



61st JECFA - Chemical and Technical Assessment (CTA), 2003

SODIUM DICHLOROISOCYANURATE

(NaDCC – anhydrous and dihydrate)

Chemical and Technical Assessment (CTA)

First draft prepared by Paul M. Kuznesof

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1 Summary

NaDCC (anhydrous: CAS no. 2893-78-9; dihydrate: CAS no. 51580-86-0) was placed on the agenda of the 61st JECFA at the request of the WHO Unit on Water, Sanitation, and Health for an evaluation of its safe use for disinfection of drinking water. NaDCC is produced as a white crystalline powder or in granular form and elicits a slight odor of chlorine. Dissolution in water produces a series of complex equilibria among a variety of chlorinated and non-chlorinated isocyanurates and free available chlorine (FAC) in the form of hypochlorous acid (HOCl). The latter is widely used for treating potable water, wastewater, water in swimming pools and spas, and many industrial water systems. Elemental chlorine, sodium hypochlorite solution, and calcium hypochlorite are other commonly used sources of FAC. Regardless of the source of FAC, HOCl is the active antimicrobial agent, effective against a wide range of bacteria, fungi, algae, viruses and other microorganisms. Cyanuric acid, the end product from use of chloroisocyanurates in bleaching, sanitizing, and disinfection applications, is unusually stable to hydrolysis, only slowly hydrolyzing in hot aqueous alkali; it is virtually inert to acid hydrolysis.

NaDCC is manufactured in a multi-step process. Pyrolysis of urea produces crude cyanuric acid. A slurry of cyanuric acid (dihydrate), previously purified (> 99%) by digestion with a strong mineral acid, is made alkaline with strong base (sodium hydroxide). This slurry is exposed to elemental chlorine. Dichloroisocyanuric acid monohydrate (DCCA) precipitates and is separated by filtration. DCCA monohydrate is reslurried and treated with strong base to form NaDCC dihydrate, which can be recovered as such for commercial purposes or be subjected to heating to produce anhydrous NaDCC. Anhydrous NaDCC is marketed at > 97% and the dihydrate at > 99%, based on analyzed deliverable FAC. On a dry basis, both products meet an assay of > 98%. The principal impurity in NaDCC is sodium chloride.

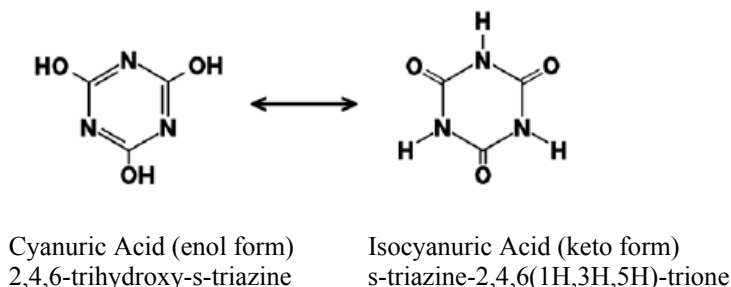
A typical value of FAC for effectiveness in drinking water is about 1 mg/L. Above this concentration water becomes increasingly unpalatable. Approximately 1.6 mg NaDCC (anhydrous) delivers about 1 mg FAC per liter of water. Assuming a daily intake of water of 2 L/person/day, the daily intake of "NaDCC" would be 3.2 mg. Given that cyanuric acid is the ultimate end-product of application of NaDCC, ingestion of 3.2 mg "NaDCC" per day leads to an exposure estimate for cyanuric acid of 1.9 mg/person/day. With regard to ingested FAC, essentially all is rapidly reduced to chloride ion in the saliva and the stomach. Therefore, little FAC would be available in the stomach to undergo chlorination reactions, such as reactions with proteins and amino acids to form N-chlorinated compounds or with phenols and unsaturated lipids to form C-chlorinated compounds.

