TABLE III (*Concluded*)

ANALYSES OF HYDROCHLORIDE

	Formula	Chlorine (as AgCl) Calcd., %	Chlorine (as AgCl) Found, %	Nitrogen (Kjeldahl) Calcd., %	Nitrogen (Kjeldahl) Found, %
1	$C_{11}H_{15}ON{\cdot}HCl$	16.60	16.15 16.20	6.57	6.71
2	$C_{10}H_{13}ON{\cdot}HCl$	17.76	17.43 17.42	7.01	6.73 6.72
3	$C_{11}H_{15}ON{\cdot}HCl$	16.60	16.63 16.67	..	..
4	$C_{11}H_{15}ON{\cdot}HCl$	16.60	16.55 16.37	..	..
5	$C_{12}H_{17}ON{\cdot}HCl$	...	[a]	..	..
6	$C_{12}H_{17}ON{\cdot}HCl$	15.58	15.05 15.37	..	..

[a] Chlorine and nitrogen determinations were unsatisfactory.

Summary

1. Acylanilines and acyltoluidines have anesthetic properties, particularly their hydrochlorides, if there is at least a butyrophenone skeleton.
2. Data concerning eight such compounds are given.
3. Six of the eight amines are new and are described together with their intermediates and, in some cases, their derivatives.

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[CONTRIBUTION FROM THE INSECTICIDE DIVISION, BUREAU OF CHEMISTRY AND SOILS]

ROTENONE. I. REDUCTION PRODUCTS OF ROTENONE

BY F. B. LAFORGE AND L. E. SMITH

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Rotenone, the active insecticidal principle of *Derris elliptica*, has been the subject of numerous investigations in recent years.[1] As it is perhaps natural to expect in the case of such a complicated compound, the published chemical articles contain numerous errors, corrections and retractions. It is indeed only in the past year that the empirical formula of rotenone has been established with certainty.

A large share of what is known of the chemistry of the compound has been contributed by the Japanese. Nagai[2] seems to have been the first to isolate it in a pure state. He gave it its present name and showed that it contained a keto group. Ishikawa[3] found that the compound was optically active, and Kariyone[4] showed the presence of methoxyl and an unsaturated bond, and obtained tubaic acid of the formula $C_{12}H_{12}O_4$

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[1] The Insecticide Division of the Bureau of Chemistry and Soils has collected and abstracted over 250 articles on *Derris*, and a publication entitled "A Bibliography of *Derris* (*Deguelia*) Species Used as Insecticides," by Dr. R. C. Roark, will be issued shortly in multigraphed form.

[2] Nagai, *J. Tokyo Chem. Soc.*, **23**, 740 (1902).

[3] Ishikawa, *Tokyo Medical J.*, **31**, No. 4 (1917).

[4] Kariyone and Atsumi, *J. Pharm. Soc.* (Japan), No. 491, 10 (1923); Kariyone, Kimura and Kondo, *ibid.*, No. 514, 1049 (1924); Kariyone and Kondo, *ibid.*, No. 518, 376 (1925).

by fusion with potassium hydroxide. Takei[5] in his first article added some facts to those found by the other Japanese investigators, but most of his results were reinterpreted in another publication[6] in which he finally decided upon the empirical formula $C_{23}H_{22}O_6$, which brought it into agreement with the figures obtained by him and others with respect to the methoxyl content and the analytical figures obtained for the phenylhydrazone, various empirical formulas having been proposed by previous investigators.

Butenandt[7] repeated some of the work of the Japanese chemists and established with certainty the empirical formula finally decided upon by Takei. He also prepared a number of new and important derivatives, but the facts so far obtained are insufficient to establish its constitution.

The investigations which will be described in this article deal principally with results obtained by the direct hydrogenation of rotenone and by the action of metallic zinc in alkaline alcoholic solution.

The simplest reduction product of rotenone, dihydrorotenone, was prepared by Kariyone by hydrogenation with palladium–barium sulfate catalyst and is described as prisms, melting at 216°, $[\alpha]_D^{20}$ −222°, yielding an oxime of melting point 258°.

Butenandt repeated the preparation of dihydrorotenone, according to the directions of Kariyone, confirmed the results of the Japanese investigator with respect to its physical properties and revised the formula to $C_{23}H_{24}O_6$. He employed dihydrorotenone for subsequent reactions to test a theory regarding the formation of his "derritol."

