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N,*N*′-(Pyridine-2,6-diyl)bis(naphthalenedicarboximide) trifluoroacetic acid monohydrate

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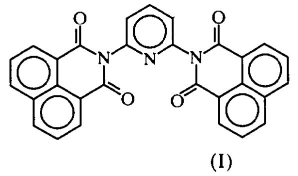
Abstract

The asymmetric unit of the title compound, $C_{29}H_{15}N_3O_4{\cdot}C_2HF_3O_2{\cdot}H_2O$, contains two independent molecules (*A* and *B*) of *N*,*N*′-(pyridine-2,6-diyl)bis(naphthalenedicarboximide), two trifluoroacetic acid molecules and two water molecules. Molecules *A* and *B* differ in the conformations of their imide fragments and in the rotation of the naphthoyleneimide moieties with respect to the pyridine plane. In molecule *A*, the imide rings are planar, while in *B*, the N atoms deviate from the least-squares planes through the other ring atoms by 0.10 and 0.09 Å.

Comment

Recently, it has been found that bis-derivatives of naphthalene-1,8-dicarboximide may be used as potential antitumour (Cherney *et al.*, 1997; Brana *et al.*, 1995, 1996) and antiviral (Hayes *et al.*, 1996) agents. This fact confers great interest on the molecular structures of such compounds. However, according to the Cambridge Structural Database (Allen & Kennard, 1993), the necessary structural information is not available. Therefore, we carried out an X-ray diffraction study of the the title compound, (I).



$\cdot C_2HF_3O_2{\cdot}H_2O$

(I)

The asymmetric unit contains two molecules (*A* and *B*) of *N*,*N*′-(pyridine-2,6-diyl)bis(naphthalenedicarboximide), two trifluoroacetic acid (TFAA) molecules and two water molecules. The imide fragments in molecule *A* are planar, as found for other *N*-aryl-substituted derivatives of naphthalene-1,8-dicarboxoimide (Batchelor *et al.*, 1997; Lindeman *et al.*, 1995; Dromzee *et al.*, 1995). However, the corresponding rings in molecule *B* are slightly non-planar; the deviations of N1*C* and N1*D* from the least-squares mean plane of the other ring atoms are 0.10 and 0.09 Å, respectively. Similar results were obtained for the *N*-ethyl derivative (Easton *et al.*, 1992). The non-planar geometry of the imide rings in molecule *B* may be explained by the high conformational flexibility of this fragment (Shishkin & Ponomarev, 1997). Therefore, intermolecular interactions in the crystal may easily deform the conformation of these rings. Molecules *A* and *B* differ also in the rotation angles of their naphthaleneimide fragments with respect to the pyridine planes, the torsion angles being C9*A*—N1*A*—C11*A*—C12*A* −79.6 (5) and C9*B*—N1*B*—C15*A*—N2*AB* −65.6 (4)° in molecule *A*, and C9*C*—N1*C*—C11*C*—C12*C* −85.4 (5) and C9*D*—N1*D*—C15*C*—C14*C* −68.0 (5)° in molecule *B*. Molecules of *N*,*N*′-(pyridine-2,6-diyl)bis(naphthalenedicarboximide), water and TFAA form a three-dimensional network of hydrogen bonds (Table 1). It should be noted that the presence of the H atom on one O atom of each TFAA molecule indicates that the acid exists in molecular form in the crystal, despite the formation of strong hydrogen bonds with water molecules.

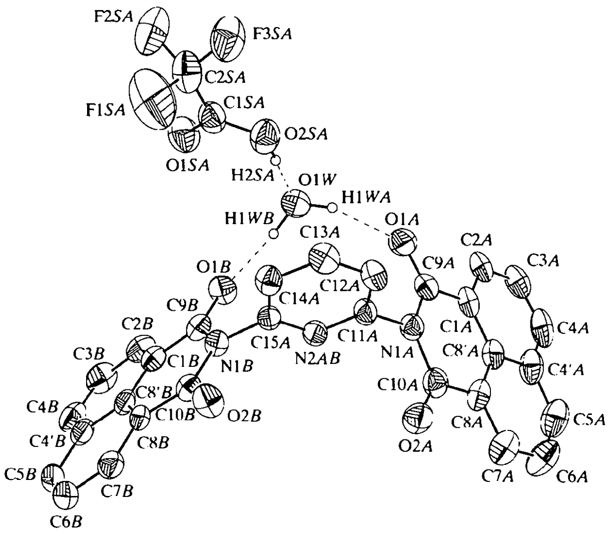


Fig. 1. A view of (I) with displacement ellipsoids drawn at the 50% probability level. Excepting those of the H_2O and TFAA molecules, H atoms have been omitted for clarity.

