# Tri-*t*-butyl Phosphite and Some of Its Reactions

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Tri-*t*-butyl phosphite, m.p. 4–5°, obtained in high conversion, was characterized by $H^1$ and $P^{31}$ nuclear magnetic resonance (n.m.r.) and infrared spectroscopy. It reacted spontaneously with methyl bromide and methyl iodide to yield (in addition to the Michaelis–Arbuzov product, di-*t*-butyl methylphosphonate) di-*t*-butyl phosphonate as the major product. It reacted also with hexachlorocyclopentadiene to yield 5-*t*-butylpentachlorocyclopentadiene. Several quadruply connected derivatives were prepared and characterized by n.m.r. and infrared spectroscopy. The high reactivity of tri-*t*-butyl phosphite is attributed to both inductive and steric influences resulting from the presence of the three *t*-butoxy groups.

The need of tri-*t*-butyl phosphite, $(t\text{-}C_4H_9O)_3P$ (I), of known purity arose in the course of a separate investigation. Perusal of the literature indicated, however, a controversial background for this compound which has not been isolated and characterized in pure form.[2] $P^{31}$ n.m.r. spectroscopy, shown to be uniquely apt for distinguishing between and characterizing various classes of phosphorus compounds,[3a] appeared to offer a convenient method to analyze both qualitatively and quantitatively for (and hence to follow the fate of) I in its formation and in its reactions.

A preliminary experiment, in which the literature procedure[2b,f,g] was followed, indicated that the reaction betwen phosphorus trichloride, *t*-butyl alcohol, and triethylamine, carried out between 0 and 5°, yielded, after filtration, a solution in which the precipitation of the amine hydrochloride continued during the time that the filtrate warmed up to room temperature. In subsequent experiments carried out under similar conditions, the reaction mixture was kept, therefore, at room temperature for 12–24 hr. before filtration. The weights of both the amine hydrochloride and of the residue, obtained after the evaporation of the solvent *in vacuo* at or below room temperature, indicated an essentially quantitative formation of I, which was characterized by $H^1$ n.m.r., $P^{31}$ n.m.r., and infrared spectroscopy, refractive index, and melting point.

Although proven to be very reactive, a sample of the phosphite was kept under nitrogen at 25° for days and at *ca.* 0° for weeks without any noticeable decrease in the assay as indicated by $P^{31}$ n.m.r. spectroscopy. When heated, however, above 50° *in vacuo*, a rapid evolution of isobutylene occurred so that, after 2 hr. at 80° and 50 mm., an essentially quantitative yield of di-*t*-butyl phosphonate, $(t\text{-}C_4H_9O)_2P(O)H$ (II), was obtained. The rapid decomposition above 50° thus indicates that distillation[2b,d,f,g] of the crude phosphite is not a suitable method of purification and that any operation at elevated temperature results in rapid decrease in the assay of the sample.

While handling I exposed to air, the ready formation of a crystalline crust was noted, which was identified as tri-*t*-butyl phosphate[2f,g,h] (III), m.p. 72–74°. When air was introduced in I at room temperature, a spontaneous exothermic reaction started and III was obtained in high yield. The ready formation of III contrasts with the resistance of triethyl phosphite to oxidation by air.[2f,g]

Both sulfur and selenium reacted exothermally with I and yielded the corresponding adducts, O,O,O-tri-*t*-butyl phosphorothioate, $(t\text{-}C_4H_9O)_3PS$ (IV), and O,O,O-tri-*t*-butyl phosphoroselenoate, $(t\text{-}C_4H_9O)_3PSe$ (V). On heating above 80°, IV started to decompose and yielded O,O-di-*t*-butyl thiolphosphate, $(t\text{-}C_4H_9O)_2P(O)SH$ (VI), m.p. 90–93°.

