Norsk institutt for luftforskning
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Analyses of selected organic contaminants and metals in drinking bottles

Technical report

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NILU PROJECT NO.

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CONTRACT REF.

ABSTRACT

On behalf of Norwegian Consumer Council NILU has conducted analyses of organic contaminants and metals in the leachate from selected drinking bottles. The simulation of the leakage is conducted based on a compilation of the methods described within NS-EN-1186-9 and NS-EN-13130-1. The instrumental analytical methods used were already established at NILU and NIVA. A number of different organic contaminants and metals have been found in trace amounts in the different products.

NORWEGIAN TITLE

Analyse av utvalgte miljøgifter og metaller i drikkeflasker og kaffekopper

KEYWORDS

Environmental contaminants | Heavy metals | Elements

ABSTRACT (in Norwegian)

NILU har på vegne av Forbrukerrådet bestemt innholdet av organiske forurensninger og tungmetaller i utlekkingsvæske fra utvalgte drikkeflasker. Simuleringen av lekkasjen er basert på en sammenstilling av metodene beskrevet i NS-EN-1186-9 og NS-EN-13130-1. De instrumentelle analysemetodene som ble benyttet var allerede etablert hos NILU og NIVA. En rekke forskjellige organiske forurensninger og metaller har blitt funnet i spormengder i de ulike produktene.

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Preface

Norwegian Consumer Council was interested in checking for possible leakage of potential harmful substances from drinking bottles that could be drunk by Norwegian consumers.
The aim of the project was to test out for leakage of selected organic contaminants and metals in bottles provided for analyses.
The simulation of the leakage is conducted based on a compilation of the methods described within NS-EN-1186-9 and NS-EN-13130-1.
Analyses of different compounds were conducted with methods established in NILU and NIVA laboratories.

Contents

Summary

On behalf of Norwegian Consumer Council NILU has conducted analyses of organic contaminants and metals in the leachate from selected drinking bottles. The simulation of the leakage was conducted based on a compilation of the methods described within NS-EN-1186-9 and NS-EN-13130-1. The instrumental analytical methods used were already established at NILU and NIVA. A number of different organic contaminants and metals have been found in trace amounts in the different products.

Analyses of selected organic contaminants and metals in drinking bottles

Technical report

1 Introduction

The project was conducted as described in the offer that was sent to Norwegian Consumer Council on 20.12.2017, Nets nr. 1303 and that was accepted a day after. 11 different drinking bottles from various suppliers were purchased in different stores by the staff of Norwegian Consumer Council and delivered to NILU in January 2018. The aim of the project was to test out for leakage of selected organic contaminants and from the bottles provided for analyses. The simulation of the leakage was conducted based on the compilation of the methods described within NS-EN-1186-9 and NS-EN-13130-1.
All analyses of the different compounds were conducted with methods established at NILU, except for UV-filters that were analysed by NIVA.

2 Materials and Methods

2.1 Samples

In order to effectively conduct different analyses in laboratories in different locations, 11 drinking bottles were purchased in December 2017 and delivered in triplicate. A total of 33 bottles were delivered to NILU. Details are described in Table 1. One of the triplicated samples delivered was used for analyses of metals in NILU laboratory in Kjeller, while another two for organic analyses in NILUs laboratories in Kjeller and Tromsø, respectively.

Table 1: Information about bottles subjected to analyses

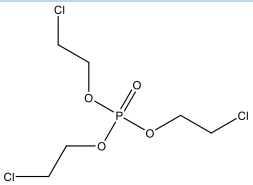
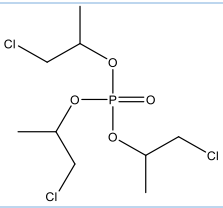
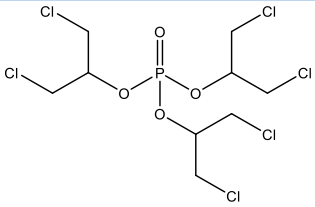
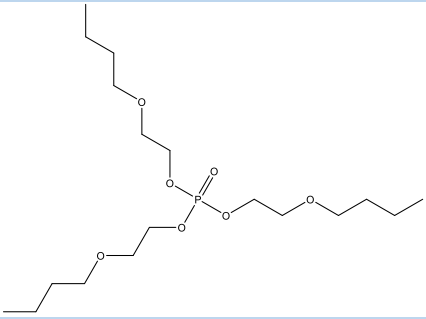
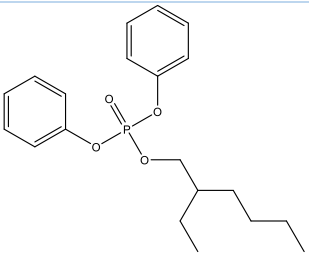
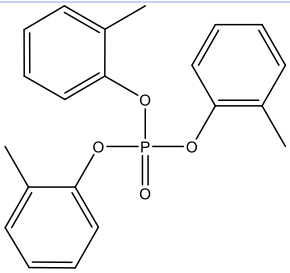
No	Shop	Location of purchase
1	XXL	Alna center
2	G- Sport	Alna center
3	Ikea	Furuset
4	Nille	Jernbanetorget
6	Jernia	Strøget
7	Biltema	Alna center
8	Rusta	Alna
9	Europris	Lindeberg
10	Coop Obs	Alna
12	ToysRus	Alna center
13	Clas Ohlson	Alna center

