Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5788 measured reflections
4448 independent reflections
2609 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$

$\theta_{\rm max} = 27.50°$
$h = -25 \rightarrow 24$
$k = -7 \rightarrow 1$
$l = -1 \rightarrow 21$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.138$
$S = 0.891$
4448 reflections
337 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0873P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.214$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.163$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N—C20	1.315 (2)	C13—C14	1.370 (2)
N—C1	1.360 (2)	C13—C18	1.413 (2)
C1—C10	1.412 (2)	C14—C15	1.406 (3)
C1—C2	1.445 (2)	C15—C16	1.365 (3)
C2—C3	1.401 (2)	C16—C17	1.411 (3)
C2—C7	1.410 (2)	C17—C18	1.398 (2)
C3—C4	1.373 (2)	C17—C21	1.409 (2)
C4—C5	1.389 (3)	C18—C19	1.406 (2)
C5—C6	1.350 (3)	C19—C23	1.376 (2)
C6—C7	1.406 (2)	C19—C20	1.476 (2)
C7—C8	1.421 (2)	C21—C22	1.369 (3)
C8—C9	1.344 (2)	C22—C23	1.429 (2)
C9—C10	1.437 (2)	C23—C24	1.489 (2)
C10—C11	1.406 (2)	C24—C25	1.528 (3)
C11—C12	1.360 (2)	C25—C26	1.484 (3)
C12—C20	1.435 (2)	C26—C27	1.511 (4)
C12—C13	1.468 (2)		
C20—N—C1	116.45 (12)	C17—C18—C13	122.93 (15)
N—C1—C10	122.74 (13)	C19—C18—C13	112.50 (13)
N—C1—C2	117.63 (13)	C23—C19—C20	134.31 (14)
C11—C12—C13	133.84 (14)	C18—C19—C20	105.71 (12)
C20—C12—C13	108.03 (12)	N—C20—C12	124.98 (12)
C14—C13—C18	118.89 (15)	N—C20—C19	127.10 (12)
C14—C13—C12	135.27 (15)	C12—C20—C19	107.92 (12)
C18—C13—C12	105.83 (13)	C19—C23—C22	115.8 (2)
C18—C17—C21	115.0 (2)	C19—C23—C24	123.23 (14)
C18—C17—C16	116.5 (2)	C23—C24—C25	112.15 (15)
C21—C17—C16	128.5 (2)	C26—C25—C24	114.3 (2)
C17—C18—C19	124.57 (15)	C25—C26—C27	113.5 (3)
C19—C23—C24—C25	84.7 (2)	C23—C24—C25—C26	−175.1 (2)
C22—C23—C24—C25	−93.9 (2)	C24—C25—C26—C27	−179.4 (3)

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from difference Fourier maps and refined isotropically. Program used for geometrical calculations: *PARST* (Nardelli, 1983).

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*.

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. One of the authors (KC) thanks the Universiti Sains Malaysia for a Visiting Post Doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1329). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1865–1867

N-(4-Nitrophenyl)-*trans*-2-aminocyclohexanol

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(*Received 14 May 1997; accepted 11 July 1997*)

Abstract

The phenyl ring in $C_{12}H_{16}N_2O_3$ is planar and the nitro group is distorted from coplanarity with it. The cyclohexane ring adopts a boat conformation. The molecules exist as centrosymmetrically-related dimers and pack as linear chains parallel to the *b* axis.

Comment

The β-aminoalcohol sequence plays an important part in organic and in medicinal chemistry (Goodman & Gilman, 1980). Specifically, the β-aminoalcohol subunit has been of particular value in the study of acetylcholine metabolism in intact nerve-terminal preparations (Rogers *et al.*, 1989). The crystal structure determination

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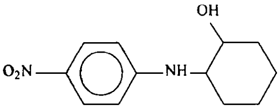
Acta Crystallographica Section C
ISSN 0108-2701 ©1997

of the title compound, (I), one of the above derivatives, was performed in order to elucidate its molecular conformation.



