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2-Phenylmalonamide

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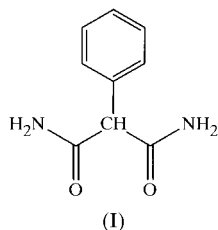
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In the title compound, C₉H₁₀N₂O₂, the amide groups are rotated out of the C(OH₂)—C(HPh)—C(OH₂) plane by *ca* 25–47° and the phenyl ring is almost perpendicular to this plane. The crystal structure is stabilized by intra- and intermolecular N—H···O hydrogen bonds.

Comment

The malonamides have the stronger extracting capacity than other diamides, which is due to the larger electron delocalization and the higher chelating abilities of malonamides (Siddall & Good, 1967). The title malonamide derivative, (I), had been synthesized with the aim of studying its lipophilic suitability as an extractant.



In the crystal, the amide N atoms are involved in intra- and intermolecular hydrogen bonds (Table 2). These hydrogen bonds form eight-membered [*R*₂²(8)] and 16-membered [*R*₈⁰(16)] hydrogen-bonded rings. These rings link the molecules into an infinite three-dimensional network.

Experimental

A suspension of diethyl phenylmalonate and NH₃ solution was refluxed for 1 week with vigorous stirring. Crystals of (I) were obtained by slow evaporation of an ethanol solution.

Crystal data

C₉H₁₀N₂O₂
M_r = 178.19
 Monoclinic, *P*2₁/*n*
a = 9.079 (2) Å
b = 7.555 (2) Å
c = 13.015 (2) Å
 β = 104.10 (1)°
V = 865.8 (3) Å³
Z = 4

D_x = 1.367 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 14.6–15.0°
 μ = 0.099 mm⁻¹
T = 296.2 K
 Prismatic, colorless
 0.20 × 0.20 × 0.10 mm

Data collection

Rigaku AFC-5R diffractometer
 ω -2 θ scans
 2275 measured reflections
 1994 independent reflections
 1347 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.016
 θ _{max} = 27.5°

h = 0 → 11
k = -9 → 0
l = -16 → 16
 3 standard reflections every 150 reflections
 intensity decay: 2%

Refinement

Refinement on *F*²
R(*F*) = 0.038
wR(*F*²) = 0.104
S = 1.23
 1658 reflections
 158 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + \{0.050[\text{Max}(F_o^2, 0) + 2F_c^2]/3\}^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.0039$
 $\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.230 (2)	C1—C2	1.535 (2)
O2—C3	1.237 (2)	C2—C3	1.528 (2)
N1—C1	1.321 (2)	C2—C4	1.530 (2)
N2—C3	1.324 (2)		
C1—C2—C3	115.0 (1)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H6···O2	0.87 (3)	2.03 (3)	2.723 (2)	136 (2)
N1—H7···O1 ⁱ	0.85 (3)	2.14 (3)	2.978 (2)	167 (2)
N2—H8···O2 ⁱⁱ	0.91 (2)	2.13 (2)	3.027 (2)	168 (2)
N2—H9···O1 ⁱⁱⁱ	0.95 (2)	2.04 (2)	2.9753 (19)	167.1 (15)

Symmetry codes: (i) -1 - *x*, -*y*, -*z*; (ii) -*x*, -1 - *y*, -*z*; (iii) -½ - *x*, *y* - ½, ½ - *z*.

All the H atoms were located from the difference Fourier map and were refined isotropically. The C—H range is 0.94 (2)–1.02 (2) Å and the N—H range is 0.85 (2)–0.94 (2) Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

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