

110. Studies in Co-ordination Chemistry. Part XVII.*
Complexes of Ruthenium with a Di(tertiary Arsine).

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Complexes of bivalent and trivalent ruthenium with *o*-phenylenebisdimethylarsine have been prepared and studied. Bivalent ruthenium forms compounds of the general formula $\text{Ru}(\text{Diarsine})_2\text{X}_2$, where X is a halogen atom. These are diamagnetic and are non-electrolytes in nitrobenzene and hence the ruthenium atom is sexicovalent. Oxidation of these bivalent complexes yields trivalent ruthenium salts containing the cation $[\text{Ru}(\text{Diarsine})_2\text{X}_2]^+$. Magnetic measurements indicated the presence of one unpaired electron in the trivalent complexes. Attempts to isolate quadrivalent ruthenium derivatives were unsuccessful.

An earlier investigation¹ showed that bivalent and trivalent iron give rise to complexes of the type $[\text{Fe}(\text{Diarsine})_2\text{X}_2]^0$ and $[\text{FeX}_2(\text{Diarsine})_2]\text{ClO}_4$ with *o*-phenylenebisdimethylarsine, and recently² it has been possible to oxidise the iron(III) complexes to form $[\text{Fe}^{\text{IV}}\text{Cl}_2(\text{Diarsine})_2][\text{ReO}_4]_2$, a derivative of quadrivalent iron. The investigation of the ruthenium complexes was prompted mainly by interest in their magnetic behaviour, but also in order to compare the complexes with those of iron and osmium. Special interest attaches to the quadrivalent state since this gives rise to anomalous magnetic behaviour in the case of osmium. An earlier³ investigation of the complexes of ruthenium with methyl diphenylarsine yielded non-electrolytes of the types $[\text{Ru}^{\text{II}}\text{X}_2, 4\text{Ph}_2\text{AsMe}]^0$ and $[\text{Ru}^{\text{III}}\text{Cl}_3, 3\text{Ph}_2\text{AsMe}]^0$ but the magnetic properties were not investigated. No previous work on the ruthenium complexes of this chelate group has been reported.

Bivalent Ruthenium Complexes.—When an alcoholic solution of *o*-phenylenebisdimethylarsine is added to ruthenium(III) halides in aqueous ethanol, and the mixture refluxed for a few minutes, crystals of the compound $[\text{RuX}_2(\text{Diarsine})_2]^0$ are precipitated. The diarsine reduces the Ru^{III} to the bivalent stage. As shown in the Table, these compounds vary in colour from pale yellow to orange. In the case of the iodide it was also possible to isolate a purple form by using more concentrated solutions in the original preparation. On heating, the violet form reverts to the orange, and both forms give the same pale yellow solution in chloroform. Addition of light petroleum to this solution yields only the orange form. A possible explanation for the existence of two forms is geometrical isomerism since the two iodine atoms in an octahedral complex could be either *cis* or *trans*.† It was not possible to check this idea by electric dipole-moment measurements owing to the insolubility of the compounds in dioxan and benzene, the two solvents likely to be of value for such a purpose. In nitrobenzene and in acetone, in which the complexes are sparingly soluble, they dissolve to yield the light-coloured form. Ebullioscopic molecular-weight determinations in chloroform and molecular conductivities in nitrobenzene show that the complexes are non-electrolytes. This, together with the diamagnetism, clearly indicates a bivalent ruthenium atom in a sexicovalent, presumably octahedral inner-orbital ($4d^25s5p^3$) complex. The corresponding thiocyanate was also prepared in the hope that it might be more soluble in benzene or dioxan to enable electric dipole-moment measurements to be carried out. Unfortunately, this thiocyanate is also only sparingly soluble. The properties of the compounds are summarised in the Table.

Trivalent Complexes.—Potentiometric titrations of the bivalent chlorine complex with

* Part XVI, preceding paper.

† Alternatively—and we think more likely—the purple form might be a salt of the type $[\text{Ru}(\text{Diarsine})_3][\text{RuDiarsineI}_4]$.

¹ Nyholm, J., 1950, 851.

² Nyholm and Parish, *Chem. and Ind.*, 1956, 470.

