

All of the atoms of the discrete complex described here ($[\text{Ni}\{\text{S}_2\text{CN}(\text{CH}_2\text{CH}_3)_2\}_2]$ in the solid state, β form), apart from the methyl groups, lie approximately in a plane. The deviation from the least-squares fit of all the atoms to the plane, excluding the CH_3 groups, is quite small with a maximum deviation of 0.039 \AA for C(3).

Of particular interest are the Ni–S, S–C and C–N bond lengths. The Ni–S distances [2.204 (1) and 2.192 (1) Å] are similar to those reported for the α form (Bonamico *et al.*, 1965), 2.207 (2) and 2.195 (2) Å . As compared to the Ni–S distances of the cation tris(di-*n*-butyldithiocarbamate) Ni^{IV} , $[\text{Ni}\{\text{S}_2\text{CN}(\text{C}_4\text{H}_9)_2\}_3]^+$, reported by Fackler *et al.* (1973), 2.261 (1) Å , the Ni–S distances observed here are approximately 0.06 \AA shorter. The observation that four-coordinate NiS_4 cores have somewhat shorter Ni–S bonds than do NiS_6 cores has been previously noted (Bonamico *et al.*, 1965). The S–C bond lengths observed here, 1.711 (3) and 1.725 (3) Å , compare well with those of the α form, 1.713 (7) and 1.700 (7) Å . Similar S–C distances are reported for thiourea derivatives and coordination complexes thereof (Lopez-Castro & Truter, 1963; Dias & Truter, 1964) and are considered consistent with an S–C(sp^2) formulation. The C–N bond distance observed for the β form, 1.319 (4) Å , is only slightly shorter than the 1.33 (1) Å reported for the α form. As discussed (Bonamico *et al.*, 1965) this C–N bond has considerable double-bond character. Indeed, it is the significant contribution of the thioureide form (Fackler, 1976) which is believed to lend stability to unusual oxidation of some metal centers, *i.e.* Au^{II} , Ni^{IV} (Calabro *et al.*, 1981; Fackler *et al.*, 1973) and stabilize M–C bonds by minimizing the

importance of the radical pathway for metal–carbon dissociation (Stein, Fackler, Papparizos & Chen, 1981).

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(4-Dimethylaminopyridine)tris(η -methylcyclopentadienyl)uranium(III)

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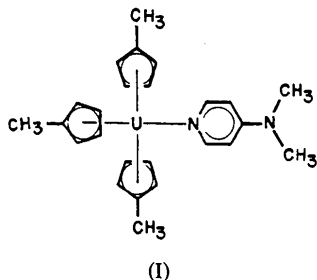
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Abstract. $[\text{U}(\text{C}_6\text{H}_7)_3(\text{C}_7\text{H}_{10}\text{N}_2)]$, $M_r = 597.57$, triclinic, $P\bar{1}$, $a = 15.541$ (6), $b = 17.155$ (7), $c = 10.303$ (4) Å , $\alpha = 106.83$ (4), $\beta = 106.52$ (4), $\gamma = 67.04$ (4)°, $V = 2374.7 \text{ \AA}^3$, $Z = 4$, $D_x = 1.671 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$, $\mu = 64.9 \text{ cm}^{-1}$, $F(000) = 1148$, $T = 296 \text{ K}$, $R = 0.033$ for 3258 independent reflections with $F^2 >$

$3\sigma(F^2)$. Two independent but chemically similar molecules are found in the unit cell. U^{III} is at the center of a distorted tetrahedron consisting of the pyridine N atom and the centroids of the three cyclopentadienyl rings. Distances are: U–N 2.662 (13) and 2.627 (12) Å ; av. U–C (ring), 2.82 (4) Å ; av. U–Cp, 2.56 (2) Å .

Introduction. The coordination chemistry of (C₅H₄R)₃U cannot be adequately described using a purely ionic bonding model; this was dramatically illustrated by the recent observation that, at room temperature, (C₅H₄Me₃Si)₃U readily forms a carbon monoxide complex with a reduced CO stretching frequency (Brennan, Andersen & Robbins, 1986). As part of a study on how steric and electronic factors influence the coordinative affinity of a given Lewis base towards trivalent uranium, we have undertaken the structural characterization of (C₅H₄Me)₃U coordination complexes. Herein is described the structure of (C₅H₄Me)₃U(4-Me₂NC₅H₄N) (I).



