Summary

It has been shown that tetraphenyldiarsyl reacts readily with the following substances: sulfur, sulfur chloride, thionyl chloride, bromocyanogen, phenylmagnesium bromide, arsenic trichloride, phenylarsine oxychloride, phosphorus trichloride and sodium-potassium alloy.

Compounds which contain reactive halogen attached to carbon, such as acetyl chloride, benzoyl chloride, benzophenone chloride, 2,4-dinitrobromobenzene and tetraphenyldichloroethane, as well as ethylene oxide, do not react with the diarsyl, in benzene solution, at ordinary temperature.

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A Study of Phenyl- and Diphenylarsine

BY F. F. BLICKE AND L. D. POWERS

Phenylarsine, dissolved in bromobenzene, absorbs oxygen very rapidly at first; the absorption then proceeds at a slower rate until the material has absorbed approximately one molecular equivalent of the gas. When the arsine was exposed to air we obtained the same oxidation products mentioned by Dehn[1] but the relative amounts varied in different experiments. Phenylarsine and sulfur react to form phenylarsine sulfide and hydrogen sulfide.

It has been reported[2] that phenylarsine reacts with iodine, dissolved in potassium iodide solution, to yield phenylarsonic acid and phenyldiiodoarsine. It has been shown that two molecular equivalents of this arsine, dissolved in ether, reacts instantly with four molecular equivalents of iodine to form phenyldiiododiarsine in quantitative yield. However, when only two molecular equivalents of iodine are used arsenobenzene is formed and by the use of three equivalents diphenyldiiododiarsyl is obtained.

Equimolecular quantities of phenylarsine and triphenylarsine dichloride react in accordance with the following equation

$$2C_6H_5AsH_2 + 2(C_6H_5)_3AsCl_2 = 2(C_6H_5)_3As + C_6H_5As{=}AsC_6H_5 + 4HCl$$

Dehn and Wilcox[3] reported that diphenylarsine, when allowed to oxidize in air, was converted almost completely into diphenylarsinic acid; a very small amount of tetraphenylarsyl oxide was also obtained. We found that diphenylarsine, dissolved in bromobenzene, absorbs oxygen as rapidly as a solution of triphenylmethyl or tetraphenyldiarsyl. The quantity of oxygen absorbed was approximately that required for the interaction of the arsine

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(1) Dehn, *Am. Chem. J.*, **33**, 149 (1905).

(2) Dehn, *ibid.*, **33**, 151 (1905); Fleury, *Bull. soc. chim.*, [4] **27**, 490 (1920).

(3) Dehn and Wilcox, *Am. Chem. J.*, **35**, 47 (1906).

with two molecular equivalents of the gas. Diphenylarsinic acid was isolated as an oxidation product in 93% yield.

It might be expected that the initial product of oxidation would be the unstable diphenylhydroxyarsine, $(C_6H_5)_2As(OH)$, which would decompose spontaneously into tetraphenylarsyl oxide. Diphenylarsinic acid might then be formed as the result of further oxidation of this substance. However, this explanation of the formation of diphenylarsinic acid cannot be correct since tetraphenylarsyl oxide, dissolved in bromobenzene, remains unchanged after long contact with oxygen; hence it seems that the acid is not produced through the intermediate formation of the oxide.[4]

It has been stated by Dehn and Wilcox[5] that diphenylarsine, dissolved in ether, reacts with iodine to form diphenyliodoarsine. It has been shown that the latter compound is obtained from equimolecular amounts of the arsine and iodine; however, if one molecular equivalent of iodine is added to two equivalents of the arsine, tetraphenyldiarsyl is produced; the diarsyl may have been formed as the result of the action of diphenyliodoarsine on diphenylarsine.[6]

Since tetraphenyldiarsyl and iodine react instantly[7] to form diphenyliodoarsine, the mechanism of the formation of diphenyliodoarsine from diphenylarsine and iodine is uncertain; the iodoarsine might be produced by direct replacement of the arsine hydrogen by iodine or as the result of the action of iodine on tetraphenyldiarsyl.

Diphenylarsine and tetraphenylarsyl oxide react in alcohol to form tetraphenyldiarsyl in excellent yield. The arsine and tetraphenylarsyl sulfide react in an analogous manner.

