phenol, methylphenol and dimethylphenol

(thermally desorbed)

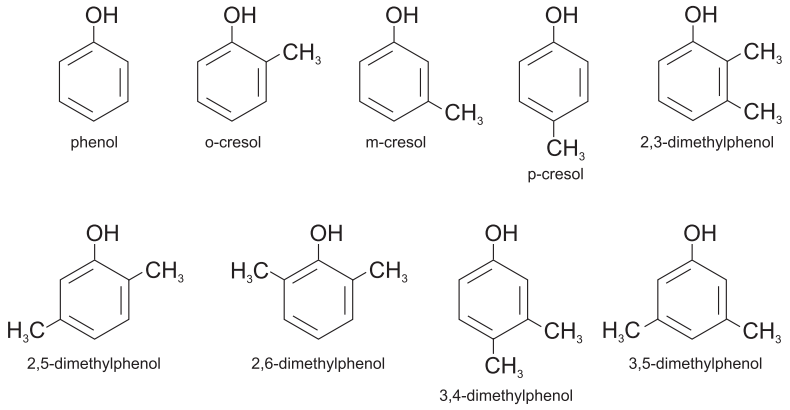
Radiello components to be used

White diffusive body code 120
Supporting plate code 121
Vertical adapter code 122 (optional)
Adsorbing cartridge code 147

Principle

Code 147 cartridge is a stainless steel net cylinder with 100 mesh grid opening and 4.8 mm diameter, packed with 250 ± 10 mg of Tenax-TA, particle size 20-35 mesh. Phenols are trapped by adsorption and recovered by thermal desorption, analysis is performed by capillary gas chromatography and MS detection.

The method has been optimized for the following compounds:



Sampling rates

Sampling rate values Q at 298 K (25 °C) and 1013 hPa are listed in the table on the right. All of the values shown have been experimentally measured.

Effect of temperature, humidity and wind speed

Sampling rate varies from the value at 298 K on the effect of temperature (in Kelvin) as expressed by the following equation:

$$Q_K = Q_{298} \left(\frac{K}{298}\right)^{1.5}$$

where Q_K is the sampling rate at the temperature **K** and Q_{298} is the reference value at 298 K. Sampling rate is invariant with humidity in the range 15 - 90% and with wind speed between 0.1 and 10 $m \cdot s^{-1}$.

	Q_{298} $ml \cdot min^{-1}$	limit of detection[1] $\mu g \cdot m^{-3}$	uncertainty at 2σ %
phenol	**38**	**0.3**	**24.1**
o-cresol	**45**	**0.4**	**17.5**
m-cresol	**48**	**0.4**	**8.0**
p-cresol	**48**	**0.4**	**8.0**
2,3-dimethylphenol	**53**	**0.4**	**26.0**
2,5-dimethylphenol	**51**	**0.3**	**25.2**
2,6-dimethylphenol	**46**	**0.4**	**7.6**
3,4-dimethylphenol	**60**	**0.4**	**22.1**
3,5-dimethylphenol	**61**	**0.4**	**22.2**

[1]after 24 hours exposure and with MS detection; analytical conditions as described in the Analysis paragraph.

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Calculations

The listed sampling rate values take already into account the recovery yields of adsorbed compounds. **The average concentration over the sampling period is therefore calculated from sampled mass of analyte and exposure time without introducing any other corrective factor,** apart from temperature variations of Q.

Average concentration ***C*** in $\mu g \cdot m^{-3}$ over the whole exposure time is calculated according to the following expression:

$$C\ [\mu g \cdot m^{-3}] = \frac{m\ [\mu g]}{Q_K\ [ml \cdot min^{-1}] \cdot t\ [min]}\ 1{,}000{,}000$$

where:
m = mass of analyte in µg
t = exposure time in minutes
Q_K = sampling rate at temperature K

Exposure

Workplace environment

Exposure time can range from 2 to 8 hours.

Other indoor sampling experiments and outdoor campaigns

The recommended exposure times range from 8 hours to 7 days.

