[CONTRIBUTION FROM THE COLLEGE OF PHARMACY, UNIVERSITY OF MICHIGAN]

Tertiary Arsines and Arsine Oxides[1]

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Since it has been shown[3] that triphenylarsine oxide reacts with phenylmagnesium bromide to form tetraphenylarsonium bromide, it seemed desirable to study the interaction of other tertiary arsine oxides with various Grignard reagents. In this paper we have described the preparation of a number of new tertiary arsines and arsine oxides. The reactions of some of these arsine oxides with Grignard reagents will be reported later.

Triphenylarsine has been obtained by three new procedures, namely, by interaction of bromobenzene and sodium amalgam with each of the three following compounds—phenylarsine oxide, tetraphenylarsyl oxide and tetraphenyldiarsyl. Presumably the mechanism of these reactions is expressed by formulations A–F.

A $\mathrm{2C_6H_5AsO + 4Na = 2C_6H_5As(ONa)Na}$

B $\mathrm{2C_6H_5As(ONa)Na + 2C_6H_5Br = 2(C_6H_5)_2As(ONa) + 2NaBr}$

C $\mathrm{2(C_6H_5)_2As(ONa) = (C_6H_5)_2AsOAs(C_6H_5)_2 + Na_2O}$

D $\mathrm{(C_6H_5)_2AsOAs(C_6H_5)_2 + 2Na = (C_6H_5)_2AsNa + (C_6H_5)_2As(ONa)}$

E $\mathrm{(C_6H_5)_2AsNa + C_6H_5Br = (C_6H_5)_3As + NaBr}$

F $\mathrm{(C_6H_5)_2AsAs(C_6H_5)_2 + 2Na = 2(C_6H_5)_2AsNa}$

If tetraphenylarsyl oxide reacts with sodium according to D, then, upon exposure of the reaction mixture to air, the diphenylarsyl sodium, $\mathrm{(C_6H_5)_2AsNa}$, should be oxidized to the sodium salt of diphenylarsinic acid. This was found to be the case and both diphenylarsinic acid and tetraphenylarsyl oxide were obtained in good yield in accordance with the following equation

$$
\begin{array}{lll}
\mathrm{D} & \multicolumn{2}{l}{\mathrm{2(C_6H_5)_2AsOAs(C_6H_5)_2 + 4Na =}} \\
 & \mathrm{2(C_6H_5)_2AsNa} \quad + & \mathrm{2(C_6H_5)_2AsONa} \\
 & \text{Oxygen}\downarrow & \text{Water}\downarrow \\
 & \mathrm{2(C_6H_5)_2As(O)ONa} & \mathrm{(C_6H_5)_2AsOAs(C_6H_5)_2 + 2NaOH}
\end{array}
$$

Nitration of triphenylarsine oxide or dihydroxide yielded tri-(3-nitrophenyl)-arsine oxide which, upon reduction with hypophosphorous acid, was converted into tri-(3-nitrophenyl)-arsine (m. p. 206–207°). Michaelis[4] treated tri-(3-nitrophenyl)-arsine oxide (m. p. 254°) with phosphorous acid and claimed that he obtained tri-(3-nitrophenyl)-arsine (m. p. 250°). It is quite evident that his "arsine" was merely unreduced arsine oxide.

Reduction of the nitroarsine oxide with stannous chloride yielded tri-(3-aminophenyl)-arsine.

The last mentioned investigator[5] stated that he prepared 3-amino-phenyldi-(3-nitrophenyl)-arsine (m. p. 205°) by reduction of tri-(3-nitrophenyl)-arsine oxide with hydrogen sulfide. We found that his compound is actually tri-(3-nitrophenyl)-arsine (m. p. 206–207°).

Tri-(3-hydroxyphenyl)-arsine was obtained by diazotization of tri-(3-aminophenyl)-arsine oxide and subsequent reduction of the tri-(3-hydroxyphenyl)-arsine oxide produced with hydriodic and hypophosphorous acids.

The tri-(3-methoxyphenyl)-arsine, obtained by methylation of tri-(3-hydroxyphenyl)-arsine with dimethyl sulfate, proved to be identical with the compound prepared from 3-anisylmagnesium iodide and arsenic trichloride.

