

# HYDROCHLORIC ACID

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**SYNONYMS** Muriatic acid, INS No. 507

## DEFINITION

Chemical names Hydrochloric acid

C.A.S. number 7647-01-0

Chemical formula HCl

Formula weight 36.46

Assay Not less than 97.0% and not more than 103.0% of the labelled amount

**DESCRIPTION** Clear colourless or slightly yellowish liquid with a pungent odour. Various concentrations are supplied as products of commerce.

**FUNCTIONAL USES** Acid

## CHARACTERISTICS

### IDENTIFICATION

Solubility (Vol. 4) Soluble in water and in ethanol

Test for acid A 1 in 100 solution of the sample is acid to litmus paper

Test for chloride (Vol. 4) Passes test

### PURITY

Non-volatile residue Not more than 0.5%  
Transfer 1 g into a tared glass dish, evaporate to dryness on a steam bath, dry at 110° for 1 h, cool in a desiccator and weigh. The weight of the residue does not exceed 5 mg.

Reducing substances Not more than 70 mg/kg as sulfur dioxide ; Transfer 1 ml of reagent grade hydrochloric acid into a 30-ml test tube, dilute to 20 ml with freshly boiled and cooled water, and add 1 ml of potassium iodide TS, 1 ml of starch TS and 2 ml of 0.001N iodine. Stopper the test tube and mix thoroughly. The blue colour produced is not discharged by 1 ml of the sample.

Oxidizing substances Not more than 30 mg/kg as chlorine  
Transfer 1 ml of the sample into a 30-ml test tube, dilute to 20 ml with freshly boiled and cooled water, and add 1 ml of potassium iodide TS and 1 ml of starch TS. Stopper the test tube and mix thoroughly. The intensity of any blue colour developed does not exceed that produced in a control

prepared similarly but containing 1 ml of 0.001N iodine (instead of potassium iodide TS) and 1 ml of reagent grade concentrated hydrochloric acid (instead of sample).

Sulfate

Not more than 0.5%

Dilute 1 g of the sample to 100 ml with water, transfer 5 ml of this dilution to a 50-ml tall-form Nessler tube and dilute to 20 ml with water. Add a drop of phenolphthalein TS, neutralize the solution with ammonia TS, and then add 1 ml of hydrochloric acid TS prepared from reagent grade hydrochloric acid. To the clear solution (filtered if necessary) add 3 ml of barium chloride TS, dilute to 50 ml with water and mix. Prepare a control consisting of 1 ml of reagent grade concentrated hydrochloric acid and 250 µg of sulfate (SO<sub>4</sub>) and the same quantities of the reagents as used for the sample. Any turbidity shown in the sample does not exceed that in the control.

Total organic compounds Total organic compounds (non-fluorine): Not more than 5 mg/kg  
Benzene: Not more than 0.05 mg/kg  
Fluorinated organic compounds (total): Not more than 25 mg/kg  
See description under TESTS

Iron

Not more than 5 mg/kg

Dilute 5 g (4.3 ml) of the sample to 40 ml and add about 40 mg of ammonium persulfate and 10 ml of ammonium thiocyanate TS. Any red colour developed does not exceed that in a control prepared by mixing 2.5 ml of Iron standard solution in an equal volume of a solution containing the same quantities of reagent grade hydrochloric acid and the reagents as used in the test.

Lead (Vol. 4)

Not more than 1 mg/kg

Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

## TESTS

### PURITY TESTS

Organic compounds

Carry out analyses by gas chromatography employing Vapour Partitioning or Solvent Extraction, depending upon the characteristics of the compound being determined. It is necessary, however, to use the Vapour Partitioning method for the determination of benzene.

Vapour Partitioning Method

This method is suitable for the determination of extractable organic compounds at 0.05 to 100 mg/kg but is most appropriate for organic compounds with a vapour pressure greater than 10 mm Hg at 25°. Use a gas chromatograph equipped with a flame ionization detector and a 4-m x 2-mm (id) stainless-steel column packed with 15%, by weight, methyl trifluoropropyl silicone (DCFS 1265, or QF-1, or OV-210, or SP-2401) stationary phase on 80/100 mesh Gas Chrom R or the equivalent. A newly packed column should be conditioned at 120° and 30 ml/min helium flow for at least 2 h (preferably overnight) before it is attached to the detector. For

analysis, the column is maintained isothermally at 105°, the injection port and detector are maintained at 250°, the carrier gas flow rate is set at 11 ml/min, fuel gas flows should be optimized for the gas chromatograph and detector in use. The experimental conditions may be changed as necessary for optimal resolution and sensitivity. The signal-to-noise ratio should be at least 10:1.

