391. *Bridged Complexes of Platinum*(II) *containing Chelate Ligands.*

By STANLEY E. LIVINGSTONE.

Bridged complexes of Pt^{II} with the chelate ligands 3-dimethylarsinopropane-1-thiol and 3-ethylthiopropane-1-thiol are described. Proof that the platinum atoms in these complexes are bridged by sulphur atoms is furnished by the isolation of an ammino-complex chloride and perchlorate, in which the bridged structure occurs in the cation. The chloride can be reconverted into the original bridged complex on heating, with loss of ammonia. Binuclear complexes containing Pt^{II} and Hg^{II}, and Pt^{II} and Pd^{II}, are also described. Some of the platinum bridged complexes crystallize from acetone and benzene with solvent of crystallization.

BRIDGED complexes of palladium with a number of different chelates have been described in the preceding paper.[1] In this investigation, the chelate ligands, 3-dimethylarsinopropane-1-thiol, $Me_2As\cdot[CH_2]_3\cdot SH$, and 3-ethylthiopropane-1-thiol $EtS\cdot[CH_2]_3\cdot SH$, were used to prepare binuclear platinum complexes. The compounds obtained are listed in Table 1.

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[1] Livingstone, preceding paper.

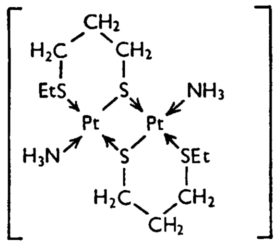
TABLE 1. *Binuclear platinum complexes.*

Chelating compound	Compound	Halogen
$Me_2As·[CH_2]_3·SH$	I	Cl
$EtS·[CH_2]_3·SH$	II	Cl
$EtS·[CH_2]_3·SH$	III	Br
,,	IV	I

The compounds (I) and (II) can be prepared by heating the dry platinum inner complex, $Pt(Chel)_2$ (where ChelH = 3-dimethylarsinopropane-1-thiol or 3-ethylthiopropane-1-thiol) with platinous chloride to temperatures in the range 140—160°. This method was used by Chatt [2] to prepare a platinum bridged compound containing tri-*n*-propylphosphine. When the chloro-complex (II) is dissolved in hot alcohol, and ammonium bromide is added, the resulting solution, on being concentrated, deposits bright yellow crystals of the bromo-complex (III), which can be recrystallized from alcohol. An *X*-ray powder photograph of this compound shows that it is isomorphous with the corresponding bromo-palladium complex.[1] The iodo-complex (IV) is obtained by treating a benzene solution of the chloro-complex (II) with an ethereal solution of methylmagnesium iodide. The chloro- and iodo-complexes recrystallize from benzene with one-half, and one molecule, respectively, of benzene of crystallization.

If a benzene solution of the chloro-complex (II) is refluxed with *p*-toluidine, the original compound (II) can be recovered unchanged. This is in contrast to the chloro-bridged platinum complex of tri-*n*-propylphosphine which reacts with *p*-toluidine in cold benzene solution to give *trans-p*-toluidine-tri-*n*-propylphosphinedichloroplatinum.[2] This suggests that the two platinum atoms in the complex are bridged by sulphur and not halogen atoms. Further evidence in support of this is given by the action of ammonia on the chloro-compound (II). If ammonia is passed into a benzene solution of (II) the solution slowly loses its yellow colour and deposits a white precipitate of diamminodi-(3-ethylthiopropane-1-μ-thiolo)diplatinum chloride (V) which contains two-thirds of a molecule of benzene of crystallization. The compound is soluble in water and has a molecular conductance expected for a bi-univalent electrolyte. If the compound is heated to 140°, the benzene and ammonia are driven off and the compound is converted into the original yellow chloro-complex (II). Treatment of an aqueous solution of the chloride (V) with excess of sodium perchlorate precipitates the perchlorate (V). The structure of these compounds is undoubtedly that shown in (V).

