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Key indicators

Single-crystal X-ray study
 $T = 120$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.040
 wR factor = 0.112
 Data-to-parameter ratio = 15.1

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

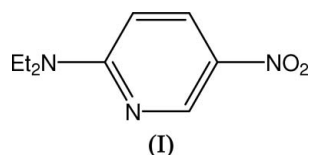
N,N-Diethyl-5-nitropyridin-2-amine

In the title compound, $\text{C}_9\text{H}_{13}\text{N}_3\text{O}_2$, the asymmetric unit contains two almost identical but crystallographically independent molecules. The molecules are linked together by pairs of weak $\text{C}-\text{H}\cdots\text{O}$ interactions into zigzag chains, which, in turn, form corrugated layers perpendicular to the a axis.

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Comment

In continuation of our studies of organic molecules with non-linear optical properties (Yufit *et al.*, 2006), an attempt to grow crystals of 2-adamantylamino-5-nitropyridine (AANP) (Tomaru *et al.*, 1991; Antipin *et al.*, 2001) by sublimation has been made. As a result, two types of crystals formed in the reaction vessel. An X-ray study of the small cubic-shaped ones revealed that they are, in fact, crystals of *N,N*-diethyl-5-nitropyridine-2-amine, (I), which is a side product in the synthesis of AANP. Here, we briefly describe the structural features of this compound.



The asymmetric unit of (I) contains two virtually identical but crystallographically independent molecules (Fig. 1). These molecules differ slightly in the positions of the terminal C atoms of the ethyl groups (Fig. 2).

The packing of the molecules of (I) is quite different from that of its benzene analogue *N,N*-diethyl-*p*-nitroaniline (Maurin & Krygowski, 1988), in which numerous $\text{C}-\text{H}\cdots\pi$ interactions are present.

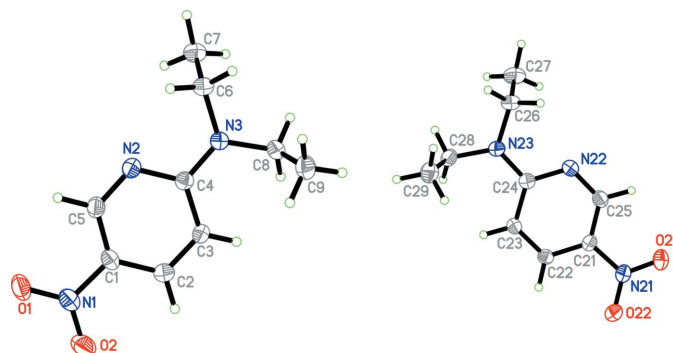


Figure 1
 The asymmetric unit of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

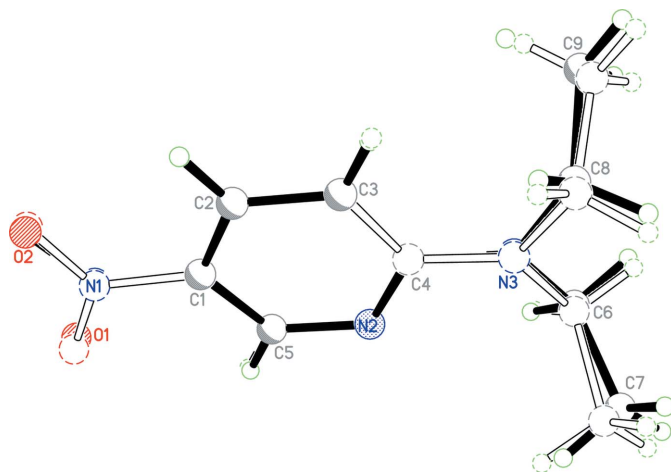


Figure 2
A least-squares fit of the pyridine rings of the two independent molecules.

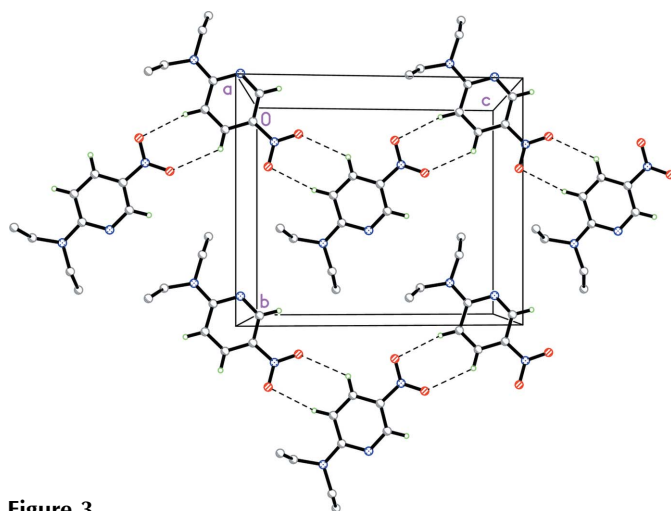


Figure 3
The chains of molecules of (I) in a single layer. H atoms of ethyl groups have been omitted for clarity. Dashed lines indicate hydrogen bonds.

