

Complexes of Dioxouranium(VI) with Zwitter-Ionic Forms of Bi- and Tetra-dentate *Schiff* Bases

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Potentially bi- and tetra-dentate *Schiff* bases derived from salicylaldehyde react with hydrated uranyl salts to give complexes: $\text{UO}_2\text{H}_2\text{LX}_2$, $\text{UO}_2\text{H}_2\text{L}'\text{X}_2$ and $\text{UO}_2(\text{HL})_2\text{X}_2$ [$\text{H}_2\text{L} = \text{N}, \text{N}'$ -propane-1,3-diylbis(salicylideneimine), $\text{H}_2\text{L}' = \text{N}, \text{N}'$ -ethylenebis(salicylideneimine) and $\text{HL} = \text{N}$ -phenylsalicylideneimine; $\text{X}^- = \text{Cl}^-$, Br^- , I^- , NO_3^- , ClO_4^- , and NCS^-]. Because of marked spectral similarities with the structurally known $\text{Ca}(\text{H}_2\text{L})(\text{NO}_3)_2$, the *Schiff* bases are coordinated through the negatively charged phenolic oxygen atoms and not the nitrogen atoms of the azomethine groups which carry the protons transferred from phenolic groups on coordination. Halide, nitrate, perchlorate and thiocyanate groups are covalently bonded to the uranyl ion, resulting a 6-coordinated uranium ion in the halo and thiocyanato complexes and 8-coordinated in nitrate and perchlorato complexes.

(Keywords: Complexes; Dioxouranium; Infra red; *Schiff* base complexes; Zwitter ion)

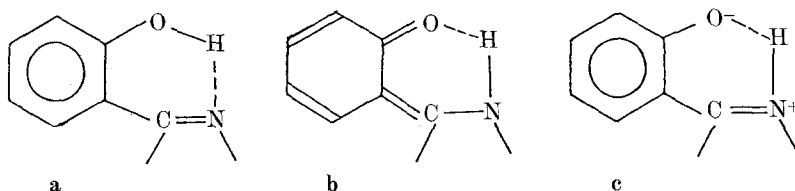
Komplexe von Dioxouranyl(VI) mit zwitterionischen Formen von zwei- und vierzähligen *Schiff*-Basen

Von Salizylaldehyd abgeleitete zwei- und vierzählige *Schiff*-Basen reagieren mit hydratisierten Uranylsalzen zu Komplexen folgenden Typs: $\text{UO}_2\text{H}_2\text{LX}_2$, $\text{UO}_2\text{H}_2\text{L}'\text{X}_2$ und $\text{UO}_2(\text{HL})_2\text{X}_2$ [$\text{H}_2\text{L} = \text{N}, \text{N}'$ -Propan-1,3-diylbis(salicylidenimin), $\text{H}_2\text{L}' = \text{N}, \text{N}'$ -Ethylen-bis(salicylidenimin) und $\text{HL} = \text{N}$ -Phenylsalicylidenimin; $\text{X}^- = \text{Cl}^-$, Br^- , I^- , NO_3^- , ClO_4^- und NCS^-]. Auf Grund eindeutiger spektraler Ähnlichkeiten mit dem bekannten $\text{Ca}(\text{H}_2\text{L})(\text{NO}_3)_2$ wird auf Koordination über die negativ geladenen phenolischen Sauerstoffatome (und nicht über die Azomethin-Stickstoffe) geschlossen. Die Anionen X^- sind kovalent an das Uranyl-Ion gebunden; damit ergibt sich ein hexakoordiniertes Uranyl-Ion für die Halogen- und Thiocyanat-Komplexe und Oktakoordination für die Nitrat- und Perchlorat-Komplexe.

Introduction

There have been many reports¹⁻¹⁰ of U(IV) and U(VI) complexes with non-ionised *Schiff* bases mainly derived from salicylaldehyde, but the mode of coordination of the non-ionised base has been the subject of much controversy. *Doretto* et al.²⁻⁴ suppose that the *Schiff* base is coordinated through the azomethine nitrogen atoms only; evidence for this came from infrared spectroscopy. Whereas others⁵⁻¹⁰ interpreted essentially identical infrared results in terms of coordination through both nitrogen and phenolic oxygen atoms. Until recently no crystallographic work was reported to resolve the problem.

