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2-Isopropylthioxanthone (2-ITX) in food and food packaging materials on the German market

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2-Isopropylthioxanthone (2-ITX) in food and food packaging materials on the German market

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Abstract

The occurrence of the photo-initiator 2-isopropylthioxanthone (2-ITX) in products on the German market was determined in more than 100 foods packed in cartons as well as in plastic cups and foils. A fast method to detect 2-ITX in food packaging materials was established. In case of positive findings the accompanying foodstuffs were analyzed in a subsequent step using different extraction methods, depending on the fat content of the food. The determination of the photo-initiator was by high performance liquid chromatography with diode array and fluorescence detection (HPLC-DAD/FLD). The recoveries achieved ranged between 94 and 106% for non-fatty (RSD \leq 1.1) and between 80 and 105% for fatty foods (RSD \leq 8.5), respectively. The limit of detection and the limit of quantification of 2-ITX were established as 2 and 5 $\mu\text{g l}^{-1}$ respectively. 2-ITX was detected in 36 out of 137 packages (26%) and significant migration occurred in 75% of the packaging materials tested positive. The amounts of 2-ITX ranged up to a maximum of 357 $\mu\text{g kg}^{-1}$ found in orange juice.

Keywords: *2-isopropylthioxanthone, 2-ITX, 2,4-diethylthioxanthone, 2,4-DTX, HPLC, UV curing inks, photo-initiator, juice, dairy products, baby food*

Introduction

In September 2005 the Italian authorities informed the European Commission by a notification transmitted through the Rapid Alert System for Food and Feed (RASFF) that they found baby milk contaminated with a substance called 2-isopropylthioxanthone (2-ITX, Figure 1, RASFF 2005).

UV curing inks are usually made of multifunctional acrylates, acrylated oligomers and pigments. Photo-initiators like 2-ITX are used to trigger the radical polymerization of the acrylic component of such inks, thus causing the liquid ink film to dry (EFSA 2005).

