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Final report

Screening study on hazardous substances in marine mammals of the Baltic Sea

Wide-scope target and suspect screening

by: Jaroslav Slobodnik1, Georgios Gkotsis2, Maria-Christina Nika2, Konstantinos Vasilatos2, Nikolaos S. Thomaidis2, Nikiforos Alygizakis1,2, Peter Oswald1, Simon Rohner3, Ursula Siebert3. Farina Reif4, Michael Dähne4, Sara Persson5, Anders Galatius6, Iwona Pawliczka7, Anita Künitzer8

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Jaroslav Slobodnik[1], Georgios Gkotsis[2], Maria-Christina Nika[2], Konstantinos Vasilatos[2], Nikolaos S. Thomaidis[2], Nikiforos Alygizakis[1,2], Peter Oswald[1], Simon Rohner[3], Ursula Siebert[3]. Farina Reif[4], Michael Dähne[4], Sara Persson[5], Anders Galatius[6], Iwona Pawliczka[7], Anita Künitzer[8]
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Abstract: Screening study on hazardous substances in marine mammals of the Baltic Sea

As stated by HELCOM Expert Group on Marine Mammals (EG MAMA; portal.helcom.fi, 2021), limited information is available on the occurrence, (eco)toxicity, and potential health effects of contaminants of emerging concern (CECs) in marine mammals. CECs are introduced into the environment by various anthropogenic activities andsome of these substances may have the potential to enter marine, freshwater and/or terrestrial food webs, where they can accumulate. Currently, exposure information is often missing and there is an urgent need for sufficient occurrence and effects data to be able to assess CECs and initiate risk mitigation measures where appropriate.

The objective of the project was to screen for potentially hazardous CECs in marine mammals from the Baltic Sea using state-of-the-art wide-scope target and suspect screening analytical methodologies. For this purpose, 11 pooled livers and one non-pooled muscle sample from 11 marine mammals' samples (Harbour porpoise (*Phocoena phocoena*), Common dolphin (*Delphinus delphis*), Grey seal (*Halichoerus grypus*), Harbor seal (*Phoca vitulina*)) were provided by HELCOM contracting parties from Germany, Sweden, Denmark and Poland.

The contaminants of interest were extracted from the freeze-dried matrices through generic methods of extraction and the final extracts were analyzed by both liquid and gas chromatography coupled to high resolution mass spectrometry (HRMS; LC-ESI-QToF and GC-APCI-QToF). The samples were quantitatively screened for the presence of more than 2,500 organic pollutants including compounds of different classes such as pharmaceuticals, personal care products, biocides, plant protection products, illicit drugs, stimulants, sweeteners, and industrial chemicals (e.g. per- and polyfluoroalkyl substances (PFASs), flame retardants, corrosion inhibitors, plasticizers, surfactants), as well as their transformation products (TPs). Additionally, a method using different sample preparation procedure for analysis of 23 compounds contained in explosives dumped historically into the Baltic Sea has been developed. A specific target method using the same sample preparation has been applied for 13 novel organophosphorous flame retardants (OPFRs) and two dechlorane-plus compounds as well.

Suspect screening of 65,690 environmentally relevant substances from the NORMAN Substance Database was performed in all raw HRMS chromatograms. The chromatograms were also uploaded into the NORMAN Digital Sample Freezing Platform (DSFP), and thus made available for retrospective screening of even more compounds as the information allowing for their screening becomes available.

Overall, 47 contaminants from different chemical classes were determined in the analyzed samples. Most of the detected compounds were PFAS, followed by plant protection products & their TPs, industrial chemicals and pharmaceuticals & their TPs. The most predominant compounds were PCB 101, l-PFOS, hexachlorobenzene and 4,4-DDE (TP of DDT), which were detected in all studied samples. The measured concentration levels of individual substances were benchmarked against their Predicted No-effect Concentration (PNEC) values for marine fish retrieved from the NORMAN Ecotoxicology Database and 33 compounds exceeded these ecotoxicological threshold values, indicating potential adverse effects on the affected marine mammals health. None of the targeted explosives was detected above its Limit of Detection in any of the samples. Five OPFRs were determined in at least one sample, with tris(3-chloropropyl)phosphate being present in ten out of 12 samples.

The suspect screening revealed presence of additional 30 substances in the studied samples and allowed for semi-quantitative estimate of their concentrations. These compounds were then

prioritised following the same procedure as in the wide-scope target screening. As a result, the industrial chemicals 12-aminododecanoic acid and 1,3-dimethyl-3-phenylbutyl acetate were the top ranking substances followed by the UV filter octinoxate. The majority of the detected chemicals were registered in the ECHA database indicating their annual high tonnage production.

Kurzbeschreibung: Screening-Studie zu gefährlichen Stoffen in Meeressäugern der Ostsee

Wie die HELCOM-Expertengruppe für Meeressäugetiere (EG MAMA; portal.helcom.fi, 2021) feststellt, liegen nur begrenzte Informationen über das Vorkommen, die (Öko-)Toxizität und die potenziellen gesundheitlichen Auswirkungen von Neuen Schadstoffen bei Meeressäugern vor. Neue Schadstoffe werden durch verschiedene anthropogene Aktivitäten in die Umwelt eingebracht, und einige dieser Stoffe haben das Potenzial, in Meeres-, Süßwasser- und/oder terrestrische Nahrungsnetze zu gelangen, wo sie sich anreichern können. Gegenwärtig fehlen häufig Informationen über die Exposition, und es besteht ein dringender Bedarf an ausreichenden Daten zum Vorkommen und die Auswirkungen, um CEC bewerten und gegebenenfalls Maßnahmen zur Risikominderung einleiten zu können.

Ziel des Projekts war das Screening auf potenziell gefährliche Neue Schadstoffe in Meeressäugetieren aus der Ostsee unter Verwendung modernster analytischer Methoden für ein weitreichendes Ziel- und Verdachtsscreening. Zu diesem Zweck wurden 11 gepoolte Leber- und eine nicht gepoolte Muskelprobe von 11 Meeressäugern (Schweinswal (*Phocoena phocoena*), Gewöhnlicher Delphin (*Delphinus delphis*), Kegelrobbe (*Halichoerus grypus*), Seehund (*Phoca vitulina*)) von HELCOM-Vertragsparteien aus Deutschland, Schweden, Dänemark und Polen zur Verfügung gestellt.

Die interessierenden Verunreinigungen wurden aus den gefriergetrockneten Matrizes mit Hilfe allgemeiner Extraktionsmethoden extrahiert, und die endgültigen Extrakte wurden sowohl mit Flüssig- als auch mit Gaschromatographie in Verbindung mit hochauflösender Massenspektrometrie (HRMS; LC-ESI-QToF und GC-APCI-QToF) analysiert. Die Proben wurden quantitativ auf das Vorhandensein von mehr als 2,500 organischen Schadstoffen untersucht, darunter Verbindungen verschiedener Klassen wie Arzneimittel, Kosmetika, Biozide, Pflanzenschutzmittel, illegale Drogen, Stimulanzien, Süßstoffe und Industriechemikalien (z. B. Per- und Polyfluoralkylsubstanzen (PFAS), Flammschutzmittel, Korrosionsinhibitoren, Weichmacher, Tenside) sowie deren Umwandlungsprodukte (TPs). Darüber hinaus wurde eine Methode zur Analyse von 23 Verbindungen entwickelt, die in Sprengstoffen enthalten sind, die in der Vergangenheit in die Ostsee verklappt wurden, wobei ein anderes Verfahren zur Probenvorbereitung verwendet wurde. Eine spezifische Ziel-Screeningmethode, die dieselbe Probenvorbereitung verwendet, wurde auch für 13 neue phosphororganische Flammschutzmittel (OPFR) und zwei Dechloran-plus-Verbindungen angewandt.Das Verdachtsscreening von 65.690 umweltrelevanten Substanzen aus der NORMAN-Stoffdatenbank wurde an allen HRMS-Rohchromatogrammen durchgeführt. Die Chromatogramme wurden auch in die NORMAN Digital Sample Freezing Platform (DSFP) hochgeladen und stehen somit für das retrospektive Screening von noch mehr Verbindungen zur Verfügung, sobald die Informationen für deren Screening verfügbar sind.

Insgesamt wurden in den untersuchten Proben 47 Schadstoffe aus verschiedenen chemischen Klassen festgestellt. Bei den meisten der nachgewiesenen Verbindungen handelte es sich um PFAS, gefolgt von Pflanzenschutzmitteln und deren TP, Industriechemikalien und Arzneimitteln und deren TP. Die am häufigsten vorkommenden Verbindungen waren PCB 101, l-PFOS, Hexachlorbenzol und 4,4-DDE (TP von DDT), die in allen untersuchten Proben nachgewiesen

wurden. Die gemessenen Konzentrationen der einzelnen Stoffe wurden mit den PNEC-Werten (Predicted No-Effect Concentration) für Meeresfische aus der NORMAN-Ökotoxikologie-Datenbank verglichen, und 33 Verbindungen überschritten diese ökotoxikologischen Schwellenwerte, was auf mögliche negative Auswirkungen auf die Gesundheit der betroffenen Meeressäuger hinweist. Keiner der untersuchten Sprengstoffe wurde in einer der Proben oberhalb seiner Nachweisgrenze nachgewiesen. Fünf OPFRs wurden in mindestens einer Probe nachgewiesen, wobei Tris(3-chlorpropyl)phosphat in zehn von 12 Proben vorhanden war.

Das Verdachtsscreening ergab das Vorhandensein von weiteren 30 Substanzen in den untersuchten Proben und ermöglichte eine halbquantitative Schätzung ihrer Konzentrationen. Diese Verbindungen wurden dann nach demselben Verfahren wie beim breit angelegten Ziel-Screening priorisiert. Das Ergebnis war, dass die Industriechemikalien 12-Aminododecansäure und 1,3-Dimethyl-3-phenylbutylacetat an erster Stelle standen, gefolgt von dem UV-Filter Octinoxat. Die meisten der entdeckten Chemikalien waren in der ECHA-Datenbank registriert, was auf ihre jährliche Produktion in großen Mengen hindeutet.

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ASE	Accelerated Solvent Extraction
APCI	Atmospheric Pressure Chemical Ionization
BQL	Below the Limit of Quantification
bbCID	broad-band Collision Induced Dissociation
DCTs	Data Collection Templates
DSFP	Digital Sample Freezing Platform
ESI	Electrospray Ionization
EI	Electron Impact Ionization
EQS	Environmental Quality Standard
FoA	Frequency of Appearance
FoE	Frequency of Exceedance
EoE	Extent of Exceedance
GC	Gas Chromatography
HR	High Resolution
LOD	Limit of Detection
LOQ	Limit of Quantification
LC	Liquid Chromatography
MS	Mass Spectrometry
MEC	Measured Environmental Concentration
NKUA	National and Kapodistrian University of Athens
NA	Not Available
ND	Not Detected
OPFRs	Organophosphorous flame retardants
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
PCPs	Personal Care Products
PFAS	Polyalkylfluorinated substances
Pharms	Pharmaceuticals
PNEC	Predicted No-effect Concentration
PPPs	Plant Protection Products
Q-ToF MS	Quadrupole Time of Flight Mass Spectrometer
QSAR	Quantitative Structure Activity Relationship
RTI	Retention Time Index
SDL	Screening Detection Limit
SPE	Solid Phase Extraction
SOP	Standard Operating Procedure
TPs	Transformation Products
UHPLC	Ultra High Performance Liquid Chromatography

ASE	Accelerated Solvent Extraction
WFD	Water Framework Directive
w.w.	Wet Weight

Summary

The pilot project "Screening study on hazardous substances in marine mammals of the Baltic Sea" has been initiated by the German Environment Agency the in collaboration with the NORMAN Association. A screening of several thousands of organic pollutants and their transformation products has been conducted by wide-scope target (2,540 substances) and suspect (65,960 substances) screening methodologies. For this purpose, five HELCOM contracting parties delivered 12 pooled or individual samples from 43 marine mammals' specimens, gathered from different locations of the Baltic Sea. The detected organic pollutants were prioritised as potential Baltic Sea Specific Contaminants on the basis of their frequency of occurrence in the samples and exceedance of ecotoxicological threshold values.

Results of this study are planned to be used as basis for further work regarding assessment of contaminant pollution of marine mammals in the Baltic Sea and health status of marine mammals in general (portal.helcom.fi, 2021). The assessment also addressed an extensive number of substances that are not covered by the existing selection of 11 HELCOM indicators on the topic of hazardous substances. The approach therefore has the ability both to support overall status assessments (e.g. HOLAS III) and work in relation to other policies (e.g. the EU Marine Strategy Framework Directive (MSFD)).

A wide-scope target and suspect screening of the samples was carried out by LC-ESI-HR-MS and GC-APCI-HR-MS techniques. Additionally, a GC-EI-MS method has been developed for the determination of 23 compounds contained in explosives formerly dumped into the Baltic Sea. A specific study on 13 organophosphorous flame retardants and two chlordane-plus compounds has been carried out. The suspect screening provided information on presence/absence of the suspect substances and semi-quantitative estimate of detected substances.

Overall, 47 contaminants were determined through wide-scope target analysis. Most of the detected compounds were per- and polyfluoroalkyl substances (PFAS) (23.4%), followed by plant protection products & TPs (19.1%), industrial chemicals (19.1%), pharmaceuticals & TPs (8 17.0%) and the group of personal care products, stimulants & TPs and preservatives (21.3%). The samples were dominated by presence of old legacy compounds such as PCB 101, hexachlorobenzene and 4,4-DDE (TP of DDT), PCB 138, PCB 153 and 4,4-DDD (TP of DDT).

PFAS (PFDeA and the substituted (branched) isomers of PFOS), as well as methylparaben (anti-fungal agent often used in a variety of cosmetics and personal care products) were present in the majority of samples. Among the detected contaminants, the highest concentration levels were observed for l-PFOS (mean concentration 334 µg/kg w.w.), methylparaben (mean concentration 296 µg/kg w.w.), N-butylbenzenesulfonamide (mean concentration 469 µg/kg w.w.) and the surfactant and antiseptic compound benzododecinium (benzyl-dimethyl-dodecylammonium; 272 µg/kg w.w.).

Additionally, 11 PFAS were determined in the studied samples. The cumulative PFAS concentration was ranging from 179 (UBA-HELCOM 3a) to 1143 (UBA-HELCOM 1) µg/kg w.w. in the liver samples, whereas in the only analyzed muscle sample (UBA-HELCOM 3b) the total concentration was 5.12 µg/kg w.w. and the number of the detected PFAS was significantly lower (n = 4). Moreover, five PCBs were detected in almost all tested samples. The sample with the highest total concentration of PCBs (745 µg/kg w.w.) was UBA-HELCOM 2 (liver of Harbour Porpoise, Germany, sampling years; 2016, 2017, 2020). The lowest total concentrations of PCBs

were observed in the Danish samples (mean ∑PFAS; 37.9 µg/kg w.w.), whereas in Germany, Sweden and Poland the mean cumulative concentrations were comparable. Additionally, the long-term banned organochlorine insecticide 2,4-DDT and its two TPs 4,4-DDD and 4,4-DDE were frequently determined. The total concentration levels were significantly higher in the two harbour porpoise samples from Germany (UBA-HELCOM 2 and 10; 255 and 245 µg/kg w.w., respectively) in comparison with the rest of liver samples (mean concentration; 45.8 µg/kg w.w.).

As a proof of 'screening' concept, high concentrations of specific pharmaceuticals used for treatment of marine mammals (baytril, enrofloraxin) were revealed in one of the pooled samples (UBA-HELCOM 10, Germany). It has been retrospectively found out that one of the stranded dolphins whose liver had been subjected for analysis was indeeed treated with pharmaceuticals identified by this independent screening (https://www.ndr.de/nachrichten/schleswig-holstein/Delfin-aus-Eckernfoerder-Bucht-ist-tot,delfin350.html).

Furthermore, 11 (bio)transformation products [(bio)TPs] of emerging contaminants were detected in the samples, including three TPs of the analgesic drug Tramadol: Nor-Tramadol (N-desmethyl-Tramadol), O-Desmethyl-Tramadol, O-Desmethyldinor-Tramadol; two TPs of Nicotine: Anabasine and Nor-Nicotine; and the metabolite of Gabapentin: 3,3-Pentamethylene-butyrolactam.

With regard to a limited number of samples only a simplified risk assessment of individual contaminants could be carried out based on exceedance of available toxicity threshold values. PNEC values for biota were derived from existing PNECs for freshwater (PNECfw; available in the NORMAN Ecotoxicity Database for 64,447 NORMAN SusDat compounds; see also https://www.norman-network.com/nds/ecotox/).

The detected substances were prioritised based on three indicators: (i) Frequency of Appearance (FoA); (ii) Frequency of PNEC Exceedance (FoE), and (iii) Extent of PNEC Exceedance (EoE). According to the NORMAN Prioritisation Framework (Dulio, 2013), the first indicator expresses at how many sites the compound was detected above the limit of detection (LOD). The second indicator considers the frequency of monitoring sites with observations of a compound above a certain effect threshold. The third indicator ranks compounds with regard to the extent of the effects expected. The Risk Score is the linear combination of the indicators scaled from 0 to 1.

Analyses of 11 (pooled) liver samples of marine mammals revealed the presence of 33 compounds, which exceeded their ecotoxicological threshold value in at least one sample. Most of the compounds exceeded their PNEC values (FoE) in less than four samples.

4,4-DDE, the stable metabolite of DDT, PCB 101, l-PFOS and PFDeA seem to be of high environmental concern, as their concentrations exceeded the respective PNEC values in all tested samples. More PCB congeners (PCB 138 and 153) and the personal care product methylparaben exceeded their ecotoxicological threshold in ten samples, whereas PCB 180 and 52 were detected at concentration levels above their PNECs in nine samples.

For the majority of compounds (64%) the maximum detected concentrations were less than 100-fold higher than their ecotoxicological thresholds, whereas for seven compounds (4,4-DDE, dieldrin and PCBs 52, 101, 138, 153 and 180) the maximum reported concentration levels were more than three orders of magnitude higher than their respective PNECs, indicating a potential high environmental risk.

Each sample was tested for the presence of 23 explosives (list provided by UBA). None of the studied substances has been detected in any of the samples above its LOD.

Five OPFRs were determined in at least one sample, with tris(3-chloropropyl)phosphate being present in ten out of 12 samples. This is of concern, since OPFRs are a possible replacement of the already banned polybrominated diphenyl ethers, are persistent and their cooncentrations may rise in future.

The samples were screened for presence of 65,690 substances. Next to the substances determined by wide-scope target screening, additional 32 contaminants were detected**.** The majority of the detected substances are widely used and produced in or imported to Europe. 21 out of the 32 chemicals were classified as industrial chemicals and all of them are registered in the ECHA database. The detected substances, which are known to be produced at a very high tonnage (10000-100000 t/a) were: acetyl tributyl citrate, erucamide, octanedioic, 10-undecenoic acid, pentyl ester and nonanedioic acid. Substances falling into the high tonnage band (100-1000 t/a) were diethylene glycol monophenyl ether , 12-aminododecanoic acid and 3,9-dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecane. Substances produced at 10-100 t/a were 2-propen-1-yl 2-(cyclohexyloxy)acetate and 1,3-dimethyl-3-phenylbutyl acetate. All other industrial chemicals were reported either as produced in the low tonnage band (1-10 t/a) or this information was not accessible. The next most important class of detected substances was that of pharmaceuticals (nine compounds). Finally, the UV filter octinoxate was detected at level 2A (tentative identification with library spectrum match).

A simplified risk assessment was conducted using the same methodology as in the wide-scope target screening, the only difference being that suspect screening produced semi-quantitative concentration levels based on the structural most similar compound from among a set of internal standard compounds. The purpose of the risk assessment was to rank the detected suspects based on the exceedance of their toxicity threshold values and FoA.

In addition to the three risk assessment scores (see Section 7.1.1), the Exposure Score developed by KEMI, Sweden, was used to confirm the relevance of a compound. KEMI score is based on normalised values (between 0-1) reflecting (i) the degree of uncontrolled release during use, (ii) annual tonnage and (iii) range of use on the market. This index (value ranging from 0 to 1) was higher than 0.3 for 17 compounds, indicating that these top-ranked substances are produced in large annual tonnage with widespread use.

All LC-HR-MS and GC-APCI-HR-MS chromatograms were uploaded into the NORMAN DSFP and thus are available for future retrospective screening for any compound detectable by those techniques without the need for additional sampling and analysis. An access to these data is restricted only to the persons identified as eligible by UBA Germany or HELCOM Expert Group on Marine Mammals.

The wide-scope screening data organised in NORMAN Data Collection Templates were uploaded into the LIFE APEX Database System (https://www.norman-network.com/apex/lacod/; a part of the NORMAN Database System), currently accessible only to the project partners (including UBA) and sample providers. A dedicated area was created accessible only to the persons identified as eligible by UBA Germany or HELCOM Expert Group on Marine Mammals. In the end of the LIFE APEX project the data are planned to be transferred to the open access NORMAN Database System – EMPODAT database (https://www.norman-network.com/nds/empodat/), which is sharing its content with the EC IPCHEM on a regular basis.

A platform for interactive visual presentation of the results of the wide-scope target and suspect screening of UBA-HELCOM samples has been developed. It is accessible at https://norman-data.eu/UBA-HELCOM/

It is recommended to systematically store data from further screening campaigns of HELCOM countries in the NORMAN Database System (https://www.norman-network.com/nds/), which would allow for their review in comparison with data from other European countries and North America. The occurrence data of this project are now fully comparable to the LIFE APEX project (https://lifeapex.eu/), where samples from marine mammals from several European sea regions are being analyzed following the identical analytical protocols. Also, it is recommended to encourage HELCOM Expert Group on Marine Mammals to provide NORMAN with commonly agreed biota ecotoxicity threshold values (PNECs) for as many substances as possible. This is to facilitate more precise prioritisation of the contaminants detected in biota samples in the Baltic Sea based on the exceedance of these values. Strategies of contaminant analyses should be compared between OSPAR and HELCOM where, additionally to liver, blubber is adviced to be analysed.

Additional efforts are taking place within NORMAN to develop a specific prioritisation scheme taking into account model-predicted PBT (persistence, bioaccumulation, toxicity) values for all substances listed in the Substance Database (https://www.norman-network.com/nds/susdat/). Once ready (expected in the end of 2021), they can be re-applied on the substances identified in the analyzed samples in the current screening/monitoring programmes. Also, there is an on-going discussion with European Chemicals Agency (ECHA) to increase the importance of environmental occurrence data in the substance evaluation scheme and receiving feedback which of the REACH substances (including their transformation products) might be preferably targeted in the updated WFD and MSFD monitoring schemes.

Zusammenfassung

Das Pilotprojekt "Screening-Studie zu gefährlichen Stoffen in Meeressäugern der Ostsee" wurde vom Umweltbundesamt in Zusammenarbeit mit dem NORMAN Netzwerk initiiert. Ein Screening von mehreren tausend organischen Schadstoffen und deren Umwandlungsprodukten wurde mit Hilfe von breit angelegten Ziel- (2.540 Substanzen) und Verdachts-Screeningmethoden (65.960 Substanzen) durchgeführt. Zu diesem Zweck lieferten fünf HELCOM-Vertragsparteien 12 Poolproben aus Einzelproben von 43 Meeressäugern, die an verschiedenen Stellen der Ostsee gesammelt wurden. Die gefundenen organischen Schadstoffe wurden aufgrund der Häufigkeit ihres Auftretens in den Proben und der Überschreitung ökotoxikologischer Schwellenwerte als potenzielle ostseespezifische Schadstoffe eingestuft.

Die Ergebnisse dieser Studie sollen als Grundlage für weitere Arbeiten zur Bewertung der Schadstoffbelastung der Meeressäuger in der Ostsee und des Gesundheitszustands der Meeressäuger im Allgemeinen dienen (portal.helcom.fi, 2021). Bei der Bewertung wurde auch eine große Anzahl von Stoffen berücksichtigt, die nicht durch die bestehende Auswahl von 11 HELCOM-Indikatoren zum Thema gefährliche Stoffe abgedeckt sind. Der Ansatz eignet sich daher sowohl zur Unterstützung von allgemeinen Zustandsbewertungen (z. B. HOLAS III) als auch für die Arbeit im Zusammenhang mit anderen Politiken (z. B. der EU-Meeresstrategie-Rahmenrichtlinie (MSRL)).

Ein umfassendes Ziel- und Verdachtsscreening der Proben wurde mit LC-ESI-HR-MS- und GC-APCI-HR-MS-Techniken durchgeführt. Darüber hinaus wurde eine GC-EI-MS-Methode für die Bestimmung von 23 Verbindungen entwickelt, die in Sprengstoffen enthalten sind, die früher in Munition in die Ostsee versenkt wurden Es wurde eine spezielle Studie zu 13 phosphororganischen Flammschutzmitteln und zwei Chlordan-plus-Verbindungen durchgeführt. Das Verdachtsscreening lieferte Informationen über das Vorhandensein bzw. Nichtvorhandensein der verdächtigen Stoffe und eine halbquantitative Schätzung der nachgewiesenen Stoffe.

Insgesamt wurden 47 Schadstoffe durch eine breit angelegte Ziel-Screeninganalyse ermittelt. Bei den meisten nachgewiesenen Verbindungen handelte es sich um Per- und Polyfluoralkylsubstanzen (PFAS) (23,4 %), gefolgt von Pflanzenschutzmitteln und TP (19,1 %), Industriechemikalien (19,1 %), Arzneimitteln und TP (17,0 %) und der Gruppe der Kosmetika, Genussmittel und TP sowie Konservierungsmittel (21,3 %). In den Proben waren vor allem alte Schadstoffe wie PCB 101, Hexachlorbenzol und 4,4-DDE (TP von DDT), PCB 138, PCB 153 und 4,4-DDD (TP von DDT) enthalten.

PFAS (PFDeA und die substituierten (verzweigten) Isomere von PFOS) sowie Methylparaben (ein Mittel gegen Pilzbefall, das häufig in einer Vielzahl von Kosmetika und Körperpflegeprodukten verwendet wird) waren in den meisten Proben vorhanden. Unter den nachgewiesenen Kontaminanten wurden die höchsten Konzentrationen für l-PFOS (mittlere Konzentration 334 µg/kg Feuchtgewicht), Methylparaben (mittlere Konzentration 296 µg/kg Feuchtgewicht), N-Butylbenzolsulfonamid (mittlere Konzentration 469 µg/kg Feuchtgewicht) und das Tensid und die antiseptische Verbindung Benzododecinium (Benzyl-Dimethyl-Dodecylammonium; 272 µg/kg Feuchtgewicht) festgestellt.

Zusätzlich wurden 11 PFAS in den untersuchten Proben bestimmt. Die kumulative PFAS-Konzentration lag in den Leberproben zwischen 179 (UBA-HELCOM 3a) und 1143 (UBA-HELCOM 1) µg/kg Feuchtgewicht, während in der einzigen untersuchten Muskelprobe (UBA-HELCOM 3b) die Gesamtkonzentration 5,12 µg/kg Feuchtgewicht betrug und die Anzahl der nachgewiesenen PFAS deutlich niedriger war (n = 4). Außerdem wurden fünf PCB in fast allen untersuchten Proben nachgewiesen. Die Probe mit der höchsten Gesamtkonzentration an PCB (745 µg/kg

Feuchtgewicht) war UBA-HELCOM 2 (Leber von Schweinswalen, Deutschland, Probenahmejahre; 2016, 2017, 2020). Die niedrigsten PCB-Gesamtkonzentrationen wurden in den dänischen Proben festgestellt (mittlerer ∑PFAS; 37,9 µg/kg Feuchtgewicht), während in Deutschland, Schweden und Polen die mittleren kumulativen Konzentrationen vergleichbar waren. Auch das seit langem verbotene Organochlorin-Insektizid 2,4-DDT und seine beiden TPs 4,4-DDD und 4,4-DDE wurden häufig bestimmt. Die Gesamtkonzentrationen waren in den beiden Schweinswalproben aus Deutschland (UBA-HELCOM 2 und 10; 255 bzw. 245 µg/kg Feuchtgewicht) signifikant höher als in den übrigen Leberproben (mittlere Konzentration; 45,8 µg/kg Feuchtgewicht).

Als Beweis für das "Screening"-Konzept wurden in einer der gepoolten Proben (UBA-HELCOM 10, Deutschland) hohe Konzentrationen spezifischer Arzneimittel für die Behandlung von Meeressäugern (Baytril, Enrofloraxin) nachgewiesen. Im Nachhinein wurde festgestellt, dass einer der gestrandeten Delfine, dessen Leber untersucht wurde, tatsächlich mit Arzneimitteln behandelt worden war, die bei diesem unabhängigen Screening identifiziert wurden (https://www.ndr.de/nachrichten/schleswig-holstein/Delfin-aus-Eckernfoerder-Bucht-ist-tot,delfin350.html).

Darüber hinaus wurden in den Proben 11 (Bio-)Transformationsprodukte [(Bio-)TPs] von Neuen Schadstoffen nachgewiesen, darunter drei TPs des Schmerzmittels Tramadol: Nor-Tramadol (N-Desmethyl-Tramadol), O-Desmethyl-Tramadol, O-Desmethyldinor-Tramadol; zwei TPs von Nikotin: Anabasin und Nor-Nikotin; und der Metabolit von Gabapentin: 3,3-Pentamethylen-Butyrolactam.

Wegen der begrenzten Anzahl von Proben konnte nur eine vereinfachte Risikobewertung einzelner Schadstoffe auf der Grundlage der Überschreitung von verfügbaren Toxizitätsschwellenwerten durchgeführt werden. PNEC-Werte für Biota wurden von bestehenden PNECs für Süßwasser abgeleitet (PNECfw; verfügbar in der NORMAN Ecotoxicity Database für 64.447 NORMAN SusDat Verbindungen; siehe auch https://www.norman-network.com/nds/ecotox/).

Die ermittelten Stoffe wurden anhand von drei Indikatoren priorisiert: (i) Häufigkeit des Auftretens (FoA), (ii) Häufigkeit der PNEC-Überschreitung (FoE) und (iii) Ausmaß der PNEC-Überschreitung (EoE). Gemäß dem NORMAN-Priorisierungsrahmen (Dulio, 2013) gibt der erste Indikator an, an wie vielen Standorten die Verbindung oberhalb der Nachweisgrenze (LOD) nachgewiesen wurde. Der zweite Indikator berücksichtigt die Häufigkeit der Überwachungsstandorte, an denen eine Verbindung oberhalb einer bestimmten Wirkungsschwelle beobachtet wurde. Der dritte Indikator ordnet die Verbindungen im Hinblick auf das Ausmaß der zu erwartenden Auswirkungen ein. Der Risikowert ist die lineare Kombination der Indikatoren auf einer Skala von 0 bis 1.

Die Analyse von 11 (gepoolten) Leberproben von Meeressäugern ergab das Vorhandensein von 33 Verbindungen, die ihren ökotoxikologischen Schwellenwert in mindestens einer Probe überschritten. Die meisten der Verbindungen überschritten ihren PNEC-Wert (FoE) in weniger als vier Proben.

4,4-DDE, der stabile Metabolit von DDT, PCB 101, l-PFOS und PFDeA scheinen für die Umwelt besonders besorgniserregend zu sein, da ihre Konzentrationen in allen untersuchten Proben die jeweiligen PNEC-Werte überschritten. Weitere PCB-Kongenere (PCB 138 und 153) und das Körperpflegemittel Methylparaben überschritten in zehn Proben ihren ökotoxikologischen Schwellenwert, während PCB 180 und 52 in neun Proben in Konzentrationen oberhalb ihrer PNEC-Werte nachgewiesen wurden.