The crystal structure¹¹ of $\text{Ca}(\text{H}_2\text{L})(\text{NO}_3)_2$, (H_2L = see abstract) has been determined recently, in which the *Schiff* base is best described by the tautomer **c**, namely, the charge separated (zwitter-ionic) form as well as two other possible tautomeric conversions, an enol-imine **a** to ketoenamine **b** which was reported¹². Co-ordination takes place through the negatively charged phenolic oxygen atoms (and not the nitrogen atoms of the azomethine groups). The changes observed on comparing the infrared spectra of this compound and the free *Schiff* base in the C=N stretching region are the same for the present work and the other¹⁻¹⁰ complexes.



Experimental

Materials. Hydrated $\text{UO}_2(\text{NO}_3)_2$ and UO_2Cl_2 were (BDH) reagent grade and used as supplied. Ethanolic solution of UO_2Br_2 , UO_2I_2 , $\text{UO}_2(\text{NCS})_2$ and $\text{UO}_2(\text{ClO}_4)_2$ ¹³⁻¹⁵ were obtained by metathesis of $\text{UO}_2(\text{NO}_3)_2$ or UO_2Cl_2 with BaBr_2 , BaI_2 , KNCS and AgClO_4 respectively, and filtering off the insoluble metal nitrate or chloride and silver chloride. The ligands were prepared by routine method¹⁶.

Synthesis of the Complexes. Most of the complexes were prepared by the addition of the stoichiometric amount of the uranyl salt in hot tetrahydrofuran (*THF*) or ethanol to the solution of the ligand in the same solvent. A yellow precipitate formed almost immediately, this was filtered off and washed with *THF* several times and dried (CaCl_2). The ionised compounds were prepared by refluxing the stoichiometric amount of the hydrated uranyl acetate and the *Schiff* base in dimethylformamide (*DMF*) for two h. An orange

precipitate was formed, this was filtered off and washed with ether and dried (CaCl_2). The compounds are not soluble in common organic solvents, only dissolve in *DMF* and dimethylsulphoxide (*DMSO*) and sparingly in hot alcohol.

Measurements. The infrared spectra in the region $4000\text{--}200\text{ cm}^{-1}$ were recorded by using a Perkin-Elmer 598 as nujol and hexachlorobutadien mulls. Conductance measurements were carried out at room temperature (300°K) in propanol and *DMF* solution ($10^{-3}M$) with a conductivity type CDM2e No. 207421 produced by a radiometer company in Copenhagen.

Analysis. Uranium was determined compleximetrically¹⁷. Halides and thiocyanate as silver halide and silver thiocyanate. Nitrate and perchlorate analysed by precipitation with nitron¹⁸. The nitrogen was estimated by a standard method¹⁷.

Results and Discussion

21 *Schiff* base complexes of dioxouranium(VI) described here are presented with analytical data and millimolar conductance in Table 1. The low molar conductivity in propanol solution indicating of the non-electrolytic¹⁹ behaviour of the complexes in this solvent, whereas the high values observed in dimethylformamide solution show considerable dissociation due to some ligand exchange between the strong donor *DMF* and the weakly bonded anions in the coordination sphere of the uranyl ion in this solvent²⁰.

Infrared spectroscopy

(a) $4000\text{--}2500\text{ cm}^{-1}$

The free *Schiff* base shows a strong intramolecular hydrogen bonding ($\text{OH}\dots\text{N}$) which gives rise to a broad absorption near 2700 cm^{-1} (Ref. ²¹). This band shifts to 3000 cm^{-1} in the complexes as hydrogen bonding arrangements change ($\text{O}\dots\text{H}+\text{N}$)¹¹. On comparing the infrared spectra of the complexes with the fully ionised *Schiff* base compounds, there is neither absorption near 2700 cm^{-1} nor at 3000 cm^{-1} only the aliphatic and the aromatic C—H vibrations observed; suggesting the coordination of the neutral ligands in these series of non-ionised *Schiff* base complexes.

(b) $1700\text{--}600\text{ cm}^{-1}$

The C=N stretching vibration for the free ligands and the complexes compared with $\text{Ca}(\text{H}_2\text{L})(\text{NO}_3)_2$ in Table 2 and Fig. 1. The other compounds had very similar spectra in this region¹⁻¹¹, the C=N stretching vibration of the free base increased in frequency (by $5\text{--}30\text{ cm}^{-1}$) on complex formation. It has been shown²² that $\text{C}=\text{N}^+$ groups have in general higher frequencies than the parent C=N groups,

Table 1. Analytical data and millimolar conductance data of dioxouranium complexes

