Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(4-Fluorobenzoyl)-3,4,5-trimethoxybenzohydrazide monohydrate

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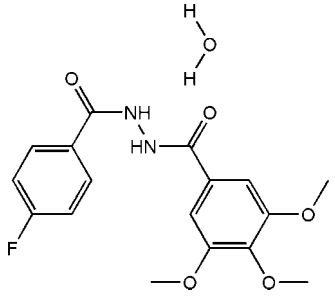
Received 17 May 2007; accepted 25 May 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.145; data-to-parameter ratio = 12.7.

The asymmetric unit of the title compound, $C_{17}H_{19}FN_2O_5\cdot H_2O$, contains two *N*′-(4-fluorobenzoyl)-3,4,5-trimethoxybenzohydrazide molecules and two water molecules. It is an important intermediate for the synthesis of biologically active heterocyclic compounds. The dihedral angles between the planar aromatic rings of the two molecules are 65.52 (3) and 68.48 (3)°. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen bonds link the molecules to form a supramolecular structure.

Related literature

For general backgroud, see: Zheng *et al.* (2003); Al-Talib *et al.* (1990); Yousif *et al.* (1986); Ahmad *et al.* (2001); Al-Soud *et al.* (2004); El-Emam *et al.* (2004); Allen *et al.* (1987). For related literature, see: Zareef & Iqbal (2007).



Experimental

Crystal data

$C_{17}H_{17}FN_2O_5\cdot H_2O$
M_r = 366.34
Monoclinic, $P2_1/c$
a = 8.3845 (5) Å
b = 13.2688 (9) Å
c = 31.833 (2) Å
β = 94.615 (1)°
V = 3530.0 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.34 × 0.23 × 0.16 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.97, T_{max} = 0.98
16960 measured reflections
6146 independent reflections
4451 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.145
S = 1.02
6146 reflections
485 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O9	0.86	2.06	2.814 (2)	145
N2—H2A···O11	0.86	2.01	2.821 (3)	157
N3—H3A···O4^{i}	0.86	2.00	2.814 (2)	157
N4—H4B···O12^{ii}	0.86	2.00	2.765 (2)	147
O11—H11A···O10^{iii}	1.01 (5)	1.93 (5)	2.862 (3)	152 (4)
O11—H11B···O5^{iv}	0.96 (5)	1.92 (5)	2.867 (3)	165 (5)
O12—H12A···O10^{v}	1.08 (5)	1.88 (5)	2.857 (3)	149 (4)
O12—H12B···O5^{v}	0.91 (5)	1.94 (5)	2.798 (3)	157 (4)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors gratefully acknowledge funds from the URF project, Quaid-I-Azam University, Islamabad, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2252).

References

Ahmad, R., Iqbal, R., Akhtar, R. H., Haq, Z. U., Duddeck, H., Stefaniak, L. & Sitkowski, J. (2001). *Nucleosides Nucleotides Nucleic Acids*, **20**, 1671–16.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Al-Soud, Y. A., Al-Deeri, M. N. & Al-Mosoudi, N. A. (2004). *Il Farm.*, **59**, 775–783.
Al-Talib, M., Tastoush, H. & Odeh, N. (1990). *Synth. Commun.* **20**, 1811–1814.
Bruker (1998). *SMART* Version 5.630. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* (Version 6.36a) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
El-Emam, A. A., Al-Deeb, O. A., Al-Omar, M. & Lehmann, J. (2004). *Bioorg. Med. Chem.* **12**, 5107–5113.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Yousif, M. Y., Ismail, A. M., Elman, A. A. & El-Kerdawy, M. M. (1986). *J. Chem. Soc. Pak.* **8**, 183–187.
Zareef, M. & Iqbal, R. (2007). *Phosphorus, Sulfur, Silicon Relat. Elem.*, **182**, 281–298.
Zheng, X., Li, Z., Wang, Y., Chen, W., Huang, Q., Liu, C. & Song, G. (2003). *J. Fluorine Chem.* **117**, 163–169.

supplementary materials

Acta Cryst. (2007). E**63**, o3052 [**doi:10.1107/S1600536807025524**]

N'-(4-Fluorobenzoyl)-3,4,5-trimethoxybenzohydrazide monohydrate

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Comment

N,*N*-Diacylhydrazines are important intermediates especially for the synthesis of various biologically active five-membered heterocycles such as 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In view of the versatility of these compounds, we have synthesized the title compound, (I), using a literature method (Zareef & Iqbal, 2007) and reported its crystal structure.

The asymmetric unit of the title compound, (I), contains two *N*'-(4-fluorobenzoyl)-3,4,5-trimethoxybenzohydrazide and two water molecules (Fig. 1), in which the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

Rings A (C1—C6), B (C12—C17) and C (C18—C23), D (C29—C34) are, of course, planar and the dihedral angles between them are A/B = 65.52 (3)° and C/D = 68.48 (3)°.

