TITLE PREPARATION OF VINYL CHLORIDE SAMPLE FOR POLYMERIZATION RATE TEST

I. SCOPE

This method describes the laboratory procedure for removing phenol inhibitor from liquid vinyl chloride by scrubbing with a solution of sodium hydroxide. The uninhibited monomer is then tested to determine the rate of polymerization.

II. THEORY

Phenol can be removed completely from liquid vinyl chloride at atmospheric pressure with a 20% sodium hydroxide solution, provided that the temperature is maintained below the boiling point of vinyl chloride (-13.7°C.). The sodium hydroxide solution does not freeze at -30°C. This method avoids the loss of those non-volatile impurities which are not removed in our plant caustic scrubber and which might be lost by any method involving flashing or distillation of the monomer.

III. TEST SPECIMEN

The procedure for removing a sample of vinyl chloride from a tank car is described in SDP-112-70. The sampling bomb containing the vinyl chloride will be delivered to the Control Laboratory and shall be properly identified as follows:

A. Date
B. Tank Car Number
C. Material Being Tested

IV. METHOD

A. Reagents

1. Distilled water
2. Sodium hydroxide pellets (C.P. Grade)
3. Ethylene glycol (60)-water (40) solution

B. Apparatus

1. Pyrex dish, flat-bottomed (diam. 7-1/2", height 4") or other suitable cooling bath.

2. Stirring motor-variable speed.

Reference 3. Turbo-agitator, stainless steel, 4-blade (length of shaft 8", blade diam. 1-3/4" - approx.).

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IV. METHOD (Cont'd.)

B. Apparatus (Cont'd.)

4. Large Dewar flask or other container partially filled with dry ice.

5. Thermometer, +50 to -50°C.

6. Beaker, one-liter.

7. Erlenmeyer flask, 250 cc and 500 cc.

C. Procedure

1. Prepare a solution of 50 gm. of sodium hydroxide in 200 ml. of distilled water in a 1-liter beaker. Cool to room temperature.

2. Set beaker in the cooling bath and adjust temperature of the bath and the caustic solution to minus 25°C. ± 5°C.

3. Chill the vinyl chloride sampling bomb in a dry ice chest, and transfer approximately 300 cc of monomer to a 500 cc Erlenmeyer Flask which has been previously cooled by dry ice.

4. Allow the vinyl chloride to warm up to -25°C. ± 5°C. and transfer 250 cc of monomer to the beaker containing caustic at -25°C.

5. Agitate the mixture vigorously for ten minutes. The agitation must be vigorous enough to give a finely divided homogeneous dispersion.

6. Shut off the stirring motor and remove the agitator from the mixture. Allow the vinyl chloride and caustic solution to separate.

7. Decant about 150 cc of the vinyl chloride into a 250 cc Erlenmeyer that has been previously chilled by dry ice. Do not allow any caustic solution to carry over.

8. Keep the monomer at dry ice temperature until it is to be used in the Polymerization Rate Test.

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V. PRECAUTIONS

1. The vinyl chloride must be below its boiling point at all times while handling and scrubbing.

2. The scrubbed monomer may be stored for 24 hours before proceeding with the rate test provided that the temperature is kept below -25°C.

3. If there is any doubt that the phenol has not been removed completely from the vinyl chloride, the phenol content may be determined by the method described in SDP-112-70-10.

VI. TIME

The charge will be 10 units per test.

Reference F. J. Lucht - 11/2/50

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