3-Nitroacetanilide

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.154
Data-to-parameter ratio = 14.8

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

The title compound, also known as N-(3-nitrophenyl)acetamide, $C_8H_8N_2O_3$, is an organic non-linear optical material. It crystallizes in the monoclinic system in the chiral space group $P2_1$, and there are four independent molecules in the asymmetric unit.

Comment

The title compound, (I), is found to be of interest as an organic non-linear optical (NLO) material. The crystals show optical second harmonic generation with the fundamental beam ($\lambda = 1064$ nm) of Nd–YAG laser. The single-crystal structure elucidation further reinforces this observation.
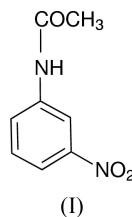

(I)

Fig. 1 shows the asymmetric unit of the title compound. The torsion angles about the C(phenyl)—N(amide) and C(phenyl)—N(nitro) bonds show that the amide and nitro groups lie in the plane of the phenyl ring (Table 1). The carbonyl group is involved in hydrogen bonding, unlike the case of 2-nitroacetanilide (Kashino et al., 1986). The packing in (I) is stabilized by N—H···O and C—H···O hydrogen bonds (Fig. 2 and Table 2).

Experimental

The title compound was prepared by the direct reaction of 3-nitroaniline and acetic anhydride at room temperature for 10 min. Crystals of (I), suitable for single-crystal diffraction study, were grown at ambient temperature by slow evaporation of a methanol solution.

Crystal data

$C_8H_8N_2O_3$	$D_x = 1.425$ Mg m^{-3}
$M_r = 180.16$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 1024
$a = 9.767$ (2) Å	reflections
$b = 13.298$ (3) Å	$\theta = 2.2$–27.6°
$c = 13.272$ (3) Å	$\mu = 0.11$ mm^{-1}
$\beta = 102.991$ (5)°	$T = 293$ (2) K
$V = 1679.8$ (6) Å^3	Pyramid, pale orange
$Z = 8$	$0.39 \times 0.35 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	5863 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.026$
Absorption correction: none	$\theta_{max} = 28.0°$
14297 measured reflections	$h = -12 \rightarrow 12$
6994 independent reflections	$k = -17 \rightarrow 16$
	$l = -17 \rightarrow 17$

Refinement

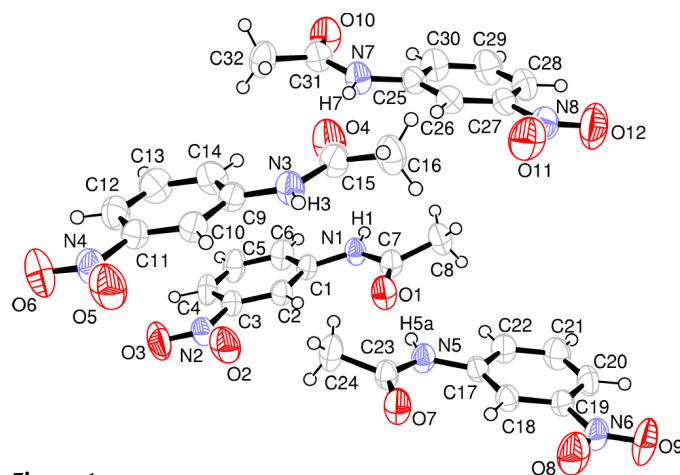|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1044P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.059$	$+ 0.024P]$
$wR(F^2) = 0.154$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
6994 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
473 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
H-atom parameters constrained	Absolute structure: (Flack, 1983),
	817 Friedel pairs
	Flack parameter = -0.01 (10)

