atom is bound to two bridging sulfur atoms (SB), two sulfur donor atoms from the diethyldithiocarbamate ligands (SL), and one terminal sulfur atom (ST).

Of particular interest are the Mo1–ST1 and Mo2–ST2 bond lengths which average 2.092 Å. This is well within the range that we have reported[4] for *syn*- and *anti*-$[Mo_2S_4(dme)_2]^{2-}$. The dimer exhibits the usual sharing of an edge by two square-pyramidal five-coordinate Mo(V) centers with the apex atoms, ST1 and ST2, in the syn conformation.[4] Each molybdenum atom is displaced from the basal plane of its square pyramid toward ST1 or ST2 by 0.73 and 0.72 Å, respectively. The dihedral angle between planes formed by SB1–Mo1–SB2 and SB1–Mo2–SB2 is 147.9 (2)°. The average Mo–SL distance is 2.453 (4) Å.

Except for the disordered C9–C10 ethyl group, the diethyldithiocarbamate ligands present no unusual geometries. There are five residual electron density peaks within 1 Å of C9, but no combination of these positions yields a more reasonable geometry for the disordered ethyl group, which lies in an open pocket between molecules.

The primary interest in this investigation are the Mo–ST bond distances. We concur with Spivack, Dori, and Stiefel[6] that such distances are substantially shorter than Mo–S single bonds of ~2.45 Å which have been observed for dithiocarbamate,[6] 1,2-dimercaptoethane,[4] and cysteine[17,18] ligands. Thus, terminal sulfido groups can be written as Mo=S, analogous to the well-known molybdenyl group, Mo=O. However, our five determinations of Mo=S distances in three different complexes[4] containing $[Mo_2S_4]^{2+}$ moieties give Mo=S distances in the range 2.085–2.129 Å, with an average distance of 2.10 (2) Å. These Mo=S distances are more than 0.1 Å longer than those given in the preliminary report[6] of the structure of $Mo_2S_4(S_2CN(n\text{-}C_4H_9)_2)_2$.

Acknowledgment. We thank Dr. William E. Newton for a sample of the compound. We thank Dr. K. O. Hodgson for a preprint of ref 3. We gratefully acknowledge support of this research by the National Institutes of Health (Grant ES-00966) and a generous allocation of computer time by the University of Arizona Computer Center. We thank the National Science Foundation for funds to purchase the diffractometer (Grant CHE 76-05481).

Registry No. $[Mo_2S_4(S_2CN(C_2H_5)_2)_2]$, 67814-43-1.

Supplementary Material Available: Table II, atomic thermal parameters; Table III, hydrogen parameters; Table IV, listings of the structure factor amplitudes ×10 (9 pages). Ordering information is given on any current masthead page.

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Contribution from the Materials and Molecular
Research Division, Lawrence Berkeley Laboratory,
and Department of Chemistry, University of California,
Berkeley, California 94720

Structure of Diethylammonium Ethoxybis(diethylmonothiocarbamato)dioxouranate(VI), $(C_2H_5)_2NH_2^+[UO_2((C_2H_5)_2NCOS)_2OC_2H_5]^-$ [1]

Dale L. Perry,*[2] David H. Templeton,* and Allan Zalkin*

Received May 25, 1978

While a rather large number of metal dithiocarbamate complexes[3,4] have been extensively investigated, relatively little attention has been given to the analogous monothiocarbamate species. This is particularly unfortunate in light of recent reports[5-9] which indicate that the chemistry of this class of compounds will be as interesting and novel as that of the corresponding dithiocarbamates. The tris(dialkylmonothiocarbamato)iron(III) chelates,[5] $Fe(R_2mtc)_3$, for example, have been shown to exhibit anomalous magnetic behavior arising from ^2T(low spin, $S = {}^1/_2$) $\rightleftarrows$ ^6A(high spin, $S = {}^5/_2$) spin-state interconversion processes. As a result, these compounds with their FeS_3O_3 metal centers are electronically and structurally similar to their iron(III) dithiocarbamate analogues containing the FeS_6 core.

In the uranyl monothiocarbamate alkoxide series[6] of complexes, however, one sees several features that are markedly different from those displayed by the corresponding dithiocarbamates. The reaction of dialkylammonium monothiocarbamates with uranyl salts affords a new synthetic route for the preparation of uranyl alkoxides; also, these compounds represent the first examples of a mixed alkoxide–bidentate chelate system (where the donor atoms are sulfur and oxygen) of an actinide ion. Since these compounds also contain three different types of uranium–oxygen bonds in the same molecule, their structural and bonding parameters are of considerable interest. The present paper represents the first structural investigation of this class of uranyl compounds and is part of an ongoing research program centered around the chemistry of the lanthanide and actinide–sulfur bond.