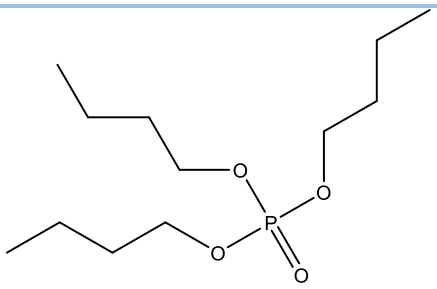
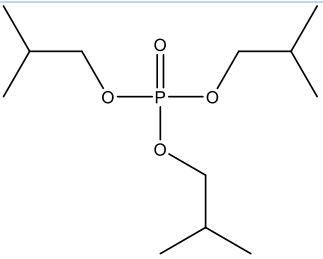
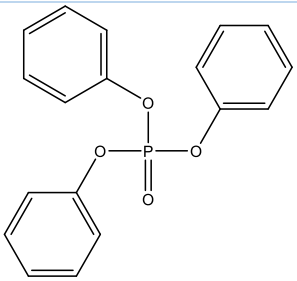
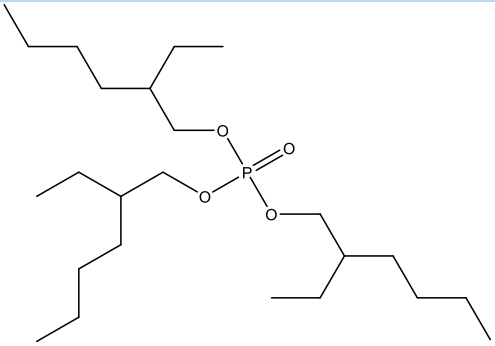
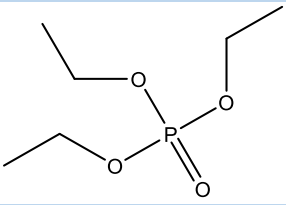
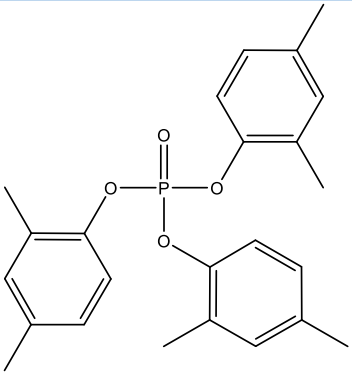
2.2 Compounds tested

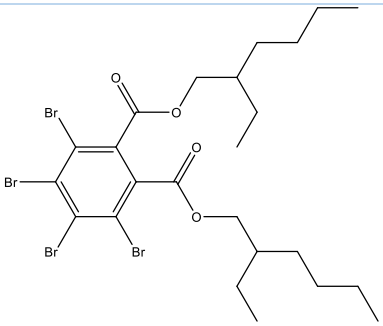
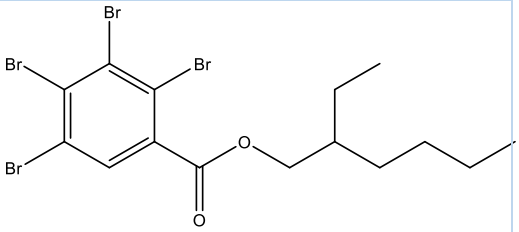
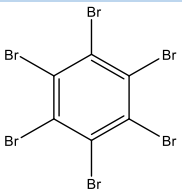
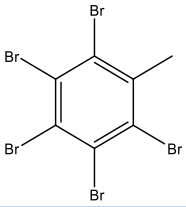
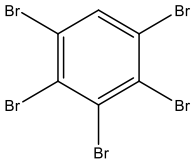
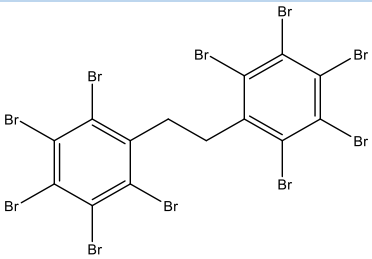
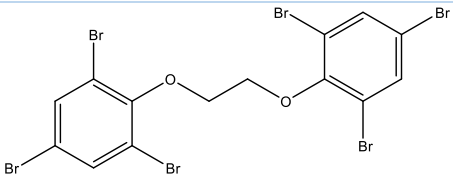
2.2.1 Organic contaminants

Details about the contaminants tested are included in tables 2-5.

Table 2: Selected flame retardants and chlorinated paraffins

Name	Chemical name	CAS number	Structure
Organophosphorus flame retardants			
TCEP	Tris(2-chloroethyl) phosphate	115-96-8	
TCPP	Tris(1-chloro-2-propyl) phosphate	13674-84-5	
TDCPP	Tris(1,3-dichloro-2-propyl)phosphate	13674-87-8	
TBEP	tris-(2-butoxyethyl)-phosphate	78-51-3	
EHDPP	2-ethylhexyl diphenyl phosphate	1241-94-7	
TCP	Tricresyl phosphate	1330-78-5	

Name	Chemical name	CAS number	Structure
TnBP	Tri-n-butyl phosphate	126-73-8	
TiBP	Tri-iso-butyl phosphate	126-71-6	
TPP	Triphenyl phosphate	115-86-6	
TEHP	tris(2-ethylhexyl)phosphate	78-42-2	
TEP	Triethyl phosphate	78-40-0	
TXP	Trixylenyl Phosphate	25155-23-1	