Storage

If cartridges are kept in a cool place without phenol and related compounds contamination, blank level and adsorbing capacity stay unaltered for at least 24 months.

After exposure the cartridges, well capped and kept in a cool and solvent-free place, maintain their content unaltered for at least three months.

Analysis

The analytical method hereafter described have been set up by the Perkin-Elmer Turbomatrix thermal desorber and Agilent 5973 MSD mass spectrometer detector. They may be implemented on other instruments by introducing minor adjustements as suggested by the analyst's experience and characteristics of employed instrumentation.

Desorption

The thermal desorber is equipped with 1/4" OD SS sample tubes, they have to be hollow and free: discard the stainless steel gauze disk which is fitted to the groove and discard also the springs if present.

Code 147 cartridge has been dimensioned to fit the diameter of Turbomatrix thermal desorption tubes. Its length is such that, when the cartridge is introduced into the tube and is stopped by the groove, it is positioned exactly centrally with respect to the tube length.

Inner diameter of Perkin-Elmer tubes is not always exactly the same; it may be the case therefore that a cartridge code 147 does not slide easily into the tube. Some pushing tool may be helpful then, such as a 500 µl syringe piston, a glass bar or an iron wire 2-3 mm thick. In some cases the tube inner diameter is sligthly larger than the cartridge outer diameter: the cartridge can therefore be pushed out from the tube during desorption due to the desorption gas pressure. If this is the case, just press sligthly one end of the cartridge to make it oval.

Once capped, the Turbomatrix steel tube has to be positioned in the carousel with the grooves on the bottom.

Temperatures and timing

- ✓ Desorption: 280°C for 10 minutes
- ✓ Cryofocusing trap (Tenax TA): during primary desorption mantain at 2 °C, secondary desorption at 99 °C/sec up to 290 °C, maintain at 290 °C for 1 minute
- ✓ Six port valve: 150 °C
- ✓ Transfer line: 200 °C

Flows

- ✓ Carrier gas: helium, 24 psi
- ✓ Desorption flow: 100 ml·min^{-1}
- ✓ Inlet split: 80 ml·min^{-1} (flow from tube to cryofocusing trap: 20 ml·min^{-1})
- ✓ Outlet split: 25 ml·min^{-1}

Instrumental analysis

Column

100% dimethylpolysiloxane, length 50m x 0.2mm, film thickness 0.5 µm; (e.g. Petrocol DH 50.2, Supelco Code 24133-U) the column is directly fitted to the six-port valve of Turbomatrix apparatus