Use of chlorine (FAC) as a disinfectant in drinking water systems (and, e.g., process waters) containing naturally occurring organic compounds can result in formation of trihalomethanes (THM). THM, such as chloroform, are regarded as carcinogens. Thus, the potential exists for THM to form in water treated with chlorinated isocyanurates. Disinfectants that reduce the activity of FAC by binding to some of it and "stabilizing" it, can, under properly controlled conditions, minimize THM formation to levels well below those produced using elemental chlorine. Monochloroamine (NH₂Cl), in which chlorine exists as "combined" (stabilized) chlorine rather than FAC, is a common alternative to the use of FAC where THM are of concern. Other chlorine stabilizers should also be effective to varying degrees in limiting THM

formation, depending on how strongly they stabilize the available chlorine. The chloroisocyanurates are not as effective as monochloroamine in limiting THM but, under controlled conditions of use, will generally produce lower levels than chlorine.

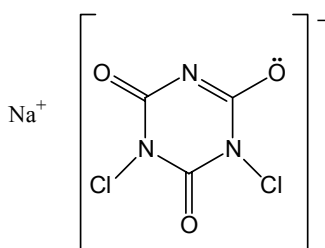
2 Description

Chloroisocyanurates are best introduced by describing the parent compound, cyanuric acid, an odorless white crystalline solid with a melting point above 330o, which can exist in two tautomeric forms:



X-ray crystallographic and neutron diffraction studies confirm the planar three-fold symmetry of cyanuric acid (Coppens, P. and Vos, A. 1971)

NaDCC is the sodium salt of dichloroisocyanuric acid:



Both the anhydrous and hydrated forms are white crystalline powders or granules with a slight odor of chlorine. They have the following properties:

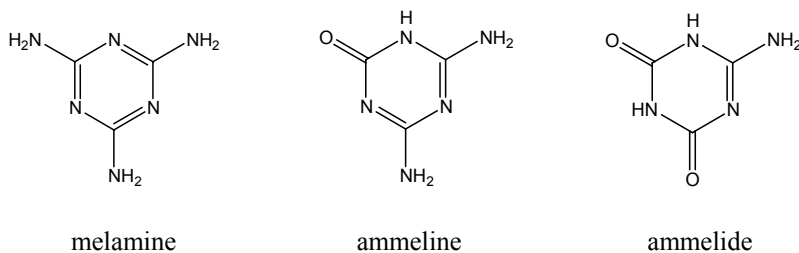
	<i>Anhydrous</i>	<i>Dihydrate</i>
CAS number:	2893-78-9	51580-86-0
Chemical formula:	NaC ₃ N ₃ O ₃ Cl ₂	NaC ₃ N ₃ O ₃ Cl ₂ •2H ₂ O
Formula weight:	219.95	255.98
Assay (dry basis):	> 98%	> 98%
Available chlorine:	> 62.0 %	55.0 % - 57.0 %
Solubility in water:	24 g/100 g	28 g/100 g
pH (1% aqueous solution at 25°):	6.0-7.0	6.0-7.0
Melting range:	240° (decomposes)	loses 1st H ₂ O at > 40 °; 2nd H ₂ O at > 80 °; 240° (decomposes)

3 Manufacturing (Wojtowicz, J. 1993; FDA 1997)

3.1 Preparation of cyanuric acid

Urea is first pyrolyzed (250o, ca. 1 hour) to produce crude cyanuric acid (see figure above), which is then purified (> 99%) by digestion with acid (e.g., hydrochloric, nitric, or sulfuric).