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3 Compared to solvent-based inks, UV curing seemed to be a good alternative because the
4 packaging material was no longer able to contaminate food with residues of organic solvents
5 from the printing process. But there are new possible contaminants in the packaging
6 material, particularly with regard to acrylates and photo-initiators (Papilloud et al. 2002).
7 Generally 2-ITX can transfer from the packaging into the foods by migration. As intermediate
8 layers of aluminium do not allow ink components to pass through packaging material, it was
9 assumed that in this particular case, 2-ITX contaminated the food by the so-called set-off
10 effect. When the printed material is rolled into spools (e.g. carton base packaging materials)
11 or stacked (e.g. plastic cups), the external layer comes into contact with the internal layer.
12 During this storage 2-ITX is transferred to the surface intended to come into contact with food
13 and finally can migrate into the foodstuff after packaging. At the beginning of this study 2-ITX
14 was detected only in food packed in cartons. It is even unknown if food, that is packed in
15 materials other than cartons can be affected. To assess the contamination of food by this
16 photo-initiator, there is need to investigate food packed in cartons as well as in other
17 packaging materials like plastic cups or foils.
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29 [Insert Figure 1 about here]
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32 According to the opinion No 044/2005 of the German Federal Institute of Risk Assessment
33 (BfR) on the 25th November 2005 (BfR 2005) and the press statement of the European Food
34 Safety Authority (EFSA) on the 9th December 2005 (EFSA 2005), 2-ITX toxicological data
35 was still not sufficient. However, the existing *in vivo* genotoxicity studies do not indicate a
36 genotoxic potential for 2-ITX (EFSA 2005) and at a maximum migration level of 50 µg kg⁻¹
37 food 2-ITX is not likely to pose a health risk (BfR 2005).
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43 Inks applied to the outer surface of food packaging materials are not covered by specific
44 European legislation. One exception is the Commission Directive 93/10/EEC (EEC 1993a).
45 According to this the printed surfaces of regenerated cellulose film must not come into
46 contact with the foodstuffs. However, the only European legislation concerning 2-ITX in food
47 and packaging materials other than regenerated cellulose film are the Framework Regulation
48 (EC) No 1935/2004 (EC 2004), the Regulation (EEC) No 315/93 (EEC 1993b) and the
49 Regulation (EC) No 178/2002 (EC 2002). Pursuant to article 3 of Regulation (EC)
50 No. 1935/2004, materials and articles intended to come into contact with food shall not
51 transfer their constituents in food in quantities which could endanger human health or bring
52 about unacceptable changes in composition or characteristics of foodstuffs. Following
53 article 14 of Regulation (EC) No 178/2002, the food itself must not be placed on the market if
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3 it is injurious to health or unfit for human consumption, whether by extraneous matter or
4 otherwise.
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8 Another document concerning printing inks is the Resolution ResAP(2005)2 on packaging
9 inks applied to the non-food contact surface of food packaging materials and articles
10 intended to come into contact with foodstuffs of the Council of Europe (ResAP 2005). This
11 resolution is not a legal norm, but it is assumed that the general requirements of article 3 of
12 Regulation (EC) No. 1935/2004 for food contact materials are fulfilled if the packaging inks
13 are in accordance with the requirements made.
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19 Manufacturers of food packaging materials are responsible for ensuring that their products
20 comply with the above mentioned regulations and that, from a technological viewpoint, they
21 are suitable for the use for which they are intended. Therefore, the European Printing Ink
22 Association (EuPIA) defined a guideline on printing inks (EUPIA 2006). According to this
23 guideline, if only insufficient toxicological data are available, a substance is acceptable
24 whether its specific migration does not exceed $10 \mu\text{g kg}^{-1}$. If three negative mutagenicity tests
25 as requested by the EFSA-Guidelines are available, as in the case of 2-ITX, the specific
26 migration limit is raised up to $50 \mu\text{g kg}^{-1}$. To assess the migration of 2-ITX into food, several
27 methods have been developed. With respect to UV curing inks and the determination of
28 major acrylates and widely used photo-initiators migrating into simulating solvents, gas
29 chromatography coupled to a mass selective detector (GC-MS) was applied successfully
30 with a recovery rate of 70-100% depending on the simulant used (Papilloud et al. 2002). In
31 milk, yoghurt and fat 2-ITX was determined using accelerated solvent extraction and high
32 performance thin layer chromatography (HPTLC)– fluorescence detection; confirmation of
33 the results was done by HPTLC-mass spectrometry (Morlock et al. 2006). The recoveries of
34 this method ranged from 6-70%, corrected by internal standard up to 70-130% with a limit of
35 detection of $1 \mu\text{g kg}^{-1}$ in butter. A method to determine 2-ITX in fruit juices using pressurized
36 liquid extraction and high performance liquid chromatography (HPLC) coupled to a single
37 quadrupole, ion trap, or triple quadrupole MS detection systems led to recoveries of about
38 70% and detection limits up to $0.05 \mu\text{g l}^{-1}$ (Sagrati et al. 2006).
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54 In this paper, a fast and reliable method to determine 2-ITX in food and food contact
55 materials to enable an effective routine surveillance of commodities on the German market is
56 described. Due to the UV-activity of photo-initiators it is conceivable to determine this
57 compound in a fast and reliable way using its characteristic UV-spectra and fluorescence
58 activity. Therefore, a method based on HPLC coupled to a diode array (DAD) and a
59 fluorescence detector (FLD) for the quantification of 2-ITX and other photo-initiators
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(Figure 1) was developed. Following a step-wise procedure, identification was carried out in food contact materials (multi-layer cartons, plastic cups, and foil) at first and in case of positive findings analysis was carried out in the wrapped foodstuffs also.

