must remember, however, that real growth on any particular diet will not take place unless all the vitamines are present. It is evident, therefore, that any one of the vitamines, or for that matter the mineral content of the diet, may be the limiting factor of growth.

Regarding the questions raised by Dr. Taylor, the alcoholic extract of cod liver oil, while it is antirachitic, is not antiophthalmic. The oil remaining after the alcoholic extraction still contains the major portion of the antirachitic and antiophthalmic vitamines originally present.

The acid extract is both antirachitic and antiophthalmic. The residual oil, after acid extraction, is no longer active. In other words, the acid has extracted the active principles of the oil.

The concentrate made by means of these acid extractions followed by saponification contains the antiophthalmic and antirachitic vitamines; therefore, this particular process is an advantage in that we have been able to extract both vitamines.

As regards the statement of Dr. Wood that the concentrate does not represent all of the therapeutic value of the cod liver oil, I feel that it is not valid, for this reason: The activity of the oil at one time was attributed to its nitrogen or phosphorus content. This has been disproven because the concentrate contains neither nitrogen nor phosphorus; and yet if it is mixed with sugar, for example, the therapeutic activity of the mixture is exactly the same as that of fresh cod liver oil. In other words, the oil has been replaced by sugar but the therapeutic effect is the same because of the presence of the concentrate.

The oil remaining after acid extraction is no longer effective in those nutritive disturbances for which cod liver oil is used. In view of the foregoing, we are justified in saying that the therapeutic value of cod liver oil, as far as experimental evidence is concerned, is due to its vitamine content.

As regards the stability, we have found that the product is stable even after seven or eight months, so far. As long as the concentrate is protected against oxidation, there is no loss of activity. This is assured by carrying out the process in a non-oxidizing atmosphere.

As to the details of manufacture, they are largely given in the detailed paper to be published shortly in the *Journal of Metabolic Research.*

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DIETHYLPHTHALATE III.*

BY J. A. HANDY AND L. F. HOYT.

1. INTRODUCTION.

A survey of the data on diethylphthalate appearing since our last paper on this subject (1) and a study of some recently proposed detection tests, have been made.

2. METHODS OF DETECTION.

A detection test for diethylphthalate involving the use of pyrogallol, quite different in its results from the fluorescein type test or the phenolphthalein test of Calvert, has been proposed by Eilles (2). This author deprecates the use of phenol-H_2SO_4 (*i. e.*, the Calvert Test) as being of little use (no reasons given) and states that the resorcinol- H_2SO_4 tests, (*i. e.*, like those of Lyons, Handy and Hoyt, and Andrew) are uncertain because of fluorescence in blanks. The Eilles test, which is official at the German Bureau of Internal Revenue (Hamburg) is carried out as follows:

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* Scientific Section, A. PH. A., Buffalo meeting, 1924.

(1) In the case of spirits 20 to 50 cc. of sample to which are added 5 drops of a 15% solution of sodium hydroxide, are evaporated on the steam-bath in a flat porcelain dish.

(2) In the case of liquors which contain sugar, or extractive matter and other substances, particularly formic acid, 20 to 50 cc. of sample are diluted with an equal volume of water and shaken out with 25 cc. of petroleum ether (30–50° b. p.). Tinctures (extracts of drugs) are treated with the necessary amount of lead acetate and filtered before extraction. Petroleum ether extract is evaporated in a flat porcelain dish without the addition of caustic soda.

The evaporated residues from (1) and (2) are mixed with 5 to 10 drops of 1.84 H_2SO_4 with the aid of a glass stirring rod and heated over a small flame with continuous stirring, until the fumes of the acid begin to be given off. Then the dish is heated somewhat more strongly at the spot where the pyrogallol is to be applied, in order that the pyrogallol will melt immediately. By tilting the dish the sulphuric acid is allowed to flow away somewhat and a few crystals of pyrogallol are immediately placed on the heated spot. In the presence of diethylphthalate a genuine violet-red color is immediately produced.

The test has been severely criticized in Germany. Wewers (3) claims that blank tests made on alcohol of known purity and even blanks with the reagents only, all gave a violet-red color. Wewers prefers to rely on the sense of taste for detection of diethylphthalate, which taste is pronounced even in high dilutions, although he gives no quantitative data.

