

Structures of Two  $M(S_5C_3)_2NBu_4$  ( $M = Pt, Ni$ ) Semiconductor Salts

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**Abstract.** (I) Tetrabutylammonium bis(2-thioxo-1,3-dithiole-4,5-dithiolato)platinate:  $[N(C_4H_9)_4][Pt(C_3S_5)_2]$ ,  $M_r = 830.2$ , monoclinic,  $C2/c$ ,  $a = 20.378$  (5),  $b = 13.384$  (3),  $c = 12.172$  (3) Å,  $\beta = 105.89$  (2)°,  $V = 3193$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.74$ ,  $D_x = 1.726$  Mg m<sup>-3</sup>,  $Mo K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 5.222$  mm<sup>-1</sup>,  $F(000) = 1668$ ,  $R = 0.0243$  for 1670 observed reflections with  $F_o \geq 6.0\sigma(F_o)$ ,  $T = 296$  (2) K. (II) Tetrabutylammonium bis(2-thioxo-1,3-dithiole-4,5-dithiolato)nickelate:  $[N(C_4H_9)_4][Ni(C_3S_5)_2]$ ,  $M_r = 693.9$ , triclinic,  $P\bar{1}$ ,  $a = 11.702$  (2),  $b = 12.120$  (2),  $c = 12.358$  (2) Å,  $\alpha = 100.04$  (1),  $\beta = 91.93$  (1),  $\gamma = 105.44$  (1)°,  $V = 1657.9$  (4) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.36$ ,  $D_x = 1.390$  Mg m<sup>-3</sup>,  $Mo K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 1.170$  mm<sup>-1</sup>,  $F(000) = 726$ ,  $R = 0.0366$  for 2760 observed reflections with  $F_o \geq 6.0\sigma(F_o)$ ,  $T = 296$  (2) K. The anions form stacks but the planes of the adjacent molecules within the stack are at angles of 76.1° (I) or 78.90° (II) to each other and the intermetal distances are very large. Thus there is no metal-metal interaction and only weak interstack S...S contacts. (II) is a new triclinic phase of a previously reported monoclinic phase.

**Introduction.** The 1,2-dithiolene complexes of transition metals have been the subject of several reviews (see Eisenberg, 1970; Burns & McAuliffe, 1979). A great deal of attention was paid to the square-planar complexes, formed by  $d^8$  metal ions such as  $Ni^{2+}$ ,  $Pd^{2+}$ ,  $Pt^{2+}$ , possessing many of the properties required for the formation of one-dimensional metals (Underhill & Ahmad, 1982; Ahmad, Turner, Underhill, Kobayashi, Sakaki & Kobayashi, 1984; Kobayashi, Mori, Sasaki, Kobayashi, Ahmad & Underhill, 1984). Syntheses and crystal structures have been reported for the  $[NBu_4][Ni(C_3S_5)_2]$  ( $H_2C_3S_5 = 4,5$ -dimercapto-1,3-dithiole-2-thione), and  $[NBu_4][Ni(C_3S_5)_2]$  salts by Lindqvist, Sjolín, Sieler, Steimecke & Hoyer (1979) and Lindqvist, Andersen, Sieler, Steimecke & Hoyer (1982), and for

the mixed-valence salts  $[NBu_4]_{0.28}[Ni(C_3S_5)_2]$  by Valade, Legros, Bousseau, Cassoux, Garbaskas & Interrante (1985). Papavassiliou (1981, 1983) has reported on the synthesis and conductivity of a new phase of  $[NBu_4][Ni(C_3S_5)_2]$  and the corresponding Pt salt. Here we report on  $[NBu_4][Ni(C_3S_5)_2]$  which crystallizes in space group  $P\bar{1}$  (while the Lindqvist phase crystallizes in  $P2_1/c$ ), and the corresponding Pt salt which crystallizes in  $C2/c$ .

