amorphous substances, which are nearly related to crystalline aloin, give this fluorescence with borax solution also.

The following experiments, which were made quite a number of years ago, may be of interest.

A large quantity of aloes was worked up in the process of making aloin, and the material was tested by this method both before and after the extraction of the crystals.

The test gave a higher figure for aloin content *after* the aloin had been removed than before, and neither figure had any relation to the actual amount found, which was normal.

This again shows that these resins are nearly related to crystalline aloin, a claim which is also corroborated by the fact, that when treated with Caro's acid both the crystalline aloin and these resinous substances yield tri- and tetra-oxi-methylanthraquinones.

The precipitation of aloin as tri-brom-aloin by means of bromine water gives unreliable results also, because the amorphous oximethylanthraquinones are precipitated together with the aloin.

The only way we know of to determine the amount of aloin in aloes is to work up about 10 pounds of aloes; but such a process is not very feasible, because while at times under certain conditions the aloin may crystallize out within a few days, under other conditions, especially when a larger amount of amorphous substances than usual is present, the complete crystallization may take weeks.

It is to be regretted that up to the present time no reliable assay process is at our disposal for such a valuable and widely used drug as aloes.

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A SOURCE OF ERROR IN EMPLOYING FUCHSIN-SULPHUROUS ACID SOLUTION AS A TEST FOR FORMALDEHYDE IN ETHYL ALCOHOL.*

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The U. S. P. test for methyl in ethyl alcohol depends upon the oxidation of the methanol by means of potassium permanganate to formaldehyde and the detection of the latter by fuchsin-sulphurous acid in the presence of sulphuric acid.

The fuchsin-sulphurous acid solution is known as Schiff's Reagent and is employed for the detection of aldehydes in general, but taking advantage of the fact that sufficient sulphuric acid prevents development of color by acetaldehyde which is the product formed when ethyl alcohol is oxidized, Denigès suggested that the fuchsin-sulphurous acid solution be added in the presence of sulphuric acid, and in this form the test is official in the U. S. P.

In the final steps of the test there are added to the oxidized material one cc of strong sulphuric acid and five cc of fuchsin-sulphurous acid solution and if a distinct blue or violet color is produced formaldehyde is indicated; as this results from oxidizing methyl alcohol the latter is reported.

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* Read before New York State Pharmaceutical Association, Stamford meeting, 1923.

In an effort to employ this test for the detection of formaldehyde in ethyl alcohol I conducted the following experiments:

(*A*) To 5 cc of ethyl alcohol, which had been redistilled from sulphuric acid and then over potassium hydroxide and containing 94.78% of absolute alcohol, there were added 1 cc concentrated sulphuric acid and 5 cc fuchsin-sulphurous acid solution. The result was an immediate purple color, indicating the presence of formaldehyde.

(*B*) Repeating the test as above but increasing the quantity of sulphuric acid to two cc the result was a garnet-colored solution.

(*C*) In this test the quantity of sulphuric acid was increased to three cc and the result was the same as in *B*.

(*D*) The quantity of sulphuric acid in this test was increased to four cc and the result was the same as with *B*.

(*E*) Increasing the quantity of the sulphuric acid in this test to five cc produced the same result as in *B*.

(*F*) Five cc of the above 94.78% ethyl alcohol without previous distillation from acid or alkali, when treated as under *A*, yielded results identical with those under *A*.

Another series of tests was made as follows:

(*G*) The above 94.78% alcohol was reduced with water to 10 per cent. by volume and 5 cc of the diluted sample was tested by adding one cc of concentrated sulphuric acid and five cc fuchsin-sulphurous acid solution. The result was a colorless solution proving the absence of formaldehyde.

(*H*) Five cc of a 5% dilution of the above 94.78% alcohol when tested as under *G* yielded a colorless solution, thus indicating the absence of formaldehyde.

