Dunaj-Jurčo, M., Kabešová, M., Kettman, V., Císařová, I. & Mikloš, D. (1993). *Acta Cryst.* C**49**, 1476–1479.
Dunaj-Jurčo, M., Ondrejovič, G., Melník, M. & Garaj, J. (1988). *Coord. Chem. Rev.* **83**, 1–28.
Harrison, W. D. & Hathaway, B. J. (1980). *Acta Cryst.* B**36**, 1069–1074.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Pavelčík, F. (1986). *XP21*. Comenius Univ., Pharmaceutical Faculty, Bratislava, Czechoslovakia.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Ugliengo, P., Borzani, G. & Viterbo, D. (1988). *J. Appl. Cryst.* **21**, 75.
Wicholas, M. & Wolford, T. (1974). *Inorg. Chem.* **13**, 316–318.

Acta Cryst. (1993). C**49**, 1482–1483

Structure of Bis(pentamethyldiethylenetriamine)disodium Hexasulfide

Susanne Besser, Regine Herbst-Irmer and
Dietmar Stalke

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

Alan T. Brooker, Ronald Snaith and
Domenec S. Wright

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

(*Received* 9 *September* 1992; *accepted* 1 *February* 1993)

Abstract

The new sodium sulfide compound, μ-hexasulfido-$1\kappa^2S^1,S^6:2\kappa^2S^1,S^6$-bis{[*N*-(dimethylaminoethyl)-*N*,*N*',-*N*'-trimethyl-1,2-ethanediamine-κ^3N,N',N'']sodium}, $[Na(pmdeta)]_2[S_6]$ (1) [pmdeta = $(Me_2NCH_2CH_2)_2NMe$], was obtained by reacting two equivalents of sodium hydride with elementary sulfur in toluene containing an excess of pmdeta at approximately 238 K. The crystal structure contains a central Na_2S_2 ring in which both S atoms are part of an S_6^{2-} residue. In addition, each Na atom is coordinated by a pmdeta molecule.

Comment

The structure is a contact ion pair where the S_6^{2-} ion adopts a bent zigzag chain and the two Na^+ ions are both bridging the terminal S atoms. Thus a four-membered Na_2S_2 ring is formed. Furthermore, each Na atom is coordinated by a pmdeta molecule. Therefore, the overall structure is comparable to that of $[Li(tmeda)]_2[S_6]$ (tmeda = $Me_2NCH_2CH_2NMe_2$) (Banister, Barr, Brooker, Clegg, Cunnington, Doyle, Drake, Gill, Manning, Raithby, Snaith, Wade & Wright, 1990), but it is quite different from that of the ionic and acyclic polysulfides BaS_3, BaS_4, K_2S_5 and Ca_2S_6 (Wells, 1984). The four-membered Na_2S_2 ring is not planar; it is bent along the Na1$\cdots$Na1^i vector. The normal of the Na1-Na1^i-S3 plane intersects that of the Na1-Na1^i-S3^i plane with an angle of 19.80 (2)°. The bond lengths within the ring are 2.9073 (9) (Na1—S3) and 2.8245 (9) Å (Na1—S3^i). The bond distances in the S_6^{2-} chain are not of the same length. Whereas the terminal bonds are 2.0344 (7) (S2—S3) and 2.0487 (7) Å (S1—S2), the central S1—S1^i bond is 2.1158 (9) Å long, hence 0.074 Å longer than the others. However, the average S—S bond length is in good agreement with the values for comparable derivatives, such as Cs_2S_6 (Abrahams & Grison, 1953) (average 2.054 Å) and $[Li(tmeda)]_2[S_6]$ (Tatsumi, Inoue, Nakamura, Cramer, VanDoorne & Gilje, 1990) (average 1.983 Å).

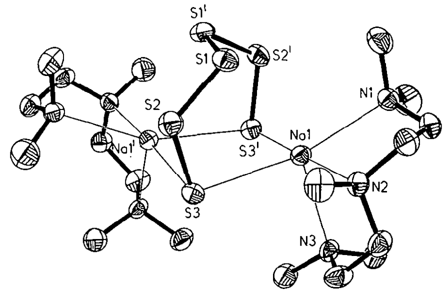

Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

Crystal data

$[Na_2(C_9H_{23}N_3)_2][S_6]$
M_r = 584.95
Tetragonal
$P\bar{4}2_1c$
a = 13.2390 (10) Å
c = 18.400 (2) Å
V = 3225.0 (5) Å^3
Z = 4
D_x = 1.205 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 64 reflections
θ = 10–12.5°
μ = 0.469 mm^{-1}
T = 293.0 (10) K
0.6 × 0.5 × 0.4 mm
Red

Data collection

Stoe Siemens AED four-circle diffractometer
Profile data from $2\theta/\omega$ scans
6917 measured reflections
3567 independent reflections
3273 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int}$ = 0.0304

$\theta_{\rm max}$ = 27.49°
$h = -14 \rightarrow 17$
$k = -12 \rightarrow 12$
$l = -23 \rightarrow 23$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
Final $R(F) = 0.0269$ for $F > 4\sigma(F)$ data
$wR(F^2) = 0.0616$ for all data
$S = 1.057$
3565 reflections
150 parameters
Calculated weights $w = 1/[\sigma^2(F_o^2)+(0.0202P)^2 + 0.8121P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.194$ e Å^{-3}
$\Delta\rho_{\min} = -0.150$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration according to Flack (1983)