Note: We have previously pointed out (JOURNAL OF THE AMERICAN PHARMACEUTICAL ASSOCIATION, 13, p. 607) that 0.1% diethylphthalate can be easily detected in miscellaneous beverages by the sense of taste.

Utz (4) likewise finds the official German method faulty. Other colors whose interpretation is doubtful are liable to be produced. Formic acid, against the presence of which Eilles warns as giving a reaction very similar to diethylphthalate, was found by Utz to give more of a brick-red, which would not be mistaken for the color produced by the ester.

Utz proposes a modification of Lyons method in which he eliminates the use of caustic soda and heats a 1-cc. sample (of spirits, or of samples free from appreciable amounts of interfering substances) with 1 cc. 1.84 H_2SO_4 until white fumes of the acid appear. A few crystals of resorcinol are then added and the mixture is heated until it turns brownish red, cooled and poured into 500 cc. water made alkaline with ammonia.

Leffman (4) reports his experience with the Utz method, which he finds simpler and quicker than methods previously suggested. This author states, however, that although the Utz method will detect very small amounts of diethylphthalate, it gives positive "blank" tests which are apt to be misleading. Good results were obtained in the presence of commercial methanol and reasonable amounts of isopropyl alcohol, though an excess of the latter tended to obscure the result.

Leffmann recommends Andrew's Method (6) as simple and dependable, satisfactory results being obtained on 2 milligrams of diethylphthalate in the presence of small amounts of acetaldehyde and commercial methanol and isopropyl alcohol.

3. FLUORESCENCE PHENOMENA.

Considerable data has appeared in the recent literature concerning the fluorescence of fluorescein and analogous compounds. In our second paper (JOURNAL OF THE AMERICAN PHARMACEUTICAL ASSOCIATION 13, p. 606) we stated that possibly the examination of the absorption spectra of genuine fluorescein and that of the resorcinol-saccharin compound, *i. e.*, sulphonefluorescein, so similar to the naked eye, might show differences. Orndorff and Vose (7) have reported just such an expected difference. They examined the absorption spectra of true fluorescein and that of sulphonefluorescein (which they prepared in the pure crystalline condition from several compounds including saccharin). Both compounds in equal dilutions, which could scarcely be differentiated with the naked eye, showed single absorption bands but with different maxima, that of sulphonefluorescein lying at $\lambda = 5249$ and being 10% more intense than that of fluorescein lying at $\lambda = 5209$.

Additional data on the tendency of products made by condensation with resorcinol to exhibit fluorescence in alkaline solution is furnished by Sen and Sinha (8). These investigators condensed resorcinol with a number of aldehydes and among others report the following, listed in Table I.

TABLE I.

Resorcinol condensed with	Color of product. In conc. H_2SO_4.	 In alkaline solution.
Benzaldehyde	Orange	Red with red-green fluorescence
Salicylic Aldehyde	....	Orange with red-blue fluorescence
Vanillin	Pink	Red-violet with green fluorescence
Chloral Hydrate	Yellow	Red with red-green fluorescence
Furfurole	Orange	Red with red-green fluorescence
Anisaldehyde	Dark red	Red with bluish fluorescence
Paraldehyde	....	Red with red-green fluorescence

They noted that the substitution of the trihydric phenol, pyrogallol, for resorcinol in condensation experiments with benzaldehyde gave a product devoid of fluorescence in alkaline solution.

4. MISCELLANEOUS DATA ON DIETHYLPHTHALATE.

Utz makes some interesting observations on diethylphthalate. It appears that the authorization of this denaturant in Germany on May 12, 1922 in the proportion of 2 volumes per 100 volumes of alcohol (subsequently reduced on Jan. 19, 1923 to 1 volume per 100) was followed quickly by complaints and hesitancy in its use. According to Utz it was claimed by Dr. Payr, director of the surgical clinic at Leipzig, that alcohol denatured with (2%) diethylphthalate irritated the skin, which it reddened with a burning, itching sensation and caused it to become rough and scaly. He stated further that it produced a numbing sensation in the finger tips.

