Data Article

Data on the optimisation of GC-MS/MS method for the simultaneous determination of compounds from food contact material

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ARTICLE INFO

ABSTRACT

Food contact materials (FCM) made of plastic materials contain various additives, e.g. plasticisers, UV-stabilisers, preservatives, antioxidants, etc. These compounds can migrate from the material to the food and display adverse health effects in consumers. Inertness of FCM is established by migration testing with appropriate food simulants [1]. A GC-MS/MS method for the simultaneous determination of several different groups of additives to plastics has been developed to perform a migration testing and to determine these compounds in real samples, as described in the research publication "Development of a SPME-GC-MS/MS method for the determination of some contaminants from food contact material in beverages" [2]. Here, we present the data on the optimisation of GC-MS/MS parameters: GC column and temperature programme choice, MS/MS parameters optimisation, and choice of internal standard. Subsequently, SPME parameters were also optimised as described in [2].

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Specifications Table

Subject	Chemistry
Specific subject area	Analytical Chemistry. Method Optimization.
Type of data	Figure, Scheme
How data were acquired	GC-MS, GC-MS/MS, GC-HRMS
Data format	Raw
Parameters for data collection	Different GC columns and temperature programmes. Mass spectra in total ion current (TIC) mode, product ion mode, selected reaction monitoring mode.
Description of data collection	Chromatograms and mass spectra were recorded during GC method optimisation and MS/MS data optimisation. Optimisation was done one-factor-at-a-time. Raw data were analysed to extract the needed information. Descriptive analysed data shown.
Data source location	City: Novo mesto & Ljubljana
	Country: Slovenia
Data accessibility	With the article (Supplementary Material, SM1-SM5; Figs. 5 and 6 are raw spectra)
Related research article	Luka Žnideršič, Anita Mlakar, Helena Prosen.
	Development of a SPME-GC-MS/MS method for the determination of some contaminants from food contact material in beverages.
	Food and Chemical Toxicology 134 (2019) 110829 https://doi.org/10.1016/j.fct.2019.110829

Value of the Data

- The data provide the optimisation strategy and insights for method development in case of chemically widely differing groups of analytes.
- The data are of benefit for analytical chemists developing GC-MS and GC-MS/MS methods.
- The data show the possibility of GC analysis of parabens without previous derivatisation by choosing an appropriate GC column.
- It is shown that deuterated internal standards may not always be the best option in GC-MS analysis.

1. Data

In the first part, data related to GC method optimisation are shown: a chromatogram recorded for the analysed compounds on the polar GC column DB-624 (Fig. 1 & Suppl. Mat. SM1); and comparison of the chromatographic peak shape for a polar analyte on two less polar GC columns — HP-5 or HP-5MS UI (Fig. 2; see also Fig. 1 in Ref. [2]).
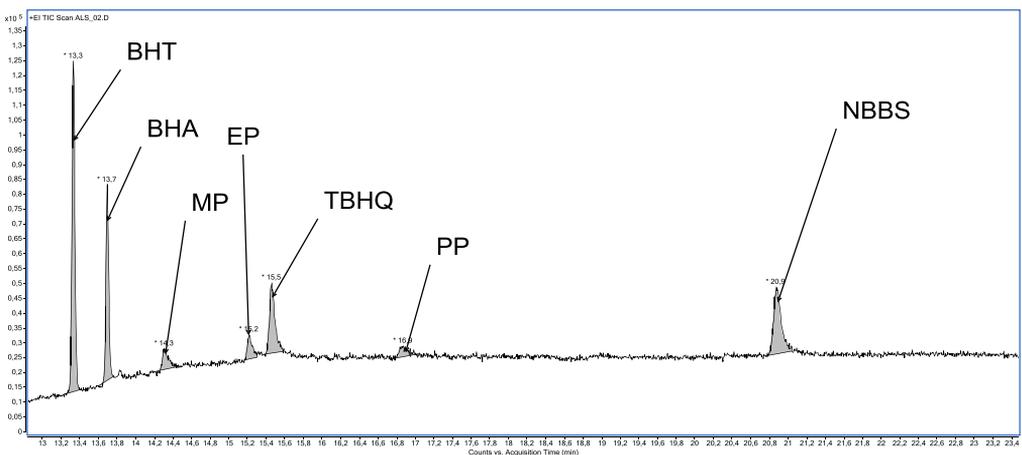
Fig. 1. Chromatogram of standard mix of analytes (0.1 mg/mL) on GC column DB-624.
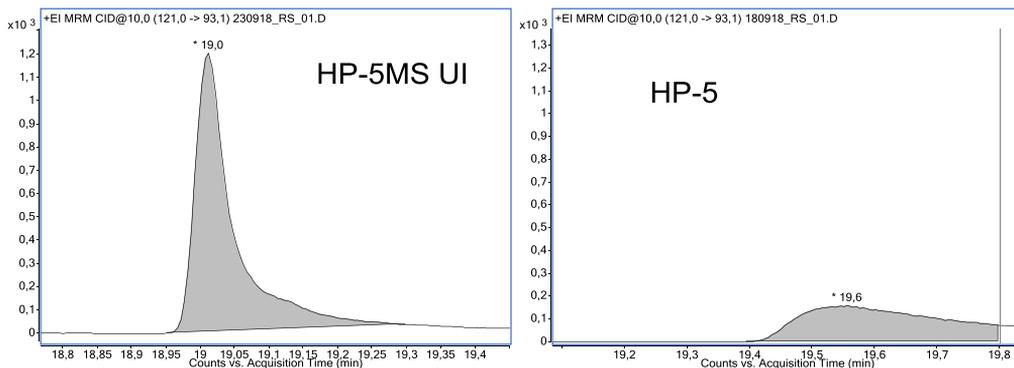
Fig. 2. Comparison of chromatographic peaks for methyl paraben on GC column HP-5MS UI and equivalent GC column HP-5.

