A New Synthesis of Compounds with Cobalt-to-Tin Bonds

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Insertion of tin(II) halides into $[Co(CO)_3L]_2$ compounds gives $[trans-(CO)_3LCO]_2SnX_2$ [X = Cl, Br, I; L = CO, Ph₃P, Buⁿ₃P, or (PhO)₃P], which were converted into the corresponding diethyl-, diphenyl-, or monophenylchloro-derivatives by Grignard reagents. The infrared spectra and a possible mechanism are discussed.

2

WE report a syntheses 1 and some properties of compounds² obtained by insertion of tin(II) halide into different cobalt carbonyl complexes containing cobaltcobalt bond. Similar complexes containing tin-cobalt bond were known; ³ they were prepared by the classical route ⁴ involving the use of an ionic derivative of a metal carbonyl and of an appropriate non-transition metal compound.

Preparation.—When a compound with general formula $[Co(CO)_{3}L]_{2}^{5}$ $[L = CO, Ph_{3}P, (PhO)_{3}P, Bu_{3}^{n}P]$ reacts with anhydrous tin(II) halide, the following reaction takes place:

$$[Co(CO)_3L]_2 + SnX_2 = [(CO)_3LCo]_2SnX_2 (X = CI, Br, or I)$$

The reaction was generally carried out in a polar solvent; when a non-polar solvent, such as benzene, was used, more time was required for stannous chloride than for stannous iodide, probably because the latter is soluble in benzene. Yields were generally very good when L was not CO; when $Co_2(CO)_8$ was employed, only a 20-40% yield was obtained, according to condition, and some gas was evolved during the reaction; this behaviour might be due to the instability of the [(CO)₄Co]₂SnX₂ compounds, or to side reactions, leading to untractable oily by-products. When $[Co(CO)_{3}L]_{2}$ was used, where L is a ligand such as R_3P or $(RO)_3P$, the steric configuration of the product according to infrared evidence was the same as that of the starting material, that is both trans for the trigonal bipyramidal configuration. The same reaction occurred and the same isomer was isolated when $[Co(CO)_{3}L_{2}][Co(CO)_{4}]$ was used instead of $[Co(CO)_3L]_2$. From the reaction course it appears that the former yields the latter on heating,⁵ losing carbon monoxide; this then reacts with tin(II) chloride. Again the same trans-isomer was obtained when $[(CO)_{4}CO]_{2}SnX_{2}$ reacted with the appropriate ligand, X being either chloride or phenyl; Ph₃As and Ph₃Sb reacted similarly.

To obtain independent evidence for the suggested formula [(CO)₄Co]₂SnCl₂ was treated with phenylmagnesium bromide; the product ⁶ was identical with that obtained from $[Co(C_5H_5N)_6][Co(CO)_4]_2$ by the reaction

$$[Co(CO)_4]^- + Ph_2SnBr_2 = [(CO)Co_4]_2SnPh_2 + 2Br^-$$

This reaction is not convenient for obtaining compounds with Co-Sn bonds, since the alkyl- or aryl-tin bromoderivatives are remarkably less stable than the corresponding chlorides, oils often being obtained.³

The compounds obtained are decomposed by hydrogen chloride (even in ethanol) and by dilute nitric acid, but not by cold concentrated sulphuric acid. They react with Grignard reagents, affording the corresponding organic derivatives. In the case of $[(CO)_3(Bu^n_3P)Co]_2SnX_2$, with phenylmagnesium bromide only $[(CO)_3(Bu^n_3P)Co]_2SnXPh$ was obtained (X = Cl or Br), while ethylmagnesium bromide yielded the corresponding diethyl derivative. This behaviour may be due to the bulky cobalt-containing moiety which hinders the entrance of a second phenyl group; indeed rather unstable [(CO)₃(Buⁿ₃P)Co]₂SnPh₂ was obtained by reaction of $[(CO)_4Co]_2SnPh_2$ with the ligand.

Infrared Spectra.-These were recorded for all the compounds synthesised (Table). All showed strong carbonyl stretching absorption. According to the lower π -acceptor properties of Group V donors, their intro-

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³ W. Hieber and H. Breu, Chem. Ber., 1957, 90, 1270.

⁴ F. Hein, H. Pobloth, and E. Heuser, Z. anorg. Chem., 1941, 248, 84; R. D. Gorsich, J. Amer. Chem. Soc., 1962, 84, 2486.
⁵ W. Hieber and W. Freyer, Chem. Ber., 1960, 93, 462; A.

