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Preparation of Sucrose Octaacetate–A Bitter-Tasting Compound

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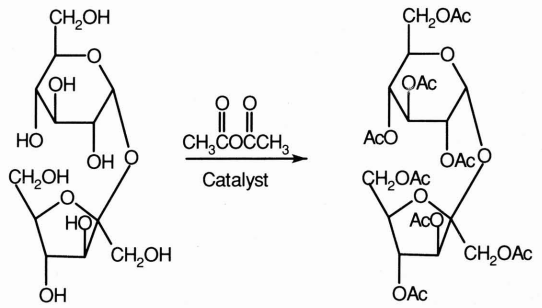
Sucrose (**1**) is the most abundant disaccharide found in plants and is prepared commercially from sugar cane, sugar beets, and the sap of the sugar maple. It is known for its sweet taste and is used in many foods as a sweetener. Acetylation of the eight hydroxy groups on this sugar change the sweet taste to the intensely bitter tasting compound, sucrose octaacetate (**2**).[2]



1 **2**

Sucrose octaacetate is a natural product that has been isolated from the roots of several species of *Clematis* (*1*). The root of *Clematis japonica* contains 0.15% of this compound by dry weight. Presumably, this bitter-tasting compound discourages predation on these plants. Humans have used this chemical commercially as a gustatory repellant because of this bitter taste. It has been used to denature alcohol (*2*), as a deterrent to fingernail biting (*3*), and to make sugar for animal feed inedible to humans (*4*). At a concentration of 0.06%, this compound makes sugar too bitter to eat (*5*). This compound has been shown to be nontoxic and does not effect the taste of the flesh of food animals or the milk of cows that consume it (*5*).

CAUTION: Do not taste the product of this experiment. If students want to taste this chemical it must be done with a commercial sample prepared for human consumption.[3] Also, it is so potent, that the bitter taste from a very small crystal will persist for a number of hours so it must be diluted before tasting. A procedure for tasting this compound is described in the experimental section.

This compound has been prepared by a number of different researchers using pyridine or sodium acetate as a catalyst (*6–8*). Procedures that use pyridine generally give a better yield than those that use sodium acetate.

Like many sugar derivatives, sucrose octaacetate can supersaturate solutions or form a syrup that resists crystallization. These problems can be solved by using seed crystals from a previous experiment to initiate crystallization.[4] It should be noted that sucrose octaacetate has two crystal forms. One form melts at 69–70 °C and the other at 89 °C (*6*). When the higher melting form is produced in a laboratory, all lower melting forms convert to the more stable higher melting form (*6*).

Experimental

*Sucrose Octaacetate (**2**)*

To a dry round-bottomed flask fitted with a reflux condenser and a drying tube is added 2.0 g (5.8 mmole) of sucrose, 1.0 g (12 mmole) of anhydrous sodium acetate and a boiling chip. Carefully add 10 mL (10.8 g, 106 mmole) of acetic anhydride and heat until the mixture just refluxes. (**Caution:** Wash with copious amounts of water if this comes in contact with skin. Remove the flame for a minute until the exothermic reaction is over and then continue heating. Keep the reaction refluxing until all of the reactants have dissolved (5–10 min). Then heat for an additional 5 min. Cool until the flask is warm to the touch and then pour the contents of the reaction flask into an Erlenmeyer flask that contains 50 g of ice and 50 mL of water. Use a glass stirring rod to stir the mixture for 5–10 min until the product collects as a thick syrup on the sides and bottom of the flask and the glass rod. Decant the water from the syrup. Add 100 mL of distilled water to the flask and stir for 5 min so all of the product is washed by the water. Decant this water wash and repeat twice using an additional 100 mL of distilled water each time. Carefully decant all of the final wash water from the product. At this point, there are two methods by which this compound can be crystallized depending on time constraint.

Method A

If the product must be obtained in a single lab period add 20 mL of 95% ethanol to the flask and heat on a steam bath. After the product has dissolved, cool it in an ice bath and add seed crystals when it is cold. Stir the solution for several seconds until the seed crystals have been evenly distributed and then let it stand until crystallization is complete. Collect it by suction filtration, wash with 5 mL of cold 95% ethanol, and air dry the product. Yield is 2.0 g (50%).

Method B

This method of recrystallization gives larger crystals and can be used if time allows. Add 10 mL of 95% ethanol to the flask. Heat this on a steam bath until the product dissolves. Stopper the flask and let it stand overnight at room temperature (20 °C). If the mixture fails to crystallize, add a seed crystal and wait an additional 24 h. The crystals are collected by suction filtration, washed with 5 mL of cold 95% ethanol and air dried. Yield is 2.4 g (60%).

The product has the following properties: FT–IR ($CHCl_3$), 2946, 1744, 1369, 1222, 1074, and 1043 cm^{-1} and mp = 89 °C (lit. 89 °C).

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[2]Other names for this compound are octa-O-acetylsucrose and the *Chemical Abstracts* entry name, 1,3,4,6-tetra-O-acetyl-β-D-fructofuranosyl-α-D-glucopyranoside, tetraacetate (*Chemical Abstracts* Registry No. 126-14-7)

[3]This compound is listed in the Aldrich Chemical Co., Flavors and Fragrances Catalog, Compound No. W30380-1.

[4]Seed crystals may be obtained from commercial sources or are available on request from W. F. W.

Taste Test

(Do not taste your product. Obtain a commercial sample of sucrose octaacetate prepared for human consumption.[3] To reduce the potency, this compound must be diluted before tasting.) Prepare a solution that contains 10 mg of sucrose octaacetate per 100 mL of ethanol. Wet clean filter paper with this solution, air dry and cut into 1 cm^3 squares. A piece of the paper must be chewed for about 30 s, because the bitter taste is not immediately evident.

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