

Fig. 1. ORTEP (Johnson, 1965) view of the cation showing the disordered arrangements of the bis(diethylphosphino)ethane ligands. Anisotropic thermal ellipsoids have been drawn at the 50% probability level. See footnotes for origin of atom designations.

2.334 (3) Å (equatorial, X-ray data) with an axial P—Os—P angle of 165.2 (1)° (Hart, Bau & Koetzle, 1977) while in the pentagonal bipyramidal trihydrides [Re(dppe)₂H₃] and [Re(dppe)H₃(PPh₃)₂] the axial P—Re—P bond angles are smaller with values of 167.4 (5) and 159.1° respectively [the equatorial P—Re—P angle in the bis(diphenylphosphino)ethane structure is 151.5 (5)°; Albano & Bellon, 1972]. Thus, despite the smaller POsP bond angle the shorter Os—P distances in the present compound suggest that P(2) and P(3) are the axial atoms in a pentagonal bipyramid with P(1) and P(4) and the *trans*-related hydride and dihydrogen (with a long H—H bond from NMR data) in the equatorial plane. The disorder, however, prevented any location of the H atoms so that the above interpretation of the structure is still somewhat in doubt.

The tetraphenylborate ion is a slightly 'stretched' tetrahedron with two Ph—B—Ph angles of 102.3 (8) and 103.8 (8)° and four angles averaging 112.8°; the average B—C distance is 1.643 (8) Å.

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Amminetris(η -methylcyclopentadienyl)uranium(III)

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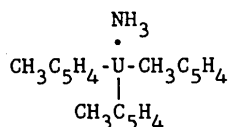
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Abstract. [U(C₅H₇)₃(NH₃)], $M_r = 492.43$, monoclinic, *Im*, $a = 12.129$ (3), $b = 24.197$ (5), $c = 8.725$ (3) Å, $\beta = 92.85^\circ$, $V = 2557.5$ Å³, $Z = 6$, $D_x = 1.92$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 90.2$ cm⁻¹, $F(000) = 1386$, $T = 296$ K, $R = 0.037$ [4247 data, $F^2 > 3\sigma(F^2)$].

There are two independent molecules, one of which is across the mirror plane. The U atom is bonded to three cyclopentadienyl rings and to an ammonia molecule, with $\langle \text{U—C} \rangle$, $\langle \text{U—ring} \rangle$ and $\langle \text{U—N} \rangle$ distances of 2.81 (5), 2.55 (3) and 2.61 (3) Å, respectively.

Introduction. The crystal structure of (MeC₅H₄)₃U-NH₃ was determined in order to examine the role of steric effects on the U—L bond length in a series of (MeC₅H₄)₃U.L compounds, where L = ammonia, 4-dimethylaminopyridine and quinuclidine. A structural representation of the complex is shown below.



Experimental. The title compound was prepared by the addition of an ammonia-saturated toluene solution to a solution of (MeC₅H₄)₃U.OC₄H₈, and the red-brown needles were crystallized from ether at 253 K. An air-sensitive crystal, 0.1 × 0.2 × 0.7 mm, was sealed inside a quartz capillary in an argon-filled drybox. X-ray diffraction intensities (θ -2 θ scans) were obtained using a modified Picker FACS-I automatic diffractometer equipped with a graphite monochromator. Cell dimensions from 30 reflections, $20 < 2\theta < 33^\circ$; 2θ scan width (1.5 + 0.693tan θ) $^\circ$, 2θ scan speed 2–8 $^\circ$ min⁻¹; analytical absorption correction, range 1.5–2.9; max. $\sin\theta/\lambda = 0.65 \text{ \AA}^{-1}$, h –15 to 15, k 0 to 31, l –11 to 11; three standard reflections, 1.6%, 1.2%, 1.6% variation in standards' intensities from average, intensities adjusted isotropically; 6046 data [4247 with $F^2 > 3\sigma(F^2)$], Friedel pairs not averaged; structure solved by Patterson and Fourier methods; refined on F , 246 parameters; 37 H atoms in calculated positions with fixed isotropic thermal parameters; anisotropic thermal parameters for 26 of the 32 non-H atoms; distance restraints applied to ten ring C—C and to four C—C (methyl) distances, 1.39 and 1.54 \AA respectively; $R = 0.075$ for all the data, $R = 0.037$ for $F^2 > 3\sigma(F^2)$ data, $wR = 0.043$, $S = 1.07$; $w = 4F^2[\sigma^2(F^2) + (0.055F^2)^2]^{-1}$; max. (shift/ σ) < 0.09; no extinction correction indicated; max. and min. of ΔF synthesis 2.4 and -2.0 e \AA^{-3} ; Rogers (1981) parameter η (an indicator of absolute configuration and twinning in non-centrosymmetric structures) = 0.95 (3); atomic f for neutral U, Cl and C, and spherical-bonded H, from *International Tables for X-ray Crystallography* (1974); local unpublished programs and ORTEP (Johnson, 1965). Atomic parameters are listed in Table 1.* A list of selected distances and angles is given in Table 2. Figs. 1 and 2 show the two independent molecules and the numbering scheme.

