

4-(2-Furylmethylene)-2-(4-morpholino)-1-phenyl-1H-imidazol-4(5H)-one

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Key indicators

Single-crystal X-ray study

T = 292 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.044

wR factor = 0.099

Data-to-parameter ratio = 9.1

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

In the title compound, $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}_3$, the packing of the molecules in the crystal structure is mainly due to π - π and intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.

Comment

Imidazolinone derivatives are of great importance because of their remarkable biological properties (Kiec-Kononowicz *et al.*, 1998). In recent years, we have been engaged in the preparation of heterocyclic derivatives *via* the aza-Wittig reaction (Hu *et al.*, 2004). The title compound, (I), may be used as a new precursor for obtaining bioactive molecules. The crystal structure of (I) is presented here.

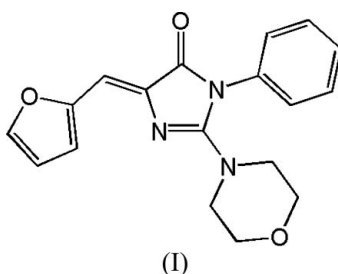


Fig. 1 shows the molecular structure of (I), with the atomic numbering scheme. In the crystal structure, there are some weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions (Table 1, Fig. 2) and intermolecular π - π interactions in which the centroid-to-centroid distance is 3.759 (17) Å .

Experimental

To a solution of iminophosphorane (1.32 g, 3 mmol) in dry dichloromethane (15 ml) was added phenyl isocyanate (3 mmol) under nitrogen at room temperature. After the reaction mixture had been allowed to stand for 10 h at room temperature, the solvent was removed under reduced pressure and diethyl ether/petroleum ether (1:2, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solvent was removed to give the carbodiimide, (II), which was used directly without further purification. To a solution of (II) in dichloromethane (15 ml) was added morpholine (3 mmol). After the reaction mixture had been allowed to stand for 6 h, the solution was concentrated under reduced pressure and the residue was recrystallized from dichloromethane/petroleum ether (1:4) to give the title compound, (I), in a yield of 85% (m.p. 491 K). Suitable crystals were obtained by vapour diffusion of ethanol and dichloromethane at room temperature. Spectroscopic analysis: ^1H NMR (CDCl_3 , 200 MHz) 3.33 (*t*, 4H, NCH_2 , $J = 4.4$ Hz), 3.64 (*t*, 4H, OCH_2 , $J = 4.9$ Hz), 6.80 (*s*, 1H, $=\text{CH}$), 6.51–7.49 (*m*, 8H, Ar–H). MS (EI, 70 eV) m/z (%): 323 (M^+ , 100), 266 (17), 189 (30), 77 (77). Elemental analysis: calculated for $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}_3$: C 66.86, H 5.30, N 13.00%; found: C 66.71, H 5.42, N 13.05%.

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Crystal data

$C_{18}H_{17}N_3O_3$
 $M_r = 323.35$
 Monoclinic, $P2_1$
 $a = 5.5926$ (12) Å
 $b = 8.8449$ (19) Å
 $c = 16.442$ (4) Å
 $\beta = 97.536$ (3)°
 $V = 806.3$ (3) Å³
 $Z = 2$

$D_x = 1.332$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2460 reflections
 $\theta = 2.5$ – 24.9 °
 $\mu = 0.09$ mm⁻¹
 $T = 292$ (2) K
 Block, yellow
 $0.20 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 5883 measured reflections
 1970 independent reflections

1705 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.082$
 $\theta_{max} = 27.5$ °
 $h = -7 \rightarrow 7$
 $k = -11 \rightarrow 11$
 $l = -20 \rightarrow 21$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.099$
 $S = 1.00$
 1970 reflections
 217 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.051P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.16$ e Å⁻³
 $\Delta\rho_{min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C18-H18B\cdots O2^i$	0.97	2.55	3.345 (4)	139
$C15-H15A\cdots N1^{ii}$	0.97	2.55	3.374 (4)	143
$C5-H5\cdots O3^{iii}$	0.93	2.39	3.303 (3)	166

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

H atoms were placed in calculated positions and treated as riding atoms (C–H in the range 0.93–0.98 Å), with $U_{iso}(H) = 1.2$ (CH) or 1.5 (CH₃) times U_{eq} (parent atom). In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 2001); software used to prepare material for publication: SHELXTL.

References

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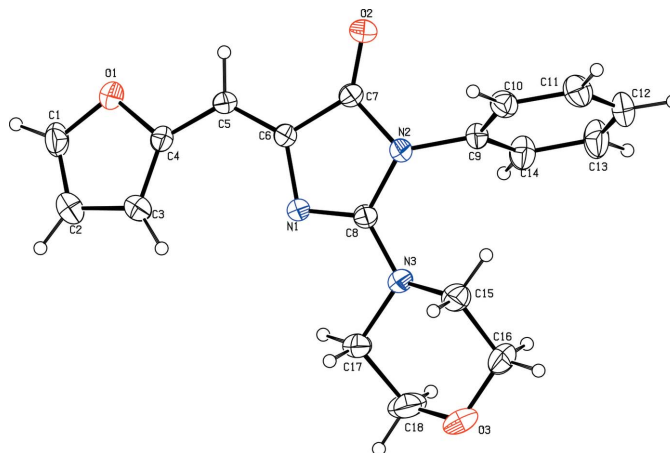


Figure 1

View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

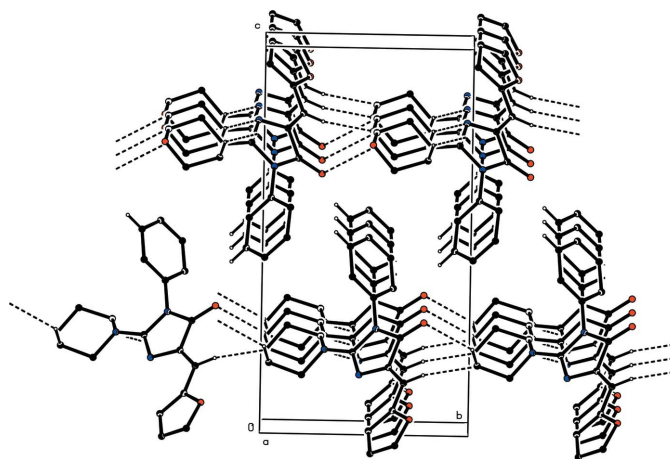


Figure 2

Packing diagram of (I), showing hydrogen-bonding associations (dashed lines) and the π – π stacking interactions. H atoms have been omitted.

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