coke oven or gas retort where a measure of coking power is wanted.

3—Benzene-pressure extraction does not remove all of the bitumen from a coal; what is left behind may well have a strong influence on coking power. 4—A large portion of the bitumens of the Utah coal investigated distil at low temperatures, probably without extensive decomposition. It is believed that this is the main reason why the coal does not produce a well-cemented coke.

Rapid Determination of Alcohol in Distilled Spirits and of Color in Whisky^{1,2}

By John F. Williams

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A simple, rapid method is given for the determination

A color reagent for detection of added coloring matter

of alcohol, which is suitable for a very small amount of

sample, uses no apparatus except a graduated cylinder,

in whisky is described. The determination of the

alcoholic content and the detection of caramel coloring

and requires no special skill to manipulate.

are combined in a single operation.

THERE is need for a simple method for the determination of alcohol in distilled spirits which can be applied away from the facilities of a chemical laboratory. The drug and medical professions need a test for the "proof" of medical spirits; the industries need a quick test in certain processes where specially denatured alcohol is used; certain public inspectors and chemists need a method for making preliminary investigations; finally, a quick test is desired at court which can be used on the spot to check previously ob-

tained analytical results. A test has been devised which depends upon the fact that a special reagent extracts the alcohol from distilled spirits, leaving the water it contained as a lower layer. The height of the

lower layer is the reading, and this may be converted into alcoholic percentages. At the same time, if the sample under examination

is whisky, the lower water layer retains certain artificial coloring matters, so that the test will reveal their presence. This test is not a precision method, and it is not intended to replace any of the standard laboratory methods. Its use is primarily for the field where a hydrometer set or pycnometer and balance are not available; or where the sample is too small for convenient use with these instruments.

Special Reagent

The special reagent is made up as follows:

	MI.
Amyl alcohol (fusel oil)	70
Toluene, C. P.	28
Tartaric acid (50% aqueous solution)	2

The materials are mixed together thoroughly. This reagent has a specific gravity of 0.8333 (20°/4° C.).

The fusel oil was redistilled and the fraction boiling between 120° and 130° C. taken (specific gravity 0.8106). The material need not exactly conform to these figures, however. Two other grades of fusel oil were tried and both were found to be suitable. Their physical properties were: (1) boiling range 110° to 130° C., specific gravity 0.8119 (20°/4° C.); and (2) boiling range 127° to 129° C., specific gravity 0.8103 (20°/4° C.).

The boiling range of the toluene was 110° to 111° C.

¹ Received March 22, 1926.

² Published by permission of the Head, Industrial Alcohol and Chemical Division, Bureau of Internal Revenue.

Procedure for Determination of Alcohol

Into a carefully graduated, clean, 20-ml. glass-stoppered cylinder or tube, which has been previously rinsed with the sample, pipet exactly 10 ml. of the sample and add 10 ml. of the reagent. The temperature before mixing should be approximately 20° C. (68° F.). Invert the tube gently five or six times, then rotate slowly or allow to stand until the layers separate, and measure the height of the lower layer in millili-

ters, reading the "middle" meniscus. Invert the tube again, allow the layers to separate, and repeat the reading. The last reading should be accepted.

From the reading in milliliters the percentage of alcohol may be obtained from Table I. In order to prepare this table of conversion a set of twenty-four standard alcoholic solutions

ranging from 5 to 66 per cent was made up by placing the calculated quantity of 95.5 per cent alcohol in 100-ml. volumetric flasks and diluting to the mark at 20° C. The strength of these solutions was verified by means of a pycnometer. Ten milliliters of each solution with 10 ml. of the reagent were extracted and the readings recorded. The room, reagent, and solutions were kept at 20° C.

Above 64 per cent no result is obtained, but by a simple modification the method may still be applied. Place exactly 5 ml. in the tube and dilute with enough water to make 10 ml. at 20° C. Add the reagent and read as before, but multiply the percentage of alcohol thus obtained by 2.

For field work a tall 10-ml. cylinder may be employed, using one-half the amounts given above, and multiplying the milliliter reading by 2 before consulting the table to obtain the percentage of alcohol. A 10-ml. cylinder 15 cm. (6 inches) long, having graduations extending approximately 13 cm. (5 inches), has been found most practicable.

Should an emulsion form it will discharge itself on standing. To hasten this discharge the cylinder may be rotated gently for a few seconds; or, after partial separation has taken place, the emulsion may be discharged at once by gently warming the cylinder a few degrees but cooling to 20 degrees before accepting the reading. By mixing the liquids gently an emulsion is not likely to form.

Droplets of water adhering to sides of the glass tube because of the presence of dirt or grease may be forced down by suddenly rapping the cylinder with a pencil.

Samples of pure as well as denatured alcohol, whisky,

brandy, gin, etc., were tested by this method and found to give results in agreement with those obtained by using a U. S. standard alcohol hydrometer.



Temperature Changes

The effect of temperature changes was studied by mixing with the reagent a solution of 50 per cent alcohol by volume at the temperatures indicated in Table II.

	Table II	
emperature ° C.	Reading	Alcohol (from Table I)
10	5.40	48.6
20	5.10	50.0
30	4.80	51.4

It is not difficult to keep the temperature within 1° or 2° from 20° C., and within these limits the error is not appreciable. The three values in the table do show, however, that the matter of temperature cannot be neglected.

With this method the effect of temperature changes is less than with the alcohol hydrometer method, as shown in Table III.



^a U. S. Internal Revenue Gaugers' Manual, 1913.

Only a slight change in the temperature was noted when mixing the sample and reagent, usually not more than 1° or 2° C. rise.

Presence of Solids

The effect of the presence of glycerol or cane sugar was determined at 20° C. with the results shown in Table IV.

