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Analytical chemical control of phthalates in toys

Analytical chemical control of chemical substances and products

NERI Technical Report, No. 404

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Suresh C. Rastogi
Gitte H. Jensen
Inge M. Worsøe

Data sheet

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Authors:	Suresh C. Rastogi, Gitte H. Jensen, and Inge M. Worsøe
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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, 35 products (toys and other articles for children up to 3 years of age) were analysed for the content of phthalates on the request of the 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-, diis*o*nonyl- and diis*o*-decyl phthalate. The subsamples of the products were soxhlet extracted in dichloromethane, followed by analysis of the extracts by gas chromatography and mass spectrometry. The content of one or more phthalates in 12 of the investigated products/part(s) of the products was found to be higher than the maximum authorised concentration, 0.05%.

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Summary

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The content of one or more phthalates in 12 of the investigated products / part(s) of the products was found to be higher than the maximum authorised concentration, 0.05%.

Present work has been performed as technical support to DEPA

Resume

I følge Miljø- og Energiministeriets bekendtgørelse nr. 151 af 15. marts 1999 må legetøj og andre artikler til børn i alders gruppe 0- 3 år ikke indeholde mere end 0,05% phthalater. I nærværende undersøgelse er indholdet af phthalater bestemt i 35 produkter (legetøj og andre artikler til børn i aldersgruppen 0-3 år) efter anmodning af Miljøstyrelsen. Produkterne modtaget fra Miljøstyrelsen blev analyseret for indholdet af dimethyl-, diethyl-, dibutyl-, butylbenzyl-, dicyclohexyl-, diethylhexyl-, di-*n*-octyl-, di-*n*-nonyl- , diisononyl- and diisodecyl phthalat. Delprøver af produkterne blev soxhlet ekstraheret i dichlormethan, efterfulgt af analyse af ekstrakter ved gaskromatografi og massespektrometri.

Indholdet af en eller flere phthalater i 12 af de undersøgte produkter / delprodukter var højere end 0,05%, den højest tilladte koncentration.

Arbejdet er udført som bistandsopgave til Miljøstyrelsen.

1 Introduction

The Statutory Order of the Danish Ministry of Environment and Energy No. 151 of 15th March 1999 regulates the content of phthalates in toys and other children articles for the age group 0-3 years (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. In the Statutory Order, all diesters of o-phthalic acid have been considered as phthalates. This means that most of the commonly used phthalates in children's products are covered by the Danish regulation. The European Commission (EC) has also implemented a restriction on phthalate content in toys and other articles for children in the age group 0-3 years (2). The EC regulation is, however, temporary and that covers 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 EC regulation, the maximum authorised concentration of these phthalates (total phthalate content) in toys and other articles for children in the age group 0-3 years is ≤ 0.1% (m / m).

The Danish Environmental Protection Agency (DEPA) requested the National Environmental Research Institute (NERI) to determine the contents of phthalates in toys and other articles for children in the age group 0-3 years in order to check the compliance of these products with the Danish Statutory Order. In the present investigation, the contents of some commonly used phthalates in consumer products, including those regulated by EU (in toys), have been determined in 35 products provided by DEPA.

Present work has been performed as technical support to DEPA.

2 Products

DEPA provided 35 products for the analysis of phthalate content (Table 1). The products were collected in December 2001 from Danish retail out-lets. DEPA specified the parts of the products, which should be analysed for the content of phthalates.

