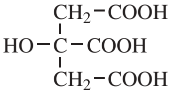
CITRIC ACID

1. Introduction

Citric acid [77-92-9] (2-hydroxy-1,2,3-propanetricarboxylic acid), is a natural component and common metabolite of plants and animals. It is the most versatile and widely used organic acid in foods, beverages, and pharmaceuticals.



$$\begin{array}{c} CH_2-COOH \\ | \\ HO-C-COOH \\ | \\ CH_2-COOH \end{array}$$

Because of its functionality and environmental acceptability, citric acid and its salts (primarily sodium and potassium) are used in many industrial applications for chelation, buffering, pH adjustment, and derivatization. These uses include laundry detergents, shampoos, cosmetics, enhanced oil recovery, and chemical cleaning.

Citric acid specifications are defined in a number of compendia including *Food Chemicals Codex* (FCC), *United States Pharmacopoeia* (USP), *British Pharmacopoeia* (BP), *European Pharmacopoeia* (EP), and *Japanese Pharmacopoeia* (JP).

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Historically, about AD 1200, the alchemist Vincentius Bellovacensis recognized that lemon and lime juices contained an acid substance. In 1784 Scheele first isolated crystalline citric acid from lemon juice. In 1834 Liebig recognized citric acid as a hydroxy tribasic acid, and in 1893 Wehmer indicated that certain fungi produce citric acid when grown on sugar solutions. The microbial fermentation of a carbohydrate substrate is virtually the exclusive commercial procedure to produce citric acid.

2. Occurrence

Citric acid occurs widely in the plant and animal kingdoms (1). It is found most abundantly in the fruits of the citrus species, but is also present as the free acid or as a salt in the fruit, seeds, or juices of a wide variety of flowers and plants. The citrate ion occurs in all animal tissues and fluids (1). The total circulating citric acid in the serum of humans is approximately 1 mg/kg body weight. Normal daily excretion in human urine is 0.2–1.0 g. This natural occurrence of citric acid is described in Table 1.

2.1. Physiological Role of Citric Acid.

Citric acid occurs in the terminal oxidative metabolic system of virtually all organisms. This oxidative metabolic system (Fig. 1), variously called the Krebs cycle (for its discoverer, H. A. Krebs), the tricarboxylic acid cycle, or the citric acid cycle, is a metabolic cycle involving the conversion of acetate derived from carbohydrates, fats, or proteins to carbon dioxide and water. This cycle releases energy necessary for an organism's growth, movement, luminescence, chemosynthesis, and reproduction.

Table 1. **Natural Occurrence of Citric Acid**

Fruits and vegetables		Animal tissues and fluids	
Plant	Citric acid, wt%	Location	Citric acid, ppm
lemons	4.0–8.0	human whole blood	15
grapefruit	1.2–2.1	human blood plasma	25
tangerines	0.9–1.2	red blood cells	10
oranges	0.6–1.0	human milk	500–1250
currants		urine	100–750
black	1.5–3.0	semen	2000–4000
red	0.7–1.3	thyroid gland	750–900
raspberries	1.0–1.3	kidney	20
strawberries	0.6–0.8	bone	7500
apples	0.008	saliva	4–24
potatoes	0.3–0.5	sweat	1–2
tomatoes	0.25	tears	5–7
asparagus	0.08–0.2		
turnips	0.05–1.1		
peas	0.05		
corn kernels	0.02		
lettuce	0.016		
eggplant	0.01		

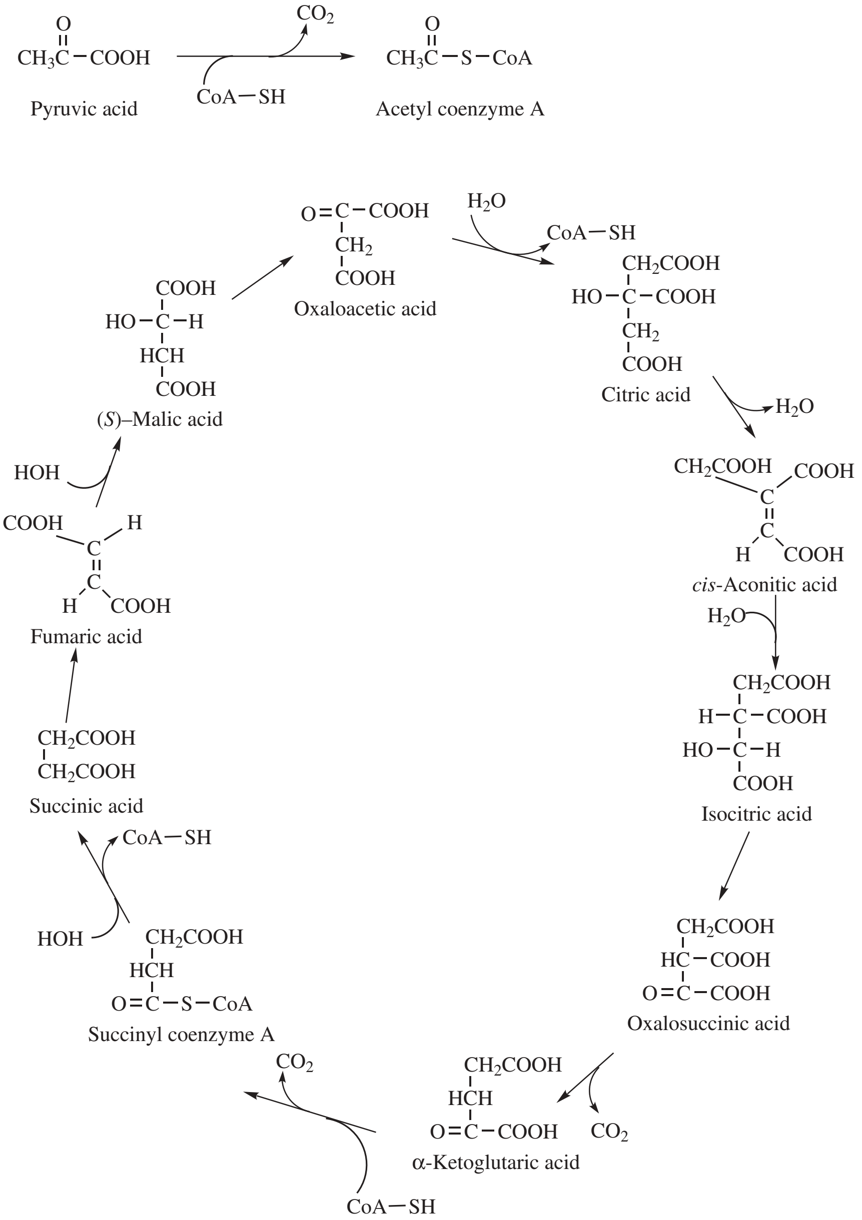


Fig. 1. Krebs (citric acid) cycle. Coenzyme A is represented CoA–SH. The cycle begins with the combination of acetyl coenzyme A and oxaloacetic acid to form citric acid.

