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Crystal Structure of Dichloro Dinitro Oxouranium(VI) Lignocaine Complex

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Crystal and molecular structure of Dichloro dinitro oxouranium(VI) lignocaine complex, $C_{28}H_{44}O_{10}N_6Cl_2U$, is reported. The sample crystallizes in the monoclinic space group $P2_1/a$ with $a = 15.719(3)$ Å, $b = 12.363(5)$ Å, $c = 19.647(4)$ Å, $\beta = 101.34(2)°$, $V = 3744(2)$ Å^3, $Z = 4$. The structure was solved using 6916 reflections measured with CuKα radiation. The stucture was refined to a final wR of 0.2391 ($R = 0.087$). The two nitrate groups bound to the uranium atom are planar and are almost perpendicular to each other. The packing of the molecules shows a layered arrangement.

Keywords: Complexes, crystal structure, lignocaine, uranium

INTRODUCTION

Literature survey reveals that uranyl complexes with different ligands have been studied[1–11]. Studies of complexes of uranium with ligands which are medicinally important are relatively few. Antifungal activity of dioxouranium(VI) with *N*-alkyl-phenothiazines is determined.[12] In this paper, we report the crystal structure of the complex of lignocaine hydrochloride with uranium nitrate.

EXPERIMENTAL

Good single crystals were obtained by slow evaporation technique in ethanol medium. The crystals were yellow in colour. The cell parameters were refined using 25 reflections in the 2θ range 40–50° on an Enraf Nonius CAD4 diffractometer with CuKα radiation. $\omega - 2\theta$ scan was employed for data collection. The crystal data are given in Table 1. Data were collected up to a 2θ maximum of 149.5°. A total of 7696 reflections were measured of which 7662 were unique. Lorentz and polarization corrections were

TABLE 1

Crystal Data

Empirical formula	$C_{28}H_{44}O_{10}N_6Cl_2U$
Formula weight	933.62
Temperature	293(2)K
Space group	$P2_1/a$
Unit cell dimensions	$a = 15.719(3)$Å $b = 12.363(5)$Å, $\beta = 101.34(2)°$ $c = 19.647(4)$Å
Volume	3744(2)Å^3
Z	4
Density (calculated)	1.657 Mg/m^3
μ	14.009 mm^{-1}
F_{000}	1840
Crystal size	0.50 × 0.40 × 0.30 mm
θ range for data collection	2.29°–74.76°
Index ranges	$-19 \leq h \leq 19, 0 \leq k \leq 15, 0 \leq l \leq 24$

applied. An empirical absorption correction was made. The minimum and maximum transmission factors were 0.71 and 0.98 respectively.[13] Wilson plot[14] gave the value of U as 0.06Å^2, where U is the mean-square amplitude of atomic vibration. Out of the 7662 unique reflections 6916 reflections were found with $I > 2\sigma(I)$.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved using SHELXS-86[15] and refined by full-matrix least squares refinement using SHELXL-93.[16] The hydrogen atoms were placed at calculated positions and refined using the riding model. Weights based on counting statistics were used. Extinction coefficient was also refined.[17] Refinement was on F^2 for all the 7662 reflections except for 12 with very negative F^2 which were omitted for potential

TABLE 2

Details of the Final Least-Squares Cycle

Refinement	Full-matrix least-squares on F^2
Function minimized	$\sqrt{\frac{\sum w(F_o^2 - F_c^2)^2}{\sum w(F_o^2)^2}}$
Least-squares weights	Counting statistics
Number of reflections	7650 reflections
Number of atoms	91
Number of parameters	433
R (all reflections)	0.0925
R (observed reflections)	0.0868
wR (all reflections)	0.2496
wR (observed reflections)	0.2391
Goodness of fit (all)	1.028
Goodness of fit (observed)	1.050
Maximum shift/sigma	4.421
Extinction coefficient	0.0014(5)
Maximum and minimum peak in the final difference map	2.01 and -3.55 e Å^{-3}

systematic errors. Weighted R factors, wR and all goodnesses of fit are based on F^2. The conventional R-factors are based on F, with F set to zero for negative F^2. The details of the last least-squares refinement and the subsequent difference Fourier map are given in Table 2.

RESULTS AND DISCUSSION

The final positional parameters, thermal parameters, bond lengths and angles are given in Tables 3–6. The bond lengths and bond angles are in good agreement with standard values. The U—Cl bond lengths are similar to those for U—Br reported by Spirlet *et al.*[8] The uranyl U—O bond distances are 1.773 Å (U—O1) and 1.766 Å (U—O2). These values agree well with the values obtained by neutron diffraction studies of Eller and Penneman[18] and Sitran *et al.*[1] It differs slightly from those quoted by Charpin *et al.*[7,9] and Cousson *et al.*[10]

The projection of the molecule on the best plane is shown in Figure 1. The two nitrate groups bound to the uranium are not symmetrical about the uranium atom. The nitrate groups are planar and are nearly perpendicular to each other. The uranyl group exists separately between the two ligands and the oxygens of the linear uranyl group are coordinated only to uranium. The seven coordination of the uranium is completed by five oxygen atoms and two chlorine atoms. The coordination polyhedron is severely strained which is evident from the bond angles given in Table 6. The packing of the molecules down **b** is shown in Figure 2. The molecules show a partial layered arrangement with ligands of one layer penetrating into the other layer unlike in the case of the complexes of platinum chloride[19] and zinc chloride[20] with lignocaine hydrochloride. The high thermal parameters of the terminal carbon atoms may be surmised as due to the increased flexibility of the chain. The nitrogen atom N1 bound to uranium is disordered which accounts for the high residual peak which occurs in the vicinity of the atom N1.

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