(V)



Cl_2 [or $(ClO_4)_2$]

However, the palladium analogue of (II), dichlorodi-(3-ethylthiopropane-1-μ-thiolo)-dipalladium (VI) [1] reacts differently. When ammonia is passed into a benzene solution of (VI), tetramminopalladous chloride is precipitated. Continued passage of ammonia fails to remove the orange colour from the solution. Evaporation of the benzene from the filtrate leaves a reddish-brown oil. The compound, $Pd\{EtS·[CH_2]_3·S\}_2$, has never been isolated in the pure state.[3] The reaction can therefore be represented :

$$\{EtS·[CH_2]_3·S\}_2Pd_2Cl_2 + 4NH_3 \longrightarrow [Pd(NH_3)_4]Cl_2 + Pd\{EtS·[CH_2]_3·S\}_2$$

Similarly, treatment of a benzene solution of (VI) with ethylamine results in precipitation of tetrakisethylaminopalladous chloride dihydrate (VII). However, these reactions do not invalidate the assumption of thiolo-bridges in the palladium complex (VI), since

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[2] Chatt, *J.*, 1950, 2301.
[3] Livingstone, *J.*, 1956, 437.

the chloro-bridged palladium complex of (*o*-methylthio)benzoic acid[4] reacts with pyridine in aqueous solution to yield $[\mathrm{Pd\,py_4}]^{++}$ (py = pyridine). Acidification of the solution yields a precipitate from which dichlorodipyridinepalladium and *o*-(methylthio)benzoic acid can be isolated:

$$\begin{array}{rcl} (\mathrm{MeS{\cdot}C_6H_4{\cdot}CO_2})_2\mathrm{Pd_2Cl_2} + 8\mathrm{py} & \longrightarrow & [\mathrm{Pd\,py_4}]^{++} + 2\mathrm{Cl}^- + 2\mathrm{MeS{\cdot}C_6H_4{\cdot}CO_2}^- \\ & & \quad\quad\quad\quad \Big\downarrow \mathrm{HCl} \\ & & 2\mathrm{Pd\,py_2Cl_2} + 2\mathrm{MeS{\cdot}C_6H_4{\cdot}CO_2H} + 4\mathrm{py,HCl} \end{array}$$

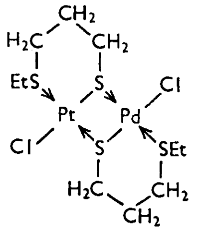
Other amines, such as ammonia, ethylenediamine, and propylenediamine, give a similar reaction with this compound.

Bridged complexes containing $\mathrm{Cd^{II}}$ and $\mathrm{Hg^{II}}$, $\mathrm{Pd^{II}}$ and $\mathrm{Hg^{II}}$, $\mathrm{Pd^{II}}$ and $\mathrm{Sn^{IV}}$, and $\mathrm{Hg^{II}}$ and $\mathrm{Sn^{IV}}$, have been reported by Mann *et al.*;[5, 6, 7] binuclear complexes containing $\mathrm{Pd^{II}}$ and $\mathrm{Hg^{II}}$, and $\mathrm{Pd^{II}}$ and $\mathrm{Cu^{II}}$, have been described.[8] By the use of 3-ethylthiopropane-1-thiol, it has been possible to isolate a series of bridged complexes containing one atom of platinum and one atom of palladium in the molecule. Also a bridged complex containing platinum and mercury has been obtained. These compounds are listed in Table 2. The structure of (IX) is as shown.

TABLE 2. *Binuclear complexes of* 3-*ethylthiopropane*-1-*thiol containing two different metal atoms.*

Compound	Metal atoms	Halogen	Colour	M. p.
VIII	Pt and Hg	Br	Pale cream	81°
IX	Pt and Pd	Cl	Yellowish-orange	227
X	Pt and Pd	Br	Deep orange	183
XI	Pt and Pd	I	Reddish-orange	206

These compounds are obtained by heating the platinum complex $\mathrm{Pt\{EtS{\cdot}[CH_2]_3{\cdot}S\}_2}$ with the appropriate anhydrous metallic halide, $\mathrm{MX_2}$, and extracting the cooled melt with acetone or benzene. The mercury complex (VIII) recrystallizes from acetone as pale yellow microcrystals, which are only sparingly soluble in acetone and benzene, but moderately soluble in nitrobenzene. The compounds (IX), (X), and (XI) recrystallize from benzene as beautiful orange crystals containing ½ mol. of benzene of crystallization, which is not lost *in vacuo* or even if the compound is heated to 100°. However, heating for one hour at 140° causes the crystal faces to lose their lustre and a loss in weight, corresponding to ½ mol. of benzene, occurs. The bromo-compound (X) recrystallizes from acetone with 1 mol. of acetone of crystallization, which is lost at 140°. These three compounds are soluble in benzene, chloroform, and nitrobenzene, but somewhat less so in acetone and alcohol. They are non-conductors in nitrobenzene solution.


