A Pharmaceutical Study of the Purity and Preservation of American Oils of Turpentine*,†

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It is recognized that the therapeutic value of oil of turpentine is limited (1). However, a monograph on the oil and another on the rectified oil are included in the twelfth revision of the U.S. Pharmacopœia (2), and several preparations of these oils are officially recognized in this country. Oil of turpentine, which is obtained from both the oleoresin (gum) and wood of several species of the pine, is the chief item in the Naval Stores industry of the South, and as a result of its abundance the oil is widely used as an internal and an external medicament by the laity. It is, moreover, one of the principal ingredients in several proprietary medicinal preparations.

This study of oil of turpentine had four objectives: (a) to determine which of the six common, commercial, American oils of turpentine, before and after rectification, conform to the requirements of the U.S. Pharmacopœia for Oil of Turpentine and Rectified Oil of Turpentine, respectively; (b) to determine the relative resistance of these oils to physical and chemical changes, and the best conditions for storing them to retard these changes; (c) to discover an efficient and practical preservative for these oils; and (d) to survey critically the U.S. Pharmacopœia monographs on Oil of Tur-pentine and Rectified Oil of Turpentine.

The specifications of the U.S. Pharmacopœia XII for Oil of Turpentine and Rectified Oil of Turpentine, which are virtually identical with those of the U. S. Pharmacopœia XI (3), are stated briefly in Table I.

PART I. PURITY OF SOME AMERICAN OILS OF TURPENTINE WITH RESPECT TO THE TESTS OF THE U. S. PHARMACOPŒIA XII

Most of the previous work done to determine the composition, purity and properties of the various oils of turpentine produced in different countries is from a commercial rather than a medicinal standpoint (4). A more recent report from Germany deals with five German and five foreign oils (5).

EXPERIMENTAL.

Fresh samples of six commercial American oils of turpentine, before and after rectification, were subjected to the tests outlined in Table I. The results of these tests are given after the description of each

oil and rectified oil, respectively. Oil No. 1. Steam Distilled Wood Turpentine Oil.—This oil is obtained chiefly by extracting shredded stumps of the longleaf yellow pine (Pinus palustris Miller) and/or the Cuban or slash pine (P. caribaea or heterophylla Morelet) with a petroleum solvent at a maximum temperature of 120° C. The crude terpene oils are separated from the solvent, chemically treated and then fractionated to give the commercial product which is known as 'steam and solvent turpentine.'

(It should be pointed out here that the U.S. Pharmacopœia definition, Specification A in Table I, denies official recognition to all oils of turpentine obtained from wood.)

With the above exception, which is to be understood throughout this paper, this oil met all re-quirements of the U. S. Pharmacopeia XII except in specific gravity (0.852) and refractive index (1.4672). While the odor of this oil is characteristic of turpentine, it is possible to distinguish its odor from that of an oil obtained from the oleoresin. The American Society for Testing Materials resolves this difficulty by stating in their specifications that the odor of a sample of turpentine oil shall be characteristic of the type specified, i. e., wood or oleoresin.

A quantity of this oil and of each of the other five oils was rectified by the U. S. Pharmacopœia XI method which consists of treating some of the oil with an equal volume of 5% sodium hydroxide solution, distilling off three-fourths of the oil, separating it from water, and finally drying it over anhydrous sodium sulfate.

After rectification the sample of oil No. 1 con-formed to all requirements of the U. S. Pharmacopœia XII for Rectified Oil of Turpentine except in refractive index (1.4652).

Oil No. 2. Destructively Distilled Wood Turpen-tine Oil.—This oil is one of the first fractions obtained by distilling the resinous wood of several species of *Pinus*. The oil is further purified, but is generally considered unfit for medicinal purposes.

This oil failed to conform to 4 of the 13 official tests. Its specific gravity was 0.852; only 88% distilled between 154-70° C.; and it failed with respect to both tests for other foreign substances.

Åfter rectification this oil failed to meet 3 of the After rectification this oil failed to meet 3 of the 13 U. S. Pharmacopœia XII requirements for Rectified Oil of Turpentine. Its specific gravity was 0.847; it contained 0.023 Gm. of residue per 5 cc. of oil; and it contained foreign substances B. Oil No. 3. Fire-Distilled Dehydrated Gum Tur-pentine Oil.—This oil is obtained from the oleoresin of the living trees, P. palustris and/or P. caribaea, by distilling it with a little water in a large copper the title heated directly with fire. Afterward the oil

kettle heated directly with fire. Afterward the oil is separated from the water and dried with sodium chloride (6).