Sacco and M. Freni, Ann. Chim. (Rome), 1958, **48**, 218; A. Davison, J. A. McCleverty, and G. Wilkinson, J. Chem. Soc., 1965, 3890.

⁶ F. Hein and W. Jehn, Annalen, 1965, 684, 4.

duction in place of CO shifts the adsorption to lower wavenumber. When the compound

[(CO)₃(Buⁿ₃P)Co]₂SnX₂ is considered the values of $v_{C=0}$ are lowered by replacing chloride with ethyl and phenyl group, as expected for an inductive effect.

According to the local symmetry in [(CO)₃LCo]₂SnCl₂, a bipyramidal trigonal disposition around each cobalt atom should require two infrared bands $(A_1 + E)$ for a trans- and three bands for a cis-configuration (C_{3v}) and C_s , respectively). According to the true molecular symmetry, if the ligands are considered as points, five infrared bands are required for a trans- and six for a cisconfiguration: C_{2v} and C_2 configuration, respectively,

Compound	Solv
$[(CO)_4Co]_2SnCl_2$	Hex
$[(CO)_4Co]_2SnBr_2$	Hex
$[(CO)_4Co]_2SnI_2$	Hex
······	CHC
$[(CO)_{a}Co]_{a}SnPh_{a}$	Hexa
$[(CO)], (Bu^n, P)Co], SnCl, \dots$	Hexa
$[(CO)]_{a}(Bu^{n},P)Co]_{a}SnBr_{a}$	CHC
$(CO)_{a}(Bu^{n}_{a}P)Co]_{a}SnEt_{a}$	Hex
$[(CO)]$, $(Bu^n, P)Co]$, $SnPh$,	Hex
[(CO)],(Bu ⁿ ,P)Co],SnClPh	Hex
(CO), (Bu ⁿ , P)Co], SnBrPh	Hex
······································	CHC
$[(CO)_{a}(Ph_{a}P)Co]_{a}SnCl_{a}$	CH,
(CO), (Ph, As)Co], SnCl,	CH.
(CO), (Ph,Sb)Co], SnCl,	CH.
[(CO) ₃ ({PhO} ₃ P)Co] ₂ SnCl ₂	CH ₂

if a symmetric disposition around the Cl-Sn-Cl plane is assumed. The observed bands in some solvents are generally more numerous than required by local but less than required by total symmetry.

In chlorinated solvents the presence of only a stronger and a weaker band is evidence for trans-substitution in our compounds. However, when a non-polar solvent, such as n-hexane, can be used, the number of bands increases. This increase cannot be explained as for some pentacarbonylalkylrhenium compounds 7 where the ligand lacks axial symmetry; besides, more bands are present than can be accounted for in this way. The increase cannot be explained by conformation isomerism,⁸ for the same reason; besides, the 2000 cm.⁻¹ region is unaffected by a temperature change from 15 to 73° (n-hexane solution) for $[Co(CO)_3(Bu_3^nP)]_2$ and

 $[(CO)_3(Bu_3^nP)Co]_2SnCl_2.$

Similar considerations apply to the [(CO)₄Co]₂SnX₂ compounds, which require seven infrared-active transitions in the carbonyl region in the case of total symmetry (C_{2v}) ; only three or four bands are required by a local

⁷ J. B. Wilford and F. G. A. Stone, J. Organometallic Chem., 1964, 2, 371.

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bipyramidal trigonal arrangement, with the tin atom on the top or on the basal plane, respectively. Here six (X = Ph or I) or five bands (X = Cl or Br) can be observed.

It seems then that some caution ought to be employed when assigning bands of compounds with metal-tin bonds since local symmetry, usually assumed, is not enough, though all the bands required by total symmetry could not be observed in our experimental conditions.

