



2-Phenoxyethanol derivatization in ink dating determination

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significant increase in enzyme activities (SOD, GST, CAT) and TAC levels, after 14 days of exposure suggesting that this concentration of NPs may cause oxidative stress in fish. In addition, the variability found in some results may be due to fish not ingesting the same amount of food containing NPs, as they compete for food. Overall, the present study contributes to a better understanding of the risk of exposure to NPs for aquatic biota.

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2-Phenoxyethanol derivatization in ink dating determination

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ABSTRACT

Introduction: 2-Phenoxyethanol (PE) is a volatile compound present in the composition of inks. After the deposition in a document, it starts to evaporate over time. This ageing process potentially allow to estimate the date of an ink in a document [1], however this is a complex system where the PE derivatization with different derivatization agents and methods can contribute to improve ink dating validation [2]. The aim of this work is to determine if PE derivatization with MSTFA:TMCS, will increase the sensitivity of the method contributing to the estimation of the ink age for a longer period of time.

Materials and methods: In order to compare peak resolution between PE and derivatized PE, a solution of 50 µg/ml of PE was prepared in hexane. The derivatized sample was prepared using chemical derivatization with MSTFA:TMCS (95:5) at a 80 °C during 30 min. The samples were analysed using GC/MS with an Agilent Technologies 5973 – 6890 N. GC MEGA-5 MS; 0.25 µm, 0.32 mm, 30 m capillary column was used. Chromatographic analysis was carried out under the following conditions: injection volume 2 µl, split ratio of 2:1 and injection temperature at 280 °C. The oven temperature program starting at 90 °C during 8 min, then ramped at 10 °C min⁻¹ to 100 °C, and increased to 240 °C at a rate of 30 °C min⁻¹ during 4.67 min. The MS analysis was carried out in SIM mode, which *m/z* of PE was 77, 94, 138 and PE-TMS was 151, 195, 210.

Results: The chromatogram results (Figure 1) showed that PE-TMS has a longer retention time (23.2 min) and a better resolution than PE (retention time of 13.2 min).

Discussion and conclusions: This preliminary study showed an increase of the sensitivity of the PE-TMS. Our results revealed that PE derivatization with MSTFA:TMCS, might be useful in ink dating determination and can contribute to the decreasing of LOQ. However, more work has to be done. In conclusion, derivatization proves to be promising in the field of ink ageing, allowing getting sensitivity to estimate the age of an ink, in a long period of time.

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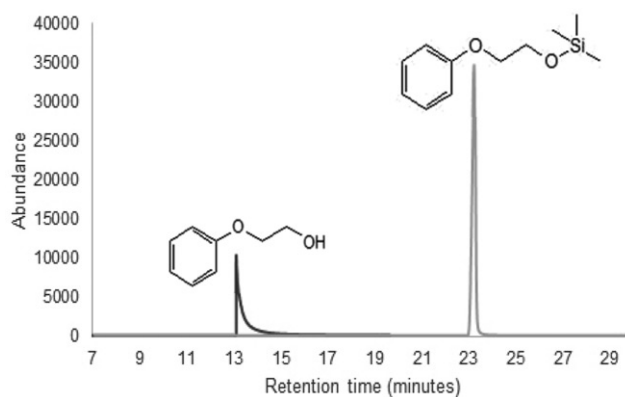


Figure 1. GC-MS chromatogram of PE (tr = 13.2) and PE-TMS (tr = 23.2) carried out in SIM mode using a GC MEGA-5 MS; 0.25 μ m, 0.32mm, 30 m capillary column.

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Assessing the content of a package of SGT-151 sold online

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ABSTRACT

Introduction: Synthetic cannabinoids (SCs) are novel psychoactive substances that mimic the effects of cannabis [1–3]. These products were sold *via* online shops and consisted of herbal mixtures sprayed with SCs [3]. Since then, a deluge of chemical variations of SCs has been occurring worldwide due to their synthesis in clandestine laboratories, often based on pharmaceutical research and patents, posing a growing challenge for authorities regarding regulation of these substances [1,3]. More recently, several highly potent compounds have emerged on the drug market, synthesised as stated in the patent application of Bowden and Williamson (“SGT-compounds”). These drugs are characterised by a cumyl substituent, which is attached to an indole, indazole or azaindole structure. One of the first cumyl-derivatives, CUMYL-PEGACLONE, was found in 2016 on the German drug market, being sold under the street name SGT-151 [1,2]. The present study aims to understand what is inside of a SGT-151 package sold in the internet as a ‘research chemical’.

Materials and methods: The cannabinoid identification was based on its mass spectra using the Cayman database (Chemical C. Cayman Spectral Library, vol. v08302018). GC/MS was the technique used to analyse the compound using a MEGA-5 MS capillary column (0.25 μ m, 0.32 mm, 30m). Chromatographic analysis was carried out under the following conditions: injection volume 1 μ L and splitless injection at 280 $^{\circ}$ C. The initial oven temperature was 100 $^{\circ}$ C for 3 min, ramped to 310 $^{\circ}$ C at a rate of 30 $^{\circ}$ C/min and held at 310 $^{\circ}$ C for 10 min. The MS conditions were as follows: ionisation energy was set at 70 eV; acquisition was carried out in a scan mode range of *m/z* 30–450. Helium was used as the carrier gas. For purification, a HPLC/DAD, operated by Clarity software, was used with a reversed-phase column. The mobile phase was a solvent gradient system consisting of (A) 5% 10 mM ammonia format and (B) 95% acetonitrile, optimised to achieve the best resolution. The results were recorded at $\lambda = 252$ nm.

Results: Figure 1(A) shows the GC/MS chromatogram of SGT-151. It is clear the presence of a peak corresponding to SGT-151 and several other peaks from other compounds. It was not possible to identify the remaining peaks. Figure 1(B) shows a GC/MS chromatogram after purification of the package content.