Bei den meisten Verbindungen (64 %) lagen die maximal festgestellten Konzentrationen weniger als 100-fach über den ökotoxikologischen Schwellenwerten, während bei sieben Verbindungen

(4,4-DDE, Dieldrin und PCB 52, 101, 138, 153 und 180) die maximal festgestellten Konzentrationen mehr als drei Größenordnungen über den jeweiligen PNEC-Werten lagen, was auf ein potenziell hohes Umweltrisiko hinweist.

Jede Probe wurde auf das Vorhandensein von 23 Explosivstoffen untersucht (Liste vom UBA zur Verfügung gestellt). Keiner der untersuchten Stoffe wurde in einer der Proben oberhalb seines LOD-Wertes nachgewiesen.

Fünf OPFRs wurden in mindestens einer Probe nachgewiesen, wobei Tris(3-chlorpropyl)phosphat in zehn von 12 Proben vorhanden war. Dies ist besorgniserregend, da OPFRs ein möglicher Ersatz für die bereits verbotenen polybromierten Diphenylether sind, persistent sind und ihre Konzentrationen in Zukunft ansteigen könnten.

Die Proben wurden auf das Vorhandensein von 65.690 Stoffen untersucht. Neben den Stoffen, die im Rahmen eines breit angelegten Zielscreenings ermittelt wurden, konnten weitere 32 Schadstoffe nachgewiesen werden. Die meisten der entdeckten Stoffe werden in großem Umfang verwendet und in Europa hergestellt oder nach Europa eingeführt. 21 der 32 Chemikalien wurden als Industriechemikalien eingestuft und sind alle in der Datenbank der ECHA registriert. Bei den nachgewiesenen Stoffen, die bekanntermaßen in sehr großen Mengen (10.000-100.000 t/a) hergestellt werden, handelte es sich um: Acetyltributylcitrat, Erucamid, Octandisäure, 10-Undecensäure, Pentylester und Nonandisäure. Stoffe, die in den oberen Mengenbereich (100-1000 t/a) fallen, sind Diethylenglykolmonophenylether, 12-Aminododecansäure und 3,9-Dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecan. Bei den mit 10-100 t/a produzierten Stoffen handelte es sich um 2-Propen-1-yl 2-(cyclohexyloxy)acetat und 1,3-Dimethyl-3-phenylbutylacetat. Alle anderen Industriechemikalien wurden entweder als im unteren Mengenbereich (1-10 t/a) hergestellt gemeldet oder diese Informationen waren nicht zugänglich. Die nächstgrößere Klasse der nachgewiesenen Stoffe war die der Arzneimittel (neun Verbindungen). Schließlich wurde der UV-Filter Octinoxat auf Stufe 2A nachgewiesen (vorläufige Identifizierung mit Übereinstimmung des Spektrums der Bibliothek).

Es wurde eine vereinfachte Risikobewertung durchgeführt, wobei dieselbe Methodik wie beim Ziel-Screening angewandt wurde, mit dem einzigen Unterschied, dass das Screening auf Verdachtsstoffe halbquantitative Konzentrationswerte auf der Grundlage der strukturell ähnlichsten Verbindung aus einer Reihe interner Standardverbindungen ergab. Der Zweck der Risikobewertung bestand darin, die entdeckten Verrdachtsstoffe auf der Grundlage der Überschreitung ihrer Toxizitätsschwellenwerte und FoA in eine Rangfolge zu bringen.

Zusätzlich zu den drei Risikobewertungs-Scores (siehe Abschnitt 7.1.1) wurde der von KEMI, Schweden, entwickelte Expositions-Score verwendet, um die Relevanz einer Verbindung zu bestätigen. Der KEMI-Score basiert auf normalisierten Werten (zwischen 0 und 1), die (i) den Grad der unkontrollierten Freisetzung während der Verwendung, (ii) die jährliche Tonnage und (iii) den Verwendungsbereich auf dem Markt widerspiegeln. Dieser Index (Wert zwischen 0 und 1) war bei 17 Verbindungen höher als 0,3, was darauf hindeutet, dass diese am höchsten eingestuften Stoffe in großen jährlichen Mengen hergestellt und weit verbreitet verwendet werden.

Alle LC-HR-MS- und GC-APCI-HR-MS-Chromatogramme wurden in das NORMAN DSFP hochgeladen und stehen somit für künftige retrospektive Untersuchungen auf alle mit diesen Techniken nachweisbaren Verbindungen zur Verfügung, ohne dass zusätzliche Probenahmen und Analysen erforderlich sind. Der Zugang zu diesen Daten ist nur den Personen vorbehalten, die vom UBA Deutschland oder der HELCOM-Expertengruppe für Meeressäugetiere als berechtigt ausgewiesen wurden.

Die in NORMAN-Datensammelvorlagen organisierten weitreichenden Screening-Daten wurden in das LIFE APEX-Datenbanksystem (https://www.norman-network.com/apex/lacod/; ein Teil des NORMAN-Datenbanksystems) hochgeladen, das derzeit nur den Projektpartnern (einschließlich des UBA) und den Probenlieferanten zugänglich ist. Es wurde ein spezieller Bereich eingerichtet, der nur für die Personen zugänglich ist, die vom UBA Deutschland oder der HELCOM-Expertengruppe für Meeressäugetiere als geeignet eingestuft wurden. Am Ende des LIFE APEX-Projekts ist geplant, die Daten in die frei zugängliche Datenbank NORMAN Database System - EMPODAT (https://www.norman-network.com/nds/empodat/) zu übertragen, die ihren Inhalt regelmäßig mit der EC IPCHEM teilt.

Es wurde eine Plattform für die interaktive visuelle Darstellung der Ergebnisse des breit angelegten Ziel- und Verdachtsscreenings von UBA-HELCOM-Proben entwickelt. Sie ist unter https://norman-data.eu/UBA-HELCOM/ zugänglich.

Es wird empfohlen, die Daten aus weiteren Screening-Kampagnen der HELCOM-Länder systematisch im NORMAN-Datenbanksystem (https://www.norman-network.com/nds/) zu speichern, was eine Überprüfung im Vergleich mit Daten aus anderen europäischen Ländern und Nordamerika ermöglichen würde. Die Vorkommensdaten dieses Projekts sind nun vollständig mit dem LIFE APEX-Projekt (https://lifeapex.eu/) vergleichbar, bei dem Proben von Meeressäugern aus mehreren europäischen Meeresregionen nach denselben Analyseprotokollen untersucht werden. Außerdem wird empfohlen, die HELCOM- Expert Group on Marine Mammals zu ermutigen, NORMAN gemeinsam vereinbarte Schwellenwerte für die Biota-Ökotoxizität (PNECs) für so viele Stoffe wie möglich zur Verfügung zu stellen. Dies soll eine genauere Priorisierung der in Biota-Proben in der Ostsee nachgewiesenen Schadstoffe auf der Grundlage der Überschreitung dieser Werte ermöglichen. Die Strategien der Schadstoffanalysen sollten zwischen OSPAR und HELCOM verglichen werden, wo zusätzlich zur Leber auch der Blubber analysiert werden sollte.

Im Rahmen von NORMAN werden zusätzliche Anstrengungen unternommen, um ein spezifisches Priorisierungsschema zu entwickeln, das die modellmäßig vorhergesagten PBT-Werte (Persistenz, Bioakkumulation, Toxizität) für alle in der Stoffdatenbank (https://www.norman-network.com/nds/susdat/) aufgeführten Stoffe berücksichtigt. Sobald sie fertig sind (voraussichtlich Ende 2021), können sie auf die Stoffe angewandt werden, die in den analysierten Proben im Rahmen der laufenden Screening- und Überwachungsprogramme identifiziert wurden. Außerdem laufen Gespräche mit der Europäischen Chemikalienagentur (ECHA), um die Bedeutung von Daten über das Vorkommen in der Umwelt im Stoffbewertungsschema zu erhöhen und Rückmeldungen darüber zu erhalten, welche REACH-Stoffe (einschließlich ihrer Umwandlungsprodukte) in den aktualisierten WRRL- und MSRL-Überwachungsprogrammen bevorzugt berücksichtigt werden könnten.

1 Introduction

In 2020, the German Environment Agency initiated the pilot project "Screening study on hazardous substances in marine mammals of the Baltic Sea" in collaboration with the NORMAN Association. Environmental Institute, Slovakia (EI), as one of the founding members of the NORMAN network, has been assigned to organise a specialized package of services, comprising screening of several thousands of organic pollutants and their transformation products by wide-scope target (>2,500 substances) and suspect (>65,000 substances) screening methodologies. For this purpose, five HELCOM contracting parties delivered 12 pooled or individual samples from 43 marine mammals' specimens to the Laboratory of Analytical Chemistry of the National and Kapodistrian University of Athens, Greece (NKUA), gathered from different locations of the Baltic Sea. The detected organic pollutants were prioritised on the basis of their frequency of occurrence in the samples and exceedance of ecotoxicological threshold values. The above services are being increasingly recognized by the EU and regional sea conventions as being of importance at identifying potential contaminant threats to the aquatic environment.

1.1 Background and problem definition

One priority under the German Chairmanship of HELCOM (1 July 2020 - 30 June 2022) is strengthening marine biodiversity. Marine biodiversity in the Baltic Sea has been subject to anthropogenic impairments. Contaminants are among the main threats for marine mammal populations in the Baltic Sea (portal.helcom.fi, 2021). For example, reduced fertility of seals and population decline of certain seal species have been attributed to high levels of organochlorines such as DDT and PCBs especially around the 1970/80s. It is now planned to screen marine mammals for a range of known and novel pollutants and link these data to the state of their health. As top predators in the food webs of the Baltic Sea, the state of their health needs to be enhanced to ensure their conservation.

1.2 Aim and content of the project

Germany initiated a non-target and target screening study on hazardous substances in samples of marine mammals' tissue starting in early 2021. The chemical screening approach should be comparable to that used in the LIFE APEX project (https://lifeapex.eu/). Results of this study are planned to be used as basis for further work regarding assessment of contaminant pollution of marine mammals in the Baltic Sea and health status of marine mammals in general.

The assessment carried out using this screening approach provides an overview of the presence of an extensive number of hazardous contaminants of emerging concern (CEC) in marine mammals. The selection of wide-scope target substances that can be determined and suspect substances that can be screened for their semi-quantitative presence/absence in samples with this approach is at present >2,400 and >65,000, respectively. These numbers are steadily increasing. The list of substances covers a large number of substances of priority concern, identified through policy initiatives and research studies. The assessment also addressed an extensive number of substances that are not covered by the existing selection of 11 HELCOM indicators on the topic of hazardous substances. The approach therefore has the ability both to support overall status assessments (e.g. HOLAS III) and work in relation to other policies (e.g. the EU Marine Strategy Framework Directive (MSFD)).

To ensure a sound, comparable and up to date database on contaminants in marine mammals with a good spatial coverage of the Baltic Sea area, cooperation with HELCOM contracting parties was striven for. It was expected that two samples per HELCOM contracting party could be analyzed

within the project. The kind of samples (species, tissue, quality of material) were to be defined according to availability and feedback from contracting parties.

Germany (UBA) presented its planned screening study on hazardous substances in marine mammals to the HELCOM EG MAMA 14-2020 meeting on 22 September as the document 2-4. Six contracting parties (SE, FI, DK, DE, PL and LT) indicated their interest to participate in the screening study and proposed contact persons. The remaining contracting parties were invited to nominate contact persons for providing samples for the study to UBA by 20 October 2020. The meeting discussed the focus of such screening studies. It was noted that the proposed sample size of two samples per country may not be sufficient for a comprehensive overview of all marine mammals and it would be useful to focus on one species with a pan-Baltic distribution, for example the grey seal. The meeting agreed that Germany will prepare a more detailed document of what kind of information is needed from to-be-provided samples and send it to those parties providing samples. A procedure how to collect and prepare samples, as well as how to transport them to the laboratory, has been developed and tested by the LIFE APEX project and detailed information was provided to the participating contracting parties (cf. **Appendix A**).

Preliminary results were presented at the HELCOM MAMA-15 meeting on 14 – 16 September 2021 (portal.helcom.fi, 2021).

2 Objectives

The specific objectives of the study were as follows:

a) A wide-scope target screening analysis of each sample for ca. 2,500 substances by LC-ESI-HR-MS and GC-APCI-HR-MS;
b) Suspect screening of 65,000+ compounds in each of the samples including their semi-quantification using LC-ESI-HR-MS;
c) A target analysis of 23 explosive compounds by GC-EI-MS;
d) Upload of the LC-HR-MS and GC-APCI-HR-MS chromatograms into NORMAN Digital Sample Freezing Platform (DSFP) for retrospective screening;
e) Prioritisation of detected compounds in order to assess results of the screening for environmental toxicological relevance;
f) Report on the occurrence of targeted substances including heatmaps on presence of suspect compounds.

3 Tasks/ technical requirements/ work packages

3.1 Work package 1: Samples

HELCOM Contracting parties provided sample tissue of marine mammals to the contractor. It was planned that each of the nine contracting parties provided two pooled samples, 18 samples in total. The species (grey seal, ringed seal, harbor seal or harbor porpoise) had to be agreed upon among Contracting parties, taking into consideration that the most widely distributed is the grey seal. Liver was the recommended matrix and it had been agreed upon that pooled samples were preferred. At the sampling, the decomposition status of the carcass needed to be considered; preferably specimens in state of preservation 1 and 2 (IJsseldijk et al., 2020) should be collected. Samples needed to be sampled with clean equipment, wrapped in aluminum foil and immediately frozen at -20°C (preferred to avoid contamination during sample pre-processing) or lyophilized.

In total 11 biota samples (marine mammals) consisting of 43 specimen from the Baltic Sea were delivered to NKUA in the period from December 2020 to May 2021. The species and sample codes are listed in **Table 1**. In order to investigate the variation of the chemical profile (detected contaminants/ concentration levels) within different matrices of analysis of the same organism, five sub-samples (liver, muscle, brain, blubber and kidney) of UBA-HELCOM 3 sample, were collected for research purposes. Results of analysis for liver and muscle are included in this report, however the applicability of the developed methodologies in the different matrices (brain, blubber and kidney) should still be tested and validated. These results were therefore not available for this report.

A guidance on sample preparation and shipment of samples (**Appendix A**) was communicated to HELCOM contracting parties by the German Environment Agency (UBA) in consultation with the contractor. Data Collection Templates (DCTs) for the collection of metadata developed by the EU LIFE APEX project were agreed upon to be used (**Appendix B**).

3.2 Work package 2: Analysis

The task of the contractor was to analyse biota samples by wide-scope target and suspect screening workflows. Analyses were carried out in the following steps (portal.helcom.fi, 2021).:

- STEP 1: Wide-scope target screening analysis (>2400 substances with LC-HR-MS and GC-APCI-HR-MS);
- STEP 2: Suspect screening (>65000 substances) and semi-quantification with LC-HR-MS with quality check;
- STEP 3: GC-APCI-HR-MS for retrospective screening for identification of unknowns.

The steps were based on the approach developed by the NORMAN Association, which has already been implemented by the LIFE APEX, OSPAR CONNECT and Black Sea EMBLAS projects. The analytical strategy has been approved for use in the HELCOM Pre-EMPT proposal of a wide-scope and suspect screening project of water, sediment and fish across the Baltic Sea to take place during 2021/2022. The list of substances screened for in this study (see Step 2 above) is available at https://www.norman-network.com/nds/susdat/index.php (only substances with Retention Time Index (RTI) and mass spectrometry information).

Based on the specific request by UBA, a dedicated method for traces of explosives in marine mammals has been developed (see details in Sections 4.3 and 5.3 below).

Table 1: List of samples.

Short code	Species	Matrix	Country	Year(s) of sampling	Provider	No. of specimens (labels by sample providers)
UBA-HELCOM 1	Harbour porpoise (*Phocoena phocoena*)	pooled liver	Germany	2015, 2018	Institute for Terrestrial and Aquatic Wildlife Research (ITAW)	4 (1, 4, 5, 8)
UBA-HELCOM 2	Harbour porpoise (*Phocoena phocoena*)	pooled liver	Germany	2016, 2017, 2020	Institute for Terrestrial and Aquatic Wildlife Research (ITAW)	4 (2, 3, 6, 7)
UBA-HELCOM 3a	Common dolphin (*Delphinus delphis*)	liver	Germany	2021	Institute for Terrestrial and Aquatic Wildlife Research (ITAW)	1 (9)
UBA-HELCOM 3b	Common dolphin (*Delphinus delphis*)	muscle	Germany	2021	Institute for Terrestrial and Aquatic Wildlife Research (ITAW)	1 (9)
UBA-HELCOM 4	Grey seal (*Halichoerus grypus*)	pooled liver	Sweden	2015, 2016, 2018	Swedish Museum of Natural History	5 (A2019/05101-05141, A2015/05551-05570, A2016/05631)
UBA-HELCOM 5	Grey seal (*Halichoerus grypus*)	pooled liver	Sweden	2015, 2016, 2017	Swedish Museum of Natural History	5 (A2015/05561, A2016/05516, -05555, A2017/05797, A2018/05167)
UBA-HELCOM 6	Harbor seal (*Phoca vitulina*)	pooled liver	Denmark	2017	Aarhus University	3 (pool I)
UBA-HELCOM 7	Grey seal (*Halichoerus grypus*)	pooled liver	Denmark	2020	Aarhus University	3 (pool II)
UBA-HELCOM 8	Grey seal (*Halichoerus grypus*)	pooled liver	Germany	2016, 2017	German Oceanographic Museum	3 (Pool 1: M157/18, M69/19, B58/16)
UBA-HELCOM 9	Grey seal (*Halichoerus grypus*)	pooled liver	Germany	2017	German Oceanographic Museum	5 (Pool 2: B63/17, B64/17, B68/17, B61/17, B55/17)
UBA-HELCOM 10	Harbour porpoise (*Phocoena phocoena*)	pooled liver	Germany	2017, 2018	German Oceanographic Museum	2 (Pool 3: M096/18, B46/17)

Short code	Species	Matrix	Country	Year(s) of sampling	Provider	No. of specimens (labels by sample providers)
UBA-HELCOM 11	Grey seal (*Halichoerus grypus*)	pooled liver	Poland	2016, 2017, 2018, 2019	University of Veterinary Medicine Hanover (TiHo Hannover)	8 (1-8)

3.3 Work package 3: Data storage

The contractor integrated all data into the NORMAN Database System (NDS) as a basis for analysis against their ecotoxicity threshold values (risk score), hazard score and exposure score (see Sections 7.1.1 and 7.3.1 below) and comparison with the LIFE APEX Chemical Occurrence Database as well as further analysis.

It has been agreed (portal.helcom.fi, 2021) that the data will be maintained under password protected conditions in the LIFE APEX Database System (https://www.norman-network.com/apex/lacod/) and NDS (https://www.norman-network.com/nds/) until they have been published in a scientific paper by UBA and the contractor and then made public subsequent to this.

Within the NDS the collected high resolution mass spectrometry data (fingerprints of typically hundreds of currently known as well as unknown chemicals present in each analysed sample) were also 'frozen' in the Digital Sample Freezing Platform (DSFP; https://norman-data.net/Verification/) module for future re-analysis against future updates of the database, if needed (e.g. should the information allowing for identification of more 'known' substances in the database increase). The number of substances screened in this report (see STEP 2 above) is expected to increase to more than 90,000 and 120,000 compounds by the end of 2021 and 2022, respectively. Digitally 'frozen' data can be reevaluated as the NORMAN Substance Database grows in the future without any need for new sampling and analytical efforts.

As all data should be used as input for the development of a HELCOM indicator, which is linking contamination of marine mammals with their health status, the data need to be submitted to ICES, who is maintaining the database for hazardous substances of HELCOM. For this, the contractor provides the data including the metadata as Excel tables, with clear distinction of samples per country. These Excel tables will be made available to every HELCOM contracting party for submission to ICES. In the case of Germany, this submission will be done via integration of the data into the German Marine Environment Database (MUDAB). The non-target screening data might be further imported into the non-target screening data portal "Gewässerbeobachtung der Zukunft".

The final data delivery and storage includes:

a. Data Collection Templates with raw data and metadata per HELCOM contracting party for submission to ICES.

b. All HR-MS data uploaded into the NORMAN Database System (NORMAN DSFP, https://www.norman-network.com/nds/; Slobodnik, 2019; Dulio, 2021) and accessible under restricted access rights.

c. All wide-scope target screening and target analyses data uploaded into the LIFE APEX Chemical Occurrence Database and NORMAN EMPODAT database and accessible under restricted access rights.

3.4 Work package 4: Assessment Report

The contractor compiles an assessment report, which provides the screening results as diagrams, identifies which hazardous substances occur in higher amounts in mammals from the Baltic Sea, identifies hot spots of contamination and subregional characteristics, as well as compares the results with the results from the LIFE APEX project and puts the findings into a European context.

The final assessment report includes:

a. Quantitative results of substances included in wide-scope target screening list and (optional) target analyses list.
b. Heat maps (presence/absence and level of confidence of identification) of suspect substances.
c. Semi-quantitative estimates of concentrations of identified suspect substances with quality check.
d. Comparison of wide-scope target and semi-quantified substances against the NORMAN Lowest PNEC values (derived for biota from freshwater PNECs; https://www.norman-network.com/nds/ecotox/lowestPnecsIndex.php).

The report is planned to be submitted by UBA to the relevant HELCOM Working groups (as EG MAMA, EN HZ, WG State and Conservation, WG PRESSURE).

It is envisaged that the results will additionally be published as scientific paper by UBA and the contractor.

4 Sample preparation

4.1 Pre-treatment

The fresh biota samples were lyophilized at -55°C and 0.05 mbar using a Telstar Lyoquest Freeze Dryer (**Figure 1**) and homogenized prior to analysis.

Figure 1: LyoQuest-55 laboratory freeze dryer, Telstar®.

Source: https://www.telstar.com/lab-hospitals-equipment/laboratory-freeze-dryers/lyoquest/ (last visit: 04/08/2021).

The % water content of UBA-HELCOM samples, used to express the results in wet weight, is provided in **Table 2**.

Table 2: %Water content of UBA-HELCOM samples.

Short code	%Water content	Short code	%Water content
UBA-HELCOM 1	69.0	UBA-HELCOM 6	70.9
UBA-HELCOM 2	71.0	UBA-HELCOM 7	67.8
UBA-HELCOM 3a	73.0	UBA-HELCOM 8	71.0
UBA-HELCOM 3b	70.0	UBA-HELCOM 9	72.0
UBA-HELCOM 4	71.5	UBA-HELCOM 10	72.0
UBA-HELCOM 5	70.2	UBA-HELCOM 11	67.0

4.2 Analysis of contaminants of emerging concern

The methodology has been described in detail and published previously in several publications (Slobodnik J, 2021; Alygizakis, 2019; Gkotsis, 2019; Androulakakis, 2021; Diamanti K., 2020; Nikolopoulou V., 2021; Badry, 2022) and has been applied here as described. A simultaneous extraction of emerging contaminants with different physicochemical properties was carried out using generic sample preparation protocols (Gkotsis, 2019). An Accelerated Solvent Extraction (ASE) and solid phase extraction (SPE) were employed prior to the analysis with wide-scope

target and suspect screening methodologies followed by liquid (LC) and gas chromatography (GC) coupled with high resolution mass spectrometry (HRMS).

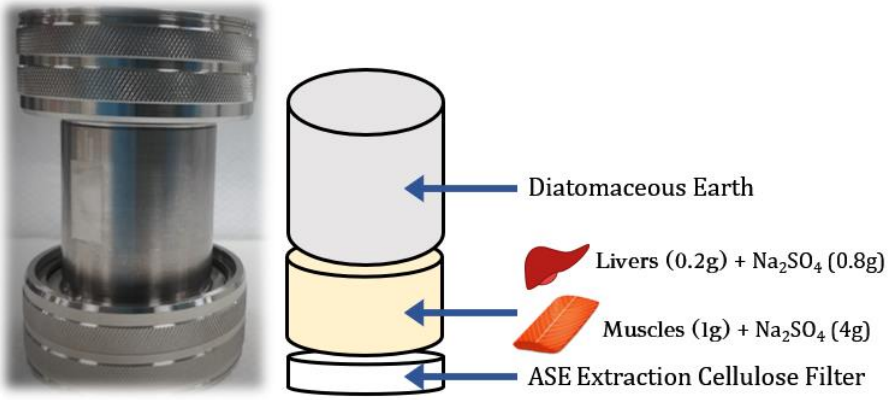
Two generic sample preparation methods per sample were used. More polar, less volatile and thermally unstable compounds were extracted by the method specific for LC-amenable compounds, whereas a different sample preparation method was followed for the extraction of more volatile and thermostable GC-amenable compounds.

4.2.1 Extraction of Liquid Chromatography-amenable contaminants

Accelerated Solvent Extraction (ASE) was used for the extraction of emerging contaminants from the biota matrices, followed by a clean-up step using in-house mixed mode Solid Phase Extraction (SPE) cartridges. Individual steps of the sample preparation protocol are presented in **Figure 5** and described below:

- 0.2 g of liver (or 1 g of muscle) were weighted and mixed with 0.8 g (or 4 g) of samples' dispersant sodium sulfate (Na2SO4), using mortar and pestle.
- A mix of isotopically labelled internal standards was spiked in each sample and left in contact with the matrix for at least 30 min prior to the extraction. Representative compounds from different classes of the LC target list of NKUA were selected.
- The samples were placed in extraction cells (**Figure 2**) and the analytes were extracted by ASE (Dionex™ ASE™ 350, Thermo Fisher Scientific, **Figure 3**). The extraction conditions are provided in **Table 3**.

Figure 2: Samples' loading in the Accelerated Solvent Extraction cell.



Source: own illustration, NKUA.

Figure 3: Dionex™ ASE™ 350, Thermo Fisher Scientific.

Source: https://www.selectscience.net/products/thermo-scientific-dionex-ase-350-accelerated-solvent-extractor-system/?prodID=103880 (last visit: 04/08/2021).

Table 3: Accelerated Solvent Extraction conditions for Liquid Chromatography -amenable compounds.

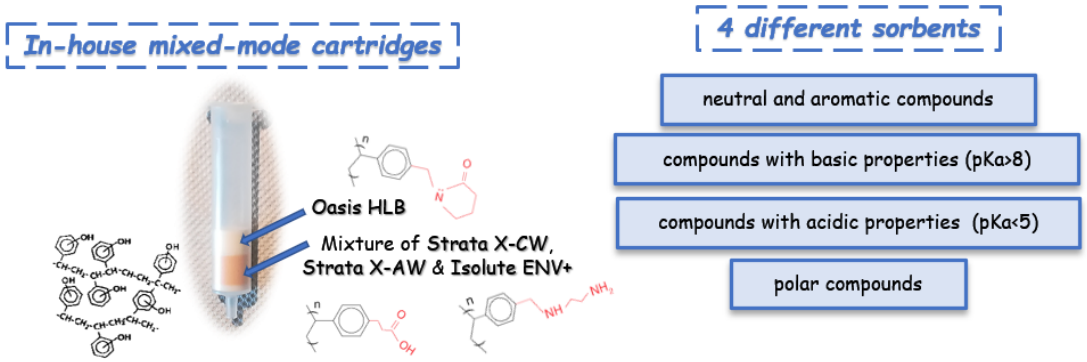
Parameter	Conditions
Temperature (°C)	50
Pressure (psi)	1500
Heating Time (s)	300
Static Time (s)	420
Number of Static Cycles	3
Flush Volume (%)	60
Purge Time (s)	180
Extraction Solvent ratio	Methanol: Acetonitrile (2:1)
Total volume of extraction solvents (mL)	60

- If not transparent, the extract was filtered through a filter paper.
- The extract was pre-concentrated using rotary evaporator (at 40°C) till reaching the final volume of 3-4 mL.
- Milli-Q water was added to adjust the final volume to 15 mL.
- Clean-Up Step 1: Defatting step. 5 mL of n-hexane were added into each sample, then the sample was mixed using Vortex stirring for 1 min and centrifuged for 10 min at 4000 rpm. Finally, the hexane layer was discarded.
- Milli-Q water was added to adjust the final volume to 50 mL.
- Clean-Up Step 2: SPE.

The samples were then cleaned-up by SPE. Layered 'mixed-mode' cartridges (depicted in **Figure 4**) consisted of Oasis HLB (200 mg) and a mixture of Strata-X-AW (weak anion exchanger), Strata-X-CW (weak cation exchanger) and Isolute ENV+ (300 mg of total mixture). The conditioning of

the cartridges was performed with 3 mL of methanol and 3 mL of Milli-Q water and then the samples were loaded in the SPE cartridges. The cartridges were dried by passing air through them for 0.5 to 1 h (applying vacuum facility in the SPE box; cartridges were visually inspected for complete dryness). The elution of the analytes from the adsorbent material was performed by a basic solution (6 mL of ethylacetate/methanol (50/50, v/v) containing 2% ammonia hydroxide (v/v)), followed by an acidic solution (4 mL of ethylacetate/methanol (50/50, v/v) containing 1.7% formic acid (v/v)).

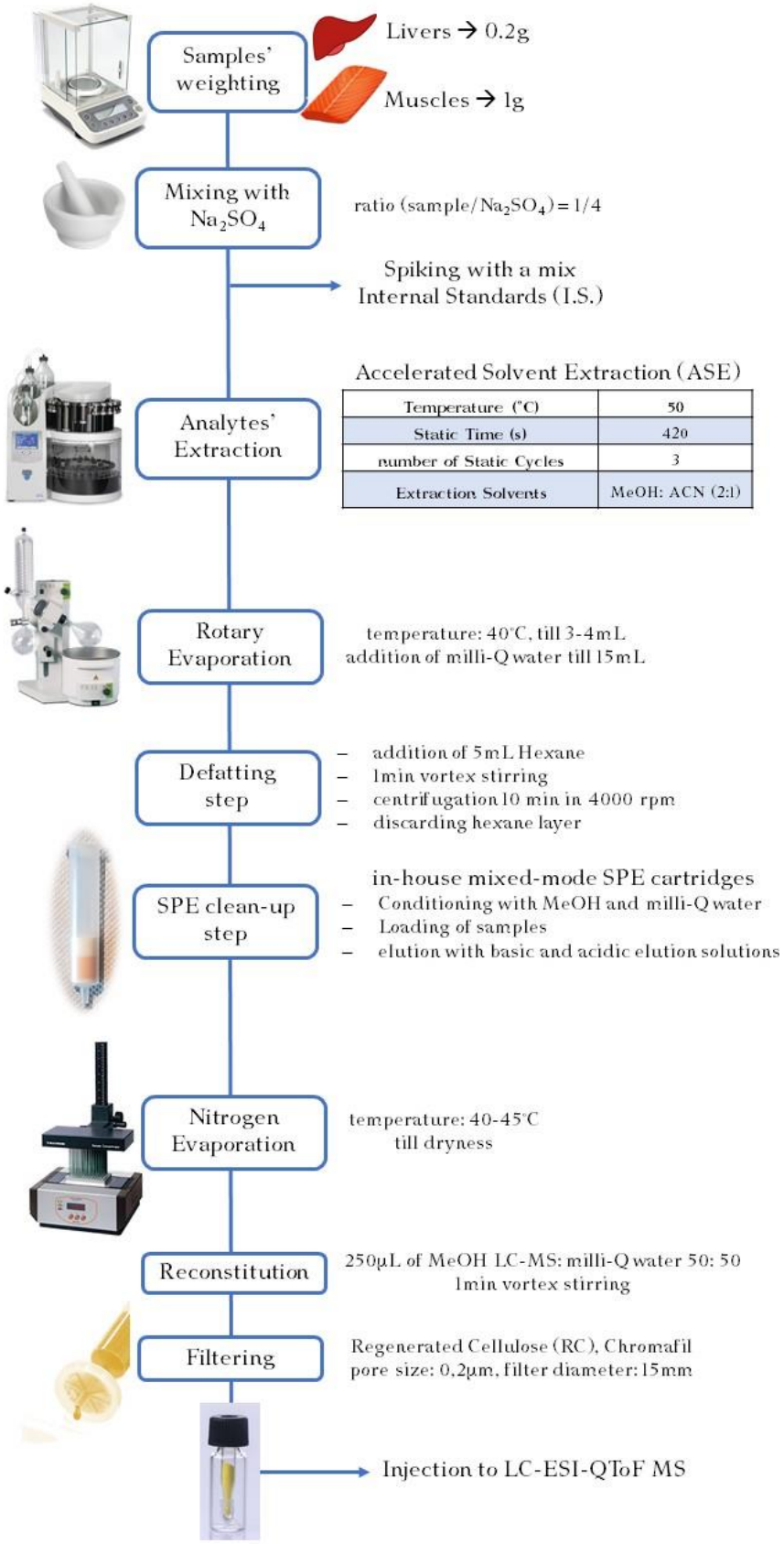
Figure 4: Mixed-mode Solid Phase Extraction (SPE) cartridges.