Experimental Section

The title compound was prepared according to a method described elsewhere[6] which essentially consisted of bubbling carbonyl sulfide (Matheson, 97.5% purity) through a solution of diethylamine in ethanol at 0 °C for 5–10 min followed by the addition of a saturated ethanolic solution of $UO_2Cl_2 \cdot 3H_2O$ (Alfa Products) with stirring. The bright yellow complex that precipitated from solution was filtered on a Büchner funnel and air-dried. The yield was 60% or better. Anal. Calcd for $(C_2H_5)_2NH_2^+[UO_2((C_2H_5)_2NCOS)_2OC_2H_5]^-$: C, 29.46; H, 5.67; N, 6.44; S, 9.81. Found: C, 29.47; H, 5.64; N, 6.42; S, 9.38.

A crystal suitable for data collection was inserted into a 0.2-mm quartz capillary and sealed. It was later ascertained that the crystals are stable in air and the precaution of sealing them in capillaries was not necessary. The crystal had the shape of a hexagonal prism with dimensions approximately 0.2 mm in cross section and 0.25 mm long. It was examined with a Picker FACS-I automatic diffractometer equipped with a graphite monochromator and a Mo X-ray tube ($\lambda(K\alpha_1)$ 0.709 30 Å). ω scans of several low-angle reflections showed peaks with half-widths of 0.09–0.12°. The space group is $P2_1/n$. The setting angles of 12 manually centered reflections ($39° < 2\theta < 45°$) were used to determine by least squares the following cell parameters: $a = 9.518$ (4) Å, $b = 12.457$ (5) Å, $c = 22.490$ (10) Å, $\beta = 93.77$

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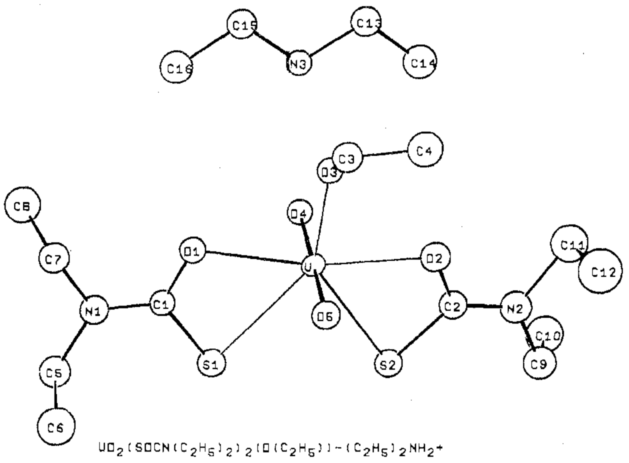

Figure 1. ORTEP view of the structure.

(5)°, and $V = 2661$ Å^3. For $Z = 4$ and a molecular weight of 653.6 the calculated density is 1.63 g cm^{-3}.

Intensity data were collected using the θ–2θ scan technique with a scan speed of 2°/min on 2θ. Each peak was scanned 0.7° before the Kα_1 peak to 0.7° after the Kα_2 peak, and backgrounds were counted for 4 s at each end of the scan range. The temperature during data collection was 23 ± 1 °C. Three standard reflections were measured after every 200th scan. A total of 10257 scans were performed of which over 3400 were rejected because of misalignment due to the crystal moving in the capillary. Some were rejected because the proximate standard reflections had low values and others whenever the ratio of the two background counts exceeded 1.5. Additional measurements were made to avoid gaps in the data. The 6357 scans, excluding the standard reflections, resulted in 4622 unique reflections of which 2542 had $I > 3\sigma$. No absorption correction was applied, the maximum and the minimum transmission factors are estimated to be 0.40 and 0.34, respectively, for an estimated absorption coefficient of 60 cm^{-1}.

A three-dimensional Patterson calculation showed the positions of the uranium atom, two sulfur atoms, and two oxygen atoms. Subsequent least-squares refinements and difference Fouriers revealed all of the atoms in the structure shown in Figure 1. Least-squares refinements, in which the function $\sum w(|F_o| - |F_c|)^2/\sum wF_o^2$ was minimized, converged rapidly to the final structure. The expressions that were used in processing the data and estimating the weights are given in the supplementary material; the "ignorance factor", p, was set to 0.05. Scattering factors from Doyle and Turner[10] were used, and anomalous dispersion corrections[11] were applied. Anisotropic thermal parameters were applied only to the uranium atom. Hydrogen atoms were neither located nor included. The discrepancy indices for 2542 data where $I > 3\sigma$ are

$$R = \sum||F_o| - |F_c||/\sum|F_o| = 0.048$$

$$R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0.055$$

R for all 4622 data is 0.11. The error in an observation of unit weight is 1.29. In the last cycle no parameter changed more than 0.06σ. The largest peak in the final difference Fourier was 1.5 electrons and seems to be an anisotropy associated with a sulfur atom.

