

Analyses of selected organic contaminants and metals in coffee cups

Technical report

Pawel Rostkowski, Hilde T. Uggerud, Mikael Harju, Vladimir Nikiforov,
Anders Borgen, Alfhild Kringstad, Morten Bjørklund, Silje Thomassen,
Marit Vadset, Mebrat Ghebremeskel, Heidi Eikenes, Malcolm Reid



NILU report 24/2018

Preface

Norwegian Consumer Council was interested in checking for possible leakage of potential harmful substances from coffee-cups that could be used by Norwegian consumers.

The aim of the project was to test for leakage of selected organic contaminants and metals in cups provided for analyses.

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.

Analyses of different compounds were conducted with methods established in NILU's and NIVA's laboratories.

Contents

Preface	2
Summary	4
1 Introduction	5
2 Materials and Methods	5
2.1 Samples	5
2.2 Compounds tested	5
2.2.1 Organic contaminants	5
2.2.2 Metals.....	16
2.3 Methods	17
2.3.1 Organic contaminants	17
2.3.2 Metals.....	17
3 Results	17
3.1 Analytical uncertainty	17
3.2 Concentrations of phenolic compounds.....	18
3.3 Concentrations of selected phthalates	18
3.4 Concentrations of selected organophosphorus flame retardants	19
3.5 Concentrations of selected UV-filters.....	19
3.6 Chlorinated paraffins	19
3.7 New brominated flame retardants and hexachlorocyclohexane	20
3.8 Metals.....	20
4 Summary	20

Summary

On behalf of Norwegian Consumer Council, NILU has conducted analyses of organic contaminants and metals in the leachate from selected coffee-cups. 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 were found in trace amounts in the different products.

Analyses of selected organic contaminants and metals in coffee cups

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. 2 coffee-cups 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 for leakage of selected contaminants from the cups 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, 2 different types of coffee-cups were purchased in December 2017 and delivered in triplicate. A total of 6 cups were delivered to NILU. Details are described in Table 1. One of the triplicated samples delivered was used for analyses of metals in NILU's laboratory in Kjeller, while another two for organic analyses in NILU's laboratories in Kjeller and Tromsø, respectively.

Table 1: Information about cups subjected to analyses

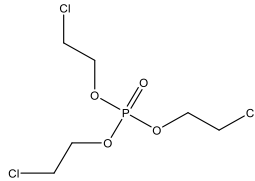
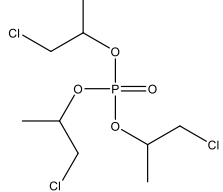
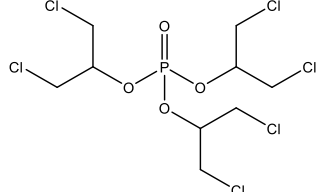
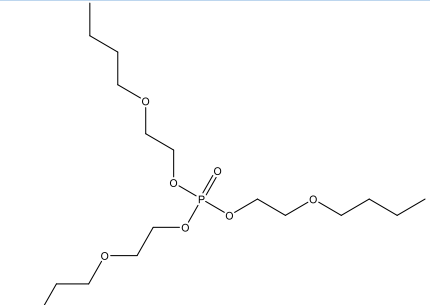
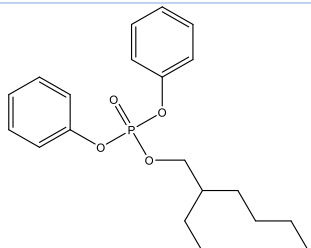
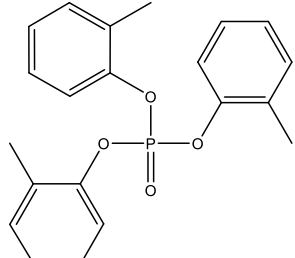
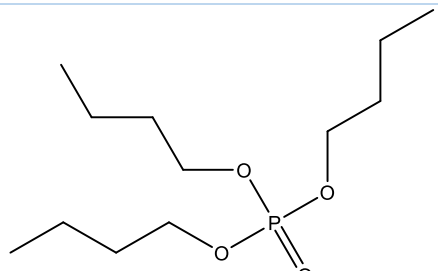
No	Shop	Location of purchase
1	Indiska	Storgata
2	Espresso house	Bussterminalen