Table 1. Toys and other articles analysed

NERI Reg. No.	MST No.	Product identification	Play toy description
2-0001	551	Holstein Bull	Toy
2-0002	552	Paddington Bear bib	Bib
2-0003	553	Chicco Little hand and foot	Teethers, shaped as hand and foot
2-0004	554	Fischer Price Play Family	A set of toy family
2-0005	555	Bathtime Duck	Play toy
2-0006	556	My first bathtime stacking boats	Boats
2-0007	557	Lamaze Celeste the Sun	Sun figure of synthetic fiber with a plastic handle
2-0008	558	Slice a riffic Sanswich set	Slices of bread, sausages etc.
2-0009	559	Vikingtoys	Lorry
2-0010	560	Picnic-basket	Basket, glass, thermos flask, etc.
2-0011	561	Play food	Slices of meat, chicken, vegetables, etc.
2-0012	562	Bondegårdsdyr	A set of toy animals
2-0013	563	Miracle Baby	Doll with feeding bottle
2-0014	564	Farmdyr med hegn	Farm animals with fence
2-0015	565	Toy Club Funny Toy	A calf with feeding bottle
2-0016	566	Aquafresh tandbørste	Tooth brush (elephant figure)
2-0017	567	My cuddly baby, doll	Doll
2-0018	568	Pegebog	Small laminated cardboard book
2-0019	569	Missers Fødselsdagsbog, mobile telephone book	Laminated cardboard book with built- in mobile telephone
2-0020	570	Disney's Peter Plys i klemme	A learning and feeling book, laminated cardboard book
2-0021	571	Hvad siger dyrene på bondegården	A learning (sounds of farm animals) book, laminated cardboard book
2-0022	572	And med kæde og bundprop i til badekarret	A duck with chain and stopper for bath tub
2-0023	573	Frog	Frog that can sprinkle water from mouth
2-0024	574	Disney's Mickey Mouse Floating water park	Boats and children figures
2-0025	575	Baby Mickey Activity keys	Several toys in a chain
2-0026	576-1	Monchichi boy, 28 cm	Monchichi boy made of synthetic fiber with hands, feet and head of soft plastic
2-0027	577	Happy street Fun time School and Playground	A set of various toys
2-0028	578	Winnie the Pooh, Musical activity ring	A set of figures formed in a ring
2-0029	579	Winnie the Pooh, The wet set Baby pool	Toy swimming pool

Table 1. Continued

DMU Reg. No.	MST No.	Product identification	Play toy description
2-0030	580	Winnie the Pooh, Squeeze' em	Winnie the Pooh, making sound when squeezed
2-0031	581	Sand Wand	A cylindrical plastic toy with restriction pockets
2-0032	582	102 Dalmatinas ball	A ball made of soft plastic
2-0033	583	Lego Babyspejl m. sol og bi	A baby mirror with bee, made of synthetic fiber with some small plastic parts
2-0034	584	Lego Biderangle	Teething rattle made of synthetic fiber and plastic
2-0593	576-2	Bølle Monchichi	Monchichi boy made of synthetic fiber with hands, feet and head of soft plastic

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), di-isononylphthalate (DINP), diisodecyl phthalate (DIDP) and butyl-benzyl phthalate (BBP). Duplicate subsamples of each product was soxhlet extracted in dichloromethane followed by analysis employing gas chromatography - mass spectrometry (GC-MS) and GC - flame ionisation detection (GC-FID), as described in earlier publications (3, 4).

3.1 Experimental

For qualitative analysis, 1 g sample was soxhlet extracted in 100 ml dichloromethane for 16 h at 60°C (waterbath). 90 ml of the extract was concentrated to 10 ml employing rotary evaporator. 1 µl of the concentrated as well as non-concentrated extract was analysed by GC-MS as described in 3.2.1. For quantification, fresh extracts of the samples were prepared so that concentration / dilution of the extracts was avoided, and they were analysed by GC-FID as described in 3.2.2. All samples were analysed in duplicate, both for qualitative and for quantitative analysis.

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 for 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 200 mg / L-10000 mg / L solutions of this chemical. DIDP was determined as DINP, because some of the GC peaks of isomers these substances overlapped. Thus, it was not necessary to prepare calibration curve for DIDP. In each GC sequence, 3 calibration standard solutions of the phthalates concerned were analysed after every 2-3 samples (4-6 extracts). These calibration curves were used for the quantification. Each standard solution as well as sample extracts were analysed two times by GC-FID. The contents of phthalates in a sample extract were calculated using the calibration standards analysed closest (in GC-sequence) to the sample extract.

Repeatability of the determination (precision) was calculated by 10 consecutive GC analysis of phthalate standard solutions 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.

The identification of phthalates was performed by comparing the GC retention times and mass spectra of the sample peaks with the retention times and mass spectra of phthalate standards. Moreover, chromatographic pattern of isomeric DINP and DIDP was considered for the identification of these phthalates. From the results of qualitative 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). In every second set of soxhlet extraction (8 extractions per .set), a blank was included.

