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2-Methoxycarbonyl-4-nitroacetanilide: π-stacked chains linked in pairs by C—H···O hydrogen bonds

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.050
wR factor = 0.130
Data-to-parameter ratio = 13.2

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

Molecules of the title compound, $C_{10}H_{10}N_2O_5$, lie on mirror planes in space group *Ibam*. The molecules are linked into [001] chains by an aromatic π–π stacking interaction, and pairs of these chains are linked by a single C—H···O hydrogen bond.

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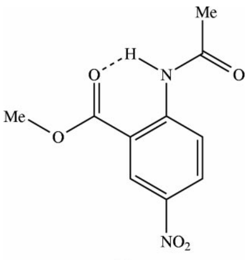
Comment

The title compound (I) (Fig. 1), crystallizes in the uncommon space group *Ibam* with Z' = 0.5: all of the atoms apart from the methyl H atoms lie on a mirror plane, chosen for the reference molecule as that at z = 0.5. Each of the H atoms in the methyl groups is disordered over two sites with equal occupancy. The inter-bond angles at N1, at C11 and at C21 (Table 1) show marked deviations from 120°, possibly indicative of repulsive non-bonded intramolecular contacts (Table 2). The bond distances show no unusual values.



(I)

The molecules of compound (I) are linked into chains by a single aromatic π–π stacking interaction, and these chains are

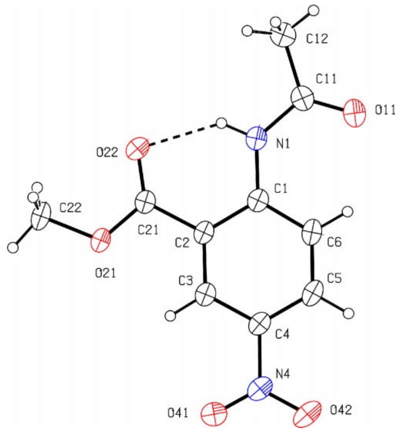


Figure 1
The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. For the sake of clarity, only one orientation is shown for each methyl group.

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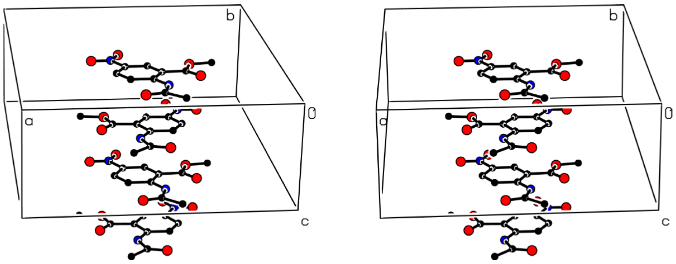

Figure 2
Stereoview of part of the crystal structure of compound (I), showing the formation of a π-stacked chain along [001]. For the sake of clarity, the H atoms have all been omitted.

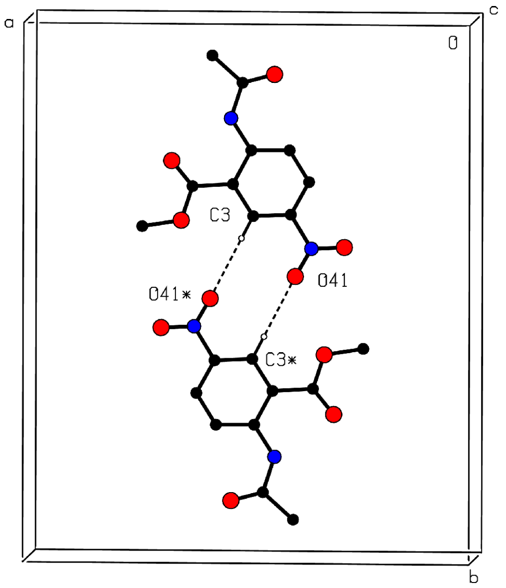

Figure 3
Part of the crystal structure of compound (I), showing the formation of the $R_2^2(10)$ motif which links the [001] chains into pairs. For the sake of clarity, the H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 1 - y, 0.5)$.