Results and Discussion

Atomic parameters, distances, and angles are listed in Tables I–III.

The structure is a salt consisting of $(C_2H_5O)UO_2(SOCN(C_2H_5)_2)_2$ anion and diethylammonium cation. The nitrogen atom of the cation, N(3), hydrogen bonds to the ethoxide oxygen atom, O(3), of one anion and to the uranyl oxygen atom, O(4), of another anion across the center of symmetry, to form a dimer of two formula units about the origin.

Table I. Positional and Thermal Parameters with Estimated Deviations[a] for $(C_2H_5)_2NH_2^+[UO_2((C_2H_5)_2NCOS)_2(OC_2H_5)]^-$

atom	x	y	z	B, Å^2
U	0.09224 (5)	0.19283 (4)	0.40256 (2)	*b*
S(1)	0.3101 (4)	0.2909 (3)	0.4779 (2)	5.38 (8)
S(2)	0.3500 (4)	0.1083 (3)	0.3623 (3)	5.25 (8)
O(1)	0.0402 (9)	0.3059 (8)	0.4848 (4)	4.6 (2)
O(2)	0.097 (1)	0.0705 (9)	0.3217 (4)	6.0 (2)
O(3)	−0.1374 (8)	0.1839 (7)	0.3832 (4)	4.4 (2)
O(4)	0.0808 (8)	0.0819 (7)	0.4527 (4)	4.4 (2)
O(5)	0.096 (1)	0.3038 (8)	0.3536 (4)	5.5 (2)
N(1)	0.162 (1)	0.406 (1)	0.5535 (5)	5.6 (3)
N(2)	0.254 (1)	−0.034 (1)	0.2800 (6)	6.8 (3)
N(3)	−0.237 (1)	0.0409 (9)	0.4590 (5)	4.4 (2)
C(1)	0.160 (1)	0.336 (1)	0.5083 (6)	4.3 (3)
C(2)	0.223 (2)	0.043 (1)	0.3166 (7)	5.3 (3)
C(3)	−0.234 (2)	0.239 (1)	0.3450 (8)	6.3 (4)
C(4)	−0.248 (2)	0.172 (2)	0.2895 (9)	8.7 (5)
C(5)	0.301 (2)	0.447 (1)	0.5824 (7)	6.6 (4)
C(6)	0.328 (2)	0.555 (2)	0.546 (1)	9.9 (6)
C(7)	0.031 (2)	0.446 (1)	0.5769 (7)	6.3 (4)
C(8)	−0.007 (2)	0.376 (2)	0.630 (1)	8.8 (5)
C(9)	0.403 (2)	−0.070 (2)	0.2716 (8)	7.5 (4)
C(10)	0.431 (2)	−0.175 (2)	0.302 (1)	9.6 (6)
C(11)	0.130 (2)	−0.105 (2)	0.247 (1)	9.7 (6)
C(12)	0.113 (3)	−0.042 (2)	0.195 (1)	13.6 (8)
C(13)	−0.302 (2)	−0.050 (1)	0.4206 (7)	6.3 (4)
C(14)	−0.195 (2)	−0.103 (2)	0.3871 (8)	7.8 (5)
C(15)	−0.338 (2)	0.094 (1)	0.4963 (7)	6.0 (4)
C(16)	−0.263 (2)	0.177 (2)	0.5363 (8)	7.3 (4)

[a] Here and in the following tables the numbers in parentheses are the estimated standard deviations for the least significant figures. [b] The anisotropic temperature factor for U has the form exp$(-0.25(B_{11}h^2a^{*2} + 2B_{12}hka^*b^* + \ldots))$, with $B_{11} = 3.64$ (2), $B_{12} = -0.12$ (3), $B_{13} = 0.42$ (1), $B_{22} = 3.62$ (2), $B_{23} = 0.54$ (3), and $B_{33} = 3.41$ (2).

