

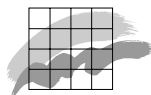


Ministry of Environment and Energy
National Environmental Research Institute

Analytical chemical control of phthalates in toys

Analytical chemical control of chemical substances
and products

NERI Technical Report No. 373



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Abstract: The content of phthalates in toys and other articles for children up to 3 years of age is regulated by the Statutory Order of Danish Ministry of Environment and Energy, No. 151 of 15th March 1999. In the present investigation, 20 products (toys and other articles for children up to 3 years of age) were analysed for the content of phthalates on the request of Danish Environmental Protection Agency (DEPA). The products received from DEPA were analysed for the contents of dimethyl-, diethyl-, dibutyl-, butylbenzyl-, dicyclohexyl-, diethylhexyl-, di-*n*-octyl-, di-*n*-nonyl-, di-*iso*-nonyl- and di-*iso*-decyl phthalate. The subsamples of the products were Soxhlet extracted in dichloromethane, followed by analysis of the extracts by gas chromatography. The content of one or more phthalates in 8 of the investigated products was found to be higher than the maximum allowed concentration, 0.05%.

Keywords: Toys, phthalates, gas chromatography, Statutory Order

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Summary

The content of phthalates in toys and other articles for children up to 3 years of age is regulated by the Statutory Order of Danish Ministry of Environment and Energy, No. 151 of 15th March 1999. In the present investigation, 20 products (toys and other articles for children up to 3 years of age) were analysed for the content of phthalates on the request of Danish Environmental Protection Agency (DEPA). The products received from DEPA were analysed for the contents of dimethyl-, diethyl-, dibutyl-, butylbenzyl-, dicyclohexyl-, diethylhexyl-, di-*n*-octyl-, di-*n*-nonyl-, di-*iso*-nonyl- and di-*iso*-decyl phthalate. The subsamples of the products were soxhlet extracted in dichloromethane, followed by analysis of the extracts by gas chromatography.

The content of one or more phthalates in 8 of the investigated products was found to be higher than the maximum allowed concentration, 0.05%.

Present work has been performed as technical support to DEPA

Resumé

I følge Miljø- og Energiministeriets bekendtgørelse nr. 151 af 15. marts 1999 må legetøj og andre artikler til børn i aldersgruppe 0- 3 år ikke indeholde mere end 0.05% phthalater. I nærværende undersøgelse er indholdet af phthalater bestemt i 20 produkter (legetøj og andre artikler til børn i aldersgruppe 0-3 år) efter anmodning fra Miljøstyrelsen. Produkterne modtaget fra Miljøstyrelsen blev analyseret for indholdet af dimethyl-, diethyl-, dibutyl-, butylbenzyl-, dicyclohexyl-, diethylhexyl-, di-*n*-octyl-, di-*n*-nonyl-, di-*iso*-nonyl- og di-*iso*-decyl phthalat. Delprøver af produkterne blev soxhlet ekstraheret i dichlormethan, efterfulgt af analyse af ekstrakter ved gaskromatografi.

Indholdet af en eller flere phthalater i 8 af de undersøgte produkter var højere end 0.05%, den højest tilladte koncentration.

Arbejdet er udført som bistandsopgave til Miljøstyrelsen.

1 Introduction

The content of phthalates in toys and other children articles for children in the age group 0-3 years is regulated by the Statutory Order of Danish Ministry of Environment and Energy No. 151 of 15th March 1999 of (1). The toys as well as other articles, which children may put in the mouth, should not contain $> 0.05\%$ phthalate according to the Statutory Order. The phthalates are defined in the Statutory Order as diesters of o-phthalic acid. This means that most of the commonly used phthalates in consumer and industrial products are covered by the Danish regulation. A restriction on phthalate content in toys and other articles for children in the age group 0-3 years has also been proposed by EU (2). The EU restriction is, however, temporary and that concerns only 6 phthalates: dibutyl phthalate (DBP), butylbenzyl phthalate (BBP), di-n-octyl phthalate (DnOP), diethylhexyl phthalate (DEHP), diisononyl phthalate (DINP) and diisodecyl phthalate (DIDP). Furthermore, according to the EU proposal, the maximum allowed concentration of these phthalates (total phthalate content) in toys and other articles for children in the age group 0-3 years is $\leq 0.1\%$ (m/m)

To check the compliance of toys and other articles for children in the age group 0-3 years with the Danish Statutory Order, Danish Environmental Protection Agency (DEPA) requested National Environmental Research Institute (NERI) to determine contents of phthalates in these products. In the present investigation contents of some commonly used phthalates in consumer products, including those regulated by EU (in toys), have been determined in 20 products provided by DEPA.

Present work has been performed as technical support to DEPA.

2 Products

DEPA provided 20 products for the analysis (Table 1). Some of the products were a collection of several articles in a package. The products were collected, in the first half of the January 2001, from the retail outlets in Denmark.

The products were made of some kind of plastic material (polymers), partly or completely. The plastic parts of all of the products were analysed for the content of phthalates.

Table 1. Toys and other articles analysed.

<i>DMU Reg. No.</i>	<i>MST No.</i>	<i>Product identification</i>
0-1735	959	Donald Duck (for bathtub)
0-1736	960	Paddington bear bib
0-1737	188	My First Melody Teething Keys
0-1738	189	Musical Teething Keys
0-1739	190	Sassy Stabelkopper (Stacking cups)
0-1740	191	Berchet baby nurse (doll with accessories)
0-1741	192	My Little Lovelies (baby doll)
0-1742	193	Anne Geddes Baby Bees (doll dressed as a bee)
0-1743	194	Learn'n Dress Pooh
0-1744	195	Musical Activity Ring
0-1745	196	Book with mirror (for pushchair)
0-1746	197	Boat Stacking Ring
0-1747	198	Lala 1 st smile (rattle made of fabric and plastic)
0-1748	199	Tiny Trio (three rattles: terry the teether, Freddy the frog and Harry the Hair)
0-1749	200	Giraffe
0-1750	201	Little Lessons Elephant
0-1751	202	Black & White and bright all over (fabric rattle with plastic rings)
0-1752	203	Teletubbies beanbag
0-1753	204	Loop (rattle with transparent plastic tube and wood blocks)
0-1754	205	Bambino Vride & Biderangle (teething rattle)