**Experimental.** Crystal dimensions 0.09 × 0.11 × 0.35 (I) and 0.15 × 0.25 × 0.30 (II) mm. Density measured by flotation. Syntex  $P2_1$  diffractometer. Lattice parameters from 15 reflections,  $9^\circ < 2\theta < 21^\circ$  (I) and  $25^\circ < 2\theta < 27^\circ$  (II). Zr-filtered Mo radiation,  $\theta/2\theta$  scans. Data in range  $2\theta < 47^\circ$  (I),  $2\theta < 42^\circ$  (II) [range of  $hkl$ :  $-22 \rightarrow 22$ ,  $-14 \rightarrow 14$ ,  $0 \rightarrow 13$  (I);  $-11 \rightarrow 11$ ,  $-12 \rightarrow 12$ ,  $0 \rightarrow 12$  (II)]. Scan speed 1.5–12.0 (I), 1.0–8.0 (II) ( $2\theta$ ) min<sup>-1</sup>, scan width 1.8° ( $2\theta$ ) plus  $\alpha_1$ – $\alpha_2$  divergence. Three standard reflections, measured every 67 reflections, showed < 3.0% intensity fluctuation. Lp and numerical (I) ( $T_{max}/T_{min} = 0.6752/0.5422$ ) (Sheldrick, 1976) or empirical (II) (Syntex, 1976) absorption correction applied. Data collected/unique/ $R_{int}$ , 5021/2376/0.025 (I), 4003/3582/0.014 (II). Positional coordinates of Pt and one S atom (I) deduced from a Patterson synthesis. All the other non-H atoms revealed from  $\Delta F$  calculations (Sheldrick, 1976). The structure of (II) solved by the SIR-85 program† (Casarano *et al.*, 1985). Attempts to refine the structures in  $Cc$  (I) and  $P1$  (II) were unsuccessful leading to negative  $U$ 's and very poor bond distances. Refinement proceeded in  $C2/c$  (I) and  $P\bar{1}$  (II) by blocked full-matrix least squares, in which  $\sum w\Delta^2$  was minimized, with SHELX76 (Sheldrick, 1976). All non-H atoms refined using anisotropic temperature factors. H atoms (calculated) isotropic riding on carbons at 0.98 Å. Final refinement  $wR$

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Table 1. Positional and equivalent isotropic thermal parameters ( $\times 10^4$ ) of the non-hydrogen atoms; e.s.d.'s of the positional parameters in parentheses

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

Compound (I)	x	y	z	$U_{eq}(\text{\AA}^2)$
Pt	0	0	0	571
S(1)	1028.2 (8)	490 (1)	1153 (2)	747
S(2)	539.2 (7)	-924 (1)	-1059 (1)	757
S(3)	2468.5 (7)	2 (2)	1114 (2)	827
S(4)	2027.7 (8)	-1276 (2)	-848 (2)	905
S(5)	3508.1 (9)	-955 (2)	158	1204
C(1)	1590 (3)	-129 (5)	550 (5)	651
C(2)	1379 (3)	-726 (4)	-371 (5)	652
C(3)	2711 (3)	-759 (5)	138 (6)	851
N	0	2870 (5)	7500	638
C(11)	118 (3)	3549 (4)	8540 (5)	754
C(12)	302 (4)	3038 (6)	9673 (5)	995
C(13)	456 (4)	3755 (6)	10659 (6)	1083
C(14)	706 (6)	3321 (8)	11797 (7)	1486
C(21)	606 (3)	2182 (5)	7594 (6)	876
C(22)	1286 (4)	2685 (8)	7782 (7)	1252
C(23)	1867 (5)	2020 (10)	7950 (10)	2818
C(24)	2391 (8)	1890 (10)	8420 (10)	2444

