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INDUSTRIAL HYGIENE MONITORING METHOD

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CK-IHM-82-3

The Determination of Methyl Chloride in Air (Gas Chromatographic Method)

1. Scope:

- 1.1 Analyte: methyl chloride
- 1.2 Matrix: air
- 1.3 DING:
8 hour time-weighted average (TWA)
methyl chloride 50 ppm v/v
- 1.4 Procedure: adsorption onto charcoal at 30 mL/minute flow for 3 hours, thermal desorption and analysis by gas chromatography with flame ionization detection (GC/FID).
- 1.5 Range: 3 hour TWA
10 - 500 ppm v/v
- 1.6 Recovery and Precision (at 95% confidence level):
95.6% $\pm$ 20%
- 1.7 Humidity Effect: not determined
- 1.8 Validation Date:
81/09/14 - 81/11/19

2. Chemical Safety Precautions:

Methyl chloride is a colourless, tasteless gas. Its faint ethereal odour and mild narcotic effect are seldom sufficient to provide warning of overexposure. Exposure to methyl chloride causes central nervous system depression which leads to incoordination and drunkenness. There is no appreciable hazard from skin irritation or absorption from the gas. Methyl chloride should be handled in the fumehood.

3. Principle:

- 3.1 A measured volume of air is drawn through two stainless steel tubes in series containing Pittsburgh Coconut Base charcoal.

- 6.2 Thermally desorb the front and back sections of both tubes. Analyse by GC/FID using operating conditions as in section 6.5.
- 6.3 Analyse a PTD blank and a charcoal/PTD blank with each set of samples.
- 6.4 Prepare three volume/volume standards using the Bi-M Gas Blender over the range of 10 - 1000 ppm. Analyse the standards by GC and plot a calibration curve or calculate the response factor at each level.

6.5 Operating Conditions

PTD: desorption temperature 130°C
desorption time 2 minutes
desorption gas ambient air

GC: Column - 5 foot X 1/8 inch stainless steel packed with 80/100 mesh Porapak P.

column temperature 100°C
injector temperature 200°C
detector temperature 250°C
nitrogen flow 15 mL/minute
hydrogen flow 30 mL/minute
air flow 240 mL/minute
injection volume 1μL
attenuation 32
chart speed 0.5
analysis time 5 minutes

A typical chromatogram is shown in Figure 1.

7. Calculations:

- 7.1 Calculate the concentration of methyl chloride by comparing areas with external standards.

$$A = \frac{B \times C}{D}$$

where A = concentration of methyl chloride in sample (nL/mL)

B = concentration of methyl chloride in standard (nL/mL)

C = area of methyl chloride in sample (integrator counts)

D = area of methyl chloride in standard (integrator counts)

8.2 The analytical limit of detection for methyl chloride is 3 nL/mL which corresponds to a TWA concentration of 0.2 ppm v/v for a 6 litre sample.

8.3 Under the collection conditions described in section 6.1 breakthrough did not exceed 10% in the second tube at ten times the TLV concentration.

9. Storage Stability

Storage studies indicated samples could be stored at -3°C for 3 days without loss of recovery.

10. Sample Tube Packing

Sample tubes were packed with PCB charcoal in the laboratory. The collector tube described is the only one available for the PTD and has a 0.5g capacity (0.4g front/0.1g back). Tubes were packed on a volume basis. Studies indicated tubes could be reused after desorption or be repacked.

11. References

Kerr, R.W., "The Validation of a Gas Chromatographic Method: Determination of Methyl Chloride in Air", Report No. CK-82-45, January, 1982, Western Canada Division.

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