DISCUSSION

A reaction course through breakage of cobalt-cobalt bond and formation of free radicals is rather unlikely,

Solvent	Carbonyl stretching frequencies (cm1)
Hexane	2109w, 2102s, 2058m, 2043s, 2023m
Hexane	2112w, 2092s, 2057m, 2042s, 2022m
Hexane,	2100m, 2098s, 2080m, 2046m, 2035s, 2018m
CHCl ₃	2103m, 2089s, 2082m, 2028s
Hexane	2098m, 2081s, 2029m, 2020s, 2011s, 1998m
Hexane	2020w, 1979s, 1954w
CHCl ₃	2016w, 1967s (broad)
Hexane	1991w, 1951s, sh, 1943s, 1931m, sh
Hexane	2002w, 1961s, 1951s, 1936m
Hexane	2008w, 1970s, 1962s, 1948s, 1933m
Hexane	2001w, 1968s, 1961s, 1946m, 1925sh
CHCl3	2005w, 1965s (asymmetric)
CH ₂ Cl ₂	2025w, 1988s
CH ₂ Cl ₂	2030w, 1989s
CH ₂ Cl ₂	2025w, 1987s
CH2Cl2	2044w, 2006s

though not completely excluded by the rather controversial existence of [Co(CO)₄] radicals.^{9,10} However, the observation¹¹ that paramagnetic

 $[Co(Ph_2P \cdot CH_2 \cdot CH_2 \cdot PPh_2)_2]$ reacts with tin(II) chloride yielding tin and no compound with tin-cobalt bond seems to exclude a radical intermediate; this view is supported by the retention of steric configuration during our reactions. A second reaction course may involve the donor properties ¹² of tin(II) halide, as such (in hydrocarbon solvent), or as an intermediate anion (SnX_3^{-}) , or as SnX₂ solvated. Indeed tin(II) chloride reacts rather smoothly with compounds liable to accept another donor molecule, such as amine, or water to yield, e.g., $[(\pi - C_5 H_5) Fe(H_2 O)(CO)_2]^+$ ¹³ or

 $[Co(CO)_4(piperidine)][Co(CO)_4].$ ¹⁴ The first step may then be a nucleophilic attack.

Another possible reaction course involves an electrophilic attack by a carbenoid entity ¹⁵ such as tin(II) halide, indium(I) bromide,¹⁶ germanium(II) iodide,¹⁷ metallic zinc,¹⁸ cadmium, and mercury.^{18,19} This might be our case because the reactivity of $[Co(CO)_3L]_2$ with tin(II) chloride decreases in the order $Bu_3^n P > Ph_3 P >$ $(PhO)_{2}P$. The π -accepting ability goes in the order

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- ¹⁷ D. Harbourne and F. G. A. Stone, ref. 40 in F. G. A. Stone,
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- ¹⁹ S. V. Dighe and M. Orchin, J. Amer. Chem. Soc., 1964, 86, 3895; Inorg. Chem., 1962, 1, 965.

 $(PhO)_{3}P > Ph_{3}P > Bun_{3}P$ and is parallel to the increasing electronic density on the cobalt atoms, as shown by the infrared carbonyl stretching frequencies and by the very different acidity constants of HCo(CO)₃L.²⁰ Indeed the colour change of the solution from reddish to vellow, which is evidence for complete transformation of the reagents, is detected earlier with the Bun₃P derivative than in the other derivatives (8 hr. at 25° ; 2 and



Flow-sheet of the reactions

4 days under reflux, respectively). No choice between the electrophilic and nucleophilic attack by SnX₂ can yet be made for $[Co(CO)_4]_2$ and for $[\pi-C_5H_5Fe(CO)_2]_2$, since in solution they exist as an equilibrium of forms with and without carbonyl bridges.^{9,21} Further preparative and kinetic ²² work is in progress.

EXPERIMENTAL

Infrared spectra were taken on Perkin-Elmer models 137 and 237 instruments; molecular weights were measured on a Mechrolab osmometer. All reactions were carried out initially in nitrogen, and evaporation under vacuum (water-pump) unless stated otherwise.

The compounds are orange-yellow or yellow diamagnetic non-conducting solids, generally soluble in organic solvents but not in water. Their stability, e.g., to air during storage, seems to be good (a few months) for [(CO)₃LCo]₂SnCl₂ compounds; the presence of tin-bonded phenyl groups or of four carbonyl groups attached to cobalt gives less stable compounds.

Dichlorobis(tetracarbonylcobalt)tin.-To anhydrous tin(II) chloride (3.5 g.) in diethyl ether (35 ml.), octacarbonyldicobalt (5.6 g.) was added in a sealed tube. This was subjected to 100 atmospheres' pressure of carbon monoxide in an autoclave, the sealed tube was broken, and the whole shaken for 4.5 hr. After being kept overnight, the contents plus the ether washings were evaporated to dryness. The orange-red crystalline residue was extracted with boiling light petroleum (b. p. 60-80°) and the filtered solution, on cooling to -18° , gave the orange compound, m. p. 112° (decomp.) (3.85 g.) (Found: C, 18.0; H, 0.0; Cl, 13.2; Co, 21·2; CO, 42·1%; M (CHCl₃), 552. C₈Cl₂Co₂O₈Sn requires C, 18.2; H, 0.0; Cl, 13.3; Co, 22.2; CO, 42.2%; M, 531.6). The compound is very soluble in organic solvents, non-conducting in nitrobenzene and unstable in alcoholic solution.