Name	Chemical name	CAS number	Structure
New brominated flame retardants			
BEHTBP	Bis(2-ethylhexyl)tetrabromophthalate	26040-51-7	
EHTBB	2-Ethylhexyl-2,3,4,5-tetrabromobenzoate	183658-27-7	
HBB	Hexabromobenzene	87-82-1	
PBT	Pentabromotoluene	87-83-2	
PBBZ	Pentabromobenzene	608-90-2	
DBDPE	Decabromodiphenylethane	84852-53-9	
BTBPE	1,2-Bis(2,4,6-tribromophenoxy)ethane	37853-59-1	
α-TBECH	α-Tetrabromoethylcyclohexane	1232836-48-4	
B-TBECH	B-Tetrabromocyclohexane	1232836-49-5	

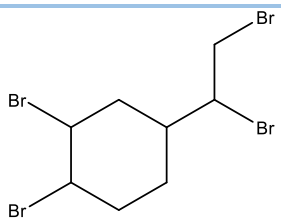
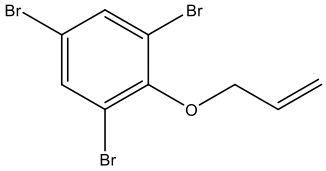
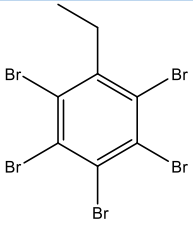
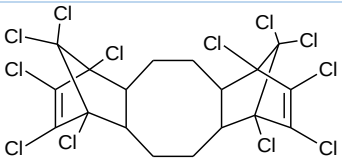
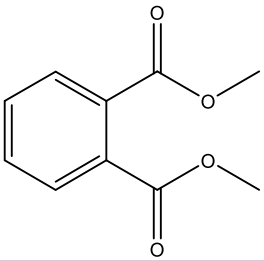
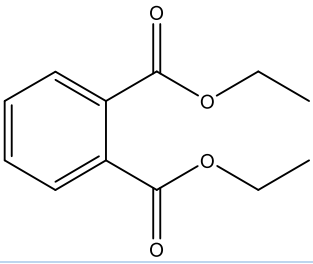
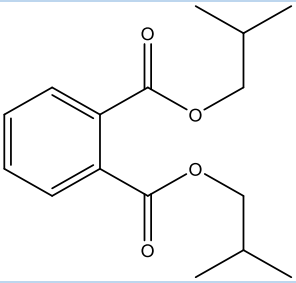
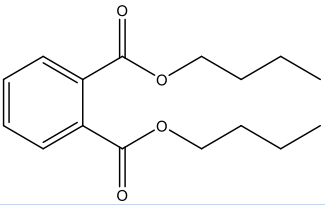
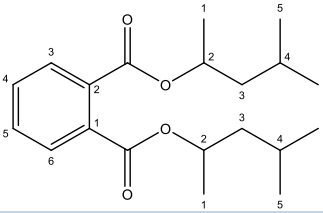
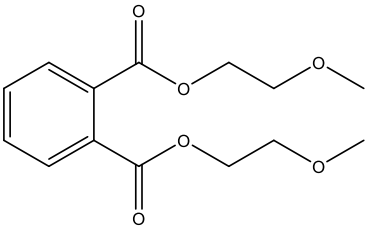
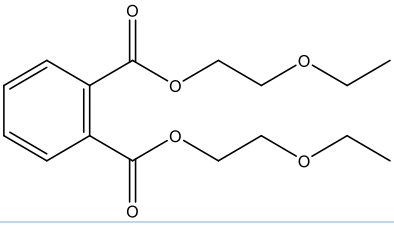
Name	Chemical name	CAS number	Structure
g/d-TBECH	Γ/δ- Tretrabromocyclohexane	Not available	
ATE (TBP-AE)	2,4,6-tribromophenyl allyl ether	3278-89-5	
PBEB	Pentabromoethylbenzene	85-22-3	
Dechlorane Plus (syn/anti)	1,4:7,10-Dimethanodibenzo[a,e]cyclooctene	135821-03-9/135821-74-8	
Chlorinated paraffins			
SCCP	Short chain chlorinated paraffins	85535-84-8	Group of compounds, not a single structure
MCCP	Medium chain chlorinated paraffins	**85535-85**-9	Group of compounds, not a single structure

Table 3: Selected phthalates

Name	Chemical name	CAS number	Structure
DMP	**Dimethylphthalate**	131-11-3	
DEP	**Diethylphthalate**	84-66-2	
DiBP	**diisobutyl phthalate**	84-69-5	
DBP	**di-n-butyl-phthalate**	84-74-2	
BMPP	**Bis(4-metyl-2-pentyl) phthalate**	84-63-9	
BMEP	**Bis(2-metoxyethyl) phthalate**	117-82-8	
BEEP	**Bis(2-etoxyethyl) phthalate**	605-54-9	