In the crystal structure, the intramolecular N—H···O and intermolecular N—H···O and O—H···O hydrogen bonds (Table 1) link the molecules to form a supramolecular structure, in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of (I), 4-fluorobenzoyl chloride (0.8 g, 5.1 mmol) was added in portions to a suspension of 3,4,5-trimethoxybenzoic hydrazide monohydrate (1.22 g, 5.0 mmol) in dry acetonitrile (50 ml), and the reaction mixture stirred for 7 h at 296 K. Then, the mixture was concentrated, and the solid product filtered and recrystallized from aqueous ethanol to afford the title compound (yield; 93%, m.p. 464–465 K). Suitable crystals were grown from a solution of (I) in ethanol by slow evaporation at room temperature.

Refinement

H atoms of water molecules were located in difference syntheses and refined isotropically [O—H = 0.91 (4)–1.08 (5) Å and U_{iso}(H) = 0.150 (18)–0.21 (3) Å^2]. The remaining H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*N*), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

Figures

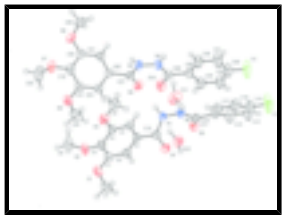


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. The systhesis route for the formation of the title compound.

N'-(4-Fluorobenzoyl)-3,4,5-trimethoxybenzohydrazide monohydrate

Crystal data

$C_{17}H_{17}FN_2O_5 \cdot H_2O$	F_{000} = 1536
M_r = 366.34	D_x = 1.379 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 464(1) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.3845 (5) Å	Cell parameters from 876 reflections
b = 13.2688 (9) Å	θ = 2.3–24.1º
c = 31.833 (2) Å	μ = 0.11 mm^{-1}
β = 94.615 (1)º	T = 294 (2) K
V = 3530.0 (4) Å^3	Plate, colorless
Z = 8	0.34 × 0.23 × 0.16 mm

Data collection

Bruker SMART CCD diffractometer	6146 independent reflections
Radiation source: fine-focus sealed tube	4451 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 294(2) K	θ_{max} = 25.0º
ω and φ scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −9→9
T_{min} = 0.97, T_{max} = 0.98	k = −15→15
16960 measured reflections	l = −37→29

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.145$	$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2 + 0.8812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.007$
6146 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
485 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}
C1	0.1353 (3)	0.62029 (16)	0.07074 (6)	0.0405 (5)
C2	0.2948 (3)	0.59103 (15)	0.07040 (6)	0.0394 (5)
C3	0.3980 (2)	0.59930 (15)	0.10683 (6)	0.0361 (5)
C4	0.3424 (2)	0.63769 (15)	0.14381 (6)	0.0347 (5)
H4A	0.4112	0.6443	0.1681	0.042*
C5	0.1821 (2)	0.66597 (14)	0.14378 (6)	0.0324 (4)
C6	0.0785 (3)	0.65717 (16)	0.10768 (6)	0.0377 (5)
H6A	−0.0283	0.6758	0.1082	0.045*
C7	−0.1246 (3)	0.6025 (2)	0.03532 (8)	0.0664 (8)
H7A	−0.1742	0.5944	0.0073	0.100*
H7B	−0.1631	0.6632	0.0474	0.100*
H7C	−0.1505	0.5458	0.0522	0.100*
C8	0.3834 (4)	0.6156 (2)	0.00231 (9)	0.0735 (9)
H8A	0.4212	0.5784	−0.0208	0.110*
H8B	0.4637	0.6630	0.0127	0.110*
H8C	0.2874	0.6512	−0.0072	0.110*
C9	0.6570 (3)	0.5665 (2)	0.14045 (8)	0.0581 (7)
H9A	0.7592	0.5409	0.1339	0.087*
H9B	0.6152	0.5250	0.1617	0.087*
H9C	0.6692	0.6343	0.1507	0.087*
C10	0.1128 (2)	0.70154 (14)	0.18295 (6)	0.0329 (5)
C11	0.1289 (2)	0.71749 (15)	0.27885 (6)	0.0356 (5)
C12	0.0682 (3)	0.75824 (16)	0.31808 (6)	0.0390 (5)
C13	0.0654 (4)	0.6931 (2)	0.35163 (8)	0.0656 (8)
H13A	0.1003	0.6271	0.3489	0.079*