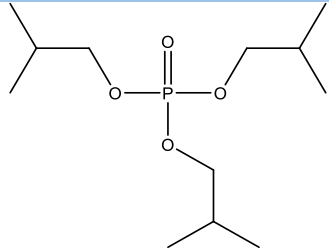
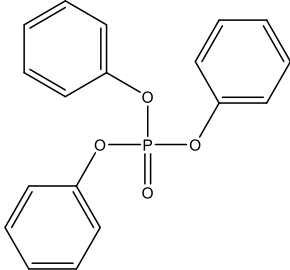
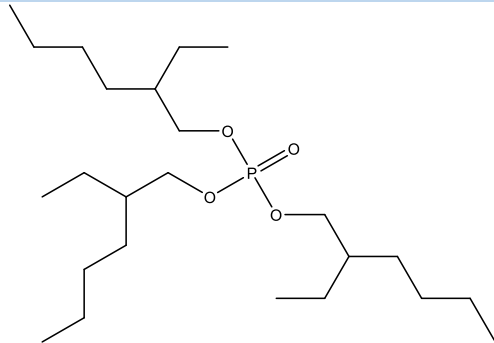
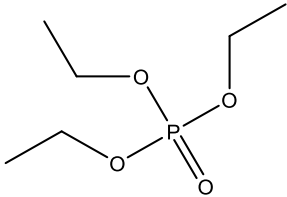
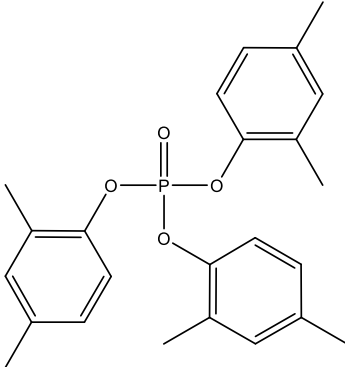
2.2 Compounds tested

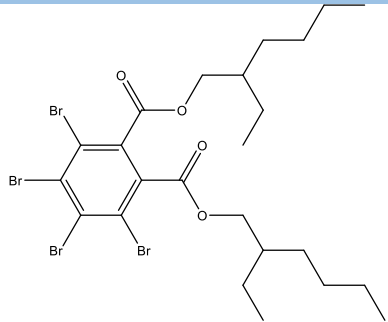
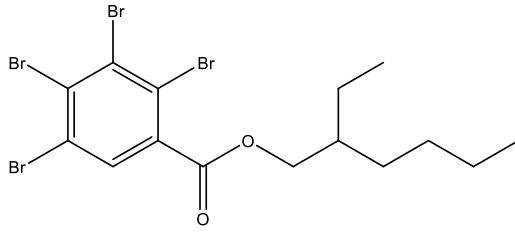
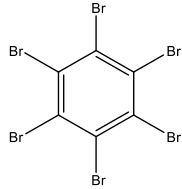
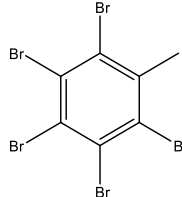
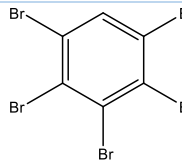
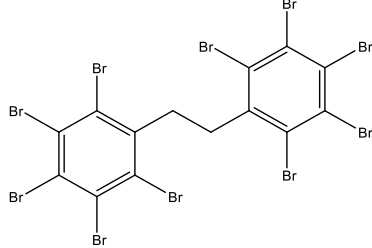
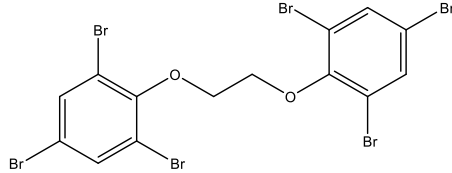
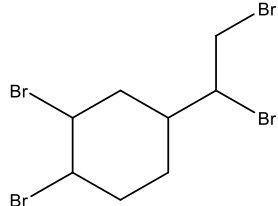
2.2.1 Organic contaminants

