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N,*N*′-Bis(2,3,4,5-tetrafluorobenzoyl)-piperazine

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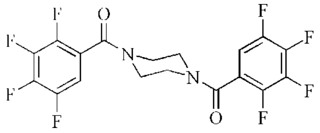
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 13.4.

The molecule of the title compound, $C_{18}H_{10}F_8N_2O_2$, is located on an inversion center. The piperazine ring adopts a chair conformation and the two tetrafluorophenyl rings are strictly parallel to each other. The molecules are connected by C—H⋯O hydrogen bonds into a one-dimensional chain-like supramolecular structure.

Related literature

For related literature, see: Kartha *et al.* (1981); Luo & Palmore (2002); Sarangarajan *et al.* (2005); Varughese & Kartha (1982); Zheng *et al.* (2005).



Experimental

Crystal data

$C_{18}H_{10}F_8N_2O_2$
M_r = 438.28
Monoclinic, $C2/c$
a = 30.158 (4) Å
b = 8.9113 (12) Å
c = 6.5744 (9) Å
β = 96.594 (2)°
V = 1755.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 291 (2) K
0.21 × 0.17 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.964, T_{max} = 0.982
6914 measured reflections
1816 independent reflections
1505 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.05
1816 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C9—H9A⋯O1^{i}	0.97	2.55	3.161 (2)	121

Symmetry code: (i) $x, y, z + 1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL97* and *XP*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2210).

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supplementary materials

Acta Cryst. (2007). E**63**, o4178 [**doi:10.1107/S1600536807046776**]

N,*N*'-Bis(2,3,4,5-tetrafluorobenzoyl)piperazine

X.-M. Zhang and **J.-T. Lu**

Comment

Recently, the supramolecular structures of systems containing piperazine unit, which includes both of hydrogen-bonded systems and of metal coordination complexes, have been extensively studied. (Kartha *et al.*, 1981; Luo & Palmore, 2002; Varughese & Kartha, 1982). To date, many piperazine derivatives with various substitutes have been synthesized (Zheng *et al.*, 2005; Sarangarajan *et al.*, 2005). Herein, we report the structure of a new compound *N*,*N*'-bis(2,3,4,5-tetrafluorobenzoyl)piperazine (I).

The geometry and labeling scheme of the title compound are depicted in Figure 1. The compound is a centrosymmetric compound *N*,*N*'-bis(2,3,4,5-tetrafluorobenzoyl)piperazine. The piperazine ring adopts a chair conformation and the two tetrafluorophenyl rings are strict parallel to each other due to the centrosymmetry. All atoms from tetrafluorophenyl ring is nearly strict planar with the largest deviation of 0.0105 (18) Å from the mean plane. The carbonyl group bond distance is 1.2275 (18) Å for C7—O1 and the amide bond distance is 1.3409 (19) Å for C7—N1, which are very similar to those of its derivative *N*,*N*'-dibenzoylpiperazine (Zheng *et al.*, 2005). The C—F bond lengths range from 1.334 (2) to 1.334 (2) Å.

In the title compound, the *N*,*N*'-bis(2,3,4,5-tetrafluorobenzoyl)piperazine units were connected together by the intermolecular C—H···O hydrogen bonds, leading to a one-dimensional chain-like supramolecular structure as shown in Figure 2.

Experimental

The title compound *N*,*N*'-bis(2,3,4,5-tetrafluorobenzoyl) piperazine was synthesized by the reaction of tetrafluorobenzoyl chloride (1.4 g, 10 mmol) with piperazine (0.42 g, 5 mmol) in the presence of triethylamine (1.01 g, 10 mmol) in ethanol (50 ml). Colorless block crystals were obtained by recrystallization from an ethanol solution (yield 80%). Elemental analysis, calculated for $C_{18}H_{10}F_8N_2O_2$: C 49.33, H 2.30, N 6.39%; found: C 49.26, H 2.28, N 6.30%.

