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Michal Voldřich

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The occurrence of extractible ink residuals in packaging materials used in the Czech Republic.

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Abstract

Residues of printing ink components were determined in 94 samples of packaging materials commercially used in the Czech Republic for food packaging. The samples which were tested included printed polyethylene and polypropylene films, co-extruded and laminated films, paperboard beverages boxes, foils for thermo sealing of polystyrene cups and polypropylene cups. Printing ink components were extracted with diethylether, then separated and determined using GC/MS. Fifty compounds potentially originating from printing were isolated, identified and quantified. No acute health risk for consumers were identified even though several findings of high levels of photoinitiators (e.g. in PET films for thermo sealing of polystyrene cups) as well as plasticizers (acetyl tributyl citrate, tributyl aconitate, 2-butoxyethyl oleate and 2-ethylhexyl diphenyl phosphate in co-extruded films) indicate that their real migration into food and or food simulants needs to be undertaken for a proper safety evaluation.

Keywords: food packaging, printing inks, migration, food safety

Introduction

Printing inks used in food packaging production contain many substances that have not been toxicologically evaluated, since they are not coming into direct contact with food. However, these substances (e.g. pigments, plasticisers, solvents, photoinitiators, stabilizers, their breakdown products, etc.) may be transferred from the printing to the food contact surface either by penetration through the packaging material or via the set-off transfer phenomenon (transport of printing ink constituents from printed surface to the unprinted side of another sheet in a stack or roll).

When the printing ink components reach the food contact layer of the package, they could migrate into the packaged food due to extraction by food, evaporation and/or steam distillation during storage and/or heating in original package (Sun Chemical 2007). As printing is not in a direct contact with food no specific legislation concerning printing inks for food packaging has been agreed so far in EU. The Framework Regulation (EC 2004)

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3 applicable to all food packages only states that printing for food packages in EU will be in
4 future controlled by particular system of directives and regulations. At present materials and
5 articles intended to come in contact with foods should comply with the general criteria laid
6 down in Art. 3 of above mentioned Framework Regulation, i.e. should not transfer their
7 constituents in food in quantities, which could endanger the human health or bring about
8 unacceptable changes in composition or characteristics of foodstuffs. These criteria are also
9 reiterated in the Council of Europe Resolution (Council of Europe 2005). To prove the
10 conformity with above mentioned demand the risk assessment is necessary. This is the
11 responsibility of packaging material and/or food contact article producer. The Framework
12 Regulation (EC 2004) also requires the traceability of food contact materials and articles at all
13 stages as well as production by Good Manufacturing Practice (EC 2006, EuPIA 2008, Sun
14 Chemical 2007).

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27 The influence of the printing system on food packaging safety, mainly migration of solvents,
28 plasticizers and photoinitiators, has been already studied. Skjevraak et al (2005) tested
29 migration from 75 samples of plastic laminates for food packaging into oil and/or distilled
30 water and they found eight major migrants with level of migration above 0.3 mg/kg, six of
31 them originated (or could have originated) from printing and/or coating, i.e. dioctyl adipate,
32 diisooctyl adipate, 2-(2-butoxyethoxy)ethanol, bisphenol A diglycidyl ether (BADGE),
33 methyl ester of dehydroabiatic acid and N-ethyl-toluenesulfonamide. They also identified four
34 primary aromatic amines originated from printing (2-methyl-aniline, 2,4,-dimethylaniline, 2-
35 methoxyaniline and 4,4'-methylene-dianiline) at migration level <0.02 mg/kg. Nevertheless
36 their results showed that migration of non-authorized substances in found level did not
37 present a risk to consumer health. Migration of plasticizers (mainly esters of phthalic acid,
38 adipic acid, sebacic acid, citric acid and mellitic acid, but also N-methyl- and N-ethyl-
39 toluenesulfonamide) from printing inks into packaged foodstuffs was also reported (Nerin et al.
40 1993, Balafas et al. 1999, Piergiovanni et al. 1999).

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55 The danger of residues of photoinitiators (PI) migrating from UV-cured printing of food
56 packages has been reported (Castle 1997). Their transfer by set-off caused significant damage
57 for producers of beverages like milk, cocoa or juice packaged in paperboard packages in
58 2005, when the substances 2-isopropyl thioxanthone (ITX) and 2-ethylhexyl-4-
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3 dimethylaminobenzoate (EHDAB) were detected in packaged products at levels up to
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5 300 µg/kg food. (EFSA 2005, EuPIA 2005b) These findings often exceeded a maximum
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7 migration level of 50 µg/kg food, which was announced by the European Food Safety
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9 Authority (EFSA) as an acceptable threshold for substances with no genotoxic potential
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11 (EFSA 2005, Gil-Vergara et al. 2007, Benetti et al. 2008). Kleinschnitz and Schreier (1998)
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13 reported the determination of 2,4,7,9-tetramethyl-5-decyne-4,7-diol in mineral water
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15 packaged in paperboard beverage boxes up to levels of 50 µg/l. The origin of this substance
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17 was a surfactant in water-based printing inks. Aurela et al. (2001) described the identification
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19 of alkylbenzenes (alkyl chain C₁₀-C₁₃) in 10 out of 15 samples of offset-printed food
20
21 packaging made of paperboard. Alkylbenzenes are used as solvent components in some offset
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23 printing inks and the authors found migration into packaged product up to 2 mg/kg.
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26 The main aim of this study was to screen polymer printed packaging materials commercially
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28 used in Czech Republic to obtain objective data for an evaluation of possible contamination of
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30 packaged food with printing ink constituents.
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33 **Materials and methods**

