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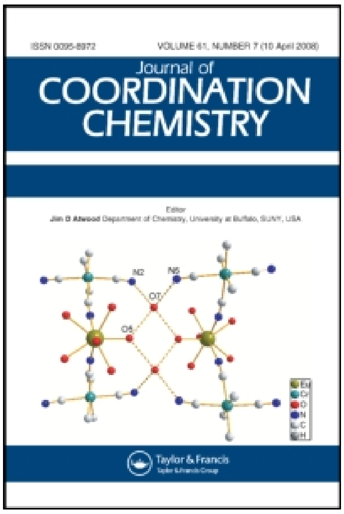

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COORDINATION COMPOUNDS OF URANIUM(VI) WITH LEWIS BASES

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A variety of Lewis bases react with uranyl salts in ethyl acetate to give yellow crystalline complex compounds in which U(VI) has different coordination numbers according to the type and size of the ligand used.

No evidence has been obtained for the formation of any complexes between uranyl salts and some organic phosphorus ligands.

Infrared spectra, thermal stability and conductivity of the soluble adducts obtained have been discussed.

INTRODUCTION

The salts of acids of the divalent uranyl radical $[O{=}U{=}O]^{2+}$, are very stable, and also have a strong tendency to form complex compounds.[1] Uranyl nitrate is the best known of the uranyl salts,[2] and occurs with 2, 3 and 6 molecules of water. The hexahydrate was early[3] formulated as $[UO_2(H_2O)_6](NO_3)_2$. Recent work led to the conclusion that the formula $[UO_2(NO_3)_2(H_2O)_2].4H_2O$ is also compatible. The neutron diffraction data indicated that the latter structure is more likely.[3] The linear UO_2^{2+} ion is surrounded in the equatorial plane by 2 bidentate NO_3 groups and 2 H_2O molecules while 4 H_2O molecules are in the outer sphere of the UO_2^{2+} ion.

Bogustawa and Helena[4] reported that the donor properties of N-oxide derivatives as ligands, have been found to be comparable with those of water and only a little stronger, since they displace water from the inner coordination sphere of uranium.

Both anionic and cationic complexes of uranyl ion are known.[5] With halide ions the complexes, UO_2X^+, $UO_2X_3^-$ and UO_2X^{2-} are reported. With phosphate, species are such as $UO_2H_2PO_4^+$, and $UO_2H_3PO_4^{2+}$ are known. Generally, the coordination chemistry of uranium(IV) has been investigated extensively[6–11] while for uranium(VI) the range is few.[12,13] The present work was carried out in an attempt to provide further information.

EXPERIMENTAL

The experiments were performed under an atmosphere of dry nitrogen. Considerable care was taken with the drying of solvents.[14] Uranyl chloride hexahydrate, BDH and uranyl nitrate hexa and tetrahydrates, May and Baker, were used during the present work. The method adopted for dehydration was reported earlier.[15]

Lewis Bases Some organic compounds used as ligands were prepared as mentioned previously.[16,17] Others were generally reagent grade materials, and were recrystallised from the appropriate solvent before use.

Preparations of the complex compounds

Reactions between the uranyl salts and the Lewis bases in the mole ratio 1:2, were carried out in anhydrous ethyl acetate. The experimental techniques used during this work were as mentioned previously.[16,17] Yields, m.ps. and analytical data for the products obtained are given in Table I. No complex compounds were obtained from the attempted reactions of uranyl salts with 1,2-bis-(diphenylphosphino)ethane and -methane.

RESULTS

Uranyl chloride and nitrate dissolve in dry ethyl actate and ethanol to give yellow solutions. These solutions are used during the present work.

