monitoring data in chemical risk assessment and environmental monitoring, in particular quality assurance measures for sampling, processing and archiving of apex predator samples as well as means to improve accessibility of the data, including the digital sample freezing of high-resolution mass spectrometric (HRMS) data.
- The second policy brief ('*Towards better use of monitoring data from apex predators in support of prioritisation and risk assessment of chemicals in Europe*') highlights the added value of LIFE APEX methodologies for prioritising thousands of chemicals with regard to hazard endpoints, in particular bioaccumulation, to improve future risk assessments across regulations by using real-world data from apex predators.

Challenge

Apex predators and chemical pollution—continuing and emerging threats

Legacy pollutants, such as dichlorodiphenyltrichloroethane (DDT) and polychlorinated biphenyls (PCBs), not only caused population declines of many species during

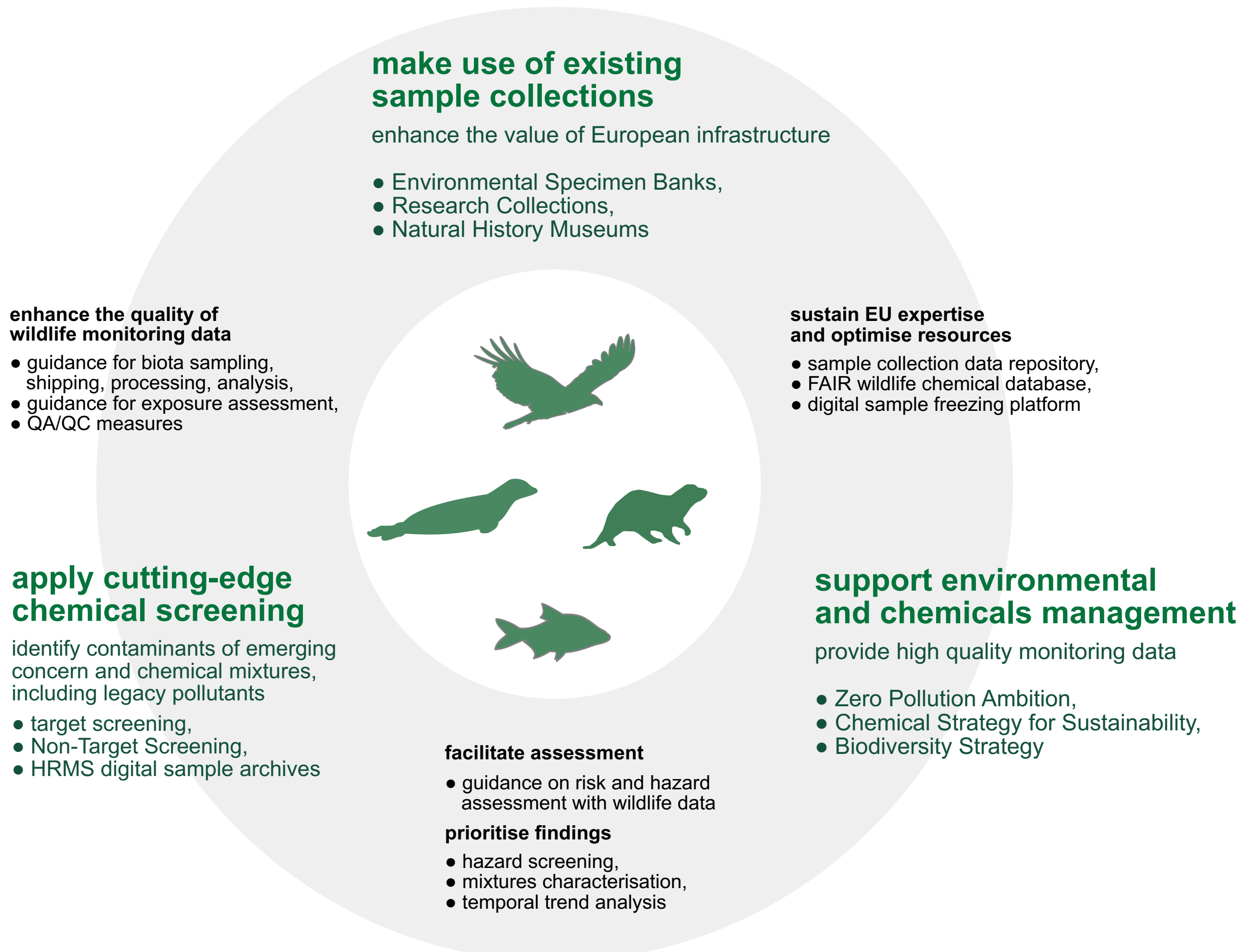

Fig. 1 LIFE APEX cycle displaying the connection between wildlife samples, novel analytical techniques and chemicals management

the twentieth century, they also triggered chemical risk assessment and mitigation measures for substances and their uses [3–5]. The population decline was particularly evident in apex predators, which in turn resulted in increased societal and political awareness of chemical contamination, as many apex predators are perceived as charismatic indicators of high conservation value and ecosystem diversity [6, 7]. The chemical contamination of apex predators has also triggered screening and monitoring studies of legacy and emerging chemicals in these species [8–10]. Their well-known ecology, long lifespan, high trophic position and sensitivity to many anthropogenic chemicals make them suitable sentinel species for bioaccumulating substances [11–13] in the Persistent, Bioaccumulative and Toxic (PBT) assessment of chemicals (EC 1907/2006, REACH) and in the context of the Stockholm Convention [14]. Furthermore, apex predators have been identified as key sentinel species of ecosystem health and ecosystem services in environmental legislation and are protection goals in the Water Framework Directive (WFD, 2000/60/EC), specifically the Directive on Environmental Quality Standards (2008/105/EU, as amended) and Descriptors 1, 8 and 9 of the Marine Strategy Framework Directive (MSFD, 2008/56/EC).

While most European apex predators seem to have recovered from the contaminant-induced population declines of the twentieth century [15–17], some species are still threatened by POPs [e.g. 12, 18]. Together with other stressors, such as climate change and increasing habitat loss, this is expected to result in cumulative impacts. Monitoring data from apex predators are therefore important not only for the general assessment of the state of the environment, particularly the threat of bioaccumulating and biomagnifying substances, but also for species conservation programmes. Due to the long-range transport of chemicals also remote areas, such as the Arctic and Antarctic, are of concern, posing a particular challenge in terms of sample availability [19, 20].