Experimental

Chemicals

HPLC grade acetonitrile was purchased from Mallinckrodt Chemicals (Griesheim, Germany) and hexafluoro-2-propanol from Sigma-Aldrich (Taufkirchen, Germany). All further solvents were of gradient grade or distilled prior to use. Dist. water was produced by a Milli-Q water purification system (Millipore, Schwalbach, Germany). The sorbent used for the cleanup Bondesil-PSA (40 µm) came from Varian (Darmstadt, Germany). Analytical standard of 2-isopropylthioxanthone (2-ITX, purity 98%) was provided by IGM Resins (Krefeld, Germany) and 2,4-diethylthioxanthone (2,4-DTX, Figure 1) as internal standard (purity 98%) by Sigma-Aldrich (Taufkirchen, Germany). All further chemicals were minimum of analytical quality.

For preparation of 0.1 M citrate phosphate buffer (pH 6.0) 21 g citric acid monohydrate and 14.2 g disodium hydrogen phosphate are dissolved in approximately 900 ml water. After adjusting to pH 6.0 with concentrated sodium hydroxide, the solution is diluted up to 1000 ml with water. Solutions of 2-ITX and 2,4-DTX were prepared in acetonitrile and stored at 4 °C in the dark.

Samples

137 samples of fatty and non-fatty food were collected since October 2005 on the German market at retail randomly and partly direct from the food manufacturer. Following a stepwise procedure the food contact materials were tested for 2-ITX at first while storing the homogenized fillings at -18 °C for further analysis in case of positive findings.

Sample preparation

Food contact material. After separation of the food contact material from the filling approximately 4 square centimeters of the printed packaging material are cut into small pieces and extracted with 1 ml hexafluoro-2-propanol (EU DG XII Research Programme AIR 941025 (1994-1997) 1997) in an ultrasonic bath for 45 min. Then 4 ml ethanol are added and the mixture is shaken intensively for 1 min. The precipitate is removed by filtration prior to HPLC analysis.

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3 If 2,4-DTX was found originally in food packaging materials, no internal standard was added
4 during the sample preparation (as described below) and both photoinitiators were determined
5 by external standard calculation.
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10 *Non-fatty foods (e.g. juices, tomato puree).* Sample preparation for non-fatty foods was
11 based on the QuEChERS-method (Anastassiades et al. 2003). To 10 g of the homogenized
12 sample material 10 ml acetonitrile was added. Extraction was carried out by shaking
13 intensively for 1 min. Then 4 g magnesium sulfate and 1 g sodium chloride was added and
14 the mixture was shaken again intensively for 1 min. After addition of the internal standard
15 2,4-diethylthioxanthone and gently shaking for 30 s the mixture was centrifuged for 5 min at
16 3000 rpm. An aliquot (8 ml) of the supernatant was mixed with 1.2 g anhydrous magnesium
17 sulfate and 200 mg PSA. After shaking intensively for 30 s, the mixture was centrifuged for
18 1 min at 5300 rpm. The supernatant was directly subjected to HPLC analysis.
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26 *Fatty foods (e.g. yogurt, milk, sausage).* To approximately 5 g of homogenized fatty food 5 ml
27 0.1 M buffer solution (pH 6.0) was added and the sample was extracted by shaking
28 intensively for 1 min. After addition of the internal standard and 30 ml acetonitrile the mixture
29 was shaken for 10 min and quantitatively filtrated through a filter paper. Flask and filter paper
30 were rinsed with 10 ml acetonitrile/water (3/1 v/v) respectively. After addition of 1.5 g sodium
31 chloride and intensively shaking for 30 s the filtrate was mixed with 20 ml *tert.*-butyl methyl
32 ether/isohehexane (80/20 v/v) before gently shaking again. The lower aqueous phase was
33 discarded. The organic layer was washed twice with 20 ml water. After addition of 10 ml
34 *tert.*-butyl methyl ether/isohehexane (50/50 v/v) and, if necessary, separation of the aqueous
35 phase, the organic layer was dried over anhydrous sodium sulfate. The solution was
36 evaporated to dryness under vacuum. Finally, the residue was dissolved in 1 ml acetonitrile
37 and directly subjected to HPLC analysis.
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47 *High performance liquid chromatography with diode array and fluorescence detection*
48 *(HPLC-DAD/FLD)*
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50 Analysis was performed on a Agilent 1100 high performance liquid chromatograph (Agilent,
51 Waldbronn, Germany) equipped with a LC-PAH Supelcosil (250 mm; 4.6 mm ID; 5 µm)
52 column (Sigma-Aldrich, Taufkirchen, Germany) coupled to a diode array (260 nm; spectra
53 recorded from 200 up to 500 nm) and a fluorescence detector (excitation 272 nm/emission
54 440 nm) connected in series. The system was run at 40 °C (stop time 10 min) in isocratic
55 mode (dist. water/acetonitrile 15/85 v/v) with a flow rate of 1 ml min⁻¹ and an injection volume
56 set to 10 µl.
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3 The limit of detection and quantitation (2-ITX) as well as the linearity range (2-ITX and 2,4-
4 DTX) were determined by HPLC-FLD analysis of solutions of the photo-initiators in
5 acetonitrile in absence of matrix interferences according to DIN standard 32645 (DIN 1994).
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8 9 *Spiking procedure*