The contention of Ehrlich and Bertheim[6] that 4-aminophenylarsine oxide, when heated with hydrochloric acid, yields tri-(4-aminophenyl)-arsine is based merely on an analysis of the reaction product. By means of the series of reactions shown below we have demonstrated that the product of Ehrlich and Bertheim really is tri-(4-aminophenyl)-arsine.

$$
\begin{array}{l}
\mathrm{(4)\ H_2NC_6H_4AsO \longrightarrow (H_2NC_6H_4)_3As \longrightarrow} \\
\mathrm{(CH_3CONHC_6H_4)_3As \longrightarrow (CH_3CONHC_6H_4)_3AsO \longrightarrow} \\
\qquad\qquad \mathrm{(H_2NC_6H_4)_3AsO \longrightarrow (HOC_6H_4)_3AsO \longrightarrow} \\
\qquad\qquad\qquad\qquad\qquad \mathrm{(CH_3OC_6H_4)_3As(OH)_2} \\
\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad \uparrow \\
\mathrm{AsCl_3 + (4)\ CH_3OC_6H_4MgI \longrightarrow (CH_3OC_6H_4)_3As} \mathrel{-\!-\!-\!-\!\lrcorner}
\end{array}
$$

Experimental Part

Triphenylarsine. (a) From Bromobenzene, Sodium Amalgam and Phenylarsine Oxide.—A mixture of 6.7 g. of phenylarsine oxide, 100 cc. of benzene, 4 cc. of 40% sodium amalgam and 9 cc. of bromobenzene was shaken for twelve hours in a stoppered bottle, the material filtered and the solvent removed from the filtrate. The residue was subjected to steam distillation, the gummy product dried and dissolved in absolute ether. The arsine was precipitated as $\mathrm{(C_6H_5)_3As{\cdot}HgCl_2}$ by the addition of mercuric chloride dissolved in ether; yield 4.4 g.; m. p. 243–245°.

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[3] Blicke and Monroe, THIS JOURNAL, **57**, 720 (1935).

[4] Michaelis, *Ann.*, **321**, 180 (1902).

[5] Michaelis, *ibid.*, **321**, 185 (1902).

[6] Ehrlich and Bertheim, *Ber.*, **43**, 919 (1910).

(b) **From Bromobenzene, Sodium Amalgam and Tetraphenylarsyl Oxide.**—After a mixture of 4.7 g. of tetraphenylarsyl oxide, 100 cc. of benzene, 3 cc. of 40% sodium amalgam and 18 cc. of bromobenzene had been shaken for twelve hours the solution was decanted, the benzene and bromobenzene removed by steam distillation, the residue dissolved in ether and the arsine precipitated with mercuric chloride; the addition product weighed 2.9 g.; m. p. 244–245°.

(c) **From Bromobenzene, Sodium Amalgam and Tetraphenyldiarsyl.**[7]—A mixture of 3.9 g. of tetraphenyldiarsyl, 50 cc. of benzene and 1.7 g. of 40% sodium amalgam was shaken for twelve hours in an atmosphere of nitrogen; 10 cc. of bromobenzene was then added and the mixture shaken again for twelve hours. The material was treated as described whereupon 7.9 g. of mercuric chloride addition product was obtained; m. p. 244–245°.

Tetraphenylarsyl Oxide and Sodium Amalgam.—A solution of 2.37 g. of tetraphenylarsyl oxide in 100 cc. of benzene was shaken with 0.6 cc. of 40% sodium amalgam in an atmosphere of nitrogen for twelve hours, the solvent allowed to evaporate, the residue treated with water and filtered. The undissolved material consisted of mercury and tetraphenylarsyl oxide. The latter weighed 0.90 g.; calcd. yield 1.18 g.; m. p. 96–97° after recrystallization from petroleum ether. Upon acidification of the filtrate 0.90 g. of diphenylarsinic acid precipitated; calcd. yield 1.31 g.; the crude acid melted at 168–170°.

4-Bromodiphenylbromoarsine.—Ten grams of 4-bromodiphenylarsinic acid,[8] 20 cc. of hydrobromic acid and three drops of hydriodic acid were treated with sulfur dioxide. The oily product was washed with water and sodium acid sulfite solution, mixed with a few cubic centimeters of acetic acid and cooled with solid carbon dioxide. The crystalline bromide weighed 11 g.; m. p. 45–46° after recrystallization from acetic acid. It is soluble in ether and benzene.