Table 1. Atomic parameters

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

	x	y	z	B*/B _{eq}
U1	0	0.18783 (2)	0	3.75 (1)
U2	0.51053 (6)	0	0.25804 (8)	3.47 (2)
N1	-0.1344 (10)	0.2713 (6)	-0.0174 (15)	4.7 (3)
N2	0.7237 (16)	0	0.2139 (25)	5.8 (6)
C1	0.0938 (15)	0.2091 (8)	0.2965 (18)	6.3 (5)
C2	0.1735 (14)	0.1816 (8)	0.2149 (23)	6.8 (5)
C3	0.2080 (12)	0.2180 (11)	0.1077 (20)	7.7 (6)
C4	0.1510 (16)	0.2689 (8)	0.1165 (20)	7.5 (6)
C5	0.0771 (13)	0.2623 (6)	0.2397 (16)	5.6 (4)
C6	-0.1489 (17)	0.1448 (8)	0.2051 (21)	7.3 (6)
C7	-0.0729 (16)	0.1008 (9)	0.1803 (24)	8.0 (6)
C8	-0.0861 (21)	0.0796 (10)	0.035 (3)	9.4 (8)
C9	-0.1712 (25)	0.1076 (15)	-0.0291 (27)	11.1 (10)
C10	-0.2106 (13)	0.1460 (11)	0.0670 (28)	9.1 (8)
C11	0.0374 (29)	0.1355 (15)	-0.300 (4)	15.1 (11)*
C12	-0.0430 (18)	0.1780 (11)	-0.3231 (19)	8.8 (8)
C13	0.0046 (21)	0.2310 (9)	-0.2965 (18)	7.9 (7)
C14	0.1123 (20)	0.2197 (10)	-0.2560 (19)	8.1 (7)
C15	0.1338 (15)	0.1651 (9)	-0.2459 (20)	7.0 (5)
C16	0.4931 (18)	0.0605 (9)	0.5354 (26)	7.8 (5)*
C17	0.4143 (17)	0.0802 (6)	0.4316 (23)	10.3 (8)
C18	0.4521 (14)	0.1088 (7)	0.3039 (18)	6.5 (5)
C19	0.5640 (15)	0.1085 (6)	0.3296 (20)	8.9 (7)
C20	0.5833 (18)	0.0821 (10)	0.4709 (26)	9.8 (6)*
C21	0.362 (4)	0	0.006 (3)	9.8 (14)
C22	0.4255 (25)	0.0466 (12)	-0.0202 (27)	12.8 (12)
C23	0.5295 (21)	0.0290 (8)	-0.0394 (21)	12.7 (14)
C24	0.0039 (19)	0.3063 (8)	0.3041 (25)	9.3 (7)
C25	0.0020 (21)	0.0781 (17)	0.313 (3)	16.9 (16)
C26	0.054 (4)	0.0725 (16)	-0.307 (5)	19.7 (16)*
C27	0.6900 (28)	0.0666 (21)	0.565 (5)	20.4 (16)*
C28	0.244 (4)	0	0.065 (10)	26.7 (34)*

* Atoms refined isotropically.

Table 2. Selected distances (\AA) and angles ($^\circ$)

Cp1, Cp2, Cp3, Cp4 and Cp5 are the centroids of cyclopentadienyl ring atoms C1–C5, C6–C10, C11–C15, C16–C20 and (C21, C22, C22', C23, C23'), respectively. Primed positions represent the equivalent groups or atoms at $x, -y, z$.