The cycle also provides the carbon-containing materials from which cells synthesize amino acids and fats. Many yeasts, molds, and bacteria conduct the citric acid cycle, and can be selected for their ability to maximize citric acid production in the process. This is the basis for the efficient commercial fermentation processes used today to produce citric acid.

3. Physical Properties

Citric acid, anhydrous, crystallizes from hot aqueous solutions as colorless translucent crystals or white crystalline powder. Its crystal form is monoclinic holohedra. Citric acid is deliquescent in moist air and is optically inactive. Some physical properties are given in Table 2 (2,3). The solubility of citric acid in water and some organic solvents is given in Table 3. The pH and specific gravity of aqueous solutions of citric acid are shown in Table 4.

Aqueous solutions of citric acid make excellent buffer systems when partially neutralized because citric acid is a weak acid and has three carboxyl groups, hence three pK_as. At 20°C p$K_1 = 3.14$, p$K_2 = 4.77$, and p$K_3 = 6.39$ (1). The buffer range for citrate solutions is pH 2.5 to 6.5. Buffer systems can be made using a solution of citric acid and sodium citrate or by neutralizing a solution of citric acid with a base such as sodium hydroxide. In Table 5 stock

Table 2. **Physical Properties of Citric Acid, Anhydrous**

Property	Value
molecular formula	$C_6H_8O_7$
mol wt	192.13
gram equivalent weight	64.04
melting point, °C	153
thermal decomposition temp., °C	175
density, g/mL	1.665
heat of combustion,[a] MJ/mol[b]	1.96
heat of solution, J/g[b]	117

[a] At 25°C.

[b] To convert J to cal, divide by 4.184.

Table 3. **Solubility[a] of Citric Acid, Anhydrous**

Temperature, °C	g/100 g satd soln
In water	
10	54.0
20	59.2
30	64.3
40	68.6
50	70.9
60	73.5
70	76.2
80	78.8
90	81.4
100	84.0
In organic solvents at 25°C	
amyl acetate	4.2
diethyl ether[b]	1.0
ethyl alcohol[b]	38.3

[a] Ref. 4.

[b] Absolute.

Table 4. **pH and Specific Gravity of Aqueous Citric Acid Solutions**

Concentration, % w/w	pH	Specific gravity at 25°C
0.1	2.8	
0.5	2.4	
1.0	2.2	
5.0	1.9	
10.0	1.7	1.035
20.0		1.084
30.0	1.2	1.131
40.0		1.182
50.0	0.8	1.243
60.0		1.294

Table 5. **Citric Acid Buffer Solutions**

0.1 *M* Citric acid, mL	0.1 *M* Sodium citrate, mL	Buffer solution pH
46.5	3.5	3.0
33.0	17.0	4.0
20.5	29.5	5.0
9.5	41.5	6.0

solutions of 0.1 *M* (0.33 *N*) citric acid are combined with 0.1 *M* (0.33 *N*) sodium citrate to make a typical buffer solution.

Citric acid monohydrate [5949-29-1] has a molecular weight of 210.14 and crystallizes from cold aqueous solutions. When gently heated, the crystals lose their water of hydration at 70–75°C and melt in the range of 135–152°C. Rapid heating causes dehydration at 100°C to form crystals that melt sharply at 153°C. Citric acid monohydrate is available in limited commercial quantities since most applications now call for the anhydrous form.

4. Chemical Properties

Citric acid undergoes most of the reactions typical of organic hydroxy polycarboxylates. Reactions such as esterification, salt formation and anhydride reactions can be easily perfomed. However, the tertiary hydroxyl group does not undergo all the common reactions. If a reaction requires more strenous conditions, dehydration to aconitic acid is promoted.

4.1. Decomposition.

When heated above 175°C, citric acid decomposes to form aconitic acid [499-12-7], citraconic acid [498-23-7], itaconic acid [97-65-4], acetonedicarboxylic acid [542-05-2], carbon dioxide, and water, as shown in Figure 2.

4.2. Esterification.

Citric acid is easily esterified with many alcohols under azeotropic conditions in the presence of a catalyst such as sulfuric acid,

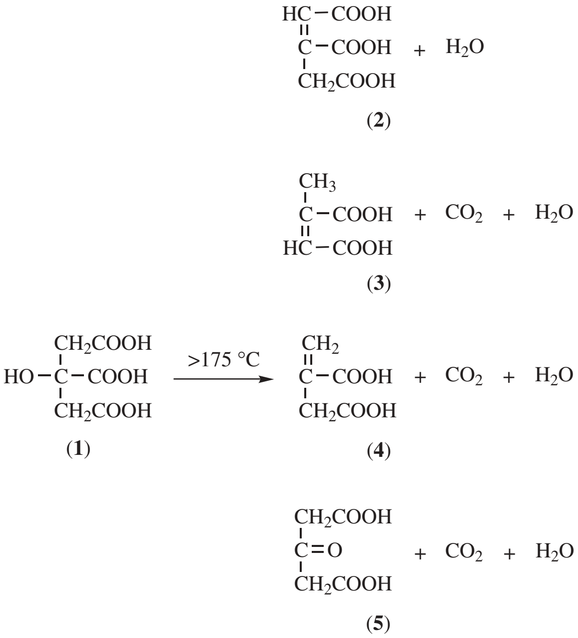
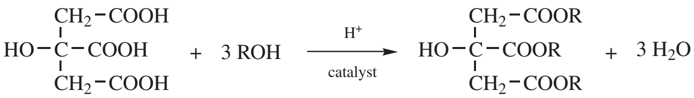


Fig. 2. Thermal decomposition of citric acid (**1**) to aconitric acid (**2**), citraconic acid (**3**), itaconic acid (**4**), and oxidation to acetonedicarboxylic acid (**5**).

p-toluenesulfonic acid, or sulfonic acid-type ion-exchange resin. Alcohols boiling above 150°C esterify citric acid without a catalyst (5–8).

