



Trichloroisocyanuric Acid and
Sodium dichloroisocyanuric acid

Method number: ID-101-SG

Collection Procedure: A known volume of air is drawn through a glass fiber filter with an entrained impinger. The filter is used for the collection of particulate Trichloroisocyanuric acid or Sodium dichloroisocyanuric acid with the impinger, containing 10 mL of 0.1% Sulfamic acid, used to collect chlorine gas.

Recommended air
volume and sampling rate: 240 Liters at 1.0 Lpm (240 minutes)

Procedure: An aliquot of the sample is reacted with acidic Potassium iodide. Trichloroisocyanuric acid and Sodium dichloroisocyanuric acid react with the Potassium iodide to form iodine which is measured with an ion specific electrode. Chlorine gas is analyzed separately by following method ID-101.

Detection Limit: 0.4 mg/m³ with a 15-liter air volume.

Status of Method: Partially validated method. This method has been partially evaluated and is presented for information and trial use only.

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Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.

1 Introduction

1.1 Scope

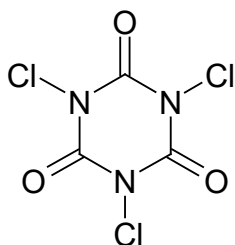
This method describes the collection and analysis of airborne Trichloroisocyanuric acid (TCCA) and Sodium dichloroisocyanuric acid (DCCA). The analysis is based on an Iodometric technique utilizing an ion specific electrode.

1.2 Synonyms

Trichloroisocyanuric acid, CAS#: 87-90-1

1,3,5,-Trichloro-s-triazine-2,4,6(1H,3H,5H)-trione; ACL-85; Fichlor 91; Trichloroisocyanurate; Symclosen

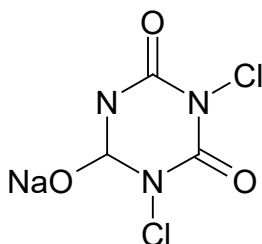
Structure:



Sodium Dichloroisocyanuric acid, CAS#: 2893-78-9

ACL-60; s-Triazine-2,4,6(1H,3H,5H)-trione, dichloro, sodium salt; SDIC; Sodium salt of dichloro-s-tria-zinetriene

Structure:



1.3 Uses

Trichloroisocyanuric acid and Dichloroisocyanuric acid are used as a source of available chlorine in "dry type" bleaches, scouring powders, dishwashing compounds, and sanitizing compounds.

1.4 Physical and Chemical Properties

The physical and chemical properties of trichloro and dichloroisocyanuric acids are:

- 1.4.1 **Trichloroisocyanuric acid** – $C_3Cl_3N_3O_3$; Mole. wt. = 232.41; C = 15.50%, Cl = 45.77%, N = 18.08%, O = 20.65%. Prepared by chlorinating cyanuric acid in 5% NaOH solution: Hands, Whitt., Soc. Chem. Ind. 67,66 (1948); Hardy U.S. pat. 2,607,738 (1952 to Monsanto). Purification by dissolving in conc. H_2SO_4 and diluting with ice water: Lorenz, U.S. pat. 2,828,308 (1958 to Purex Corp.). May be stored in the dry state for at least a year. Releases hypochlorous acid on contact with water. Available chlorine about 90%. pH of aqueous soln. about 4.4. Solubility in water at 25 °C about 0.2%. Soluble in chlorinated and highly polar solvents.
- 1.4.2 **Sodium dichloroisocyanuric acid** – $C_3HCl_2N_3O_3Na$; Mole. wt. = 220.96. C = 15.26%, Cl = 30.04%, Na = 16.56%, N = 17.80%, O = 20.33%. Structure studies and preparation: Petterson et. al., J. Org. Chem. 25, 1595 (1960). Preparation from trisodium isocyanurate and gaseous Cl: Symes et. al., U.S. pats. 3,035,056 and 3,035,057 (1962 to Monsanto).

2 Range and Detection Limit

A lower analytical limit of 0.1 mg/mL, was selected for routine analysis.

3 Precision and Accuracy:

Within the operating range of the electrode, reproducibility is reported to be independent of concentration. With calibration every two hours, electrode measurements can be obtained which are reproducible to 2%.