10 Spiking of homogenized blank samples was performed by adding 2-ITX standard solution
11 directly into the matrix. The spiked matrices were shaken briefly and left to stand quite a time
12 (2 min) before extraction to enable the photo-initiator to distribute. For non-atty foods the
13 recovery tests were conducted on blank homogenized orange and vegetable juice by spiking
14 (5 times each matrix and level) with 0.05 and 0.5 mg kg⁻¹ of 2-ITX. For fatty foods blank
15 homogenized milk and oil was spiked (6 times each matrix and level) with 0.1 mg kg⁻¹ of
16 2-ITX (Table 1).
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24 **Results and Discussion**

25 26 27 *Validation of the method*

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31 *Limits of detection and quantitation.* The method for the determination of 2-ITX with HPLC-
32 DAD/FLD (Figure 2) in acetonitrile was linear in the range of 6 up to 120 µg L⁻¹ (Mandel test
33 of linearity) for the FLD-signal with a correlation coefficient of 0.9995. Due to the similar
34 molecular structure and chemical properties 2,4-DTX was used as internal standard (linearity
35 5 up to 100 µg L⁻¹). According the DIN standard 32645 (DIN 1994), the limit of detection and
36 quantification of 2-ITX by fluorescence detection were determined to 2 and 5 µg L⁻¹ in
37 acetonitrile, respectively. Confirmation of 2-ITX by its UV-spectrum could be achieved above
38 12 µg L⁻¹. The results of the analyzed samples were only accepted, if the presence of the
39 photo-initiator was approved by the characteristic UV spectrum (Figure 2).
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47 [Insert Figure 2 about here]
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50 *Recoveries.* For non-fatty foods the recovery rates were above 85% (data not shown). By
51 application of the internal standard 2,4-DTX the recovery was increased to 94 - 106% with a
52 relative standard deviation (RSD) below 1.1% (Table I). For fatty foods, there was partial
53 interference with the signal of the internal standard. Therefore, determination of 2-ITX in fatty
54 foods was carried out by external calibration (Table I).
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60 [Insert Table I about here]

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3 [Insert Figure 3 about here]
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6 *Analysis of real samples*