3 Analysis

The products were analysed for the content of following phthalates: dimethyl phthalate (DMP), diethyl phthalate (DEP), dibutyl phthalate (DBP), dicyclohexyl phthalate (DCHP), di-*n*-octyl phthalate (DnOP), diethylhexyl phthalate (DEHP), di-*n*-nonyl phthalate (DNP), diisononyl-phthalate (DINP), diisodecyl phthalate (DIDP) and butylbenzyl phthalate (BBP). Duplicate subsamples of each product were soxhlet extracted in dichloromethane followed by GC-FID analysis as described before (3, 4). Experimental conditions in brief are described the Section 3.1. In every second set of soxhlet extraction (8 extractions per set), a blank was included. The recoveries of all phthalates at two concentration levels (600 mg/L and 6000 mg/L for DINP and DIDP, and 60 mg/L and 600 mg/L for all other phthalates) were determined by the extraction of known amounts of phthalates under the same conditions as the samples. Calibration curves of all phthalates, except DINP and DIDP, were prepared by analysing solutions of respective standard chemicals at 7 concentrations (40 mg/L-2000 mg/L). The DINP calibration curve was prepared by analysing 400 mg/L-2000 mg/L solutions of this chemical. Calibration curve for DIDP was not prepared, because DIDP was determined as DINP due to overlap of GC peaks of these substances by the present method. Repeatability of the determination (precision) was checked by 10 consecutive GC analysis of solutions of phthalate standards at two concentration levels: 2000 mg/L and 6000 mg/L for DINP; 4000 mg/L and 10000 mg/L for DIDP; and 200 mg/L and 1000 mg/L for all other phthalates.

All of the samples were first screened, by duplicate analysis, for the presence of phthalates. From the results of screening analysis, approximate concentrations of the phthalates present in the respective samples were calculated. On the basis of these results, fresh extracts of the samples were prepared where amount of the sample and the volume of extraction solvent were adjusted so that no concentration/dilution of the extract was necessary for the quantification (in most cases). Calibration curves prepared by the analysis of solutions of standard phthalates, analysed in the same GC-sequence as the samples, were used for the quantification. In each GC run, 3 calibration standard solutions of the phthalates concerned were analysed after every 2-3 samples (4-6 extracts). Each standard solution as well as sample extract was analysed two times by GC-FID. The contents of phthalates in a sample extract was determined using the calibration standards analysed closest (in GC-sequence) to the sample extract.

3.1 Experimental

For screening analysis, 1 g sample was soxhlet extracted in 100 ml dichloromethane for 16 h at 66°C. 90 ml of the extract was concentrated to 10 ml employing rotatory evaporator. 1µl of the concentrated as well as non-concentrated extract was analysed by GC as described in 3.1.1. For quantification, fresh extracts of the samples were prepared so that concentration/dilution of the extracts was avoided, in most cases. All sam-

ples were analysed in duplicate, both for screening analysis as well as for quantification.

Identification of the phthalates was performed by comparing the GC retention times of the sample peaks with the retention times of the peaks of standard phthalates, besides considering the chromatographic pattern of DINP and DIDP. The confirmation of the identified phthalates was done by the analysis of sample extracts spiked with appropriate amounts of the relevant standard phthalates.

3.1.1 GC conditions for phthalate analysis

Apparatus: Hewlett Packard (HP) gas chromatograph 5890A with split/splitless injector, flame ionisation detector, HP auto-sampler 7673A and HP Chemstation.

GC column: Chrompack fused silica column CP-Sil-5CB, 50 m x 0.32 mm, d_f 0.12 μm .

Temperature program: Start temperature 150°C, 5°C to 280°C, 5 min at 280°C

Carrier gas: He, 55ml/min, column head pressure 19.5 psi

Injector: Split, 300°C, injection volume 1 μl

Detector: Flame ionisation, 300°C, make-up gas N_2 30 ml/min

4 Results and Discussion

The analytical method used in the present investigation has previously been demonstrated to be suitable for the identification and determination of phthalates in toys (3, 4). As described below, the important characteristics of the method (stability of the GC-retention time, calibration range, recovery, repeatability of determination, etc.) were checked in the present investigation, and they were found satisfactory for the analysis of phthalate content in toys. The analysis of phthalate content in the products was performed in 2 steps. In the first step, a screening analysis for the identification of phthalates as well as estimation of their concentration in the sample extracts was performed. This was followed by quantitative determination of the identified phthalates in fresh sample extracts, where relevant calibration standards were analysed together with the sample extracts.

The GC retention time (t_R) of the investigated phthalates were stable through out the study, with a maximum variation <1%. The GC retention times of the phthalates were used for the identification of all phthalates, besides the chromatographic pattern of DINP and DIDP were considered for the presence of these substances in the sample extracts. The GC separation of the phthalates under study (as well as chromatographic pattern of DINP and DIDP) is shown in Figure 1. The detection limit considered as the concentration of a phthalate in a solution showing visible peak, without any noise at the base line, was 0.05% (w/w) DINP and DIDP, and that was 0.01% (w/w) for other phthalates. The identification was performed both by analysing the sample extracts (approximately 1 g sample /100 ml dichloromethane) extracts as well as by analysing concentrated (9:1) sample extracts, so that phthalates present in low concentration ($\leq 0.01\%$) could be identified. The sample extracts containing high concentration of phthalates were reanalysed after appropriate dilution of the sample extracts. The identification was confirmed by the GC analysis of the sample extracts spiked with the appropriate amounts of the identified phthalates, for example DEHP in the sample 0-1743 (Figure 2). All of the samples were analysed in duplicate. The phthalates identified in some selected samples are shown in Figures 3-5. The analysis of blank extracts by GC revealed only the signal of the extraction solvent.

The calibration curves for all of the phthalates were linear ($R^2 \geq 0.9999$) in the investigated concentration range: 200-10000 mg/L for DINP and 40 mg/L - 2000 mg/L for all other phthalates (Figure 6A and 6B). The recovery of all of the investigated phthalates under the experimental conditions was 93-106%. The DINP recovery was determined at concentration levels 600 mg/L and 6000 mg/L, and the recoveries of other phthalates were determined at concentration levels 60 mg/L and 600 mg/L. The repeatability of determination was determined by 10 consecutive GC injections of the phthalate standard solutions at 2 concentration levels: 2000 mg/L and 6000 mg/L for DINP, 4000 mg/L and 10000 mg/L for DIDP, and 200 mg/L and 1000 mg/L for all other phthalates. The relative standard deviation (RSD) for the determination of all phthalates was within 4%.