The German Druggists Association applied to the German Health Office for a ruling in regard to the use of Alcohol denatured with diethylphthalate in cosmetics. In reply (*Pharm. Ztg.*, 68, 643 (1923)) the association was assured that the ester could be used without risk, in the quantities in which it was added to denatured alcohol, in toilet waters and preparations for external use.

Utz mentions the use of diethylphthalate as an adulterant, reported by Schimmel as being found by them in Oil of Rose and Oil of Lavender. He further states that the ester is better known perhaps on account of its war uses, among which was its employment as a glycerin substitute by the German Army in filling the heating jackets of field kitchens.

5. Experimental Part: Methods of Detection.

(A) EILLES PYROGALLOL TEST.

This test was applied to diethylphthalate and to a number of other compounds. In our experience this Official German Test for Diethylphthalate was found to be of little or no value for the following reasons:

1. The intensity and duration of the color reaction vary according to the degree of heat applied.
2. Impossible to standardize the procedure so that different operators can consistently get the same result.
3. Not sufficiently characteristic for diethylphthalate.
   (*a*) Blanks are highly colored and of a bluish violet quite similar to the reddish violet produced with diethylphthalate.
   (*b*) Saccharin and Vanillin were found to give a test with pyrogallol indistinguishable from that produced with diethylphthalate.
   (*c*) Other substances such as succinic and formic acids, as well as chloral hydrate, give similar and confusing color reactions.
4. At best the test is very fugitive and fades quickly to a brown. Unless the test is carried out very carefully the color reaction is liable to be obscured by the charring or blackening of the reagent.
5. Less sensitive than the fluorescein type test.

Some experiments were made on the effect of substituting other phenols for pyrogallol in the Eilles test. The procedure was as follows:

Five drops 1.84 H_2SO_4 were heated in a small porcelain capsule *until white fumes of the acid appeared.* Then a few fragments of the phenol under consideration were applied to the heated area and the color reaction noted. All of the phenols tried give a bewildering series of color changes in blank tests, as follows:

	Reagent.	Colors in blank test.
(1)	(Mono.) Phenol	Amber, reddish brown, dark brown
(2)	(di.) Hydroquinone	Rose, reddish violet, brown, dark green
(3)	(di.) Resorcinol	Yellow, orange, olive-green
(4)	(tri.) Pyrogallol	Rose, reddish violet, blue-violet
(5)	(tri.) Phloroglucinol	Deep yellow, orange, olive-green
(6)	α-Napthol	Red-brown, surrounded by a bright lavender ring
(7)	β-Napthol	Brown, brownish green

The reaction mixtures were poured into dilute alkali. No. 3 Resorcinol showed a green fluorescence, No. 7 β-Napthol a greenish blue fluorescence, the others no fluorescence.

Such intense and fugitive colors were obtained in these blank tests that it was considered a waste of time to continue experiments with a test of this type.

(B) REACTION BETWEEN VARIOUS PHENOLS, AND CERTAIN AROMATIC AND ALIPHATIC COMPOUNDS.

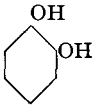
A systematic study was made of the possibility that some phenol other than resorcinol could be used to better advantage in the detection of diethylphthalate, by formation of a condensation product rather than a spot color test. In order that conditions should be as uniform as possible, the procedure of heating for 3 minutes in an oil-bath at 160° C. was adopted. In addition to a blank test and a test on diethylphthalate, we applied the same procedure, at the same time, to a number of other substances which we, or others, have found to condense with resorcinol to form compounds showing fluorescence in alkaline solution.

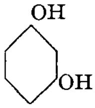
The available phenols and the substances with which they were condensed are listed in Table II.

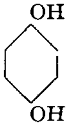
TABLE II.

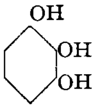
A. Phenols.

