

## 2-(3-Carboxymethyl-4-nitrophenyl)propionic acid

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## Key indicators

Single-crystal X-ray study

T = 298 K

Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$ 

Disorder in main residue

R factor = 0.067

wR factor = 0.195

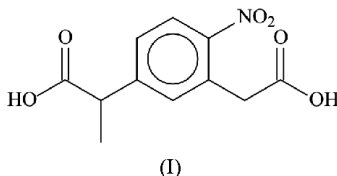
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecules of the title dicarboxylic acid,  $\text{C}_{11}\text{H}_{11}\text{NO}_6$ , are linked by two pairs of hydrogen bonds into a linear chain structure; the nitro group distorts the bond dimensions of the aromatic ring.

## Comment

Our studies have been directed toward the synthesis of metal salts of benzene-1,3-dicarboxylic acids; such acids are rarely studied in contrast to, for example, the 1,2- and 1,4-dicarboxylic acids. 3-Carboxyphenoxyacetic acid crystallizes as a monohydrate whose water molecules link the carboxyl groups into a hydrogen-bonded network motif (Gu *et al.*, 2004). Benzene-1,3-di(oxyacetic acid) has been characterized as its manganese (Liu *et al.*, 2004) and nickel (Gao, Liu, Huo *et al.*, 2004) derivatives; the dianionic group has also been isolated as a mixed sodium/chromium salt (Gao, Liu, Dong *et al.*, 2004). The title compound, (I) (Fig. 1), is a substituted benzene-1,3-diacetic acid; the nitro group is twisted by  $22.9(2)^\circ$  with respect to the aromatic ring but it appears to distort the distances and angles of the C atoms *ortho* and *para* to it [ $\text{C5}-\text{C4}-\text{C9} = 118.0(3)^\circ$  and  $\text{C5}-\text{C6}-\text{C7} = 116.2(3)^\circ$ ]. Adjacent molecules are linked by hydrogen bonds into a linear chain structure (Fig. 2).



The energy of the geometry-optimized structure was calculated to be  $146.6 \text{ kcal mol}^{-1}$  more stable than the X-ray structure; however, the theoretical structure shows no significant distortion of the aromatic ring. Atom C4 is more positive than the adjacent methylene C atom, whereas atom C6 is more negative than the adjacent methine C atom. Atoms C4 and C6 are both more positive than their adjacent neighbors in the crystal structure.

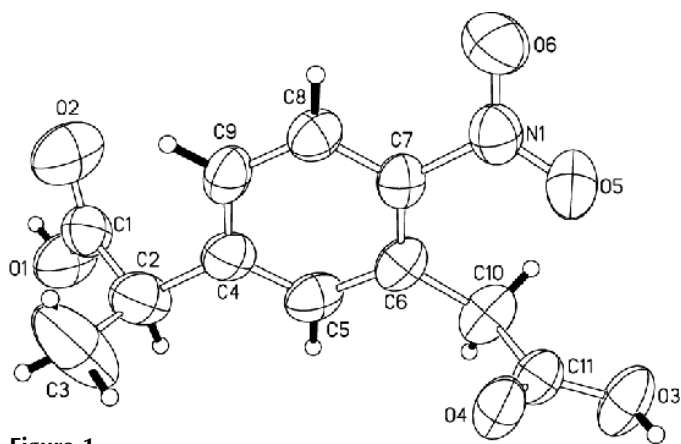
## Experimental

The dicarboxylic acid was synthesized according to a literature procedure (Peng & Chen, 2001) by replacing the chlorine in 2,4-dichloronitrobenzene with the potassium salt of diethyl methylmalonate. Potassium *tert*-butylate was used to catalyse the reaction. Crystals were obtained when the hydrolysed mixture was set aside. Elemental analysis calculated for  $\text{C}_{11}\text{H}_{11}\text{NO}_6$ : C 52.13, H 4.34, N 5.53%; found: C 52.38, H 4.21, N 5.49%. For the geometry-optim-

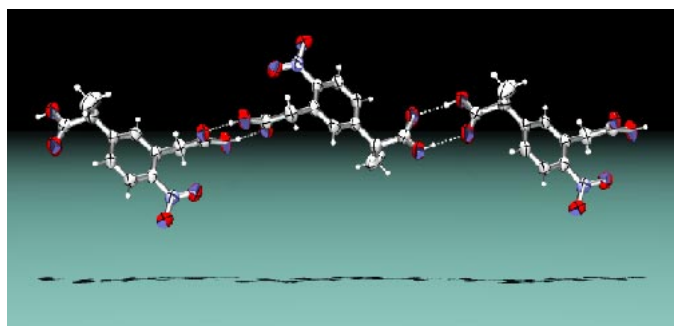
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**Figure 1**  
ORTEP (Johnson, 1976) plot of (I); displacement ellipsoids are drawn at the 50% probability level. The minor component of the disordered methyl group is not shown.