³ Dwyer, Humpoletz, and Nyholm, *J. Proc. Roy. Soc. N.S.W.*, 1946, 80, 217.

potassium dichromate in dilute acid solution show that a one-electron oxidation per ruthenium atom can be effected. Owing to the insolubility of the bivalent complex in water the reaction was carried out in chloroform-acetone. The acetone also ensured that the resulting ruthenium(III) complex remained in solution, since the estimated oxidation-reduction potential for the system $[\text{Ru}(\text{Diarsine})_2\text{Cl}_2]^0 - e \longrightarrow [\text{Ru}(\text{Diarsine})_2\text{Cl}_2]^+$ is $+0.64$ v. This explains how the Ru^{II} complex can be oxidised to the Ru^{III} compound by ferric chloride; also how the Ru^{III} complex ion is reduced by iodide ions.

The ruthenium(III) complexes of the diarsine were prepared conveniently by oxidation of the corresponding ruthenium(II) complexes with chlorine or bromine in chloroform. However, the reactions are complicated by the easy removal of one molecule of the diarsine with the formation of complexes of the type $[\text{Ru}(\text{Diarsine})_2\text{X}_2][\text{Ru}(\text{Diarsine})\text{X}_4]$. No corresponding anion of trivalent iron was obtained in the earlier investigation but complexes with the FeCl_4^- ion can be isolated. Tervalent chromium also forms semi-covalent anionic complexes similar to those of ruthenium. The bromo- and chloro-complexes were precipitated as fine emerald-green and bluish-green crystals respectively. The analyses agreed with the empirical formula $\text{Ru}_2(\text{Diarsine})_3\text{X}_6$; the possible

Conductivities and magnetic moments of ruthenium complexes.

Compound *	Colour	Conductivity in PhNO_2		$\chi_g \times 10^{-6}$ at 20.0°	$\chi_M \times 10^{-6}$	Diamag. corr.	$\chi_M' \times 10^{-6}$	μ (B.M. per Ru atom)
		Λ_M at 25°	Concn. (10^{-3}M)					
RuD_2Cl_2	Pale yellow	0.4	0.33	-0.13	-98	428	—	0.0 †
RuD_2Br_2	Yellow- orange	0.6	0.38	-0.19	-161	447	—	0.0 †
RuD_2I_2	Orange	0.6	0.28	-0.16	-150	467	—	0.0 †
RuD_2I_2	Purple	0.8	0.28	-0.18	-175	467	—	0.0 †
$\text{RuD}_2(\text{CNS})_2$	Silver- grey	1.1	0.4	-0.12	-97	439	—	0.0 †
$[\text{RuD}_2\text{Cl}_2]\text{Cl}$	Green	27.3	1.54	1.26	981	449	1430	1.83
$[\text{RuD}_2\text{Cl}_2]\text{ClO}_4$	Green	29.4	0.50	1.29	1088	456	1544	1.91
$[\text{RuD}_2\text{Cl}_2][\text{RuDCl}_4]$	Blue- green	34.9	2.1	1.75	2230	699	2929	1.86
$[\text{RuD}_2\text{Br}_2]\text{Br}$	Green	26.0	0.54	1.23	1114	482	1596	1.94
$[\text{RuD}_2\text{Br}_2][\text{RuDBr}_4]$	Green	35.4	1.7	1.67	2570	762	3332	1.97
$[\text{RuD}_2\text{I}_2]\text{ClO}_4$	Dark green	27.2	1.0	1.01	1038	497	1535	1.90
$[\text{RuDCl}_4]^-$	—	—	—	—	1249	273	1522	1.89
$[\text{RuDBr}_4]^-$	—	—	—	—	1456	315	1771	2.04

* D = *o*-Phenylenebisdimethylarsine.

† These moments are regarded as formally zero. The small net paramagnetism after diamagnetic correction is attributed to small amounts of impurity and/or Van Vleck paramagnetism.

alternative formulation as $[\text{Ru}(\text{Diarsine})_3][\text{RuX}_6]$ is unlikely for the following reasons. (1) The conductivities in nitrobenzene indicate that the complexes are uni-univalent electrolytes. (2) The $[\text{RuX}_6]^{3-}$ ion is orange-red, but the complexes isolated are green. Even if the $[\text{Ru}(\text{Diarsine})_3]^{3+}$ ion were highly coloured, it is unlikely that the complex would be green if the red $[\text{RuCl}_6]^{3-}$ anion were present. (3) No other simple complexes of the $[\text{Ru}(\text{Diarsine})_3]^{3+}$ could be prepared. These trivalent ruthenium complexes of the diarsine are very soluble in acetone or ethanol, but are rapidly reduced at the boiling point in these solvents. Reduction is slow at room temperature, however.

