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Biphenyl-4,4′-dicarboxylic acid *N*,*N*-dimethylformamide monosolvate

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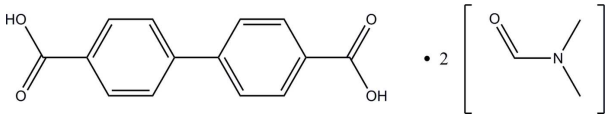
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.175; data-to-parameter ratio = 16.6.

Biphenyl-4,4′-dicarboxylic acid was recrystallized from *N*,*N*-dimethylformamide (DMF) yielding the title compound, $C_{14}H_{10}O_4\cdot 2C_3H_7NO$. The acid molecules are located on crystallographic centres of inversion and are hydrogen bonded to DMF molecules. These hydrogen-bonded units form infinite chains although there is no interaction between the methyl groups of neighboring DMF molecules.

Related literature

The title compound is a popular linker for the synthesis of metal-organic framework materials, for example IRMOF 10 (Eddaoudi *et al.*, 2002) and UIO-67 (Cavka *et al.*, 2008).



Experimental

Crystal data

$C_{14}H_{10}O_4\cdot 2C_3H_7NO$
M_r = 388.41
Triclinic, $P\overline{1}$
a = 7.666 (7) Å
b = 7.774 (7) Å
c = 9.099 (8) Å
α = 88.549 (10)°
β = 73.596 (10)°
γ = 65.208 (7)°
V = 469.6 (7) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 150 K
0.2 × 0.2 × 0.1 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.980, T_{max} = 0.990
3968 measured reflections
2136 independent reflections
1635 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.175
S = 1.11
2136 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O3^{i}	0.82	1.76	2.575 (2)	172

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97* and *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5277).

References

Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cavka, J. H., Jakobsen, S., Olsbye, U., Guillou, N., Lamberti, C., Bordiga, S. & Lillerud, K. P. (2008). *J. Am. Chem. Soc.* **130**, 13850–13851.
Eddaoudi, M., Kim, J., Rosi, N. L., Vodak, B. T., Wachter, J., O'Keeffe, M. & Yaghi, O. M. (2002). *Science*, **295**, 469–472.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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Biphenyl-4,4′-dicarboxylic acid *N*,*N*-dimethylformamide monosolvate

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S1. Comment

The title compound, (I) (Fig. 1), which is a popular linker for the synthesis of metal-organic framework materials, for example IRMOF 10 (Eddaoudi *et al.*, 2002) and UIO-67 (Cavka *et al.*, 2008), comprises units of one biphenyl-4,4′-dicarboxylic acid molecule hydrogen bonded to two DMF molecules *via* O—H···O links. These units pack as chains (Fig. 2), although there is no interaction between the methyl groups of neighboring DMF molecules. The chains are arranged in layers with no stacking interactions between the benzene rings (Fig. 3).

S2. Experimental

Biphenyl-4,4′-dicarboxylic acid and *N*,*N*-Dimethylformamide (DMF) were purchased from Sigma-Aldrich and used without further purification. 1.0 g Biphenyl-4,4'dicarboxylic acid was suspended in 100 ml DMF and heated to 100°C. DMF was added in small portions until the acid had just dissolved (app. 50 ml) and the solution left in aluminium foil over night for slow cool-down to RT. Filtration of the now 125 ml DMF suspension yielded 0.57 g white powder of Biphenyl-4,4-dicarboxylic acid after drying under vacuum. The mother liquor was placed at 5°C over night which gave a small amount of colourless crystals, which gave the structure presented here.

S3. Refinement

Hydrogen atoms were placed in ideal positions and refined with a riding model with C-H = 0.93Å and U(H)=1.2U_{eq}(C) or with C-H = 0.96Å and U(H)=1.5U_{eq}(C_{methyl}).

