

take out the tin with nitric acid, the lead as sulphate, copper by electrolysis and iron with ammonia as usual. Add five grams of ammonium chloride. Make the solution just neutral with hydrochloric acid, and add four-tenths of a gram of dimethylglyoxime dissolved in a little alcohol for every tenth of a gram of nickel supposed to be present. Add ammonia, drop by drop, until the solution smells slightly ammoniacal. Let stand just below the boiling point for one-half hour (more or less according as the nickel separates completely). Filter hot through counterpoised filters or on a weighed gooch, wash with hot water and dry in the air bath at 105° C. to constant weight. Weigh and calculate the nickel.



Until a little experience has been acquired, the filtrate should be tested to make sure that no nickel has escaped precipitation. Add 0.05 gram more of the dimethylglyoxime in a little alcohol, see that the solution is just ammoniacal and let stand for a few minutes. There should be no reddening of the solution, which would mean more nickel. Make the filtrate just acid with concentrated hydrochloric acid and add an excess of ten cubic centimeters. Boil for ten minutes to break up the dimethylglyoxime in the solution, add ten grams of microcosmic salt dissolved in a little water, neutralize with ammonia and hydrochloric or acetic acid until the solution neither turns blue litmus paper red nor red paper blue, and let stand just below the boiling point (do not let bump) until the precipitate has become granular. Filter under suction, wash with hot water, ignite in a weighed porcelain crucible, cool and weigh as zinc pyrophosphate.  $\text{Zn}_2\text{P}_2\text{O}_7 \times 0.42913 = \text{Zn}.$

Dimethylglyoxime can be bought for \$2.50 per ounce.

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#### A RAPID VOLUMETRIC METHOD OF SUGAR ESTIMATION.

By W. F. SUTHERST.

Received February 14, 1911.

While carrying out a number of sugar estimations in citrus fruits, at the Experiment Station of the Bonita Union High School, Lordsburg, Cal., an immense amount of time seemed to be taken up in finding the point when all the Fehling solution was reduced. The writer adopted the following method:

Fifty cc. of normal Fehling solution were heated to boiling in a porcelain casserole, and the trial sugar solution run in from a small burette or graduated pipette, till a faint blue color is apparent at the edge of the vessel. From this to the finish as further sugar is run in, a drop of the mixture is placed on the top side of a filter folded in half; the filtrate passes through, free from copper oxide, to the under side and the spot moistened with a drop of a solution containing 10 grams glacial acetic acid, 1 gram potassium ferrocyanide in 100 cc. water. On holding up to the light the faintest trace of copper ferrocyanide can be perceived, and the end of the reaction plainly indicated at the first trial.

#### THE IMPORTANCE OF A STANDARD TEMPERATURE FOR SPECIFIC GRAVITY DETERMINATIONS, AND FOR STANDARDIZING MEASURES OF CAPACITY.<sup>1</sup>

By G. W. THOMPSON.

Received February 11, 1911.

Chemists generally appreciate the advantage of the metric system of weights and measures. Weights and measures in this system are related so simply that the terms, in many practical cases, become interconvertible.

In this country, where the old system of weights and measures prevail, the common measure of capacity is the gallon. The Bureau of Standards and its antecedents adopted the temperature of the maximum density of water as that at which gallon measures should be standardized. A gallon is 231 inches. A great many materials, such as oils and prepared paints, are sold by measure. In very many cases these materials have values dependent on measure. Thus, in the case of raw linseed oil, commercially a gallon weighs 7.50 pounds, although the actual weight is 7.75 pounds. Some purchasers, however, insist on a gallon close to 7.75 pounds. The movement which is growing in strength for the sale of all commodities by accurate net weights and measures makes it very desirable that weights should be as far as possible convertible to measures.

On the other hand, there are probably cases where weights cannot so readily be determined as volume, in which case the ready conversion from volume to weight is a thing to be desired.

Considering these ends as of great commercial desirability, the question arises as to the best means to be used to bring them about. If specific gravity determinations were made at 4° C., it would be a simple matter to calculate the weight of a gallon at that temperature by multiplying that specific gravity by 8.336, the weight of a gallon of water in air. There are two difficulties, however, in the way of this procedure. One is that 4° C. is not a good temperature at which to make specific gravity determinations. Furthermore, with very few materials are their volume values fully measurable at that temperature. The volume value of a material should be measured at that temperature at which it is used.

Calculations have been made of the capacity of gallon measures at 20° C. standardized to contain 231 cubic inches at 4° C. It is found that such a copper measure contains at 20° C. 231.19 cubic inches and a similar brass measure 231.20 cubic inches. There are good reasons for basing our calculations at 20° C. although 25° C. has its advocates.

An important phase of this subject which should be considered is that most liquids expand with a higher coefficient of expansion than the materials from which measures are made. Commercially, therefore, the higher the temperature at which a material is measured, the less the weight will be. It might be considered a hardship to the consumer if a relatively high temperature should be adopted for measuring liquids,

<sup>1</sup> Read at the meeting of the Industrial Section of the American Chemical Society at Minneapolis, Dec. 28-31, 1910.

as the consumer would apparently receive less in pounds and it would appear as though the manufacturer would be correspondingly benefited. The benefit, on the other hand, which the consumer would derive from a standard temperature for measuring liquids, would ultimately be greater than any possible temporary loss he might sustain.