It was found that the chlorine as well as the ethoxy group in phenylchloroethoxyarsine was removed when the latter reacted with diphenylarsine; diphenylchloroarsine and arsenobenzene were isolated as reaction products.

$$2(C_6H_5)_2AsH + 2C_6H_5AsCl(OC_2H_5) = 2(C_6H_5)_2AsCl + C_6H_5As{=}AsC_6H_5 + 2C_2H_5OH$$

When the arsine was allowed to react with triphenylbromomethane in a molecular equivalent amount, diphenylbromoarsine and triphenylmethane were produced.

With triphenylmethyl the arsine yielded triphenylmethane and tetraphenyldiarsyl.

It was found that interaction of triphenylarsine dichloride with two molecular equivalents of diphenylarsine yields triphenylarsine and tetraphenyldiarsyl (A), while equimolecular amounts of the two compounds react to produce triphenylarsine and diphenylchloroarsine (C). It is possible in the latter instance that tetraphenyldiarsyl is formed as an intermediate

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[4] Phenylarsine oxide, likewise, dissolved in bromobenzene does not absorb oxygen.

[5] Ref. 3, p. 45.

[6] Blicke and Powers, THIS JOURNAL, **54**, 3358 (1932).

[7] Blicke and Smith, *ibid.*, **51**, 2276 (1929).

$$\begin{array}{ll} \mathrm{A} & (C_6H_5)_3AsCl_2 + 2(C_6H_5)_2AsH = (C_6H_5)_3As + (C_6H_5)_2As\text{—}As(C_6H_5)_2 + 2HCl \\ \mathrm{B} & (C_6H_5)_3AsCl_2 + (C_6H_5)_2As\text{—}As(C_6H_5)_2 = (C_6H_5)_3As + 2(C_6H_5)_2AsCl \\ \hline \mathrm{C} & 2(C_6H_5)_3AsCl_2 + 2(C_6H_5)_2AsH = 2(C_6H_5)_3As + 2(C_6H_5)_2AsCl + 2HCl \end{array}$$

product and that this substance then reacts with triphenylarsine dichloride according to formulation B. At least we have shown in a separate experiment that the arsine dichloride and the diarsyl react to yield triphenylarsine and diphenylchloroarsine.

Equimolecular amounts of diphenylarsine and triphenylarsine hydroxychloride react, undoubtedly, in accordance with formulation F. A separation of the compounds formed, however, was not found possible until the

$$\begin{array}{ll} \mathrm{D} & 4(C_6H_5)_2AsH + 2(C_6H_5)_3As(OH)Cl = 2(C_6H_5)_3As + 2(C_6H_5)_2As\text{—}As(C_6H_5)_2 + \\ & \qquad 2HCl + 2H_2O \\ \mathrm{E} & 2(C_6H_5)_2As\text{—}As(C_6H_5)_2 + 2(C_6H_5)_3As(OH)Cl = (C_6H_5)_2As\text{—}O\text{—}As(C_6H_5)_2 + \\ & \qquad 2(C_6H_5)_2AsCl + 2(C_6H_5)_3As + H_2O \\ \hline \mathrm{F} & 4(C_6H_5)_2AsH + 4(C_6H_5)_3As(OH)Cl = 4(C_6H_5)_3As + (C_6H_5)_2As\text{—}O\text{—}As(C_6H_5)_2 + \\ & \qquad 2(C_6H_5)_2AsCl + 3H_2O + 2HCl \end{array}$$

mixture of reaction products had been treated with alkali, which converted the diphenylchloroarsine into tetraphenylarsyl oxide. The arsine, oxide and chloride in formulation F are probably produced as a result of the interaction of diphenylarsine and the hydroxychloride with the formation of triphenylarsine and tetraphenyldiarsyl (D); tetraphenyldiarsyl and the hydroxychloride then react to yield tetraphenylarsyl oxide, diphenylchloroarsine and triphenylarsine (E). In separate experiments we have shown that the reactions formulated (D and E) take place readily.

Experimental Part

All reactions were carried out in a modified free-radical bulb[8] at ordinary temperature and all compounds sensitive toward oxygen were isolated in a carbon dioxide atmosphere. Melting points of compounds which react with oxygen were determined in a sealed tube filled with nitrogen.

Although it was evident that some of the reactions took place rapidly and were, no doubt, completed in a short time, all of the reaction mixtures were shaken for at least twelve hours unless otherwise indicated.