Source: own illustration, NKUA.

- The extract was evaporated using nitrogen stream at 40-45 °C till dryness.
- 250 µL of the mix of methanol (LC-MS grade) and Milli-Q water (50/50, v/v) were used for the final reconstitution of each sample and the reconstituted sample was homogenized using Vortex stirring for 1 min.
- The final extract was filtered through the Regenerated Cellulose (RC) filter (Chromafil - pore size: 0,2 µm; filter diameter: 15 mm), using a syringe, into a 2 mL glass vial with an insert placed inside.
- After the analysis by LC-ESI-QToF MS the vials were stored in the freezer at -80°C.

Figure 5: Sample preparation protocol for the Liquid Chromatography-amenable compounds.



Source: own illustration, NKUA.

4.2.2 Extraction of Gas Chromatography-amenable contaminants

Individual steps of the sample preparation for the determination of GC-amenable compounds are presented in **Figure 6** and below:

- 0.2 g of liver (or 1 g of muscle) were weighted and mixed with 0.8 g (or 4 g) of samples' dispersant sodium sulfate (Na2SO4), using mortar and pestle.
- A mix of isotopically labelled internal standards was spiked into each sample and left in contact with the matrix for at least 30 min prior to the extraction. Representative compounds from different classes of the GC target list of NKUA were selected.
- The analytes were extracted by ASE (Dionex™ ASE™ 350, Thermo Fisher Scientific). The extraction conditions are provided in **Table 4**.

Table 4: Accelerated Solvent Extraction conditions for Gas Chromatography-amenable compounds.

Parameter	Conditions
Temperature (°C)	100
Pressure (psi)	1500
Heating Time (s)	300
Static Time (s)	300
Number of Static Cycles	3
Flush Volume (%)	60
Purge Time (s)	180
Extraction Solvent ratio	Hexane:Dichloromethane (2:1)
Total volume of extraction solvents (mL)	70

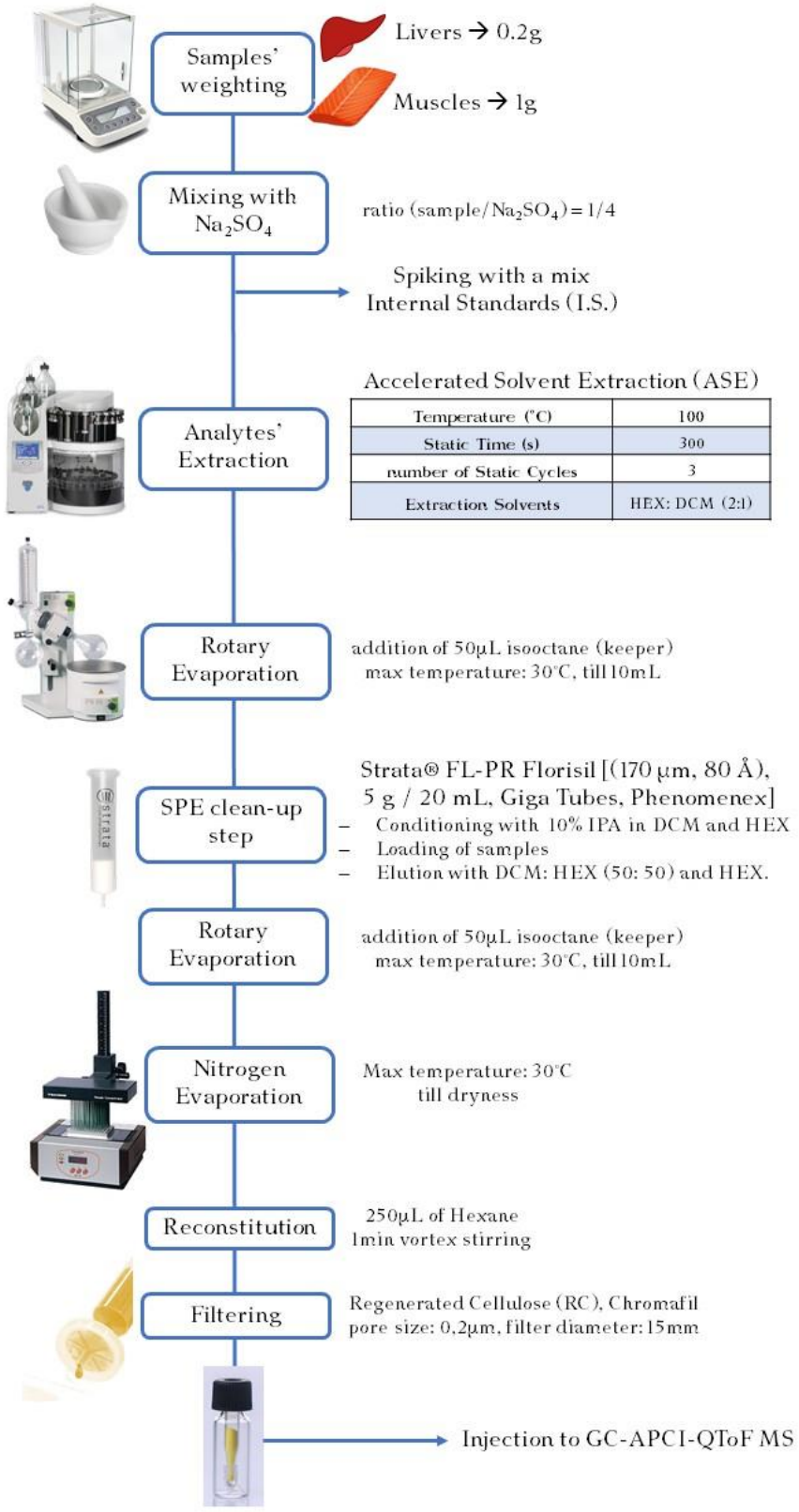
- If not transparent, the extract was filtered through a filter paper.
- 50 μL of isooctane, used as a keeper, were added.
- The extract was pre-concentrated by rotary evaporation (max. temperature 30 °C) till 10 mL.
- Clean-Up Step: SPE.

The samples were then cleaned-up by SPE. Strata® FL-PR Florisil ((170 μm, 80 Å), 5 g/ 20 mL, Giga Tubes, Phenomenex) cartridges were used. The conditioning of the cartridges was performed using 20 mL of 10% isopropanol in dichloromethane, followed by 30 mL of hexane. After conditioning, the samples were loaded in the SPE cartridges and the eluent was collected. The elution of the analytes from the adsorbent material was performed using 20 mL of dichloromethane : hexane (50/50 v/v), followed by 20 mL of hexane. The whole extract (cleaned extract and elution solvents) was placed into an evaporation flask. After that:

- 50 μL of isooctane was added.

- The extract was pre-concentrated by rotary evaporation (max. temperature 30 °C) till 10 mL.
- The extract was evaporated using nitrogen stream (max. temperature 30 °C) to a final volume of 250 µL hexane and homogenized using Vortex stirring for 1 min.
- The final extract was filtered through the Regenerated Cellulose (RC) filter (Chromafil - pore size: 0,2 µm; filter diameter: 15 mm), using a syringe, into a 2 mL glass vial with an insert placed inside each vial.
- After the analysis by GC-APCI-QToF MS the vials were stored in the freezer at -80°C.

Figure 6: Sample preparation protocol for the Gas Chromatography-amenable compounds.



Source: own illustration, NKUA.

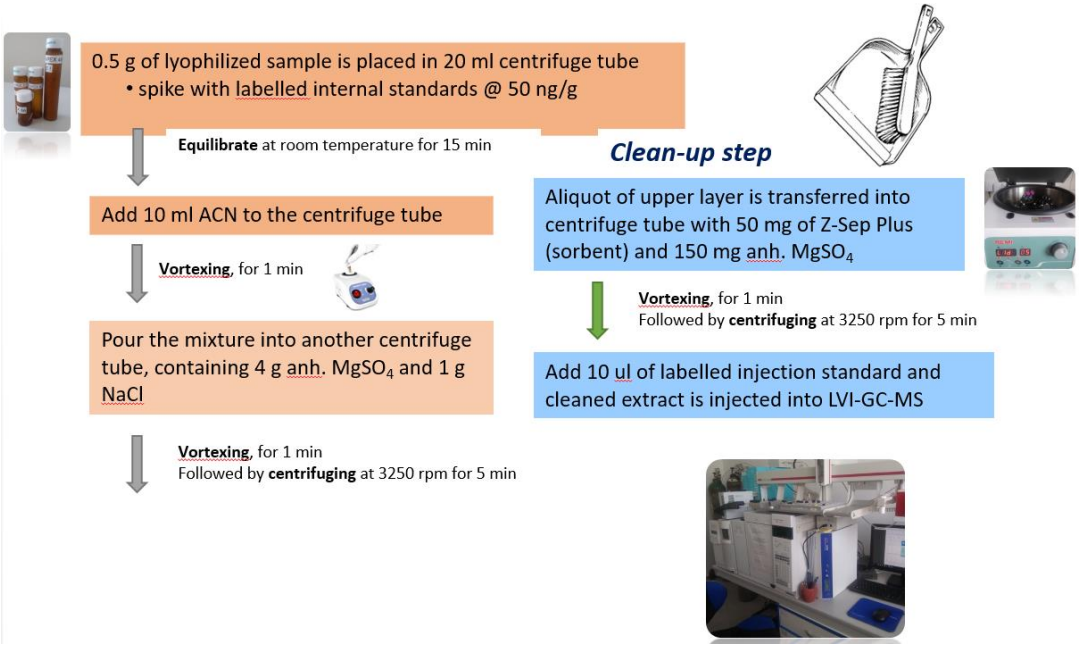
4.2.3 Quality Assurance & Quality Control (QA/QC)

A thorough quality assurance and quality control (QA/QC) was applied during the sample preparation and instrumental analysis. A mix of internal standards was added into each sample prior to extraction to assure satisfactory recovery of the target compounds. Samples spiked with a mix of known emerging contaminants were also analyzed in each batch of samples. Moreover, procedural blank (reagent blanks) were prepared to assess any external contamination which might have been brought in during the sample preparation of the extracts and analysis. A mix of known analytes (RTI calibrant substances) was used to assess the stability of retention time during instrumental analysis. A QC sample was running every 10 injections to ensure the good operation and high sensitivity of the instrument.

4.3 Analysis of explosives

All samples to be tested for the presence of selected 23 explosive compounds, were prepared in the laboratory of EI using QuEChERS ("quick, easy, cheap, effective, rugged, and safe" SPE) approach using the following procedure (**Figure 7**):

Figure 7: Sample preparation protocol for the explosive compounds.



Source: own illustration, EI.

The studied nitroaromatic explosives are thermally sensitive and unstable, therefore a GC-MS method differing from the general screening approach (Section 4.2.2) had to be developed. Sample processing at low temperatures, very fast sample introduction onto the analytical column followed up by rapid separation were needed to avoid decomposition of the studied analytes.

4.4 Analysis of novel organophosporous flame retardants and dechlorane-plus compounds

The samples were prepared in the same way as those subjected to analysis of explosives (see paragraph 4.3 above), however, a different GC-MS method has been used for their analysis (see paragraph 5.3 below).

5 Instrumental analysis

The samples were analysed by both liquid and gas chromatography hyphenated with a high resolution mass spectral analyser. A detailed information on the instrumental analysis of the RPLC-ESI-QTOF and GC-APCI-QTOF system is provided below in sections 5.1 and 5.2, respectively. The methodology has been described in detail and published previously in several publications (Slobodnik J, 2021; Alygizakis, 2019; Gkotsis, 2019; Androulakakis, 2021; Diamanti K., 2020; Nikolopoulou V., 2021; Badry, 2022) and has been applied here as described.

5.1 Reversed-Phase Liquid Chromatography High Resolution Mass Spectrometry

Figure 8: UHPLC-ESI-QToF MS, Maxis Impact, Bruker Daltonics.

Source: https://www.directindustry.com/prod/bruker-daltonics/product-30029-991983.html (last visit: 04/08/2021).

Apparatus

- Chromatographic System: Ultra High Performance Liquid Chromatography (UHPLC) apparatus with an HPG-3400 pump (Dionex UltiMate 3000 RSLC, Thermo Fisher Scientific) (**Figure 8**)
- Column: Acclaim TM RSLC 120 C18 (100 × 2.1 mm, 2.2 μm; Thermo Fisher Scientific)
- Mass Spectrometer: Hybrid Quadrupole Time of Flight Mass Analyzer (QTOF-MS) (Maxis Impact, Bruker Daltonics)

Solvents - Buffers

- Ultrapure Water by Milli-Q-UV system (Millipore)
- Methanol, LC-MS Grade (Merck)
- Formic acid - eluent additive for LC-MS (Fluka Analytical)
- Ammonium formate, LC-MS Ultra and ammonium acetate for mass spectrometry - eluent additives for UHPLC-MS (Fluka Analytical)

Gradient elution program

The gradient elution programme of the reversed-phase liquid chromatographic system, for both positive and negative ESI mode is provided in **Table 5**.

Positive Ionization:

Aqueous solvent: H_2O/methanol 90/10 (v/v), 5 mM $HCOONH_4$, 0.01% formic acid

Organic Solvent: Methanol, 5 mM $HCOONH_4$, 0.01% formic acid

Negative Ionization:

Aqueous solvent: H_2O/methanol 90/10 (v/v), 5 mM CH_3COONH_4

Organic Solvent: Methanol, 5 mM CH_3COONH_4

Table 5: Reversed Phase Liquid Chromatography (RPLC) gradient elution programme.

Time (min)	*Flow rate (mL min^{-1})*	*Aqueous solvent %*	*Organic solvent %*
0	0.2	99.0	1.0
1	0.2	99.0	1.0
3	0.2	61.0	39.0
14	0.4	0.1	99.9
16	0.48	0.1	99.9
16.1	0.48	99.0	1.0
19.1	0.2	99.0	1.0
20.0	0.2	99.0	1.0

Column temperature: 30 °C

Injection volume: 5 µL

Ion source

QTOF-MS was equipped with an electrospray ionization interface (ESI) operated in both positive and negative mode.

MS parameters

- Scan mode: a) 1st run in Data Independent mode: broad band Collision Induced Dissociation (bbCID) acquisition mode (acquisition of full scan MS spectra (4 eV) and MS/MS (25 eV) spectra in a single run) and b) 2nd run in Data Dependent mode (acquisition of full scan MS spectra and MS/MS spectra of the 5 most abundant ions per MS scan in a single run)
- m/z (mass to charge ratio) range: 50 - 1000 Da
- Scan rate: 2 Hz
- External calibration of QToF-MS was performed just prior to analysis with 10 mM of sodium formate in a mixture of water/isopropanol (50/50, v/v). The theoretical exact masses of

calibration ions with formulas Na(NaCOOH)1-14 in the range of 50–1000 Da were used. Also, internal calibration was performed by calibrant injection at the beginning of each chromatogram (1st segment, 0.1–0.25 min).

5.2 Gas Chromatography High Resolution Mass Spectrometry

The GC-APCI-QTOF system consisted of a Bruker 450 GC, a CP-8400 AutoSampler and a hybrid quadrupole time of flight mass spectrometer (QTOF-MS) (Maxis Impact, Bruker Daltonics) **(Figures 9 and 10)**.

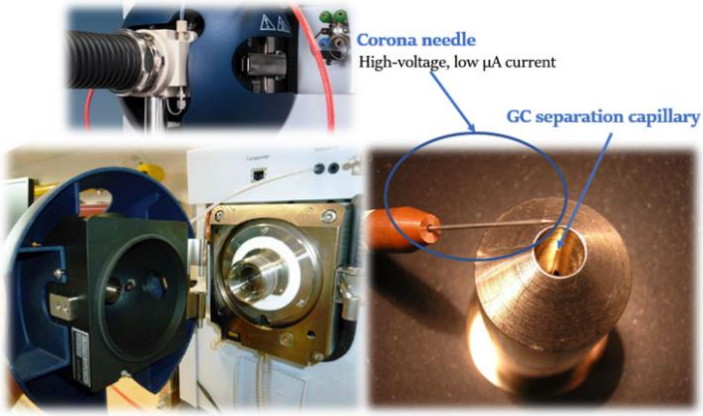
GC was operated in splitless injection mode (Restek Split liner w/Glass Frit (4 mm x 6.3 x 78.5)) and the splitless purge valve was activated 1 min after injection. The injection volume was 1 µL. A Restek Rxi-5Sil MS column of 30 m (0.25 mm i.d. x 0.25 µm film thickness) was used with helium as a carrier gas at the constant flow of 1.5 mL min^{-1}.

The GC oven was programmed as follows: 55 °C initial hold for 3 min, increase at a rate of 15 °C min^{-1} to 180 °C, then increase with a step of 6.5 °C min^{-1} to 280 °C and hold for 5 min followed by an increase of 10 °C min^{-1} to 300 °C and hold for 5.28 min. The temperature of splitless injector port, GC-MS transfer line and MS source were maintained at 280, 290 and 250 °C, respectively.

Figure 9: UHPLC-ESI-QToF MS, Maxis Impact, Bruker Daltonics.

Source: own illustration, NKUA.

Figure 10: The Atmospheric Pressure Chemical Ionization (APCI) source.



Source: own illustration, NKUA.

The operating parameters of APCI interface were: capillary voltage, 5000 V; corona voltage, 2000 V; end plate offset, 500 V; nebulizer, 3.5 bar; drying gas, 1.5 L min^{-1}. The QToF MS system operated in two different acquisition modes (Data Independent and Data Dependent), as previously described for LC-HRMS analysis. The scan rate was 8 Hz per cycle.

The QToF mass spectrometer was calibrated with perfluorotributylamine (FC43) prior to each analysis (external calibration) and at the first seconds (1st segment, 0.1–0.25 min) of every chromatogram (internal calibration).

5.3 Gas Chromatography Mass Spectrometry

For analysis of explosives GC – Agilent 7890 equipped with a split/splitlessinjector (SSL) and a programmable temperature vaporization (PTV) injector coupled to a Agilent 5973 quadrupole mass spectrometer with electron ionization source was used. The GC was equipped with a DB-5MS 15 m × 0.25 mm × 0.25 µm column (J&W, USA) and connected to the respective injector. PTV-injector with packed quartz wool liners was used for introducing 20 µl of an extract. Hydrogen served as a carrier gas for the GC (Linde, purity 99.999%). Spectra were recorded and analyzed in Chemstation software. The GC oven was programmed as follows: 50 °C initial hold for 5 min, increase at a rate of 20 °C min^{-1} to 315 °C and hold for 5 min. The temperature of GC-MS transfer line and MS source were maintained at 280 °C and 250 °C, respectively. PTV injector was programed as follows: 70 °C initial hold for 0.20 min, increase at a rate of 700 °C min^{-1} to 180 °C and hold for 5 min.

For analysis of OPFRs a dechlorane-plus compounds, aample extracts were analyzed by GC-MS/MS system using an electron impact (EI) ionization. The GC-MS system (Agilent 7890 (Waldbronn, Germany) has been upgraded to MS/MS by Chromtech, Germany. The GC was operated in splitless injection mode (Restek Split liner w/Glass Frit (4 mm x 6.3 x 78.5)) and the splitless purge valve was activated 1 min after injection. The injection volume was 2 µL. A Restek Rxi-5Sil MS column of 30 m (0.25 mm i.d. x 0.25 µm film thickness) was used with hydrogen as a carrier gas at the constant flow of 1.6 mL min^{-1}. The MS/MS system was operated in multiple reaction monitoring (MRM) mode. Quantification was based on external calibration curves established for the compounds of interest.

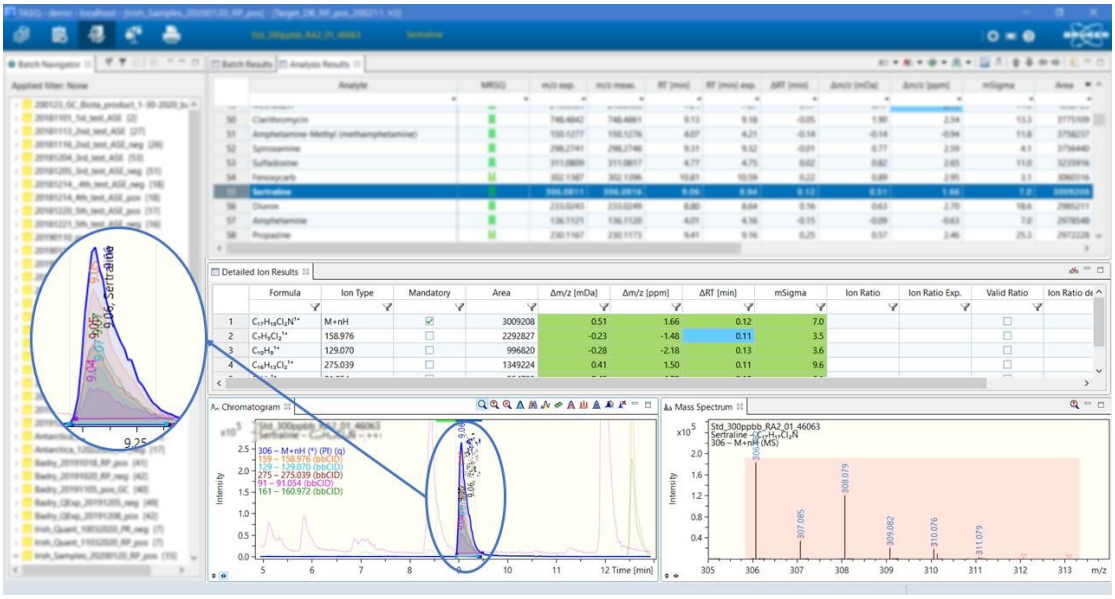
6 Data treatment

Two different approaches were used for screening of organic contaminants: (a) wide-scope target screening providing fully-quantitative results on the detected contaminants and (b) suspect screening resulting in information on presence/absence of a compound in the sample and semi-quantification of the detected substances.

6.1 Wide-scope target screening

Target screening was performed with the use of in-house developed databases of 2,540 contaminants (Alygizakis, 2018; Damalas, 2020, Diamanti K., 2020; Badry, 2022) and the software TASQ Client 2.1 and DataAnalysis 5.1 (Bruker Daltonics, Bremen, Germany). The graphical user interface of TASQ Client 2.1 is presented in **Figure 11.** The detection was based on specific screening parameters (mass accuracy <2 mDa, retention time shift ±0.2 min, isotopic fitting <100 mSigma (only for confirmation of positive findings)), whereas the presence of adduct and fragment ions confirmed the analytes (Gago-Ferrero, 2020). The Screening Detection Limit (SDL) was established as the lowest concentration level tested for which a compound is detected in all spiked samples, at the expected retention time and with specific mass error of the precursor ion. The SDL was not compound-specific, but a generic reporting value derived after method validation. Furthermore, compound-specific validation was performed for quantification purposes of the compounds detected with the screening method. Compound-specific limit of detection (LOD) and limit of quantification (LOQ) values were calculated after the treatment and analysis of samples spiked with the detected compounds and structure-related isotope labeled compounds. The contaminants that were detected in traces below the LOQ (concentration levels between the LOD and LOQ values) were reported as BQL (below the quantification limit). For statistical treatment of the results, substitution of BQL with LOQ/2 may be performed, as indicated by the QA/QC Directive (2009/90/EC).

Figure 11: A typical outcome of analysis provided by the software TASQ Client 2.1 (Bruker Daltonics, Bremen, Germany).



Source: own illustration, NKUA.

6.2 Suspect screening

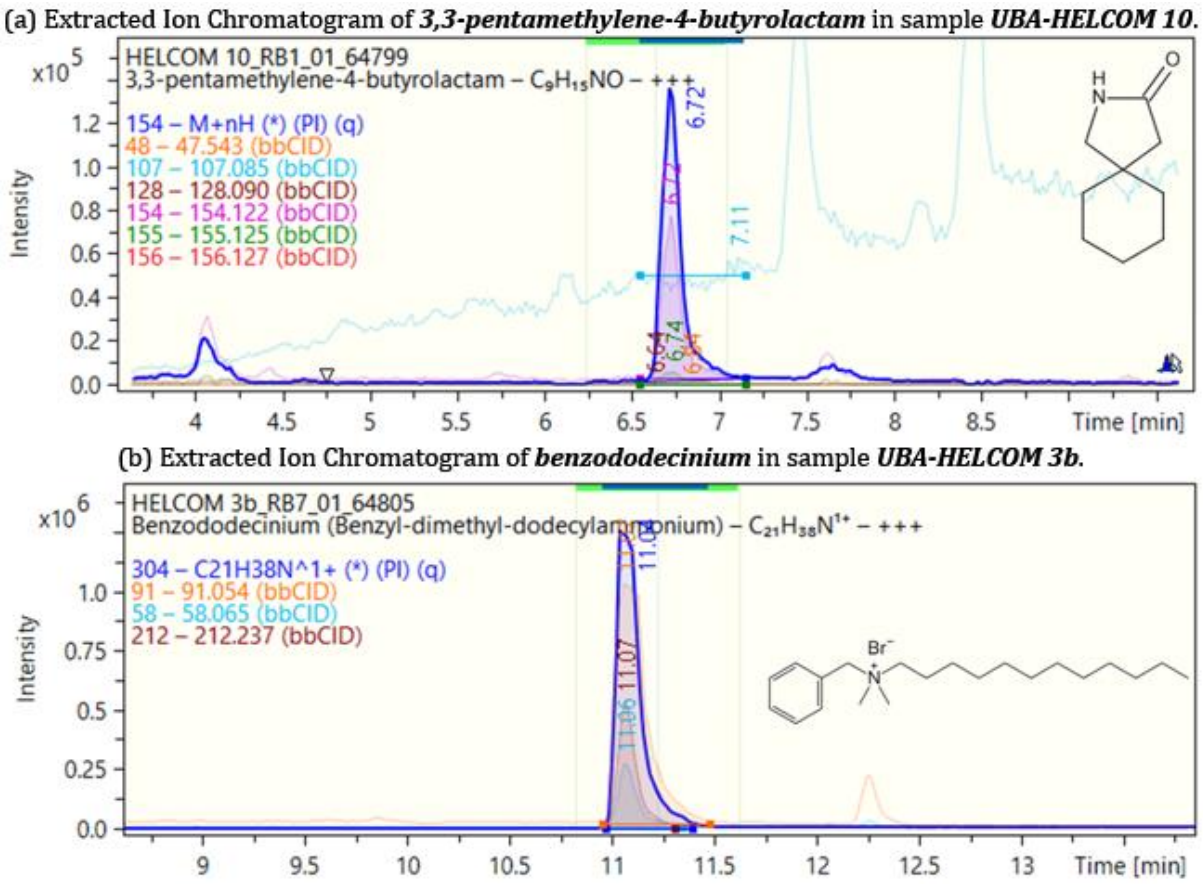
Suspect screening was performed for environmentally relevant pollutants from the NORMAN Substance Database (SusDat; https://www.norman-network.com/nds/susdat/) in all raw chromatograms which were imported into the NORMAN Digital Sample Freezing Platform (DSFP) (http://www.norman-data.eu/) - a novel tool developed for revealing the presence of suspects and identification of unknown compounds in environmental samples (Alygizakis, 2019). The methodology has been described in detail and published previously in several publications (Alygizakis, 2019; Alygizakis, 2019; Slobodnik J, 2021; Diamanti K., 2020; Nikolopoulou V., 2021) and has been applied here as described. The calibrant masses were used to recalibrate the whole chromatogram using HPC fitting algorithm, which is embedded in DataAnalysis 5.1 (Bruker Daltonics, Bremen, Germany). This calibration method ensured mass accuracy below 2 mDa during the whole chromatographic run for ions with m/z 50-1000. For exporting files in mzML format, CompassXport 3.0.9.2 (Bruker Daltonics, Bremen, Germany) was used. Chromatograms acquired under bbCID were separated in low and high collision energy layer chromatograms. All mzML files and their meta-data (instrumental, sample meta-data, matrix-specific meta-data and retention time of RTI calibrant substances) were uploaded to DSFP. DSFP is based on an integrated workflow, which follows standard operating procedure (SOP) to process the mzML files and all meta-data for generation of harmonized Data Collection Templates (DCTs). This data reduction technique resulted in an automatic generation of DCTs, which include condensed information from bulky raw LC-HRMS files.

7 Results

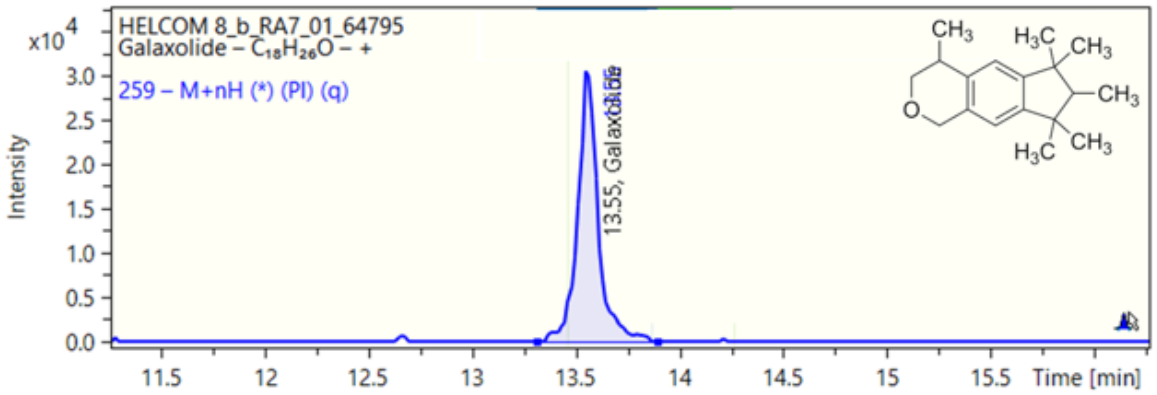
7.1 Wide-scope target screening

Overall, 47 contaminants were determined through wide-scope target analysis, out of which seven were detected only in the range between their LOD and LOQ (BQL). All samples were livers from marine mammals from the Baltic Sea, with the exception of the sample UBA-HELCOM 3, where also muscle (UBA-HELCOM 3b) from the same specimen was analysed. Examples of selected extracted chromatograms of the detected compounds are provided in **Figure 12**. Results of analyses for all samples, expressed in µg/kg wet weight, are provided in **Appendix C** as well as in the separately submitted DCT. Data on the frequency of detection (Frequency of Appearance (FoA; a number between 0 and 1) of a compound in analysed samples; FoA = n/N where n is No. of samples in which the compound was determined and N is the total number of samples) and detected concentration ranges in livers are provided in **Table 6**. Based on the information available about substances' main use, chemical class or application, their main use category was proposed, although some compounds may have multiple uses. For every compound detected, LOD and LOQ values were derived. The risk associated with the exceedance of toxicity threshold values has been assessed by comparing the measured concentrations with the environmental quality standards (EQS) for biota (fish) as in the Directive 2013/39/EU and PNEC values derived for fish in the marine environment from the NORMAN Ecotoxicology Database (https://www.norman-network.com/nds/ecotox/lowestPnecsIndex.php). Compounds whose concentrations were below the method's Screening Detection Limit (1.71 µg/kg and 0.375 µg/kg wet weight for livers and muscles, respectively) are listed in **Appendix D**.

