Data collection: *MolEN* (Fair, 1990). Cell refinement: *MolEN*. Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1378). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,1,3,3-Tetramethylurea

Christopher S. Frampton and Kevin E. B. Parkes

Roche Research Centre, PO Box 8, Welwyn Garden City, Hertfordshire, AL7 3AY, England. E-mail: framptoc@rwep03.dnet.roche.com

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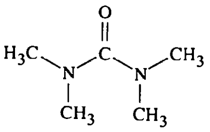
Abstract

Molecules of 1,1,3,3-tetramethylurea, $C_5H_{12}N_2O$, exhibit twofold symmetry and the geometry at the N atom deviates slightly from planarity.

Comment

The structure of 1,1,3,3-tetramethylurea, (I), was determined to investigate the degree of planarity at the N atoms. The molecule lies on a crystallographic twofold axis, as shown in Fig. 1. Bond lengths and angles are generally as expected (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The N atom, N(1), lies 0.201 (1) Å out of the plane defined by C(1), C(2) and C(3). The geometrical data correspond well with the structure of aqua-dioxo-bis(μ_2-hydrophosphito)uranium bis(1,1,3,3-tetramethylurea) (Mistryukov, Kanishcheva & Mikhailov, 1983) in which the 1,1,3,3-tetramethylurea is present as molecules of solvation. Other related structures are hexakis(1,1,3,3-tetramethylurea) trihydrophosphido-tetracontaoxa-dodeca-molybdenum (Prosser-Mccartha, Kadkhodayan, Williamson, Bouchard & Hill, 1986), triiodo-(1,1,3,3-tetramethylurea-*O*)iron (Pohl, Opitz, Saak & Haase, 1993) and trichloro-(1,1,3,3-tetramethylurea-*O*)aluminium (Bittner, Mannig & Noth, 1986). The first of these contains 1,1,3,3-tetramethylurea bonded through the O atom *via* a bifurcated hydrogen bond, the last two contain 1,1,3,3-tetramethylurea directly bonded to iron and aluminium, respectively, through the O atom. This has the effect of increasing the C—O distance from 1.226 (6) Å as found in the



(I)

present structure to 1.248, 1.279 (6) and 1.303 (3) Å, respectively. These structures all demonstrate a higher degree of planarity about the N atoms which lie on average 0.1 Å from the plane of the three bonded C atoms.

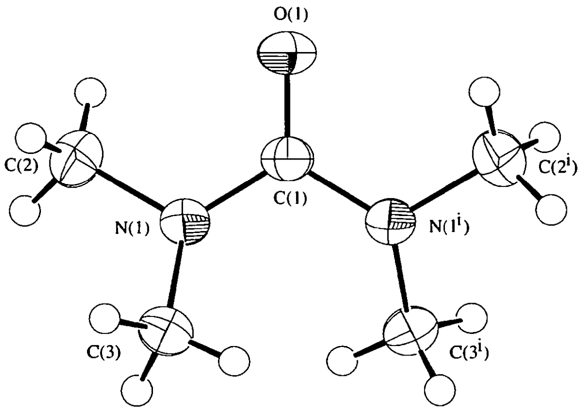


Fig. 1. View of the molecule showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level; H atoms are drawn as small circles of arbitrary radii. Symmetry transformation used to generate equivalent atoms: (i) $-x$, y, $3/2 - z$.

Experimental

A small drop of 1,1,3,3-tetramethylurea (99% purity, Aldrich Chemicals Ltd) was sealed in a 0.2 mm glass capillary and centred on the diffractometer. The sample was cooled with a stream of cold nitrogen gas from an Oxford Cryosystems Cryostream cooler (Cosier & Glazer, 1986) to approximately 10 K below the melting point of 272 K. A single crystal suitable for data collection was grown using a seed crystal from the polycrystalline solid by careful repeated cycles of warming and cooling.

Crystal data

$C_5H_{12}N_2O$
$M_r = 116.17$
Monoclinic
$C2/c$
$a = 10.6297\,(10)$ Å
$b = 6.2597\,(4)$ Å
$c = 9.9677\,(7)$ Å
$\beta = 93.034\,(7)^\circ$
$V = 662.31\,(9)$ Å^3
$Z = 4$
$D_x = 1.165$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 43.40\text{–}52.25^\circ$
$\mu = 0.669$ mm^{-1}
$T = 200\,(1)$ K
Cylinder
0.38 × 0.20 × 0.20 mm
Colourless

Data collection

Rigaku AFC-7R diffractometer
$\omega/2\theta$ scans
Absorption correction: none
718 measured reflections
678 independent reflections
569 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.011$
$\theta_{\rm max} = 69.81^\circ$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 7$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 150 reflections intensity decay: 0.29%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0337$
$wR(F^2) = 0.1309$
$S = 1.064$
615 reflections
63 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.27P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0063 (12)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
O(1)	0	0.2072 (2)	3/4	0.0521 (5)
N(1)	0.08543 (9)	−0.1035 (2)	0.68114 (10)	0.0388 (4)
C(1)	0	0.0113 (2)	3/4	0.0336 (4)
C(2)	0.18719 (13)	0.0125 (2)	0.6225 (2)	0.0484 (4)
C(3)	0.05292 (14)	−0.3034 (2)	0.61223 (13)	0.0441 (4)