Absolute grades of alcohol and ether were used and all solvents were thoroughly saturated with nitrogen prior to use.

Reactions of Phenylarsine

Oxygen.—In a typical experiment 0.543 g. of the arsine, dissolved in bromobenzene, absorbed 57 cc. (N. T. P.) of oxygen in one minute, 66 cc. in sixty minutes and 81 cc. after forty-eight hours. Interaction of the above amount of arsine with one molecular equivalent of oxygen would require 79 cc. of oxygen.

Sulfur.—To 3.5 g. (2.6 cc.) of phenylarsine, dissolved in 10 cc. of benzene, there was added 0.64 g. of sulfur, dissolved in 50 cc. of the same solvent. After twenty-four hours all of the solvent was removed; during this process a strong odor of hydrogen sulfide was noticeable. The oily residue was dissolved in 20 cc. of benzene. Upon the addition

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(8) Ref. 6, p. 3356.

of 50 cc. of absolute alcohol the phenylarsine sulfide precipitated in crystalline form; yield 0.81 g.; calcd. yield 1.8 g.; m. p. 161–163°.[9]

Iodine.—The iodine was decolorized at once when 2.54 g. (0.01 mole) of iodine, dissolved in 40 cc. of ether, was added to 1.54 g. (1.14 cc., 0.01 mole) of phenylarsine, dissolved in 10 cc. of ether, and an orange, crystalline precipitate separated. This material was arsenobenzene contaminated by a small amount of by-product, probably diphenyldiiododiarsyl. The mixture was shaken for forty-eight hours and then treated with alkali. After separation of the alkaline layer and removal of the solvent, 1 g. of arsenobenzene was obtained; calcd. yield 1.5 g. After recrystallization from toluene the product melted at 208–210°.

To 1.54 g. (0.01 mole) of the arsine, dissolved in ether, there was added 3.81 g. (0.015 mole) of iodine, dissolved in 75 cc. of the same solvent. Diphenyldiiododiarsyl crystallized slowly from the solution. The ether was decanted and the product washed three times with ether. The yield was 2.5 g.; calcd. yield 2.8 g.; m. p. 175–177°.[10]

To a solution prepared from 1.54 g. (0.01 mole) of the arsine and 10 cc. of benzene, there was added 5.1 g. (0.02 mole) of iodine, dissolved in 80 cc. of benzene. The iodine was decolorized immediately. The red oil, phenyldiiodoarsine, obtained after complete removal of the solvent, was isolated as phenylarsylene N-pentamethylenedithiocarbamate; the yield of the latter was 4.6 g.; calcd. yield 4.7 g.; m. p. 183–184°.[11]

Triphenylarsine Dichloride.—A solution prepared from 3.77 g. (0.01 mole) of triphenylarsine dichloride and 40 cc. of dry benzene was added to 1.54 g. (0.01 mole) of phenylarsine, dissolved in 10 cc. of the same solvent. After a short time the stopcock of the radical bulb was opened for an instant in order to allow hydrogen chloride to escape. The mixture was shaken and after twelve hours the liquid was decanted from the precipitated arsenobenzene into a second radical bulb. The residue of arsenobenzene was suspended in ether and shaken with alkali in order to remove traces of hydrogen chloride,[12] and then with alcohol and ether; yield 0.9 g.; calcd. yield 1.5 g.; mixed m. p. 210–212°.[13]

The decanted benzene solution was shaken with alkali and the benzene was then removed. The oily, yellow residue was boiled with 50 cc. of alcohol and the alcoholic solution filtered in air. When the filtrate cooled triphenylarsine precipitated. A further amount of product was obtained from the filtrate; yield 2.5 g.; calcd. yield 3.1 g.; m. p. 60–61°.[14]

Reactions of Diphenylarsine

Oxygen.—The data obtained from two typical experiments are as follows: 0.565 g. of the arsine, dissolved in bromobenzene, absorbed 49 cc.[15] of the gas in one minute and 58 cc. in sixty minutes. If the arsine reacted with two molecular equivalents of oxygen, 55 cc. would be required. In another experiment 0.868 g. of arsine absorbed 89 cc. of the gas after sixty minutes; calcd. amount, 85 cc. Extraction with ammonia

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[9] The melting point recorded for this compound by Schulte [*Ber.*, **15**, 1953 (1882)] is 152°. It was reported [Blicke and Smith, THIS JOURNAL, **52**, 2948 (1930)] that preparation of the sulfide from phenyldichloroarsine and hydrogen sulfide yielded material which melted at 174–176°. When this experiment was repeated, sulfide was obtained which melted at 162–164°. Mixed with the sulfide obtained from phenylarsine the mixed melting point was found to be 162–164°. It is not strange that different samples of phenylarsine sulfide possess different melting points in view of the fact that marked variations in melting point have been observed among preparations of phenylarsine oxide.

