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## Tris[bis(trimethylsilyl)amido](trimethylsilylimido)uranium(V)

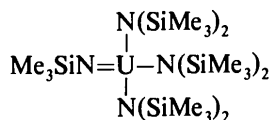
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**Abstract.**  $\{[(\text{CH}_3)_3\text{Si}]_2\text{N}\}_3\text{U}=\text{NSi}(\text{CH}_3)_3$ ,  $M_r = 806.40$ , rhombohedral,  $R\bar{3}c$ ,  $a = 12.495(4) \text{ \AA}$ ,  $\alpha = 89.83(3)^\circ$ ,  $V = 1950.8 \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 1.373 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 41.7 \text{ cm}^{-1}$ ,  $F(000) = 814$ ,  $T = 296 \text{ K}$ ,  $R = 0.027$  for 1194 data with  $F^2 > 3\sigma(F^2)$ . The uranium(V) atom is on a threefold axis at the center of a tetrahedron of N atoms. Distances: U=N, 1.910(16); U–N 2.295(10);  $\langle\text{Si–N}\rangle$ , 1.728(27);  $\langle\text{Si–C}\rangle$ , 1.91(4) \text{ \AA}.

**Introduction.** The title compound was prepared during a systematic synthetic and comparative X-ray crystallographic study of monomeric pentavalent uranium compounds. The only other pentavalent uranium organoimide that has been crystallographically examined is  $(\text{MeC}_5\text{H}_4)_3\text{U}(\text{NC}_6\text{H}_5)$  (Brennan & Andersen, 1985). Since the phenylimide was unique it was of interest to examine other members of this class of molecule so that the structural systematics could be elucidated. To this end, the title compound was prepared and crystallographically examined. A structural representation of the complex is shown below.



**Experimental.** The compound was prepared from  $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{U}$  and  $\text{Me}_3\text{SiN}_3$  in hexane. The crystal used in the X-ray studies was grown from hexane (253 K) (Brennan, 1985). A red air-sensitive crystal,  $0.2 \times 0.2 \times 0.35 \text{ mm}$ , was sealed inside a thin-walled quartz capillary in an argon-filled dry box. X-ray diffraction intensities ( $\theta$ – $2\theta$  scans) were obtained using a modified Picker FACS-I automatic diffractometer equipped with a graphite monochromator. Cell dimensions from 22 reflections  $22 < 2\theta < 33^\circ$ ; no absorption

Table 1. Positional and equivalent isotropic thermal parameters with e.s.d.'s

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

	x	y	z	$B_{\text{eq}}(\text{Å}^2)$
U	0	0	0	3.42(1)
N(1)	–0.0880(17)	–0.0880	–0.0880	5.0(2)
N(2)	0.1633(19)	–0.0072(17)	–0.0842(19)	4.0(7)
Si(1)	–0.1690(6)	–0.1690	–0.1690	8.0(2)
Si(2)	0.2809(12)	–0.0256(10)	–0.0165(11)	4.6(5)
Si(3)	0.1594(7)	–0.0096(6)	–0.2211(7)	5.2(2)
C(1)	–0.170(4)	–0.122(4)	–0.310(4)	11.5(19)
C(2)	0.3843(22)	0.0836(25)	–0.036(3)	6.3(10)
C(3)	0.3465(25)	–0.1585(22)	–0.0557(27)	5.7(10)
C(4)	0.2553(23)	–0.0281(27)	0.1377(21)	5.4(10)
C(5)	0.0402(20)	0.0797(22)	–0.2729(15)	7.7(7)
C(6)	0.2835(26)	0.0457(25)	–0.2882(26)	7.2(11)
C(7)	0.1474(29)	–0.1485(27)	–0.2779(26)	6.5(11)

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

U–N(1)	1.910(6)	Si(2)–C(2)	1.896(13)
U–3N(2)	2.295(10)	Si(2)–C(3)	1.914(12)
N(1)–Si(1)	1.759(17)	Si(2)–C(4)	1.953(12)
N(2)–Si(3)	1.711(10)	Si(3)–C(7)	1.884(13)
N(2)–Si(2)	1.713(11)	Si(3)–C(6)	1.893(14)
Si(1)–3C(1)	1.858(22)	Si(3)–C(5)	1.970(15)
N(1)–U–N(2)	103.10(23)	N(2)–Si(2)–C(4)	110.6(5)
N(2)–U–N(2)	115.02(17)	C(2)–Si(2)–C(3)	107.6(6)
U–N(1)–Si(1)	180.00	C(2)–Si(2)–C(4)	104.1(7)
U–N(2)–Si(2)	122.8(5)	C(3)–Si(2)–C(4)	108.0(6)
U–N(2)–Si(3)	115.6(5)	N(2)–Si(3)–C(5)	109.9(7)
Si(2)–N(2)–Si(3)	121.2(6)	N(2)–Si(3)–C(6)	114.2(6)
N(1)–Si(1)–C(1)	111.6(7)	N(2)–Si(3)–C(7)	113.5(6)
C(1)–Si(1)–C(1)	107.3(8)	C(5)–Si(3)–C(6)	105.5(8)
N(2)–Si(2)–C(2)	115.4(6)	C(5)–Si(3)–C(7)	109.6(8)
N(2)–Si(2)–C(3)	110.8(6)	C(6)–Si(3)–C(7)	103.7(7)

