SODIUM DICHLOROISOCYANURATE (ANHYDROUS and DIHYDRATE)

New specifications prepared at the 61st JECFA (2003), published in FNP 52 Add 11 (2003). An ADI of 2.0 mg/kg bw for the anhydrous form was established at the 61st JECFA (2003).

SYNONYMS Anhydrous: NaDCC; sodium dichloro-*s*-triazinetrione

Dihydrate: NaDCC dihydrate; sodium dichloro-*s*-triazinetrione dihydrate

DEFINITION NaDCC (anhydrous/dihydrate) is prepared by first reacting elemental chlorine with cyanuric acid in aqueous alkaline slurry to produce dichloroisocyanuric acid monohydrate. The latter is converted to the dihydrate of NaDCC, which may be heated to yield the anhydrous material. Both the dihydrate and the anhydrous material are produced as dry powders that can be granulated and packaged.

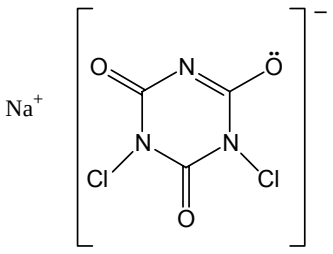
Chemical names 1,3-dichloro-1,3,5-triazine-2,4,6(1H,3H,5H)-trione, sodium salt
Triazine, 2,4,6(1H,3H,5H)-trione, 1,3-dichloro-, sodium salt

1,3-dichloro-1,3,5-triazine-2,4,6(1H,3H,5H)-trione, sodium salt, dihydrate
Triazine, 2,4,6(1H,3H,5H)-trione, 1,3-dichloro-, sodium salt, dihydrate

C.A.S. number Anhydrous: 2893-78-9
Dihydrate: 51580-86-0

Chemical formula Anhydrous: $NaC_3N_3O_3Cl_2$
Dihydrate: $NaC_3N_3O_3Cl_2$•2H2O

Structural formula



(anhydrous form)

Formula weight Anhydrous: 219.95
Dihydrate: 255.98

Assay Available chlorine: not less than 62.0% (anhydrous) and 55.0% - 57.0% (dihydrate) Not less than 98% of NaDCC on the dried basis for both anhydrous and the dihydrate

DESCRIPTION Hygroscopic white crystalline powder or granules with a slight chlorine odour

FUNCTIONAL USES Antimicrobial agent for use in drinking water systems

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Soluble in water, slightly soluble in acetone

Melting range (Vol. 4) 240° (decomposes)

Infrared spectrum The infrared spectra of anhydrous NaDCC and NaDCC dihydrate obtained using a diamond anvil compression cell are given in the Appendix.

Sodium (Vol. 4) Passes test

PURITY

pH (FNP 5) 6.0 - 7.0 (1 % soln)

Sodium chloride Not more than 2 %
See description under TESTS

Loss on drying (Vol. 4) Anhydrous: not more than 3.0% (127°, 1.5 h)
Dihydrate: between 11.0% and 14.0% (127°, 1.5 h)

Lead (Vol. 4) Not more than 2 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 methods described in Volume 4, "Instrumental Methods."

TESTS

PURITY TESTS

Sodium chloride

Principle:
Chloride ion is determined using a chloride-specific electrode.

Apparatus:
- Ion-selective electrode meter
- Chloride ion electrode
- Reference electrode

Reagents:
(Note: Use only distilled water. Do not use de-ionized water.)
Sodium nitrate, 5 M: Dissolve 42.5 g of reagent-grade material in 100 ml of water.
Chloride standards for calibration of the ion-selective electrode meter:
- *Stock solution (1000 mg/l):* Transfer 1.650 g of reagent grade sodium chloride to a 1-litre volumetric flask. Add ca. 500 ml of water to dissolve the salt and dilute to volume. Mix well.
- *Chloride standard (20 mg/l):* Pipette 10 ml of the stock solution into a 500-ml volumetric flask, dilute to volume and mix.
- *Chloride standard (2 mg/l):* Pipette 10 ml of the 20 mg/l chloride standard into a 100-ml volumetric flask, dilute to volume and mix.

Procedure:
Calibrate the ion-selective electrode meter according to the manufacturer's instructions.

Weigh 0.30 g of the sample. Quantitatively transfer the sample to a 500-ml volumetric flask. Add about 300 ml of water and shake the flask until the sample has completely dissolved. Dilute to volume and mix. Transfer 100 ml of the solution to a 150-ml beaker and, *via* pipette, add 2 ml of 5 M sodium nitrate. Stir the solution slowly. Introduce the electrodes into the solution. Record the concentration of chloride ion, C (mg/l), directly from the meter display. (Note: If the concentration reading is above 20 mg/l (i.e., 20 ppm), repeat the analysis, decreasing the sample weight as necessary.) Remove the electrodes from the solution, rinse them with water and blot dry. Store the electrodes in a beaker of water until needed.