[10] Blicke and Smith, THIS JOURNAL, **52**, 2943 (1930).

[11] Blicke and Oakdale, *ibid.*, **54**, 2995 (1932). Due to an error the melting point was recorded previously as 173–174°.

[12] Arsenobenzene absorbs oxygen rapidly in the presence of halogen acids [Blicke and Smith, *ibid.*, **52**, 2946 (1930)].

[13] Michaelis and Schäfer [*Ber.*, **46**, 1742 (1913)] recorded the m. p. as 212°.

[14] The m. p. has been recorded as 58–59° by Pfeiffer [*ibid.*, **37**, 4621 (1904)].

[15] About two-thirds of this volume was absorbed after fifteen seconds.

water of the combined bromobenzene solutions yielded 1.51 g. or 93% of the calcd. amount of diphenylarsinic acid; m. p. 168–170°.[16]

Iodine.—When 2.30 g. (0.01 mole) of diphenylarsine reacted with 2.54 g. (0.01 mole) of iodine in 50 cc. of alcohol, a light yellow solution was obtained which yielded 3.60 g. of diphenyliodoarsine; m. p. 41–42°.[17] The calcd. yield is 3.56 g.

To 2.30 g. (177 cc., 0.01 mole) of diphenylarsine, dissolved in 10 cc. of absolute alcohol, there was added 1.27 g. (0.005 mole) of iodine, dissolved in 40 cc. of alcohol. The iodine was decolorized instantly and crystals of tetraphenyldiarsyl began to separate rapidly. The alcohol was decanted, the product suspended in about 25 cc. of ether and shaken with water until hydriodic acid had been completely removed. The water layer was separated and most of the ether removed by distillation under reduced pressure. The diarsyl was then washed three times with 10-cc. portions of absolute alcohol and dried in a bath at 60–70° under diminished pressure. The material melted at 127–129°;[18] yield 2.0 g.; calcd. yield 2.3 g. Dissolved in bromobenzene 0.769 g. of the compound absorbed 40 cc. (N. T. P.) of oxygen; calcd. absorption 37 cc.

Tetraphenylarsyl Oxide.—To 3.45 g. (0.015 mole) of diphenylarsine, dissolved in 20 cc. of absolute alcohol, there was added 3.56 g. (0.0075 mole) of tetraphenylarsyl oxide, dissolved in 80 cc. of the same solvent. After several days, the solvent was decanted from the precipitated tetraphenyldiarsyl and the latter washed four times with 20-cc. portions of alcohol; yield 6.25 g. or 91% of the calcd. amount; m. p. 125–127°. Dissolved in bromobenzene 1.378 g. of the material absorbed 65 cc. of oxygen in less than two minutes; calcd. absorption 67 cc.

Tetraphenylarsyl Sulfide.—Tetraphenyldiarsyl began to crystallize from the solution soon after the addition of 2.45 g. of tetraphenylarsyl sulfide, dissolved in alcohol, to 2.30 g. of diphenylarsine, dissolved in 20 cc. of the same solvent. The alcohol was decanted from the diarsyl, the latter washed twice with alcohol and then with ether; m. p. 129–130°; yield 1.9 g.; calcd. yield 4.6 g.

Phenylchloroethoxyarsine.—To 4.6 g. of phenylchloroethoxyarsine (0.02 mole), dissolved in 80 cc. of absolute alcohol there was added 4.60 g. (0.02 mole) of diphenylarsine, dissolved in 20 cc. of the same solvent. Arsenobenzene began to precipitate at once in crystalline form. The precipitate was filtered through a Jena filter, in an inert atmosphere,[19] and washed with four 50-cc. portions of alcohol. The alcohol washings were added to the alcoholic filtrate. Finally, the arsenobenzene was washed thoroughly with absolute ether; m. p. 208–209°; yield 2.83 g.; calcd. yield 3.04 g.