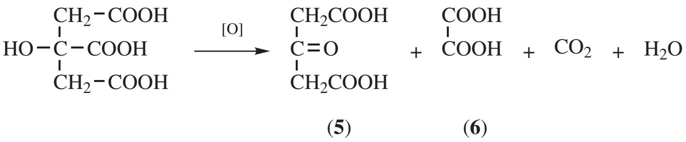


$$\text{HO-C(CH}_2\text{-COOH)}_2\text{-COOH} + 3\,\text{ROH} \xrightarrow[\text{catalyst}]{\text{H}^+} \text{HO-C(CH}_2\text{-COOR)}_2\text{-COOR} + 3\,\text{H}_2\text{O}$$

Alcohols typically used in citric acid esterification are methyl, ethyl, butyl, and allyl alcohols.

4.3. Oxidation.

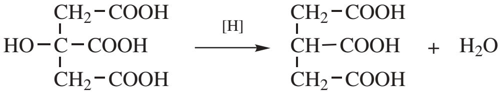
Citric acid is easily oxidized by a variety of oxidizing agents such as peroxides, hypochlorite, persulfate, permanganate, periodate, hypobromite, chromate, manganese dioxide, and nitric acid. The products of oxidation are usually acetonedicarboxylic acid (**5**), oxalic acid (**6**), carbon dioxide, and water, depending on the conditions used (5).



$$\text{HO-C(CH}_2\text{-COOH)}_2\text{-COOH} \xrightarrow{[\text{O}]} \underset{(\mathbf{5})}{\text{O=C(CH}_2\text{COOH)}_2} + \underset{(\mathbf{6})}{\text{HOOC-COOH}} + \text{CO}_2 + \text{H}_2\text{O}$$

4.4. Reduction.

The hydrogenation of citric acid yields 1,2,3-propanetricarboxylic acid [99-14-9]. (5)



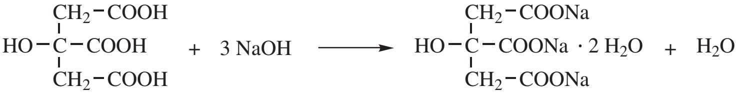
$$\begin{array}{l}CH_2-COOH\\ \quad|\\ HO-C-COOH\\ \quad|\\ CH_2-COOH\end{array} \xrightarrow{[H]} \begin{array}{l}CH_2-COOH\\ |\\ CH-COOH\\ |\\ CH_2-COOH\end{array} + H_2O$$

Catalytic hydrogenation is more difficult. Hydrogenolysis yields carbon dioxide, water, methane, formic acid, acetic acid and a small amount of methyl succinic acid (5).

Hydrogenation of trisodium citrate over a Ni catalyst at 8.6 MPa (85 atm) and a temperature of 220–230°C results in hydrogenolysis fragments.

4.5. Salt Formation. Citric acid forms mono-, di-, and tribasic salts with many cations such as alkalies, ammonia, and amines. Salts may be prepared by direct neutralization of a solution of citric acid in water using the appropriate base, or by double decomposition using a citrate salt and a soluble metal salt.

Trisodium citrate is more widely used than any of the other salts of citric acid. It is generally made by neutralization of a water solution of citric acid using sodium hydroxide. The neutralization reaction is highly exothermic giving off 1109 J/g of citric acid. To conserve energy, the heat evolved can be used in the sodium citrate concentration and crystallization steps.



$$\begin{array}{l}CH_2-COOH\\ \quad|\\ HO-C-COOH\\ \quad|\\ CH_2-COOH\end{array} + 3\,NaOH \longrightarrow \begin{array}{l}CH_2-COONa\\ \quad|\\ HO-C-COONa\cdot 2\,H_2O\\ \quad|\\ CH_2-COONa\end{array} + H_2O$$

Other sources of sodium ion that are used to make sodium citrate are sodium carbonate and sodium bicarbonate. These reactions evolve large volumes of carbon dioxide gas, resulting in much foaming but less exotherm.

The mono- and disodium citrate salts are made by limiting the amount of sodium available by using only one mole of base for each mole of citric acid for the monosodium citrate and two moles for the disodium citrate. The result is primarily the mono or disalt with small amounts of the other forms and citric acid being present. Other salts that have been offered commercially are shown in Table 6.

4.6. Chelate Formation. Citric acid complexes with many multivalent metal ions to form chelates (9,10). This important chemical property makes citric acid and citrates useful in controlling metal contamination that can affect the color, stability, or appearance of a product or the efficiency of a process.

Citric acid, with its one hydroxyl and three carboxyl groups, is a multidentate ligand. Two or more of these sites are utilized to form a ring structure. The normal molar ratio of metal-to-ligand is 1:1. With some metal ions, under certain conditions, more than one ring can be formed allowing a higher metal-to-ligand ratio (see CHELATING AGENTS).

When a metal ion is chelated by a ligand such as citric acid, it is no longer free to undergo many of its chemical reactions. A metal ion that is normally colored may, in the presence of citrate, have little or no color. Under pH conditions that may precipitate a metal hydroxide, the citrate complex may be soluble. Organic molecules that are catalytically decomposed in the presence of metal ions can be made stable by chelating the metal ions with citric acid.

Table 6. **Salts of Citric Acid**

Salt	CAS Registry number	Molecular formula
ammonium citrate	[3012-65-5]	$(NH_4)_2HC_6H_6O_7$
calcium citrate	[813-94-5]	$Ca_3(C_6H_5O_7)_2$
calcium citrate tetrahydrate	[5785-44-4]	$Ca_3(C_6H_5O_7)_2 \cdot 4H_2O$
cobalt citrate	[866-81-9]	$Co_3(C_6H_5O_7)_2$
copper citrate	[866-82-0]	$C_6H_8O_7 \cdot 2Cu$
ferric ammonium citrate	[1185-57-5]	$C_6H_8O_7\mathrm{xFe} \cdot x\mathrm{NH}_3$
ferric citrate	[2338-05-8]	$C_6H_8O_7 \cdot \mathrm{xFe}$
lead citrate	[512-26-5]	$Pb_3(C_6H_5O_7)_2$
lithium citrate	[919-16-4]	$Li_3C_6H_5O_7$
magnesium citrate	[3344-18-1]	$Mg_3(C_6H_5O_7)_2$
manganese citrate	[5968-88-7]	$MnC_6H_5O_7$
nickel citrate	[6018-92-4]	$Ni_3(C_6H_5O_7)_2$
potassium citrate	[866-84-2]	$K_3C_6H_5O_7$
potassium citrate hydrate	[6100-05-6]	$K_3C_6H_5O_7 \cdot H_2O$
sodium citrate dihydrate	[6132-04-3]	$Na_3C_6H_5O_7 \cdot 2H_2O$
zinc citrate	[546-46-3]	$Zn_3(C_6H_5O_7)_2$

Chelation is an equilibrium reaction. There are always some free-metal ions present as well as chelated metal ions. In a system where a metal salt is being reduced, such as in metal plating, the rate of the reaction forming the metal can be controlled by using the metal citrate chelate.

