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4-Nitroisophthalic acid

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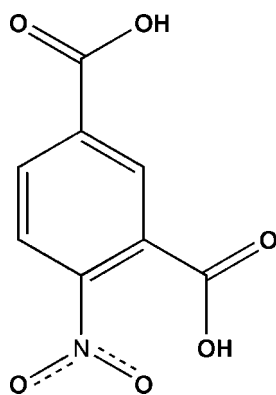
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.092; wR factor = 0.308; data-to-parameter ratio = 14.3.

In the crystal structure of the title compound, $\text{C}_8\text{H}_5\text{NO}_6$, both carboxyl groups are involved in intermolecular centrosymmetric cyclic $\text{O}-\text{H}\cdots\text{O}$ hydrogen-bonding associations, which give a zigzag chain structure extending along $(2\bar{1}1)$. Weak $\pi-\pi$ stacking interactions are also present [minimum ring centroid separation = $3.893(4)$ Å].

Related literature

For 4-nitroisophthalic acid as an intermediate in the synthesis of pharmaceutical drugs and as a ligand in transition metal complexes, see: Birk & Weihe (2009); Pan *et al.* (2011).



Experimental

Crystal data

| | |
|-----------------------------------|---|
| $\text{C}_8\text{H}_5\text{NO}_6$ | $\gamma = 75.37(3)^\circ$ |
| $M_r = 211.13$ | $V = 427.14(15) \text{ \AA}^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 7.0261(14) \text{ \AA}$ | Mo $K\alpha$ radiation |
| $b = 7.4380(15) \text{ \AA}$ | $\mu = 0.15 \text{ mm}^{-1}$ |
| $c = 8.5775(17) \text{ \AA}$ | $T = 293 \text{ K}$ |
| $\alpha = 80.09(3)^\circ$ | $0.30 \times 0.25 \times 0.20 \text{ mm}$ |
| $\beta = 86.22(3)^\circ$ | |

Data collection

| | |
|---|--|
| Rigaku SCXmini CCD-detector diffractometer | 4103 measured reflections |
| Absorption correction: multi-scan (<i>CrystalClear</i> ; Rigaku, 2005) | 1943 independent reflections |
| $T_{\min} = 0.957$, $T_{\max} = 0.971$ | 1554 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.136$ |

Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.092$ | 8 restraints |
| $wR(F^2) = 0.308$ | H-atom parameters constrained |
| $S = 0.86$ | $\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$ |
| 1943 reflections | $\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$ |
| 136 parameters | |

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{O4}-\text{H4}\cdots\text{O3}^i$ | 0.86 | 1.76 | 2.605(7) | 168 |
| $\text{O5}-\text{H5}\cdots\text{O6}^{ii}$ | 0.87 | 1.73 | 2.602(7) | 180 |

Symmetry codes: (i) $-x + 2, -y + 1, -z + 1$; (ii) $-x, -y + 2, -z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2171).

References

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supporting information

Acta Cryst. (2012). E68, o206 [doi:10.1107/S1600536811053797]

4-Nitroisophthalic acid

Yang-Hui Luo and Mei-Ling Pan

S1. Comment

4-Nitroisophthalic acid is an important chemical material because it is an intermediate in the synthesis of many pharmaceutical drugs and is also an excellent ligand for many transition metal complexes (Pan *et al.*, 2011; Birk & Weihe, 2009). As part of our interest in this compound, we report here the crystal structure of this acid.

The molecular structure of the title compound, C₈H₅NO₆ is shown in Fig. 1. All of the non-H and non-O atoms are approximately coplanar: the maximum r.m.s. deviation being 0.0202 Å. In the crystal structure, both carboxylic acid groups are involved in intermolecular centrosymmetric cyclic O—H...O hydrogen-bonding associations (Table 1) which give a zigzag chain structure extending along (2 -1 1) (Fig. 2). Weak π ... π stacking interactions are also present [minimum ring centroid separation = 3.893 (4) Å].

