organic compounds

A polymorph of terephthalaldehyde

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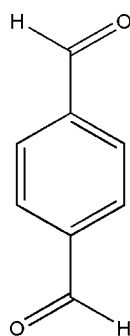Correspondence e-mail: chwangzg@yahoo.com.cn

A new orthorhombic polymorph of terephthalaldehyde, $C_8H_6O_2$, with a melting point of 372 K, has been obtained by recrystallization from ethanol. At room temperature, the crystals transform into the well known monoclinic form, with a melting point of 389 K. The crystal structure of the monoclinic form involves C—H···O hydrogen bonds, but no such bonds are observed in the orthorhombic form. The molecule is planar.

Related literature

For the structure of the monoclinic polymorph, see: Britton (1998).

Experimental

Crystal data

$C_8H_6O_2$	V = 668.01 (7) Å^3
M_r = 134.13	Z = 4
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation
a = 12.8811 (5) Å	μ = 0.10 mm^{-1}
b = 3.8933 (3) Å	T = 295 (2) K
c = 13.3202 (9) Å	$0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer	653 independent reflections
	563 reflections with $I > 2\sigma(I)$
Absorption correction: none	R_{int} = 0.030
3959 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042	1 restraint
$wR(F^2)$ = 0.112	H-atom parameters constrained
S = 1.04	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
653 reflections	$\Delta\rho_{min}$ = −0.10 e Å^{-3}
91 parameters	

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

References

Britton, D. (1998). *J. Chem. Crystallogr.* **28**, 601–604.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

A polymorph of terephthalaldehyde

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Comment

Terephthalaldehyde, (I), is a simple aromatic compound used in organic synthesis. It is known as a white substance, slightly soluble in water, with a melting point of 389 K. To the best of our knowledge, the only packing information of (I) in the solid state comes from the work of Britton (Britton, 1998), who investigated the intermolecular C—H···O arrangement in the structure of terephthalaldehyde and the crystals suitable for diffraction were obtained by recrystallization from an acetone/ethyl ether mixture.

A new polymorphic form of (I) has been obtained serendipitously during cocrystallization of pyridine and terephthalaldehyde from ethanol (Fig. 1). Flat needles which precipitated first from the solution had a melting point of 372 K and were identified as a new polymorphic form of (I) by X-ray crystallography. At room temperature, the crystals transform into the well known monoclinic form, with a melting point of 389 K. The crystal structure of the monoclinic form involves C—H···O hydrogen bonds but no such bonds are observed in the orthorhombic form (Fig. 2).

Experimental

The title compound was purchased from Aldrich.

Refinement

All H atoms were initially located in a difference Fourier map and then included with constrained bond lengths and isotropic displacement parameters: C—H=0.93Å and U_{iso}(H)=1.2U_{eq}(C) for H atoms.
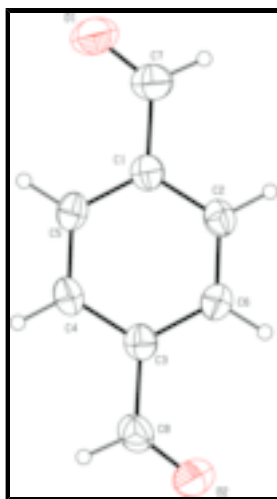
Figures

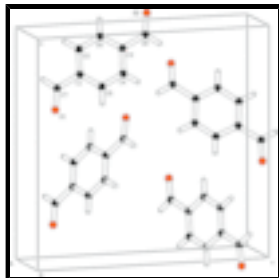Fig. 1. The molecular structure of (I) with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Fig. 2. The packing of the molecules, viewed down the *b* axis.

terephthalaldehyde

Crystal data

$C_8H_6O_2$	$F_{000} = 280$
$M_r = 134.13$	$D_x = 1.334$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2ac	Cell parameters from 1070 reflections
$a = 12.8811$ (5) Å	$\theta = 3.1–23.5°$
$b = 3.8933$ (3) Å	$\mu = 0.10$ mm^{-1}
$c = 13.3202$ (9) Å	$T = 295$ (2) K
$V = 668.01$ (7) Å^3	Block, colorless
$Z = 4$	$0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer	563 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.030$
Monochromator: graphite	$\theta_{max} = 25.5°$
$T = 295(2)$ K	$\theta_{min} = 3.1°$
φ and ω scans	$h = -15 \rightarrow 12$
Absorption correction: none	$k = -4 \rightarrow 4$
3959 measured reflections	$l = -16 \rightarrow 15$
653 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.112$	$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2 + 0.0133P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.014$
653 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
91 parameters	$\Delta\rho_{min} = -0.10$ e Å^{-3}