$\langle \text{U2}-\text{C} \rangle$	2.78 (6)	Cp1–U1–Cp3	118.1
U1–Cp1	2.54	Cp2–U1–Cp3	118.1
U1–Cp2	2.57	Cp4–U2–Cp4'	114.4
U1–Cp3	2.58	Cp4–U2–Cp5	120.5
U2–Cp4	2.53	N1–U1–Cp1	98.1
U2–Cp5	2.51	N1–U1–Cp2	97.4
U1–N1	2.595 (12)	N1–U1–Cp3	97.6
U2–N2	2.633 (19)	N2–U2–Cp4	98.5
		N2–U2–Cp5	94.5

Discussion. There are two independent molecules in the unit cell. In the first molecule, Fig. 1, the atoms are all in general positions. The second molecule is astride the mirror plane and is shown in Fig. 2. Although the orientations of components in the two molecules are different, the bond distances and angles are comparable within the estimated statistics, and chemically the two are identical.

The average U—N distance of 2.61 (3) \AA in the ammonia complex may be compared with other trivalent uranium coordination complexes with nitrogen-containing donor ligands: 2.64 (2) \AA in (MeC₅H₄)₃U.4-NC₅H₄(NMe₂) (Zalkin & Brennan, 1987), and 2.764 (4) \AA in (MeC₅H₄)₃U-N(CH₂CH₂)₃CH (Brennan, Stults, Andersen & Zalkin, 1988). The large variation in these distances reflects the relative steric demands of the ligands. The title

* Lists of anisotropic thermal parameters, calculated H-atom positions, additional distances and angles, distance restraints and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51789 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

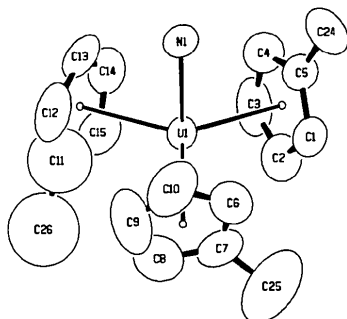


Fig. 1. ORTEP drawing of the molecule in the general position, with thermal ellipsoids at 50% probability level.

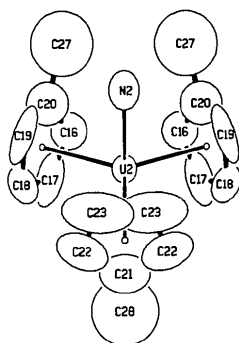


Fig. 2. ORTEP drawing of the molecule on the mirror plane, with thermal ellipsoids at 50% probability level.

compound has a U—N distance similar to that found in the dimethylaminopyridine adduct, consistent with their similar steric nature. This is in marked contrast to the quinuclidine complex. Solution-based competition studies further reflect this difference: the ammonia and dimethylaminopyridine complexes are similar in their

coordinative affinity for uranium, and both are better Lewis bases than quinuclidine (Brennan, 1985; Rosen, 1988).

The average Cp—U—Cp and Cp—U—N angles also reflect the steric differences between these ligands, although the differences are small. In $(\text{MeC}_5\text{H}_4)_3\text{U}\cdot\text{NH}_3$, these angles are 118 and 97° , respectively, while in $(\text{MeC}_5\text{H}_4)_3\text{U}\cdot 4\text{-NC}_5\text{H}_4(\text{NMe}_2)$ and $(\text{MeC}_5\text{H}_4)_3\text{U}\cdot\text{N}(\text{CH}_2\text{CH}_2)_3\text{CH}$ the Cp—U—Cp angles are 117 and 116° and the Cp—U—N angles are 100 and 101° . Thus, the title compound shows a slightly greater tendency towards an idealized geometry of trigonal pyramidal, suggesting that ammonia is slightly less bulky than either of these other ligands. Although these values are consistent with the solution equilibrium studies, they are not significant crystallographically.

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Structure of *trans*-Bis(DL- α -alaninato)copper(II) Monohydrate

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Abstract. $[\text{Cu}\{\text{NH}_2\text{CH}(\text{CH}_3)\text{COO}\}_2]\cdot\text{H}_2\text{O}$, $[\text{Cu}(\text{C}_3\text{H}_6\text{NO}_2)_2]\cdot\text{H}_2\text{O}$, $M_r = 257.76$, monoclinic, $C2/c$, $a =$

$12.074(2)$, $b = 9.567(2)$, $c = 8.944(1)$ Å, $\beta = 110.87(1)^\circ$, $V = 965.3(5)$ Å³, $Z = 4$, $D_x = 1.790$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 2.35$ mm⁻¹, $F(000) = 532$, $T = 295$ K. Conventional R factor: 0.053 for 1825 unique reflections. The Cu^{II} ion

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