S2. Experimental

4-Nitroisophthalic acid was obtained commercially from ChemFuture PharmaTech, Ltd (Nanjing, Jiangsu). Crystals of it suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

S3. Refinement

All H atoms attached to C atoms and O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å, and O—H = 0.86±1 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

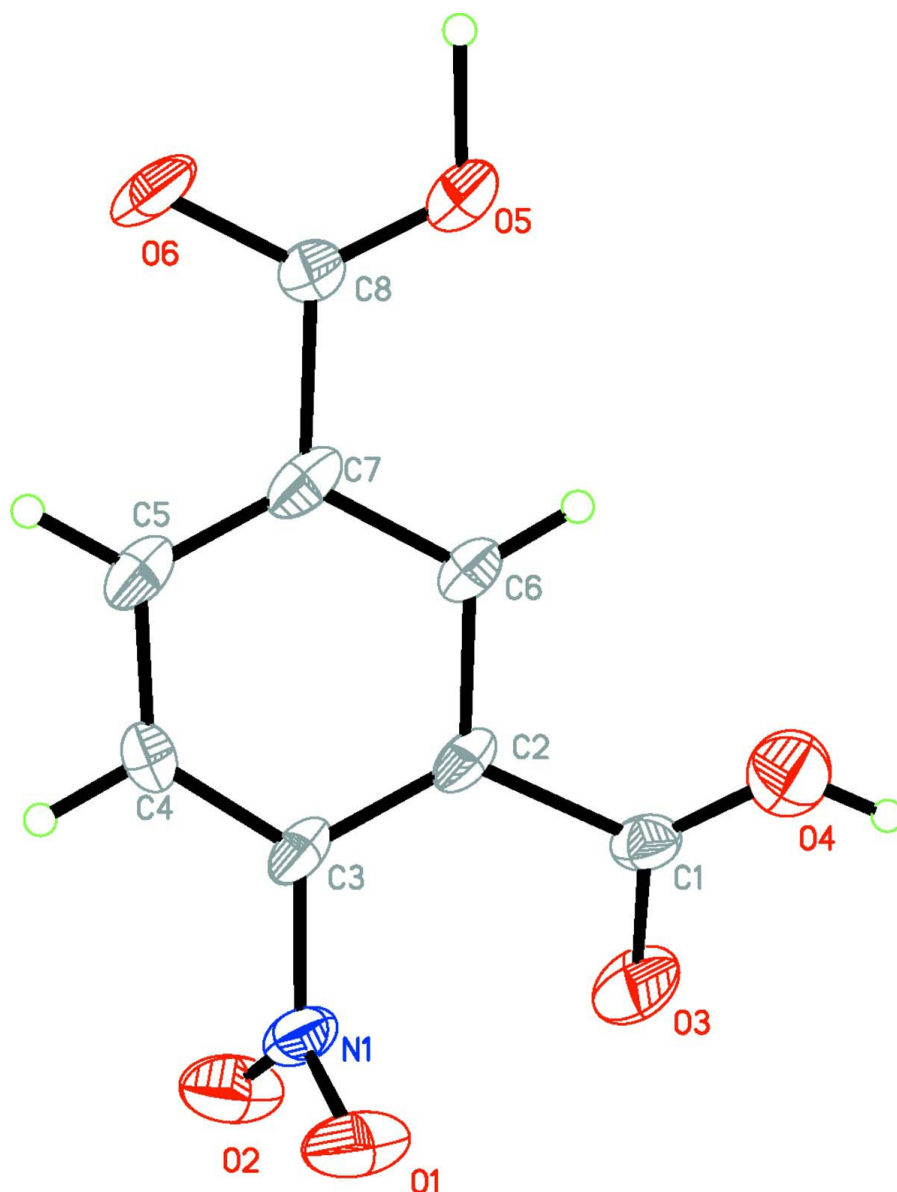
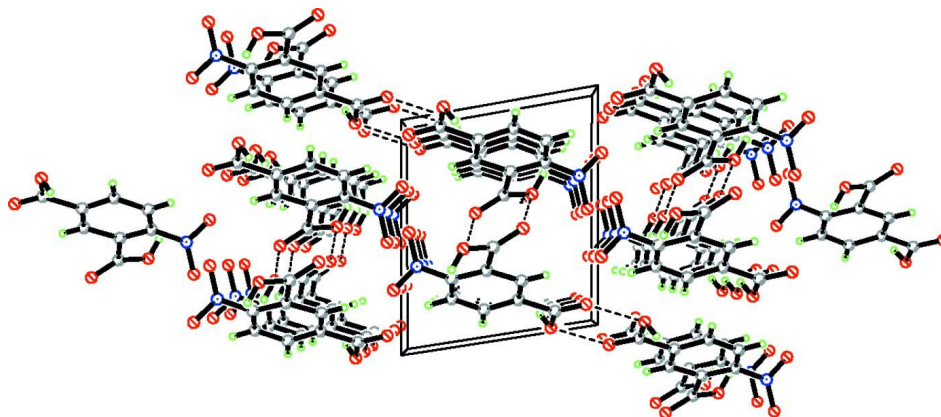


