The first distillates may be added to the small amount remaining in the distilling flask (which should not be driven down to complete dryness), and a fresh portion of purified alcohol recovered.

The rationale of the proceeding appears to be that the permanganate oxides and destroys chiefly the fusel oil, furfurol and other compounds of that nature, the acids resulting from the reaction are neutralized by the calcium carbonate added before distillation, and by distilling slowly, the aldehyde at least is concentrated in the first portions of the distillate. Distillation of alcohol containing caustic potash or soda seemed to cause a constant formation of aldehyde. The alcohol thus purified is perfectly neutral, and gives most satisfactory results when used as a solvent for caustic alkalies or silver nitrate, the solutions remaining as colorless as distilled water, even after boiling and standing indefinitely, if properly protected from dust and other external influences.

THE EXAMINATION OF COMMERCIAL GLYCERINE.

BY J. H. WAINWRIGHT, PH. B., F. C. S.

The Tariff law of 1883 imposes upon "Glycerine, crude, brown or yellow, of the specific gravity of one and twenty-five hundredths or less at a temperature of sixty degrees Fahrenheit, not purified by distilling or refining," a duty of two cents per pound; and upon "Glycerine, refined" a duty of five cents per pound.

In view of possible attempts to enter, through the Custom House, a partially refined glycerine as the crude article, a sample of every importation of so-called crude glycerine is submitted to the U. S. Laboratory for examination as to whether it is properly invoiced, or is partially or wholly refined; in the latter case it should pay the higher rate of duty. It has been always considered by the chemists at this port that the extent of "refining" properly permissible in a glycerine in order to bring it within the definition of *crude*, is the allowing of impurities to subside by long

standing, or removing them by a process of straining or filtering, and all samples showing evidence of having been treated further than this are reported as *refined* and subjected to the higher rate of duty.

For purposes of classification under the tariff law referred to above *distillation* has always been regarded as the dividing line between crude and refined, and efforts have therefore been made to determine by means of physical and chemical tests whether this process of refining has been employed, since nearly all the glycerine imported as crude is bought by distillers for the manufacture of glycerine to be used in making dynamite (for which purpose distilled glycerine alone is used), and the white C. P. article used for medicinal and pharmaceutical purposes.

I will now call attention to some of the characteristics of crude and refined glycerine. Glycerine, crude, is defined by the tariff [T. I. new, A. 4]), to be "brown or yellow, of the specific gravity of one and twenty-five hundredths, or less, at a temperature of sixty degrees Fahrenheit, not purified by refining or distilling." Crude glycerine is the product obtained directly by stearine candle manufacturers from the saponification or disintegration of their fats and oils, and is a bye-product in the manufacture of soap, being obtained from the waste lyes.

Its specific gravity is usually 1.25, or less, at 60° F., but the gravity of waste lye glycerines sometimes runs much higher owing to the large amounts of chlorides, etc., present.

Its color is yellow, brown or black, but never white, sometimes light yellow, usually medium to dark yellow and often dark brown or black.

It possesses a strong characteristic odor of fatty acids usually disagreeable, sometimes even fœtid.

Reaction to litmus paper may be either acid or alkaline, never neutral.

Refined glycerine is usually the product of the distillation of the crude article, the distillation being repeated several times and the resulting glycerine concentrated and further purified by decolorization, etc., according to the uses for which it is intended. Its gravity is usually about 1.25.

Its color ranges all the way from dark brown or even black to white, according to the extent to which the process of distilling or refining has been carried.

If distilled it should have either no odor at all or a more or less burnt odor. This odor in a *distilled* glycerine is highly characteristic and suggests the odor obtained on burning gunpowder. If refined without distillation, as would be shown by other tests, the odor of fatty acids, if any, should be very faint. Distilled glycerine also possesses a characteristic taste faintly suggestive of garlic. This taste, however, as well as the burnt odor, is only exhibited in distilled glycerine of inferior qualitiy.

Reaction neutral or very faintly acid.

In determining the nature of a sample of glycerine a problem is often met with, inasmuch as it is often a matter of great doubt whether a sample has been distilled or not. The following tests, however, I regard as extremely reliable, and, with experience and practice, they afford results which admit of very nice distinction between crude and refined (or distilled) glycerine.

A 10 grm. sample is heated in a tared platinum capsule until it inflames, the source of heat is removed and it is allowed to burn spontaneously; the residue is then weighed and its per cent. calculated. Distilled or refined glycerine will yield from 0. to 0.5 per cent. of "carbonaceous residue," rarely more than 0.5 and never as much as 1 per cent. (unless in the case of an otherwise apparently high grade article it has been adulterated, which would be shown by other tests), whereas crude glycerine frequently yields as high as 10 per cent. I have found the per cent. of "carbonaceous residue" as determined by this test to admit of duplication within very narrow limits.

If desired, a determination of the ash may be made by igniting the "carbonaceous residue."

According to Sulman and Berry (Analyst, **11**, 12) the determination of the ash will definitely decide whether the glycerine is crude or distilled; this is somewhat of an error, as it does not necessarily follow that a small proportion of ash alone would indicate that the glycerine has been distilled, since a large proportion of the glycerine manufactured is made directly from fats by

"saponification" with steam, in which case the impurities would necessarily be organic and would be destroyed in the process of incineration, hence, other tests to show the presence of fatty impurities must be employed.

