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1-(3-Phenylpropyl)urea

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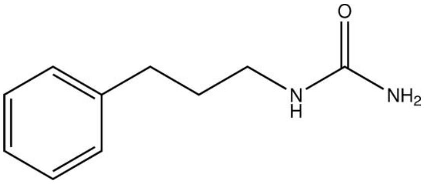
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.144; data-to-parameter ratio = 17.4.

In the crystal of the title compound, $C_{10}H_{14}N_2O$, double supramolecular layers of $PhCH_2CH_2CH_2NHC(O)NH_2$ are formed parallel to the *bc* plane by intermolecular N—H···O hydrogen bonding, with $R_2^2(8)$ and $R_2^1(6)$ motifs in the *b*- and *c*-axis directions, respectively. The mean plane of the C_{ar}—C—C group makes a dihedral angle of 84.8 (2)° with the benzene ring.

Related literature

For related structural information see Bernstein *et al.* (1995). For background chemistry, see: Gray *et al.* (2005); Hua & Woollins (2009); Renodon-Cornière *et al.* (2002).



Experimental

Crystal data

$C_{10}H_{14}N_2O$
M_r = 178.23
Monoclinic, $P2_1/c$
a = 17.002 (4) Å
b = 6.4953 (15) Å
c = 9.171 (2) Å
β = 91.401 (8)°
V = 1012.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 93 K
0.25 × 0.04 × 0.03 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2004)
T_{min} = 0.981, T_{max} = 0.998
6696 measured reflections
2126 independent reflections
1360 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.144
S = 1.03
2126 reflections
122 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.88	2.10	2.936 (2)	159
N1—H1*B*···O1^{ii}	0.88	2.09	2.8788 (19)	148
N2—H2···O1^{ii}	0.863 (18)	2.127 (19)	2.9240 (19)	153.2 (17)

Symmetry codes: (i) $-x+1, -y+3, -z+1$; (ii) $x, -y+\frac{5}{2}, z-\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5070).

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supplementary materials

Acta Cryst. (2009). E**65**, o2556 [**doi:10.1107/S1600536809038501**]

1-(3-Phenylpropyl)urea

Y. Li, G. Hua, A. M. Z. Slawin and J. D. Woollins

Comment

2,4-Bis(phenyl)-1,3-diselenadiphosphetane-2,4-diselenide [PhP(Se)(μ-Se)]$_2$, which is known as Woollins reagent, **WR**, has received increasing attention due to its relatively unpleasant chemical properties and since it can be prepared readily and safely handled (Gray *et al.* 2005). Now it is becoming a very useful selenium source or reagent in synthetic chemistry (Hua *et al.* 2009; Hua & Woollins, 2009). We report here the synthesis and X-ray structure of $C_6H_4(CH_2)_3NHCONH_2$. The title compound was prepared by the reaction of Woollins' reagent with 3-phenylpropan-1-amine.

Double supramolecular layers of the title compound (Fig. 2), $PhCH_2CH_2CH_2NHC(O)NH_2$, are formed parallel to the *bc* plane by intermolecular N—H···O hydrogen bonding (see Table 1), with $R_2^2(8)$ and $R_2^1(6)$ motifs in approximately b and c directions respectively (Bernstein *et al.*, 1995). The mean plane of the C3—C5 group makes dihedral angle of 84.8 (2)° with the benzene ring.

Experimental

A toluene suspension of Woollins reagent (0.54 g, 1.0 mmol) and $Ph(CH_2)_3NHCN$ (0.29 g, 2.0 mmol), which were prepared from cyanogen bromide with primary or secondary amine in dry methanol in the presence of excess of anhydrous CH_3COONa at room temperature in almost quantitative yield (Renodon-Cornière *et al.* 2002), was refluxed for 4 h under nitrogen during which time the mixtures became a clear reddish brown solution. After cooling and addition of water (1 cm^3) the reflux was continued for another 1 h. The solvent was removed in vacuum, and the residue was purified by column chromatography (silica gel, 9:1 dichloromethane/ethyl acetate as eluant) to afford the title compound in the yield of 95%. The single crystals of the title compound for the X-ray crystallographic analysis were obtained by recrystallization from dichloromethane/hexane as colorless blocks.