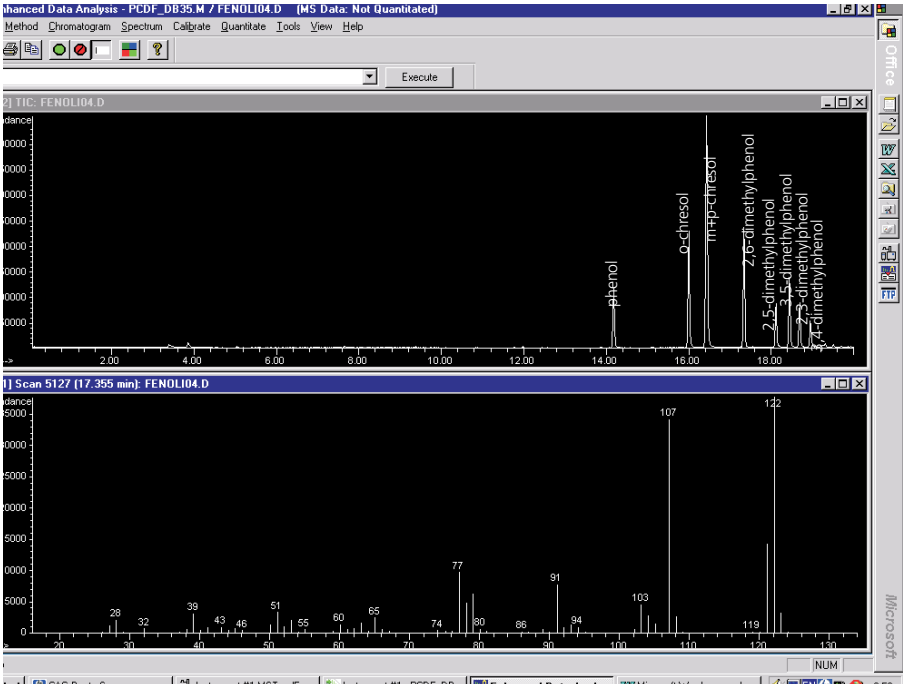
Temperatures

- ✓ GC oven: 50 °C for 2 minutes, 8 °C/min up to 160 °C, 12 °C/min up to 260 °C, final isotherm 2 minutes
- ✓ GC-MS interface: 260 °C

Flows

- ✓ helium carrier gas: 0.8 ml·min^{-1}

In the figure on the right a typical chromatogram (as total ion current) is shown.



Calibration

Calibration curves are obtained by gas-phase injections of methanol solutions of the analyzed compounds onto blank cartridges. Injections are performed through a GC injector, where a short piece of wide-bore (0.53 mm i.d.) deactivated uncoated column is installed. The other end bears a Swagelock reducing connection (1/16" to 1/4"). The 1/4" Swagelock nut has to be equipped with a PTFE ***ferrule*** instead of the original steel one (use PTFE ferrules that come along with the Turbomatrix caps).

Introduce a blank code 147 cartridge in a Turbomatrix tube and fit the tube to the Swagelock nut. Keep the injector at 200 °C but do not heat the oven. Slowly inject 1 µl of each calibration solution under nitrogen flow (50 ml·min^{-1}) and let the system purge for 2 minutes. Analyze the cartridge as you would do with a sample.

We suggest you to prepare a complete set of calibration solutions by subsequent dilutions such as they contain, for example, 4, 2, 1, 0.05 and 0.010 µg·µl^{-1} of each compound.

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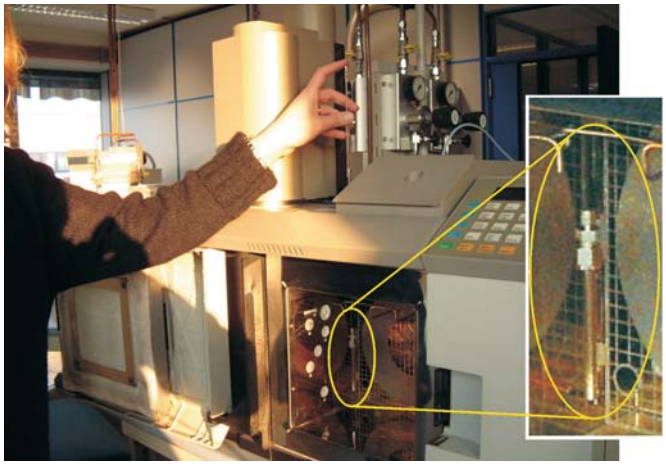
Cartridge recovery

Recovery yield of phenol and related compounds is higher than 98%. Nevertheless, traces of analyzed compounds remain on the cartridge, but a thorough regeneration can be performed as follows.

Wash the cartridge with methanol (5 ml in a glass tube are enough) stirring from time to time. Let it dry in the air and finally condition it at 300 °C for two hours under nitrogen or helium flow.

Thermal stability of Tenax-TA is good enough to allow a great number of sampling, analysis and conditioning cycles, provided that conditioning temperature does not exceed 300 °C and nitrogen or helium employed do not contain more than 10 ppm of oxygen.



Calibration standards are easy to prepare by applying to the gas chromatograph injector a Swagelock reducing connection (1/16 to 1/4"); injections are performed through a GC injector, where a short piece of wide-bore (0.53 mm i.d.) deactivated uncoated column is installed.

Analytical service for radiello diffusive sampler

Analytical services are also available to European users directly through the Fondazione Salvatore Maugeri. Users can send the sampled cartridges in for analysis and results. For more information on prices and conditions of this service, please contact directly

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