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	
TnBP	Tri-n-butyl phosphate	126-73-8	

Name	Chemical name	CAS number	Structure
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	
New brominated flame retardants			

Name	Chemical name	CAS number	Structure
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	
g/d-TBECH	Γ/δ -Tetrabromocyclohexane	Not available	

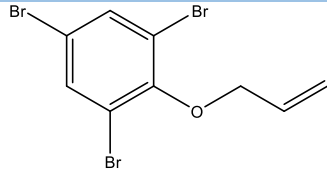
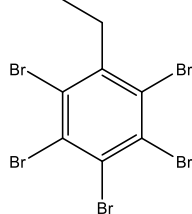
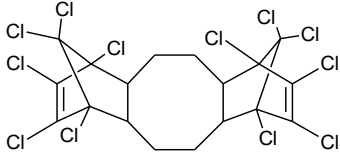
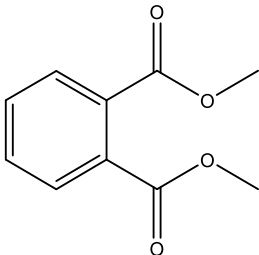
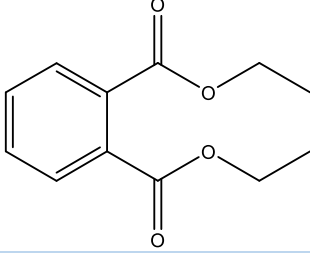
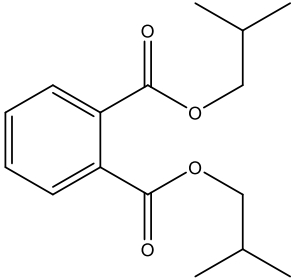
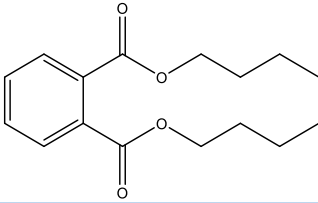
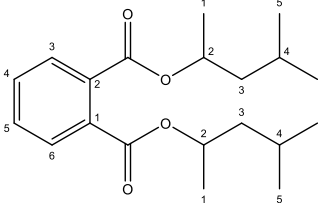
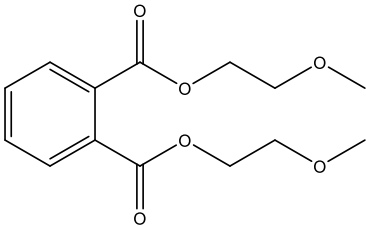
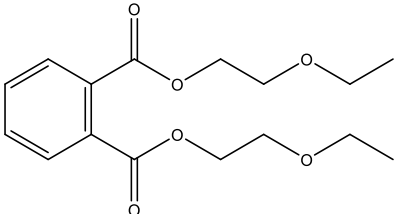
Name	Chemical name	CAS number	Structure
ATE (TBP-AE)	2,4,6-tribromophenyl allyl ether	3278-89-5	
PBEB	Pentabromoethylbenzene	85-22-3	
Dechlorane Plus (syn/anti)	1,4:7,10-Dimethano-dibenzo[a,e]cyclooctene	135821-03-9/ 135821-74-8	
Chlorinated paraffins			
SCCP	Short chain chlorinated paraffins	85535-84-8	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-metoxylethyl) phthalate	117-82-8	
BEEP	Bis(2-etoxyethyl) phthalate	605-54-9	