In the second part, data on MS/MS optimisation are shown: mass or tandem mass spectra for the analyte methyl paraben recorded in total ion current (TIC) mode, product ion (PI) mode, and selected reaction monitoring (SRM) mode are shown in Fig. 3; a chromatogram of a standard mix of analytes in TIC, PI, and SRM mode is shown in Fig. 4. For the raw data, see Suppl. Mat. SM2, SM3, and SM4.

The third part of data show the mass spectrum of internal standard phenyl dimethoxyphosphate (Fig. 5) obtained by complete transformation of precursor compound phenyl dichlorophosphate, PDCP, in methanol (Scheme 1), which was confirmed by the HRMS mass spectrum (Fig. 6). A comparison of chromatographic peak shape of the chosen internal standard and isotopically labelled internal standard deuterated 2,6-di-*tert*-butyl-4-methyl-phenol, dBHT (Fig. 7 & Suppl. Mat. SM5).
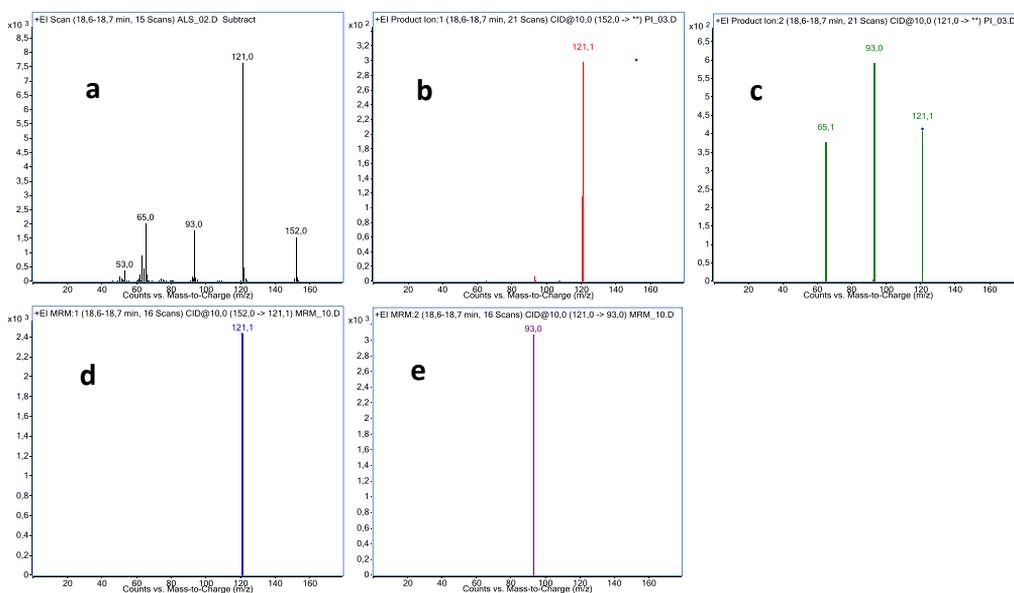
Fig. 3. Mass spectra for methyl paraben during MS/MS method optimisation. **a** − total ion current mode; **b** − product ion mode, m/z 152 → 20-200; **c** − product ion mode, m/z 121 → 20-200; **d** − selected reaction monitoring, transition m/z 121 → 93 (quantifier); **e** − selected reaction monitoring, transition m/z 152 → 121 (qualifier).
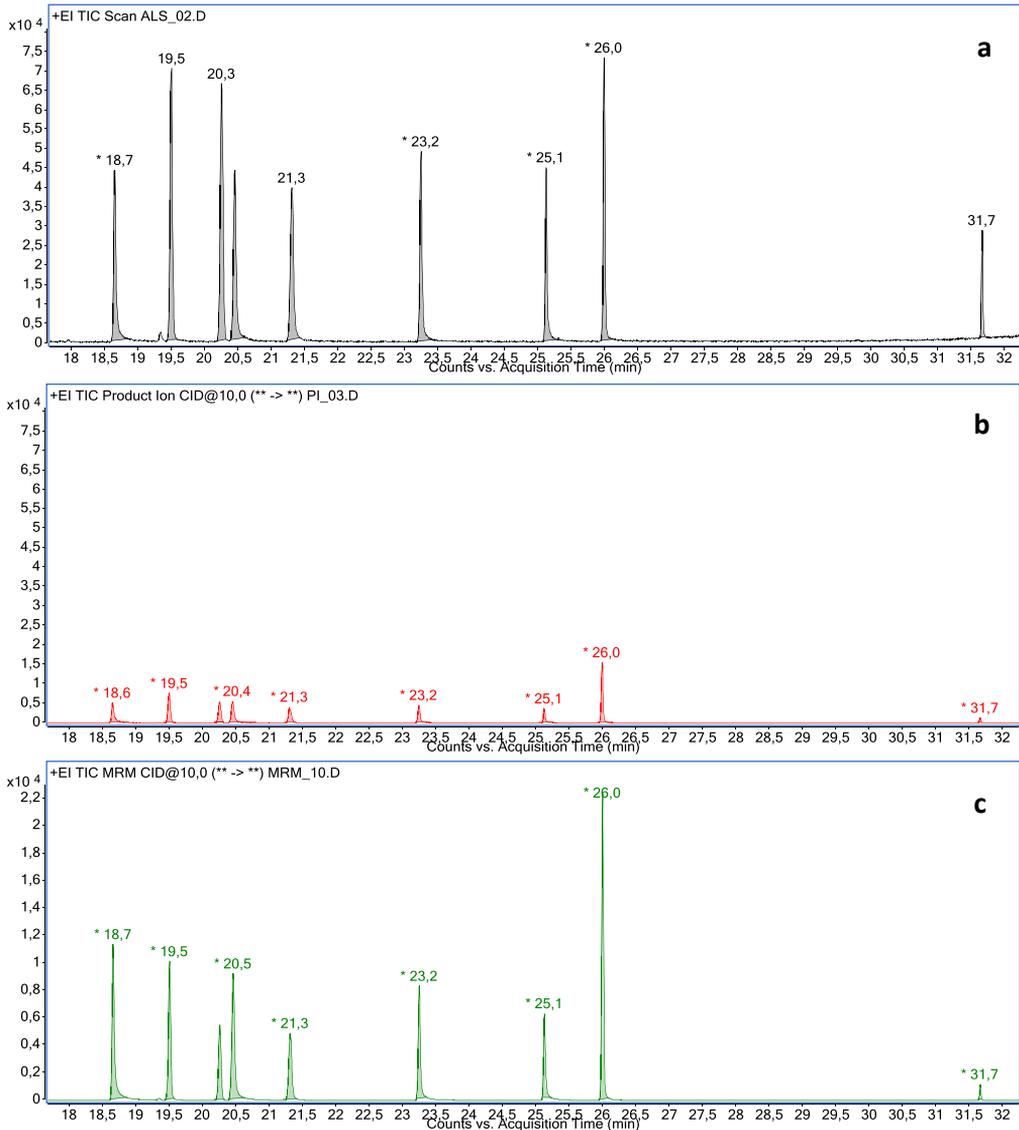
Fig. 4. Chromatograms of standard mix of analytes (0.1 mg/mL). **a** − TIC mode; **b** − PI mode; **c** − SRM mode at optimised collision energies. Analytes: MP (18.7 min), BHA (19.5 min), BHT (20.3 min), EP (20.5 min), TBHQ (21.3 min), PP (23.2 min), BP (25.1 min), NBBS (26.0 min), TBEP (31.7 min).