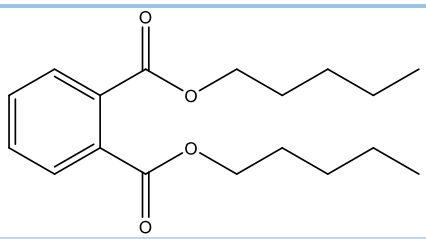
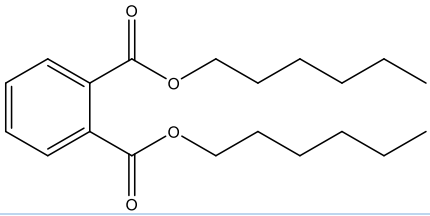
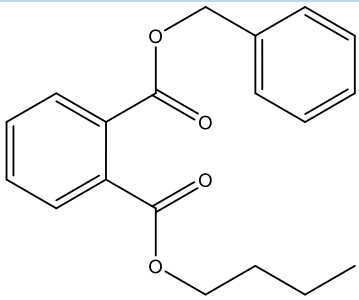
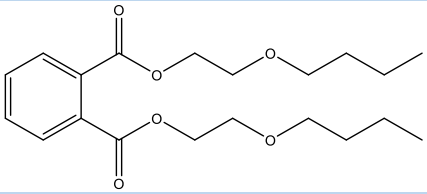
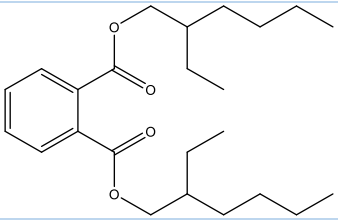
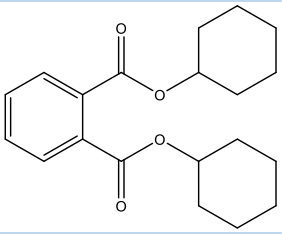
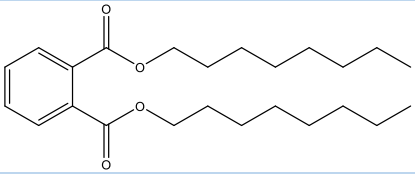
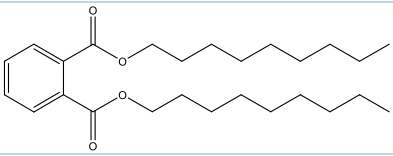
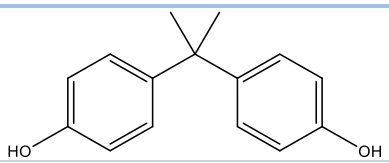
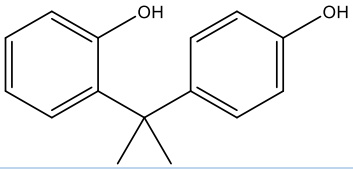
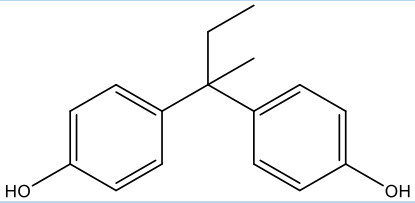
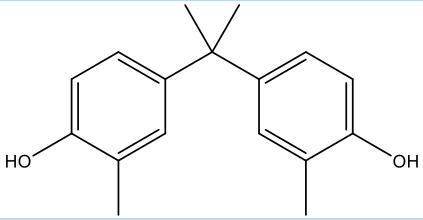
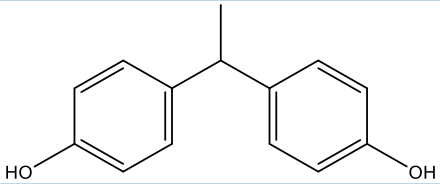
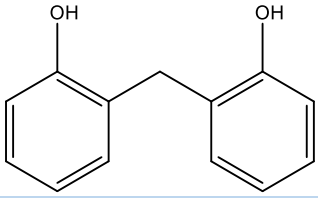
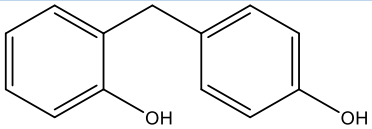
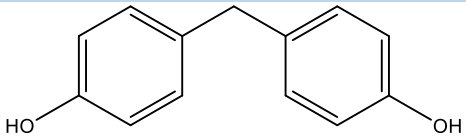
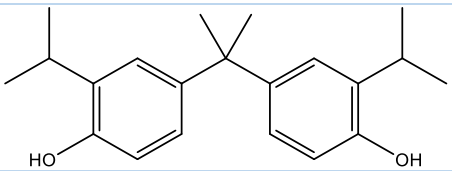
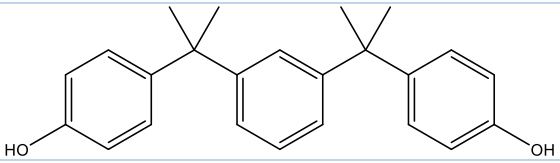
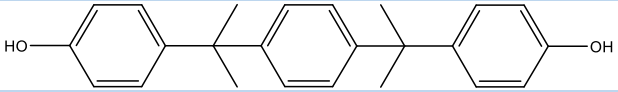
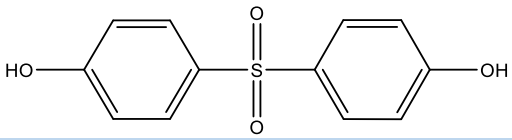
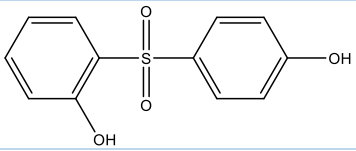
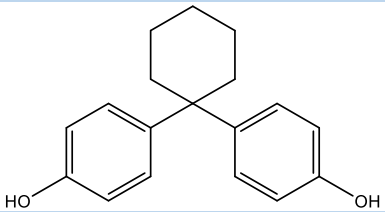
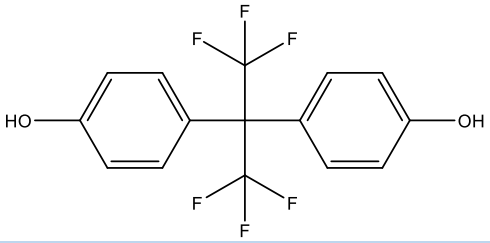
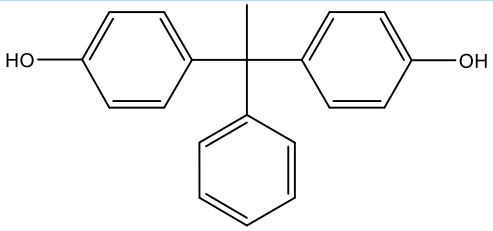
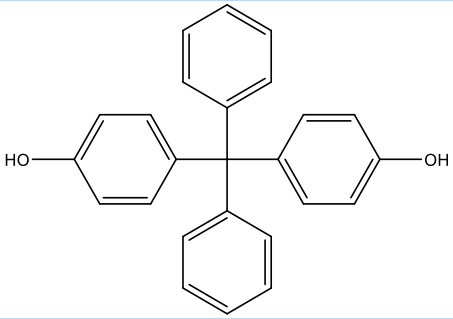
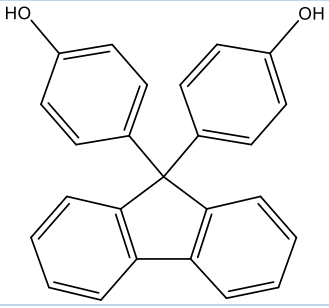
Name	Chemical name	CAS number	Structure
DPP	**Dipentyl phthalate**	131-18-0	
DHXP	**di-n-hexyl phthalate**	84-75-3	
BBP	**benzyl butyl phthalate**	85-68-7	
BnBP	**Bis(2-*n*-butoxyethyl) phthalate**	117-83-9	
DEHP	**Bis(2-ethylhexyl) phthalate**	117-81-7	
DCHP	**dicyclohexyl phthalate**	84-61-7	
DOP	**di-n-octyl phthalate**	117-84-0	
DNP	**di-nonyl phthalate**	84-76-4	