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8 Samples from the German market (137) were analyzed for 2-ITX by the method reported
9 here (Figure 3). Most of the samples were packed in multi-layer cartons and plastic cups, but
10 also in plastic foil like butter and sausages. The results of the foodstuffs analyzed are shown
11 in Table II. Of all the packages analyzed, 2-ITX was detected in 36 samples (26%, Figure 4).
12 In 27 of 36 positive tested food packaging materials (75%), significant migration of 2-ITX into
13 the food could be observed with the highest amounts of 2-ITX found in orange juice
14 (357 $\mu\text{g kg}^{-1}$) and baby food (208 $\mu\text{g kg}^{-1}$). In 13 samples (10%) the recommended migration
15 level of 50 $\mu\text{g kg}^{-1}$ was exceeded.
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22 [Insert Figure 4 about here]

23 [Insert Table II about here]
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28 Furthermore, the migration of 2-ITX into food was not only limited to printed multilayer
29 cartons, but also occurs in food packed in printed plastic cups and foils (Figure 4). However,
30 in these fillings analyzed the migration levels were always below 50 $\mu\text{g kg}^{-1}$ food, at which
31 level 2-ITX is not likely to pose a health risk (BfR 2005).
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35 The latest results showed that the internal standard 2,4-DTX used in this study can be found
36 in food packaging materials also, e.g. in plastic cups of yoghurt. The concentration of this
37 contaminant in yoghurts was determined to be in the range of 15 to 48 $\mu\text{g kg}^{-1}$ without
38 addition of the internal standard as described in the experimental section. Like 2-ITX,
39 complete toxicological data concerning 2,4-DTX is not available so far.
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45 **Conclusion**

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49 With the method presented above, it is possible to determine 2-ITX as well as 2,4-DTX in
50 various food packaging materials and foods in a quick and reliable way. By the strategy
51 chosen to analyze at first the wrappings - and only in case of positive findings - the
52 corresponding fillings, a rapid throughput on a routine basis could be achieved. The recovery
53 rates of 2-ITX in food ranged between 94 and 105% with a relative standard deviation
54 between 0.4 and 8.5%. In practice, the limit of quantification for 2-ITX was below 50 $\mu\text{g kg}^{-1}$
55 and thus allowed effective control of the maximum migration level of 50 $\mu\text{g kg}^{-1}$
56 recommended by the German Federal Institute of Risk Assessment (BfR 2005).
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3 Significant migration of 2-ITX from packaging materials into foodstuff could be detected in 20
4 % of the samples from the German market investigated, particularly up to 357 $\mu\text{g kg}^{-1}$ in
5 orange juice and 208 $\mu\text{g kg}^{-1}$ in baby food. The occurrence of 2-ITX and 2,4-DTX in various
6 food packaging materials, not being limited to multilayer cartons, should guide the industry to
7 apply other, less-migrating photo-initiators. Apart from the efforts of the industry, the
8 implementation of legislative standards for good manufacturing practice with a positive list for
9 printing inks and maximum migration limits especially for substances with incomplete
10 toxicological assessment is inevitable.
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17 **Acknowledgement**

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19 We wish to thank Ms Pechstein, Ms Drewnik and Ms Moser for the skilful technical
20 assistance, Mr. Dr. Weißhaar and Mr. Altkofer for valuable discussions and IGM resins for
21 providing 2-ITX.
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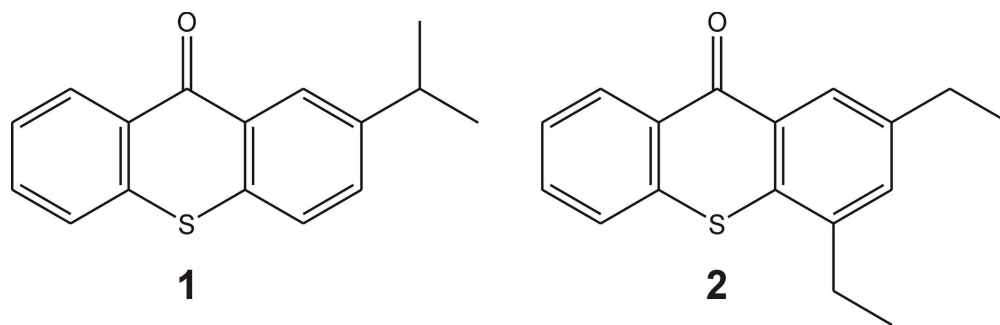