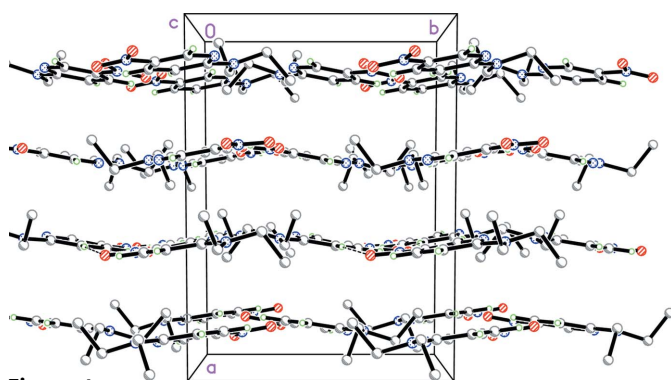


Figure 4
A packing diagram for (I), viewed down the *a* axis.

In the crystal structure, molecules are linked together by pairs of C—H...O [$O1^i \cdots H2(-C2) = 2.74(1) \text{ \AA}$, $O2^i \cdots H3(-C3) = 2.63(1) \text{ \AA}$, $O21^{ii} \cdots H22(-C22) = 2.67(1) \text{ \AA}$ and $O22^{ii} \cdots H23(-C23) = 2.51(1) \text{ \AA}$; symmetry codes: (i) $x, \frac{5}{2} - y, \frac{1}{2} + z$, (ii) $x, \frac{5}{2} - y, -\frac{1}{2} + z$] interactions in zigzag chains parallel to the *c* axis (Fig. 3); each independent molecule forms separate chains. These chains form corrugated

layers perpendicular to *a* axis (Fig. 4). The pyridine rings of the molecules in adjacent layers are partially overlapped, the shortest interplanar distance being 3.42 \AA , which is within the normal range for π - π aromatic interactions (Janiak, 2000).

Experimental

The title compound, (I), was isolated by chromatography from the mixture of products of the reaction between 2-chloronitropyridine (0.948 g, 5.979 mmol) and adamantylamine (0.984 g, 6.594 mmol). The crystals of (I) were formed on heating the powder sample at 343 K for 3 d (yield 0.99 g, 61%; m.p. 440 K).

Crystal data

$C_9H_{13}N_3O_2$
 $M_r = 195.22$
Monoclinic, $P2_1/c$
 $a = 14.6723(7) \text{ \AA}$
 $b = 10.6920(5) \text{ \AA}$
 $c = 12.4224(5) \text{ \AA}$
 $\beta = 96.820(1)^\circ$
 $V = 1934.99(15) \text{ \AA}^3$
 $Z = 8$

$D_x = 1.340 \text{ Mg m}^{-3}$
Mo- $K\alpha$ radiation
Cell parameters from 4483 reflections
 $\theta = 2.4\text{--}30.4^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 120(2) \text{ K}$
Block, yellow
 $0.26 \times 0.14 \times 0.12 \text{ mm}$

Data collection

Bruker SMART CCD 6000 diffractometer
 ω scans
Absorption correction: none
19597 measured reflections
5389 independent reflections

3536 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.048$
 $\theta_{max} = 29.5^\circ$
 $h = -20 \rightarrow 20$
 $k = -14 \rightarrow 14$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.112$
 $S = 0.97$
5389 reflections
357 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.29 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.19 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—N1	1.2387 (14)	N3—C8	1.4658 (15)
O2—N1	1.2378 (15)	N3—C6	1.4692 (16)
O21—N21	1.2368 (14)	N21—C21	1.4333 (15)
O22—N21	1.2356 (14)	N22—C25	1.3276 (17)
N1—C1	1.4365 (16)	N22—C24	1.3612 (16)
N2—C5	1.3263 (16)	N23—C24	1.3509 (15)
N2—C4	1.3626 (15)	N23—C28	1.4620 (17)
N3—C4	1.3455 (15)	N23—C26	1.4660 (16)
O2—N1—O1	122.69 (11)	O22—N21—C21	118.76 (10)
O2—N1—C1	118.57 (11)	O21—N21—C21	118.83 (11)
O1—N1—C1	118.74 (11)	C25—N22—C24	117.57 (11)
C5—N2—C4	117.73 (11)	C24—N23—C28	121.83 (10)
C4—N3—C8	121.56 (10)	C24—N23—C26	121.11 (11)
C4—N3—C6	121.56 (10)	C28—N23—C26	117.04 (10)
C8—N3—C6	116.44 (10)	N3—C4—N2	116.80 (11)
O22—N21—O21	122.40 (11)	N3—C4—C3	121.76 (11)

H atoms were located in a difference synthesis and refined isotropically [$C-H = 0.922(14)\text{--}0.976(14)$ for CH, $0.965(13)\text{--}1.007(14)$ for CH_2 and $0.970(17)\text{--}1.028(16) \text{ \AA}$ for CH_3].

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine

structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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