When heated, urea initially dissociates into isocyanic acid (HNCO) and ammonia. These recombine to form equilibrium amounts of ammonium cyanate, along with the intermediates biuret and triuret, through interactions of urea with the isocyanic acid. Isocyanic acid trimerizes to cyanuric acid; amination of biuret and triuret results in cyclic aminotriazine by-products, primarily ammelide, ammeline, and minor amounts of melamine:



AMINOTRIAZINES

The acid hydrolysis of this crude cyanuric acid converts acyclic impurities to carbon dioxide and ammonia; the cyclic aminotriazine by-products, which may constitute as much as 20% of the crude cyanuric acid, are converted to additional cyanuric acid and ammonia. And because amination of urea and its intermediates results in the formation of water, any unreacted urea will also hydrolyze to produce ammonia and carbon dioxide. The purified cyanuric acid (> 99%) is recovered as the dihydrate and washed with water to remove residual acid and acid salts. Other means of purification of cyanuric acid are described by Wojtowicz (Wojtowicz, J. 1993); references are to the patent literature. However, the process described above is likely the most widely used.

3.2 Preparation of NaDCC

A slurry of purified cyanuric acid dihydrate is allowed to react with 50% sodium hydroxide at room temperature or slightly below to produce a solution of disodium cyanurate. In a continuous process, this solution is fed into a tank along with elemental chlorine. Dichloroisocyanuric acid (DCCA) monohydrate precipitates. Sodium chloride is a major by-product. Time, temperature, and pH of the reaction are carefully controlled. The precipitated DCCA monohydrate is filtered from the slurry and washed. Acyclic organics and ammonia are destroyed by the chlorine while any residual aminotriazines that may have been present in the cyanuric acid are either chlorinated or destroyed.

DCCA monohydrate is reslurried and allowed to react with 50% sodium hydroxide to form NaDCC dihydrate. The resulting slurry is dewatered until a wet cake with about 20% water is achieved. The wet cake is dried with mild heat using a forced-air dryer at temperatures ranging between 65o and 105o and residence times of less than one second. The resulting dry NaDCC dihydrate powder is then granulated and packaged. This material begins to lose its water of hydration at 40o. Therefore, care must be taken to ensure proper storage conditions. Water liberated due to elevated temperatures will result in lumping of the product and other caking and heat effects in products formulated with the dihydrate.

To obtain anhydrous NaDCC, some manufacturers dry the NaDCC dihydrate wet cake directly, using a forced-air dryer at temperatures ranging between 300o and 375o and residence times of less than one second. The dried powder is then granulated and packaged. Other manufacturers first produce NaDCC dihydrate granules. Then, in a separate step, dry the dihydrate at temperatures between 80o and 130 o to produce anhydrous NaDCC granules.

4 Chemical characterization

4.1 Physicochemical properties

NaDCC (both the dihydrate and anhydrous material), as well as cyanuric acid, are well-characterized substances. Physical and chemical properties are described in the Kirk-Othmer Encyclopedia of Chemical Technology (Wojtowicz, J. 1993), in a web-based document on chloroisocyanurates by Occidental Chemical Corporation (OxyChem 2003), in a monograph developed by OxyChem on the chemistry of the chloroisocyanurates (OxyChem 1997), and in a Food Additive Petition (FAP) submitted by Occidental to the U.S. Food and Drug Administration (FDA 1997).

Occidental Chemical Corporation literature (OxyChem 2003) declares purities for anhydrous NaDCC and NaDCC dihydrate of > 97% and > 99%, respectively, based on analyzed deliverable FAC. Sales specifications from July and August 2002 cite a range for moisture of 12.2-14.0% for the dihydrate and not more than 3.0% for the anhydrous material. Other manufacturers may produce product that differs slightly from these values. Thermal stabilities, pH of water solutions, and water solubilities are given in the Table above. The pH range is a critical parameter related to the purity of NaDCC as it permits an assessment of the presence of unacceptable levels of tri- or mono-chloroisocyanurates. NaDCC has a characteristic infrared spectrum (FDA 1997) and X-ray powder diffraction data are available (FDA 1997; OxyChem 1997) for distinguishing between anhydrous NaDCC and the dihydrate.

4.2 Possible impurities

The principal impurity in NaDCC is sodium chloride; minor amounts of unreacted cyanuric acid might also be present. The chlorinated aminotriazines, according to data in the FAP (FDA 1997), would likely each be present at levels no greater than 200 mg/kg or non-detectable at the reported detection limits.