I have frequently found samples yielding as high as three per cent. of "carbonaceous residue" and less than 0.05 per cent. of ash. The proportion of mineral matter, however, affords a good indication of the character of the sample, since, in refined glycerine, the ash is never higher than 0.2 per cent. and rarely as high as 0.1, whereas, in crude glycerine made from soaps, lyes and by other processes of saponification, the ash frequently runs as high as 14 per cent.

If deemed desirable, an examination of the ash may be made, this also will often afford an indication of the source of manufacture from which the glycerine was obtained (Allen's Com. Org. Anal., 2d ed., 2, 297). Of all the other tests which are recommended for the examination of glycerine the two most important are those made with solutions of silver nitrate and basic lead acetate in distilled water, the former being a two per cent. solution and the latter being made by adding 10 grams. of C.P. lead acetate and 8 grammes of litharge to 500 c. c. distilled water, boiling for some time and filtering. The tests are made as follows :

The silver nitrate test.

Dissolve 5 c. c. of the sample to 20 c. c. distilled water in a large test tube and add 5 c. c. silver solution, shake, allow to stand at rest for one hour. In refined glycerine a darkening of the solution may occur, with even a slight reduction of silver, after standing some time : it will be quite heavy if allowed to stand long enough, even in highly refined samples, but if it is not quite heavy at the end of one hour the result may safely be considered an indication of refined glycerine. In crude glycerine a considerable precipitation takes place, usually at once, and is nearly always *flocculent*. The precipitate may be of any color from black to white, according to the nature of the impurities present.

The lead test.

This test is made by adding to the lead solution in a large test tube, its own volume of a solution containing equal volumes of

the sample and distilled water, and shaking and allowing to stand at rest for one hour, as in the silver test. Refined glycerine will remain unchanged or will show a slight precipitation or cloudiness, but never a *flocculent* precipitate, even on standing for a long time, whilst in crude glycerine there will always be more or less of a flocculent precipitate.

In applying these tests it is *never* safe to rely on either of them *alone*, as it will frequently happen that a sample will be met with which will stand one test and not the other; however, if it will not stand *both* of these tests it is perfectly safe to call it crude, and, with some experience, a fair idea of the *value* of the sample may be obtained. By boiling, the tests may be greatly hastened, although this is not recommended except in extremely doubtful cases, as the test thereby becomes much more delicate.

If deemed desirable, other tests may be made, and among them the following will be found useful:

The addition of an equal volume of distilled water. Refined glycerine will remain clear. In *crude* glycerine if much oil or fat be present they may be separated from the glycerine in this manner:

Ammonia.—*Refined*, no change; *crude*, precipitate indicates presence of *iron* and *alumina*.

Ammonium Oxalate.—*Refined*, no change; *crude*, precipitate indicates *lime* salts.

Barium Chloride.—Refined, no change; *crude*, precipitate indicates *sulphates*.

Nitrogen Peroxide (Gas).—*Refined*, no change; *crude*, curdling indicates *fatty impurities*.

Fehling's Solution.—*Refined*, no change; *crude*, shows the presence of *glucose*, etc.

For other tests recommended for the examination I would refer to the very able paper of Messrs. Sulman and Berry (Analyst, **11**, 12 and 34) and to Allen's Com. Org. Anal. (2d ed., vol. **2**, 292 et seq.).

With a fair amount of experience a discrimination between crude or raw and refined or distilled glycerine will readily be accomplished by means of the foregoing tests and, moreover, a fair

idea as to the quality of the sample may be obtained, and it may be ascertained beyond a doubt whether it is fit for the manufacture of nitro-glycerine for which so much of the refined glycerine of commerce is used.

UNITED STATES LABORATORY,

New York, December 6th, 1889.

THE NOMENCLATURE AND NOTATION OF ALKALOIDAL SALTS.

BY S. W. WILLIAMS.

Now that the U. S. Pharmacopœia is about to be revised, it seems eminently proper that the American Chemical Society should recommend a system of nomenclature and notation for alkaloidal salts more consistent than that adopted by the last Committee of Revision.

Four names and four formulas fairly representative of the terminology and symbolic representation of salts formed by acids, with alkalies and alkaloids, should be sufficient to demonstrate that a wider divergence from consistency could hardly have been attained :

Pharmacopœial Name.	*Pharmacopœial* Formula.
Ammonium bromide.	NH_4 Br.
Quinine hydrobromate.	C_{20} H_{24} N_2 O_2 H Br.
Potassium sulphate.	K_2 S O_4
Quinine sulphate.	$(C_{20}$ H_{24} N_2 $O_2)_2$ H_2 SO_4.

Note as inconsistencies :

1. Writing a salt as a hydrobromide and naming it a hydrobromate.

2. Calling one salt of hydrobromic acid a hydrobromate and another salt of the same acid a bromide.

3. Recognizing in the name of one salt the unreplaced hydrogen of the combining acid and ignoring the same unreplaced hydrogen in another.