

or three times and let stand until the precipitate settles.

If caramel be present the precipitate will be dark brown in color, while the supernatant liquid will be dark colored also. After the color of the precipitate and liquid have been observed, add 5 cc. glacial acetic acid and shake; in the presence of a pure extract, or, of an extract colored with caramel, the precipitate

the eighth edition of the U. S. Pharmacopeia; the prune juice was prepared by extracting dried California prunes with 70 per cent. and with 30 per cent. alcohol, the Sherry color was made by evaporating down on the steam bath a pure California Sherry to $\frac{1}{10}$ of its original volume.

Below is given in tabular form the results obtained

Color of precipitate with basic lead acetate.	Color of supernatant liquid when precipitate settles.	Action on addition of glacial acetic acid to lead acetate ppt.	Appearance of the ethereal layer.	Quantity of resins.	Indicates.
None	Dark brown	All dissolves	No color	None	An entirely artificial extract colored with caramel.
Dark brown	Colorless	Gelatinous reddish brown residue	No color	None	An entirely artificial extract colored with prune juice.
Dark brown	Brown	Gelatinous reddish brown ppt.	No color	None	An entirely artificial extract colored with prune juice and caramel.
Dark brown	Brown	Gelatinous reddish brown ppt.	Small amt. color	Small quantity	An extract containing some natural vanilla color together with caramel and prune juice.
Voluminous, light colored	Colorless	Quan. of colorless ppt. undissolved	Fair amt. color	Small	An extract containing some natural vanilla color together with sherry residues.
Voluminous, light colored	Colorless	All dissolves	Much color	Large	A straight vanilla extract.
Voluminous, light colored	Colorless	All dissolves	Much color	None	Is probably an extract made by extracting the beans with glycerine.

will immediately dissolve. If prune juice has been used, there will be obtained, on the addition of basic lead acetate to the extract after removal of alcohol, resins and vanillin in the manner above described a very voluminous dark colored precipitate, while the supernatant liquid will be almost or entirely colorless. On addition of 5 cc. glacial acetic acid to this precipitate it will be found that there is present a lead salt insoluble in acetic acid. This insoluble substance is of a gelatinous reddish brown appearance much resembling the precipitate of ferric hydroxid.

The following precautions are necessary in applying the test:

1. The coloring matter of Sherry wine is said to be sometimes used to color and flavor vanilla extract. This vehicle gives with basic lead acetate a copious light colored precipitate closely resembling in appearance, the precipitate produced under like conditions from a pure vanilla extract but differing from the latter in containing a body insoluble in acetic acid, which body is left as a light flocculent precipitate after treatment of the lead acetate precipitate with this acid. If, however, a mixture of Sherry and caramel have been used, this precipitate will be colored brown and is thus very difficult to distinguish from a mixture of caramel and prune juice.

2. The observation as to whether or not acetic acid has dissolved all of the precipitate produced by basic lead acetate should be made immediately after adding the acid, as on standing for several hours or over night, traces of such precipitate begin to appear when extracts of known purity are under examination. We have found that the extraction with ether described above apparently does not remove from a pure extract any lead-precipitating substance except the vanillin so that with such a product after extraction with ether, there is obtained, on the addition of basic lead acetate, a copious very light colored precipitate.

The pure vanilla extract used in working out the above method was prepared from a medium grade of Bourbon beans according to the directions given in

in the examination of various pure and artificial extracts by the above method.

METHOD FOR NICKEL-ZINC SEPARATION IN GERMAN SILVER AND OTHER ALLOYS.

By LA VERNE W. SPRING.

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The nickel-zinc separation has always been a serious matter for the chemist who had to do it only occasionally and nearly as forbidding for him who was so unfortunate as to encounter it regularly. It was one of those things from which he was praying to be spared, but which he felt he could sometimes do little to avoid.

Some of the modifications of the sulphide separation can be made to work but it is no method for a busy laboratory or for inexperienced hands. The fixed alkali hydroxide method is open to objections. The titration of the nickel with potassium cyanide in the presence of sodium pyrophosphate works at least approximately under the proper restrictions, but does not allow of a direct determination of the zinc.

Since the announcement during the past three or four years of the German methods for direct precipitation of nickel as nickel glyoxime or as nickel dicyandiamidine from solutions of almost any other metals, our own troubles have ceased quite completely. The analysis of German silver means now little more than analysis of a brass and the new methods could be used advantageously by many more chemists than appear to be using them. I do not claim originality in the method as used in our laboratory for the past two years, for though I do not know that certain parts of the method have ever been published, they have, in a way, been forecast by the German chemists, Grossmann, Brunck, *et al.*, in articles in the German chemical magazines. The method is as follows:

Weigh out one-half gram of the drillings, or more, according to the percentage of the constituents,

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.

CRANE COMPANY LABORATORY, CHICAGO.

A RAPID VOLUMETRIC METHOD OF SUGAR ESTIMATION.

By W. F. SUTHERST.

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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.¹

By G. W. THOMPSON.

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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,

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