

pigment from the fiber, which, if ground wood pulp be absent, and if the concentration of the caustic be regulated, and if the temperature be not too high, serves to remove a large proportion of carbon ink. Too great a concentration may bring about some mercerization. More weakly alkaline solutions, for example, sodium silicate, sodium phosphate, borax, soap, etc., also lift the ink in part and do little damage to the fiber. However, the detergent effect calls for scouring or rubbing, which so embeds the carbon in the fiber as to make it almost impossible to separate the two.

Certain solvents, as kerosene or gasoline, tend to loosen the ink by dissolving the binder. This may be combined with an alkaline solution, for example, a borax or a soap solution. During agitation the suds or skim, which forms on the surface of the water and entangles the carbon particles, may be washed away.

Rosin is extensively used as a filler and binder for the fibers of the paper, which have a "surface." As mentioned, some of the cheaper magazine papers contain wood pulp, which retains natural gums and resins. They serve in part as binders for the ink pigments. Pine oil is one of the normal solvents for rosin, gums, and resins, so its addition to the old printed matter helps materially to lift the ink.

In practice in reclaiming bookstock, we have therefore used borax (10 lbs.), soap (10 lbs.), kerosene (2 gal.), and pine oil (2 gal.) to 2000 lbs. of bookstock in water to make a 3 to 6 per cent pulp. The stock is soaked and gently pulled apart in a beater or other device, thus reducing the mechanical injury to the fibers to the minimum. Time is saved by heating the mixture up to 75° to 90° C. by introducing live steam. After pulping, which requires one hour or less, depending upon the machine used, the ink and chemicals are washed away by one of several well-known washers. The pulp may then be bleached or tinted as desired. A selected combination of the chemicals may be used instead of all four with selected lots of waste paper when the composition (including ink and the binder) is known.

A superior product of desired strength, length of fiber and cleanliness has been obtained by the process.<sup>1</sup>

The City of New York, through the Board of Education and Bureau of Vocational Activities, has established a textile school under the direction of William H. Dooley, who has had considerable experience in the textile industry and in the establishment of similar schools in other cities. Day courses extending over two years are offered in marketing of textiles, costume design, general textiles, applied textile design, chemistry and dyeing, textile manufacturing and engineering, and knitting and sweater course. Evening courses open only to those engaged in the trade include woollens and worsteds, cotton converting, general cotton, textile chemistry, experimental dyeing, loom-fixing, fabric analysis, costume design, draping costume design, garment design, operating sweater and knitting machinery, general knitting, and applied textile design. A complete experimental dye laboratory has been donated by H. A. Metz & Co.

<sup>1</sup> U. S. Patent, 1,351,092.

## A NEW CRYSTALLINE FORM OF POTASSIUM CHLORATE<sup>1</sup>

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Crystals of potassium chlorate having a long, fibrous, silky appearance, as distinguished from the plates of the ordinary form, have been prepared by dissolving the latter in water and adding thereto an aqueous solution of hydrocarbons such as is obtained by treating crude petroleum (California) with concentrated sulfuric acid and slowly concentrating this mixture as on a water bath, until the potassium chlorate crystallizes out. In the presence of an excess of the water-soluble hydrocarbon, the potassium chlorate appears in the form of long silky fibers, as shown in the accompanying illustration. These crystals do not always separate out in parallel groups as shown, but may, under certain conditions, separate out radially from various nuclei.



The crystalline form of these crystals has been investigated by Dr. E. T. Wherry, who reported as follows:

The essential optical properties of these crystals are in every respect identical with those of a typical commercial sample ("analyzed reagent") of the salt. The refractive indices are:  $\alpha$ , 1.440;  $\beta$ , 1.515;  $\gamma$ , 1.525, making the double refraction 0.080. The optical axial angle  $2E$  is 45°, and the sign negative. The same exact numerical values are given by both samples. The two substances must, therefore, be identical, in so far as crystal system is concerned, and the difference lies merely in the habit of relative development of different forms. Ordinary potassium chlorate is crystallized tabular parallel to the base,  $c$ , 001, the plates being bounded chiefly by faces of a prism,  $m$ , 110. In parallel polarized light, nicols crossed, the extinction angle of such crystals, with reference to this prism, is 38°; with reference to the faces of a side dome sometimes present, the extinction angle is parallel (straight). The sample in question shows, however, long rod-shaped crystals, most of them with an ex-

<sup>1</sup> Published by permission of the National Research Council.

These crystals were first produced in the laboratory of the Western Precipitation Company, Los Angeles, Cal. Production and tests were continued under the direction of Dr. Charles E. Munroe, chairman of the Committee of Explosives Investigations of the National Research Council.

inction angle of  $42^\circ$ . Consideration of the optical orientation, which is: obtuse bisectrix = axis  $b$  and acute bisectrix emerges through the base, making an angle of about  $60^\circ$  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  $20^\circ$  Bé., 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 OILS<sup>1</sup>

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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.<sup>2</sup>

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

<sup>1</sup> 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.

<sup>2</sup> Leach, "Food Inspection and Analysis," 3rd Edition, p. 32.