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4,4′-Bis(di-*n*-butylaminostyryl)-2,2′-bipyridine: a space group revision

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Key indicators

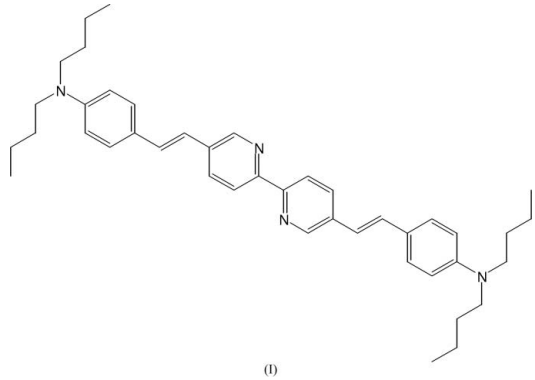
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.079
wR factor = 0.270
Data-to-parameter ratio = 17.6

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

The structure of the title compound, $C_{42}H_{54}N_4$, has originally been refined in space group $P1$ (No. 1) with two molecules in the asymmetric unit. However, successful refinement clearly demonstrates that the structure is better described in space group $P\overline{1}$ (No. 2) with two half molecules in the asymmetric unit, both molecules being located on centres of inversion.

Comment

The question of whether a structure is centrosymmetric or not is probably the most irksome problem affecting all crystallographers, because refining a centrosymmetric structure in a non-centrosymmetric space group will lead to serious problems (Marsh, 1995). In order to avoid these problems symmetry-checking programs such as *MISSYM* (Le Page, 1988) should always be used to check that no centre of inversion has been overlooked. These programs are easily available as stand-alone programs, e.g *PLATON* (Spek, 2003), or *via* the Internet, *e.g.* http://journals.iucr.org/services/cif/checking/checkform.html. As a result, it should be unlikely that nowadays a structure is published in an incorrect space group. However, the title compound was published in space group $P1$ (No. 1) with Z = 2 (Maury *et al.*, 2001). The authors noted that the two molecules in the asymmetric unit had a pseudo-centre of symmetry in the mid-point of the bond connecting the two pyridyl rings. Furthermore, the structure did not refine satisfactorily; the absolute maximum value of parameter shift to s.u. ratio was 0.851. The poor convergence of the refinement in this case is a result of the overlooked inversion centre. Several bond lengths adopted strange values (*e.g.* C46—C47 = 1.01 Å, C80—C81 = 1.09 Å, C20—C21 = 0.84 Å and C77—C78 = 1.73 Å) and several atoms had strange displacement parameters (*e.g.* C20, C21, C46, C47, C80, C81 and C111; Figs. 3 and 4).



(I)

Refining the structure in space group $P\overline{1}$ gives significantly better results (Table 1, and Figs. 1 and 2). The data-to-para-

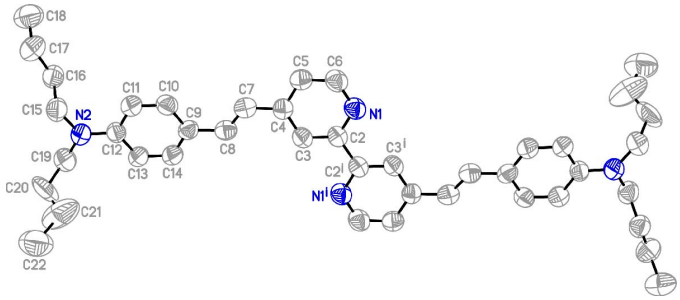


Figure 1
Molecule 1 of the title compound after refinement in $P\overline{1}$ (present work). Displacement ellipsoids are drawn at the 50% probability level. H atoms and one of the two disordered sets of atoms have been omitted for clarity. [Symmetry code (i): $-x, -1-y, -z$.]

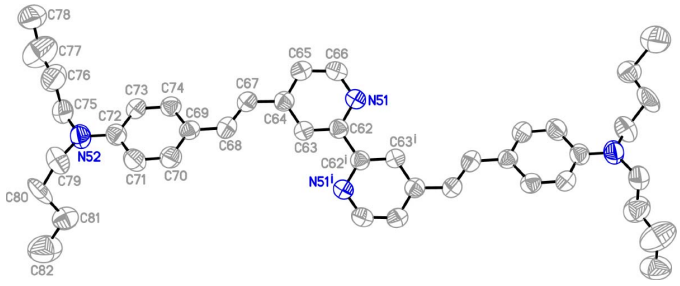


Figure 2
Molecule 2 of the title compound after refinement in $P\overline{1}$ (present work). Displacement ellipsoids are drawn at the 50% probability level. H atoms and one of the two disordered sets of atoms have been omitted for clarity. [Symmetry code (i): $-x, -y, -z$.]

