

Determination of Methyl methacrylate monomer by GC

Analytical conditions

GC conditions

zone

temperatures: 250°C (injector)
300°C (detector)

column program: initial temp at 120°C, increase temp at 5°C/min to 150°C, hold for 5 min

column gas flow: 1.35 mL/min (hydrogen)

septum purge: 1.5 mL/min (hydrogen)

injection size: 1.0 μ L (5.4:1 split)

column: 60 m \times 0.32-mm i.d. capillary GC Column (4.0- μ m film thickness)

retention times: 9.8 min (MME)
9.3 min (benzene)

FID conditions

hydrogen flow: 36.5 mL/min

air flow: 444 mL/min

nitrogen makeup
flow: 46.5 mL/min

An internal standard (ISTD) calibration method is used. A calibration curve can be constructed by plotting micrograms of analyte per milliliter versus ISTD-corrected response of standard injections. Bracket the samples with freshly prepared analytical standards over a range of concentrations.

Analysis:

Methyl methacrylate can be determined in air by gas chromatography with flame ionization detection. The sample is adsorbed on fused silica (XA-2 resin) or charcoal coated with 4-tert-butylcatechol and desorbed with carbon disulfide or toluene. The estimated limit of detection is 0.01 mg per sample. A method involving desorption with 5% isopropanol in carbon disulfide from charcoal has a detection limit of 0.8 mg/m³.

Interferences (analytical)

Any compound that produces an FID response and has a similar retention time as the analyte or internal standard is a potential interference. If any potential interferences were reported, they should be considered before samples are desorbed. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

Retention time on a single column is not considered proof of chemical identity. Analysis by an alternate GC column or confirmation by mass spectrometry are additional means of identification.

Calculations

The amount of analyte per milliliter is obtained from the appropriate calibration curve in terms of micrograms per milliliter uncorrected for desorption efficiency. The back (55-mg) section is analyzed primarily to determine the extent of sample saturation during sampling. If any analyte is found on the back section, it is added to the amount on the front section. This total amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentration is calculated using the following formulae.

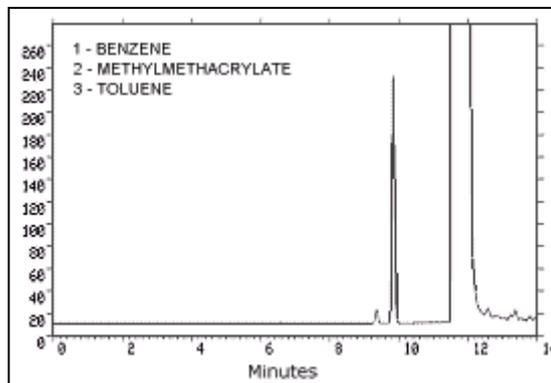
$$\text{mg/m}^3 = \frac{A \times B}{C \times D}$$

where A = micrograms of analyte per milliliter
B = desorption volume
C = liters of air sampled
D = desorption efficiency

$$\text{ppm} = \frac{24.46 \times \text{mg/m}^3}{\text{MW}}$$

where 24.46 = molar volume (liters) at 101.3 kPa (760 mmHg) and 25°C

$$\% \text{Purity} = \frac{\text{Mass of Pure Product(Std)}}{\text{Mass of Product Obtained}} \times 100$$



Chromatogram of Methyl methacrylate monomer