Experimental. Red air-sensitive crystals of the complex, prepared by the addition of 4-Me₂NC₅H₄N to a solution of (C₅H₄Me)₃U(OC₄H₈) (Brennan, Andersen & Zalkin, 1986a) and crystallized from ether, were sealed inside quartz capillaries under argon. Crystal 0.16 × 0.18 × 0.24 mm with six faces; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 21 reflections, 21 < 2θ < 33°; analytical absorption correction, range 1.99 to 3.69; max. (sinθ)/λ = 0.54 Å⁻¹, h-16 to 16, k-18 to 18, l-11 to 11; three standard reflections, 1.1%, 1.6%, 1.4% variations in standard intensities from average, intensities adjusted isotropically; 12468 data, 6240 independent [including 3258 with F² > 3σ(F²)], R_{int} = 0.043; structure solved by Patterson and Fourier methods; refined on F, 475 parameters; 32 H atoms in calculated positions with fixed isotropic thermal parameters; anisotropic thermal parameters for non-hydrogen atoms with the exception of C(13)-C(18); R = 0.099 for 6240 data, R = 0.033 for 3258 reflections for which F² > 3σ(F²), wR = 0.038, S = 1.26; w = [σ(F)]⁻², derived from σ²(F²) = {[σ(F²), counting statistics only] + (0.04F²)²}; max. (shift/σ) = 0.05; empirical extinction correction, F_{corr} = (1 + 1.8 × 10⁻⁸I); max. and min. of ΔF synthesis 1.6 and -1.2 e Å⁻³; atomic f for neutral U, N, C and spherical-bonded H from *International Tables for X-ray Crystallography* (1974); local unpublished programs and ORTEP (Johnson, 1965).

Very large thermal parameters in cyclopentadienyl ring, C(13)-C(18), indicated severe thermal motion or disorder. To get convergence in the least-squares refinements, the following distance constraints (Waser,

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

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B _{eq} /B
U(1)	0.01034 (4)	0.28930 (4)	0.14804 (5)	4.84
N(1)	0.0786 (9)	0.1576 (9)	-0.0500 (13)	6.32
N(2)	0.1538 (10)	-0.0232 (9)	-0.4101 (16)	7.31
C(1)	0.0288 (15)	0.2064 (16)	0.3641 (20)	7.68
C(2)	0.0138 (15)	0.1474 (11)	0.2375 (23)	7.51
C(3)	-0.0803 (14)	0.1836 (14)	0.1725 (17)	6.47
C(4)	-0.1204 (14)	0.2632 (15)	0.2605 (26)	8.08
C(5)	-0.0539 (16)	0.2777 (15)	0.3806 (24)	8.12
C(6)	-0.0670 (19)	0.3547 (16)	0.5095 (23)	13.70
C(7)	-0.0856 (18)	0.4332 (16)	0.028 (4)	9.96
C(8)	-0.0603 (18)	0.3695 (26)	-0.075 (3)	11.21
C(9)	-0.1143 (21)	0.3177 (17)	-0.104 (3)	11.36
C(10)	-0.1739 (15)	0.3503 (20)	-0.011 (3)	9.37
C(11)	-0.1527 (18)	0.4234 (18)	0.0795 (24)	9.13
C(12)	-0.2038 (29)	0.4907 (27)	0.194 (4)	24.38
C(13)	0.1847 (12)	0.3156 (11)	0.1690 (18)	8.5 (5)
C(14)	0.1248 (14)	0.3963 (12)	0.2218 (20)	10.2 (6)
C(15)	0.1065 (15)	0.3992 (13)	0.3460 (21)	12.0 (7)
C(16)	0.1563 (12)	0.3182 (11)	0.3750 (17)	8.5 (5)
C(17)	0.2047 (12)	0.2662 (11)	0.2671 (17)	8.2 (5)
C(18)	0.2688 (21)	0.1740 (13)	0.274 (3)	19.9 (12)
C(19)	0.0555 (12)	0.0848 (12)	-0.0953 (19)	7.05
C(20)	0.0776 (13)	0.0249 (10)	-0.2140 (18)	7.15
C(21)	0.1294 (10)	0.0358 (11)	-0.2931 (18)	5.84
C(22)	0.1533 (11)	0.1108 (12)	-0.2443 (18)	6.79
C(23)	0.1301 (12)	0.1673 (11)	-0.1274 (19)	7.48
C(24)	0.1279 (15)	-0.1044 (11)	-0.4526 (19)	9.33
C(25)	0.2073 (13)	-0.0082 (11)	-0.4960 (17)	8.29
U(2)	0.50706 (4)	0.76605 (3)	0.17079 (5)	4.50
N(3)	0.5736 (9)	0.6405 (8)	0.3081 (13)	5.94
N(4)	0.6630 (9)	0.4571 (9)	0.5664 (16)	7.39
C(26)	0.5247 (16)	0.6826 (15)	-0.1078 (18)	8.00
C(27)	0.5203 (13)	0.6214 (10)	-0.0409 (15)	6.19
C(28)	0.4265 (13)	0.6486 (11)	-0.0208 (15)	6.10
C(29)	0.3803 (12)	0.7243 (13)	-0.0722 (17)	7.17
C(30)	0.4398 (16)	0.7490 (13)	-0.1207 (18)	7.32
C(31)	0.4187 (18)	0.8222 (13)	-0.1908 (20)	11.96
C(32)	0.6384 (17)	0.8095 (22)	0.0925 (21)	8.87
C(33)	0.6923 (14)	0.7394 (15)	0.148 (3)	8.86
C(34)	0.6961 (12)	0.7589 (16)	0.2821 (29)	8.01
C(35)	0.6438 (16)	0.8466 (18)	0.3241 (19)	8.12
C(36)	0.6061 (15)	0.8802 (16)	0.204 (3)	8.81
C(37)	0.5533 (20)	0.9766 (15)	0.1918 (29)	15.26
C(38)	0.3245 (17)	0.8221 (19)	0.241 (4)	10.75
C(39)	0.3921 (21)	0.8041 (15)	0.356 (3)	9.40
C(40)	0.4356 (15)	0.8659 (19)	0.4068 (23)	9.08
C(41)	0.4010 (17)	0.9212 (12)	0.321 (3)	8.40
C(42)	0.3295 (18)	0.8975 (19)	0.2142 (25)	9.73
C(43)	0.2716 (18)	0.9509 (17)	0.1003 (26)	15.43
C(44)	0.6187 (11)	0.6548 (9)	0.4425 (16)	5.42
C(45)	0.6488 (10)	0.5969 (10)	0.5315 (15)	5.38
C(46)	0.6326 (10)	0.5159 (10)	0.4808 (17)	5.51
C(47)	0.5862 (11)	0.5022 (9)	0.3404 (16)	6.14
C(48)	0.5614 (11)	0.5630 (10)	0.2648 (14)	6.02
C(49)	0.6502 (13)	0.3702 (11)	0.5023 (19)	8.65
C(50)	0.7082 (12)	0.4763 (11)	0.7118 (17)	7.94