Complexes	M%	X%	N%	Molar Conductance* ($\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$) in <i>n</i> -propanol	Molar Conductance* ($\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$) in <i>DMF</i>
$\text{UO}_2(\text{H}_2\text{L})\text{Cl}_2$	38.85 (39.05)	11.52 (11.63)	4.45 (4.59)	10.50	34.22
$\text{UO}_2(\text{H}_2\text{L})\text{Br}_2$	34.00 (34.12)	22.70 (22.91)	3.85 (4.01)	19.20	113.00
$\text{UO}_2(\text{H}_2\text{L})\text{I}_2$	29.80 (30.03)	31.75 (32.02)	3.70 (3.53)	24.60	150.00
$\text{UO}_2(\text{H}_2\text{L})(\text{NO}_3)_2$	35.75 (35.92)	18.50 (18.71)	—	16.30	137.00
$\text{UO}_2(\text{H}_2\text{L})(\text{ClO}_4)_2$	32.10 (32.27)	26.72 (26.96)	3.55 (3.79)	11.30	110.00
$\text{UO}_2(\text{H}_2\text{L})(\text{NCS})_2$	36.20 (36.35)	17.50 (17.73)	8.45 (8.56)	16.10	75.60
$\text{UO}_2\text{L} \cdot \text{DMF}$	38.90 (39.03)	—	6.75 (6.89)	1.30	1.22
$\text{UO}_2(\text{H}_2\text{L})\text{Cl}_2$	37.90 (38.17)	11.40 (11.36)	4.35 (4.49)	8.60	24.50
$\text{UO}_2(\text{H}_2\text{L})\text{Br}_2$	33.25 (33.40)	22.20 (22.42)	3.80 (3.93)	20.50	102.00
$\text{UO}_2(\text{H}_2\text{L})\text{I}_2$	29.35 (29.51)	31.20 (31.46)	3.35 (3.47)	31.50	100.00
$\text{UO}_2(\text{H}_2\text{L})(\text{NO}_3)_2$	34.95 (35.17)	18.10 (18.32)	—	25.40	128.00
$\text{UO}_2(\text{H}_2\text{L})(\text{ClO}_4)_2$	31.45 (31.67)	26.30 (26.46)	3.60 (3.72)	22.30	99.70
$\text{UO}_2(\text{H}_2\text{L})(\text{NCS})_2$	35.30 (35.58)	17.10 (17.36)	8.15 (8.38)	19.40	67.30
$\text{UO}_2\text{L} \cdot \text{DMF}$	37.85 (38.16)	—	6.59 (6.73)	0.42	1.15
$\text{UO}_2(\text{HL})_2\text{Cl}_2$	32.10 (23.36)	9.80 (9.64)	3.65 (3.80)	12.20	21.60
$\text{UO}_2(\text{HL})_2\text{Br}_2$	28.65 (28.87)	19.50 (19.38)	3.20 (3.39)	24.10	87.50
$\text{UO}_2(\text{HL})_2\text{I}_2$	26.70 (25.92)	27.45 (27.63)	2.90 (3.05)	37.20	107.00
$\text{UO}_2(\text{HL})_2(\text{NO}_3)_2$	29.95 (30.18)	15.50 (15.72)	—	13.70	120.00
$\text{UO}_2(\text{HL})_2(\text{ClO}_4)_2$	27.37 (27.56)	22.85 (23.03)	3.10 (3.24)	19.20	25.20
$\text{UO}_2(\text{HL})_2(\text{NCS})_2$	30.15 (30.49)	14.60 (14.87)	6.90 (7.17)	15.20	80.40
UO_2L_2	35.55 (35.82)	—	4.10 (4.22)	2.20	14.30

* Millimolar conductance of the known compound $\text{CaH}_2\text{L}(\text{NO}_3)_2$ in *n*-propanol and *DMF* solutions were 8.5 and 110 ($\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$) respectively.

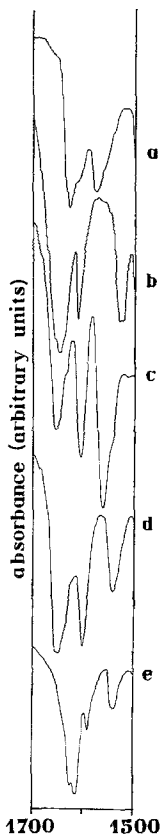


Fig. 1. Infra-red spectra for the ligand and the metal complexes in the region 1700—1500 cm^{-1} for *a* H_2L ; *b* $\text{CaH}_2\text{L}(\text{NO}_3)_2$; *c* $\text{UO}_2\text{H}_2\text{L}(\text{NO}_3)_2$; *d* $\text{UO}_2\text{H}_2\text{LI}_2$; *e* UO_2L

thus the results are consistent with the presence of charge-separated forms, and therefore these and the other complexes¹⁻¹¹ are likely to be structurally related in that the coordination takes place through the negatively charged phenolic oxygen atoms rather than the positively charged nitrogen atoms of the azomethine groups. A sharp absorption at 1280 cm^{-1} assignable to C—O stretching vibration in the free ligands changes little on complex formation (Table 2). The C—O stretching vibration must include considerable contribution from the skeletal vibrations of the aromatic ring, and because the vibration changed little in the complexes it was suggested¹¹ that the bonding arrangements of the oxygen atoms were the same in the free ligands and the complexes.