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This oil met all U. S. Pharmacopœia XII requirements for Oil of Turpentine, and after rectification it met all requirements for Rectified Oil of Turpentine.

Oil No. 4. Fire-Distilled Undehydrated Gum Turpentine Oil.—This oil is identical with oil No. 3 except that it is not dehydrated with sodium chloride. This is the type oil produced by operators in the small individual distilling outfits.

This oil met all U. S. Pharmacopœia XII requirements for Oil of Turpentine, and after rectification it met all requirements for Rectified Oil of Turpentine.

Oil No. 5. Steam-Distilled Dehydrated Gum Turpentine Oil.—This oil is produced by distilling the oleoresin of P. palustris and/or caribaea in an experiseveral bicyclic hydrocarbons, oil of turpentine easily ozonizes, resinifies, polymerizes and undergoes other chemical reactions on standing which changes its composition and its physical and chemical properties and renders it unfit for oral use (8).

Using the following U. S. Pharmacopœia XII specifications and tests, each of which is given a number corresponding to its position in Table I—1, color; 2, odor and taste; 3, solubility; 4, specific gravity; 7, reaction; 8, distillation range; 9, fixed oils; 11, mineral or rosin oils; 12, foreign

TABLE	Ι
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	Specification	Oil of Turpentine	Rectified Oil
А.	Definition	The volatile oil distilled from the oleoresin ob- tained from <i>P. palustris</i> Miller and other species of <i>Pinus</i> (Fam. <i>Pinaceae</i>) which yield exclusively terpene oils	The same
1.	Color	Colorless liquid	The same
2.	Odor, Taste	Characteristic, becoming stronger on exposure	The same
3.	Solubility	Oil is soluble in 5 vol. alcohol	The same
4.	Specific gravity	0.854 to 0.868 at 25° C.	0.853 to 0.862 at 25° C.
5.	Optical rotation	Active, but variable	The same
6.	Refractive index	1.4680 to 1.4780 at 20° C.	The same
7.	Reaction	Alcoholic solution (1–5) neutral or only slightly acid to litmus	The same
8.	Distillation range	90% of 100 cc. distils between 154 and 170° C.	The same
9.	Fixed oils	3 drops on unsized paper leaves no stain	The same
10.	Mineral oil	Does not exceed 1% as determined by poly- merization with sulfuric acid	The same
11.	Mineral or rosin oils	5 cc. contains 0.1 Gm. or less of residue after evaporation on steam bath	0.015 Gm. or less
12.	Foreign substances A	5 cc. of the oil shaken with 5 cc. KOH (5%) does not become darker than light straw yellow within 24 hrs.	The same
13.	Foreign substances B	5 cc. oil shaken with 5 cc. HCl does not become darker than light straw yellow in either layer within 5 mins.	The same

mental still heated with steam (7). The oil is then dehydrated with sodium chloride.

This oil met all U. S. Pharmacopœia XII requirements for Oil of Turpentine, and after rectification it met all requirements for Rectified Oil of Turpentine.

Oil No. 6. Vacuum-Distilled Gum Turpentine Oil.—This oil is produced in large central distilling plants by distilling precleaned oleoresin of *P*. *palustris* and/or *caribaea* under reduced pressure with the use of steam. The number of these central plants is increasing.

This oil met all U. S. Pharmacopœia XII requirements for Oil of Turpentine, and after rectification it met all requirements for Rectified Oil of Turpentine.

It is noteworthy that the samples of oils Nos. 3, 4 and 5 were pure enough even before they were rectified to meet all U. S. Pharmacopœia XII requirements for Rectified Oil of Turpentine.

PART II. THE RELATIVE RESISTANCE OF SOME AMERICAN OILS OF TURPENTINE TO PHYSICAL AND CHEMICAL CHANGES UNDER SEVERAL DIFFERENT STORAGE CONDITIONS

Due to the presence of unsaturated linkages and the strained ring structures in its substances A; and 13, foreign substances B—an investigation was made to determine the resistance of the six oils to the physical and chemical changes as indicated by the above tests. Although fixed oils and mineral or rosin oils were known to be absent, tests Nos. 9 and 11 were included because both tests afford an excellent means of determining the presence of a nonvolatile residue, probably a polymerization product, which develops in the oil as it ages.