3.2 Chromatographic conditions for phthalate analysis

3.2.1 GC-MS

Instrument:	Perkin Elmer Turbots Mass spectrometer coupled to AutoSystem XL gas chromatograph with split / splitless injector
Transfer line:	Direct to ion source, 310°C
GC column:	Chrompack fused silica column CP-Sil-5CB, 50 m x 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, total flow 55 ml / min, column head pressure 19.5 psi
Injector:	Split, 300°C, injection volume 1 µl
Ion source:	7 eV, 175°C, +ve ion mode, scan m / z 33-300 in 1 sec
MS Library:	NBS and Wiley

3.2.2 GC-FID

Instrument:	Hewlett Packard (HP) gas chromatograph 5890A with split / splitless injector, flame ionisation detector, HP autosampler 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, total flow 55 ml / 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 analyses of phthalate contents in the products were 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 qualitative analysis of phthalates was performed employing GC-MS. GC-FID was used for the quantification of the identified phthalates. The GC-MS method used in the present investigation is an extension of the GC-FID method (3, 4), that has previously been demonstrated to be suitable for the identification and determination of phthalates in toys. 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 GC retention times (t_R) of the investigated phthalates were stable through out the study, with a maximum variation <1%. The t_R of standard phthalates as well as their mass spectra were used for the identification of all phthalates. Moreover, 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 a visible GC-peak (without any visible noise at the base line) was 0.05% (w/w) for 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) as well as by analysing concentrated (9:1) sample extracts, so that phthalates present in low concentration (≤ 0.01%) could also be identified. The sample extracts containing high concentration of phthalates were reanalysed after appropriate dilution of the extracts. All of the samples were analysed in duplicate. The phthalates identified in some selected samples are shown in Figures 2-4. 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 ≥0.999) in the investigated concentration range: 200-10000 mg/L for DINP and 40 mg/L - 2000 mg/L for all other phthalates. The recovery of all of the investigated phthalates under the experimental conditions was 93-105%. The DINP recovery was determined at concentration levels 600 mg/L and 6000 mg/L, and the recoveries of all other phthalates were determined at concentration levels 60 mg/L and 600 mg/L.

The repeatability of determination was calculated 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 5%.

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 phthalate contents in the investigated products are described in Table 2. Of the 35 products (toys and other articles for children) investigated in the present study, one or more of the phthalates were found in 12 products. Among the investigated phthalates, BBP was present in 1 sample, DBP in 3 samples, DEHP in 4 samples, DINP in 6 samples and DIDP was present in 1 sample. None of the other investigated phthalates were identified in any of the products. It should be noted that when a product contains both DnOP and DINP, DnOP in such a product could not be determined by the present method because of interference by isomeric GC peak (s) of DINP (Figure 1).

According to Statutory Order of the Ministry of Environment, 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 12 samples containing phthalates, phthalate content in 4 samples were rather low (DMU No. 2-0009, 2-0018, 2-0019, 2.0021). Three of these products, laminated cardboard books (DMU No. 2-0018, 2-0019, 2.0021), contained < 0.30 % DBP. The manufacturer of these products declared that no phthalate was present in the lamination or the printing. The source of DBP in these product may thus be either the cardboard or the material used for gluing the lamination on cardboard. The fourth product with rather low phthalate content was a lorry: 0.08% DEHP was found in a wheel, while no phthalates were identified in the lorry platform. A rather low DINP content in this product may be due to contamination during the manufacturing process. The content of phthalates (mainly DEHP and DINP) in 8 other products was 3-40%.

Two samples of Monchichi boy (NERI No. 2-0026 and 2-0593, from two different batches) were analysed for the content of phthalates, in the present investigation. The plastic parts of the first sample ('Monchichi boy', NERI No. 2-0026) contained approximately 30-31% DINP (Table 2). The importer of the product, in response to the report of results of analysis, informed that this product is not marketed any more. The new product 'Bølle Monchichi' (NERI No. 2-0593) was then analysed to check the phthalate content. The new 'Monchichi' did not contain any of the investigated phthalates (Figure 4).

Table 2: Phthalate content in the investigated toys.