34 Packaging materials:

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36 *Printing ink residuals testing.* 94 food packaging materials were tested. All of them are
37
38 currently used for food packaging in Czech Republic. The set of samples included:
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- 41 i. 3 films made of low density polyethylene (LDPE) and 2 samples from polypropylene
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43 (PP) with direct printing on outside surface.
- 44 ii. 8 laminates PP-metalized/LDPE, 2 laminated films of metalized polyethylene
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46 terephthalate (PET) and LDPE (PET-met/LDPE) and one laminate PP/Al/LDPE with
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48 reverse printing on inside surface of upper layer.
- 49 iii. 2 laminates PET/LDPE, 9 coextruded films PP/LDPE, 16 films of laminates
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51 paper/Al/LDPE and 10 laminates paper/LDPE with printing on the outside surface.
- 52
53 iv. 15 laminates paper/Al/LDPE for production of beverages boxes with printing on
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55 outside paper surface.
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57 v. 8 aluminium foils and 15 PET films coated with thermosealable lacquer for sealing
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59 polystyrene (PS) cups with printing on the outside surface.
- 60 vi. 2 cups of PP printed on the outside surface.

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3 Concerning printing techniques used for tested packaging materials production 53 samples
4 and 26 packaging materials were printed with flexography using UV-cured and not UV cured
5 inks, respectively. 15 samples were produced using gravure print.
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10 Analytical methods:

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12 *Determination of free printing ink constituents by diethyl ether extraction.* 1 dm² of tested
13 packaging material was extracted with 50 ml of diethyl ether (p.a., Sigma-Aldrich
14 Chemie,GmbH) in Soxtec™ 2043 extractor (Foss Analytical, DK) for two hours. Diethyl
15 ether extract was evaporated to dryness at 40 °C using a vacuum evaporator and redissolved
16 in 2 ml of diethyl ether. 1 µl of this solution was injected into a gas chromatograph coupled
17 with a mass spectrometry detector and analysed at following conditions: gas chromatograph -
18 Agilent 6890N (Agilent Technologies); detector – Agilent 5973N mass detector, electron
19 impact ionisation 70 eV, temperature 280 °C; capillary column - DB-5 (30 m x 0.25 mm x
20 0.25 µm); mobile phase - He, flow rate 0.6 ml/min, linear speed 28.9 cm/s; injection - 1 µl
21 using split 1 : 100, temperature 300°C; column temperature - 70 °C for 5 minutes, increase 15
22 °C/min to 300 °C, 300 °C to the analysis end. Identification of separated substances consisted
23 in comparison of obtained mass spectra with the spectrum library of used chromatography
24 software (NIST MS Search 2.0) and its confirmation using the mass spectra of analysed
25 standards. The quantification was done using diethyl phthalate (p.a., standard for GC, Sigma-
26 Aldrich Chemie, GmbH) as the internal standard. At least three parallel samples for each
27 tested packaging film were analysed. The results in the next text are expressed as mean
28 values± standard deviations.
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55 The analytical deviation for reference compounds at 10 mg/l (according with migration level
56 of 10 µg/dm²) concentration level in diethylether solution typically corresponded to 5-10%
57 RSD and at 100mg/l (100 µg/dm²) to 3-5% RSD.
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61 **Results and discussion**

62 Having planned this study, we had to overpass some problems. Firstly we did not get either
63 interest for cooperation from printing ink producers so that we had to obtain the packaging
64 material samples from food and packaging material producers, i.e. we could not exploit any
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3 more detailed knowledge on tested printing parameters. Unfortunately there was no
4 possibility to obtain and test unprinted packaging materials as controls. As printing inks form
5 quite complex chemical systems and due to little information on tested printings composition,
6 we decided to analyze the packaging materials samples using the gas chromatography coupled
7 with mass spectroscopy detector as this technique offers the most convenient identification of
8 separated substances. Moreover the potential migrants capable of transfer from printing to
9 packaged food, which could present the threat for consumer, should be first of all organic
10 substances of lower molecular weight with sufficiently low boiling temperature.
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20 Three techniques of isolation of printing ink constituents were originally tested, i.e.
21 adsorption on Tenax TA sorbent, application of solid phase micro extraction method (SPME)
22 and direct extraction with diethyl ether. The last procedure was found the most efficient, as
23 the number of isolated and identified substances was higher comparing with the other
24 techniques. The examples of typical chromatograms are shown in Figure 1
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32 *Identification of ink components released from tested films*