EXPERIMENTAL

A sample of each of the six oils, before and after rectification, was stored for a period of one year under these four storage conditions: (a) a partly filled, tightly stoppered prescription bottle; (b) a partly filled prescription bottle closed with a loose cotton plug; (c) a partly filled, tightly stoppered amber bottle; and (d) a tin can closed with a screw cap. All samples were stored at room temperature for 12 months, and then subjected to the above ten tests.

The results with the rectified oils are given in Table II. The results with the unrectified samples

are omitted since there were no significant differences in the behavior of the two qualities of oils under these storage conditions except possibly that the unrectified samples of the two wood oils proved to be more stable in the cotton-plugged bottles than did the corresponding rectified samples.

In a second restricted series of storage experiments it was found that samples of oil No. 6 stored in aluminum cans (9) for six months both in the presence and absence of air deteriorated rapidly according to tests Nos. 4, 8, 9, 11 and 13. An identical sample tightly stoppered and stored in a refrigerator changed, but to a lesser extent.

To observe the effects of light on oil of turpentine preserved in the absence of air, rectified samples of oils Nos. 1 and 6, selected as representatives of wood and gum turpentine oils, respectively, were put up patent literature on synthetic antioxidants developed as preservative for rubber (17, 18).

EXPERIMENTAL

The problem of finding a suitable preservative agent was approached by preparing seven samples of each of the six rectified oils in half-filled, screw-capped prescription bottles. One of the seven samples of each oil was left untreated to serve as a control, and to each of the other six samples of each oil were added, respectively, 0.1% (in the case of ethyl alcohol 0.5%) of the following preservative agents: (a) an aliphatic alcohol (ethyl alcohol), (b) a phenol (thymol), (c) an ether containing a phenolic hydroxyl (guaiacol), (d) an alicyclic alcohol

TABLE II.--RECTIFIED OILS OF TURPENTINE AFTER ONE YEAR OF STORAGE

Rectified Oil	Closed Prescription Bottle	Cotton-Plugged Prescription Bottle ^a	Closed Amber Bottle ^a	Closed Tin Can ^a
Steam distilled wood	None (passed all tests)	(1) (4) (7) (8) $(9)(11)$ (12) (13)	None	None
Destructively distilled wood	None	(1)(8)(9)(12)	(1)(11)(13)	(1)(13)
Fire distilled gum (dehydrated)	None	(1) (4) (7) (8) $(9)(11)$ (12) (13)	None	None
Fire distilled gum (undehydrated)	None	(1) (4) (8) (9) $(11)(12)$ (13)	None	None
Steam distilled gum (dehydrated)	None	(1) (4) (8) (9) $(11)(12)$ (13)	None	None
Vacuum distilled gum	None	(1) (4) (8) (9) $(11)(12)$ (13)	None	None

^a Numerals refer to the numbered specifications and tests given in Table I with which the respective oils failed to comply after one year of storage under the conditions specified.

under a vacuum in clear glass. One sample of each oil was stored in the dark and another in an east window for six months. The two samples stored in the dark showed no change according to the ten selected tests. The sample of oil No. 1 stored in the light failed to pass test No. 1 while the corresponding sample of oil No. 6 failed to pass tests Nos. 1 and 11.

A freshly rectified sample of oil No. 6 which passed all U. S. Pharmacopœia XII tests for purity, having an exact specific gravity of 0.8576 and a residue content of 0.0089 Gm. per 5 cc., was stored in a prescription bottle under a carbon dioxide atmosphere for one year. An examination of the oil at the end of the period showed that it was practically unchanged, having a specific gravity of 0.8610 and a residue content of 0.0147 Gm. per 5 cc., and passing all other U. S. Pharmacopœia XII tests.

PART III. THE PRESERVATION OF TURPEN-TINE OILS WITH CHEMICAL PRESERVATIVES

Since most of the changes in oil of turpentine are caused by oxidation, and in view of the fact that these reactions are influenced by moisture (10) the preservatives usually found to be effective in the oil have been antioxidants, reducing and dehydrating agents. According to previous reports the most promising of these agents are: hydroquinone (11, 12, 13), pyrogallol (12, 14), lower aliphatic alcohols (15), sodium sulfite (16), calcium oxide (16) and metallic magnesium and tin (9). The more recent reports on this subject have come from the (α -terpineol), (e) an ester (methyl salicylate), and (f) a cyclic ketone (camphor). The low concentration of 0.1% of the preservatives was used since appreciable amounts of these compounds could not be tolerated in medicinal turpentine oil. The prepared samples and the controls were stored for 12 months, and then unstoppered and exposed to the air for an additional period of three months (July 29 to October 20, 1942). At the end of this period all samples were tested by the ten selected official tests.