Compound (II)	x	y	z	$U_{eq}(\text{\AA}^2)$
Ni(1)	5000	0	0	701
Ni(2)	0	0	0	765
S(11)	3602 (1)	237 (1)	8955 (1)	868
S(12)	6353 (1)	1342 (1)	9465 (1)	867
S(13)	3675 (1)	1892 (1)	7358 (1)	991
S(14)	6187 (1)	2923 (1)	7848 (1)	950
S(15)	4904 (2)	3778 (1)	6211 (1)	1224
S(21)	9392 (1)	1510 (1)	10597 (1)	1021
S(22)	10416 (1)	526 (1)	8438 (1)	973
S(23)	9168 (1)	3511 (1)	9512 (2)	1285
S(24)	10138 (1)	2631 (1)	7545 (1)	1121
S(25)	9396 (2)	4810 (2)	7676 (2)	1715
C(11)	4360 (4)	1331 (3)	8323 (3)	758
C(12)	5543 (4)	1813 (3)	8549 (3)	753
C(13)	4919 (5)	2903 (4)	7093 (3)	864
C(21)	9549 (4)	2211 (4)	9499 (4)	877
C(22)	9996 (4)	1797 (4)	8581 (4)	845
C(23)	9557 (4)	3709 (4)	8215 (5)	1152
N	3342 (3)	2383 (3)	2776 (3)	732
C(31)	3032 (5)	1109 (4)	2851 (4)	952
C(32)	3270 (5)	842 (4)	3978 (5)	1204
C(33)	2970 (7)	-449 (5)	3913 (6)	1760
C(34)	3224 (8)	-847 (6)	4952 (7)	2095
C(41)	2997 (4)	2415 (4)	1592 (4)	829
C(42)	3237 (5)	3615 (4)	1288 (4)	946
C(43)	2972 (6)	3484 (5)	60 (4)	1267
C(44)	3131 (6)	4542 (5)	-383 (5)	1471
C(51)	2698 (4)	3051 (4)	3574 (4)	839
C(52)	1376 (4)	2596 (5)	3471 (4)	1097
C(53)	907 (5)	3319 (6)	4468 (6)	1707
C(54)	-357 (8)	2888 (9)	4423 (9)	2861
C(61)	4652 (4)	2978 (4)	3089 (4)	834
C(62)	5489 (5)	2499 (6)	2403 (5)	1413
C(63)	6794 (6)	3041 (7)	2788 (8)	1953
C(64)	7240 (10)	3931 (9)	2900 (10)	3634

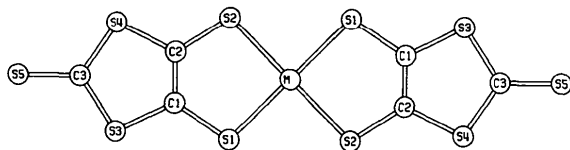
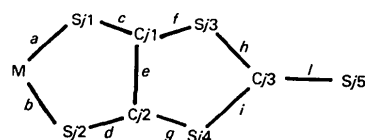


Fig. 1. An ORTEP view of the anion.

= 0.0278 (I) and 0.0527 (II) for observed data with  $1/w = \sigma^2(F_o) + kF_o^2$  where  $k = 0.00016$  (I) and 0.00077 (II). Five reflections showing poor agreement were given zero weight during final refinement cycles.  $R/wR$ : 0.0474/0.0334 (I) and 0.0524/0.0668 (II) for

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) of the anions; e.s.d.'s in parentheses



The digit  $j (=1,2)$  is used only in the case of (II).

