



Analysis of volatile organic compounds in the workplace air using two chromatographic columns

Overview

High concentration of volatile organic compounds is detected when analyzing the workplace air. It is often the case that certain components do not separate completely at chromatographic column. In order to make the separation of composite compounds more efficient it is suggested to use two chromatographic columns for parallel analysis.

The concentration of certain volatile organic compounds (VOC) in the workplace air is constantly monitored unlike in the ambient air and the air in closed premises. Normally the list of these compounds is known in advance and determined by specific nature of production processes (for example, usage of certain solvents, varnishes, enamels etc.). Therefore, in most of the cases to define the concentration of VOC in the workplace air typical and less expensive chromatographic detectors (FID, PID, ECD, TID, FPD) are used instead of highly sensitive and informative mass-spectrometry detector.

The one of the main issues when using typical detectors is obtaining the accurate identification of analyzed components.

Up-to-date standards for VOC concentration in the workplace air, such as ISO 16017, ISO 16200, US EPA TO-17, regulate the determination of a wide range of components during one measurement: hydrocarbons, halogenated halocarbons, alcohols, ethers, ketones etc. Using sample tubes during sampling enables capturing most of the VOC concentrated in the air. In certain cases dozens or hundreds of various components are captured by absorbent during sampling. Therefore, there is an issue of separating a large number of analyzed compounds during chromatographic analysis. To solve this issue analyzed components can be separated between two parallel chromatographic columns: non-polar (BPX-Volatiles) and polar (Sol Gel-WAX).



Analysis methods

1. ISO 16017-1-2007. Ambient air, workplace air and air in closed premises. Sampling volatile organic compounds with sample tube followed by thermal desorption and gas chromatograph analysis in capillary columns. Part 1. Sampling by pumping method.
2. ISO 16017-2-2007. Ambient air, workplace air and air in closed premises. Sampling volatile organic compounds with sample tube followed by thermal desorption and gas chromatograph analysis in capillary columns. Part 2. Sampling by diffusional method.
3. ISO 16200-1-2007. Quality of workplace air. Sampling volatile organic compounds followed by solvent desorption and gas chromatograph analysis. Part 1. Sampling by pumping method.
4. ISO 16200-2-2007. Quality of workplace air. Sampling volatile organic compounds followed by solvent desorption and gas chromatograph analysis. Part 2. Sampling by diffusional method.
5. US EPA TO-17. Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes.
6. ASTM D6196. Standard Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air.

7. EN 13649. Stationary Source Emissions – Determination Of The Mass Concentration Of Individual Gaseous Organic Compounds.
8. NIOSH 2549. Volatile Organic Compounds.

Equipment

- Gas chromatograph Chromatec-Crystal 9000
- Chromatographic columns
 - BPX-Volatiles 60 m × 0.32 mm × 1.8 μm, Cat. N 054983
 - Sol Gel-WAX 60 m × 0.32 mm × 0.5 μm, Cat. N 054792
- Split/Splitless inlet
- 2x FID detectors,
- Liner (1.2mm i.d., P/N 630-2005)
- Thermal Desorber Chromatec TDA
- Sorbent tube Tenax TA
- Trap Tenax TA
- Carrier-gas – helium

The gas circuit with thermal desorber and two chromatographic columns is shown on the Figure 1.

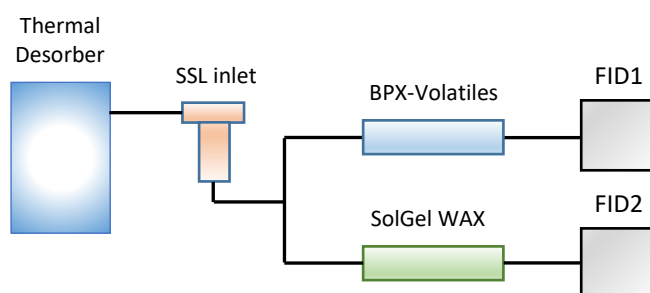


Figure 1 – Flow diagram

Analysis procedure

Samples were collected on Tenax TA sorbent tube using the Sampling pump PV-2, P/N 400-1911.

The analyzed components are carried from thermal desorber via transfer line into GC inlet with a T-branch splitter (P/N 505-0102) located at the outlet to divide the sample between two columns. The outlets of both columns are connected to the flame ionization detectors (FID 1 and FID 2).

Operating mode

Chromatograph		
Run time	25 min	
Column		
Carrier-gas pressure	120 kPa	
Flow split	1:10	
Column temperature		
Isotherm 1:	40 °C	3 min 10 °C/min
Isotherm 2:	190 °C	0 min 20 °C/min
Isotherm 3:	220 °C	
Inlet		
Inlet temperature	250 °C	
FID detector		
Hydrogen flow rate	25 ml/min	
Air flow rate	250 ml/min	
Makeup gas flow	20 ml/min	
Detector temperature	250 °C	
Thermal desorber		
Desorption temperature	270 °C	
Purge gas flow rate	20 ml/min	
Desorption time	7 min	
Trap temperature (low)	-10 °C	
Trap temperature (upper)	280 °C	
Trap heating time	2 min	
Trap heating rate	2000 °C/min	
Tap temperature	220 °C	
Transfer line temperature	220 °C	

The present document contains the description of analysis of the standard VOC mixture in methanol (components concentration 1 μg/μl, sample volume 1 μl). Injection unit for TD tubes (P/N 400-1931) was applied to inject standard liquid sample the sorbent tube, then kept 5 min enabling to blow the solvent (methanol) off the sorbent tube before inserting it into the thermal desorber.