4 Interferences

Strong oxidizing agents including iodate, bromine, cupric ion and manganese dioxide reportedly interfere with the analysis. It is also reported that silver and mercuric ion concentrations in the sample must be below 10 to 20 ppm or they will also interfere with the analysis.

5 Collection and Shipping of Samples

- 5.1 Assemble the filter in the three-piece filter cassette holder and close firmly to insure that the center ring seals the edge of the filter. The glass fiber filter is supported with a second glass fiber filter, if necessary, and the filter holder is held together by plastic tape or a shrinkable cellulose band. If the filter does not lie flat and the spacer ring does not fit snugly into the bottom of the filter holder, sample leakage will occur around the filter. Remove the cassette plugs. Use pieces of flexible tubing to connect the filter holder to the impinger and the impinger to the pump. Fill the impinger with 10 mL of 0.1% Sulfamic acid as in the procedure for Chlorine (OSHA method ID-101).
- 5.2 Clip the collection devices to the worker's lapel. Air being sampled should not be passed through any hose or tubing before entering the filter cassette.
- 5.3 A sample size of 240 liters is recommended. Sample at a flow rate of 1.0 liter per minute for 4 hours. The flow rate should be known with an accuracy of at least $\pm 5\%$ with the sampling device in-line.
- 5.4 Turn the pump on and begin sample collection. Since it is possible for filters to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, the pump rotameter should be observed frequently, and the sampling should be terminated at any evidence of a problem.

- 5.5 Terminate sampling at the predetermined time and note sample flow rate, collection time, and ambient temperature and pressure. If a pressure reading is unavailable, record the elevation. Replace the filter plugs.
- 5.6 Transfer the solutions in the midjet glass impingers to 20-mL scintillation vials which have caps with PTFE liners. Cap the vials and seal the caps with tape. Wrap the tape around the cap in the direction the cap is turned.
- 5.7 Blank. With each batch of ten samples, submit one filter from the same lot of filters used for sample collection. Filter should be subjected to the same handling as for the samples except that no air is drawn through it. Also, submit a sample of the unused 0.1% Sulfamic acid. Label these as blanks.
- 5.8 The sample vials and cassettes are sealed with a Form OSHA-21 seals.
- 5.9 The sample vials and cassettes are packed and shipped to the laboratory for analysis as soon as possible.
- 6 Analytical Procedure
- 6.1 Apparatus
- 6.1.1 Equipment for sample collection
- Glass fiber filters, Gelman A/E, 37-mm in diameter.
 - Plastic three piece 37-mm filter holders (cassettes).
 - The filter is supported in the cassette by a second glass fiber filter if necessary.
 - A personal air-sampling pump capable of operating at 1.5 to 2.0 liters/min.
 - Midjet fritted glass impingers.
- 6.1.2 Equipment for sample analysis
- Orion Model 97-70 residual chlorine electrode or equivalent
 - Millivolt meter - Orion Model 940 or equivalent
 - Laboratory glassware including volumetric flasks, pipettes, beakers, disposable beakers, etc.
- 6.2 Reagents:
- All reagents used should be ACS analyzed reagent grade or better.
- 6.2.1 Sulfamic acid, 0.1% - Sulfamic acid (1.0 g) is dissolved in deionized water and the solution is diluted to one liter.
- 6.2.2 Potassium iodide, 0.5M - Potassium iodide (21.75 g) is dissolved in deionized water and the solution is diluted to 250 mL. The solution should be made fresh the day it is used.
- 6.2.3 Acid reagent (buffer) (6.4M Acetic acid, 1.8M Sodium acetate) - Sodium acetate (37.1 g) is dissolved in 100 mL of deionized water. Glacial acetic acid (92 mL) is added and the solution is diluted to 250 mL using deionized water.
- 6.2.4 Standard Stock Solution - Potassium iodate (0.500 g) is dissolved in deionized water and diluted to 500 mL in a volumetric flask. This solution is equivalent to 1000-ppm chlorine.