Table 2. *Selected geometric parameters (Å, °)*

O(1)—C(1)	1.226 (2)	C(2)—H(2)	1.00 (2)
N(1)—C(1)	1.3706 (13)	C(2)—H(3)	0.98 (2)
N(1)—C(2)	1.451 (2)	C(3)—H(4)	1.01 (2)
N(1)—C(3)	1.460 (2)	C(3)—H(5)	0.98 (2)
C(2)—H(1)	0.95 (2)	C(3)—H(6)	0.94 (2)
C(1)—N(1)—C(2)	117.82 (11)	O(1)—C(1)—N(1)	121.64 (7)
C(1)—N(1)—C(3)	122.59 (10)	O(1)—C(1)—N(1^i)	121.64 (7)
C(2)—N(1)—C(3)	113.73 (10)	N(1)—C(1)—N(1^i)	116.71 (14)
C(2)—N(1)—C(1)—O(1)	8.11 (12)		
C(3)—N(1)—C(1)—O(1)	−143.21 (9)		
C(2)—N(1)—C(1)—N(1^i)	−171.89 (12)		
C(3)—N(1)—C(1)—N(1^i)	36.79 (9)		

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

Cell determination and data collection were performed at low temperature (200 K). The scan width in ω was $(1.47 + 0.14\tan\theta)^\circ$ with an ω scan rate of 8.0° per minute. The weak reflections $[I < 25\sigma(I)]$ were rescanned (maximum of 4 rescans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1063). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An Inorganic *N*-Amino Analogue of Pyrrole: a Five-Membered Ring with Methylhydrazine and Two B Atoms as Building Blocks

Udo Engelhardt* and Seung Soon Park

Institut für Anorganische und Analytische Chemie der Freien Universität Berlin, Fabeckstrasse 34-36, D-14195 Berlin, Germany. E-mail: udoengel@blume.chemie.fu-berlin.de

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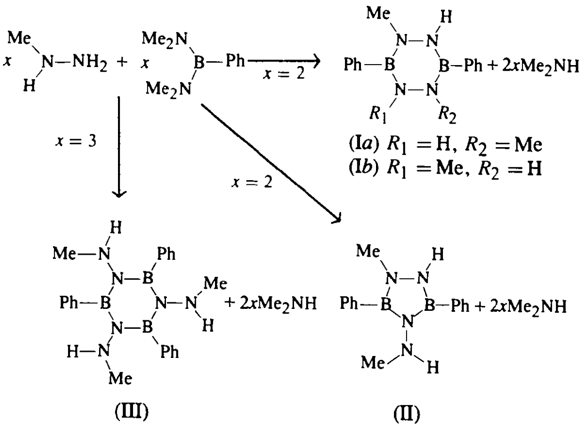
Abstract

The compound 1-methyl-4-methylamino-3,5-diphenyl-1,2,4-triaza-3,5-diborole, $C_{14}H_{18}B_2N_4$, has been synthesized from bis(dimethylamino)phenylborane and methylhydrazine. The molecular structure of the planar ring system is best described as an inorganic *N*-methylaminopyrrole with two rather short and two normal B—N bonds [1.40 (1) and 1.44 (1) Å, mean] and a short N—N bond [1.414 (5) Å] in the ring [exocyclic N—N 1.441 (4) Å].

Comment

Inorganic rings containing boron atoms and hydrazine building blocks have long been known (Nöth & Regnet, 1969; Nöth, Reichenbach & Winterstein, 1977; Nölle & Nöth, 1978; Barlos & Nöth, 1980; Dirschl, Nöth & Wagner, 1984; Kumpfmüller, Nölle, Nöth, Pommerenning & Staudigl, 1985; Boese & Klingebiel, 1986). Since boron is capable of exchanging different amino moieties in aminoboranes, we began several years ago to prepare phosphorus(V) hydrazine heterocycles with boron in the ring starting from the corresponding dihydrazido derivatives of phosphoric acid and bis(dimethylamino)phenylborane (Scheffler, 1989; Engelhardt & Park, 1995). The transamination reaction may lead to equilibria involving several different compounds. In this context, we now have tested the reaction of methylhydrazine with bis(dimethylamino)phenylborane (see scheme below).



Of the most probable reaction products (I), (II) and (III), only (II) could be isolated from the hexane solution (yield 91% based on methylhydrazine) besides some insoluble material. The molecular peak at *m*/*e* = 264 is the strongest signal in the mass spectrum, the isotope pattern corresponding to two B atoms in the molecule. The constitution in solution ($CDCl_3$) is confirmed by the NMR spectral data (see *Experimental*). The X-ray structure analysis reveals the same constitution in the solid phase. The asymmetric unit contains one molecule (Fig. 1). The central inorganic five-membered ring is essentially planar (Table 2). The N1—N2 distance in the ring of 1.414 (5) Å is not exceptionally short and compares well with other values in non-planar hydrazine rings with Nsp^2 atoms (Engelhardt & Hartl, 1975, 1976). This is in contrast, however, to the longer exocyclic N3—N4 distance of 1.441 (4) Å, where N3 has a trigonal pyramidal coordination [the sum of the bond angles around N3 is 326 (4)°]. There are two rather short B—N distances [B2—N1 1.399 (6) and B3—N2 1.404 (6) Å] and two significantly longer ones [B2—N4 1.446 (6) and B3—N4 1.432 (6) Å]. Out of several resonance forms (Fig. 2), the aromatic form (*b*), analogous to pyrrole, is apparently the most important. The phenyl rings bonded to B2 and B3 are almost coplanar with and normal to the central inorganic ring, respectively [interplanar angles 5.0 (2) and 84.3 (2)°].

It is remarkable that only the five-membered ring product (II) is obtained in this study. In the case of the