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#### Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
R factor = 0.056  
wR factor = 0.171  
Data-to-parameter ratio = 13.7

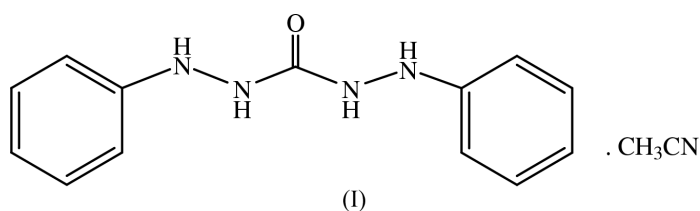
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Diphenylcarbazide acetonitrile solvate

In the crystal lattice of the title compound, 1,5-diphenylcarbonohydrazide acetonitrile solvate,  $\text{C}_{13}\text{H}_{14}\text{N}_4\text{O} \cdot \text{C}_2\text{H}_3\text{N}$ , the diphenylcarbazide molecules create a network structure through hydrogen bonds. The crystal structure is stabilized by  $\text{N}-\text{H} \cdots \text{N}$  and  $\text{N}-\text{H} \cdots \text{O}$  hydrogen bonds. The FT-IR spectra clearly show the presence of acetonitrile molecules in the crystal lattice.

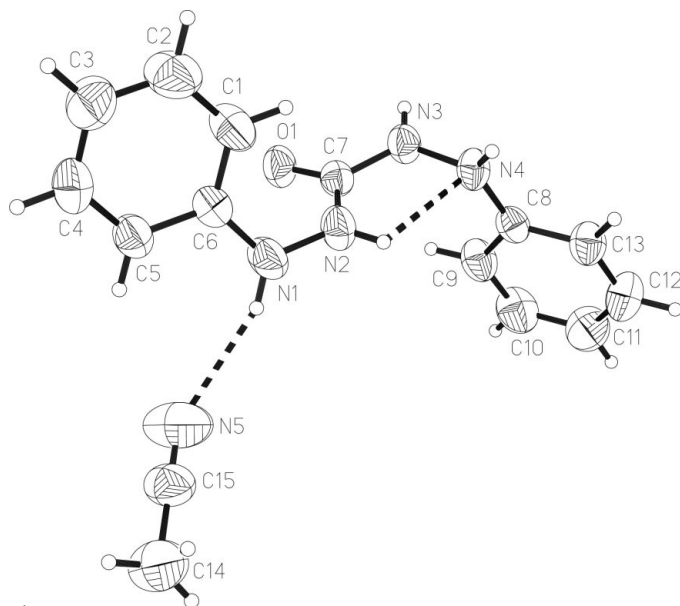
#### Comment

Diphenylcarbazide is an artificial electron-donor material frequently used in analytical chemistry for the calorimetric determination of chromium and as a sensitive reagent for metal ions (mercury and cadmium) (El-Kabbany *et al.*, 1997). It has various applications, especially in the fields of biophysics and microbiology. With the help of exogenous electron donors, diphenylcarbazide photo-inactivated sites on the electron-transfer chain were delineated (Verma & Singh, 1995). It is also used as an artificial donor during charge separation in photochemical reactions (Melis *et al.*, 1992) and in photosynthetic electron transport (Prasad *et al.*, 1991; Sundari & Raghavendra, 1990; Mishra *et al.*, 1993). The crystal structure of diphenylcarbazide ( $\text{C}_{13}\text{H}_{14}\text{N}_4\text{O}$ ) was determined (De Ranter *et al.*, 1979) and found to be orthorhombic with space group *Pbnm*. In this paper, we report the crystal structure of diphenylcarbazide acetonitrile solvate, (I).



A displacement ellipsoid plot with the atom-numbering scheme is shown in Fig. 1. The bonds and angles observed in this structure are normal and in agreement with those of related structures reported earlier (De Ranter *et al.*, 1979; Burke-Laing & Laing, 1976; Blaton *et al.*, 1979). The  $\text{C}15 \equiv \text{N}5$  triple bond is in quite good agreement with the average value of  $1.136(10) \text{ \AA}$  given by Allen *et al.* (1987). The torsion angles defining the conformation of the central part of the host molecule are  $68.3(3)$  and  $93.3(2)^\circ$ , respectively, for  $\text{C}6-\text{N}1-\text{N}2-\text{C}7$  and  $\text{C}7-\text{N}3-\text{N}4-\text{C}8$ . The dihedral angle between the two planes of the phenyl rings is  $52.3(1)^\circ$ ; these two rings make angles of  $79.5(1)$  and  $85.4(1)^\circ$  with the central ureylene moiety ( $\text{N}2/\text{C}7/\text{O}1/\text{N}3$ ). It is also noted that not only the central ureylene moiety is planar, but  $\text{N}1$  and  $\text{N}4$  lie in its

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**Figure 1**  
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

plane. The acetonitrile molecule also lies almost in the plane of the central ureylene group with a dihedral angle of  $14.9(2)^\circ$ .