The high reactivity of I was indicated also by its participation at room temperature in the Michaelis–Arbuzov reaction. When the theoretical amount of methyl iodide was added to I at 5–10°, formation of white crystals was noted, the amount of which increased as the temperature rose owing to the mildly exothermic nature of the subsequent reaction. Soon (in about 15–20 min.), the crystals began to disappear, and after 30–40 min. a pale yellow solution resulted and the $P^{31}$ n.m.r. showed it to be a mixture of II (major) and the Michaelis–Arbuzov product, di-*t*-butyl methylphosphonate (VII), $CH_3P(O)(O\text{-}t\text{-}C_4H_9)_2$. The structure of VII was proven, in addition to $H^1$ and $P^{31}$ n.m.r. and infrared spectroscopy, also by its synthesis from sodium di-*t*-butyl phosphite and methyl iodide. I yielded VII spontaneously when brought into contact with methyl bromide also but not, under similar conditions, with methyl chloride.

The formation of II in major proportion in the reaction of I with methyl iodide requires some comment. The precipitation of the transient white crystals safely can be attributed to the formation of the phosphonium adduct[3b] (VIII).

$$(C_4H_9O)_3P\colon + CH_3I \longrightarrow \underset{\textbf{VIII}}{(C_4H_9O)_3P^{+}\text{—}CH_3\ I^-} \qquad (1)$$

The decay of VIII to VII takes place probably by an $S_N1$ route[4] (eq. 2), which can be assisted by the partici-

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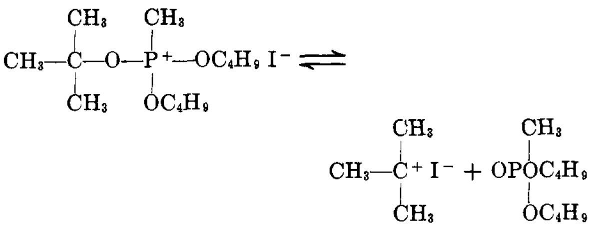
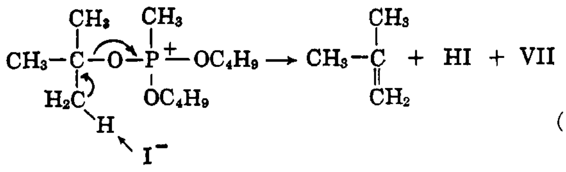
[2] (a) T. Milobendzki and A. Sachnowski, *Chem. Polski*, **15**, 34 (1917); (b) G. M. Kosolapoff, *J. Am. Chem. Soc.*, **74**, 4953 (1952); (c) W. Gerrard, M. J. D. Isaacs, M. Machell, K. B. Smith, and P. L. Wyvill, *J. Chem. Soc.*, 1920 (1953); (d) R. W. Young, *J. Am. Chem. Soc.*, **75**, 4620 (1953); (e) H. Goldwhite and B. C. Saunders, *J. Chem. Soc.*, 2409 (1957); (f) J. R. Cox, Jr., and F. H. Westheimer, *J. Am. Chem. Soc.*, **80**, 5441 (1958); (g) J. R. Cox, Jr., Ph.D. thesis, Harvard University, 1959; (h) J. R. Cox, Jr., M. G. Newton, and O. B. Ramsay, Abstracts, Joint Southeastern-Southwestern Regional Meeting of The American Chemical Society, New Orleans, La., Dec., 1961.

[3] (a) N. Muller, P. C. Lauterbur, and J. Goldenson, *J. Am. Chem. Soc.*, **78**, 3557 (1956); J. R. Van Wazer, C. F. Callis, J. N. Shoolery, and R. C. Jones, *ibid.*, **78**, 5715 (1956); H. Finegold, *Ann. N. Y. Acad. Sci.*, **70**, 875 (1958); R. A. Y. Jones and A. R. Katritzky, *Angew. Chem.*, **74**, 60 (1962); L. C. D. Groenweghe, L. Maier, and K. Moedritzer, *J. Phys. Chem.*, **66**, 901 (1962). (b) Low solubility of phosphonium salts in nonpolar solvents, such as ether, is well-known and contrasts with the good solubility of the related triply and quadruply connected uncharged structures in the same solvent. For instance, in the preparation of the methiodide of triphenyl phosphite, *i.e.*, $CH_3P^+(OC_6H_5)_3I^-$, ether is used to elute the unconverted starting materials [S. R. Landauer and H. N. Rydon, *J. Chem. Soc.*, 2224 (1953)].