TABLE I
Analytical results

1. $(MePh_3P)_2UO_2Cl_4$	(Found: C, 47.0; H, 3.7; P, 6.4. $C_{38}H_{36}Cl_4O_2P_2U$ required: C, 47.2; H, 3.75; P, 6.4%)
2. UO_2Cl_2, Diphos ethane dioxide	(Found: C, 39.9; H, 3.3; P, 7.4. $C_{26}H_{24}Cl_4O_4P_2U$ required: C, 40.5; H, 3.1; P, 8.0%)
3. $UO_2(NO_3)_2$, Diphos ethane dioxide	(Found: C, 38.2; H, 2.9; P, 7.15. $C_{26}H_{24}N_2O_{10}P_2U$ required: C, 37.9; H, 2.9; P, 7.5%)
4. $UO_2(NO_3)_2$, Diphos methane dioxide, $2H_2O$	(Found: C, 35.4; H, 2.9; P, 7.1. $C_{25}H_{26}N_2O_{12}P_2U$ required: C, 35.5; H, 3.1; P, 7.3%)
5. UO_2Cl_2, 2phen, $4H_2O$	(Found: C, 37.6; H, 3.2; P, 6.7. $C_{24}H_{24}Cl_2N_4O_6U$ required: C, 37.3; H, 3.1; P, 7.2%)
6. UO_2Cl_2, 2DM-phen, $2H_2O$	(Found: C, 42.6; H, 3.7; N, 7.5. $C_{28}H_{28}Cl_2N_4O_4U$ required: C, 42.4; H, 3.6; N, 7.1%)
7. UO_2Cl_2, 2terpy, $4H_2O$	(Found: C, 40.7; H, 3.6; N, 4.7. $C_{30}H_{30}Cl_2N_6O_6U$ required: C, 41.0; H, 3.4; N, 4.6%)
8. $UO_2(NO_3)_2$, 1.5terpy, $8H_2O$	(Found: C, 32.9; H, 3.4; N, 10.8. $(C_{45}H_{33}N_{13}O_{16}U_2)$, $8H_2O$ required: C, 33.1; H, 3.0; N, 11.1%)
9. $UO_2(NO_3)_2$, 2bipy, $4H_2O$	(Found: C, 30.1; H, 2.9; N, 10.5. $C_{20}H_{24}N_6O_{12}U$ required: C, 30.9; H, 3.1; N, 10.8%)
10. UO_2Cl_2, 2dipyamine, $4H_2O$	(Found: C, 32.2; H, 3.6; N, 10.6. $C_{20}H_{26}Cl_2N_6O_6U$ required: C, 31.8; H, 3.5; N, 11.1%)
11. UO_2Cl_2, 2hexamine, $8H_2O^a$	(Found: C, 18.2; H, 4.8; N, 16.0. $C_{12}H_{40}Cl_2N_8O_{10}U$ required: C, 18.8; H, 5.3; N, 14.6%)
12. $UO_2(NO_3)_2$, dipyamine	(Found: C, 21.8; H, 2.0; N, 11.9. $C_{10}H_9N_5O_8U$ required: C, 21.2; H, 1.6; N, 12.4%)
13. $UO_2(NO_3)_2$, hexamine[b]	(Found: C, 14.0; H, 2.2; N, 14.05. $C_6H_{12}N_6O_8U$ required: C, 13.5; H, 2.3; N, 15.7%)
14. $UO_2(NO_3)_2$, pyrazine	(Found: C, 10.3; H, 1.4; N, — $C_4H_4N_4O_8U$ required: C, 10.1; H, 0.85; N, —)
15. UO_2Cl_2, 2pyrazine, $8H_2O$	(Found: C, 14.8; H, 3.1; N, 8.1. $C_8H_{24}Cl_2N_4O_{10}U$ required: C, 15.0; H, 3.3; N, 8.7%)
16. UO_2Cl_2, 2pipy, $2H_2O^c$	(Found: C, 21.7; H, 5.0; N, 4.5. $C_{10}H_{26}Cl_2N_2O_4U$ required: C, 21.9; H, 4.8; N, 5.1%)
17. $UO_2(NO_3)_2$ 2pipy, $4H_2O$	(Found: C, 18.6; H, 4.7; N, 8.4. $C_{10}H_{30}N_4O_{12}U$ required: C, 18.9; H, 4.8; N, 8.8%)
18. UO_2Cl_2, 2py-N-oxide	(Found: C, 23.0; H, 1.7; N, 5.4. $C_{10}H_{10}Cl_2N_2O_4U$ required: C, 22.6; H, 1.9; N, 5.3%)
19. $UO_2(NO_3)_2$, 2py-N-oxide	(Found: C, 20.7; H, 1.8; N, 9.9. $C_{10}H_{10}N_4O_{10}U$ required: C, 20.6; H, 1.7; N, 9.6%)
20. $UO_2(NO_3)_2$, 2picolinic acid, $2H_2O$	(Found: C, 21.6; H, 2.5; N, 8.4. $C_{12}H_{14}N_4O_{14}U$ required: C, 21.3; H, 2.1; N, 8.3%)

[a] m.p. decomp. 191°C, m. 225°C; [b] m.p. 218°C. [c] m.p. 186°C. Other complexes do not melt up to 240°C.