The assessment of chemical contaminants in apex predators furthermore contributes to the aims of the United Nations Sustainable Development Goal 14.1 regarding the reduction of marine pollution.

Lack of standardisation and guidance for using chemical monitoring data from wildlife

In general, fish and mussels are addressed in guidance documents for monitoring biota in regulatory monitoring frameworks [21–23], while similar recommendations for apex predators are lacking. The risk for mammals, and often other terrestrial vertebrates, is usually estimated using results from experimental animal studies designed specifically for assessing human health effects. The wildlife samples that could be used for chemical analysis appear to have been underestimated, as a central database for biota samples in collections across Europe has been lacking. Further constraints for using monitoring data from apex predators were indicated in a survey that was addressed to European institutions involved in chemical risk assessment (e.g. national competent authorities, industry, academia). The respondents indicated that a lack of standardisation and guidance on how to use the data was missing [24]. The gaps often concerned measures to ensure sufficient quality in all steps of the workflow—including sampling, archiving and sending samples to the analytical laboratory. A second survey among European sample archives revealed great differences in sampling approaches and quality assurance measures among environmental specimen banks (ESBs), research collections (RCs) and natural history museums (NHMs) [24]. Additionally, harmonisation of novel analytical methods, such as suspect and non-target screening, including digital sample freezing and retrospective analysis, is needed to ensure a regulatory uptake of (semi-)quantitative data. The majority of the respondents among chemical risk assessment institutions indicated that they were currently only using contaminant data from lower trophic levels or abiotic matrices [24]. These data mostly stem from standardised laboratory tests and regulatory monitoring programmes that focus on, e.g. fish and mussels with readily available guidelines [e.g. 25]. In general, the respondents did not routinely consider contaminant data from wildlife and apex predators. However, they indicated that—provided that there are sufficient data quality assurance/control measures—they would be interested in integrating such data into their assessments in the future [24].

