

Table 6. Hydrogen-bonding geometry (Å, °) for (II)

D	H	A	H...A	D...A	D—H...A
N(1)	H(1A)	Cl(14 ⁱ)	2.824 (26)	3.563 (4)	161.3 (4.7)
N(1)	H(1B)	Cl(25)	2.853 (26)	3.594 (4)	161.7 (4.7)
N(2)	H(2A)	Cl(16 ⁱ)	2.778 (24)	3.537 (5)	167.7 (5.3)
N(2)	H(2B)	Cl(24)	3.030 (31)	3.740 (5)	154.0 (5.1)

Symmetry code: (i) $1 - x, 1 - y, 2 - z$; (ii) $-x, 1 - y, 2 - z$.

This work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55969 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1034]

References

- Barner, C. J., Collins, T. J., Mapes, B. E. & Santarsiero, B. D. (1986). *Inorg. Chem.* **25**, 4322–4323.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Flack, H. D. & Schwarzenbach, D. (1988). *Acta Cryst.* **A44**, 499–506.
- Hursthouse, M. B., Short, R. L., Kelly, P. F. & Woollins, J. D. (1988). *Acta Cryst.* **C44**, 1731–1733.
- Hursthouse, M. B., Walker, N. P. C., Warrens, C. P. & Woollins, J. D. (1985). *J. Chem. Soc. Dalton Trans.* pp. 1043–1047.
- Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1990b). *SHELXTL-Plus*. Version 4.3. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1992). *SHELXL92*. Program for crystal structure refinement. Univ. of Göttingen, Germany.

Acta Cryst. (1993). **C49**, 1283–1284

Structure of Potassium Silanolate at 153 K

FRANK PAUER AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie, Universität Göttingen, Tammannstrasse 4, 3400 Göttingen, Germany

(Received 13 November 1992; accepted 3 February 1993)

Abstract

The structure of tetrapotassium tetrakis(2-methyl-2-sila-2-propanolate), $(\text{KOSiMe}_3)_4$, is reported. The cubane-like tetramer lies on a position of crystallographic symmetry 23; the Me_3SiO unit and the K atom lie on a crystallographic threefold axis.

Comment

The structure of $(\text{KOSiMe}_3)_4$ has been determined previously from powder diffraction data. It was published in the

space group $P\bar{4}3m$ [$a = 8.844(1) \text{ \AA}$ (Weiss, Hoffmann & Grützmacher, 1990)]. The single-crystal X-ray diffraction data show that after doubling the axes [$a = 17.573(2) \text{ \AA}$] additional weak uuu reflections are present. This leads to an F -centred lattice and the space group $F\bar{4}3c$. Omitting the reflections with uuu indices gives the primitive cell mentioned above. Presumably, these uuu reflections were overlooked in the powder diffraction experiment because of their relative weakness. The good agreement of the single-crystal data with the $F\bar{4}3c$ model makes it very probable that this is the correct space group. This may also be true for the isostructural Rb and Cs species. The change of space group reduces the symmetry of the tetramer from $T_d(43m)$ to $T(23)$ and involves a rotation of the Me_3Si group by 19.5° about the threefold axis. There are no close intermolecular contacts.

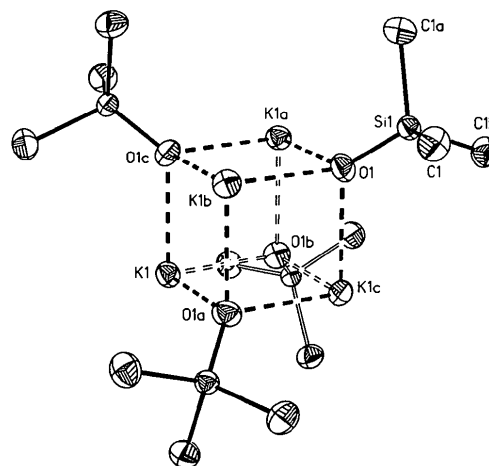


Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

Crystal data

$4\text{K}^+ \cdot 4\text{C}_3\text{H}_9\text{OSi}^-$

$M_r = 513.2$

Cubic

$F\bar{4}3c$

$a = 17.573(2) \text{ \AA}$

$V = 5426.7(11) \text{ \AA}^3$

$Z = 8$

$D_x = 1.256 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71072 \text{ \AA}$