Table 2. Selected infrared bands (cm^{-1}) of the ligands and dioxouranium(VI) complexes

Complexes	C=N	C=O	$\nu_3 \text{UO}_2$	$\nu_2 \text{UO}_2$	U—O—ligand	other bands
H_3L	1630 s	1280 s	—	—	—	—
$\text{CaH}_2\text{L}(\text{NO}_3)_2$	1645 vs	1280 m	—	—	—	—
$\text{UO}_2\text{H}_2\text{L}(\text{NO}_3)_2$	1655 vs	1280 m	920 s	260 s	450 s	400 s U—O ₂ NO
$\text{UO}_2\text{H}_2\text{LCl}_2$	1660 vs	1290 s	920 vs	—	405 s	260 s U—Cl
$\text{UO}_2\text{H}_2\text{LBr}_2$	1655 vs	1285 s	900 s	255 s	400 s	—
$\text{UO}_2\text{H}_2\text{LI}_2$	1650 vs	1280 s	895 s	260 s	400 s	—
$\text{UO}_2\text{H}_2\text{L}(\text{NCS})_2$	1648 vs	1282 bb	900 s	260 s	410 s	—
$\text{UO}_2\text{H}_2\text{L}(\text{ClO}_4)_2$	1655 vs	1285 s	900 vs	260 s	400 s	—
UO_2LDMF	1625 vs	1300 vs	900 vs	240 m	370 vs	450 s U—N
$\text{H}_2\text{L}'$	1630 s	1280 s	—	—	—	—
$\text{UO}_2\text{H}_2\text{L}'(\text{NO}_3)_2$	1650 vs	1280 m	920 vs	250 s	460 s	380 s U—O ₂ NO
$\text{UO}_2\text{H}_2\text{L}'\text{Cl}_2$	1650 vs	1290 s	920 vs	—	365 s	250 s U—Cl
$\text{UO}_2\text{H}_2\text{L}'\text{Br}_2$	1660 vs	1280 s	910 vs	250 s	390 s	—
$\text{UO}_2\text{H}_2\text{L}'\text{I}_2$	1640 vs	1285 m	910 vs	240 m	370 s	—
$\text{UO}_2\text{H}_2\text{L}'(\text{NCS})_2$	1635 vs	1280 s	920 vs	250 m	380 s	—
$\text{UO}_2\text{H}_2\text{L}'(\text{ClO}_4)_2$	1640 vs	1285 vs	890 vs	260 s	375 s	—
$\text{UO}_2\text{L}'\text{DMF}$	1620 vs	1310 s	900 vs	260 s	380 s	460 s U—N
HL	1620 s	1280 s	—	—	—	—
$\text{UO}_2(\text{HL})_2(\text{NO}_3)_2$	1650 vs	1260 s	930 vs	255 s	450 s	300 s U—O ₂ NO
$\text{UO}_2(\text{HL})_2\text{Cl}_2$	1650 vs	1290 vs	925 vs	—	350 s	240 s U—Cl
$\text{UO}_2(\text{HL})_2\text{Br}_2$	1640 vs	1280 s	920 vs	250 s	330 s	—
$\text{UO}_2(\text{HL})_2\text{I}_2$	1640 vs	1275 s	920 vs	250 s	335 m	—
$\text{UO}_2(\text{HL})_2(\text{NCS})_2$	1630 vs	1290 m	890 s	260 s	330 m	—
$\text{UO}_2(\text{HL})_2(\text{ClO}_4)_2$	1640 vs	1280 s	920 vs	250 s	340 m	—

s, strong; b, broad; v, very; m, medium.

On examining the infrared spectra of these complexes and the fully ionised *Schiff* base compounds one can see that the C=N stretching frequency of the ionised ligand has fallen on complex formation and the C—O stretching frequency increased to a higher frequency (Table 2 and Fig. 1). Therefore the results are consistent with those of the structurally known ionised compounds²³⁻²⁴ where the coordination took place via oxygen atoms and the nitrogen atoms of the azomethine groups.