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34 From the tested films 50 substances (see Table I) were isolated and identified in amount
35 exceeding 0.001 mg/dm^2 . According to the published data, these substances could originate
36 from printing inks even it is obvious that extraction with diethyl ether did not enable to
37 distinguish between additives released from polymers and those really extracted from printing
38 as only the printed packaging materials were tested. Extractible levels of identified substances
39 ranged from $0.0015 \pm 0.0002 \text{ mg/dm}^2$ for lauramide to $0.898 \pm 0.017 \text{ mg/dm}^2$ for acetyl
40 tributyl citrate.
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50 The presence of substances obviously originated from printing, which are not allowed for
51 polymer food contact materials, is interesting, e.g. dichlor benzenamine (probably the
52 breakdown product of aryl pigments), six photoinitiators and/or their breakdown derivatives
53 (isopropyl thioxantone, 4-phenyl benzophenone, 4-hydroxy benzophenone, ethyl-4-dimethyl
54 aminobenzoate, 2-ethylhexyl-4-dimethylaminobenzoate and trimethyl benzaldehyde). On the
55 other hand there were substances present in amount exceeding 0.500 mg/dm^2 , e.g.
56 diethylhexyl adipate (up to $0.698 \pm 0.014 \text{ mg/dm}^2$), 2-ethylhexyl diphenyl phosphate ($0.873 \pm$
57 0.026 mg/dm^2) and acetyl tributyl citrate ($0.898 \pm 0.017 \text{ mg/dm}^2$).
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3 The most frequent substance found in tested samples of packaging materials was diethylhexyl
4 adipate, which was identified in 24 samples included beverage board boxes, laminated and
5 co-extruded packaging films and polyethylene terephthalate films for sealing of cups with
6 yoghurt. The maximum content 0.698 ± 0.014 mg/dm² was found in beverage board box
7 printed using flexography. In 23 samples (paper board boxes, laminated and/or co-extruded
8 films) oleamide was identified in maximal level of 0.220 ± 0.004 mg/dm². The highest
9 concentration of printing ink constituent (i.e. 0.898 ± 0.017 mg/dm²) was found for acetyl
10 tributyl citrate in co-extruded film for biscuit packaging. This substance was identified in
11 twelve more packaging films. 2-ethylhexyl diphenyl phosphate was identified in 8 samples of
12 co-extruded and/or laminated films for biscuit packaging at maximum level of 0.873 ± 0.026
13 mg/dm². Erucamide was found in one sample of beverage board box at level 0.232 ± 0.007
14 mg/dm².
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27 Seven PIs were identified in samples printed with UV curable inks. Ethyl 4-
28 dimethylaminobenzoate was the identified in ten samples of PET films for sealing of
29 polystyrene cups at maximal level of 0.151 ± 0.005 mg/dm², 4-phenyl benzophenone was
30 found in 9 samples including laminated films for packaging of spices, soup condiment and/or
31 dehydrated soups as well as PET films and aluminium foils for sealing polystyrene cups at
32 maximal level 0.139 ± 0.006 mg/dm². The more known PIs 2-isopropyl thioxanthone (ITX)
33 and 2-ethylhexyl 4-dimethylaminobenzoate (EHDAB) were found only in 4 and 2 cases,
34 respectively. This fact is probably the consequence of the attitude of food producers in Czech
35 Republic, whose generally refuse packaging materials printed with inks containing ITX and
36 EHDAB. The highest found contents of ITX and EHDAB were 0.182 ± 0.010 mg/dm² and
37 0.0759 ± 0.0023 mg/dm², respectively.
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50 Tributyl aconitate was identified in 14 samples of packaging materials (beverage boxes,
51 laminated films) at level up to 0.0764 ± 0.0046 mg/dm², toluene-2,4-diisocyanate in 12
52 samples (PET and aluminium sealing foils, co-extruded and laminated films) up to $0.0611 \pm$
53 0.0048 mg/dm², dibutyl sebacate in two films for biscuit and spice packaging up to $0.0616 \pm$
54 0.0043 mg/dm² and 2-butoxy ethyl oleate in one laminated film for vanilla sugar packaging at
55 level 0.0616 ± 0.0037 mg/dm².
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3 The above mentioned results are generally comparable with findings of Skjevrak et al. (2005)
4 for laminated food packaging films. The main difference is an absence of N-ethyl-toluene
5 sulfonamide (NETSA). NETSA was used as plasticizer in resins, cellulose acetate,
6 nitrocellulose, lacquers adhesives and inks and in Norwegian study this substance was
7 isolated from printed laminated films in by far the highest amount. Its absence in materials
8 tested in this study should certify the intent of packaging film producers not to use NETSA in
9 printing inks (Skjevrak et al. 2005).
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18 Besides the above mentioned residues, fifteen substances were detected but not identified, and
19 so far were characterised only by mass spectra, but were present at quite significant levels
20 (about 50-100 $\mu\text{g}/\text{dm}^2$), too. Their identification will pose one of the main tasks for the
21 authors.
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28 *Evaluation of possible risk of potential migrants originated from printing inks*