The addition of a slight excess of chlorine or bromine (X) to the bivalent ruthenium complex of iodine, $\text{Ru}(\text{Diarsine})_2\text{I}_2$, results first in the formation of $[\text{Ru}(\text{Diarsine})_2\text{I}_2]\text{X}$, then decomposition occurs forming $[\text{Ru}(\text{Diarsine})_2\text{X}_2][\text{Ru}(\text{Diarsine})\text{X}_4]$, iodine in the cation being oxidised to the free element. Iodine cannot be used as the oxidant because its oxidation potential is not high enough. Reduction of the trivalent ruthenium complexes can also be effected with iodide ion or sulphites, as well as with solvents like

acetone and alcohol. The perchlorates of the ruthenium(III) cation $[\text{Ru}(\text{Diarsine})_2\text{X}_2]^+$ are difficult to obtain once the $[\text{Ru}(\text{Diarsine})\text{X}_4]^-$ ion concentration becomes appreciable owing to the fact that the perchlorate is the more soluble salt. They may be formed if excess of perchloric acid in ethanol is added to the solutions of the bivalent complexes in chloroform before treatment with the oxidising agent (chlorine or bromine).

Potentiometric titrations with silver nitrate were carried out with the chloride $[\text{Ru}(\text{Diarsine})_2\text{Cl}_2]\text{Cl}$ and with the bromide $[\text{Ru}(\text{Diarsine})_2\text{Br}_2]\text{Br}$. The potentiometric end-points agreed well with the calculated titres for one-third of the total halogen in both cases, thus indicating that only one halogen atom is ionised. Conductivity measurements of the trivalent ruthenium complexes in nitrobenzene gave molecular conductivities between 26 and 35 mhos. The higher figures were obtained when the anion was $[\text{Ru}(\text{Diarsine})\text{X}_4]^-$, presumably owing to some dissociation of the anion. This effect was also observed with the Cr^{III} complexes. This dissociation is also indicated by the unduly low molecular weight of the $[\text{Ru}(\text{Diarsine})_2\text{X}_2][\text{Ru}(\text{Diarsine})\text{X}_4]$ complexes in nitrobenzene solution. The conductivities of the ruthenium(III) complexes agree well with the figures for other uni-univalent electrolytes. Measurements of magnetic susceptibility gave magnetic moments of approximately 1.9 B.M. (see Table) for the ruthenium(III) complexes, indicating one unpaired electron.

All attempts to obtain tris-diarsine complexes of ruthenium were unsuccessful. Prolonged refluxing of excess of diarsine with ruthenium halides in ethanol yielded complexes containing two molecules only of the ligand to each ruthenium atom. Attempts to prepare a tris-complex using ruthenium(III) perchlorate were also not successful. The colourless solution was refluxed with diarsine in ethanol for several hours, but all of the diarsine could be recovered. This reluctance of most trivalent metals to form tris-diarsine complexes has been referred to previously. Tri-oxalato- and tri-glycine complexes of ruthenium(III) were also treated unsuccessfully with the diarsine in attempts to prepare the required tris-diarsine complex. Repeated attempts to prepare ruthenium(IV) complexes were not successful. The use of 15N-nitric acid as oxidant, which yields Fe^{IV} , Ni^{IV} , and Os^{IV} complexes, resulted in decomposition. As will be described in the next paper, stable osmium(IV) complexes can be obtained by this procedure. The failure to isolate ruthenium(IV) complexes similar to the iron(IV) compounds probably arises from their reactivity, since the quadrivalent state of ruthenium is well known in many complexes such as K_2RuCl_6 of which the iron analogues cannot be prepared.