Refinement

H atoms were located in a difference map and were refined as riding, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

Fig. 1. The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for non-H atoms. The suffix A corresponds to symmetry code (−*x* + 1/2, −*y* + 3/2, −*z* + 1).

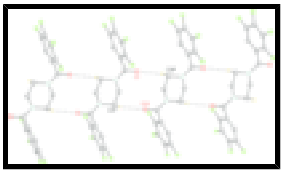

Fig. 2. A part of the crystal structure of (I), showing the one-dimensional chain-like supramolecular structure. Dashed lines indicate C—H···O hydrogen bonds. For the sake of clarity, H atoms not involved in the hydrogen bonds have been omitted. Atoms marked with asterisk (*), hash (#) and dollar ($) signs are at the symmetry positions ($-x + 1/2$, $-y + 3/2$, $-z + 1$), (x, y, $z - 1$) and ($-x + 1/2$, $-y + 3/2$, $-z$), respectively.

N,*N*'-Bis(2,3,4,5-tetrafluorobenzoyl)piperazine

Crystal data

$C_{18}H_{10}F_8N_2O_2$
$M_r = 438.28$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 30.158$ (4) Å
$b = 8.9113$ (12) Å
$c = 6.5744$ (9) Å
$\beta = 96.594$ (2)°
$V = 1755.2$ (4) Å^3
$Z = 4$

$F_{000} = 880$
$D_x = 1.659$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 1816 reflections
$\theta = 1.4$–26.5°
$\mu = 0.17$ mm^{-1}
$T = 291$ (2) K
Block, colourless
0.21 × 0.17 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 293(2)$ K
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.964$, $T_{max} = 0.982$
6914 measured reflections

1816 independent reflections
1505 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 26.5$°
$\theta_{min} = 1.4$°
$h = -37 \rightarrow 37$
$k = -11 \rightarrow 11$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.109$
$S = 1.05$
1816 reflections
136 parameters

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.9211P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ 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 > 2sigma(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*	$U_{\rm iso}$*/$U_{\rm eq}$
F1	0.35977 (4)	0.55322 (14)	0.1733 (2)	0.0882 (5)
F2	0.44756 (5)	0.51144 (15)	0.2847 (3)	0.1033 (5)
F3	0.49652 (4)	0.72402 (17)	0.4995 (2)	0.0858 (4)
F4	0.45729 (4)	0.97811 (16)	0.6065 (2)	0.0817 (4)
O1	0.30498 (4)	0.85478 (16)	0.07758 (16)	0.0567 (4)
N1	0.28391 (4)	0.80494 (15)	0.38982 (18)	0.0394 (3)
C1	0.38334 (6)	0.6604 (2)	0.2813 (3)	0.0540 (4)
C2	0.42821 (6)	0.6382 (2)	0.3369 (3)	0.0624 (5)
C3	0.45290 (5)	0.7449 (2)	0.4468 (3)	0.0566 (5)
C4	0.43232 (6)	0.8737 (2)	0.5014 (3)	0.0511 (4)
C5	0.38765 (5)	0.89670 (19)	0.4480 (2)	0.0454 (4)
H5	0.3743	0.9846	0.4870	0.054*
C6	0.36230 (5)	0.78895 (17)	0.3356 (2)	0.0401 (4)
C7	0.31433 (5)	0.81783 (17)	0.2572 (2)	0.0394 (3)
C8	0.23774 (5)	0.84858 (19)	0.3295 (2)	0.0410 (4)
H8A	0.2329	0.8623	0.1822	0.049*
H8B	0.2318	0.9433	0.3936	0.049*
C9	0.29394 (5)	0.76986 (19)	0.6077 (2)	0.0412 (4)
H9A	0.2912	0.8601	0.6880	0.049*
H9B	0.3244	0.7340	0.6347	0.049*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0746 (8)	0.0620 (7)	0.1262 (12)	−0.0065 (6)	0.0041 (8)	−0.0388 (7)
F2	0.0810 (9)	0.0719 (9)	0.1567 (15)	0.0310 (7)	0.0121 (9)	−0.0272 (9)
F3	0.0411 (6)	0.1097 (11)	0.1053 (10)	0.0177 (6)	0.0028 (6)	−0.0016 (8)
F4	0.0482 (6)	0.1015 (10)	0.0933 (9)	−0.0141 (6)	−0.0017 (6)	−0.0367 (7)
O1	0.0500 (7)	0.0867 (9)	0.0341 (6)	−0.0045 (6)	0.0081 (5)	0.0078 (6)
N1	0.0338 (7)	0.0526 (8)	0.0319 (6)	−0.0005 (5)	0.0046 (5)	0.0055 (5)
C1	0.0522 (10)	0.0468 (9)	0.0638 (11)	−0.0052 (8)	0.0103 (8)	−0.0084 (8)