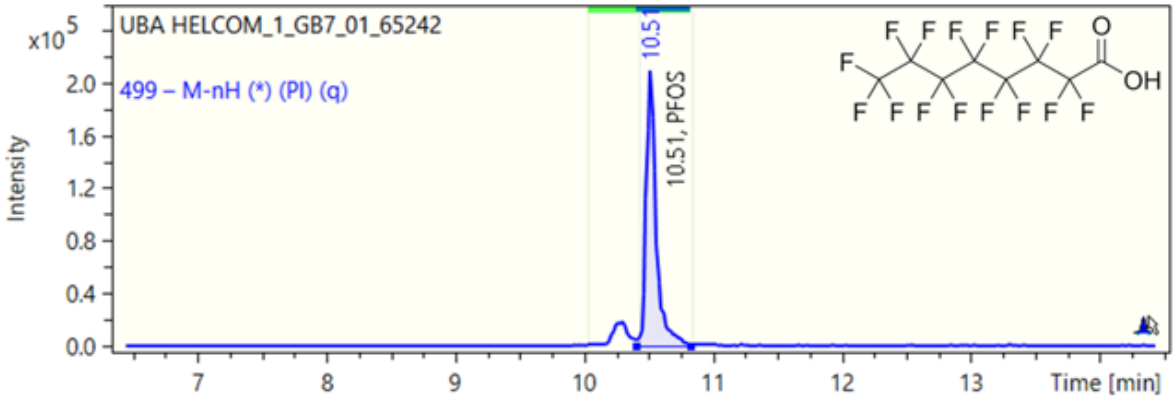
Figure 12: Selected examples of Extracted Ion Chromatograms of detected contaminants acquired by high resolution mass spectrometric analysis.



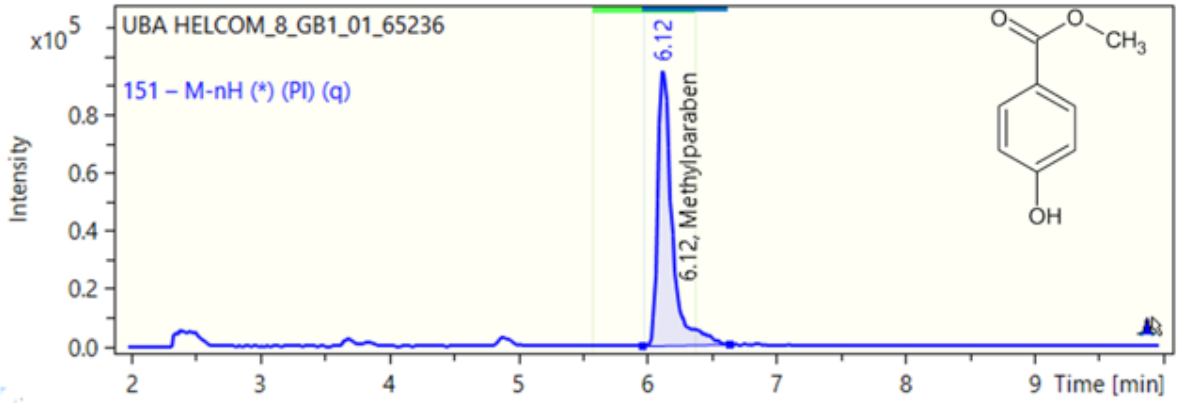
(c) Extracted Ion Chromatogram of ***galaxolide*** in sample ***UBA-HELCOM 8***.



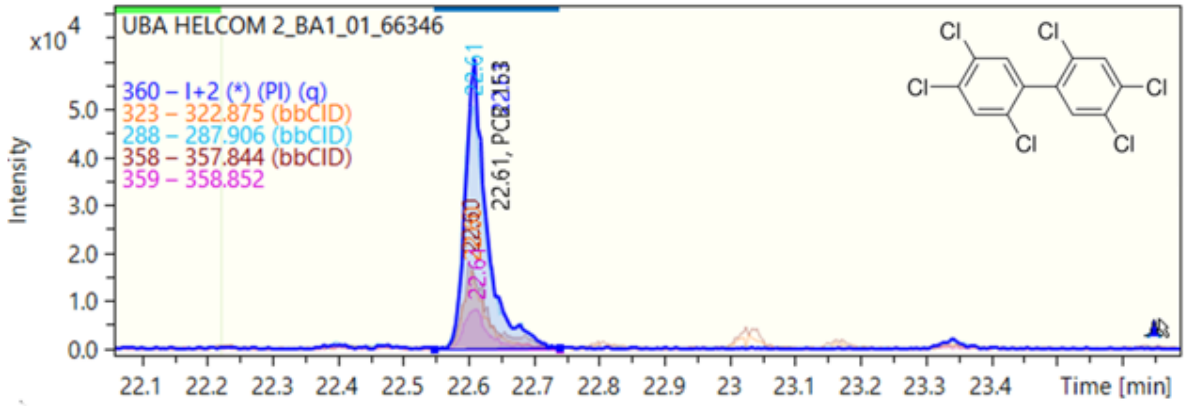
(d) Extracted Ion Chromatogram of ***Perfluorooctane sulfonic acid (l-PFOS)*** in sample ***UBA-HELCOM 1***.

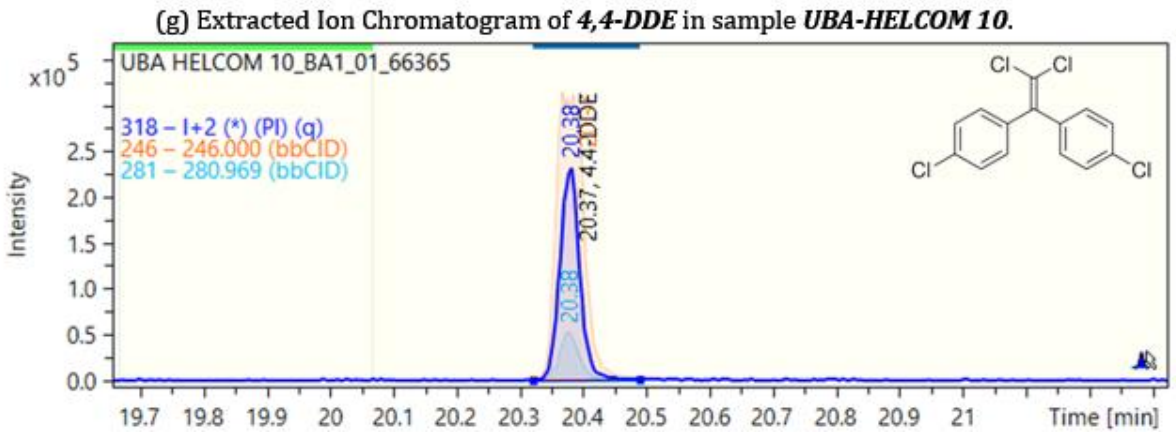


(e) Extracted Ion Chromatogram of ***methylparaben*** in sample ***UBA-HELCOM 8***.



(f) Extracted Ion Chromatogram of ***PCB 153*** in sample ***UBA-HELCOM 2***.





Source: own illustration using TASQ Client 2.1 (Bruker Daltonics, Bremen, Germany), NKUA

Table 6: Summary of wide-scope target screening anlaysis results, expressed in µg/Kg w.w.

Analyte	CAS No.	Classification	Analytical technique	FoA liver	Detection range	LOQ	PNEC
Benzophenone-3	131-57-7	PCPs	LC-ESI-QTOF	0.09	BQL	6.17	10.5
Galaxolide	1222-05-5	PCPs	LC-ESI-QTOF	0.82	29.7-306	9.00	859
Octocrylene	80135-31-5	PCPs	LC-ESI-QTOF	0.55	8.68-56.2	8.60	1.26
Ethylparaben	120-47-8	PCPs	LC-ESI-QTOF	0.18	BQL-23.3	4.43	7.15
Methylparaben	99-76-3	PCPs	LC-ESI-QTOF	0.91	27.3-964	4.49	2.56
Carazolol	57775-29-8	Pharms	LC-ESI-QTOF	0.09	160	10.6	0.280
Enrofloxacin	93106-60-6	Pharms	LC-ESI-QTOF	0.09	138	29.4	1.41
Sulfadoxine	2447-57-6	Pharms	LC-ESI-QTOF	0.09	BQL	17.6	0.330
3,3-Pentamethylene-butyrolactam	64744-50-9	Pharms TPs	LC-ESI-QTOF	0.73	5.55-1069	3.70	66.7
2-Hydroxy-ibuprofen	51146-55-5	Pharms TPs	LC-ESI-QTOF	0.09	46.7	22.2	2.33
Nor-Tramadol (N-Desmethyl-Tramadol)	73806-55-0	Pharms TPs	LC-ESI-QTOF	0.36	BQL-22.7	11.5	12.4
O-Desmethyl-Tramadol	73986-53-5	Pharms TPs	LC-ESI-QTOF	0.09	BQL	28.9	N.A.
O-Desmethyldinor-Tramadol	522648-42-6	Pharms TPs	LC-ESI-QTOF	0.36	BQL-77.3	21.7	N.A.

Analyte	CAS No.	Classification	Analytical technique	FoA liver	Detection range	LOQ	PNEC
Perfluorobutane sulfonic acid (PFBuS)	375-73-5	PFAS	LC-ESI-QTOF	0.09	BQL	0.385	17.7
Perfluorodecanoic acid (PFDeA)	335-76-2	PFAS	LC-ESI-QTOF	1.00	BQL-15.9	9.78	0.820
Perfluoroheptanoic acid (PFHpA)	375-85-9	PFAS	LC-ESI-QTOF	0.27	BQL	1.07	4.66
Perfluoroheptane sulfonic acid (PFHpS)	375-92-8	PFAS	LC-ESI-QTOF	0.64	BQL	0.862	21.8
Perfluorohexane sulfonic acid (PFHxS)	355-46-4	PFAS	LC-ESI-QTOF	0.91	BQL-0.694	0.233	15.3
Perfluorononanoic acid (PFNA)	375-95-1	PFAS	LC-ESI-QTOF	1.00	BQL-46.2	4.80	16.5
Perfluorooctanoic acid (PFOA)	335-67-1	PFAS	LC-ESI-QTOF	0.27	BQL	2.69	0.041
Perfluorooctane sulfonic acid (l-PFOS)	1763-23-1	PFAS	LC-ESI-QTOF	1.00	159-1059	2.79	9.10*
Perfluorooctane sulfonic acid isomers (sum branched isomers)	N.A.	PFAS	LC-ESI-QTOF	1.00	BQL-173	2.66	9.10*
Perfluorooctane sulfonamide (PFOSA)	754-91-6	PFAS	LC-ESI-QTOF	0.09	6.49	1.88	0.0098
Perfluoroundecanoic acid (PFUnDA)	2058-94-8	PFAS	LC-ESI-QTOF	1.00	BQL-12.0	9.35	22.3
2,4-DDT	789-02-6	PPPs	GC-APCI-QTOF	0.27	14.7-72.5	8.63	31.0
Dieldrin	60-57-1	PPPs	GC-APCI-QTOF	0.18	104-257	8.39	0.200
Hexachlorobenzene	118-74-1	PPPs	GC-APCI-QTOF	1.00	BQL-49.4	2.89	10.0
Fenuron	101-42-8	PPPs	LC-ESI-QTOF	0.09	18.1	9.62	0.640
Pyrethrins: Cinerin II	121-20-0	PPPs	LC-ESI-QTOF	0.18	BQL-39.7	25.4	0.150
4,4-DDD	72-54-8	PPPs TPs	GC-APCI-QTOF	0.91	BQL-76.5	3.16	741
4,4-DDE	72-55-9	PPPs TPs	GC-APCI-QTOF	1.00	2.54-155	0.196	0.005
Metazachlor OA	1231244-60-2	PPPs TPs	LC-ESI-QTOF	0.09	54.3	8.62	0.55

Analyte	CAS No.	Classification	Analytical technique	FoA liver	Detection range	LOQ	PNEC
Propachlor-OXA	70628-36-3	PPPs TPs	LC-ESI-QTOF	0.18	58.4-64.5	34.4	2.32
Harman	486-84-0	Stimulants	LC-ESI-QTOF	0.09	7.05	3.92	0.480
Nicotine	54-11-5	Stimulants	LC-ESI-QTOF	0.36	BQL-50.8	35.0	4.69
Anabasine	143924-51-0	Stimulants TPs	LC-ESI-QTOF	0.27	42.6-121	32.8	N.A.
Nor-Nicotine	5746-86-1	Stimulants TPs	LC-ESI-QTOF	0.64	BQL-248	13.6	21.0
Benzotriazole (BTR)	95-14-7	Ind. Chem.	LC-ESI-QTOF	0.09	37.3	14.0	4.44
Triethyl-Phosphate	78-40-0	Ind. Chem.	LC-ESI-QTOF	0.27	BQL	18.1	81.4
N-Butylbenzenesulfonamide	3622-84-2	Ind. Chem.	LC-ESI-QTOF	0.36	201-765	11.5	9.85
PCB 101	37680-73-2	Ind. Chem.	GC-APCI-QTOF	1.00	BQL-104	2.44	0.080
PCB 138	35065-28-2	Ind. Chem.	GC-APCI-QTOF	0.91	BQL-138	4.84	0.100
PCB 153	35065-27-1	Ind. Chem.	GC-APCI-QTOF	0.91	2.50-287	1.30	0.050
PCB 180	35065-29-3	Ind. Chem.	GC-APCI-QTOF	0.82	BQL-176	2.63	0.080
PCB 52	35693-99-3	Ind. Chem.	GC-APCI-QTOF	0.82	BQL-123	5.72	0.080
Triethylcitrate	77-93-0	Preservatives	LC-ESI-QTOF	0.18	BQL	3.67	18.1

BQL: Below the Limit of Quantification, LOQ: Limit of Quantification; PNEC: predicted no-effect concentration; TPs: Transformation products, Pharms: Pharmaceuticals, PCPs: Personal Care Products, PPPs: Plant Protection Products, Ind. Chem.: Industrial Chemicals, PCBs: Polychlorinated Biphenyls, FoA: Frequency of appearance, N.A.:not available
*EQS from 2013/39/EU was used;

In total 47 contaminants from different chemical classes were determined in the samples from 11 pooled samples of 43 marine mammals' specimen samples (12 samples if calculating separate muscle and liver samples for UBA-HELCOM 3). Most of the detected compounds were per- and polyfluoroalkyl substances (PFAS) (11 compounds, 23.4%), followed by plant protection products & TPs (9 compounds, 19.1%), industrial chemicals (9 compounds, 19.1%) and pharmaceuticals & TPs (8 compounds, 17.0%). The rest (personal care products, stimulants & TPs and preservatives) accounted for 21.3% of the detected compounds (n=10). PCB 101, l-PFOS, hexachlorobenzene and 4,4-DDE (TP of DDT) were detected in all tested samples. PCB 138, PCB 153, two PFAS (PFDeA and the substituted (branched) isomers of PFOS), 4,4-DDD (TP of DDT), as well as methylparaben

(anti-fungal agent often used in a variety of cosmetics and personal care products) were present in the majority of samples (FoA: 0.91). Among the detected contaminants, the highest concentration levels were observed for l-PFOS (12 samples, concentration range: 159-1059 µg/Kg w.w., mean concentration 334 µg/Kg w.w.), methylparaben (11 samples, concentration range: 27.3-964 µg/Kg w.w., mean concentration 296 µg/Kg w.w.), N-butylbenzenesulfonamide (4 samples, concentration range: 201-765 µg/Kg w.w., mean concentration 469 µg/Kg w.w.) and the surfactant and antiseptic compound benzododecinium (benzyl-dimethyl-dodecylammonium), which was detected only in the UBA-HELCOM 3b (muscle of common dolphin from Germany) at the concentration 272 µg/Kg w.w. The total number of contaminants per sample ranged from 18 to 28. The highest number of the compounds dominated by pharmaceuticals and their TPs was determined in the sample UBA-HELCOM 10. This might be partly attributed to the contribution of specimen B46/17 (coding of provider), in the pooled sample, which was gathered in poor condition (illness, potential tumors in liver and stomach). However, it is unclear how most substances are accumulated and therefore any process leading to an increased contaminant load must be monitored closely. Sample UBA-HELCOM 10 also consisted of M096/18 – a live stranding on the coast of Heiligendamm, Germany. The sample contained pharamceuticals that are due to the treatment prior to death (baytril, enrofloraxin), but also showed substances that are rather used in human treatment and are potentially dangerous for marine mammals, such as tramadol, ibuprofen and gabapentin. These substances are not due to treatment and were found in xx of the other samples. These two cases show that illness and cases of abnormal behavior prior to death would be especially important to investigate in future studies. These cases also highlight, that additional information from the provider is required in order to evaluate whether the detection of pharmaceuticals is due to environmental pollution or their deliberate administration or a combination of both. This highlights as well, that pooled samples are a good method for general screening, but are not appropriate to determine whether the found substances have larger effects on any tested individual.

Eleven PFAS were determined in the studied samples. The cumulative PFAS concentration was ranging from 179 (UBA-HELCOM 3a) to 1143 (UBA-HELCOM 1) in the liver samples, whereas in the only analyzed muscle sample (UBA-HELCOM 3b) the total concentration was 5.12 µg/Kg w.w. and the number of the detected PFAS was significantly lower (n = 4). Moreover, five PCBs were detected in almost all tested samples. The sample with the highest total concentration of PCBs (745 µg/kg w.w.) was UBA-HELCOM 2 (liver of Harbour Porpoise, Germany, sampling years; 2016, 2017, 2020). The lowest total concentrations of PCBs were observed in the Danish samples (mean ∑PFAS; 37.9 µg/Kg w.w.), whereas in Germany, Sweden and Poland the mean cumulative concentrations were comparable. Additionally, the long-term banned organochlorine insecticide 2,4-DDT and its two TPs 4,4-DDD and 4,4-DDE were frequently determined. The total concentration levels were significantly higher in the two harbour porpoise samples from Germany (UBA-HELCOM 2 and 10; 255 and 245 µg/Kg w.w., respectively) in comparison with the rest of liver samples (mean concentration; 45.8 µg/Kg w.w.).

Furthermore, 11 (bio)transformation products [(bio)TPs] of emerging contaminants were detected in the samples, including three TPs of the analgesic drug Tramadol: Nor-Tramadol (N-desmethyl-Tramadol), O-Desmethyl-Tramadol, O-Desmethyldinor-Tramadol; two TPs of Nicotine: Anabasine and Nor-Nicotine; and the metabolite of gabapentin: 3,3-pentamethylene-butyrolactam.

7.1.1 Risk assessment

With regard to a limited number of samples only a simplified risk assessment of individual contaminants could be carried out based on exceedance of available toxicity threshold values. PNEC values for biota were derived from existing PNECs for freshwater (PNECfw; available in the NORMAN Ecotoxicity Database for 64,447 NORMAN SusDat compounds; see also https://www.norman-network.com/nds/ecotox/), using the equation (PNECbio_mw=PNECfw*BCF)/10); where PNECbio_mw is the PNEC for marine water biota (fish), PNECfw is the PNEC for fresh water and BCF is the bioconcentration factor for fish from the US EPA CompTox Database (for values, see NORMAN Substance Factsheets at https://www.norman-network.com/nds/factsheets/). For risk assessment purposes, the lowest PNEC was selected based on availability of background data in the following order (a) existing EQS values; (b) existing experimental PNEC values from reference laboratories; (c) *in silico* predicted PNEC. Please, note that for several compounds either PNECs or BCFs were not available and, consequently, no risk assessment could be carried out. In cases where contaminants were detected at BQL levels, LOQ/2 concentration was used for risk estimation. Such outcomes suggest that there is a need for more sensitive analytical method for these specific analytes.

The detected substances were prioritised based on three indicators: (i) Frequency of Appearance (FoA); (ii) Frequency of PNEC Exceedance (FoE), and (iii) Extent of PNEC Exceedance (EoE). According to the NORMAN Prioritisation Framework (Dulio, 2013), the first indicator expresses at how many sites the compound was detected above the limit of detection (LOD). For more details, see also text above.

The second indicator considers the frequency of monitoring sites with observations of a compound above a certain effect threshold. For the calculation of this indicator, a compound's maximum observed concentration at each site (MEC_{site}) is compared to the lowest PNEC. Subsequently, the number of sites where the threshold was exceeded was divided by the total number of sites where the respective compound was monitored.

Frequency of Exceedance = n / N; n is the number of sites with MECsite > Lowest PNEC; N is the total number of sites where the substance was measured.

The third indicator ranks compounds with regard to the extent of the effects expected. It is defined as the 95th percentile of all MEC_{site} values per compound (MEC_{95}) divided by the PNEC. The resulting hazard ratio is then scaled from 0 to 1 as follows:

EoE = MEC95 / Lowest PNEC; MEC95/lowest PNEC <1 = 0; 10≥ MEC95/lowest PNEC≥1 =0.1; 100≥ MEC95/lowest PNEC>10 = 0.2; 1000≥ MEC95/lowest PNEC>100 =0.5; MEC95/lowest PNEC>1000 = 1.

The Risk Score is the linear combination of the indicators scaled from 0 to 1. Considering that in this project only one concentration measurement per site was available, values of MEC (not MEC_{95}) were taken for calculations.

Analyses of 11 (pooled) liver samples of marine mammals revealed the presence of 33 compounds, which exceeded their ecotoxicological threshold value in at least one sample (**Table**

7). Most of the compounds exceeded their PNEC values (FoE) in less than four samples (FoE <0.634).

4,4-DDE, the stable metabolite of DDT, PCB 101, l-PFOS and PFDeA seem to be of high environmental concern, as their concentrations exceeded the respective PNEC values in all tested samples. More PCB congeners (PCB 138 and 153) and the personal care product methylparaben exceeded their ecotoxicological threshold in 10 (FoA 0.909) samples, whereas PCB 180 and 52 were detected at concentration levels above their PNECs in 9 samples.

Table 7: Risk assessment of compounds exceeding their PNEC/EQS values (for fish, marine environment). Maximum detected concentrations and PNECs are expressed in µg/kg w.w. Compounds are ranked based on their final score (Sum of FoA+FoE+EoE).

Compound	*Maximum detected conc.*	*PNEC*	*EoE score*	*FoE score*	*FoA score*	*Final score*
4,4-DDE	155	0.005	1.0	1.000	1.0	3.0
PCB 138	138	0.1	1.0	0.909	0.9	2.8
PCB 153	287	0.05	1.0	0.909	0.9	2.8
PCB 180	176	0.08	1.0	0.818	0.8	2.6
PCB 52	124	0.08	1.0	0.818	0.8	2.6
PCB 101	104	0.08	0.5	1.000	1.0	2.5
Methylparaben	964	2.56	0.5	0.909	0.9	2.3
Perfluorodecanoic acid (PFDeA)	16	0.82	0.2	1.000	1.0	2.2
Perfluorooctane sulfonic acid (l-PFOS)	1060	9.1*	0.2	1.000	1.0	2.2
Perfluorooctane sulfonic acid isomers (sum branched isomers)	173	9.1*	0.2	0.727	1.0	1.9
PFNA	46	16.5	0.1	0.364	1.0	1.5
Hexachlorobenzene	49	10*	0.1	0.273	1.0	1.4
Dieldrin	258	0.2	1.0	0.182	0.2	1.4
Octocrylene	56	1.26	0.2	0.545	0.5	1.3
Nor-Nicotine	248	21	0.2	0.364	0.6	1.2
3,3-pentamethylene-butyrolactam	1069	66.7	0.2	0.273	0.7	1.2
N-Butylbenzenesulfonamide	765	9.85	0.2	0.364	0.4	0.9
Pyrethrins: Cinerin II	40	0.15	0.5	0.182	0.2	0.9
Nicotine	51	4.69	0.1	0.364	0.4	0.8

Compound	*Maximum detected conc.*	*PNEC*	*EoE score*	*FoE score*	*FoA score*	*Final score*
Perfluorooctanoic acid (PFOA)	1.35**	0.041	0.2	0.273	0.3	0.7
Perfluorooctane sulfonamide (PFOSA)	6	0.0098	0.5	0.091	0.1	0.7
Carazolol	160	0.28	0.5	0.091	0.1	0.7
Nor-Tramadol (N-Desmethyl-Tramadol)	23	12.4	0.1	0.182	0.4	0.6
Propachlor-OXA	65	2.32	0.2	0.182	0.2	0.6
2,4-DDT	72	31	0.1	0.182	0.3	0.6
2-Hydroxy-ibuprofen	47	2.33	0.2	0.091	0.1	0.4
Enrofloxacin	138	1.41	0.2	0.091	0.1	0.4
Sulfadoxine	8.78**	0.33	0.2	0.091	0.1	0.4
Fenuron	18	0.64	0.2	0.091	0.1	0.4
Metazachlor OA	54	0.55	0.2	0.091	0.1	0.4
Harman	7	0.48	0.2	0.091	0.1	0.4
Ethylparaben	23	7.15	0.1	0.091	0.2	0.4
Benzotriazole (BTR)	37	4.44	0.1	0.091	0.1	0.3

*EQS from 2013/39/EU was used; **LOQ/2 was used; PNEC: predicted no-effect concentration; EoE: Extent of PNEC exceedance; FoE: Frequency of PNEC exceedance; FoA: Frequency of appearance

Regarding the Extent of PNEC Exceedance (EoE), values of EoE≥0.5 were observed for six compounds. The highest EoE was recored for 4,4-DDE, dieldrin and PCBs 52, 138, 153 and 180, whereas the highest final risk score was calculated for 4,4-DDE.

For he majority of the compounds (64%) the maximum detected concentrations were less than 100-fold higher than their ecotoxicological thresholds, whereas for seven compounds (4,4-DDE, dieldrin and PCBs 52, 101, 138, 153 and 180) the maximum reported concentration levels were more than three orders of magnitude higher than their respective PNECs, indicating a potential high environmental risk.

One should be aware that the risk assessment of the detected contaminants was implemented using PNECs for biota at the lower trophic levels (marine fish) as a proxy, since PNEC values for marine mammals are not currently available. A careful scrutiny of the ecotoxicological threshold values and further experimental toxicity evidence is suggested to support the outcomes of this risk assessment.

An alternative approach would be to use the FoA only, however, this might not be representative due to a small number of samples analysed. A novel methodology assessing environmental relevance of detected contaminants in marine mammals via their persistence and bioaccumulation properties in combination with FoA is currently being tested within the LIFE

APEX project (https://lifeapex.eu/). Also, there is a consideration within the NORMAN network to use human toxicity threshold values derived from rat and mice toxicity studies.

7.2 Target analysis of explosives

Each sample was tested for the presence of 23 explosives (list provided by UBA) using QuEChERS sample preparation method followed by LVI-GC-MS analysis. LODs of the studied compounds obtianed by spiking lyophilized biota samples are in **Table 8**.

Table 8: The list of studied nitroaromatic explosives and their LODs in biota samples.

No.	Compound	CAS No.	LOD μg/kg ww
1	2-Amino-4,6-dinitrobenzene	35572-78-2	5
2	4-Amino-2,6-dinitrobenzene	19406-51-0	5
3	2-Amino-4-nitrotoluene	99-55-8	10
4	2-Amino-6-nitrotoluene	603-83-8	10
5	1,2-Dinitrobenzene	528-29-0	5
6	1,3-Dinitrobenzene	99-65-0	5
7	1,4-Dinitrobenzene	100-25-4	5
8	Hexogen	121-82-4	15
9	Nitrobenzene	98-95-3	10
10	Nitroguanidine	556-88-7	15
11	Nitropenta	78-11-5	15
12	2-Nitrotoluene	88-72-2	5
13	3-Nitrotoluene	99-08-1	5
14	4-Nitrotoluene	99-99-0	5
15	Octogen	2691-41-0	15
16	Tetryl	479-45-8	15
17	1,3,5-Trinitrobenzene	99-35-4	5
18	2,4,6-Trinitrophenol	88-89-1	15
19	2,4,6-Trinitrotoluene	118-96-7	2
20	Hexyl	131-73-7	15
21	2,4-Dinitrotoluene	121-14-2	5
22	2,4-Diamino-6-nitrotoluene	6629-29-4	15
23	2,6-Diamino-4-nitrotoluene	59229-75-3	15

None of the studied substances has been detected in any of the samples above its LOD. The newly developed method has been proved robust for analysis of the thermally sensitive and unstable nitroaromatic explosives, featuring very fast sample introduction onto the analytical column followed by rapid separation of substances to avoid their decomposition. The LODs could be further improved by transferring the method to GC-MS/MS system.

7.3 Target analysis of organophosphorous flame retardants and dechlorane-plus compounds

The samples were subjected to target analysis of 13 organophosphorous flame retardants (OPFRs) and two dechlorane-plus compounds using QuEChERS sample preparation method followed by LVI-GC-MS analysis. Five OPFRs were detected, with Tris(3-chloropropyl)phosphate being present in ten out of 12 samples (see **Table 9**). None of the OPFRs exceeded the provisional

PNEC from the NORMAN Ecotoxicology Database, however, their presence is of concern since OPFRs are possibly replacing widely used and already banned polybrominated flame retardants (PBDEs), and their concentrations might increase in future. Based on the model-derived values of PBT properties by JANUS tool (UBA), the OPFRs detected in the marine mammals samples are ranked among persistent compounds.

Table 9: The list of studied organophosphorous flame retardants and dechlorane-plus compounds in biota samples. All concentrations are in µg/kg ww. Lowest PNEC = Lowest PNECbio_marine [µg/kg ww] - for fish.