Results

Figures 2 and 3 show the checkout chromatograms of a standard VOC mixture with simultaneous separation of components between two columns with different polarity: BPX-Volatiles and Sol Gel-WAX. The list of the components is given in Table 1. As chromatograms show *m*- and *p*-xylenes, *o*-xylene and styrene are not separated on the column BPX-Volatiles while methanol, *n*-nonane, *n*-decane, 1,2-dichloroethane, chloroform, tetrachloroethylene, 1-butanol and *p*-xylene are not separate on the column SolGel-WAX. If peaks are not separated on one of the columns during the analysis with two columns identification can be done on the second column.

Chromatec Analytic software allows configuring identification parameters when recording chromatograms from two detectors. The feature *Identification* in Components list is applied (see table 2). The peak identification on the main channel (*Usual* peak) is confirmed, provided that this peak is found on the second channel (*Confirming* peak).

To ensure the correct identification of the compounds under analysis the list of *components* with the same names for each channel is drawn up in the Table Components. Identification column in this table contains the component feature: on one of the channels it is *Usual* and on the other – *Confirming*. The Regular feature is specified for such channel where the component is better separated from the other components under analysis and extraneous peaks. On the second channel where the overlapping with other components is more probable the component will have the *Confirming* feature. Component list configuration in Chromatec Analytic SW is shown on the Figure 3.

When identification parameters are set Chromatec Analytic will identify chromatographic peaks only if the given component was detected on the both columns. This increases the component identification accuracy when analyzing composite compounds.

