

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Benzene-1,4-dicarboxylic acid-*N,N*-dimethylacetamide (1/2)

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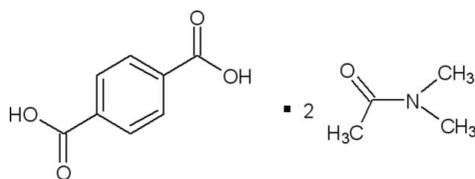
Received 8 June 2009; accepted 3 July 2009

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.118; data-to-parameter ratio = 13.1.

The asymmetric unit of title compound, $\text{C}_8\text{H}_6\text{O}_4 \cdot 2\text{C}_4\text{H}_9\text{NO}$, contains one half-molecule (an inversion centre in $P21/n$ generates the other half of the molecule) of terephthalic acid (TA) and one molecule of *N,N*-dimethylacetamide (DMAC). The DMAC molecules are linked to TA by strong $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For the crystal structure of terephthalic acid-bis(*N,N*-dimethylformamide), see: Dale & Elsegood (2004). For the polymorphism of terephthalic acid, see: Bailey & Brown (1967); Sledz *et al.* (2001).



Experimental

Crystal data

 $\text{C}_8\text{H}_6\text{O}_4 \cdot 2\text{C}_4\text{H}_9\text{NO}$ $M_r = 340.37$

Monoclinic, $P2_1/n$
 $a = 10.191$ (2) Å
 $b = 8.5228$ (17) Å
 $c = 10.719$ (2) Å
 $\beta = 110.67$ (3)°
 $V = 871.0$ (3) Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 113$ K
 $0.60 \times 0.51 \times 0.38$ mm

Data collection

Rigaku R-AXIS RAPID
diffractometer
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.940$, $T_{\max} = 0.961$

6605 measured reflections
1522 independent reflections
1395 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.118$
 $S = 1.08$
1522 reflections
116 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\max} = 0.58$ e Å⁻³
 $\Delta\rho_{\min} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H2} \cdots \text{O1}^i$	0.91 (3)	1.65 (3)	2.551 (2)	173 (2)

Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Xu Wei for the X-ray data collection and would like to express sincere thanks to Professor Li Xi for providing the study environment and helpful comments.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2216).

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supporting information

Acta Cryst. (2009). E65, o1794 [doi:10.1107/S1600536809025793]

Benzene-1,4-dicarboxylic acid–*N,N*-dimethylacetamide (1/2)

Xia Guo, Youwei Cheng and Xi Li

S1. Comment

Terephthalic acid (TA) is an important intermediate in the production of polyesters for plastics and fiber applications. According to Bailey & Brown (1967), TA exists in two polymorphic modifications (forms 1 and 2), both triclinic. Recently Sledz *et al.* (2001) reported a new crystalline form of TA which is monoclinic and designated as form 3.

N,N-Dimethylformamide (DMF) and *N,N*-dimethylacetamide (DMAC) are the two of a few organic solvents capable of dissolving TA. The crystal structure of the 2:1 DMF solvate of terephthalic acid was reported recently (Dale & Elsegood, 2004). The solvent molecules and TA form a centrosymmetric discrete planar assembly with both carboxylic acid groups hydrogen bonded to DMF molecules *via* $R_2^2(7)$ motif (O—H \cdots O/C—H \cdots O interactions). Recently we have obtained single crystals of the DMAC solvate of TA and here we report its crystal structure.