For the determination of the phthalate content in the products, fresh sample extracts were prepared so that concentration/dilution of the extracts before GC analyses was avoided in most cases. The determination was performed in duplicate samples using 3 point calibration curves prepared by the analysis of the relevant standard solutions run in the same sequence as the samples. The GC-sequence was designed so that appropriate standard solutions were analysed before and after each set of 2-3 samples (in duplicate). The calibration standards were selected to match the estimated concentrations of phthalates in the sample extract. The regression line equation was used for the quantification. The determination revealed that the content of phthalates in a product was similar or very close to the estimated concentrations derived from the screening analysis. When a product contained DINP as well as DIDP, GC chromatograms of which overlap partly (Figure 1), the content of both of these phthalates in the product was determined together as DINP.

The results of the analysis of investigated phthalates are described in Table 2. Of the 20 products (toys and other articles for children) investigated in the present study, one or more of the phthalates were found in 9 products. Among the investigated phthalates, DBP was present in 1 sample, DEHP in 5 samples, DINP in 6 samples and DIDP was present in 3 samples. None of the other investigated phthalates were identified in any of the products. It should be noted, however, that if a product contains both DnOP and DINP, DnOP peak in such a product can not be identified by the present method because of overlap by GC peaks of DINP (Figure 1).

According to Statutory Order of the Ministry of Environment and Energy, toys and other articles for children in the age group 0- 3 years should not contain >0.05% phthalate, defined as diester of o-phthalic acid. Of the 9 products containing phthalates, 8 products did not conform to the maximum allowed concentration of phthalate set by the statutory order. One product (DMU No. 0-1743, raincoat and hat of Winnie the Pooh) containing 0.06% (m/m) DEHP may be considered as conforming to the regulation, when analytical uncertainty (recovery and repeatability) is taken into account. The manufacturer of this product informed that the material used for raincoat and the hat was urethane. The reason for presence of DEHP in this product may be a contamination either in the raw material, or the product might have contaminated during the manufacturing process. It is also possible that the lining of these articles (raincoat and hat) contained DEHP.

A book made of cardboard (DMU No. 0-1745), covered with soft plastic material (lining), contained 0.15% DBP. As the cardboard and the printing ink may not contain DBP, the phthalate content of the plastic lining may be rather high.

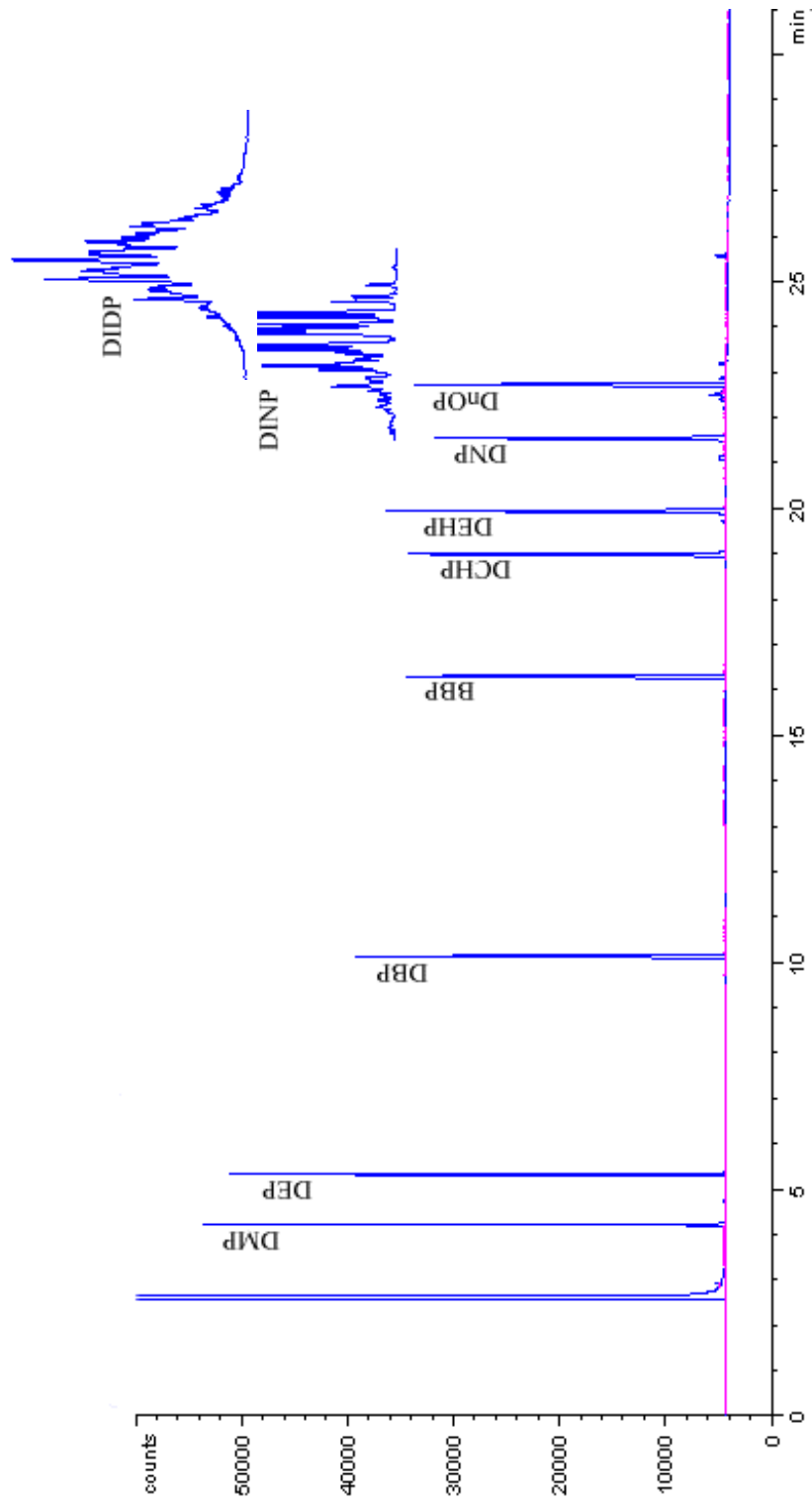


Figure 1: GC separation of investigated phthalates. DINP 0.4%, DIDP 0.6% and all other phthalates 0.04%

