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3,3′-(1,3,5,7-Tetraoxo-2,3,6,7-tetrahydro-1*H*,5*H*-pyrrolo[3,4-*f*]isoindole-2,6-diyl)dipropanoic acid *N*,*N*-dimethylformamide disolvate

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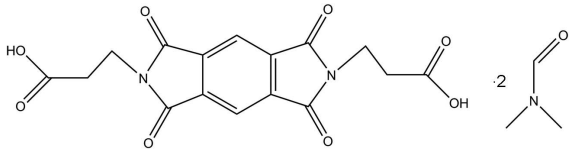
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.133; data-to-parameter ratio = 14.3.

In the title compound, $C_{16}H_{12}N_2O_8\cdot 2C_3H_7NO$, the complete tricyclic compound is generated by a crystallographic centre of symmetry. In the crystal, the tricycle is linked to two adjacent *N*,*N*-dimethylformamide solvent molecules by O—H···O hydrogen bonds.

Related literature

For a related structure and background, see: Wang & Wei (2005).



Experimental

Crystal data

$C_{16}H_{12}N_2O_8\cdot 2C_3H_7NO$
M_r = 506.47
Monoclinic, $P2_1/c$
a = 12.542 (8) Å
b = 8.611 (6) Å
c = 12.902 (9) Å
β = 118.774 (8)°
V = 1221.3 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.33 × 0.31 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
12363 measured reflections
2386 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 1.06
2386 reflections
167 parameters
15 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O5^{i}	0.82	1.78	2.583 (3)	166

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{3}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5179).

References

Bruker (2001). *SAINT-Plus* and *SMART*. Bruker AXS, Inc., Madison, Wisconsin, USA.
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Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Wang, Z.-L. & Wei, L.-H. (2005). *Acta Cryst.* E**61**, o3129–o3130.

supporting information

Acta Cryst. (2009). E**65**, o2975 [doi:10.1107/S1600536809045437]

3,3′-(1,3,5,7-Tetraoxo-2,3,6,7-tetrahydro-1*H*,5*H*-pyrrolo[3,4-*f*]isoindole-2,6-diyl)dipropanoic acid *N*,*N*-dimethylformamide disolvate

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S1. Experimental

3-Bromopropanoic acid (2 mmol, 0.306 g) and pyrrolo[3,4-*f*]isoindole-1,3,5,7(2*H*,6H)-tetraone (1 mmol, 0.360 g) were dissolved in 20 ml of the mixed solvent of *N*,*N*-dimethylformamide and water in a ratio of 1:2 (v/v). The mixture was heated in a Teflon-lined steel autoclave inside a programmable electric furnace at 353 K for three days. After cooling the autoclave to room temperature, colourless slabs of (I) were obtained.

S2. Refinement

H9A aom was located by Fourier map, other H atoms were geometrically placed with C—H = 0.93–0.97 Å and O—H = 0.82 Å, and were refined as riding with $U_{iso}(H)$=1.2U_{eq}($C_{methylene}$ and C in phenyl ring) and U_{iso}(H)=1.5U_{eq}(O and C_{methyl}).

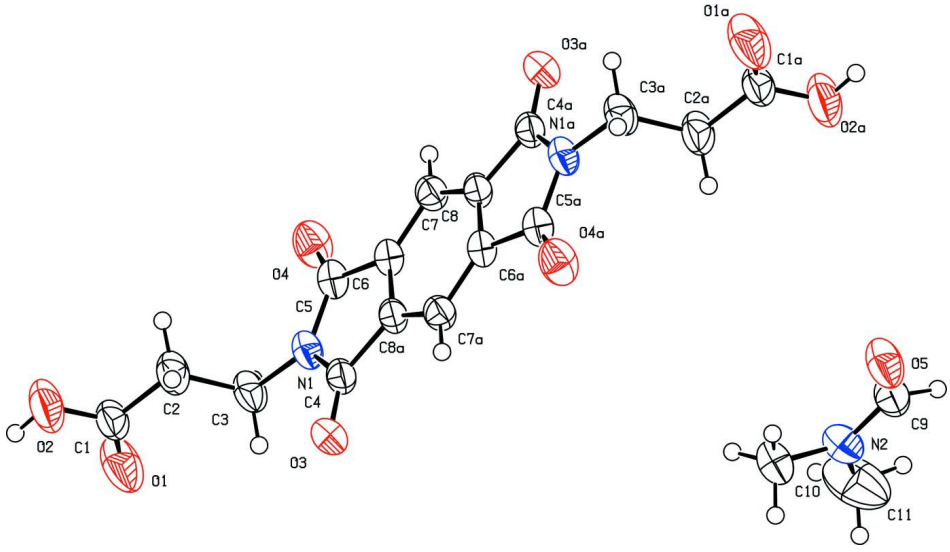

Figure 1

The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level.