The log function of the ratio of chelated metal ions to free-metal ions is expressed as the stability constant or formation constant as shown in Table 7. The higher the stability constant the greater the percentage of metal ions that are chelated (11).

Stability constants are measured at their optimum pH. Conditional stability constants are measured at a specific pH. In general, stability constants for metal citrates are very low below pH 2–3, high at pH 3–10, and low above pH 10–12.

4.7. Corrosion. Aqueous solutions of citric acid are mildly corrosive toward carbon steels. At elevated temperatures, 304 stainless steel is corroded

Table 7. **Stability Constants for Metal Citrates**

Metal	Valence	$\log K$
Fe	+3	12.5
Al	+3	7.00
Pb	+2	6.50
Ni	+2	5.11
Co	+2	4.80
Zn	+2	4.71
Ca	+2	4.68
Cu	+2	4.35
Cd	+2	3.98
Mn	+2	3.67
Mg	+2	3.29
Fe	+2	3.08
Ba	+2	2.98

by citric acid, but 316 stainless steel is resistant to corrosion. Many aluminum, copper, and nickel alloys are mildly corroded by citric acid. In general, glass and plastics such as fiber glass reinforced polyester, polyethylene, polypropylene, poly(vinyl chloride), and cross-linked poly(vinyl chloride) are not corroded by citric acid.

5. Manufacturing and Processing

Historically, citric acid was isolated by crystallization from lemon juice and later was recognized as a microbial metabolite. This work led to the development of commercial fermentation technology (12). The basic raw materials for making citric acid include Molasses (mainly beet), sucrose, dextrose (mainly from corn, wheat or tapioca) and unrefined sweet potato.

5.1. Fermentation. Several microorganisms (yeasts and molds) have been identified as citric acid accumulators (i.e., *Yarrowia sp., Candida sp.* and *Aspergillus niger*). However, the most common microorganisms used for the commercial production for citric acid is *Aspergillus niger*. Commercial scale fermentation was begun in 1923 utilizing certain strains of *Aspergillus niger* to produce citric acid on the surface of a sucrose and salt solution. Surface or tray fermentation is still used for production of citric acid in areas of the world that are agriculturally rich and still accounts for a reasonable proportion of worldwide citric acid production. Surface fermentation is more labor intensive and is easier to maintain aseptically in industrial operations. Nowadays, the preferred method for large-volume industrial production is a submerged process known as deep tank fermentation (13–21).

In the deep tank submerged process, *Aspergillus niger* mold spores are grown under controlled aseptic conditions on a test-tube slant and transferred to a seed tank or inoculum which is added to a fermentor along with pasteurized syrup. The pH is adjusted and nutrients added. Sterile air is sparged into the fermentor while the sugar is converted to citric acid. The complete fermentation cycle can take as long as 7 days or even less if yeasts are used.

Several patents have recently been granted for production of citric acid/ citrates by fermentation. One is a process for continuous production of citric acid/ citrate comprising the steps of continuously feeding to a fermentor containing yeasts, oxygen, sugar (especially glucose), and an ammonium compound. Sugar continuously fed to the fermentor is transformed to citric acid and the product is withdrawn continuously from the fermentor (22).

Another uses an aqueous solution of raw material containing citric acid by means of fermentation. Second, third, and fourth solutions are obtained by cell separation, filtration, and ion exchangers. A final solution is obtained containing citric acid/ citrate in water (23).

5.2. Recovery. Citric acid fermentation broth is generally separated from the biomass using filtration or centrifugation. The citric acid is usually purified using either a lime-sulfuric acid method or a liquid extraction process (24). Choice between these two methods is dictated in part by the fermentation feedstock. Lime-sulfuric extraction is more traditional, so it is used in many of the older plants. It is still used in newer production facilities where crude feedstocks

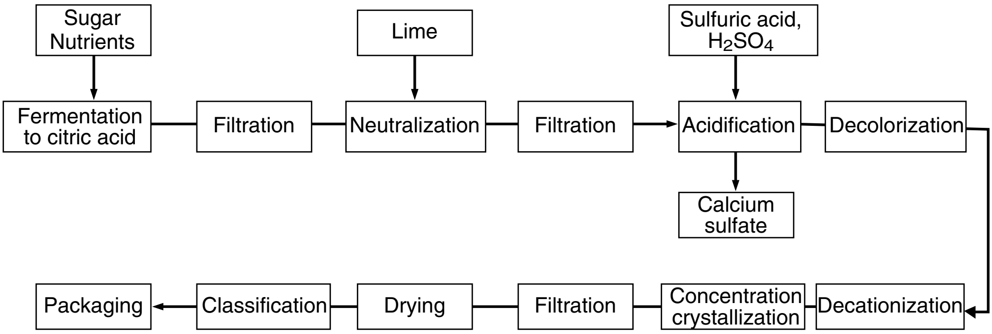


Fig. 3. Lime-sulfuric recovery process for citric acid.

are still used. Solvent extraction is currently viable where pure substrate is used for fermentation.

Lime-Sulfuric. Recovery of citric acid by calcium salt precipitation is shown in Figure 3. Although the chemistry is straightforward, the engineering principles, separation techniques, and unit operations employed result in a complex commercial process. The fermentation broth, which has been separated from the insoluble biomass, is treated with a calcium hydroxide (lime) slurry to precipitate calcium citrate. After sufficient reaction time, the calcium citrate slurry is filtered and the filter cake washed free of soluble impurities. The clean calcium citrate cake is reslurried and acidified with sulfuric acid, converting the calcium citrate to soluble citric acid and insoluble calcium sulfate. Both the calcium citrate and calcium sulfate reactions are generally performed in agitated reaction vessels made of 316 stainless steel and filtered on commercially available filtration equipment.