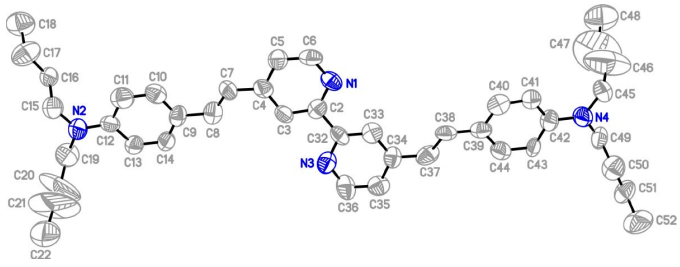


Figure 3
Molecule 1 of the title compound after refinement in *P*1 (Maury *et al.* 2001). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

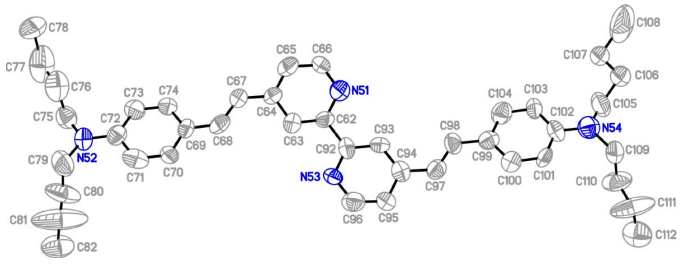


Figure 4
Molecule 2 of the title compound after refinement in *P*1 (Maury *et al.* 2001). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

meter ratio is increased from 10.2 to 17.6, and, as a result, the s.u. values are significantly lower. Furthermore, geometric parameters that should be in the same range differ significantly when the structure is refined in *P*1, but these values are either exactly equal because a centre of inversion is imposed on the structure or they adopt similar values if the correct space group ($P\overline{1}$) is chosen. In addition, strange bond lengths are avoided by using a disorder model.

Experimental

The coordinates were obtained from the website of the New Journal of Chemistry. The atom names were not changed in order to facilitate comparison between the two refinements. The reflection data were provided by the Editorial Office of New Journal of Chemistry.

Crystal data

$C_{42}H_{54}N_4$
$M_r = 614.89$
Triclinic, $P\overline{1}$
$a = 11.915\ (3)$ Å
$b = 12.050\ (4)$ Å
$c = 13.544\ (2)$ Å
$\alpha = 105.67\ (2)^\circ$
$\beta = 99.141\ (10)^\circ$
$\gamma = 93.40\ (2)^\circ$
$V = 1837.8\ (8)$ Å^3
$Z = 2$
$D_x = 1.111$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\mu = 0.07$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.45 × 0.35 × 0.32 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
8328 measured reflections
7957 independent reflections
3290 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 27.0^\circ$
$h = 0 \rightarrow 15$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.080$
$wR(F^2) = 0.270$
$S = 1.02$
7957 reflections
453 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1309P)^2 + 0.0377P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Results of the refinement in $P\overline{1}$ and *P*1.

	$P\overline{1}$	*P*1
Reflections	7957	8401
Parameters	453	820
$R[F^2 > 2\sigma(F^2)]$	0.0795	0.0776
$wR(F^2)$	0.2702	0.3179
S	1.023	0.928
$(\Delta/\sigma)_{max}$	<0.001	0.851
N1—C2	1.339 (4)	1.366 (13)
N1—C6	1.325 (4)	1.295 (14)
N3—C32	–	1.311 (12)
N3—C36	–	1.383 (15)
N51—C62	1.340 (4)	1.349 (12)
N51—C66	1.330 (4)	1.315 (14)
N53—C92	–	1.324 (12)
N53—C96	–	1.353 (14)
C6—N1—C2	116.4 (3)	114.0 (9)
C32—N3—C36	–	119.4 (9)
C66—N51—C62	115.7 (3)	116.0 (8)
C92—N53—C96	–	115.8 (9)

H atoms were geometrically positioned and refined with fixed individual displacement parameters (set to 1.2 times U_{eq} of the parent atom or 1.5 for methyl groups) using a riding model, with C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively. The methylene groups C20, C21, C80 and C81 were refined as disordered over two positions. The site occupation factors refined to 0.551 (15) for C20 and C21, and to 0.464 (13) for C80 and C81. In addition, equivalent distances of the disordered groups were restrained to be equal.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1990); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; method used to solve structure: coordinates taken from previous study; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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