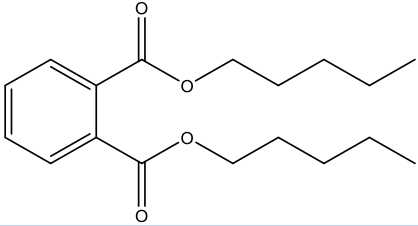
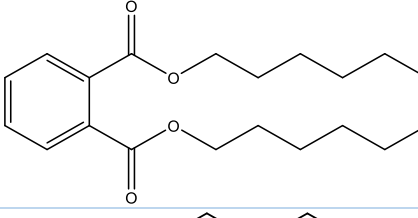
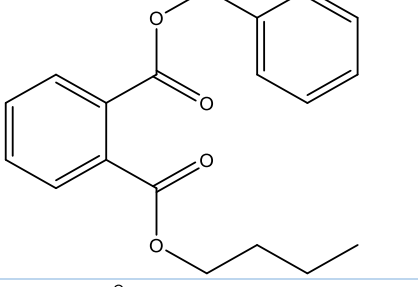
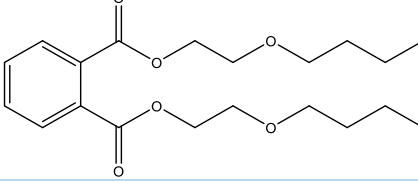
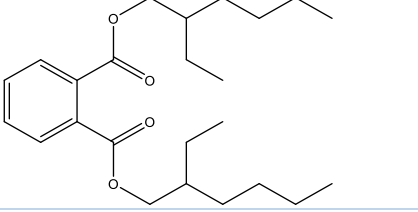
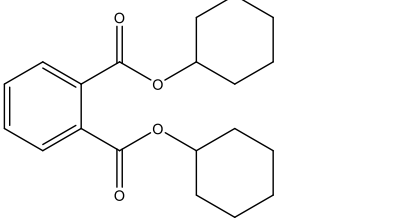
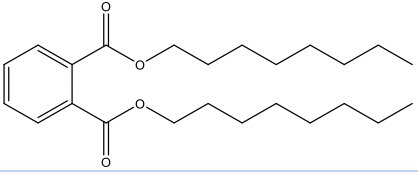
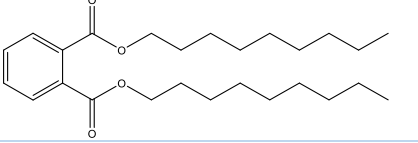
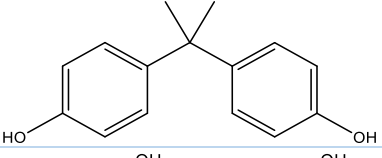
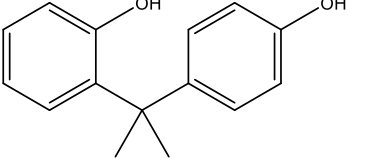
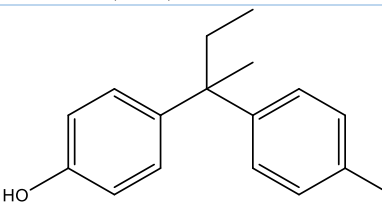
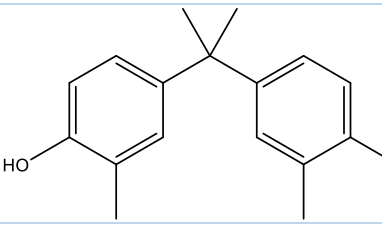
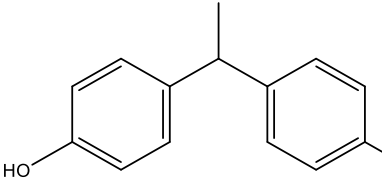
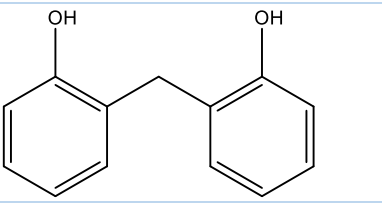
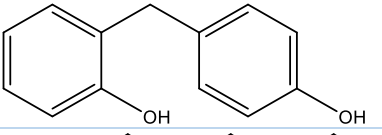
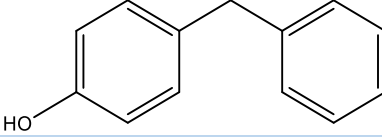
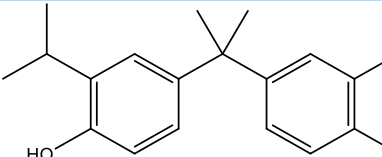
