as guides, prepare three fresh portions of 10 grams each and add linne-water as before, except that the amount added to a dish differs from that added to another by 1 or 2 cc. Dry, take up with 100 cc. of water, allow to stand, draw off, and treat exactly as before. The smallest amount of linne-water which gives the characteristic pink color is taken as the acidity equivalent of the soil. Each cubic centimeter of standard linne-water is equivalent to an acidity of 0.01 per cent. expressed as calcium oxide.

It is essential that the distilled water used be free from alkalies and acid.

## THE USE OF BASIC ALUMINUM ACETATE AS A PRESERV-ATIVE IN SAUSAGE.

BY ED. MAC-KAY CHACE.

Received March 30, 1904.

IN THE course of the usual work in this laboratory upon imported foods, the writer some time since received a sample of imported canned sausage which was preserved with a salt of aluminum. As aluminum compounds are not among those usually employed as preservatives in canned meats, an investigation was started in order to ascertain the extent of their use, the amount used, etc.

Up to the time of the present writing they have been found in several samples of imported canned sausage, all from the produce of two manufacturers, however. They have not been found in any samples of imported smoked, or domestic canned or smoked sausage.

The two manufacturers in whose goods aluminum was found acknowledge the use of the basic acetate of aluminum in small quantities. The detection of the addition of aluminum is easily carried out inasmuch as it is not a normal constituent of the ash of flesh.<sup>1</sup>

The following method was found to work satisfactorily: About 25 grams of the ground sausage are ashed (complete ashing is not necessary) and the ash dissolved in strong hydrochloric acid. Sodium hydroxide is then added in excess and the whole boiled, the precipitate and insoluble ash filtered off, the filtrate made

<sup>1</sup> See the works of Bunge and Halliburton on "Physiological Chemistry."

662

acid with hydrochloric acid and the aluminum precipitated with ammonia, partly as hydroxide, partly as phosphate. The precipitate may be filtered off and tested on charcoal with cobalt nitrate. Whenever possible, the work should be carried on in platinumware to avoid solution of aluminum from glass or porcelain and the reagents should always be tested.

With the amount added as a preservative no trouble was found in detecting it in the above manner, enough of the precipitate being obtained to render further identification possible, if desired. For the quantitative determination it was found that the method of Wachenroder and Fresenius gave accurate results.<sup>1</sup>

The finely ground sausage is heated over a low flame until danger of spurting is past, when the mass is thoroughly charred. It is then cooled and digested on the water-bath for some time, with strong hydrochloric acid, filtered, slightly washed, and the filter and residue reignited. This ash is usually of a good gray color and small in quantity; it is dissolved as before, in the strongest hydrochloric acid, filtered, and the filtrate added to the former one. Any residue left on the filter-paper should be examined further for aluminum.

The combined filtrates obtained above are made slightly alkaline with ammonia, and barium chloride added until no further precipitate is formed, the hydroxide and phosphate of aluminum and the phosphate of barium filtered off, slightly washed, and dissolved in the least possible amount of hydrochloric acid. The solution obtained is saturated with barium carbonate, potassium hydroxide added in excess, and the whole digested for some time. Finally, the barium in solution is precipitated with sodium carbonate, the phosphate and carbonate of barium filtered off, and thoroughly washed. The filtrate is acidified with hydrochloric acid and the aluminum determined in the usual way.

The cans upon which the quantitative determinations were carried out were of the one-pound type, containing four sausages, weighing from 400 to 450 grams, and from 175 to 200 cc. of liquor. The sausages were removed from the liquor, and ground, without removing the casing. The liquor was evaporated to dryness, ashed, and the aluminum determined, as in the sausage. The amount of the preservative found was as follows: No. I averaged on several different lots, 11.2 mg. of Al<sub>2</sub>O<sub>3</sub> per 100

<sup>4</sup> See Fresenius' "Quantitative Analysis," American edition of 1904, Vol. I, p. 4<sup>2</sup>9.

grams of sausage, and 12.6 mg. to the total liquor, making the entire content in the can between 60 and 70 mg. No. 2 averaged, on the two lots examined, 31.3 mg. per 100 grams of sausage, and 54.3 in the total liquor, making 175 to 200 mg. to the can.