EXPERIMENTAL

Dichlorodi-(o-phenylenebisdimethylarsine)ruthenium(II).—Ruthenium(III) chloride (0.6 g.) in water (15 ml.) was added to *o*-phenylenebisdimethylarsine (1.2 g.) in purified ethanol (150 ml.) to give one phase. No reaction appeared to take place in the cold and the solution was heated under reflux for a few minutes. The pale yellow crystalline precipitate (0.6 g.) which formed was allowed to settle, separated by centrifuging, washed with cold ethanol, and dried *in vacuo*. A second crop of crystals was obtained by adding water, in which the complex is insoluble, to the mother-liquors. The use of hypophosphorous acid to assist in the reduction gave no greater yield (Found: C, 32.0; H, 4.2; Cl, 9.5%; *M*, ebullioscopic in 1.54% solution in chloroform, 680. $\text{C}_{20}\text{H}_{32}\text{Cl}_2\text{As}_4\text{Ru}$ requires C, 32.2; H, 4.3; Cl, 9.5%; *M*, 745). The compound, which is stable to above 360°, is soluble in chloroform and slightly soluble in warm ethanol, in which pale yellow solutions are obtained. It is also slightly soluble in methanol, acetone, nitrobenzene, and 1:4-dioxan, but insoluble in benzene, ether, and water. The compound is stable when freely exposed to air or water at room temperature. In dilute ethanol, silver nitrate gave only a slight turbidity when cold, but decomposition occurred on boiling, yielding a copious precipitate of silver chloride.

Dibromodi-(o-phenylenebisdimethylarsine)ruthenium(II).—Ruthenium(III) chloride (0.6 g.) in water (15 ml.) was treated with sodium bromide (1.5 g.), followed by diarsine (1.2 g.) in ethanol (150 ml.), filtered and brought to boiling as before. The subsequent treatment was the same as

that for the chloride. Orange-pink acicular crystals of the *bromide* (0.66 g.) were obtained and recrystallised from ethanol. As before, dilution with water gave a further crop of crystals (Found: C, 29.3; H, 4.0; Br, 19.5%; *M*, ebullioscopic in 1.14% solution in chloroform, 836. $C_{20}H_{32}Br_2As_4Ru$ requires C, 28.8; H, 3.8; Br, 19.2%; *M*, 834). The compound dissolves, but to a smaller extent, in the same solvents as the chloride. It was stable on heating to above 360°.

Di-iodo-(o-phenylenebisdimethylarsine)ruthenium(II).—*Orange form*. Ruthenium(III) chloride (0.6 g.) in water (15 ml.) was treated with sodium iodide (1.5 g.) and diarsine (1.2 g.) in ethanol (150 ml.), and the solution heated to boiling as before. The orange crystalline precipitate was allowed to settle before being separated by centrifuging. Owing to its low solubility in warm ethanol recrystallisation was not possible, but purification was effected by washing with water to remove alkali halide and ethanol, and then with ether to remove traces of diarsine. The *complex* was sparingly soluble in chloroform, dioxan, and nitrobenzene, and virtually insoluble in other common organic solvents. The practically quantitative yield (0.9 g.) may be attributed to the low solubility in ethanol (Found: C, 25.9; H, 3.5; I, 27.3; Ru, 11.4%; *M*, ebullioscopic in 1.24% solution in chloroform, 870. $C_{20}H_{32}I_2As_4Ru$ requires C, 25.9; H, 3.51; I, 27.4; Ru, 11.0%; *M*, 928).

Purple form. Ruthenium(III) chloride (0.6 g.) in water (10 ml.) was treated with sodium iodide (3.0 g.) and diarsine (1.2 g.) in ethanol (30 ml.), and the solution filtered and heated under reflux as above. The purple crystalline precipitate settled rapidly, and was separated as above. Purification was effected by washing consecutively with water, ethanol, and ether. Solutions in chloroform or nitrobenzene, in which the substance is sparingly soluble, were pale yellow, and the orange form was precipitated therefrom by concentration (Found: C, 25.95; H, 3.3; I, 27.8%). Molecular-weight determinations were not carried out because isomerism to the orange form took place in dilute solutions. On heating the compound is transformed into the orange form at about 370°.