Figure 1

The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

A packing diagram viewed down the a axis showing the three-dimensional network. Intermolecular hydrogen bonds are shown as dashed lines.

4-nitrobenzene-1,3-dicarboxylic acid

Crystal data

$C_8H_5NO_6$

$M_r = 211.13$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.0261$ (14) Å

$b = 7.4380$ (15) Å

$c = 8.5775$ (17) Å

$\alpha = 80.09$ (3)°

$\beta = 86.22$ (3)°

$\gamma = 75.37$ (3)°

$V = 427.14$ (15) Å³

$Z = 2$

$F(000) = 216$

$D_x = 1.642$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1943 reflections

$\theta = 3.0$ – 27.5 °

$\mu = 0.15$ mm⁻¹

$T = 293$ K

Prism, red

$0.30 \times 0.25 \times 0.20$ mm

Data collection

Rigaku SCXmini CCD-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 13.6612 pixels mm⁻¹

CCD profile-fitting scans

Absorption correction: multi-scan

CrystalClear (Rigaku, 2005)

$T_{\min} = 0.957$, $T_{\max} = 0.971$

4103 measured reflections

1943 independent reflections

1554 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.136$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.0$ °

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 9$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.092$

$wR(F^2) = 0.308$

$S = 0.86$

1943 reflections

136 parameters

8 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

$w = 1/[\sigma^2(F_o^2) + (0.1225P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.017$

