

Starter kit - sampler for 1,3-butadiene and isoprene (Cat. No. RAD141S)

CAUTION: Do not swallow. Do not smoke. Wash the hands thoroughly after use. Avoid contact with the eyes, skin and clothes. In case of contact with eyes, flush with large amounts of running water for at least 15 minutes. See MSDS for complete safety information.

Principle

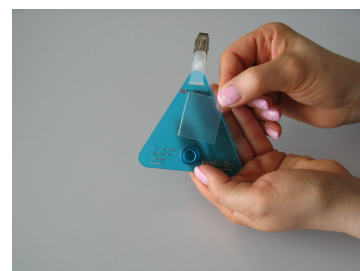
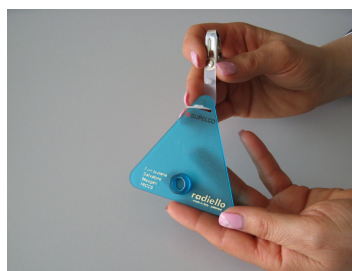
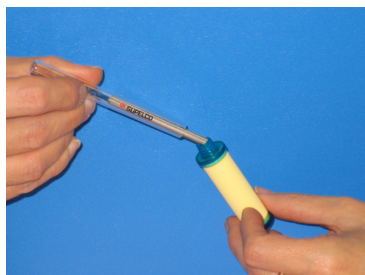
The starter kit for sampling 1,3-butadiene and isoprene (thermal desorption) contains the following:

- 2 graphitised carbon (Carbopack X) cartridges with barcode labels (sample + blank, Cat. No. RAD141)
- 1 support plate (separate parts, Cat. No. RAD121)
- 1 yellow diffusive body (Cat. No. RAD1202)
- 1 vertical adapter for personal sampling (Cat. No. RAD122)

It allows the sampling of 1,3-butadiene and isoprene by diffusion through a high-thickness microporous polyethylene membrane (part code RAD1202) and adsorption onto a graphitised carbon (Carbopack X 40/60 mesh) cylindrical cartridge (part code RAD141). The analytes are recovered by thermal desorption, then analysed by gas chromatography-mass spectrometry.

Procedure for sampling and analysis

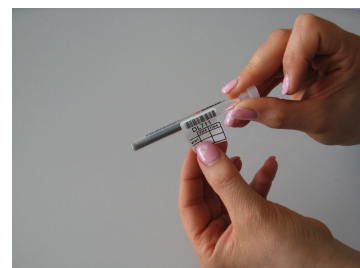
- 1) Assemble the support plate: insert the clip strip in the slot, ply and insert the peg into the hole; peel off the transparent pocket and adhere it onto the plate in a central position.
- 2) Take one cartridge, draw it out of its storage glass tube and place it in the diffusive body. Keep the glass tube and stopper to restore the cartridge after sampling. Keep the second cartridge to be analysed as a blank.



- 3) Be sure that the cartridge is correctly centred in the diffusive body ensuring that the cartridge lays completely inside. If the cartridge is sticking out, gently tap on the diffusive body.



- 4) While keeping the diffusive body in vertical position, screw it onto the support plate.
- 5) Expose your sampler. For outdoor sampling, use of a shelter (Cat. No. RAD196) is recommended.
- 6) Record the date and time of start of exposure on the barcode label (AVOIDING THE USE OF MARKER PENS, which contain solvents!) and place it in the sampler's pocket.
- 7) Once the exposure is finished, unscrew the diffusive body and transfer the exposed cartridge back in its original storage glass tube.
- 8) Record the date and time of end of exposure, place the adhesive label onto the glass tube so that the barcode is parallel to the axis of the tube.



The recommended analytical procedure includes:

- Thermal desorption by commercial thermal desorption apparatuses (i.e. Perkin Elmer, Dani, Markes...): the outer diameter of Cat. No. RAD141 cartridges is fitted to the inner diameter of most stainless steel desorption tubes that have a ID of 4.9 mm;
- GC/MS determination, split injection, external standard quantitation.

Typical analytical conditions are listed below as guidelines. Adjust the temperature program in order to separate and elute all the compounds present in your samples. Look at the possible presence of 1-butene, 2-butene (*trans* and *cis*), 2-methyl-1-propene (isobutylene), which show the ion 54 a.m.u. in their mass spectra and thus could interfere with 1,3-butadiene quantitation (see below).

Automated thermal desorber

Carrier gas: Helium at 12 psi
 Primary desorption temperature: 320 °C for 6 min
 Total primary desorption flow-rate: 120 ml·min⁻¹
 Inlet split flow-rate: 100 ml·min⁻¹
 Outlet split flow-rate: 40 ml·min⁻¹
 Overall split ratio: 1:150
 Valve temperature: 150 °C
 Cold trap packing: Tenax TA 60/80 mesh
 Cold trap temperature: -20 °C
 Secondary desorption temperature: 290 °C for 1 min
 Transfer line temperature: 200 °C

GC/MS system

Column: any capillary column capable of separating permanent gases
 Column temperature program: 80 °C, 0.5 min; 25°C·min⁻¹ to 175°C; stand for 4 min; 25 °C·min⁻¹ to 250°C; 7 min
 Total run time: 20 min
 MS mode: SCAN from 25 to 250 a.m.u.
 EM setting: autotune value
 Target ions for quantitation: 54 a.m.u. (1,3-butadiene), 67 a.m.u. (isoprene)

Analyse the samples in your laboratory following the guidelines reported in the manual ([http:// www.radiello.com/english/cov_term_en.htm](http://www.radiello.com/english/cov_term_en.htm)) or send them in for **analysis by Fondazione Salvatore Maugeri – Centro di Ricerche Ambientali** – Via Svizzera, 16 – 35127 PADOVA (ITALY). Details on the analytical service provided by the Fondazione are available on the web site <http://www.radiello.com/english/LabService.htm>.