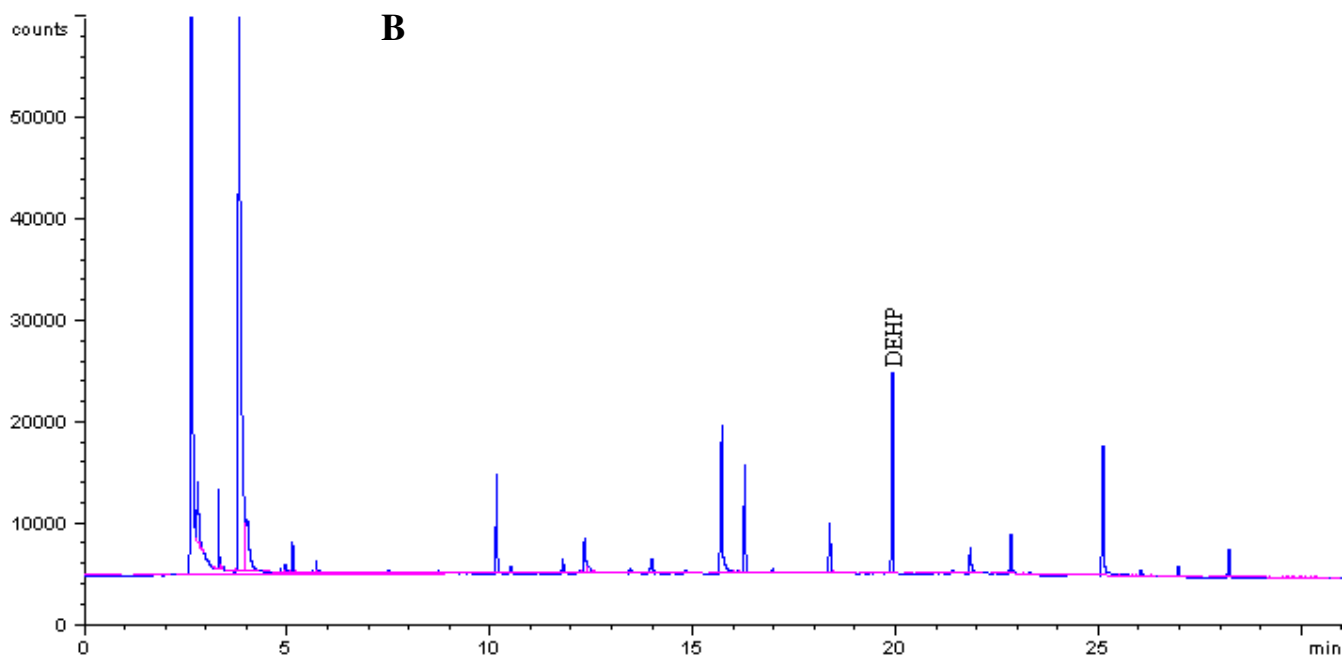
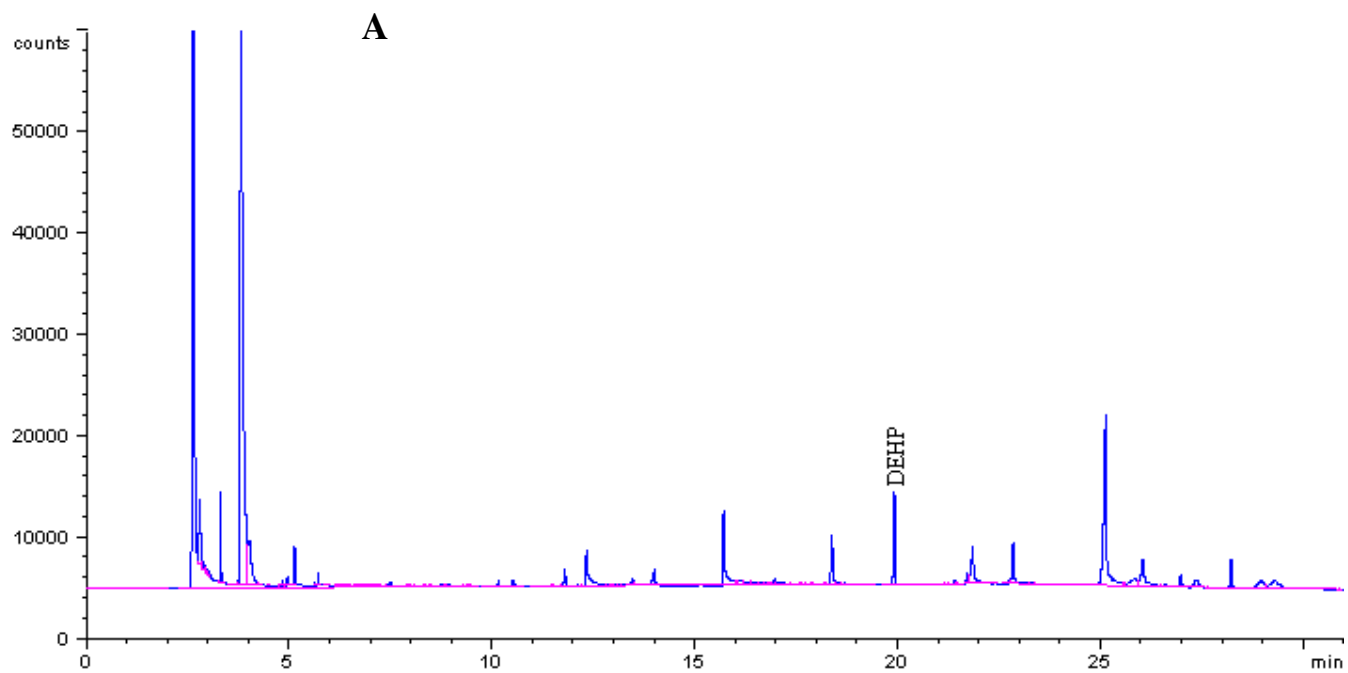


Figure 2: Identification of DEHP in the sample (0-1743, raincoat) extract (ca. 2.0 g/10 ml) by GC-FID analysis (A). The identification of DEHP was confirmed by the GC-FID analysis of the sample extract spiked with 0.02% DEHP, where the DEHP peak of the sample is completely overlapped by the GC-peak of the standard DEHP (B)

