

Fig. 2. Elementarzelle von $[\text{Ni}(\text{TolNNNNNTol})_2]_2$. Ursprung in $\bar{1}$. Abgebildet sind zwei der acht Komplexe sowie die 4-Zentren, auf denen die übrigen Komplexe zentriert sind [die Achse $(\frac{1}{2}, \frac{1}{2}, z)$ ist der Übersichtlichkeit halber weggelassen].

in den N_5 -Ketten sprechen für eine teilweise Delokalisierung der π -Elektronen. Zwischen den Atomen $\text{N}(11)\text{--}\text{N}(12)$ bzw. $\text{N}(21)\text{--}\text{N}(22)$ und $\text{N}(14)\text{--}\text{N}(15)$ bzw. $\text{N}(24)\text{--}\text{N}(25)$ werden mit $1,28 \text{ \AA}$ stets kürzere Abstände als zwischen den Atomen $\text{N}(12)\text{--}\text{N}(13)$ bzw.

$\text{N}(22)\text{--}\text{N}(23)$ und $\text{N}(13)\text{--}\text{N}(14)$ bzw. $\text{N}(23)\text{--}\text{N}(24)$ mit etwa $1,34 \text{ \AA}$ gefunden. Die kürzeren Bindungen sind gegenüber einer $\text{N}=\text{N}$ -Doppelbindung geringfügig aufgeweitet, während die längeren Bindungen eine verkürzte $\text{N}\text{--}\text{N}$ -Einfachbindung darstellen.

Die Komplexe haben damit bezüglich der inneren $[\text{Ni}(\text{N}_5)_2]_2$ -Einheit eine weitgehend identische Struktur wie die analogen Einheiten in der monoklin kristallisierenden, solvathaltigen Form der Verbindung (Beck & Strähle, 1985). Der entscheidende Unterschied zwischen den Komplexen der tetragonalen und monoklinen Form besteht darin, daß der Komplex im monoklinen Kristall deutlich von der S_4 -Symmetrie abweicht. Die Interplanarwinkel zwischen der Ebene der N_5 -Ketten und den zugehörigen Tolyrinen in der monoklinen Verbindung überstreichen mit Werten von $17,3$ bis $65,0^\circ$ einen weiten Bereich.

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Structure of Bis(4-benzoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-onato- O,O')-(dimethyl sulfoxide- O)dioxouranium(VI)*

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Abstract. $[\text{UO}_2(\text{C}_{17}\text{H}_{13}\text{N}_2\text{O}_2)_2(\text{C}_2\text{H}_6\text{OS})]$, $M_r = 902.77$, triclinic, $P\bar{1}$, $a = 10.330(2)$, $b = 11.286(3)$,

$c = 15.341(4) \text{ \AA}$, $\alpha = 77.53(2)$, $\beta = 79.59(2)$, $\gamma = 82.68(2)^\circ$, $V = 1710.3 \text{ \AA}^3$, $Z = 2$, $D_x = 1.75 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha_1) = 0.70930 \text{ \AA}$, $\mu = 46.1 \text{ cm}^{-1}$, $F(000) = 859.0$, ambient temperature, final $R = 4.3\%$ for 3758 reflections. Crystals are composed of discrete molecules containing a linear uranyl ion equatorially

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coordinated by two bidentate acyl pyrazolone anions and an O-bonded dimethyl sulfoxide molecule. The U—O bond lengths for the oxygen atoms on the pyrazolone rings (av. 2.338 Å) are significantly shorter than those for the acyl oxygen atoms (av. 2.396 Å).

Introduction. The compound 4-benzoyl-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (HA) and related derivatives have been extensively studied as extractants for the actinides and lanthanides (Navratil, 1977; Ramakrishna & Patil, 1984; Chun-Hui & Freiser, 1986). Detailed structural information on isolated complexes of these ligands could give further insight into the extraction systems, especially in comparison to the extensive structural and extraction data available for related β -diketone compounds. Complexes of the composition UO₂A₂L where L = dialkyl- or diaryl-sulfoxide have been isolated from the organic phase after extraction of uranium(VI) with HA and a sulfoxide in benzene (Subramanian, Sabnis & Nagar, 1983). We report here the crystal structure of such an adduct.

