



IPC-TM-650 TEST METHODS MANUAL

1 Scope This test is a method for determining acid acceptance of chlorinated solvents.

Acid acceptance is applicable for evaluating the condition of the following inhibited chlorinated solvents.

- Perchloroethylene
- 1,1,1-Trichloroethane
- Methylene Chloride
- Trichloroethylene

2 Applicable Documents

ASTM D2111-95 Standard Test Methods for Specific Gravity of Halogenated Organic Solvents and Their Admixtures

3 Test Specimen

3.1 25 ml of solvent (see 5.1 for sampling procedure)

4 Apparatus

4.1 One flask, Erlenmeyer, 100 ml, with glass stoppers (see 6.5)

4.2 Two flasks, Erlenmeyer, 250 ml, with glass stoppers (see 6.5)

4.3 Two flasks, Volumetric, 1000 ml, with glass stoppers (see 6.5)

4.4 Two pipets, 25 ml, with bulb (see 6.5)

4.5 One pipet, graduated (calibrated), with bulb (see 6.5)

4.6 One eye dropper (see 6.5)

4.7 One balance, analytical (see 6.5)

4.8 Two pieces of weighing paper (see 6.5)

4.9 Sodium hydroxide 0.1 N in C.P. anhydrous methanol (5.2.1 & 6.5)

Number 2.3.36	
Subject Acid Acceptance of Chlorinated Solvents	
Date 10/85	Revision
Originating Task Group N/A	

4.10 Hydrochlorination reagent, 0.1 N Hydrochloric acid in anhydrous isopropanol (see 5.2.2 & 6.5)

4.11 Bromothymol blue indicator, 0.1% solution

Note: Make sure all glassware is clean and dry.

5 Procedure

5.1 Sampling Procedure

5.1.1 Securely tie a copper wire around the neck of a 100 ml Erlenmeyer flask. Carefully lower, allow to fill, stopper, and store in cool, dark place.

5.1.2 Since a representative sample is desired, sampling should be done when the tank is half full. Most tanks (or hoses from them) have sampling points. From here, fill a 100 ml Erlenmeyer flask, stopper, and store in a cool, dark place.

5.1.3 See 6.3 for safety and handling and 6.4 for first aid.

5.2 Reagent Preparation

5.2.1 Sodium Hydroxide Solution Preparation

5.2.1.1 Weigh 4.0 g reagent grade NaOH.

5.2.1.2 Pour approximately 500 ml C.P. anhydrous methanol into a 1 liter volumetric flask.

5.2.1.3 Add NaOH and swirl until dissolved.

5.2.1.4 Dilute solution to exactly 1 liter with methanol and stopper. See 6.3 for safety and handling information and 6.4 for first aid information.

5.2.2 Hydrochlorination Reagent Preparation

5.2.2.1 Pour about 500 ml anhydrous isopropanol into a 1 liter volumetric flask.

5.2.2.2 Bulb pipet exactly 8.50 ml 37% HCl into the flask, then swirl.

IPC-TM-650		
Number 2.3.36	Subject Acid Acceptance of Chlorinated Solvents	Date 10/85
Revision		

5.2.2.3 Dilute solution to exactly 1 liter and stopper. See 6.3 for safety and handling information and 6.4 for first aid information.

5.3 Test Procedure

5.3.1 Measure specific gravity of sample using ASTM Method D 2111-95.

5.3.2 Bulb pipet exactly 25 ml sample into a 250 ml Erlenmeyer flask.

5.3.3 Bulb pipet exactly 25 ml hydrochlorination reagent into sample, stopper, then mix thoroughly.

5.3.4 Let sit 15 to 20 minutes for complete reaction. During reaction time, a blank may be run (see 5.3).

5.3.5 When reaction time is complete, add five drops of bromothymol blue 0.1% solution with an eye dropper.

5.3.6 Titrate the sample to endpoint with 0.1 N NaOH solution. The endpoint is reached when solution color changes from yellow to blue.

5.4 Blank

5.4.1 Bulb pipet exactly 25 ml hydrochlorination reagent into a 250 ml Erlenmeyer flask.

5.4.2 Add five drops bromothymol blue 0.1% solution with an eye dropper.

5.4.3 Titrate blank to endpoint with 0.1 N NaOH solution. The endpoint is reached when solution color changes from yellow to blue.