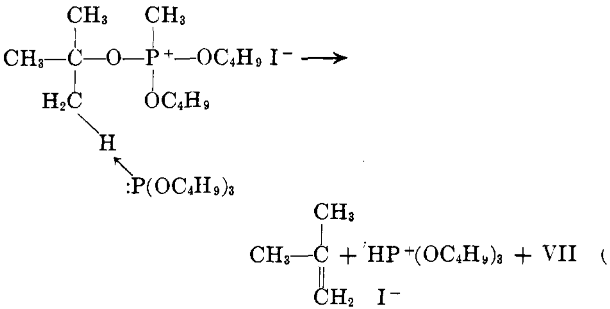


$$(CH_3)_3C{-}O{-}\overset{+}{P}(CH_3)(OC_4H_9)_2\ I^- \rightleftharpoons (CH_3)_3C^+\ I^- + OP(CH_3)(OC_4H_9)_2 \quad (2)$$

VII



$$CH_3{-}C(CH_3)(CH_2H){-}O{-}\overset{+}{P}(CH_3)(OC_4H_9)_2\ I^- \longrightarrow CH_3{-}C(CH_3){=}CH_2 + HI + VII \quad (2a)$$



$$CH_3{-}C(CH_3)(CH_2H){-}O{-}\overset{+}{P}(CH_3)(OC_4H_9)_2\ I^- + :P(OC_4H_9)_3 \longrightarrow CH_3{-}C(CH_3){=}CH_2 + HP^+(OC_4H_9)_3\ I^- + VII \quad (2b)$$

IX

pation of the halide (eq. 2a) or of the phosphite (I) (eq. 2b).

Decay of IX probably follows a route similar to the decay of VIII by eq. 2, 2a, or 2b.

$$IX \longrightarrow II + \text{coproducts} \qquad (3, 3a, 3b)$$

Consumption of I by one of the coproducts of eq. 2a, HI, can also take place.[5] This step also results in the formation of IX.

Compound I reacted readily also with hexachlorocyclopentadiene and yielded 5-*t*-butylpentachlorocyclopentadiene (X)[4] and di-*t*-butylphosphorochloridate, $(t\text{-}C_4H_9O)_2P(O)Cl$(XI), identified by $P^{31}$ n.m.r. The $H^1$ and $P^{31}$ n.m.r. frequencies of the phosphorus compounds listed are presented in Table I.

TABLE I

$P^{31}$ AND $H^1$ N.M.R. CONSTANTS

| | Compound | $\delta_{P^{31}}$, p.p.m.[a] | $J_{H-P}$, c.p.s. | $\delta_{H^1}$ ($t$-$C_4H_9$), p.p.m.[b] |
|---|---|---|---|---|
| I | $(t\text{-}C_4H_9O)_3P$ | −138.2 | | 1.34 |
| II | $(t\text{-}C_4H_9O)_2P(O)H$ | +3.8 | 678 | 1.45[c] |
| III | $(t\text{-}C_4H_9O)_3PO$ | +13.3 | | 1.46 |
| IV | $(t\text{-}C_4H_9O)_3PS$ | −41.2 | | 1.53 |
| V | $(t\text{-}C_4H_9O)_3PSe$ | −31.1 | | 1.57 |
| VI | $(t\text{-}C_4H_9O)_2P(O)SH$ | −25.0 | | 1.54 |
| VII | $(t\text{-}C_4H_9O)_2P(O)CH_3$ | −21.2 | 17.3 | 1.44[d] |
| XI | $(t\text{-}C_4H_9O)_2P(O)Cl$ | +5.8 | | |
| XII | $(t\text{-}C_4H_9O)_2PCl$ | −170.3 | | |