Refinement

All H atoms except H2 (which was freely refined) were included in calculated positions (C—H distances are 0.98 Å methyl H atoms, 0.99 Å for methylene H atoms and 0.95 Å for aryl H atoms, N—H 0.88 Å) and were refined as riding atoms with $U_{iso}(H) = 1.2\ U_{eq}$ (parent atom, amino, methylene and aryl H atoms) or $U_{iso}(H) = 1.5\ U_{eq}$ (parent atom, methyl H atoms).

Figures

Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

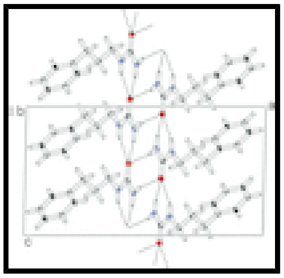

Fig. 2. View along the b direction of the crystal packing of the title compound with hydrogen bonding shown as dashed lines.

1-(3-Phenylpropyl)urea

Crystal data

$C_{10}H_{14}N_2O$

$M_r = 178.23$

Monoclinic, $P2_1/c$

$a = 17.002$ (4) Å

$b = 6.4953$ (15) Å

$c = 9.171$ (2) Å

$\beta = 91.401$ (8)°

$V = 1012.5$ (4) Å^3

$Z = 4$

$F_{000} = 384$

$D_x = 1.169$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2697 reflections

$\theta = 2.4$–28.1°

$\mu = 0.08$ mm^{-1}

$T = 93$ K

Platelet, colorless

0.25 × 0.04 × 0.03 mm

Data collection

Rigaku Mercury CCD diffractometer

Radiation source: rotating anode

Monochromator: confocal

Detector resolution: 0.83 pixels mm^{-1}

$T = 93$ K

ω scans

Absorption correction: Multi-scan (CrystalClear; Rigaku, 2004)