Recommendations

Making more efficient use of existing European sample collections

The LIFE APEX project and the simultaneous COST Action 'European Raptor Biomonitoring Facility' (CA16224) have demonstrated that samples from various environmental compartments and with wide European geographical coverage are available in ESBs, RCs and NHMs and can be cost-efficiently used [26–28]. However, harmonisation of quality assurance measures across ESBs, RCs and NHMs still remains a challenge as sampling programmes can have different strategies (systematic/opportunistic) and the implementation of quality assurance measures and availability of relevant metadata vary among institutions [24]. However, as a first step towards harmonisation, protocols have been established that address the collection, storage and analysis of samples [24, 29], which together with identified key criteria for species and matrix selection [11, 30] can support pan-European monitoring programmes. This is particularly relevant for assessing the effectiveness of risk mitigation measures at a European scale as chemicals assessments are harmonised and implemented through common European Union (EU) directives and regulations.

Requirements

Harmonised sampling strategy

One of the purposes of using samples of apex predators is to support chemicals management by evaluating the effectiveness of chemical policies in general and substance regulations in particular, to provide data for risk assessment and to identify emerging issues. However, the screening and monitoring data have to meet certain quality requirements before such data can be used in a regulatory context. The most obvious first step for large-scale monitoring involving samples from different collections is to select a sentinel species or, if this proves difficult, a common taxonomic group with similar ecological traits that meets the purpose of the monitoring campaign [11]. Besides the objectives of the programme, the selection of a sentinel species depends on species distribution across broad spatial regions together with key ecological criteria, such as feeding ecology, habitat use and residential/migratory behaviour. For marine mammals, target species (e.g. dolphins) are often highly mobile, i.e. national frameworks cannot accomplish the necessary logistics and coordination. Thus, such activities require large-scale (EU and beyond) frameworks for a successful implementation, in line with the ocean-scale impact of chemical contaminants.

A second step that is critical for monitoring includes the selection of sample matrix for chemical analysis [29, 30]. Many apex predators are protected species and sample collections usually archive specimens that originate from opportunistic sampling approaches based on the availability of carcasses [28]. Exceptions represent the sampling of feathers or eggs in case of raptors, whereas the collection of, e.g. blood requires further ethical considerations. Furthermore, the choice of the sample matrix depends on the physicochemical properties of the target analytes, as well as on the use of the data in chemicals management. Different goals could include to (a) assess exposures to a certain chemical class, e.g. for a pending risk assessment/exposure evaluation, (b) identify as many chemicals as possible, including chemical mixtures, to which an apex predator is exposed, or (c) to qualitatively or quantitatively assess the bioaccumulation of contaminants in food webs. In the liver, both lipophilic and more hydrophilic chemicals can be detected [e.g. 31], as it contains lipids and is also the metabolically most competent organ. The fat content generally varies with the nutritional condition of the individual, which may lead to the remobilisation of lipophilic compounds into the bloodstream in the case of starved individuals. This is particularly relevant for the sampling of apex predators as the majority of samples in collections are opportunistic samples from deceased individuals, which may have died after starvation. The availability of sufficient metadata in addition to the lipid weight, such as nutrition condition, but also age class or sampling year and sex are therefore crucial for interpreting analytical results. Despite these variations, the liver is considered to represent the most suitable organ for screening studies with the aim of detecting a broad range of chemicals with different physicochemical properties, including stable metabolites and transformation products. However, blood might be more suitable when focussing on rather mobile and persistent substances [32].

Quality assurance and quality control measures

Sample collections that can provide samples from apex predators include ESBs, established in support of chemical monitoring, RCs, typically with some experience in chemical monitoring, and NHMs, where there may be less focus on contaminant research [24, 28]. However, only a few ESBs regularly collect samples of apex predators as many ESBs were established during the 1980–1990s when apex predators were scarce. Staff training as well as harmonised guidance, best practice documents and tutorials, e.g. from ESBs, are important elements in building capacity for apex predator and prey sample collections, helping to ensure comparability and consistency of monitoring results [14].