In the crystal lattice, the molecules create a network structure through hydrogen bonds. There are four intermolecular hydrogen bonds which form the hydrogen-bond network. The acetonitrile molecule acts as an acceptor connecting two diphenylcarbazide molecules. The O atom of the ureylene group is involved in two  $O \cdots H-N$  hydrogen bonds with neighboring molecules. Finally, there is an intramolecular  $N2-H2A \cdots N4$  hydrogen bond.

Comparing the structure of (I) with the crystal structure of diphenylcarbazide reported by De Ranter *et al.* (1979), some differences are noted. Firstly, the phenylhydrazine groups are not crystallographically equivalent to each other; secondly, there are no hexagonal units linked into a herring-bone structure; thirdly, there is no marked bond delocalization in the hydrazo chain ( $C6-N1-N2-C7$  and  $C7-N3-N4-C8$ ), for  $C6-N1$  and  $C8-N4$  are indicative of considerable partly single-bond character. Because of the effect of the acetonitrile molecule, the  $N1-N2$ ,  $N1-C6$  and  $N2-C7$  bond lengths are shorter than those of  $N3-N4$ ,  $N4-C8$  and  $N3-C7$ , respectively. The FT-IR spectra clearly show a strong peak at  $2251\text{ cm}^{-1}$ , which was tentatively assigned as the  $\nu(C\equiv N)$  stretching vibration of the acetonitrile molecules in the crystal lattice.

## Experimental

The title compound was prepared by refluxing an acetonitrile solution of diphenylcarbazide (0.484 g, 2 mmol) and  $PdCl_2$  (0.1 mg). The deep-red solution was filtered and the filtrate was left to stand undisturbed. Single crystals suitable for X-ray analysis were obtained by slow evaporation at 338–340 K from the acetonitrile solvent.

## Crystal data

$C_{13}H_{14}N_4O \cdot C_2H_3N$   
 $M_r = 283.34$   
 Monoclinic,  $P2_1/n$   
 $a = 5.7818(2)\text{ \AA}$   
 $b = 15.320(1)\text{ \AA}$   
 $c = 17.469(1)\text{ \AA}$   
 $\beta = 97.476(1)^\circ$   
 $V = 1534.20(14)\text{ \AA}^3$   
 $Z = 4$

$D_x = 1.227\text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 4025 reflections  
 $\theta = 1.8\text{--}24.8^\circ$   
 $\mu = 0.08\text{ mm}^{-1}$   
 $T = 293(2)\text{ K}$   
 Parallelepiped, light yellow  
 $0.48 \times 0.34 \times 0.18\text{ mm}$

## Data collection

Siemens SMART CCD area-detector  
 $\omega$  scans  
 8214 measured reflections  
 2624 independent reflections  
 1756 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.056$   
 $\theta_{\text{max}} = 24.7^\circ$   
 $h = -6 \rightarrow 6$   
 $k = -18 \rightarrow 17$   
 $l = -14 \rightarrow 20$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.171$   
 $S = 1.01$   
 2624 reflections  
 191 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1025P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.37\text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.47\text{ e \AA}^{-3}$   
 Extinction correction: *SHELXTL*  
 Extinction coefficient: 0.019 (5)

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—C7	1.235 (2)	N3—C7	1.361 (3)
N1—N2	1.386 (3)	N3—N4	1.392 (2)
N1—C6	1.394 (3)	N4—C8	1.414 (3)
N2—C7	1.345 (3)	N5—C15	1.124 (4)
N2—N1—C6	119.8 (2)	C7—N3—N4	119.3 (2)
C7—N2—N1	121.1 (2)		

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A $\cdots$ N5	0.86	2.39	3.210 (4)	159
N2—H2A $\cdots$ N5 <sup>i</sup>	0.86	2.42	3.226 (4)	155
N3—H3A $\cdots$ O1 <sup>ii</sup>	0.86	2.14	2.933 (2)	154
N4—H4A $\cdots$ O1 <sup>i</sup>	0.86	2.53	3.035 (2)	118
N2—H2A $\cdots$ N4	0.86	2.28	2.645 (3)	106

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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