1963) were applied: C(ring)-C(ring) 1.39 (1) Å, C(ring)-CH₃ 1.54 (1) Å, H₃C...C (nearest ring neighbor) 2.52 (3) Å. Isotropic thermal parameters were assigned to these atoms.

Atomic parameters are listed in Table 1,* and distances and angles are listed in Table 2. Figs. 1 and 2 show the two independent molecules with numbering scheme.

* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positions, distances and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44134 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

U(1)—N(1)	2.662 (13)	U(2)—N(3)	2.627 (12)
U(1)—C(1)	2.866 (15)	U(2)—C(26)	2.850 (16)
U(1)—C(2)	2.819 (15)	U(2)—C(27)	2.771 (15)
U(1)—C(3)	2.792 (15)	U(2)—C(28)	2.791 (13)
U(1)—C(4)	2.832 (17)	U(2)—C(29)	2.814 (14)
U(1)—C(5)	2.927 (17)	U(2)—C(30)	2.853 (17)
U(1)—C(7)	2.775 (20)	U(2)—C(32)	2.801 (17)
U(1)—C(8)	2.759 (22)	U(2)—C(33)	2.800 (18)
U(1)—C(9)	2.807 (23)	U(2)—C(34)	2.801 (17)
U(1)—C(10)	2.821 (18)	U(2)—C(35)	2.835 (15)
U(1)—C(11)	2.753 (18)	U(2)—C(36)	2.826 (18)
U(1)—C(13)	2.856 (18)	U(2)—C(38)	2.850 (19)
U(1)—C(14)	2.843 (21)	U(2)—C(39)	2.751 (19)
U(1)—C(15)	2.860 (23)	U(2)—C(40)	2.779 (19)
U(1)—C(16)	2.837 (18)	U(2)—C(41)	2.800 (18)
U(1)—C(17)	2.833 (17)	U(2)—C(42)	2.857 (18)
U(1)—Cp(1)	2.584	U(2)—Cp(4)	2.551
U(1)—Cp(2)	2.528	U(2)—Cp(5)	2.555
U(1)—Cp(3)	2.590	U(2)—Cp(6)	2.549
N(1)—U(1)—Cp(1)	101.7	N(3)—U(2)—Cp(4)	100.5
N(1)—U(1)—Cp(2)	94.9	N(3)—U(2)—Cp(5)	101.0
N(1)—U(1)—Cp(3)	103.2	N(3)—U(2)—Cp(6)	95.8
Cp(1)—U(1)—Cp(2)	117.7	Cp(4)—U(2)—Cp(5)	115.3
Cp(1)—U(1)—Cp(3)	116.0	Cp(4)—U(2)—Cp(6)	118.0
Cp(2)—U(1)—Cp(3)	117.7	Cp(5)—U(2)—Cp(6)	119.3
U(1)—N(1)—C(21)	170.2 (6)	U(2)—N(3)—C(46)	173.4 (5)