(IX)

Confirmation that these compounds are not equimolar mixtures or mixed crystals of the platinum and the palladium bridged complexes is furnished by the absorption spectra of the three bromo-complexes. The curve obtained for the mean of the optical densities of the bromo-diplatinum complex (III) and the corresponding dipalladium complex[1] is not identical with the curve obtained for the bromo-platinum-palladium complex (X) (see Fig.).

Di-(3-ethylthiopropane-1-μ-thiolo)platinum was heated separately with anhydrous nickel bromide and cupric bromide in an attempt to prepare binuclear complexes containing $\mathrm{Pt^{II}}$ and $\mathrm{Ni^{II}}$, and $\mathrm{Pt^{II}}$ and $\mathrm{Cu^{II}}$, respectively. No compounds soluble in benzene, acetone, or nitrobenzene were obtained from the cooled melts. The question arises why $\mathrm{Pd^{II}}$,

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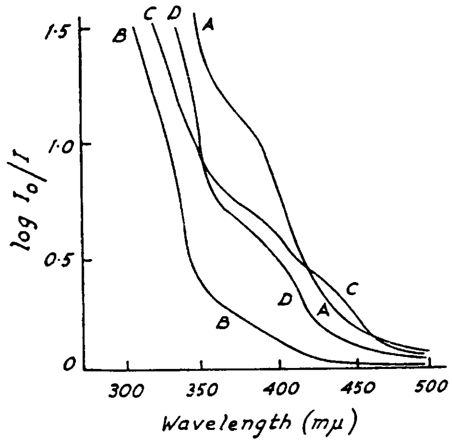
[4] Livingstone and Plowman, *J. Proc. Roy. Soc., New South Wales*, 1950, **84**, 188.
[5] Evans, Mann, Peiser, and Purdie, *J.*, 1940, 1209.
[6] Mann and Purdie, *J.*, 1940, 1230.
[7] Allison and Mann, *J.*, 1949, 2915.
[8] Livingstone and Plowman, *J. Proc. Roy. Soc., New South Wales*, 1951, **85**, 116.

which has a square planar configuration in complexes, and Hg^{II} (tetrahedral) form binuclear complexes containing Pt (planar), and Cu^{II} and Ni^{II}, both of which are planar, do not. It is unlikely that the configuration of the mercury atom is altered in these complexes. An *X*-ray powder photograph of the bromo-bridged palladium–mercury complex of *o*-(methylthio)-benzoic acid [8] shows that the compound is not isomorphous with the bromo-bridged di-palladium complex of this ligand. This is what would be expected if the mercury atom retained a tetrahedral configuration. The explanation appears to be that Pt^{II}, Pd^{II}, and Hg^{II} have the strongest affinity for thiols; hence they are the three elements most likely to form mixed polynuclear compounds containing sulphur bridges.



A, $(C_5H_{11}S_2)_2Pd_2Br_2$. *C*, $(C_5H_{11}S_2)_2PtPdBr_2$.
B, $(C_5H_{11}S_2)_2Pt_2Br_2$. *D*, $\frac{1}{2}$(A + B).

(2 × 10^{-4}M solutions in benzene)

EXPERIMENTAL

Dichlorodi-(3-dimethylarsinopropane-1-μ-thiolo)diplatinum (I).—Bis-(3-dimethylarsinopropane-1-thiolo)platinum (Livingstone [3]) (1·94 g.) and platinous chloride (0·95 g.) were ground together, placed in a test-tube, and heated in a paraffin-oil bath. At approximately 170° the mixture melted, then solidified to a dark greenish mass. After cooling, the product was finely ground, and extracted with boiling acetone. The insoluble material was collected and the pale yellowish-green filtrate concentrated to 20 ml. to yield pale yellow crystals of the bridged *complex* (I) (0·7 g.), which recrystallized from acetone (350 ml.) in pale yellow diamond-shaped crystals (0·46 g.), which are only sparingly soluble in nitrobenzene (Found: C, 14·9; H, 2·8; Cl, 8·5. $C_{10}H_{24}Cl_2S_2As_2Pt_2$ requires C, 14·7; H, 3·0; Cl, 8·7%). The crystals are unmolten at 310°.

