Monsanto Chemical Company
PLASTICS DIVISION

STANDARD PROCEDURE. **Numb r** 508-70-5A

D partment EAST CONTROL LABORATORY 6 **Sheets.** **Pag** 1

TITLE DETERMINATION OF ALDEHYDES IN VINYL CHLORIDE

I. SCOPE

This test is applicable for determination of aldehyde concentration in Vinyl Chloride as acetaldehyde.

II. THEORETICAL

This test depends upon extraction of the aldehyde with Ammonium Hydroxide and the formation of a water insoluble compound with α-methyl indole. The turbidity of this compound in water suspension can be determined as a measure of the amount of aldehyde originally present.

III. TEST SPECIMENS

Samples of Vinyl Chloride Monomer will be delivered to the Control Laboratory in approved type sample bombs, adequately identified as to:

A. Date
B. Material
C. Lot, Tank, or Batch
D. Tests Required

IV. METHOD

A. Equipment

1. Betz-Hellige Turbidimeter (Fig. II), with
   a. 10 mm. liquid depth Turbidimeter Tube (8012-E).
   b. Glass Plunger (8015).
2. 100 ml. Volumetric Flask.
3. Coca-Cola Bottles and Caps.
4. "Wrist Action" Shaker with guard.
5. 100 ml. Graduated Cylinder.
6. Sharp Pointed Instrument (Ice Pick).

Reference

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RSV0026806

IV. METHOD

A. Equipment (Cont'd.)

7. 2 ml. Graduated Pipette.

8. Triple Beam Balance.

9. Other Ordinary Laboratory Equipment.

B. Reagents

1. α-Methyl Indole Solution.

a. Add 60 ml. concentrated cp HCl to 30 ml. of distilled water in a 250 ml. Erlenmeyer Flask, then dissolve in this, 0.15 $\pm$ 0.05 grams α-methyl indole.

2. Ammonium Hydroxide, c.p. conc.

3. Acetaldehyde - Technical.

C. Procedure

1. Preparation of Samples

a. Pipette 1.0 ml. of conc. NH_4OH, into an 8 oz. bottle.

b. Weigh the bottle and add weights equal to 100 grams plus the weight of the bottle and NH_4OH.

c. Pour approximately 105 grams of Vinyl Chloride into the bottle from the cold sample bomb.

d. Allow the Vinyl Chloride to evaporate until the weights are just balanced by the remainder (100 grams of sample). Quickly cap the bottle.

NOTE: This must be done on the fume exhaust bench.

e. Allow the sealed bottle to come to room temperature then place on the Wrist Shaker and shake vigorously and continuously for 1 hour.

Reference

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IV. METHOD (Cont'd.)

C. Procedure (Cont'd.)

NOTE: Be sure the guard is in place over the shaker.

f. Remove from the shaker and cautiously puncture the bottle cap with an ice pick. Allow the monomer to evaporate slowly on the fume exhaust bench.

b. When all the Vinyl Chloride has evaporated, wash the contents of the bottle into a 100 ml. volumetric flask with several portions of distilled water. The total volume of distilled water shall not exceed 50 ml.

h. To the washings add 20 ml. of the α-methyl indole test solution. Shake to mix thoroughly.

i. Allow the solution to stand for 10 minutes.

j. Dilute to 100 ml. with distilled water. Shake to mix thoroughly.

2. Testing of Sample

a. Fill the clean Helliger Turbidimeter (8012-E 10 mm. liquid depth) to the mark with a portion of the test solution.

b. Close the tube with the glass plunger (8015).

c. Open the door on the front of the instrument and place the sample on the frame of the mirror in the circular groove provided for it.

d. Close the door, plug the cord into an electrical outlet, and turn the light switch.

e. Observe the sample on the eyepiece provided on the top of the instrument. By revolving the dial on the right side of the instrument, regulate the brightness of the spot in the center of the observation field so that it matches the surrounding area.

