Cl_2 had formed). The substance was washed successively with oxygen-free anhydrous ethylenediamine, absolute alcohol and ether and dried under an infrared lamp. It was completely decomposed by dilute acids to give the usual pink solutions characteristic of Co^{2+} ion; C, 20.81; H, 8.09; N, 24.28. Calcd. for $[\mathrm{Coen}_3]\mathrm{Cl}_2 \cdot 2\mathrm{H}_2\mathrm{O}$: Found: C, 20.93; H, 7.88; N, 24.29.

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CANBERRA, AUSTRALIA

[CONTRIBUTION FROM THE JOHN CURTIN SCHOOL OF MEDICAL RESEARCH, AUSTRALIAN NATIONAL UNIVERSITY]

Stereospecific Influences in Metal Complexes Containing Optically Active Ligands. Part IV. The Preparation and Resolution of "Mixed" Tris-(diamine)-platinum(IV) Complexes

BY FRANCIS P. DWYER AND ALAN M. SARGESON

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Bis-(ethylenediamine)-*l*-propylenediamine-platinum(IV), bis-(*l*-propylenediamine)-ethylenediamine-platinum(IV) and bis-(*d*-propylenediamine)-*l*-propylenediamine-platinum(IV) chlorides have been prepared in dimethylformamide solution by the reactions between tetrachloro-(*l*-propylenediamine)-platinum(IV) or tetrachloro-(ethylenediamine)-platinum(IV) and either ethylenediamine or *d*-propylenediamine. The optical isomers of each compound, separated through the *d* and *l*-tartrates, showed no evidence of disproportionation or racemization in boiling aqueous solution.

In previous papers in this series[1] it has been proposed that the failure of workers to isolate "mixed" tris-(diamine) complexes such as *d*-propylenediaminebis-(*l*-propylenediamine)-platinum(IV) and -cobalt(III) chlorides is not due to the inherent instability of the complexes[2-4] but to the establishment of equilibrium among all the possible species during the preparative reactions. As a result, the thermodynamically more favored complexes such as $[\mathrm{Pt}\ l\text{-pn}_3]\mathrm{Cl}_3$ and $[\mathrm{Pt}\ d\text{-pn}_3]\mathrm{Cl}_4$ which predominate in the equilibrium mixture have been isolated and the small amount of the "mixed" tris-(diamine) complex has been overlooked. In general it appears that equilibrium is attained either through a 7- or 8- covalent transition state or through small catalytic amounts of the Co(II) and Pt(II) complexes.

The "mixed" tris-(diamines) $[\mathrm{Pten}_2\mathrm{pn}]\mathrm{Cl}_4$, $[\mathrm{Pt}\text{-}\mathrm{en}\ \mathrm{pn}_2]\mathrm{Cl}_4$ and $[\mathrm{Pt}\ d\text{-pn}_2.l\text{-pn})\mathrm{Cl}_4$ have now been prepared by the reaction between tetrachloroethylenediamine or tetrachloropropylenediamine-platinum(IV) and the appropriate diamine base in dimethylformamide at 20–25°. The "mixed" tris-(diamine) complexes were sparingly soluble in dimethylformamide and separated immediately on formation.

$$[\mathrm{Pt}\ l\text{-pn}\ \mathrm{Cl}_4] + 2\mathrm{en} = \mathrm{DL}\text{-}[\mathrm{Pten}_2\ l\text{-pn}]\mathrm{Cl}_4 \quad (1)$$

$$[\mathrm{Pten}\ \mathrm{Cl}_4] + 2l\text{-pn} = \mathrm{DL}\text{-}[\mathrm{Pten}\ L\text{-pn}_2]\mathrm{Cl}_4 \quad (2)$$

$$[\mathrm{Pt}\ l\text{-pn}\ \mathrm{Cl}_4] + 2d\text{-pn} = \mathrm{DL}\text{-}[\mathrm{Pt}\ d\text{-pn}_2\text{-}l\text{-pn}]\mathrm{Cl}_4 \quad (3)$$

