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Publication date:
2012

Document Version
Publisher's PDF, also known as Version of record

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Extraction method for the collection of volatile organic compounds in paper and cardboard food packaging materials

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Introduction **The migration of potentially harmful substances from paper and cardboard into foods is far less studied than migration for other food contact materials. Two important migration mechanisms of substances from cellulose-based packaging is mass transfer in air and direct contact. In order to toxicologically screen for, and asses for, substances present in paper- and cardboard food packaging materials, we developed an efficient and reproducible extraction strategy to collect VOCs from paper- and cardboard.**

Materials and methods

Virgin paper samples were spiked with a standard mix to a final concentration of 100ng/dm^2. The paper samples were then placed in a 2L glass bottle in an a 60 C oven, and volatiles were sampled, with a sampling time of 60 min. The inlet air was cleaned through a carbon filter outside the oven, and all connecting tubing was made of the chemically inert material Teflon.

For the collection of VOCs from paper and board, double-bed thermal desorption tubes containing in total 300 mg (200+100 mg) MPPO (Tenax® TA 60/80) was used. A GilAir 5 pump, set at 70 mL/min, was used to drive the VOCs through the desorption tube. In order to optimize collection, the tubes were placed outside the oven and cooled with dry ice to between -15 C to -20 C.

The analytes were extracted from the MPPO resin by the addition of 1 mL ethanol and vigorously shaken for 30 min., before injection to an Agilent 6890/5973N MSD GC-MS-MS system. All compounds were separated within 10 min.

Table 1. VOCs spiked on paper matrix and analysed collected on MPPO resin. Analytes were then extracted by ethanol and analysed on GC/MS. For the two analytes with the lowest boiling point, the second portion of resin was also included in the calculation of recovery.

Compound	CAS number	Mw (g/mol)	Boiling point (760 Torr)	Vapour pressure (Torr, 25 C)	Recovery (%)	Standard deviation
1,2-dichlorbenzene	95-50-1	147	181	1	70	4
o-Xylene	95-47-6	106	146	6	65	0
Styrene	100-42-5	104	145	6	73	6
m-Xylene	108-38-3	106	141	8	74	6
p-Xylene	106-42-3	106	140	8	72	5
Ethylbenzene	100-41-4	106	136	9	72	4
Chlorbenzene	108-90-7	113	132	11	73	5
Tetrachlorethene	127-18-4	166	119	19	75	6
Toluene	108-88-3	92	111	28	76	7
Cis-1,2-dichlorethylene	156-59-2	97	48	333	83	0
Trans-1,2-dichlorethylene	156-60-5	97	48	333	73	8

