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3,4-Lutidine

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Key indicators

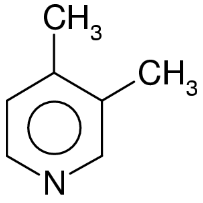
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.053
wR factor = 0.135
Data-to-parameter ratio = 19.1

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

The crystal structure of 3,4-lutidine (3,4-dimethylpyridine, C_7H_9N), has been determined at 150 (2) K following *in situ* crystal growth from the liquid. In space group *C*2/*c*, there are four independent molecules in the asymmetric unit, linked into dimers *via* C—H···N interactions.

Comment

As part of a study devoted to improving the techniques for determining the crystal structures of substances that are liquids at room temperature, we have reported previously the crystal structures of 2,6-lutidine (Bond *et al.*, 2001), 3,5-lutidine (Bond & Davies, 2002*a*) and 2,5-lutidine (Bond & Davies, 2002*b*). We report here the crystal structure of 3,4-lutidine, (I), determined at 150 (2) K following *in situ* crystal growth from the liquid.



(I)

Compound (I) crystallizes in the monoclinic space group *C*2/*c* with four independent molecules in the asymmetric unit (Fig. 1). Within the asymmetric unit, molecules are linked into dimers *via* C—H···N interactions (Table 1). In the structures of the other lutidines reported to date, molecules are linked into linear chains *via* C—H···N interactions involving the H atom at the 4-position; this interaction is clearly prohibited in (I). Two orientations may be envisaged for dimerization in which a centrosymmetric motif would result: either both molecules interact through the H atoms at their 2-positions, or both interact through the H atoms at their 6-positions. The observed dimer involves one molecule of (I) interacting through H at the 2-position and one interacting through H at the 6-position, giving rise to an asymmetric motif (point symmetry 1). In both independent dimers within the asymmetric unit, the interaction C6—H6···N1 is significantly shorter and closer to linear than the C2—H2···N1 interaction (Table 1). This is not obviously an intra-dimer effect and may be a result of inter-dimer interactions between methyl substituents. The dimers may be considered to form layers parallel to (001), with the planes through the dimer units lying alternately parallel to (110) and ($\bar{1}$10) in adjacent layers (Figs. 2 and 3).

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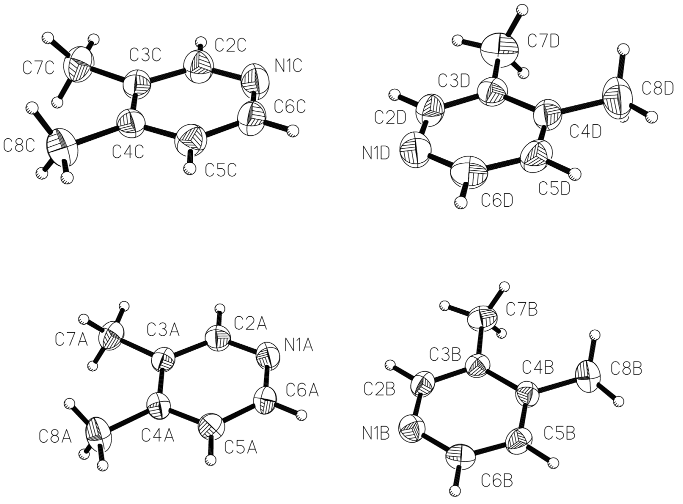

Figure 1
The asymmetric unit and atom-labelling scheme in (I), showing displacement ellipsoids (C/N atoms) at the 50% probability level (*XP*; Sheldrick, 1993). Independent molecules are denoted by the suffixes *A*, *B*, *C* and *D*.

Experimental

The sample (99%) was obtained from the Aldrich company and used without further purification. The crystal was grown in a 0.3 mm glass capillary tube at *ca* 235 K (a temperature only slightly less than the melting point of the solid in the capillary) using a technique described earlier (Davies & Bond, 2001). Once grown, the crystal was cooled to 150 (2) K for data collection. The length of the cylindrical crystal was not estimated, but it exceeded the diameter of the collimator (0.35 mm).