$T_{\min} = 0.981$, $T_{\max} = 0.998$

6696 measured reflections

2126 independent reflections

1360 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 28.5$°

$\theta_{\min} = 3.4$°

$h = -16 \rightarrow 20$

$k = -8 \rightarrow 7$

$l = -9 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.144$

$S = 1.03$

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.23$ e Å^{-3}

2126 reflections	$\Delta\rho_{\min} = -0.20$ e Å^{-3}
122 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.030 (5)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.56839 (7)	1.2854 (2)	0.55596 (11)	0.0367 (4)
N2	0.61033 (9)	1.1114 (2)	0.35832 (16)	0.0321 (4)
C5	0.81085 (11)	0.5672 (3)	0.32854 (16)	0.0325 (5)
N1	0.54532 (10)	1.4186 (3)	0.33030 (14)	0.0401 (5)
H1A	0.5219	1.5274	0.3665	0.048*
H1B	0.5499	1.4060	0.2353	0.048*
C2	0.64786 (11)	0.9469 (3)	0.44128 (17)	0.0342 (5)
H2A	0.6075	0.8490	0.4749	0.041*
H2B	0.6756	1.0048	0.5282	0.041*
C10	0.79059 (12)	0.3888 (3)	0.25485 (18)	0.0396 (5)
H10	0.7413	0.3258	0.2721	0.047*
C6	0.88327 (12)	0.6552 (3)	0.3003 (2)	0.0451 (6)
H6	0.8987	0.7777	0.3497	0.054*
C3	0.70581 (11)	0.8358 (3)	0.34725 (18)	0.0338 (5)
H3A	0.7425	0.9378	0.3063	0.041*
H3B	0.6768	0.7703	0.2646	0.041*
C4	0.75349 (11)	0.6713 (3)	0.42853 (18)	0.0354 (5)
H4A	0.7828	0.7353	0.5114	0.042*
H4B	0.7174	0.5671	0.4684	0.042*
C9	0.84088 (13)	0.3000 (3)	0.1561 (2)	0.0457 (6)
H9	0.8259	0.1765	0.1074	0.055*
C8	0.91183 (13)	0.3890 (4)	0.1284 (2)	0.0475 (6)
H8	0.9459	0.3288	0.0598	0.057*
C7	0.93348 (13)	0.5670 (4)	0.2009 (2)	0.0533 (6)
H7	0.9828	0.6294	0.1828	0.064*
C1	0.57407 (10)	1.2717 (3)	0.42002 (17)	0.0305 (5)
H2	0.6095 (11)	1.110 (3)	0.264 (2)	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0480 (9)	0.0409 (8)	0.0211 (7)	0.0078 (6)	0.0029 (5)	−0.0016 (5)
N2	0.0408 (10)	0.0356 (9)	0.0199 (8)	0.0082 (7)	0.0011 (6)	−0.0016 (6)
C5	0.0355 (11)	0.0340 (11)	0.0280 (9)	0.0034 (8)	0.0013 (7)	0.0026 (7)
N1	0.0575 (12)	0.0397 (10)	0.0232 (8)	0.0145 (8)	0.0041 (7)	−0.0004 (6)
C2	0.0405 (12)	0.0357 (11)	0.0264 (9)	0.0050 (8)	0.0023 (7)	0.0009 (7)
C10	0.0426 (13)	0.0348 (11)	0.0415 (11)	−0.0006 (9)	0.0055 (8)	−0.0006 (8)
C6	0.0402 (13)	0.0472 (13)	0.0480 (12)	−0.0050 (10)	0.0030 (9)	−0.0113 (9)
C3	0.0385 (12)	0.0351 (11)	0.0281 (10)	0.0043 (8)	0.0043 (7)	−0.0005 (7)
C4	0.0397 (12)	0.0363 (11)	0.0303 (10)	0.0037 (8)	0.0008 (8)	0.0013 (7)
C9	0.0541 (14)	0.0392 (12)	0.0440 (12)	0.0029 (10)	0.0029 (9)	−0.0079 (9)
C8	0.0447 (14)	0.0572 (15)	0.0408 (12)	0.0130 (11)	0.0071 (9)	−0.0058 (10)
C7	0.0388 (13)	0.0659 (16)	0.0557 (13)	−0.0033 (11)	0.0092 (10)	−0.0079 (11)
C1	0.0327 (11)	0.0361 (11)	0.0228 (10)	0.0010 (8)	0.0022 (7)	0.0001 (7)

Geometric parameters (Å, °)

O1—C1	1.2559 (18)	C10—H10	0.9500
N2—C1	1.342 (2)	C6—C7	1.387 (3)
N2—C2	1.450 (2)	C6—H6	0.9500
N2—H2	0.863 (18)	C3—C4	1.525 (2)
C5—C10	1.380 (3)	C3—H3A	0.9900
C5—C6	1.388 (3)	C3—H3B	0.9900
C5—C4	1.514 (3)	C4—H4A	0.9900
N1—C1	1.344 (2)	C4—H4B	0.9900
N1—H1A	0.8800	C9—C8	1.367 (3)
N1—H1B	0.8800	C9—H9	0.9500
C2—C3	1.509 (2)	C8—C7	1.379 (3)
C2—H2A	0.9900	C8—H8	0.9500
C2—H2B	0.9900	C7—H7	0.9500
C10—C9	1.386 (3)		