[a] External (capillary), 85% $H_3PO_4$ reference. [b] Downfield from internal tetramethylsilane reference. [c] $\delta_{H-P}$ = 6.61 p.p.m.; $J_{P-H}$ = 676 c.p.s. [d] $\delta_{CH_3P}$ = 1.26 p.p.m.; $J_{P-H}$ = 17.4 c.p.s.

The high reactivity of I is probably the result of two major influences: the inductive effect of the *t*-butyl groups and the steric conditions around both the phosphorus and the central carbon atom in the *t*-butyl groups. The effect of the electron-releasing *t*-butyl substituent *via* induction in organic reactions has been studied extensively and interpreted.[6] The $P^{31}$ n.m.r. shifts support the specific role attributable to the *t*-butyl group in increasing the electron density on the site of substitution and suggest that, compared with the normal alkyl analogs, the *t*-butyl increases the electron density on phosphorus *via* the linking oxygen, as indicated by the more effective shielding (high-field shift) in the n.m.r. of the derivatives of I.[7] The high electron density around the phosphorus thus is partly responsible for the enhanced nucleophilicity of I, as, for instance, reflected by the Michaelis–Arbuzov reaction.

Analysis of $P^{31}$ n.m.r. data on I and related structures indicates considerable steric congestion in the trivalent species.[7] Although measurements of bond angles of the organic P(III) esters are not reported in the literature, the interpretations of n.m.r. chemical shifts are compatible with structures in which the esters of phosphorous acid primarily utilize p-orbitals[7] and hence have bond angles not far from the theoretical value of 90°. When the substituents are bulky, as in the case of *t*-butyl, steric congestion arises and the repulsion between the substituent groups results in widening of the bond angles,[7] which occurs through increased utilization of $sp^3$-orbitals.[8] The enhanced reactivity of I over its straight chain analogs thus is partly attributable to the "abortive bond"[8] formed by the inclusion of the unshared pair of electrons of the phosphorus in the $sp^3$ hybrid and to the decrease of strain in the transformation of I into quadruply connected, tetrahedral structures, which are known to have expanded bond angles close to the theoretical value of 109° 28′.

Much of the chemistry of I is attributable to the ready detachment of this bulky substituent as a *t*-butyl carbonium ion. The ease with which SN1 or SN1 type of reactions (eq. 2–3b) takes place[2e,9] is due to the formation of $(CH_3)_3C^+$, which eliminates isobutylene and makes the step, when carried out at or above room temperature and in an open system, essentially irreversible.[10]

## Experimental

**N.m.r. Spectra.**—Proton spectra were obtained with an A-60 n.m.r. spectrometer, manufactured by Varian Associates, Palo Alto, Calif., using tetramethylsilane as internal reference. $P^{31}$ measurements were made on a Varian Associates HR-60 high-resolution spectrometer equipped with a Model V-4311 fixed-frequency RF unit, operating at 24.288 Mc. in a magnetic field of 14,092 gauss. Referencing was done by an audio-side-band

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(4) The transfer of *t*-butyl in preference to ethyl was demonstrated in the alkylation of hexachlorocyclopentadiene by *t*-butyl diethyl phosphite [V. Mark, *Tetrahedron Letters*, 295 (1961)] and interpreted as taking place *via* an SN1 route [V. Mark, Abstracts, 139th National Meeting of the American Chemical Society, St. Louis, Mo., March 1961, p. 46-O].

(5) For a study of the dealkylation of tributyl phosphite by hydrogen halides, see W. Gerrard and E. G. G. Whitebread, *J. Chem. Soc.*, 914 (1952).