(*I*) To 100 cc of the above 94.78% alcohol there was added 0.02 cc of 37% solution of formaldehyde. This solution was then diluted with water to contain 5 per cent. by volume of alcohol. Five cc of this solution tested as under *G* immediately produced a purple color showing the presence of formaldehyde.

The fuchsin-sulphurous acid solution of the U. S. P. not being very satisfactory I have for some time employed the following modification of Elvove:[1]

Two-tenth grams of fuchsin are dissolved in 120 cc of hot water; after cooling to room temperature there are added 2 grams anhydrous sodium sulphite, dissolved in 20 cc of water, followed by 2 cc concentrated HCl and then the whole diluted to 200 cc. Allow to stand one hour before using. Stopper in amber-colored bottles. The solution will keep several weeks.

In connection with this work I made comparative examinations of the commonly employed tests for the detection of formaldehyde and, as a result, conclude that in addition to the fuchsin-sulphurous acid among the most sensitive and easily applied tests are the following:

Dodsworth and Lyons' modification of Hehner's test suggested by A. B. Lyons in his paper "Detection of Wood Spirit in Alcoholic Beverages,"[2] which is employed is as follows:

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[1] *J. Ind. Eng. Chem.*, 9, 295, 1917.

[2] JOUR. A. PH. A., 10, 13, 1922.

To 1 cc of alcohol add 10 drops of albumen solution from a pipette which delivers 30 drops to the cc. Shake thoroughly and then underlay with 1 cc of ferrated sulphuric acid. In the presence of formaldehyde a beautiful purple ring will form.

In determining the sensitiveness of this test I added 0.01 cc of the 37% formaldehyde solution to 100 cc of the above 94.78% alcohol. One cc of this solution was then added to 99 cc of the 94.78% alcohol, and to one cc of this solution (which contained 0.000001 cc of the 37% formaldehyde solution per cc) there were added 10 drops of albumin solution and, after shaking thoroughly, 1 cc of ferrated sulphuric acid so as to form a distinct layer. Upon slightly twirling the tube to produce a very gentle mixing a beautiful purple color made its appearance.

Morphine-sulphuric acid solution produces with formaldehyde a beautiful purple color. The test was made by taking 1 cc of the above very dilute solution of formaldehyde (containing 0.000001 cc of 37% formaldehyde solution per cc) adding five drops of morphine solution and underlaying with sulphuric acid. After gently rotating the tube to produce a slight admixture a beautiful purple color appeared.

Alcohol free from formaldehyde tested in various dilutions did not show the presence of formaldehyde when these tests were applied.

It should be remembered that both of these tests are more sensitive in the presence of traces of formaldehyde than when larger amounts are present.

The following reagents were employed in making the tests:

Ferrated Sulphuric Acid.—Dissolve 0.030 Gm. of ferric-ammonium alum in 1 cc distilled water and add to 100 cc of sulphuric acid, U. S. P.

Solution of Albumen.—0.100 Gm. dried egg albumen, dissolved in 10 cc of distilled water.

Solution of Morphine.—Dissolve 0.50 Gm. of morphine in enough diluted sulphuric acid to aid solution and add sufficient distilled water to make 100 cc.

SUMMARY.

1. Formaldehyde-free undiluted ethyl alcohol when tested for formaldehyde by means of fuchsin-sulphurous acid solution in the presence of sulphuric acid will give a strong positive reaction.

2. Formaldehyde-free ethyl alcohol diluted to 5% or 10% with water will not give a positive reaction for formaldehyde when tested with fuchsin-sulphurous acid in the presence of sulphuric acid.

The test will detect 0.00005 cc of the 37% formaldehyde in the amount of sample (5 cc) taken for analysis.

3. The ferrated-sulphuric-acid-albumen and the morphine-sulphuric acid reagents are both very sensitive, detecting 0.000001 cc of 37% formaldehyde solution in the 1 cc of sample taken for analysis.

4. All three tests are easily made and do not indicate the presence of formaldehyde in ethyl alcohol which is free from it.

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