$\Delta\rho_{\max} = 0.40$ e Å⁻³

$\Delta\rho_{\min} = -0.50$ e Å⁻³

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|------------|-------------|------------|----------------------------------|
| O1 | 0.7042 (7) | 0.0658 (7) | 0.5109 (6) | 0.0760 (19) |
| O2 | 0.8052 (7) | -0.0122 (8) | 0.2874 (6) | 0.081 (2) |
| O3 | 0.9385 (6) | 0.3293 (8) | 0.4061 (6) | 0.076 (2) |
| O4 | 0.7716 (6) | 0.6167 (8) | 0.4485 (6) | 0.074 (2) |
| O5 | 0.2226 (6) | 0.9309 (7) | 0.0974 (5) | 0.0637 (18) |
| O6 | 0.0288 (6) | 0.7626 (7) | 0.0224 (6) | 0.0706 (18) |
| N1 | 0.7089 (7) | 0.1015 (8) | 0.3680 (6) | 0.0470 (19) |
| C1 | 0.7884 (8) | 0.4702 (9) | 0.3938 (7) | 0.041 (2) |
| C2 | 0.6144 (7) | 0.4407 (9) | 0.3070 (6) | 0.0396 (19) |
| C3 | 0.5813 (8) | 0.2668 (9) | 0.2931 (7) | 0.044 (2) |
| C4 | 0.4265 (8) | 0.2656 (9) | 0.2113 (7) | 0.0446 (19) |
| C5 | 0.2929 (8) | 0.4233 (10) | 0.1433 (7) | 0.049 (2) |
| C6 | 0.4838 (7) | 0.6123 (9) | 0.2397 (6) | 0.0397 (19) |
| C7 | 0.3223 (7) | 0.5954 (10) | 0.1615 (6) | 0.045 (2) |
| C8 | 0.1809 (8) | 0.7812 (9) | 0.0882 (7) | 0.045 (2) |
| H4 | 0.87560 | 0.63520 | 0.48410 | 0.0880* |
| H4A | 0.40800 | 0.14920 | 0.19970 | 0.0540* |
| H5 | 0.13830 | 1.03360 | 0.05700 | 0.0770* |
| H5A | 0.18720 | 0.41500 | 0.08750 | 0.0590* |
| H6A | 0.50480 | 0.72900 | 0.24730 | 0.0480* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-----------|-----------|-----------|------------|------------|------------|
| O1 | 0.075 (3) | 0.068 (4) | 0.066 (3) | 0.011 (3) | -0.018 (3) | 0.005 (3) |
| O2 | 0.098 (4) | 0.058 (4) | 0.067 (3) | 0.017 (3) | -0.008 (3) | -0.006 (3) |
| O3 | 0.034 (2) | 0.080 (4) | 0.105 (4) | 0.007 (3) | -0.030 (2) | -0.010 (3) |
| O4 | 0.045 (2) | 0.097 (4) | 0.085 (4) | -0.009 (3) | -0.029 (2) | -0.032 (3) |
| O5 | 0.049 (2) | 0.067 (4) | 0.074 (3) | -0.006 (3) | -0.034 (2) | -0.008 (3) |
| O6 | 0.042 (2) | 0.086 (4) | 0.079 (3) | -0.001 (3) | -0.034 (2) | -0.010 (3) |
| N1 | 0.046 (3) | 0.053 (4) | 0.036 (3) | -0.006 (3) | -0.015 (2) | 0.005 (3) |
| C1 | 0.035 (3) | 0.047 (4) | 0.041 (4) | -0.012 (3) | -0.007 (3) | -0.001 (3) |
| C2 | 0.025 (3) | 0.052 (4) | 0.038 (3) | 0.000 (3) | -0.012 (2) | -0.007 (3) |
| C3 | 0.032 (3) | 0.055 (4) | 0.043 (4) | -0.002 (3) | -0.012 (3) | -0.012 (3) |
| C4 | 0.048 (3) | 0.032 (3) | 0.060 (4) | -0.017 (3) | -0.011 (3) | -0.010 (3) |
| C5 | 0.038 (3) | 0.067 (5) | 0.044 (4) | -0.010 (4) | -0.016 (3) | -0.011 (3) |

| | | | | | | |
|----|-----------|-----------|-----------|------------|------------|------------|
| C6 | 0.032 (3) | 0.052 (4) | 0.036 (3) | -0.011 (3) | -0.012 (2) | -0.004 (3) |
| C7 | 0.030 (3) | 0.071 (5) | 0.032 (3) | -0.004 (3) | -0.009 (2) | -0.010 (3) |
| C8 | 0.037 (3) | 0.046 (4) | 0.054 (4) | -0.006 (3) | -0.013 (3) | -0.012 (3) |