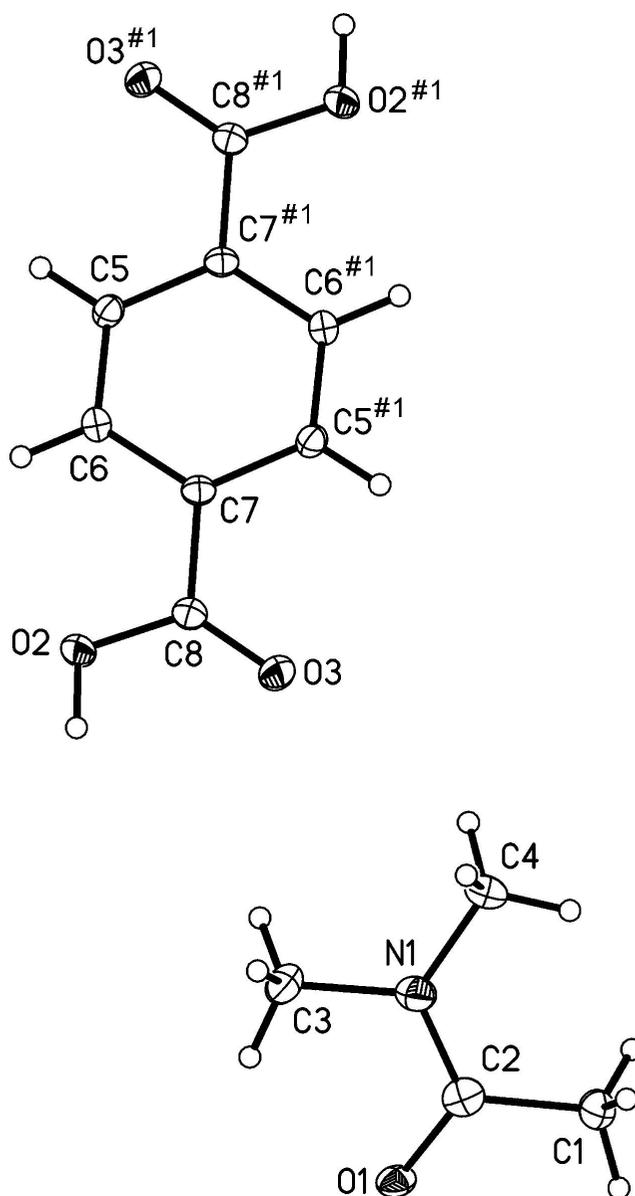
The asymmetric unit of title compound contains one half-molecule of TA and one *N,N*-dimethyl acetamide (DMAC) molecule (Fig. 2). The DMAC molecules are linked to TA by strong O—H \cdots O hydrogen bonds (Fig.3 and Table 1), which may be effective in stabilizing the crystal structure. The carboxylic group is roughly coplanar with the benzene ring forming dihedral angle of 0.6 (3) $^\circ$. The dihedral angle between TA and the dimethylacetamide molecule is 21.7 (1) $^\circ$.

S2. Experimental

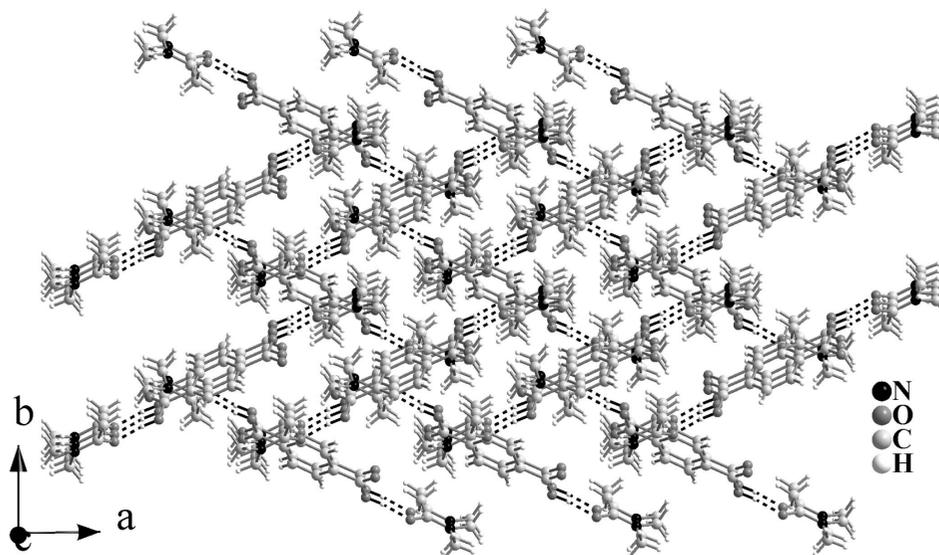
Single crystals were obtained by dissolving TA (1.0 g) in DMAC (20 ml) at 80 $^\circ$ C and then allowing the solvent to cool to room temperature. The sample proved unstable in the air.

