

Finally it is noteworthy that *trans*-azobenzene and *trans*-stilbene are not only isostructural and have almost the same packing coefficients (Kitaigorodsky, 1973), which are 0.75 and 0.74 respectively, but also show the same kind and degree of disorder. With this similarity in mind we can imagine that these molecules form a continuous series of mixed crystals.

We thank Drs A. J. M. Duisenberg for collecting the intensity data.

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Acta Cryst. (1984). C40, 431–433

15,30-Dihydroxy-12,27-dimethyl-5,20-dioxa-2,8,17,23-tetrathia[9.9]metacyclophane, C₂₆H₃₆O₄S₄

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(Received 21 July 1983; accepted 24 October 1983)

Abstract. $M_r = 540.83$, orthorhombic, $Pbca$, $a = 28.25$ (1), $b = 9.14$ (1), $c = 10.27$ (1) Å, $V = 2652$ (4) Å³, $Z = 4$, $D_m = 1.34$, $D_x = 1.35$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.40$ cm⁻¹, $F(000) = 1152$, $T = 293$ K. Final $R = 0.092$ for 1248 observed reflections. The molecule is a centrosymmetric 24-membered metacyclophane macrocycle. The intraannular hydroxyl groups are hydrogen-bonded to oxa ring atoms leading to the formation of two internal eight-membered rings; moreover, they are involved in transannular non-bonding interactions.

Introduction. Polycondensation in benzyl alcohol of 2,6-bis[(dimethylamino)methyl]-4-methylphenol with 2,2'-oxydiethanedithiol gives both a linear polymer and a macrocycle, which were characterized by several techniques (Costa Bizzarri, Della Casa, Ferruti, Ghedini, Pilati & Scapini, 1983); molecular-weight determination and a mass spectrum suggested a 12-membered ring for the macrocycle, while ¹H NMR spectra seem to indicate the presence of a larger ring. In order to elucidate this problem single-crystal X-ray analysis was undertaken.

Experimental. Crystals, in form of colourless prisms, obtained by evaporating a toluene solution for several days, and supplied by Costa Bizzarri

et al. D_m measured by flotation. Crystal of dimensions 0.21 × 0.24 × 0.35 mm used for data collection on an automatic Siemens AED diffractometer using Zr-filtered Mo $K\alpha$ radiation. Accurate cell dimensions and orientation matrix obtained from least-squares fit of θ , χ , φ values of 14 reflections in range $15 \leq \theta \leq 18^\circ$, measured by use of a narrow counter aperture. 2318 reflections up to $2\theta = 50^\circ$ (hkl range: 0–33, 0–10, 0–12) measured by θ – 2θ scan technique; intensities of three standard reflections, 512, $\bar{5}12$, $\bar{5}\bar{1}\bar{2}$, measured at 4 h intervals, no significant variation throughout data collection. Systematic absences: $Ok\bar{l}$, $k = 2n + 1$; $h0l$, $l = 2n + 1$; $hk0$, $h = 2n + 1$ consistent with space group $Pbca$ (No. 61). Intensities corrected for Lorentz and polarization factors. Structure solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by full-matrix least squares using 1248 reflections having $I \geq 3\sigma(I)$; refined quantity $\sum w\Delta F^2$, where $w = 1/\sigma^2$. A difference Fourier synthesis in final stages of refinement clearly indicated positions of all H atoms, except those of methyl group; residual electron density within -0.30 and 0.32 e Å⁻³. H contribution included and held fixed in final least-squares cycles, where S atoms refined anisotropically; $R = 0.092$, $wR = 0.086$, $S = 3.3$; max. $\Delta/\sigma = 0.19$. *SHELXTL* used throughout (Sheldrick, 1981). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

those about C(3)—C(4) and C(6)—C(7) which are antiperiplanar and synclinal respectively, the first favouring extension of the chain along the longer side of the rectangular cavity, the second favouring the folding of the shorter one; both push the two S atoms out of the cavity.

The molecules lie almost on planes perpendicular to the *c* axis; the crystal packing is regulated by normal van der Waals contacts.

This work was supported by CNR; computations were performed on the Univac 1100 of the Centro di Calcolo, University of Cagliari.

Acta Cryst. (1984). C40, 433–434

Structure of 2,2,4,4,6,6-Hexa-*tert*-butylcyclotrisilazane, [(C₄H₉)₂SiNH]₃

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(Received 20 September 1983; accepted 24 October 1983)

Abstract. $M_r = 472.0$, rhombohedral, space group $R\bar{3}c$, $a = 10.227(1)$, $c = 49.051(7)$ Å on hexagonal axes, $U = 4443.0$ Å³, $Z = 6$, $D_x = 1.058$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.17$ mm⁻¹, $T = 291$ K, $F(000) = 1584$, $R = 0.060$ for 736 observed reflections. The Si₃N₃ ring is planar, with internal angles at Si and N of 104.1 (2) and 135.9 (2)°. The Si—N bond length is 1.727 (2) Å.

Introduction. Compounds of the type $R_2\text{SiXY}$, in which X and Y are the functional groups OH or NH₂ and R is an alkyl substituent, readily undergo condensation reactions to give chain and ring siloxanes (Si—O—Si) and silazanes (Si—N—Si); when R is *tert*-butyl (Bu), such condensation is not observed, probably because of the steric bulk of the Bu substituents (Sommer & Tyler, 1954). An indirect synthetic route is required, and has been achieved *via* the intermediates Bu₂SiFOH and Bu₂SiFNH₂ (Klingebiel, 1981; Clegg, Klingebiel & Sheldrick, 1982; Klingebiel & Vater, 1983). The cyclotrisiloxane (Bu₂SiO)₃ contains a planar Si₃O₃ ring (Clegg, 1982). The cyclotrisilazane (Bu₂SiNH)₃ is found to be isostructural with it.

Experimental. Crystal obtained from *n*-hexane, 0.2 × 0.2 × 0.3 mm. Stoe—Siemens AED diffractometer. Unit-cell parameters from 2θ values of 38 reflections ($20 < 2\theta < 25^\circ$). 2049 reflections with $2\theta < 55^\circ$ and all indices ≥ 0 . Profile analysis on-line (Clegg, 1981). No

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significant variation for three standard reflections. No absorption corrections. $R_{\text{int}} = 0.017$, 1137 unique reflections, 736 with $F > 4\sigma(F)$. Automatic multi-solution direct methods. Blocked-cascade refinement on F ; $w^{-1} = \sigma^2(F) + 0.0004F^2$. Bu H atoms constrained to give C—H = 0.96 Å, H—C—H = 109.5°, $U(\text{H}) = 1.2U(\text{C})$; N—H refined freely subject to space-group symmetry constraints, with isotropic U . Anisotropic thermal parameters for non-H atoms. No extinction correction. Scattering factors from *International Tables for X-ray Crystallography* (1974). 58 parameters, $R = 0.060$, $wR = 0.058$. Slope of normal probability plot = 1.32. $(\Delta/\sigma)_{\text{max}} = 0.006$, $(\Delta/\sigma)_{\text{mean}} = 0.002$. Largest peak in final difference map 0.35 e Å⁻³, largest hole -0.29 e Å⁻³. Programs used: *SHELXTL* (Sheldrick, 1978); diffractometer control program by WC.

Discussion. Atomic coordinates are given in Table 1,* bond lengths and angles in Table 2. The molecular structure is shown in Fig. 1. (Bu₂SiNH)₃ is isostructural with (Bu₂SiO)₃ (Clegg, 1982), three N—H groups replacing the three O atoms. The Si₃N₃ ring is

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38978 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.