INDUSTRY CIRCULAR

OFFICE OF THE COMMISSIONER OF INTERNAL REVENUE
ALCOHOL AND TOBACCO TAX DIVISION
WASHINGTON 25, D. C.

Industry Circular No. 62-2

January 24, 1962

APPROVED METHODS FOR TESTING CERTAIN CHEMICALS
(INCLUDING CHEMICAL BY-PRODUCTS)
FOR SPIRITS CONTENT

Proprietors of distilled spirits
plants and others concerned:

Purpose. This circular is issued to advise you that a revenue procedure will soon be published in the Internal Revenue Bulletin announcing approved methods for testing for spirits content certain chemicals where such chemicals are produced and collected in the production facility of a distilled spirits plant.

Background. Section 201.278 of the Distilled Spirits Plant regulations provides that all chemicals produced, including chemical by-products of the spirits production system, shall be substantially free of spirits content before being transferred to storage tanks or removed from the production facilities; the spirits content of such chemicals shall not, except as authorized by the Director, Alcohol and Tobacco Tax Division, exceed 10 percent by volume; and the testing of such chemicals for spirits content will be conducted by the proprietor in accordance with methods approved by the Director.

Required apparatus and reagents. Required apparatus and reagents are as follows:

Fusel oil tube - A&TTD type (The bulb holds three times the volume of the graduated neck portion. The graduations are from the top line marked 0 to the bottom line marked 100.)
Flask or graduate - 300 to 500 ml. capacity
3 graduates - 25 ml. capacity
Saturated salt solution (sodium chloride)
Sulfuric acid, concentrated, specific gravity 1.84
2% sulfuric acid solution (2 ml. concentrated sulfuric acid diluted with distilled water to 100 ml.)
Sodium sulfate, anhydrous (Na_2SO_4)
Ceric ammonium nitrate reagent (Triturate 10 g. of ceric ammonium nitrate ($(NH_4)_2Ce(NO_3)_6$)) with 3.5 ml. concentrated nitric acid (HNO_3) and dilute with distilled water to 125 ml. Filter if cloudy.)
Standard acetone - alcohol solution (To 90 ml. of acetone (C.P.) add 10 ml. of ethyl alcohol (C.P.).)

Standard alcohol solution (2% ethyl alcohol (C.P.) 98% saturated salt solution)
Chloroform ($CHCl_3$)
Nitric acid, concentrated (HNO_3)

Approved methods for testing. The approved methods for testing acetone, butyl alcohol, ethyl ether, ethyl oil, and fusel oil are listed in subparagraphs (a), (b), (c), (d), and (e) of this paragraph, respectively. Samples meeting test requirements contain no more than 10 percent ethyl alcohol by volume and, thus, the tests serve as a rapid screening procedure. Failure to meet test requirements is not conclusive proof of the presence of more than 10 percent ethyl alcohol by volume since other chemicals may be present which affect solubilities or oxidation rate as in the case of acetone. Where samples fail the approved method for testing and other laboratory analyses indicate the sample to contain less than 10 percent ethyl alcohol by volume, additional samples should be taken and forwarded to the regional laboratory for analyses by other methods.

(a) Acetone

Put 5 ml. of ceric ammonium nitrate reagent in a 25 ml. graduate, add 5 ml. of acetone sample, close the graduate, and shake several times to mix. Immediately compare the color with that produced when 5 ml. of standard acetone-alcohol solution is similarly treated. If the color of the acetone sample being tested is not greater than the color of the standard (made with 5 ml. of standard acetone-alcohol solution), the acetone sample contains no more than 10 percent ethyl alcohol by volume. If desired, both the acetone sample and the standard may be diluted with equal quantities of distilled water. The difference in color is more apparent at 5 percent alcohol since at 10 percent alcohol the color is quite dark.

(b) Butyl alcohol.

Put 20 ml. of saturated salt solution in the fusel oil tube and add 2% sulfuric acid solution until the 100 mark on the fusel oil tube is reached. Add butyl alcohol sample until the 0 mark on the fusel oil tube is reached, close the fusel oil tube, shake for about 5 minutes, and place in an upright position to allow the two layers to separate (30 minutes may be required to get a good separation). A reading of 90 or less at a temperature of 60° to 80° F. indicates more than 10 percent ethyl alcohol by volume.*

(c) Ethyl ether.

Put saturated salt solution in the fusel oil tube until the 100 mark on the fusel oil tube is reached. Add ethyl ether sample until

the 0 mark on the fusel oil tube is reached, close the fusel oil tube, shake, and place in an upright position to allow the two layers to separate (about 2 or 3 minutes). A reading of 90 or less at a temperature of 60° to 80° F. indicates more than 10 percent ethyl alcohol by volume.*

(d) Ethyl oil.

Either of the following methods may be used:

(i) Put saturated salt solution in the fusel oil tube until the 100 mark on the fusel oil tube is reached. Add ethyl oil sample until the 0 mark on the fusel oil tube is reached, close the fusel oil tube, shake, and place in an upright position to allow the two layers to separate. A reading below 90 at a temperature of 60° to 80° F. indicates more than 10 percent ethyl alcohol by volume.*

(ii) Put 19 ml. of chloroform in a 25 ml. graduate, add 1 ml. of ethyl oil sample, close the graduate, shake several times to mix, and then add 5 ml. of saturated salt solution. Into another 25 ml. graduate put 20 ml. of chloroform, add 5 ml. of standard alcohol solution, close the graduate, shake for 3 minutes, and place in an upright position to allow the two layers to separate. Into each of these two graduates add 5 ml. of ceric ammonium nitrate reagent and stir several times to mix reagent with the upper layer in the graduate. If the color in the graduate containing the ethyl oil sample is not greater than the color in the graduate containing the standard alcohol solution, the ethyl alcohol is not more than 10 percent by volume.

(e) Fusel oil.

Put saturated salt solution in the fusel oil tube until the 100 mark on the fusel oil tube is reached. Add fusel oil sample until the 0 mark on the fusel oil tube is reached, close the fusel oil tube, shake, and place in an upright position to allow the two layers to separate. A reading below 90 at a temperature of 60° to 80° F. indicates more than 10 percent ethyl alcohol by volume.*

(*The ethyl alcohol in the sample is dissolved in the saturated salt solution and remains in the lower layer. The decrease in the upper layer gives a measure of the amount of ethyl alcohol contained in the sample.)

Temperature effects on sample. Oxidation rate of acetone is increased by elevated temperatures and decreased by lower temperatures. An increase in temperature increases the solubility of the solvents in the saturated salt solution.

Moisture effects on sample. Except for acetone, appreciable quantities of water have a decided effect on the test results. The water is completely dissolved in the saturated salt solution and, thus, would be read in the fusel oil tube as alcohol. When the solvents meet the requirements of the test, there is no need to make a correction for water; but, if the test indicates the presence of more than 10 percent ethyl alcohol by volume, then the following procedure may be used to dry the sample before making the test:

> Dehydration of sample - Put 200 ml. of sample in the 300 ml. to 500 ml. flask or graduate and add approximately 80 grams of anhydrous sodium sulfate. Shake, let stand for 1 hour (butyl alcohol requires 36 hours contact), and decant supernatant liquid which is used as the test sample in the preceding tests. The anhydrous sodium sulfate is a fairly effective drying agent and as such it removes most of the water.

Inquiries. Inquiries concerning this circular should refer to its number and be addressed to your Assistant Regional Commissioner (Alcohol and Tobacco Tax).

Dwight E. Avis
Director, Alcohol and Tobacco Tax Division