Dichlorodi-(3-ethylthiopropane-1-μ-thiolo)diplatinum (II).—Bis-(3-ethylthiopropane-1-thiolo)platinum (2·7 g.) and platinous chloride (1·51 g.) were ground together and heated until molten (140—150°). After 1 min. at this temperature, the mixture solidified. The powdered product was extracted with boiling acetone. The undissolved residue was filtered off, and the filtrate concentrated to 150 ml. Yellow crystals (2·51 g.) of the pure *compound* (II) separated on cooling; it is readily soluble in benzene, nitrobenzene, and chloroform, somewhat less so in acetone and alcohol, and has m. p. 237° (without decomp.) (Found: C, 16·8; H, 3·2; Cl, 9·2; Pt, 53·3%; *M*, cryoscopically in 0·76% nitrobenzene solution, 825. $C_{10}H_{22}Cl_2S_4Pt_2$ requires C, 16·4; H, 3·0; Cl, 9·7; Pt, 53·3%; *M*, 732). The compound recrystallizes from benzene, as a bright yellow solvate, m. p. 237° (Found: Pt, 50·5. $C_{10}H_{22}Cl_2S_4Pt_2,\frac{1}{2}C_6H_6$ requires Pt, 50·6%).

Dibromodi-(3-ethylthiopropane-1-μ-thiolo)diplatinum (III).—The chloro-compound (II) (0·72 g.) was dissolved in hot alcohol (300 ml.), and ammonium bromide (1 g.) added. The pale yellow solution was kept at the b. p. for 1 hr., then concentrated to 100 ml. and, on cooling, yielded bright yellow crystals (0·68 g.) which were recrystallized from alcohol (100 ml.), containing ammonium bromide (1 g.), to give the pure *bromo-complex* (III) (0·60 g.), m. p. 202° (Found: C, 14·8; H, 2·9; Pt, 47·5%; *M*, cryoscopically in 0·68% nitrobenzene solution, 880. $C_{10}H_{22}Br_2S_4Pt_2$ requires C, 14·6; H, 2·7; Pt, 47·6%; *M*, 821).

Di-iododi-(3*-ethylthiopropane-*1*-μ-thiolo)diplatinum* (IV).—A solution of the chloro-compound (II) (1·2 g.) in benzene (150 ml.) was added to an ethereal solution (35 ml.) of methylmagnesium iodide [from magnesium (0·28 g.) and methyl iodide (1·6 g.)]. The mixture was kept at the reflux for 30 min., a deep orange colour developing; it was then cooled, and water (50 ml.) added. The benzene layer was separated, dried, and concentrated to 10 ml., whereupon it deposited deep yellow crystals (m. p. 208°) of the solvated *iodo-complex* (IV) [Found: C, 19·2; H, 2·8; Pt, 39·6; loss on heating to 150°, 7·5%; *M*, cryoscopically in 0·69% benzene solution, 800. $C_{10}H_{22}I_2S_4Pt_2,C_6H_6$ requires C, 19·3; H, 2·8; Pt, 39·3; loss of C_6H_6, 7·9%; *M* (unsolvated), 915].

Non-reaction of (II) *with* p*-Toluidine.*—The chloro-complex (II) (1 g.) was dissolved in hot benzene (60 ml.), and *p*-toluidine (0·3 g.) added to the solution, which was kept at the b. p. for 15 min.; on cooling, it deposited bright yellow crystals (m. p. 237°) of the original complex (II), containing benzene of crystallization (Found: Pt, 50·5%).

Reaction of (II) *with Ammonia.*—The chloro-compound (II) (1 g.) was dissolved in benzene (350 ml.), and ammonia passed into the yellow solution. Within 5 min. an amorphous white precipitate commenced to form and after 2 hr. precipitation was complete, the supernatant liquor being colourless. The mixture was heated and kept at the b. p. for 20 min., the precipitate becoming crystalline, while some of the product dissolved. Ammonia was passed in until the liquor again became colourless. After the precipitate was filtered off and washed with benzene, there remained colourless crystals (1 g.) of solvated *diamminodi-*(3*-ethylthiopropane-*1*-μ-thiolo)-diplatinum chloride* (V) (Found: N, 3·6; Cl, 8·8; Pt, 47·7; loss on heating to 140°, 9·8. $C_{10}H_{28}N_2Cl_2S_4Pt_2,\frac{2}{3}C_6H_6$ requires N, 3·4; Cl, 8·7; Pt, 47·7; loss of $2NH_3 + \frac{2}{3}C_6H_6$, 10·5%). When heated in a closed tube, the compound gave a strong smell of ammonia and a colourless condensate, leaving a yellow residue identified as the chloro-complex (II), m. p. 237°. The compound (V) in water had $\Lambda_{2000} = 225$ mho cm.$^{-2}$ at 25°.