Table 4: Selected phenolic compounds and alkylphenol ethoxylates

Name	CAS number	Structure
Bisphenol A	**80-05-7**	
2,4'-Bisphenol A	**837-08-1**	
Bisphenol B	**77-40-7**	
Bisphenol C	79-97-0	
Bisphenol E	**2081-08-5**	
2,2'-Bisphenol F	**2467-02-9**	
2,4'-Bisphenol F	**2467-03-0**	
4,4-Bisphenol F	**620-92-8**	
Bisphenol G	**127-54-8**	

Bisphenol M	**13595-25-0**	
Bisphenol P	**2167-51-3**	
Bisphenol S (4,4')	**80-09-1**	
2,4'-Bisphenol S	**5397-34-2**	
Bisphenol Z	**843-55-0**	
Bisphenol AF	**1478-61-1**	
Bisphenol AP	**1571-75-1**	
Bisphenol BP	**1844-01-5**	
Bisphenol FL	**3236-71-3**	

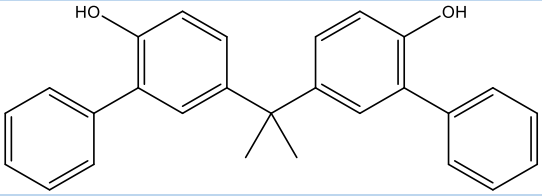
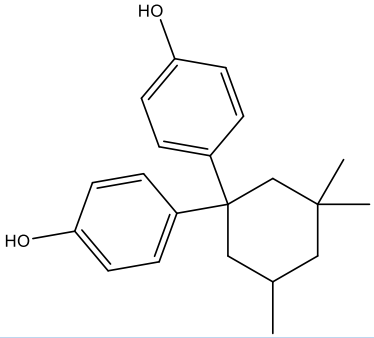
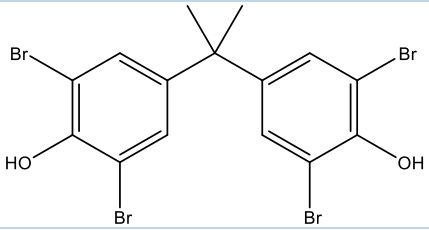
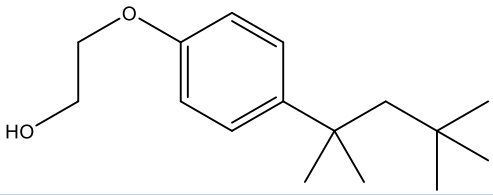
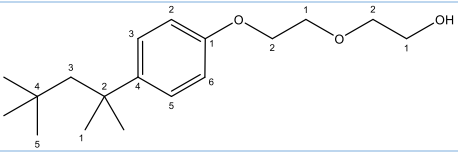
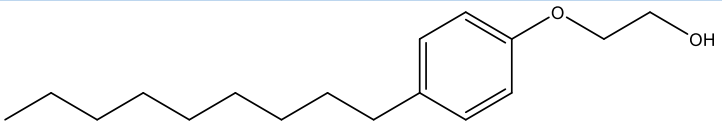
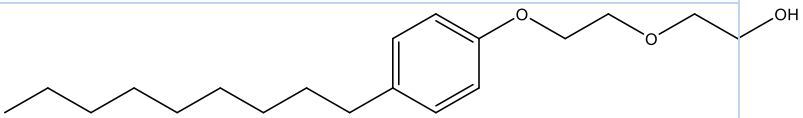
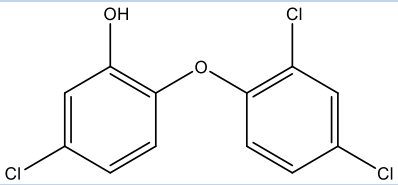
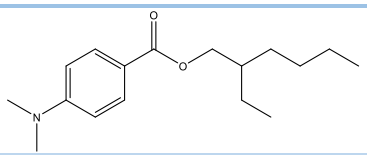
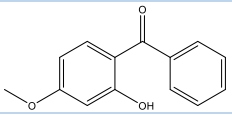
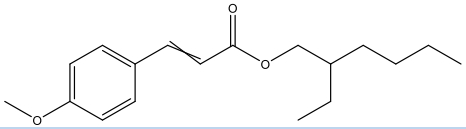
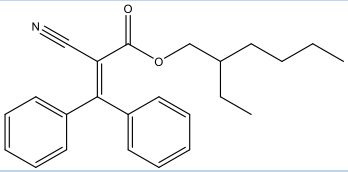
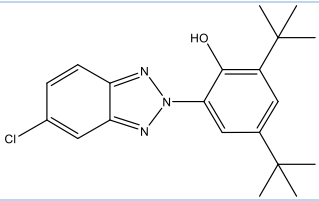
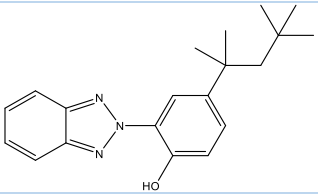
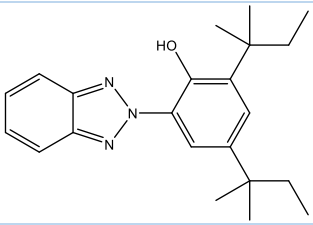
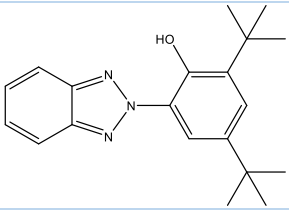
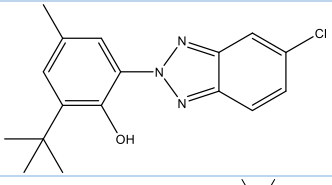
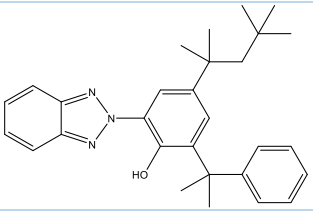
Bisphenol PH	24038-68-4	
Bisphenol TMC	129188-99-4	
Tetrabromobisphenol A	**79-94-7**	
Octylphenol monoethoxylate (OPEO-1)	2315-67-5	
Octylphenol diethoxylate (OPEO-2)	2315-61-9	
Nonylphenol monoethoxylate (NPEO-1)	104-35-8	
Nonylphenol diethoxylate (NPEO-2)	20427-84-3	
Triclosan	3380-34-5	

Table 5: Selected benzotriazole based UV-filters

Name	CAS number	Structure
2-ethylhexyl-4-Dimethylaminobenzoate (ODPABA)	21245-02-3	