**Figure 2**  
POV-Ray (Cason, 2002)/ORTEP (Johnson, 1976) plot of the hydrogen-bonded chain in (I).

ization calculations with *HyperChem* (Hypercube, 2000), the starting structure was taken from the crystal structure, and this was optimized at the *PM3* level.

#### Crystal data

$C_{11}H_{11}NO_6$	$Z = 2$
$M_r = 253.21$	$D_x = 1.431 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.468 (3) \text{ \AA}$	Cell parameters from 5376 reflections
$b = 8.550 (4) \text{ \AA}$	$\theta = 3.4\text{--}27.5^\circ$
$c = 9.525 (5) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$\alpha = 104.03 (3)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 91.42 (5)^\circ$	Block, yellow
$\gamma = 94.33 (3)^\circ$	$0.42 \times 0.31 \times 0.25 \text{ mm}$
$V = 587.8 (5) \text{ \AA}^3$	

#### Data collection

Rigaku R-AXIS RAPID diffractometer	2347 independent reflections
$\omega$ scans	1680 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan ( <i>ABSCOR</i> ; Higashi, 1995)	$R_{\text{int}} = 0.023$
$T_{\text{min}} = 0.695$ , $T_{\text{max}} = 0.971$	$\theta_{\text{max}} = 26.2^\circ$
4881 measured reflections	$h = -9 \rightarrow 8$
	$k = -10 \rightarrow 10$
	$l = -11 \rightarrow 11$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0907P)^2 + 0.3164P]$
$R[F^2 > 2\sigma(F^2)] = 0.067$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.195$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.47 \text{ e \AA}^{-3}$
2347 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
165 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—C1	1.260 (4)	C2—C4	1.523 (4)
O2—C1	1.235 (4)	C4—C5	1.385 (4)
O3—C11	1.304 (3)	C4—C9	1.395 (4)
O4—C11	1.218 (3)	C5—C6	1.392 (4)
O5—N1	1.213 (3)	C6—C7	1.389 (4)
O6—N1	1.222 (3)	C6—C10	1.510 (4)
N1—C7	1.475 (4)	C7—C8	1.374 (4)
C1—C2	1.514 (5)	C8—C9	1.371 (4)
C2—C3	1.465 (7)	C10—C11	1.503 (4)
O5—N1—O6	122.9 (3)	C5—C6—C7	116.2 (3)
O5—N1—C7	119.3 (3)	C5—C6—C10	119.1 (3)
O6—N1—C7	117.8 (3)	C7—C6—C10	124.7 (3)
O1—C1—O2	123.2 (3)	C6—C7—C8	122.3 (3)
O1—C1—C2	115.3 (3)	C6—C7—N1	121.2 (2)
O2—C1—C2	121.4 (3)	C8—C7—N1	116.5 (2)
C1—C2—C3	109.6 (4)	C7—C8—C9	120.0 (3)
C1—C2—C4	113.3 (3)	C4—C9—C8	120.4 (3)
C3—C2—C4	113.0 (3)	O3—C11—C10	113.5 (2)
C2—C4—C5	119.8 (3)	C6—C10—C11	112.5 (2)
C2—C4—C9	122.2 (3)	O3—C11—O4	123.3 (3)
C5—C4—C9	118.0 (3)	O4—C11—C10	123.2 (2)
C4—C5—C6	123.1 (3)		

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O1—H1 $\cdots$ O2 <sup>i</sup>	0.82	1.82	2.641 (4)	173
O3—H3 $\cdots$ O4 <sup>ii</sup>	0.82	1.84	2.658 (3)	176

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

The H atoms were placed in calculated positions [aromatic C—H = 0.93  $\text{\AA}$ , methylene C—H = 0.97  $\text{\AA}$  and methyl C—H = 0.98  $\text{\AA}$ , with  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5U_{\text{eq}}(\text{C})$ ; O—H = 0.82  $\text{\AA}$ , with  $1.2U_{\text{eq}}(\text{O})$ ]. The acid H atoms were positioned geometrically by allowing the OH units to rotate so as to fit the electron density. The crystal was measured to  $\theta_{\text{max}} = 27.5^\circ$ , but as it did not diffract at high angle, only the reflections up to  $26.2^\circ$  were used in the refinements.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1976) and *POV-Ray* (Cason, 2002); software used to prepare material for publication: *SHELXL97*.

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