We have made a large number of experiments on the hydrogenation of rotenone with both palladium–barium sulfate and platinum oxide catalysts in acetone and acetic ester solutions, but our results differ widely from those reported by Kariyone and Butenandt.

With the first-mentioned catalyst in acetone solution, absorption of hydrogen takes place until about 56 cc. per gram of rotenone is consumed, when the reaction practically stops. With platinum catalyst, it proceeds rapidly to about the same point, but continues at a much slower rate without any definite end-point. In both cases two compounds are formed, if the reaction is discontinued after about one molecule of hydrogen has been absorbed. Since one product is an acid and the other a neutral compound, the two are easily separated by agitating the ethereal solution of the mixture with dilute sodium hydroxide solution. The acid is precipitated in crystalline form from the alkaline solution by the addition of mineral acid, whereas the neutral compound crystallizes on evaporation of the ethereal solution. Both are easily purified, the

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[5] Takei, *Biochem. Z.*, **157**, 1 (1925).

[6] Takei, *Ber.*, **61**, 1003 (1928).

[7] Butenandt, *Ann.*, **464**, 253 (1928).

acid by crystallization from dilute alcohol, and the neutral compound from 95% alcohol. The combined yields of the two are about equal to the quantity of rotenone reduced. The acid crystallizes in needles and melts at 209°, the neutral substance in prisms melting at 164°. Both have the same empirical formula, $C_{23}H_{24}O_6$.

The optical rotation of the neutral compound was found to be $(\alpha)_D^{20} = 225°$. It yielded an oxime melting at 258°, the same compound that was obtained by hydrogenation of rotenone oxime. There seems to be no doubt that the neutral compound is dihydrorotenone, and except for the melting point, where we have noted a very great discrepancy, our results are in agreement with those found by Kariyone.

Both the acid and dihydrorotenone are capable of further reduction. On long continued reduction with platinum catalyst in acetic ester solution, the acid takes up another molecule of hydrogen, yielding a new acid of the formula $C_{23}H_{26}O_6$, melting point 215°.

Dihydrorotenone is more slowly reduced under the same conditions, and yields as one of the end-products the same acid as does the original acid product, perhaps together with some of the dihydrodesoxyrotenone of Butenandt, in which the keto group is reduced.

From these results, it is apparent that the rotenone molecule is simultaneously attacked in two places on catalytic reduction. One reaction probably involves the simple reduction of an unsaturated bond, yielding dihydrorotenone, the other, the opening of a lactone linkage to form an acid which still contains the original double bond of rotenone. Further reduction of either compound then leads to the same end-product, an acid containing four hydrogen atoms more than rotenone.

Butenandt obtained an acid $C_{23}H_{26}O_6$, melting point 206°, by the reduction of rotenone with platinum catalyst in saturated alcoholic ammonia, which may be identical with the acid described above of the same formula.

The isorotenone described by Takei does not absorb hydrogen as does rotenone, and it seems likely that it contains no unsaturated bond, but a ring structure instead.

Among the derivatives prepared by Butenandt is a phenolic compound of a bright yellow color, containing the original keto group, but possessing two carbon atoms less than rotenone, and named by him "derritol." It is formed with a compound called rotenol in which the keto group is probably reduced to an alcoholic group. These compounds result from the reduction of rotenone with zinc dust in alkaline alcoholic solution.

We have found that by slight modification of this method, either of these compounds may be made the predominating product. Two or more by-products of lower molecular weight are formed in this reaction, and have been isolated in crystalline form.

According to Butenandt's theory, the presence of a double bond is essential to the formation of derritol, since he was unable to obtain a corresponding compound from dihydrorotenone. It seems doubtful, however, that Butenandt was working with dihydrorotenone, for this compound readily gives a derivative of the formula of dihydroderritol as well as dihydrorotenol, when subjected to the alkaline zinc reduction process. The first-mentioned compound is, however, colorless instead of yellow, but it gives the same phenol reaction with ferric chloride as does derritol. Isorotenone, which probably contains no double bond, is also readily converted into isoderritol and isorotenol by reduction with zinc in alkaline solution. It seems probable, therefore, that whatever the mechanism of the reaction leading to the formation of derritol may be, it is not dependent upon the presence of a double bond.