Figure 1. Chemical structures of the photo-initiators 2- ITX (**1**), and 2,4-DTX (**2**).
113x35mm (600 x 600 DPI)

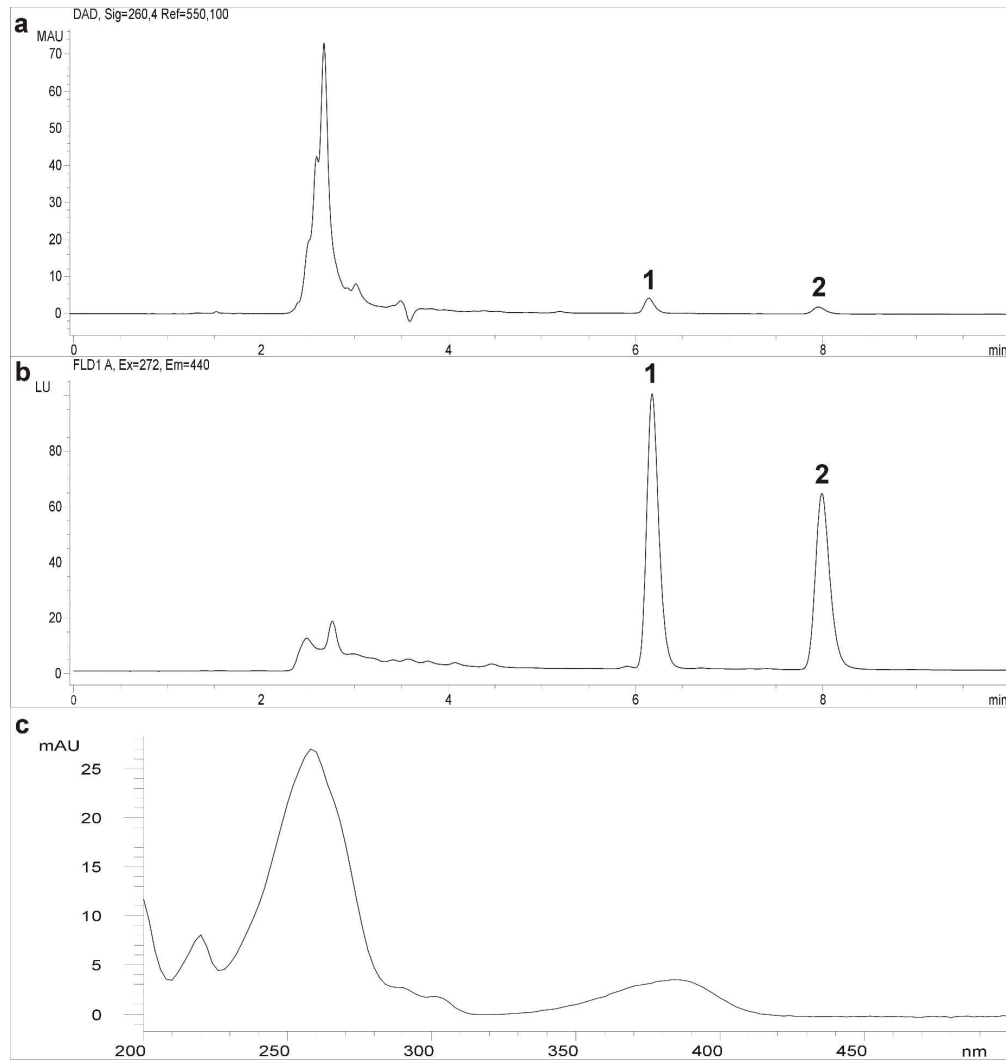


Figure 2. HPLC separation of an olive oil extract spiked with about $300 \mu\text{g kg}^{-1}$ 2-ITX (1) and 2,4-DTX (2). The DAD (a) and FLD signals (b) are shown as well as the characteristic UV spectrum (c) of 2-ITX.