C14	0.0112 (5)	0.7247 (3)	0.38937 (9)	0.0876 (11)
H14A	0.0098	0.6807	0.4121	0.105*
C15	−0.0398 (4)	0.8209 (3)	0.39262 (9)	0.0763 (9)
C16	−0.0401 (4)	0.8875 (2)	0.36048 (9)	0.0693 (8)
H16A	−0.0765	0.9530	0.3637	0.083*
C17	0.0147 (3)	0.85632 (17)	0.32281 (7)	0.0520 (6)
H17A	0.0157	0.9014	0.3004	0.062*
C18	0.6326 (3)	0.88247 (16)	0.06547 (6)	0.0404 (5)
C19	0.7901 (3)	0.91489 (16)	0.06329 (6)	0.0410 (5)
C20	0.8925 (3)	0.92140 (16)	0.09979 (7)	0.0395 (5)
C21	0.8393 (2)	0.89549 (15)	0.13871 (6)	0.0374 (5)
H21A	0.9079	0.8993	0.1631	0.045*
C22	0.6817 (2)	0.86384 (14)	0.14043 (6)	0.0331 (4)
C23	0.5787 (3)	0.85736 (16)	0.10412 (6)	0.0387 (5)
H23A	0.4736	0.8362	0.1057	0.046*
C24	0.3742 (3)	0.8852 (2)	0.02942 (9)	0.0699 (8)
H24A	0.3242	0.8818	0.0012	0.105*
H24B	0.3470	0.9478	0.0421	0.105*
H24C	0.3373	0.8302	0.0457	0.105*
C25	0.8852 (5)	0.8736 (3)	−0.00250 (10)	0.0913 (11)
H25A	0.9205	0.9053	−0.0272	0.137*
H25B	0.7937	0.8321	−0.0103	0.137*
H25C	0.9698	0.8327	0.0105	0.137*
C26	1.1576 (3)	0.9561 (2)	0.12892 (8)	0.0651 (8)
H26A	1.2562	0.9837	0.1207	0.098*
H26B	1.1747	0.8879	0.1383	0.098*
H26C	1.1206	0.9957	0.1514	0.098*
C27	0.6136 (2)	0.84363 (14)	0.18144 (6)	0.0332 (5)
C28	0.6198 (2)	0.71404 (15)	0.26891 (6)	0.0364 (5)
C29	0.5681 (3)	0.71889 (15)	0.31265 (6)	0.0378 (5)
C30	0.5624 (3)	0.80734 (17)	0.33588 (7)	0.0569 (7)
H30A	0.5917	0.8679	0.3240	0.068*
C31	0.5136 (4)	0.8065 (2)	0.37639 (8)	0.0707 (9)
H31A	0.5091	0.8659	0.3918	0.085*
C32	0.4727 (4)	0.7175 (2)	0.39295 (8)	0.0637 (7)
C33	0.4743 (3)	0.62851 (19)	0.37147 (8)	0.0605 (7)
H33A	0.4433	0.5688	0.3837	0.073*
C34	0.5231 (3)	0.62949 (17)	0.33105 (7)	0.0486 (6)
H34A	0.5259	0.5696	0.3160	0.058*
F1	−0.0941 (3)	0.85145 (18)	0.42954 (6)	0.1304 (9)
F2	0.4249 (3)	0.71612 (15)	0.43261 (5)	0.1053 (7)
H11A	0.227 (6)	1.024 (4)	0.2480 (16)	0.21 (3)*
H12A	0.524 (6)	0.029 (4)	0.2616 (14)	0.166 (19)*
H11B	0.020 (6)	1.030 (4)	0.2369 (15)	0.17 (2)*
H12B	0.732 (5)	0.038 (4)	0.2605 (13)	0.150 (18)*
N1	0.2139 (2)	0.75011 (13)	0.21121 (5)	0.0383 (4)
H1A	0.3115	0.7602	0.2059	0.046*
N2	0.1603 (2)	0.78397 (13)	0.24905 (5)	0.0386 (4)
H2A	0.1475	0.8474	0.2532	0.046*