Anal. Calcd. for $C_{12}H_9AsBr_2$: As, 19.32. Found: As, 19.27.

Tri-(3-nitrophenyl)-arsine.—A mixture of 13.7 g. of tri-(3-nitrophenyl)-arsine oxide, 85 cc. of acetic acid, 15 cc. of 50% hypophosphorous acid and two drops of hydriodic acid was heated at 65–70° for four hours, the liquid decanted from the precipitate and the product washed with acetic acid, then with alcohol and recrystallized from pyridine. The yellow needles, which weighed 11.5 g., are only slightly soluble in the common organic solvents; m. p. 206–207° after recrystallization from pyridine.

Anal. Calcd. for $C_{18}H_{12}O_6N_3As$: As, 16.99. Found: As, 16.88.

When oxidized with potassium permanganate the compound is converted into the arsine oxide; m. p. 255–256°[9] after recrystallization from pyridine.

Tri-(3-aminophenyl)-arsine.[10]—To 8.8 g. of the nitroarsine oxide, dissolved in 80 cc. of acetic acid, there was added 50 g. of stannous chloride. A rapid stream of dry hydrogen chloride was passed into the mixture whereupon the tin salt dissolved and a crystalline precipitate formed rapidly. After twelve hours the product was filtered on a Jena filter, dissolved in water and treated with 10% sodium hydroxide solution until the mixture was neutral; 75 cc. more of sodium hydroxide solution was added and the liquid decanted from the gummy amine. After the latter had become granular it was washed with water; yield 5 g.; m. p. 178–179° after recrystallization from xylene. The compound is colorless, stable in air, and is soluble in dilute mineral acids.

Anal. Calcd. for $C_{18}H_{12}N_3As$: As, 21.34. Found: As, 21.15.

Tri-(3-acetylaminophenyl)-arsine was obtained in quantitative yield when 2.1 g. of the arsine was boiled for a few minutes with 6 cc. of acetic anhydride.

Tri-(3-acetylaminophenyl)-arsine Dihydroxide and Tri-(3-aminophenyl)-arsine Oxide.—Twenty-two grams of tri-(3-acetylaminophenyl)-arsine was suspended in a mixture of 700 cc. of water and 200 cc. of acetone, stirred vigorously and an aqueous solution of potassium permanganate added until the pink color persisted for an hour. The filtered solution was concentrated under reduced pressure whereupon 15.5 g. of tri-(3-acetylaminophenyl)-arsine dihydroxide separated in glistening, white crystals. Upon extraction of the manganese dioxide precipitated with 500 cc. of warm alcohol 3 g. more of the product was obtained. The dihydroxide was recrystallized from dilute acetic acid; m. p. above 300°.

Anal. Calcd. for $C_{24}H_{26}O_5N_3As$: As, 14.66. Found: As, 14.28.

The acetyl derivative was hydrolyzed by heating 3 g. of the latter with 45 cc. of alcohol, 30 cc. of water and 9 g. of sodium hydroxide for seven hours. Upon removal of the alcohol 2 g. of crystalline tri-(3-aminophenyl)-arsine oxide was obtained. After recrystallization from dilute alcohol the oxide melted at 272°.

Anal. Calcd. for $C_{18}H_{18}ON_3As$: As, 20.41. Found: As, 20.26.

The oxide was reduced to the arsine when 1.8 g. of the oxide, 2.5 cc. of 50% hypophosphorous acid, two drops of hydriodic acid and 50 cc. of alcohol were refluxed for four hours. The alcohol was removed and the arsine precipitated by the addition of alkali; yield 1.5 g.; mixed m. p. 178–179°.

Tri-(3-hydroxyphenyl)-arsine Oxide.—A mixture of 3.6 g. of tri-(3-aminophenyl)-arsine oxide, 50 cc. of water, 15 cc. of sulfuric acid and 50 g. of ice was treated with 2.1 g. of sodium nitrite. After twelve hours it was filtered, heated to 75–80° for one hour, cooled and the gummy precipitate dissolved in 100 cc. of dilute sodium hydroxide solution, the solution partially neutralized with hydrochloric acid, treated with charcoal and then made acid to litmus. The precipitated oxide weighed 2.2 g. It was dissolved in the least possible amount of methyl alcohol and water added until the solution was slightly turbid. After twelve hours the oxide separated in glistening white needles; m. p. above 300°.