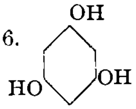
1.  Phenol

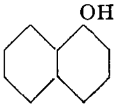
2.  Catechol (o)

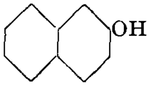
3.  Resorcinol (m)

4.  Hydroquinone (p)

5.  Pyrogallol (1-2-3)

6.  Phloroglucinol (1-3-5)

7.  α-Napthol

8.  β-Napthol

B. Substances tested.

1. Diethylphthalate
2. Acetone
3. Anisaldehyde
4. Benzaldehyde
5. Chloral Hydrate
6. Formic Acid
7. Malic Acid
8. Saccharin
9. Salicylaldehyde
10. Succinic Acid
11. Vanillin

The procedure was as follows:

0.05 gram of each phenol, 0.05 Gm. or 1 drop of each substance (if liquid) to be tested and 1 cc. 1.84 H_2SO_4 were placed in hard glass test-tubes and the tubes heated for 3 minutes in an oil-bath maintained at 160° C. After cooling the reaction mixtures were added to 100 cc. of a 5% solution of NaOH and the color and fluorescence noted. (It is quite probable that in some cases no condensation took place under the above conditions of concentration and temperature, but this is of little importance in this investigation, since we are concerned only in comparing reactions of diethylphthalate and other substances under identical conditions.)

A blank test was, of course, made on each of the phenols. None of these series of tests afforded a method of detection equal to the fluorescein test, *i. e.*, condensation with resorcinol. The intensity and brilliance of fluorescein, and particularly its permanence, are much greater than in the case of any of the condensation products from equal weights of substances (particularly formic acid, saccharin, and succinic acid), reacting in a similar manner with resorcinol.

To study the effect of temperature, the condensation with each of the phenols and diethylphthalate was also made at a higher temperature.

The colors of the final alkaline solution of the blanks and of the condensation products of diethylphthalate with the several phenols are recorded as Table III.

Procedure: A. 0.05 Gm. each of the phenols and diethylphthalate, 1 cc. 1.84 H_2SO_4, heated together for 3 min. at 160° C.

Procedure: B. 0.05. Gm each of the phenols and diethylphthalate, 12 drops 1.84 H_2SO_4, heated over a free flame until fumes of the acid started to appear (*i. e.*, procedure like the Calvert Test).

TABLE III.

	Procedure A, Color in alkaline solution.		Procedure B, Color in alkaline solution.	
	Blank.	Test with diethylphthalate.	Blank.	Test with diethylphthalate.
Phenol	Colorless	[1]Colorless	Colorless	Pink
Catechol	Bright yellow	Bright yellow	Greenish yellow	Dark brown
Resorcinol	Colorless	Yellow with intense greenish fluorescence	Colorless	Reddish brown, green fluorescence
Hydroquinone	Pale olive-green, bluish fluorescence	Light brown with bluish fluorescence	Amber	Purplish brown
Pyrogallol	Greenish yellow	Yellowish green	Olive-green	Olive-green
Phloroglucinol	Amber	Brownish red	Amber	Reddish brown
α-Napthol	Pale amber	Amber	Pale amber	Light brown, bluish fluorescence
β-Napthol	Pale greenish yellow, green fluorescence	Exactly like blank, same degree of fluorescence	Brown, green fluorescence	Brown, green fluorescence

[1] Condensation to form phenolphthalein does not take place apparently at 160° C. under the conditions of our test. A higher temperature, as in the Calvert test, appears to be necessary.

It will be noted that procedure B, which was tried with the various phenols and diethylphthalate only, heating at a higher temperature than A, tended to produce deeper colored final solutions.

In the course of the series of tests made according to procedure A with each of the 8 phenols and 11 substances listed in Table II, two outstanding facts were observed, *viz.:*

(1) The fact that some phenols give rise to condensation products which are fluorescent in alkaline solution whereas other phenols do not.

Classification of Phenols with regard to the fluorescence, or lack of it, shown in alkaline solution by the condensation products made by heating 0.05 Gm. of a phenol (column A, Table II), with 0.05 Gm. of certain substances (column B, Table II) and 1 cc. 1.84 H_2SO_4 for 3 min. at 160° C., is tabulated in Table IV.

TABLE IV.

Phenols whose condensation products are fluorescent in alkaline solution.	Phenols whose condensation products show no fluorescence in alkaline solution.
Resorcinol—Green Fluorescence	Phenol
Hydroquinone—Blue Fluorescence	Catechol
β-Napthol—Blue Fluorescence	Pyrogallol
	Phloroglucinol
	α-Napthol

With resorcinol, hydroquinone, and β-napthol, all of the final test solutions from the 11 reacting substances showed fluorescence of varying intensity, but unmistakable in all cases.