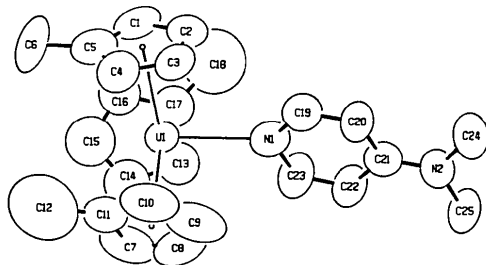
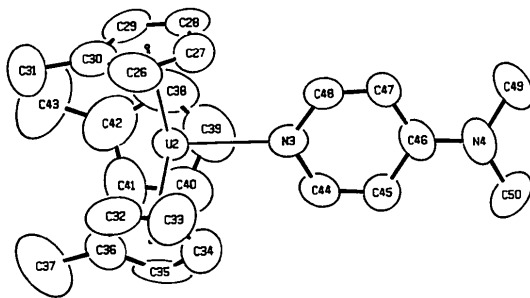
Fig. 1. ORTEP drawing of the $(C_5H_4Me)_3U(4-Me_2NC_5H_4N)$ molecule (1), drawn with 50% probability atomic thermal ellipsoids.

Fig. 2. ORTEP drawing of molecule (2), drawn with 50% probability atomic thermal ellipsoids.

Discussion. The complex $(C_5H_4Me)_3U(4-Me_2NC_5H_4N)$ is monomeric in the solid state. Three cyclopentadienyl centroids and a pyridine N form a distorted tetrahedron about the metal atom. The corresponding bond distances and angles of the two chemically similar but crystallographically independent molecules are within 3 e.s.d.'s of each other. Coordination of the pyridine N rather than the dimethylamino N to the U atom is consistent with solution measurements which indicate that pyridine

quantitatively displaces tertiary amines from $(C_5H_4Me)_3U$.

The average Cp—M—Cp angle in $(C_5H_4Me)_3UL$, $L = 4-Me_2NC_5H_4N$, of 117° is comparable to 118° for $L = SC_4H_8$ (Zalkin & Brennan, 1985), 118° for $L = OPPh_3$ (Brennan, Andersen & Zalkin, 1986b), and 118° in $(C_5H_5)_3U(OC_4H_8)$ (Wasserman, Zozulin, Moody, Ryan & Salazar, 1983). These are distinctly different from the pattern of Cp—M—Cp angles (106 , 109 and 119°) found in $(C_5H_4Me)_3U(PMe_3)$ (Brennan & Zalkin, 1985), where the phosphine complex is more sterically crowded. There are no exceptionally close contacts between the pyridine ligand and the cyclopentadienyl groups.

The U—N distance of $2.64(2)$ Å in the title compound can be compared with those in the following three uranium—pyridine complexes: $2.659(4)$ Å in $U^{III}(BH_4)_3(4-Ph_2PC_5H_4N)$ (Wasserman, Moody, Paine, Ryan & Salazar, 1984); $2.61(1)$ Å in $UO_2(\text{tropolonate})_2(C_5H_4N)$ (Degetto, Marangoni, Bombieri, Forsellini, Baracco & Graziani, 1974); and $2.47(1)$ Å in bis(pentane-2,4-dionato)pyridinedioxouranium (Alcock, Flanders & Brown, 1984). The U—N distance in the title compound is similar to all but the bis(pentane-2,4-dionato)pyridinedioxouranium value, indicating an insensitivity of this distance to metal oxidation state or coordination number. The bis(pentane-2,4-dionato)pyridinedioxouranium complex appears to be unusual in that a relatively short U—N distance is achieved at the expense of a nonlinear uranyl geometry [$173.5(8)^\circ$].