No.	Compound	CAS No.	Lowest PNEC	No. of Analyses	No. of Analyses with conc > LoQ	Min. Conc.	Max. Conc.
1	Tris(3-chloropropyl)phosphate	1067-98-7		12	10	0.56	1.78
2	Triisobutyl phosphate (TIBP)	126-71-6	13.7	12	3	0.45	0.88
3	Triethyl phosphate (TEP)	78-40-0	81.4	12	2	0.51	1.07
4	Tributyl phosphate	126-73-8	218	12	2	0.65	0.91
5	Tris(3,5-dimethylphenyl)phosphate	25653-16-1		12	1	0.88	0.88
6	Tri-o-cresyl phosphate	78-30-8	20	12	0		
7	Tripropyl phosphate	513-08-6	1.3	12	0		
8	2-Ethylhexyl diphenyl phosphate (EHDP)	1241-94-7	0.5	12	0		
9	Triphenyl phosphate (TPHP)	115-86-6	202	12	0		
10	Tris(2-chloroethyl) phosphate (TCEP)	115-96-8	0.5	12	0		
11	Tris(1,3-dichloro-2-propyl) phosphate (TDCIPP)	13674-87-8	1.3	12	0		
12	Tris(2-butoxyethyl) phosphate	78-51-3	13.8	12	0		
13	Tris(2-isopropylphenyl)phosphate (T2iPPP)	64532-95-2		12	0		
14	Syn-dechlorane plus	135821-03-3		12	0		
15	Anti-dechlorane plus	135821-74-8		12	0		

7.4 Suspect screening

Each sample was screened for presence of 65,690 substances. All substances detected by wide-scope target screening (cf. above) and naturally occurring substances were excluded from the further assessment. Overall, 32 additional contaminants were detected**. Table 10** presents the substance NORMAN ID, common compound name, level of identification (Schymanski, 2014), and CAS number. The substances were ordered based on their use category.

Table 10: Compounds detected by suspect screening in the studied marine mammals samples. The compounds are sorted based on their use category.

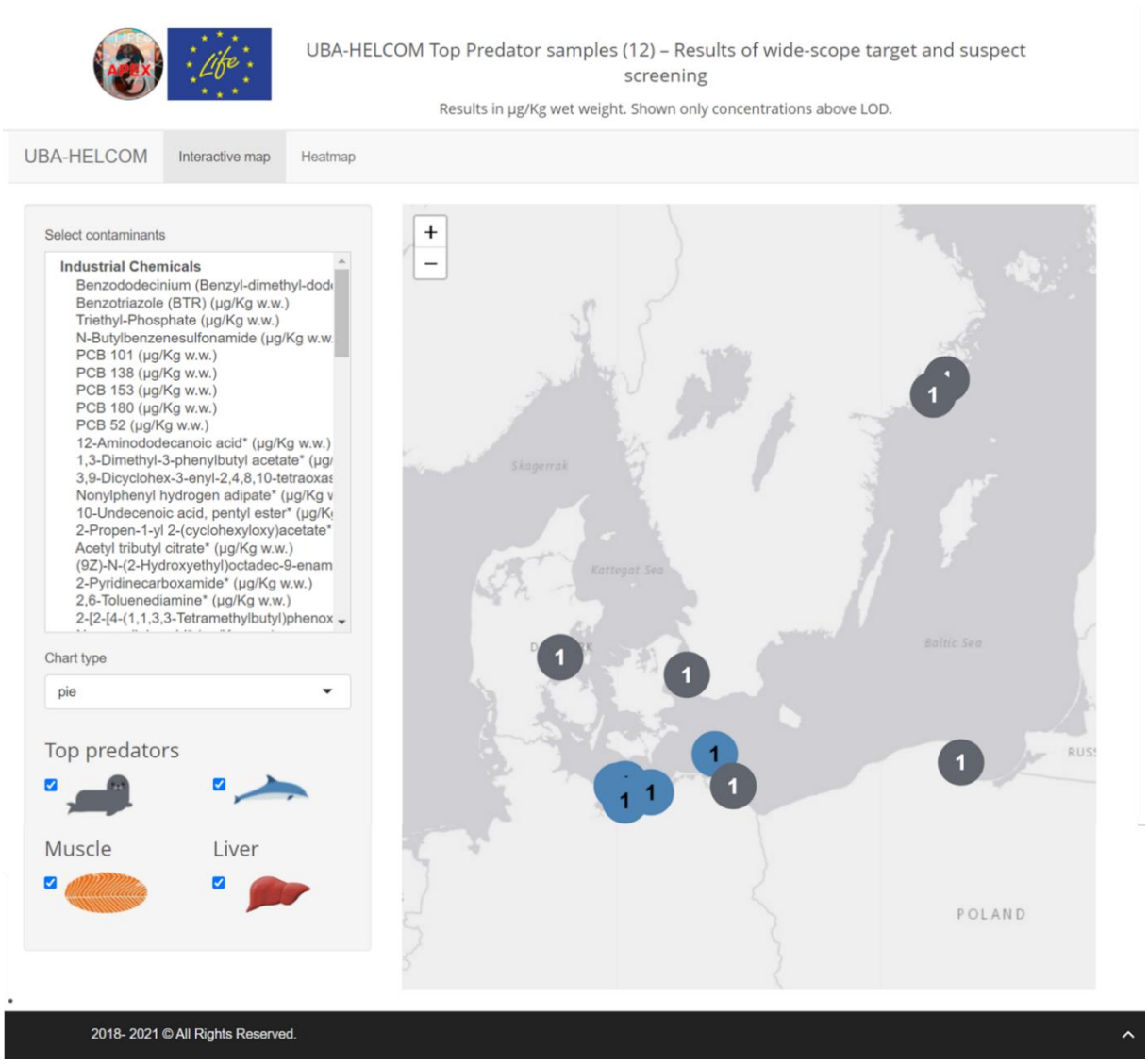
Category	NORMAN ID	Compound name	Level	CAS
Industrial chemicals	NS00002714	2,6-Toluenediamine	3	823-40-5
	NS00004766	Diethylene glycol monophenyl ether	3	104-68-7
	NS00006072	12-Aminododecanoic acid	3	693-57-2
	NS00010302	2-[2-[4-(1,1,3,3-Tetramethylbutyl)phenoxy]ethoxy]ethanol	3	2315-61-9
	NS00010307	Acetyl tributyl citrate	3	77-90-7
	NS00010316	8-Hydroxychinolin	2A	611-36-9
	NS00010726	Erucamide	2A	112-84-5
	NS00011498	Nonanedioic acid	3	123-99-9
	NS00011527	3,9-Dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecane	3	6600-31-3
	NS00011940	2-Propen-1-yl 2-(cyclohexyloxy)acetate	3	68901-15-5
	NS00012325	1,3-Dimethyl-3-phenylbutyl acetate	3	68083-58-9
	NS00013916	(9Z)-N-(2-Hydroxyethyl)octadec-9-enamide	3	111-58-0
	NS00015186	Octanedioic acid	3	505-48-6
	NS00018817	Perfluoro-3,7-dimethyloctanoic acid	3	172155-07-6
	NS00020357	1,3-Benzenedimethanol, 2-hydroxy-5-methyl-	3	91-04-3
	NS00024723	2-Pyridinecarboxamide	3	1452-77-3
	NS00026057	10-Undecenoic acid, pentyl ester	3	18451-96-2
	NS00028312	Bis(methylcyclohexyl) adipate	3	27479-35-2
	NS00049403	2-Ethyloctanedioic acid	3	3971-33-3
	NS00062722	Nonylphenyl hydrogen adipate	3	93982-14-0
Pharmaceuticals	NS00001253	Artemether	3	159573-83-8
	NS00007916	Dobutamine	3	34368-04-2
	NS00014859	2-Quinolinecarboxylic acid, 4-hydroxy-	3	492-27-3
	NS00023357	Betazole	3	105-20-4
	NS00025280	Hydroxyvalerenic acid	3	1619-16-5
	NS00029235	Sobrepin	3	32226-54-3
	NS00031030	4-Piperidone	3	41661-47-6
	NS00039361	Ethylphenylacetylurea	3	90-49-3

Category	NORMAN ID	Compound name	Level	CAS
	NS00040751	2-(4-{[(1R,2S)-2-Hydroxycyclopentyl]methyl}phenyl)propanoic acid	3	371753-19-4
UV filters	NS00004841	Octinoxate	2A	5466-77-3

The majority of the detected substances are widely used and produced in or imported to Europe. 21 out of the 32 chemicals were classified as industrial chemicals and all of them are registered in the ECHA database. The detected substances, which are known to be produced at a very high tonnage (10000-100000 t/a) were: acetyl tributyl citrate, erucamide, octanedioic, 10-undecenoic acid, pentyl ester and nonanedioic acid. Substances falling into the high tonnage band (100-1000 t/a) were diethylene glycol monophenyl ether , 12-aminododecanoic acid and 3,9-dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecane. Substances produced at 10-100 t/a were 2-propen-1-yl 2-(cyclohexyloxy)acetate and 1,3-dimethyl-3-phenylbutyl acetate. All other industrial chemicals were reported either as produced in the low tonnage band (1-10 t/a) or this information was not accessible. The next most important class of detected substances was that of pharmaceuticals (9 compounds). Finally, the UV filter octinoxate was detected at level 2A (tentative identification with library spectrum match). Globally, octinoxate or 2-ethylhexyl-4-methoxycinnamate (EHMC) is one of the most commonly used UV filters in sunscreen and personal care products. Due to its widespread usage, the occurrence of EHMC in the aquatic environment has frequently been documented. In the EU, EHMC is listed under the European Community Rolling Action Plan (CoRAP) as suspected to be persistent, bioaccumulative, and toxic (PBT) and as a potential endocrine disruptor. It was included in the first watch list under the Water Framework Directive (WFD) referring to a sediment PNEC of 200 µg/kg dry weight (dw).

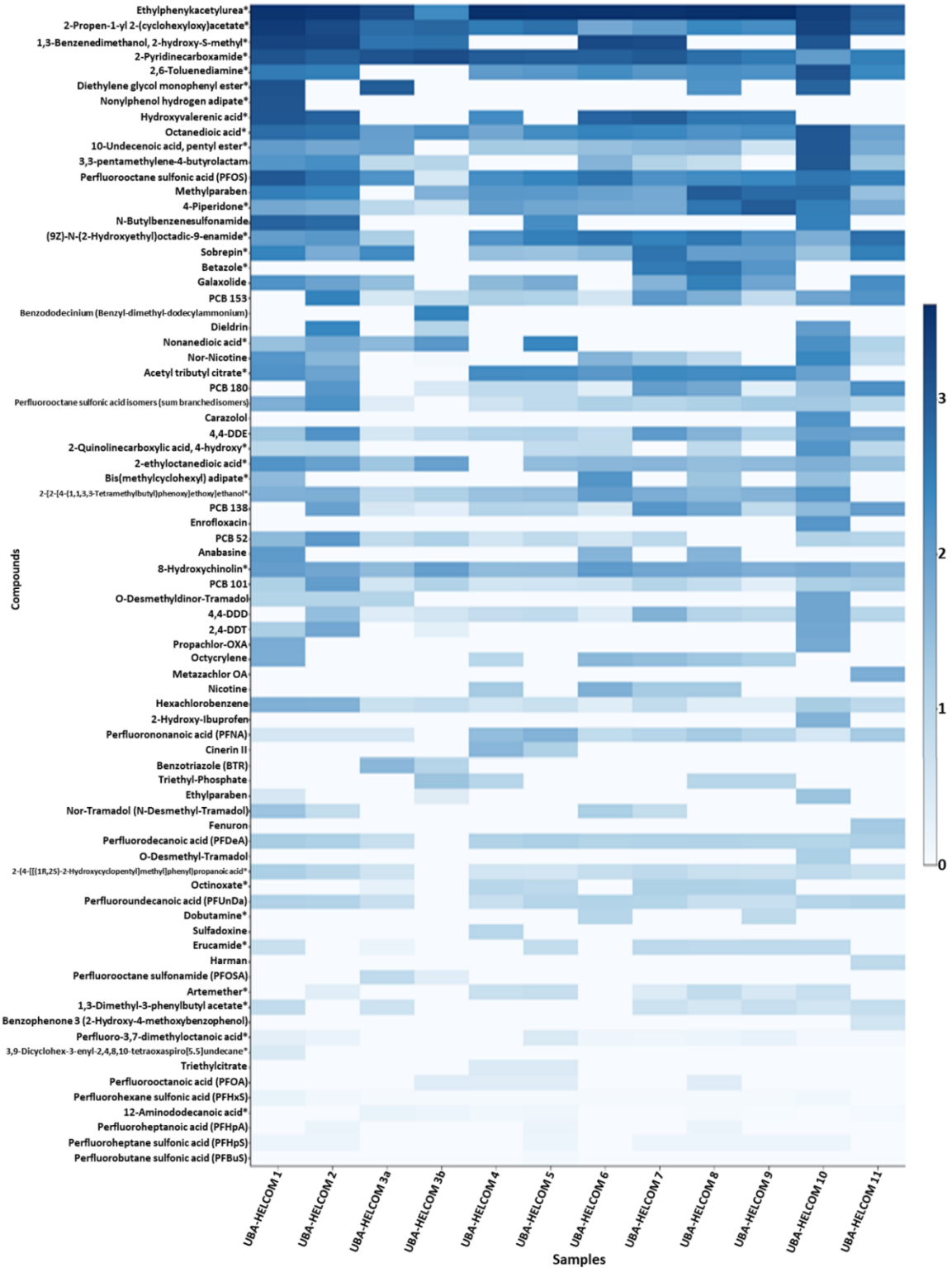
The findings of both target and suspect screening are presented in an application hosted in NORMAN website (**Figure 13**). The results are also summarized in the heatmap (**Figure 14**).

Figure 13: A platform for interactive visual presentation of the results of the wide-scope target and suspect screening of UBA-HELCOM samples. The platform is accessible at https://norman-data.eu/UBA-HELCOM/.



Source: own illustration, EI

Figure 14: Heatmap summarizing the findings of wide-scope target suspect screening of UBA-HELCOM samples. Results from wide-scope target screening are noted with a star (*).Different colour shades represent the concentration levels in µg/kg w.w. (logarithmic scale; see also a bar on the right).



Source: own illustration, EI

7.4.1 Risk assessment

A simplified risk assessment was conducted using the same methodology as in the wide-scope target screening, the only difference being that suspect screening produced semi-quantitative concentration levels based on the structural most similar compound from among a set of internal standard compounds. The purpose of the risk assessment was to rank the detected suspects based on the exceedance of their toxicity threshold values and FoA. PNECs, as presented in **Table 11**, were retrieved from the NORMAN Ecotoxicology Database.

Table 11: Compounds detected by suspect screening analysis in the studied UBA-HELCOM samples and their predicted no-effect concentrations (PNECs).

NORMAN ID	Name in SusDat	Lowest PNECbio_marine [µg/kg ww] - for fish
NS00002714	2,6-Toluenediamine	8.31
NS00004766	Diethylene glycol monophenyl ether	67.2
NS00006072	12-Aminododecanoic acid	34.1
NS00010302	2-[2-[4-(1,1,3,3-Tetramethylbutyl)phenoxy]ethoxy]ethanol	25.6
NS00010307	Acetyl tributyl citrate	2.24
NS00010316	8-Hydroxyquinoline	1.44
NS00010726	Erucamide	0.11
NS00011498	Nonanedioic acid	13.6
NS00011527	3,9-Dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecane	23.6
NS00011940	2-Propen-1-yl 2-(cyclohexyloxy)acetate	7.34
NS00012325	1,3-Dimethyl-3-phenylbutyl acetate	11.4
NS00013916	(9Z)-N-(2-Hydroxyethyl)octadec-9-enamide	1.60
NS00015186	Octanedioic acid	22.6
NS00018817	Perfluoro-3,7-dimethyloctanoic acid	0.46
NS00020357	1,3-Benzenedimethanol, 2-hydroxy-5-methyl-	47.2
NS00024723	2-Pyridinecarboxamide	9.53
NS00026057	10-Undecenoic acid, pentyl ester	5.71
NS00028312	Bis(methylcyclohexyl) adipate	2.24
NS00049403	2-ethyloctanedioic acid	7.04
NS00062722	Nonylphenyl hydrogen adipate	0.47
NS00001253	Artemether	4.25
NS00004841	Octinoxate	295
NS00007916	Dobutamine	2.33

NORMAN ID	Name in SusDat	Lowest PNECbio_marine [µg/kg ww] - for fish
NS00014859	2-Quinolinecarboxylic acid, 4-hydroxy-	2.01
NS00023357	Betazole	6.91
NS00025280	Hydroxyvalerenic acid	1.3
NS00029235	Sobrepin	13.9
NS00031030	4-Piperidone	12.7
NS00039361	Ethylphenylacetylurea	1.58
NS00040751	2-(4-{[(1R,2S)-2-Hydroxycyclopentyl]methyl}phenyl)propanoic acid	1.00

For the final ranking of the compounds, see **Table 8**.

In addition to the three risk assessment scores (see Section 7.1.1), the Exposure Score developed by KEMI, Sweden, was used to confirm the relevance of a compound. KEMI score is based on normalised values (between 0-1) reflecting (i) the degree of uncontrolled release during use, (ii) annual tonnage and (iii) range of use on the market. The underlying data are confidential, but the index allows use of this information for prioritisation purposes and is available in the NORMAN Database System. This index (value ranging from 0 to 1) was higher than 0.3 for 17 compounds (**Table 8)**, indicating that these top-ranked substances are produced in large annual tonnage with widespread use. The 'low scoring' substances were almost exclusively PPPs, pharmaceutical, stimulants & their TPs, which do not fall under REACH legislation. Therefore, it is expected that their KEMI Exposure Score could be underestimated. The NORMAN network is currently working on the development of specific indices for pharmaceuticals and biocides.

Table 8: Substances detected by suspect screening prioritised based on their Risk Score. The table presents the concentration range in µg/kg w.w., Frequency of Appearance (FoA), Frequency of PNEC Exceedance (FoE Score), Extent of PNEC Exceedance (EoE Score) and the total Risk Score (sum of the FoA + FoE + EoE).

Analyte	Concentration range	FoA	FoE Score	EoE Score	Final Risk Score	KEMI Exposure Score
12-Aminododecanoic acid	0.08-0.68	0.58	1.00	1.00	2.58	0.33
1,3-Dimethyl-3-phenylbutyl acetate	2.6-5.8	0.58	1.00	1.00	2.58	0.62
Octinoxate	0.89-12.9	0.50	1.00	1.00	2.50	0.67
3,9-Dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecane	1.92	0.08	1.00	1.00	2.08	0.56
Nonylphenyl hydrogen adipate	1191	0.08	0.92	1.00	2.00	0.09
Ethylphenylacetylurea	222-4114	1.00	0.00	1.00	2.00	0.13
10-Undecenoic acid, pentyl ester	3.6-1093	0.92	0.17	0.50	1.58	0.2
2-Propen-1-yl 2-(cyclohexyloxy)acetate	67.8-2795	1.00	0.00	0.50	1.50	0.67

Analyte	Concentration range	FoA	FoE Score	EoE Score	Final Risk Score	KEMI Exposure Score
Hydroxyvalerenic acid	207-1160	0.58	0.42	0.50	1.50	0
Acetyl tributyl citrate	84.1-230	0.75	0.25	0.50	1.50	0.85
(9Z)-N-(2-Hydroxyethyl)octadec-9-enamide	13.8-513	0.92	0.08	0.50	1.50	0.53
2-Pyridinecarboxamide	109-1540	1.00	0.00	0.50	1.50	0.24
2,6-Toluenediamine	114.7-1305	0.83	0.17	0.50	1.50	0.41
Perfluoro-3,7-dimethyloctanoic acid	0.17-1.9	0.75	0.58	0.10	1.43	0.02
4-Piperidone	3.3-949	1.00	0.17	0.20	1.37	0.13
Artemether	1.3-6.1	0.58	0.67	0.10	1.35	0.02
2-[2-[4-(1,1,3,3-Tetramethylbutyl)phenoxy]ethoxy]ethanol	6.2-143	0.92	0.33	0.10	1.35	0.38
Nonanedioic acid	11.0-251	0.58	0.50	0.20	1.28	0.77
Sobrepin	23.4-458	0.92	0.08	0.20	1.20	0.09
2-ethyloctanedioic acid	20.6-151	0.92	0.08	0.20	1.20	0.13
Bis(methylcyclohexyl) adipate	22.1-148	0.33	0.67	0.20	1.20	0.09
Octanedioic acid	70.1-1127	1.00	0.00	0.20	1.20	0.49
Erucamide	0.57-7.5	0.58	0.42	0.20	1.20	0.7
Diethylene glycol monophenyl ether	160.1-1283	0.33	0.67	0.20	1.20	0.71
2-(4-{[(1R,2S)-2-Hydroxycyclopentyl]methyl}phenyl)propanoic acid	2.7-14.4	0.92	0.08	0.20	1.20	0.02
2-Quinolinecarboxylic acid, 4-hydroxy-	5.8-152	0.58	0.42	0.20	1.20	0.17
Betazole	143-457	0.25	0.75	0.20	1.20	0.13
1,3-Benzenedimethanol, 2-hydroxy-5-methyl-	457-2063	0.58	0.42	0.20	1.20	0.38
8-Hydroxychinolin	32.0-115	1.00	0.00	0.20	1.20	0.62
Dobutamine	7.1-9.3	0.17	0.83	0.10	1.10	0.17

8 Conclusions and recommendations

A wide-scope target and suspect screening of 12 pooled or individual samples from 43 marine mammals' specimen from the Baltic Sea, provided by five HELCOM contracting parties, was carried out by LC-ESI-HR-MS and GC-APCI-HR-MS techniques. Additionally, a GC-EI-MS method has been developed for the determination of 23 compounds contained in explosives formerly dumped into the Baltic Sea. A specific GC-MS target analysis of 13 OPFRs and two dechlorane-plus compounds has been carried out. The wide-scope target screening comprised analysis of 2,540 substances in each sample, whereas suspect screening provided information on presence/absence of 65,690 substances and semi-quantitative estimate of detected substances.

Overall, 47 contaminants were determined through wide-scope target analysis, Most of the detected compounds were per- and polyfluoroalkyl substances (PFAS) (23.4%), followed by plant protection products & TPs (19.1%), industrial chemicals (19.1%) and pharmaceuticals & TPs (8 17.0%). The rest (personal care products, stimulants & TPs and preservatives) accounted for 21.3% of the detected compounds. PCB 101, l-PFOS, hexachlorobenzene and 4,4-DDE (TP of DDT) were detected in all tested samples. PCB 138, PCB 153, two PFAS (PFDeA and the substituted (branched) isomers of PFOS), 4,4-DDD (TP of DDT), as well as methylparaben (anti-fungal agent often used in a variety of cosmetics and personal care products) were present in the majority of samples. Among the detected contaminants, the highest concentration levels were observed for l-PFOS (mean concentration 334 µg/kg w.w.), methylparaben (mean concentration 296 µg/kg w.w.), N-butylbenzenesulfonamide (mean concentration 469 µg/kg w.w.) and the surfactant and antiseptic compound benzododecinium (benzyl-dimethyl-dodecylammonium), which was detected only in the UBA-HELCOM 3b (muscle of common dolphin from Germany) at the concentration of 272 µg/kg w.w.

The highest number of compounds, dominated by pharmaceuticals and their TPs, was determined in the sample UBA-HELCOM 10. The sample contained pharamceuticals that are due to the treatment prior to death (baytril, enrofloraxin), but also showed substances that are rather used in human treatment and are potentially dangerous for marine mammals, such as tramadol, ibuprofen and gabapentin.

The cumulative PFAS concentration of 11 detected compounds was ranging from 179 (UBA-HELCOM 3a) to 1143 (UBA-HELCOM 1) µg/Kg w.w. in the liver samples, whereas in the only analyzed muscle sample (UBA-HELCOM 3b) the total concentration was 5.12 µg/Kg w.w. and the number of the detected PFAS was significantly lower (n = 4). Moreover, five PCBs were detected in almost all tested samples. The sample with the highest total concentration of PCBs (745 µg/kg w.w.) was UBA-HELCOM 2 (liver of Harbour Porpoise, Germany, sampling years; 2016, 2017, 2020). The lowest total concentrations of PCBs were observed in the Danish samples (mean ∑PFAS; 37.9 µg/Kg w.w.), whereas in Germany, Sweden and Poland the mean cumulative concentrations were comparable. Additionally, the long-term banned organochlorine insecticide 2,4-DDT and its two TPs 4,4-DDD and 4,4-DDE were frequently determined. The total concentration levels were significantly higher in the two harbour porpoise samples from Germany (UBA-HELCOM 2 and 10; 255 and 245 µg/Kg w.w., respectively) in comparison with the rest of liver samples (mean concentration; 45.8 µg/Kg w.w.).

Furthermore, 11 (bio)transformation products [(bio)TPs] of emerging contaminants were detected in the samples, including three TPs of the analgesic drug Tramadol: Nor-Tramadol (N-desmethyl-Tramadol), O-Desmethyl-Tramadol, O-Desmethyldinor-Tramadol; two TPs of Nicotine: Anabasine and Nor-Nicotine; and the metabolite of Gabapentin: 3,3-Pentamethylene-butyrolactam.

Regarding limited number of samples only a simplified risk assessment of individual contaminants could be carried out based on exceedance of available toxicity threshold values. The detected substances were prioritised based on three indicators: (i) Frequency of Appearance (FoA); (ii) Frequency of PNEC Exceedance (FoE), and (iii) Extent of PNEC Exceedance (EoE).

Analyses of 11 (pooled) liver samples of marine mammals revealed the presence of 33 compounds, which exceeded their ecotoxicological threshold value in at least one sample. Most of the compounds exceeded their PNEC values (FoE) in less than four samples.

4,4-DDE, the stable metabolite of DDT, PCB 101, l-PFOS and PFDeA seem to be of high environmental concern, as their concentrations exceeded the respective PNEC values in all tested samples. More PCB congeners (PCB 138 and 153) and the personal care product methylparaben exceeded their ecotoxicological threshold in 10 samples, whereas PCB 180 and 52 were detected at concentration levels above their PNECs in nine samples.

For he majority of the compounds (64%) the maximum detected concentrations were less than 100-fold higher than their ecotoxicological thresholds, whereas for seven compounds (4,4-DDE, dieldrin and PCBs 52, 101, 138, 153 and 180) the maximum reported concentration levels were more than three orders of magnitude higher than their respective PNECs, indicating a potential high environmental risk.

One should be aware that the risk assessment of the detected contaminants was implemented using PNECs for biota at the lower trophic levels (marine fish) as a proxy, since PNEC values for marine mammals are not currently available. A careful scrutiny of the ecotoxicological threshold values and further experimental toxicity evidence is suggested to support the outcomes of this risk assessment.

An alternative approach would be to use the FoA only, however, this might not be representative due to a small number of samples analysed. A novel methodology assessing environmental relevance of detected contaminants in marine mammals via their persistence and bioaccumulation properties in combination with FoA is currently being tested within the LIFE APEX project (https://lifeapex.eu/). Also, there is a consideration within the NORMAN network to use human toxicity threshold values derived from rat and mice toxicity studies.

The samples were tested for the presence of 23 explosives (list provided by UBA). None of the studied substances has been detected in any of the samples above its LOD. The LODs could be further improved by transferring the method to GC-MS/MS system.

Five OPFRs were determined in at least one sample, with tris(3-chloropropyl)phosphate being present in ten out of 12 samples. This is of concern, since OPFRs are a possible replacement of the already banned polybrominated diphenyl ethers, are persistent and their cooncentrations may rise in future.

Each sample was screened for presence of 65,690 substances. All substances detected by wide-scope target screening (cf. above) and naturally occurring substances were excluded from the further assessment. Overall, 32 additional contaminants were detected.

The majority of the detected substances are widely used and produced in or imported to Europe. 21 out of the 32 chemicals were classified as industrial chemicals and all of them are registered in the ECHA database. A simplified risk assessment was conducted using the same methodology as in the wide-scope target screening, the only difference being that suspect screening produced semi-quantitative concentration levels based on the structural most similar compound from among a set of internal standard compounds. The purpose of the risk assessment was to rank the detected suspects based on the exceedance of their toxicity threshold values and FoA.

In addition to the three risk assessment scores (see Section 7.1.1), the Exposure Score developed by KEMI, Sweden, was used to confirm the relevance of a compound. KEMI score is based on normalised values (between 0-1) reflecting (i) the degree of uncontrolled release during use, (ii) annual tonnage and (iii) range of use on the market. The underlying data are confidential, but the index allows use of this information for prioritisation purposes and is available in the NORMAN Database System. This index (value ranging from 0 to 1) was higher than 0.3 for 17 compounds indicating that these top-ranked substances are produced in large annual tonnage with widespread use.

All LC-HR-MS and GC-APCI-HR-MS chromatograms were uploaded into the NORMAN DSFP and thus are available for future retrospective screening for any compound detectable by those techniques without the need for additional sampling and analysis. An access to these data is restricted only to the persons identified as eligible by UBA Germany or HELCOM Expert Group on Marine Mammals.

The wide-scope screening data organised in NORMAN Data Collection Templates were uploaded into the LIFE APEX Database System (https://www.norman-network.com/apex/lacod/; a part of the NORMAN Database System), currently accessible only to the project partners (including UBA) and sample providers. A dedicated area was created accessible only to the persons identified as eligible by UBA Germany or HELCOM Expert Group on Marine Mammals. In the end of the LIFE APEX project the data are planned to be transferred to the open access NORMAN Database System – EMPODAT database (https://www.norman-network.com/nds/empodat/).

It is recommended to systematically store data from further screening campaigns of HELCOM countries in the NORMAN Database System (https://www.norman-network.com/nds/), which would allow for their review in comparison with data from other European countries and North America. The occurrence data of this project are now fully comparable to the LIFE APEX project (https://lifeapex.eu/), where samples from marine mammals from several European sea regions are being analyzed following the identical analytical protocols. Also, it is recommended to encourage HELCOM Expert Group on Marine Mammals to provide NORMAN with commonly agreed biota ecotoxicity threshold values (PNECs) for as many substances as possible. This is to facilitate more precise prioritisation of the contaminants detected in biota samples in the Baltic Sea based on the exceedance of these values. Strategies of contaminant analyses should be compared between OSPAR and HELCOM where, additionally to liver, blubber is adviced to be analysed.

Additional efforts are taking place within NORMAN to develop a specific prioritisation scheme taking into account model-predicted PBT (persistence, bioaccumulation, toxicity) values for all substances listed in the Substance Database (https://www.norman-network.com/nds/susdat/). Once ready (expected in the end of 2021), they can be re-applied on the substances identified in the analyzed samples in the current screening/monitoring programmes. Also, there is an on-going discussion with European Chemicals Agency (ECHA) to increase the importance of environmental occurrence data in the substance evaluation scheme and receiving feedback which of the REACH substances (including their transformation products) might be preferably targeted in the updated WFD and MSFD monitoring schemes.

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A Appendix - Sample requirements and shipping instructions

Sample requirements

Species and matrix:

Two pooled samples per country are required, preferably one pooled sample from grey seals and one pooled sample from harbour porpoise. In countries where no whales occur, the second pooled sample could be another seal species. As matrix, liver should be provided for comparability with LIFE APEX results.

To smooth some of the variability between individuals without adding hugely to the analytical costs, a 'sample' will consist of pooled liver tissues from at least 3, but ideally 5 individuals (fewer than 5 would be considered if otherwise a country can't participate, but a minimum of 3 is essential).

The required sample amounts for the **non-target screening (NTS) workflow analyses** (wide-scope target, suspect screening and GC-APCI-HR-MS) are as follows:

- Marine mammals - liver: 29 g ww (minimum) or 52 g ww (optimum) liver weight of pool from three to five individuals in total. Individual samples collected from the same region.

For the analysis, a pooled sample from three to five individuals represents one sample.

- Organisms should preferably be collected in recent years (2015-2020)
- Samples must be stored at -20°C or lower temperatures.
- To avoid any contamination by organic compounds, please wrap each individual sample in aluminium foil scrupulously in two layers and put them then in a zip-bag.