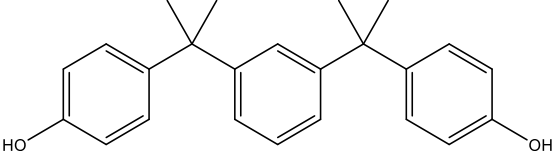
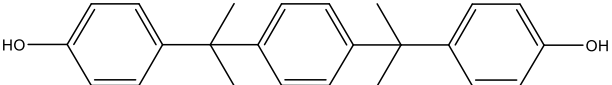
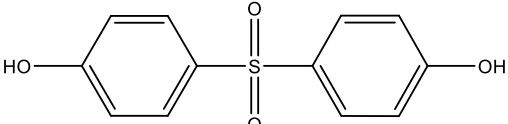
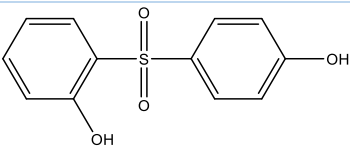
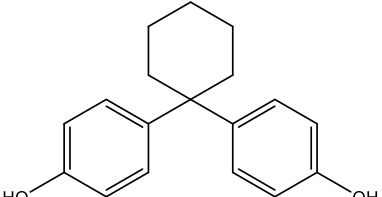
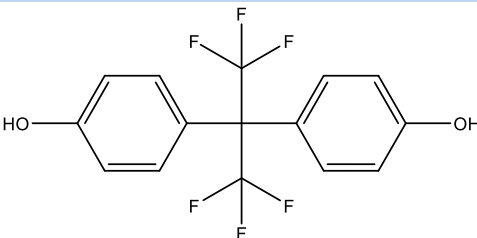
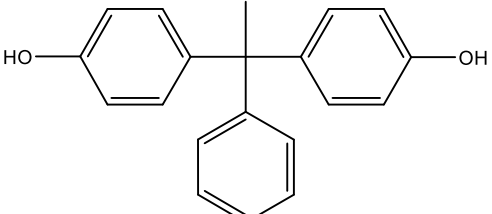
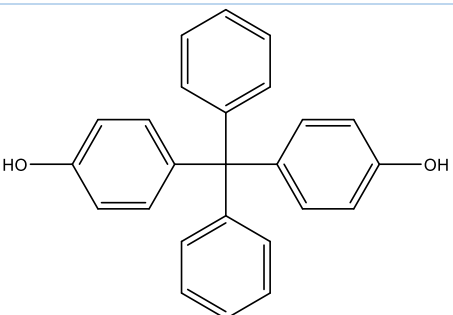
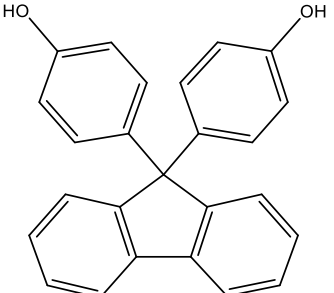
Name	Chemical name	CAS number	Structure
DPP	Dipentyl phthalate	131-18-0	
DHXP	di-n-hexyl phthalate	84-75-3	
BBP	benzyl butyl phthalate	85-68-7	
BnBP	Bis(2-n-butoxyethyl) phthalate	117-83-9	
DEHP	Bis(2-ethylhexyl) phthalate	117-81-7	
DCHP	dicyclohexyl phthalate	84-61-7	
DOP	di-n-octyl phthalate	117-84-0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