Rotenone.—The preparation of rotenone is so simple as to require no detailed description. Three or four extractions of the coarsely ground, dried root with ether is sufficient to dissolve out all the rotenone, which crystallizes in nearly pure state from the concentrated extract. It is recrystallized from about 20 parts of boiling absolute alcohol. Crystallization takes place so rapidly on cooling that it is necessary to filter on a hot funnel. A little decolorizing carbon is used to obtain a pure white product. Rotenone melts at 163°. The yield of pure product varies with the quality of the *Derris* root. A fairly good material yields 2.5% of recrystallized rotenone, although we have analyzed samples containing less than 0.5%, while some Japanese chemists claim a yield of more than 6%.

Dihydrorotenone.[4,7]—Four grams of rotenone was added to 30 cc. of acetic ester containing about 0.3 g. of platinum oxide catalyst,[8] previously saturated with hydrogen, and the solution was shaken in a hydrogen atmosphere. About 250 cc. is absorbed in three or four minutes, but if sufficient time is allowed, 400 cc. or more will be taken up. The reduction in this case, however, was discontinued after the absorption of about one equivalent of hydrogen. The solution was poured off from the catalyst, diluted with 100 cc. of ether and extracted several times with 5% aqueous potassium hydroxide.

The ether acetic ester solution was dried and evaporated to a colorless sirup which readily crystallized on cooling. The crystalline cake was rubbed up with a few cubic centimeters of alcohol, filtered and dried. The yield was in one experiment 1.9 g.; in another carried out under the same conditions, 1.7 g. The material is easily recrystallized from 95% alcohol, from which it is deposited in prisms often 1 mm. or more in length. The loss on recrystallization is small, and hence the compound is easily purified. After one recrystallization the compound melts at 164°, and further recrystallization does not alter the melting point. The analytical figures agree for the expected formula for dihydrorotenone.

Anal.[9] Subs., 0.0893, 0.0820, 0.0821: CO_2, 0.2285, 0.2100, 0.2107; H_2O, 0.0490,

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[8] R. Adams and R. L. Shriner, THIS JOURNAL, **45**, 2171 (1923).

[9] It is necessary to mix the substance with copper oxide in order to obtain satisfactory results.

0.0448, 0.0465. Subs., 0.0228, 0.0260: AgI, 0.0265, 0.0313. Calcd. for $C_{23}H_{24}O_6$: C, 69.69; H, 6.06; $2OCH_3$, 15.65. Found: C, 69.77, 69.82, 69.98; H, 6.09, 6.07, 6.28; OCH_3, 15.27, 15.90. 0.3153 g. of subs. in 15 cc. of benzene rotated in a 2-dm. tube $-9.46°$; $[\alpha]^{20}_{D} = -225.2°$.

Acid Reduction Product.—The aqueous alkaline extract from the reduction mixture yields a crystalline product on the addition of an excess of hydrochloric acid. The compound is best purified by dissolving in hot alcohol and adding an equal volume of hot water to the solution. The compound crystallizes in needles which melt at 209°.

If the reduction has been carried too far, a mixture of two acids is obtained, which will be referred to later.

The yield is, as in the first case described, 1.7 g., and 1.9 g. in the second. In another experiment, in which 6 g. of rotenone was reduced, 3 g. of acid was obtained, together with 2 g. of pure dihydrorotenone. Similar results were obtained in a number of other runs. The compound may be titrated but the end-point is not sharp.

Anal. Subs., 0.0833, 0.0828, 0.0805: CO_2, 0.2124, 0.2106, 0.02045; H_2O, 0.0462, 0.0446, 0.0455. Subs., 0.0222, 0.0182: AgI, 0.0264, 0.0218. Calcd. for $C_{23}H_{24}O_6$: C, 69.69; H, 6.06; $2OCH_3$, 15.65. Found: C, 69.55, 69.36; 69.28; H, 6.16, 5.99, 6.27; OCH_3, 15.71, 15.82. 0.3154 g. of subs. in 15 cc. of chloroform rotated in a 1-dm. tube $+0.76°$; $[\alpha]^{20}_{D} = +36.2°$; 0.0562 subs. = 1.24 cc. of $N/10$ acid; M, calcd., 396; found, 453.