The following information on sample requirements is necessary for the assessment of the analytical results. The information on the samples provided by you needs to be filled into an **Excel table "Biota_sampling protocol"** and provided separately from the samples. This Excel Table (Biota_sampling protocol) contains two sheets:

- Organisation_details – meta-data on sampling provider,
- Sampling_info – absolutely necessary meta-data to be provided for sampling. The grey fields having the drop-down menu, the white ones must be completed in writing.

Information on pooling:

- Same sample region in national waters
- Same age class (preferentially adults)
- Same sex and preferably adult males
- Comparable size
- All individual samples will be pooled and freeze-dried at the University of Athens, Greece. A registration number in accordance to Regulation (EC) No 1069/2009 for receiving animal by-

products and derived products can be forwarded on request. **It should be clearly indicated in the parcel with the samples which individual samples belong to one pool.**

Requested accompanying data:

- Species name
- Sampling approach (e.g. opportunistic or systematic sampling)
- Location (preferentially GPS data)
- Date of sampling/finding
- Weight of every liver sample within each pool
- Biometric data e.g.:
  - Weight, size/length
  - Age or Age class (adult, juvenile, immature)
  - Sex
- Preferably and if available, all animals included in the study should have a full necropsy report including histopathological examination with a summary on the main pathological changes and cause of death. This will be needed to link the contamination to the health status.

Required data on quality assurance:

- Date of freezing and temperature of storage
  - The sample should be maintained in a cold chain (at least -20°C) and ideally send in labeled glass vials
- Recording on confounding factors (euthanisation, medical treatment, potential cause of death, if applicable)
- Description of the state of the sample (for opportunistic samples, e.g., state of autolysis and, if possible, estimated time of death)
  - The sample should originate from a freshly found organism
- Description of handling procedures (e.g. examination, organs dissected)

Shipping instructions

The samples should be shipped by courier (DHL as other couriers might have difficulties to transport dry ice) to:

For the attention of
Prof. Nikolaos S. Thomaidis
Laboratory of Analytical Chemistry (4th floor, laboratory No 8),
Department of Chemistry, University of Athens,
Panepistimiopolis Zografou, GR-15771 Athens, GREECE
Contact person: Dr. Maria-Christina Nika
E-mail: nika_mar-chr@chem.uoa.grailto:, Tel: +30 6937698531, +30 210727-4756, -4576, -4795

IMPORTANT: Prior to shipping, please provide the following sample information by e-mail to Dr. Maria-Christina Nika (nika_mar-chr@chem.uoa.gr):

- Matrix (species, liver/muscle tissue, pooled/not pooled)
- Date of sampling/death
- Region of sampling - coordinates

1. Shipping of non-lyophilised samples with dry ice: Please request dry ice from a local supplier within your country/region:

- Due to sublimation processes, dry ice is produced only on demand;
- Please ask your local supplier for quantities (depending on the weight of sample material) and request adequate boxes for shipment;
- Dry ice needs to be stored at least at -20°C at your institution until the courier (e.g. DHL) will pick the package up to avoid the excessive sublimation.

2. Courier Order: Once you know the delivery date of the dry ice gather the following information:

1. **Size of the parcel (cm x cm x cm)**
2. **Weight of dry ice (kg)**
3. **Weight of the parcel in total (kg)**
4. **Address from where the samples have to be picked up (+ contact details of contact person at your institute, e-mail, phone number).**

IMPORTANT: The transport should ideally take place at the same day or alternatively on the next day of dry ice delivery (around 20% of dry ice sublimates during 24h at 20°C. It is much less if the dry ice is kept at 20°C).
Please ship the samples from Monday to Wednesday.
The shipment should be organised latest on Wednesday in order to ensure the arrival of the samples until Thursday/Friday.
It would be best to send the samples to the University of Athens **before 19 December 2020**, latest shipment on 21 December (Monday). If that is not possible, send the samples in early January 2021.

Receiver:
Please note on the parcel: **for 21 312/4 PROJECT, Prof. N. Thomaidis.**

3. Attached you will also find a template of "Non-Hazardous Content Declaration", required for your shipment. Please replace the marked phrases, according to your shipment information.

B Appendix - Obligatory meta data of sampling, analysis and QA/QC information to be used for filling up Data Collection Template (DCT)

Based on the LIFE APEX Chemical Occurrence Database needs – see https://www.norman-network.com/apex/lacod/downloadDCT.php.

C Appendix – Wide-scope target analysis results

Table 9: Wide-scope target analysis results; unit: µg/kg w.w.

Analyte	UBA-HELCOM 1	UBA-HELCOM 2	UBA-HELCOM 3a	UBA-HELCOM 3b	UBA-HELCOM 4	UBA-HELCOM 5	UBA-HELCOM 6	UBA-HELCOM 7	UBA-HELCOM 8	UBA-HELCOM 9	UBA-HELCOM 10	UBA-HELCOM 11	LOQ (livers)	LOQ (muscles)
3,3-pentamethylene-4-butyrolactam	146	191	7.08	9.60	<LOD	<LOD	44.6	11.4	5.55	<LOD	1069	22.0	3.70	0.727
2-Hydroxy-Ibuprofen	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	46.7	<LOD	22.2	6.95
Nor-Tramadol (N-Desmethyl-Tramadol)	22.7	BQL	<LOD	<LOD	<LOD	<LOD	14.3	BQL	<LOD	<LOD	<LOD	<LOD	11.5	3.59
O-Desmethyl-Tramadol	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	BQL	<LOD	28.9	9.02
O-Desmethyldinor-Tramadol	BQL	BQL	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	77.3	<LOD	21.7	6.77
Carazolol	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	160	<LOD	10.6	0.582
Enrofloxacin	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	138	<LOD	29.4	9.18
Sulfadoxine	<LOD	<LOD	<LOD	<LOD	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	17.6	5.49
Benzophenone 3 (=2-Hydroxy-4-methoxybenzophenon)	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	BQL	6.17	1.93
Galaxolide	159	86.6	29.7	<LOD	34.4	59.6	<LOD	45.0	306	78.6	<LOD	191	9.00	2.81
Octocrylene	56.2	<LOD	<LOD	<LOD	8.68	<LOD	37.2	29.7	21.0	14.8	<LOD	<LOD	8.60	2.69
Ethylparaben	BQL	<LOD	<LOD	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	23.3	<LOD	4.43	2.95
Methylparaben	322	254	<LOD	49.0	129	125	88.6	67.6	964	606	622	27.3	4.49	7.73
Perfluorobutane sulfonic acid (PFBuS)	<LOD	<LOD	<LOD	<LOD	<LOD	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	0.385	0.179
Perfluorodecanoic acid (PFDeA)	15.9	13.5	BQL	<LOD	11.5	14.0	10.6	11.2	11.4	10.8	10.0	14.2	9.78	2.54

Analyte	UBA-HELCOM 1	UBA-HELCOM 2	UBA-HELCOM 3a	UBA-HELCOM 3b	UBA-HELCOM 4	UBA-HELCOM 5	UBA-HELCOM 6	UBA-HELCOM 7	UBA-HELCOM 8	UBA-HELCOM 9	UBA-HELCOM 10	UBA-HELCOM 11	LOQ (livers)	LOQ (muscles)
Perfluoroheptanoic acid (PFHpA)	<LOD	BQL	<LOD	<LOD	<LOD	BQL	<LOD	<LOD	BQL	<LOD	<LOD	<LOD	1.07	0.237
Perfluoroheptane sulfonic acid (PFHpS)	BQL	BQL	<LOD	<LOD	<LOD	BQL	<LOD	BQL	BQL	BQL	BQL	<LOD	0.862	0.355
Perfluorohexane sulfonic acid (PFHxS)	0.694	0.271	BQL	BQL	BQL	BQL	BQL	BQL	BQL	BQL	0.298	<LOD	0.233	0.223
Perfluorononanoic acid (PFNA)	BQL	BQL	BQL	<LOD	30.5	46.2	6.21	9.15	17.3	9.73	BQL	17.5	4.80	1.50
Perfluorooctanoic acid (PFOA)	<LOD	<LOD	<LOD	1.41	BQL	BQL	<LOD	<LOD	BQL	<LOD	<LOD	<LOD	2.69	1.15
Perfluorooctane sulfonic acid (l-PFOS)	1060	500	159	2.38	194	272	454	169	212	243	421	322	2.79	0.253
Perfluorooctane sulfonic acid isomers (sum branched isomers)	51.4	173	BQL	<LOD	3.45	6.83	13.5	9.35	12.8	18.3	19.4	9.20	2.66	0.830
Perfluorooctane sulfonamide (PFOSA)	<LOD	<LOD	6.49	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	1.88	2.44
Perfluoroundecanoic acid (PFUnDA)	12.0	11.1	BQL	<LOD	BQL	10.0	10.9	10.2	BQL	BQL	9.84	11.4	9.35	1.30
2,4-DDT	14.7	70.3	<LOD	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	72.5	<LOD	8.63	2.08
4,4-DDD	<LOD	29.2	BQL	3.01	5.39	6.51	BQL	46.4	10.4	7.85	76.5	9.08	3.16	1.00
4,4-DDE	22.5	155	2.52	6.65	11.2	12.0	5.97	114	44.7	11.9	95.3	83.5	0.196	0.0784
Dieldrin	<LOD	258	<LOD	10.7	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	104	<LOD	8.39	2.62
Hexachlorobenzene	49.4	47.7	4.24	5.46	2.99	4.52	BQL	5.93	BQL	BQL	15.8	7.44	2.89	0.803
Fenuron	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	18.1	9.62	0.800
Pyrethrins: Cinerin II	<LOD	<LOD	<LOD	<LOD	39.7	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	25.4	7.95
Metazachlor OA	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	54.3	8.62	2.69

Analyte	UBA-HELCOM 1	UBA-HELCOM 2	UBA-HELCOM 3a	UBA-HELCOM 3b	UBA-HELCOM 4	UBA-HELCOM 5	UBA-HELCOM 6	UBA-HELCOM 7	UBA-HELCOM 8	UBA-HELCOM 9	UBA-HELCOM 10	UBA-HELCOM 11	LOQ (livers)	LOQ (muscles)
Propachlor-OXA	58.4	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	64.5	<LOD	34.4	10.7
Benzododecinium (Benzyl-dimethyl-dodecylammonium)	<LOD	<LOD	<LOD	272	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	1.23	2.16
Benzotriazole (BTR)	<LOD	<LOD	37.3	9.66	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	14.0	0.523
Triethyl-Phosphate	<LOD	<LOD	<LOD	23.5	BQL	<LOD	<LOD	<LOD	BQL	BQL	<LOD	<LOD	18.1	5.67
N-Butylbenzenesulfonamide	765	598	<LOD	<LOD	<LOD	201	<LOD	<LOD	<LOD	<LOD	312	<LOD	11.5	3.59
PCB 101	13.3	104	3.08	11.4	3.01	3.20	3.67	10.5	5.43	BQL	15.0	17.5	2.44	1.63
PCB 138	<LOD	91.0	BQL	BQL	10.6	9.89	BQL	138	75.8	6.42	34.3	101	4.84	2.43
PCB 153	<LOD	287	2.45	6.63	14.1	11.8	2.85	124	52.9	5.85	79.8	149	1.30	0.771
PCB 180	<LOD	139	<LOD	2.03	6.87	6.88	BQL	102	69.0	BQL	24.7	176	2.63	1.27
PCB 52	34.5	124	6.33	13.3	BQL	6.52	BQL	7.79	<LOD	<LOD	11.4	9.78	5.72	2.51
Triethylcitrate	<LOD	<LOD	<LOD	<LOD	BQL	BQL	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	3.67	1.15
Harman	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD	7.05	3.92	0.562
Nicotine	<LOD	<LOD	<LOD	<LOD	BQL	<LOD	50.8	BQL	BQL	<LOD	<LOD	<LOD	35.0	2.82
Anabasine	121	<LOD	<LOD	<LOD	<LOD	<LOD	43.2	<LOD	42.6	<LOD	<LOD	<LOD	32.8	10.2
Nor-Nicotine	142	40.5	<LOD	BQL	<LOD	<LOD	42.4	19.8	BQL	<LOD	248	BQL	13.6	0.352

*BQL: Below the Limit of Quantification, LOD: Limit of Detection, LOQ: Limit of Quantification

Table 10: Suspect screening analysis results; unit: µg/kg w.w.

Analyte	UBA-HELCOM 1	UBA-HELCOM 2	UBA-HELCOM 3a	UBA-HELCOM 3b	UBA-HELCOM 4	UBA-HELCOM 5	UBA-HELCOM 6	UBA-HELCOM 7	UBA-HELCOM 8	UBA-HELCOM 9	UBA-HELCOM 10	UBA-HELCOM 11
12-Aminododecanoic acid	N.D.	N.D.	0.68	0.48	0.29	0.33	N.D.	N.D.	0.12	0.08	N.D.	0.17
1,3-Dimethyl-3-phenylbutyl acetate	5.83	N.D.	3.62	N.D.	N.D.	N.D.	N.D.	3.71	2.58	4.82	3.05	5.25
Octinoxate	N.D.	N.D.	0.89	N.D.	9.10	7.34	N.D.	12.9	12.7	12.3	N.D.	N.D.
3,9-Dicyclohex-3-enyl-2,4,8,10-tetraoxaspiro[5.5]undecane	1.92	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Nonylphenyl hydrogen adipate	1191	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
Ethylphenylacetylurea	3474	3144	1587	222	3931	3730	3662	3541	3975	4115	2468	953
10-Undecenoic acid, pentyl ester	108	71.3	91.3	N.D.	17.8	17.3	27.4	35.8	39.2	3.61	1094	61.7
2-Propen-1-yl 2-(cyclohexyloxy)acetate	2795	1638	565	678	360	494	67.8	112	249	199	2102	649
Hydroxyvalerenic acid	1160	791	N.D.	N.D.	207	N.D.	736	874	408	399	N.D.	N.D.
Acetyl tributyl citrate	157	84.1	N.D.	N.D.	199	197	127	230	209	221	91.6	N.D.
(9Z)-N-(2-Hydroxyethyl)octadec-9-enamide	106	135	13.8	N.D.	160	313	425	288	403	158	56.0	513
2-Pyridinecarboxamide	1238	848	1232	1540	930	848	825	1034	523	378	109	328
2,6-Toluenediamine	366	315	N.D.	N.D.	115	135	202	137	177	166	1305	240
Perfluoro-3,7-dimethyloctanoic acid	1.05	0.61	N.D.	N.D.	N.D.	1.94	0.38	0.35	0.49	0.73	0.17	0.20
4-Piperidone	69.75	54.52	7.78	3.27	105	74.3	64.9	63.2	430	949	327	57.8
Artemether	N.D.	1.28	N.D.	N.D.	4.54	5.10	N.D.	1.71	6.12	2.21	4.79	N.D.
2-[2-[4-(1,1,3,3-Tetramethylbutyl)phenoxy]ethoxy]ethanol	50.2	55.1	6.24	12.81	24.64	27.78	106	56.4	36.0	45.4	143	N.D.
Nonanedioic acid	26.5	62.1	35.9	128	N.D.	251	N.D.	N.D.	N.D.	N.D.	181	10.956
Sobrepin	275	57.7	207	N.D.	27.0	23.9	36.1	458	98.9	101	23.4	309
2-ethyloctanedioic acid	151	91.4	20.6	90.3	N.D.	31.0	39.0	40.9	29.6	33.9	52.4	26.4
Bis(methylcyclohexyl) adipate	33.8	N.D.	N.D.	N.D.	N.D.	N.D.	148	N.D.	22.1	N.D.	28.6	N.D.
Octanedioic acid	566	474	98.6	173	70.2	203	282	241	165	192	1127	86.1
Erucamide	4.50	N.D.	0.57	N.D.	N.D.	5.76	N.D.	7.54	6.82	7.11	6.89	N.D.

Analyte	UBA-HELCOM 1	UBA-HELCOM 2	UBA-HELCOM 3a	UBA-HELCOM 3b	UBA-HELCOM 4	UBA-HELCOM 5	UBA-HELCOM 6	UBA-HELCOM 7	UBA-HELCOM 8	UBA-HELCOM 9	UBA-HELCOM 10	UBA-HELCOM 11
Diethylene glycol monophenyl ether	1283	N.D.	906	N.D.	N.D.	N.D.	N.D.	N.D.	160	N.D.	731	N.D.
2-(4-{[(1R,2S)-2-Hydroxycyclopentyl]methyl}phenyl)propanoic acid	14.4	8.44	3.29	N.D.	2.97	2.66	5.06	6.96	4.38	4.00	6.64	4.49
2-Quinolinecarboxylic acid, 4-hydroxy-	8.00	9.31	N.D.	N.D.	N.D.	5.79	6.43	N.D.	6.82	N.D.	152	7.89
Betazole	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	370	458	143	N.D.	N.D.
1,3-Benzenedimethanol, 2-hydroxy-5-methyl-	2063	1922	457	489	N.D.	N.D.	1688	1533	N.D.	N.D.	1163	N.D.
8-Hydroxychinolin	101	73.4	32.4	100	31.9	32.2	115	75.7	70.2	52.4	63.0	38.3
Dobutamine	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	9.31	N.D.	N.D.	7.08	N.D.	N.D.

D Appendix - Contaminants below the method's Screening Detection Limit

Table 11: Contaminants that were below the method's Screening Detection Limit (1.71 and 0.375 µg/kg wet weight for livers and muscles, respectively) in the samples analysed by wide-scope target screening.

#	Compound	#	Compound
1	1-(3-carboxypropyl)-3,7-dimethylxanthine	1248	Ibuprofen-Carboxy
2	1,2,3,4,6,7,8-HpCDD	1249	Ifosfamid
3	1,2,3,4,6,7,8-HpCDF	1250	Imatinib
4	1,2,3,4,7,8,9-HpCDF	1251	Imazalil
5	1,2,3,4,7,8-HxCDD	1252	Imazamethabenz-methyl
6	1,2,3,4,7,8-HxCDF	1253	Imazamethabenz-Methyl
7	1,2,3,6,7,8-HxCDD	1254	Imazamox
8	1,2,3,6,7,8-HxCDF	1255	Imazapyr
9	1,2,3,7,8,9-HxCDD	1256	Imazaquin
10	1,2,3,7,8,9-HxCDF	1257	Imazethapyr
11	1,2,3,7,8-PeCDD	1258	Imazosulfuron
12	1,2,3,7,8-PeCDF	1259	Imibenconazole
13	1,2,3-Trichlorobenzene	1260	Imidacloprid
14	1,2-Benzisothiazolinone	1261	Imidacloprid-guanidine
15	1,2-Dinitrobenzene	1262	Imidacloprid-urea
16	1,3-Dinitrobenzene	1263	Imidazolidinon- 1-3-Dimethyl-2-
17	1,4-Dinitrobenzene	1264	Imidocarb
18	11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	1265	Iminostilbene
19	11-Ketotestosterone	1266	Imipramine
20	16-OH E1 (1-3-5(10)-estratrien-3 16-diol-17-one/16-hydroxyestrone)	1267	Inabenfide
21	17alpha-Hydroxyprogesterone	1268	Indanazoline
22	17beta-Estradiol	1269	Indapamide
23	1H,1H,2H,2H-perfluorodecan sulfonate (8:2)	1270	Indeno(1,2,3-cd)pyrene
24	1H,1H,2H,2H-perfluorohexanesulfonate (4:2)	1271	Indinavir
25	1H,1H,2H,2H-perfluorooctane sulfonate (6:2)	1272	Indomethacin

#	Compound	#	Compound
26	2 2 3-triazole-5-OH (1.2.3)	1273	Indoprofen
27	2 C-D	1274	Indoramin
28	2 C-P	1275	Indoxacarb
29	2-(2-(Chlorophenyl)amino)benzaldehyde	1276	Iobitridol
30	2-(4-Morpholinyl)benzothiazole	1277	Iodixanol
31	2,3,4,6,7,8-HxCDF	1278	Iodofenphos
32	2,3,4,7,8-PeCDF	1279	Iodofenphos (Jodfenphos)
33	2,3,5-Trimethacarb	1280	Iohexol
34	2,3,7,8-TCDD	1281	Iomeprol
35	2,3,7,8-TCDF	1282	Iopamidol
36	2,4,5,6-Tetrachloro-m-xylene	1283	Iopodic acid
37	2,4,6-Tri-tert-butylphenol	1284	Iopromid
38	2,4-Diamino-6-Nitrotoluene	1285	Ioversol
39	2,4-Diaminobenzenesulfonic acid	1286	Ioxitalamin acid
40	2,4-Dinitrotoluene	1287	Ioxynil
41	2,6-Diamino-4-Nitrotoluene	1288	Ipratropium
42	2.4.5-T	1289	Iprindol
43	2.4.5-T-methylester	1290	Iprobenfos
44	2.4-D	1291	Iprodione
45	2.4-DB	1292	Iprodione
46	2.4-DB-methylester	1293	Iprovalicarb
47	2.4-D-butylester	1294	Iprovalicarb Isomer 1
48	2.4-D-methylester	1295	Iprovalicarb Isomer 2
49	2-4-Dinitrophenol (DNP)	1296	IQ
50	2-5-DMA	1297	Irbesartan
51	25I-NBoMe	1298	Irgarol
52	2-Amido-3,5,6-trichloro-4-cyanobenzenesulfonic acid	1299	Irgarol-descyclopropyl
53	2-Benzothiazolesulfonic acid	1300	Isazofos
54	2C-B	1301	Isazophos

#	Compound	#	Compound
55	2C-C	1302	Isoaminile
56	2-C-C-NBoMe	1303	Isocarbamid (Azolamide)
57	2C-E	1304	Isocarbophos
58	2C-H	1305	Isoconazole
59	2C-I	1306	Isodrin
60	2CT-2	1307	Isofenphos
61	2C-T-4	1308	Isofenphos Isomer 1
62	2C-T-7	1309	Isofenphos Isomer 2
63	2F-Methamphetamine	1310	Isofenphos methyl
64	2-Hydroxycarbamazepine	1311	Isofenphos-methyl
65	2-Methyl-3,5-Dinitroaniline	1312	Isoniazide
66	2-Methyl-3-Nitroaniline	1313	Isophorone diamine
67	2-Methyl-4-amino-6-methoxy-s-triazine	1314	Isoprocarb
68	2-Methyl-5-Nitroaniline	1315	Isopropalin
69	2-Morpholinothiobenzothiazole	1316	Isoproturon
70	2-Naphthalin sulfonic acid	1317	Isoproturon-didemethyl (1-(4-Isoprophenyl)urea)
71	2-Nitrotoluene	1318	Isothipendyl
72	2-Octyl-4-isothiazolin-3-one	1319	Isoxaben
73	2-Oxo-3-hydroxy-LSD	1320	Isoxadifen-ethyl
74	2-Trifluoromethyl-benzenesulfonamide	1321	Isoxaflutole
75	3,3,4,4,5,5,6,6,7,7,8,8,8-Tridecafluoro-1-octanesulfonic acid	1322	Isoxathion
76	3,4,5-Trichlorophenol	1323	Isoxsuprine
77	3,4,5-Trimethacarb	1324	Isradipine
78	3.5.6-Trichloro-2-pyridinol	1325	Ivermectin B1a
79	3-4-(dichlorophenyl)-3-methyl urea	1326	Ivermectin B1b
80	3-4-(dichlorophenyl)-urea	1327	JWH-007
81	3-Iodopropynyl butylcarbamate	1328	JWH-015
82	3-Nitrotoluene	1329	JWH-018
83	4,4'-Dichlorobenzophenone	1330	JWH-018-1-Methyl-Hexyl

#	Compound	#	Compound
84	4,4'-Dichlorobenzophenone	1331	JWH-018-6-Methoxy-Ind
85	4.4-DDT	1332	JWH-018-Adamantoyl. AB-001
86	4`-Aminoacetanilide	1333	JWH-018-M-2-OH-Ind
87	4-5-Dichloro-2-n-octyl-isothiazol-3(2H)-on (DCOIT)	1334	JWH-018-M-4-OH-Ind
88	4-Acetamido-Antipyrine	1335	JWH-018-M-5-OH-Ind
89	4-AcO-DIPT	1336	JWH-018-M-6-OH-Ind
90	4-Amino-2,6-Dinitrotoluene	1337	JWH-018-M-7-OH-Ind
91	4-Aminobenzamide	1338	JWH-018-M-N-4-OH-Pentyl
92	4-Androsten-11beta-ol-3,17-dione	1339	JWH-018-M-N-5-OH-Pentyl
93	4-Fluorobenzoylpropionic acid	1340	JWH-018-M-N-Pentanoic acid
94	4-Formylamino-Antipyrine	1341	JWH-019
95	4-Formyl-antipyrine	1342	JWH-073
96	4-Hydroxybenzotriazole	1343	JWH-073-2-Methyl
97	4-Hydroxycoumarin	1344	JWH-073-3-Methyl
98	4-Hydroxytamoxifen	1345	JWH-073-M-2-OH-Ind
99	4-Methoxy-1.3-phenylenediamine	1346	JWH-073-M-3-OH-Butyl
100	4-Nitrotoluene	1347	JWH-073-M-4-OH-Butyl
101	4-n-Nonylphenol (NP)	1348	JWH-073-M-4-OH-Ind
102	4-n-Nonylphenol di-ethoxylate (NPE2EO)	1349	JWH-073-M-5-OH-Ind
103	4-n-Nonylphenol mono-ethoxylate (NPE1EO)	1350	JWH-073-M-6-OH-Ind
104	4-n-Octylphenol-di-ethoxylate	1351	JWH-073-M-7-OH-Ind
105	4-n-Octylphenol-mono-ethoxylate	1352	JWH-073-M-N-Butanoic acid
106	4-Trifluoromethylphenol	1353	JWH-081
107	5-Aminonaphthalene-2-sulfonic acid	1354	JWH-122
108	5-Androstan-3-17-diol-3-glucosiduronate (3-diol-3G)	1355	JWH-122-F-Pentyl
109	5-Carboline	1356	JWH-122-M-N-5-OH-Pentyl
110	5-Chloro-2-methyl-4-isothiazolin-3-on (CMI)	1357	JWH-147
111	5-Chlorobenzotriazole	1358	JWH-200
112	5-Fluorouracil	1359	JWH-200-M-4-OH-Ind

#	Compound	#	Compound
113	5-Hydroxyindole-3-acetic acid	1360	JWH-201
114	6-beta-Hydroxycortisol	1361	JWH-203
115	6-Mercaptopurine	1362	JWH-210
116	7-Hydroxymethotrexat	1363	JWH-250
117	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	1364	JWH-251
118	A4 (4-androsten-3 17-dione)	1365	JWH-302
119	ABICA-5F	1366	JWH-370
120	Acamprosat	1367	JWH-398
121	Acebutolol	1368	JWH-412
122	Aceclidine	1369	Kavain
123	Aceclofenac	1370	Ketamine
124	Acemetacin	1371	Ketamine-Nor
125	Acenapthene	1372	Ketazolam
126	Acenapthylene	1373	Ketobemidone
127	Acephate	1374	Ketoconazole
128	Acephate	1375	Ketoprofen
129	Acepromazine	1376	Ketorolac
130	Aceprometazine	1377	Ketotifen
131	Acesulfame	1378	Kresoxim-methyl
132	Acetaminodantrolene	1379	Labetalol
133	Acetamiprid	1380	Lacosamide
134	Acetamiprid	1381	Lactofen
135	Acetazolamide	1382	Lamotrigine
136	Acetiamine	1383	Lansoprazole
137	Acetic acid, (4-chloro-2-methylphenoxy)-	1384	Laudanosine
138	Acetochlor	1385	Lauramidopropylbetaine
139	Acetochlor	1386	Lauric isopropanolamide
140	Acetylsalicylic acid	1387	Lauryl diethanolamide
141	Acibenzolar-S-Methyl	1388	Lauryl sulfate

#	Compound	#	Compound
142	Aciclovir	1389	Lenacil
143	Acifluorfen	1390	Leptophos
144	Aclonifen	1391	Lercanidipine
145	Aconitine	1392	Lethane 384
146	Acridone	1393	Letrozole
147	Acrinathrin	1394	Leuco malachite green (LMG)
148	Acrinathrin	1395	Levamisol
149	Acrivastine	1396	Levetiracetam
150	Actinoquinol	1397	Levobunolol
151	ADB-CHMICA	1398	Levocabastine
152	ADBICA-5F	1399	Levofloxacin
153	ADONA (4,8-dioxa-3H-perfluorononanoate)	1400	Levomepromazine
154	AES-C12, n=0 (Dodecyl hydrogen sulfate)	1401	Levomepromazine-Nor
155	AES-C12, n=1 (2-(Dodecyloxy)ether hydrogen sulfate)	1402	Levomepromazine-sulfoxide
156	AES-C12, n=2 (2-(2-(Dodecyloxy)ethoxy)ether hydrogen sulfate)	1403	Levopropylhexedrine
157	AES-C12, n=3 (2-(2-(2-(Dodecyloxy)ethoxy)ethoxy)ethyl hydrogen sulfate)	1404	Lidocaine
158	AES-C12, n=4 (3,6,9,12-Tetraoxatetracosyl hydrogen sulfate)	1405	Lidocaine-Nor
159	AES-C12, n=5 (3,6,9,12,15-Pentaoxaheptacosyl hydrogen sulfate)	1406	Lidocaine-N-oxide
160	AES-C12, n=6 (3,6,9,12,15,18-Hexaoxatriacontyl hydrogen sulfate)	1407	Lincomycin
161	AES-C12, n=7 (3,6,9,12,15,18,21-Heptaoxatritriacontyl hydrogen sulfate)	1408	Linezolid
162	AES-C12, n=8 (3,6,9,12,15,18,21,24-Octaoxahexatriacontyl hydrogen sulfate)	1409	Linuron
163	AES-C12, n=9 (3,6,9,12,15,18,21,24,27-Monaoxanonatriacontyl hydrogen sulfate)	1410	Linuron
164	Agomelatine	1411	Lisinopril
165	a-HCH	1412	Lisuride
166	Ajmaline	1413	Lonazolac
167	Alachlor	1414	Lopinavir
168	Alachlor	1415	Loratadine

#	Compound	#	Compound
169	Alachlor-ESA	1416	Lorazepam
170	Alachlor-OXA	1417	Lormetazepam
171	Alanycarb	1418	Losartan
172	Albendazole	1419	Lovastatin
173	Albendazole sulfone	1420	LSD-Iso
174	Albuterol / Salbutamol	1421	LSD-Nor
175	Aldicarb	1422	LSD-OH (2-oxo-3-OH-LSD)
176	Aldicarb-sulfone (Aldoxycarb)	1423	L-Thyroxine
177	Aldicarb-sulfoxide	1424	Lufenuron
178	Aldosterone	1425	Lycopsamine
179	Aldrin	1426	Lycopsamine-N-oxide
180	Alfentanyl	1427	Mabuterol
181	Alimemazine	1428	Malachite green (MG)
182	Aliskiren	1429	Malaoxon
183	Alitame	1430	Malaoxon
184	Alizapride	1431	Malathion
185	Allethrin I	1432	Malathion
186	Allethrin II	1433	MAM (6-O-Monoacetylmorphine)
187	Allethrin Isomer 1	1434	Maprotiline
188	Allethrin Isomer 2	1435	Marbofloxacin
189	Allidochlor	1436	Mazindol
190	Almitrine	1437	MBDB
191	Alprazolam	1438	MBZP (3-)
192	Alprazolam- alpha-Hydroxy	1439	MBZP (N-)
193	Alprenolol	1440	MCPA
194	Althiazide	1441	MCPA-methylester
195	Altretamine	1442	MCPB
196	Alypin	1443	MDA
197	AM-1220	1444	MDAI

