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Key indicators

Powder X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.090 \text{ \AA}$
R factor = 0.19

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

4-(4'-Dimethylaminostyryl)pyridine N-oxide from powder data

The crystal structure of 4-(4-dimethylaminophenylethenyl)pyridine N-oxide, $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}$, has been determined from X-ray laboratory powder diffraction data. The powder pattern was indexed with a monoclinic unit cell with a long *a* axis. The structure was solved by a grid search technique. The subsequent bond-restrained Rietveld refinement gave bond lengths and angles within expected ranges.

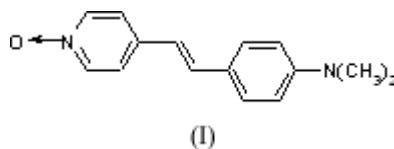
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Comment

4-(4'-Dimethylaminostyryl)pyridine N-oxide, (I), is an unusual substance. It has two potential coordinating groups, N—O and NMe_2 , which are conjugated with each other through an extensive π -system. The question of the first site of protonation has been discussed previously (Titski *et al.*, 1996; Korzhenevskaya & Rybatschenko, 1999).



Compound (I) is the subject of much research devoted to the investigation of complexes formed with Lewis acids (Andreev *et al.*, 2002), intramolecular charge transfer (Andreev *et al.*, 1998), nucleophilic properties (Rybatschenko *et al.*, 2001; Lobanova, 2001), and biological activity (Anisimov *et al.*, 2000). This compound has apoptogenic and erythroid differentiation induction activity towards K-562 cells (Volkova *et al.*, 2001). According to our data, the molecule of (I) studied here is the *trans* isomer. The IR spectrum contains a band at 970 cm^{-1} , which shows the presence of a *trans* $\text{H}-\text{C}=\text{C}-\text{H}$ group. The NMR spectrum contains two signals for vicinal olefin protons with $J = 16 \text{ Hz}$, characteristic of a *trans* isomer (Silverstein *et al.*, 1977).

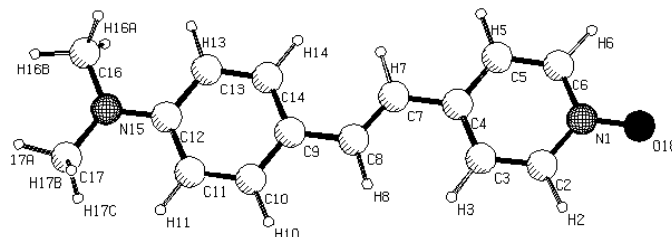


Figure 1
The molecular structure of (I), with the atomic numbering.

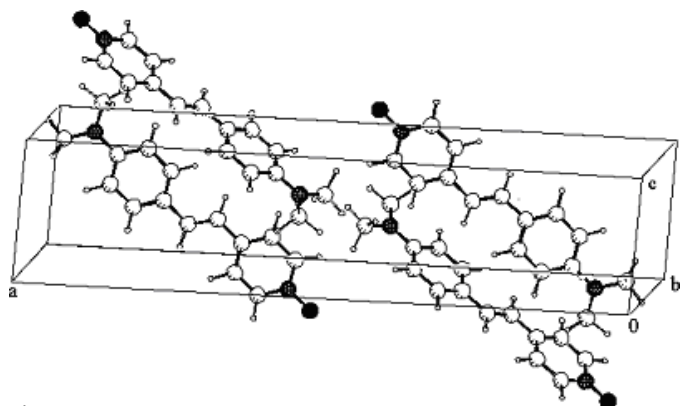


Figure 2
A packing diagram for (I).

Experimental

Compound (I) was synthesized according to the method of Titski & Turovskaya (1990). IR spectra were measured from KBr discs, using a Specord M-80 spectrometer. NMR spectra were measured with a Bruker 300 spectrometer (300 MHz), in CDCl_3 solution. Electronic spectra were measured using a Specord UV-vis spectrometer, in 96% ethanol and in chloroform.