J. Chem. Soc. (A), 1966

The reaction was run also in acetic acid, diethyl ether, or tetrahydrofuran solution, under nitrogen (1 atm.), giving the same compound in 20% yield (based on octacarbonyldicobalt). When benzene was the solvent, four days' stirring under nitrogen (1 atm.) was required, yields being about 20%.

Dibromobis(tetracarbonylcobalt)tin.—Tin(II) bromide (2.60 g.) was added to an ether solution (50 ml.) of octacarbonyldicobalt (2.84 g.). Gas was evolved; the solution became deep orange; after 20 minutes some orange precipitate was formed. After 3 hr. the solvent was removed under vacuum and the dark brown residue was extracted with boiling light petroleum. When the filtered solution was cooled to -80° , brown-orange needles (1.13 g.) separated, m. p. 115° (decomp.) (Found: C, 15.0; H, 0.1; CO, 35.0. C₈Br₂Co₂O₈Sn requires C, 15.5; H, 0.0; CO, 36.1%). The compound is very soluble in all organic solvents.

Di-iodobis(tetracarbonylcobalt)tin.-A benzene solution (30 ml.) of octacarbonyldicobalt (1.96 g.) was treated at room temperature with tin(II) iodide (2.20 g.); gas was evolved and some precipitate was formed. After 3 days, benzene was removed in vacuo, the residue was extracted with light petroleum, and the filtered extract cooled to -80° to yield brick-red crystals of the compound (1.05 g.), m. p. ca. 105° (decomp.) (Found: C, 13·3; H, 0·2; Co, 16·0; CO, 31.0. $C_8Co_2I_2O_8Sn$ requires C, 13.5; H, 0.0; Co, 16.5; CO, 31.3%). The compound is very soluble in all organic solvents; but either in solution or in the solid state it is not stable for long. The compound was similarly obtained when tetrahydrofuran was used for benzene.

Bis(tetracarbonyl)diphenyltin.-(a) To the orange solution of dichlorobis(tetracarbonylcobalt)tin (1.00 g.) in ether (60 ml.) a solution of phenylmagnesium bromide in ether (8 ml.; 15.6 g./100 ml.) was added; the now dark brown solution was left overnight. The residue from the evaporated solution crystallised from ethanol as straw-yellow crystals (0.31 g.), decomposing from ca. 140° (Found: C, 39·3; H, 1·8. $C_{20}H_{10}Co_2O_8Sn$ requires C, 39·4; H, 1·7%). It is soluble in organic solvents, but its solutions are not stable for long.

(b) A solution of octacarbonyldicobalt (1.48 g) in ether (10 ml.) was treated with pyridine (2 ml.) in the same solvent (20 ml.) with stirring. The colourless ether layer was decanted, the residual brown paste was washed with ether $(2 \times 30 \text{ ml.})$ and light petroleum (20 ml.); then it was covered with light petroleum (50 ml.) and dichlorodiphenyltin(IV) (1.40 g.) in the same solvent (20 ml.) was added. The brown layer disappeared slowly, and then the orange ether solution was washed thrice with water, dried (Na₂SO₄), and evaporated to 9-10 ml. On cooling to -80° ochre-yellow crystals (0.74 g.) separated, decomposing from ca. 140°, with the same infrared spectrum as the compound described above.

Hexacarbonylbis(tri-n-butylphosphine)dicobalt. A solution of octacarbonyldicobalt (3.75 g.) in benzene (30 ml.) was treated with tri-n-butylphosphine (4.46 g.) in the same solvent (10 ml.); gas was evolved immediately and during the following 0.5 hour's heating under reflux. After removal of the solvent in vacuo, the brownish-black crystalline residue was extracted with CH₂Cl₂, the extract evaporated again, and crystallised from methanol (-80°) , yielding

 ²⁰ W. Hieber and E. Lindner, Chem. Ber., 1961, 94, 1417.
²¹ E. O. Fischer, Angew. Chem., 1965, 76, 724; G. Bor, Spectrochim. Acta, 1963, 19, 2065.

