

THE NITROGEN METHOD

From what has been said it is evident that any method of crucible heating by which air is in contact with the graphite is not accurate. The true value of the volatile matter in graphite can be determined only by heating in an atmosphere of pure nitrogen, hydrogen, or some other inert or nonreducible gas. This is best carried out by placing about a 1-g. sample in a platinum boat, and placing the boat in a silica tube through which a steady stream of pure nitrogen is passed. When the air has been completely expelled, the tube under the boat is heated for 30 min. with a 30-mm. Méker burner. It has been shown that 15 min. is long enough, but a half hour is recommended as a precaution. Various forms of heating and temperature control were tried, such as electric and gas heated combustion furnaces, electric tube furnace, multiple burners, etc., but the single Méker burner was found to be sufficient. It is important that pure nitrogen be used. Attempts to use commercial nitrogen containing 3.5 per cent of oxygen gave inaccurate results, the amount of loss depending upon the time of heating.

SUBSTITUTE METHOD

The nitrogen method will consume over an hour and will

require the preparation and purification of nitrogen, so that as a working method the following is suggested: Place a 1-g. sample of the graphite in a platinum crucible provided with a well-fitting lid. Support this in such a way that it can be heated to 700° to 750° C. (about 10 mm. above a good Méker burner), and heat for exactly 30 sec. As a method for the determination of volatile matter in coke and anthracite, the short heating process is entirely unsatisfactory.

SUMMARY

1—It is pointed out that the method of determining volatile matter in graphite by heating in a crucible is rendered inaccurate by the oxidation of the graphite.

2—The error is dependent on the temperature, and time of heating, as well as on other factors.

3—The method of heating in a boat in an atmosphere of nitrogen gives accurate results, but is time-consuming, and requires pure nitrogen.

4—Results very close to the true value may be obtained by heating in a crucible in the air under certain stated conditions.

Discoloration in Canned Sweet Potatoes¹

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The producer of canned foods must impart to his goods at least two prime and essential qualities before he can consider himself in any measure successful in his art. The first requisite is wholesomeness, secured by the sanitary packing of sound raw material when it is in that particular state of maturity most suitable for food. The second is palatability and appearance. On first thought it might seem that palatability and appearance are a necessary consequence of wholesomeness. This is not always the case, as is illustrated by various forms of discoloration in canned foods.

The discoloration in canned sweet potatoes is of this character. Sweet potatoes frequently become black in the can, unaccompanied by any other noticeable feature. Then discoloration apparently begins where the potatoes are in contact with the can, but may eventually permeate its entire contents. As there is usually a small amount of semi-liquid starch paste in the bottom of the can, the discoloration is more pronounced there in the early stages, as the result of closer contact. How soon after packing discoloration sets in is a variable factor. From the experiments below, it would seem that in extreme cases it may occur very early.

All foods evolve hydrogen sulfide upon being heated. This sometimes leads to a black discoloration in canned foods, due to the formation of ferrous sulfide. From general appearance one cannot distinguish the discoloration of canned sweet potatoes from that due to the formation of ferrous sulfide, but a few qualitative tests sufficiently demonstrate its entirely different character. If black sweet potatoes are treated with dilute hydrochloric acid, the color is discharged, but when the acid is neutralized with ammonia, even after heating to drive out any hydrogen sulfide, the black color is again obtained. Hydrogen peroxide does not bleach the discolored sweet potatoes.

EFFECT OF AIR

Into a number of cans of sweet potatoes from each of two distinct commercial packs, both of which were normal in

every way, air was admitted under sterile conditions by a very small puncture. The cans were placed in boiling water for some time to relieve the vacuum and thus to prevent the rapid inrush of air which would augment the chances of contamination. Alcohol was then burned on the top of the can. Just as the last had burned away, a puncture was made, and this was at once covered with a pad of sterilized cotton which was held in place by setting another can upon it. These cans were left undisturbed for periods of time ranging from a week to a month. At the end of a week, typical and distinct black discoloration of the sweet potatoes had set in in the bottom of the can, where there was the most moisture and the best contact between the can and its contents. This became more and more abundant as time elapsed. After varying periods of a week or more, some of the cans were resealed. These resealed cans retained the black that had formed in them to the end of the experiment. It could not be decided whether there was any increase of the black in the cans after resealing, because only a limited number of cans were used in the experiment.

This experiment makes very evident the necessity of air-tight seams in the cans. It does not answer the question as to the manner in which the admitted air brings about the discoloration, whether by direct action upon the sweet potatoes or by aiding the solution of the iron, which in turn is the immediate cause.

EFFECT OF IRON SALTS

To throw more light upon this point, sweet potatoes were canned with the addition of iron salts. The sweet potatoes were steamed in the usual way until soft, then peeled, and packed into No. 3 cans and into quart glass fruit jars. A varying amount of concentrated iron solution ranging from 100 mg. to 700 mg. per can was then added. It had already been found by analysis that black sweet potatoes contained amounts of iron as high as this. When this solution of iron was poured over the potatoes they immediately became black. Where a thick peel had been removed or where the ends were cut away, there was no appreciable darkening.

¹ Presented before the Division of Agricultural and Food Chemistry, at the 61st Meeting of the American Chemical Society, Rochester, N. Y., April 26 to 29, 1921.

After processing, however, the entire potato was more or less permeated.

