VC SOURCE TEST METHOD BACKGROUND

For stack sampling purposes, vinyl chloride (CH_2 = CHCl, molecular weight 62.50) must be assumed to exist in conjunction with other hydrocarbons, both chlorinated and otherwise. Accordingly, the accepted method for VC (vinyl chloride) analysis has consisted of first separating the VC from other hydrocarbons, followed by measuring the quantity of VC with a flame ionization detector. However, between various groups concerned with measuring VC, non-uniformity in procedures was found to exist in the following areas: (1) sample collection, (2) introduction of sample to gas chromatograph, (3) chromatographic column and associated operating parameters, and (4) chromatograph calibration.

Two of the approaches for VC sample collection involved an evacuated flask for grab samples and tubes containing activated charcoal for an integrated sample. Since it was established that a two-hour average concentration was desired, the integrated sample approach offered a greater advantage over the grab sample approach because emission fluctuations due to process variations would be automatically averaged. In addition, the integrated approach naturally minimizes the number of samples that need to be analyzed. Upon investigating the activated charcoal sampling tubes, it was found that they were basically designed for sampling ambient concentration levels of VC. Since source effluent concentrations are expected to be higher, there was the uncertainty involved with predicting sample breakthrough or when the sample should be terminated. In addition, the amount of charcoal that would be required for some sources would be excessive for a two-hour sampling period.

In view of these deficiencies, it was decided that collection of the sample in Tedlar bags might be a better approach. To check the flexibility of this approach a study of VC stability, or deterioration in Tedlar bags in the presence of various process-associated gases was undertaken.[1] The study showed no significant deterioration of VC over a period of 48 hours. Consequently the integrated bag technique was deemed suitable.

Introduction of a collected gas sample to a gas chromatograph can be done either through use of a gas-tight syringe or an automated sample loop. The latter approach was selected since it has less possibility of leakage and provides a more reproducible sample volume.

Several columns, used by the various groups, are mentioned in the literature as being suitable for the separation of VC from other gases; most notable among them have been Carbopak A[2] and Chromosorb 102.[3] Carbopak A is the more sensitive of the two; however, the VC elution time is not as great as with the Chromosorb 102. For this reason Chromosorb 102 was selected for further study. A program was undertaken to establish whether various hydrocarbons that were known to be associated with VC in stack emissions interferred with the VC peak from the Chromosorb column. The study revealed no such problems.[4] Furthermore, analysis of actual source samples of the peak reported as VC with this column by two qualitative techniques, spectroscopy and electron capture, indicated the peak was only VC.[5] It should be noted that selection of Chromosorb 102 does not mean that Carbopak A or some other column(s) may not work equally well.

Calibration has been accomplished by two techniques, the most common being the dilution of 99+% VC with nitrogen into a series of lower concentrations in Tedlar bags. The second technique utilizes a set of cylinder standards. Both techniques have been found to produce acceptable results and will be included in the reference method.

Based on the study of VC stability in Tedlar bags, possible interferences by various process associated gases, and calibration methods, and as a result of a field study and tests conducted at a source of vinyl chloride, Method 104 was prepared for determining compliance with new source performance standards. This method is essentially the same as used for the data gathering process for setting the standards. Leak tests for the flexible bags and calibration procedures to insure accuracy, precision, and reliability are also specified.

REFERENCES

1. "Evaluation of a Collection and Analytical Procedure for Vinyl Chloride in Air," by G.D. Clayton and Associates. December 13, 1974. EPA Contract No. 68-02-1408, Task Order No. 2. EPA Report No. 75-VCL-1.

2. "Vinyl Chloride Monitoring Near the B.F. Goodrich Chemical Company in Louisville, Kentucky." Region IV, U.S. Environmental Protection Agency, Surveillance and Analysis Division, Athens, Georgia. June 24, 1974.

3. "The Evaluation of Airborne Concentrations of Vinyl Chloride," Richard R. Keenan, G.D. Clayton & Associates, 1974.

4. Same as 1

5. EPA Report No. 75-VCL-2