Geometric parameters (Å, °)

| | | | |
|-------------|------------|-------------|------------|
| O1—N1 | 1.209 (7) | C2—C6 | 1.424 (9) |
| O2—N1 | 1.225 (8) | C2—C3 | 1.396 (9) |
| O3—C1 | 1.281 (8) | C3—C4 | 1.335 (8) |
| O4—C1 | 1.235 (9) | C4—C5 | 1.370 (9) |
| O5—C8 | 1.237 (8) | C5—C7 | 1.383 (10) |
| O6—C8 | 1.289 (7) | C6—C7 | 1.398 (7) |
| O4—H4 | 0.8600 | C7—C8 | 1.543 (9) |
| O5—H5 | 0.8700 | C4—H4A | 0.9300 |
| N1—C3 | 1.406 (8) | C5—H5A | 0.9300 |
| C1—C2 | 1.552 (8) | C6—H6A | 0.9300 |
| C1—O4—H4 | 118.00 | C4—C5—C7 | 117.0 (5) |
| C8—O5—H5 | 116.00 | C2—C6—C7 | 116.2 (6) |
| O1—N1—C3 | 118.7 (5) | C5—C7—C8 | 120.9 (5) |
| O2—N1—C3 | 119.2 (5) | C6—C7—C8 | 116.3 (6) |
| O1—N1—O2 | 121.5 (6) | C5—C7—C6 | 122.7 (6) |
| O4—C1—C2 | 120.2 (6) | O6—C8—C7 | 115.3 (5) |
| O3—C1—O4 | 126.0 (6) | O5—C8—O6 | 126.5 (6) |
| O3—C1—C2 | 113.8 (5) | O5—C8—C7 | 118.2 (5) |
| C3—C2—C6 | 121.0 (5) | C3—C4—H4A | 118.00 |
| C1—C2—C6 | 113.4 (5) | C5—C4—H4A | 118.00 |
| C1—C2—C3 | 125.6 (5) | C4—C5—H5A | 122.00 |
| N1—C3—C4 | 122.9 (6) | C7—C5—H5A | 121.00 |
| N1—C3—C2 | 118.9 (5) | C2—C6—H6A | 122.00 |
| C2—C3—C4 | 118.2 (6) | C7—C6—H6A | 122.00 |
| C3—C4—C5 | 124.8 (6) | | |
| O1—N1—C3—C2 | 69.8 (8) | C3—C2—C6—C7 | 0.6 (8) |
| O1—N1—C3—C4 | -108.8 (7) | N1—C3—C4—C5 | 176.5 (6) |
| O2—N1—C3—C2 | -118.3 (6) | C2—C3—C4—C5 | -2.1 (9) |
| O2—N1—C3—C4 | 63.1 (8) | C3—C4—C5—C7 | 0.0 (9) |
| O3—C1—C2—C3 | 23.2 (8) | C4—C5—C7—C6 | 2.5 (8) |
| O3—C1—C2—C6 | -157.0 (5) | C4—C5—C7—C8 | 179.5 (5) |
| O4—C1—C2—C3 | -154.2 (6) | C2—C6—C7—C5 | -2.7 (8) |
| O4—C1—C2—C6 | 25.5 (8) | C2—C6—C7—C8 | -179.9 (5) |
| C1—C2—C3—N1 | 2.8 (9) | C5—C7—C8—O5 | -174.4 (5) |
| C1—C2—C3—C4 | -178.6 (5) | C5—C7—C8—O6 | 5.5 (8) |
| C6—C2—C3—N1 | -177.0 (5) | C6—C7—C8—O5 | 2.8 (8) |
| C6—C2—C3—C4 | 1.7 (8) | C6—C7—C8—O6 | -177.3 (5) |
| C1—C2—C6—C7 | -179.2 (5) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O4—H4 \cdots O3 ⁱ | 0.86 | 1.76 | 2.605 (7) | 168 |
| O5—H5 \cdots O6 ⁱⁱ | 0.87 | 1.73 | 2.602 (7) | 180 |
| C5—H5A \cdots O6 ⁱⁱⁱ | 0.93 | 2.56 | 3.423 (8) | 154 |

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x, -y+2, -z$; (iii) $-x, -y+1, -z$.