NERI Reg. No.	DEPA No.	The part/piece of the toy analysed	Phthalate content	
			Phthalate	Content (% W/W)
2-0001	551	Bull	BBP DINP+DIDP	0.075 28.61
2-0002	552	Backside of the bib	DINP	3.13
2-0003	553	Foot	-	-
2-0003	553	Hand	-	-
2-0004	554	Bear	-	-
2-0004	554	Doll	-	-
2-0004	554	Tiger	-	-
2-0004	554	Zebra	-	-
2-0005	555	Duck	-	-
2-0006	556	Boat, red	-	-
2-0007	557	Yellow handle	-	-
2-0008	558	Cheese	-	-
2-0008	558	Salad	-	-
2-0008	558	Tomato	-	-
2-0009	559	Lorry wheel, white	DEHP	0.08
2-0009	559	Lorry platform, yellow	-	-
2-0010	560	Cup, yellow	-	-
2-0010	560	Straw	-	-
2-0010	560	Fork, red	-	-
2-0011	561	Sausage, red	-	-
2-0011	561	Carrot	-	-
2-0011	561	Pommesfrit,	-	-
2-0011	561	Chicken	-	-
2-0012	562	Pig	-	-
2-0012	562	Bull	DEHP DINP	11.41 0.47
2-0012	562	Dog	DEHP DINP	18.99 0.41
2-0013	563	Neck	-	
2-0013	563	Foot	-	-
2-0013	563	Hand	-	-
2-0014	564	Pig	-	-
2-0014	564	Fence	-	-
2-0014	564	Dog	-	-
2-0015	565	Horse	DINP	38.50

NERI Reg. No.	DEPA No.	The part/piece of the toy analysed	Phthalate content	
			Phthalate	Content (% W/W)
2-0016	566	Elephant figure	-	-
2-0017	567	Head	-	-
2-0017	567	Arm	-	-
2-0017	567	Leg	-	-
2-0018	568	Part of a page	DBP	0.28
2-0019	569	Part of a page	DBP	0.20
2-0020	570	Part of a page	-	-
2-0021	571	Part of a page	DBP	0.07
2-0022	572	Duck, yellow	-	-
2-0022	572	Duck hat, blue	-	-
2-0023	573	Frog, green part	DINP	41.91
2-0023	573	Frog, yellow part	DINP	40.89
2-0024	574	Goofy	-	-
2-0024	574	Minnie	-	-
2-0025	575	Hand	-	-
2-0026	576-1	Face	DINP	31.15
2-0026	576-1	Foot	DINP	31.69
2-0026	576-1	Hand	DINP	30.09
2-0027	577	A boy figure	-	-
2-0027	577	Pine tree	-	-
2-0027	577	Dog	-	-
2-0028	578	Winnie the Pooh	-	-
2-0028	578	Hammock	-	-
2-0029	579	Transparent material	DEHP	23.58
2-0029	579	Painted material	DEHP	21.25
2-0030	580	Belly of Winnie the Pooh	-	-
2-0031	581	Soft plastic, yellow	-	-
2-0032	582	Ball	DEHP	23.28
2-0033	583	Wings of the bee	-	-
2-0034	584	Red plastic material	-	-
2-0593	576-2	Face	-	-
2-0593	576-2	Hand	-	-
2-0593	576-2	Foot	-	-