(I)

Both N atoms are in the planar configuration and the N1—C1 distance is shorter than the N2—C4 distance as a result of delocalization of the electron cloud within the nitro group. The cyclohexane ring adopts a chair conformation with atoms C9 and C12 deviating from the mean plane formed by C7, C8, C10 and C11 by 0.654 (3) and −0.682 (2) Å, respectively. This mean plane makes a dihedral angle of 71.45 (8)° with the phenyl-ring plane. The nitro group is twisted out of the phenyl-ring plane by 8.0°.

In the crystal, the molecules exist as O—H···O hydrogen-bonded dimers around inversion centres and these dimers are linked by N—H···O hydrogen bonds to form linear chains parallel to the **b** direction (Table 2).

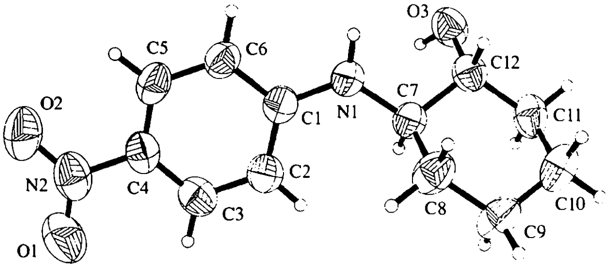


Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as small spheres of arbitrary radii.

Experimental

The title compound was synthesized by ring opening of cyclohexane oxide with *p*-nitroaniline (Sriraghavan & Ramakrishnan, 1997). Single crystals were grown by slow evaporation from a chloroform–methanol (1:2) solution.

Crystal data

$C_{12}H_{16}N_2O_3$
$M_r = 236.27$
Monoclinic
$C2/c$
$a = 18.703\ (2)$ Å
$b = 9.955\ (1)$ Å
$c = 15.016\ (1)$ Å
$\beta = 122.30\ (1)°$
$V = 2363.2\ (4)$ Å^3
$Z = 8$
$D_x = 1.328$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 44 reflections
$\theta = 5.32–12.52°$
$\mu = 0.096$ mm^{-1}
$T = 293\ (2)$ K
Rectangular slab
0.48 × 0.36 × 0.26 mm
Light brown

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3285 measured reflections
2700 independent reflections
1293 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$

$\theta_{\rm max} = 27.49°$
$h = -1 \rightarrow 24$
$k = -1 \rightarrow 12$
$l = -19 \rightarrow 16$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.096$
$S = 0.817$
2700 reflections
218 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.11$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (°)*

C1—N1—C7	124.5 (2)	C7—N1—H1N1	116 (1)
C1—N1—H1N1	118 (1)		
O1—N2—C4—C3	8.3 (3)	O2—N2—C4—C3	−171.3 (2)
O1—N2—C4—C5	−172.8 (2)	O2—N2—C4—C5	7.6 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O3$^{\rm i}$	0.88 (2)	2.07 (2)	2.931 (2)	165 (2)
O3—H1O3···O2$^{\rm ii}$	0.87 (3)	2.17 (2)	3.034 (2)	172 (2)

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

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometric calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Post Doctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1172). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1867–1869

Two Cyclotetrasiloxanes at 143 K

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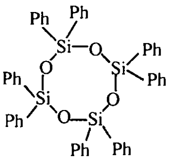
(*Received 6 May 1997; accepted 9 July 1997*)