Crystal data

$\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}$	Wavelength of incident radiation:
$M_r = 240.2$	1.54056 Å
Monoclinic, $P2_1/n$	Cell parameters from 33 reflections
$a = 26.822$ (4) Å	$\theta = 8.9\text{--}34.4^\circ$
$b = 7.758$ (2) Å	$\mu = 0.64$ mm $^{-1}$
$c = 6.079$ (3) Å	$T = 293$ (2) K
$\beta = 94.03$ (1) $^\circ$	Yellow
$V = 1262$ (1) Å 3	Specimen shape: flat sheet
$Z = 4$	$20 \times 20 \times 0.1$ mm
$D_x = 1.266$ Mg m $^{-3}$	Particle morphology: no specific habit
Cu $K\alpha_1$ radiation	

Data collection

Philips Analytical XPert PRO X-ray diffraction system diffractometer	$h = 0 \rightarrow 17$
Specimen mounted in reflection mode	$k = 0 \rightarrow 5$
Absorption correction: none	$l = 0 \rightarrow 3$
$\theta_{\text{max}} = 30.0^\circ$	$2\theta_{\text{min}} = 6.00$, $2\theta_{\text{max}} = 60.00^\circ$
	Increment in $2\theta = 0.01^\circ$

Refinement

Refinement on I_{net}	$w(i) = 1/I_{\text{obs}}(i)$ at each point i
$R_p = 0.19$	$(\Delta/\sigma)_{\text{max}} = 0.01$
$R_{\text{wp}} = 0.29$	$\Delta\rho_{\text{max}} = 0.54$ e Å $^{-3}$
$R_{\text{exp}} = 0.19$	$\Delta\rho_{\text{min}} = -0.43$ e Å $^{-3}$
$S = 1.5$	Preferred orientation correction:
Profile function: split-type pseudo-Voigt (Toraya, 1986)	Spherical harmonics expansion (Ahtee <i>et al.</i> , 1989) up to the 6th order.
162 parameters	
H-atom parameters constrained	

Two X-ray powder diffraction patterns were measured in transmission mode on a Giniuer-de-Wolf diffractometer and in reflection mode on an XPert X-ray powder diffraction system equipped with a standard resolution goniometer PW 3050/60 and proportional point detector PW 3011/20. The powder was sprinkled on the sample holder using a fine sieve to avoid preferred orientation. The thickness of the sample was no more than 0.1 mm. During the exposures the specimen was spun in its plane to improve particle statistics. The unit-cell

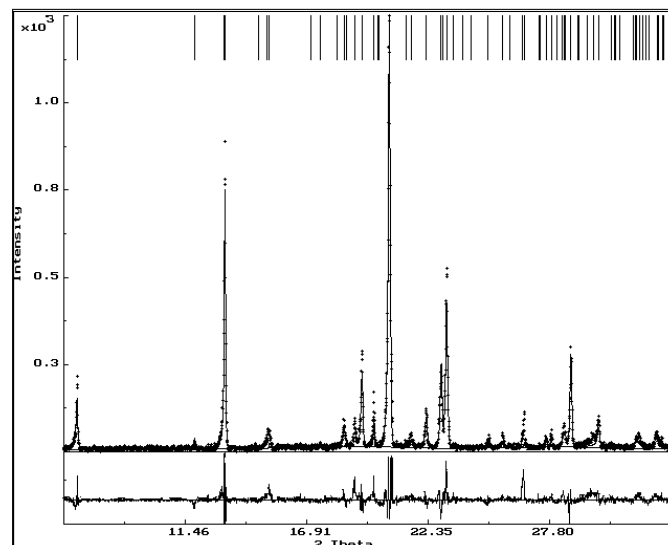


Figure 3
The Rietveld plot for (I), showing the observed and difference profiles. The reflection positions are shown above the observed profile.

dimensions were determined with the indexing program *TREOR* (Werner *et al.*, 1985) with $M_{20} = 14$ and $F_{30} = 31$, using the first 30 peak positions. The structure was solved by the grid-search procedure (Chernyshev & Schenk, 1998) and refined with the use of bond restraints by the *MRIA* program (Zlokazov & Chernyshev, 1992). The strength of restraints was a function of interatomic separation and, for intramolecular bond lengths, corresponds to an r.m.s. deviation of 0.03 Å. An additional restraint was applied to the planarity of the $\text{C}_{15}\text{N}_2\text{O}$ fragment. H atoms were placed in geometrically calculated positions and allowed to refine using riding model constraints, with a common isotropic displacement parameter U_{iso} fixed at 0.05 Å 2 . A single overall U_{iso} parameter for non-H atoms was refined. The diffraction profiles and the differences between the measured and calculated profiles are shown in Fig. 3.

Data collection: local program; data reduction: local program; program(s) used to solve structure: *MRIA* (Zlokazov & Chernyshev, 1992); program(s) used to refine structure: *MRIA*; software used to prepare material for publication: *MRIA* and *PARST* (Nardelli, 1983).

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