Reduction of Rotenone with Palladium–Barium Sulfate Catalyst.—One and a half grams of rotenone was reduced in acetone solution with 0.5 g. of palladium–barium sulfate catalyst, according to the directions of Butenandt and Kariyone.

The absorption of hydrogen ceased in about thirty minutes, when 84 cc. had been taken up. This volume of hydrogen corresponds to the theoretical quantity for two atoms per molecule of rotenone. The solvent was evaporated and the residue dissolved in ether, and the two resulting compounds were separated in the manner described above. With palladium–barium sulfate catalyst the dihydrorotenone predominates in the mixture, 1.04 g. being isolated. The same acid was formed as in the cases where platinum was used, the yield amounting to 0.45 g. In another experiment in which 2 g. of rotenone was employed, 1.5 g. of dihydrorotenone was obtained.

Reduction of Dihydrorotenone.—One gram of dihydrorotenone was subjected to long-continued hydrogenation with 0.2 g. of platinum oxide catalyst in acetic ester solution. After about three hours, 20 cc. of hydrogen had been absorbed and the product was worked up as in previous cases. The ethereal solution of the reaction products was extracted with dilute potassium hydroxide, and on acidification yielded 0.08 g. of acid. It was recrystallized from 50% acetic acid. The product crystallizes in needles melting at 215°.

Anal. Subs., 0.0728: CO_2, 0.1854; H_2O, 0.0436. Calcd. for $C_{23}H_{26}O_6$: C, 69.30; H, 6.58. Found: C, 69.44; H, 6.65.

The neutral reduction product was probably mostly dihydrorotenone, but may have contained the dihydrodesoxyrotenone of Butenandt.

Reduction of the Acid Reduction Product of Rotenone.—One-half gram of the acid product described above, as obtained by reduction of rotenone, was subjected to a second reduction with platinum catalyst in acetic ester solution. About 36 cc. of hydrogen was taken up in fifteen minutes. The solution was evaporated and the crystalline product recrystallized from hot 50% alcohol. It crystallized in needles which melted at 215°. A mixture of this product and the acid obtained from dihydrorotenone melted also at 215°.

Anal. Subs., 0.0903: CO_2, 0.2285; H_2O, 0.0524. Calcd. for $C_{23}H_{26}O_6$: C, 69.30; H, 6.58. Found: C, 69.00; H, 6.46.

Apparently the two acids obtained—the one from dihydrorotenone and the other from the first acid reduction product—are identical.

If rotenone is allowed to absorb more than two atoms of hydrogen, a mixture of the partly and fully hydrogenated acids is obtained.

Oxime of Dihydrorotenone.—The oxime of dihydrorotenone is readily obtained either by hydrogenation of rotenone oxime[6] or by the action of hydroxylamine on dihydrorotenone.

Two grams of rotenone oxime in 50 cc. of acetone containing 0.4 g. of platinum catalyst, which had been saturated with hydrogen, absorbed 120 cc. of hydrogen in three to four minutes. The solvent was evaporated and the product recrystallized from absolute alcohol, in which it is difficultly soluble. It crystallizes in long needles and melts with decomposition at 256–257°. The yield is quantitative.

The same compound is also obtained when 1.6 g. of dihydrorotenone, 1.1 g. of hydroxylamine hydrochloride, 1.2 g. of anhydrous sodium acetate and a few drops of acetic acid in 20 cc. of absolute alcohol are boiled for from four to five hours under reflux. The oxime crystallized out on cooling and was recrystallized from absolute alcohol. The yield was 1.2 g. of product, melting at 256° with decomposition. A mixed melting point of the compound with that obtained by reduction of rotenone oxime showed no depression.

Kariyone also records 256° as the melting point of dihydrorotenone oxime.

Anal. Subs., 0.0948: CO_2, 0.2325; H_2O, 0.0512. Subs., 0.0212, 0.0229: AgI, 0.0249, 0.0264. Calcd. for $C_{23}H_{25}O_6N$: C, 67.15; H, 6.08; $2OCH_3$, 15.09. Found: C, 66.88; H, 6.00; OCH_3, 15.50, 15.24.