The chloride (V) (0·3 g.) was dissolved in water (3 ml.) and treated with 10% sodium perchlorate solution, yielding a gummy white precipitate, which, when kept for several hours, became crystalline. The colourless crystals of *diamminodi-*(3*-ethylthiopropane-*1*-μ-thiolo)-diplatinum perchlorate* (as V) were filtered off, washed with a little water, and dried *in vacuo* (P_2O_5) (yield, 0·14 g.) (Found: N, 2·8; Pt, 43·5. $C_{10}H_{28}O_8N_2Cl_2S_4Pt_2$ requires N, 3·1; Pt, 43·7%). The perchlorate had $\Lambda_{2000} = 218$ mho cm.$^{-2}$ in water at 25°.

Reaction of Dichlorodi-(3*-ethylthiopropane-*1*-μ-thiolo)dipalladium* (VI) *with Ammonia.*—Ammonia was passed into a solution of the chloro-palladium complex (VI) (1 g.) in benzene (400 ml.). Precipitation started at once and was complete in 30 min. although the liquor was still orange. The precipitate of tetramminopalladous chloride was filtered off, and washed with hot benzene (yield 0·43 g.) (Found: Pd, 43·1. Calc. for $H_{12}N_4Cl_2Pd$: Pd, 43·3%). The filtrate, on evaporation, yielded a reddish-brown oil, which could not be induced to crystallize.

Reaction of (VI) *with Ethylamine.*—The palladium complex (VI) (1 g.) was dissolved in benzene and treated with ethylamine (5 ml. of 33% alcoholic solution). After standing for 2 hr. the precipitate was collected and dissolved in water (3 ml.) containing a few drops of ethylamine solution. Addition of acetone precipitated *tetrakisethylaminopalladous chloride dihydrate* (VII) (0·2 g.) (Found: Cl, 17·9; Pd, 26·8. $C_8H_{28}N_4Cl_2Pd,2H_2O$ requires Cl, 18·0; Pd, 27·0%). The compound loses water when heated to decomposition. It had $\Lambda_{1000} = 225$ mho cm.$^{-2}$ in water at 25°.

Reaction of Di-[o*-*(*methylthio*)*benzoato-μμ′-dichloro*]*dipalladium with Pyridine.*—The dihydrate of the complex (1·5 g.) was treated with water (50 ml.) and 10% aqueous pyridine (15 ml.) to give a pale yellow solution, which was acidified with hydrochloric acid, and the resulting pale cream precipitate filtered off. The product was warmed with acetone, and the pale yellow crystals of dichlorodipyridinepalladium were isolated (Found: Pd, 31·5. Calc. for $C_{10}H_{10}N_2Cl_2Pd$: Pd, 31·8%). *o*-(Methylthio)benzoic acid (m. p. 168°) was recovered from the acetone filtrate.

Dibromodi-(3*-ethylthiopropane-*1*-μ-thiolo*)*platinum-mercury* (VIII).—Bis-(3-ethylthiopropane-1-thiolo)platinum (1·0 g.) and mercuric bromide (0·78 g.) were ground together and heated. The mixture commenced to soften at 100°, became pasty at 115°, and melted at 140°. The deep yellow mass was then cooled, pulverized, and extracted with boiling acetone, most of the material remaining undissolved. The acetone extract was filtered and evaporated to 40 ml., and, on cooling, deposited the *platinum-mercury complex* (VIII) as a fine pale cream powder (0·21 g.), m. p. 81° (Found: C, 14·5; H, 2·7; Br, 18·9; Pt, 23·7%; *M*, cryoscopically in 0·31% nitrobenzene solution, 880. $C_{10}H_{22}Br_2S_4HgPt$ requires C, 14·5; H, 2·7; Br, 19·3; Pt, 23·6%; *M*, 826).

Dichlorodi-(3-*ethylthiopropane*-1-μ-*thiolo*)*platinum-palladium* (IX).—Bis-(3-ethylthiopropane-1-thiolo)platinum (1·0 g.) and palladous chloride (0·39 g.) were heated as for the platinum-mercury complex (VIII). After the mass had become molten, it was cooled and extracted with boiling benzene (80 ml.). The reddish-orange extract was filtered and evaporated to 10 ml. The solution, on cooling, deposited yellowish-orange prisms (0·53 g.) of the *complex* (IX), containing ½ mol. of benzene of crystallization which is lost at 140°; it had m. p. 227° (Found : C, 22·6; H, 3·5; Cl, 9·8; Pd, 14·9; Pt, 29·5; Pd + Pt, 44·4; loss on heating, 5·6. $C_{10}H_{22}Cl_2S_4PdPt,\frac{1}{2}C_6H_6$ requires C, 22·9; H, 3·7; Cl, 10·4; Pd, 15·6; Pt, 28·6; Pd + Pt, 44·2; loss of $\frac{1}{2}C_6H_6$, 5·8%). The molecular weight was determined on the unsolvated material (dried at 140°) cryoscopically in 0·42% nitrobenzene solution (Found : *M*, 630. Required : *M*, 641).