Table	IV	
(50 per cent a	lcohol used)	
	Reading Ml.	Indicated alcohol Per cent
Glycerol, 5 per cent	5.00	50.3
Cane sugar, 5 per cent	5.07	- 50.1
Without additions	5.10	50.0

No appreciable interference was noted by the presence of small amounts of glycerol or cane sugar on 50 per cent alcoholic solutions.

Determination of Color

For determination of color in whisky, the tube was placed near light against a sheet of white paper, and the approximate percentage of color in the upper and lower layers noted. So-called natural color in whisky appeared in the upper layer, while any caramel or other water-soluble colors dissolved in the lower or aqueous layers (Figure 2).

Extension of Method

In the laboratory the method is applicable to the distillates from wines and cordials, and to the concentrated distillates from vinegar, near-beet, ale, cider, etc. In the case of liquors having a low alcoholic content (under 10 per cent)



Figure 2-Results of Test when Applied to Typical Samples of Colored Distilled Spirits

distil 100 ml. of the sample very slowly, collecting 20 ml. Apply the test to 10 ml. of the distillate, and divide the percentage of alcohol thus obtained by 5.

No statement can be made at this time as to the application of the method directly to wines, extracts, cordials, medicinal preparations, etc., before distillation. It is expected, however, that by slight modification the method will be applicable to these liquids. The perfume industry, as well as other fields where alcohol is used as a solvent, is yet to be investigated with reference to this test.

Instead of using the regular graduated cylinder described above, a special tube can be made and so graduated as to show the percentage of alcohol direct. Such a tube may be made 120 mm. long and have an inside diameter approximately 11 mm.

Acknowledgment

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Destruction of Vitamin A in Milk by Ultra-Violet Light^{1,2}

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S TEENBOCK, Hart, Hoppert, and Black³ have called attention to the fact that the antirachitic vitamin in cow's milk is increased eight or more times by the exposure to the radiations of the quartz-mercury vapor lamp. They suggest the possibility of the practical utilization of this principle in the preparation of infant foods, and suggest to dairymen the beneficial results which may be expected from such treatment. In a report of the Medical Research Council⁴ it is stated, "the plan of exposing milk, to ultraviolet rays, in order to increase its nourishing qualities is proving highly successful."

In this connection it should be pointed out that, while the irradiation of milk enhances its nutritive value as far as its vitamin D content is concerned, it materially decreases its nutritive value with respect to its vitamin A content. This is of particular importance in infant feeding, since milk is the main source of vitamin A in such cases. The experiment described herein substantiates these facts.

Since chicks have been shown⁵ to be very susceptible to the lack of vitamin A, they were chosen as the experimental animals for testing the influence of ultra-violet light on the destruction of vitamin A. Two lots of fourteen each, day-old chicks (white Leghorns) were taken for the experiment. From previous experiments we have learned that chicks treated with ultra-violet light and fed a ration of white corn and tankage, when supplied with vitamin A, make satisfactory growth. These two lots of chicks were accordingly irradiated with a quartz-mercury vapor lamp 15 minutes per day, fed a ration of 90 per cent white corn chop and 10 per cent tankage, and given 5 cc. of fresh whole milk as the only source of vitamin A. The only difference in the treatment of the two lots was that the milk given to Lot I was irradiated and that given to Lot II was nonirradiated.

The milk, in a sheet-iron pan, was irradiated by being subjected to the rays of the quartz mercury vapor lamp, placed 45 to 50 cm. (18 to 20 inches) above its surface. The pan was rocked back and forth about once every 3 minutes in order to expose the milk to a greater extent, its depth being about 2 mm. or less. The two lots of chicks were kept in small pens, about 1×2 meters (3 \times 6 feet), inside the animal house. The experiment was carried on for 7 weeks, with the results as shown by the chart.

At the end of the experiment the chicks of Lot II showed no signs of vitamin A deficiency. The one remaining chick of

¹ Presented by Titus, Hughes, and Fitch before the Division of Biological Chemistry at the 71st Meeting of the American Chemical Society, Tulsa, Okla., April 5 to 9, 1926.

² Contribution No. 121 from the Department of Chemistry, Kansas State Agricultural College.

³ J. Biol. Chem., 66, 441 (1925).

4 J. Am. Med. Assoc., 86, 565 (1926).

⁵ Hart, Steenbock, Lepkovsky, and Halpin, J. Biol. Chem., **60**, 341 (1924).

Lot I was apparently an outstanding individual and was able to survive the unfavorable conditions. With the one exception, all chicks of Lot I died, showing typical vitamin A deficiency. The chicks showed general unthrift and in two cases well-advanced stages of opthalmia developed before death. This agrees with the finding of Emmett and Peacock,⁶ who have reported the occurrence of ophthalmia in their chicks suffering from vitamin A deficiency. The most marked lesion observed on autopsy was an enlargement of the kidneys, which were paler than normal owing to an excessive amount of urates in the tubules.⁷ Another common



Destruction of Vitamin A Milk Subjected to Irradiations of the Ouartz-Mercury Vapor Lamp as Shown by High Mortality of Chicks in Lot I

lesion observed was a pericarditis, in which was observed a marked deposit of a urate-like material in the pericardial sac and over the pericardium. It was not uncommon to see this same urate-like deposit covering the liver and intestines. Beach⁸ and Hart, Steenbock, and co-workers⁵ have called attention to the white streaks on the surface of the liver.

⁷ Bushnell and Hinshaw, Dept. of Bacteriology, Kansas Expt. Sta., Circ. 106.

8 Am. Vet. Med. Assoc., 11, 303 (1921).

⁶ J. Biol. Chem., 56, 679 (1923).