inction angle of 42° . Consideration of the optical orientation, which is: obtuse bisectrix = axis b and acute bisectrix emerges through the base, making an angle of about 60° with the vertical crystallographic axis c ; this leads to the conclusion that these crystals are elongated parallel to the prism m , *i. e.*, that the habit is prismatic (instead of tabular, as more usual).

The following is a specific example of the method of procedure in obtaining these crystals:

Equal parts of California crude oil, about 20° B $\acute{e}$., and fuming sulfuric acid were mixed and agitated for 1 hr., the acid being preferably added gradually so as not to heat the mixture above room temperature. The mixture was then allowed to stand for several hours, until a solid tar-like material had separated out on top, the residual acid being in the bottom of the beaker. This acid, which amounted to about one-third the total bulk, was drawn off, and the tar-like material (probably due to the sulfonation of the unsaturated hydrocarbons of the oil) was then dissolved in hot water to form a solution of specific gravity 1.066, or about 6 per cent strength.

To a saturated solution of 200 g. of ordinary potassium chlorate were added 10 cc. of the above solution of water-soluble hydrocarbon, and the whole diluted with water to a volume of 800 cc. The solution was brought to a boil, filtered, and the filtrate allowed to crystallize. The resulting crystals were removed from the mother liquor, dried, redissolved in water, and recrystallized.

As will be apparent, the amount of this water-soluble hydrocarbon compound in the above example is very small, being less than one-third of one per cent of the amount of the original chlorate present. Even smaller amounts may be used to produce a like result. In some cases, particularly when concentration is effected by boiling the solution, some oxidation of the hydrocarbon occurs, and then more of the latter is necessary to alter the habit of crystallization.

The above procedure may be widely varied. Thus, for the manufacture of the water-soluble hydrocarbons, instead of fuming sulfuric acid, concentrated sulfuric acid or liquid sulfur dioxide may be used, the amount of acid needed varying through wide limits, as does the temperature at which the reaction may be effected. The tests have shown that all grades of California oil, from the residuum of topping plants to the very light oils found in some fields, may be used to produce the soluble hydrocarbons above referred to. These crystals were also produced from neutral solutions of the soluble hydrocarbons, as when neutralized by caustic soda or ammonia.

Various stages in the transformation of the crystals from plates to fibers have been obtained by using an insufficient amount of the hydrocarbon, or some which had been partially oxidized.

Preliminary tests of these crystals as to explosion by friction were made by grinding them with sulfur in a wooden mortar with a wooden pestle, and the results seemed to indicate less sensitiveness, but further tests made with the frictional pendulum at the Bureau of Mines showed no essential difference under standard conditions. It is possible, however, that these crystals

might be better adapted to being coated with a protecting film, which would make them less sensitive. It was originally planned to use the water-soluble hydrocarbon for this purpose. However, the tests were discontinued at the signing of the armistice, and these possibilities were not investigated.

A TEST FOR ANNATTO IN FATS AND OILS¹

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The usual test for annatto in butter, oleomargarine, and other fatty foods, in which the clarified fats are mixed with sodium hydroxide solution, the mixture of the two allowed to pass through filter paper, and the dried paper tested for annatto with stannous chloride solution, is somewhat unsatisfactory. This is due to the fact that the paper becomes saturated with the fat and oftentimes so large an amount of fat remains in the paper that the sodium hydroxide solution with the annatto does not get an opportunity to come in contact with greaseless paper fiber so that it may be properly absorbed. Consequently, when the stannous chloride solution is dropped on the dry filter paper, the pink color is oftentimes faint or obscure, even when there is plenty of annatto present in the sample to give a positive test.

The above test being at times quite unsatisfactory, the writer tried some modifications in the endeavor to render it more positive and consequently more satisfactory. The method described below has been in use for some time, and has proved quite satisfactory for the identification of annatto in butter, oleomargarine, cream, ice cream, and some other food materials. The procedure is quite simple and does not consume much time or material. The annatto is removed from the fat, and subsequent manipulation is free from the general messiness of oil and fat determinations. Small amounts of annatto that might easily escape identification by the older method are easily and positively identified.

METHOD

Have ready some paper pulp made by disintegrating a fair grade of filter paper in water. The ordinary grade of Munktell Swedish paper answers very well. Too coarse a paper makes a lumpy pulp.

Prepare a solution of sodium hydroxide by dissolving 5 g. of sodium hydroxide in 95 cc. of water.

Prepare a stannous chloride solution as follows: Saturate concentrated hydrochloric acid with tin, dilute with an equal volume of water, and from time to time add a slight excess of acid. Keep pieces of tin in the reagent bottle.²

In a large test tube holding at least 60 cc. place 15 cc. of the melted and filtered fat, free from moisture, salt, and curd. Add to the fat 15 cc. of chloroform and mix well. Add 15 cc. sodium hydroxide solution, cork the test tube, and shake thoroughly for a few

¹ Presented by title before the Division of Agricultural and Food Chemistry at the 60th Meeting of the American Chemical Society, Chicago, Ill., September 6 to 10, 1920.