1 restraint

Extinction correction: none

Primary atom site location: structure-invariant direct methods

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	$U_{iso}*/U_{eq}$
C1	0.8164 (2)	0.3463 (7)	0.2145 (2)	0.0509 (7)
C2	0.9236 (2)	0.3850 (7)	0.2147 (3)	0.0545 (7)
H2	0.9566	0.4847	0.1599	0.065*
C3	0.9315 (2)	0.1279 (7)	0.3776 (2)	0.0515 (7)
C4	0.8239 (2)	0.0894 (7)	0.3770 (3)	0.0547 (7)
H4	0.7907	−0.0106	0.4316	0.066*
C5	0.7668 (2)	0.1993 (6)	0.2955 (3)	0.0543 (7)
H5	0.6950	0.1744	0.2953	0.065*
C6	0.9805 (2)	0.2776 (7)	0.2951 (3)	0.0547 (6)
H6	1.0523	0.3040	0.2949	0.066*
C7	0.7563 (4)	0.4635 (8)	0.1269 (4)	0.0707 (10)
H7	0.7934	0.5547	0.0733	0.085*
C8	0.9916 (3)	0.0084 (8)	0.4657 (4)	0.0627 (9)
H8	0.9550	−0.0874	0.5190	0.075*
O1	0.6637 (2)	0.4514 (7)	0.1187 (3)	0.0930 (10)
O2	1.0834 (2)	0.0266 (7)	0.4731 (4)	0.0876 (9)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0535 (16)	0.0494 (14)	0.0498 (17)	0.0041 (11)	0.0057 (14)	−0.0068 (13)
C2	0.0564 (18)	0.0551 (15)	0.0520 (17)	0.0022 (13)	0.0123 (15)	0.0015 (15)
C3	0.0547 (16)	0.0509 (14)	0.0490 (17)	−0.0008 (12)	0.0064 (15)	−0.0078 (13)
C4	0.0593 (19)	0.0573 (14)	0.0476 (17)	−0.0067 (14)	0.0140 (16)	0.0002 (14)
C5	0.0463 (13)	0.0570 (14)	0.0595 (15)	−0.0016 (12)	0.0102 (17)	−0.0083 (14)
C6	0.0448 (13)	0.0582 (14)	0.0610 (15)	−0.0003 (12)	0.0043 (18)	−0.0008 (14)
C7	0.071 (2)	0.077 (2)	0.063 (3)	0.0121 (16)	−0.006 (2)	−0.0016 (18)
C8	0.067 (2)	0.0704 (19)	0.0508 (19)	0.0030 (13)	0.009 (2)	0.0003 (14)
O1	0.075 (2)	0.129 (2)	0.075 (2)	0.0162 (14)	−0.0158 (19)	−0.0026 (18)
O2	0.0617 (17)	0.125 (2)	0.0761 (19)	0.0064 (12)	−0.0077 (16)	0.0074 (15)

Geometric parameters (Å, °)

C1—C5	1.378 (4)	C4—C5	1.379 (5)
C1—C2	1.389 (4)	C4—H4	0.9300
C1—C7	1.473 (6)	C5—H5	0.9300
C2—C6	1.363 (5)	C6—H6	0.9300
C2—H2	0.9300	C7—O1	1.199 (5)
C3—C4	1.393 (4)	C7—H7	0.9300
C3—C6	1.395 (4)	C8—O2	1.188 (4)
C3—C8	1.482 (6)	C8—H8	0.9300
C5—C1—C2	120.2 (3)	C1—C5—C4	119.9 (3)
C5—C1—C7	120.4 (3)	C1—C5—H5	120.1
C2—C1—C7	119.4 (3)	C4—C5—H5	120.1
C6—C2—C1	120.2 (3)	C2—C6—C3	120.2 (2)
C6—C2—H2	119.9	C2—C6—H6	119.9
C1—C2—H2	119.9	C3—C6—H6	119.9
C4—C3—C6	119.4 (3)	O1—C7—C1	125.6 (5)
C4—C3—C8	119.4 (3)	O1—C7—H7	117.2
C6—C3—C8	121.2 (3)	C1—C7—H7	117.2
C5—C4—C3	120.1 (3)	O2—C8—C3	124.5 (4)
C5—C4—H4	120.0	O2—C8—H8	117.7
C3—C4—H4	120.0	C3—C8—H8	117.7
C5—C1—C2—C6	0.1 (4)	C1—C2—C6—C3	0.0 (4)
C7—C1—C2—C6	−179.9 (3)	C4—C3—C6—C2	0.0 (4)
C6—C3—C4—C5	−0.2 (4)	C8—C3—C6—C2	179.9 (3)
C8—C3—C4—C5	180.0 (3)	C5—C1—C7—O1	1.8 (5)
C2—C1—C5—C4	−0.2 (4)	C2—C1—C7—O1	−178.2 (3)
C7—C1—C5—C4	179.7 (3)	C4—C3—C8—O2	179.2 (3)
C3—C4—C5—C1	0.2 (4)	C6—C3—C8—O2	−0.6 (5)

Fig. 1

Fig. 2