Figure 1
View of the asymmetric unit of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
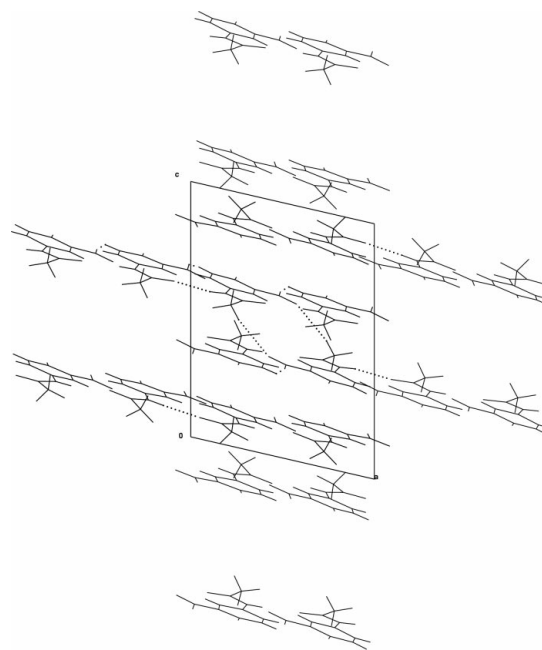
Table 1
Selected geometric parameters (Å, °).

O1—C7	1.216 (3)	N1—C1	1.386 (3)
O2—N2	1.207 (3)	N1—C7	1.362 (3)
O3—N2	1.204 (3)	N2—C3	1.476 (3)
O4—C15	1.209 (3)	N3—C9	1.404 (3)
O5—N4	1.208 (3)	N3—C15	1.347 (3)
O6—N4	1.225 (3)	N4—C11	1.469 (3)
O7—C23	1.225 (3)	N5—C23	1.345 (3)
O8—N6	1.221 (3)	N5—C17	1.395 (3)
O9—N6	1.207 (3)	N6—C19	1.474 (3)
O10—C31	1.212 (3)	N7—C31	1.344 (3)
O11—N8	1.214 (3)	N7—C25	1.405 (3)
O12—N8	1.225 (4)	N8—C27	1.468 (4)
C7—N1—C1—C6	−171.9 (2)	C17—N5—C23—C24	176.6 (2)
C1—N1—C7—C8	−179.0 (2)	O9—N6—C19—C18	176.4 (2)
O2—N2—C3—C4	179.9 (2)	O8—N6—C19—C20	175.7 (2)
O3—N2—C3—C2	−179.8 (2)	C31—N7—C25—C26	172.5 (2)
C9—N3—C15—C16	175.6 (3)	C25—N7—C31—C32	−178.3 (2)
O5—N4—C11—C12	−179.2 (2)	O11—N8—C27—C28	178.7 (3)
O6—N4—C11—C10	−179.2 (2)	O11—N8—C27—C26	−1.1 (4)
C23—N5—C17—C22	178.3 (2)	O12—N8—C27—C26	178.2 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O7^i	0.86	2.06	2.911 (3)	169
N3—H3···O10^{ii}	0.86	2.03	2.894 (3)	178
N5—H5A···O1^{iii}	0.86	2.06	2.922 (3)	178
N7—H7···O4^{iv}	0.86	2.06	2.908 (3)	171
C2—H2···O1	0.93	2.29	2.861 (3)	119
C5—H5···O9^v	0.93	2.51	3.203 (4)	131
C8—H8A···O2^{vi}	0.96	2.51	3.443 (3)	163
C14—H14···O4	0.93	2.27	2.865 (4)	121
C18—H18···O7	0.93	2.27	2.851 (3)	120
C21—H21···O3^{vi}	0.93	2.54	3.178 (4)	126
C30—H30···O10	0.93	2.30	2.874 (4)	120

Symmetry codes: (i) $-x, y - \frac{1}{2}, 1 - z$; (ii) $1 + x, y - 1, z$; (iii) $1 - x, \frac{1}{2} + y, 1 - z$; (iv) $x, 1 + y, z$; (v) $-x, \frac{1}{2} + y, 1 - z$; (vi) $1 - x, y - \frac{1}{2}, 1 - z$.

H atoms were positioned geometrically and allowed to ride on their respective parent atoms; C—H = 0.93–0.96 Å, and U_{iso} = $1.2U_{eq}$(NH and ring CH) or $1.5U_{eq}$(CH$_3$ and CH$_2$).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *PLATON* (Spek, 1990).

Figure 2
Packing diagram of the title compound, viewed down the *b* axis.

References

Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Kashino, S., Matsushita, T., Iwamoto, T., Yamaguchi, K. & Haisa, M. (1986). *Acta Cryst.* C**42**, 457–462.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.