C2	0.0555 (11)	0.0535 (11)	0.0804 (13)	0.0136 (9)	0.0170 (10)	−0.0038 (9)
C3	0.0374 (9)	0.0736 (12)	0.0596 (11)	0.0064 (8)	0.0095 (8)	0.0056 (9)
C4	0.0412 (9)	0.0640 (11)	0.0484 (9)	−0.0066 (8)	0.0068 (7)	−0.0071 (8)
C5	0.0417 (9)	0.0508 (9)	0.0448 (9)	0.0009 (7)	0.0099 (7)	−0.0076 (7)
C6	0.0393 (8)	0.0467 (8)	0.0360 (7)	−0.0025 (6)	0.0121 (6)	0.0016 (6)
C7	0.0409 (8)	0.0431 (8)	0.0348 (8)	−0.0057 (6)	0.0068 (6)	−0.0014 (6)
C8	0.0377 (8)	0.0507 (9)	0.0345 (7)	0.0029 (7)	0.0038 (6)	0.0072 (6)
C9	0.0356 (7)	0.0561 (9)	0.0317 (7)	−0.0019 (7)	0.0025 (6)	0.0043 (6)

Geometric parameters (Å, °)

F1—C1	1.344 (2)	C3—C4	1.372 (3)
F2—C2	1.334 (2)	C4—C5	1.368 (2)
F3—C3	1.3343 (19)	C5—C6	1.386 (2)
F4—C4	1.338 (2)	C5—H5	0.9300
O1—C7	1.2275 (18)	C6—C7	1.501 (2)
N1—C7	1.3409 (19)	C8—C9^{i}	1.513 (2)
N1—C8	1.4563 (19)	C8—H8A	0.9700
N1—C9	1.4635 (18)	C8—H8B	0.9700
C1—C2	1.375 (3)	C9—C8^{i}	1.513 (2)
C1—C6	1.376 (2)	C9—H9A	0.9700
C2—C3	1.363 (3)	C9—H9B	0.9700
C7—N1—C8	120.27 (12)	C1—C6—C5	118.18 (15)
C7—N1—C9	125.08 (13)	C1—C6—C7	120.56 (15)
C8—N1—C9	114.04 (11)	C5—C6—C7	120.98 (14)
F1—C1—C2	118.85 (16)	O1—C7—N1	123.36 (15)
F1—C1—C6	119.81 (16)	O1—C7—C6	118.82 (13)
C2—C1—C6	121.34 (17)	N1—C7—C6	117.81 (13)
F2—C2—C3	119.90 (18)	N1—C8—C9^{i}	110.70 (13)
F2—C2—C1	120.01 (18)	N1—C8—H8A	109.5
C3—C2—C1	120.09 (17)	C9^{i}—C8—H8A	109.5
F3—C3—C2	120.17 (18)	N1—C8—H8B	109.5
F3—C3—C4	120.72 (18)	C9^{i}—C8—H8B	109.5
C2—C3—C4	119.11 (16)	H8A—C8—H8B	108.1
F4—C4—C5	120.50 (16)	N1—C9—C8^{i}	110.51 (12)
F4—C4—C3	118.17 (16)	N1—C9—H9A	109.5
C5—C4—C3	121.32 (16)	C8^{i}—C9—H9A	109.5
C4—C5—C6	119.97 (16)	N1—C9—H9B	109.5
C4—C5—H5	120.0	C8^{i}—C9—H9B	109.5
C6—C5—H5	120.0	H9A—C9—H9B	108.1
F1—C1—C2—F2	−0.4 (3)	F1—C1—C6—C7	−6.4 (2)
C6—C1—C2—F2	178.96 (18)	C2—C1—C6—C7	174.31 (16)
F1—C1—C2—C3	−179.86 (18)	C4—C5—C6—C1	0.1 (2)
C6—C1—C2—C3	−0.5 (3)	C4—C5—C6—C7	−173.87 (14)
F2—C2—C3—F3	1.1 (3)	C8—N1—C7—O1	−5.7 (2)
C1—C2—C3—F3	−179.42 (18)	C9—N1—C7—O1	−176.22 (15)
F2—C2—C3—C4	−179.17 (18)	C8—N1—C7—C6	172.75 (13)