#### Preparation of Standard Solutions

Prepare a standard solution of the organic compounds to be quantitated in Hydrochloric Acid (known to be free of interfering impurities) at approximate concentrations of 5 mg/kg, or within ±50% of the concentrations in the samples to be analyzed.

Place a stirring bar in a one-litre volumetric flask equipped with a ground-glass stopper, and tare the combination. Fill the flask with reagent-grade hydrochloric acid so that no air space is present when the flask is stoppered, and determine the weight of the Hydrochloric Acid. Calculate the volume (V) in 1l of each organic component to be added from the formula

$$V = (C \times W)/(D \times 1000)$$

where C is the desired concentration, in mg/kg; W is weight, in g, of the Hydrochloric Acid; D is the density, in mg/1l, of the organic compound; and 1000 is a conversion factor with the units g/kg. Add the calculated amount of each component to the Hydrochloric Acid with a syringe (ensure that the syringe tip is under the solution surface), stopper the flask, and stir the solution for at least 2 h using a magnetic stirrer.

#### Calibration

Treat the standard in the same way as described for the sample under Procedure (below). Determine a blank for each lot of reagent-grade Hydrochloric Acid, and calculate a response factor (R) by dividing the concentration (C) in mg/kg for each component by the peak area (A) for that component (subtract any area obtained from the blank sample):  
 $R = C/(A - \text{area of blank})$

Gaseous compounds present special problems in the preparation of standards. Therefore, to determine response factors for gaseous compounds use the following method, which will be referred to as the Method of Multiple Extractions. Dilute a sample of Hydrochloric Acid known to contain the gaseous compound of interest with an equal volume of water. Draw 20 ml of this solution into a 50-ml glass syringe; then draw 20 ml of air into the syringe, cap with a rubber septum, and place the syringe on a shaker for 5 min. Withdraw 1 ml of the vapour through the septum, and inject it into the chromatograph. Expel the vapour phase from the 50-ml syringe, draw in another 20 ml of air, repeat the extraction, and inject another 1-ml vapour sample into the gas chromatograph. Repeat the extraction, and GC analysis on the same sample of acid a total of six times. For each impurity, plot the area ( $A_N$ ) determined for extraction N against the difference between  $A_N$  and the area determined for extraction (N + 1); that is, plot  $A_N$  against  $[A_N - A_{N+1}]$ . The slope of this line is the extraction efficiency (E) for that impurity into the air.

Inject into the chromatograph 1 ml of 0.1% (by volume) standard gas sample of each impurity in air and determine the absolute factor ( $F_A$ ) in g, per peak area (A) by the following formula:

$$F_A = (M \times 4.0816 \times 10^{-8})/A,$$

where M is the molecular weight of the compound.

The concentration (C), in mg/kg, of the component in the original sample is calculated by the formula

$$C = (A \times F_A \times 1.6949 \times 10^6)/E.$$

where A is the peak area corresponding to the compound (as above),  $F_A$  is the absolute factor, and E is extraction efficiency. The response factor is then calculated as  $R = C/A$

### Procedure

Dilute a 10-ml sample of Hydrochloric Acid to be analyzed with an equal volume of water. Draw this solution into a 50-ml glass syringe. Then draw 20 ml of air into the syringe, cap with a rubber septum, and place the syringe on a shaker for 5 min. Draw 1 ml of the vapour through the septum, and inject it into the gas chromatograph. Approximate elution times in min for some specific organic compounds are as follows:

Methane and acetylene: 1.70  
Methyl chloride: 2.21  
Vinyl chloride: 2.29  
1,1,1-Trichlorofluoromethane: 2.62  
Ethyl chloride: 2.90  
Vinylidene chloride: 3.20  
Methylene chloride: 3.64  
Chloroform: 4.49  
1,1-Dichloroethane: 4.53  
Carbon tetrachloride: 4.86  
1,1,1-Trichloroethane: 5.50  
Benzene: 6.00  
Trichloroethylene: 6.22  
Ethylene dichloride: 6.61  
Propylenedichloride: 8.41  
Perchloroethylene: 9.73

Alternate columns may be required to resolve some combinations of components. Methyl chloride and vinyl chloride are resolved by a 3.7-m x 3-mm (id) squalene column at 45° and a helium flow of 10 ml/min. Chloroform and 1,1-dichloroethane are resolved by a 4-m x 3-mm (id) DC 550R column at 110° and a helium flow of 12 ml/min.