Dibromodi-(3-*ethylthiopropane*-1-μ-*thiolo*)*platinum-palladium* (X).—Di-(3-ethylthiopropane-1-thiolo)platinum (1·5 g.) and palladous bromide (0·86 g.) were heated together as described for the mercury complex (VIII). The cooled melt was treated with boiling benzene (120 ml.); a red solution resulted, leaving only a small insoluble residue. The filtered benzene solution, after being concentrated to 35 ml., yielded deep orange prisms (1·64 g.) of the bridged *complex* (X), containing solvent of crystallization, which is not lost *in vacuo*. When the compound was heated to 100° for 1 hr., no loss in weight occurred, but heating at 140° for 1 hr. caused the crystal faces to become dull and the benzene to be driven off; the compound melted at 184° (Found : C, 20·5; H, 3·4; Br, 20·7; Pd, 13·9; Pt, 25·3; loss on heating to 140°, 5·04%; *M*, on sample dried at 140°, cryoscopically in 0·85% nitrobenzene solution, 730; cryoscopically in 0·37% benzene solution, 650. $C_{10}H_{22}Br_2S_4PdPt,\frac{1}{2}C_6H_6$ requires C, 20·2; H, 3·3; Br, 20·7; Pd, 13·8; Pt, 25·3; $\frac{1}{2}C_6H_6$, 5·06%; *M*, unsolvated compound, 732). The *compound* is soluble in acetone from which it recrystallizes as thin orange needles, containing solvent of crystallization, which is lost on heating to 140° for 1 hr. (Found : C, 18·9; H, 3·3; Pt + Pd, 38·2; loss on heating, 6·4. $C_{10}H_{22}Br_2S_4PdPt,C_3H_6O$ requires C, 19·7; H, 3·6; Pt + Pd, 38·2; loss of C_3H_6O, 7·3%). The conductivity of a 0·001M-solution of the compound in nitrobenzene was measured at 25° : $\Lambda_{1000} = 0·02$ mho cm.$^{-2}$.

Di-iododi-(3-*ethylthiopropane*-1-μ-*thiolo*)*platinum-palladium* (XI).—Di-(3-ethylthiopropane-1-thiolo)platinum (1·0 g.) and palladous iodide (0·78 g.) were ground together and heated. The mixture began to soften at 110°, became pasty at 120°, and resolidified at 150°. The product was then cooled, ground to a powder, and extracted with boiling benzene (100 ml.). The deep red benzene solution was filtered, and, on evaporation to 10 ml., yielded thin reddish-orange prisms (1·30 g.) of the *complex* (XI) (Found : Pt + Pd, 34·6. $C_{10}H_{22}I_2S_4PdPt,\frac{1}{2}C_6H_6$ requires Pt + Pd, 34·9%). The benzene of crystallisation was driven off by heating the compound for 1 hr. at 145°, the m. p. then being 206° (Found : C, 14·8; H, 2·4; I, 31·0; Pd, 12·9; Pt, 23·7%; *M*, cryoscopically in 0·48% nitrobenzene solution, 900; in 0·41% benzene solution, 690. $C_{10}H_{22}I_2S_4PdPt$ requires C, 14·5; H, 2·7; I, 30·7; Pd, 12·9; Pt, 23·6%; *M*, 826). A solution of the complex in nitrobenzene at 25° had $\Lambda_{1000} = 0·30$ mho cm.$^{-2}$.

X-*Ray Photographs*.—The diffraction patterns were recorded with Cu-$K_{\alpha\ 1,2}$ radiation and a " Unicam " 9-cm. powder camera.

The author is indebted to a Referee for helpful criticism, to Dr. C. M. Harris for some useful suggestions, to Dr. E. Challen for microanalyses for carbon, hydrogen, and nitrogen, and to Mr. N. C. Stephenson for taking and interpreting the *X*-ray photographs.

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[*Received, June* 16*th*, 1955.]