Experimental

Crystal data

$C_{29}H_{15}N_3O_4{\cdot}C_2HF_3O_2{\cdot}H_2O$
$M_r = 601.49$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Triclinic
$P\bar{1}$
$a = 12.984\,(7)$ Å
$b = 13.034\,(5)$ Å
$c = 16.749\,(8)$ Å
$\alpha = 82.04\,(3)°$
$\beta = 72.31\,(4)°$
$\gamma = 87.45\,(4)°$
$V = 2674\,(2)$ Å^3
$Z = 4$
$D_x = 1.494$ Mg m^{-3}
D_m not measured

Cell parameters from 22 reflections
$\theta = 10–11°$
$\mu = 0.121$ mm^{-1}
$T = 213\,(2)$ K
Parallelepiped
0.30 × 0.30 × 0.20 mm
Yellow

Data collection

Syntex $P2_1$/PC diffractometer
ω scans
Absorption correction: none
9781 measured reflections
9325 independent reflections
4276 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.074$
$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -18 \rightarrow 19$
2 standard reflections every 98 reflections intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.178$
$S = 1.154$
9322 reflections
793 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.092P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.027$
$\Delta\rho_{\rm max} = 0.81$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.45$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*—H···*A*
O1*W*—H1*WA*···O1*A*$^{\rm i}$	1.90	158
O1*W*—H1*WB*···O1*B*$^{\rm i}$	1.86	159
O2*W*—H2*WA*···O1*C*	1.76	173
O2*W*—H2*WB*···O2*D*	1.95	168
O2*SA*—H2*SA*···O1*W*	1.74	176
O2*SB*—H2*SB*···O2*W*	1.68	172

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

Data collection: *P*3 (Siemens, 1989*a*). Cell refinement: *P*3. Data reduction: *XDISK* (Siemens, 1989*b*). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1342). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.

Batchelor, R. A., Hunter, C. A. & Simpson, J. (1997). *Acta Cryst.* C**53**, 1117–1119.

Brana, M. F., Castellano, J. M., Moran, M., Devega, M. J. P., Perron, D., Conlon, D., Bousquet, P. F., Romerdahl, C. A. & Robinson, S. P. (1996). *Anticancer Drug Des.* **11**, 297–309.

Brana, M. F., Castellano, J. M., Moran, M., Devega, M. J. P., Qian, V. D., Romerdahl, C. A. & Keilhauer, G. (1995). *Eur. J. Med. Chem.* **30**, 235–239.

Cherney, R. J., Swartz, S. G., Patten, A. D., Akamike, E. & Sun, J. H. (1997). *Bioorg. Med. Chem. Lett.* **7**, 163–168.

Dromzee, Y., Kossanyi, J., Wintgens, V. & Valat, P. (1995). *Z. Kristallogr.* **210**, 760–764.

Easton, C. J., Gulbis, J. M., Hoskins, B. F., Scharfbillig, I. M. & Tiekink, E. R. T. (1992). *Z. Kristallogr.* **199**, 249–254.

Hayes, B. A., Gupta, S., Chang, S. C., Utecht, R. E. & Lewis, D. E. (1996). *J. Labelled Compd Radiopharm.* **38**, 607–612.

Lindeman, S. V., Ponomarev, I. I. & Rusanov, A. L. (1995). *Acta Cryst.* C**51**, 2157–2161.

Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5.02. *Program Package for Crystal Structure Solution and Refinement*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Shishkin, O. V. & Ponomarev, I. I. (1997). *Russ. Chem. Bull.* **46**, 59–61.

Siemens (1989*a*). *P3. Program for Data Collection*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1989*b*). *XDISK. Data Reduction Program*. Version 3.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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2,3-Dihydro-3-methyl-2′,4′,6′-trinitrospiro[1,3-thiazolotropylium-2,1′-cyclohexadienide]

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Abstract

In molecules of the title compound, $C_{14}H_{10}N_4O_6S$, the geometry of the six-membered ring is typical for Meisenheimer complexes of 2,4,6-trinitrobenzene. However, the difference between the C—N bond lengths of the nitro groups *para* and *ortho* to the saturated C atom is less pronounced than in related compounds. There is significant conjugation between the seven-membered