1,6-Diazidodiamantane

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Key indicators

Single-crystal X-ray study
$T = 294$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.182
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecule of the title compound, $C_{14}H_{18}N_6$, lies on a crystallographic centre of inversion. The unique C—N—N and N—N—N angles are 115.8 (3) and 173.3 (4)°, respectively.

Comment

The title compound, (I), is a derivative of diamantane and the azido functionality is one of the precusor groups for the synthesis of nitrogen-containing organic compounds. For example, compound (I) can easily be transformed into 1,6-diamantandiamine under reduction conditions. As a member of the diamondoid family, (I) is a potentially valuable molecule in materials science.

(I)
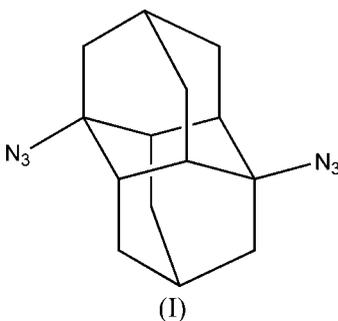
The molecular structure of (I) is shown in Fig. 1. The molecule lies on a centre of inversion and the unique C—N—N and N—N—N angles are 115.8 (3) and 173.3 (4)°, respectively. The steric effect of the attached azide groups appears to cause a distortion from the expected values (based on hybridization principles) of the N—C1—C2 and N1—C1—C5 bond angles of 106.0 (2) and 111.3 (2)°, respectively. The N1—N2, N2—N3 and N1—C1 bond lengths are 1.202 (2), 1.106 (2) and 1.464 (2) Å, respectively. We have recently published details of the crystal structure of 1-azidodiamantane (Yu *et al.*, 2006).

Experimental

The title compound was prepared according to the procedure of Surya Prakash *et al.* (1986). To a stirred solution of 1,6-dibromo-diamantane (2.0 g, 6.0 mmol) and trimethylsilyl azide (1.41 ml, 10.76 mmol) in dry dichloromethane (40 ml) under nitrogen at 273 K, stannic chloride (0.8 ml) was added. After the addition, the mixture was brought to room temperature, followed by prolonged stirring for 11 h. After the reaction was complete, the mixture was quenched with ice–water (50 ml), followed by dichloromethane extraction (3 × 30 ml). The organic layer was dried over anhydrous MgSO$_4$ and then evaporated to obtain the crude product. The crude product was further purified on a silica-gel column using petrolum ether–dichloromethane (30:1, v/v) as the eluant to give the title compound

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as a white solid (1.5 g, 96% yield). Suitable crystals were obtained by evaporation of a dichloromethane solution of (I) (m.p. 362 K).

Crystal data

$C_{14}H_{18}N_6$	$V = 308.3\ (7)\ \text{Å}^3$
$M_r = 270.33$	$Z = 1$
Triclinic, $P\bar{1}$	$D_x = 1.456\ \text{Mg m}^{-3}$
$a = 6.574\ (9)\ \text{Å}$	Mo $K\alpha$ radiation
$b = 7.036\ (9)\ \text{Å}$	$\mu = 0.09\ \text{mm}^{-1}$
$c = 7.478\ (10)\ \text{Å}$	$T = 294\ (2)\ \text{K}$
$\alpha = 76.36\ (2)°$	Block, colourless
$\beta = 67.78\ (2)°$	$0.22 \times 0.16 \times 0.14\ \text{mm}$
$\gamma = 77.75\ (2)°$	

Data collection

Bruker SMART CCD area-detector diffractometer	1555 measured reflections
φ and ω scans	1084 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	633 reflections with $I > 2\sigma(I)$
$T_{min} = 0.790$, $T_{max} = 0.990$	$R_{int} = 0.020$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$+ 0.0788P]$
$wR(F^2) = 0.182$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1084 reflections	$\Delta\rho_{max} = 0.20\ \text{e Å}^{-3}$
91 parameters	$\Delta\rho_{min} = -0.24\ \text{e Å}^{-3}$
H-atom parameters constrained	

All H atoms were initially located in a difference Fourier map. They were repositioned with ideal geometry (C—H = 0.97–0.98 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:
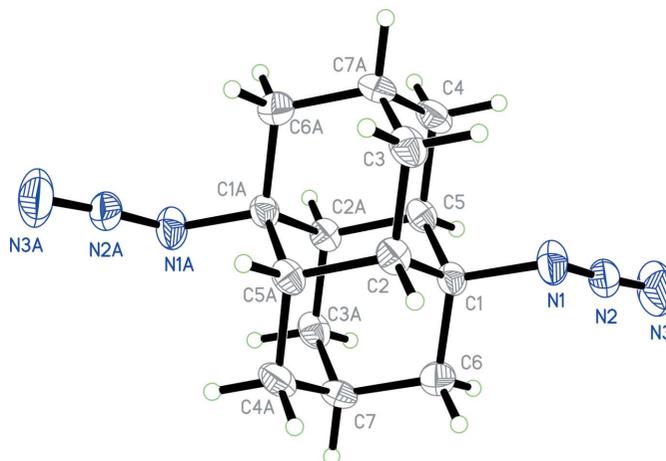
Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Atoms labelled with the suffix 'A' are related by the symmetry operator $(-x + 1, -y, -z + 1)$.

SHELXTL (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0), *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELX97*. University of Göttingen, Germany.

Surya Prakash, G. K., Stephenson, M. A., Shih, J. G. & Olah, G. A. (1986). *J. Org. Chem.* **51**, 3215–3217.

Yu, C.-Y., Su, Q.-Y., Li, Q. & Wang, L.-B. (2006). *Acta Cryst.* E**62** o3430–o3431.