(2) A remarkable parallelism was found to exist in the appearance, *i. e.*, as regards color and fluorescence or lack of it, of the final alkaline solutions of the condensation products of diethylphthalate, formic acid, saccharin, and succinic acid with each of the phenolic substances of column A, Table II. This data supplements and elaborates the data previously reported in our second paper under part II B (4) "Fluorescent Phenomena and color reactions resulting from condensation of resorcinol and phenol with substances other than phthalic acid derivatives."

While acetone, chloral hydrate, and the various aldehydes gave in general dark brown, red, or purple in the final test solutions throughout the series, the above four similarly reacting substances gave in general clear light-colored solutions.

(C) COMPARISON OF THE SENSITIVENESS OF THE FLUORESCEIN TESTS FOR DIETHYLPHTHALATE OF UTZ, ANDREW, AND HANDY-HOYT.

Each test was carried out carefully according to its specific directions on 0.2 mgms. and 10 mgms. of diethylphthalate in 1 cc. absolute ethyl alcohol. Results are shown in Table V.

TABLE V.

	A, Utz.	B, Andrew.	C, Handy-Hoyt.
Blank (1 cc. absolute ethyl alcohol)	Reddish brown strong fluorescence	Colorless	Colorless
1 cc. absolute ethyl alcohol and 0.2 mgm. diethylphthalate	Solution less red than blank. Strong fluorescence	Faint fluorescence	Distinct—stronger than B
1 cc. absolute ethyl alcohol and 10 mgms. diethylphthalate. (Equivalent to 1 cc. 39-C Spec. Den. Alcohol, 1% diethylphthalate).	Solution dark amber. Intense fluorescence	Intense yellow-green fluorescence	Intense yellow-green fluorescence

Due to the high temperature employed in the Utz test, a strongly positive blank is, in our opinion, unavoidable, and renders this test of little value in detecting traces of diethylphthalate. In the above tests the fluorescence of the blank was greater when fresh than that with 0.2 mgm. diethylphthalate.

The Andrew test, we belive, is rightly regarded by all who have tried it as a very satisfactory test, convenient, rapid, and sensitive; although our experience has been that the maximum amount of fluorescein is not produced at the moderate temperature employed and that the fluorescence is correspondingly less intense than that obtained on the same weight of diethylphthalate in a test like our own made at 160° C. The Andrew Test is the least liable to give positive "blanks," because of the very moderate condensation temperature employed.

We would again point out that the matter of positive blanks may be due primarily, to (1) too high a temperature, especially if part of the resorcinol remains undissolved in the acid and exposed to the air on the sides of the dish or tube during the condensation. (2) Impurities of various kinds in the sample under test. In this connection we have repeatedly obtained blanks entirely devoid of fluorescence when working with absolute or otherwise specially purified ethyl alcohol, by both the Andrew Test and our own, and have likewise obtained temporarily positive blanks by both methods on commercial alcohols free from diethylphthalate.

(D) USE OF DILUTE H_2SO_4 IN PETROLEUM ETHER EXTRACTION OF DIETHYLPHTHALATE.

Comparison of extractions with petroleum ether from solutions diluted (1) with water and (2) with dilute H_2SO_4 (10%).

A small quantity of "39-C." was made in the laboratory, using 50 cc. Absolute Alcohol and 0.50 cc. Diethylphthalate.

1. *Gravimetric Test.*—In order to secure weighable quantities of the ester, extractions were made under the following conditions.

	Extraction from: Aqueous solution.	Dilute H_2SO_4.
Test solution of the ester		
5 cc. Dist. H_2O		
4 cc. Absolute Alcohol		
1 cc. 39-C.	10 cc.	10 cc.
Added Liquid	10 cc. (H_2O)	10 cc. (10% H_2SO_4)
Petroleum Ether	20 cc.	20 cc.
Theoretical weight of diethylphthalate	0.0111 Gm.	0.0111 Gm.
Wt. of ester recovered in petroleum ether extract	0.0104 Gm.	0.0105 Gm.

Both extractions were shaken 20 times in the same manner in separatory funnels and allowed to stand over night. In the above test the concentration of diethylphthalate in the original 10 cc. test portion is 0.1%, and 0.05% after dilution with the added liquid. The concentration of alcohol in the final liquid under extraction was 25% by volume.