Experimental. Crystals were obtained by dissolving UO₂A₂·2EtOH in hot dimethyl sulfoxide (Me₂SO)/10% water and allowing the solution to cool slowly to room temperature. The complex UO₂A₂·2EtOH was prepared by dissolving 10 mmol of UO₂(NO₃)₂·6H₂O and 20 mmol of HA in 150 ml of warm ethanol and cooling slowly to room temperature. The crystal selected for study was an orange parallelepiped with dimensions 0.1 × 0.15 × 0.3 mm. Enraf-Nonius CAD-4 diffractometer, graphite monochromator. Lattice parameters from 25 reflections ($10 \leq \theta \leq 16^\circ$). Absorption correction was made by measuring the intensity of a low-angle reflection as a function of ψ (translated to ϕ in the actual correction), normalizing to the $\psi = 0$ value and superimposing a spherical correction, assuming a radius which is the average of the three most prominent directions of crystal development ($r = 0.1$ mm); transmission = 0.29–0.20. Variable-speed θ – 2θ scans [to $\sigma(I)/I = 0.02$ or 90s], $2\theta_{\max} = 45^\circ$, (min., max.) $h, k, l = (-10, 10), (-11, 12), (0, 14)$; 4461 total reflections, 3758 for which $I \geq 2\sigma(I)$, $R_{\text{int}} = 1.2\%$ (381 reflections), two standard reflections, no systematic variation during data collection. The structure was solved with standard Patterson and difference Fourier methods and refined by full-matrix least-squares method (Larson, 1977) with anisotropic temperature factors for atoms heavier than hydrogen (H atoms not located) and a secondary extinction factor (not significantly > 0) (Zachariasen, 1967; Larson, 1967), $\sigma(I) = \sigma_c(I) + (0.03I)^2$, $R(F) = 4.3$, $wR = 5.5\%$, $w = 4F^2/\sigma^2(I)$, $S = 2.5$, $(\Delta/\sigma)_{\max} = 0.01$, $(\rho_{\max}, \rho_{\min})$ (2.3, –1.5 e Å^{–3}) not in chemically interpretable positions. Neutral-atom scattering factors, f' and f'' , from Cromer & Waber (1974) and Cromer (1974).

Discussion. Final parameters are given in Table 1, and relevant bond distances and angles are given in Table 2. Numbering of atoms is shown in Fig. 1.*

The crystal contains discrete molecules of UO₂·A₂(Me₂SO) with the uranyl ion bound to two bidentate acylpyrazolone anions and a neutral Me₂SO molecule in the equatorial plane, forming a pentagonal bipyramid of O atoms about the U atom. Many structures of this general type have been determined where the bidentate ligand is acetylacetonate (acac) or a substituted analogue and with a variety of monodentate

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43795 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters (Å²)*