Abstract

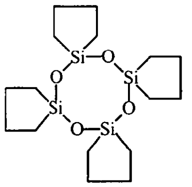
The crystal structures of octaphenylcyclotetrasiloxane [$C_{48}H_{40}O_4Si_4$, (I)] and 2,4,6,8-tetrakis(tetramethylene)cyclotetrasiloxane [a tetraspiro compound: cyclotetrasiloxane-2,4,6,8-tetraspiro-tetrakis(cyclopentane), $C_{16}H_{32}O_4Si_4$, (II)] were determined at 143 K. Compound (I) is compared with the room-temperature structure and the structure of the monoclinic polymorph, both reported previously. Whereas the siloxane ring of (I) is nearly planar, the siloxane ring of (II) exhibits a chair conformation. The angles between the siloxane ring and the various phenyl substituents of (I) vary considerably and are on average smaller in the triclinic form than in the monoclinic form. Greatly differing Si—O—Si angles, as found in the monoclinic form (153 and 167°), appear only in one of the molecules in the asymmetric unit of (I) [153.4 (1) and 163.7 (1)°]; the Si—O—Si angles of the second molecule differ by only 3.0°. The Si—O—Si angles of (II) display a difference of 5.2°. The silacyclopentane rings of (II) exhibit different conformations.

Comment

Octaphenylcyclotetrasiloxane, (I), and 2,4,6,8-tetrakis(tetramethylene)cyclotetrasiloxane, (II), were obtained from the reaction of diphenyldibromosilane or 1,1-dibromosilacyclopentane with *N*-methylimidazole in low yields. During preparation, the reactants must have been affected by atmospheric humidity and the associated hydrolysis must be the cause of the formation of the cyclosiloxanes.



(I)



(II)

Compound (I) crystallizes in the triclinic crystal system with two half molecules in the asymmetric unit, each molecule being situated on an inversion centre (Fig. 1). In contrast, the monoclinic form of (I) [as determined by Hossain, Hursthouse & Malik (1979), hereinafter referred to as mon1, and as determined by Braga & Zanotti (1980), hereinafter mon2] does not show any crystallographic symmetry. A comparison with the crystal structure of the triclinic form of (I), which has been determined at room temperature by Ovchinnikov *et al.*, (1988) and is hereinafter referred to as tric1, shows some remarkable differences. Firstly, there are differences in the cell parameters. *a* and *b* of (I) are slightly smaller than in tric1 [10.731 (1) and 10.764 (2) Å, respectively], whereas *c* is considerably shorter than in tric1 [19.147 (2) Å]. γ is nearly equal in both structures [76.12 (1)° in tric1], but whereas α was found to be larger than β in tric1 [83.79 (1) and 83.08 (1)°, respectively], the reverse was found for (I) at low temperature.

The siloxane ring is nearly planar [r.m.s. deviations: 0.051 Å in molecule 1 of (I) and 0.057 Å in molecule 2 of (I); 0.057 and 0.048 Å in tric1; 0.046 and 0.048 Å in mon1 and mon2, respectively]. All Si—O bond lengths of (I), ranging from 1.620 (1) to

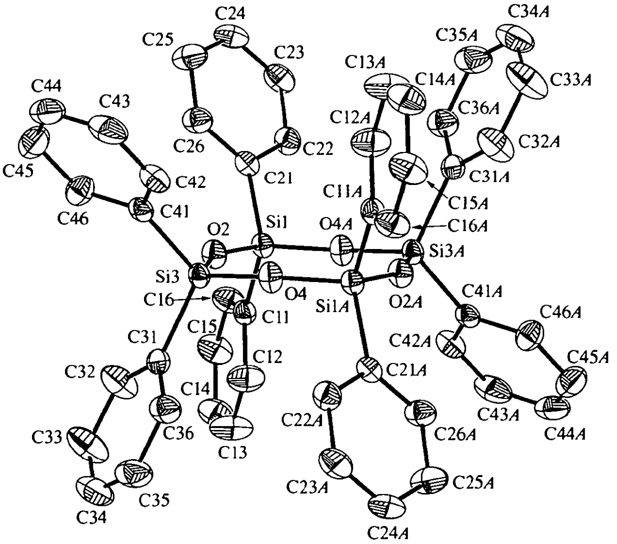


Fig. 1. Perspective view of molecule 1 of (I) with the atom numbering. Displacement ellipsoids are at the 50% probability level and H atoms have been omitted for clarity.