Calculations:

$$\% \text{ sodium chloride} = C \times 0.0824/W$$

where
W = sample weight (g)
0.0824 = 500/(10000 × FWCl/FWNaCl), and FWCl/FWNaCl is the ratio of the formula weight of chlorine to that of sodium chloride

METHOD OF ASSAY

Principle:
The sample is dissolved in a solution of potassium iodide. The chlorine of the sample oxidizes iodide to free iodine, which is titrated against sodium thiosulfate. “Available chlorine” is calculated from the titration result. The sample purity on the dried basis is calculated from the available chlorine and the loss on drying.

Procedure*:*
Add 200 ml of freshly boiled and cooled distilled water to a 500 ml iodine flask with stirring bar. Add 25 ml of potassium iodide TS. Accurately weigh about 0.23 g - 0.26 g of sample. Transfer to the flask; stir until dissolved. Add either 10 ml of a 1:3 sulfuric acid solution or 10 ml of glacial acetic acid. Titrate with standardized 0.1 N sodium thiosulfate solution (FNP 5) until the solution turns yellow. Add 1 ml of starch TS and continue with the titration until the blue colour just disappears. Record the volume (ml) of titrant used (V). The percent available chlorine (%AvCl) is:

$$\%\text{AvCl} = 100 \times V \times N \times 0.03546 / W.$$

where

N = normality of the titrant
W = weight of the sample (g)
0.03546 = molecular weight of chlorine divided by 1000
(Note: For the dihydrate, to ensure the absence of free water, the material is normally dried to slightly less than the theoretical water content, i.e., to < 2:1 water:NaDCC.
In this case, the %AvCl can be slightly higher than the theoretical available chlorine,
TAC, of 55.40 %.)

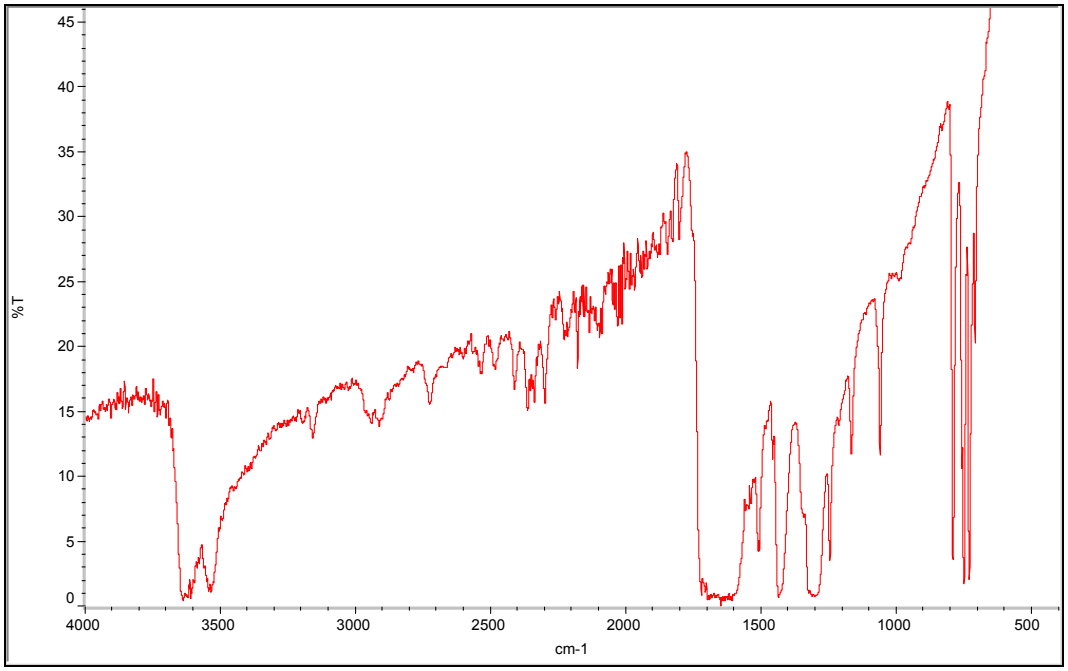
The content of sodium dichloroisocyanurate is:

$$\% \text{ NaDCC (dried basis)} = 100 \times [\ \%\text{AvCl} /64.47] \times [100 /(100 - \text{LOD}]$$

where
64.47 = percent TAC for anhydrous NaDCC
LOD is the percent loss on drying previously determined.

IR Spectrum of NaDCC in Diamond Cell

Peaks at: 3636, 3533, 2910, 2725, 2536, 2482, 2411, 2362, 2297, 1647, 1508, 1433, 1310, 1243, 1164, 1057, 788, 748, 728, 706 cm-1 Source: Occidental Chemical Corp., 2003



IR Spectrum of NaDCC Dihydrate in Diamond Cell:

Peaks at: 3592, 3472, 3225, 2898, 2699, 2466, 2288, 2052, 1841, 1717, 1502, 1427, 1309, 1235, 1163, 1057, 789, 752, 739, 717 cm-1 Source: Occidental Chemical Corp., 2003