Quality assurance and quality control measures are particularly important when analysing contaminants that have a high potential for cross-contamination such as personal care products (e.g. parabens, fragrances, insect repellents) that may be used by staff members involved in sampling and sample processing. Other potential sources of external chemical contamination may be related to the veterinary treatment of individuals in rehabilitation centres (e.g. veterinary antibiotics and analgesics), to the taxidermy of dead animals using biocides or to frequently used industrial chemicals in laboratories (e.g. plasticizers, flame retardants, solvents, etc.). Whereas clinical records can help identify veterinary treatments, preventing cross-contamination from personal care products or industrial chemicals used in laboratories remains challenging. Besides using field blanks, we propose a scoring system for the quality assurance measures applied during sampling processing and storage of each sample based on identified key criteria [24], similar to the approach established for reporting and evaluating ecotoxicity data [33]. Together, these guidance documents will (1) improve the quality and comparability of screening and monitoring data of chemicals in wildlife, an important factor to enhance the regulatory uptake of wildlife monitoring data in chemicals management, and (2) ensure that the valuable resources of European sample archives are efficiently used to produce valid and plausible results for supporting hazard and risk assessment.

Regulatory requirements

Besides quality assurance aspects in sampling and sample processing, which help discriminate the animal's original exposure to environmental contaminants from cross-contamination, the reliable identification of chemicals in the samples becomes increasingly important, including the distinction of metabolites and transformation products from naturally occurring substances. Comprehensive high-resolution analytical tools are state-of-the-art in the identification of unknown or suspected compounds. While the value of monitoring data is well recognised for developing risk assessment models, their use in actual assessments is limited by the lack of information influencing the exposure conditions [34]. Adding information on, e.g. chemical uses, exposure indices and tonnages (if available) to existing suspect screening databases that contain information for substance identification (i.e. chromatographic and spectrometric parameters, such as precursor ion, predicted retention time and fragment ions predicted *in-silico* or retrieved from existing literature), will help develop comprehensive databases for identifying contaminants of emerging concern. Furthermore, additional matrices from lower trophic level (prey-) species (e.g. fish) as well as abiotic matrices (e.g. water) collected in the same spatiotemporal context as the apex predators can provide extensive information (e.g. biomagnification factors) that will likely increase the regulatory uptake of the data and ultimately improve risk assessments. These approaches combined with the ongoing regulatory pressure for development landscape risks assessment models will maximise the capacity of monitoring data for supporting the risk assessment and its translation into environmental impacts.

Achievements

Development of online database systems

The centralised NORMAN Digital Sample Freezing Platform [35] contains chemical information, e.g. from all five European marine regions (biota and non-biota) and can be used by regulatory European monitoring frameworks, such as those of HELCOM, OSPAR or the EMBLAS Black Sea monitoring campaign. This offers new opportunities for prioritisation and screening exercises, for example studying ubiquitous or region-specific exposure patterns. In the future it can be envisaged that this approach can be complemented with more data from freshwater and terrestrial ecosystems to achieve holistic exposure assessments in support of the MSFD, the EU Zero Pollution strategy and the One Substance—One Assessment strategy across the compartment and substance specific legislations [1, 36, 37]. The online databases developed as part of the LIFE APEX project can overcome the hurdles of the low visibility and accessibility, for both existing sample collections and data generated from these samples (https://www.norman-network.com/apex/):

- A first database provides a sample catalogue of European apex predator and prey collections, including information on available species, number of samples and archived sample matrices (e.g. liver, muscle) from various institutions across Europe.
- A second database provides a substance database where users can access a list of different compound groups and their uses, including key parameters for chemical identification in HRMS suspect screening.
- The chemical occurrence database provides a geo-referenced database where concentrations of detected substances and relevant metadata are available for each sample.
- Finally, the digital sample freezing platform is a next-generation data platform where HRMS chromatograms are stored for retrospective non-target screening (data mining).