Although it might be supposed that the addition of the two molecules of base would be stepwise, no evidence was found for the formation of the *trans*-dichlorobis-(diamine) complex, nor could the *cis*-dichlorobis-(diamine) complex be prepared when only one additional molecule of base was allowed to react in very dilute solution. From studies of the reaction between ethylenediamine and ethylenediaminetetrachloroplatinum(IV) in aqueous solution, other workers concluded that the first molecule of the base attached rather slowly as a monodentate ligand, replacing one of the two chlorine atoms *trans* to each other. Rapid replacement of a second chlorine atom, necessarily in the *cis* position, gave the *cis*-dichlorobis-(ethylenediamine) complex which immediately formed the tris-(ethylenediamine) complex.[5] Bis-(ethylenediamine)-*l*-propylenediamineplatinum(IV) ion can exist in only two isomeric forms, (D*l* and L*l*). Because propylenediamine also is unsymmetrical the ions $(\mathrm{Pt}\ \mathrm{D}\text{-pn}_2\text{-}l\text{-pn}]^{4+}$ and $[\mathrm{Pt}\ l\text{-pn}_2\mathrm{en}]^{4+}$ can exist in eight and six isomeric forms,[6] respectively.

Resolution of all three substances was effected through the *d*- and *l*-tartrates. The specific rotations of the chlorides are shown in Table I and for comparison the values for $[\mathrm{Pten}_3]\mathrm{Cl}_4$ and $[\mathrm{Pt}\ \mathrm{pn}_3]\mathrm{Cl}_4$. The values show an obvious regularity when it is remembered that the contribution of the coordinated propylenediamine is opposite in sign to the free base.[7] The approximate composition of the mixture of isomers as prepared has been calculated from the rotation of the mixture and the specific rotations of the pure isomers (Table I). The accuracy of the estimate is limited by the difficulty of isolating the whole of the material, especially the more soluble isomer, from the dimethylformamide solution. It is evident that the relative amounts of the isomers formed in each reaction are necessarily not the equilibrium amounts. If it is assumed that the reaction proceeds through the *cis*-dichlorobis-(diamine) complex and the tris-(diamine) complex is formed therefrom, with retention of con-

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(1) F. P. Dwyer, F. L. Garvan and A. Shulman, THIS JOURNAL, **81**, 290 (1959); F. P. Dwyer and F. L. Garvan, *ibid.*, **81**, 1043 (1959); F. P. Dwyer and A. M. Sargeson, *ibid.*, **81**, 5269 (1959).

(2) F. M. Jaeger and H. B. Blumendal, *Proc. Acad. Wetensch. Amsterdam*, **29**, 575 (1926).

(3) A. P. Smirnoff, *Helv. Chim. Acta*, **3**, 177 (1920).

(4) L. Tschugaeff and W. Sokolov, *Ber.*, **40**, 3461 (1907); **42**, 56 (1909).

(5) F. Basolo, J. C. Bailar and B. R. Tarr, THIS JOURNAL, **72**, 2433 (1950).

(6) The isomeric forms could be designated by the assignment of the usual octahedral position number to the methyl group in proximity. The four isomeric forms of the *levo series* of $\mathrm{DL}[\mathrm{Pt}\cdot d\text{-pn}_2\text{-}l\cdot\mathrm{pn}]^{4+}$ ion can then be written L-(2,3,5)-*ddl*, L-(1,3,6)-*ddl*, L-(1,3,5)-*ddl* and L-(2,3,6)-*ddl*.

(7) T. D. O'Brien and R. C. Toole, *ibid.*, **77**, 1368 (1955).

TABLE I

Isomer	[α]D	% in isomeric mixture
D-[Pten$_2$-*l*-pn]Cl$_4$	+133	50
L-[Pten$_2$-*l*-pn]Cl$_4$	− 74	50
D-[Pten-*l*-pn$_2$]Cl$_4$	+176	55
L-[Pten-*l*-pn$_2$]Cl$_4$	− 29	45
D-[Pt-*d*-pn$_2$-*l*-pn]Cl$_4$	+ 83	65
L-[Pt-*d*-pn$_2$-*l*-pn]Cl$_4$	−158	35
D-[Pt-*l*·pn$_3$]Cl$_4$	+212	60,[a] 85[b]
L-[Pt-L·pn$_3$]Cl$_4$	− 1	40,[a] 15[b]
D-[Pten$_3$]Cl$_4$	+ 90	..