	$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$		
	x	y	z
U(1)	0.22603 (4)	0.15344 (4)	0.22266 (3)
O(2)	0.2942 (8)	0.2012 (8)	0.3048 (5)
C(1)	0.426 (1)	0.286 (1)	0.045 (1)
N(2)	0.562 (1)	0.336 (1)	–0.090 (1)
C(3)	0.532 (1)	0.194 (1)	0.040 (1)
O(4)	0.4397 (6)	0.0621 (6)	0.1675 (5)
C(6)	0.644 (1)	–0.124 (1)	0.125 (1)
C(8)	0.880 (1)	–0.166 (1)	0.121 (1)
C(10)	0.781 (1)	0.043 (1)	0.101 (1)
C(12)	0.365 (1)	0.475 (1)	–0.070 (1)
C(14)	0.331 (2)	0.650 (1)	–0.185 (1)
C(16)	0.154 (1)	0.592 (1)	–0.070 (1)
O(5)	0.2428 (7)	–0.0502 (7)	0.3003 (5)
N(3)	0.178 (1)	–0.242 (1)	0.371 (1)
C(19)	–0.034 (1)	–0.204 (1)	0.415 (1)
C(21)	–0.042 (1)	0.029 (1)	0.357 (1)
C(22)	–0.188 (1)	0.061 (1)	0.378 (1)
C(24)	–0.368 (1)	0.176 (1)	0.454 (1)
C(26)	–0.411 (1)	0.046 (1)	0.358 (1)
C(28)	–0.168 (1)	–0.234 (1)	0.465 (1)
C(30)	0.293 (1)	–0.437 (1)	0.412 (1)
C(32)	0.529 (1)	–0.472 (1)	0.369 (1)
C(34)	0.417 (1)	–0.271 (1)	0.325 (1)
S(1)	0.0714 (6)	0.4370 (5)	0.2647 (6)
C(36)	–0.063 (2)	0.453 (2)	0.327 (2)
O(1)	0.1528 (8)	0.1069 (7)	0.1434 (6)
O(3)	0.3297 (7)	0.2975 (6)	0.1082 (5)
N(1)	0.447 (1)	0.370 (1)	–0.034 (1)
C(2)	0.612 (1)	0.233 (1)	–0.045 (1)
C(4)	0.537 (1)	0.089 (1)	0.107 (1)
C(5)	0.656 (1)	0.000 (1)	0.111 (1)
C(7)	0.756 (1)	–0.208 (1)	0.129 (1)
C(9)	0.893 (1)	–0.042 (1)	0.109 (1)
C(11)	0.734 (1)	0.166 (1)	–0.094 (1)
C(13)	0.418 (1)	0.551 (1)	–0.147 (1)
C(15)	0.202 (1)	0.668 (1)	–0.146 (1)
C(17)	0.232 (1)	0.492 (1)	–0.029 (1)
C(18)	0.155 (1)	–0.121 (1)	0.344 (1)
N(4)	0.061 (1)	–0.293 (1)	0.419 (1)
C(20)	0.017 (1)	–0.091 (1)	0.368 (1)
O(6)	0.0269 (7)	0.1189 (7)	0.3273 (6)
C(23)	–0.233 (1)	0.144 (1)	0.434 (1)
C(25)	–0.455 (1)	0.128 (1)	0.415 (1)
C(27)	–0.274 (1)	0.012 (1)	0.338 (1)
C(29)	0.297 (1)	–0.317 (1)	0.370 (1)
C(31)	0.412 (1)	–0.516 (1)	0.409 (1)
C(33)	0.533 (1)	–0.351 (1)	0.327 (1)
O(7)	0.0855 (7)	0.3383 (6)	0.2115 (6)
C(35)	0.153 (3)	0.548 (2)	0.216 (1)
			17.3

ligands. All of the uranyl acac-type structures with monodentate donor ligands have exhibited the pentagonal bipyramidal geometry. Monodentate donors include H_2O (Frasson, Bombieri & Panattoni, 1966), Hacac (Lenner, 1979), methanol (Mackinnon & Taylor, 1983), nitrate (Graziani, Marangoni, Paolucci & Forsellini, 1978), pyridine (Alcock, Flanders & Brown, 1984), triphenylphosphine oxide (Taylor & McLaren, 1979), or any of several ketoamines (Haigh, Nassimbeni, Pauptit, Rodgers & Sheldrick, 1976; Nassimbeni, Orpen, Pauptit, Rodgers & Haigh, 1977; Rodgers, Nassimbeni & Haigh, 1977; Rodgers, Nassimbeni, Pauptit, Orpen & Haigh, 1977). The hexafluoroacac structures have been given special attention, presumably owing to the volatility of the complexes (Taylor & Waugh, 1977; Taylor, Ekstrom & Randall, 1978; Johnson, Taylor & Waugh, 1979; Kramer, Dines, Hall, Kaldor, Jacobson & Scanlon, 1980; Prasad, Gabe, Glavincevski & Brownstein, 1983).