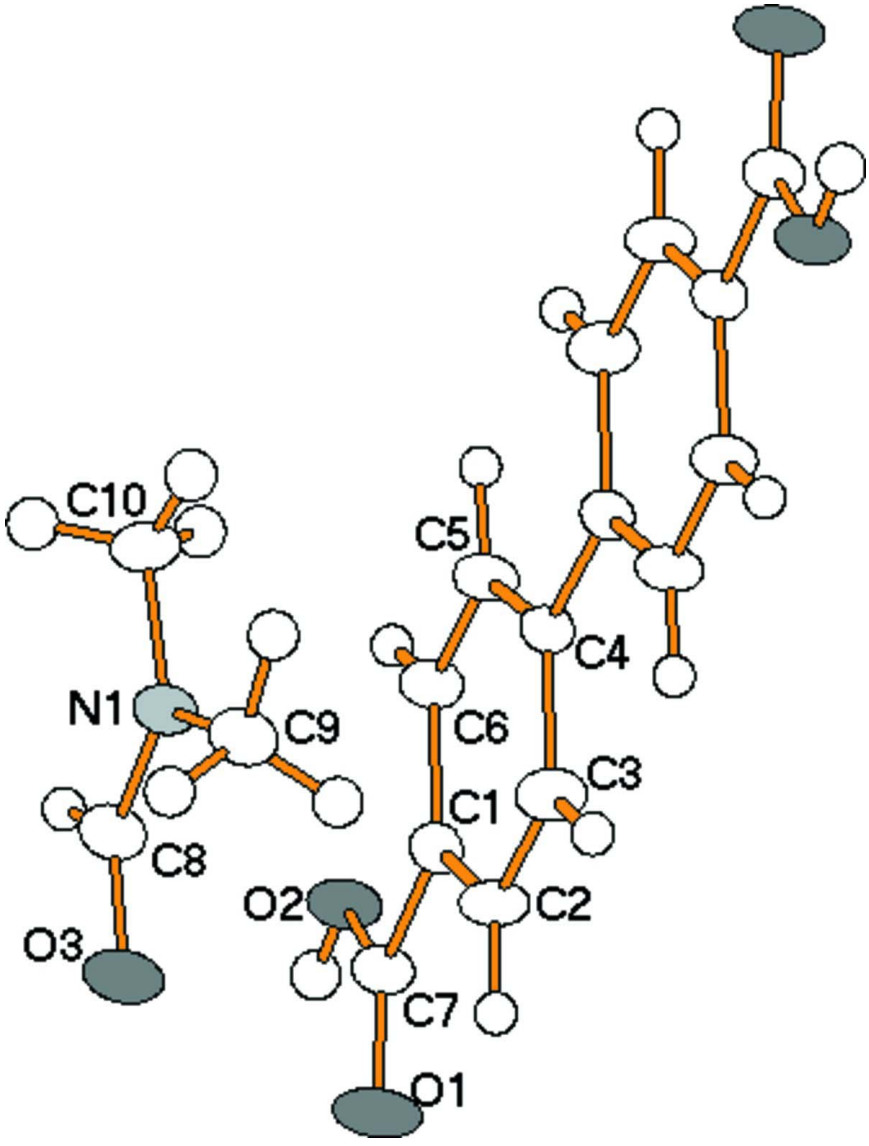

Figure 1

The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. Unlabeled atoms are related to the labeled ones by an inversion centre.

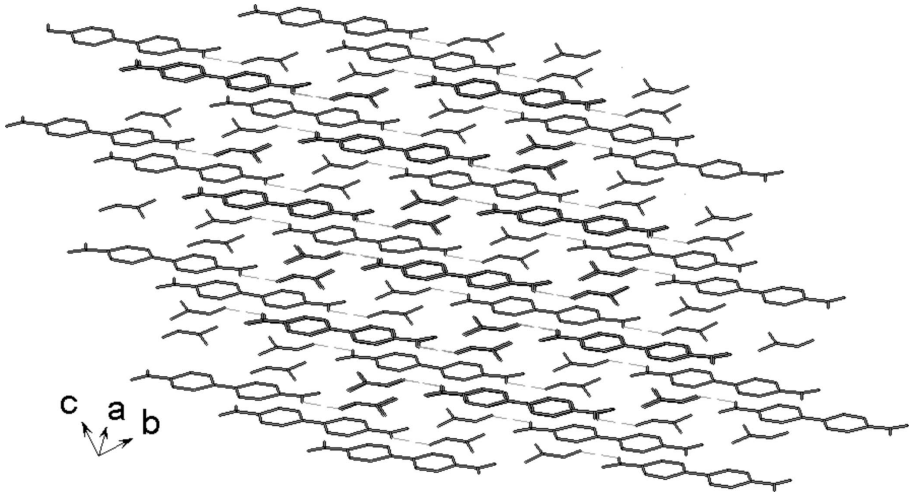

Figure 2

The packing of (I), showing the hydrogen bonded chains. Hydrogen atoms are omitted and hydrogen bonds are shown as dashed lines.

