DETERMINATION OF VINYL CHLORIDE (VCM) IN AIR

SCOPE

This method is used for personnel monitoring and spot checking the air at various points in and about the plant for vinyl chloride content.

OUTLINE OF METHOD

Representative air samples are collected in clean, plastic bags, and the VCM content of the air is determined by gas chromatography.

APPARATUS

Almost any commercial gas chromatograph equipped with a flame ionization detector, gas sampling valve, and column backflush valve is satisfactory. An example is the Carle Model 9500 flame ionization chromatograph.

The chromatogram is recorded on a potentiometric recorder with a full scale deflection of one millivolt, a response time no greater than one second, and a chart ten inches wide.

Separating Column - 6' x 1/8" aluminum tube packed with 80/100 mesh "Chromosorb 101"

Gas Sample Bags - Valve-type, polyester-protected, vinyl-aluminum bag with hose bib and shut-off valve, 7- or 15-liter capacity Calibrated Instruments, Inc., Ardsley, New York

Chromatograph Gases - Nitrogen or helium carrier gas, hydrogen, and air -- all "zero" grade

Calibration Gas - Approximately 10 ppm (v/v) vinyl chloride in nitrogen, supplied by Precision Gas Products, Rahway, N. J.

PROCEDURE

Set the chromatograph column oven temperature to 90°C and the carrier gas flow rate to 20 mℓ per minute. The detector and injection port temperatures should be 20-to-30 degrees higher than the column. Adjust the hydrogen and air flow rates to the manufacturer's recommended values, and then readjust the hydrogen flow rate to maximize the sensitivity of the detector. Make sure to obtain a quiet, stable baseline before beginning the determinations.

Attach a pressure regulator to the calibration gas cylinder and connect the outlet of the regulator to the inlet of the sample valve (one mℓ) with a clean metal or Teflon line. Purge the valve thoroughly with calibration gas. Shut off the flow of calibration gas and wait a few seconds for the pressure in the valve to fall to ambient. Turn the valve and inject the sample. Mark the point of injection on the recorder chart, and also write the sample number and sensitivity settings (range and attenuator) on the chart. Keep all peaks on scale with the attenuator, and write down any changes made in the attenuator setting.

Use the first standard run as a check on the vinyl chloride retention time and the instrument sensitivity (Note 1).

Connect the hose bib of a sample bag to the inlet of the sample bag with a short length of clean Teflon tubing. Open the valve on the bag and squeeze it to purge the sample loop. After purging the valve, release the pressure and allow the pressure in the valves to come to ambient. Turn the valve to inject the sample. Vinyl chloride in the sample is identified by retention time comparison with the standard. Begin and end the determinations with standard runs; and, if there are more than six samples, intersperse calibration runs with sample determinations.

After the samples have been run, evacuate the bags, fill with zero nitrogen, knead the bag thoroughly, and evacuate. Repeat this procedure at least three times. The shut-off valve on the bag is closed after the final evacuation, and the bag is again ready for sample collection.

CALCULATION

The area of the vinyl chloride peak is directly proportional to the mass of vinyl chloride injected. For carefully controlled isothermal chromatography, peak heights are proportional to peak areas, and they may be used as a measure of detector response. Peak areas can be measured faster and more accurately than peak areas.

Multiply the measured peak height by the attenuator setting to obtain the corrected peak height. Calculate the concentration of VCM in the air

$$\text{VCM (ppm)} = \frac{Q \times R}{P}$$

where Q = concentration of standard (ppm)

R = corrected peak height for sample

P = corrected peak height for standard.

NOTES

(1) The vinyl chloride retention time and the instrument sensitivity will remain fairly constant from day to day. Any significant change from previous values is a warning signal. A change in the retention time is indicative of an instrumental problem (usually improper column temperature and/or flow or a leaky septum). Identify the problem and correct it before proceeding. A change in sensitivity can be due to an instrument malfunction, a bad standard, or faulty injection technique. Determine the nature of the problem by running a second standard.

(2) Vinyl chloride is flammable, toxic, and a suspected carcinogen. Handle it with due care in a hood.

(3) The concentration of vinyl chloride in the standards and samples should be of the same order of magnitude.

(4) It is permissible and often necessary to change the attenuator setting. However, the range switch setting should not be changed.

(5) Place a suction line near the exit of the sampling valve so that the air passing through the valve is vented into the hood.

PREPARED BY: C. B. Herrin

DATE PREPARED: 3 December 1976