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Bis(methyltriphenylphosphonium) tetrachlorodioxouranate(VI)

The addition of methyl triphenylphosphonium iodide in ethanol to an ethanolic solution of uranyl chloride saturated with HCl gave a yellow crystalline product, which was recrystallised several times from ethanol saturated with HCl. The product obtained was identified by analysis as $[MePh_3P]_2UO_2Cl_4$. This salt was previously reported,[6] but the method of preparation is different.

Reactions with 1,2-bis-(diphenylphosphino)ethane, (Diphosethane) and 1,1-bis-(diphenylphosphino)methane, (Diphosmethane)

The reaction of Diphosethane with uranyl chloride was studied in ethyl acetate. An impure sample of the dioxide adduct was obtained after washing with ethyl acetate. Recrystallisation of the impure sample from acetonitrile gave UO_2Cl_2(Diphosethanedioxide). The same adduct was obtained on using (Diphosethanedioxide) as a ligand. $UO_2(NO_3)_2$(Diphosethanedioxide) and $UO_2(NO_3)_2$(Diphosmethanedioxide).$2H_2O$ were obtained from the reactions of Diphosethane or Diphosmethane in ethyl acetate with uranyl nitrate in the same solvent. An authentic sample of the Diphosdioxides adduct was obtained on using Diphosethanedioxide and Diphosmethanedioxide as ligands.

Similarly, it is reported[18a,b] that the reaction between triphenylphosphine and uranium(VI) in the presence of air gave triphenylphosphine oxide complexes, UO_2X_2,-$2Ph_3PO$ (X = Cl, Br, and NO_3).

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Reactions of Uranium(VI) *With Compounds Containing Nitrogen, o-Phenanthroline(phen) and 2,9-Dimethyl-o-phenanthroline (DM-phen)*

The reactions of phen, and DM-phen with uranyl chloride were carried out, when the adducts UO_2Cl_2,2phen, $4H_2O$ and $UO_2Cl_2$2DM-phen,$2H_2O$, were obtained. When an ethyl acetate solution of phen was added to a solution of uranyl nitrate in the same solvent the product obtained proved by analysis and ir to be $UO_2(NO_3)_2$2-phen,EtAc. It seems that the organic ligand and the solvent compete fairly well in the coordination sphere of uranium(VI). The complex compound $UO_2(NO_3)_2$,2-DM-phen,$2H_2O$ was obtained from the reaction of uranyl nitrate with DM-phen. in ethyl acetate.

Reactions of uranyl chloride and uranyl nitrate with 2,2′,2″-terpyridyl

The adducts obtained on using 2,2′,2″-terpyridyl (terpy) as a ligand were $UO_2Cl_2$2-terpy,$4H_2O$ and $UO_2(NO_3)_2$1.5terpy,$8H_2O$, as found from the analysis and ir.

Reactions with 2,2′-bipyridyl(bipy) and 4,4′-dimethyl-2,2′-bipyridyl (DM-bipy)

When the reactions of bipy. and DM-bipy with uranyl chloride were studied in ethyl acetate the adducts $UO_2Cl_2$2bipy and $UO_2Cl_2$1.5 DM-bipy were obtained on using the mole ratio of UO_2Cl_2: ligand as 1 : 2. The complex compound $UO_2(NO_3)_2$,2bipy.,$4H_2O$ was obtained from the reaction of uranyl nitrate in ethyl acetate with bipy in the same solvent.

Dipyridyl amine (dipyamine) and Hexamine Complexes

The reactions of dipyridyl amine and hexamine with uranyl chloride in ethyl acetate gave the products UO_2Cl_2.2dipyamine4H_2O, and UO_2Cl_2.2hexamine.8H_2O, and $UO_2(NO_3)_2$.-dpyamine and $UO_2(NO_3)_2$.hexamine were obtained from the reaction with uranyl nitrate.

Reactions with Pyrazine and Piperidine (pipy)

The reactions of pyrazine and piperidine with uranyl chloride gave the products UO_2Cl_2.2pyrazine.8H_2O and UO_2Cl_2.2pipy.2H_2O. Uranyl nitrate reacted with pyrazine in ethyl acetate to give the product $UO_2(NO_3)_2$(pyrazine), at a mole ratio of $UO_2(NO_3)_2$: ligand of 1 : 1. The same product was obtained on using excess of the ligand. The complex compound $UO_2(NO_3)_2$.2pipy.4H_2O was obtained from the reaction of uranyl nitrate with piperidine.

Reaction of Uranyl chloride with Pyridine-N-oxide (Py-N-oxide)

The reaction of pyridine-*N*-oxide in ethyl acetate with uranyl chloride in the same solvent gave the product UO_2Cl_2.2py. *N*-oxide.