The results of these tests are shown in Table III. The numerical values are given in the case of specific gravity, distillation range, and mineral or rosin oil residue, and a capital X is placed after the value if it is not within in the U. S. Pharmacopœial XII range. In the case of the other tests a plus sign is used to indicate passing, a capital X indicates failure to pass, and a question mark indicates a border line result.

In view of the definite superiority of the phenols, guaiacol and thymol, in preserving all of the oils, rectified samples of each of these six oils were treated with 0.1% of an additional series of substituted phenols, including hydroquinone and pyrogallol which were used for comparison purposes. Several nitrogen derivatives, analogs of which are known to act as antioxidants, were included in this series of experiments. The samples, with controls, were stored in partly filled, cotton-plugged, prescription bottles at room temperature from December 11, 1942, until June 14, 1943. After this period the samples were tested by subjecting them to the simple tests Nos. 1, 9, 12 and 13, and if the preserved sample looked promising with these tests it was carried through the entire series of ten tests. The most effective preservatives of those tried are listed in Table IV which gives the results of these preservatives when used with oil No. 6 (vacuum-distilled gum turpentine).

While the preservatives listed in Table IV were the most effective they represent only about onefourth of the compounds studied. To focus more attention on these preservatives, some of the compounds which significantly failed to preserve the oil are classified and listed below.

Alcohols: glycerin, "Cellosolve" solvent, tocopherol. Nitrogen derivatives: N-acetyl-p-aminothymo and dibenzoyl-p-aminothymol.

PART IV. A CRITICAL SURVEY OF THE U. S. PHAR-MACOPŒIA XII MONOGRAPHS ON OIL OF TUR-PENTINE AND RECTIFIED OIL OF TURPENTINE *Definition.*—While the official tests would

TABLE III

				TABLE II	.1					
Oil No. 1. Control Ethyl alcohol Thymol Guaiacol & Terpineol Methyl salicylate Camphor Oil No. 2. Control Ethyl alcohol Thymol Guaiacol & Terpineol Methyl salicylate Camphor (not avail-	$XXXXXX + + + + X + \cdot Color (1)$	++++++++++++++ Odor and Taste (2)	++++++++++++ Solubility (3)	(f) 0.982 X 0.933 X 0.935 X 0.935 X 0.935 X 0.935 X 0.935 X 0.938 X 0.938 X 0.938 X 0.938 X 0.929 X 0.928 X 0.928 X 0.928 X 0.928 X 0.928 X 0.928 X	+++++X++++++X Reaction (7)	99 89 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.	(0) SILO PEIXE LIXE LIXE (0)	silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo silo	XXX VXX XXX++ VX Foreign Substance A	XXXXX XXX++XX ^{Foreign} Substance B
able) Oil No. 3. Control Ethyl alcohol Thymol Guaiacol α -Terpineol Methyl salicylate Camphor Oil No. 4. Control Ethyl alcohol Thymol Guaiacol α -Terpineol Methyl salicylate Camphor Oil No. 5. Control Ethyl alcohol Thymol Guaiacol α -Terpineol Methyl salicylate Camphor Oil No. 6. Control Ethyl alcohol Thymol Guaiacol α -Terpineol Methyl salicylate Camphor Oil No. 6. Control Ethyl alcohol Thymol Guaiacol α -Terpineol Methyl salicylate Camphor	x ? x x ? ? ? ? x ? x + x x x x ? x + ? ? ? x x x + x x x	******	*******	$\begin{array}{c} 0.991 \ \ \text{X} \\ 0.936 \ \ \text{X} \\ 0.936 \ \ \text{X} \\ 0.939 \ \ \text{X} \\ 0.939 \ \ \text{X} \\ 0.937 \ \ \text{X} \\ 0.937 \ \ \text{X} \\ 0.943 \ \ \text{X} \\ 0.937 \ \ \text{X} \\ 0.938 \ \ \text{X} \\ 0.941 \ \ \text{X} \\ 0.944 \ \ \text{X} \\ 0.943 \ \ \text{X} \\ 0.941 \ \ \text{X} \\ 0.944 \ \ \text{X} \\ 0.944 \ \ \text{X} \\ 0.943 \ \ \text{X} \\ 0.944 \ \ \text{X} \\ 0.944 \ \ \text{X} \\ 0.944 \ \ \text{X} \\ 0.939 \ \ \text{X} \\ 0.938 \ \ \text{X} \\ 0.939 \ \ \text{X} \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \ 0.939 \ \$	X++++++X++++++X+++++++++++++++++++++++	49 XX 94 XX 94 XXX 94 XXX 94 XXX 94 88 XX 94 94 88 XX 94 94 88 XX 95 XXX 95 55 XXX 96 70 XX 96 70 XX 96 70 XX 96 70 XX 97 70 XX	xx+xxxx xx++xxx xx++xxx xx~+xxx	$\begin{array}{c} 2.611 \ {\rm X} \\ 1.352 \ {\rm X} \\ 0.029 \ {\rm X} \\ 1.339 \ {\rm X} \\ 1.339 \ {\rm X} \\ 1.424 \ {\rm X} \\ 0.643 \ {\rm X} \\ 2.652 \ {\rm X} \\ 1.660 \ {\rm X} \\ 0.022 \ {\rm X} \\ 0.015 \\ 1.610 \ {\rm X} \\ 1.478 \ {\rm X} \\ 0.613 \ {\rm X} \\ 2.666 \ {\rm X} \\ 1.507 \ {\rm X} \\ 1.507 \ {\rm X} \\ 0.023 \ {\rm X} \\ 0.023 \ {\rm X} \\ 0.010 \\ 1.501 \ {\rm X} \\ 1.526 \ {\rm X} \\ 0.025 \ {\rm X} \\ 1.620 \ {\rm X} \\ 0.025 \ {\rm X} \\ 1.554 \ {\rm X} \\ 0.554 \ {\rm X} \\ 0.568 \ {\rm X} \\ \end{array}$	~x+~xxx xx++xxx xx++xxx xx++xxx	xx+xxxx xx++xxx xx++xxx xx+xxx

