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Performance evaluation of a CTC CombiPAL-GC-FID set-up for the quantification of volatiles emission according to VDA 277

Introduction

Nowadays polymers are widely used by automobile constructors as materials for car interiors. This leads to health risks linked to the exposure to volatiles emitted from the polymers into the vehicle that might be inhaled by the car user. This risk becomes very significant especially under elevated temperature conditions that increase the amount of volatiles released. For these reasons car manufacturers are required to monitor and quantify the volatiles emitted from the different polymers.

A procedure used for this purpose is regulated by VDA 277[1]. This analytical method consists in static head-space sampling followed by gas chromatography coupled to a flame ionization detector (HS-GC-FID). Hereby we investigate the performance of a CTC CombiPAL autosampler coupled to a GC-FID instrument applied to the VDA 277 norm.

Norm specifications

- Calibration curve with R^2>0.995 for acetone in n-butanol in the range 0.1-100 g/L.
- RSD <10% for 10 injections of acetone at 0.5 g/L in n-butanol.
- Retention time for butylated hydroxytoluene (BHT) <16 minutes.
- Full resolution of all individual target compounds from the peaks present in the blank.

Method and experimental details

The norm indicates that the amount of volatiles emitted should be expressed as total carbon emission (µg C/g sample). This amount is calculated based on the response of acetone used as external standard. Calibration solutions of acetone in n-butanol are prepared at nominal concentrations of approximately 0.1-0.5-10-5-10-50-100 g/L.

If quantification of specific compounds is needed, individual external calibration lines must be prepared for the targets. As example, we assess sensitivity and linearity (0.05-10 g/L in n-butanol) for dangerous compounds potentially present in real-life cases such as BHT, benzene, phenol, dodecane, methyl styrene and dicylohexylamine.

Table 1 – Instrumental set-up and method parameters.

Instrument	GC-FID equipped with a CTC CombiPAL
Incubation temperature	120°C
Incubation time	1 h
Injection volume	1.0 mL
Split ratio	1:20
Inlet temperature	200°C
Column	VF-WAXms (30 m x 0.25 mm x 0.25 µm)
Detector temperature	250°C

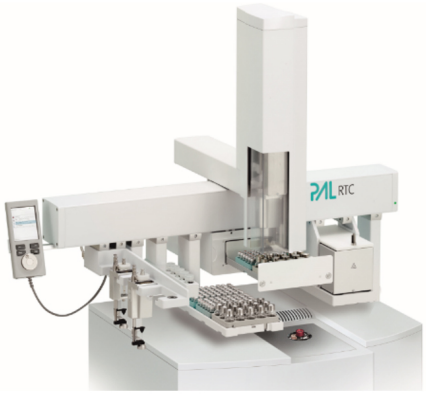

Fig. 1 – CTC CombiPAL.

For all solutions an aliquot of 2.0 µL is placed in a 10-mL vial then closed with a crimp cap with PTFE septa. The vials are analyzed with the instrument and parameters described in Table 1. For real-life samples the method used is the same but the incubation time is increased to 5 hours to allow equilibration of the volatile emission profile.

Results

Acetone

Repeatability was evaluated injecting 10 different vials spiked with the 0.5 g/L acetone standard. Repeatability is very satisfactory with an RSD of 6.5%.

The calibration curve of acetone is shown in Fig. 2. As can be seen, the linearity requirements is fulfilled (R2=0.999).

Sensitivity is suitable to detect acetone at a concentration of least 0.05 g/L.

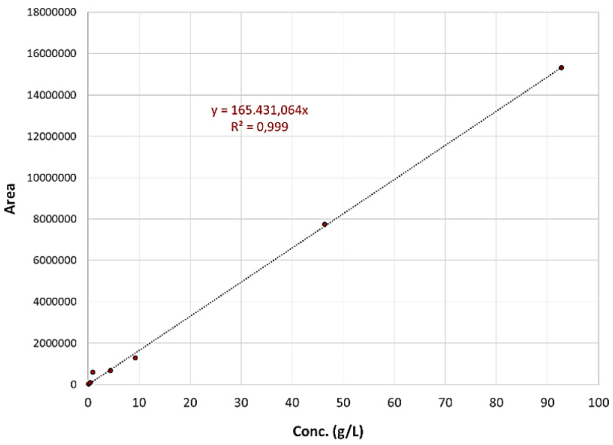

Fig. 2 – Calibration line of acetone in n-butanol.

Target compounds

The retention time of BHT is 15.1 minutes, complying with the norm specification.

All peaks are resolved from each other and from minor peaks coming from n-butanol.

All compounds are detected at 0.05 g/L with signal-to-noise ratio adequate for quantification (Fig. 3) and provide good linearity in the range investigated.

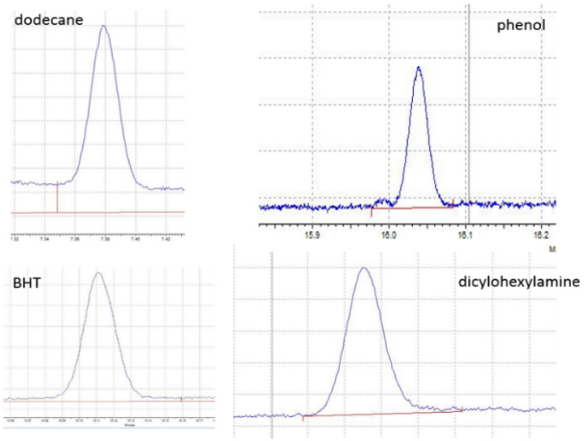

Fig. 3 – Examples of peaks at 0.05 g/L.

HS-GC-FID/MS

The system can be enhanced with the addition of a mass spectrometer detector (MS). This can be easily done simply by splitting part of the column outlet flow to the MS. This configuration allows using FID for quantification while adding the possibility to identify unknown compounds. The tests performed confirms that the HS-GC-FID/MS set-up performance remains fully up to specification.

Conclusions

- CTC CombiPAL-GC-FID is suitable for volatiles emission quantification according to norm VDA 277.
- Linearity, repeatability, sensitivity and chromatographic resolution fulfill the norm requirements.
- The detection limit is at least 0.05 g/L for all compounds investigated.
- The CTC CombiPAL autosampler grants flexibility by allowing quick transition to liquid injection simply by changing the syringe.
- The configuration can be upgraded to HS-GC-FID/MS for identification of unknowns.

References

[1] Verband der Automobilindustrie e.V. – VDA 277 (1995).

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