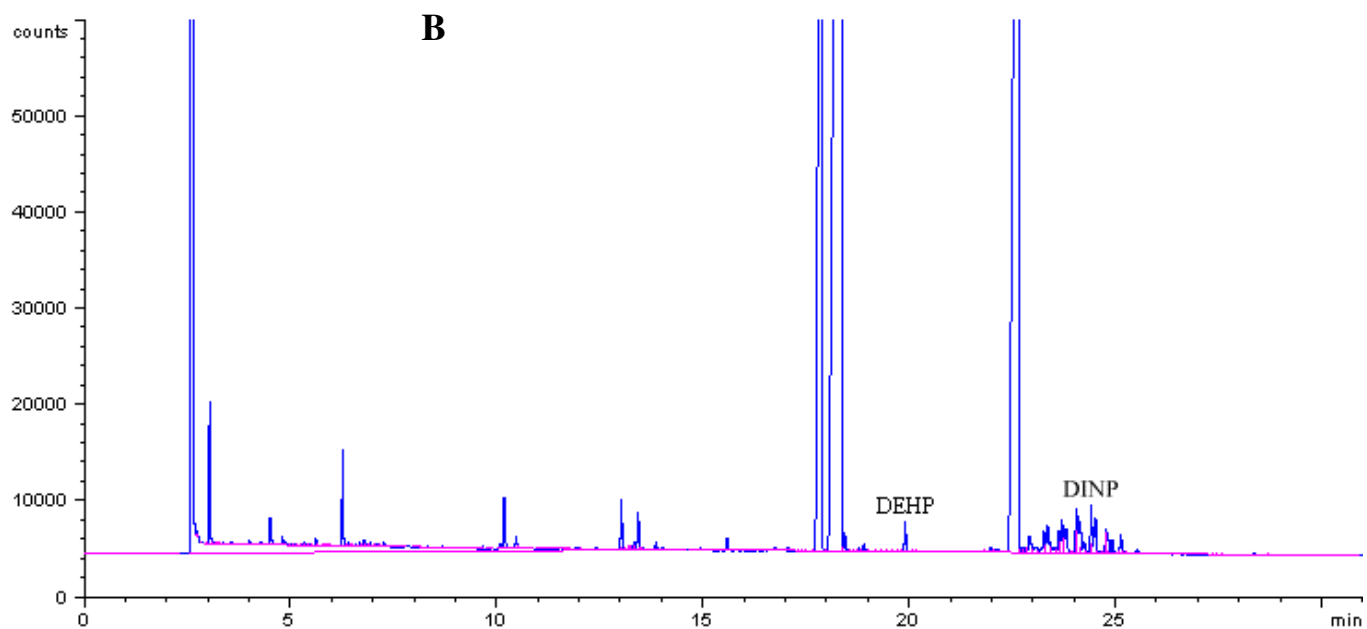
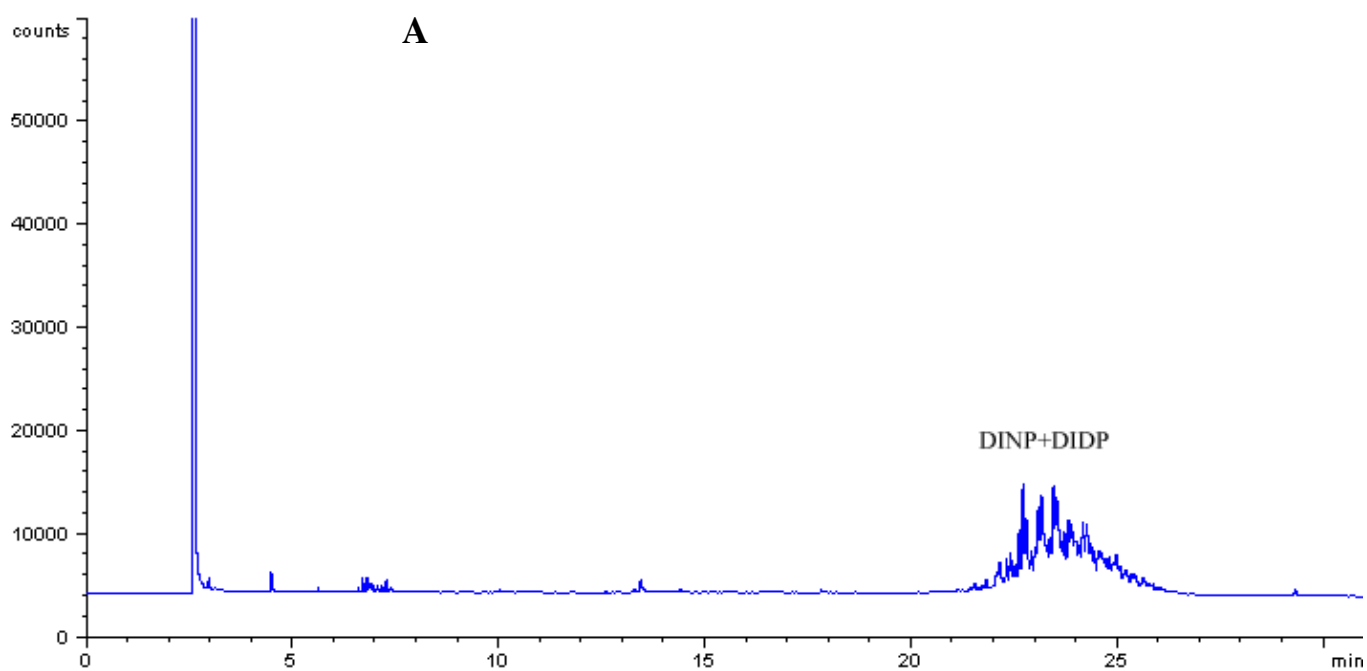


Figure 3: Screening analysis of extracts (ca. 0.5 g/50 ml) of products 0-1735 (A) and 0-1736 (B) by GC-FID. 0-1735 was analysed as without concentration of the soxhlet extract, and 0-1736 was analysed after 9x concentration.