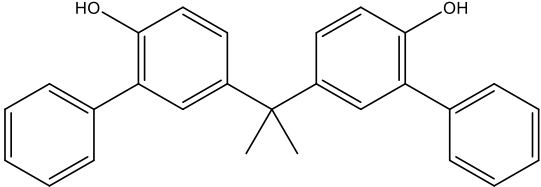
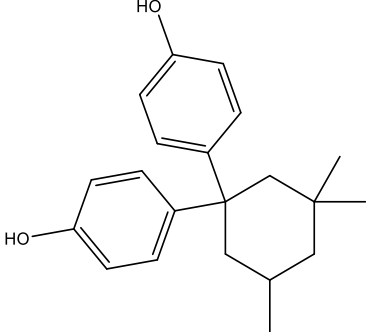
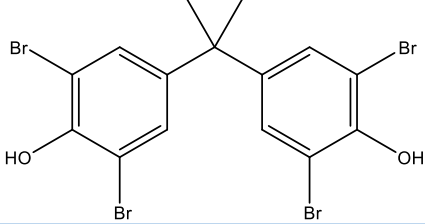
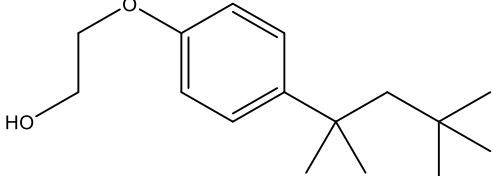
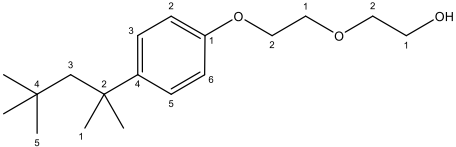
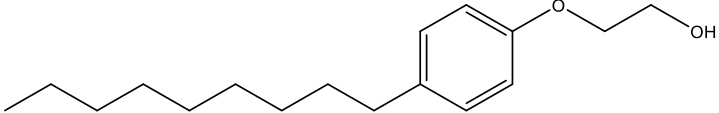
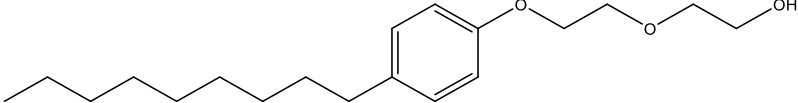
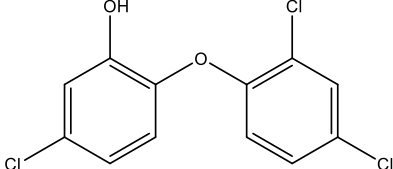
Bisphenol PH	24038-68-4	
Bisphenol TMC	129188-99-4	
Tetrabromobisphenol A	79-94-7	
Octylphenol monoethoxylate (OPEO-1)	2315-67-5	
Octylphenol diethoxylate (OPEO-2)	2315-61-9	
Nonylphenol monoethoxylate (NPEO-1)	104-35-8	
Nonylphenol diethoxylate (NPEO-2)	20427-84-3	
Triclosan	3380-34-5	