#	Compound	#	Compound
198	AM-2201	1445	MDAT
199	AM-2233	1446	MDDMA
200	AM-694	1447	MDDMA-bk
201	Amantadine	1448	MDEA
202	Ambroxol	1449	MDMA
203	Amcinonide	1450	MDPBP
204	AMDOPH	1451	MDPPP
205	Ametryn	1452	MDPV
206	Amfepramone	1453	Mebendazole
207	Amidephrine	1454	Mebeverine
208	Amidosulfobetaine-14	1455	MEC (4-)
209	Amidosulfuron	1456	Mecarbam
210	Amiloride	1457	Meclofenamic Acid
211	Aminobenzimidazole (2-)	1458	Meclonazepam
212	Aminocarb (Metacil)	1459	Meclozine
213	Aminoglutethimide	1460	Mecoprop
214	Aminoheptan (2-)	1461	Mecoprop-methylester
215	Aminophenazone. Amidopyrin	1462	Medazepam
216	Aminorex Isomer 1	1463	Medroxyprogesterone
217	Aminorex Isomer 2	1464	Medroxyprogesteroneacetate
218	Amiodarone	1465	Mefenacet
219	Amisulpiride	1466	Mefenamic acid
220	Amisulpride-N-Oxide	1467	Mefenorex
221	Amitraz	1468	Mefenpyr-diethyl
222	Amitriptyline	1469	Mefexamide
223	Amitrole	1470	Mefloquine
224	Amlodipine	1471	Mefluidide
225	Amorolfine	1472	Mefruside
226	Amoxapine	1473	Megestrol-17-acetate

#	Compound	#	Compound
227	Amoxicillin	1474	MeIQX
228	Amphetamin -2-Fluoro	1475	Melatonin
229	Amphetamine	1476	Melitracen
230	Amphetamine (3-F-)	1477	Meloxicam
231	Amphetamine (4-F-)	1478	Melperone
232	Amphetamine-dehydrometh (DHMEPH)	1479	Melphalan
233	Amphetamine-Methyl (methamphetamine)	1480	Memantine
234	Amphetamine-N-Ethyl	1481	MeO-AMT (5-)
235	Amphetamine-N-Propyl	1482	MeO-DALT (5-)
236	Amphetamine-P-hydroxy	1483	MeO-DIPT (5-)
237	Ampicillin	1484	MeO-DMT (5-)
238	Amrinone	1485	MeOT (5-)
239	Anastrozole	1486	MeO-TMT (5-)
240	Anatabine	1487	Mepanipyrim
241	Ancymidol	1488	Mepanipyrim
242	Andarine met	1489	Meperidine
243	Androstenedione-6a-hydroxy	1490	Mephedrone
244	Androsterone glycuronide	1491	Mepindolol
245	Anilazine	1492	Mepivacaine
246	Anilofos	1493	MePPP (4-4-Methyl-pyrrolidinopropiophenone)
247	Antazoline	1494	Meprobamate
248	Anthracene	1495	Mepronil
249	APB (5-)	1496	Meptazinol
250	APB (6-)	1497	Mepyramine. Pyrilamine. Bromth
251	Apomorphine	1498	Mequitazine
252	Apophedrin (Phenylethanolamine)	1499	Mercaptopurine (6-)
253	Apraclonidin	1500	Meropenem
254	Aprindine	1501	Mescaline
255	Apronalide	1502	Mesocarb-P-Hydroxy

#	Compound	#	Compound
256	Aramite (total)	1503	Mesoridazine
257	Arecoline	1504	Mesotrion-MNBA
258	Aripiprazole	1505	Mesterolone
259	Arprinocid	1506	Metaclazepam
260	Asana (Esfenvalerate)	1507	Metalaxyl
261	Asenapine	1508	Metamitron
262	Aspartame	1509	Metamitron-desamino
263	Aspon	1510	Metaxalone
264	Astemizole	1511	Metazachlor
265	Asulam	1512	Metazachlor
266	Atazanavir	1513	Metazachlor BH 479-11
267	Atenolol	1514	Metazachlor BH 479-9
268	Atenolol acid (Metoprolol acid)	1515	Metazachlor BH479-12
269	Atenolol-desisopropyl	1516	Metazachlor-ESA
270	Atomoxetine	1517	Metconazole
271	Atorvastatin	1518	Metconazole
272	Atraton	1519	Metenolone acetate
273	Atrazine	1520	Metformin
274	Atrazine	1521	Methabenzthiazuron
275	Atrazine-2-hydroxy	1522	Methacrifos
276	Atrazine-desethyl	1523	Methacrifos
277	Atrazine-desethyl-2-hydroxy (=Prometon-Hydroxy-Desisopropyl)	1524	Methacrylamide
278	Atrazine-desethyl-desisopropyl	1525	Methadone
279	Atropine	1526	Methadone-Nor
280	AvermectinB1a (Abamectin)	1527	Methamidophos
281	AvermectinB1b (Abamectin)	1528	Methamphetamine (3-F-)
282	Axeen Isomer 1 (Proxabarbal. Proxibarbital)	1529	Methamphetamine (4-F-)
283	Azaconazole	1530	Methamphetamine-P-Hydroxy

#	Compound	#	Compound
284	Azacyclonol	1531	Methaphenilene
285	Azadirachtin	1532	Methaqualone
286	Azamethiphos	1533	Methazolamide
287	Azapropazone	1534	Methcathinone
288	Azatadine	1535	Methcathinone (3-F-)
289	Azelastine	1536	Methcathinone (4-F-/ 4-FMC. Flephedrone)
290	Azimsulfuron	1537	Methedrone
291	Azinphos-ethyl	1538	Methenolone
292	Azinphos-methyl (Guthion)	1539	Methfuroxam
293	Aziprotryne	1540	Methidathion
294	Azithromycin	1541	Methidathion
295	Azoxystrobin	1542	Methimazole
296	Azoxystrobin acid	1543	Methiocarb (Mercaptodimethur)
297	Baclofen	1544	Methiocarb-sulfone
298	Bambuterol	1545	Methiocarb-sulfoxide
299	Bamifylline	1546	Methiocarb-sulfoxide phenol
300	Bamipine	1547	Methiopropamine
301	Barban	1548	Methocarbamol
302	Barbital	1549	Methohexital
303	Barbital-Amo	1550	Methomyl
304	Barbital-Brallo	1551	Methomyl
305	Barbital-Secbuta	1552	Methoprene
306	Barbital-Seco	1553	Methoprotryne
307	Barverin	1554	Methoprotryne
308	BDB	1555	Methotrexate
309	Beclamide	1556	Methoxetamine
310	Beflubutamid	1557	Methoxychlor (DMTD)
311	Beflubutamide	1558	Methoxyfenozide
312	Befunolol	1559	Methoxyphenamine

#	Compound	#	Compound
313	Benactyzine	1560	Methsuximide
314	Benalaxyl	1561	Methyl 2-dimethoxyphosphinothioylsulfanylacetate (formothion methanolyse)
315	Benalaxyl	1562	Methyl arachidate
316	Benazolin	1563	Methyl behenate
317	Bendiocarb	1564	Methyl decanoate
318	Bendroflumethiazide	1565	Methyl dodecanoate
319	Benfluralin	1566	Methyl hexanoate
320	Benfuracarb	1567	Methyl myristate
321	Benodanil	1568	Methyl palmitate
322	Benomyl (decomposed to Carbendazim)	1569	Methyl stearate
323	Benorilate	1570	Methylaminophenazone (4-)
324	Benoxacor	1571	Methylclothiazide
325	Benperidol	1572	Methylephedrine
326	Benproperine	1573	Methylone (MDMC)
327	Benserazide	1574	Methylphenidate
328	Bensulfuron-methyl	1575	Methysergide
329	Bensulide	1576	Metipranolol
330	Bensultap	1577	Metixene
331	Bentazepam	1578	Metobromuron
332	Bentazone	1579	Metoclopramide
333	Benthiavalicarb-isopropyl	1580	Metolachlor
334	Benzamidine	1581	Metolachlor
335	Benzatropine	1582	Metolachlor CGA 357704
336	Benzenesulfonamide	1583	Metolachlor CGA 368208
337	Benzenesulfonate-3-nitro	1584	Metolachlor-ESA
338	Benzenesulfonate-4-hydroxy	1585	Metolachlor-morpholinon
339	Benzenesulfonic acid	1586	Metolachlor-NOA 413173
340	Benzethonium	1587	Metolachlor-OXA

#	Compound	#	Compound
341	Benzo(a)anthracene	1588	Metolazone
342	Benzo(a)pyrene	1589	Metolcarb
343	Benzo(b)fluoranthene	1590	Metolcarb
344	Benzo(g,h,i)perylene	1591	Metominostrobin 1 (Z-Isomer)
345	Benzo(k)fluoranthene	1592	Metominostrobin 2 (E-Isomer)
346	Benzocaine	1593	Metoprolol
347	Benzoctamine	1594	Metopropol tartrate
348	benzoic acid	1595	Metosulam
349	Benzoic acid-3 5-dibromo-4-hydroxy-	1596	Metoxuron
350	benzoic acid-3-Phenoxy	1597	Metrafenone
351	Benzophenone- 2-Amino-5-chloro	1598	Metribuzin
352	benzophenone -2-Amino-5-nitro	1599	Metribuzin
353	Benzophenone-4	1600	Metribuzin-Desamino (DA)
354	Benzothiazole (BTH)	1601	Metribuzin-Diketo (DK)
355	Benzothiazole- 2-Amino	1602	Metronidazole
356	Benzothiazole- 2-Me-S	1603	Metsulfuron-methyl
357	Benzothiazole -2-OH	1604	Metyrapone
358	benzothiazole-Mercapto	1605	Mevinphos
359	Benzotriazole -1-Methyl	1606	Mevinphos (Phosdrin)
360	Benzotriazole -1-OH	1607	Mexacarbate
361	Benzotriazole- 4-Hydroxy	1608	Mexiletine
362	Benzotriazole- 5-Me / Benzotriazole -4-Me	1609	MGK-264
363	Benzotriazole-5 6-di-Me	1610	Mianserine
364	Benzotriazole-5-carboxylic acid	1611	Mianserine-Nor
365	benzotriazole-chloro	1612	Mianserine-N-oxide
366	Benzoximate	1613	Miconazole
367	Benzoxonium	1614	Midazolam
368	Benzoylecgonine	1615	Midazolam -1-Hydroxy
369	Benzoylprop-ethyl	1616	Milnacipran

#	Compound	#	Compound
370	Benzthiazide	1617	Minoxidil
371	Benzthiazuron	1618	Mirtazapine
372	Benzyldimethylhexadecylammonium	1619	Mirtazapine- 8-OH
373	Benzyldimethyltetradecylammonium	1620	Mirtazapine-Desmethyl (normirtazapine)
374	Benzylpiperazine	1621	Mizolastine
375	Benzyltetronic acid (2-)	1622	Moclobemide
376	betamethasone acetate	1623	Modafinil
377	Betamethasone dipropionate	1624	Molinate
378	Betamethasone-17-valerate	1625	Molindone
379	Betaxolol	1626	Mometasone fuorate
380	Bethanidine	1627	Monensin
381	Bexarotene	1628	Monocrotaline
382	Bezafibrate	1629	Monocrotaline-N-oxide
383	b-HCH	1630	Monocrotophos
384	Bicalutamide	1631	Monocrotophos
385	Bifenazate	1632	Monolinuron
386	Bifenazate	1633	Montelukast
387	Bifenox	1634	Monuron
388	Bifenox	1635	Moperone
389	Bifenox acid	1636	Morantel
390	Bifenthrin	1637	Morphine (MOR)
391	Bifenthrin	1638	Morphine-3-beta-D-glucuronide
392	Bioresmethrin	1639	Morphine-6-beta-D-glucuronide
393	Biperidene	1640	Morphine-Dihydro
394	Bis(2-ethylhexyl)phthalat	1641	Morphine-Ethyl
395	Bisoprolol	1642	Morphine-Nor
396	Bisphenol A (BPA)	1643	Moxaverine
397	Bisphenol S	1644	Moxidectin
398	Bispyribac	1645	Moxisylyte

#	Compound	#	Compound
399	Bitertanol	1646	Moxonidine
400	Bornaprine	1647	MPPH (5-(p-Methylphenyl)-5-phenylhydantoin)
401	Boscalid	1648	MTA (4-)
402	Bosentan	1649	Myclobutanil
403	Brefedrone	1650	Myclobutanil
404	Brinzolamide	1651	Mycophenolic acid
405	Brodifacoum	1652	N'-(2 4-Dimethylphenyl)-N-methylformamidine
406	Bromacil	1653	N,N-Dimethyldecylamine
407	Bromadiolone	1654	N,N-Dimethyldodecylamine
408	Bromazepam	1655	N,N-Dimethyldodecylamine N-oxide
409	Bromhexine	1656	N,N-Dimethylsulfamide
410	Bromochlorophen	1657	N,N-Dimethyltetradecylamine
411	Bromocyclen	1658	N,N-Dimethyltetradecylamine-N-oxide
412	Bromodragonfly	1659	N-2-4-Dimethylphenylformamide (DMF. Metabolite Amitraz)
413	Bromophos (Bromophos-methyl)	1660	Nabumetone
414	Bromophos Ethyl	1661	N-Acetyl mesalazine
415	Bromophos Methyl	1662	Nadolol
416	Bromophos-ethyl	1663	Naftifine
417	Bromopropylate	1664	Nalbuphine
418	Bromoxynil	1665	Naled
419	Bromperidol	1666	Nalidixic acid
420	Brompheniramine	1667	Nalorphine
421	Bromuconazole	1668	Naloxone
422	Bromural (Bromisoval)	1669	Naltrexone
423	Bronopol	1670	Nandrolone
424	Brotizolam	1671	Nandrolone phenylpropionate
425	Brucine	1672	Naphazoline
426	Bucetin	1673	Naphthalene
427	Budesonide	1674	Naphthalin sulfonic acid (2-)

#	Compound	#	Compound
428	Budipine	1675	Naphthyl acetic acid (1-)
429	Bufexamac	1676	Naphyrone
430	Bulbocapnine	1677	Napropamide
431	Bumetanide	1678	Napropamide
432	Bunitrolol	1679	Naproxen
433	Buphedrone	1680	Naptalam (N-1-Naphthylphthalamic acid)
434	Buphedrone-4-Methyl	1681	Narasin
435	Bupirimate	1682	Nateglinide
436	Bupirimate	1683	N-bisdesmethyl-Tramadol (dinor-tramadol)
437	Bupivacaine	1684	N-Cyclohexyl-2-benzothiazol-amine
438	Bupranolol	1685	N-Desmethyl-tapentadol
439	Bupranolol- 5-Carboxy	1686	Nebivolol
440	Buprenorphine (BN)	1687	Neburon
441	buprenorphine-Nor	1688	Nefazodone
442	Buprofezin	1689	Nefopam
443	Buprofezin (Z-isomer. Buprofexin)	1690	Neotam
444	Bupropion	1691	N-EtFOSE-M [2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol] acetate adduct
445	Buspirone	1692	N-ethyl-4-methoxybenzamide
446	Butachlor	1693	N-ethylperfluorooctane sulfonamide (N-EtFOSA)
447	Butafenacil	1694	Nicardipine
448	Butamifos	1695	Niclosamide
449	Butizide	1696	Nicosulfuron
450	Butocarboxim	1697	Nifedipine
451	Butocarboxim-sulfoxide	1698	Nifenazone
452	Butoxycaine	1699	Niflumic acid
453	Butoxycarboxim	1700	Nifoxipam
454	Butralin	1701	Nigericin
455	Butter yellow	1702	Nikethamide

#	Compound	#	Compound
456	Buturon	1703	Nilvadipine
457	Butylate	1704	Nimesulide
458	Butylone	1705	Nimorazole
459	Butylparaben	1706	Nisoldipine
460	Cadusafos	1707	Nitenpyram
461	Cadusafos	1708	Nitrazepam
462	Cafaminol	1709	Nitrazepam- 7-Amino
463	Caffeine	1710	Nitrendipin
464	Calteridol	1711	Nitrofen
465	Camazepam	1712	Nitrofen
466	Cambendazol	1713	Nitrofurantoin
467	Candesartan	1714	Nitrothal-isopropyl
468	Cannabidiol	1715	Nizatidine
469	Cannabinol	1716	N-Methyl-2-pyrrolidone
470	Canrenone	1717	N-Methyldodecylamine
471	Capecitabin	1718	N-methylperfluorooctane sulfonamide (N-MeFOSA)
472	Caproylresorcinol	1719	N-methylperfluorooctane sulfonamidoacetic acid
473	Capsaicin	1720	Nomifensine
474	Captafol	1721	Nordextropropoxyphene
475	Captan	1722	Norfloxacin
476	Captopril	1723	Norflurazon
477	Carbamazepine	1724	Norflurazon
478	Carbamazepine -10-Hydroxy	1725	Norgestimate
479	Carbamazepine-10.11-dihydro-10.11 dihydroxy	1726	Norgestrel
480	Carbamazepine-10.11-epoxide	1727	Nor-Phemiramine
481	Carbamazine-Diethyl	1728	Noscapine
482	Carbaryl	1729	Novaluron
483	Carbendazim	1730	Noviflumuron
484	Carbetamide	1731	Novobiocin

#	Compound	#	Compound
485	Carbinoxamine	1732	Nuarimol
486	Carbofuran	1733	Nystatin
487	Carbofuran-3-hydroxy	1734	Obidoxime
488	Carbophenothion	1735	OCDD (Octachlorodibenzo-p-dioxin)
489	Carbophenothion	1736	OCDF (Octachlorodibenzofuran)
490	Carbosulfan	1737	Octachloronapthalene
491	Carboxin	1738	Octopamine
492	Carbutamide	1739	O-Desmethylnor-Tramadol
493	Carbuterol	1740	Ofloxacin
494	Carfentrazone Ethyl	1741	Ofloxacin-N-desmethyl (Impurity E)
495	Carfentrazone-ethyl	1742	Ofurace
496	Carisoprodol	1743	Olanzapine
497	Carprofen	1744	Olopatadine
498	Carteolol	1745	Olsalazine
499	Carticaine (Articaine)	1746	Omeprazole
500	Carvedilol	1747	Omethoate
501	Cathine	1748	Omethoate
502	Cathinone	1749	Ondansetron
503	Cathinone-Dimethyl	1750	Opipramol
504	Cathinone-Ethyl	1751	Orbencarb
505	Cefaclor	1752	ORG 27569
506	Cefadroxil	1753	Ornidazole
507	Cefalexin	1754	Orphenadrine
508	Cefalonium	1755	Orphenadrine-Nor (Tofenacin. Elamol)
509	Cefazolin	1756	Oryzalin
510	Cefoperazone	1757	Oseltamivir
511	Cefquinome	1758	Oseltamivir-carboxylate
512	Ceftazidime	1759	Oxacillin
513	Ceftiofur	1760	Oxadiargyl

#	Compound	#	Compound
514	Celecoxib	1761	Oxadiazon
515	Celiprolol	1762	Oxadiazon
516	Cerivastatin	1763	Oxadixyl
517	Cetirizine	1764	Oxadixyl
518	Cetirizine-Methyl ester	1765	Oxamyl
519	Cetirizine-N-Oxide	1766	Oxasulfuron
520	CGA 321113 (Trifloxystrobin Metabolite)	1767	Oxatomide
521	c-HCH (Lindane)	1768	Oxazepam
522	Chloramben	1769	Oxcarbazepine
523	Chloramphenicol	1770	Oxeladin
524	Chlorantraniliprole	1771	Oxetacaine
525	Chlorazanil	1772	Oxfendazole
526	Chlorbenzoxamine	1773	Oxitropium
527	Chlorbromuron	1774	Oxolinic acid
528	Chlorbufam	1775	Oxomemazine
529	Chlorcyclizine	1776	Oxprenolol
530	Chlordiazepoxide	1777	Oxybuprocaine
531	Chlordiazepoxide-Desmethyl	1778	Oxybutynin
532	Chlordimeform	1779	Oxycarboxin
533	chlorendate-Dibutyl	1780	Oxycodone
534	Chlorfenapyr	1781	Oxycodone-Nor
535	Chlorfenapyr	1782	Oxydemeton-methyl
536	Chlorfenprop-methyl	1783	Oxyfedrine
537	Chlorfenson	1784	Oxyfluorfen
538	Chlorfenvinphos mix of Z&E isomers 1	1785	Oxyfluorfen
539	Chlorfenvinphos mix of Z&E isomers 2	1786	Oxymetazoline
540	Chlorfluazuron	1787	Oxymorphone
541	Chloridazone	1788	Oxypendyl
542	Chloridazone-methyl-desphenyl	1789	Oxypertine

#	Compound	#	Compound
543	Chlorimuronethyl	1790	Oxyphenbutazone
544	Chlormequat	1791	Oxyphencyclimine
545	Chlormezanone	1792	Oxytetracycline
546	Chlorobenzilate	1793	Paclobutrazol
547	Chlorophacinone	1794	Paclobutrazole
548	Chlorophene	1795	Papaverine
549	Chloropropylate	1796	Paracetamol
550	Chloropyramine	1797	Paramethasone
551	Chloroquine	1798	Paraoxon
552	Chloroquine-Hydroxy	1799	Paraoxon Eth
553	Chlorothalonil R611965	1800	Paraoxon-methyl
554	Chlorothalonil-4-hydroxy	1801	Parathion
555	Chlorothiamide	1802	Parathion-Ethyl
556	Chlorothiazide	1803	Parathion-methyl
557	Chlorotoluron	1804	Parathion-Methyl
558	Chloroxuron	1805	Paroxetine
559	Chlorphenethazine	1806	PCB 209
560	Chlorpheniramine	1807	PCB 28
561	Chlorpromazine	1808	Pebulate
562	Chlorpropamide	1809	Pemoline
563	Chlorpropham	1810	Penbutolol
564	Chlorprotixene	1811	Penciclovir
565	Chlorpyrifos Ethyl	1812	Penconazole
566	Chlorpyrifos Methyl	1813	Penconazole
567	Chlorpyriphos	1814	Pencycuron
568	Chlorpyriphos-methyl	1815	Pendimethalin
569	Chlorsulfuron	1816	Pendimethalin
570	Chlortetracycline	1817	Penfluridol
571	Chlorthal-dimethyl	1818	Penfluron

#	Compound	#	Compound
572	Chlorthal-dimethyl (DCPA. Dacthal)	1819	Penicillin V
573	Chlorthalidon	1820	Pentabromobenzyl acrylate
574	Chlorthion	1821	Pentabromoethylbenzene
575	Chlozolinate	1822	Pentachloroaniline
576	Chromafenozide	1823	Pentachlorobenzene
577	Chrysene	1824	Pentachloronapthalene (PCN 52)
578	Cilastatin	1825	Pentachloronapthalene (PCN 54)
579	Cilazapril	1826	Pentachlorophenol
580	Cimaterol	1827	Pentachlorothioanisole
581	Cimetidine	1828	Pentachlorphenol
582	Cinchocaine	1829	Pentanochlor
583	Cinchofen	1830	Pentazocine
584	Cinidon-ethyl	1831	Pentedrone
585	Cinnarizine	1832	Pentifylline
586	Cinosulfuron	1833	Pentobarbital
587	Cinoxacin	1834	Pentoxifylline
588	Ciprofloxacin	1835	Pentoxyverine
589	cis Chlordane	1836	Pentylone
590	Cisapride	1837	Perazine
591	Citalopram	1838	Perfluorobutanoic acid (PFBuA)
592	Citalopram 3-oxo	1839	Perfluorodecane sulfonic acid (PFDS)
593	Citalopram amide	1840	Perfluorododecanoic acid (PFDoDA)
594	Citalopram carboxylic acid	1841	Perfluorohexane sulfonic acid (PFHxS) monosubstituted isomer
595	Citalopram desmethyl (Citalopram-Nor)	1842	Perfluorononane sulfonic acid (PFNS)
596	Clanobutin	1843	Perfluorohexanoic acid (PFHxA)
597	Clarithromycin	1844	Perfluoropentane sulfonic acid (PFPeS)
598	Clarithromycin-N-desmethyl	1845	Perfluoropentanoic acid (PFPeA)
599	Clazuril	1846	Perfluorotetradecanoic acid (PFTeDA)

#	Compound	#	Compound
600	Clemastine	1847	Perfluorotridecanoic acid (PFTrDA)
601	Clenbuterol	1848	Pergolide
602	Clephedrone (4-Chloromethcathinone)	1849	Periciazine
603	Clethodim Peak 1	1850	Perindopril
604	Clibucaine	1851	Permethrin
605	Clidinium	1852	Permethrin Isomer 1
606	Climbazole	1853	Permethrin Isomer 2
607	Climbazole	1854	Perphenazine
608	Clindamycin	1855	Perthane
609	Clobazam	1856	Pethidine
610	Clobenzepam	1857	Pethoxamide
611	Clobetasol	1858	PFHxDA (Perfluoro-n-hexadecainoic acid)
612	Clobetasol propionate	1859	PFODA (Perfluoro-n-octadecanoic acid)
613	Clobetasone butyrate	1860	Phenacetin
614	Clobutinol	1861	Phenanthrene
615	Clodinafop-propargyl	1862	Phenazepam
616	Clofentezine	1863	Phenazepam- 3-Hydroxy
617	Clofibrate	1864	Phenazocine
618	Clofibric acid	1865	Phenazone
619	Clomazone (Command)	1866	Phenazopyridine
620	Clomazone PEAK 1	1867	Phencyclidine
621	Clomazone PEAK 2	1868	Phenelzine
622	Clomethiazole	1869	Phenformin
623	Clomipramine	1870	Pheniramine
624	Clomipramine-Nor	1871	Pheniramine N-Oxide
625	Clonazepam	1872	Pheniramine-Nor
626	Clonazepam- 7-Amino	1873	Phenmedipham
627	Clonidine	1874	Phenmetrazine
628	Clopamide	1875	Phenobarbital

#	Compound	#	Compound
629	Clopenthixol	1876	Phenothrin (tech)
630	Clopidogrel	1877	Phenothrin Isomer 1
631	Clopidogrel Carbon acid	1878	Phenothrin Isomer 2
632	Clopidol	1879	Phenoxybenzamide
633	Clopyralid	1880	Phenoxybenzoic acid (3-)
634	Cloquintocet- 1 -methylhexyl ester	1881	Phenprocoumon
635	Clorprenaline	1882	Phentermine
636	Closantel	1883	Phenthoate
637	Clothiandin	1884	Phenthoate
638	Clothiapine	1885	Phentolamine
639	Clotiazepam	1886	Phenylbenzimidazole sulfonic acid
640	Clotrimazole	1887	Phenylbutazone
641	Clozapine	1888	Phenylephrine
642	Clozapine-Nor	1889	Phenylphenol (2-)
643	Cocaethylene	1890	Phenyltoloxamine
644	Cocaine	1891	Phenytoin
645	Codeine	1892	PHIP
646	Codeine-Dihydro	1893	Pholcodine
647	Codeine-Nor	1894	Pholedrine
648	Colchicine	1895	Phorate
649	Corticosterone	1896	Phorate
650	Cortisol F (4-Pregnene-11b,17a,21-triol-3,20-dione)	1897	Phorate-oxon
651	Cortisone E (4-Pregnene-17,21-diol-3,11,20-trione)	1898	Phosalone
652	Cotinine	1899	Phosalone
653	Cotinine-Hydroxy	1900	Phosmet
654	Coumachlor	1901	Phosmet
655	Coumaphos	1902	Phosphamidon (Dimecron)
656	CP 47-497	1903	Phosphamidon isomer 1
657	CP 47-497-C8	1904	Phosphamidon isomer 2

#	Compound	#	Compound
658	CP 55-940	1905	Phosphate-triethyl
659	CPP (m-)	1906	Phoxim
660	Crimidine	1907	Phthalamic acid
661	Croconazole	1908	Phthalate-Benzyl butyl
662	Crotamiton	1909	Phthalate-Diethyl
663	Crotethamide	1910	Phthalate-Dimethyl
664	Crotoxyphos	1911	Phthalate-Di-n-butyl
665	Crufomate	1912	Phthalate-Di-n-octyl
666	Cyamemazine	1913	Phthalate-Diphenyl
667	Cyanazine	1914	Physostigmine
668	Cyanazine	1915	Picaridin (Icaridin)
669	Cyanofenphos	1916	Picloram
670	Cyanophos	1917	Picolinafen
671	Cyanuric acid	1918	Picoxystrobin
672	Cyazofamid	1919	Pilocarpine
673	Cyclamic acid	1920	Pimozide
674	Cyclizine	1921	Pindolol
675	Cycloate	1922	Pioglitazone
676	Cyclobenzaprine	1923	Pipamperone
677	Cycloheximide	1924	Piperazine
678	Cyclopentolate	1925	Piperazine - 1-3-Trifluoromethylphenyl
679	Cyclophosphamide	1926	Piperazine -1[(4-chlorophenyl) phenyl methyl]
680	Cyclothiazide	1927	Piperazine -1-Piperonyl
681	Cyclovalone	1928	Piperazine-o-Chlorophenyl
682	Cycloxydim	1929	Piperidin carboxamide (4-)
683	Cycluron	1930	Piperonyl butoxide
684	Cyfluthrin (Baythroid)	1931	Pipotiazine
685	Cyfluthrin Isomer 1	1932	Pipradrol
686	Cyfluthrin Isomer 2	1933	Piprozolin

#	Compound	#	Compound
687	Cyfluthrin Isomer 3	1934	Pirenzepine
688	Cyfluthrin Isomer 4	1935	Piretanide
689	Cyhalothrin (lambda-)	1936	Pirimicarb
690	Cyhalothrin-lambda major (mix 2 isomers)	1937	Pirimicarb
691	Cyhalothrin-lambda minor (mix 2 isomers)	1938	Pirimicarb-desmethyl
692	Cymoxanil	1939	Pirimicarb-desmethyl-formamido
693	Cypermethrin Isomer 1	1940	Pirimiphos ethyl
694	Cypermethrin Isomer 2	1941	Pirimiphos-ethyl
695	Cypermethrin Isomer 3	1942	Pirimiphos-methyl
696	Cypermethrin Isomer 4	1943	Piritramide
697	Cyprazin	1944	Pirmenol
698	Cyproconazole	1945	Pitofenone
699	Cyproconazole	1946	Pizotifen
700	Cyprodinil	1947	PMA
701	Cyprodinil	1948	PMMA
702	Cyproheptadine	1949	Polythiazide
703	Cyproterone	1950	PPP- alpha
704	Cyromazine	1951	Practolol
705	Cytarabin	1952	Prajmaline
706	Cythioate	1953	Pramipexole
707	Cytidine -2' 3'-di-O-acetyl-5'-deoxy-5-fluoro	1954	Pravastatin
708	Cytosine- 5-Fluoro	1955	Prazepam
709	D L-N N-Didesmethyl-Venlafaxine	1956	Praziquantel
710	D L-N O-Didesmethyl-Venlafaxine	1957	Prazosin
711	D617 (met. of verapamil)	1958	Prednisolone
712	Dabigatran etexilate	1959	Prednisolone-Methyl
713	Daidzein	1960	Prednisone
714	Daimuron (Dymron)	1961	Pregabalin
715	Dalapon	1962	Prenylamine

