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2-Amino-7,7-dimethyl-5-oxo-4-phenyl-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 13.4

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

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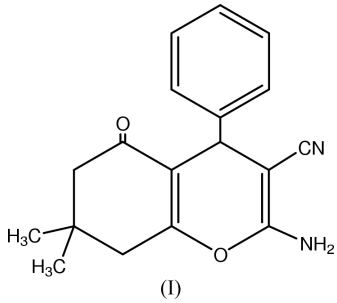
In the molecule of the title compound, $C_{18}H_{18}N_2O_2$, the C7/C8/C9/O1/C10/C11 plane forms an angle of 92.5 (4)° with the phenyl plane. The most striking feature of the title compound is the formation of a three-dimensional network through N—H···N and N—H···O hydrogen bonds.

Comment

Polyfunctionalized benzo-4*H*-pyrans are structural units of a number of natural products (Hatakeyama *et al.*, 1988) and, because of their inherent reactivity, the pyran rings are versatile synthons (Singh *et al.*, 1996). On the other hand, substituted benzo-4*H*-pyrans possess varied biological activity (Hassanien *et al.*, 1999). Since the stereochemistry is so important in the rational design of new functional molecules, we report herein the crystal structure of the title compound, (I).



(I)

In the structure (Fig. 1) of the neutral molecule (I), the C7/C8/C9/O1/C10/C11 plane forms an angle of 92.5 (4)° with the phenyl plane. The nitrile group is typical [N≡C 1.1448 (19) Å].

A notable feature of (I) is the formation of a three-dimensional network through hydrogen bonds, as shown in Fig. 2. These are in the normal range of weak interactions (Sasada, 1984). The amino N2 atom of one molecule links through H2*B* to the nitrile N1 atom of another molecule, creating a dimer. The amino N2 atom of one molecule also links through H2*A* to the keto O2 atom of another molecule to form the three-dimensional framework.

Experimental

The title compound was prepared by the reaction of 2-cyanocinnamonitrile with 5,5′-dimethyl-1,3-cyclohexanedione in ethylene under reflux for 4 h. The colorless solid was purified by recrystallization from ethanol to obtain single crystals suitable for X-ray diffraction. The product was characterized by NMR, IR and elemental analyses, giving results consistent with those in the literature (Singh *et al.*, 1996).

Crystal data

$C_{18}H_{18}N_2O_2$
M_r = 294.34
Monoclinic, $P2_1/c$
a = 11.307 (1) Å
b = 9.475 (1) Å
c = 14.919 (2) Å
β = 99.34 (1)°
V = 1577.1 (3) Å^3
Z = 4
D_x = 1.240 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 3.3–15.1°
μ = 0.08 mm^{-1}
T = 296 (2) K
Prism, colorless
0.56 × 0.50 × 0.40 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
3252 measured reflections
2783 independent reflections
1942 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 25.0°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 11$
$l = -17 \rightarrow 17$
3 standard reflections
every 97 reflections
intensity decay: 2.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.02
2783 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.044 (3)

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.3746 (17)	N2—C9	1.335 (2)
O1—C10	1.3767 (17)	C8—C9	1.351 (2)
O2—C12	1.2219 (18)	C10—C11	1.333 (2)
N1—C16	1.1448 (19)		
C9—O1—C10	118.41 (11)	O2—C12—C11	121.14 (15)
N2—C9—C8	129.01 (15)	O2—C12—C13	121.42 (15)
N2—C9—O1	109.64 (14)	N1—C16—C8	178.11 (17)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···N1^{i}	0.875 (17)	2.156 (18)	3.021 (2)	169.8 (15)
N2—H2A···O2^{ii}	0.88 (2)	2.13 (2)	3.006 (2)	171.6 (18)

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

The H atoms of the amino group were located in difference Fourier syntheses. The other H atoms were located by geometry and included in the structure-factor calculations.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976).

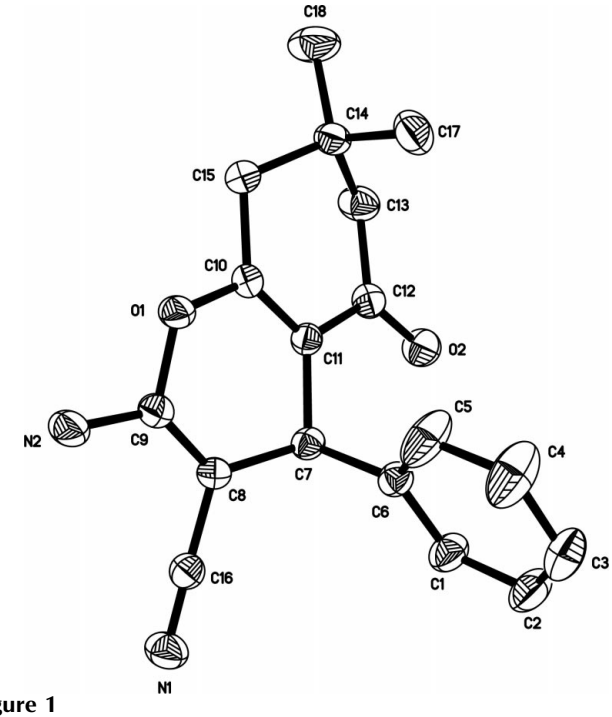


Figure 1
*ORTEP*II view (Johnson, 1976) of the title complex with displacement ellipsoids at the 30% probability level

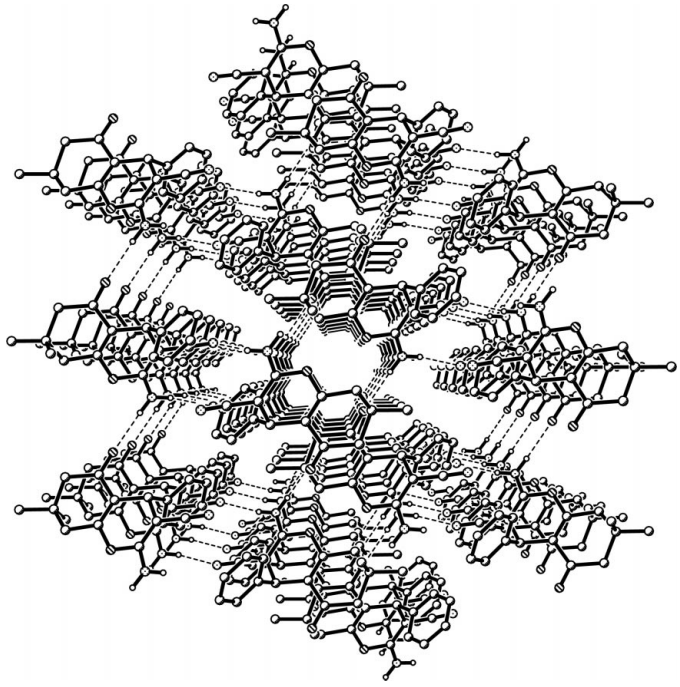


Figure 2
The molecular packing diagram of the title complex.

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