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)
- 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 cups were thoroughly rinsed with ultra-pure Milli-Q water prior to extraction.

It was concluded that coffee-cups can be used with fat containing drinks, for example coffee with cream or milk, so the cups were filled with a fat simulant consisting of a 1:1 mixture of ultra-pure MilliQ-water and ethanol 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 to 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 coffee-cups.

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 cups were thoroughly rinsed with ultra-pure Milli-Q water prior to extraction.

In the next step they 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. ¹¹⁵In 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 and the use of validated, but not accredited, methods, analytical uncertainty for organic contaminants measured in this study has been established at the level of 60%. 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

25 ng/kg of Octylphenol monoethoxylate (OPEO-1) and 241 ng/kg nonylphenol monoethoxylate (NPEO-1) was detected in a coffee cup from Indiska.

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

Table 6: Selected phenolic contaminants found in leachate from cups (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
cup	Indiska	12	<1	<1	<1	<1	2	<0.6
cup	Espresso House	8	6	8	<1	<1	11	1.2

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	cup	1619	161.1	997	65.9	56.7	1342	63.6	129

Table 8: Concentrations of phthalates in a leachate from cups (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
cup	Indiska	630	31669	15727	8723	<55	2249
cup	Espresso House	51	<588	310	659	<55	<1342

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
cup	0.6	0.9	0.8	9.0	3.5	3.4	8.1	1.1	2.2	2.3

Table 10: Concentrations of organophosphorus flame retardants in a leachate from cups (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	T CPP	TiBP	TnBP	TPP	TBEP
cup	Indiska	422	<89.2	135	866	116	813	<331
cup	Espresso House	<22.5	<89.2	34.4	<381	<16.1	19.5	<331

3.5 Concentrations of selected UV-filters

350 ng/kg of BP3, 60 ng/kg of UV-329 and 400ng/kg of total EHMC was found in the leachate from the coffee-cup from Indiska.

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

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
coffee cups	6	0.05	1	2	12	2	1	100	20

3.6 Chlorinated paraffins

Concentrations of chlorinated paraffins are presented in Table 12.

Table 12: Concentrations of chlorinated paraffins (ng/kg)

Type of the container	Details	SCCP	MCCP
cup	Indiska	<54	<6
cup	Espresso House	<54	<6

3.7 New brominated flame retardants and hexachlorocyclohexane

Only trace amounts of isomers of hexachlorocyclohexane were detected in the cup from Indiska (

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

Type of container	details	a-HCH	b-HCH	g-HCH	DBDPE
cup	Indiska	0.02	0.01	<0.01	<2
Type of container	details	a-HCH	b-HCH	g-HCH	DBDPE
cup	Espresso House	<0.01	<0.01	<0.01	<2
cup	Indiska	0.02	0.01	<0.01	<2
cup	Espresso House	<0.01	<0.01	<0.01	<2

leachate from cups (ng/kg). Note: To facilitate presentation only compounds detected in at least one of the samples are presented.

3.8 Metals

Germanium, silver (LODs = 10 ng/kg), arsenic, tin (LOD = 50 ng/kg), tellurium, beryllium, thallium and bismuth (LODs = 5 ng/kg) were not detected in any of the samples. Detected concentrations of other metals are presented in 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 container	Details	Al	V	Cr	Co	Cu	Zn	Ga	Cd	Pb
Coffee Cup	Indiska	22 826	16.0	228	8.99	556	254 754	203	7.11	164
Coffee Cup	Espresso House	1 267	<5	<50	<4	38.3	358	<50	<5	<9

4 Summary

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

NILU – Norwegian Institute for Air Research is an independent, nonprofit institution established in 1969. Through its research NILU increases the understanding of climate change, of the composition of the atmosphere, of air quality and of hazardous substances. Based on its research, NILU markets integrated services and products within analyzing, monitoring and consulting. NILU is concerned with increasing public awareness about climate change and environmental pollution.

NILU
—

NILU's values: Integrity - Competence - Benefit to society

NILU's vision: Research for a clean atmosphere

NILU – Norwegian Institute for Air Research
P.O. Box 100, NO-2027 KJELLER, Norway

E-mail: nilu@nilu.no

<http://www.nilu.no>

Norwegian Institute for Air Research

ISBN: 978-82-425-2943-5

