

Starch

Business



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animal Chapter 1: SYNTHESIS OF OXALIC ACID FROM SAWDUST History.—

Oxalic acid was discovered by Scheele, in 1776, and it is found in the organic as well as in the inorganic kingdoms. In plants it is generally met in combination with calcium or potassium; rhubarb, *Rumex acetosa*, *Oxalis acetosella*, *phytolacca*, *belladonna*, etc., contain that to History.—Oxalic acid was discovered by Scheele, in 1776, and it is found in the organic as well as in the inorganic kingdoms. In plants it is generally met in combination with calcium or potassium; rhubarb, *Rumex acetosa*, *Oxalis acetosella*, *phytolacca*, *belladonna*, etc.

, contain the acid or bin-oxalate of potassium; rhubarb also contains oxalate of calcium, as likewise do many lichens, and in the human body it forms the mulberry calculus, a frequent form of gravel. In combination with calcium it is found in ginger, orris-root, squill, valerian, curcuma, quassia, and other drugs. As an ammonium oxalate it is present in the fertilizer guano. In the *Cicer arietinum* or chick pea it occurs in a free condition. It may also be formed artificially by the action of nitric acid on sugar, molasses, rice, starch, gum, wool, silk, hair, and many other organic compounds, which are free from nitrogen.

Berthelot has obtained it synthetically from acetylene, ethylene, propylene, and allylene, by oxidation with permanganate of potassium. Preparation.—

There are several methods by which oxalic acid may be procured; the following are considered among the best: 1. Gently heat 1 part of pure starch with 8 parts of nitric acid of sp. gr.

1. 200 or 1. 250. A powerful reaction ensues, with an evolution of red nitrous acid vapors; when this diminishes, heat must be applied, and continued until no more red vapors are given off; if sufficiently evaporated, a large quantity of crystals of hydrated oxalic acid are deposited as the liquid cools. These are dried on a porous tile, then dissolved in a little hot water, and pure oxalic acid is deposited as the solution cools. The mother liquor remaining after the first deposit of crystals contains much free nitric acid, saccharic acid, and other products.

2. Digest 1 pint of sugar dried at 100° C. (212° F.) with 8. 25 pints of nitric acid of sp. gr.

1, 380. Evaporate the mixture to a sixth, and leave to crystallize. This process requires but an hour or two, and yields from 50 to 60 per cent of handsome crystals. 3. An ingenious and economical method of manufacturing oxalic acid from sawdust has been devised by Mr.

Dale, of England. It is as follows: two equivalents of caustic soda and one equivalent of caustic potash are mixed together, dissolved in water, and the solution evaporated until it has a sp. gr. 1. 350, when enough sawdust is to be stirred in to form a thick paste.

This paste is heated on iron plates, being constantly stirred. At first, water escapes; the mass then swells; inflammable gases, hydrogen, and hydrocarbons are evolved, along with a peculiar aromatic odor. When the temperature has been maintained at 204. 4° C. (400° F.

) for an hour or two, this stage of the process is complete. The mass has a dark color, contains from 1 to 4 per cent of oxalic acid, and about 0.5 per cent of formic acid; the balance of the mass consists of an unknown substance, which is intermediate between cellulose and oxalic acid. The next stage consists in heating the mass till quite dry, being careful that no charring takes place; a gray powder is formed, containing from 28 to 30 per cent of oxalic acid, in combination with sodium and potassium. The gray powder is now washed on a filter with a solution of carbonate of sodium, which decomposes the oxalate of potassium, and converts it into oxalate of sodium, which is decomposed by boiling milk of lime, oxalate of calcium being precipitated while the sodium hydroxide remains in solution. The oxalate of calcium being placed in leaden vessels, is treated with sulphuric acid, which precipitates the calcium and leaves the oxalic acid in solution, which may be obtained in crystals by evaporation.

The sodium hydroxide left in solution after the addition of milk of lime, may be recovered by boiling, and be again made use of with fresh sawdust. The same may be done with the potassium salt which filters through in the last stage. By this process 2 pounds of sawdust are made to yield 1 pound of oxalic acid. Description.—Oxalic acid crystallizes in colorless, transparent, oblique, quadrilateral prisms with two-sided summits. The crystals are inodorous, have a strongly acid taste, faintly effloresce in a dry atmosphere, redden litmus paper, and when pure are completely volatilized by heat, and without becoming blackened.