weakly linked in pairs by a C—H···O hydrogen bond. The reference molecule at $(x, y, 0.5)$ forms π-stacking interactions with the two molecules at $(1 - x, y, 0)$ and $(1 - x, y, 1)$. The common interplanar spacing is $c/2$ and the ring-centroid separation is 3.634 (2) Å, corresponding to a near-ideal centroid offset of 1.341 (2) Å. Propagation of this interaction thus produces a chain running parallel to the [001] direction and generated by the c-glide plane at $x = 0.5$ (Fig. 2). Four chains of this type run through each unit cell; two are generated by the c-glide plane at $x = 0.5$ and lie wholly within the domain $0.25 < y < 0.75$, while the two others generated by the c-glide plane at $x = 0.0$ lie within the domain $-0.25 < x < 0.25$.

Within each domain of x, the pairs of [001] chains in the domains $0 < y < 0.5$ and $0.5 < y < 1.0$ are linked by a single C—H···O hydrogen bond which, although it is rather long, is effectively linear (Table 2). Atom C3 in the molecule at $(x, y, 0.5)$ acts as donor to nitro atom O41 in the molecule at $(1 - x, 1 - y, 0.5)$, so forming an $R_2^2(10)$ motif generated by the twofold rotation axis along $(0.5, 0.5, z)$ (Fig. 3).

Experimental

2-Carboxymethylacetanilide was nitrated using fuming nitric acid at 273 K, following a published procedure (Adams *et al.*, 1954). The reaction mixture was poured on to ice and the resulting solid (I) was collected. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in acetone (m.p. 447–449 K).

Crystal data

$C_{10}H_{10}N_2O_5$
$M_r = 238.20$
Orthorhombic, *Ibam*
$a = 16.4482$ (14) Å
$b = 19.9095$ (17) Å
$c = 6.7549$ (6) Å
$V = 2212.1$ (3) Å^3
$Z = 8$
$D_x = 1.430$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1384 reflections
$\theta = 2.1$–$27.5°$
$\mu = 0.12$ mm^{-1}
$T = 291$ (2) K
Prism, colourless
0.43 × 0.18 × 0.13 mm

Data collection

Bruker SMART 1000 CCD area detector diffractometer
φ–ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.930$, $T_{\max} = 0.985$
7820 measured reflections

1384 independent reflections
828 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.057$
$\theta_{\max} = 27.5°$
$h = -21 \rightarrow 21$
$k = -24 \rightarrow 25$
$l = -8 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.130$
$S = 1.01$
1384 reflections
105 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.1650P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected bond angles (°).

C1—N1—C11	129.6 (2)	C2—C21—O21	111.55 (19)
N1—C11—O11	122.8 (2)	C2—C21—O22	125.9 (2)
N1—C11—C12	114.1 (2)	O21—C21—O22	122.5 (2)
O11—C11—C12	123.1 (2)	C21—O21—C22	117.2 (2)

Table 2
Geometric parameters (Å, °) for hydrogen bonds and short intramolecular contacts.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O22	0.86	1.98	2.691 (2)	139
C3—H3···O41$^{\rm i}$	0.93	2.51	3.440 (3)	180
C3—H3···O21	0.93	2.32	2.660 (3)	101
C6—H6···O11	0.93	2.23	2.854 (3)	124

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

All of the non-H atoms lie on mirror planes and the reference molecule was chosen to lie on the mirror plane at $z = 0.5$. The H atoms were located in difference maps and then treated as riding atoms, with C—H distances 0.93 (aromatic) or 0.95 Å (methyl) and an N—H distance of 0.86 Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C,N), or $1.5U_{\rm eq}$(C) for the methyl groups. Each methyl group was modelled using six H-atom sites, each with 0.5 occupancy, offset by 60° intervals.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and PRPKAPPA (Ferguson, 1999).

X-ray data were collected at the University of Aberdeen using a Bruker SMART 1000 CCD diffractometer; the authors thank the University of Aberdeen for funding the purchase of the diffractometer. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work. JLW thanks CNPq and FAPERJ for financial support.

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