156x164mm (600 x 600 DPI)



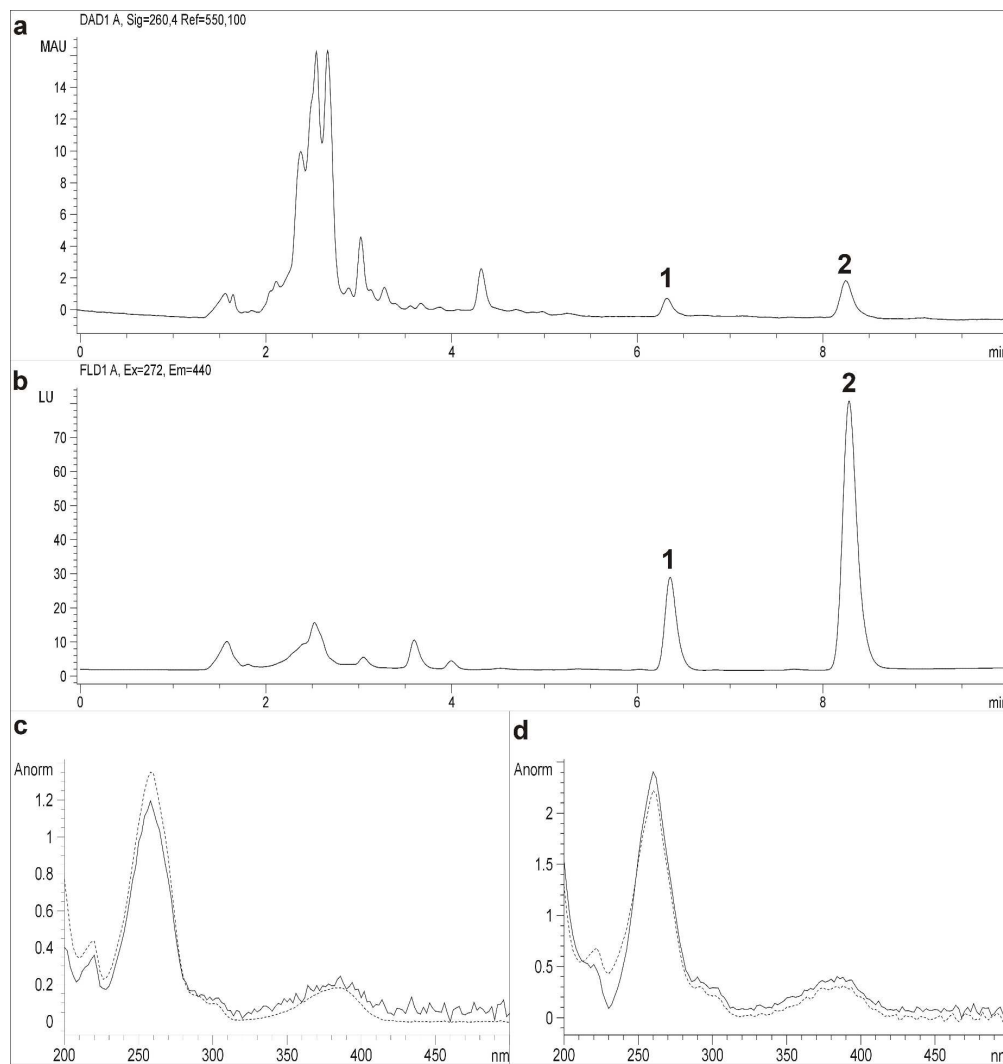


Figure 3. HPLC separation of a yoghurt from the German market with an 2-ITX content determined to $18 \mu\text{g kg}^{-1}$. The DAD (a) and FLD signals (b) are shown as well as the correlation of the UV spectra recorded (continuous line) with the spectra of 2-ITX (c) and 2,4-DTX (d) of standard solutions recorded (dotted line).
155x164mm (600 x 600 DPI)

