STANDARD PROCEDURE.

Plant Springfield **Number** SDP 435-70-7B

Department Control Lab **Sheets** **Page** 1

TITLE Quantitative Determination of Trace Amounts of HCl or NaOH in Vinyl Chloride Monomer

I. SCOPE:

This procedure describes a method for the determination of trace amounts of hydrochloric acid or sodium hydroxide in vinyl chloride monomer. Detections on the amount of less than 0.2 ppm is possible.

II. SIGNIFICANCE:

Hydrochloric acid in vinyl chloride monomer is known to cause rust problems in tank cars and storage tanks. The rust and acid are suspect in difficulties in particle size control and formation of wall scale in kettles. In the case of scrubbed,monomer, sodium hydroxide is sometimes present as a result of carryover from the caustic scrubbers. This method permits the determination of excess amounts of either in the vinyl chloride monomer.

III. SUMMARY:

A sample of vinyl chloride monomer is added to a slightly acidified Ethylene dichloride-thymol blue-methanol solution. This solution is then titrated along with a blank solution to the thymol blue endpoint with standard base. The difference in titer is then calculated to either HCl or NaOH in ppm.

IV. TEST SPECIMENS:

Samples should be delivered to the laboratory in specially designed glass sample containers. These sample containers should be kept in a dry ice chest at all times except when sampling from the container. Each container should be tagged indicating tank car number, date, supplier and tare weight of the container.

V. METHOD:

A. Equipment

1. 500 ml volumetric flasks
2. 500 ml Erlenmeyer flasks, glass stoppered
3. 5 ml microburette, 0.01 ml graduations.

Reference Source of supply: 1. Curtin - Cat. # 2703c
2. Harshaw - " # H-6350
3. Sargent - " # 10935 or 10937

This supersedes / supplements instruction issued on

FORM 126

ISSUED NOV. 1, 1962
STANDARDS DEPARTMENT
BY A.O. Eichsberg

V. METHOD: (Cont'd.)

A. Equipment (Cont'd.)

4. pHydrion paper (From Micro Essential Lab., Brooklyn, N.Y.)
5. Glass or stainless steel sample containers for vinyl chloride monomer. (See Texas City Final Report #34 "Safe Handling of Glass Sample Bomb for VCM Liquid".)

B. Reagents

1. Thymol blue in methanol, 0.1%.
2. Anhydrous methanol (Monsanto methanol satisfactory)
3. Ethylene Dichloride (Neutral)

   Check with pH paper to determine if neutral. If not, wash with water in a separatory funnel until the washings are neutral to pH paper. Dry the EDC over $CaCl_2$ for about 1 hour and filter.

4. Potassium hydroxide, 0.005N in methanol. Standardize with potassiumacid phthalate.

C. Procedure

1. Prepare a supply of solvent mixture as follows:

   a. Place 250 ml (approximately) of neutral ethylene dichloride, 15 ml of 0.1% thymol blue indicator solution and 25 ml of anhydrous methanol in a 500 ml volumetric flask.
   b. Dilute to volume with neutral ethylene dichloride.
   c. If the solvent mixture does not show a bright red color at this point, bubble a very small amount of dry HCl into the solution until it is a bright red color.

   Note: The dry HCl may be obtained by placing the solvent mixture in a clean dry side arm suction flask fitted with a rubber stopper containing a glass tube which will reach below the surface of the mixture. This tube can then be connected to one tube of another two holed rubber stoppered Erlenmeyer flask containing concentrated HCl.

Reference

V. METHOD: (Cont'd.)

C. Procedure (Cont'd.)

1. Note: This tube will extend to just above the HCl liquid level. Another tube should be placed in the stopper for intake of air. The side arm of the suction flask may then be attached to an aspirator or other vacuum source to draw HCl gas into the solvent mixture.

2. Pipette 100 ml of the solvent mixture into each of two 500 ml glass stoppered Erlenmeyer flasks.
3. Add 100 ml of neutral ethylene dichloride to one of the flasks. This is the blank.
4. Stopper and chill both flasks to about - 10°C in an acetone dry ice bath. Also chill a 100 ml graduated cylinder.
5. When the flasks are cooled, measure 100 ml of vinyl chloride monomer into the graduated cylinder and add to the second or sample flask.
   Caution: Observe the usual safety precautions in handling vinyl chloride monomer. Wear face shield and leather palm gloves. Work should be done from this point on down draft hood.
6. Swirl the flask gently to mix.
   Caution: In order to prevent pressure build-up, do not stopper the flask.
7. Titrate both flasks with standard 0.005N KOH in methanol to the normal thymol blue end point (average to bright red) observing the color against a white background.

   Note: The titer for both sample and blank should fall within 0.1 ml to 5.0 ml for greatest accuracy. If the titer is outside the range, the acidity of the solvent mixture prepared under 1 above must be adjusted to this range.

8. When titration is completed, the vinyl chloride should be allowed to evaporate off on the down draft hood.

Reference