Phenols: phenol, *m*- and *p*-cresols, carvacrol, *o*and *p*-nitrophenols, resorcinol monomethyl ether, *o*-hydroxybenzyl alcohol, 2-hydroxy-3methoxybenzaldehyde, salicylic acid, salol, dithymol sulfide, phloroglucinol and phenolphthalein.

Ethers: anisole and beechwood creosote.

indicate that oil of turpentine from destructively distilled wood is inferior to that from the oleoresin, the original purity and keeping qualities of the oil obtained by extracting wood closely approach those of the oil from the oleoresin. Whether the presence of some 18% of monocyclic hydrocarbons and

Naphthols: β -naphthol, 3-hydroxy-2-naphthoic acid.

2% of alcohols and aldehydes (19) in the extracted oil renders it unfit for oral use is, of course, another question.

Solubility.—This test may serve to detect certain adulterants in turpentine oils, but it is worthless in eliminating oils which have deteriorated on aging. All of the samples in this study, even those containing 40% of nonvolatile matter, easily passed this test. The test could be strengthened by requiring that oil of turpentine dissolve in an equal *Mineral Oil.*—While this test is probably the most practical and dependable yet used to detect petroleum products in turpentine oil, it measures low percentages of kerosene only approximately, and gasoline not at all without the use of the centrifuge.

Mineral or Rosin Oils.—This test is most useful in determining the amount of nonvolatile material formed in an aged oil in addition to being a test for mineral or rosin oils. The terminal point of the evaporation

TABLE	IV
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Oil No. 6. Control Guaiacol o-Cresol Thymol p-Chlorothymol Hydroquinone Pyrogallol a-Naphthol Resorcinol p-Methoxyphenol Catechol Quinhydrone p-Nitrosothymol	XXX + XXX + X Color (J)	+++++~++++++ ~ Odor and Taste (2)	+++++++++++++ Solubility (3)	(f) Aixing Constraints (f) Aixing Constraints (f) (f) (f) (f) (f) (f) (f) (f)	++++++XXX+X Reaction (7)	666 76 76 76 76 76 76 76 76 76 76 76 76	+++++++×++×+++************************	sic Sic Sic Sic Sic Sic Sic Sic S	++++++×~××+× ^{Foreign} Substance A	·+++X+··+XXX~X Foreign Substance B
(0.05%) p-Nitrosophenol so-	+	+-	+	0.863 X	+	90	+	0.020 X	+	+
dium salt (0.05%) m-Aminophenol (0.05%)	x	+	+	0.863 X	+	90	+	0.018 X	+	+
p-Benzylaminophenol Phenyl α-naphthyl- amine	X X	+ +	+ +	0.862 0.865 X	+ +	92 92	++++	0.014 0.040 X	+ ?	;
2-Hydroxybenzyli-	x	+	+	0.867 X	+	92	?	0.042 X	+	+
dene-4-aminothymol 4-Methoxybenzyli- dene-4-aminothy- mol	x	+	+	0.865 X	+	89 X	+	0.030 X	+	+
3-Methoxy-4-hy- droxybenzylidene- 4-aminothymol	x	+	+	0.865 X	+	88 X	+	0.029	+	+

volume of 95% ethyl alcohol and/or in 6 volumes of 85% ethyl alcohol.