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30 When migration of substances not included in the EU positive list of monomers and/or
31 additives for food contact polymer is identified, assessments of health risks are usually carried
32 out. In this study the content of substances which could be released and potentially transferred
33 into packaged product was determined, but no data on real migration were obtained. It means
34 that no proper health risk assessment could be done. Nevertheless the possible danger due to
35 presence of identified substances could be estimated.
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44 From substances not included in the EU positive list identified in Table 1 the occurrence of
45 four PIs (ethyl 4-dimethylaminobenzoate, EHDAB, 4-phenyl-benzophenone and ITX) at
46 levels up to 0.182 mg/dm^2 could pose problems. These substances were identified in the
47 printing of barrier films for spice, condiment and or dehydrated soups packaging. These
48 materials eliminate the possibility of PIs penetration through the package. Considering the
49 extent of set-off transfer in agreement with this study results, i.e. 1 % of PI amount in lacquer
50 per month at 40 °C, we can expect the safety of packaging material for approximately 2 year
51 storage at 20 °C. Similar situation can be expected for aluminium foils for thermo sealing of
52 polystyrene cups with yoghurt, moreover there is advantage in smaller area of foil in contact
53 with food. In the PET films for thermo sealing of polystyrene cups the maximal found amount
54 of PI reached the level 0.151 mg of ethyl 4-dimethylaminobenzoate per dm^2 . Due to good
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3 barrier parameters of PET film and the layer of thermo sealable lacquer on the base of
4 polyvinyl dichloride (PVdC) as well as the fact that the area of such packaging material in the
5 contact with food is quite limited the real risk for consumer could be considered as quite low.
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7 Nevertheless the migration tests of these materials would be advisable.
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13 Acetyl tributyl citrate was identified in different types of packaging materials including
14 coextruded PP/LDPE films in levels up to 0.898 mg/dm². Due to the absence of efficient
15 barrier it would be suitable to verify migration of this additive through the tested packaging
16 films even if acetyl tributyl citrate is accounted the material of low toxicity. Similar situation
17 could be stated for tributyl aconitate, which was found in 14 tested samples in maximal level
18 0.0764 mg/dm², and 2-butoxyethyl oleate that was identified only in one tested sample in
19 amount 0.0616 ± 0.0037 mg/dm².
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28 Considering identified substances included in EU positive list the high content of 2-ethylhexyl
29 diphenyl phosphate in five tested samples of co-extruded films (up to 0.873 mg/dm²) is
30 suspicious. The set specific migration limit for this additive is 2.4 mg/kg, i.e. 0.4 mg/dm², and
31 in agreement with the results obtained by this study it could be surpassed in contact with food
32 simulants. In this case also the migration test of relevant packaging materials should be done
33 for final assessment.
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42 The migration tests of PIs, acetyl tributyl citrate, tributyl aconitate, 2-butoxyethyl oleate and
43 2-ethylhexyl diphenyl phosphate from tested packaging materials samples will be carried out
44 in the next part of project solution.
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50 Conclusion

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52 The main results of carried out experiments can be summed as follows:

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54 i) The determination of residues of printing inks in 94 food packaging materials
55 commercially used in Czech Republic did not indicate any acute health risk for food
56 consumer.
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59 ii) Suspicious findings of higher levels of PIs in PET films for thermosealing of
60 polystyrene cups as well as acetyl tributyl citrate, tributyl aconitate, 2-butoxyethyl

oleate and 2-ethylhexyl diphenyl phosphate should be completed with information on real migration into food and or food simulants.

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Table I – List of identified potential residuals from printing inks found in 94 examined samples of packaging materials extracted in level above 0.001 mg/dm^2 (PL = included in EU positive list of Directive 2002/72/EC, SCF-L = Scientific Committee of Food list as defined e.g.in EFSA 2008, TSCA = Toxic Substances Control Act)

No.	Printing ink residue	CAS-no.	Toxicological data	highest content (mg/dm^2)	number of occurrence	origin/function	Included in EU positive list for polymers (4, 24)
1	Acetyl tributyl citrate (1,2,3-Tributyl 2-acetyloxypropane-1,2,3-tricarboxylate)	77-90-7	<ul style="list-style-type: none"> LD50 intraperitoneal, mouse: >4g/kg; low toxicity by ingestion, skin contact, and intraperitoneal routes; TSCA listed (1) 	0.898 ± 0.017	13	Solvent, film-former and plasticizer for inks, adhesives, coatings (1)	PL
2	Butyl hydroxyl toluene (4-methyl-2,6-ditert-butyl-phenol)	128-37-0	<ul style="list-style-type: none"> ADI 0.05 mg/kg bw/d (5) 	0.020 ± 0.002	7	Antioxidant	SML = 0.5 mg/dm^2 (SCF-L 1)
3	Butyl palmitate (butyl hexadecanoate)	111-06-8	<ul style="list-style-type: none"> Maximised Survey-derived Daily Intakes (MSDI-EU) : 0.79 $\mu\text{g/capita/day}$; modified Theoretical Added Maximum Daily Intake (mTAMDI) : 3500 $\mu\text{g/person/day}$; Threshold of concern : 1800 $\mu\text{g/person/day}$ (2) 	0.0045 ± 0.0005	1	Lubricant (1)	PL (SCF-L 3)
4	2-Butoxyethyl oleate (2-butoxyethyl cis-octadec-9-enoate)	109-39-7		0.0616 ± 0.0037	1	Plasticizer, lubricant (3)	