Benzophenone 3 (BP3)	131-57-7	
Ethylhexylmethoxycinnamate (EHMC)	5466-77-3	
Octocrylene (OC)	6197-30-4	
UV-327	3864-99-1	
UV-329	3147-75-9	
UV-328	25973-55-1	
UV-320	3846-71-7	
UV-326	3896-11-5	
UV-928	73936-91-1	

2.2.2 Metals

Following metals were quantified in extracts:

- Aluminum (Al)
- Antimony (Sb)
- Arsenic (As)
- Beryllium (Be)
- Lead (Pb)
- Gallium (Ga)
- Germanium (Ge)
- Cadmium (Cd)
- Cobalt (Co)
- Copper (Cu)
- Chromium (Cr)
- Mercury (Hg)
- Molybdenum (Mo)
- Nickel (Ni)
- Zinc (Zn)
- Silver (Ag)
- Thallium (Tl)
- Tellurium (Te)
- Tin (Sn)
- Vanadium (V)

2.3 Methods

2.3.1 Organic contaminants

To avoid contamination and possible false identifications, all the bottles were thoroughly rinsed with ultra-pure Milli-Q water prior to extraction.

Drinking bottles were filled with MilliQ-water again and extracted in 40 °C for 24-hours.
After 24-hrs, the extracts containing possible leachate were spiked with mixtures of isotopically labelled standards and subjected for further cleanup and concentration suitable to different type of analyses (liquid-liquid and/or solid phase extraction).
To avoid possible false positives in analytical results due to contamination in the laboratory, lab blank samples were prepared with each batch of processed samples. In brief, clean glass containers were treated exactly the same way as drinking bottles.

The final extracts were subjected to analyses with either gas or liquid-chromatography high resolution mass spectrometry.

2.3.2 Metals

To avoid contamination and possible false identifications, all the bottles were thoroughly rinsed with ultra-pure Milli-Q water prior to extraction.
Drinking bottles were filled with acetic acid diluted in MilliQ-water to 3% and extracted in 40 °C for 24-hours.