correction was made due to the fractured nature of the crystal with its lack of measurable faces; max.  $(\sin\theta)/\lambda = 0.60 \text{ \AA}^{-1}$ ,  $h$  0 to 12;  $k$  –14 to 14,  $l$  –14 to 14; three standard reflections, 1.9% 3.0%, 2.2% variation in intensities of standards from average, intensities adjusted isotropically; 6769 data, 2045

unique (including 1194,  $F^2 > 3\sigma$ ),  $R_{int} = 0.057$ ; structure solved by Patterson and Fourier methods; refined on  $F$ , 99 parameters, H atoms not included, anisotropic thermal parameters;  $R = 0.072$  (2045 data),  $R = 0.027$  [ $F^2 > 3\sigma(F^2)$ ],  $wR = 0.035$ ,  $S = 1.24$ ,  $w = 4F^2[\sigma(F^2) + (0.04F^2)^2]^{-1}$ ; max. (shift/ $\sigma$ ) = 0.01; empirical extinction correction,  $F_{corr} = (1 + 9.5 \times 10^{-7})^{-1}$ ; max. and min. of  $\Delta F$  synthesis 0.9 and  $-1.2 \text{ e \AA}^{-3}$ . Atomic scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974). Local unpublished programs and ORTEP (Johnson, 1965) used. Atomic parameters are listed in Table 1. A list of selected bond distances and angles is given in Table 2. An ORTEP view of the molecule is shown in Fig. 1.\*

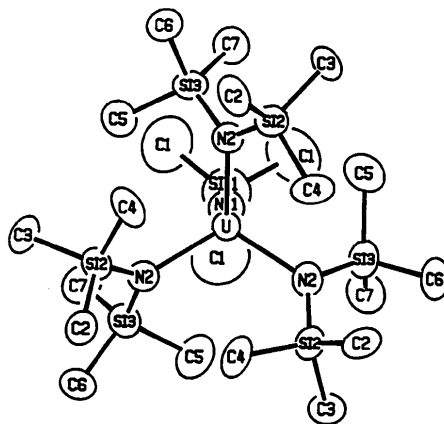


Fig. 1. ORTEP drawing (Johnson, 1965) of the molecule looking down and slightly off the threefold axis.

**Discussion.** The U–N(1)–Si(1) angle is required by the space group to be linear. The U–N(1) distance is 1.91 (2) Å and is substantially shorter than that found in  $(\text{MeC}_5\text{H}_4)_3\text{U}(\text{NC}_6\text{H}_5)$  of 2.019 (6) Å (Brennan & Andersen, 1985), where the U–N–C angle is slightly non-linear [167.4 (6)°]. Clearly there is a profound change in the geometrical parameters upon replacing the methylcyclopentadienyl groups with the bis(trimethylsilyl)amido groups in molecules of the same general stereochemistry,  $L_3\text{UL}'$ . The U–N(2) bond length is *ca* 0.4 Å longer than U–N(1), while the Si–N distances in the imido and amido ligands are nearly

equal, as are the Si–C distances. The N(2) atom is 0.07 Å from the plane consisting of U, Si(3) and Si(4).

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\* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44989 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of a Styrylbenzothiazole Platinum(II) Complex: $[\text{Net}_4][\text{PtBr}_3(\text{asb})]$

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**Abstract.** Tetraethylammonium tribromo[2-(2-acetoxystyryl)benzothiazole]platinate(II),  $[(\text{C}_2\text{H}_5)_4\text{N}][\text{PtBr}_3(\text{C}_{17}\text{H}_{13}\text{NO}_2\text{S})]$ ,  $M_r = 860.4$ , monoclinic,  $P2_1/c$ ,  $a = 11.230$  (9),  $b = 19.333$  (4),  $c = 13.685$  (6) Å,  $\beta = 101.06$  (4)°,  $V = 2916$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.96 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 90.3 \text{ cm}^{-1}$ ,  $F(000) = 1648$ ,  $T = 296 \text{ K}$ , final  $R = 0.047$  for 3384

unique observed reflections. The  $[\text{PtBr}_3(\text{asb})]^-$  unit has square-planar geometry about the Pt, with the asb coordinated to the Pt through the N of the thiazole ring and a Pt–N bond distance of 2.010 (8) Å. The average Pt–Br distance is 2.426 (7) Å. The ligand is non-planar with a dihedral angle of 22.4 (7)° between the benzothiazole and the acetoxystyrene rings. The