Nitrato Complexes. The infrared spectra of three nitrato complexes showed no strong and broad absorption band near 1380 cm^{-1} where the free nitrate ion is known to absorb²⁵. However on comparing the spectra of the free ligands and the halide complexes with the nitrato complexes, the nitrate spectra were dominated by the strong absorption from ν_1 ($1480\text{--}1500\text{ cm}^{-1}$), ν_2 ($1020\text{--}1030\text{ cm}^{-1}$), ν_4 ($1280\text{--}1300\text{ cm}^{-1}$) and ν_6 ($810\text{--}820\text{ cm}^{-1}$) of the nitrato group in C_{2v} symmetry²⁵. Since these spectra were essentially the same as that¹¹ of $\text{Ca}(\text{H}_2\text{L})(\text{NO}_3)_2$ and the other²⁵⁻²⁶ compounds, the nitrato groups are bidentate in these series of *Schiff* base complexes. The weak fundamentals ν_3 and ν_5 were masked by the strong ligand absorption ($700\text{--}800\text{ cm}^{-1}$).

Thiocyanato Complexes. The thiocyanato complexes exhibited very strong absorption band between $2050\text{--}2100\text{ cm}^{-1}$ corresponding to previous studies on the empirical ranges for the observed bands²⁷ for both S-bonding (thiocyanato-) or N-bonding (isothiocyanato-) indicated that all the complexes contain N-bonded thiocyanate groups. The same conclusion came from the consideration of the C—S stretching ($800\text{--}830\text{ cm}^{-1}$) and N—C—S deformation ($460\text{--}480\text{ cm}^{-1}$) and thus the class A character of the uranyl ion is emphasized.

Perchlorato Complexes. The two perchlorato complexes: $\text{UO}_2\text{H}_2\text{L}(\text{ClO}_4)_2$ and $\text{UO}_2\text{H}_2\text{L}'(\text{ClO}_4)_2$ showed strong absorption bands at $1150, 1100, 1030, 965, 945, 620, 650, 470\text{ cm}^{-1}$ indicating that the perchlorato group must act as a bidentate ligand on C_{2v} symmetry²⁸ in these compounds. Since in $\text{UO}_2(\text{HL})_2(\text{ClO}_4)_2$ the strong ligand absorption bands obscured the perchlorate absorption bands in the region interested it was not possible to draw any conclusion on the bonding mode of the perchlorate group, only the low molar conductivity of the said compound indicates the non-ionic character of the perchlorate group.

The infrared bands due to the UO_2^{+2} group are observed near 900 and 800 cm^{-1} as strong and weak bands corresponding to asymmetric stretching frequency ν_3 (UO_2) and the symmetric stretching frequency ν_1 (UO_2) respectively. The bending vibrations, ν_2 (OUO) are obtained at

250–270 cm^{-1} region and they are listed in Table 2. These observations indicate that the UO_2^{+2} moiety in the present compounds is virtually linear.

The two additional bands found near 1660 cm^{-1} and 650 cm^{-1} in the spectra of the ionised *Schiff* base compounds, UO_2LDMF and $\text{UO}_2L'\text{DMF}$ were assigned to the coordinated dimethylformamide²⁹ in these compounds.

(c) 600–200 cm^{-1}

Halide Complexes. The far i.r. spectra of the chloro complexes showed a strong absorption band in the region of the U—Cl ^{30–31} stretching vibration between 240–260 cm^{-1} (Table 2). We were not able to find the absorption bands due to the other metal-halide (U—Br and U—I) stretching frequencies which probably lie below 200 cm^{-1} (Ref.³¹).

The non-ligand bands occurring in 450–350 cm^{-1} and 300–400 cm^{-1} in the spectra of the non-ionised *Schiff* base complexes may be tentatively assigned to ν (U—O -ligand) and ν ($\text{U—O}_2\text{NO}$) modes respectively³¹ while an additional band found at 400–500 cm^{-1} in the spectra of the ionised compounds could be attributed to the U—N stretching frequency (see Table 2).

Conclusion

On the basis of the properties of the complexes studied some features can be emphasised: (a) the non-ionised *Schiff* base coordinated through the negatively charged phenolic oxygen atoms only. (b) The halo and thiocyanato groups bonded unidentally while the nitrato and perchlorato groups are bidentate, suggesting the 6-coordinated uranium in halo and iso-thiocyanato and 8-coordinated in nitrato and perchlorato complexes.

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