### Calculation

Calculate the concentration (C) in mg/kg of each compound by multiplying its corresponding peak area (A) by the appropriate response factor (R) determined in the Calibration protocol:

$$C = R \times A$$

### Precision

The relative standard deviation at 5 mg/kg should not exceed 15% for five analyses.

#### Solvent Extraction Method

The solvent extraction technique is suitable for the determination of extractable organic compounds at 0.3 to 100 mg/kg, but is most appropriate for organic compounds with vapour pressures less than 10 mm Hg at 25°. The conditions for the gas chromatograph are the same as for the Vapour Partitioning method, except that the column temperature is 120°, and the carrier-gas flow is 21 ml/min.

#### Preparation of Standards

Prepare the Standard Solution as described under Vapour Partitioning.

#### Calibration

Extract a sample of the Standard Solution as directed under Procedure (below) and inject it into the gas chromatograph. Determine a blank for each lot of reagent-grade Hydrochloric Acid and perchloroethylene by extracting the Hydrochloric Acid in the same way as the standard. Calculate a response factor (R) by dividing the concentration (C) in mg/kg for each component by the peak area (A) for that component (subtract any area obtained from the blank sample):

$$R = C/(A - \text{area of blank})$$

#### Procedure

Accurately transfer 90 ml of the Hydrochloric Acid sample and 10 ml of perchloroethylene (free of interfering impurities) into a narrow-mouth, 125-ml bottle. Place the bottle in a mechanical shaker for 30 min. Separate the two phases (perchloroethylene on the bottom) and inject 3 µl of the perchloroethylene extract into the gas chromatograph. Approximate elution times in min for some chlorinated organic compounds are as follows:

Vinylidene chloride: 2.94  
Methylene chloride: 3.27  
Chloroform: 3.83  
Carbon tetrachloride: 4.07  
1,1,1-Trichloroethane: 4.50  
Trichloroethylene: 4.97  
Ethylene dichloride: 5.26  
Propylene dichloride: 6.36  
Perchloroethylene: 6.95  
1,1,1,2-Tetrachloroethane: 10.12  
1,1,2,2-Tetrachloroethane: 13.70  
Pentachloroethane: 16.19

To determine perchloroethylene and higher-boiling impurities, substitute methylene chloride (free of interfering impurities) for perchloroethylene in the extraction step. For higher-boiling impurities such as monochlorobenzene and the three dichloro-benzenes, use a 2.74-m x 2.1-mm (id) stainless steel column packed with 10% Carbowax 20M/20% KOH on 80/100 mesh Chromasorb W (acid washed) at 150° and a nitrogen flow of 35 ml/min.

#### Calculation

Calculate the concentration (C), in mg/kg, of each compound by multiplying the corresponding peak area (A) (subtracted any area obtained from the blank sample) by the appropriate response factor (R) determined in the Calibration protocol:

$$C = R \times (A - \text{area of blank})$$

#### Precision

The relative standard deviation at 5 mg/kg should not exceed 15% for five analyses.

### **METHOD OF ASSAY**

Tare accurately a 125-ml glass-stoppered conical flask containing 50 ml of 1N sodium hydroxide. Partially fill, without the use of vacuum, a 10-ml serological pipet from near the bottom of a representative sample, remove any acid adhering to the outside and discard the first ml flowing from the pipet. Hold the tip of the pipet just above the surface of the sodium hydroxide solution, and transfer between 2.5 and 3 ml of the sample into the flask, mix the contents, and weigh accurately to obtain the weight of the sample. Add methyl orange TS and titrate the excess of sodium hydroxide with 1N hydrochloric acid. Each ml of 1N sodium hydroxide is equivalent to 36.46 mg of HCl.