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5	Caprolactam (azepan-2-one)	105-60-2	<ul style="list-style-type: none"> ADI 0.25 mg/kg bw/d (24) 	$\frac{0.0265 \pm 0.0008}{}$	1	Monomer, component of external colour printing (1,5)	PL, SML(T) = 2.5 mg/dm^2 (SCF-L 2)	<p>Deleted: .</p> <p>Deleted: 15</p> <p>Deleted: kg</p> <p>Formatted: Superscript</p>
6	Dibutyl sebacate (dibutyl decanedioate)	109-43-3	<ul style="list-style-type: none"> LD50 oral, rat 16 g/kg; Mildly toxic by ingestion, experimental reproductive effects; TSCA listed (1) 	$\frac{0.0616 \pm 0.0043}{}$	2	Plasticizer, solvent (1)	PL (SCF-L 3)	<p>Deleted: .</p>
7	2,5-Dichloroaniline	95-82-9		$\frac{0.0031 \pm 0.0004}{}$	1	Azo dyes decomposition product (6)	(limit for primary aromatic amines SML(T) = 0.0016 mg/dm^2)	<p>Deleted: .</p> <p>Deleted: 0033</p>
8	Diethylhexyl adipate (bis(2-ethylhexyl) hexanedioate)	103-23-1	<ul style="list-style-type: none"> ADI 0.3 mg/kg bw/d (24) 	$\frac{0.698 \pm 0.014}{}$	24	Plasticizer, solvent (1)	PL SML = 3 mg/dm^2 (SCF-L 2)	<p>Deleted: .2</p> <p>Deleted: 18</p> <p>Deleted: kg</p> <p>Formatted: Superscript</p>
9	Diisobutyl phthalate (bis(2-methylpropyl) benzene-1,2-dicarboxylate)	84-69-5	<ul style="list-style-type: none"> LD50 oral, rat: 15g/kg, skin, guinea pig: 10g/kg, Mildly toxic by ingestion and skin contact; Experimental teratogen; Reproductive effects; TSCA listed (1) 	$\frac{0.0152 \pm 0.0012}{}$	2	Plasticizer, solvent for paints; vehicles for pigment dispersions (1,7,8)	(SCF-L 6B)	<p>Deleted: .</p>
10	Diisooctyl phthalate (bis(6-methylheptyl) benzene-1,2-dicarboxylate)	27554-26-3	<ul style="list-style-type: none"> ADI 0.15 mg/kg bw/d (24) 	$\frac{0.0048 \pm 0.0003}{}$	13	Plasticizer, solvent (1,7,8)	PL SML(T) = 1.5 mg/dm^2 (in combination with higher molecular weight esters for DIOP maximally 0.5 mg/dm^2)	<p>Deleted: 9</p> <p>Deleted: kg</p> <p>Formatted: Superscript</p> <p>Deleted: .</p> <p>Deleted: 3</p> <p>Deleted: kg</p> <p>Formatted: Superscript</p>

11	Diocetyl adipate (<i>diocetyl hexanedioate</i>)	123-79-5	<ul style="list-style-type: none"> LD50 oral, rat: 9.1 g/kg, oral, mouse: 15g/kg (9) 	$\frac{0.0053 \pm 0}{.0005}$	4	Plasticizer, solvent and raw material for adhesives and coatings (1)	(SCF-L 6B)
12	4,4'-Diphenylmethane diisocyanate (<i>1-isocyanato-4-[(4-isocyanatophenyl)methyl]benzene</i>)	101-68-8	<ul style="list-style-type: none"> LD50 oral, rat: > 10 g/kg; LC50 Inhalation, rat: 450 mg/l/4 h (10) 	$\frac{0.0057 \pm 0}{.0004}$	2	Polyurethane inks and adhesives (1)	PL QM(T) = 1 mg/kg (as NCO) (SCF-L 4A)
13	2,4-Ditert-butylphenol	96-76-4	<ul style="list-style-type: none"> LD50 oral, mouse: 700 mg/kg; oral, rat: 2400 mg/kg; skin, rabbit: 2200 mg/kg (11) 	$\frac{0.0071 \pm 0}{.0005}$	1	UV absorber or degradation product from antioxidants (e.g. Irgafos) (1,12)	
14	Docosan-1-ol	661-19-8	<ul style="list-style-type: none"> Low toxicity; TSCA listed (1) 	$\frac{0.0057 \pm 0}{.0005}$	1	Lubricant, surfactant, plasticizer, solvent (1)	PL (<i>PM/REF No. 33120</i>) (SCF-L 3)
15	Erucamide (<i>cis-docos-13-enamide</i>)	93050-58-9	Skin, eye and respiratory irritant (13)	$\frac{0.232 \pm 0}{.007}$	1	Slip agent, lubricant (1)	PL (SCF-L 3)
16	Ethyl 4-dimethylaminobenzoate	97003-31-1		$\frac{0.151 \pm 0}{.005}$	10	UV absorber, photoinitiator (1,14)	
17	3-Ethyl-5-(2-ethylbutyl)-octadecane			Approx. 5.0	3		
18	2-Ethylhexyl 4-dimethylaminobenzoate	21245-02-3	<ul style="list-style-type: none"> No teratogenic effects; NOEL 40 mg/kg (15) 	$\frac{0.0759 \pm 0}{.0023}$	2	UV absorber, photoinitiator (1,14)	
19	2-ethylhexyl diphenyl phosphate (<i>3-(diphenoxyphosphoryloxymethyl)heptane</i>)	1241-94-7	<ul style="list-style-type: none"> ADI 0.04 mg/kg bw/d (24) 	$\frac{0.873 \pm 0}{.026}$	8	Rubber accelerator, plasticizer, lubricant, fire retardant (1)	PL SML = $\frac{0.6 \text{ mg}}{\text{dm}^2}$ (SCF-L 2)
20	Glycerol-1-oleate (<i>2,3-dihydroxypropyl cis-octadec-9-enoate</i>)	25496-72-4		$\frac{0.0074 \pm 0}{.0006}$	2	Lubricant, solvent (1)	SCF-L 1