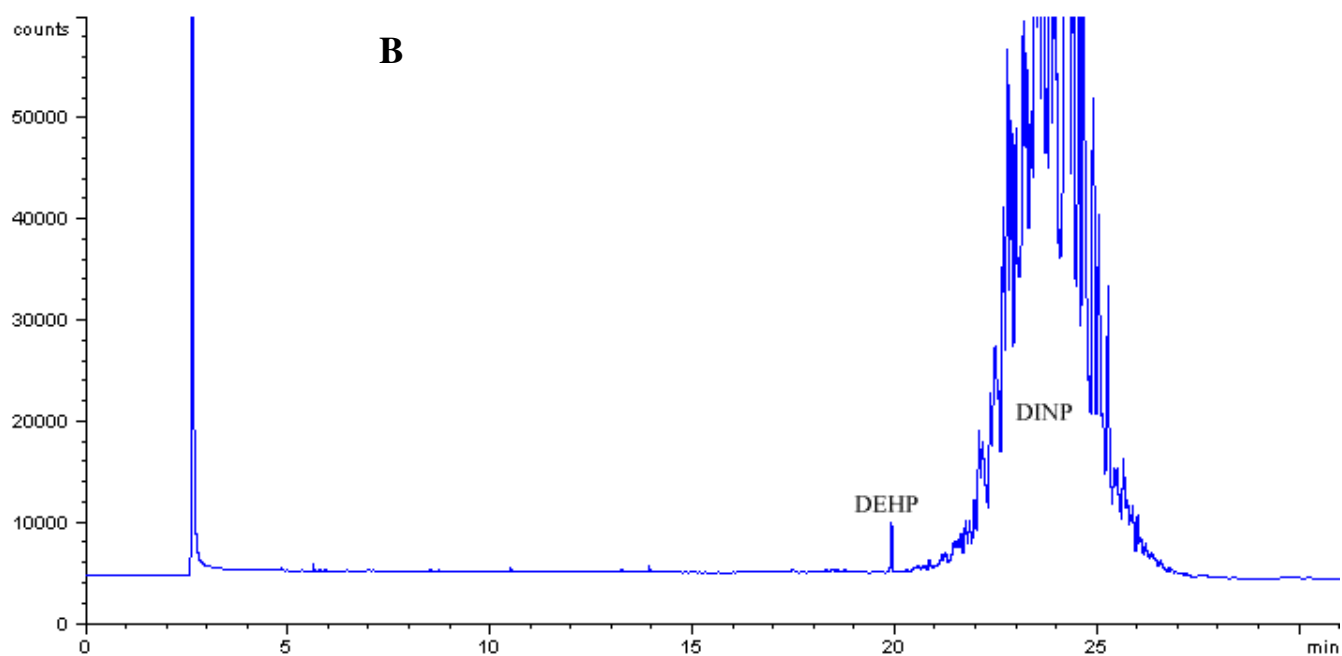
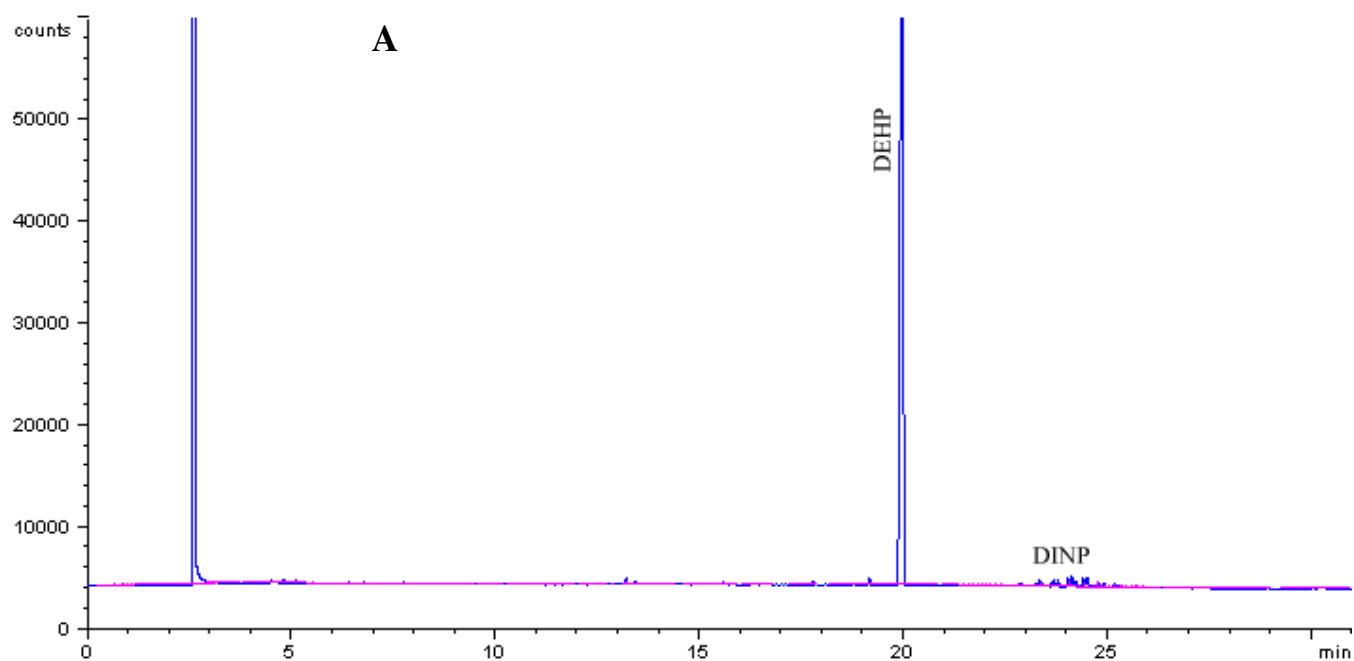


Figure 4: GC-FID analysis of extracts of 0-1740 (inflatable ring) and 0-1751 (red ring).

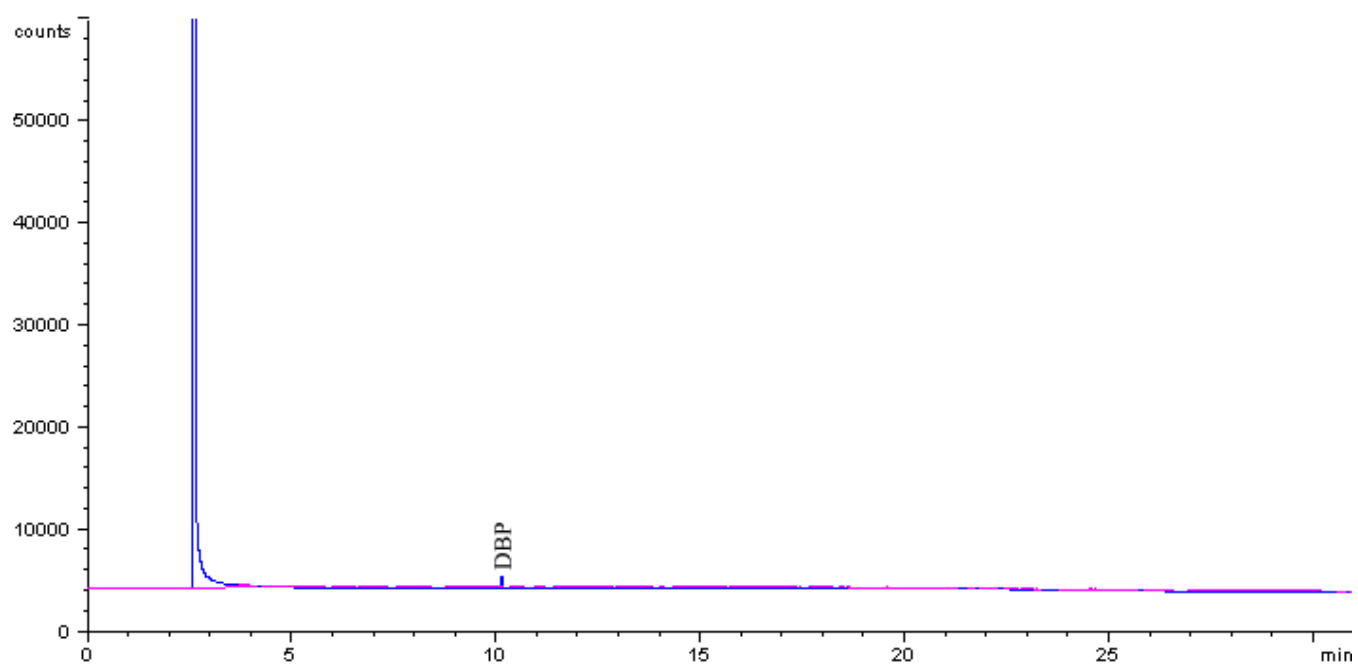


Figure 5: GC-FID of extract of 0-1745 (book)