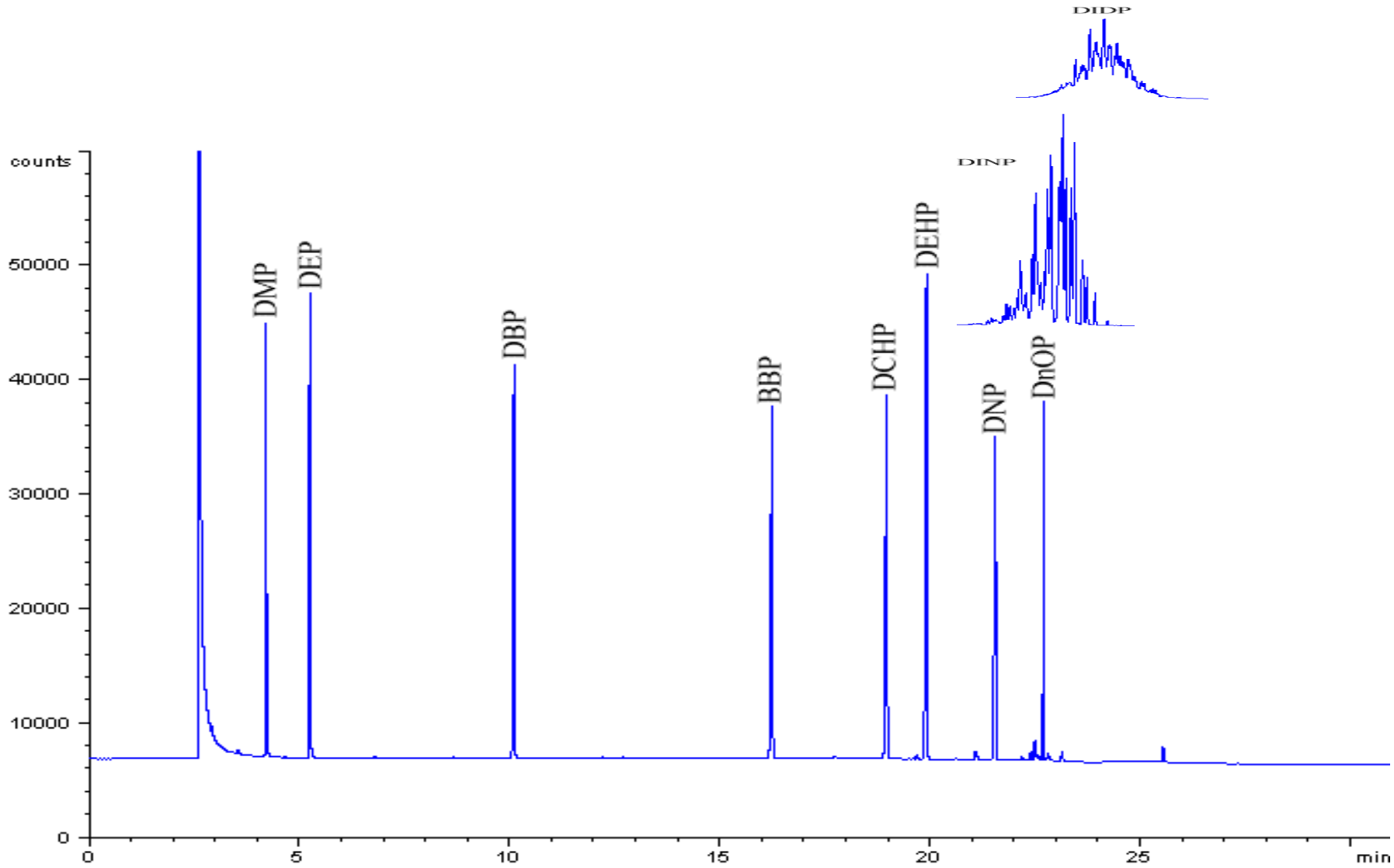

Figure 1: GC-separation of investigated phthalates. DINP and DIDP approximately 0.6% and other phthalates approximately 0.04%.