Cyanuric acid (Wojtowicz, J., 1993) is unusually stable to hydrolysis. It is only slowly hydrolyzed by hot aqueous alkali and is virtually inert to acid hydrolysis. The triazine ring can be cleaved by alkaline hypochlorite to form elemental nitrogen and bicarbonate. Wojtowicz (Wojtowicz, J., 1993) notes that although numerous mono-, di-, and tri-substituted organic derivatives of cyanuric/isocyanuric acid are referenced in the literature, many are not accessible via cyanuric acid. The extensive review of the chemistry of cyanuric acid by Wojtowicz (Wojtowicz, J., 1993) allows a conclusion that cyanuric acid residues in drinking water would not be expected to show any additional reaction chemistry.

4.3 Rationale for proposed specifications for NaDCC (anhydrous and dihydrate)

Assay – Level of purity is based on measurable FAC, for which a standard analytical method is available.

Identification tests – Solubility properties, melting range, and infrared spectrum are characteristic physical properties for NaDCC. The sodium test distinguishes NaDCC from non-ionic chloroisocyanurates and confirms a sodium salt.

Loss on drying – Determination of water content provides a means to distinguish the anhydrous material from the dihydrate and from mixtures of the two. This specification also serves as an indication of GMP.

pH – The pH range can be related to the purity of NaDCC, as it permits a qualitative assessment of the presence of unacceptable levels of tri- or mono-chloroisocyanurates.

Sodium chloride - Residual sodium chloride may be present in the final product. It is an important indicator of purity. An analytical method is available.

Lead – JECFA specifications for food additives include lead limits as a matter of course. For inorganic salts such as NaDCC, the presence of residual lead must always be considered. Moreover, as reagents such as mineral acids and sodium hydroxide are employed in the manufacture of NaDCC, a lead limit is clearly appropriate. The selected limit is based on industry information. Atomic absorption spectrophotometry is satisfactory for the analysis. International and national water quality authorities have established maximum limits for metals in addition to lead in drinking water. Therefore, no additional limits for metals other than lead are not specified for NaDCC.

Consideration was given to the need for specifying upper limits for residual chlorinated cyclic aminotriazines, which, as noted above, have been observed at levels no greater than 200 mg/kg or non-detectable at the reported detection limits. As the exposure to these cyclic triazines can be expected to be quite low and no issue

of toxicological concern has been raised, a specification limit has not been proposed. An analytical approach, which calls for dechlorinating a sample of NaDCC with thiosulfate and determining the three cyclotriazines (above figure) using High Performance Liquid Chromatography with a proprietary column, is available. In use, drinking water suppliers usually specify that substances used in water treatment do not add significantly to the concentrations of any contaminant covered by the WHO Guidelines for Drinking-water Quality.

5 Functional uses

NaDCC is being evaluated by JECFA for its use as a disinfectant in drinking water. Registrations for its use in drinking water and waste water systems have been approved by the U.S. Environmental Protection Agency. The United Kingdom (United Kingdom 2000) lists NaDCC as suitable in “emergency” situations for drinking water disinfection as do a number of other countries. NaDCC is used for routine disinfection of drinking water in some Latin American countries and by military personnel of several countries for disinfection of drinking water in the field.

“Chlorine” has been in use for the treatment of water, especially drinking water, for over a century. The term “chlorine” is often used generically in reference to various materials known to provide, when dissolved in water, “free available chlorine” (FAC), i.e., chlorine available as hypochlorous acid (HOCl) and hypochlorite ion (ClO⁻). Commonly used sources of FAC are elemental chlorine, sodium hypochlorite, calcium hypochlorite, and the chloroisocyanurates, including NaDCC. Solutions of hypochlorous acid/hypochlorite have both excellent oxidizing and disinfecting properties. Hypochlorite is a strong oxidizing agent and is highly effective for eliminating organic contaminants, whereas undissociated HOCl is the principal microbiocidal agent, effective against bacteria, fungi, algae, viruses, and other microorganisms (OxyChem 2003; Pinto, G. and Rohrig, B. 2003).