Anal. Calcd. for $C_{18}H_{15}O_4As$: As, 20.25. Found: As, 20.38.

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(7) By the action of bromobenzene on the product formed when tetraphenyldiarsyl was shaken with sodium–potassium alloy, Blicke, Patelski and Powers [This Journal, **55**, 1160 (1933)] obtained a small amount of triphenylarsine which was isolated as methyltriphenylarsonium iodide.

(8) Blicke and Webster, This Journal, **59**, 534 (1937).

(9) Prepared by a different method Michaelis [*Ann.*, **321**, 180 (1902)] reported the melting point to be 254°.

(10) Philips [*Ber.*, **19**, 1034 (1886)] and Michaelis [*Ann.*, 321, 213 (1902)] obtained this compound in an impure state.

TABLE I

ARSINES

	Arsine	Prepared[a] from	M. p., °C.	% Yield	Formula	% As Calcd.	% As Found	HgCl₂ addition product m. p., °C.
1	Methyldiphenyl	Diphenylbromoarsine + methylmagnesium iodide	(B. p. 186–188/21 mm.)[b]	..	....	...	...	188–189
2	Cyclohexyldiphenyl	Diphenylbromoarsine + cyclohexylmagnesium bromide	(B. p. 200–203/4 mm.)	80	$C_{18}H_{21}As$	24.01	23.60	233–234
3	4-Bromotriphenyl	4-Bromodiphenylbromoarsine + phenylmagnesium bromide	64–65	98	$C_{18}H_{14}BrAs$	19.46	19.47	175–176
4	Phenyldi-α-naphthyl	Phenyldichloroarsine + α-naphthylmagnesium bromide	205–206	71	$C_{26}H_{19}As$	18.45	18.18	.....
5	Diphenyl-α-naphthyl	Diphenylbromoarsine + α-naphthylmagnesium bromide	110–111	70	$C_{22}H_{17}As$	21.04	21.07	235–236
6	Diphenyl-β-naphthyl	Diphenylbromoarsine + β-naphthylmagnesium bromide	90–91	70	$C_{22}H_{17}As$	21.04	20.84	200–201
7	1-Phenyl-*o,o′*-diphenylyl	*o,o′*-Diphenylylchloroarsine[c] + phenylmagnesium bromide	87–88	90	$C_{18}H_{13}As$	24.64	24.59	204–205
8	1-(4-Methoxyphenyl)-*o,o′*-diphenylyl	*o,o′*-Diphenylylchloroarsine + 4-anisylmagnesium iodide	115–116	78	$C_{19}H_{15}OAs$	20.43	20.03	196–197
9	1-(α-Naphthyl)-*o,o′*-diphenylyl	*o,o′*-Diphenylylchloroarsine + α-naphthylmagnesium bromide	150–151	80	$C_{22}H_{15}As$	21.16	20.86	.....
10	6-Methylphenox	6-Chlorophenoxarsine + methylmagnesium iodide	(B. p. 192–195/16 mm.)[d]	90	....	...	...	.....
11	6-Phenylphenox	6-Iodophenoxarsine + phenylmagnesium bromide	108–109[e]	83	....	...	...	201–202
12	6-α-Naphthylphenox	6-Chlorophenoxarsine + α-naphthylmagnesium bromide	137–138	75	$C_{22}H_{15}OAs$	20.25	20.02	.....

Compounds 3, 5 and 12 were recrystallized from acetic acid, 7, 8 and 11 from ethyl alcohol, 6 from methyl alcohol and 4 and 9 from a mixture of benzene and alcohol. The mercuric chloride addition products precipitated when mercuric chloride, dissolved in absolute ether, was added to the arsine, dissolved in the same solvent. The addition products were all recrystallized from alcohol except that derived from methyldiphenylarsine which was recrystallized from acetic acid.