From the alcoholic filtrate 4.7 g. of diphenylchloroarsine was obtained; mixed m. p. 40–42°; calcd. yield 5.3 g.

Triphenylbromomethane.—Two and three-tenths grams (0.01 mole) of diphenylarsine and 3.23 g. of triphenylbromomethane dissolved in 70 cc. of ether were placed in a free-radical apparatus. After twenty-four hours the solution was concentrated to a small volume. Ten cc. of petroleum ether (30–60°) was added and the solution cooled, whereupon a small amount of fairly pure triphenylmethane separated in crystalline form. After recrystallization from alcohol the methane melted at 91–93°;[20] mixed m. p. 92–93°. The ether–petroleum ether filtrate was evaporated to dryness, the residue dissolved in ether and filtered to remove traces of diphenylarsinic acid. After evaporation of the solvent from the filtrate, the crystalline residue was dissolved in a small amount of

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(16) We are inclined to believe that the m. p. 178° assigned to this substance by Bart [*Ann.*, **429**, 101 (1922)] is too high. We have observed in many instances that the acid melts in the neighborhood of 170° and this corresponds to the observation of Gibson and Johnson [*J. Chem. Soc.*, 99 (1928)] who stated that an analyzed sample of the acid melted at 171°.

(17) Blicke and Smith, THIS JOURNAL, **51**, 1564 (1929).

(18) Blicke, Weinkauff and Hargreaves, *ibid.*, **52**, 782 (1930).

(19) In the presence of diphenylchloroarsine arsenobenzene absorbs oxygen rapidly.

(20) Kekulé and Franchimont, *Ber.*, **5**, 907 (1872).

hot alcohol. When the solution was cooled, practically pure triphenylmethane separated. Concentration of the filtrate yielded diphenylbromoarsine, mixed m. p. 52–54°,[21] and upon further concentration a mixture which seemed to consist of the methane and bromoarsine was obtained. The components of this mixture were not separated. In all there were isolated 1.7 g. of pure methane and 0.7 g. of pure bromoarsine.

Triphenylmethyl.—To 4.6 g. (0.02 mole) of diphenylarsine in 10 cc. of benzene there was added a solution of triphenylmethyl prepared from 6.5 g. of triphenylbromomethane, 10 g. of molecular silver and 80 cc. of benzene. The orange triphenylmethyl solution was decolorized at once. After concentration to a volume of 20 cc. triphenylmethane precipitated in the form of its benzene addition product; the benzene-free crystals, which weighed 1.6 g., melted at 90–92°. The solution was decanted from the crystals into a second radical bulb, the solvent removed and the residue recrystallized from alcohol. The tetraphenyldiarsyl obtained weighed 1.0 g.; m. p. 125–128°. Dissolved in bromobenzene 0.776 g. of the material absorbed 39 cc. (N. T. P.) of oxygen; calcd. amount 38 cc.

Triphenylarsine Dichloride (A, C).—A mixture of 3.77 g. (0.01 mole) of the arsine dichloride dissolved in 40 cc. of dry benzene and 4.60 g. (0.02 mole) of the arsine dissolved in 10 cc. of benzene was shaken for twelve hours, the benzene removed completely and 40 cc. of absolute alcohol added to the residue to dissolve the triphenylarsine formed. The alcoholic solution was decanted, the residue of tetraphenyldiarsyl washed with alcohol and then recrystallized from 200 cc. of the same solvent; yield 4.1 g.; calcd. yield 4.6 g.; m. p. 130–131°. Dissolved in bromobenzene 1.045 g. of the diarsyl absorbed 53 cc. of oxygen in ten seconds; calcd. absorption 51 cc. Upon concentration of the alcoholic solution of triphenylarsine 2.1 g. of the latter compound was obtained; calcd. yield 3.0 g.; m. p. 60–61°.