Table II. Distances (Å)

U-O(1)	2.40 (1)	C(5)-N(1)	1.52 (2)
U-O(2)	2.38 (1)	C(7)-N(1)	1.48 (2)
U-O(3)	2.20 (1)	C(9)-N(2)	1.52 (2)
U-O(4)	1.79 (1)	C(11)-N(2)	1.62 (3)
U-O(5)	1.77 (1)	C(13)-N(3)	1.47 (2)
U-S(1)	2.863 (4)	C(15)-N(3)	1.52 (2)
U-S(2)	2.869 (4)	C(5)-C(6)	1.61 (3)
S(1)-C(1)	1.72 (2)	C(7)-C(8)	1.54 (3)
S(2)-C(2)	1.74 (2)	C(9)-C(10)	1.50 (3)
O(1)-C(1)	1.28 (2)	C(11)-C(12)	1.40 (3)
O(2)-C(2)	1.27 (2)	C(13)-C(14)	1.46 (3)
O(3)-C(3)	1.40 (2)	C(15)-C(16)	1.52 (3)
N(1)-C(1)	1.33 (2)	N(3)-O(3)	2.68 (2)
N(2)-C(2)	1.32 (2)	N(3)-O(4)[a]	2.85 (2)

[a] Atom at $-x$, $-y$, $1 - z$.

Table III. Selected Angles (deg)

O(4)-U-O(5)	177.4 (4)	O(2)-U-S(2)	57.6 (3)
O(4)-U-O(1)	86.8 (4)	S(2)-U-S(1)	75.1 (1)
-O(2)	89.6 (4)	S(1)-U-O(1)	58.1 (2)
-O(3)	89.1 (4)	O(1)-C(1)-S(1)	119 (1)
-S(1)	91.6 (3)	O(2)-C(2)-S(2)	116 (1)
-S(2)	90.2 (3)	C(1)-N(1)-C(5)	121 (1)
O(5)-U-O(1)	91.9 (4)	C(1)-N(1)-C(7)	121 (1)
-O(2)	91.3 (4)	C(5)-N(1)-C(7)	118 (1)
-O(3)	88.6 (4)	C(2)-N(2)-C(9)	123 (2)
-S(1)	89.6 (3)	C(2)-N(2)-C(11)	120 (1)
-S(2)	92.3 (3)	C(9)-N(2)-C(11)	117 (2)
O(1)-U-O(3)	86.0 (3)	C(13)-N(3)-C(15)	114 (1)
O(3)-U-O(2)	83.4 (3)	O(3)-N(3)-O(4)[a]	127 (1)

[a] Atom at $-x$, $-y$, $1 - z$.

The uranium atom is at the center of a pentagonal bipyramid and is heptacoordinate. The two uranyl oxygen atoms are at the apices of the bipyramid, and two sulfur and three oxygen atoms form an irregular pentagon in a plane about the uranium atom. Pentagonal bipyramid type coordination is also found in such structures as $UO_2SO_4 \cdot 3^1/_2H_2O$,[12] $UO_2(H_2O)(CO(NH_2)_2)_4(NO_3)_2$,[13] and various uranyl fluorides.[14–16]

The two sulfur atoms are adjacent to each other; see Figure 1. The U–S distance averages 2.866 Å and compares with an average value of 2.80 Å found in the uranyl tris(diethyldithiocarbamate) anion,[17] and an average value of 2.84 Å found in the triphenylphosphine oxide and triphenylarsine oxide adducts of uranyl diethyldithiocarbamate.[18]

Three types of U–O distances are found in this structure; they are the uranyl-, ethoxide-, and carbamate-type bonds with distances of 1.78, 2.20, and 2.39 Å, respectively.

Registry No. $(C_2H_5)_2NH_2^+[UO_2((C_2H_5)_2HCOS)_2OC_2H_5]^-$, 67827-33-2.

Supplementary Material Available: Data processing formulas and the listing of structure factor amplitudes (19 pages). Ordering information is given on any current masthead page.

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(1) This work was supported by the Division of Basic Energy Sciences of the Department of Energy.
(2) National Science Foundation Postdoctoral Fellow, 1976–1977; Miller Fellow, 1977–1979.
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Contribution from the Materials and Molecular Research
Division, Lawrence Berkeley Laboratory,
University of California, Berkeley, California 94720

Structure of a New Uranyl Sulfate Hydrate, α-$2UO_2SO_4·7H_2O$[1]

Allan Zalkin,* Helena Ruben, and David H. Templeton

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When an aqueous solution of uranyl sulfate and (+)-tartaric acid was allowed to evaporate slowly, white crystals of tartaric acid and yellow crystals of a new uranyl sulfate hydrate both came out of solution. The X-ray crystal structure determination reported in this paper established that the yellow crystals are $2UO_2SO_4·7H_2O$ with identical composition but different structure than a previously reported "metastable hydrate"[2,3] of uranyl sulfate; this latter compound will be referred to here as β-$2UO_2SO_4·7H_2O$. The α-form crystal used for this study is air stable and showed no evidence of decomposition during the 4 weeks it was being studied and exposed to X-rays.