They dissolve in from 8 to 11 parts of water at 15.5° C. (60° F.), in their own weight of water at 100° C. (212° F.)

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), and in 4 parts of alcohol; the addition of a small quantity of nitric acid to the water causes them to dissolve more readily. Nearly all the oxalates are insoluble in water, excepting the alkaline. Oxalate of calcium is insoluble, and hence oxalic acid is useful as a test for calcium, and is usually employed in the form of oxalate of ammonium; if the liquor to be examined contains any free acid, this must first be neutralized, as the oxalate can only detect calcium in neutral or alkaline fluids. Oxalic acid reduced by hydrogen is converted into glycolic and acetic acids, and if the action be kept up sufficiently long the glycolic becomes wholly formed into acetic acid. Oxalic acid may be detected in any solution, by being entirely volatilized by heat; by yielding a white precipitate with nitrate of silver, soluble in nitric acid; and by giving a white precipitate with lime water, which is insoluble in water, readily soluble in nitric acid, insoluble in acetic acid, and which, when dried and heated to low redness, is converted, without blackening, into carbonate of calcium.

Solution of sulphate of calcium produces a bluish-white precipitate with oxalic acid. Oxalic acid is sometimes contaminated with nitric acid, which gives a faint odor to it, and stains the cork of the bottle in which it is kept, yellow. If a very dilute solution of sulphate of indigo, containing the impure crystals, be boiled, the nitric acid present will decolorize the solution. On account of the resemblance between crystals of this acid and of magnesium sulphate, the latter has been used as an adulterant. This resemblance has also led to cases of poisoning, the person believing the acid to be Epsom salts. The acid may likewise be used for removing iron-rust and ink-stains

from linen, and is employed in calico printing as a bleaching and discharge agent.

SALT OF SORREL, Salt of lemon, or Essential salts of lemon (acid or binoxalate of potassium), is well known to the laity as an agent to remove iron-stains, though oxalic acid is generally used now, and is often sold under these popular names. Action and Toxicology.—Oxalic acid and the oxalates poison the nervous system and the blood, producing, as well, gastrointestinal lesions. A dose of 60 grains killed a boy (Taylor). Again, by prompt treatment, two cases recovered after a half ounce had been swallowed. Death takes place in varying lengths of time, a circumstance that can not readily be accounted for.

Some cases die in from 10 minutes to an hour. The above-mentioned boy died in 8 hours. The symptoms are an intensely pure, acid taste, burning of the parts over which the poison passes, intense pain, vomiting, especially a bloody material, an extremely feeble pulse, an inability to assume the upright posture, collapse, and stupor. These symptoms, with the rapidity with which death takes place, will point to oxalic acid as the cause. Still, persons have been known to live for 22 days, death being produced by a slow poisoning.

The post-mortem changes are a whitened oesophageo-gastric tract, though the stomach may contain a dark, gelatinous liquid, appearing like disorganized blood. The mucous coats are softened and loosened, but rarely perforated. The blood is excessively red, and in some instances oxalates have been found in the tubuli uriniferi of the kidneys. Koch regards it as a

heart poison. The same lethal symptoms may be produced from salt of sorrel.

Poisoning by oxalic acid, oxalate of ammonium, or oxalate of potassium, is best remedied by the speedy administration of chalk, suspended in water; when chalk can not be had, magnesia may be used; either of these forms insoluble oxalates. Medical Uses and Dosage.—This article, unless in great attenuation, is an unfit agent for internal administration, though it has been given in doses of three-quarters of a grain every 3 hours in diphtheria. Such a procedure is certainly dangerous, and an infusion of sorrel, sumac-bobs, and similar substances containing the acid in combination, would even be hazardous. These infusions have, however, given excellent results in diphtheria and certain sore throats when used simply as a gargle.

Webster (*Dynamical Therapeutics*, 175), praises the action of oxalic acid in lagging functions of the spinal cord due to over-exertion, thereby resulting in intensive pains, marked lumbar weakness, and insomnia. One or two grains of the 6x trituration every 4 or 5 hours, are recommended in these conditions. A solution of oxalic acid in water promptly removes iron stains. For a number of years past I have used a saturated aqueous solution of it as an external application in cutaneous cancer, acne, scald head, and several forms of cutaneous disease, since which, on my recommendation, others have employed it with success in similar affections, sometimes alone, and again with a small portion of creosote added. The saturated solution, neutralized by caustic potash, forms an excellent application to discuss indolent tumors (J. King).