N3	0.7156 (2)	0.80515 (13)	0.21211 (5)	0.0392 (4)
H3A	0.8085	0.7838	0.2066	0.047*
N4	0.6678 (2)	0.80059 (13)	0.25281 (5)	0.0399 (4)
H4B	0.6692	0.8544	0.2679	0.048*
O1	0.0433 (2)	0.60855 (14)	0.03384 (5)	0.0601 (5)
O2	0.3502 (2)	0.54784 (12)	0.03520 (5)	0.0515 (4)
O3	0.55010 (18)	0.56535 (12)	0.10374 (5)	0.0499 (4)
O4	−0.02783 (17)	0.68598 (12)	0.18909 (5)	0.0460 (4)
O5	0.1502 (2)	0.62589 (11)	0.27442 (5)	0.0493 (4)
O6	0.5408 (2)	0.87890 (15)	0.02826 (5)	0.0631 (5)
O7	0.8433 (2)	0.94808 (13)	0.02612 (5)	0.0571 (5)
O8	1.04171 (19)	0.95777 (14)	0.09416 (5)	0.0576 (5)
O9	0.47413 (17)	0.86429 (12)	0.18701 (5)	0.0448 (4)
O10	0.6223 (2)	0.63466 (11)	0.24890 (5)	0.0556 (5)
O11	0.1199 (3)	0.99373 (14)	0.23830 (7)	0.0737 (6)
O12	0.6459 (3)	0.00454 (12)	0.26922 (5)	0.0567 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0457 (13)	0.0455 (12)	0.0300 (11)	−0.0021 (10)	0.0022 (9)	−0.0024 (9)
C2	0.0484 (14)	0.0371 (11)	0.0343 (11)	0.0001 (10)	0.0132 (9)	−0.0059 (9)
C3	0.0339 (12)	0.0360 (11)	0.0398 (12)	0.0024 (9)	0.0115 (9)	−0.0010 (9)
C4	0.0334 (12)	0.0372 (11)	0.0337 (11)	−0.0007 (9)	0.0047 (8)	−0.0037 (8)
C5	0.0325 (11)	0.0325 (10)	0.0331 (11)	−0.0004 (8)	0.0074 (8)	−0.0019 (8)
C6	0.0340 (12)	0.0426 (12)	0.0368 (12)	0.0024 (9)	0.0054 (9)	−0.0008 (9)
C7	0.0518 (17)	0.092 (2)	0.0533 (16)	−0.0062 (14)	−0.0099 (12)	−0.0093 (14)
C8	0.098 (2)	0.0757 (19)	0.0520 (16)	0.0077 (17)	0.0375 (15)	0.0017 (14)
C9	0.0367 (14)	0.0726 (17)	0.0648 (16)	0.0087 (12)	0.0016 (12)	−0.0125 (13)
C10	0.0325 (12)	0.0340 (10)	0.0329 (11)	0.0031 (8)	0.0067 (8)	0.0006 (8)
C11	0.0357 (12)	0.0336 (11)	0.0378 (11)	−0.0021 (9)	0.0039 (9)	−0.0024 (9)
C12	0.0393 (12)	0.0433 (12)	0.0349 (11)	−0.0050 (9)	0.0060 (9)	−0.0057 (9)
C13	0.094 (2)	0.0544 (15)	0.0513 (16)	0.0097 (14)	0.0241 (14)	0.0082 (12)
C14	0.136 (3)	0.085 (2)	0.0462 (17)	0.009 (2)	0.0385 (18)	0.0165 (15)
C15	0.105 (3)	0.086 (2)	0.0426 (16)	−0.0062 (18)	0.0323 (15)	−0.0172 (15)
C16	0.098 (2)	0.0535 (15)	0.0608 (18)	−0.0028 (15)	0.0338 (16)	−0.0194 (13)
C17	0.0719 (17)	0.0432 (13)	0.0433 (13)	0.0008 (12)	0.0189 (12)	−0.0027 (10)
C18	0.0456 (13)	0.0433 (12)	0.0318 (11)	0.0008 (10)	−0.0001 (9)	−0.0005 (9)
C19	0.0515 (14)	0.0390 (11)	0.0338 (11)	0.0031 (10)	0.0109 (10)	0.0068 (9)
C20	0.0372 (12)	0.0406 (11)	0.0421 (12)	−0.0018 (9)	0.0118 (9)	0.0062 (9)
C21	0.0360 (12)	0.0417 (12)	0.0348 (11)	−0.0021 (9)	0.0044 (9)	0.0046 (9)
C22	0.0341 (12)	0.0332 (10)	0.0328 (11)	0.0007 (8)	0.0067 (8)	0.0018 (8)
C23	0.0336 (12)	0.0429 (12)	0.0399 (12)	−0.0025 (9)	0.0053 (9)	0.0007 (9)
C24	0.0629 (19)	0.086 (2)	0.0573 (17)	0.0096 (15)	−0.0176 (13)	−0.0017 (14)
C25	0.133 (3)	0.086 (2)	0.0627 (19)	−0.009 (2)	0.054 (2)	−0.0037 (17)
C26	0.0387 (14)	0.089 (2)	0.0673 (17)	−0.0166 (14)	0.0037 (12)	0.0139 (15)
C27	0.0322 (12)	0.0315 (10)	0.0364 (11)	−0.0038 (8)	0.0066 (9)	−0.0020 (8)
C28	0.0364 (12)	0.0345 (11)	0.0387 (11)	0.0035 (9)	0.0051 (9)	−0.0004 (9)