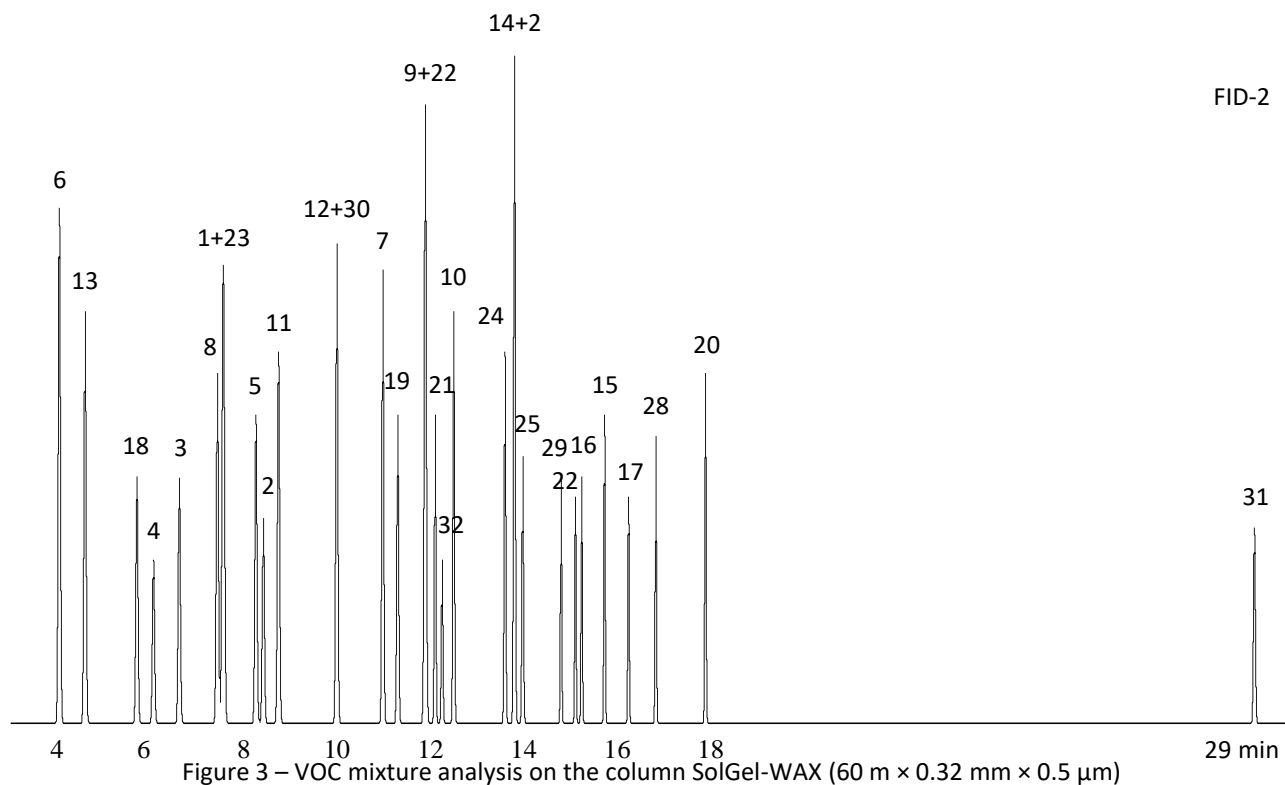
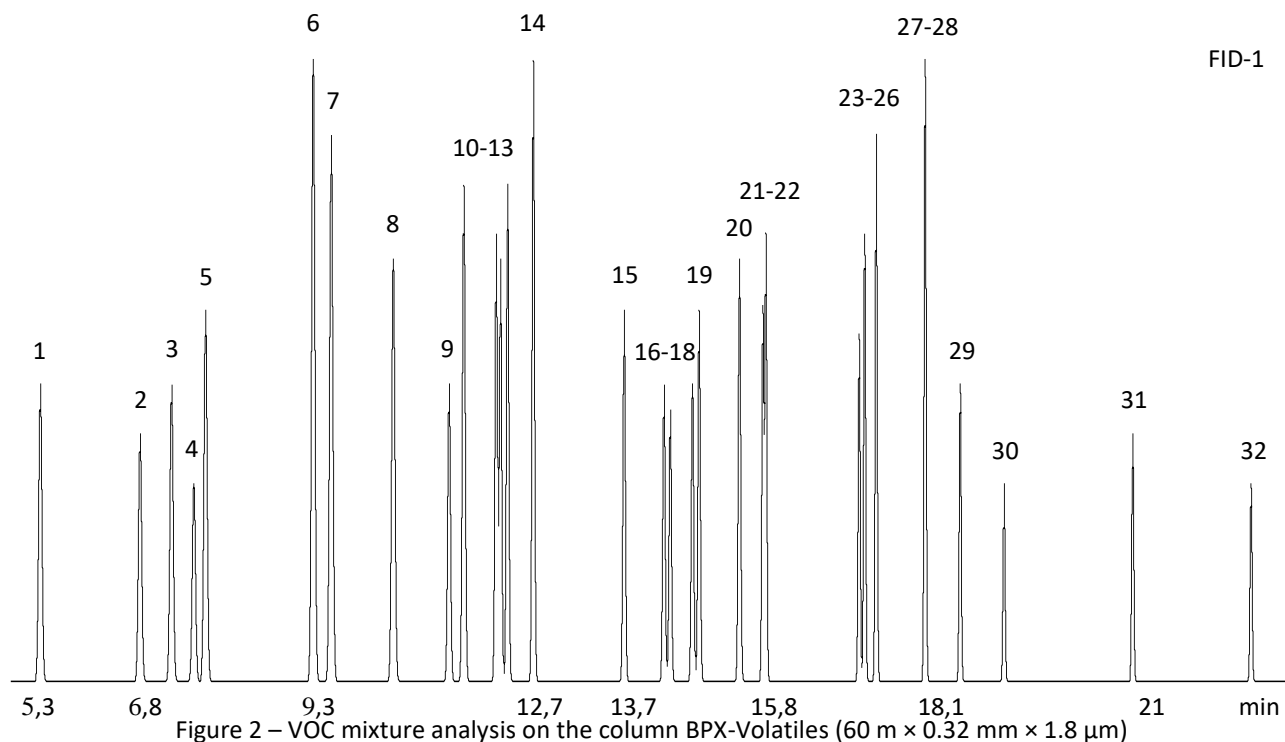
Table 1 – List of components

1	Methanol	17	2-Methyl-1-butanol
2	Etanol	18	n-Octane
3	Acrolein	19	Toluene
4	Acetone	20	1-Pentanol
5	2-Propanol	21	Butyl acetate
6	n-Hexane	22	Tetrachloroethylene
7	1-Propanol	23	n-Nonane
8	Ethyl acetate	24	Ethylbenzene
9	Chloroform	25	<i>m</i> -Xylene
10	2-Methyl-1-propanol	26	<i>p</i> -Xylene
11	Benzene	27	<i>o</i> -Xylene
12	1,2-Dichloroethane	28	Styrene
13	n-Heptane	29	Isopropyl benzene
14	1-Butanol	30	Decane
15	2-Ethoxyethanol	31	Phenol
16	3-Methyl-1-butanol	32	Undecane

Table 2 – Configuration of identification parameters in Chromatec Analytic during the analysis with two columns

Name	Detector	Time, min	Screen, %	Identification
Acetone	FID-2	6.124	5.000	Usual
Acrolein	FID-2	6.685	5.000	Usual
Ethyl acetate	FID-2	7.512	5.000	Confirming
Methanol – n-Nonane	FID-2	7.642	5.000	Usual
2-Propanol	FID-2	8.351	5.000	Confirming
Ethanol	FID-2	8.511	5.000	Confirming
Benzene	FID-2	8.842	5.000	Usual
1,2-Dichloroethane + n-Decane	FID-2	10.109	5.000	Usual

Chromatograms



Conclusion

Two chromatographic columns with different polarity enable the efficient separation of VOC that may be abundant in an air sample. Identification of components on the two columns is carried out using features of Chromatec Analytic software. To increase the accuracy one of the columns is assigned as confirming in order to prevent the extraneous peak from overlapping the peaks under analysis.