[a] This work, prepared in dimethylformamide. [b] Reference 1, prepared in alcohol.

figuration, then the final isomeric composition, when the reaction has gone to completion, is determined with the addition of the first molecule of diamine. This mechanism is consistent with the common intermediate DL-[Pten-*l*-pnCl$_2$]Cl$_2$ of reactions 1 and 2 above and the close similarity of the relative amounts of the tris complexes formed. The rates of formation of the optical isomers of [Pten-*l*-pnCl$_2$]Cl$_2$ appear to be nearly equal, but there is a little discrimination in the formation of [Pt *l*-pn-*d*-pnCl$_2$]Cl$_2$. The optical isomers were not racemized at 100° in dilute acid or alkali and showed no evidence of disproportionation. The molar ratios pn/en and *d*-pn/*l*-pn, determined after decomposition of the complexes with a fused mixture of sodium sulfide and hydroxide, were close to the expected values.

Careful fractionation of the optical isomers of [Pten-*l*-pn$_2$]Cl$_4$ and [Pt-*l*-pn-*d*-pn$_2$]Cl$_4$ failed to effect any separation of *cis* and *trans* geometrical isomers, and it is concluded, as with [Ptpn$_3$]Cl$_4$ and [Copn$_3$]Cl$_3$ described previously,[1] that the solubilities must be similar. Because of the discrimination shown toward the resolving agent and the similarity with the optical forms of [Pten$_2$pn]Cl$_4$, which cannot exist in geometrical isomers, it is highly probable that all, in fact, are optical and not geometrical isomers.

Experimental

Unless otherwise stated all rotations were measured in water at 20° in a 2-dm. tube. The active[1] propylenediamine used had [α]D 34.2° in benzene.

Dichloro-*l*-propylenediamine-platinum(II).—This substance has been prepared previously[8] by the action of optically impure *l*-propylenediamine on potassium tetrachloroplatinate(II). To a boiling solution of sodium acetate (50 g. in 120 ml. of water) were added *l*-propylenediamine dihydrochloride[1] (5 g.) and then immediately with stirring, potassium tetrachloroplatinate(II) (11 g.). The yellow [Ptpn Cl$_2$]^0 crystallized almost at once, and after 5 minutes was filtered off and washed with a little cold water. The filtrate after reheating for a further 5 minutes gave a second smaller amount of the material. The final filtrate acidified with concentrated hydrochloric acid (50 ml.) and cooled, deposited a little more (total yield 90%). The slight pink tint due to traces of [Ptpn$_2$]PtCl$_4$ was removed by recrystallization from 5 *N* hydrochloric acid. A 0.1% solution in water (4-dm. tube) gave [α]D +35°. *Anal.* Calcd. for [Pt-$C_3H_{10}N_2Cl_2$]: Pt, 57.35. Found: Pt, 57.4.

Tetrachloro-(*l*-propylenediamine)-platinum(IV).—Dichloro-(*l*-propylenediamine)-platinum(II) (12.5 g.), suspended in cold water (250 ml.), was treated with chlorine gas for ten minutes. The material changed to dark orange-red (Pt(III) ?) and then became orange. The mixture was heated in the water-bath for 15 minutes, then cooled in ice, saturated with chlorine and reheated. This procedure was carried out three times. The orange solution then was evaporated on the steam-bath in a current of air to 25 ml. The orange yellow [Ptpn Cl$_4$] was washed with a little ice-water (yield 14 g., 93%). A 0.1% aqueous solution (4 dm. tube) gave [α]D +108°, [α]$_{5461}$ + 140°. *Anal.* Calcd. for Pt-($C_3H_{10}N_2$)Cl$_4$: C, 8.76; H, 2.45; N, 6.81. Found: C, 8.75; H, 2.49; N, 6.62.

D-Bis-(ethylenediamine)-*l*-propylenediamine-platinum-(IV) Chloride 1-Hydrate.—Tetrachloro-(*l*-propylenediamine)-platinum(IV) (15 g.) in dimethylformamide (200 ml.) was treated with ethylenediamine (5.5 ml., 89% w./v.). The mixture became more viscous, a pale yellow solid separated after 10 minutes and after 3 hr. a thick gel had formed. Acetone (11 l.), containing concentrated hydrochloric acid (30 ml.), was added, the mixture warmed gently to effect coagulation, then cooled. The white precipitate was removed and washed with acetone (yield 19.5 g., 96%; [α]D +31°).