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21	Glyceryl diacetate (<i>(2-Acetyloxy-3-hydroxy-propyl) acetate</i>)	25395-31-7	<ul style="list-style-type: none"> LD50 oral, mouse: 8.5 g/kg; subcut, rat: 4 g/kg; TSCA listed (1) Low toxicity, official food additive E1517 (16); 	$\frac{0.0031 \pm 0}{.0002}$	1	Plasticizer, solvent (1)	PL, SCF-L 1	Deleted: .
22	Hexadecan-1-ol	8032-89-1	<ul style="list-style-type: none"> LD50 oral, rat: 6.4 g/kg (1) Skin, eye and respiratory irritant (1,17) 	$\frac{0.0037 \pm 0}{.0002}$	2	Dispersion stabilizer, solvent (1)	PL (<i>PM/REF No. 33120</i>) (SCF-L 3)	Deleted: .
23	4-hydroxybenzophenone (<i>phenyl-(4-hydroxyphenyl)-methanole</i>)	1137-42-4	<ul style="list-style-type: none"> LD50 oral, mouse: 3724 g/kg; oral, rat: 12086 mg/kg (18) 	$\frac{0.0064 \pm 0}{.0004}$	3	UV stabilizer, photoinitiator (14)		Deleted: .
24	Isobutyl stearate (<i>isobutyl octadecanoate</i>)	646-13-9	<ul style="list-style-type: none"> Primary skin irritant, TSCA listed (1) 	$\frac{0.0393 \pm 0}{.0035}$	1	Additive into inks, dyes (1)	(SCF-L-7)	Deleted: .
25	Isodecyl diphenyl phosphate (<i>1-diphenoxyphosphoryloxy-8-methyl-nonane</i>)	59800-46-3	<ul style="list-style-type: none"> LD50 oral/rat: 15.8 g/kg; LC 50: 96 hr/bluegill: 6.7 g/l (19, 20) 	$\frac{0.0038 \pm 0}{.0004}$	1	Plasticizer, lubricant, fire retardant (19)		Deleted: .
26	Isopropyl 9,10-epoxyoleate (<i>propan-2-yl 8-(3-octyloxiran-2-yl)octanoate</i>)	95007-80-0		Approx. 4.0	2	Component of light aged varnishes ?		
27	Isopropyl-2-ethylhexanoate (<i>propan-2-yl 2-ethylhexanoate</i>)	67024-46-8		$\frac{0.0023 \pm 0}{.0002}$	1	Plasticizer, solvent, ink gallant (21)		Deleted: .
28	Isopropylthioxanthone (<i>2-Propan-2-ylthioxanthen-9-one</i>)	5495-84-1		$\frac{0.182 \pm 0}{.010}$	4	UV absorber, photoinitiator (14)		Deleted: 1.9
29	Lauramide (<i>dodecanamide</i>)	1120-16-7		$\frac{0.0015 \pm 0}{.0002}$	1	Polymer additive; release agent for food packaging (1)		Deleted: .
30	Methyl 2-benzoylbenzoate	606-28-0		$\frac{0.0143 \pm 0}{.0011}$	3	Photoinitiator (22)		Deleted: .