2. Experimental design, materials, and methods

2.1. Compounds

Methyl paraben (MP, >99%), ethyl paraben (EP, >99%), propyl paraben (PP, >99%), butyl paraben (BP, >99%), *tert*-butylhydroquinone (TBHQ, >97%), *N*-butylbenzenesulfonamide (NBBS, >99%), tris(2-butoxyethyl)phosphate (TBEP, >94%), 2,6-di-*tert*-butyl-4-methyl-phenol (BHT, >99%), 3-*tert*-butyl-4-hydroxyanisole (BHA, >99%), phenyl dichlorophosphate (PDCP, >95%), 2,6-di(*tert*-butyl-d$_9$)-4-
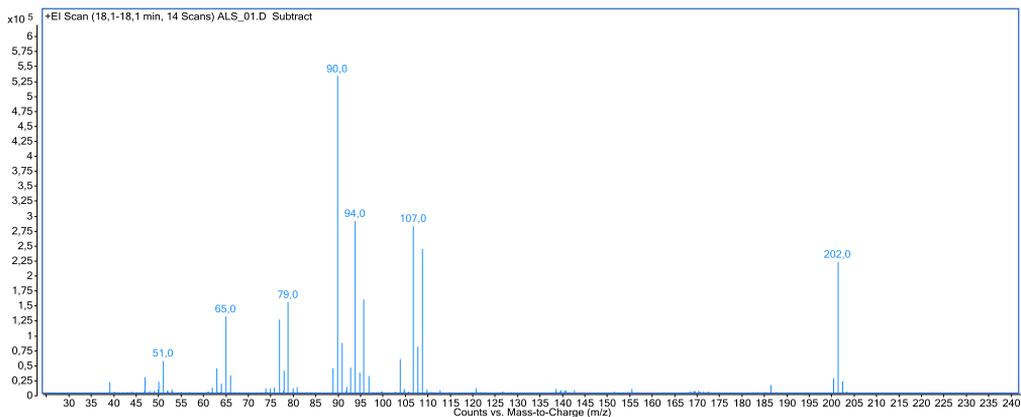
Fig. 5. Mass spectrum (EI ionisation) of internal standard phenyl dimethoxyphosphate obtained by dissolving phenyl dichlorophosphate (PDCP, 1 mg/mL) in methanol (raw spectrum).
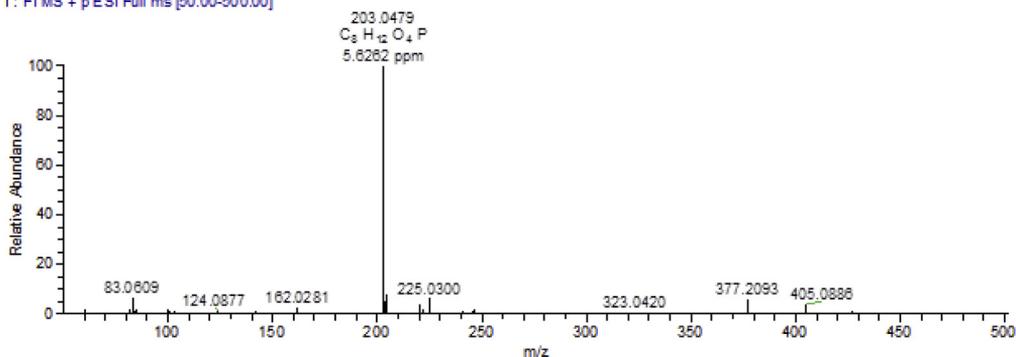
Fig. 6. HRMS mass spectrum of phenyl dimethoxyphosphate (0.01 mg/mL) in ultrapure water (raw spectrum).
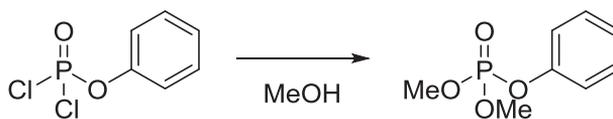
Scheme 1. Transformation of phenyl dichlorophosphate (PDCP) to phenyl dimethoxyphosphate in methanol.
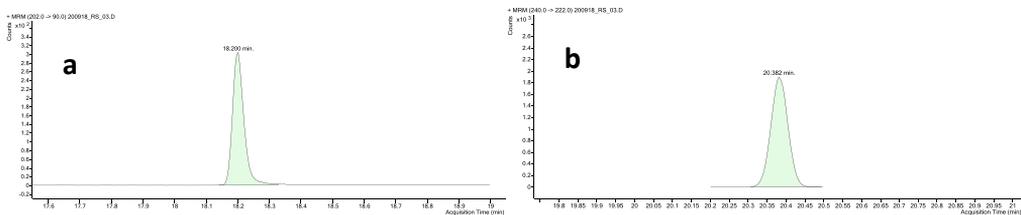
Fig. 7. Comparison of chromatographic peak shapes of phenyl dimethoxyphosphate (**a**) and deuterated BHT (**b**) on HP-5MS UI column.