The citric acid solution is deionized at this stage to remove trace amounts of residual calcium, iron, other cationic impurities, and to improve crystallization. In some processes, trace-impurity removal and decolorization are accomplished with the aid of adsorptive carbon.

The aqueous citric acid solution is concentrated in a series of crystallization steps to achieve the physical separation of citric acid from remaining impurities. Standard evaporation, crystallization, and filtration equipment can be employed in this operation. The choice of crystallizer temperature dictates the formation of anhydrous or monohydrate citric acid. Above 37°C, the transition point, anhydrous citric forms whereas below 37°C, the monohydrate will crystallize. The finished citric acid is dried and classified. Because anhydrous citric acid is hygroscopic, to protect against caking care must be taken to avoid handling, packaging, and storage of the crystals in areas of high temperature and high humidity.

Liquid Extraction. The recovery process, shown in Figure 4, was developed in the 1970s and involves the extraction of citric acid from fermentation broth using a mixture of trilaurylamine,*n*-octanol, and C_{10} or C_{11} hydrocarbon, followed by re-extraction of the citric acid from the solvent phase into water (25). Efficient citric acid extraction is achieved through a series of countercurrent steps that ensure intimate contact of the aqueous and nonaqueous phases. When transfer of the citric acid to the solvent phase is complete, the citric acid is

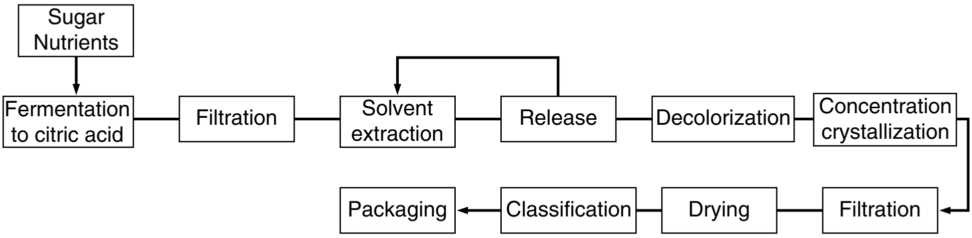


Fig. 4. Liquid extraction recovery process for citric acid.

re-extracted into water, again using a multistage countercurrent system. The two steps differ mainly in the temperature at which they are performed.

The final processing steps are a wash of the aqueous citric acid solution by the hydrocarbon solvent, followed by passage of the acid solution through granular activated carbon columns. Effluent from the carbon columns is processed through a conventional sequence of evaporation/crystallization/drying and packaging steps to complete the manufacturing process.

Citric acid is also commercially available as a 50% w/w solution made either by dissolving crystalline citric acid in water, or a combination or crystalline citric acid, and one of the citric acid process streams. There are several grades of citric acid solutions available, each made according to quality which is measured by color and trace impurities. The citric acid content of each grade can be identical, 50% w/w, which is near the solubility limit.

Several methods of recovery of citric acid from a medium in which it is contained have been reported in ref. 26. The preferred method involves contact of citric acid with a solid-phase free base resin having a tertiary amine group to absorb the citric acid.

5.3. By-Products. The biomass from the fungal fermentation process is called mycellium and can be used as a supplement for animal feed since it contains digestable nutrients (27,28). The lime–sulfuric purification and recovery process results in large quantities of calcium sulfate cake, which is usually disposed of into a landfill but can find limited use in making plaster, cement, wallboard, or as an agricultural soil conditioner. The liquid extraction purification and recovery process has the advantage of little solid by-products.

5.4. Energy. In recent years the concern for energy conservation has resulted in many innovative process improvements to make the manufacture of citric acid more efficient. For example, heat produced by the exotherm of the neutralization of citric acid with lime is used in another part of the process where heat is required, such as the evaporation/crystallization step.

5.5. Chemical Synthesis. The chemical synthesis of citric acid was reported in 1880 (29). Since then, many different synthetic routes have been investigated, reported, and patented (30–38). However, none of these have proven to be commercially feasible.

6. Shipment and Storage

Crystalline citric acid, anhydrous, can be stored in dry form without difficulty, although conditions of high humidity and elevated temperatures should be

Table 8. **Citric Acid[a] Corrosion Rates[b]**

Material	Temperature, °C	Corrosion rate, mm/yr
316 stainless steel	25	0.03
	50	0.03
304 stainless steel	25	0.03
	50	0.23
carbon steel	25	4.6
	50	32.8

[a] 50 wt% solution.

[b] Ref. 37.

avoided to prevent caking. Storage should be in tight containers to prevent exposure to moist air. Several granulations are commercially available with the larger particle sizes having less tendency toward caking.

Liquid citric acid is commercially available in a variety of concentrations with 50% w/w being most common. Grades are available that vary in appearance, purity, and color. Packaging is usually in drums, tank trucks, or rail cars. Liquid citric acid should be kept above 0°C to prevent crystallization.

Solutions of citric acid are corrosive to normal concrete, aluminum, carbon steel, copper, copper alloys, and should not be used with nylon, polycarbonates, polyamides, polyimides, or acrylics.

Recommended materials of construction for pipes, tanks, and pumps handling citric acid solutions are 316 stainless steel, fiber glass-reinforced-polyester, polyethylene, polypropylene, and poly(vinyl chloride). At elevated temperatures, 304 stainless steel is not recommended (Table 8).

Although not as corrosive as the acid, the sodium and potassium salts of citric acid should be handled in the same type of equipment as the acid to avoid corrosion problems.

7. Economic Aspects

Citric acid is manufactured in over 20 countries with 1998 worldwide production estimated at approximately 879,000 MT, distributed as shown in Figure 5. Most of this production is used for foods and beverages; however, industrial applications, eg, detergents, metal cleaning, of citric acid are becoming more important on a worldwide basis.

It was estimated that 1990 U.S. citric acid and citrate salt consumption was 152,000 t. Citric acid represents approximately 90% of this volume. This citric acid/citrate use and its historical distribution in various markets is described in Table 9.

In the 1980s, citric acid was produced mainly by pharmaceutical companies. However, in 1990–1991, when corn wet miling companies entered the market, back integration with a source of fermentation feedstock (dextrose, sugar, or molasses) became important. This marked the movement of citric acid from a pharmaceutical company product to a commodity chemical product.

