

Summary

Ambient air samples were collected on three types of multimedia adsorption tubes packed with combinations of glass beads, graphitic carbons and carbon molecular sieves as recommended by US EPA Method TO-2 and TO-17.

Method TO-2 is used to collect and determine highly volatile, non-polar organics (Vinyl Chloride, Vinylidene Chloride, Benzene, Toluene) that can be captured on carbon molecular sieve and determined by thermal desorption techniques. The compounds to be determined by this technique have boiling points in the range of - 15 to 120°C.

Method TO-17 has been drafted by the US EPA in order to update Methods TO-1 and TO-2 for a new generation of thermal desorption systems as well as new types of solid adsorbents. It is an informative method for ambient air monitoring.

The tubes were desorbed and analyzed on an Analytical Volatile Organic Compound System (AVS). Vinyl Chloride was determined by GC-MSD in Selective Ion Mode (SIM) employing ion 62 as the target ion.

Preparation of Tubes

Approximately 0.2 mL of water was introduced into the back end of each C, MB or SS tube to aid in cleaning and conditioning by steam distillation. Purified helium gas was purged through the tubes (in reverse direction to collection) while they were heated to 280-310°C for 45 minutes on an Envirochem Model 7856/6 Thermal Desorption Unit (TDU). Cleaned tubes were packed hot in appropriate clean glass containers and closed with teflon lined screw caps.

CARBOTRAP C/Y, CARBOTRAP B/X, CARBOXEN 569 and CARBOSIEVE SIII were obtained from Supelco, Oakville, Ontario and were used to pack sample collection tubes as described in the following table.

| PACKING ORDER | 1 | | 2 | | 3 | 4 |
|------------------|-------------------------------------|-------------------------------------|---|-------------------------------------|--------------------------------------|---|
| PACKING MATERIAL | CARBOTRAP C (mg) (20-40 mesh) | CARBOTRAP Y (mg) (20-40 mesh) | CARBOTRAP B (mg) (20-40 mesh)/ CARBOPACK B (mg) (60-80 mesh) | CARBOTRAP X (mg) (20-40 mesh) | CARBOXEN 569 (mg) (20-45 mesh) | CARBOSIEVE SIII (mg) (60-80 mesh) |
| SAMPLE TUBE | | | | | | |
| C | 200 | 120 | 160 | 200 | - | 150 |
| MB | 400 | 250 | 400 | 500 | 300 | 300 |
| SS | - | 80 | - | 160 | 90 | 120 |

OSB Lab replaced sorbents C/B with Y/X to minimize artifact formation. Artifact formation had been observed since the inception of OSB Lab in late winter 1995. Tests showed that conversion occurred almost entirely on the focusing traps. Supelco was contacted about these observations and provided research grade 40/60 mesh Y/X to replace C/B sorbents. Supelco indicated that Y/X were now manufactured in-house and were cleaner. Also, version C was going to be discontinued and a new version B would be produced by Supelco. Instrument focusing traps were converted to Y/X on April 9 1996. A standardized packing size 20-40 mesh was established for Y/X sorbents except the traps which required a finer particle size. It must be noted that manufacturing techniques have changed substantially at Supelco over the past 10 years and consequently the sorbent properties.

Sample Collection

Field samples were collected on three different types of adsorption tubes (C, MB and SS). Envirochem C tubes did not require additional handling unless moisture was present, otherwise they were analyzed directly. If high moisture was suspected to be present, the tube was purged

with dry helium for 15 minutes or more at room temperature. Samples collected on MB and SS tubes had to be transferred onto C tubes with a stream of dry helium while being heated to 270-300°C for 15 minutes on an Envirochem Model 7856/6 Thermal Desorption Unit (TDU) prior to instrumental analysis.

Sample Analysis

Analysis was carried out on the Analytical VOC System (AVS) which consisted of an Envirochem 810A Concentrating Capillary Inletting System coupled to a Hewlett Packard 5890E Series II Plus Gas Chromatograph via a 260°C heated nickel steel transfer tube (line). The gas chromatograph contained a 60 m HP 624 fused silica capillary column in line with an HP 5972A Mass Selective Detector (MSD).

The C tube was desorbed at 260°C on the Envirochem Unit sequentially onto a primary and then secondary internal analytical trap. The secondary smaller focusing trap then automatically injected by means of thermal desorption, any Vinyl Chloride into the GC via the transfer line. The GC employed cryogenic oven control (liquid CO₂ cooled) initiated at 0°C for superior separation. Vinyl Chloride was detected by the MSD in SIM mode at enhanced sensitivity for ions M/Z 62, 61 and 35 (ion 62 was used for quantitation while ions 61 and 35 were used for confirmation). Ions 27 and 64 were not used for quantitation due to too much background interference. The MSD was interfaced to an HP ChemStation Data System G1701AA-A.03.02 with a National Institute for Science and Technology (NIST/EPA/NIH) MS compound library, with NIST98/HP-PBM98 combination mass spectral search programs obtained from ChemSW.

Calibration and Standardization

The HP ChemStation Data System was capable of plotting calibration data in a number of ways. An evaluation of a number of equations was made to determine the best consistent fit for the sample data. Sample concentrations were calculated from standards using two regression ranges developed for this project. The standards were split into a high and low range, above and below 2.5 ng. The high range was obtained from a Tedlar bag containing 100 ppm Vinyl

Chloride transferred directly from a Scotty II Mix 73 certified gas reference standard cylinder and was designated as the high standard. The low range was obtained from a Tedlar bag containing approximately 1 litre of Zero air to which was added 50 mL of 100 ppm Vinyl Chloride from the high standard and was designated as the low standard. Standards above 2.5 ng were plotted using linear regression. Standards below 2.5 ng were fitted with a quadratic equation forced through zero. High and low standards were routinely compared to ensure that linearity was maintained. Calibration was accomplished via direct gas injection through the AVS.

A previous study evaluated Vinyl Chloride sample breakthrough volume, Vinyl Chloride Tedlar bag stability of diluted gas, and demonstrated an MDL of 0.014 ng per tube.