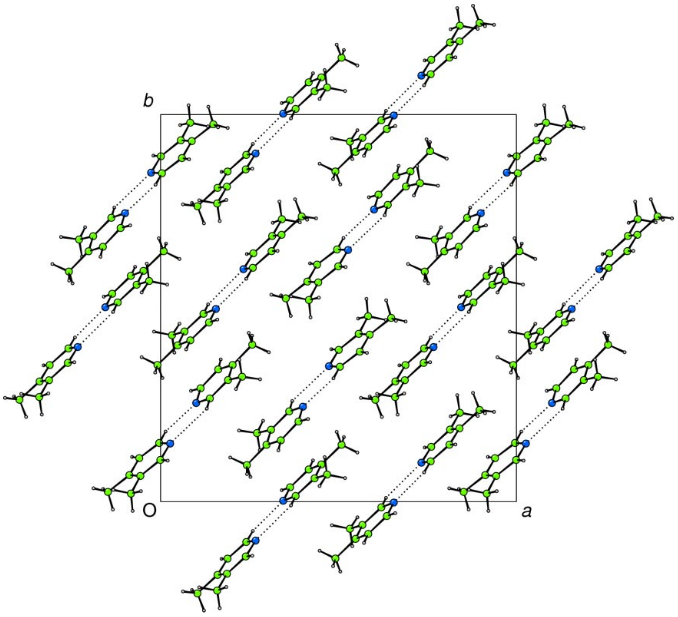

Figure 2
Projection on to (001) of a single layer in (I), showing dimers linked by C—H···N interactions orientated parallel to (110) (*CAMERON*; Watkin *et al.*, 1996).

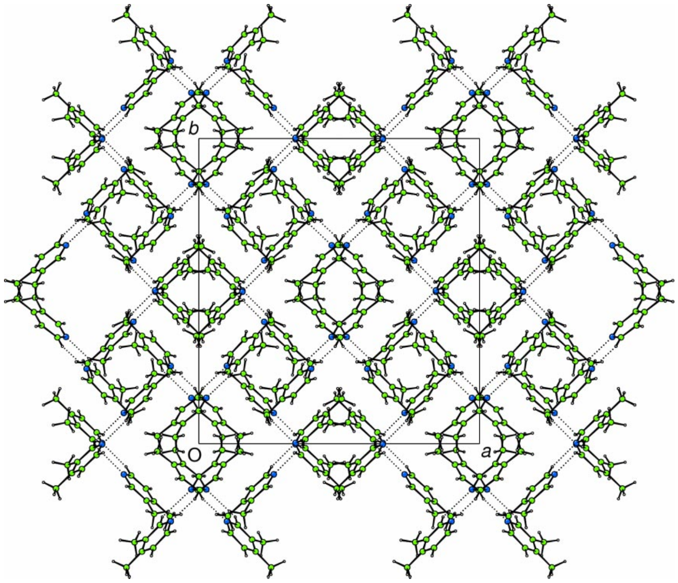

Figure 3
Projection on to (001) of the entire structure of (I), showing layers of dimers oriented alternately parallel to (110) and ($\bar{1}$10) (*CAMERON*; Watkin *et al.*, 1996).

Crystal data

C_7H_9N
$M_r = 107.15$
Monoclinic, $C2/c$
$a = 20.9006$ (8) Å
$b = 20.6112$ (9) Å
$c = 12.9921$ (3) Å
$\beta = 115.135$ (2)°
$V = 5066.9$ (3) Å^3
$Z = 32$

$D_x = 1.124$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9883 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.07$ mm^{-1}
$T = 150$ (2) K
Cylinder, colourless
0.15 mm (radius)

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: none
9752 measured reflections
5740 independent reflections
4041 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.026$
$\theta_{\rm max} = 27.5°$
$h = 0 \rightarrow 26$
$k = 0 \rightarrow 26$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.135$
$S = 1.03$
5740 reflections
301 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 2.9233P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6A—H6A···N1B	0.95	2.48	3.368 (2)	156
C2B—H2B···N1A	0.95	2.83	3.630 (2)	143
C6C—H6C···N1D	0.95	2.45	3.339 (2)	156
C2D—H2D···N1C	0.95	2.88	3.661 (2)	140

H atoms were placed geometrically and refined with isotropic displacement parameters, with common parameters assigned to chemically equivalent H atoms (one parameter for all methyl H atoms, four parameters in total). Each methyl group was allowed to rotate about its local threefold axis.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*-92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1993) and *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL*97.

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