C29	0.0423 (13)	0.0372 (11)	0.0345 (11)	0.0032 (9)	0.0073 (9)	0.0063 (9)
C30	0.091 (2)	0.0363 (12)	0.0460 (14)	−0.0039 (12)	0.0246 (13)	0.0037 (10)
C31	0.122 (3)	0.0474 (15)	0.0465 (15)	0.0005 (15)	0.0322 (16)	−0.0049 (12)
C32	0.091 (2)	0.0657 (17)	0.0374 (14)	0.0067 (15)	0.0253 (13)	0.0089 (12)
C33	0.087 (2)	0.0482 (14)	0.0496 (15)	−0.0017 (13)	0.0222 (13)	0.0181 (12)
C34	0.0644 (16)	0.0367 (12)	0.0461 (13)	0.0004 (11)	0.0129 (11)	0.0033 (10)
F1	0.205 (3)	0.1345 (18)	0.0614 (12)	0.0040 (18)	0.0715 (14)	−0.0247 (12)
F2	0.181 (2)	0.0936 (13)	0.0492 (10)	0.0023 (13)	0.0573 (11)	0.0106 (9)
N1	0.0356 (10)	0.0485 (10)	0.0327 (9)	−0.0055 (8)	0.0138 (7)	−0.0089 (8)
N2	0.0494 (11)	0.0326 (9)	0.0352 (9)	−0.0036 (8)	0.0124 (8)	−0.0065 (7)
N3	0.0387 (10)	0.0498 (11)	0.0309 (9)	0.0065 (8)	0.0138 (8)	0.0039 (8)
N4	0.0548 (12)	0.0353 (9)	0.0314 (9)	−0.0017 (8)	0.0141 (8)	−0.0013 (7)
O1	0.0526 (11)	0.0931 (13)	0.0337 (9)	0.0022 (9)	−0.0013 (7)	−0.0112 (8)
O2	0.0663 (11)	0.0540 (10)	0.0363 (9)	0.0052 (8)	0.0180 (7)	−0.0095 (7)
O3	0.0385 (9)	0.0629 (10)	0.0497 (10)	0.0115 (7)	0.0117 (7)	−0.0084 (8)
O4	0.0300 (9)	0.0627 (10)	0.0464 (9)	−0.0008 (7)	0.0102 (7)	−0.0091 (7)
O5	0.0638 (11)	0.0341 (9)	0.0513 (10)	0.0055 (7)	0.0138 (8)	−0.0020 (7)
O6	0.0544 (11)	0.0962 (14)	0.0374 (9)	−0.0030 (10)	−0.0036 (8)	0.0010 (9)
O7	0.0713 (12)	0.0643 (11)	0.0379 (9)	−0.0013 (9)	0.0181 (8)	0.0126 (8)
O8	0.0398 (9)	0.0815 (12)	0.0524 (10)	−0.0144 (8)	0.0102 (7)	0.0173 (9)
O9	0.0314 (9)	0.0547 (9)	0.0496 (9)	−0.0010 (7)	0.0115 (7)	0.0003 (7)
O10	0.0804 (13)	0.0363 (9)	0.0528 (10)	−0.0035 (8)	0.0222 (9)	−0.0072 (7)
O11	0.0760 (15)	0.0430 (10)	0.1049 (16)	0.0036 (10)	0.0246 (12)	0.0115 (10)
O12	0.0779 (14)	0.0358 (9)	0.0578 (11)	−0.0084 (9)	0.0150 (9)	0.0026 (7)

Geometric parameters (Å, °)

C1—O1	1.361 (3)	C20—O8	1.366 (3)
C1—C6	1.393 (3)	C20—C21	1.393 (3)
C1—C2	1.393 (3)	C21—C22	1.392 (3)
C2—O2	1.373 (2)	C21—H21A	0.9300
C2—C3	1.394 (3)	C22—C23	1.389 (3)
C3—O3	1.364 (2)	C22—C27	1.490 (3)
C3—C4	1.396 (3)	C23—H23A	0.9300
C4—C5	1.396 (3)	C24—O6	1.403 (3)
C4—H4A	0.9300	C24—H24A	0.9600
C5—C6	1.389 (3)	C24—H24B	0.9600
C5—C10	1.494 (3)	C24—H24C	0.9600
C6—H6A	0.9300	C25—O7	1.407 (3)
C7—O1	1.415 (3)	C25—H25A	0.9600
C7—H7A	0.9600	C25—H25B	0.9600
C7—H7B	0.9600	C25—H25C	0.9600
C7—H7C	0.9600	C26—O8	1.413 (3)
C8—O2	1.425 (3)	C26—H26A	0.9600
C8—H8A	0.9600	C26—H26B	0.9600
C8—H8B	0.9600	C26—H26C	0.9600
C8—H8C	0.9600	C27—O9	1.228 (2)
C9—O3	1.415 (3)	C27—N3	1.346 (3)
C9—H9A	0.9600	C28—O10	1.232 (2)