The substance (17 g.) in hot water (200 ml.), was treated with potassium *l*-tartrate (13.5 g.), in hot water (100 ml.). The least soluble diastereoisomer, D-[Pten$_2$-*l*-pn](*l*-Tart.)$_2$, separated on scratching the sides of the vessel with a glass rod. The precipitate was removed from the hot solution and shaken up with two portions of boiling water (100 ml.). The filtrate and washings were reserved. *Anal.* Calcd. for [Pt($C_2H_8N_2$)$_2$·$C_3H_{10}N_2$]($C_4H_4O_6$)$_2$·2H$_2$O; C, 24.98; H, 5.31; Pt, 27.05. Found: C, 24.7; H, 5.32; Pt, 27.0.

The diastereoisomer (5 g.) was treated with concentrated hydrochloric acid (10 ml.) and alcohol (50 ml.), warmed in a water-bath, and the complex chloride filtered and washed with alcohol. It was then crystallized to constant rotation from dilute hydrochloric acid. A 0.5% solution gave [α]D +133°, [α]$_{5461}$ +156°. The molar ratio pn/en determined as described previously[1] was 0.49. *Anal.* Calcd. for [Pt-($C_2H_8N_2$)$_2$·$C_3H_{10}N_2$]Cl$_4$·H$_2$O; C, 15.31; H, 5.13; N, 15.14; Pt, 35.52; Cl, 25.81. Found: C, 15.26; H, 5.22; N, 15.18; Pt, 35.56; Cl, 26.0.

L-Bis-(ethylenediamine)-*l*-propylenediamine-platinum-(IV) Chloride 1.5 Hydrate.—The filtrate and washings from the diastereoisomer above were concentrated in such a way as to yield four fractions of crystalline material. The two most soluble fractions were converted to the chloride with hydrochloric acid, the solution evaporated to dryness and the *l*-tartaric acid removed by washing with alcohol. The chloride was then fractionally crystallized from a little dilute hydrochloric acid, and the fractions recrystallized in such a way as to concentrate the least soluble material in the head fraction ([α]D −74°). The least soluble material was then converted to the sulfate by double decomposition with the theoretical weight of silver sulfate and then to the *l*-tartrate, with a slight excess of barium *l*-tartrate. The diastereoisomer was recrystallized from water to give the pure L-[Pten$_2$-*l*-pn] (*l*-Tart.)$_2$. *Anal.* Calcd. for [Pt($C_2H_8N_2$)$_2$·$C_3H_{10}N_2$]($C_4H_4O_6$)$_2$·3H$_2$O: C, 24.36; H, 5.45; Pt, 26.38. Found: C, 24.74; H, 5.51; Pt, 26.3.

The diastereoisomer was converted to the chloride as above and the latter recrystallized from aqueous methanol to constant rotation. A 0.5% solution gave [α]D −74°, [α]$_{5461}$ −88°.

Anal. Found: C, 14.97; H, 5.23; Pt, 34.85; Cl, 25.45; ratio pn/en, 0.51.

L-Bis-(*d*-propylenediamine)-*l*-propylenediamine-platinum-(IV) Chloride.—Dextro propylenediamine[1] (8.0 ml. 86%, w./v.) was added to tetrachloro-(*l*-propylenediamine)-platinum(IV) (16.4 g.) in dimethylformamide (200 ml.). A gel soon formed and after 3 hr. acetone (30%)–alcohol (70%) mixture (1 l.), containing concentrated hydrochloric acid (30 ml.), was added, and the precipitate centrifuged down. The precipitate, after dissolution in water, was obtained in crystalline form by evaporation to dryness and was then washed with alcohol (yield 20 g. (87%); [α]D −71°). The substance was recrystallized from water, and the mother liquor evaporated to dryness in order to obtain approximately two equal portions. The less soluble part was then transformed in boiling solution to the sulfate (using silver sulfate) and to the *d*-tartrate (using a slight excess of barium *d*-tartrate). The diastereoisomer L-[Pt-*d*-pn$_2$-*l*-pn](*d*-Tart)$_2$·5H$_2$O separated on cooling. *Anal.* Calcd. for [Pt·($C_3H_{10}N_2$)$_3$]·($C_4H_4O_6$)$_2$·5H$_2$O: C, 25.39; H, 6.02, Pt, 24.29. Found: C, 25.37; H, 6.06; Pt, 23.9.