(6) See, for example, C. K. Ingold, "Structure and Mechanism in Organic Chemistry," Cornell Univ. Press, Ithaca, N. Y., 1953, pp. 64-71, and several pertinent chapters listed in the Index.

(7) V. Mark and J. R. Van Wazer, a correlation of molecular structure with the n.m.r. shifts of phosphorus compounds having tertiary alkyl substituents will be published separately.

(8) J. R. Van Wazer, "Phosphorus and Its Compounds," Interscience Publishers, Inc., New York, N. Y., 1958, pp. 70, 53.

(9) For a comparison and a contrasting of the hydrolysis of tri-*t*-butyl phosphate with the primary trialkyl phosphates (in which the P-O, and not the C-O, bonds are cleaved), see ref. 2h.

(10) The formation of isobutylene was actually observed in the Michaelis-Arbuzov reactions of I.

modulation technique, using (as an external reference) 85% phosphoric acid in a sealed 1.0-mm.-o.d. capillary tube inserted in the sample. Peak-position measurements could be reproduced to within ±0.1 p.p.m. Positive shifts ($\delta$) are upfield.

**Tri-*t*-butyl Phosphite** (I).—To a three-necked flask equipped with a Hershberg stirrer, dropping funnel, thermometer, and a vent line leading to a nitrogen atmosphere, there was charged a solution of 74.1 g. (1.0 mole) of *t*-butyl alcohol and 111.3 g. (1.1 moles) of triethylamine in 400 ml. of anhydrous ether. The solution was cooled to +2 to +5°, after which a solution of 45.8 g. (0.33 mole) of phosphorus trichloride in 100 ml. of anhydrous ether was added at such a rate as to keep the temperature of the resultant slurry around +5°. After the addition was completed in about 2 hr., the slurry was stirred for an additional hour at +2 to +5°. A filtered sample of a parallel experiment taken at this time deposited, on warming to room temperature, triethylamine hydrochloride. The $P^{31}$ n.m.r. of a sample stripped of the solvent and triethylamine below +5° indicated the presence of di-*t*-butyl phosphorochloridite, $(t\text{-}C_4H_9O)_2PCl$ (XII), $\delta = -170.3$ p.p.m.) and of I ($\delta = -138.2$ p.p.m.).

The ice bath was removed and the slurry was kept at room temperature (24°) for 12–18 hr. Filtration through a sintered-glass funnel and rinsing of the filter cake (which, after drying, weighed 136.2 g., 99%) yielded a clear solution. After stripping of the solvent *in vacuo* at 5–15°, a pale yellow oil was obtained, $n^{25}D$ 1.4205, the weight (80.6 g.) of which indicated a 96.7% conversion. $P^{31}$ n.m.r. of the sweet smelling oil showed a composition of 90% of I and 10% of II. The yield of I accordingly was 87%. The oil solidified at 0° to a white crystalline mass. The large crystals, which had a feather-like structure, melted at 4–5°. Infrared maxima of the oil were 3.35 (s)[11] C–H; 6.82 (w), 7.21 (m), and 7.34 (s) $t\text{-}C_4H_9$; 7.91 (m) P=O, due to the presence of II; 8.04 (m); 8.52 (s); 9.67 (m); 10.10 (s) P–O–C; 10.58 (s); 11.04 (s); 12.40 (m); 13.68 (w); and 14.56 (m) $\mu$. The n.m.r. data are summarized in Table I.