S3. Refinement

The H atom of the carboxylic group was located from a difference Fourier map and fully refined. The remaining H atoms were placed in geometrically calculated positions and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

**Figure 1**

View of the title compound showing 50% probability displacement ellipsoids. Symmetry operation for atoms with '#': $-x, -y, -z + 1$.

**Figure 2**

The packing diagram for the title compound; dashed lines indicate hydrogen bonds.

Benzene-1,4-dicarboxylic acid-*N,N*-dimethylacetamide (1/2)

Crystal data

$C_8H_6O_4 \cdot 2C_4H_9NO$

$M_r = 340.37$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 10.191(2) \text{ \AA}$

$b = 8.5228(17) \text{ \AA}$

$c = 10.719(2) \text{ \AA}$

$\beta = 110.67(3)^\circ$

$V = 871.0(3) \text{ \AA}^3$

$Z = 2$

$F(000) = 364$

$D_x = 1.298 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 6873 reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 113 \text{ K}$

Block, colorless

$0.60 \times 0.51 \times 0.38 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 0 pixels mm^{-1}

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.940$, $T_{\max} = 0.961$

6605 measured reflections

1522 independent reflections

1395 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.1^\circ$

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 10$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.118$

$S = 1.08$

1522 reflections

116 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 atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.6867P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.58 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.26 \text{ e } \text{Å}^{-3}$$

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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.74204 (15)	0.07465 (18)	0.88639 (15)	0.0233 (4)
O1	0.95506 (11)	0.13078 (14)	0.88125 (11)	0.0207 (3)
O2	0.30574 (13)	-0.23616 (15)	0.52621 (12)	0.0248 (3)
O3	0.35578 (12)	-0.10972 (15)	0.72025 (11)	0.0237 (3)
C1	0.89721 (19)	0.2372 (2)	1.06449 (18)	0.0266 (4)
H1A	0.9097	0.1670	1.1378	0.040*
H1B	0.8203	0.3067	1.0557	0.040*
H1C	0.9813	0.2973	1.0806	0.040*
C2	0.86637 (18)	0.1442 (2)	0.93832 (17)	0.0224 (4)
C3	0.71443 (18)	-0.0191 (2)	0.76458 (17)	0.0241 (4)
H3A	0.7248	0.0458	0.6954	0.036*
H3B	0.6206	-0.0598	0.7363	0.036*
H3C	0.7799	-0.1046	0.7824	0.036*
C4	0.63337 (19)	0.0830 (3)	0.9455 (2)	0.0304 (5)
H4A	0.6764	0.0888	1.0408	0.046*
H4B	0.5755	-0.0090	0.9216	0.046*
H4C	0.5768	0.1746	0.9131	0.046*
C5	-0.08828 (17)	-0.03522 (19)	0.37140 (16)	0.0171 (4)
H5	-0.1477	-0.0586	0.2852	0.020*
C6	0.04270 (17)	-0.10394 (19)	0.42154 (16)	0.0175 (4)
H6A	0.0712	-0.1734	0.3692	0.021*
C7	0.13228 (16)	-0.06918 (18)	0.55081 (15)	0.0152 (4)
C8	0.27579 (16)	-0.13973 (18)	0.60876 (15)	0.0163 (4)
H2	0.394 (3)	-0.276 (3)	0.563 (2)	0.052 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0191 (8)	0.0254 (8)	0.0268 (8)	-0.0007 (6)	0.0097 (6)	0.0006 (6)
O1	0.0166 (6)	0.0255 (7)	0.0209 (6)	-0.0043 (5)	0.0080 (5)	0.0014 (5)
O2	0.0174 (7)	0.0324 (7)	0.0224 (7)	0.0083 (5)	0.0044 (5)	-0.0055 (5)
O3	0.0182 (6)	0.0282 (7)	0.0200 (6)	0.0047 (5)	0.0012 (5)	-0.0035 (5)