C9—H9B	0.9600	C28—N4	1.333 (3)
C9—H9C	0.9600	C28—C29	1.493 (3)
C10—O4	1.228 (2)	C29—C34	1.389 (3)
C10—N1	1.349 (3)	C29—C30	1.390 (3)
C11—O5	1.238 (2)	C30—C31	1.384 (3)
C11—N2	1.337 (3)	C30—H30A	0.9300
C11—C12	1.488 (3)	C31—C32	1.349 (4)
C12—C13	1.376 (3)	C31—H31A	0.9300
C12—C17	1.389 (3)	C32—F2	1.355 (3)
C13—C14	1.383 (4)	C32—C33	1.365 (4)
C13—H13A	0.9300	C33—C34	1.381 (3)
C14—C15	1.353 (4)	C33—H33A	0.9300
C14—H14A	0.9300	C34—H34A	0.9300
C15—C16	1.351 (4)	N1—N2	1.393 (2)
C15—F1	1.356 (3)	N1—H1A	0.8600
C16—C17	1.381 (3)	N2—H2A	0.8600
C16—H16A	0.9300	N3—N4	1.388 (2)
C17—H17A	0.9300	N3—H3A	0.8600
C18—O6	1.360 (3)	N4—H4B	0.8600
C18—C23	1.385 (3)	O11—H11A	1.01 (6)
C18—C19	1.396 (3)	O11—H11B	0.97 (6)
C19—O7	1.370 (2)	O12—H12A	1.08 (5)
C19—C20	1.391 (3)	O12—H12B	0.91 (4)
O1—C1—C6	124.0 (2)	C22—C21—H21A	120.6
O1—C1—C2	116.06 (18)	C20—C21—H21A	120.6
C6—C1—C2	119.88 (19)	C23—C22—C21	120.98 (18)
O2—C2—C1	120.72 (19)	C23—C22—C27	117.48 (18)
O2—C2—C3	118.93 (19)	C21—C22—C27	121.36 (18)
C1—C2—C3	120.23 (18)	C18—C23—C22	119.9 (2)
O3—C3—C2	115.82 (17)	C18—C23—H23A	120.0
O3—C3—C4	123.94 (19)	C22—C23—H23A	120.0
C2—C3—C4	120.21 (19)	O6—C24—H24A	109.5
C5—C4—C3	118.95 (19)	O6—C24—H24B	109.5
C5—C4—H4A	120.5	H24A—C24—H24B	109.5
C3—C4—H4A	120.5	O6—C24—H24C	109.5
C6—C5—C4	121.08 (18)	H24A—C24—H24C	109.5
C6—C5—C10	117.25 (18)	H24B—C24—H24C	109.5
C4—C5—C10	121.59 (17)	O7—C25—H25A	109.5
C5—C6—C1	119.6 (2)	O7—C25—H25B	109.5
C5—C6—H6A	120.2	H25A—C25—H25B	109.5
C1—C6—H6A	120.2	O7—C25—H25C	109.5
O1—C7—H7A	109.5	H25A—C25—H25C	109.5
O1—C7—H7B	109.5	H25B—C25—H25C	109.5
H7A—C7—H7B	109.5	O8—C26—H26A	109.5
O1—C7—H7C	109.5	O8—C26—H26B	109.5
H7A—C7—H7C	109.5	H26A—C26—H26B	109.5
H7B—C7—H7C	109.5	O8—C26—H26C	109.5
O2—C8—H8A	109.5	H26A—C26—H26C	109.5
O2—C8—H8B	109.5	H26B—C26—H26C	109.5

H8A—C8—H8B	109.5	O9—C27—N3	122.46 (18)
O2—C8—H8C	109.5	O9—C27—C22	121.52 (18)
H8A—C8—H8C	109.5	N3—C27—C22	115.97 (17)
H8B—C8—H8C	109.5	O10—C28—N4	121.32 (19)
O3—C9—H9A	109.5	O10—C28—C29	122.51 (19)
O3—C9—H9B	109.5	N4—C28—C29	116.15 (18)
H9A—C9—H9B	109.5	C34—C29—C30	118.3 (2)
O3—C9—H9C	109.5	C34—C29—C28	117.77 (19)
H9A—C9—H9C	109.5	C30—C29—C28	123.89 (19)
H9B—C9—H9C	109.5	C31—C30—C29	121.0 (2)
O4—C10—N1	122.27 (18)	C31—C30—H30A	119.5
O4—C10—C5	121.50 (18)	C29—C30—H30A	119.5
N1—C10—C5	116.22 (17)	C32—C31—C30	118.3 (2)
O5—C11—N2	121.91 (19)	C32—C31—H31A	120.8
O5—C11—C12	120.96 (19)	C30—C31—H31A	120.8
N2—C11—C12	117.12 (18)	C31—C32—F2	118.6 (2)
C13—C12—C17	118.6 (2)	C31—C32—C33	123.3 (2)
C13—C12—C11	116.9 (2)	F2—C32—C33	118.1 (2)
C17—C12—C11	124.5 (2)	C32—C33—C34	118.3 (2)
C12—C13—C14	120.7 (3)	C32—C33—H33A	120.8
C12—C13—H13A	119.6	C34—C33—H33A	120.8
C14—C13—H13A	119.6	C33—C34—C29	120.8 (2)
C15—C14—C13	118.7 (3)	C33—C34—H34A	119.6
C15—C14—H14A	120.6	C29—C34—H34A	119.6
C13—C14—H14A	120.6	C10—N1—N2	120.11 (16)
C16—C15—C14	122.7 (2)	C10—N1—H1A	119.9
C16—C15—F1	118.7 (3)	N2—N1—H1A	119.9
C14—C15—F1	118.6 (3)	C11—N2—N1	119.74 (17)
C15—C16—C17	118.8 (3)	C11—N2—H2A	120.1
C15—C16—H16A	120.6	N1—N2—H2A	120.1
C17—C16—H16A	120.6	C27—N3—N4	118.66 (17)
C16—C17—C12	120.5 (2)	C27—N3—H3A	120.7
C16—C17—H17A	119.8	N4—N3—H3A	120.7
C12—C17—H17A	119.8	C28—N4—N3	121.00 (17)
O6—C18—C23	124.3 (2)	C28—N4—H4B	119.5
O6—C18—C19	116.01 (19)	N3—N4—H4B	119.5
C23—C18—C19	119.73 (19)	C1—O1—C7	118.31 (18)
O7—C19—C20	118.6 (2)	C2—O2—C8	115.80 (18)
O7—C19—C18	121.13 (19)	C3—O3—C9	118.12 (17)
C20—C19—C18	120.05 (18)	C18—O6—C24	118.01 (19)
O8—C20—C19	115.02 (18)	C19—O7—C25	116.67 (19)
O8—C20—C21	124.5 (2)	C20—O8—C26	118.12 (18)
C19—C20—C21	120.5 (2)	H11A—O11—H11B	124 (4)
C22—C21—C20	118.83 (19)	H12A—O12—H12B	123 (4)
O1—C1—C2—O2	−3.5 (3)	C20—C21—C22—C27	−174.65 (19)
C6—C1—C2—O2	175.32 (19)	O6—C18—C23—C22	−179.8 (2)
O1—C1—C2—C3	−179.37 (19)	C19—C18—C23—C22	−0.5 (3)
C6—C1—C2—C3	−0.5 (3)	C21—C22—C23—C18	0.1 (3)
O2—C2—C3—O3	1.8 (3)	C27—C22—C23—C18	175.33 (18)