Bond lengths or angles	(II)		
	(I)	Ni(1)	Ni(2)
a	2.276 (1)	2.160 (1)	2.158 (1)
b	2.275 (2)	2.160 (1)	2.167 (1)
c	1.730 (6)	1.713 (4)	1.713 (6)
d	1.708 (6)	1.717 (5)	1.720 (5)
e	1.348 (7)	1.350 (6)	1.340 (7)
f	1.743 (5)	1.743 (5)	1.745 (5)
g	1.745 (6)	1.743 (4)	1.751 (6)
h	1.736 (7)	1.718 (5)	1.720 (6)
i	1.716 (7)	1.719 (5)	1.730 (6)
l	1.639 (6)	1.651 (5)	1.641 (6)
a-b	90.0 (1)	93.1 (0)	93.0 (1)
a-c	101.8 (2)	102.2 (2)	102.2 (2)
b-d	102.3 (2)	102.0 (1)	101.6 (2)
c-f	120.8 (4)	123.0 (3)	122.6 (3)
c-e	122.7 (4)	121.3 (4)	121.4 (4)
d-g	121.4 (4)	122.6 (3)	122.2 (3)
d-e	123.2 (4)	121.4 (3)	121.8 (4)
f-e	116.5 (4)	115.7 (3)	116.0 (4)
g-e	115.4 (4)	116.0 (4)	116.1 (4)
f-h	97.3 (3)	97.6 (2)	97.8 (3)
g-i	98.1 (3)	97.4 (2)	97.3 (3)
h-i	112.7 (3)	113.3 (3)	112.9 (3)
h-l	123.4 (4)	123.7 (3)	123.4 (3)
i-l	123.9 (4)	123.1 (3)	123.7 (4)

all data. S: 1.46 (I) and 1.70 (II).  $|\Delta/\sigma|_{\max} = 0.194$  for (I) and 0.768 for (II) [ $U_{33}$  of C(64) lying at the end of a chain]. Number of refined parameters 177 (I) and 327 (II).  $(\Delta\rho)_{\max}/(\Delta\rho)_{\min}$ : 0.47/-0.46 (I) and 0.31/-0.39 (II) e  $\text{\AA}^{-3}$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,\* bond lengths and angles of the anions in Table 2, an atom-numbering scheme in Fig. 1 and ORTEP (Johnson, 1976) stereoviews of the molecular packing in Figs. 2 (I) and 3 (II).

**Discussion.** The unit cell of (I) contains one independent Pt(C<sub>3</sub>S<sub>5</sub>) moiety with Pt at the origin and one N(C<sub>4</sub>H<sub>8</sub>)<sub>2</sub> moiety with N on the twofold axis, while in (II) there are two independent Ni(C<sub>3</sub>S<sub>5</sub>) moieties (symmetry  $\bar{1}$ ) and one NBU<sub>4</sub><sup>+</sup> cation. The anions are very nearly planar with greatest deviations from the least-squares plane of 0.036  $\text{\AA}$  [S(5)] for (I) and 0.026  $\text{\AA}$  [S(2)] for (II). Corresponding bond lengths

\* Lists of observed and calculated structure factors, anisotropic thermal parameters of the non-H atoms, positional and thermal parameters of the H atoms and bond lengths and angles of the cations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44978 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and angles are very close to those observed elsewhere for Pt (Kobayashi *et al.*, 1984; Ahmad *et al.*, 1984) or Ni (Lindqvist *et al.*, 1979, 1982; Valade *et al.*, 1985) dithiolate complexes and there is a very good agreement in the bond lengths between the two independent Ni complexes. All the atoms show an unusually high

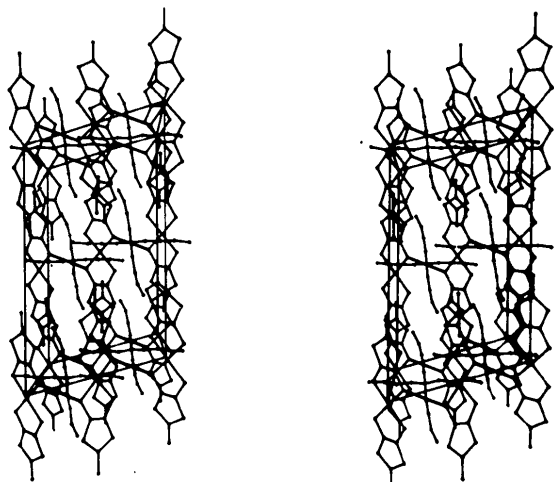


Fig. 2. Stereoview of (I) looking down the  $b$  axis. Heavy lines indicate  $S \cdots S$  contacts.