Data collection: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS*-92 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*-92 (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*-92.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S1	0.07958 (3)	0.50720 (4)	0.98797 (2)	0.0327 (2)
S2	0.11554 (3)	0.63303 (3)	0.92854 (2)	0.0319 (2)
S3	0.10418 (3)	0.60140 (3)	0.82065 (2)	0.0276 (2)
Na1	0.10709 (5)	0.38329 (5)	0.83890 (3)	0.0247 (3)
N1	0.13872 (12)	0.21307 (11)	0.90150 (8)	0.0313 (8)
N2	0.29592 (11)	0.36127 (11)	0.85166 (8)	0.0289 (7)
N3	0.17224 (11)	0.36322 (12)	0.71214 (8)	0.0325 (8)
C11	0.0722 (2)	0.1363 (2)	0.87043 (13)	0.0511 (13)
C12	0.1193 (2)	0.2189 (2)	0.98012 (10)	0.0433 (12)
C13	0.24482 (14)	0.18790 (15)	0.88728 (11)	0.0347 (10)
C21	0.3395 (2)	0.4542 (2)	0.88127 (12)	0.0433 (11)
C22	0.31559 (14)	0.27586 (14)	0.90035 (10)	0.0338 (9)
C23	0.33531 (14)	0.3415 (2)	0.77781 (10)	0.0349 (9)
C31	0.1173 (2)	0.4340 (2)	0.66560 (10)	0.0431 (12)
C32	0.1640 (2)	0.2622 (2)	0.68081 (11)	0.0424 (12)
C33	0.27755 (13)	0.3961 (2)	0.71878 (10)	0.0378 (10)

Table 2. *Geometric parameters* (Å, °)

S1—S2	2.0487 (7)	S3—Na1	2.9073 (9)
S1—S1$^{\rm i}$	2.1158 (9)	Na1—N3	2.501 (2)
S2—S3	2.0344 (7)	Na1—N2	2.528 (2)
S3—Na1$^{\rm i}$	2.8245 (9)	Na1—N1	2.565 (2)
S2—S1—S1$^{\rm i}$	107.74 (3)	N3—Na1—S3$^{\rm i}$	103.79 (4)
S3—S2—S1	109.65 (3)	N2—Na1—S3$^{\rm i}$	177.09 (4)
S2—S3—Na1$^{\rm i}$	86.70 (2)	N1—Na1—S3$^{\rm i}$	106.14 (4)
S2—S3—Na1	95.21 (2)	N3—Na1—S3	90.14 (4)
Na1$^{\rm i}$—S3—Na1	94.05 (2)	N2—Na1—S3	97.95 (4)
N3—Na1—N2	74.53 (5)	N1—Na1—S3	157.90 (4)
N3—Na1—N1	105.62 (5)	S3$^{\rm i}$—Na1—S3	84.37 (2)
N2—Na1—N1	72.28 (5)		

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

DS thanks the University of Göttingen and the DAAD (Nato scholarship) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71149 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1023]

References

Abrahams, S. C. & Grison, E. (1953). *Acta Cryst.* **6**, 206-213.

Banister, A. J., Barr, D., Brooker, A. T., Clegg, W., Cunnington, M. J., Doyle, M. J., Drake, S. R., Gill, W. R., Manning, K., Raithby, P. R., Snaith, R., Wade, K. & Wright, D. S. (1990). *J. Chem. Soc. Chem. Commun.* pp. 105-107.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876-881.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1992). *SHELXL*-92. Program for crystal structure refinement. Univ. of Göttingen, Germany.

Stoe & Cie (1988). *DIF*4. Version 7.08. Diffractometer control program. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1988). *REDU*4. Data reduction program. Stoe & Cie, Darmstadt, Germany.

Tatsumi, K., Inoue, Y., Nakamura, A., Cramer, R. E., VanDoorne, W. & Gilje, J. W. (1990). *Angew. Chem.* **102**, 455-457; *Angew. Chem. Int. Ed. Engl.* **29**, 422-424.

Wells, A. F. (1984). *Structural Inorganic Chemistry*, 5th ed. Oxford Univ. Press.

Acta Cryst. (1993). C**49**, 1483–1485

Structure of μ-[Bis(chloroacetato-*O*)mercury(II)-κ^2*Hg*]-μ-[1,8-naphthalenediamino-κ^2*NN'*:κ^2*NN'*]-bis[*cis*-dicarbonyl(triisopropylphosphine-*P*)ruthenium(I)](*Ru*—*Ru*)

S. García-Granda* and
J. F. Van der Maelen Uría

Departamento de Química Física y Analítica,
Facultad de Química, Universidad de Oviedo,
Julián Clavería s/n, 33006 Oviedo, Spain

J. A. Cabeza, J. M. Fernández-Colinas
and V. Riera

Departamento de Química Orgánica e Inorgánica,
Facultad de Química, Universidad de Oviedo,
Julián Clavería s/n, 33006 Oviedo, Spain

(*Received* 8 *October* 1992; *accepted* 11 *February* 1993)

Abstract

The molecule shows a twofold symmetry through the Hg atom and the diaminonaphthalene ligand. Ru—Ru and Ru—Hg distances are 2.799 (1) and 2.795 (1) Å, respectively.

Comment

This work is part of a project concerning the reactivity of the Ru—Ru bonds of binuclear ruthenium(I)