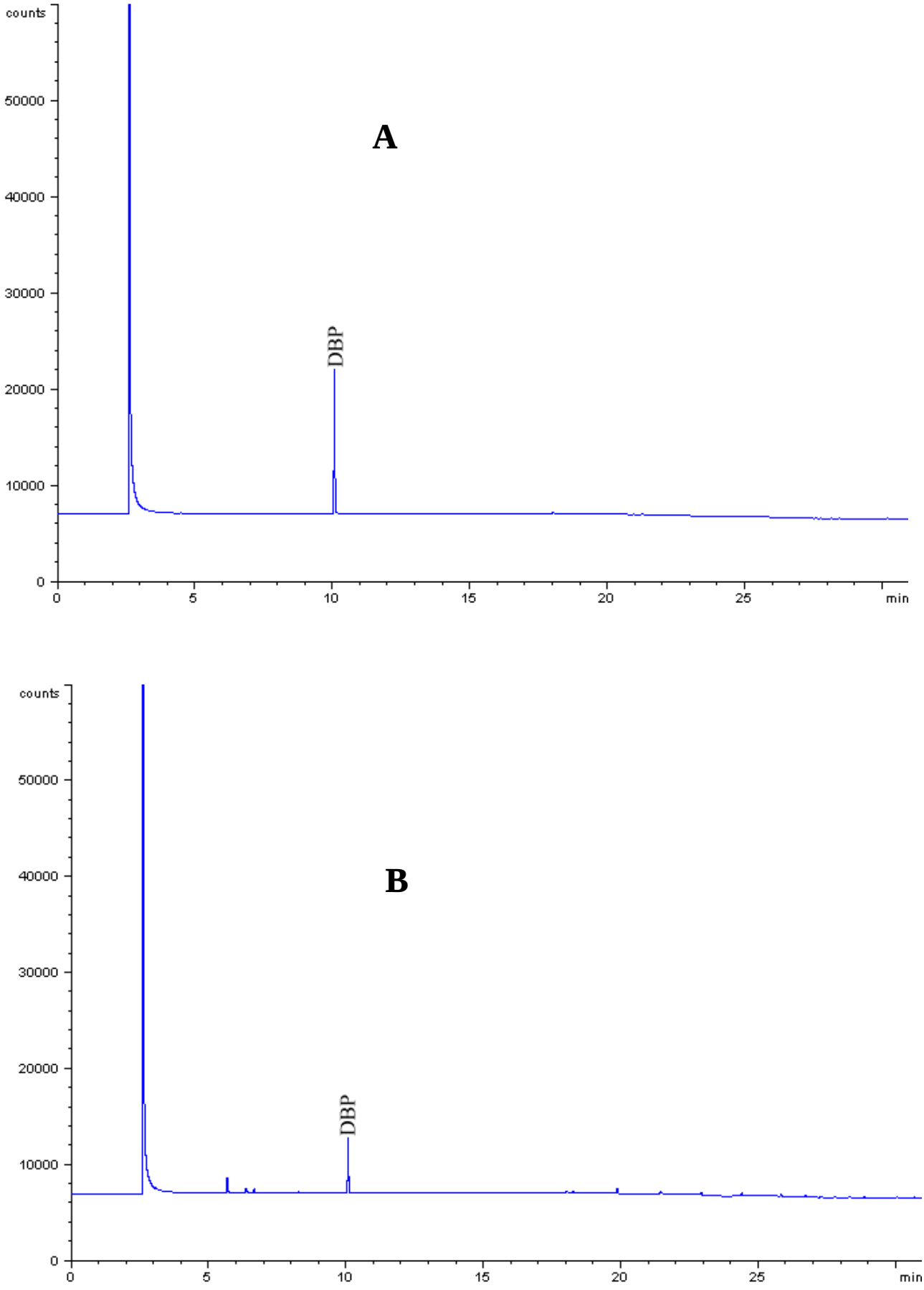

Figure 2: GC chromatogram of extracts laminated cardboard books 2-0019 (**A**) and 2-0021 (**B**)