Reactions of Uranyl nitrate with Pyridine-N-oxide and Picolinic acid

Pyridine-*N*-oxide and picolinic acid reacted with uranyl nitrate in ethyl acetate to give the products $UO_2(NO_3)_2$.2-py-*N*-oxide and $UO_2(NO_3)_2$.2-icolinic acid.2H_2O, respectively.

DISCUSSION

Infrared Spectra

The infrared spectra of the compounds investigated are an important guide for identification of their structure. The spectra were measured between 650 and 4000 cm^{-1}. The configuration of the uranyl ion $(UO_2)^{2+}$ in simple salts and in its complex compounds was a subject of considerable interest in past years. On the basis of the X-ray and infrared data of a number of compounds containing $(UO_2)^{2+}$, Jones[19] derived a general formula relating the U=O distance with the U=O stretching force constant. From the infrared spectral evidence, McGlynn *et al.*[20] reported that in a series of complexes of uranyl compounds the stretching frequency of the U=O decreases on complex formation.

The U=O stretching band in UO_2Cl_2 and $UO_2(NO_3)_2$ occurs at 940 cm^{-1}. In salts containing the $(UO_2Cl_4)^{2-}$ anion or in the phosphineoxide complex compounds, UO_2Cl_2.2Ph_3PO, it occurs at 925 cm^{-1}, and the intensity of this band is comparable to that of C=O or P=O stretching bands. The P=O stretching band in Diphosethanedioxide or methanedioxide occurs at 1190 cm^{-1} as a very strong band.[21] This band shifts to lower frequency on complex formation, and is found at 1135 and 1140 cm^{-1} in the spectra of the adducts UO_2Cl_2.Diphosethanedioxide and UO_2Cl_2.Diphosmethanedioxide, respectively. The shifts for these complex ($\Delta\nu_{PO} \sim 55$ cm^{-1}) is almost identical to those found for similar complexes with $SnCl_4$ and $InCl_3$,[16,17] but are smaller than the shifts reported for U(IV) and

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Th(IV)[22] complexes with the same ligands ($\Delta\nu_{PO} \sim 125-100\ cm^{-1}$). In the case of the complex $UO_2(NO_3)_2$.Diphosmethanedioxide.$2H_2O$ this band appears at $1160\ cm^{-1}$.

Nitrogen Donors

The infrared spectra of the adducts obtained from the reaction of UO_2Cl_2 and $UO_2(NO_3)_2$ with the organic ligands phen 2,9-dimethyl-1,10-phenanthroline, 4,4′-dimethyl-2,2′-bipyridyl, 2,2′,2″-terpyridyl and pyrazine show certain deviations from those of the free ligands. The most clear were general shifts of the ring vibrations in the region 1650–1500 cm^{-1} to lower

TABLE II
Thermal decomposition of some uranyl chloride complexes

Temp. (°)	$M_{obs.}$	Formula	$M_{cal.}$
$UO_2Cl_2.2Ph_3PO$			
20–100	897.55	$UO_2Cl_2.2Ph_3PO$	897.55
160–180	861.6	$UO_2Cl_2.2Ph_3P$	865.55
510–800		volatile	
$[MePh_3P]_2UO_2Cl_4$			
20–280	966.5	$[MePh_3P]_2UO_2Cl_4$	966.5
500–530	339.6	UO_2Cl_2	340.95
550–800	261.2	UO_2	270
UO_2Cl_2.Diphosethanedioxide			
20–240	771.4	UO_2Cl_2.Diphosethanedioxide	771.4
320–340	740.5	UO_2Cl_2.Diphosethane	739.3
460–800	401.1	U, Ph_2P	423
UO_2Cl_2.2phen.$4H_2O$			
20–300	773.4–742.5	UO_2Cl_2.2phen.$2H_2O$	773.4–737.4
540–800	278.4	UO_2	270
UO_2Cl_2.2DM-phen.$2H_2O$			
20–60	793.5	UO_2Cl_2.2DM-phen.$2H_2O$	793.5
150–200	761.76	UO_2Cl_2.2DM-phen.	757.5
500–800	269.8	UO_2	270
UO_2Cl_2.2terpy.$4H_2O$			
20–70	879.5	UO_2Cl_2.2terpy.$2H_2O$	879.5
80–150	844.3	UO_2Cl_2.2terpy.$2H_2O$	843.5
510–800	225	U	238.04
UO_2Cl_2.1.5DM-bipy			
20–180	617.3	UO_2Cl_2.1.5DM-bipy	617.3
420–460	345.7	UO_2Cl_2	340.95
520–800	246	U	238.04
$UO_2.Cl_2$.2dipyamine.$4H_2O$			
20–280	755.4–694.9	UO_2Cl_2.2dipyamine.$4H_2O$	755.4–683.4
540–800	287.1	UO_3	286.04
UO_2Cl_2.2pyridine-N-oxide			
20–200	531.15	UO_2Cl_2.2pyridine-*N*-oxide	531.15
380–420	350.6	UO_2Cl_2	340.95
540–800	297.4	UO_3	286

