The reported method was developed for the determination of possible migrants in paperboard packaging material by usage of solid phase micro extraction and gas chromatography coupled to mass spectrometry. The method can be used for monitoring the content of unwanted compounds in paperboard intended for use in the contact with food. During method development were investigated all important parameters in order to reach the best method performance for the group of 19 important compounds covering the representatives of phthalates, photoinitiators, phenols, and off-flavors deriving from the degradation of paperboard components including printing, coating and adhesives. The final method was successfully validated as a quantitative screening method for a group of 12 target contaminants. The final method was applied in a small survey covering paperboard samples of various quality including both virgin and recycled paperboard.

Method

Sample Preparation

- Cut paperboard into small pieces (2 mm x 2 mm)
- Add 6 ml 13% CH3OH in H2O
- Automatic SPME
- GC-MS/MS

SPME and Instrumental analysis

Automated SPME

- Fiber: 100 µm PDMS (polydimethylsiloxane)
- Extraction time and temperature: 45 min at 65°C
- Desorption time and temperature: 7 min at 270°C
- Conditioning fiber: 20 min
- Swirling the vial: all the time

Instrumentation

- System: Thermo Scientific™ TSQ 8000 Triple Stage Quadrupole MS coupled to Trace 1310 GC equipped with TriPlus RSH Autosampler
- Column: TG – 5 SiMS(0.25mm x 30m; 0.25 µm)
- Injection: S/SL injector – splitless mode, at 270°C
- Carrier flow: 1.2 ml/min
- Transfer line: 250°C
- MS/MS parameters: EI Positive
  - SRM ion mode
  - at 70 eV

Method Development

Different commercial SPME fibers and other parameters affecting the performance of extraction process including extraction temperature (see the Figure 1) were investigated during method development.

FIGURE 1. Peak areas for 12 target compounds determined at different extraction temperatures (65°C was chosen as optimal)

Survey Samples

After validation method was applied on the small group of survey samples covering virgin and recycled paperboard in printed and non-printed version. The results confirmed the presumption of higher content of packaging contaminants in recycled and printed paperboard samples as it is shown in the Table 2.

Method Validation

In-house validation of the developed method was carried out for paperboard and 12 target compounds. Due to the difficulty to gain a pure blank paperboard for quantitation the standard addition procedure was employed. The measured parameters were specificity, linearity, precision, accuracy, limit of detection and limit of quantification (LOD and LOQ). The partial results are shown in the Table 1. The example of chromatogram with 12 target compounds is shown in the Figure 2.

Conclusion

- The reported method enables determination and quantification of 12 possible migrants from paperboard
- The method is fully automated thanks to the usage of automated SPME
- Thanks to the usage of automated SPME the developed method is very fast, robust and saving significantly manpower
- The good results obtained from in-house validation confirmed the suitability of this method for monitoring the content of unwanted contaminants in paperboard intended to be used in contact with food