The diastereoisomer after treatment with hydrochloric acid and alcohol as before gave the pure *l*-chloride, [α]D +11.54° in benzene, whence *d*-pn/*l*-pn = 2.1 ± 0.1, (calculated from [α]D ±34.8° for pure propylenediamine).

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[8] L. A. Tschugaeff and W. Sokolov, *Ber.*, **40**, 3461 (1907).

Anal. Calcd. for $[Pt(C_3H_{10}N_2)_3]Cl_4$: C, 19.32; H, 5.41; N, 15.03; Pt, 34.89; Cl, 25.35. Found: C, 19.5; H, 5.64; N, 15.16; Pt, 34.94; Cl, 25.2.

D-Bis-(*d*-propylenediamine)-*l*-propylenediamine-platinum (IV) Chloride.—The more soluble portion of the mixture of isomers above was converted to the *d*-tartrate in the usual manner and combined with the filtrate remaining after the crystallization of the less soluble diastereoisomer. The solution was then concentrated to 60 ml. and the less soluble diastereoisomer, which crystallized, was removed. The more soluble diastereoisomer in solution was treated with concentrated hydrochloric acid and the mixture evaporated to dryness. The *d*-tartaric acid was completely removed by washing with warm alcohol, and the complex chloride transformed with silver sulfate and then barium *l*-tartrate to the *l*-tartrate. Fractional crystallization of the latter material gave the pure diastereoisomer, D-$[Pt\text{-}d\text{-}pn_2\text{-}l\text{-}pn](l\text{-tart.})_2 \cdot 7H_2O$; in the least soluble fractions.

Anal. Calcd. for $[Pt(C_3H_{10}N_2)_3](C_4H_4O_6)_2 \cdot 7H_2O$: C, 24.30; H, 6.24; Pt, 23.24. Found: C, 24.40; H, 6.14; Pt, 23.45.

The chloride obtained in the usual way, after recrystallization from water gave $[\alpha]_D$ +83°, $[\alpha]_{5461}$ +100°. The recovered propylenediamine gave $[\alpha]_D$ +11.80° in benzene, whence d-pn/l-pn $= 1.96 \pm 0.1$.

Anal. Found: C, 19.16, H, 5.36; N, 15.06; Pt, 34.92; Cl, 25.1.

D-Bis-(*l*-propylenediamine)-ethylenediamine-platinum-(IV) Chloride.—The intermediate substances $[Pten\ Cl_2]$ and $[Pten\ Cl_4]$ have been described previously.[5] The former is best prepared in the same manner as $[Ptpn\ Cl_2]$ above, using potassium tetrachloroplatinate(II), (10 g.) and ethylenediamine dihydrochloride (4 g.) yield 6.5 g., 82%). Levopropylenediamine (6.6 g.) was added to a solution of tetrachloro-(ethylenediamine)-platinum(IV) (15.8 g.) in dimethylformamide (250 ml.). A gel gradually formed and, after 3 hr., dry hydrogen chloride gas was passed until the mixture was acid and coagulation effected by the addition of acetone (70%), alcohol (30%) mixture (1 l.). The white solid (17.5 g., 80%) gave $[\alpha]_D$ +85°. The substance was dissolved in hot water and evaporated until approximately half of the complex separated on cooling. This portion was then converted to the *d*-tartrate as above, in boiling solution. The diastereoisomer d-$[Pt\text{-}l\text{-}pn_2en](d\text{-tart})_2 \cdot 5H_2O$ that separated on cooling was recrystallized from hot water. *Anal.* Calcd. for $[Pt(C_3H_{10}N_2)_2 \cdot C_2H_8N_2](C_4H_4O_6)_2 \cdot 5H_2O$: C, 24.33; H, 5.87; Pt, 24.71. Found: C, 24.62; H, 6.03; Pt, 24.6.

