

Acetone 3-nitrophenylhydrazone, redetermined at 120 K: sheets built from N—H···O, C—H···O and C—H···N hydrogen bonds

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Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 R factor = 0.058
 wR factor = 0.146
 Data-to-parameter ratio = 16.4

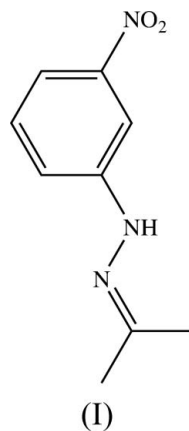
For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

Molecules of the title compound, $\text{C}_9\text{H}_{11}\text{N}_3\text{O}_2$, are linked into sheets by a combination of N—H···O, C—H···O and C—H···N hydrogen bonds.

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Comment

The structure of the title compound, (I) (Fig. 1), was determined many years ago [Cambridge Structural Database, Version 7.27 (Allen 2002) refcode NPHYAC; Menczel, 1969], using diffraction data collected at ambient temperature; no H atom coordinates were reported, and the structure was refined only to $R = 0.169$. We have now redetermined this structure using diffraction data collected at 120 K, and we report here the details of the supramolecular aggregation.



The molecules are linked into sheets by a combination of N—H···O, C—H···O and C—H···N hydrogen bonds (Table 1), and the sheet formation is readily analysed in terms of a hydrogen-bonded dimer as the basic building block. Atoms N1 and C2 in the molecule at (x, y, z) both act as hydrogen-bond donors to atom O31 in the molecule at $(\frac{3}{2} - x, \frac{1}{2} - y, 1 - z)$, so forming a centrosymmetric dimer centred at $(\frac{3}{4}, \frac{1}{4}, \frac{1}{2})$ containing three edge-fused rings, one of $R_2^2(10)$ type (Bernstein *et al.*, 1995) flanked by two of $R_2^2(6)$ type (Fig. 2).

Atoms C5 in the molecules at (x, y, z) and $(\frac{3}{2} - x, \frac{1}{2} - y, 1 - z)$, which form the dimer centred at $(\frac{3}{4}, \frac{1}{4}, \frac{1}{2})$, act as hydrogen-bond donors to atoms N2 in the molecules at $(\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and $(x, -y, -\frac{1}{2} + z)$, which lie in the dimers centred at $(\frac{3}{4}, \frac{3}{4}, 1)$ and $(\frac{3}{4}, -\frac{1}{4}, 0)$, respectively. Similarly, atoms N2 at (x, y, z) and $(\frac{3}{2} - x, \frac{1}{2} - y, 1 - z)$ accept hydrogen bonds from atoms C5 in the molecules at $(\frac{3}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z)$ and $(x, 1 - y, -\frac{1}{2} + z)$, which themselves form parts of the dimers centred at $(\frac{3}{4}, -\frac{1}{4}, 1)$ and $(\frac{3}{4}, \frac{3}{4}, 0)$, respectively. Hence each dimer is linked by C—H···N hydrogen bonds to four other dimers and