#	Compound	#	Compound
716	Daminozide	1963	Pretilachlor
717	Danofloxacin	1964	Prilocaine
718	Dapiprazole	1965	Primaquine
719	Dapsone	1966	Primidone
720	Darunavir	1967	Proadifen
721	Dazomet	1968	Probenecid
722	Debrisoquine	1969	Procainamide
723	Decoquinate	1970	Procaine
724	DEET (Diethyltoluamide)	1971	Prochloraz
725	Deflazacort	1972	Prochloraz BTS40348
726	Deltamethrin	1973	Prochloraz BTS44596
727	Demeton	1974	Prochlorperazine
728	Demeton-S-methylsulfone	1975	Procyclidine
729	Demoxepam	1976	Procymidone
730	Denatonium	1977	Procymidone
731	Denaverine	1978	Profenophos
732	Deprenyl / Selegiline	1979	Profenophos
733	deprenyl-Nor	1980	Profoxydim
734	Deprenyl-N-oxide	1981	Proguanil
735	Deschloroetizolam	1982	Prohexadione
736	Desethylhydroxy-Chloroquine	1983	Prolinamide
737	Desipramine	1984	Prolintane
738	desisopropyl-Atrazine	1985	Promazine
739	Desloratadine	1986	Promazine-amino
740	Desmedipham	1987	Promazine-Propionyl
741	Desmetryn	1988	Promecarb
742	Desonide	1989	Promecarb
743	Desoxycortone 21-(3-phenylpropionate)	1990	Promethazine
744	Desoxycortone enantate	1991	Prometon

#	Compound	#	Compound
745	Desoxypipradol	1992	Prometryn
746	Desphenyl chloridazon	1993	Propachlor
747	DET	1994	Propachlor
748	Detajmium	1995	Propachlor-ESA
749	Dexamethasone	1996	Propafenone
750	Dexfenfluramine	1997	Propafenone-N-Desmethyl
751	Dextromethorphan	1998	Propamocarb
752	Dextropropoxyphene	1999	Propamocarb
753	d-HCH	2000	Propanil
754	Dhydroepiandrosterone (DHEA) / Androstenolone	2001	Propanil
755	Di(2-ethylhexyl)phthalate (DEHP)	2002	Propantheline
756	Diafenthiuron	2003	Propaphos
757	Dialifos	2004	Propaquizafop
758	Diallate	2005	Propargite
759	Diatrizoate (Amidotrizoate acid)	2006	Propazine
760	Diaveridine	2007	Propazine
761	Diazepam	2008	Propazine-2-hydroxy (Prometon-Hydroxy)
762	Diazepam-Nor	2009	Propetamphos
763	Diazinon	2010	Propham
764	Diazinon	2011	Propham
765	Diazinon-O-analog	2012	Propiconazole
766	Dibenzepin	2013	Propipocaine
767	Dibenzo(a,h)anthracene	2014	Propiverine
768	Dibucaine	2015	Propoxur
769	Dibutylone	2016	Propoxycarbazone
770	Dicamba	2017	Propranolol
771	Dicamba-methyl	2018	Propylparaben
772	Dicapthon	2019	Propylthiouracil
773	Dichlobenil	2020	Propyphenazone

#	Compound	#	Compound
774	Dichlofenthion	2021	Propyzamide (Pronamide)
775	Dichlofenthion	2022	Proquazone
776	Dichlofluanid	2023	Proquinazid
777	Dichlofluanid	2024	Proquinazid
778	Dichlormid	2025	Prosulfocarb
779	Dichlorobenzamide	2026	Prosulfuron
780	Dichlorophen	2027	Prothioconazole
781	Dichlorphenamide	2028	Prothioconazole-desethio
782	Dichlorprop	2029	Prothiophos
783	Dichlorprop-methyl	2030	Prothipendyl
784	Dichlorvos	2031	Protionamide
785	Dichlorvos	2032	Protriptyline
786	Diclazepam	2033	Proxyphylline
787	Diclazuril	2034	Psilocin
788	Diclobutrazol	2035	PVP- Alpha
789	Diclobutrazol	2036	Pymetrozine
790	Diclofenac	2037	Pyraclostrobin
791	Diclofop	2038	Pyraflufen Ethyl
792	Diclofop Methyl	2039	Pyraflufen-ethyl
793	Diclofop-methyl	2040	Pyranocoumarin
794	Dicloran	2041	Pyrazolam
795	Dicloxacillin	2042	Pyrazophos
796	Dicofol	2043	Pyrazophos
797	Dicofol	2044	Pyrazoxyfen
798	Dicrotophos	2045	Pyrene
799	Dicycloverine	2046	Pyrethrin I
800	Didecyldimethylammonium (DADMAC (C10:C10))	2047	Pyrethrin II
801	Dienogest	2048	Pyrethrins: Cinerin I
802	Diethazine	2049	Pyrethrins: Jasmolin I

#	Compound	#	Compound
803	Diethofencarb	2050	Pyrethrins: Jasmolin II
804	Difenoconazole	2051	Pyribenzamine (Tripelenamine. Azaron)
805	Difenoxuron	2052	Pyributicarb
806	Difenzoquat (Ion 1+)	2053	Pyridaben
807	Difloxacin	2054	Pyridaphenthion
808	Diflubenzuron	2055	Pyridate
809	Diflufenican	2056	Pyrifenox
810	Diflufenican	2057	Pyrilamine
811	Diflufenzopyr	2058	Pyrimethamine
812	Diflunisal	2059	Pyrimethanil
813	Diglyme	2060	Pyrimethanil
814	Dikegulac	2061	Pyrimidifen
815	Diltiazem	2062	Pyrimidinol
816	Diltiazem-Deacetyl	2063	Pyrimiphos Me
817	Diltiazem-Nor	2064	Pyriproxifen
818	Dimefuron	2065	Pyriproxyfen
819	Dimethachlor	2066	Pyritinol
820	Dimethachlor CGA369873	2067	Pyrovalerone
821	Dimethachlor-ESA	2068	Pyrvinium
822	Dimethachlor-OXA	2069	Quetiapine
823	Dimethenamid	2070	Quetiapine-Hydroxy
824	Dimethenamide	2071	Quinalphos
825	Dimethenamide-ESA	2072	Quinalphos
826	Dimethenamide-OXA	2073	Quinapril
827	Dimethipin	2074	Quinclorac
828	Dimethirimol	2075	Quinidine
829	Dimethoate	2076	Quinine
830	Dimethoate	2077	Quinmerac
831	Dimethocain	2078	Quinoxyfen

#	Compound	#	Compound
832	Dimethomorph	2079	Quintozene (Pentachloronitrobenzene)
833	Dimethyl-5-sulfoisophthalate	2080	Quizalofop
834	Dimethylanilin (N.N-)	2081	Quizalofop-ethyl
835	Dimethylaniline (2.4-) (Metabolite Amitraz)	2082	Rabenzazole
836	Dimethyldioctadecylammonium	2083	Ractopamine
837	Dimethylvinphos	2084	Rafoxanide
838	Dimetindene	2085	Raloxifene
839	Dimetotiazine	2086	Ramifenazone
840	Dimetridazole	2087	Ramipril
841	Dimoxystrobin	2088	Ranitidine
842	Dimoxystrobin	2089	Ranitidine-N-oxide
843	Dinex (2-Cyclohexyl-4.6-dinitrophenol)	2090	Ranitidine-S-oxid (peak 1)
844	Diniconazole	2091	Raubasine
845	Dinocap	2092	RCS-4
846	Dinoseb	2093	RCS-4-M-5-COOH-Pentyl
847	Dinotefuran	2094	RCS-4-M-5-OH-Pentyl
848	Dinoterb	2095	RCS-4-ortho
849	Dioctylsulfosuccinate	2096	RCS-8
850	Diosgenin	2097	Reboxetine
851	Dioxacarb	2098	Remifentanyl
852	Dioxathion	2099	Remoxipride
853	Dioxethedrin	2100	Repaglinide
854	Diphacinone	2101	Reproterol
855	Diphenamid	2102	Reserpine
856	Diphenamide	2103	Resveratrol
857	Diphenhydramine	2104	Retrorsine
858	Diphenoxylate	2105	Retrorsine-N-oxide
859	Diphenylamin-4-(dimethylbutylamino) (6PPD)	2106	Ribavirin
860	Diphenylamine	2107	Rifaximin

#	Compound	#	Compound
861	Diphenylphosphate	2108	Riluzole
862	Diphenylpyraline	2109	Rimantadin
863	Diponium	2110	Rimsulfuron
864	Dipropetryn	2111	Risperidone
865	Diprophylline	2112	Risperidone-9-OH (Paliperidone)
866	DiPT	2113	Ritalinic acid
867	DIPT- 4-OH	2114	Ritonavir
868	Dipyridamole	2115	Rivastigmine
869	Dipyron (Metamizol)	2116	Rizatriptan
870	Diquat (Ion 2+)	2117	Robenidine
871	Disopyramide	2118	Ronidazole
872	Disulfoton	2119	Ropinirole
873	Disulfoton-sulfone	2120	Ropivacaine
874	Disulfoton-sulfoxide	2121	Rosiglitazone
875	Ditalimfos	2122	Rosuvastatin
876	Ditalimfos	2123	Rotenone
877	Dithiopyr	2124	Roxithromycin
878	Diuron	2125	Saccharine
879	Diuron (3-(3,4-Dichlorophenyl)-1,1-dimethylurea) Fragm 187	2126	Salicylamide
880	Dixyrazine	2127	Salicylamide-N-Isopropyl
881	DMA (3-4-)	2128	salicylic acid-4-Benzamido
882	DMAA (Methylhexanamine)	2129	Salicylic acid-5-Amino
883	DMMC (3-4-)	2130	Salicylic acid-thio
884	DMPEA	2131	Salinomycin
885	DMSA (N N-Dimethylaminosulfanilide)	2132	Salmeterol
886	DMST (N.N-Dimethyl-N'-p-tolylsulphamide)	2133	Samoxifen-3-OH-4-MEOMT
887	DMT (Dimethyltryptamine)	2134	Sarafloxacin
888	DNOC (4.6-dinitro-o-cresol)	2135	Schradan
889	DOB	2136	Scopolamine

#	Compound	#	Compound
890	Dobutamine	2137	Scopolamine-Methyl
891	DOC	2138	Sebuthylazine
892	Dodecyl-benzenesulfonate	2139	Secbumeton
893	Dodemorph	2140	Semduramicin
894	Dodine	2141	Semicarbazide (SEM)
895	DOET	2142	Senecionine
896	DOM	2143	Senecionine-N-oxide
897	Domperidone	2144	Seneciphylline
898	Donepezil	2145	Seneciphylline-N-oxide
899	Dorzolamide	2146	Senkirkine
900	Dosulepin	2147	Sertindole
901	Doxapram	2148	Sertraline
902	Doxepin	2149	Sertraline-Nor
903	Doxepin-Nor	2150	Sethoxydim
904	Doxycycline	2151	Sibutramine
905	Doxylamine	2152	Sibutramine-Nor
906	DPT	2153	Siduron
907	Drazoxolon	2154	Sildenafil
908	Drofenine	2155	Sildenafil-Nor
909	Dronedarone	2156	Simazine
910	Droperidol	2157	Simazine 2-Hydroxy
911	Dropropizine	2158	Simetryn
912	Drospirenone	2159	Simvastatin
913	Drostanolone met.1	2160	Sitagliptin
914	Duloxetine	2161	SK8265000
915	Dydrogesterone	2162	Sotalol
916	E1 (1-3-5(10)-estratrien-3-ol-17-one estrone)	2163	Spinosad A (Spinosyn A)
917	E122	2164	Spinosad D
918	E123	2165	Spinosyn A

#	Compound	#	Compound
919	E124	2166	Spinosyn B or K
920	E128	2167	Spirapril
921	E129	2168	Spirodiclofen
922	E1-2-OH (1-3-5(10)-estratrien-2 3-diol-17-one/2-hydroxyestrone)	2169	Spirodiclofen
923	E1-4-OH (1-3-5(10)-estratrien-3 4-diol-17-one/4-hydroxyestrone)	2170	Spiromesifen
924	E3 estriol (1-3-5(10)-estratrien-3 16-17-triol)	2171	Spironolactone
925	Ebastine	2172	Spirotetramat
926	EDDP	2173	Spiroxamine
927	Edifenphos	2174	Spiroxamine Isomer 1
928	EE2 - Ethinyl estradiol	2175	Spiroxamine Isomer 2
929	Efavirenz	2176	Streptomycin-Dihydro
930	Emamectin B1a	2177	Strychnine
931	Emamectin B1b	2178	STS-135
932	Embutramide	2179	Sucralose
933	EME (Ecgonine methyl ester)	2180	Sudan I
934	Emtricitabine	2181	Sudan II
935	Enalapril	2182	Sufentanil
936	Endosulfan alpha	2183	Sulcotrione
937	Endosulfan beta	2184	Sulfabenzamide
938	Endosulfan sulphate	2185	sulfachloropyridazine
939	Endosulfan-sulfate	2186	Sulfaclomide
940	Endothal	2187	Sulfaclozine
941	Endrin	2188	Sulfadiazine
942	Enoximon	2189	Sulfadiazine- N4-Acetyl
943	Entacapon	2190	Sulfadimethoxine
944	Ephedrine	2191	Sulfadimethoxine-N4-Acetyl
945	Ephedrine-Nor (Cathin. Phenylpropanolamine)	2192	Sulfadimidine (Sulfamethazine)
946	Ephedrine-Pseudo	2193	Sulfaethidole

#	Compound	#	Compound
947	Epiandrosterone	2194	Sulfaguanidine
948	Epinephrine	2195	Sulfalene
949	Eplerenone	2196	Sulfamerazine
950	EPN	2197	Sulfameter (sulfumetin)
951	EPN	2198	Sulfamethazine-N4-Acetyl
952	Epoxiconazole	2199	Sulfamethizole
953	Epoxiconazole	2200	Sulfamethoxazole
954	Eprosartan	2201	Sulfamethoxazole (Impurity F)
955	EPTC	2202	Sulfamethoxazole hydroxylamine
956	Ergocristine-Dihydro	2203	Sulfamethoxazole-N4-Acetyl (Impurity A)
957	Ergotamine	2204	Sulfamethoxypyridazine
958	Erythromycin	2205	Sulfamonomethoxine
959	Esculin	2206	Sulfamoxole
960	Esfenvalerate/Fenvalerate Isomer 1	2207	Sulfanilamide
961	Esfenvalerate/Fenvalerate Isomer 2	2208	Sulfapyridine
962	Esmolol	2209	Sulfaquinoxaline
963	Esomeprazole	2210	Sulfasalazine
964	Esprocarb	2211	Sulfathiazole
965	Estazolam	2212	Sulfathiazole- N4-Acetyl
966	ET- alpha	2213	Sulfathiazole-Phthalyl
967	Etaconazole	2214	Sulfinpyrazone
968	Etaconazole	2215	Sulfisoxazole
969	Ethacrynic acid	2216	Sulfometuron-methyl
970	Ethalfluralin	2217	Sulfonic acid-6-Chlorothymol
971	Ethambutol	2218	Sulfotep
972	Ethenzamide	2219	Sulfotepp
973	Ethiofencarb	2220	Sulindac
974	Ethiofencarb-sulfone	2221	Sulpiride
975	Ethiofencarb-sulfoxide	2222	Sulpiride-O-Desmethyl

#	Compound	#	Compound
976	Ethion	2223	Sulprofos (Bolstar)
977	Ethion	2224	Sulthiame
978	Ethiprole	2225	Sumatriptan
979	Ethirimol	2226	Suprofen
980	Ethofumesate	2227	Suxibuzone
981	Ethopabate	2228	SWEP.MCC
982	Ethoprophos	2229	Synephrine
983	Ethoprophos	2230	Tacrine
984	Ethoxyquin	2231	Tadalafil
985	Ethoxysulfuron	2232	Talinolol
986	Ethyl glucuronide	2233	Tamoxifen
987	Ethyl sulfate	2234	TCMTB
988	Ethylone	2235	Tebuconazole
989	Ethylphenidate	2236	Tebuconazole
990	Etilefrine	2237	Tebufenozide
991	Etizolam	2238	Tebufenpyrad
992	Etodolac	2239	Tebufenpyrad
993	Etodroxizine	2240	Tebupirimphos
994	Etofenprox	2241	Tebutame
995	Etofenprox	2242	Tebuthiuron
996	Etofylline	2243	Tecnazene
997	Etoxazole	2244	Teflubenzuron
998	Etoxazole	2245	Tefluthrin
999	Etridiazole	2246	Tefluthrin
1000	Etrimfos	2247	Telmisartan
1001	Exemestane	2248	Temazepam
1002	Famotidine	2249	Temephos
1003	Famoxadone	2250	Tenofovir
1004	Famphur	2251	TEPP

#	Compound	#	Compound
1005	Famphur	2252	Tepraloxydim
1006	Fedrilate	2253	Terazosin
1007	Felodipine	2254	Terbacil
1008	Fenamidone	2255	Terbinafine
1009	Fenamiphos	2256	Terbufos
1010	Fenamiphos	2257	Terbufos
1011	Fenamiphos - sulfone	2258	Terbufos sulfone
1012	Fenamiphos-sulfone	2259	Terbufos-sulfoxide
1013	Fenarimol	2260	Terbumeton
1014	Fenazaquin	2261	Terbumeton
1015	Fenbendazole	2262	Terbutaline
1016	Fenbuconazole	2263	Terbuthylazine
1017	Fenbufen	2264	Terbuthylazine
1018	Fenbutatin Oxide	2265	Terbuthylazine-2-hydroxy
1019	Fencamfamine	2266	Terbuthylazine-desethyl
1020	Fenchlorfos	2267	Terbuthylazine-desethyl-2-hydroxy
1021	Fenchlorphos-oxon	2268	Terbutryn
1022	Fenclofos.Ronnel	2269	Terbutryn
1023	Fendiline	2270	Terbutylazine
1024	Fenefrine-Nor	2271	Terconazole
1025	Fenethylline	2272	Terfenadine
1026	Fenfluramine	2273	Ternidazole
1027	Fenfluramine-Nor	2274	Terodiline
1028	Fenfuram	2275	Tertatolol
1029	Fenhexamide	2276	Testosterone- 17-alpha-methyl
1030	Fenitrothion	2277	Testosterone-6a-hydroxy
1031	Fenitrothion	2278	Tetrabromobisphenol A (TBBP-A)
1032	Fenobucarb	2279	Tetracaine
1033	Fenofibrate	2280	Tetrachloronapthalene (PCN 27)

#	Compound	#	Compound
1034	Fenofibric acid	2281	Tetrachlorophenol-2.3.4.6
1035	Fenoprofen	2282	Tetrachlorosalicylanilide
1036	Fenoprop (Silvex.2.4.5-TP)	2283	Tetrachlorvinphos
1037	Fenoprop-methylester (Silvex-methyester)	2284	Tetrachlorvinphos (Stirofos)
1038	Fenoterol	2285	Tetraconazole
1039	Fenothiocarb	2286	Tetracycline
1040	Fenoxaprop-P	2287	Tetradecylsulfate
1041	Fenoxycarb	2288	Tetradifon
1042	Fenoxycarb	2289	Tetraethylene glycol monododecyl ether
1043	Fenpiclonil	2290	Tetrahydrophthalimide (cis-) (1.2.3.6-)
1044	Fenpipramide	2291	Tetramethrin
1045	Fenpiprane	2292	Tetramethrin Isomer 1
1046	Fenpropathrin	2293	Tetramethrin Isomer 2
1047	Fenpropathrin	2294	Tetrasul
1048	Fenpropidin	2295	Tetrazepam
1049	Fenpropidin	2296	Tetrazepam-Nor
1050	Fenpropimorph	2297	Tetroxoprim
1051	Fenpropimorph	2298	Tetryzoline
1052	Fenproporex (NARL)	2299	THC
1053	Fenpyroximate	2300	THC-COOH. 11-COOH-THC. 11-nor-9-Carboxy-THC
1054	Fenson	2301	THC-OH. 11-OH-THC
1055	Fenson	2302	THE (Tetrahydrocortisone)
1056	Fensulfothion	2303	Thebacon
1057	Fensulfothion-sulfon	2304	Thenylchlor
1058	Fentanyl	2305	Theobromine
1059	Fentanyl -3-Methyl	2306	Theophylline
1060	Fentanyl- 3-Methylnor	2307	THF (Tetrahydrocortisol)
1061	Fentanyl- Alpha-methyl	2308	Thiabendazole
1062	Fentanyl-Nor	2309	Thiacloprid

#	Compound	#	Compound
1063	Fentanyl-Para-fluoro	2310	Thiacloprid-amide
1064	Fenthion	2311	Thiadone
1065	Fenthion	2312	Thiamethoxam
1066	Fenthion-oxon	2313	Thiamphenicol
1067	Fenthion-oxon	2314	Thiazopyr
1068	Fenthion-sulfon	2315	Thidiazuron
1069	Fenthion-sulfoxide	2316	Thiethylperazine
1070	Fenticonazole	2317	Thifensulfuron-methyl
1071	Fentin (triphenylstannylium)	2318	Thiobencarb
1072	Fenvalerate	2319	Thiocyclam
1073	Fexofenadine	2320	Thiodicarb
1074	Finasteride	2321	Thiofanox
1075	Fipronil	2322	Thioguanine
1076	Fipronil	2323	Thiometon
1077	Fipronil-desulfinyl	2324	Thionazin (Zinophos)
1078	Fipronil-sulfide	2325	Thiopental
1079	Fipronil-sulfone	2326	Thiophanate-ethyl
1080	Flamprop	2327	Thiophanate-methyl
1081	Flamprop-isopropyl	2328	Thiopropazate
1082	Flazasulfuron	2329	Thioproperazine
1083	Flecainide	2330	Thioridazine
1084	Flocoumafen	2331	Thioridazine-5-sulfoxide
1085	Floctafenine	2332	Thiothixene
1086	Flonicamid	2333	Thiram (Tetramethylthiuramdisulfide.TMTD)
1087	Flonicamid	2334	Thymopentin
1088	Florfenicol	2335	Tiagabine
1089	Fluacrypyrim	2336	Tiamulin
1090	Fluanisone	2337	Tiapride
1091	Fluazifop-p-butyl	2338	Ticlopidine

#	Compound	#	Compound
1092	Fluazinam	2339	Tiemonium
1093	Fluazuron	2340	Tilidine
1094	Flubendazole	2341	Tilidine-Nor
1095	Fluchloralin	2342	Tilmicosin
1096	Fluchloralin	2343	Timolol
1097	Fluconazole	2344	Tinidazole
1098	Flucycloxuron	2345	Tiocarbazil
1099	Flucythrinate	2346	Tiocarlide
1100	Flucythrinate Isomer 1	2347	Tizanidine
1101	Flucythrinate Isomer 2	2348	TMA
1102	Fludioxonil	2349	Tokuthion (Prothiophos)
1103	Fludioxonil	2350	Tolazamide
1104	Fludrocortisone	2351	Tolazoline
1105	Flufenacet	2352	Tolbutamide
1106	Flufenacet	2353	Tolclofos Methyl
1107	Flufenacet-ESA	2354	Tolclofos-methyl
1108	Flufenacet-OXA	2355	Tolfenamic acid
1109	Flufenamic acid	2356	Tolfenpyrad
1110	Flufenoxuron	2357	Toliprolol
1111	Flufenzine (Diflovidazin)	2358	Tolmetin
1112	Flumazenil	2359	Tolnaftate
1113	Flumequine	2360	Tolpropamine
1114	Flumethasone	2361	Toltrazuril
1115	Flumethrin	2362	Toluenesulfonamide
1116	Flumetsulam	2363	Tolycaine
1117	Flumioxazin	2364	Tolyfluanid
1118	Flumioxazin	2365	Topiramate
1119	Flunisolide	2366	Topotecan
1120	Flunitrazepam	2367	Torasemide

#	Compound	#	Compound
1121	Flunitrazepam- 7-Amino	2368	Toremifene
1122	Flunitrazepam- 7-Amino-desmethyl	2369	Tralkoxydim
1123	Flunitrazepam-N-Desmethyl (Norflunitrazepam)	2370	Tralomethrin
1124	Flunixine	2371	Tramadol
1125	Fluocinolone acetonide	2372	Tramadol-N-oxide
1126	Fluometuron	2373	Tranexamic acid
1127	Fluometuron	2374	trans Chlordane
1128	Fluopicolide	2375	Transfluthrin
1129	Fluoranthene	2376	Tranylcypromine
1130	Fluorene	2377	Trapidil
1131	Fluoroglycofen-ethyl	2378	Trazodone
1132	Fluorometholone	2379	Trenbolone
1133	Fluotrimazole	2380	Triadimefon
1134	Fluoxastrobin	2381	Triadimefon
1135	Fluoxetine	2382	Triadimenol
1136	Fluoxetine-nor	2383	Triallate
1137	Fluoxymesterone met.	2384	Tri-allate
1138	Flupentixol	2385	Triamcinolone
1139	Fluphenazine	2386	Triamcinolone acetonide
1140	Flupirtine	2387	Triamterene
1141	Fluquinconazole	2388	Triasulfuron
1142	Flurazepam	2389	Triazamate
1143	Flurazepam-Desalkyl	2390	Triazolam
1144	Flurbiprofen	2391	Triazolam-1-Hydroxymethyl (alpha-Hydroxy-Triazolam)
1145	Fluridone	2392	Triazophos
1146	Flurochloridone	2393	Triazophos
1147	Fluroxypyr	2394	Triazoxide
1148	Flurprimidol	2395	Tribenuron-methyl
1149	Flurtamone	2396	Tribufos (Merphos oxide. DEF)

#	Compound	#	Compound
1150	Flusilazole	2397	Tributylamine
1151	Flusilazole	2398	Trichlorfon (Dylox)
1152	Fluspirilen	2399	trichlormethiazide
1153	Flutamide	2400	Trichloronapthalene (PCN 13)
1154	Fluticasone propionate	2401	Trichloronate
1155	Flutolanil	2402	Trichloronate
1156	Flutolanil	2403	Trichlorophenol-2.4.6
1157	Flutriafol	2404	Triclabendazole
1158	Fluvalinate (tau-)	2405	Triclocarban
1159	Fluvalinate Isomer 1	2406	Triclopyr
1160	Fluvalinate Isomer 2	2407	Triclopyr-methylester
1161	Fluvastatin	2408	Triclosan
1162	Fluvoxamine	2409	Tricyclazole
1163	Folpet	2410	Trietazine
1164	Fomesafen	2411	Triethylphosphorothioate (O.O.O-)
1165	Fonofos	2412	Trifloxystrobin
1166	Fonofos (Dyfonate)	2413	Trifloxystrobin
1167	Foramsulfuron	2414	Trifloxystrobin CGA 321113
1168	Forchlorfenuron	2415	Trifloxysulfuron
1169	Formetanate	2416	Triflumizole
1170	Formoterol	2417	Triflumizole
1171	Fosinopril	2418	Triflumuron
1172	Fosthiazate	2419	Trifluoperazine
1173	Fosthiazate Isomer 1	2420	Trifluperidol
1174	Fosthiazate Isomer 2	2421	Triflupromazine
1175	Fuberidazole	2422	Trifluralin
1176	Furalaxyl	2423	Triflusulfuron-methyl
1177	Furalaxyl	2424	Triforine
1178	Furathiocarb	2425	Triglyme

#	Compound	#	Compound
1179	Furathiocarb	2426	Trihexyphenidyl. Benzhexol
1180	Furazolidone	2427	Trimeprazine
1181	Furilazole	2428	Trimethacarb (2.3.5-)
1182	Furosemide	2429	Trimethacarb (3.4.5-)
1183	Gabapentin	2430	Trimethobenzamide
1184	Galantamine	2431	Trimethoprim
1185	Galaxolidone	2432	Trimethoprim (Impurity B)
1186	Gallopamil	2433	Trimethyloctylammonium
1187	Gamma-Hydroxybutyric acid	2434	Trimipramine
1188	Gemcitabin	2435	Trimipramine-Nor
1189	Gemfibrozil	2436	Trinexapac acid
1190	Genistein	2437	Trinexapac-ethyl
1191	Gentamycin	2438	Triperiden
1192	GenX (HFPO-DA: , 2,3,3,3-tetrafluoro-2-(heptafluoropropoxy) propanoate)	2439	Triphenyl phosphate
1193	Gestoden	2440	Triprolidine
1194	Gestrinone	2441	Triptyline-Nor
1195	Gibberellic acid	2442	Triticonazole
1196	Glaphenine	2443	Tritoqualine
1197	Glibenclamide	2444	Tromantadine
1198	Glibornuride	2445	Tropicamide
1199	Glimepiride	2446	Tropisetron
1200	Glipizide	2447	Trospium
1201	Glufosinate	2448	Tulobuterol
1202	Griseofulvin	2449	Tylosin
1203	Guaifenesin	2450	Tyrosine-3.5-Diiodo
1204	Guanabenz	2451	Uniconazole
1205	Guanoxan	2452	Uniconazole
1206	Guanylurea	2453	UR-144

#	Compound	#	Compound
1207	Halcinonide	2454	Ursodeoxycholic acid
1208	Halfenprox	2455	Valdecoxib
1209	Halofenozide	2456	Valganciclovir
1210	Halofuginone	2457	Valproic acid
1211	Haloperidol	2458	Valsartan
1212	Haloxyfop ethoxyethyl ester	2459	Vamidothion
1213	Harmaline	2460	Vancomycin
1214	Harmine	2461	Vardenafil
1215	Harpagoside	2462	Varenicline
1216	Heliotrine	2463	Vedaprofen
1217	Heliotrine-N-oxide	2464	Vegadex (Sulfallate)
1218	Heptachlor	2465	Venlafaxine
1219	Heptachlor Epoxide	2466	venlafaxine-N-Desmethyl (norvenlafaxine)
1220	Heptachloronapthalene (PCN 73)	2467	Venlafaxine-N-oxide
1221	Heptenophos	2468	Venlafaxine-O-Desmethyl (Desvenlafaxine)
1222	Heroin	2469	Verapamil
1223	Hexachlorobutadiene	2470	Verapamil-Nor
1224	Hexachloronapthalene (PCN 66)	2471	Vernolate
1225	Hexachloronapthalene (PCN 67)	2472	Vildagliptin
1226	Hexaconazole	2473	Vincamine
1227	Hexadecyltrimethylammonium	2474	Vinclozolin
1228	Hexaflumuron	2475	Vinclozolin
1229	Hexazinone	2476	Warfarin
1230	Hexazinone	2477	WIN-48-098
1231	Hexobendine	2478	WIN-55-212-2
1232	Hexythiazox	2479	Xanthinol
1233	HHMA	2480	Xipamide
1234	Histapyrrodine	2481	XMC
1235	HMA	2482	Yohimbine

#	Compound	#	Compound
1236	HMMA	2483	Zaleplon
1237	Homatropine	2484	Zidovudine
1238	Hordenine	2485	Zimelidine
1239	HU-210	2486	Ziprasidone
1240	Hydrochlorothiazide	2487	Zolmitriptan
1241	Hydrocodone	2488	Zolpidem
1242	Hydrocortisone	2489	Zonisamide
1243	Hydroflumenthiazide	2490	Zopiclone
1244	Hydromorphone	2491	Zotepine
1245	Hydroxyzine	2492	Zoxamide
1246	IAI (5-)	2493	Zuclopenthixol
1247	Ibuprofen		