[a] The halo arsine, dissolved in benzene, was heated with 1.5 molecular equivalents of the Grignard reagent for three hours, the mixture treated in the usual manner, the ether–benzene layer washed with dilute sodium hydroxide solution, dried and the solvents removed. The oily products crystallized when cooled and rubbed with a few drops of methyl alcohol or benzene. [b] Prepared by different methods Michaelis and Link [*Ann.*, **207**, 199 (1881)] reported the boiling point as 306° while Burrows and Turner [*J. Chem. Soc.*, **117**, 1381 (1920)] stated that the boiling point is 163–170° under 15 mm. pressure. [c] Blicke, Weinkauff and Hargreaves, THIS JOURNAL, **52**, 781 (1930). [d] Our procedure is a variation of that used by Aeschlimann [*J. Chem. Soc.*, 414 (1927)]; he reported the boiling point to be 185° under 20 mm. pressure and 198–200° under 40 mm. pressure. [e] Aeschlimann (*ibid.*, p. 415), who prepared the compound from 6-chlorophenoxarsine, reported the melting point to be 107°.

Tri-(3-hydroxyphenyl)-arsine.—A mixture of 2.8 g. of the arsine oxide, 3.8 cc. of 50% hypophosphorous acid, two drops of hydriodic acid and 50 cc. of alcohol was heated for four hours, cooled and 50 cc. of water added. The precipitated oily arsine soon crystallized; yield 2.4 g.; m. p. 187–188° after recrystallization from 30% alcohol.

The arsine is soluble in alcohol, ether, acetic acid and dilute sodium hydroxide solution, insoluble in benzene and chloroform.

Anal. Calcd. for $C_{18}H_{15}O_3As$: As, 21.16. Found: As, 21.06.

Tri-(3-methoxyphenyl)-arsine.—(a) A solution of 7 g. of arsenic trichloride in 20 cc. of benzene was added, dropwise, to methylmagnesium iodide prepared from 35 g. of 3-iodoanisole, 3.6 g. of magnesium, 80 cc. of ether and 20 cc. of benzene, the mixture heated for one hour and decomposed in the usual manner. The solvents were removed, the oily arsine subjected to steam distillation, dissolved in the least possible amount of warm alcohol, treated with charcoal and filtered. The methoxyarsine which separated weighed 3.6 g.; m. p. 112–113°.

Anal. Calcd. for $C_{21}H_{21}O_3As$: As, 18.92. Found: As, 18.63.

(b) A solution of 1.8 g. of tri-(3-hydroxyphenyl)-arsine in 15 cc. of 10% sodium hydroxide solution was shaken with 1.5 cc. of dimethyl sulfate. The methylation process was repeated three times. The precipitated methoxyarsine weighed 1.3 g.

The mercuric chloride addition product melted at 177–178° after recrystallization from acetic acid.

Tri-(4-methoxyphenyl)-arsine.[11]—This arsine was prepared in the manner described above from 4-anisylmagnesium iodide and arsenic trichloride. In order to purify the compound it was dissolved in benzene and precipitated by the addition of alcohol; m. p. 157–159°;[12] yield 7.7 g.

Tri-(4-methoxyphenyl)-arsine Dihydroxide.—An excess of diazomethane, in ether solution, was added to 0.5 g. of tri-(4-hydroxyphenyl)-arsine oxide, described below, dissolved in methyl alcohol. After twenty-four hours the solvents were removed and the residue, which crystallized

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(11) Prepared by J. F. Oneto.

(12) Michaelis and Weitz [*Ber.*, **20**, 49 (1887)] who used a different method reported the melting point to be 156°.