C1—C2—C3—O3	177.72 (19)	C23—C22—C27—O9	−31.8 (3)
O2—C2—C3—C4	−176.40 (18)	C21—C22—C27—O9	143.4 (2)
C1—C2—C3—C4	−0.5 (3)	C23—C22—C27—N3	150.57 (19)
O3—C3—C4—C5	−177.07 (18)	C21—C22—C27—N3	−34.2 (3)
C2—C3—C4—C5	1.0 (3)	O10—C28—C29—C34	0.7 (3)
C3—C4—C5—C6	−0.5 (3)	N4—C28—C29—C34	−177.6 (2)
C3—C4—C5—C10	176.16 (18)	O10—C28—C29—C30	−179.3 (2)
C4—C5—C6—C1	−0.5 (3)	N4—C28—C29—C30	2.4 (3)
C10—C5—C6—C1	−177.29 (18)	C34—C29—C30—C31	0.2 (4)
O1—C1—C6—C5	179.8 (2)	C28—C29—C30—C31	−179.8 (2)
C2—C1—C6—C5	1.0 (3)	C29—C30—C31—C32	0.5 (5)
C6—C5—C10—O4	28.4 (3)	C30—C31—C32—F2	179.9 (3)
C4—C5—C10—O4	−148.3 (2)	C30—C31—C32—C33	−1.2 (5)
C6—C5—C10—N1	−152.48 (19)	C31—C32—C33—C34	1.3 (5)
C4—C5—C10—N1	30.8 (3)	F2—C32—C33—C34	−179.9 (3)
O5—C11—C12—C13	11.6 (3)	C32—C33—C34—C29	−0.5 (4)
N2—C11—C12—C13	−167.8 (2)	C30—C29—C34—C33	−0.2 (4)
O5—C11—C12—C17	−168.1 (2)	C28—C29—C34—C33	179.9 (2)
N2—C11—C12—C17	12.4 (3)	O4—C10—N1—N2	0.5 (3)
C17—C12—C13—C14	−0.4 (4)	C5—C10—N1—N2	−178.61 (17)
C11—C12—C13—C14	179.8 (3)	O5—C11—N2—N1	2.0 (3)
C12—C13—C14—C15	0.3 (5)	C12—C11—N2—N1	−178.50 (18)
C13—C14—C15—C16	0.1 (6)	C10—N1—N2—C11	71.4 (3)
C13—C14—C15—F1	179.5 (3)	O9—C27—N3—N4	−8.4 (3)
C14—C15—C16—C17	−0.4 (5)	C22—C27—N3—N4	169.24 (16)
F1—C15—C16—C17	−179.8 (3)	O10—C28—N4—N3	2.9 (3)
C15—C16—C17—C12	0.3 (4)	C29—C28—N4—N3	−178.77 (18)
C13—C12—C17—C16	0.0 (4)	C27—N3—N4—C28	102.7 (2)
C11—C12—C17—C16	179.8 (2)	C6—C1—O1—C7	−20.6 (3)
O6—C18—C19—O7	4.7 (3)	C2—C1—O1—C7	158.2 (2)
C23—C18—C19—O7	−174.6 (2)	C1—C2—O2—C8	78.9 (3)
O6—C18—C19—C20	179.7 (2)	C3—C2—O2—C8	−105.2 (3)
C23—C18—C19—C20	0.3 (3)	C2—C3—O3—C9	−176.3 (2)
O7—C19—C20—O8	−2.4 (3)	C4—C3—O3—C9	1.8 (3)
C18—C19—C20—O8	−177.52 (19)	C23—C18—O6—C24	23.3 (3)
O7—C19—C20—C21	175.25 (19)	C19—C18—O6—C24	−156.0 (2)
C18—C19—C20—C21	0.2 (3)	C20—C19—O7—C25	104.8 (3)
O8—C20—C21—C22	176.9 (2)	C18—C19—O7—C25	−80.2 (3)
C19—C20—C21—C22	−0.5 (3)	C19—C20—O8—C26	−173.2 (2)
C20—C21—C22—C23	0.4 (3)	C21—C20—O8—C26	9.3 (3)