Uptake rates

The values of diffusive uptake rate were **experimentally measured** at 20°C (273 K) and 1013 hPa in a standard atmosphere chamber.

The uptake rate for **1,3-butadiene** in **workplace air** is **30.5 ± 0.3 ml·min⁻¹** (nominal value at a concentration level between 114 and 226 µg/m³ for 8-hour exposures). For longer term sampling (e.g. 7 days) of **1,3-butadiene** please refer to Strandberg et al. ⁽¹⁾⁽²⁾.

For **isoprene** the uptake rate is **41.2 ± 4.9 ml·min⁻¹** (best estimate in the range 2 – 6,680 µg/m³ for exposures from 30 to 480 min).

Effect of temperature and humidity

Both temperature and relative humidity affects the uptake rate for 1,3-butadiene. If the temperature decreases to 5 °C, the bias is + 12.9% at 20% RH or -2.4% at 80% RH, with respect to 20 °C and 50% RH. Avoid sampling at temperatures as high as 40 °C, because the uptake rate shows a significant decrease.

The effect of temperature and relative humidity on the uptake rate for isoprene is lower: at low temperature and humidity (5°C, 21% RH) the uptake rate is 10% higher, while at high temperature and humidity (41°C, 77% RH) there is a 23% decrease.

Do not expose the Radiello sampler directly to rain. Always use the shelter Cat. No. RAD196 when sampling outdoors, to avoid water entering the membrane and wetting the sorbent.

Calculations

The mean concentration *C* over the exposure time interval is calculated from the mass of analyte found onto the cartridge (corrected for the blank, if any) and from the exposure time, using the above values of the uptake rate, as follows

$$C_{[mg \cdot m^{-3}]} = \frac{m_{[mg]} - m_{b[mg]}}{U_{[ml \cdot min^{-1}]} \cdot t_{[min]}} \cdot 1,000,000$$

where:

m = mass of analyte in µg

m_b = mass of blank in µg

U = uptake rate in ml · min⁻¹

t = exposure time in minutes

The desorption efficiency, determined by repeated desorption of the same tube, is higher than 99.9% for both compounds. Therefore, there is no need of introducing correction factors.

Limit of quantitation

Blank response, the limit of detection (LOD) and the limit of quantitation (LOQ) depend on the instrumentation and on the analytical conditions.

Under the analytical conditions specified above, the blank value is not detectable, i.e. less than 0.5 ng for both compounds.

The LOQ for 8-hour workplace exposure is 0.1 µg/m³. For 7-day ambient air exposure the LOQ is 0.03 µg/m³; see also Strandberg et al. ⁽²⁾

Measurement uncertainty

The following table shows the values of the uncertainty in the measurements of **1,3-butadiene** in workplace, assessed by two different approaches. The uncertainties were first determined under **laboratory conditions**, following the methods of the ISO GUM (*Guide to Expression of Uncertainty in Measurement*, International Organization for Standardization, Geneva, 1993) and ISO 5725:1994 (*Accuracy (trueness and precision) of measurement methods and results*). In this case, the uncertainty accounts for all contributions involved in the whole measurement process (effect of time, T, RH on uptake rate, uncertainty of measured mass and so on), which were determined according to EN 838:1995. Afterwards, the uncertainty was determined by a **field comparison** with the method OSHA 56 (as the reference method), following the standard ISO 13752.

Measurement uncertainty for 8-hour diffusive sampling of 1,3-butadiene in workplace air

Relative combined expanded uncertainty (2·u _c)	200 µg/m ³	442 µg/m ³ (0.1 TLV)	2.210 µg/m ³ (0.5 TLV)	4.420 µg/m ³ (TLV-TWA ACGIH)
Laboratory tests at 20 °C, 50% RH (EN 838, calculations by ISO GUM)	48.4 %			
Field comparison (ISO 13752)	37.0 %	25.0 %	11.1 %	7.9 %

Shelf life

Kept in a cool place and away from volatile organic compounds, the cartridges maintain unchanging blank level and adsorbing capacity for at least eighteen months. Expiry date and lot number are printed onto the plastic envelope wrapping each cartridge: its integrity stands as warranty seal. Expired adsorbent cartridges can be re-conditioned. It is recommended to run a blank analysis for cleanliness. See analytical protocol above.

The leak test, performed according to EN 838, at 100 ppb for 8 hours, showed no detectable uptake of the analytes by new, sealed cartridges.

References

- 1) Strandberg et al. *Atmos. Environ.* 39(2005), 4101-4110.
- 2) Strandberg et al. *Atmos. Environ.* 40(2006), 7686-7695.