C1—N2—C2	123.43 (14)	C4—C3—H3A	108.8
C1—N2—H2	115.5 (12)	C2—C3—H3B	108.8
C2—N2—H2	121.1 (12)	C4—C3—H3B	108.8
C10—C5—C6	117.78 (17)	H3A—C3—H3B	107.7
C10—C5—C4	120.97 (17)	C5—C4—C3	111.07 (14)
C6—C5—C4	121.11 (17)	C5—C4—H4A	109.4
C1—N1—H1A	120.0	C3—C4—H4A	109.4
C1—N1—H1B	120.0	C5—C4—H4B	109.4
H1A—N1—H1B	120.0	C3—C4—H4B	109.4
N2—C2—C3	109.73 (13)	H4A—C4—H4B	108.0
N2—C2—H2A	109.7	C8—C9—C10	120.42 (19)
C3—C2—H2A	109.7	C8—C9—H9	119.8
N2—C2—H2B	109.7	C10—C9—H9	119.8
C3—C2—H2B	109.7	C9—C8—C7	119.43 (18)

H2A—C2—H2B	108.2	C9—C8—H8	120.3
C5—C10—C9	121.23 (19)	C7—C8—H8	120.3
C5—C10—H10	119.4	C8—C7—C6	120.1 (2)
C9—C10—H10	119.4	C8—C7—H7	120.0
C7—C6—C5	121.1 (2)	C6—C7—H7	120.0
C7—C6—H6	119.5	O1—C1—N2	121.38 (15)
C5—C6—H6	119.5	O1—C1—N1	121.44 (16)
C2—C3—C4	113.74 (14)	N2—C1—N1	117.17 (14)
C2—C3—H3A	108.8		

C1—N2—C2—C3	−160.22 (17)	C2—C3—C4—C5	−179.52 (16)
C6—C5—C10—C9	0.2 (3)	C5—C10—C9—C8	−0.6 (3)
C4—C5—C10—C9	175.84 (17)	C10—C9—C8—C7	0.8 (3)
C10—C5—C6—C7	0.2 (3)	C9—C8—C7—C6	−0.4 (3)
C4—C5—C6—C7	−175.50 (18)	C5—C6—C7—C8	0.0 (3)
N2—C2—C3—C4	175.12 (15)	C2—N2—C1—O1	−2.2 (3)
C10—C5—C4—C3	−92.9 (2)	C2—N2—C1—N1	176.63 (16)
C6—C5—C4—C3	82.7 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{i}	0.88	2.10	2.936 (2)	159
N1—H1B···O1^{ii}	0.88	2.09	2.8788 (19)	148
N2—H2···O1^{ii}	0.863 (18)	2.127 (19)	2.9240 (19)	153.2 (17)

Symmetry codes: (i) −*x*+1, −*y*+3, −*z*+1; (ii) *x*, −*y*+5/2, *z*−1/2.

Fig. 1

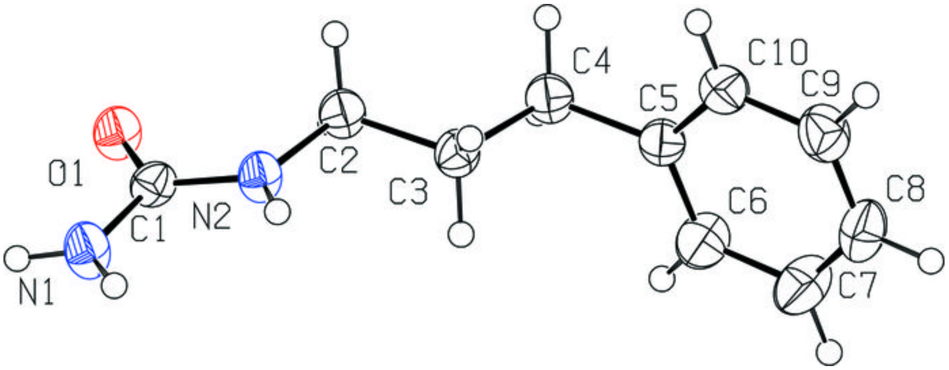

Fig. 2