TABLE II

ARSINE DIHYDROXIDES AND ARSINE OXIDES

	Dihydroxide	M. p., °C.	Formula	%As Calcd.	%As Found
1	4-Bromotriphenylarsine	180–181	$C_{18}H_{16}O_2BrAs$	17.88	18.02
2	Phenyldi-α-naphthylarsine	242–243	$C_{26}H_{21}O_2As$	17.03	17.09
3	Diphenyl-α-naphthylarsine	190–191	$C_{22}H_{19}O_2As$	19.21	19.24
4	1-Phenyl-*o,o′*-diphenylylarsine	107–108	....	...	...
5	1-α-Naphthyl-*o,o′*-diphenylylarsine	212–213	$C_{22}H_{17}O_2As$	19.31	19.27
6	6-Phenylphenoxarsine	92–93[a]	....	...	...
7	6-α-Naphthylphenoxarsine	*191–192*	$C_{22}H_{17}O_3As$	*18.64*	*18.98*
8	Tri-(4-methoxyphenyl)-arsine[b] oxide	92–94	$C_{21}H_{23}O_5As$	17.02	17.40
9	Methyldiphenylarsine	154–155[c]	....	...	...
10	6-Methylphenoxarsine	149–151[d]	$C_{13}H_{11}O_2As$	27.35	27.47
11	Cyclohexyldiphenylarsine	206–207	$C_{18}H_{21}OAs$	22.84	22.92
12	1-Phenyl-*o,o′*-diphenylylarsine	178–179	$C_{18}H_{13}OAs$	23.42	23.47
13	6-Phenylphenoxarsine	188–189[e]	....	...	...

[a] Aeschlimann [*J. Chem. Soc.*, 415 (1927)] who used different procedures reported the melting point to be 93°. We found that the compound was not hygroscopic. [b] Prepared by J. F. Oneto. [c] Burrows [*J. Proc. Roy. Soc. N. S. Wales*, **68**, 77 (1934)] used a different method and stated that the compound was very hygroscopic, water soluble and melted at 142°. Our material was not hygroscopic. [d] Although Aeschlimann[a] prepared this substance he reported no melting point or analysis. [e] Aeschlimann[a] stated that the oxide melts at 184°.

slowly, dissolved in benzene, treated with charcoal and filtered. Upon the addition of petroleum ether (30–60°) the dihydroxide separated slowly in the form of colorless needles; m. p. 92–93°. Tri-(4-methoxyphenyl)-arsine, obtained from 4-anisylmagnesium iodide, was oxidized and the product mixed with the dihydroxide just described; mixed m. p. 92–94°.

To prepare the compounds listed above the arsine was dissolved in acetone, stirred, and potassium permanganate, dissolved in the same solvent, added until the pink color remained permanent for one hour; excess permanganate was destroyed by alcohol, the mixture filtered and the solvent removed.

Based on analytical data some of the oxidation products seem to be arsine dihydroxides while others are oxides.

The arsine dihydroxides are insoluble in cold water, somewhat soluble in hot water, soluble in benzene and alcohol and insoluble in ether and petroleum ether. The oxides are soluble in alcohol. Triphenylarsine oxide and phenylphenoxarsine are partially converted into dihydroxides when heated with water. None of the oxides are hygroscopic.

Compounds 1, 2, 3, 8, 9, and 11 were recrystallized from a mixture of benzene and petroleum ether, compound 4 from water, compound 10 from benzene and compounds 5, 6 and 7 from dilute alcohol.

Tri-(4-aminophenyl)-arsine Oxide.[13]—Two grams of tri-(4-acetylaminophenyl)-arsine was oxidized with potassium permanganate and the crude oxide refluxed five hours with 30 cc. of alcohol, 20 cc. of water and 6 g. of sodium hydroxide. The mixture was filtered; 0.3 g. of nearly colorless arsine oxide separated from the filtrate and 0.2 g. of oxide was obtained by extraction of the residue with warm alcohol; m. p. above 300°.

Anal. Calcd. for $C_{18}H_{18}ON_3As$: As, 20.41. Found: As, 20.57.

Tri-(4-hydroxyphenyl)-arsine Oxide.—Four grams of tri-(4-aminophenyl)-arsine oxide, 70 cc. of water, 17 cc. of sulfuric acid and 50 g. of ice was treated with 2.3 g. of sodium nitrite. After twelve hours the mixture was filtered, heated at 80–85° for one hour and cooled; 4.0 g. of the hydroxy oxide separated; m. p. 276–278° with decomposition.

Anal. Calcd. for $C_{18}H_{15}O_4As$: As, 20.25. Found: As, 20.10.

Summary

The preparation of a number of new tertiary arsines, arsine dihydroxides and arsine oxides has been described. Several arsines described incorrectly in the literature have been characterized properly.

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[13] Morgan and Micklethwait [*J. Chem. Soc.*, **95**, 1474 (1909)] claimed to have obtained this oxide from aniline and arsenic trichloride.