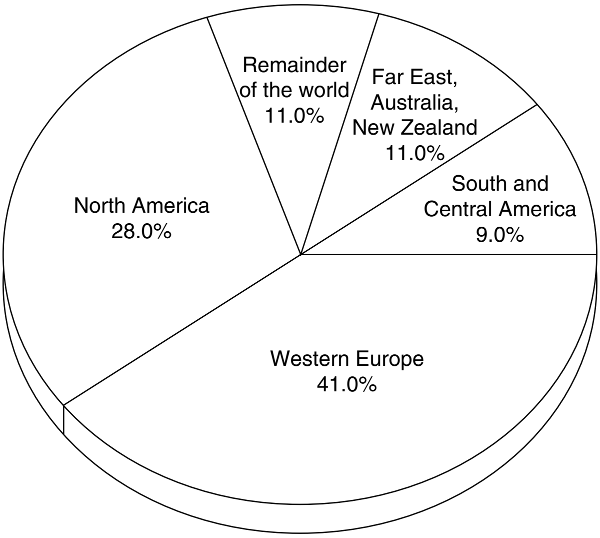


Fig. 5. Worldwide citric acid production in 1998 (40).

The list price for citric acid in the U.S. for 1998 was approximately 1.65 USD/g (40).

8. Specifications, Standards, and Quality Control

Since citric acid is produced and sold throughout the world, it must meet the criteria of a variety of food and drug compendia (41–45).

9. Analytical and Test Methods

Aqueous titration with 1 *N* NaOH remains the official method for assaying citric acid (41,42). Although not citrate-specific, the procedure is satisfactory in the absence of interfering substances. Low concentrations of citric acid can be determined by a spectrophotometic method based on the Furth and Herrmann reaction with pyridine and acetic anhydride (PAA) (46). This PAA method is

Table 9. **U.S. Citric Acid/Citrate Distribution by End Use,** %[a]

Product	1998	1996	1994
beverages	48	46	47
foods	23	22	23
pharmaceuticals and cosmetics	18	19	20
household detergents and cleaning products	6	6	6
misc. nonfood	5	7	4
Total annual consumption	*233,000*	*206,000*	*203,000*

[a] Ref. 40.

citrate-specific at 420 nm and is sensitive for citrate ions at concentrations down to 5 ppm. The PAA method can be used to quantify citrate in foods, beverages, and industrial products such as detergents.

An enzymatic method (47), which is specific for the citrate moiety, can be used as a combined assay and identification test for citric acid and its common salts down to 20 ppm.

A high performance liquid chromatography (HPLC) method to determine citric acid and other organic acids has been developed (48). The method is an isocratic system using sulfuric acid to elute organic acids onto a specific hplc column. The method is sensitive for citric acid down to ppm levels and is capable of quantifying citric acid in clear aqueous systems.

10. Health and Safety Factors

Citric acid, as well as its common sodium and potassium salt forms, are Generally Recognized As Safe (GRAS) by the U.S. Food and Drug Administration as Multiple Purpose Food Substances (49). Citric acid is also approved by the Joint FAO/WHO Expert Committee on Food Additives for use in foods without limitation (50). The use of citric acid and certain of its salts and esters has been evaluated by a Special Committee on GRAS Substances (SCOGS) of the Federation of American Societies for Experimental Biology under contract with the FDA (51). The evaluation was based largely on two scientific literature reviews prepared for the FDA that summarize the world's applicable scientific literature from 1920 through 1973 (1). Citric acid, sodium and potassium citrates are not listed under the Safe Drinking Water and Toxic Enforcement Act of 1986 (California proposition 65) that lists chemicals known to cause cancer or reproductive toxicity. These compounds are not listed by the National Toxicology Program (NTP) or the International Agency for Research on Cancer (IARC) or regulated as carcinogens by the U.S. Occupational Safety and Health Administration (OSHA) (52,53).

Tests have shown that citric acid is not corrosive to skin but is a skin and ocular irritant (54). For these reasons it is recommended that individuals use appropriate personal protection to cover the hands, skin, eyes, nose, and mouth when in direct contact with citric acid solutions or powders. This product is not hazardous under the criteria of the OSHA Hazard Communication Standard (48).

11. Environmental Considerations

Citric acid is biodegraded readily by many organisms under aerobic and anaerobic wastewater treatment conditions and in the natural environment (55). Under United States Environmental Protection Agency legislation, citric acid and its sodium and potassium salts are reported on the Toxic Substance Control Act (TSCA) inventory. They are exempt from the Emergency Planning and Community Right-to-Know Act (SARA Title III) and contain no section 313 toxic chemical although they may contain up to the FCC limits for arsenic (1 ppm), lead

(0.5 ppm), and heavy metals (5 ppm as lead). Under the U. S. Resource Conservation and Recovery Act (RCRA), if discarded in its purchased form, this product would not be a hazardous waste either by listing or by characteristic (56).

12. Uses

Citric acid is utilized in a large variety of food and industrial applications because of its unique combination of properties. It is used as an acid to adjust pH, a buffer to control or maintain pH, in a wide range (2.5–6.5), a chelator to form stable complexes with multivalent metal ions, and a dispersing agent to stabilize emulsions and other multiphase systems (see DISPERSANTS). In addition, it has a pleasant, clean, tart taste making it in the acidulant of choice in food and beverage products applications.

12.1. Food Uses.

Beverages. Citric acid, sodium citrate, and potassium citrate are used extensively in carbonated and non-carbonated beverages (57). Juice-added beverages, low calorie beverages, and thirst quenchers, also known as isotonic drinks, use citric acid alone and in combination with citrate salts for flavoring and buffering properties and to increase the effectiveness of antimicrobial preservatives. The high solubility of citric acid is important in beverage syrups. Citric acid also combines well with other acids (i.e. malic acids) to give unique flavor profiles. The amount of acid used depends on the flavor desired in the product as well as taste evaluation and customer preference. Citric acid and its salts are also used to stabilize the pH in reduced-calorie drinks that contain acid-sensitive sweeteners. The acid concentration of most fruit-flavored carbonated beverages (qv) falls in the range of 0.10–0.25% w/w.

Citric acid and its salts are used in dry beverage mixes, convenience teas, New age drinks and cocktail mixes for pH control and flavor, and are used in wine coolers at 0.10–0.55%, combining well with fruity and light flavors. Because citric acid is derived from a natural fermentation process, its use causes no problems in labeling claims. Therefore, citric acid is ideal for the marketing of health-oriented drinks.