EFFECT OF PEELING

This seemed to indicate that the substance that reacted with the iron to form the black compound was near the outer surface of the potato. Another lot was peeled rather thin in the raw state, as it was thought that the causative substance might permeate the entire potato during steaming. Then another moderately thick coat was peeled away. The potatoes and this second coat were steamed until tender and packed separately into glass jars, and iron was added to them as described above. The potatoes did not remain entirely free from discoloration, although it was very slight before processing and decidedly less after processing than with potatoes with only the usual amount of peel removed. The second coat that had been removed became very dark when iron was added.

That the troublesome substance was not all found near the outer surface was made clear by cutting raw sweet potatoes in cross-section and treating the cut surfaces with iron solution. Very soon a dark ring formed just inside the peel, and dark spots also appeared near the center. A region between the center and the outer portion remained almost free from discoloration. When the raw sweet potatoes were cut in cross-section a milky juice exuded in spots about the center. These spots were coincident with those where discoloration occurred when iron solution was applied.

Apparently the juice of the sweet potato contains a tannin-like substance which, in combination with iron, causes the discoloration. Any factor, such as a breathing can or a poor exhaust, which tends to hasten the solution of iron from the can, will augment discoloration. If no air is admitted into the can after sealing, any iron going into solution would be in the ferrous state. This does not form black compounds with tannins. Ferrous iron would very soon be oxidized upon opening the can, and it seems probable that the contents might then darken, but no such phenomenon has come under our observation.

SUMMARY

The black discoloration in canned sweet potatoes is of a different character from that caused by the formation of ferrous sulfide in certain canned foods. It is the result of the combination of a tannin-like substance in the sweet potatoes with iron from the can. Since the production of this color with tannins requires iron in the ferric state, the access of air to the can is a prerequisite of its formation. The necessity of tight seams is therefore emphasized.

The substance which is involved in the discoloration is localized largely just under the peel. Since tannin probably permeates the potatoes to some extent during steaming, and since some is also found near the center of the potato, it does not seem possible to avoid this trouble by removing a thicker peel, either in the raw state or after steaming.

Composition of Hollyhock Seed and Oil^{1,2}

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The seeds of the hollyhock (*Althaea rosea*), as they mature on the stalks, are eaten by poultry, sparrows, and wild birds. Nothing can be found in the literature concerning the composition and food value of such seeds, or the character of the oil. As the hollyhock is a member of the mallow family (Malvaceae), it was thought possible that the oil from the seed might have qualities similar to those of cottonseed and kapok oils, which are derived from plants belonging to the same family.

The following was found to be the empirical composition of mature seeds threshed from pods of different varieties:

	Per cent
Moisture	4.4
Ash	6.9
Ether extract	11.9
Crude protein	21.2
Crude fiber	25.6
Starch	9.1

The ether extract was an oil with a greenish yellow color similar to that of raw linseed oil. The unrefined oil had at first a bland taste, but later developed an unpleasant bitter flavor. It showed the following properties:

Refractive index at 25° C.	1.4722
Specific gravity 15.6°/15.6°	0.9275
Iodine number	119.0
Halphen test	Positive
Bechi test	Positive

Like cottonseed oil, the hollyhock seed oil, after being heated for 10 min. at 250° C., no longer gave a color reaction with Halphen's reagent.

The most interesting point developed in this study was that the oil gave a strong, positive Halphen test, and also responded positively to Bechi's test. This fact is interesting for two reasons: It shows that the Halphen and Bechi tests are not distinctive of cottonseed oil exclusively, and it fur-

nishes additional evidence in support of the view that the Halphen test is peculiar to the oils from the seeds of plants belonging to the mallow family¹ (Malvaceae) and the related bombax family (Bombaceae). Lewkowitsch² admits that the Halphen test cannot be considered as exclusively indicative of cottonseed oil, since kapok oil from *Bombax ceiba* or *Eriodendron anfractuosum* gives a similar color reaction.

So far as known, the chromogenic substance in cottonseed and kapok oils that gives the carmine color when heated with Halphen reagent has never been isolated or identified. Possibly the oils from the seeds of some of the other Malvaceae contain a larger amount of the chromogen, and a study of them might serve to reveal the identity of that substance. Members of this family which are commonly grown in this country are the okra (*Hibiscus* or *Abelmoschus esculentus*), the oil from the seeds of which has been recently described by Jamieson and Baughman;³ the rose mallow (*Hibiscus moscheutos*); the shrubby althaea (*Hibiscus syriacus*); the musk mallow (*Malva moschata*); and the weeds, cheese mallow (*Malva rotundifolia*); and velvet mallow (*Abutilon theophrasti*).

It is doubtful if hollyhock oil will ever be an article of commerce, as the seed contains less than 13 per cent of oil, about half as much as is contained in cottonseed. On account of the quantity of ether extract and protein present, however, the seed might serve some useful purpose as a feedingstuff. While it is very light and bulky, individual plants seem to yield heavily, an average of about 100 g. of seed per plant being obtained from six plants.

¹ Ivanov and Kokotkina, "Physiological Characters of Plants and Botanical Families;" Ivanov and Moshkova, "Data concerning the Character of Seeds of Different Varieties of Cotton," C. A., 11 (1917), 2917.

² "Oils, Fats and Waxes," 3rd Ed., II, 534.

³ J. Am. Chem. Soc., 42 (1920), 166.

¹ Received April 22, 1921.

² Published by permission of Secretary of Agriculture, U. S. Department of Agriculture.