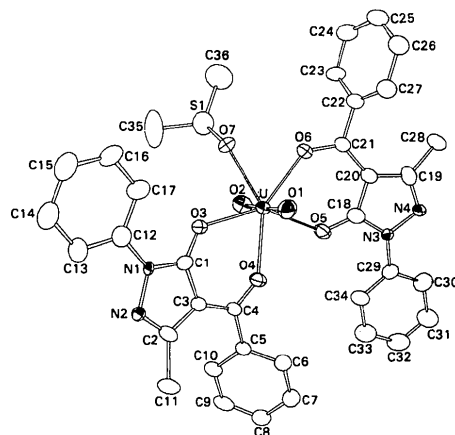


Fig. 1. Diagram of a molecule of the title compound, with atomic numbering.

Table 2. Selected bond distances (Å) and angles (°)

U—O(1)	1.742 (8)	U—O(5)	2.350 (7)
—O(2)	1.757 (8)	—O(6)	2.388 (7)
—O(3)	2.325 (7)	—O(7)	2.378 (7)
—O(4)	2.403 (6)		
C(1)—O(3)	1.27 (1)	C(18)—O(5)	1.28 (1)
C(1)—C(3)	1.41 (1)	C(18)—C(20)	1.42 (1)
C(1)—N(1)	1.37 (1)	C(18)—N(3)	1.34 (1)
N(1)—C(12)	1.43 (1)	N(3)—C(29)	1.41 (1)
N(1)—N(2)	1.39 (1)	N(3)—N(4)	1.41 (1)
N(2)—C(2)	1.31 (1)	N(4)—C(19)	1.31 (1)
C(2)—C(11)	1.54 (1)	C(19)—C(28)	1.50 (2)
C(2)—C(3)	1.43 (1)	C(19)—C(20)	1.44 (1)
C(3)—C(4)	1.39 (1)	C(20)—C(21)	1.40 (1)
C(4)—O(4)	1.26 (1)	C(21)—O(6)	1.27 (1)
C(4)—C(5)	1.48 (1)	C(21)—C(22)	1.50 (1)
O(7)—S(1)	1.50 (1)	C(35)—S(1)	1.58 (2)
C(36)—S(1)	1.55 (2)		
O(1)—U—O(2)	177.9 (4)	O(3)—U—O(6)	143.5 (3)
O(1)—U—O(3)	89.7 (3)	O(3)—U—O(7)	72.1 (2)
O(1)—U—O(4)	92.1 (3)	O(4)—U—O(5)	72.7 (2)
O(1)—U—O(5)	88.5 (3)	O(4)—U—O(6)	144.5 (3)
O(1)—U—O(6)	88.1 (4)	O(4)—U—O(7)	144.0 (3)
O(1)—U—O(7)	91.1 (3)	O(5)—U—O(6)	71.8 (2)
O(2)—U—O(3)	91.5 (3)	O(5)—U—O(7)	143.2 (3)
O(2)—U—O(4)	89.9 (3)	O(6)—U—O(7)	71.5 (3)
O(2)—U—O(5)	91.5 (3)	U—O(3)—C(1)	131.4 (6)
O(2)—U—O(6)	89.9 (4)	U—O(4)—C(4)	138.9 (6)
O(2)—U—O(7)	87.7 (3)	U—O(5)—C(18)	131.9 (7)
O(3)—U—O(4)	72.0 (2)	U—O(6)—C(21)	134.5 (7)
O(3)—U—O(5)	144.6 (2)	U—O(7)—S(1)	129.6 (6)
O(3)—C(1)—N(1)	123.8 (9)	O(5)—C(18)—N(3)	125. (1)
O(3)—C(1)—C(3)	130. (1)	O(5)—C(18)—C(20)	128. (1)
N(1)—C(1)—C(3)	106.3 (9)	N(3)—C(18)—C(20)	106.9 (9)
C(1)—N(1)—N(2)	111.2 (8)	C(18)—N(3)—N(4)	111.2 (9)
C(1)—N(1)—C(12)	130.5 (9)	C(18)—N(3)—C(29)	130.6 (9)
N(2)—N(1)—C(12)	117.8 (9)	N(4)—N(3)—C(29)	117.6 (9)
N(1)—N(2)—C(2)	106.0 (8)	N(3)—N(4)—C(19)	106.3 (9)
N(2)—C(2)—C(11)	117.8 (9)	N(4)—C(19)—C(28)	117. (1)
C(3)—C(2)—C(11)	130. (1)	C(20)—C(19)—C(28)	132. (1)
C(1)—C(3)—C(2)	104.6 (9)	C(18)—C(20)—C(19)	104.6 (9)
C(1)—C(3)—C(4)	121.8 (9)	C(18)—C(20)—C(21)	123. (1)
C(2)—C(3)—C(4)	133.3 (9)	C(19)—C(20)—C(21)	131. (1)
C(3)—C(4)—O(4)	121.8 (9)	C(20)—C(21)—O(6)	121. (1)
C(3)—C(4)—C(5)	122.4 (9)	C(20)—C(21)—C(22)	123. (1)
O(4)—C(4)—C(5)	115.8 (9)	O(6)—C(21)—C(22)	116. (1)
O(7)—S(1)—C(35)	113.6 (9)	C(35)—S(1)—C(36)	122. (1)
O(7)—S(1)—C(36)	115.9 (8)		