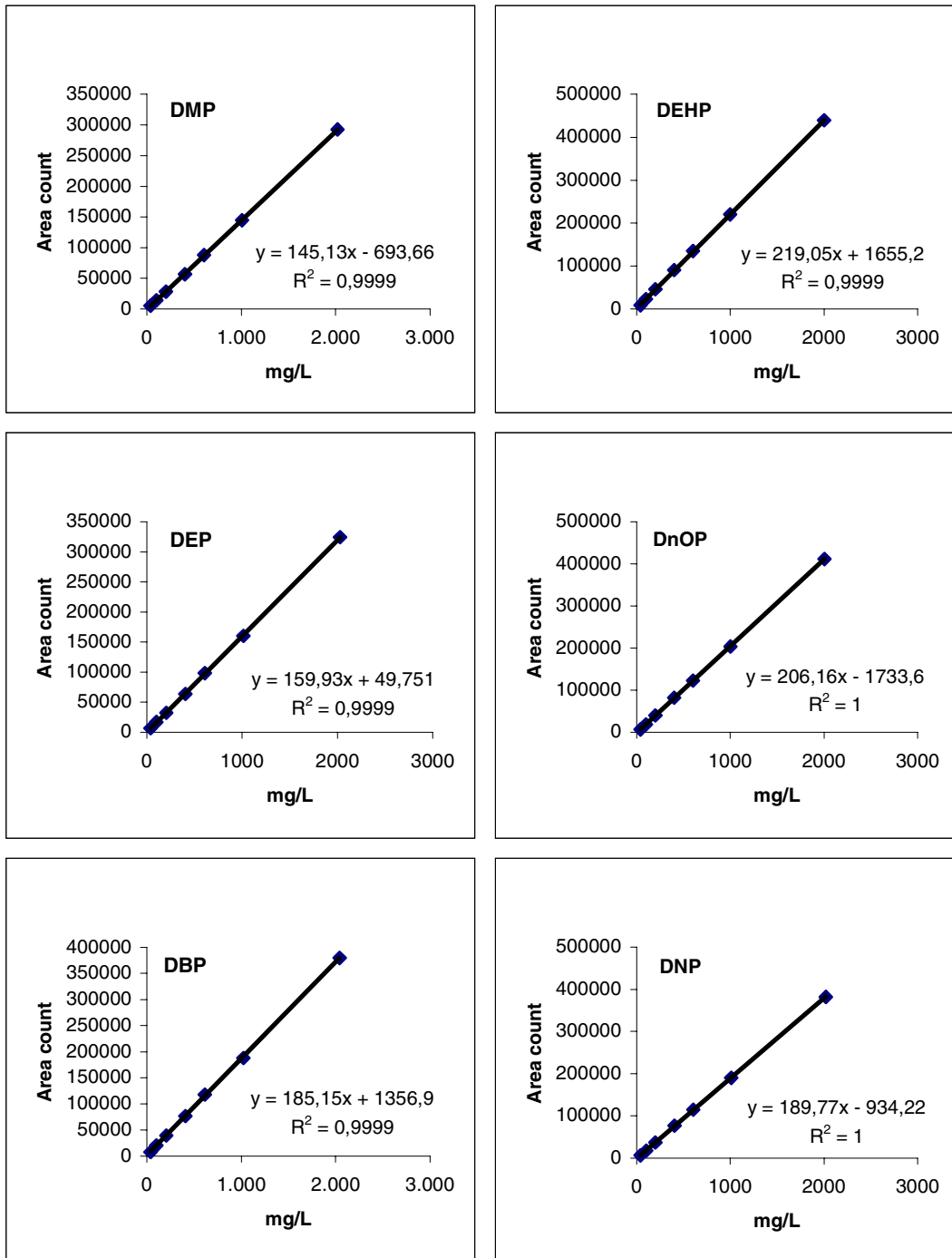


Figure 6A: Calibration curves of the investigated phthalates

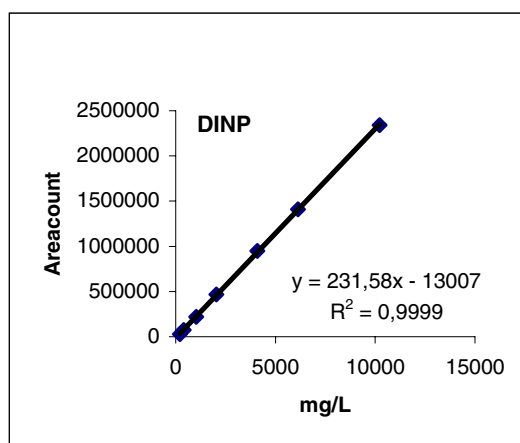
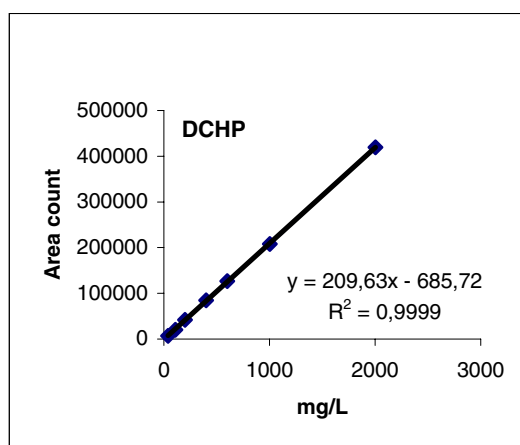
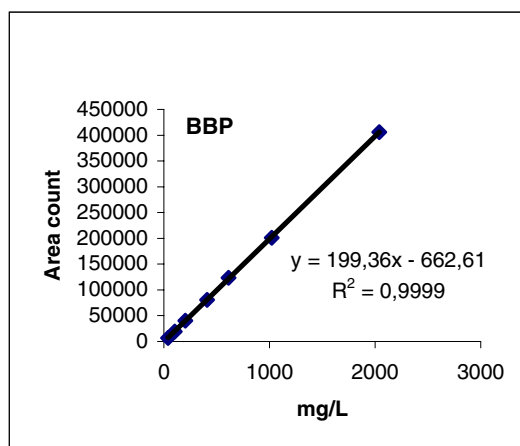


Figure 6B: Calibration curves of the investigated phthalates

Table 2. Contents of phthalates in the investigated products.