Reference

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IV. METHOD (Cont'd.)

C. Procedure (Cont'd.)

2. Testing of Sample (Cont'd.)

NOTE: To obtain a reproducible reading, always rotate the dial towards the higher value (i.e. turn the dial counter clockwise starting at zero) until the black spot in the center of the field just diaappears.

f. When the proper adjustment has been made, read the scale on the dial.

g. Read the aldehyde concentration in parts per million from a graph similar to the attached.

h. If the turbidity exceeds the range of the graph, the final solution may be diluted with distilled water and the calculation for aldehydes corrected for this dilution. For example, if the solution were diluted to three times its original volume, the value taken from the graph would be multiplied by three.

V. CALCULATION

The results are read directly from the attached graph. (See Fig. 1).

VI. REPORT

Report to the nearest part per million, the average of two determinations.

VII. PRECAUTIONS

A. Safety

1. Vinyl Chloride liquid has a high co-efficient of expansion and can create dangerous pressure if allowed to become warm in a closed container with no vapor space present.

Reference

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VII. PRECAUTIONS (Cont'd.)

A. Safety (Cont'd.)

2. Vinyl Chloride has a low boiling point (-13.9°C) so that moderate heating of its vapors may cause high pressure if they are not allowed to escape.

3. Because the monomer is kept cold and also has a cooling effect on evaporation, care must be taken to avoid contact with the body. Always wear safety goggles when handling the monomer and gloves when handling the cold sample bomb.

4. Liquid Vinyl Chloride and its vapor are highly inflammable. In addition, the vapors have anesthetic properties. Therefore, the determination and handling must be carried on in an area where there is adequate ventilation and no danger from flame or spark.

5. The analyst must wear a heavy rubber apron, heavy long gloves, and a face shield when working with Vinyl Chloride in sealed bottles.

B. Technique

1. The α-methyl indole solution must be made fresh each day.

2. Cleanliness of equipment is of utmost importance. Glass tube and plunger are best cleaned by scrubbing with soap solution. Strongly acid or alkaline cleaning mixtures should not be used. Rinse cell with clean water and a portion of the sample rather than cloth drying.

3. The cemented joints of the cell are not intended to withstand mechanical strain. Care should be taken to support the bottom of the tube or plunger whenever undue pressure is applied to them.

4. The mirror must be carefully handled to protect surface. Use only a camel's hair brush or a cotton swab to clean center aperture.

Reference

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VII. PRECAUTIONS (Cont'd.)

B. Technique (Cont'd.)

5. Restrict the length of the burning time of the bulb to the actual time required, for making the adjustments on reading. This will greatly extend the life of the bulb.

6. New charts must be made up for each instrument and for each bulb used. Be sure the chart you use is for the bulb in service.

7. The operation of filling the tube, inserting in the apparatus and reading, should be done as quickly as possible, in order to prevent the settling of particles on the bottom of the tube.

8. Bubbles at the bottom of the tube or at the lower surface of the plunger show as luminous particles. It is necessary that they be eliminated. Air bubbles adhering to the plunger can easily be removed by raising the plunger. Air bubbles on plunger bottom are best avoided by inserting the plunger one side first.

9. Always use the purest available chemicals.

VIII. ACCURACY AND PRECISION

This method is accurate to within $\pm$ 1 ppm of acetaldehyde in the Vinyl Chloride sample. Duplicate determinations should check within 2 ppm of each other.

IX. TIME

A. The overall time for this test is approximately 4 hours.

B. The charge unit value is 5 units per test.

Reference W. A. HAMILTON - January, 1951
RESEARCH TENTATIVE PROCESS "SUSPENSION POLYMERIZATION OF VINYL CHLORIDE" 7/13/48

ISSUED MAR 14 1951
STANDARDS DEPARTMENT
By E. H. Brown

This supersedes ~~supplements~~ instruction issued on 2/9/49