Benzenesulfonic Acid Derivative of Rotenone Oxime.—Five grams of rotenone oxime prepared by the directions of Takei was dissolved in 20 cc. of dry pyridine and 2 g. of benzenesulfonic acid chloride was slowly added. After about eighteen hours, the solution was poured into 100 cc. of 5% sulfuric acid. The amorphous precipitate crystallized in a few minutes. It was washed with water and recrystallized by dissolving in 30 cc. of hot alcohol and adding 15 cc. of hot water. It was again recrystallized from 90% alcohol and yielded 4 g. of white needles, melting at 143°.

Anal. Subs., 0.0242: AgI, 0.0212. Subs., 0.0225, 0.0230: 1.22, 1.22 cc. of $N/30$ HCl. Subs., 0.1481: $BaSO_4$, 0.0576. Calcd. for $C_{29}H_{26}O_8SN$: $2OCH_3$, 11.29; N, 2.55; S, 5.82. Found: OCH_3, 11.57; N, 2.53, 2.48; S, 5.34.

Numerous attempts were made to effect the Beckmann rearrangement on the compound, without success. Both alkaline and acid reagents decompose the compound in such a manner as to yield only tarry products from which nothing definite could be isolated.

Dihydrorotenone Iso-oxime.—Dihydrorotenone oxime is apparently converted into an isomeric compound by the action of concentrated hydrochloric acid. One gram was allowed to stand in contact with 15 cc. of concentrated hydrochloric acid for three days. It turned yellow, but the color disappeared on dilution with water. The product was recrystallized from absolute alcohol and formed long needles, melting at 270 to 273° with decomposition. The yield was quantitative.

Anal. Subs., 0.0999: CO_2, 0.2453; H_2O, 0.0537. Subs., 0.0225, 0.0208: AgI, 0.0252, 0.0243. Calcd. for $C_{23}H_{25}O_6N$: C, 67.15; H, 6.08; $2OCH_3$, 15.09. Found: C, 66.96; H, 5.96; OCH_3, 14.80, 15.44.

Preparation of Rotenol and Derritol.—In following the directions of Butenandt[7] for the preparation of these derivatives, it was found that while the yield of rotenol was fairly good, the product obtained from the alkaline extract was very difficult to crystallize and the yield of derritol very unsatisfactory. However, if the alcoholic potassium hydroxide solution containing the rotenone was first heated nearly to boiling, and the zinc dust was added subsequently, the yield of derritol was greatly improved. It was also found that by varying the percentage of potassium hydroxide, the quantities of both rotenol and derritol obtained could be varied at will.

Below are listed some typical runs, 3 g. of rotenone being used in each case.

Potassium hydroxide, %	5	10	15	20
Rotenol, grams	2.0	1.1	0.9	0.2
Derritol, grams	0.1	0.65	0.95	1.15

If larger quantities of rotenone were employed, mechanical stirring was necessary to obtain corresponding yields. The melting point of pure derritol was found to be 164°, instead of 161° as recorded by Butenandt, and rotenol melted sharply at 120° instead of 115°. The optical rotation of derritol was measured, with the following result: 0.1700 g. of substance in 5 cc. of $CHCl_3$ rotated in 1-dm. tube $-2.25°$; $[\alpha]_D^{20} = -66.2°$.

On concentrating the derritol mother liquors, a small quantity of another crystalline substance melting at 161° was obtained. The methoxyl content of this substance was about 1% higher than that of derritol.

Anal. Subs., 0.0207, 0.0213: AgI, 0.0279, 0.0283. Found: CH_3O, 17.80, 17.55.

Since the yield was small, no further work was done on this compound.

The mother liquors of rotenol contain traces of an oil with a terpene-like odor; this oil is volatile with steam and can be isolated from the distillate by ether extraction. It has not yet been identified.

Preparation of Dihydrorotenol and Dihydroderritol.—The dihydrorotenol and dihydroderritol were prepared in the same manner as described for the preparation of rotenol and derritol. With 15% potassium hydroxide and 3 g. of dihydrorotenone, the yield of the alkali-insoluble compound (dihydrorotenol) was 0.8 g. It was recrystallized from methyl alcohol and melted at 131°. This is in agreement with the melting point of dihydrorotenol obtained by Butenandt. The yield of the alkali-soluble fraction was 0.5 g. Recrystallized from dilute methyl alcohol, it formed white needles, melting at 122°. It gives a strong color test for phenol with ferric chloride and the analysis indicates that it is dihydroderritol.