Reaction.—This test is inconclusive. For example, a series of 5-cc. samples of three different oils, which could be said to be neutral or slightly acid to litmus, required 0.02, 1.07 and 1.81 cc., respectively, of 0.0856 N sodium hydroxide to neutralize it to phenolphthalein when titrated in neutral ethyl alcohol. In this connection it should be pointed out that the German Pharmacopœia of 1926 required that 2.5 Gm. of Rectified Oil of Turpentine require not more than 0.3 cc. of 0.1 N potassium hydroxide to neutralize it to phenolphthalein. is difficult to detect, however, especially when a sample contains a large amount of nonvolatile material. More reproducible results could be obtained by requiring a definite period of evaporation and desiccation to a constant weight. It was also found that saturation of the sample of oil with water prior to evaporation increased its amount of residue as much as 16% as shown by this test. This is significant in view of the fact that dehydrated and undehydrated oils are marketed.

Foreign Substances A.—To determine the results of this test by observing the color of only the oily layer leads to error since the

emulsification occurring during the 24 hours renders this layer opaque regardless of the purity of the oil. It was our experience that this test would not be made too severe by requiring that both the alkaline and oily layers be observed for color.

Foreign Substances B.—This test, which probably detects higher-boiling polymers in aged oils, is the strictest official test for such oils. It was observed that considerable heat is produced upon the addition of the acid. The amount of heat produced and the depth of color are in a direct ratio.

Storage.-It is known that certain metals catalyze the changes which cause deterioration of turpentine oil (9, 16). Our results would indicate that glass containers are best for storing this oil, tin containers are almost as good, and aluminum containers are unsatisfactory.

SUMMARY AND CONCLUSIONS

1. Samples of two American oils of turpentine from wood and four from the oleoresin have been described and tested for purity, before and after rectification, by the U. S. Pharmacopœia XII tests.

Of the six oils examined the four 2. oleoresin oils were definitely purer than the oils obtained from wood by extraction and destructive distillation, respectively, al-

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 (2) "United States Pharmacopeia, XII" Mack

Printing Co., Easton, Pa., 1942, p. 336. (3) "United States Pharmacopœia, XI" Mack

Printing Co., Easton, Pa., 1935, p. 269. (4) "Allen's Commercial Organic Analysis," P. Blakiston's Son and Co., Philadelphia, Pa., 1925, Vol. 4, p. 140.

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(6) U. S. Department of Agriculture Publication NS-52.

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(8) Brooks, B. T., "The Chemistry of Non-Benzenoid Hydrocarbons," Chemical Catalog Co., New York, N. V., 1922, p. 428.
(0) Kenther W. Luchkim, Bran, 4 (1925), 6

(9) Korotkov, K., Lesokhim. Prom., 4 (1935), 6.

though the purity of the wood oil obtained by extraction approaches that of the oils from the oleoresin.

As judged by the ten official tests 3. selected to determine purity, the wood oils and the oleoresin oils were about equal in their stability with respect to the number of changes undergone under various storage conditions, but the wood oils changed to a greater degree.

Light had little influence on the 4. stability of the oils, but the amount of air in contact with the sample profoundly affected it in all cases. Closed tin containers are about equal to glass for the preservation of these oils, but aluminum containers proved unsatisfactory for this purpose.

5. Under the influence of chemical preservatives, the sample of oil obtained by the extraction of wood held up better than did the other samples, while the destructively distilled wood oil was the most difficult to The most effective chemical prepreserve. servatives were in the order named: hydro*p*-benzylaminophenol, quinone, hydroquinone monomethyl ether, catechol, guaiacol and p-nitrosophenol sodium.

6. A survey has been made of the U.S. Pharmacopœia XII monographs on Oil of Turpentine and Rectified Oil of Turpentine, and several changes have been recommended with respect to these monographs.

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