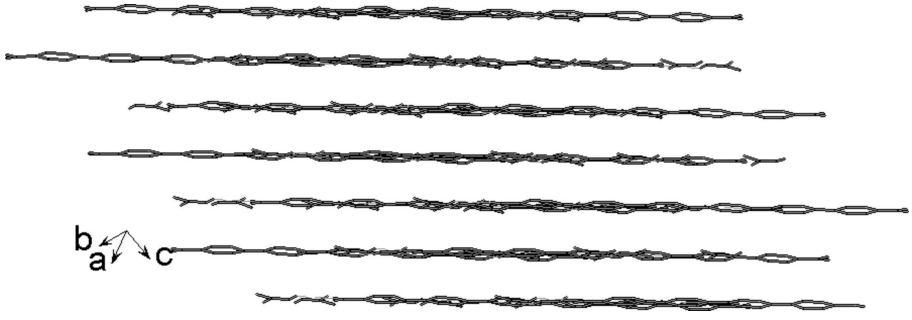

Figure 3

The packing of (I), showing the layers formed by the chains. Hydrogen atoms are omitted and hydrogen bonds are shown as dashed lines.

Biphenyl-4,4′-dicarboxylic acid *N*,*N*-dimethylformamide monosolvate

Crystal data

$C_{14}H_{10}O_4 \cdot 2C_3H_7NO$
$M_r = 388.41$
Triclinic, $P\overline{1}$
$a = 7.666\ (7)$ Å
$b = 7.774\ (7)$ Å
$c = 9.099\ (8)$ Å
$\alpha = 88.549\ (10)°$
$\beta = 73.596\ (10)°$
$\gamma = 65.208\ (7)°$
$V = 469.6\ (7)$ Å^3
$Z = 1$
$F(000) = 206$
$D_x = 1.374$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1412 reflections
$\theta = 2.4–28.2°$
$\mu = 0.10$ mm^{-1}
$T = 150$ K
Prism, colourless
0.2 × 0.2 × 0.1 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.980, T_{max} = 0.990
3968 measured reflections
2136 independent reflections
1635 reflections with $I > 2\sigma(I)$

R_{int} = 0.015
θ_{max} = 28.8°, θ_{min} = 2.9°
h = −9→10
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.175
S = 1.11
2136 reflections
129 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1177P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1394 (2)	0.7816 (2)	0.21617 (19)	0.0217 (4)
C2	−0.0126 (3)	0.8677 (2)	0.3535 (2)	0.0260 (4)
H2	−0.0795	0.9999	0.3711	0.031*
C3	−0.0653 (3)	0.7578 (2)	0.4645 (2)	0.0265 (4)
H3	−0.1653	0.8178	0.5567	0.032*
C4	0.0289 (2)	0.5590 (2)	0.44054 (18)	0.0208 (4)
C5	0.1794 (3)	0.4743 (2)	0.30105 (19)	0.0252 (4)
H5	0.2442	0.3421	0.2816	0.030*
C6	0.2336 (2)	0.5847 (2)	0.1908 (2)	0.0251 (4)
H6	0.3344	0.5255	0.0989	0.030*
C7	0.1957 (2)	0.9059 (2)	0.10229 (19)	0.0240 (4)
O1	0.1055 (2)	1.07728 (18)	0.11935 (16)	0.0363 (4)
O2	0.35493 (18)	0.80786 (17)	−0.01629 (14)	0.0282 (3)
H2A	0.3804	0.8814	−0.0753	0.042*
O3	0.44518 (19)	0.01439 (18)	0.77730 (15)	0.0333 (4)
N1	0.3688 (2)	0.3198 (2)	0.72336 (17)	0.0249 (4)
C8	0.3490 (3)	0.1893 (2)	0.8138 (2)	0.0267 (4)
H8	0.2566	0.2312	0.9116	0.032*
C9	0.5089 (3)	0.2649 (3)	0.5689 (2)	0.0292 (4)