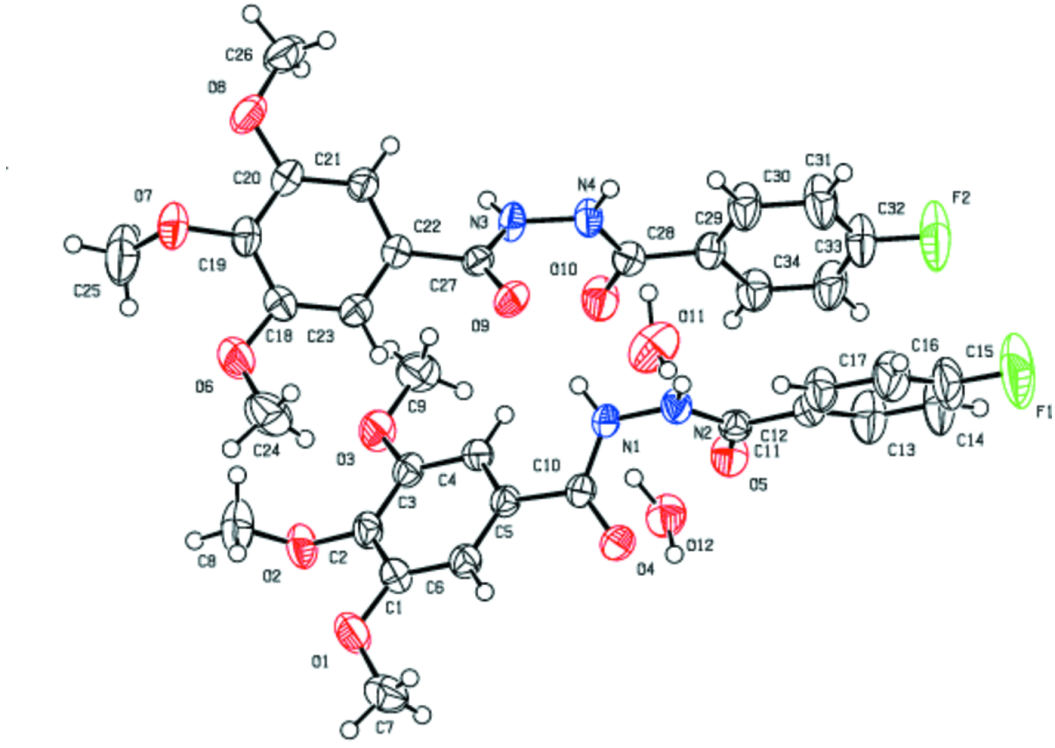
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O9	0.86	2.06	2.814 (2)	145
N2—H2A···O11	0.86	2.01	2.821 (3)	157
N3—H3A···O4^{i}	0.86	2.00	2.814 (2)	157
N4—H4B···O12^{ii}	0.86	2.00	2.765 (2)	147

O11—H11A···O10^{iii}	1.01 (5)	1.93 (5)	2.862 (3)	152 (4)
O11—H11B···O5^{iv}	0.96 (5)	1.92 (5)	2.867 (3)	165 (5)
O12—H12A···O10^{v}	1.08 (5)	1.88 (5)	2.857 (3)	149 (4)
O12—H12B···O5^{v}	0.91 (5)	1.94 (5)	2.798 (3)	157 (4)

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*, *y*+1, *z*; (iii) −*x*+1, *y*+1/2, −*z*+1/2; (iv) −*x*, *y*+1/2, −*z*+1/2; (v) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1


C26
O8
C21
C20
O7
C22
N3
N4
C28
C29
C30
C31
C32
F2
C25
C19
C27
O10
C33
C34
O11
O9
C18
C23
C17
C16
C15
O6
C9
N1
N2
C12
C13
C14
F1
C24
O3
C4
C10
C11
O5
C3
C5
O12
C8
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O4
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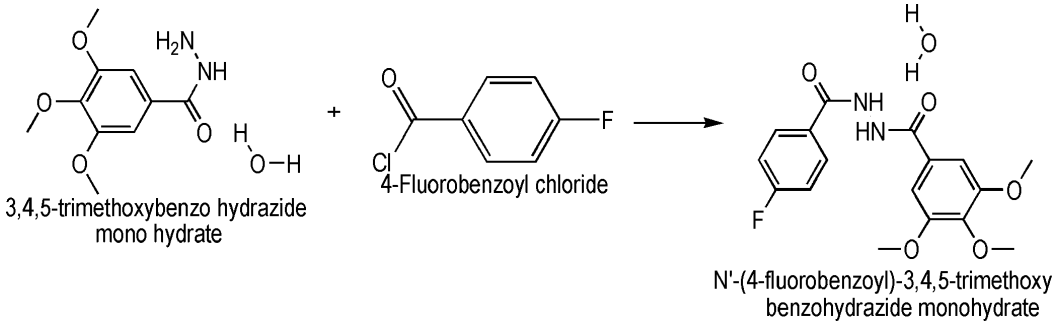
C1
O1
C7

Fig. 2


3,4,5-trimethoxybenzo hydrazide
mono hydrate
4-Fluorobenzoyl chloride
N'-(4-fluorobenzoyl)-3,4,5-trimethoxy
benzohydrazide monohydrate