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frequency. Multiple splittings were observed in the region 850–715 cm^{-1}. Similar behaviour of other metal complexes was observed by Taylor.[23]

Pyridine-N-oxide Complex

The frequency of the N—O group in pyridine-*N*-oxide occurs at 1180 cm^{-1} and shifts to 1190 cm^{-1} in the spectrum of its complex with uranyl chloride, UO_2Cl_2.2py-*N*-oxide. This indicates that the coordination is through the oxygen atom. Similar results were reported previously.[4]

Thermal Decomposition of Uranyl chloride and nitrate Complexes

Uranyl chloride as well as uranyl nitrate complexes decompose on heating. The molecular weights of the intermediate compounds have been calculated. The final products after heating up to 800°C are usually UO_3 and UO_2. Some of the uranyl chloride complexes volatilized. Table II shows the effect of heat on some of these complexes.

Conductivity Measurements

Conductivities of millimolar solutions of uranium(VI) chloride and nitrate complexes in acetonitrile, nitromethane and dimethylformamide have been measured. The conductivities reported for millimolar solutions of 1 : 1 electrolytes in these solvents are 150,[24] 70–120[25] and 70–100 ohm^{-1} cm^2. The results are recorded in Tables IIIa and b.

TABLE IIIa
Conductivities of some uranyl chloride complexes in different solvents

	ohm^{-1} cm^2		
Compound	MeCN	$MeNO_2$	DMF
$[MePh_3P]_2UO_2Cl_4$	—	348	—
UO_2Cl_2.Diphosethaneoxide	—	—	W[a]
UO_2Cl_2.2phen.$4H_2O$	—	—	W
$UO_2Cl_2$2DM-phen.$2H_2O$	—	87	—
$UO_2Cl_2$2terpy.$4H_2O$	—	—	W
UO_2Cl_2.2bipy	144	—	—
$UO_2Cl_2$1.5DM-bipy	W	—	—
UO_2CL_2.2dipyamine.$4H_2O$	—	—	117
UO_2Cl_2.2py-*N*-oxide	—	104.5	—
UO_2Cl_2.$3Ph_2SO$	—	—	W

[a]W = weak electrolyte.

All the adducts which are soluble in acetonitrile behave as weak electrolytes except the complex UO_2Cl_2.2bipy which behaves as strong electrolyte. When nitromethane was used as a solvent the salt $[MePh_3]_2UO_2Cl_4$ shows great ionization indicating that it dissociates into the ions $[MePh_3P]^+$ and $[UO_2Cl_4]^{2-}$. The adducts UO_2Cl_2.2DM-phen.$2H_2O$ and UO_2Cl_2.2py-*N*-oxide behave as strong electrolytes. The solution of the complexes UO_2Cl_2.2dipyamine.$4H_2O$, and $UO_2(NO_3)_2$.2phen.EtAc, in dimethylformamide show that they also behave as strong electrolytes.

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TABLE IIIb
Conductivities of some uranyl nitrate complexes in different solvents

	ohm^{-1} cm^2		
Compound	MeCN	$MeNO_2$	DMF
$UO_2(NO_3)_2.2Ph_3PO.2H_2O$	—	—	W[a]
$UO_2(NO_3)_2$.Diphosethanedioxide	—	—	W
$UO_2(NO_3)_2$.Diphosmethaed oxide.$2H_2O$	W	—	—
$UO_2(NO_3)_2$.2phen.EtAc	—	—	54.8
$UO_2(NO_3)_2$.1.5terpy.$8H_2O$	—	W	—
$UO_2(NO_3)_2$.2bipy.$4H_2O$	—	—	W
$UO_2(NO_3)_2$.dipyamine	—	—	W
$UO_2(NO_3)_2$.2py-*N*-oxide	—	W	—
$UO_2(NO_3)_2$.2picolinie acid.$2H_2O$	—	—	W
$UO_2(NO_3)_2.3Ph_2SO$	—	—	W

[a]W = weak electrolyte.

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