C1	0.0258 (9)	0.0256 (10)	0.0299 (10)	-0.0037 (7)	0.0117 (8)	-0.0079 (7)
C2	0.0227 (9)	0.0179 (9)	0.0248 (9)	0.0013 (7)	0.0060 (7)	0.0051 (7)
C3	0.0227 (9)	0.0265 (10)	0.0205 (9)	-0.0061 (7)	0.0047 (7)	-0.0023 (7)
C4	0.0192 (9)	0.0407 (11)	0.0344 (11)	-0.0024 (8)	0.0133 (8)	-0.0086 (8)
C5	0.0168 (8)	0.0189 (8)	0.0145 (8)	-0.0012 (6)	0.0043 (6)	-0.0011 (6)
C6	0.0187 (8)	0.0176 (8)	0.0171 (8)	0.0005 (6)	0.0076 (6)	-0.0018 (6)
C7	0.0140 (8)	0.0156 (8)	0.0170 (8)	-0.0014 (6)	0.0066 (6)	0.0022 (6)
C8	0.0164 (8)	0.0159 (8)	0.0175 (8)	-0.0009 (6)	0.0072 (6)	0.0017 (6)

Geometric parameters (Å, °)

N1—C2	1.330 (2)	C3—H3B	0.9600
N1—C4	1.459 (2)	C3—H3C	0.9600
N1—C3	1.471 (2)	C4—H4A	0.9600
O1—C2	1.263 (2)	C4—H4B	0.9600
O2—C8	1.320 (2)	C4—H4C	0.9600
O2—H2	0.91 (3)	C5—C6	1.381 (2)
O3—C8	1.213 (2)	C5—C7 ⁱ	1.397 (2)
C1—C2	1.502 (2)	C5—H5	0.9300
C1—H1A	0.9600	C6—C7	1.396 (2)
C1—H1B	0.9600	C6—H6A	0.9300
C1—H1C	0.9600	C7—C5 ⁱ	1.397 (2)
C3—H3A	0.9600	C7—C8	1.498 (2)
C2—N1—C4	123.61 (16)	N1—C4—H4A	109.5
C2—N1—C3	117.98 (15)	N1—C4—H4B	109.5
C4—N1—C3	118.39 (14)	H4A—C4—H4B	109.5
C8—O2—H2	111.4 (16)	N1—C4—H4C	109.5
C2—C1—H1A	109.5	H4A—C4—H4C	109.5
C2—C1—H1B	109.5	H4B—C4—H4C	109.5
H1A—C1—H1B	109.5	C6—C5—C7 ⁱ	120.51 (15)
C2—C1—H1C	109.5	C6—C5—H5	119.7
H1A—C1—H1C	109.5	C7 ⁱ —C5—H5	119.7
H1B—C1—H1C	109.5	C5—C6—C7	119.99 (15)
O1—C2—N1	119.73 (16)	C5—C6—H6A	120.0
O1—C2—C1	121.74 (15)	C7—C6—H6A	120.0
N1—C2—C1	118.53 (16)	C6—C7—C5 ⁱ	119.50 (15)
N1—C3—H3A	109.5	C6—C7—C8	121.80 (15)
N1—C3—H3B	109.5	C5 ⁱ —C7—C8	118.70 (14)
H3A—C3—H3B	109.5	O3—C8—O2	123.91 (15)
N1—C3—H3C	109.5	O3—C8—C7	122.83 (15)
H3A—C3—H3C	109.5	O2—C8—C7	113.26 (14)
H3B—C3—H3C	109.5		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O2—H2···O1 ⁱⁱ	0.91 (3)	1.65 (3)	2.551 (2)	173 (2)

Symmetry code: (ii) $-x+3/2, y-1/2, -z+3/2$.