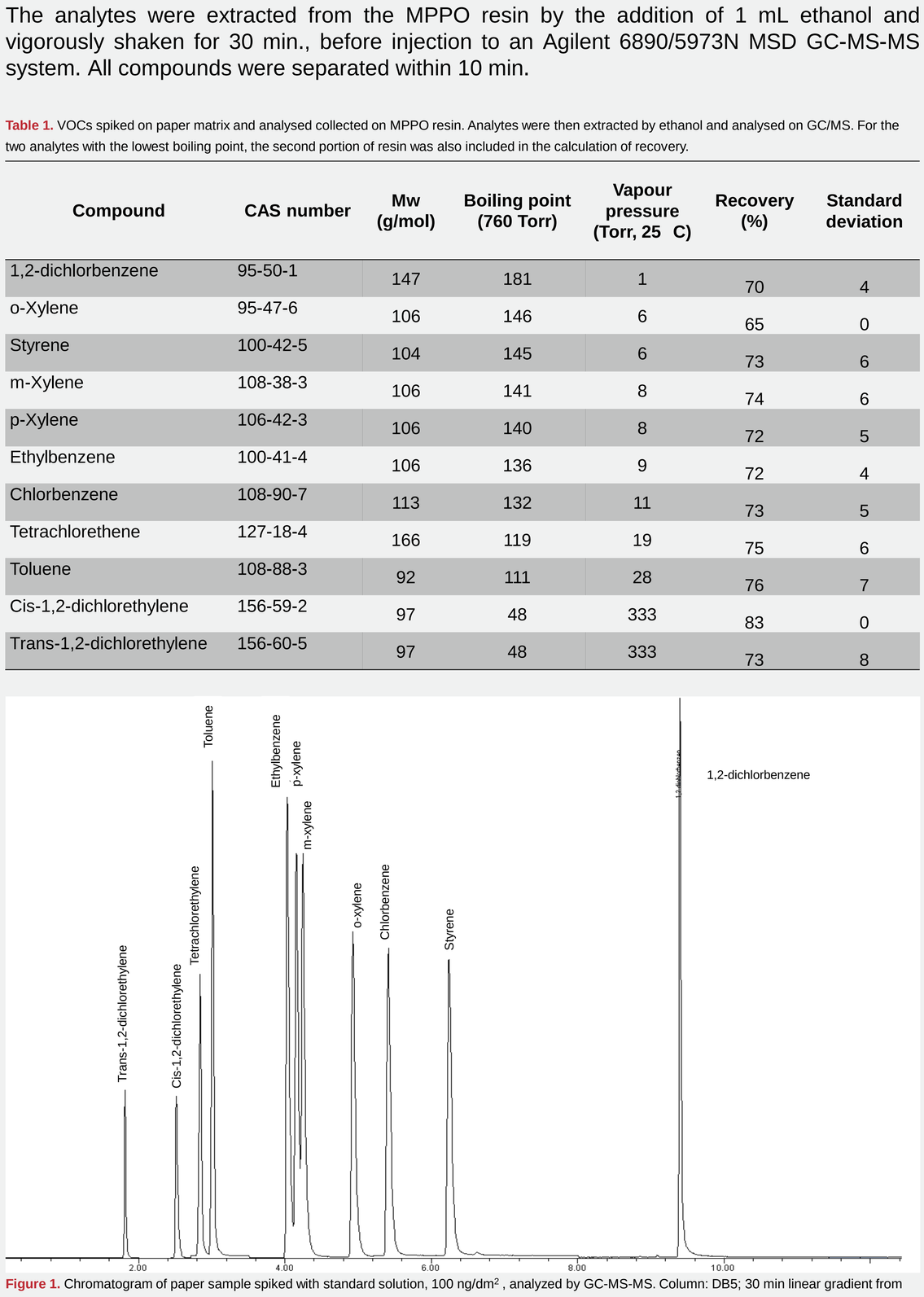

Figure 1. Chromatogram of paper sample spiked with standard solution, 100 ng/dm^2 , analyzed by GC-MS-MS. Column: DB5; 30 min linear gradient from 40 C to 200 C; injection volume: 1 µl; He flow: 40 cm/sec.

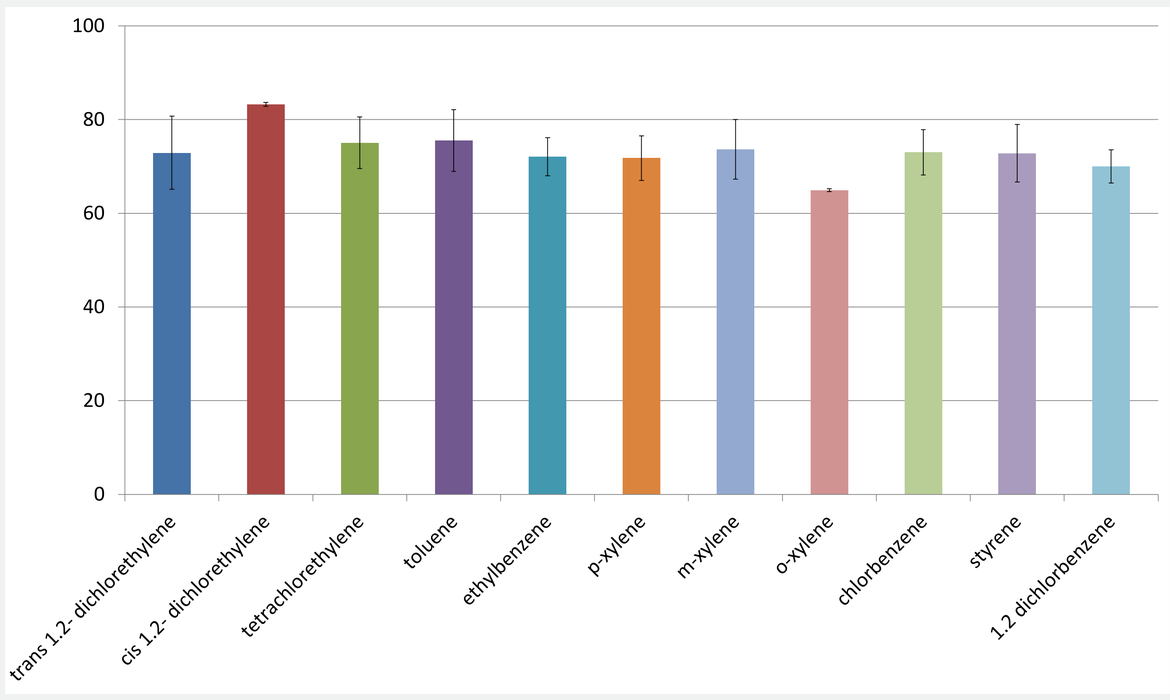

Figure 2. Mean recovery of analytes in spiked paper (100 ng/dm^2) extracted from MPPO resin with ethanol. The standard deviation is indicated as an error bar for each compound.

Results and discussion

The model compounds had boiling points within a range of 48 C to 181 C, see Table 1. A representative chromatogram for paper spiked with 100 ng/dm^2 of the standard mixture can be seen in Figure 1.

Recovery for the analytes ranged from 65-83 %, see Figure 2, with a mean value of 73% and mean relative standard deviation of 5%. Due to breakthrough to the second fraction for the two most volatile compounds, the second fraction was also included in the calculation of recovery.

Most previous studies investigating migrating compounds have mainly been focusing on non-volatile compounds[1,2,3]. The method was optimized for amount of MPPO resin, number of resin beds and flow velocity. Highest achievable flow without substantial breakthrough was 70 ml/min.

The temperature of sample and resin for optimum adhesion of volatiles to MPPO were also investigated. The MPPO tubes needed to be cooled down to below -15 C to collect the most volatile analytes. The temperature of the oven heating the sample was optimised to 60 C, as low temperature, 40-50 C, decreased recoveries and high temperatures, 80-100 C, gave high breakthrough.

Future studies and perspectives

We are now in the process of developing an extraction method for VOCs, semi-VOCs and non-VOCs in large amounts of paper and board matrix (30-90dm^2), by using both the described method and a boiling ethanol reflux system. A pooled extract is made to produce more comprehensive extracts of paper matrices for toxicological testing.

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