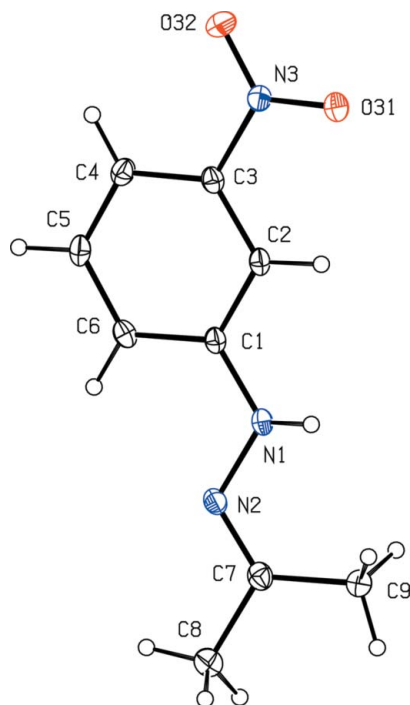


Figure 1
A molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

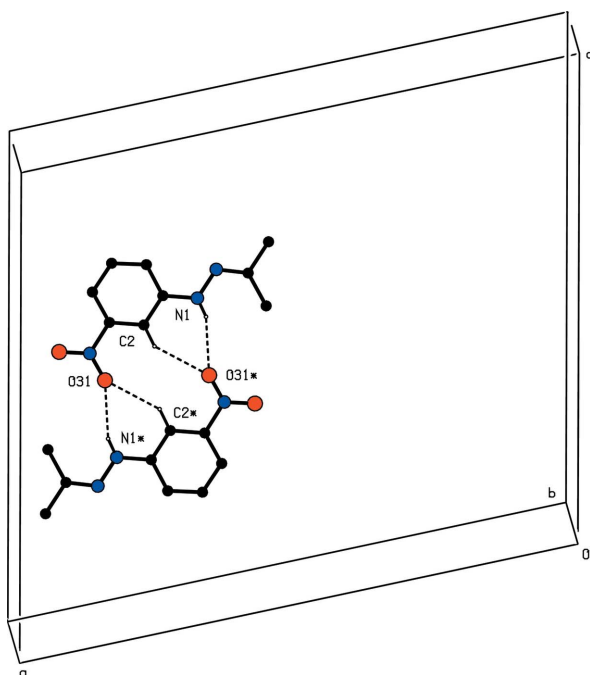


Figure 2
The molecular structure of (I), showing the formation of a centrosymmetric dimer containing three edge-fused hydrogen-bonded (dashed lines) rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted. The atoms marked with an asterisk (*) are at the symmetry position ($1-x, 1-y, 1-z$).

propagation of these interactions then generates a sheet parallel to (100) (Fig. 3).

This sheet is generated by centres of inversion with $x = \frac{3}{4}$, and it occupies the domain $0.5 < x < 1.0$; a second sheet, related to the first by the *C*-centring operation, is generated by

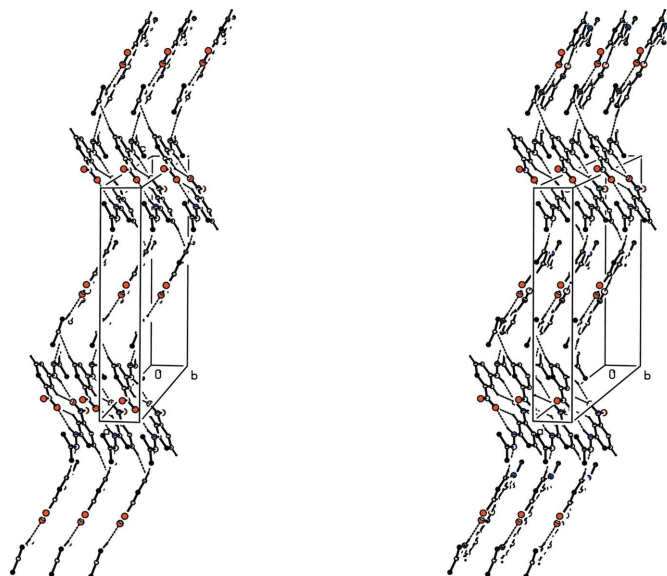


Figure 3
A stereoscopic view of part of the crystal structure of (I), showing the formation of a hydrogen-bonded (dashed lines) sheet parallel to (100). For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

centres of inversion with $x = \frac{1}{4}$, and it occupies the domain $0 < x < 0.5$; however, there are no direction-specific interactions between adjacent sheets.

Experimental

3-Nitrohydrazine hydrochloride (3 mmol) was dissolved in acetone (30 ml) and the solution was then heated under reflux for 1 h. The solution was cooled and the excess solvent was removed under reduced pressure. The resulting solid product, (I), was crystallized from ethanol.

Crystal data

$C_9H_{11}N_3O_2$
 $M_r = 193.21$
Monoclinic, $C2/c$
 $a = 22.7837$ (15) Å
 $b = 3.8307$ (2) Å
 $c = 21.7292$ (13) Å
 $\beta = 100.129$ (3)°
 $V = 1866.91$ (19) Å³

$Z = 8$
 $D_x = 1.375$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 120$ (2) K
Lath, colourless
 $0.20 \times 0.06 \times 0.02$ mm

Data collection

Bruker-Nonius KappaCCD
diffractometer
 φ and ω scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)
 $T_{\min} = 0.977$, $T_{\max} = 0.998$

10187 measured reflections
2117 independent reflections
1392 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.079$
 $\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.146$
 $S = 1.03$
2117 reflections
129 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 1.9052P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.30$ e Å⁻³

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O31^i$	0.84	2.35	3.144 (2)	158
$C2-H2\cdots O31^i$	0.95	2.49	3.305 (3)	143
$C5-H5\cdots N2^{ii}$	0.95	2.61	3.546 (3)	167

Symmetry codes: (i) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$; (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.

All H atoms were located in difference maps and then treated as riding atoms with distances $C-H = 0.95 \text{ \AA}$ or 0.98 \AA and $N-H = 0.84 \text{ \AA}$, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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