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Crystal data

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a = 12.542 (8) Å
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c = 12.902 (9) Å
β = 118.774 (8)°
V = 1221.3 (14) Å^3
Z = 2
F(000) = 532
D_x = 1.377 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 2660 reflections
θ = 3.0–23.7°
μ = 0.11 mm^{-1}
T = 296 K
Slab, colourless
0.33 × 0.31 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
12363 measured reflections
2386 independent reflections

1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.0°, θ_{min} = 1.9°
h = −15→15
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 1.06
2386 reflections
167 parameters
15 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.5597P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
O1	0.2286 (2)	0.3041 (3)	−0.14930 (17)	0.0847 (7)
O2	0.3228 (2)	0.5093 (3)	−0.16182 (17)	0.0896 (8)
H2	0.2861	0.4876	−0.2324	0.134*
O3	0.22033 (16)	0.5402 (2)	0.20189 (14)	0.0502 (5)
O4	0.55406 (18)	0.2430 (2)	0.29251 (15)	0.0579 (5)
O5	0.2066 (2)	1.0088 (2)	1.11020 (15)	0.0651 (6)
C1	0.2949 (2)	0.4100 (3)	−0.1032 (2)	0.0487 (6)
C2	0.3578 (3)	0.4407 (4)	0.0263 (2)	0.0575 (7)
H2B	0.3407	0.5465	0.0395	0.069*
H2C	0.4449	0.4321	0.0564	0.069*

C3	0.3212 (3)	0.3327 (3)	0.09466 (19)	0.0479 (6)
H3A	0.2332	0.3320	0.0597	0.057*
H3B	0.3475	0.2281	0.0903	0.057*
C4	0.3193 (2)	0.4830 (3)	0.26018 (19)	0.0388 (5)
C5	0.4876 (2)	0.3319 (3)	0.30588 (19)	0.0403 (5)
C6	0.5071 (2)	0.4122 (3)	0.41634 (19)	0.0366 (5)
C7	0.6041 (2)	0.4039 (3)	0.5291 (2)	0.0396 (5)
H7A	0.6714	0.3412	0.5482	0.048*
C8	0.5939 (2)	0.4955 (3)	0.61186 (18)	0.0368 (5)
C9	0.1607 (3)	0.8835 (4)	1.0674 (2)	0.0522 (7)
H9A	0.170 (3)	0.794 (4)	1.119 (3)	0.066 (8)*
C10	0.0679 (3)	0.9775 (4)	0.8676 (3)	0.0717 (9)
H10A	0.1090	1.0710	0.9071	0.108*
H10B	−0.0182	0.9958	0.8254	0.108*
H10C	0.0955	0.9462	0.8131	0.108*
C11	0.0398 (4)	0.7052 (5)	0.9101 (4)	0.1117 (16)
H11A	0.0643	0.6347	0.9753	0.168*
H11B	0.0663	0.6662	0.8566	0.168*
H11C	−0.0472	0.7148	0.8695	0.168*
N1	0.37472 (18)	0.3802 (2)	0.21817 (16)	0.0416 (5)
N2	0.0942 (2)	0.8560 (3)	0.95392 (19)	0.0558 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1149 (18)	0.0799 (15)	0.0394 (10)	−0.0332 (14)	0.0212 (11)	−0.0087 (10)
O2	0.1066 (17)	0.1158 (18)	0.0353 (10)	−0.0460 (15)	0.0254 (11)	0.0011 (11)
O3	0.0456 (10)	0.0599 (11)	0.0371 (9)	0.0041 (9)	0.0135 (8)	0.0081 (8)
O4	0.0728 (13)	0.0580 (11)	0.0467 (10)	0.0166 (10)	0.0319 (10)	−0.0008 (8)
O5	0.0889 (15)	0.0617 (12)	0.0383 (10)	−0.0119 (11)	0.0253 (10)	−0.0033 (9)
C1	0.0564 (14)	0.0563 (14)	0.0308 (11)	−0.0061 (12)	0.0190 (11)	−0.0015 (10)
C2	0.0695 (18)	0.0686 (18)	0.0319 (13)	−0.0228 (15)	0.0224 (13)	−0.0066 (12)
C3	0.0612 (16)	0.0484 (14)	0.0290 (12)	−0.0080 (12)	0.0178 (11)	−0.0050 (10)
C4	0.0435 (14)	0.0410 (13)	0.0314 (11)	−0.0031 (11)	0.0176 (11)	0.0046 (10)
C5	0.0514 (14)	0.0383 (12)	0.0341 (12)	0.0009 (11)	0.0228 (11)	0.0013 (9)
C6	0.0452 (13)	0.0361 (12)	0.0317 (11)	0.0017 (10)	0.0211 (10)	0.0030 (9)
C7	0.0409 (13)	0.0434 (13)	0.0352 (12)	0.0071 (10)	0.0188 (10)	0.0046 (10)
C8	0.0417 (13)	0.0408 (12)	0.0287 (11)	0.0005 (10)	0.0176 (10)	0.0044 (9)
C9	0.0573 (17)	0.0564 (17)	0.0404 (14)	0.0010 (13)	0.0215 (13)	0.0058 (13)
C10	0.0654 (19)	0.104 (3)	0.0398 (15)	0.0021 (18)	0.0207 (14)	0.0130 (15)
C11	0.110 (3)	0.078 (3)	0.088 (3)	−0.016 (2)	0.001 (2)	−0.017 (2)
N1	0.0513 (12)	0.0442 (11)	0.0278 (9)	−0.0014 (9)	0.0180 (9)	−0.0002 (8)
N2	0.0534 (13)	0.0621 (14)	0.0408 (12)	−0.0010 (11)	0.0139 (10)	−0.0024 (10)