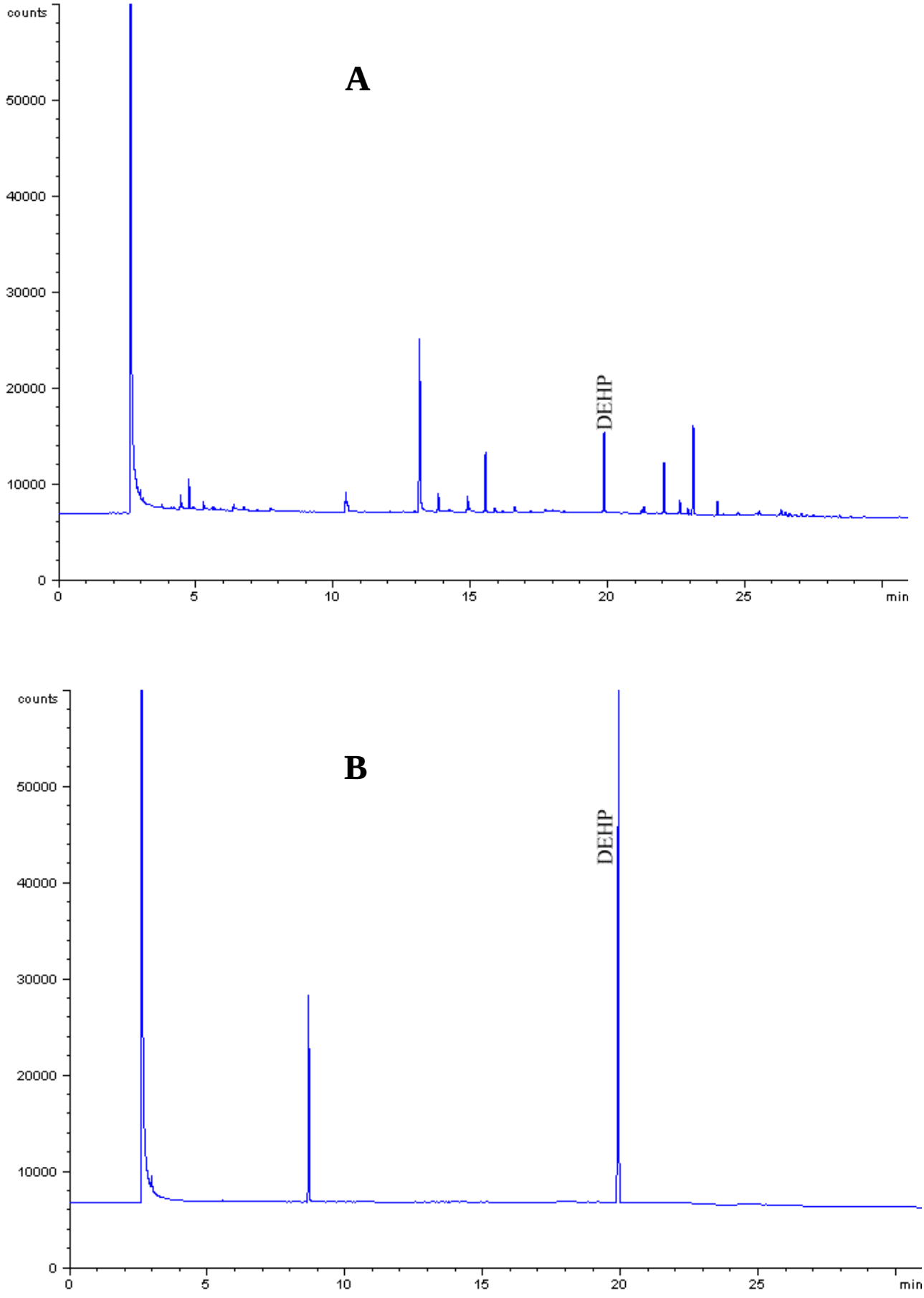

Figure 3: GC-chromatogram of extracts of **A** - lorry wheel (NERI No. 2-0009) and **B** - Dalmatinas ball (NERI No. 2-0032).

