

## 1-Azidodiamantane

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## Key indicators

Single-crystal X-ray study

T = 293 K

Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$ 

R factor = 0.045

wR factor = 0.121

Data-to-parameter ratio = 13.2

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

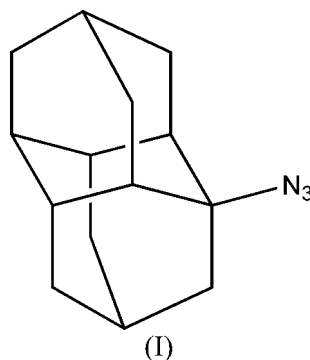
In the title structure,  $\text{C}_{14}\text{H}_{19}\text{N}_3$ , the C–N–N and N–N–N angles are 115.62 (17) and 173.3 (2)°, respectively.

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## Comment

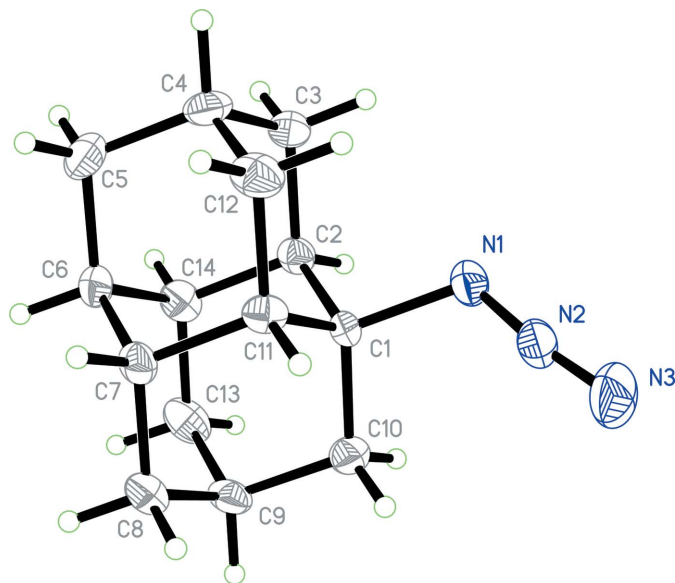
The title compound, (I), is a derivative of diamantane and the azido functionality is one of the precursor groups for the synthesis of nitrogen-containing organic compounds. For example, compound (I) can easily be transformed into 1-diamantanamine under reduction conditions. 1-Diamantanamine can be regarded as a structural analogue of amantadine (1-adamantanamine). The latter has been established as effective in the prophylaxis and treatment of influenza A virus infections. Furthermore, as a member of the diamondoid family, (I) is also a potentially valuable molecule in materials science.



The molecular structure of compound (I) is shown in Fig. 1. The C1–N1–N2 and N1–N2–N3 angles are 115.62 (17) and 173.3 (2)°, respectively. The steric effect of the attached azide group appears to cause a distortion from the expected value (based on hybridization principles) of the N–C1–C2 angle = 105.69 (15)°. The N1–N2, N2–N3 and N1–C1 bond lengths are 1.220 (2), 1.135 (2) and 1.508 (2) Å, respectively.

## Experimental

The title compound was prepared according to the literature procedure of Surya Prakash *et al.* (1986). To a stirred solution of 1-bromodiamantane (4.0 g, 15 mmol) and trimethylsilyl azide (2.96 ml, 22.5 mmol) in dry dichloromethane (40 ml), stannic chloride (2 ml) was added under nitrogen at 273 K. The temperature of the mixture was then raised 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  $\text{MgSO}_4$  and evaporated to obtain the crude product. The crude product was



**Figure 1**  
A view of the molecular structure of (I), showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

further purified on a silica-gel column using hexane as the eluant, to give the title compound as a white solid (3.1 g, 90% yield). Suitable crystals were obtained by evaporation of a dichloromethane solution of (I) (m.p. 387–388 K).

#### Crystal data

$C_{14}H_{19}N_3$   
 $M_r = 229.32$   
 Monoclinic,  $P2_1/c$   
 $a = 7.678$  (2) Å  
 $b = 12.575$  (4) Å  
 $c = 12.073$  (4) Å  
 $\beta = 96.063$  (6)°  
 $V = 1159.0$  (6) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.314$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism, colourless  
 $0.24 \times 0.16 \times 0.14$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1997)  
 $T_{\min} = 0.797$ ,  $T_{\max} = 0.994$

12765 measured reflections  
 2045 independent reflections  
 1018 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.053$   
 $\theta_{\text{max}} = 25.0^\circ$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.121$   
 $S = 1.06$   
 2045 reflections  
 155 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.28$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97 (Sheldrick, 1997)  
 Extinction coefficient: 0.046 (4)

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 in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

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

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