- 6.2.5 TCCA Stock Solution (optional) - Trichloroisocyanuric acid (0.500 g) is dissolved in deionized water and diluted to 500 mL in a volumetric flask. This solution is 1000-ppm TCCA and 915 ppm Chlorine.
- 6.2.6 DCCA Stock Solution (optional) – Sodium dichloroisocyanuric acid (0.500 g) is dissolved in deionized water and diluted to 500 mL in a volumetric flask. This solution is 1000-ppm DCCA and 645 ppm Chlorine.

6.3 Safety Precautions

- 6.3.1 Extreme care must be used when handling glacial acetic acid. Gloves and face protection should be used. The area where the acid is diluted should be well ventilated (NOTE: Do not vent into hoods designated for perchloric acid.) Inhalation of the acetic acid vapors should be avoided. The glacial acetic acid should be added to the aqueous solution, thus avoiding the splattering which can occur when water is added to a concentrated acid. If any of the acid contacts the eyes, skin, or clothes, flush the area immediately with copious amounts of water. Inform your supervisor. Medical treatment may be necessary if the burn is serious.
- 6.3.2 Care should be exercised when using laboratory glassware. Chipped pipettes, volumetric flasks, beakers, or any glassware with sharp edges exposed should not be used in order to avoid the possibility of cuts or abrasions.
- 6.3.3 Pipetting is always done using a pipetting bulb, never by mouth.

6.4 Standard Preparation *

- 6.4.1 The 1000-ppm standard stock solution (Sec 6.2.4) is diluted by serial 1/10 dilutions to 100 and 10-ppm working stock solutions using deionized water.
- 6.4.2 Standards are prepared in the analytical range of 20 to 0.2 ppm, using the dilution scheme indicated in Table 1, by using the following procedure:
- 6.4.2.1 An aliquot of the indicated stock solution (Table 1) is placed into a 50 mL volumetric flask. * NOTE: If available, TCCA or DCCA may be substituted for KIO_3 and the results read in direct concentration.
- 6.4.2.2 Add 0.5 mL acid reagent (Sec 6.2.3) to the volumetric flask.
- 6.4.2.3 Add 0.5 mL Potassium iodide (Sec 6.2.2) to the volumetric flask and swirl to mix the reagents; allow the solution to react for 2 minutes before proceeding with the procedure.
- 6.4.2.4 Dilute to volume with deionized water; mix by inverting the flask several times and store in the dark until it is time to perform the analysis.

6.5 Sample Preparation

- 6.5.1 Samples are prepared for analysis by the following procedure:
- 6.5.1.1 Place glass fiber filter into Phillips beaker. Desorb with 40 mL of 0.1% Sulfamic acid.
- 6.5.1.2 Remove 20 mL (or one-half) and place into 50-mL volumetric flask, saving the remainder in case a dilution is necessary.

- 6.5.1.3 Measure the volume of solution received and record this volume in your notebook.
- 6.5.1.4 Quantitatively transfer the sample to a 25-mL volumetric flask. Rinses for the transfer and final dilutions are made with 0.1% Sulfamic acid.
- 6.5.1.5 Add 0.5 mL acid reagent to the volumetric flask.
- 6.5.1.6 Add 0.5 mL Potassium iodide to the volumetric flask and swirl to mix; allow the solution to react for two minutes before proceeding with the procedure.
- 6.5.1.7 Dilute the sample to volume using deionized water; mix thoroughly by inverting the flask several times and store in the dark until it is time to perform the analysis. Record this volume as the solution volume.

6.6 Analysis

- 6.6.1 Analyze the impinger samples as Chlorine following OSHA method ID-101.
- 6.6.2 Working standard solutions are prepared as previously indicated. (Standards are prepared the day the analysis is to be performed.)
- 6.6.3 After inserting the electrode leads into the appropriate jacks in the millivolt meter, the instrument is set up for the analysis by following the manufacturers' instructions. For the Orion 940 the operating instructions are included in Table 2.
- 6.6.4 Generally five or six samples are prepared at a time and are read while preparing the next five or six samples. The electrode should be rinsed using deionized water and blotted dry before it is introduced into the next sample or standard solution. Standard solutions are kept in volumetric flasks in the dark. Fresh aliquots are used from the volumetric flasks each time a standard is read. Standards in the range of the samples are read after every fourth or fifth sample.