²² A. J. Poe, personal communication.

the compound as dark red-violet crystals (3·11 g.), m. p. 108° (decomp. at 110°) (Found: C, 51·3; H, 7·6; CO, $24\cdot3^{\circ}_{0}$; M (benzene), 680. $C_{30}H_{54}Co_2O_6P_2$ requires C, 52·2; H, 7·9; CO, $24\cdot3^{\circ}_{0}$; M, 690·6).

Dichlorobis(tricarbonyltri-n-butylphosphinecobalt)tin.-(a)An acetone solution (80 ml.) of dark red-violet hexacarbonylbis(tri-n-butylphosphine)dicobalt (1.00 g.), and anhydrous tin(11) chloride (0.36 g.) was kept for 8 hr. The orangeyellow solution was evaporated to dryness, the residue was extracted with CH₂Cl₂, the extract evaporated to dryness, and treated with light petroleum $(2 \times 5 \text{ ml.})$; the ochrevellow solid was filtered off and vacuum-dried (1.21 g.), m. p. 91-92°, golden-yellow rhombic crystals (0.85 g.), m. p. 96-97° (from methanol) (Found: C, 41.5; H, 6.1; Cl, 8.2; Co, 13.2; P, 6.0; CO, 19.1%; M (CHCl₃), 881. $C_{30}H_{54}Cl_2Co_2O_6P_2Sn$ requires C, 41.0; H, 6.2; Cl, 8.1; Co, 13.4; P, 7.0; CO, 19.1%; M, 880.2). It is soluble in organic solvents, but slightly soluble in light petroleum, non-conducting in nitrobenzene; its solutions are fairly stable in air.

(b) The same compound was prepared similarly using yellow tricarbonylbis(tri-n-butylphosphine)cobalt tetracarbonyl cobalt in place of red-violet hexacarbonylbis(trin-butylphosphine)dicobalt. CO was evolved during the

n-butylphosphine)dicobalt. CO was evolved during the reaction, while the colour changed from orange to blood-red, and then again to orange-yellow.

Dibromobis(tricarbonyltri-n-butylphosphinecobalt)tin.---

As for the dichloro-compound, the *compound* was crystallised from ethanol to yield 61% (based on cobalt compound) of orange-yellow leaflets, m. p. 128—130° (Found: C, 36.9; H, 5.6. $C_{30}H_{54}Br_2Co_2O_6P_2Sn$ requires C, 37.2; H, 5.6%). It is sparingly soluble in light petroleum.

Bis(tricarbonyltri-n-butylphosphinecobalt)diethyltin.— An ether solution (30 ml.) of dichlorobis(tricarbonyltri-n-butylphosphinecobalt)tin (0.53 g.) was treated with excess of ethereal ethylmagnesium iodide. Some precipitate was immediately formed. After being kept overnight, water was added, the ether layer separated and dried (CaCl₂), and the brownish-yellow residue left after evaporation was crystallised from ethanol and then from ethanol-water to yield yellow-green crystals of the *compound* (0.36 g.), m. p. 77° (Found: C, 47.3; H, 7.4; Co, 14.3; CO, 19.3. $C_{34}H_{64}Co_2O_6P_2Sn$ requires C, 47.1; H, 7.4; Co, 13.6; CO, 19.4%). The compound is soluble in organic solvents, including hexane. The same compound was obtained similarly from the corresponding bromide.

Chlorobis(tricarbonyltri-n-butylphosphinecobalt)phenyltin. —As in the preceding case, use of phenylmagnesium bromide gave canary-yellow crystals, m. p. 105—106°, in 76% yield after crystallisation from ethanol (Found: C, 47.1; H, 6.7; Cl, 4.1; CO, 19.1. $C_{36}H_{59}$ ClCo₂O₆P₂Sn requires C, 46.9; H, 6.5; Cl, 3.9; CO, 18.2%).

Bromobis(tricarbonyltri-n-butylphosphinecobalt)phenyltin. —Prepared as for the corresponding chloride, it formed straw-yellow crystals, m. p. 110—112°, from ethanol (Found: C, 45.5; H, 6.4; CO, 17.5. $C_{36}H_{59}BrCo_2O_6P_2Sn$ requires C, 44.8; H, 6.2; CO, 17.4%).