The chloride, obtained with hydrochloric acid and alcohol, after three recrystallizations from water, gave $[\alpha]_D$ +176°, $[\alpha]_{5461}$ +210° (0.25% soln.). *Anal.* Calcd. for $[Pt(C_3H_{10}N_2)_2 \cdot C_2H_8N_2]Cl_4$: C, 17.62; H, 5.17; Pt, 35.79; Cl, 26.00; molar ratio pn/en, 2.00. Found: C, 17.65; H, 5.24; Pt, 35.74, Cl, 25.86; molar ratio pn/en, 2.06.

L-Bis-(*l*-propylenediamine)-ethylenediamine-platinum-(IV) Chloride.—The more soluble portion of the mixture of isomers was converted as before to the *d*-tartrate, combined with the mother liquors from the less soluble diastereoisomer and concentrated gradually to 50 ml. Small amounts of the less soluble diastereoisomer, that separated, were removed. The *d*-tartrate was removed from the remaining sirup by the addition of hydrochloric acid, followed by evaporation to dryness and extraction with warm alcohol. The complex chloride was then transformed to the *l*-tartrate, as before, and the solution concentrated. A small amount of the diastereoisomer D-$[Pt\text{-}l\text{-}pn_2 \cdot en](l\text{-tart.})_2$ that separated was removed, and finally a gum was left. The *l*-tartrate was then replaced by chloride and the substance fractionally crystallized from aqueous methanol. The least soluble fractions gave the pure levo form, $[\alpha]_D$ −29°; $[\alpha]_{5461}$ −35°, (0.5% solution). *Anal.* Calcd. for $[Pt(C_3H_{10}N_2)_2 \cdot C_2H_8N_2]Cl_4 \cdot H_2O$: C, 17.06; H, 5.37; Pt, 34.64; Cl, 25.17, molar ratio pn/en, 2.00. Found: C, 17.46; H, 5.33; Pt, 34.62; Cl, 24.9; molar ratio pn/en, 2.01.

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CANBERRA, AUSTRALIA

[CONTRIBUTION FROM THE RESEARCH LABORATORY, KAISER ALUMINUM & CHEMICAL CORPORATION]

Preparation of Pure Anhydrous Aluminum Fluoride by Vacuum Sublimation

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Laboratory quantities of pure anhydrous aluminum fluoride have been prepared by vacuum sublimation of a calcined hydrated salt in a simple and easily constructed apparatus. By proper selection of condenser temperature and retort-condenser temperature differential, a dense pad of well-defined crystals can be produced. Individual crystals up to four millimeters in diameter are observed in the product. The purity is greater than 99% with no detectable oxyfluorides.

For many chemical and physical applications the level of impurities in technical grade aluminum fluoride and the water of hydration in reagent grade product cannot be tolerated. Technical grade aluminum fluoride may contain up to 15% alumina together with minor impurities such as cryolite, aluminum sulfate, silica and iron oxide. Reagent grade aluminum fluoride contains only traces of impurities but is a hydrated salt or mixture of hydrates containing up to 40% water of crystallization. Attempts to remove this water inevitably result in the loss of fluoride and formation of alumina by thermal hydrolysis. Removal of the last traces of water requires calcination temperatures from 500 to 800°.[1] Thermal hydrolysis takes place at temperatures as low as 150 to 200°.[2]

Pure anhydrous aluminum fluoride has been prepared by some investigators by sublimation techniques. Batashev[3] states that it may be prepared by distillation of the dry impure salt in a hydrogen fluoride atmosphere. Zhivov[4] suggests sublimation of technical aluminum fluoride from an Alundum crucible at not less than 1100° using a second Alundum crucible as a condenser. To avoid basic fluoride formation, it is necessary to employ an atmosphere of hydrogen fluoride or inert gas. The sublimation of aluminum fluoride from a mixture of fluorspar and kaolin is the subject of a Russian patent.[5]

The method described here was developed for the purpose of preparing laboratory quantities of high

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[1] T. R. Scott, Council for Scientific and Industrial Research, Bulletin 230, Melbourne, Australia, 1947.

[2] A. J. Edwards, British Patent 656,374 (August 22, 1951).

[3] K. P. Batashev, *Trans. Leningrad Ind. Inst. No. 1, Sect. Met.*, No. 1, 40 (1939).

[4] V. G. Zhivov, *Khim. Referat. Zhur.*, **6**, 98 (1940).

[5] G. S. Morozov and N. A. Ivanov, Russian Patent 52,597 (February 28, 1938).