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4-(3,4-Dimethoxyphenyl)-2-oxo-*N*,3-bis(3,4,5-trimethoxyphenyl)-2,3-dihydro-1*H*-imidazoline-1-carboxamide

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The structure of the title compound, $C_{30}H_{33}N_3O_{10}$, consists of an almost planar imidazolin-2-one system connecting two planar substituted phenyl groups and a roughly planar 3,4,5-trimethoxyanilinocarbonyl fragment which forms a dihedral angle of 95.17 (9)° with the heterocyclic ring.

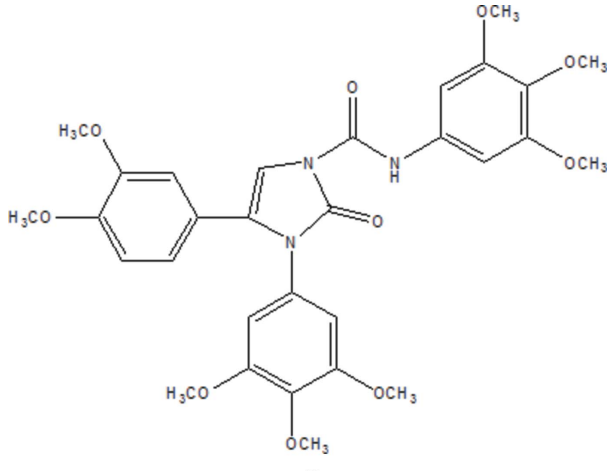
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.057
wR factor = 0.131
Data-to-parameter ratio = 15.3

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

Comment

Imidazolinones, in particular aryl-substituted imidazolin-2-ones, have become attractive targets for organic synthesis because of their broad spectrum of biological activities, being reported as antioxidants (Smith *et al.*, 1987), ATP-sensitive potassium channel openers (Gadwood *et al.*, 1995) and anti-inflammatory phosphodiesterase PDE4 inhibitors (Andrés *et al.*, 2002). Additionally, they are widely used in combinatorial chemistry in the search for pharmacologically active agents (Durant, 1985). In order to study their potential biological activity in other areas, a series of imidazolin-2-ones has been synthesized and evaluated for antitumour activity *in vitro* against a human tumour cell line (leukaemia HL-60). The title compound, (I), has a low micromolar IC50 of 6.5 μmol l^{-1} against leukaemia HL-60. To confirm its conformation, an X-ray crystallographic study of (I) has been carried out and the results are presented here (Fig. 1).



(I)

The roughly coplanar arrangement of the imidazolin-2-one and carboxamide groups may be further stabilized by an intramolecular N1—H1···O2 contact (H1···O2 = 1.97 Å and N1—H1···O2 = 142°).

Experimental

3,4,5-Trimethoxyaniline (3 mmol) in dry toluene (4 ml) was added dropwise to a stirred solution of $(COCl_2)_3$ (2.91 mmol) in dry toluene (4 ml) and cooled in an ice bath. After stirring for 1 h at room temperature and refluxing for 4 h, 3,4-dimethoxy-ω-amino-acetophenone (3 mmol) was added to the mixture directly and it was then heated under reflux overnight. After removal of the toluene, the residue was extracted with dichloromethane and dried over anhydrous sodium sulfate. After removing the solvent *in vacuo*, the residue was purified by column chromatography (silica gel, trichloromethane–petroleum ether 1:1) to afford the title compound. Crystals of (I) suitable for crystallographic study were obtained by slow crystallization from ethyl acetate at room temperature.

Crystal data

$C_{30}H_{33}N_3O_{10}$
M_r = 595.60
Triclinic, $P\overline{1}$
a = 9.429 (4) Å
b = 11.209 (4) Å
c = 15.890 (7) Å
α = 93.802 (15)°
β = 104.333 (16)°
γ = 112.476 (14)°
V = 1479.1 (10) Å^3
Z = 2
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11137 reflections
θ = 3.0–27.5°
μ = 0.10 mm^{-1}
T = 296 (1) K
Platelet, colourless
0.33 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.961, T_{max} = 0.990
14714 measured reflections
6711 independent reflections
4056 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.027
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 13$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.131
S = 1.01
6711 reflections
440 parameters
H-atom parameters constrained
$w = 1/[0.0002F_o^2 + 3\sigma(F_o^2) + 0.5]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 3.9 (3) × 10^2

All H atoms were positioned geometrically. The methyl H atoms were then constrained to an ideal geometry, with C—H = 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

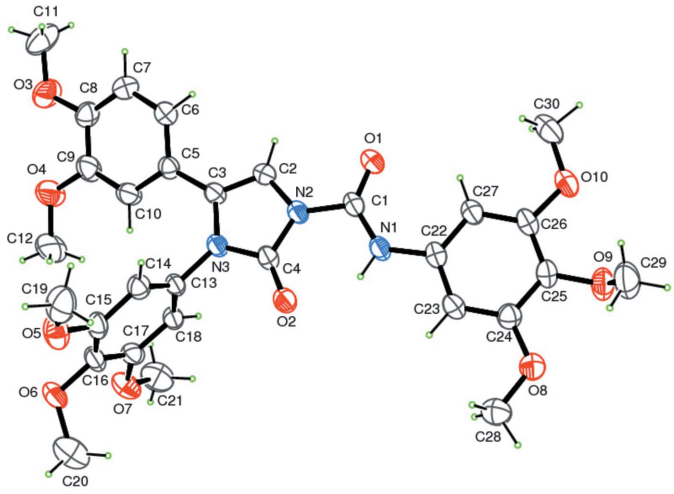


Figure 1
A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

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