Geometric parameters (Å, º)

O1—C1	1.184 (3)	C6—C7	1.378 (3)
O2—C1	1.296 (3)	C6—C8^{i}	1.387 (3)

O2—H2	0.8200	C7—C8	1.382 (3)
O3—C4	1.204 (3)	C7—H7A	0.9300
O4—C5	1.203 (3)	C8—C6^{i}	1.387 (3)
O5—C9	1.222 (3)	C8—C4^{i}	1.488 (3)
C1—C2	1.488 (3)	C9—N2	1.311 (3)
C2—C3	1.498 (4)	C9—H9A	0.99 (3)
C2—H2B	0.9700	C10—N2	1.445 (4)
C2—H2C	0.9700	C10—H10A	0.9600
C3—N1	1.458 (3)	C10—H10B	0.9600
C3—H3A	0.9700	C10—H10C	0.9600
C3—H3B	0.9700	C11—N2	1.449 (4)
C4—N1	1.387 (3)	C11—H11A	0.9600
C4—C8^{i}	1.488 (3)	C11—H11B	0.9600
C5—N1	1.384 (3)	C11—H11C	0.9600
C5—C6	1.495 (3)		

C1—O2—H2	109.5	C6—C7—H7A	122.5
O1—C1—O2	122.5 (2)	C8—C7—H7A	122.5
O1—C1—C2	124.4 (2)	C7—C8—C6^{i}	122.4 (2)
O2—C1—C2	113.1 (2)	C7—C8—C4^{i}	129.6 (2)
C1—C2—C3	113.8 (2)	C6^{i}—C8—C4^{i}	107.94 (19)
C1—C2—H2B	108.8	O5—C9—N2	124.9 (3)
C3—C2—H2B	108.8	O5—C9—H9A	120.7 (17)
C1—C2—H2C	108.8	N2—C9—H9A	114.4 (18)
C3—C2—H2C	108.8	N2—C10—H10A	109.5
H2B—C2—H2C	107.7	N2—C10—H10B	109.5
N1—C3—C2	111.0 (2)	H10A—C10—H10B	109.5
N1—C3—H3A	109.4	N2—C10—H10C	109.5
C2—C3—H3A	109.4	H10A—C10—H10C	109.5
N1—C3—H3B	109.4	H10B—C10—H10C	109.5
C2—C3—H3B	109.4	N2—C11—H11A	109.5
H3A—C3—H3B	108.0	N2—C11—H11B	109.5
O3—C4—N1	125.2 (2)	H11A—C11—H11B	109.5
O3—C4—C8^{i}	128.8 (2)	N2—C11—H11C	109.5
N1—C4—C8^{i}	106.0 (2)	H11A—C11—H11C	109.5
O4—C5—N1	125.5 (2)	H11B—C11—H11C	109.5
O4—C5—C6	128.6 (2)	C5—N1—C4	112.35 (19)
N1—C5—C6	105.9 (2)	C5—N1—C3	124.1 (2)
C7—C6—C8^{i}	122.5 (2)	C4—N1—C3	123.5 (2)
C7—C6—C5	129.7 (2)	C9—N2—C10	121.1 (3)
C8^{i}—C6—C5	107.8 (2)	C9—N2—C11	121.6 (3)
C6—C7—C8	115.0 (2)	C10—N2—C11	117.3 (3)

O1—C1—C2—C3	−5.3 (4)	C6—C5—N1—C4	0.0 (3)
O2—C1—C2—C3	176.6 (3)	O4—C5—N1—C3	−1.9 (4)
C1—C2—C3—N1	−173.2 (2)	C6—C5—N1—C3	178.2 (2)
O4—C5—C6—C7	−0.5 (4)	O3—C4—N1—C5	−179.1 (2)
N1—C5—C6—C7	179.4 (2)	C8^{i}—C4—N1—C5	0.6 (2)

O4—C5—C6—C8^{i}	179.4 (2)	O3—C4—N1—C3	2.7 (4)
N1—C5—C6—C8^{i}	−0.7 (2)	C8^{i}—C4—N1—C3	−177.6 (2)
C8^{i}—C6—C7—C8	−0.7 (4)	C2—C3—N1—C5	−88.7 (3)
C5—C6—C7—C8	179.2 (2)	C2—C3—N1—C4	89.2 (3)
C6—C7—C8—C6^{i}	0.7 (4)	O5—C9—N2—C10	−0.8 (5)
C6—C7—C8—C4^{i}	−179.0 (2)	O5—C9—N2—C11	−178.1 (4)
O4—C5—N1—C4	180.0 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O5^{ii}	0.82	1.78	2.583 (3)	166

Symmetry code: (ii) *x*, −*y*+3/2, *z*−3/2.