Bis(tricarbonyltri-n-butylphosphinecobalt)diphenyltin. A benzene solution (60 ml.) of crude bis(tetracarbonylcobalt)diphenyltin [from Co₂(CO)₈ (2.88 g.), pyridine (4.5 ml.), ether (50 ml.), and (C₆H₅)₂SnBr₂ (2.51 g.)] was treated with tri-n-butylphosphine (2.52 g.). After 3 hr. the orange solution was evaporated to dryness, the residue dissolved in benzene, and heated under reflux for 3 hr. The filtered solution was evaporated to dryness and, on standing, brownish-yellow crystals were formed which, crystallised twice from ethanol, yielded the dirty yellow *compound*, m. p. 121–122° (Found: C, 52·1; H, 6·7; Co, 11·6; CO, 17·2. $C_{42}H_{64}Co_2O_6P_2Sn$ requires C, 52·4; H, 6·7; Co, 12·2; CO, 17·4%).

Dichlorobis(tricarbonyltriphenylphosphinecobalt)tin.—(a) A mixture (0.48 g.) of sym-hexacarbonylbis(triphenylphosphine)cobalt and tricarbonylbis(triphenylphosphine)cobalt tetracarbonylcobaltate [both from $Co_2(CO)_8$ and PPh_3] was suspended in acetone (40 ml.) containing tin(II) chloride (0.14 g.). After two days on a bath at *ca*. 65°, the solution was filtered and evaporated to dryness; the residue was extracted with methylene chloride, the extract evaporated to dryness (0.39 g.), and crystallised from the same solvent (-80°) as yellow crystals, m. p. 217° (decomp.) (Found: C, 50.3; H, 3.1; Cl, 7.7; CO, 15.6. $C_{42}H_{30}Cl_2Co_2O_6P_2Sn$ requires C, 50.4; H, 3.0; Cl, 7.1; CO, 16.8%). It is sparingly soluble in benzene.

(b) When benzene solutions of dichlorobis(tetracarbonylcobalt)tin and excess of triphenylphosphine were heated together for 15 minutes a compound was obtained which was found to be the same as that obtained by method (a)according to m. p., mixed m. p., and infrared spectrum.

Dichlorobis(tricarbonyltriphenylarsinecobalt)tin.—Dichlorobis(tetracarbonylcobalt)tin (0.20 g.) and triphenylarsine (0.24 g.) were refluxed for a few hours in benzene. The residue after evaporation was precipitated from benzene with light petroleum to yield the orange-red compound (0.18 g.), m. p. 180° (Found: C, 45.6; H, 2.8; Cl, 6.5; Co, 10.6; CO, 14.6. $C_{42}H_{30}As_2Cl_2Co_2O_6Sn$ requires C, 46.3; H, 2.8; Cl, 6.5; Co, 10.8; CO, 14.5%).

Dichlorobis(tricarbonyltriphenylstibinecobalt)tin.—This was similarly prepared and purified, forming brick-red crystals, m. p. 188° (Found: C, 42.6; H, 2.5; Cl, 6.0; CO, 14.5. $C_{42}H_{30}Cl_2Co_2O_6Sb_2Sn$ requires C, 42.7; H, 2.5; Cl, 6.0; CO, 14.3%).

Dichlorobis(tricarbonyltriphenylphosphitecobalt)tin.—(a) This was similarly prepared, but crystallised from ethyl ether-light petroleum and then from ethanol, to yield yellow needles, m. p. 130—133° (Found: C, 46.4; H, 2.9; Cl, 6.5; Co, 10.7; O, 17.1; CO, 14.2%; M (CHCl₃), 1034. C₄₂H₃₀Cl₂Co₂O₁₂P₂Sn requires C, 46.1; H, 2.8; Cl, 6.5; Co, 10.7; O, 17.5; CO, 15.3%; M, 1096).

(b) Hexacarbonylbis(triphenylphosphite)dicobalt (0.31 g.), and anhydrous tin(II) chloride (0.13 g.) in acetone (60 ml.) were refluxed for four days. The residue after evaporation was extracted with CH_2Cl_2 , the extract washed with dilute Na_2CO_3 , and dried $(CaCl_2)$; the evaporated residue was treated with ligroin to yield a yellow solid, identified as the compound from (a) by mixed m. p. and infrared spectrum.

We thank Consiglio Nazionale delle Ricerche for financial support; one of us (D. M.) thanks Società Montecatini-Istituto Donegani, and another (S. C.) Accademia dei Lincei for scholarships.

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UNIVERSITÀ DI MILANO, ITALY. [6/167 Received, February 8th, 1966]