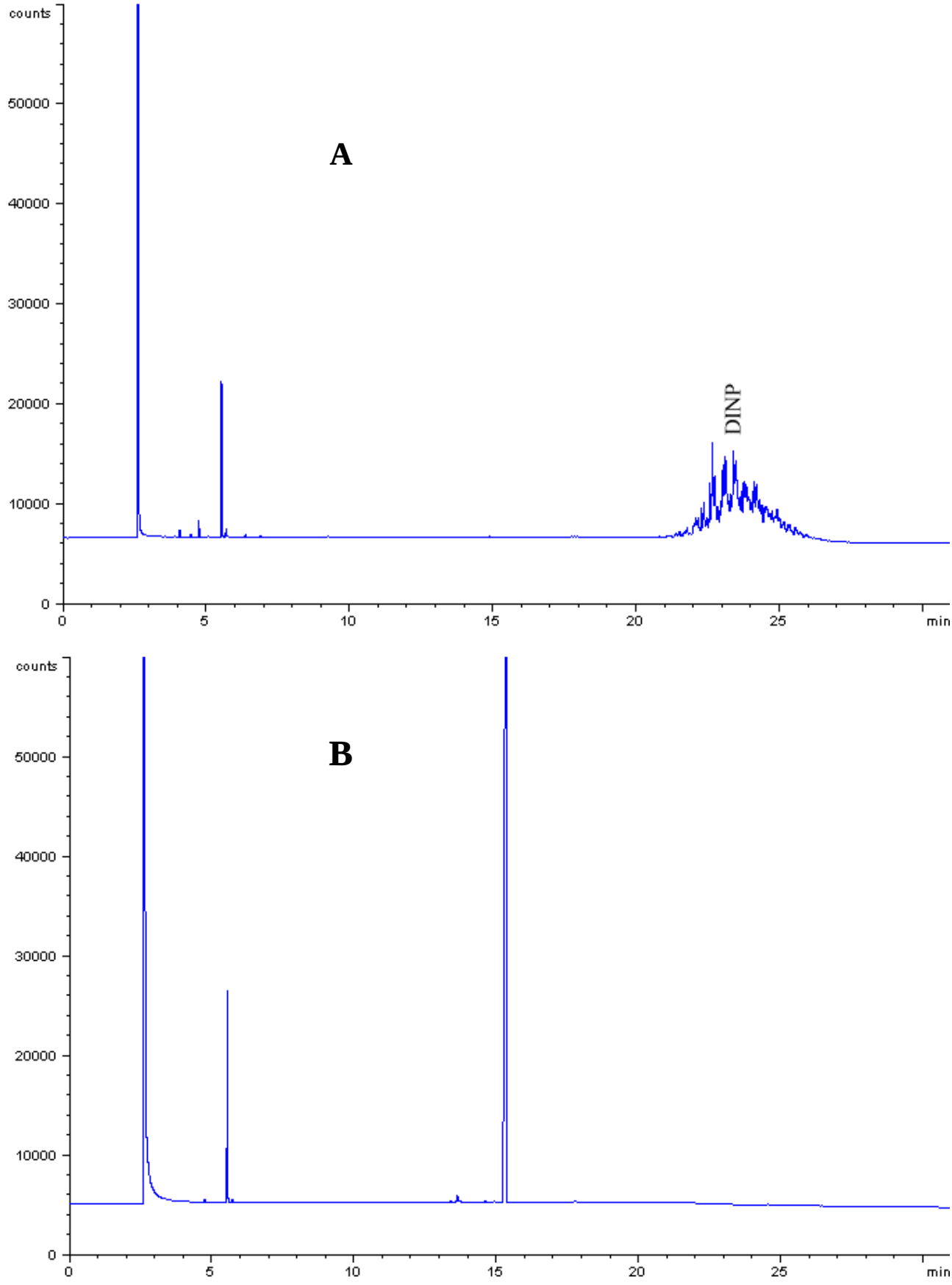

Figure 4: GC-chromatogram of face-extracts of **A -** Monchichi boy (NERI No. 2-0026) and **B** - Bølle Monchichi (NERI No. 2-0593)

5 References

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2. Forslag til Europa-Parlamentets og Rådets Direktiv om toogtyvende ændring af Direktiv 76/769/EØF om indbyrdes tilnærmelse af medlemstaternes 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).

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

Addresses:

URL: http://www.dmu.dk

National Environmental Research Institute
Frederiksborgvej 399
PO Box 358
DK-4000 Roskilde
Denmark
Tel: +45 46 30 12 00
Fax: +45 46 30 11 14

Management
Personnel and Economy Secretariat
Research and Development Section
Department of Policy Analysis
Department of Atmospheric Environment
Department of Marine Ecology
Department of Environmental Chemistry and Microbiology
Department of Arctic Environment
Project Manager for Quality Management and Analyses

National Environmental Research Institute
Vejlsøvej 25
PO Box 314
DK-8600 Silkeborg
Denmark
Tel: +45 89 20 14 00
Fax: +45 89 20 14 14

Environmental Monitoring Co-ordination Section
Department of Terrestrial Ecology
Department of Freshwater Ecology
Project Manager for Surface Waters

National Environmental Research Institute
Grenåvej 12-14, Kalø
DK-8410 Rønde
Denmark
Tel: +45 89 20 17 00
Fax: +45 89 20 15 15

Department of Landscape Ecology
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