6.7 Calculations

- 6.7.1 The OSHA Auto AA program is used for the calculations.
- 6.7.2 Calculations are made using the following equation:

$$mg / m^3 = \frac{(ppm - ppm \text{ blank})(mL \text{ sample vol.})(50 \text{ mL})}{(Air \text{ volume (L)})(mL \text{ aliquot analyzed})}$$

- 6.7.4 If determining TCCA or DCCA with KIO_3 standards, use the following equation(s):

$$TCCA \text{ } mg / m^3 = \frac{(ppm \text{ sample} - ppm \text{ blank})(mL \text{ sample vol.})(50 \text{ mL})}{(Air \text{ volume (L)})(mL \text{ aliquot analyzed})(0.915)^*}$$

$$DCCA \text{ } mg / m^3 = \frac{(ppm \text{ sample} - ppm \text{ blank})(mL \text{ sample vol.})(50 \text{ mL})}{(air \text{ volume (L)})(mL \text{ aliquor analyzed})(0.645)^{**}}$$

* TCCA acts as a 91.5% chlorinating agent.

**DCCA acts as a 64.5% chlorinating agent.

Table 1
KIO₃ Standards
Dilution Scheme

Std (ppm)	mL stock Std used	Conc (ppm) stock	final vol (mL)
0.2	1.0	10	50
0.6	3.0	10	50
1.0	5.0	10	50
10.0	5.0	100	50
20.0	10.0	100	50

The Orion 940 follows a menu driven display. The instrument is programmed for the analysis by following the promptings of the menu driven display using the "YES", "NO" and the numeric keypads to answer the questions.

Table 2
Operating Instructions For
Orion 940 IonAnalyzer

Instruction	answer
1. Operator menu?	1. Yes
2. Change Electrode ID?	2. Yes
3. Electrode 1 = F ⁻ Is this correct?	3. "NO". An array of four choices will be displayed. Continue to press "NO" until C12 is displayed and then press the number displayed for C12 on the numeric keyboard.
4. Set number of decimal places or significant digits?	4. "NO" or reset with the numeric keys.
5. Set absolute or relative millivolts?	5. "NO"
6. Change the pH or ISE limits?	6. "NO"
7. Set timer?	7. "NO"
8. Change print interval?	8. "NO"
9. Set temperature?	9. "NO"
10. Change the date and time?	10. "NO" or reset with the numeric keys.
11. Enter standby mode?	11. "NO"
Enter "speed 0" to continue after changing the electrode ID, if necessary, and making any other desired changes on the Operator Menu.	

12. Calibrate C12?	12. "YES"
13. Calibrate by direct measurement?	13. "YES"
14. Enter number of standards (1 - 5).	14. Press "2" to calibrate on the 1 and 10 ppm standards.
15. Do blank corrections -----	15. "NO" -----
16. 1 – C12 electrode in standard 1?	16. The electrode is rinsed, blotted dry and placed into the 1.0-ppm standard solution (20 mL disposable plastic beakers are used to hold the solutions). Answer "YES"
17. Std. 1 = X.XX not ready.	17. Wait for the reading to stabilize and the display to read ...
18. Std. 1 = X.XX Cal as X.XX? -----	18. If necessary, use the numeric keys to change to CAL AS 1.00? and answer "YES". Record this reading in your notebook. -----
19. 1- C12 Electrode in Std 2?	19. The electrode is removed from the 1.0-ppm solution, rinsed with deionized water, and blotted dry. Then the electrode is placed into the 10-ppm solution. Answer "YES".
20. Std. 2 = XX.XX Cal as XX.XX?	20. If necessary, change the XX.XX to 10.00 and answer "YES". Record this reading in your notebook.
21. Slope = - 59.2 -----	21. Record the slope in your notebook. -----
22. Measure 1- C12? 10:31 11-22-88	22. Answer "YES". Rinse the electrodes, blot dry and place the electrodes into the first standard.
23. 1-C12 = X.XX 10:32 11-22-88	23. Record this value with the proper sample or standard number. Rinse and dry the electrodes and repeat this step for each sample or standard. After all of the analyses are complete, enter "speed 8" to return to ENTER STANDBY? and answer "YES".