Cell parameters from 60 reflections

$\theta = 8-55^\circ$

$\mu = 0.845 \text{ mm}^{-1}$

$T = 153(2) \text{ K}$

Cube

$0.5 \times 0.5 \times 0.5 \text{ mm}$

Colourless

Data collection

Stoe-Siemens AED diffractometer

Profile data from $2\theta/\omega$ scans

Absorption correction:

none

$R_{\text{int}} = 0.0263$

$\theta_{\text{max}} = 27.46^\circ$

$h = -22 \rightarrow 22$

$k = -22 \rightarrow 22$

$l = -13 \rightarrow 13$

1010 measured reflections
526 independent reflections
400 observed reflections
[$I > 2\sigma(I)$]

3 standard reflections
frequency: 90 min

Refinement

Refinement on F^2

Final $R_1 = 0.0227$ for

$F > 4\sigma(F)$

$wR_2 = 0.0716$ for all F^2 data

$S = 1.084$

524 reflections

20 parameters

Calculated weights

$$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2 + 0.5296P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = -0.001$$

$$\Delta\rho_{\max} = 0.186 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.194 \text{ e } \text{\AA}^{-3}$$

Atomic scattering factors

from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Absolute structure determined according to Flack (1983)

Refinement was on F^2 for all reflections except for two flagged for possible systematic errors; the observed threshold $F > 4\sigma(F)$ is used only for calculating R_1 and wR_2 . The goodness-of-fit value is calculated using F^2 . The unweighted R factor is based on F and is intended for comparison with other refinements based on F . The methyl H atoms were located by difference Fourier synthesis, but in the refinement an idealized methyl group was allowed to rotate freely around the local threefold axis to optimize the torsion angle.

Data collection: *DIF4* 7.08 (Siemens diffractometer control software). Cell refinement: *DIF4* 7.08 (Siemens diffractometer control software). Data reduction: *REDU4* (Siemens diffractometer control software). Program(s) used to solve structure: *SHELXS92* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL92*.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$			
	x	y	z	U_{eq}
K1	0.82521 (2)	0.82521 (2)	0.82521 (2)	0.0284 (2)
Si1	0.62369 (3)	0.62369 (3)	0.62369 (3)	0.0225 (2)
O1	0.67561 (6)	0.67561 (6)	0.67561 (6)	0.0309 (4)
C1	0.61473 (14)	0.66142 (13)	0.52405 (10)	0.0370 (5)

Table 2. Geometric parameters (\AA , $^\circ$)

K1—O1 ⁱ	2.6290 (11)	Si1—C1	1.879 (2)
Si1—O1	1.580 (2)		
O1 ⁱ —K1—O1 ⁱⁱ	89.37 (5)	Si1—O1—K1 ⁱ	124.82 (4)
O1—Si1—C1	112.50 (7)	K1 ⁱ —O1—K1 ^{iv}	90.63 (5)
C1—Si1—C1 ⁱⁱⁱ	106.28 (8)		

Symmetry code: (i) $x, \frac{3}{2} - y, \frac{3}{2} - z$; (ii) $\frac{3}{2} - x, y, \frac{3}{2} - z$; (iii) y, z, x ; (iv) $\frac{3}{2} - x, \frac{3}{2} - y, z$.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71100 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1040]

This work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

References

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.
Sheldrick, G. M. (1990b). *SHELXTL-Plus*. Siemens X-ray Analytical Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1992). *SHELXL92*. Program for crystal structure refinement. Univ. of Göttingen, Germany.
Weiss, E., Hoffmann, K. & Grützmacher, H. F. (1990). *Chem. Ber.* **103**, 1190–1197.

Acta Cryst. (1993). **C49**, 1284–1286

Structure of a Phenyl Dioxocyclam Nickel Complex

PAULA CHINN AND DARYLE H. BUSCH†

Chemistry Department, The Ohio State University,
Columbus, Ohio, USA

NATHANIEL W. ALCOCK

Department of Chemistry, University of Warwick,
Coventry CV4 7AL, England

(Received 12 June 1992; accepted 7 January 1993)

Abstract

The molecule, (3-phenyl-1,5,8,12-tetraazacyclotetradecane-2,4-dionato-*N,N',N'',N'''*)nickel(II), has near-mirror symmetry with its two amine N—H bonds directed to the same side of the coordination plane. The Ni—N(amide) bonds are shorter than the Ni—N(amine) bonds [mean values 1.880 (2) and 1.937 (2) \AA , respectively]. The crystal contains an extensive network of hydrogen bonds.

Comment

The compound was prepared as part of a programme investigating the influence of substitution on the properties of macrocyclic ligands. Spectroscopic data and assignments are given by Chinn (1987). The X-ray structure was determined because no dioxocyclam complexes had hitherto been reported.

The overall structure of the neutral complex is as expected, with square-planar coordination about Ni;

† Present address: Chemistry Department, University of Kansas, Lawrence, Kansas 66045, USA.