A typical value of available chlorine for effectiveness in drinking water is about 1 mg/L, according to OxyChem (OxyChem 2000). OxyChem’s report also notes that above this concentration drinking water becomes increasingly unpalatable. A US-EPA-registered label for NaDCC (Occidental Chemical Corp., EPA label no. 935-41) provides detailed directions for use in a variety of water-treatment applications. For example, for disinfection of public water systems, a quantity of NaDCC necessary to achieve a residual of at least 0.2 ppm FAC is recommended. For emergency disinfection of raw or pre-treated (settled, coagulated, and/or filtered) drinking water supplies (lakes, rivers, wells, etc.), NaDCC should be introduced to achieve 10 ppm FAC initially, followed by maintenance of 1 ppm.

The United Kingdom water supply regulations (United Kingdom 2000) specify that NaDCC may be used only where the water is not grossly contaminated and only for as long as it takes to restore “conventional” treatment or “for no more than 90 days in any period of a year, whichever is applicable.” It must be used in a controlled contact time of not less than 15 minutes between dosings whereby not more than 10 mg/L of NaDCC should be applied and not more than 1 mg/L of free residual chlorine should be present at the end of the relevant contact time. Provision is also made for use in grossly contaminated water sources.

6 Reactions and Fate upon Consumption (O’Brien, J.E. et al. 1974; OxyChem 1997; OxyChem 2000)

When added to water, NaDCC (anhydrous or dihydrate) rapidly hydrolyzes to release FAC and establish a complex series of equilibria (see attached Figure) involving six chlorinated and four non-chlorinated isocyanurates. These equilibria are established on the order of seconds (Matte, D. et al. 1989). The concentration of each species depends on the concentrations of total available chlorine (TAC = FAC and “reservoir” chlorine, e.g., as DCC-) and total isocyanurates, the pH, and the values of the equilibrium constants (dependent on temperature and ionic strength). “Reservoir” chlorine refers to the bound chlorine of the various chloroisocyanurates (see Figure). The latter function as reservoirs of rapidly-released FAC, as FAC is depleted. Thus, if HOCl is consumed by reaction with organic material (oxidation), chloroisocyanurates will rapidly dissociate to release more HOCl.

The FAC for anhydrous NaDCC is ca. 63% and the dihydrate contains ca. 56% FAC; by definition, FAC for elemental chlorine is 100% (Pinto, B. and Rohrig, B. 2003). Therefore, development of 1 mg/L FAC, typical for drinking water treatment, requires ca. 1.6 mg/L of anhydrous NaDCC and ca. 1.8 mg/L for the dihydrate.

The distribution of the various chemical species in aqueous solutions of NaDCC can be calculated from their hydrolysis and acid-dissociation constants. As an example (OxyChem 1997), dissolution of NaDCC to provide 1.0 mg/L total available chlorine, at pH 7.0, gives the following: 48.1% from HOCl, 26.8% from

monochlorocyanurate, 11.8% from dichlorocyanurate, 12.8% from hypochlorite, and less than 1% from other chlorocyanurates and chlorocyanuric acids. In normal batch-type use of NaDCC, oxidative and microbiocidal demand will consume FAC until all available chlorine has been reduced, leaving only non-chlorinated isocyanurates. But, as long as NaDCC is added to water to maintain a certain level of total available chlorine or FAC, the various cyanurates will be present at levels dependent on the properties of the water (i.e., pH, temperature, etc.)