31	Methyl dehydroabietate (<i>methyl (1R,4aS,10aS)-1,4a-dimethyl-7-propan-2-yl-2,3,4,9,10,10a-hexahydrophenanthrene-1-carboxylate</i>)	1235-74-1		Approx. 4.0	8	Cellulose derivative, component of varnishes and printing inks (16)
32	Methyl margarate (<i>methyl heptadecanoate</i>)	1731-92-6		$\frac{0.0064 \pm 0}{.0005}$	2	Intermediate for detergents, emulsifiers, stabilizers, resins, lubricant, plasticizers, defoamer in food-contact coatings (1)
33	Methyl oleate (<i>methyl cis-octadec-9-enoate</i>)	112-62-9	<ul style="list-style-type: none"> low oral toxicity; mildly irritating to skin; TSCA listed (1) 	$\frac{0.0036 \pm 0}{.0004}$	2	Intermediate for detergents, emulsifiers, stabilizers, resins, lubricant, plasticizers, defoamer in food-contact coatings (1)
34	Methyl palmitate (<i>methyl hexadecanoate</i>)	112-39-0	TSCA listed (1)	$\frac{0.0073 \pm 0}{.0005}$	1	Intermediate for detergents, emulsifiers, stabilizers, resins, lubricant, plasticizers, defoamer in food-contact coatings (1)
35	Methyl stearate (<i>methyl octadecanoate</i>)	112-61-8	<ul style="list-style-type: none"> experimental tumorigen; questionable carcinogen; TSCA listed (1) 	$\frac{0.0085 \pm 0}{.0005}$	2	Intermediate for stearin acid detergents, emulsifiers, wetting agents, stabilizers, resins, lubricants, plasticizers, paints (1)

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36	7-Methyl- <i>cis</i> -tetradecen-1-ol-acetate	130-99-6		Approx. 3.0	1		
37	Mesitylaldehyde (2,4,6-trimethylbenzaldehyde)	487-68-3		<u>0.0074±0.0004</u>	1	Product of photoinitiators degradation?	
38	Oleamide (<i>cis</i> -octadec-9-enamide)	301-02-0	<ul style="list-style-type: none"> TSCA listed, LD50 (rat): > 10 g/kg (1) 	<u>0.220±0.004</u>	19	Slip agent for printing inks; processed plastics, coatings and films (1)	PL, (SCF-L-3)
	(<i>trans</i> -octadec-9-enamide)				4		
39	Palmitamide (hexadecanamide)	629-54-9		<u>0.0035±0.0003</u>	2	Slip agent for printing inks; processed plastics, coatings and films (1)	
40	4-Phenyl-benzophenone (<i>phenyl</i> -(4- <i>phenylphenyl</i>)- <i>methanone</i>)	2128-93-0		<u>0.139±0.006</u>	9	UV stabilizer, photoinitiator (14)	
41	1-Phenylbutan-2-one	1007-32-5		Approx. 3.0	2	Solvent	
42	(2-Phenyl-1,3,-dioxolan-4-yl) methylolate			Approx. 3.0	1		
43	Phthalic acid	88-99-3	<ul style="list-style-type: none"> ADI 1.0 mg/kg bw/d (24) 	<u>0.0089±0.0007</u>	9	Component of dyes, food packaging adhesives (1)	PL, (SCF-L 2)
44	<u>Squalene</u> <u>((6E,10E,14E,18E)-2,6,10,15,19,23-hexamethyltetracos-2,6,10,14,18,22-hexaene)</u>	<u>7683-64-9</u>	<ul style="list-style-type: none"> 	<u>0.0070±0.0008</u>	<u>5</u>	<u>Antioxidant (1)</u>	
45	<u>Styrene</u>	<u>100-42-5</u>	<ul style="list-style-type: none"> <u>LD50</u> <u>oral,rat: 5000 mg/kg;</u> <u>intravenous,mouse: 90 mg/kg;</u> <u>intraperitoneal, mouse): 660 mg/kg (1)</u> 	<u>0.0143±0.0011</u>	<u>1</u>	<u>Solvent, raw materials for ink production (1)</u>	<u>PL (SCF-L 4B)</u>

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46	Tetraethyleneglycol diacrylate (2-[2-[2-(2-prop-2-enoyloxyethoxy)ethoxy]ethyl prop-2-enoate)	17831-71-9		Approx. 3.0	1	Component of adhesives, varnishes and photopolymers (1)	(SCF-L 8)	Deleted: 44
47	Toluene-2,4-diisocyanate (2,4-diisocyanato-1-methyl-benzene)	584-84-9	<ul style="list-style-type: none"> LD50 oral, rat: 5.8 g/kg; inh., rat, 4h: 14 mg/kg; Strong irritant to eyes, skin, and tissue; TSCA listed (1) 	0.0611 ± 0.0048	12	Component of polyurethane foams, elastomers, coatings, adhesives, sealants (1)	PL QM(T) = 1 mg/kg (as NCO) (SCF-L 4A)	Deleted: . Deleted: 45
48	Tributoxyethyl phosphate (1-[2-[bis(2-butoxyethoxy)phosphoryloxy]ethoxy]butane)	78-51-3	<ul style="list-style-type: none"> LD50 oral, rat: 3.0 g/kg; intravenous, mouse: 180 mg/kg; skin, rabbit >16 ml/kg; TSCA listed (1) 	0.0344 ± 0.0023	1	Plasticizer for most resins and elastomers; coalescing solvent/plasticizer for acrylic-base polish, gloss paints, adhesives (1)	(SCF-L 6B)	Deleted: . Deleted: 46
49	Tributyl aconitate (1,2,3-Tributyl cis-prop-1-ene-1,2,3-tricarboxylate)	7568-58-3		0.0764 ± 0.0046	14	Plasticizer (23)		Deleted: . Deleted: 47
50	Triphenyl phosphate (diphenoxyphosphoryloxybenzene)	115-IS	<ul style="list-style-type: none"> LD50 oral, rat: 3.5 g/kg; subcut., cat: 100 mg/kg; skin, rabbit: >7.9 g/kg; toxic by inhalation, skin contact slow absorption (1) 	0.0119 ± 0.0009	1	Flame retardant plasticizer for coatings, lacquers; varnishes; solvent for adjuvants in food-contact cross linked polyesters (1,16)	(SCF-L 6B)	Deleted: . Deleted: 48 Deleted: 49