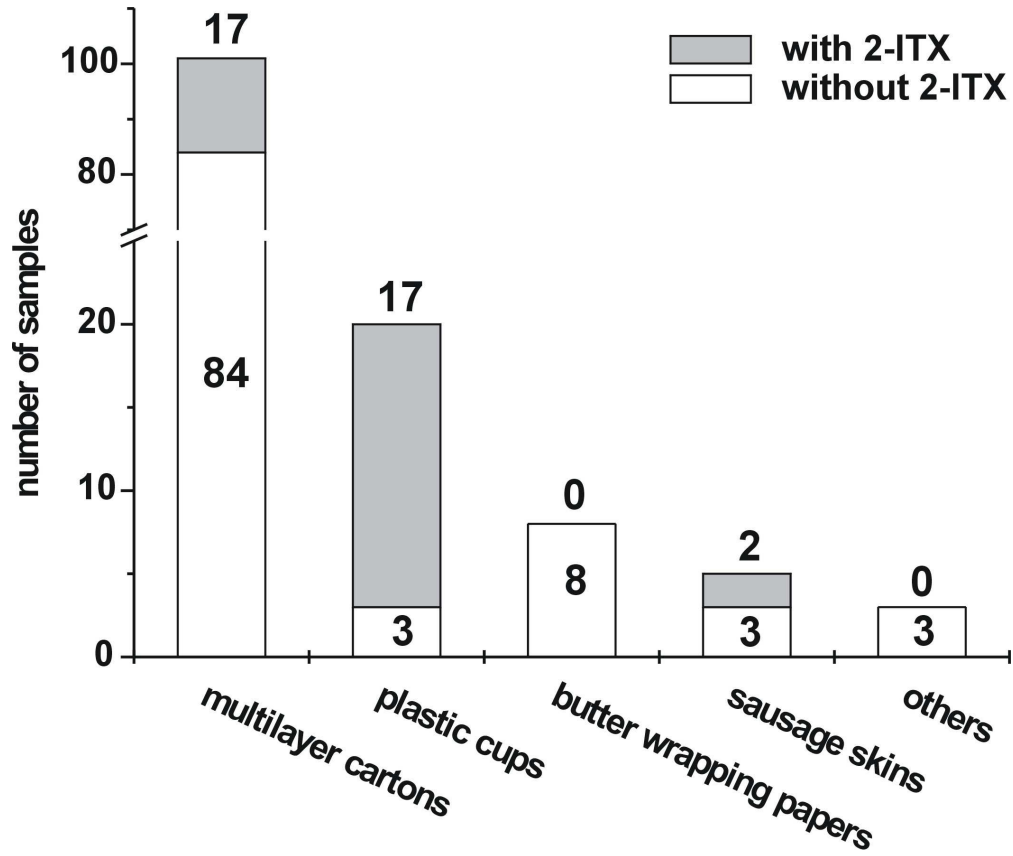


Figure 4. Overview about the 2-ITX detected in food packaging materials.
89x75mm (600 x 600 DPI)

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Table I. Recoveries rates and relative standard deviations (RSDs) from food samples spiked with 2-ITX.

food	fortification level [mg kg ⁻¹]	replicates	recovery rates [%]	mean [%]	RSD [%]
orange juice	0.05	5	95, 96, 96, 95, 95	95	0.4
	0.50	5	104, 104, 105, 103, 104	104	0.6
vegetable juice	0.05	5	94, 97, 95, 97, 97	96	1.1
	0.50	5	104, 105, 106, 105, 106	105	0.8
milk	0.10	6	80, 105, 93, 94, 100, 94	94	8.5
oil	0.10	6	95, 95, 90, 94, 95, 94	94	2.0

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