The extracts were analyzed by use of inductively coupled plasma mass spectrometry (ICP-MS) (Agilent 7700x). The calibration standards were matrix matched with 3% acetic acid. ^{115}In was used as internal standard and added to all calibration standards, blank samples and extracts during analysis.

3 Results

3.1 Analytical uncertainty

Due to the lack of available certified reference materials, replicated samples were analysed. The methods used are validated, but not accredited and analytical uncertainty for organic contaminants measured in this study has been established at the level of 60%. Although it appears to be somewhat high, for comparison, typical uncertainty in established accredited analyses of persistent organic pollutants in environmental matrices is at the level of 30-40%. Uncertainty for metals in this study was established at the level of 35%.

3.2 Concentrations of phenolic compounds

51 ng/kg of Octylphenol monoethoxilate (OPEO-1) was detected in a drinking bottle from Toys R Us. Concentration of nonylphenol monoethoxylate (NPEO-1) was lower (37 ng/kg). Octylphenol diethoxylate was found only in a drinking bottle from Toys R Us. In all other samples the levels of alkylphenol ethoxylates were below 10ng/kg.

Several other phenolic contaminants have been detected and are presented in Table 6.

Table 6: Selected phenolic contaminants found in leachate from drinking bottles (ng/kg). Note: to facilitate presentation only compounds detected in at least one of the samples are presented. Limits of detection (LOD) for all bisphenols from table 4 were established at 1ng/kg and for triclosan at 0.6 ng/kg.

Type of the container	Details	bisphenol A	(4,4')-bisphenol F	(2,4')-bisphenol F	(2,2')-bisphenol F	bisphenol AF	bisphenol S	triclosan
bottle	XXL	<1	<1	4	<1	<1	<1	<0.6
bottle	G-Sport	52	<1	<1	<1	<1	<1	<0.6
bottle	Ikea	<1	<1	<1	<1	<1	<1	<0.6
bottle	Nille	44	<1	<1	<1	<1	<1	<0.6
bottle	Jernia	6	7	9	2	<1	<1	<0.6
bottle	Biltema	16	4	5	1	<1	2	<0.6
bottle	Rusta	<1	7	9	1	8	<1	<0.6
bottle	Europris	9	<1	<1	<1	<1	0	<0.6
bottle	Coop Obs	<1	4	4	<1	<1	<1	<0.6
bottle	Toys R Us	13	8	4	1	8	<1	0.8
bottle	Clas Ohlson	<1	8	4	<1	<1	<1	<0.6

3.3 Concentrations of selected phthalates

Limits of detection for selected phthalates that were not detected in the samples are presented in Table 7, while the concentrations of phthalates (ng/kg) found in the samples are presented in Table 8.

Table 7: Limits of detection for analysed phthalates (ng/kg).

Name	Type	BMEP	BMPP	BEEP	DPP	DHXP	DCHP/DEHP	DOP	DNP
LOD	bottle	491	44.2	302	5.4	17.2	45.8	11.2	172

Table 8: Concentrations of phthalates in a leachate from drinking bottles (ng/kg). Note: To facilitate presentation only compounds detected in at least one of the samples are presented.

Type of the container	Details	DMP	DEP	DiBP	DBP	BBP	DCHP/DEHP
bottle	XXL	<24.8	113	<5.5	55.5	<136	<45.8
bottle	G-Sport	<24.8	358	<5.5	66.9	<136	<45.8
bottle	Ikea	<24.8	<35.8	<5.5	<24.1	<136	<45.8
bottle	Nille	<24.8	<35.8	<5.5	60.7	<136	<45.8
bottle	Jernia	<24.8	<35.8	25.6	90.7	<136	<45.8
bottle	Biltema	206	<35.8	19.4	103	<136	<45.8
bottle	Rusta	<24.8	<35.8	25.8	91.0	<136	<45.8
bottle	Europris	<24.8	1038	<5.5	48.0	<136	<45.8
bottle	Coop Obs	<24.8	<35.8	<5.5	<24.1	<136	<45.8
bottle	Toys R Us	<24.8	569	1222	1100	845.0	<45.8
bottle	Clas Ohlson	<24.8	172	72.2	147	<136	<45.8

3.4 Concentrations of selected organophosphorus flame retardants

Several organophosphorus flame retardants have been detected in the samples (Table 10). Limits of detection for those not being detected are presented in Table 9.

Table 9: Limits of detection for selected organophosphorus flame retardants

	TPrP	BdPhP	DBPhP	TDCPP	TCP	EHDP	TXP	TIPPP	TTBPP	TEHP
bottle	1.82	0.72	0.34	3.61	4.54	1.88	2.52	0.23	0.30	0.52

Table 10: Concentrations of organophosphorus flame retardants in a leachate from drinking bottles (ng/kg). Note: to facilitate presentation only compounds detected in at least one of the samples are presented.