Anal. Subs., 0.0635, 0.0623: CO_2, 0.1576, 0.1550; H_2O, 0.0360, 0.0365. Subs., 0.0233: AgI, 0.0295. Calcd. for $C_{21}H_{24}O_6$: C, 67.74; H, 6.45; $2OCH_3$, 16.66. Found: C, 67.68, 67.84; H, 6.30, 6.51; OCH_3, 16.72.

Preparation of Isorotenol and Isoderritol.—The isorotenone used in the preparation of isorotenol and isoderritol was prepared by the method of Takei.[6] The product so obtained had a melting point of 184°. This figure is about 7° higher than that recorded by Takei, but confirms the figure found by Wright.[10] However, since the analytical data of both Takei and Wright for carbon and hydrogen were at some variance from the theoretical, the carbon and hydrogen determination was repeated on our product.

Anal. Subs., 0.0907, 0.0906: CO_2, 0.2324, 0.2320; H_2O, 0.0460, 0.0447. Calcd. for $C_{23}H_{22}O_6$: C, 70.05; H, 5.59. Found: C, 69.88; 69.84; H, 5.63, 5.48.

Isorotenone was treated with 15% potassium hydroxide and zinc in the same manner that has been described for the preparation of rotenol and derritol. From 3.0 g. of isorotenone, 0.9 g. of the product (isorotenol) insoluble in alkali was obtained. It crystallized from methyl alcohol in white needles, melting at 133°.

Anal. Subs., 0.0653, 0.0670: CO_2, 0.1611, 0.1711; H_2O, 0.0371, 0.0363. Subs., 0.0202: AgI, 0.0240. Calcd. for $C_{23}H_{24}O_6$: C, 69.67; H, 6.10, $2OCH_3$, 15.65. Found: C, 69.35, 69.63; H, 6.31, 6.03; OCH_3, 15.69.

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[10] Wright, THIS JOURNAL, **50**, 3355 (1928).

The alkali-soluble portion gave 0.9 g. of a compound (isoderritol) which crystallized from methyl alcohol in light yellow plates, melting at 150°.

Anal. Subs., 0.0728, 0.0773: CO_2, 0.1814, 0.1925; H_2O, 0.0381, 0.0409. Subs., 0.0209: AgI, 0.0268. Calcd. for $C_{21}H_{22}O_6$: C, 68.07; H, 5.99; $2OCH_3$, 16.75. Found: C, 67.93, 68.00; H, 5.81, 5.88; OCH_3, 16.93.

Summary

Hydrogenation of rotenone results in the formation of a neutral and an acid compound as primary products.

The neutral compound is dihydrorotenone and is formed by the simple reduction of an unsaturated bond. Dihydrorotenone melts at 164°, instead of 216° as recorded by Kariyone.

The acid is easily separated from the reaction mixture and has the same empirical formula as dihydrorotenone, $C_{23}H_{24}O_6$.

On further reduction, both compounds yield the same end-product, an acid containing four more hydrogen atoms than rotenone. It seems likely that the molecule of rotenone is first simultaneously reduced in two places, while further hydrogenation results in the acid, $C_{23}H_{26}O_6$.

Contrary to Butenandt, dihydrorotenone is converted into dihydroderritol and dihydrorotenol by treatment with zinc in alkaline alcoholic solution.

Isorotenone yields isoderritol and isorotenol by the same treatment.

From these results, it follows that the formation of derritol is independent of the presence of an unsaturated bond in rotenone.

The preparation and properties of some other rotenone derivatives are described.

WASHINGTON, D. C.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE COLLEGE OF LIBERAL ARTS OF NORTHWESTERN UNIVERSITY]

SYNTHESIS IN THE DIPHENYL ETHER SERIES. I. PREPARATION OF SOME SIMPLE DERIVATIVES[1]

BY C. M. SUTER

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Since diphenyl ether is now readily available, a further study of its derivatives and their preparation from the parent substance seems desirable. Most of such derivatives previously prepared have been made by the reaction between the salt of a phenol and an aromatic halogen compound.[2]

The preparation of *o*- and *p*-nitrodiphenyl ether by the nitration of

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[1] Presented before the Division of Organic Chemistry at the Columbus meeting of the American Chemical Society, May, 1929.

[2] See Raiford and Colbert, THIS JOURNAL, **48,** 2652 (1926), for recent work and bibliography.