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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 σ(C–C) = 0.003 Å
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.

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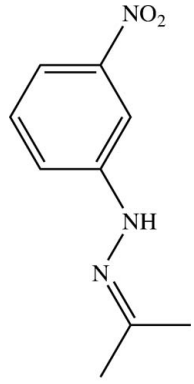
Molecules of the title compound, $C_9H_{11}N_3O_2$, are linked into sheets by a combination of N—H···O, C—H···O and C—H···N hydrogen bonds.

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.



(I)

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 $R_2^1(6)$ 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

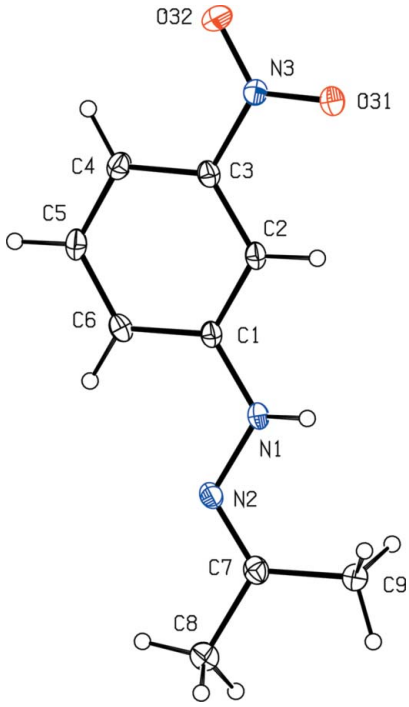


Figure 1
A molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

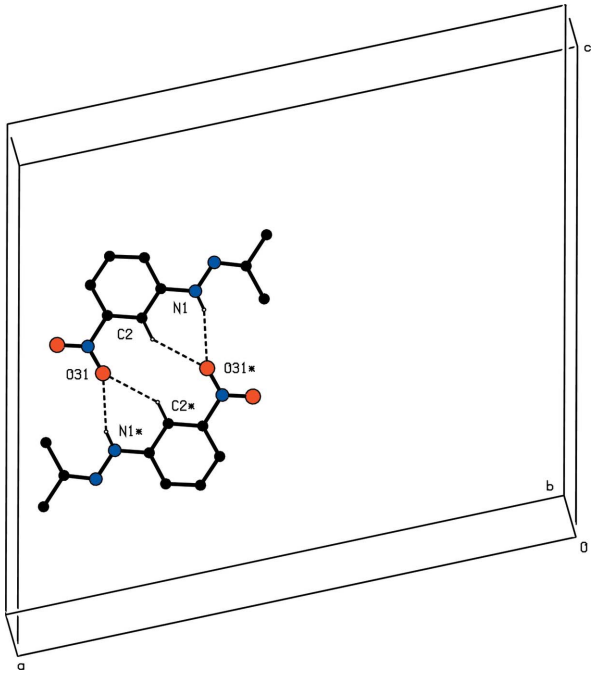


Figure 2
The molecular structure of (I), showing the formation of a centrosymmetric dimer containing three edge-fused hydrogen-bonded (dashed lines) rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted. The atoms marked with an asterisk (*) are at the symmetry position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

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

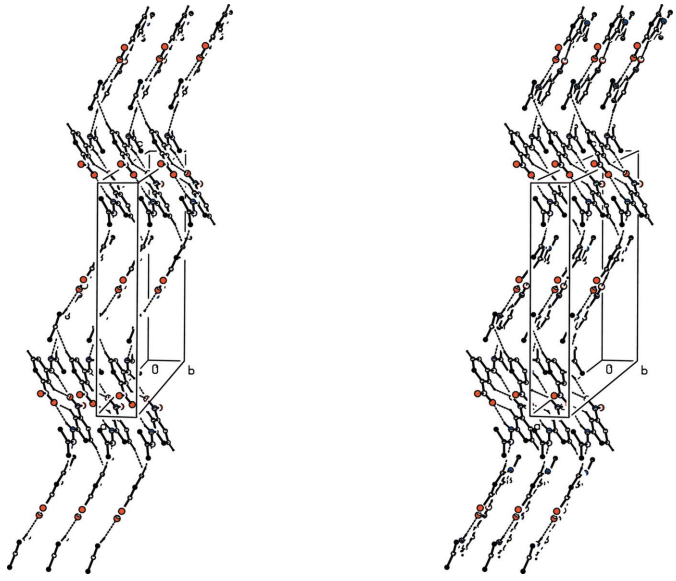


Figure 3
A stereoscopic view of part of the crystal structure of (I), showing the formation of a hydrogen-bonded (dashed lines) sheet parallel to (100). For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

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
$\beta = 100.129\ (3)^\circ$
$V = 1866.91\ (19)$ Å^3
$Z = 8$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 120\ (2)$ K
Lath, colourless
0.20 × 0.06 × 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 $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 27.5^\circ$

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···A	D—H	H···A	D···A	D—H···A
N1—H1···O31^{i}	0.84	2.35	3.144 (2)	158
C2—H2···O31^{i}	0.95	2.49	3.305 (3)	143
C5—H5···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 Å or 0.98 Å and N—H = 0.84 Å, 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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