H9A	0.4432	0.3379	0.4976	0.044*
H9B	0.5531	0.1317	0.5410	0.044*
H9C	0.6229	0.2888	0.5657	0.044*
C10	0.2551 (3)	0.5215 (2)	0.7753 (2)	0.0304 (4)
H10A	0.1787	0.5833	0.7065	0.046*
H10B	0.3461	0.5763	0.7765	0.046*
H10C	0.1653	0.5386	0.8773	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0211 (8)	0.0194 (8)	0.0226 (8)	−0.0075 (6)	−0.0056 (6)	0.0033 (6)
C2	0.0264 (8)	0.0157 (8)	0.0281 (9)	−0.0054 (6)	−0.0020 (7)	0.0009 (6)
C3	0.0262 (8)	0.0204 (8)	0.0242 (8)	−0.0076 (6)	0.0015 (6)	−0.0006 (6)
C4	0.0199 (8)	0.0187 (8)	0.0218 (8)	−0.0066 (6)	−0.0061 (6)	0.0033 (6)
C5	0.0274 (8)	0.0155 (8)	0.0243 (8)	−0.0045 (6)	−0.0029 (7)	0.0016 (6)
C6	0.0267 (8)	0.0178 (8)	0.0224 (8)	−0.0054 (6)	−0.0012 (6)	0.0010 (6)
C7	0.0244 (8)	0.0186 (8)	0.0247 (8)	−0.0069 (6)	−0.0047 (6)	0.0029 (6)
O1	0.0389 (8)	0.0173 (7)	0.0360 (8)	−0.0057 (6)	0.0031 (6)	0.0042 (5)
O2	0.0307 (7)	0.0185 (6)	0.0252 (7)	−0.0071 (5)	0.0006 (5)	0.0045 (5)
O3	0.0358 (7)	0.0216 (7)	0.0326 (7)	−0.0083 (5)	−0.0024 (6)	0.0065 (5)
N1	0.0260 (7)	0.0190 (7)	0.0265 (7)	−0.0084 (6)	−0.0053 (6)	0.0036 (6)
C8	0.0254 (8)	0.0238 (9)	0.0260 (9)	−0.0080 (7)	−0.0047 (7)	0.0051 (6)
C9	0.0308 (9)	0.0251 (9)	0.0274 (9)	−0.0108 (7)	−0.0045 (7)	0.0061 (7)
C10	0.0342 (9)	0.0199 (9)	0.0342 (9)	−0.0095 (7)	−0.0090 (7)	−0.0001 (7)

Geometric parameters (Å, º)

C1—C6	1.383 (3)	C7—O2	1.324 (2)
C1—C2	1.390 (2)	O2—H2A	0.8200
C1—C7	1.497 (2)	O3—C8	1.244 (2)
C2—C3	1.389 (3)	N1—C8	1.321 (2)
C2—H2	0.9300	N1—C10	1.449 (2)
C3—C4	1.396 (3)	N1—C9	1.451 (2)
C3—H3	0.9300	C8—H8	0.9300
C4—C5	1.398 (2)	C9—H9A	0.9600
C4—C4^{i}	1.493 (3)	C9—H9B	0.9600
C5—C6	1.392 (2)	C9—H9C	0.9600
C5—H5	0.9300	C10—H10A	0.9600
C6—H6	0.9300	C10—H10B	0.9600
C7—O1	1.206 (2)	C10—H10C	0.9600
C6—C1—C2	118.77 (15)	O2—C7—C1	112.84 (15)
C6—C1—C7	122.67 (15)	C7—O2—H2A	109.5
C2—C1—C7	118.56 (16)	C8—N1—C10	121.51 (15)
C3—C2—C1	120.52 (16)	C8—N1—C9	120.74 (15)
C3—C2—H2	119.7	C10—N1—C9	117.75 (14)
C1—C2—H2	119.7	O3—C8—N1	124.59 (17)

C2—C3—C4	121.27 (16)	O3—C8—H8	117.7
C2—C3—H3	119.4	N1—C8—H8	117.7
C4—C3—H3	119.4	N1—C9—H9A	109.5
C3—C4—C5	117.62 (14)	N1—C9—H9B	109.5
C3—C4—C4^{i}	121.26 (18)	H9A—C9—H9B	109.5
C5—C4—C4^{i}	121.12 (18)	N1—C9—H9C	109.5
C6—C5—C4	120.95 (15)	H9A—C9—H9C	109.5
C6—C5—H5	119.5	H9B—C9—H9C	109.5
C4—C5—H5	119.5	N1—C10—H10A	109.5
C1—C6—C5	120.84 (16)	N1—C10—H10B	109.5
C1—C6—H6	119.6	H10A—C10—H10B	109.5
C5—C6—H6	119.6	N1—C10—H10C	109.5
O1—C7—O2	124.29 (16)	H10A—C10—H10C	109.5
O1—C7—C1	122.87 (16)	H10B—C10—H10C	109.5
C6—C1—C2—C3	1.5 (3)	C7—C1—C6—C5	178.71 (15)
C7—C1—C2—C3	−177.92 (15)	C4—C5—C6—C1	−0.2 (3)
C1—C2—C3—C4	−1.4 (3)	C6—C1—C7—O1	175.06 (16)
C2—C3—C4—C5	0.4 (3)	C2—C1—C7—O1	−5.5 (3)
C2—C3—C4—C4^{i}	−179.51 (17)	C6—C1—C7—O2	−5.8 (2)
C3—C4—C5—C6	0.4 (3)	C2—C1—C7—O2	173.65 (15)
C4^{i}—C4—C5—C6	−179.67 (17)	C10—N1—C8—O3	−178.38 (16)
C2—C1—C6—C5	−0.7 (3)	C9—N1—C8—O3	0.5 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O3^{ii}	0.82	1.76	2.575 (2)	172

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