Establishing and maintaining an infrastructure for long-term archiving of chemical monitoring data, including quality assurance information, metadata and tools for retrospective analysis in biota and apex samples from various projects and monitoring exercises are important steps in making apex predator data available for regulatory uptake [35, 38]. The storage of HRMS chromatograms in the digital sample freezing platform is particularly important for chemicals which have so far been disregarded or overlooked in prioritisation and chemical risk assessment and may become relevant in the future. With LIFE APEX, regulators are now able to retrieve chemical data for apex predators and their prey from these databases, e.g. for substance evaluations and assessments. So far, the data collections are especially helpful for screening exercises, since the spatial resolution is low and predator–prey relationships have not been established yet. This is expected to change in the future, when more results will be added to the data repositories and links to contaminant data from other media will be explored. Such information will ultimately strengthen the connection between science and regulation [39].

The accessibility of archive information, together with information on contaminants of emerging concern, is not only relevant for regulatory purposes but also for initiatives that are now increasingly using comprehensive analytical tools to build reference databases for suspect/non-target screening in biota matrices [e.g. 40]. These initiatives further support the EU Zero Pollution Action Plan [1] for reducing the impact of chemical pollution as well as the EU Biodiversity Strategy [41], where chemical pollution is recognised as an important threat to biodiversity [42].

Making the data accessible to comply with regulatory needs

The LIFE APEX chemical occurrence database currently holds a total number of 946,651 results for 3,248 substances (LIFE APEX database, accessed 24/06/2022). All databases will be systematically extended and integrated into the NORMAN database system and can support research initiatives and partnerships, such as PARC [2, 40]. Furthermore, the data will be regularly uploaded to IPCheM (the Information Platform for Chemical Monitoring, hosted by the European Commission) and EMODnet (the European Marine Observation and Data Network) together with other chemical occurrence data from the NORMAN network. This will facilitate the sharing of data between authorities and provide data access to researchers, regulators and industry. By using publicly accessible databases, the LIFE APEX project complies with the Findability, Accessibility, Interoperability and Reusability of data (FAIR) principles. These databases will ultimately strengthen the science to policy interface, for example in the European research partnership PARC [2]. Using centralised and easily accessible databases, the authorities responsible for chemicals management can react quickly to environmental concerns that may arise in the future. Accessible online databases with intuitive (map-based) visualisation tools also support risk communication when sharing the data in public-oriented formats, such as social media, newsletters or other news outlets. Thereby, regulators can respond to high the priority given by Europeans to addressing chemical pollution [43].

Abbreviations

CA: Cost action; DDT: Dichlorodiphenyltrichloroethane; EFSA: European Food Safety Authority; EMODnet: European marine observation and data network; EMPLAS: Environmental monitoring in the Black Sea; EPA: U.S. Environmental Protection Agency; ESBs: Environmental specimen banks; EU: European Union; European Commission: European Commission; FAIR: Findability, accessibility, interoperability and reusability of data; HELCOM: Baltic Marine Environment Protection Commission (Helsinki Commission); HRMS: High-resolution mass spectrometry; IPCheM: Information platform for chemical monitoring; MSFD: Marine Strategy Framework Directive; NHMs: Natural history museums; NORMAN: Network of reference laboratories, research centres and related organisations for monitoring of emerging environmental substances; OECD: Organisation for economic co-operation and development; OSPAR: Convention for the protection of the marine environment of the North-East Atlantic (Oslo/Paris); PARC: Partnership for the assessment of risks from chemicals; PBT: Persistent, bioaccumulative and toxic; PCBs: Polychlorinated biphenyls; POPs: Persistent organic pollutants; RCs: Research collections; UNEP: United Nations Environment Programme; WFD: Water Framework Directive.

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Author contributions

AB and JK were responsible for the concept of the manuscript. AB drafted the manuscript and all the authors read, improved and approved the final manuscript.

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Declarations

Ethics approval and consent to participate

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Consent for publication

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Competing interests

The authors declare that they have no competing interests.

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