The manufacturer of No. 2 claims to add 0.5 per cent. of the basic acetate. It is supposed that the solution used is that of the German Pharmacopoeia, the preparation of which is carried out according to the following formula

	Grams.	
Aluminum sulphate	· 30	
Water	· 100	
Calcium carbonate	. :3	
Acetic acid	. 36	

This solution would contain about 5 per cent. of aluminum oxide by weight, and if added to the extent of 0.5 per cent. would introduce 25 mg. of aluminum oxide per 100 grams of sausage, slightly less than the amount found.

It is quite possible that in time the sausage might absorb the entire amount of the aluminum salt contained in the liquor by reason of its diffusion through the casing. When liquor containing the preservative comes in contact with the ground sausage the aluminum is fixed by the constituents of the meat as a compound which is insoluble in boiling water, and boiling hydrochloric acid of tenth-normal strength, and only slightly soluble in a mixture of equal parts of the strongest hydrochloric acid and water. To illustrate: 50 grams of sausage were mixed with the liquor from a can (190 cc. in volume) and allowed to stand for forty-eight hours in the refrigerator of the laboratory, the liquor filtered off, ashed and the aluminum tested for, with negative results. In this connection there have been noted in the laboratory instances of the apparent diffusion of other preservatives through the casings of sausages, manufacturers having alleged that boric acid had been used by them in the liquor only and that the edible part of the product would not become contaminated. In every case, however, we have found considerable quantities of boric acid in the sausage itself, although in less amount than was present in the liquor.

The claim is made for this, as for every other preservative, that it is harmless. It is not the purpose of this work to add

664

more testimony to that already collected on the injurious effect of soluble aluminum salts upon the human digestion. The consensus of opinion is that such salts retard the digestion, both in the stomach and intestines.

The only question to be answered then is as to the solubility of the salt present in the digestive fluids. As has been stated, it is insoluble in boiling water and dilute hydrochloric acid. If, however, 50 grams of the sausage be treated for four hours at  $40^{\circ}$  C. with 100 cc. of 0.33 per cent. hydrochloric acid containing 0.1 gram of Merck's pepsin, filtered, a portion of the filtrate ashed, and tested for aluminum, no small quantity of that metal will be found.

To determine the proportion of the preservative which would be dissolved in the stomach during digestion, the following experiments were tried: 50 grams of the two samples (Nos. I and 2), finely ground, were digested with 200 cc. of the above pepsin solution for twelve hours, an aliquot portion filtered off, and the aluminum determined. Sample No. I contained 14.9 mg. of aluminum oxide per 100 grams of the sausage, of which 12 mg. were dissolved by the solution, or 82.7 per cent. Sample No. 2 contained 34 mg. of aluminum oxide per 100 grams of sausage of which 24 mg. were dissolved, or 70.3 per cent., showing that a very large proportion of the compound is dissolved during the process of digestion and becomes a retarding factor. Acknowledgment is herewith made to Dr. W. D. Bigelow for valuable suggestions in planning this work.

FOOD LABORATORY, U. S. DEPT. OF AGR.

## THE DETECTION OF MINERAL OIL IN DISTILLED GREASE OLEINES.

## BY AUGUSTUS H. GILL AND STEPHEN N. MASON. Received April 6, 1904.

IN THE woolen industry a large quantity of soap and oil is used in cleansing, oiling and milling the wool, yarn and cloth, during the different processes of manufacture.

Formerly all the waste from this washing went into the streams and was lost, also polluting the water, and making it unfit for other purposes. At the present time this waste is recovered, and after suitable purification, is used over again.