5.5 Disposal of Solutions

5.5.1 0.1 N NaOH in Methanol, Unused This solution is dilute and readily soluble in water, hence it can be safely poured down the sink. Flush with excess water.

5.5.2 All Other Solutions DO NOT POUR DOWN THE SINK. Place the solutions in appropriate, labeled, metal con-

tainers to be buried or burned in accordance with local, state, and federal regulations.

5.6 Cleaning Glassware Use soap and water to clean any glassware. Rinse well with excess water.

5.7 Safety and Handling, First Aid See 6.3 and 6.4.

6 Notes

6.1 Calculations:

$$\frac{(B-A) \times 0.4}{\text{ml sample} \times \text{specific gravity (1)}} = \text{Total Acid Acceptance as \% of NaOH}$$

where:

A = ml of 0.1 N NaOH used to titrate sample

B = ml of 0.1 N NaOH used to titrate blank

(1) See sale specification for specific gravity of solvent choice.

6.2 Interpreting Test Results*

Chlorinated Solvent	Within Normal Range	Solvent is Borderline Check Daily	A) Discard B) Remove, distill blend with virgin solvent at 4 parts virgin solvent, 1 part distilled. <0.04%
Methylene Chloride	>0.08%	0.04%-0.08%	
1,1,1-Trichloroethane			
Perchloroethylene	>0.04%	≤0.04%	

*Note: These guidelines have been established using inhibited chlorinated solvents from the Dow Chemical Company. The solvent manufacturer should be consulted for particular interpretation.

6.3 Safety and Handling

6.3.1 Sampling Procedure Wear safety goggles and neoprene or neoprene-coated gloves. Have adequate ventilation. Avoid sparks or flames.

6.3.2 Laboratory Procedure There are no unusual hazards inherent in this method, but good ventilation and normal laboratory safety precautions should be employed. When preparing reagents, mixing should be employed in a hood and chemical goggles should be worn. Keep away from any sparks or flames.

Number 2.3.36	Subject Acid Acceptance of Chlorinated Solvents	Date 10/85
Revision		

OSHA recommended exposure limits for chlorinated solvents (all in parts per million).

Chlorinated Solvent	OSHA	ACGIH	ACC ³	AMP ⁴
	8 Hr TWA ¹			
Perchloroethylene	100	50	200	300/5 min. in any 3 hrs.
Trichloroethylene	100	50	200	200/5 min. in any 2 hrs.
1,1,1-Trichloroethane	350	350	450 ^a	
Methylene Chloride	500	100	1000	2000/5 min. in any 2 hrs.

a. No limits established by OSHA. ACGIH recommends a short term exposure limit (STEL) of 450 ppm.

(1) TWA—Time Weighted Average

(2) ACGIH—American Conference of Governmental Hygienists

TLV—Threshold limit values from ACGIH Handbook, 83/84

(3) ACC—Acceptable Ceiling Concentration

(4) AMP—Acceptable Maximum Peak

6.4 First Aid

6.4.1 Eye Contact Rinse eyes with running water for at least 30 minutes. Prompt medical attention is essential.

6.4.2 Skin Contact Flush the area thoroughly with water. Wash clothing before reusing it.

6.4.3 Oral Ingestion DO NOT INDUCE VOMITING. Call a physician or transport the worker to an emergency facility.

6.4.4 Inhalation Remove the worker to fresh air if the worker starts feeling dizzy or light-headed. Contact a physician or transport the worker to a medical facility. If breathing stops, give mouth-to-mouth resuscitation. Administer oxygen when breathing starts (see 6.3.2 for chlorinated solvent exposure limits).

6.4.5 Additional Help For any additional chemical related medical help, call Chemtree Emergency at 800) 424-9000. In Canada call (collect) Canutel (613) 996-6666.

6.5 All equipment and chemicals used were purchased from Van Waters and Rogers standard lab supply catalog.

6.6 This test is designed for the base chlorocarbons only; therefore, discretion should be used when testing bipolar solvents.

6.7 Specific Questions Any specific questions can be referred to Dow Chemical U.S.A., Inorganic Chemicals Department, Technical Service and Development, Midland, Mich. 48640.