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49	Squalene ((6E,10E,14E,18E)-2,6,10,15,19,23-hexamethyltetracos-2,6,10,14,18,22-hexaene)	7683-64-9		7.0	5	Antic
50	Styrene	100-42-5	LD50 oral,rat: 5000 mg/kg; intravenous,mouse: 90 mg/kg; intraperitoneal, mouse): 660 mg/kg (1)	14.3	1	Solve mate prod

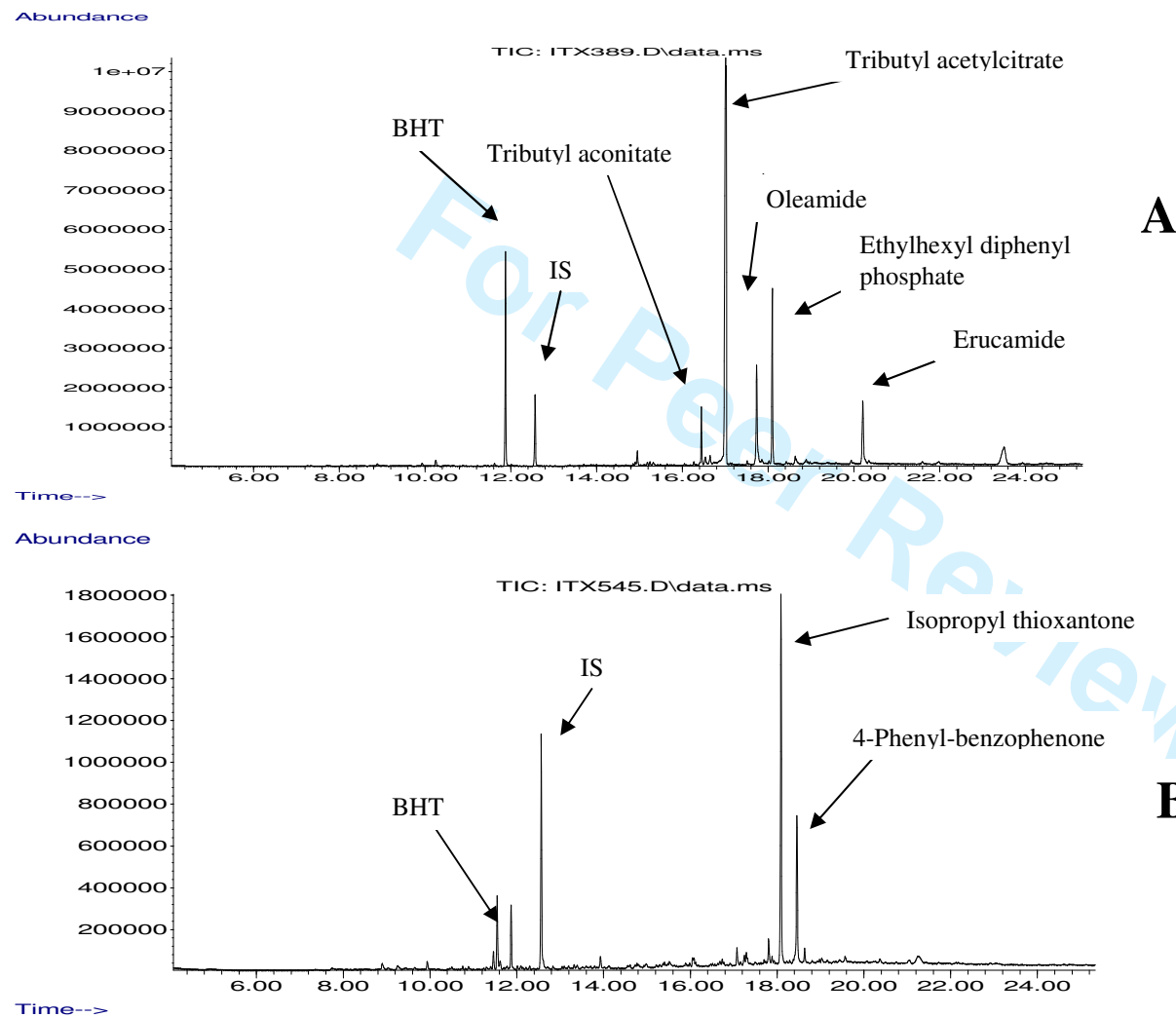


Figure 1. Chromatograms of diethylether extracts from co-extruded polypropylene film with flexography print used for bisquit packaging (A) and laminated paper/Al/LDPE film with UV cured flexography printing used for spice packaging (B) (BHT – buty lhydroxy toluene, IS – internal standard = diethyl phthalate)