The methyl carbon atom of each C_5H_4Me ligand is displaced out of the mean plane of the cyclopentadienyl C(ring) atoms by 0.04 to 0.19 Å, on the opposite side from the U atom. The N and methyl C atoms of the amino group are in the plane of each pyridine ring.

As there are no close non-bonding contacts, distorted Cp—M—Cp angles, or unusual U—L distances in the final structure, it is reasonable to suggest that the relative coordinative affinity of the pyridine ligand in this system is dominated by electronic factors. The coplanarity of the NMe_2 substituent with the pyridine ligand indicates substantial interaction of the nitrogen π electrons with the aromatic ring.

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Structure of a Second Polymorph of Calcium Acetate Monohydrate

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Abstract. Ca(C₂H₃O₂)₂·H₂O, *M_r* = 176.18, triclinic, *P*1̄, *a* = 6.700 (2), *b* = 9.801 (2), *c* = 12.257 (2) Å, α = 78.83 (2), β = 86.21 (2), γ = 73.63 (2)°, *V* = 757.6 (3) Å³, *Z* = 4, *D_x* = 1.545 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 7.7 cm⁻¹, *T* = 295 K, *F*(000) = 368, *R* = 0.029 for 2921 observed reflections with *I* > 2.5 σ(*I*). The structure consists of infinite O-bridged Ca layers that are connected in the third dimension by hydrogen bonds *via* water molecules of crystallization. The Ca atoms are seven and eight coordinated.

Introduction. Two crystal forms of Ca(OAc)₂ were obtained as a result of attempts to obtain good-quality crystals by crystallization under a variety of experimental conditions. Crystallization from pure water yielded needle-shaped crystals of a monohydrate for which the crystal structure has been reported previously (Klop, Schouten, van der Sluis & Spek, 1984). Addition of 10% ethanol resulted in large flat needles. The ethanol was added to decrease the solubility and thereby the viscosity. A high viscosity is known to hamper crystallization. A crystal structure determination of this compound was undertaken to characterize it and to find out whether the ethanol merely changed the habit or changed the complete structure.

Experimental. The colourless transparent crystal (0.2 × 0.4 × 0.6 mm) used in this study, cleaved from a large crystal, was obtained by evaporation from a saturated solution of Ca(OAc)₂ in a 10% ethanol/water mixture. Enraf–Nonius CAD-4F diffractometer, Zr-filtered Mo *K*α radiation. Cell parameters and estimated standard deviations calculated from the setting

angles of 20 reflections (10 < θ < 15°). Data set: ω-scan mode, Δω = 0.5 + 0.35 tanθ°, 1.70 < θ < 27.50°, *h*–8→0, *k*–12→12, *l*–15→15, total of 3781 reflections measured, 3480 unique. Standard reflections (3̄16, 3̄33), measured every 2h, showed a linear decay of 2% and 1% fluctuation during 60h of X-ray exposure time. Intensity data corrected for decay and *L_p* but not for absorption. Standard deviation calculated following McCandlish, Stout & Andrews (1975); σ²(*I*) = σ_{cs}²(*I*) + (*PI*)² with *P* = 0.012. 2921 unique reflections considered observed [*I* > 2.5 σ(*I*)]. Structure solved by Patterson and Fourier techniques and refined on *F* by full-matrix least-squares procedures. Anisotropic thermal parameters for all non-hydrogen atoms; isotropic thermal parameters for the water H atoms; one common isotropic temperature parameter for all methyl H atoms. All methyl groups were found to be disordered and refined with rigid methyl groups in two staggered positions with populations ranging from 40 to 60%. An empirical extinction correction was applied: *F*^{corr} = *F*_{calc}[1 – (6.5 × 10⁻⁷ *F*_{calc}² / sinθ)]. Refinement converged at *R* = 0.029; *wR* = 0.037 [*w*⁻¹ = σ²(*F*)]; 227 parameters; *S* = 0.51; (Δ/σ)_{av} = 0.03; (Δ/σ)_{max} = 0.77]. Maximum and minimum residual density 0.32 and –0.46 e Å⁻³ in the final difference Fourier map. Final atomic parameters are listed in Table 1.† Scattering factors of Cromer & Mann (1968). Anomalous-dispersion corrections from Cromer & Liberman (1970). Calculations carried out

† Lists of structure factors, anisotropic thermal parameters, methyl H-atom parameters, additional bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44114 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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