<i>DMU Reg. No.</i>	<i>MST No.</i>	<i>Sample analysed</i>	<i>Phthalate(s) identified</i>	<i>Phthalate content % (m/m)</i>
0-1735	959	Animal figure (green)	DINP + DIDP	40.842
0-1736	960	Bib	DINP	5.771
0-1737	188	Soft plastic part of the keys (pink)	-	-
0-1738	189	Soft plastic part of the keys (blue)	-	-
0-1739	190	Ring (red)	-	-
0-1740	191	Plastic Teddy bear (yellow)	DEHP	30.241
			DINP	0.406
		Doll leg	DINP + DIDP	48.545
		Doll arm	DINP + DIDP	44.336
		Doll head	DINP + DIDP	54.998
		Duck (yellow)	DINP	36.050
		Inflatable ring	DEHP	26.759
			DINP	1.554
		Rabbit (pink)	DEHP	30.754
			DINP	0.507
		Key (blue)	-	-
0-1741	192	Doll head	DINP + DIDP	35.761
		Doll leg	DINP + DIDP	35.316
		Doll arm	DINP + DIDP	41.360
0-1742	193	Doll leg	-	-
0-1743	194	Rain coat	DEHP	0.064
		Hat	DEHP	0.061
0-1744	195	Hammock with Winnie-the-Pooh	-	-
0-1745	196	Blue strap	-	-
		Book	DBP	0.147
0-1746	197	Transparent plastic (boat)	DEHP	20.436
		White plastic (boat)	DEHP	20.490
0-1747	198	Ear (orange/white)	-	-
		Ear ring (yellow)	-	-
0-1748	199	Plastic flower (red)	-	-
0-1749	200	Giraffe	-	-

Table 2. Continued.

<i>DMU Reg. No.</i>	<i>MST No.</i>	<i>Sample analysed</i>	<i>Phthalate(s) identified</i>	<i>Phthalate content % (m/m)</i>
0-1750	201	Hook (yellow)	DINP	29.271
		Leg (red)	DEHP DINP	0.610 35.045
0-1751	202	Ring (red)	DEHP	0.107
		Pendant (blue)	DINP	31.404
			DEHP DINP	0.072 35.707
0-1752	203	Plastic face	-	-
0-1753	204	Transparent tube	-	-
0-1754	205	Ring (orange)	-	-
		Covering (black)	-	-

5 References

1. Miljø- og Energiministeriets bekendtgørelse nr. 151 af 15. marts 1999. Bekendtgørelse om forbud mod phthalater i legetøj til børn i alderen 0-3 år samt i visse småbørns artikler m.v.
2. Forslag til Europa-Parlamentets og Rådets Direktiv om toogtyvende ændring af Direktiv 76/769/EØF om indbyrdes tilnærmelse af medlemsstaternes administrativt eller ved lov fastsatte bestemmelser om begrænsning af markedsføring og anvendelse af visse farlige stoffer og præparater (phthalater) og om ændring af Rådets direktiv 88/378/om indbyrdes tilnærmelse af medlemsstaternes lovgivning om sikkerhedskrav til legetøj 1999/0238 (COD).
3. Rastogi S. C. (1998) Gas chromatographic analysis of phthalate esters in plastic toys. *Chromatographia* 47: 724-726.
4. Rastogi S.C., Worsøe I.M., Køppen B., Hansen A.B. and Avnskjold J. (1997) Indhold af organiske opløsningsmidler og phthalater i legetøj. Danmarks Miljøundersøgelser. 34 s- Faglig rapport fra DMU, nr. 217.

National Environmental Research Institute

The National Environmental Research Institute, NERI, is a research institute of the Ministry of Environment and Energy. In Danish, NERI is called *Danmarks Miljøundersøgelser (DMU)*.

NERI's tasks are primarily to conduct research, collect data, and give advice on problems related to the environment and nature.

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Publications:

NERI publishes professional reports, technical instructions, and the annual report. A R&D projects' catalogue is available in an electronic version on the World Wide Web.

Included in the annual report is a list of the publications from the current year.

Faglige rapporter fra DMU/NERI Technical Reports

2000

- Nr. 337: Vandmiljø 2000. Tilstand og udvikling. Faglig sammenfatning. Af Svendsen, L.M. et al. 64 s., 75,00 kr.
- Nr. 338: NEXT I 1998-2003 Halogenerede Hydrocarboner. Samlet rapport over 3 præstationsprøvnings-runder . Af Nyeland, B. & Kvamm, B.L. 87 s., 150,00 kr.
- Nr. 339: Phthalates and Nonylphenols in Roskilde Fjord. A Field Study and Mathematical Modelling of Transport and Fate in Water and Sediment. The Aquatic Environment. By Vikelsøe, J., Fauser, P., Sørensen, P.B. & Carlsen, L. (in press)
- Nr. 440: Afstrømningsforhold i danske vandløb. Af Ovesen, N.B. et al. 238 s., 225,00 kr.
- Nr. 341: The Background Air Quality in Denmark 1978-1997. By Heidam, N.Z. 190 pp., 190,00 DKK.
- Nr. 342: Methyl t-Buthylether (MTBE) i spildevand. Metodeafprøvning. Af Nyeland, B. & Kvamm, B.L. 45 s., 75,00 kr.
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The content of phthalates in toys and other articles for children up to 3 years of age is regulated by the Statutory Order of Danish Ministry of Environment and Energy, No. 151 of 15th March 1999. In the present investigation, 20 products (toys and other articles for children up to 3 years of age) were analysed for the content of phthalates on the request of Danish Environmental Protection Agency (DEPA). The products received from DEPA were analysed for the contents of dimethyl-, diethyl-, dibutyl-, butylbenzyl-, dicyclohexyl-, diethylhexyl-, di-*n*-octyl-, di-*n*-nonyl-, di-*iso*-nonyl- and di-*iso*-decyl phthalate. The subsamples of the products were soxhlet extracted in dichloromethane, followed by analysis of the extracts by gas chromatography. The content of one or more phthalates in 8 of the investigated products was found to be higher than the maximum allowed concentration, 0.05%.

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