Jams, Jellies, and Preserves. Citric acid is used in jams and jellies to provide tartness and to adjust the product pH for optimum gelation (58).

Candy. Citric acid is added in candy for tartness (59,60). To suppress the inversion of sucrose, it should be added after the cook, at levels from 0.5 to 2.0%. The pH of pectin gel candies is adjusted with citric acid for maximum gel strength. Sodium and potassium citrate are also used to reduce and control inversion and to regulate tartness.

Salads. A combination of citric acid and ascorbic acid is used as an alternative to sulfites in prevention of enzymatic browning in fresh prepared vegetables (61).

Frozen Food. The chelating and acidic properties of citric acid enable it to optimize the stability of frozen food products by enhancing the action of antioxidants and inactivating naturally present enzymes which could cause undesirable browning and loss of firmness (62,63).

Citric acid also inhibits color and flavor deterioration in frozen fruit. Here again the function is to inhibit enzymatic and trace metal-catalyzed oxidation.

Canned Fruits and Vegetables. The use of citric acid to bring the pH below 4.6 can reduce heat treatment requirements in canned fruits and vegetables. In addition, citric acid chelates trace metals to prevent enzymatic oxidation and color degradation, and enhances the flavor, especially of canned fruits.

Fats and Oils. The oxidation of fats and oils in food products can be prevented by the addition of citric acid to chelate the trace metals that catalyze the oxidation. Citric acid is also used in the bleaching clays and the degumming process during oil refining to remove chlorophyll and phospholipids (64–68).

Confections and Desserts. Citric acid andsodium citrate are utilized in the confection industry to optimize gel-setting characteristics, provide tartness, and enhance flavor. Citric acid acts as a flavor adjunct in compressed candy, tablets, cream candies and startch-based jellies.

Pasteurized Process Cheese. Sodium citrate is used in pasteurized process and sliced cheese as an emulsifying salt to stabilize the water and oil emulsion and improve process cheese body and texture (69).

Dairy Products. Sodium citrate is an important stabilizer used in whipping cream and vegetable-based dairy substitutes. Addition of sodium citrates to ice cream, ice milk, and frozen desserts before pasteurization and homogenization reduces the viscosity of the mix, making it easier to whip.

Seafood. Citric acid is used in combination with other preservatives/antioxidants to lower the pH to retard microbial growth, which can lead to spoilage, formation of off-flavors, and colors on fish and other seafood products. It is also used to chelate trace metals that catalyze oxidation.

Meat Products. Citric acid is used in cured meat products to increase the effectiveness of the antioxidant preservatives, as a processing aid, and a texture modifier. It is often encapsulated and released at a specific temperature from a controlled release matrix. Carcass rinse solutions contain citric acid to adjust the pH and enhance antimicrobial properties.

Citric acid or sodium citrate can be used as a replacement of up to 50% of cure accelerators such as ascorbic acid, erythorbic acid, sodium ascorbate, and sodium erythorbate in the preparation of cured, dried meat and poultry products.

12.2. Medical Uses. Citric acid and citrate salts are used to buffer a wide range of pharmaceuticals at their optimum pH for stability and effectiveness (70–78). Effervescent formulations use citric acid and bicarbonate to provide rapid dissolution of active ingredients and improve palatability. Citrates are used to chelate trace metal ions, preventing degradation of ingredients. Citrates are used to prevent the coagulation of both human and animal blood in plasma and blood fractionation. Calcium and ferric ammonium citrates are used in mineral supplements. A patent derivatives a chewing gum with fluoride and citric acid for improved dental health (79).

12.3. Agricultural Use. Citric acid and its ammonium salts are used to form soluble chelates of iron, copper, magnesium, manganese, and zinc micronutrients in liquid fertilizers (80–86). Citric acid and citrate salts are used in animal feeds to form soluble, easily digestible chelates of essential metal nutrients, enhance feed flavor to increase food uptake, control gastric pH and improve feed efficiency.

12.4. Industrial Uses. *Laundry Detergents.* Sodium citrate is used in both liquid and powder laundry detergents as a builder(87–104). In many detergent applications, builder systems containing citrates are used as environmentally acceptable replacements for phosphates. Citrates chelate water hardness ions, disperse soil, and are used as processing aids. High water solubility and performance at both low and high pH and low and high temperatures are the keys to citrate use in detergents. In powder detergents citrates are used as auxiliary co-builders usually with zeolites and carbonates (see DETERGENCY).

Hard Surface Cleaners. Citric acid and sodium citrate are used in hard surface cleaners as acidifying and chelating agents for dissolving hard water deposits and as a builder to increase the efficacy of the surfactants. In carpet cleaning, citric acid stabilizes dyes and removes carpet yellowing, especially in stain-resistant carpets.

Reverse Osmosis Membrane Cleaning. Citric acid solutions are used to remove iron, calcium, and other cations that foul cellulose acetate and other membranes in reverse osmosis and electrodialysis systems. Citric acid solutions can solubilize and remove these cations without damaging the membranes (105–107).

Metal Cleaning. Citric acid, partially neutralized to ∼pH 3.5 with ammonia or triethanolamine, is used to clean metal oxides from the water side of steam boilers and nuclear reactors with a two-step single fill operation (108–126). The resulting surface is clean and passivated. This process has a low corrosion rate and is used for both pre-operational mill scale removal and operational cleaning to restore heat-transfer efficiency.

High-pressure sprays of heated neutralized citric acid solutions replace sandblasting techniques to clean stainless steel equipment and areas not easily accessible such as ship bilges.

Petroleum. Citric acid is added to hydrochloric acid solutions in acidizing limestone formations. Citric acid prevents the formation of ferric hydroxide gel in the spent acid solution by chelating the ferric ions present. Formation of the gel would plug the pores, preventing the flow of oil to the producer well (127–131).

A clear solution of aluminum citrate neutralized to pH 7 is used for *in situ* gelling of polymers in polymer flooding and well stimulation in enhanced oil recovery techniques (132–136). The citrate chelate maintains aluminum ion solubility and controls the rate of release of the aluminum cross-linker.

Citric acid is used to chelate vanadium catalyst in a process for removing hydrogen sulfide from natural and refinery gas and forming elemental sulfur, a valuable product (137).