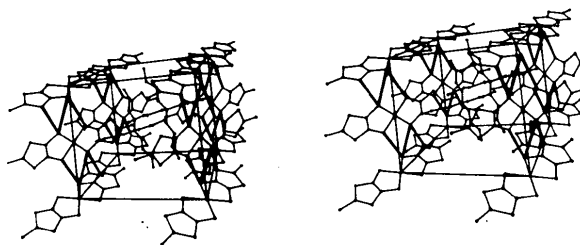


Fig. 3. Stereoview of (II) looking down the  $b$  axis. Heavy lines indicate  $S \cdots S$  contacts.

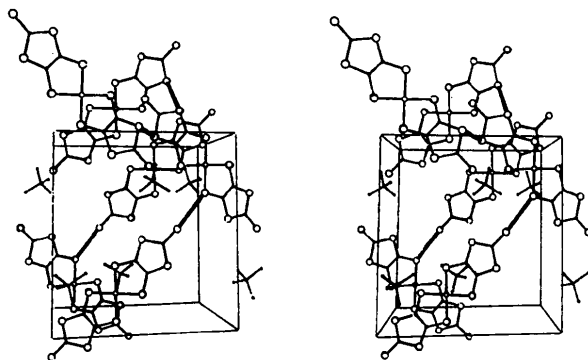


Fig. 4. Stereoview of the Lindqvist phase of (II) looking down the  $c$  axis. Heavy lines indicate  $S \cdots S$  contacts. The  $NBu_4$  is represented by  $NC_4$  to improve clarity.

thermal motion. The butyl chains are either slightly disordered (though we failed to resolve this disorder) or they have a large thermal motion which progressively increases towards the end of the butyl chains. As a consequence the C—C distances in the cation appear to differ considerably from standard values and vary from 1.430 (10) to 1.585 (9) Å, with two at the ends of the butyl chains, C(23)—C(24) and C(63)—C(64), of only 1.08 (1) and 1.05 (1) Å, respectively. Such high thermal motion in  $NBu_4$  with the consequent shortening of C—C distances has been observed before (Lindqvist *et al.*, 1979). The N—C distances are normal, in the range 1.509 (6) to 1.523 (6) Å.

The anions form stacks parallel to  $c$  (I) and  $a$  (II) axes. The planes of the adjacent anions within the stacks are not parallel, but are at angles of  $76.1^\circ$  (I) and  $78.9^\circ$  (II) to each other and the intermetal distances are large, 6.086 Å (I) and 5.851 Å (II). Thus there is no metal—metal interaction. There is one intrastack  $S \cdots S$  contact at 3.594 (3) Å in (I) (Fig. 2) and three such contacts at 3.618 (3), 3.696 (3) and 3.719 (3) Å in (II) (Fig. 3). There are no short interstack contacts. Since there is no overlap between the anions within the stack and the  $S \cdots S$  interactions are weak (sum of Van der Waals radii = 3.60 Å) these salts are expected to have low conductivity (Papavassiliou, 1983). The packing arrangement of the present phase of  $[NBu_4][Ni(C_3S_3)_2]$ , Fig. 3, is completely different from the packing of the Lindqvist phase, Fig. 4. In the present phase there is stacking of the anions but only weak  $S \cdots S$  contacts. In the Lindqvist phase there is no stacking but there are two quite strong  $S \cdots S$  contacts at 3.402 and 3.546 Å and a weaker one at 3.611 Å.

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

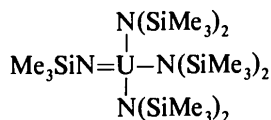
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**Abstract.**  $\{[(\text{CH}_3)_3\text{Si}]_2\text{N}\}_3\text{U}=\text{NSi}(\text{CH}_3)_3$ ,  $M_r = 806.40$ , rhombohedral,  $R\bar{3}c$ ,  $a = 12.495(4) \text{ \AA}$ ,  $\alpha = 89.83(3)^\circ$ ,  $V = 1950.8 \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 1.373 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 41.7 \text{ cm}^{-1}$ ,  $F(000) = 814$ ,  $T = 296 \text{ K}$ ,  $R = 0.027$  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