Deviations of the inner coordination atoms from the least-squares equatorial plane in the present case are less than 0.07 Å. Dihedral angles between this plane and the least-squares planes defined by the two pyrazolone planes (pyrazolone ring atoms and the C and O atoms involved in the acyl portion of the ligand) are 15 and 18°; a slight twist of the C—C bond between the pyrazolone and the acyl is apparent, *i.e.* the oxygen atom is slightly out of the plane for both ligands (0.3 and 0.1 Å). The dihedral angles between the pyrazolone planes and the inner equatorial plane are in the range (2–29°) observed for similar acac structures determined previously. Distances to the acyl oxygens (av. 2.396 Å) are equivalent within the error limits of the experiment and are significantly longer than those to the oxygens on the pyrazolone rings (av. 2.338 Å), indicating a larger negative charge on the latter. A similar difference in the binding of the pyrazolone oxygens relative to the acyl oxygens was reported for the complex $\text{EuA}_3(\text{triisobutylphosphate})_2$, where the Eu—O distances differ by 0.06, 0.12 and 0.13 Å (Karaseva, Karasev, Udovenko & Sigula, 1983). The distances to the acyl oxygens in $\text{UO}_2\text{A}_2(\text{Me}_2\text{SO})$ are even slightly longer than the U—O distance to the neutral Me_2SO ligand. Owing to the high thermal motions of the atoms in the Me_2SO ligand, the S—C distances are about 0.2 Å shorter than those observed in other Me_2SO complexes (*e.g.* Casellato, Vigato, Tamburini, Graziani & Vidali, 1983).

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Structure of Dibromodioxouranium(VI)–Tris(tetrahydrofuran)

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Abstract. [UO₂Br₂].3C₄H₈O, $M_r = 646.17$, monoclinic, $P2_1/c$, $a = 6.931(3)$, $b = 17.032(4)$, $c = 16.257(4)$ Å, $\beta = 94.25(5)^\circ$, $V = 1914(2)$ Å³, $Z = 4$, $D_x = 2.242$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 121.920$ cm⁻¹, $F(000) = 1192$, $T = 295(1)$ K, $R = 0.037$ for 965 observed reflections. The coordination polyhedron of the U atom is a bipyramid with a non-planar pentagonal base made up of two Br⁻ ions and three THF O atoms. The two uranyl O atoms are

in the apical positions. The stacking in the crystal is dictated by van der Waals interactions.

Introduction. The title compound has been prepared as part of our investigations on the bonding and coordination geometry in actinide complexes. We report here its characterization and structure analysis by X-ray diffraction.

Experimental. Light-brown single crystals obtained by reaction of a solution of [U{1,4,7-(CH₃)₃C₉H₄}]Br₃.2THF in THF with dry oxygen (5 µl per week)

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