C1—C2—C3—C4	0.3 (3)	C9—N1—C7—C6	2.3 (2)
F3—C3—C4—F4	0.7 (3)	C1—C6—C7—O1	−73.8 (2)
C2—C3—C4—F4	−178.98 (17)	C5—C6—C7—O1	100.00 (18)
F3—C3—C4—C5	179.83 (16)	C1—C6—C7—N1	107.63 (17)
C2—C3—C4—C5	0.1 (3)	C5—C6—C7—N1	−78.54 (19)
F4—C4—C5—C6	178.75 (15)	C7—N1—C8—C9^{i}	133.86 (15)
C3—C4—C5—C6	−0.3 (3)	C9—N1—C8—C9^{i}	−54.65 (19)
F1—C1—C6—C5	179.64 (15)	C7—N1—C9—C8^{i}	−134.44 (15)
C2—C1—C6—C5	0.3 (3)	C8—N1—C9—C8^{i}	54.55 (19)

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9A···O1^{ii}	0.97	2.55	3.161 (2)	121

Symmetry codes: (ii) *x*, *y*, *z*+1.

Fig. 1

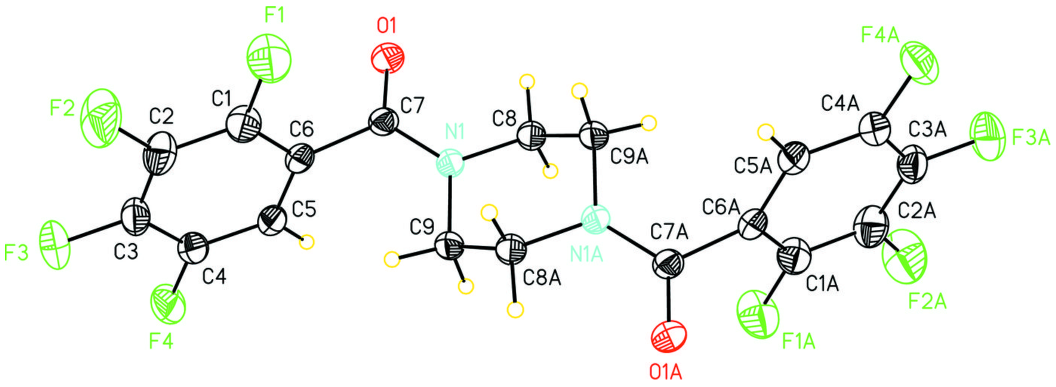
F1
O1
F4A
F2
C2
C1
C6
C7
N1
C8
C9A
C4A
C3A
F3A
C5A
C6A
C7A
C2A
C5
C9
N1A
F3
C3
C4
C8A
C1A
F2A
F4
F1A
O1A

Fig. 2