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2,3-Lutidine

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The crystal structure of 2,3-lutidine (2,3-dimethylpyridine, C_7H_9N) has been determined at 150 (2) K, following *in situ* crystal growth from the liquid. Molecules are linked into polar chains *via* C—H···N interactions. The structure is best described as disordered in space group *C*2/*c*, with half a molecule in the asymmetric unit.

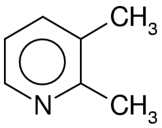
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.050
wR factor = 0.157
Data-to-parameter ratio = 33.3

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

Comment

As part of a study devoted to improving the techniques for determining the crystal structures of substances that are liquid at room temperature, we have reported previously the structures of all but one of the lutidine (dimethylpyridine) isomers (Bond *et al.*, 2001; Bond & Davies, 2002*a*,*b*,*c*; Bond & Parsons, 2002). Reported here is the structure of the remaining isomer, 2,3-lutidine, (I), determined at 150 (2) K, following *in situ* crystal growth from the liquid.



(I)

In space group *C*2/*c*, the asymmetric unit of (I) comprises half a molecule sited on a twofold axis that bisects the methyl-substituted positions and the opposite bond of the pyridine ring (Fig. 1). This leads to a disordered description of the structure in which the N atom and C—H group in the 4-position are overlaid, with their site-occupancy factors constrained to be 0.5. Discounting the energetically unfavourable possibility of two N atoms in adjacent molecules being brought into close contact, and likewise two C—H groups, the structure contains polar chains linked by C—H···N interactions [H4···N1$^{\mathrm{i}}$ = 2.55 Å, C4—H4···N1$^{\mathrm{i}}$ = 162.0°; symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}-y, -z$]. Similar chains are observed in the 2,6-, 2,5- and 3,5-isomers. The chains in (I) are arranged in layers parallel to ($10\overline{1}$) (Fig. 2). The significant distortion from linearity of the C—H···N interactions presumably accommodates the steric demands of the methyl substituents in adjacent chains. This disordered model suggests that chains are aligned in an antiparallel manner. It is not possible to comment definitively on the local nature of the alignment; adjacent chains within a given layer may always be parallel with their neighbours, but adjacent layers may be aligned in an antiparallel manner. Alternatively, there may be antiparallel alignment within layers, or even domains of parallel alignment distributed through the crystal.

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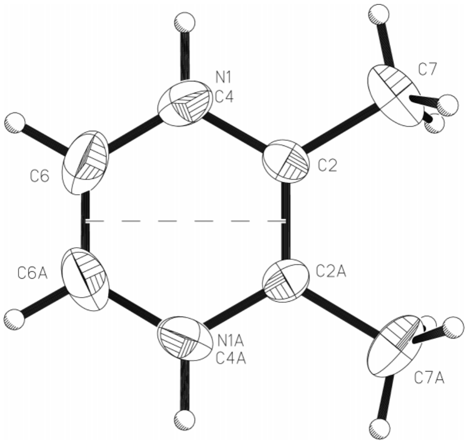

Figure 1
The molecular unit and displacement ellipsoids (50% probability) for (I) in space group *C*2/*c*. The dashed line denotes the direction of the crystallographic twofold axis, and symmetry-equivalent atoms are denoted by the suffix *A*.

An alternative description of the structure was examined in the polar space group *Cc* with a whole molecule in the asymmetric unit. This leads to a largely satisfactory refinement with $R[F^2 > 2\sigma(F^2)] = 0.053$ and $wR(F^2) = 0.149$, although the displacement ellipsoids of the N atom and the C atom in the 4-position of the ring appear slightly large and slightly small, respectively (Fig. 3). This is indicative of some disorder between the two positions, *i.e.* not all polar chains are aligned in a parallel manner. In view of this fact, and given that absolute structure determination is unfeasible, we believe that the disordered centrosymmetric model is most satisfactory.

Experimental

The sample (99%) was obtained from the Aldrich Co. and used without further purification. The crystal was grown in a 0.3 mm glass capillary tube at *ca* 257.5 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).

Crystal data

C_7H_9N
$M_r = 107.15$
Monoclinic, *C*2/*c*
$a = 11.4158$ (7) Å
$b = 7.5787$ (5) Å
$c = 7.4714$ (4) Å
$\beta = 101.900$ (4)°
$V = 632.51$ (7) Å^3
$Z = 4$

$D_x = 1.125$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3561 reflections
$\theta = 1.0$–35.0°
$\mu = 0.07$ 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
2468 measured reflections
1366 independent reflections
1035 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.023$
$\theta_{\rm max} = 35.3°$
$h = -18 \rightarrow 18$
$k = -12 \rightarrow 11$
$l = -11 \rightarrow 11$

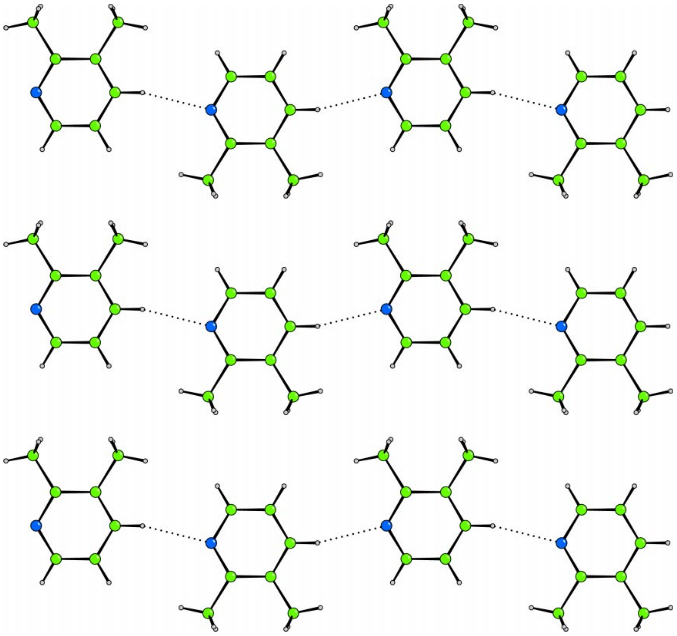

Figure 2
View of (I), projected on to (10$\overline{1}$), showing a single layer of chains linked *via* C—H···N interactions. Adjacent chains are shown arbitrarily to be coparallel.

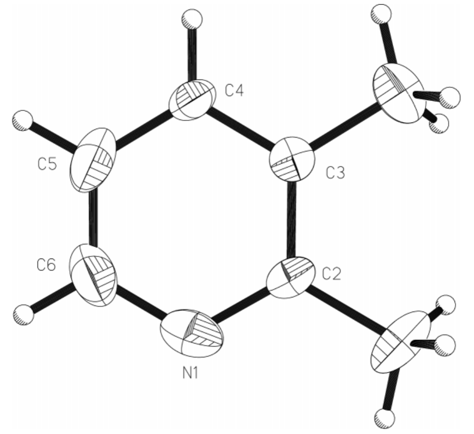

Figure 3
The molecular unit and displacement ellipsoids (50% probability) for (I) in space group *Cc*. The large/small displacement parameters for N1 and C4 indicate that the structure is best described as disordered in space group *C*2/*c* (see *Comment*).

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.157$
$S = 1.08$
1366 reflections
41 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0759P)^2 + 0.1323P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}

All H atoms were placed geometrically and independent isotropic displacement parameters were refined (one common displacement parameter for the methyl H atoms). 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 (Sheldrick, 1997).

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