Dithiocyanatodi-(o-phenylenebisdimethylarsine)ruthenium(II).—Ruthenium(III) chloride (0.25 g.) in water (6 ml.) was treated with ammonium thiocyanate (0.18 g.) and diarsine (0.65 g.) in ethanol (80 ml.), heated under reflux for 1 min., and set aside. The product which settled rapidly was removed by decantation and centrifuging. Recrystallisation from ethanol (250 ml.) gave silvery-grey crystals (0.33 g.), which were slightly soluble in chloroform, but only very sparingly soluble in dioxan and insoluble in benzene (Found: C, 33.3; H, 4.1; N, 3.2%; *M*, cryoscopic in 1.4% solution in nitrobenzene, 700. $C_{22}H_{32}N_2S_2As_4Ru$ requires C, 33.5; H, 4.1; N, 3.5%; *M*, 790).

Dichlorodi-(o-phenylenebisdimethylarsine)ruthenium(III) Tetrachloromono-(o-phenylenebisdimethylarsine)ruthenate(III).—The ruthenium(II) complexes were prepared from the corresponding ruthenium(II) compounds. The chloro-complex $[Ru(Diarsine)_2Cl_2]^0$ (0.45 g.) was dissolved in "AnalaR" chloroform (60 ml.) to a yellow solution and treated with a steady stream of dry chlorine for 10 min. at room temperature. The blue-green solution was concentrated by boiling off most of the chloroform, and precipitation of the complex assisted by the addition of a few drops of light petroleum. The *complex* (0.38 g.) was separated by centrifuging, washed once with chloroform, and dried in a vacuum. The bluish-green crystals are soluble in ethanol and acetone, reduction to the bivalent complex $[Ru(Diarsine)_2Cl_2]^0$ taking place in these solvents, slowly in cold solutions, but rapidly at the b. p. The filtrate remains reddish presumably owing to the presence of Ru^{III} from the anion (Found: C, 28.2; H, 3.8; Cl, 16.7%; *M*, cryoscopic in 0.27% solution in nitrobenzene, 493. $C_{30}H_{48}Cl_6As_6Ru_2$ requires C, 28.3; H, 3.8; Cl, 16.7%; *M*, 1275). The compound decomposes at 233°.

Dichlorodi-(o-phenylenebisdimethylarsine)ruthenium(III) Chloride.—The complex $[Ru(Diarsine)_2Cl_2]^0$ (0.25 g.) in chloroform was treated simultaneously with dry hydrogen chloride and excess of dry chlorine, both as gas, the yellow solution becoming green. The chlorine was then stopped, and hydrogen chloride passed for a few more minutes; the mixture was then stirred and set aside. The green precipitate which formed was separated by centrifuging, washed with chloroform, then with a little alcohol and then several times with water and dried in vacuum (Found: C, 30.1; H, 3.9; Cl, 13.4%; *M*, cryoscopic in 0.68% solution in nitrobenzene, 360. $C_{20}H_{32}Cl_3As_4Ru$ requires C, 30.8; H, 4.1; Cl, 13.6%; *M*, 780). On heating, the compound decomposed near 200°. On warming with alcohol it was reduced to the Ru^{II} complex $[RuCl_2(Diarsine)_2]^0$.

A potentiometric titration was carried out to determine the proportion of free chloride ion. $[Ru(Diarsine)_2Cl_2]Cl$ (0.0176 g.) in ethanol (15 ml.) was treated with acetone (35 ml.) and barium

nitrate (0.5 g.), and the solution titrated fairly rapidly with 0.00995N-silver nitrate with constant stirring. A sharp end-point after only 1 equiv. of silver ion had been added confirmed that only one Cl atom is ionised.

Dichlorodi-(o-phenylenebisdimethylarsine)ruthenium(III) Perchlorate.—The bivalent complex $[\text{Ru}(\text{Diarsine})_2\text{Cl}_2]^0$ (0.15 g.) was dissolved in warm chloroform (25 ml.), and the solution filtered and treated with 70% perchloric acid (0.1 ml.) with sufficient ethanol to give one phase. Dry chlorine was bubbled into the solution until a precipitate had commenced to form, whereupon passage of the chlorine was stopped and the mixture shaken with a few drops of light petroleum. As with the preparation of the chloride, excess of chlorine had to be avoided and the perchlorate anion introduced in excess before oxidation, because the salt containing the complex $[\text{Ru}(\text{DiarsineCl}_4)]^-$ tends to be precipitated owing to lower solubility. The rest of the procedure was the same as that already described for the other trivalent ruthenium(III) complexes (Found: C, 28.6; H, 4.2; Ru, 11.8%; *M*, cryoscopic in 0.81% solution in nitrobenzene, 360. $\text{C}_{20}\text{H}_{32}\text{O}_4\text{Cl}_3\text{As}_4\text{Ru}$ requires C, 28.4; H, 3.8; Ru, 12.1%; *M*, 844); m. p. 265° (decomp.).