2. *Qualitative Test.*—Effect of concentration of alcohol, and acid on the extraction of minute amounts of the ester in qualitative tests.

(A) Extraction of a 10 cc. portion of a sample containing 10% by volume of alcohol (comparable to sweet wine) and 0.01% of diethylphthalate after dilution with (1) water, and (2) dilute H_2SO_4.

	1.	2.
Ester	1 mgm.	1 mgm.
Alcohol	1 cc.	1 cc.
Water	9 cc.	9 cc.
Added Liquid	10 cc. Water	10 cc. H_2SO_4 (10%)
Petroleum Ether used	20 cc.	20 cc.
Fluorescein test on extract	+++	++

Note: Extraction from aqueous solution gave in this case a fluorescence of greater intensity than was obtained on the strictly parallel extraction from dilute H_2SO_4.

(B) Extraction of a 10 cc. portion of a sample containing 50% by volume of alcohol (comparable to whisky, brandy, or gin) and 0.01% of diethylphthalate after dilution with (1) water, and (2) dilute H_2SO_4.

	1.	2.
Ester	1 mgm.	1 mgm.
Alcohol	5 cc.	5 cc.
Water	5 cc.	5 cc.
Added Liquid	10 cc. Water	10 cc. H_2SO_4 (10%)
Petroleum Ether used	20 cc.	20 cc.
Fluorescein test on extract	+	++

Note: Extraction from acid solution gave in this case the stronger fluorescein reaction.

SUMMARY.

1. Extraction of 10 cc. of an alcoholic sample containing a ponderable amount (*i. e.*, 0.1%) of diethylphthalate with petroleum ether effects the same high percentage of recovery of the ester, irrespective of whether the sample is diluted with equal volumes of water or 10% sulphuric acid.

2. Qualitative fluorescein tests on the petroleum ether extracts of 10 cc. samples containing 1 mgm. (0.01%) of the ester show no consistent superiority of dilution of the sample with 10% sulphuric acid. Results are variable; several tests for the ester in beverages as well as in solutions of the ester in pure absolute alcohol show sometimes a more intense fluorescein reaction on extracts from an aqueous medium and at other times a superiority in favor of extraction from an acid medium.

3. Due, however, to the beneficial effect of the acid in preventing the formation of emulsions during extraction, its use is recommended. Samples to be extracted should then be preferably diluted with an equal volume of 10% sulphuric acid in place of water.

4. The fact that 10 mgm. diethylphthalate can be extracted nearly quantitatively from a solution whose total volume is 20 cc. and whose alcoholic content is 25% by volume, does not mean that 1 mgm. can be extracted to a like extent from an entirely similar solution. The intensity of fluorescein tests on such extractions of solutions containing 1 mgm. of the ester is much less than that of a blank of 1 mgm. of the ester.

OFFICIAL U. S. TREASURY DEPARTMENT TEST FOR DIETHYLPHTHALATE.

The Bureau of Internal Revenue of the U. S. Treasury Department employ as their official test for diethylphthalate a procedure which combines the essential features of both the Andrew method and our own. Details of this official method, furnished by Dr. J. M. Doran, Head, Industrial Alcohol and Chemical Division, Bureau of Internal Revenue, Washington, D. C., are as follows:

"A. FOR LIQUIDS OR SOLUTIONS CONTAINING A CONSIDERABLE AMOUNT OF DIETHYLPHTHALATE.

"To 10 cc. of the sample add 2 cc. of a 10% solution of sodium hydroxide and evaporate to complete dryness on the steam-bath. Add about 5 cc. of concentrated sulphuric acid and 25 milligrams of resorcin and heat for five minutes on the steam-bath. Transfer the contents to a test-tube and heat for about 5 minutes to 160° C. in a paraffin bath. Cool. Pour contents into about 300 cc. of water and make alkaline with strong sodium hydroxide solution. The presence of diethylphthalate (in the absence of other phthalates) is indicated by the greenish yellow fluorescence of fluorescein."

"B. FOR LIQUIDS OR SOLUTIONS CONTAINING A TRACE OF DIETHYLPTHALATE.