**Di-*t*-butyl Phosphonate** (II).—Considerable frothing began when a sample of I (17.7 g.) was heated to 50–60° at 20–30 mm. After the gas evolution subsided, the pale yellow oil (which now weighed 14.3 g.) was fractionated to yield II as a colorless liquid, b.p. 42° at 0.4 mm., $n^{23}D$ 1.4186.[2c,d,e] The infrared maxima checked with those reported.[2d,e]

**Tri-*t*-butyl Phosphate** (III).—The introduction of air into I (8.0 g.) caused a mildly exothermic reaction, in the course of which the temperature rose to 35°. In about 1 hr., the sample solidified. Recrystallization from pentane yielded large colorless plates of II, m.p. 72–74° (lit.[2f,g] m.p. 71–75°). Infrared maxima were 3.33 (s) C–H; 6.73 (m), 7.14 (s), and 7.26 (s) $t\text{-}C_4H_9$; 7.88 (s) P=O; 8.47 (m); 9.60 (s); 10.06 (s) P–O–C; 10.88 (m); 12.02 (w); 12.32 (w); 13.28 (w); 14.12 (m); 14.50 (w); and 15.28 (w) $\mu$.

*Anal.* Calcd. for $C_{12}H_{27}O_4P$: C, 54.11; H, 10.22; P, 11.59. Found: C, 53.9; H, 10.2; P, 11.4.

**O,O,O-Tri-*t*-butyl Phosphorothioate** (IV).—The addition of sulfur powder to a solution of 14.6 g. of I in 30 ml. of ether was accompanied by an exothermic reaction in which the sulfur was consumed. The solution was allowed to stand with excess sulfur overnight, after which filtration and evaporation of the solvent *in vacuo* yielded IV in form of a pale yellow oil (16.0 g.), $n^{25}D$ 1.4482. $P^{31}$ n.m.r. ($\delta = -41.2$ p.p.m.) confirmed the structure and indicated the presence of small amounts (~5%) of II and III. Infrared maxima were 3.36 (s), 6.82 (m), 7.21 (s), 7.35 (s), 8.05 (s), 8.58 (s), 9.68 (s), 10.16 (s), 10.95 (s), 12.17 (s), 13.56 (s), 14.16 (w), and 14.57 (m) $\mu$.

**O,O-Di-*t*-butyl Phosphorothioic Acid** (VI).—Distillation of IV (14.6 g.) was only partly successful. Although pure IV was obtained as a colorless oil, b.p. 80° at 0.4 mm., the bulk of the sample decomposed with strong gas evolution when the pot temperature surpassed 85°. Even during the decomposition of the sample, a solid began to separate in the flask and, at the end of the reaction, the entire content of the flask solidified to an off-white solid, which weighed 5.7 g. Recrystallization from ether yielded white crystals, m.p. 90–93° (decomposition with gas evolution), which were identified as O,O-di-*t*-butyl phosphorothioic acid, $(t\text{-}C_4H_9O)_2P(O)SH$, by $P^{31}$ n.m.r. ($\delta = -25.0$ p.p.m.), elemental analysis (Calcd. for $C_8H_{19}O_3PS$: C, 14.17. Found: S, 13.6.), and infrared analysis. The latter showed the following maxima in a Nujol mull: 3.4 (s, broad); 4.2 (m, broad) probably sh; 6.79 (m), 7.10 (w), and 7.25 (m) $t\text{-}C_4H_9$; 8.14 (s, broad) hydrogen-bonded P=O; 8.60 (s); 9.88 (s) P–O–C; 10.80 (s); and 12.3 (m) $\mu$.

**O,O,O-Tri-*t*-butyl Phosphoroselenoate** (V).—The addition of selenium in excess to a solution of 2.5 g. of I in 5 ml. of ether was exothermic and yielded V in form of a colorless oil. The structure of V was identified by its $P^{31}$ n.m.r. frequency ($\delta = -31.1$ p.p.m.), which showed the presence of II and III only in small amounts (~10%) in the sample.