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Preparation.—There are several methods by which oxalic acid may be procured; the following are considered among the best: 1. Gently heat 1 part of pure starch with 8 parts of nitric acid of sp. gr. 1.200 or 1.

250. A powerful reaction ensues, with an evolution of red nitrous acid vapors; when this diminishes, heat must be applied, and continued until no more red vapors are given off; if sufficiently evaporated, a large quantity of crystals of hydrated oxalic acid are deposited as the liquid cools. These are dried on a porous tile, then dissolved in a little hot water, and pure oxalic acid is deposited as the solution cools. The mother liquor remaining after the first deposit of crystals contains much free nitric acid, saccharic acid, and other products. 2. Digest 1 pint of sugar dried at 100° C.

(212° F.) with 8.25 pints of nitric acid of sp. gr. 1.380. Evaporate the mixture to a sixth, and leave to crystallize.

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manufacturing oxalic acid from sawdust has been devised by Mr. Dale, of England. It is as follows: two equivalents of caustic soda and one equivalent of caustic potash are mixed together, dissolved in water, and the solution evaporated until it has a sp. gr.

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4° C. (400° F.) for an hour or two, this stage of the process is complete. The mass has a dark color, contains from 1 to 4 per cent of oxalic acid, and about 0.5 per cent of formic acid; the balance of the mass consists of an unknown substance, which is intermediate between cellulose and oxalic acid.

The next stage consists in heating the mass till quite dry, being careful that no charring takes place; a gray powder is formed, containing from 28 to 30 per cent of oxalic acid, in combination with sodium and potassium. The gray powder is now washed on a filter with a solution of carbonate of sodium, which decomposes the oxalate of potassium, and converts it into oxalate of sodium, which is decomposed by boiling milk of lime, oxalate of calcium being precipitated while the sodium hydroxide remains in solution. The oxalate of calcium being placed in leaden vessels, is treated with sulphuric acid, which precipitates the calcium and leaves the oxalic acid in solution, which may be obtained in crystals by evaporation. The sodium hydroxide left in solution after the addition of milk of lime, may be recovered by boiling,

and be again made use of with fresh sawdust. The same may be done with the potassium salt which filters through in the last stage. By this process 2 pounds of sawdust are made to yield 1 pound of oxalic acid.

Description.—Oxalic acid crystallizes in colorless, transparent, oblique, quadrilateral prisms with two-sided summits. The crystals are inodorous, have a strongly acid taste, faintly effloresce in a dry atmosphere, redden litmus paper, and when pure are completely volatilized by heat, and without becoming blackened. They dissolve in from 8 to 11 parts of water at 15. 5° C. (60° F.

), in their own weight of water at 100° C. (212° F.), and in 4 parts of alcohol; the addition of a small quantity of nitric acid to the water causes them to dissolve more readily. Nearly all the oxalates are insoluble in water, excepting the alkaline. Oxalate of calcium is insoluble, and hence oxalic acid is useful as a test for calcium, and is usually employed in the form of oxalate of ammonium; if the liquor to be examined contains any free acid, this must first be neutralized, as the oxalate can only detect calcium in neutral or alkaline fluids.

Oxalic acid reduced by hydrogen is converted into glycolic and acetic acids, and if the action be kept up sufficiently long the glycolic becomes wholly formed into acetic acid. Oxalic acid may be detected in any solution, by being entirely volatilized by heat; by yielding a white precipitate with nitrate of silver, soluble in nitric acid; and by giving a white precipitate with lime water, which is insoluble in water, readily soluble in nitric acid, insoluble in acetic acid, and which, when dried and heated to low redness, is converted,

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This resemblance has also led to cases of poisoning, the person believing the acid to be Epsom salts. The acid may likewise be used for removing iron-rust and ink-stains from linen, and is employed in calico printing as a bleaching and discharge agent. SALT OF SORREL, Salt of lemon, or Essential salts of lemon (acid or binoxalate of potassium), is well known to the laity as an agent to remove iron-stains, though oxalic acid is generally used now, and is often sold under these popular names. Action and Toxicology.—Oxalic acid and the oxalates poison the nervous system and the blood, producing, as well, gastro-intestinal lesions.

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