Experimental Section

An irregular wedge-shaped crystal with approximate dimensions 0.30 × 0.11 × 0.15 mm was glued to a glass fiber and examined with a Picker FACS-I automatic diffractometer equipped with a graphite monochromator and a Mo X-ray tube ($\lambda(K\alpha_1)$ 0.709 30 Å). ω scans of the 600, 040, and 006 reflections showed half-widths of 0.15, 0.11, and 0.09°, respectively. Absent reflections indicated space groups *Pmca* or $P2_1ca$; the subsequent Patterson function indicated two different uranium atoms which could only be accommodated in the noncentric space group $P2_1ca$.[4] The setting angles of 12 manually centered reflections (40° < 2θ < 50°) were used to determine by least squares the cell parameters a = 11.227 (6) Å, b = 6.790 (3) Å, c = 21.186 (10) Å, and V = 1615 Å^3. For Z = 4 and a molecular weight of 858.29 the calculated density is 3.53 g/cm^3; the calculated density of the β form is 3.46 g/cm^3. No extinction correction was indicated nor applied.

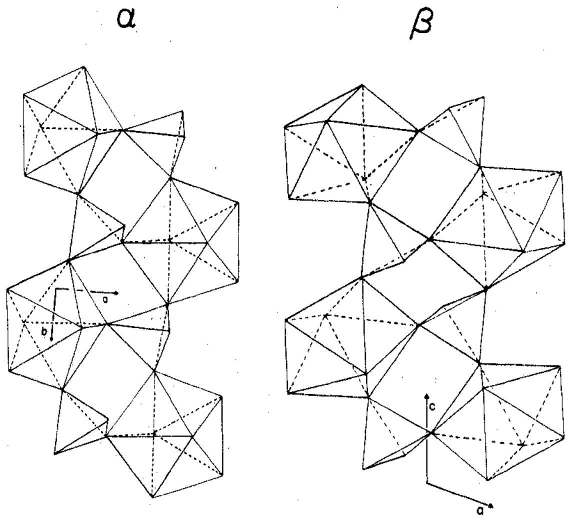

Figure 1. Comparison of the arrangement in the α and β forms of $2UO_2SO_4·7H_2O$.

Intensity data were collected using the θ–2θ scan technique with a scan speed of 2°/min on 2θ. Each reflection was scanned from 0.75° before the $K\alpha_1$ peak to 0.75° after the $K\alpha_2$ peak, and backgrounds were counted for 4 s at each end of the scan range, offset by 0.5°. The temperature during data collection was 21 ± 1 °C. Three standard reflections were measured after every 200th scan. The 6177 scans, not including standards, resulted in 5640 unique intensities in which the Friedel pairs were not averaged. The data were corrected for absorption by an analytical method[5] using an estimated absorption coefficient of 193 cm^{-1}; absorption corrections varied from 4.8 to 10.0.

A three-dimensional Patterson function calculation revealed two crystallographically unique uranium atoms, each in the general position. Subsequent least-squares calculations and Fourier maps revealed the positions of all of the light atoms. A series of least squares in which the function $\sum w(|F_o| - |F_c|)^2/\sum wF_o^2$ was minimized converged rapidly to the final structure. The expressions that were used in processing the data and estimating the weights are given in the supplementary material; the "ignorance factor", p, was set to 0.05. Scattering factors from Doyle and Turner[6] were used, and anomalous dispersion corrections[7] were applied. When f'' of uranium was treated as a variable as described elsewhere,[8] it refined to 9.4 ± 0.3, within 1σ of the literature[7] value. This result shows that the absolute orientation of the structure was chosen correctly and that the specimen was substantially free of inversion twinning. The positions of all of the hydrogen atoms were estimated from the hydrogen bonding and were included in the calculation of the structure factors but not refined. Anisotropic thermal parameters were included only for uranium. No extinction correction was indicated nor applied. The discrepancy indices for 3437 data (Friedel pairs not averaged) where $F^2 > 3\sigma$ are

$$R = \sum||F_o| - |F_c||/\sum|F_o| = 0.049$$

$$R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0.054$$

R for all 5640 data is 0.103. The error in an observation of unit weight is 1.24. In the last cycle no parameter changed more than 0.07σ.

Results and Discussion

Atomic parameters, distances, and angles are listed in Tables I–III. The uranium atom is at the center of a pentagonal bipyramid arrangement of oxygen atoms. These are joined in an infinite chain along the b axis by sulfate tetrahedra as is shown in Figure 1. The β form also contains chains made

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