FAC readily reacts with components of saliva and stomach fluid: proteins and amino acids form N-chlorinated compounds; phenols and unsaturated lipids form C-chlorinated compounds; and sulfur-containing amino acids, thiocyanate ion, and carbohydrates form chloride ion and unchlorinated oxidation products (Tan, H. et al. 1987; Ghanbari, H.A. et al. 1982; Fukayama, M.Y. et al. 1986; Wei, C.I. et al. 1987). In contact with saliva (ca. pH 7), chlorinated isocyanurates react nearly instantaneously and chlorine demand (presence of oxidizable organics) is sufficiently high to reduce nearly all FAC to chloride ion in treated drinking water, as it is swallowed (e.g., $t_{1/2} = 0.01$ s for thiocyanate at pH 7). In the stomach, pH 2-3, remaining chlorinated isocyanurates rapidly dissociate into FAC; oxidizable organics will rapidly reduce FAC to chloride. The neutral dichloroisocyanuric acid (HCl₂CY, see Figure) quickly becomes the only chloroisocyanurate of significance and dissociates as FAC continues to react. Because nearly all FAC is reduced to chloride, the quantities of chlorinated organics will be extremely low. For comparison, monochloroamine or MCA (NH₂Cl), a widely used disinfectant for drinking water, has a very short lifetime in the stomach, releasing its combined (bound) chlorine in less than one minute when present initially at concentrations below 15 mg/L. Because monochloroisocyanurate has been shown to release its reservoir (bound) chlorine almost 9000 times as fast as MCA and the dissociation constants of the chloroisocyanurates are much higher than MCA, all the chloroisocyanurates may be expected to react much more rapidly than MCA in both saliva and stomach fluids. Cyanuric acid and chloride ion are the stable end-products.

THM formation (OxyChem 1997; OxyChem 2003a): The use of elemental chlorine (FAC) as a disinfectant in drinking water systems (and e.g., process waters) where naturally occurring organic compounds are present is well-known to result in the formation of trihalomethanes (THM), such as chloroform, for which WHO drinking-water guidelines exist. Thus, the potential exists for THM to form in water treated with chloroisocyanurates. Disinfectants that can reduce the activity of FAC by binding to some of it and “stabilizing” it, can, under properly controlled conditions, reduce the formation of THM to levels well below those produced using elemental chlorine as the disinfectant. Thus, MCA, in which all the chlorine is combined (stabilized) chlorine rather than FAC, is a common alternative to the use of FAC where THM are of concern. Other chlorine stabilizers should also be effective to varying degrees in limiting THM formation, depending on how strongly they stabilize the available chlorine. The chloroisocyanurates are not as effective as MCA in limiting THM, but under controlled conditions of use will generally produce lower levels than elemental chlorine.

7 Intake of Chloroisocyanurates

A typical concentration of FAC for drinking water treatment is 1.0 mg/L (OxyChem 2000). As anhydrous NaDCC contains ca. 63% FAC, achievement of 1 mg/L FAC requires 1.6 mg/L NaDCC. Currently, the WHO (2003) uses the quantity of 2 L per day for adults, 1 L per day for a 10 kg child, or 0.75 L per day for a 5 kg bottle-fed infant as default upper-percentile drinking water intake rates. However, the WHO also recognizes that higher intake rates may occur in some tropical countries. These rates include water consumed in the form of juices and other beverages containing tap water (e.g., coffee). Thus, the daily intake of “NaDCC” resulting from consumption of 2 liters of water would be 3.2 mg/person/day. Given that 1 mole of NaDCC corresponds to 1 mole of cyanuric acid and the latter is the ultimate end-product of application of NaDCC, ingestion of 3.2 mg “NaDCC” per day leads to an exposure estimate for cyanuric acid of 1.9 mg/person/day.

The quantities of chlorinated organics will be extremely low (noted above), as nearly all FAC is reduced to chloride by the high chlorine demand in the saliva and the stomach. Therefore, exposures to such substances should be negligible and in any case be regarded in the same light as exposures to them from use of sources of FAC other than NaDCC to disinfect drinking water.

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Equilibrium Reactions for Isocyanurate and Chlorinated Isocyanurate Compounds

(Figure from “Chemistry of the Chlorinated Isocyanurates,” OxyChem, 1997)