² Leach, "Food Inspection and Analysis," 3rd Edition, p. 32.

moments. The cork must be held tightly in the test tube while shaking.

Remove the cork and immerse the tube nearly its full length in a bath of water at 50° to 60° C.

Allow the tube to remain in the hot water until the emulsion is pretty well broken up or until no fat drops can be seen dropping from the bottom of the soap froth when the tube is given a slight shake. If there is a large amount of annatto present it will not all be in the soap froth, though there will be plenty for a test.

Remove the soap froth from the tube to a small beaker with a spatula or small spoon, add 10 cc. of water and 2 cc. of the sodium hydroxide solution. Then add enough of the paper pulp to make a thin felt in a Gooch crucible. Let stand on the steam bath for about one-half hour with frequent stirring and the annatto will be absorbed by the paper pulp. If the quantity of annatto is small, it is well to leave the beaker on the steam bath until the liquid has concentrated to about one-third its original volume. In this way a more positive test is obtained with a small quantity of annatto.

Filter with light suction on a Gooch crucible containing a small disk of filter paper. When the liquid has passed through, if annatto is present, the paper pulp will have an orange color. At once drop a few drops of stannous chloride solution on the paper and again suck dry. If annatto is present the paper pulp will be colored pink. Reverse the crucible and blow the felt out in the palm of the hand. The mat may then be dried if it is to be preserved. In case the available sample is small the quantities used in the test may be reduced.

One author recommends 5 per cent citric acid in place of stannous chloride and this was found to work well.¹ Boric, tartaric, acetic, sulfuric, hydrochloric, and nitric acids of from 3 to 5 per cent also give satisfactory results. Even so dilute an acid as that obtained by blowing one's breath through water for a few minutes may be used, but its action is slower than with stronger acids. The stannous chloride solution, however, gives better results than anything else tried.

There is an aqueous solution of annatto on the market that is used by ice cream manufacturers. A few drops of this may be added to 15 cc. of colorless oleomargarine freed from moisture, salt, and curd, and the test run as described. We have used the test in this way on some food materials after extracting the annatto from the bulk of the material.

An easier way to handle an aqueous solution of annatto is to dilute the solution and make slightly acid with hydrochloric acid. The annatto will precipitate in a form resembling ferric hydroxide. Filter this on a soft Munktell paper about the size of a silver dollar. Wash once with water and place paper and precipitate in a small beaker with 10 cc. of water and 2 cc. of the sodium hydroxide solution, and proceed as usual. The small filter paper can be disintegrated

with the help of a glass rod and will furnish the necessary paper pulp.

After the soap froth has been removed from a butter or oleomargarine determination it will sometimes be noticed that the sodium hydroxide layer over the chloroform-fat mixture still has a deep color. This is the case when the annatto is present in large amounts. If desired, this sodium hydroxide layer may be pipetted off and annatto tested for as in an aqueous solution. The acidification of the solution should be carefully done and the solution placed on the water bath so that the fine precipitate may collect. It will then filter quite easily.

BENZYL SUCCINATE: PRELIMINARY REPORT ON ITS COMPOSITION, MANUFACTURE, PROPERTIES, AND PROBABLE THERAPEUTIC USES¹

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The extremely important pharmacological studies of Macht have recently led to the clinical application of benzyl esters—especially benzyl benzoate—in the treatment of cases of excessive peristalsis, or excessive spasm of smooth muscle, with surprisingly gratifying results. However, certain objections to the use of benzyl benzoate have arisen which make desirable the finding of less objectionable substitutes. The benzoate is a fluid of disagreeable taste and odor, practically insoluble in water. It is highly objectionable to most patients, and cannot be tolerated by some. By the mouth, it must be administered in a flavored solution of alcohol, or as a flavored emulsion. It may cause vomiting, or may develop severe gastric disturbances if used in large amounts or over a considerable period of time. Gelatin capsules containing the benzoate dissolved in oil are also given, but are open to one or more of the preceding objections.

With these facts in mind, and being especially interested in the production of various compounds of succinic acid, the writer was prompted to take up the study of benzyl succinate.

This substance was prepared, with certain modifications, according to the method of Bischoff and von Hedenström, by heating succinic acid with benzyl alcohol. The succinate was obtained in the form of beautiful snow-white crystals, with a very slight aromatic "benzyl" taste, and with practically no odor. The percentage of benzyl group in benzyl succinate is 61.08, or considerably greater than that present in benzyl benzoate (42.89 per cent).

Investigations so far show that benzyl succinate may be administered by the mouth in powdered, tablet, or capsule form, without fear of nausea or other intestinal disturbances, even when given in large doses or over considerable periods of time.

Exhaustive preliminary experiments have demonstrated that this product is practically nontoxic. Guinea pigs inoculated subcutaneously with as much as 4 cc. of a 12.5 per cent alcoholic solution of benzyl succinate became perfectly normal after several

¹ Presented before the Division of Medicinal Products Chemistry at the 60th Meeting of the American Chemical Society, Chicago, Ill., September 6 to 10, 1920.

¹ Bolton and Revis, "Fatty Foods, Their Practical Examination," p. 113.