**Di-*t*-butyl Methylphosphonate** (VII). A. *Via* **the Michaelis–Arbuzov Reaction.**—To an ice-cold sample of crystalline I (31.8 g., 0.127 mole) there was added, without solvent, 20.0 g. (0.141 mole) of methyl iodide. The crystals dissolved readily, but in about 6 min. a precipitate of smaller crystals began to separate, the amount of which increased for about 10 min. more, parallel with a spontaneous increase in temperature to 29°. In another 5 min., the crystals began to dissolve and, in about 20 min. more, a clear solution resulted while the temperature decreased to 22°. $P^{31}$ n.m.r. of the sample gave the following pattern: a quadruplet at $-21.2 \pm 0.1$ p.p.m. (about 5%), $J \approx 17.3$ c.p.s.; the doublet of II at $-10.4$ and $+17.4$ p.p.m., $J = 678$ c.p.s. (about 90%); and the singlet of III (about 5%) at $+13.6 \pm 0.1$ p.p.m.

Essentially the same results were obtained when methyl bromide (excess) was added at 0° to I. Transient formation of a white, crystalline precipitate was noted again during the mildly exothermic reaction. No reaction was observed within a 1-hr. test period when methyl chloride was introduced into I at 20 to 25°.

B. *Via* **the Michaelis–Becker Reaction.**—The sodium salt of II was prepared in the form of a thick paste by refluxing 16.2 g. (0.0835 mole) of II with 1.92 g. (0.0835 g.-atom) of sodium in 120 ml. of dry dioxane, with the aid of a Hershberg stirrer. After the paste was cooled to room temperature, 50 ml. of ether was added to give a stirrable mixture, and 15.0 g. (0.106 mole) of methyl iodide was added dropwise to keep the strongly exothermic reaction below 30°. After the addition was completed (in 30 min.), the slurry was refluxed for 1 hr., filtered, and the ether was removed by distillation. Pentane (30 ml.) was added to the resulting oil and the solution was washed three times with water in order to remove the dioxane. Drying of the solution and evaporation of the pentane yielded a colorless oil (8.3 g.), the $P^{31}$ n.m.r. of which exhibited the same quadruplet as under A (about 60%), and also the doublet of II (about 40%). The quadruplet accordingly belongs to di-*t*-butyl methylphosphonate (VII), and this assignment is confirmed also by $H^1$ n.m.r. (see Table I). The yield of VII in the Michaelis–Becker reaction is accordingly 29% but can be increased, if care is taken to completely transform II into its sodium salt. Attempted fractionation was not successful in separating VII from II, owing apparently to close boiling points of the two compounds (49° at 1 mm.). Vapor phase chromatography (14 ft. × 1/4 in., 5% Dow Corning 550 silicon on T-6 firebrick) was equally unsuccessful and runs at column temperatures between 190 and 50° indicated thermal decomposition.

Infrared maxima attributable to VII were 3.34 (s), 6.24 (w), 6.67 (w), 7.08 (w), 7.20 (m), 7.31 (s), 7.68 (m), 7.96 (s), 9.12 (s, broad), 9.90 (s, broad), 11.60 (m, broad), 12.55 (s, broad), 13.04 (m), 13.28 (w), 13.63 (w), and 15.12 (w) $\mu$.

**5-*t*-Butylpentachlorocyclopentadiene** (X).—To a solution of 12.5 g. (0.05 mole) of I in 80 ml. of ether, there was added dropwise, in the course of 20 min., a solution of 13.6 g. (0.05 mole) of hexachlorocyclopentadiene in 20 ml. of ether. During the resulting exothermic reaction, the temperature rose to 32° and the solution acquired a pale brown color. Evaporation of the solvent under vacuum yielded a light brown oil, the $P^{31}$ n.m.r. of which indicated the presence of di-*t*-butyl phosphorochloridate, $(t\text{-}C_4H_9O)_2P(O)Cl$ (XI), as the major component (about 70%) in addition to II and III, which were present to about 20 and 10%, respectively. Hydrolysis and chromatography over alumina yielded X (m.p. 62–63°)[4] in 40% yield (5.9 g.).

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(11) s = strong, m = medium, w = weak.