"To not less than 50 cc. (preferably more) of the sample add 0.2 cc. of a 10% solution of sodium hydroxide and evaporate to dryness on steam-bath. Add about 5 cc. concentrated sulphuric acid and 25 milligrams of resorcin and heat for about 5 minutes on the steam-bath. Transfer the contents to a test-tube and heat for about five minutes to 160° C. in a paraffin bath. Cool. Pour contents into about 150 cc. of water and make alkaline with strong sodium hydroxide solution. Allow to stand about 24 hours. A greenish yellow fluorescence of medium intensity when the solution is viewed through the long axis of a Nessler's tube is indicative of diethylphthalate.

"*Note:* A blank test on pure alcohol should be made for comparison. Final alkaline solution should always be allowed to stand for 24 hours before making decision, since fluorescence is always produced, even in the blank. In the absence of diethylphthalate, this fluorescence will entirely fade away upon standing the required length of time. All apparatus should be thoroughly cleaned, since any phthalate will respond to this test."

Attention is called to the fact that this official method recognizes the necessity of interpreting results 24 hours or more after the tests have been made A similar caution was inserted in our second paper, Diethylphthalate II (p. 706).

It is understood of course that any fluorescein-type test for diethylphthalate is to be applied directly only to samples free from interfering substances and that a preliminary extraction with petroleum ether is advisable if the sample contains sugars, extractive principles of drugs, and other interfering substances. The test is positive only for the phthalic radical.

GENERAL CONCLUSIONS.

1. The official German test for diethylphthalate, employing pyrogallol, is of very little value. Substitution of other phenols for pyrogallol is of no advantage.

2. The Utz fluorescein-type test is unsatisfactory because the high temperature employed gives rise to positive blanks showing strong and persistent fluorescence.

3. For all practical purposes the Andrew Test combines a sufficient degree of sensitiveness with rapidity and convenience. In general it is the least troublesome of the fluorescein type tests as regards "blanks."

4. In our opinion the most comprehensive and refined test so far developed is the one now in use by Internal Revenue Dept. This method combines the

essential features of the Andrew Test with a supplementary heating at 160° C. whereby the yield of fluorescein is increased, which tends of course to increase the sensitiveness of the test. In addition this test includes the caution of interpreting results on traces of diethylphthalate after the test solutions have stood at least 24 hours.

ABSTRACT OF DISCUSSION.

James M. Doran said that Mr. Handy was a little over-modest in his last statement, about the test being developed in the Laboratory of the Internal Revenue Department. Part of the work had been done there, but most valuable help was given by Mr. Handy and, in his opinion, most of the credit for the present development of this test is due Mr. Handy and his laboratory. He said further, in substance, that the test has entered largely into the work of the chemists of the Revenue Department, because an attempt is necessary to fix the purity of alcoholic samples taken by agents. It gives some clue or inference as to the possible source of alcohol of questionable nature. It is of considerable importance to pharmacists and the chemists of the Revenue Department would be very glad to see a little more work done on this and other denaturants; thereby great protection would be given pharmacists. The necessity of reliable qualitative tests is obvious; not only does the Department want to be assured of the quality of alcohols that may be used, but also that the tests do not lead to erroneous conclusions. The inferences that might be drawn from finding diethylphthalate in certain samples of alcohol, in the possession of pharmacists, might be unjust and embarrass them; the Department is desirous that these tests be developed so they can be used with assurance.

D. M. Copley said that, in his experience, these products are now nearly odorless; formerly they had a pleasant aromatic odor.

The author replied that they had samples of nearly all diethylphthalates, of commercial consequence, manufactured here and abroad. There is much interest in the subject and many samples are submitted to them for an opinion. Diethylphthalate when made from pure basic products has a slight odor; formerly, some manufacturers added a perfume oil to cover up this odor.

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NEW VETERANS' HOSPITAL.

Construction of a general hospital for war veterans near the Twin Cities or Rochester, Minn., has been approved by President Coolidge.

Plans for the hospital, as drawn up by the Federal Board of Hospitalization, included provision for an observation ward for tubercular patients.

Present hospital facilities at Hot Springs, S. D., will be extended to accommodate 300 more tuberculosis patients.