If we desire to calculate from specific gravity to weight per gallon, the question of ratio arises. Shall this specific gravity relate to water at the same temperature  $\frac{t_m}{t_m}$  or shall it relate to water at  $4^\circ \text{C}$ .  $\frac{t_m}{4^\circ \text{C}}$ ?

Practically it does not make any difference, however, as we will show by the following reasoning:

1. The gallon is 231 cubic inches.
2. A brass gallon measure standardized at  $4^\circ \text{C}$ . contains 231.20 cubic inches at  $20^\circ \text{C}$ .
3. *a*, 231.20 cubic inches of water at  $4^\circ \text{C}$ . weighs in air 8.3432 lbs. = *W*.
- b*, 231.20 cubic inches of water at  $20^\circ \text{C}$ . weighs in air 8.3285 lbs. = *W*<sub>1</sub>.
4. Specific gravity  $\frac{20^\circ}{20^\circ}$  : Specific gravity  $\frac{20^\circ}{4^\circ}$  =  
 $1 : 0.9982343 = W : W_1$

Therefore:

$$\text{Specific gravity } \frac{20^\circ}{4^\circ} \times W = \text{specific gravity } \frac{20^\circ}{20^\circ} \times W_1$$

For practical purposes, therefore, we could define the gallon in about the following terms:

"The gallon of liquid shall be considered as equal to the weight obtained by multiplying *a*, the specific gravity of the liquid at  $\frac{20^\circ \text{C}}{4^\circ \text{C}}$ , by 8.3432 lbs. or *b*,

the specific gravity of the liquid at  $\frac{20^\circ \text{C}}{20^\circ \text{C}}$ , by 8.3285."

Whether this definition would be of practical value in specifications or not is yet to be determined, but it is certain that it at least is deserving of consideration.

#### A METHOD FOR THE DETERMINATION OF TIN IN CANNED FOODS.

By HERMAN SCHREIBER AND W. C. TABER.

Received January 27, 1911.

The determination of tin in canned foods, which has recently become of increased importance, may be made by several methods, each having its advantages and defects. Ashing the material at a very low heat in a muffle will give correct results with some foods under some circumstances, but not always, and when the composition of the material is not known, it is not safe to apply this method. Moreover, tin and its salts are volatilized by heat in the presence of chlorid of ammonia and probably with other chlorids also, since it is well known that sodium chlorid volatilizes on heating strongly. When small amounts of a metal are to be recovered from a large amount of organic matter, the danger of mechanical loss in the fumes is very great. After ashing, the ash must be fused with caustic alkali if all of the tin is to be recovered, so that this process does not offer any advantage over the

method herein proposed in regard to the time necessary for making the determination.

Munson<sup>1</sup> has proposed carbonizing the organic matter with sulphuric acid and heat, and ashing in a muffle with the aid of nitric acid. Determinations made by this method did not give satisfactory duplicates, and the results represented only about 50 per cent. of the tin present, as determined by sulphuric acid digestion (Table I) in Halenke's wet ashing method, as modified by Schryver.<sup>2</sup> In this method the organic matter is destroyed by digestion with large amounts of sulphuric acid with the aid of potassium sulphate.

TABLE I.  
COMPARISON OF THE MUNSON METHOD AND THE SULPHURIC ACID OR WET COMBUSTION METHOD (SCHRYVER).

Sample.	Gram of tin found in 50 grams of sample.	
	By Munson method.	By sulphuric acid method. (Schryver).
Herring in tomato sauce.....	0.0236	0.0458
Do.....	0.0217	0.0652
Sardines.....	{ 0.0318 }	0.0652
	{ 0.0462 }	
Do.....	{ 0.0682 <sup>3</sup> }	.....
	{ 0.0801 <sup>3</sup> }	.....

Table II shows that the results obtained when the precipitates are weighed as stannic oxid ( $\text{SnO}_2$ ) are as correct as when the precipitate is dissolved and determined electrolytically<sup>4</sup> as tin. Schryver has shown that the wet combustion method gives accurate results and good duplicates. This is also shown by the determinations given in Table II. However, this method has some very serious practical defects. When tin is to be determined in meat, fish, or sirups, only 25 grams of material can be digested in one flask. This necessitates making two digestions in order that an amount can be used which will avoid the great multiplication of the analytical error which would occur if the result were calculated as milligrams per kilogram. Using such small quantities of the sample increases the error of weighing and sampling, and, moreover, limits the amount of work which can be done under ordinary conditions. The flasks have a tendency to break during the digestion, which, together with the foaming of the material, requires constant attention and considerable experience before satisfactory results can be obtained. No attempt was made to determine the amount of tin which could be recovered by the various methods by adding known amounts of soluble tin salts to food, since this would not give conditions analogous to those met in practice.

Our work indicated that the following was the most satisfactory method of making a sulphuric acid digestion: Weigh 25 grams of fish or meat or 50 grams of vegetables into a beaker, wash into an 800 or 1000 cc. Kjeldahl flask, add from 100 to 150 cc. of water, 25 grams of potassium sulphate, 50 cc. of sulphuric acid (sp. gr. 1.84), and a few glass beads. Place over a small flame, rotate a few times till it boils, then in-

<sup>1</sup> U. S. Dept. Agr., Bur. of Chem., *Bull.* 107, 62 (revised).

<sup>2</sup> Report 7 of the Local Government Board (medical department) of Great Britain on the presence of tin in certain canned goods.

<sup>3</sup> Wet combustion not made, given to show poor concordance of results.

<sup>4</sup> U. S. Dept. Agr., Bur. of Chem., *Bull.* 207, 69 (revised).