Type of the container	Details	TEP	TCEP	TCPP	TiBP	TnBP	TPP	TBEP
bottle	XXL	<9.23	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8
bottle	G-Sport	<9.23	32.3	105.5	<72.6	<2.22	<0.4	<58.8
bottle	Ikea	<9.23	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8
bottle	Nille	<9.23	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8
bottle	Jernia	<9.23	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8
bottle	Biltema	27.5	<13.1	<15.3	<72.6	<2.22	36.7	<58.8
bottle	Rusta	<9.23	<13.1	<15.3	<72.6	<2.22	1435	<58.8
bottle	Europris	<9.23	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8
bottle	Coop Obs	<9.23	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8
bottle	Toys R Us	16.3	62.3	643	<72.6	121	<0.4	1393
bottle	Clas Ohlson	25.1	<13.1	<15.3	<72.6	<2.22	<0.4	<58.8

3.5 Concentrations of selected UV-filters

Analyses of benzotriazole based UV-filters revealed presence of BP3 only in leachate drinking bottle from Toys R Us (31 ng/kg)..

The levels of UV-filters were under limits of detection (see Table 11) for all other samples.

Table 11: Limits of detection for selected UV-filters for all samples tested in this study (ng/kg).

Type of container	BP3	ODPABA	UV-320	UV-326	UV-329	UV-328	UV-327	OC	Sum EHMC
drinking bottles	3-6	0.1-0.2	0.1-0.2	0.5-1	1-2	0.2-0.4	0.1-0.3	20-50	15-40

3.6 Chlorinated paraffins

Concentrations of chlorinated paraffins are presented in Table 12.

Table 12: Concentrations of chlorinated paraffins

Type of the container	Details	SCCP (ng/kg)	MCCP (ng/kg)
drinking bottle	XXL	<265	154
drinking bottle	G-Sport	<270	76
drinking bottle	Ikea	<274	69
drinking bottle	Nille	<206	152
drinking bottle	Jernia	<563	112
drinking bottle	Biltema	<378	375
drinking bottle	Rusta	<397	90
drinking bottle	Europris	<263	65
drinking bottle	COOP Obs	<236	116
drinking bottle	Toys R Us	<406	98
drinking bottle	Clas Olson	<232	47

3.7 New brominated flame retardants and hexachlorocyclohexane

Only DBDPE and isomers of hexachlorocyclohexane were detected in some of the samples (Table 13). Other brominated flame retardants were not detected (with LOD< 0.2-0.5 ng/kg).

Table 13: Concentrations of new brominated flame retardants and hexachlorocyclohexane in a leachate from drinking bottles (ng/kg). Note: to facilitate presentation only compounds detected in at least one of the samples are presented.

Type of container	details	a-HCH	b-HCH	g-HCH	DBDPE
bottle	XXL	0.12	<0.01	0.05	<2
bottle	G-Sport	0.12	<0.01	0.08	<2
bottle	Ikea	0.02	<0.01	0.04	<2
bottle	Nille	0.01	<0.01	<0.01	5.28
bottle	Jernia	0.04	<0.01	0.07	16.93
bottle	Biltema	<0.01	<0.01	0.06	40.69
bottle	Rusta	<0.01	<0.01	0.04	25.71
bottle	Europris	<0.01	<0.01	<0.01	2.79
bottle	Coop Obs	<0.01	<0.01	<0.01	<2
bottle	Toys R Us	0.05	0.04	0.24	5.21
bottle	Clas Ohlson	<0.01	<0.01	47.1	12.38

3.8 Metals

Germanium, silver (LODs = 10 ng/kg), arsenic (LOD = 50 ng/kg), tellurium, thallium and bismuth (LODs = 5 ng/kg) were not detected in any of the samples. Concentrations of other metals are presented in table Table 14.

Table 14: Results of analyses of metals (ng/kg). Note: to facilitate presentation only compounds detected in at least one of the samples are presented.

Type of the container	details	Be	Al	V	Cr	Co	Ni	Cu	Zn	Ga	Mo	Cd	Sn	Sb	Te	Pb
Drinking bottle	XXL	<5	5306.78	<5	<50	<4	<20	17.71	3085.06	<50	<10	<5	<50	<3	<5	15.64
Drinking bottle	G-Sport	7.74	<1000	<5	<50	<4	<20	511.89	129.71	<50	<10	<5	<50	<3	<5	<9
Drinking bottle	IKEA	<5	<1000	<5	<50	<4	<20	<10	133.81	<50	<10	<5	<50	<3	<5	<9
Drinking bottle	Jernia	<5	7590.40	38.37	1259.22	31.22	273.98	234.01	816.63	6437.04	37.37	<5	<50	11.64	<5	28.92
Drinking bottle	Biltema	<5	3033.82	7.43	65.54	<4	1248.26	141.65	9776.01	323.49	<10	<5	<50	3.48	<5	93.44
Drinking bottle	Rusta	<5	<1000	<5	<50	<4	<20	<10	1376.90	<50	<10	<5	<50	6.38	<5	<9
Drinking bottle	Europris	<5	<1000	<5	<50	<4	<20	39.83	417.62	<50	<10	<5	66.91	<3	<5	14.67
Drinking bottle	COOP obs	<5	<1000	6.91	<50	<4	<20	<10	142.73	<50	<10	<5	<50	<3	<5	<9
Drinking bottle	ToysRus	<5	<1000	5.06	<50	7.07	<20	89.12	1447.81	<50	<10	<5	<50	14.32	<5	143.63
Drinking bottle	Clas Ohlson	<5	<1000	<5	<50	<4	<20	<10	<100	<50	<10	<5	<50	<3	<5	<9

4 Summary

A number of different environmental organic contaminants and metals have been found in trace amounts in selected commercial drinking bottles. As this is a data report only, the obtained results were neither compared with other reports nor discussed further.

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