A mixture of 3.77 g. (0.01 mole) of triphenylarsine dichloride, dissolved in 40 cc. of dry benzene, and 2.30 g. (0.01 mole) of diphenylarsine yielded triphenylarsine and diphenylchloroarsine. Since it was found impossible to separate these compounds, the mixture was treated with sodium hydroxide solution, whereby the diphenylchloroarsine was converted into tetraphenylarsyl oxide. The latter substance is quite insoluble in petroleum ether (30–60°) while the triphenylarsine is very soluble in this solvent. After the separation had been effected, the triphenylarsine was recrystallized from alcohol; yield 2.3 g.; calcd. yield 3.1 g.; mixed m. p. 60–61°. The yield of tetraphenylarsyl oxide was 2.0 g.; calcd. yield 2.4 g.; mixed m. p. 92–94°.[22]

Tetraphenyldiarsyl and Triphenylarsine Dichloride (B).—To 2.62 g. of tetraphenyldiarsyl, dissolved in 30 cc. of dry benzene, there was added 1.72 g. of triphenylarsine dichloride, dissolved in 30 cc. of the same solvent. After twelve hours the solvent was removed completely and the residue of triphenylarsine and diphenylchloroarsine dissolved in 20 cc. of petroleum ether (30–60°). The diphenylchloroarsine was separated in the form of tetraphenylarsyl oxide as described above. The yield of the latter product was 1.9 g.; calcd. yield 2.7 g.; m. p. 92–95°. The yield of triphenylarsine was 1.2 g.; calcd. yield 1.7 g.; m. p. 59–60°.

Triphenylarsine Hydroxychloride (D, F).—When 4.60 g. (0.02 mole) of diphenylarsine, dissolved in 20 cc. of absolute alcohol, was added to 3.58 g. (0.01 mole) of triphenylarsine hydroxychloride, dissolved in 20 cc. of the same solvent, tetraphenyldiarsyl precipitated immediately while triphenylarsine remained in solution. After twelve hours the alcoholic solution was decanted from the crystalline material into a second radical bulb. The diarsyl was washed with absolute alcohol and then recrystallized from 150 cc. of this solvent. The yield was 3.6 g.; calcd. yield 4.6 g.; m. p.

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(21) Steinkopf and Schwen, *Ber.*, **54**, 1459 (1921).

(22) The highest m. p. recorded in the literature is 95.5–96.5° [Blicke and Smith, THIS JOURNAL, **51**, 1562 (1929)].

127–129°. Dissolved in bromobenzene 0.839 g. of the diarsyl absorbed 43 cc. (N. T. P.) of oxygen; calcd. absorption 41 cc. The triphenylarsine obtained from the alcoholic solution weighed 2.4 g.; calcd. yield 3.1 g.; m. p. 59–60°.

Four and six-tenths grams of diphenylarsine (0.02 mole) dissolved in 10 cc. of alcohol, was mixed with 7.16 g. (0.02 mole) of the hydroxychloride, dissolved in 40 cc. of the same solvent. After twelve hours the solvent was removed, the diphenylchloroarsine hydrolyzed to the corresponding oxide and the reaction products separated; yields, 2.7 g. of tetraphenylarsyl oxide, m. p. 90–92°, and 5 g. of triphenylarsine, mixed m. p. 59–61°.

Tetraphenyldiarsyl and Triphenylarsine Hydroxychloride (E).—Three and three-tenths grams of the isolated diarsyl, dissolved in 70 cc. of benzene, was mixed with 2.58 g. of the hydroxychloride, dissolved in 25 cc. of alcohol. After twenty-four hours the solvents were removed, the residue dissolved in petroleum ether and shaken with 5% sodium hydroxide solution, whereby the diphenylchloroarsine was converted into tetraphenylarsyl oxide. Upon concentration of the petroleum ether layer the oxide separated. Triphenylarsine was isolated from the mother liquor. Both products were recrystallized from alcohol. The yield of oxide, m. p. 91–94°, was 1.7 g.; the yield of arsine, m. p. 59–61°, was 1.1 g.

Summary

The reactions which take place between phenylarsine and oxygen, sulfur, iodine and triphenylarsine dichloride, respectively, have been studied.

Interaction of diphenylarsine with each of the following substances has been investigated: oxygen, iodine tetraphenylarsyl oxide, tetraphenylarsyl sulfide, phenylchloroethoxyarsine, triphenylbromomethane, triphenylmethyl, triphenylarsine dichloride and triphenylarsine hydroxychloride.

In addition it has been shown that tetraphenyldiarsyl reacts with triphenylarsine dichloride and with triphenylarsine hydroxychloride.

ANN ARBOR, MICHIGAN

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