methyl(phenol-3,5,*O*-d$_3$) (dBHT, >98%). These compounds, except PDCP and dBHT, can be found in food contact material [1].

2.2. GC columns

DB-624, 6%-cyanopropyl-phenyl- and 94%-polydimethylsiloxane, 30 m × 0.25 mm i.d., film thickness 1.4 μm (Agilent Technologies, Santa Clara, CA, USA).

HP-5, 5%-phenyl-methylpolysiloxane, 30 m × 0.25 mm i.d., film thickness 0.25 μm (Agilent Technologies, Santa Clara, CA, USA).

HP-5MS UI, 5%-phenyl-methylpolysiloxane, 30 m × 0.25 mm i.d., film thickness 0.25 μm (Agilent Technologies, Santa Clara, CA, USA).

2.3. GC-MS/MS method

GC-MS/MS method was developed by using a gas chromatograph 7890A and tandem mass spectrometer 7000B (both Agilent Technologies, Santa Clara, CA, USA), equipped with a multi-purpose autosampler MPS (Gerstel, Müllheim an der Ruhr, Germany).

Temperature programme: initial temperature 50 °C (4 min), followed by temperature ramp of 8 °C/min to intermediate temperature 150 °C (5 min), and then ramp of 12 °C/min to final temperature 280 °C (5 min). Carrier gas was helium (>99.999%) with flow 1.4 mL/min. Injection was at 250 °C in splitless mode. Transfer line temperature was 280 °C; ion source temperature was 230 °C; and quadrupoles temperatures were 150 °C. Ionisation was electron impact at 70 eV. Total ion chromatograms were recorded in *m/z* range 35-700. Compounds were identified by spectral comparison using mass spectral library NIST (version 2.0, updated 19.05.2011).

To process and analyse the data, programmes Gerstel Maestro, Agilent Mass Hunter Qualitative Analysis, and Mass Hunter Quantitative Analysis B.08.00 were used. SRM method was developed and optimised by using Agilent Design SRM experiments assistant.

2.4. HRMS method

High resolution mass spectra were recorded on mass spectrometer LTQ Orbitrap XL (Thermo Fisher Scientific Company, Villebon, France) equipped with heated electrospray ionisation (HESI-II). Mass spectra were recorded in *m/z* range 100-800 following positive ESI. Aqueous solutions were directly injected into HRMS instrument.

Acknowledgments

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.105060.

References

[1] Commission Regulation (EU) No 10/2011 of 14 January 2011 on Plastic Materials and Articles Intended to Come into Contact with Food, n.d. 89. https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=celex:32011R0010.

[2] L. Žnideršič, A. Mlakar, H. Prosen, Development of a SPME-GC-MS/MS method for the determination of some contaminants from food contact material in beverages, Food Chem. Toxicol. 134 (2019) 110829, https://doi.org/10.1016/j.fct.2019.110829.