STANDARD TEST PROCEDURE NO. 555

B.F.GOODRICH CHEMICAL CO.

Gas Chromatographic Analysis of Vinyl Chloride Monomer Using the Hydrogen Flame Ionization Detector

I. SCOPE:

This test is used for measuring the impurities in vinyl chloride monomer and it may also be used for other low boiling liquids.

II. PRINCIPLE:

The principles of gas chromatographic separations are employed utilizing hydrogen flame ionization detection for high sensitivity.

III. SAFETY PRECAUTIONS:

Care should be taken in the handling of vinyl chloride, using adequate ventilation and keeping it from open flames or sources of heat and electrical spark.

IV. APPARATUS:

1. Temperature programmed gas chromatograph with hydrogen flame ionization detector and equipped with a Micro Tek universal sample inlet system.
2. Micro Tek sample cylinders.
3. Micro Tek snap sampler, 0.005 cc (5 micro liter) or smaller when adequate sensitivity can be obtained.
4. Recorder, 0-1 mv range with variable speed chart drive.
5. Freezer for storing samples.

V. PROCEDURE:

A. Instrument Operation.

The operating manual of the instrument manufacturers must be strictly adhered to and the operator should be thoroughly familiar with these manuals prior to attempting to run the equipment.

Utilizing the Beckman GC2A Thermotrac temperature programmer and Hydrogen flame ionization detector with a 3/16" x 22' SS column of 30% Carbowax 1500/ 1% H_3PO_4 on Chromosorb W, 30-60 M, the following conditions should be used

and regarded as critical to a satisfactory analysis:

Air	10 psig	400-450 cc/min.
Hydrogen	10 "	30 "
Helium	28-31	65-70 "

Temperature Programming:

0-5 minutes hold at 75-80° F

5-20 minutes - linear program

to 153-158° F and hold for the duration of the analysis.

The instrument should be kept on at all times, i.e., never shut down over night or over week-ends as stability is lost.

B. Plant Sampling

It is best to take the sample directly into vacuum clean Micro Tek cylinders, however, a technique can be used for transferring from standard cylinders, if necessary. When able, use the same cylinder for each sampling point.

The VCl should flush through the cylinder for one minute at a good rate before closing the valves. The cylinder should be at least 90% filled.

If the sample is not to be run immediately, the full cylinder should be placed in a freezer maintained at 0° C (to prevent loss and/or stratification of low boilers) and stored there until ready to analyze.

It must be realized that the analysis is only as good as the sample and sampling is one of the most important aspects of any chromatographic work. The lower the boiling point of the material and the wider the boiling point range of the various impurities, the more important sampling technique becomes.

C. Introduction of sample into the instrument.

The snap sampler is placed in the sample cylinder and allowed to remain in the liquid with the plunger down for one minute. During this time the chromatograph controls may be set and checked. Pull the plunger up and down a few times. Transfer the full snap sampler to the universal sample inlet and inject the sample as quickly as possible. The analysis is completed in the routine manner for gas chromatography.

VII CALCULATIONS:

The area of each peak is determined and from this the concentration of each component is calculated and reported in ppm. Various materials found in VCl have shown a different response (in terms of peak height and area) to the H.F. detector. It is, therefore, necessary to determine correction factors which will be used in the calculations to obtain correct mol or weight figures.

Written by: J. M. Glenn
Date: May 21, 1962

Checked by: J. M. Whitney

RSV 0005456