Dibromodi-(o-phenylenebisdimethylarsine)ruthenium(III) Tetrabromomono-(o-phenylenebisdimethylarsine)ruthenate(III).—The ruthenium(II) complex $[\text{Ru}(\text{Diarsine})_2\text{Br}_2]^0$ (0.35 g.) was dissolved in chloroform (75 ml.), and bromine added in slight excess. The resulting deep green solution was evaporated to 25 ml., and the bright green crystals which were formed on shaking with a little light petroleum were separated by centrifuging, washed with chloroform, and dried (H_2SO_4) in a vacuum (0.30 g.). The complex gave bright green solutions in ethanol or acetone, but was reduced in these solvents by warming or on standing to the yellow $[\text{Ru}(\text{Diarsine})_2\text{Br}_2]^0$, the solution remaining reddish owing to the presence of some ruthenium(III) bromide co-ordinated with solvent (Found: C, 23.4; H, 3.2%; *M*, cryoscopic in 0.26% solution in nitrobenzene, 650. $\text{C}_{30}\text{H}_{48}\text{Br}_6\text{As}_6\text{Ru}_2$ requires C, 23.4; H, 3.1%; *M*, 1542). The salt decomposed at 258°.

Dibromodi-(o-phenylenebisdimethylarsine)ruthenium(III) Bromide.—The ruthenium(II) complex $[\text{Ru}(\text{Diarsine})_2\text{Br}_2]^0$ (0.3 g.) was dissolved in chloroform (50 ml.), the solution filtered, and hydrogen bromide and bromine passed through it for a few minutes. Excess of bromine was avoided to prevent development of a high concentration of $[\text{Ru}(\text{Diarsine})\text{Br}_4]^-$ ion. Evaporation of the solution to about 20 ml. and addition of a few drops of light petroleum gave green crystals on agitation. The latter were separated by centrifuging, washed with chloroform and water, and dried in a vacuum. The bromide is soluble in ethanol and acetone but, like the corresponding chloro-complex, is reduced by these reagents in hot solution (Found: C, 26.0; H, 3.4; Br, 26.4%; *M*, cryoscopic in 0.31% solution in nitrobenzene, 420. $\text{C}_{20}\text{H}_{32}\text{Br}_3\text{As}_4\text{Ru}$ requires C, 26.3; H, 3.5; Br, 26.3%; *M*, 913); m. p. 230° (decomp.). A potentiometric titration in ethanol-acetone indicated that only one Br atom was ionised.

Di-iododi-(o-phenylenebisdimethylarsine)ruthenium(III) Perchlorate.—The ruthenium(III) complex $[\text{Ru}(\text{Diarsine})_2\text{I}_2]^0$ (0.2 g.) in warm chloroform (50 ml.) was treated with perchloric acid (0.1 g.) in ethanol (5 ml.) and the calculated proportion of bromine (0.018 g.) was added. The solution, which became deep green, was evaporated to 30 ml., and the sage-green crystals which formed were separated by centrifuging as before. After being washed with chloroform, these (0.14 g.) were dried in a vacuum (Found: C, 22.8; H, 3.0; Ru, 10.1%; *M*, cryoscopic in 1.0% solution in nitrobenzene, 480. $\text{C}_{20}\text{H}_{32}\text{O}_4\text{ClI}_2\text{As}_4\text{Ru}$ requires C, 23.4; H, 3.1; Ru, 9.9%; *M*, 1028). Addition of sodium iodide to an alcoholic solution of the complex caused liberation of free iodine, with immediate reduction to the bivalent complex $[\text{Ru}(\text{Diarsine})_2\text{I}_2]^0$. Treatment of the latter with excess of bromine in the absence of perchlorate anion gave the bromo-complex $[\text{Ru}(\text{Diarsine})_2\text{Br}_2][\text{RuDiarsineBr}_4]$. The complex $[\text{RuDiarsineI}_4]^-$ anion was not obtained (contrast the chromium complex).

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