Flue Gas Desulfurization. Citric acid can be used to buffer systems that can scrub sulfur dioxide from flue gas produced by large coal and gas-fired boilers generating steam for electrical power (138–147). The optimum pH for sulfur dioxide absorption is pH 4.5, which is where citrate has buffer capacity. Sulfur dioxide is the primary contributor to acid rain, which can cause environmental damage.

Mineral and Pigment Slurries. Citric acid can be used as a dispersing agent in slurries of ores, rocks, clays, and pigments during refining and transport. Citric acid controls swelling of clays and reduces pumping viscosity by contributing thixotropic properties to the dispersions (148–156).

Electrodeposition of Metals. Citric acid and its salts are used as sequestrants to control deposition rates in both electroplating and electroless plating of metals (157–175). The addition of citric acid to an electroless nickel plating bath results in a smooth, hard, nonporous metal finish.

Concrete, Mortar, and Plaster. Citric acid and citrate salts are used as admixtures in concrete, mortar, and plaster formulations to retard setting times and reduce the amount of water required to make a workable mixture (176–184). The citrate ion slows the hydration of Portland cement and acts as a dispersant, reducing the viscosity of the system (185). At levels below 0.1%, citrates accelerate the setting rate while at 0.2–0.4% the set rate is retarded. High early strength and improved frost resistance have been reported when adding citrate to concrete, mortar, and plaster.

Textiles. Citric acid acts as a buffer in the manufacture of glyoxal resins which are used to give textiles a high quality durable-press finish (see AMINO RESINS). It has been reported to increase the soil-release property of cotton with wrinkle-resistant finishes and is used as a buffer, a chelating agent, and a non-volatile acid to adjust pH in disperse dying operations (186–197).

Plastics. Citric acid and bicarbonate are used as an effervescent blowing agent to foam polystyrene for insulated food and beverage containers replacing blowing agents such as chlorinated fluorocarbons (198–210).

Citric acid is used as a chelating agent in catalyst systems for making resins, and citrate esters are used as plasticizers (qv) inPVC film, especially in food packaging (211).

Paper. Citric acid is added to the pulp slurry prior to bleaching to sequester metal ions and prevent discoloration (212–215). Citrates are used in cigarette paper to control the burning rate to match that for tobacco.

Tobacco. Citric acid is a natural constituent of the tobacco leaf and during tobacco processing additional citric acid is added to enhance the flavor and to effect more complete combustion of tobaccos (216).

Cosmetics and Toiletries. Citric acid and bicarbonate are used in effervescent type denture cleansers to provide agitation by reacting to form carbon dioxide gas. Citric acid is added to cosmetic formulations to adjust the pH, act as a buffer, and chelate metal ions preventing formulation discoloration and decomposition (91,217–222). The use of active such in creams to alleviate signs of dermatological aging and wrickling is discussed in Ref. 221.

Refractories and Molds. Citric acid is used as a binder for refractory cements, imparting volume stability and strength in ceramic materials for electrical condensers, foundry and glassmaking molds, and sand molds for metal castings (223–227).

13. Derivatives

13.1. Salts.

The trisodium citrate salt is made by dissolving citric acid in water at a concentration of 50% w/w or higher. A 50% solution of sodium hydroxide is carefully added to pH 8.0–8.5. The reaction is exothermic and cooling is necessary to prevent boiling. The hot solution can be treated with activated carbon to remove impurities before evaporating and crystallizing. The concentrated slurry is filtered to separate the sodium citrate dihydrate [6132-04-3], which is washed with water, dried in a hot air dryer, classified, and packaged in bags, drums, or large sacks. The tripotassium salt of citric acid is made in a similar manner using potassium hydroxide. The product crystallizes as the monohydrate [6100-05-6].

Ammonium salts of citric acid are made by adding either aqueous or anhydrous ammonia to citric acid dissolved in water. They are usually used in the liquid form rather than isolated as a dry product. Citric acid salts are listed in Table 5. Solubility data is as follows (1).

Citrate salt	Water solubility, wt%
diammonium citrate	50
calcium citrate tetrahydrate	0.10
ferric ammonium citrate	very soluble
potassium citrate monohydrate	60
sodium citrate dihydrate	42

13.2. Esters.

The significant esters of citric acid are trimethyl citrate, triethyl citrate, tributyl citrate, and acetylated triethyl- and tributyl citrate. Many other esters are available but have not been used on a commercial scale. Citric acid esters are made under azeotropic conditions with a solvent, a catalyst, and the appropriate alcohol.

Catalysts used are usually acids such as sulfuric acid, *p*-toluenesulfonic acid, sulfonic acid ion-exchange resins, and others. The water from the reaction of the citric acid and the alcohol is continuously removed as the azeotrope until no more water is formed. At this point, the reaction is usually complete and the solvent and any excess alcohol is distilled off under mild vacuum. The catalyst is neutralized using carbonate or sodium hydroxide, leaving a crude product. If a pure product is desired, the ester can be distilled under high vacuum.

The properties of citric acid esters are described in Table 10.

Citric acid esters are used as plasticizers in plastics such as poly(vinyl chloride), poly(vinylidene chloride), poly(vinyl acetate), poly(vinyl butyral), polypropylene, chlorinated rubber, ethylcellulose, and cellulose nitrate. Most citrate esters are nontoxic and are acceptable by the FDA for use in food-contact

Table 10. **Properties of Citric Acid Esters**

Name	CAS Registry number	Molecular weight	Density,[a] g/mL	Bp[b], °C
triethyl citrate	[77-93-0]	276.29	1.136	126–127
tri-*n*-butyl citrate	[77-94-1]	360.43	1.042	169–170
tricyclohexyl citrate	[4132-10-9]	438.57	1.7	57[c]
acetyl triethyl citrate	[77-89-4]	318.31	1.135	131–132
acetyl tri-*n*-butyl citrate	[77-90-7]	402.46	1.046	172–174
acetyl tri-2-ethylhexyl citrate	[144-15-0]	570.81	0.983	225

[a] At 25°C.

[b] At 133 Pa = 1 mm Hg.

[c] Melting point.

packaging and for flavor in certain foods. As a plasticizer, citrate esters provide good heat and light stability and excellent flexibility at low temperatures. Triethyl citrate, tri-*n*-butyl citrate, isopropyl citrate, and stearyl citrate are considered GRAS for use as food ingredients (228–232).

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