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1,8-Bis(dimethylethyleneguanidino)naphthalene

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.003 Å
R factor = 0.060
wR factor = 0.169
Data-to-parameter ratio = 18.1

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

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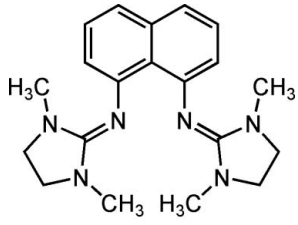
The title compound, $C_{20}H_{26}N_6$, obtained from the reaction of *o*-phenylenediamine with commercially available 2-chloro-1,3-dimethylimidazolinium chloride, crystallizes with three independent molecules in the asymmetric unit. In each molecule, the two nitrogen heterocycles are *syn* oriented with respect to the naphthalene fragment and have centroid–centroid separations in the range 3.280 (4)–3.395 (4) Å.

Comment

In a continuation of our research in guanidine chemistry (Kitani *et al.*, 2005; Kawahata *et al.*, 2005; Ishikawa *et al.*, 2006; Kawahata, Shikii *et al.*, 2006), we report here the crystal structure of the title compound, (I), which has been reported previously as a white powder (Raab *et al.*, 2003).



(I)

The title compound crystallizes with three independent molecules, viz. *A*, *B* and *C* (Fig. 1), in the asymmetric unit. All bond lengths and angles in (I) are normal (Allen *et al.*, 1987) and comparable with those observed in related structures (Kawahata, Ito *et al.*, 2006). In each molecule, the two nitrogen heterocycles are *syn* oriented with respect to the naphthalene fragment. The distances between the centroids of the two nitrogen heterocycles in molecules *A*, *B* and *C* are 3.280 (4), 3.395 (4) and 3.391 (4) Å, respectively. The crystal packing is mainly stabilized by van der Waals forces.

Experimental

To an ice-cooled solution of 1,8-diaminonaphthalene (1.00 g, 6.3 mmol) and triethylamine (2.1 ml, 28 mmol) in absolute CH_2Cl_2 (10 ml) was added dropwise a solution of commercially available 2-chloro-1,3-dimethylimidazolinium chloride (Isobe *et al.*, 2000) (2.37 g, 14 mmol) in absolute CH_2Cl_2 (10 ml). The mixture was stirred at room temperature for 1 h. The reaction mixture was then extracted with toluene (80 ml) and 20% aqueous NaOH (30 ml × 3). The organic layer was washed with water (5.0 ml × 10) and brine (30 ml × 3), then dried (Na_2SO_4), and evaporated to give purple–brown crystals (2.18 g). Recrystallization from *n*-hexane gave pink blocks (yield 2.05 g, 92.4%; m.p. 428–429 K)..

Crystal data

$C_{20}H_{26}N_6$
M_r = 350.47
Monoclinic, $P2_1/n$
a = 8.4589 (12) Å
b = 36.168 (5) Å
c = 17.943 (2) Å
β = 92.642 (2)°
V = 5483.5 (13) Å^3
Z = 12
D_x = 1.274 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 90 K
Block, pink
0.40 × 0.20 × 0.18 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
33635 measured reflections
12973 independent reflections
7208 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.169
S = 1.10
12973 reflections
715 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.0265P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

H atoms were included in calculated positions (C—H = 0.95–0.99 Å) and treated as riding, with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

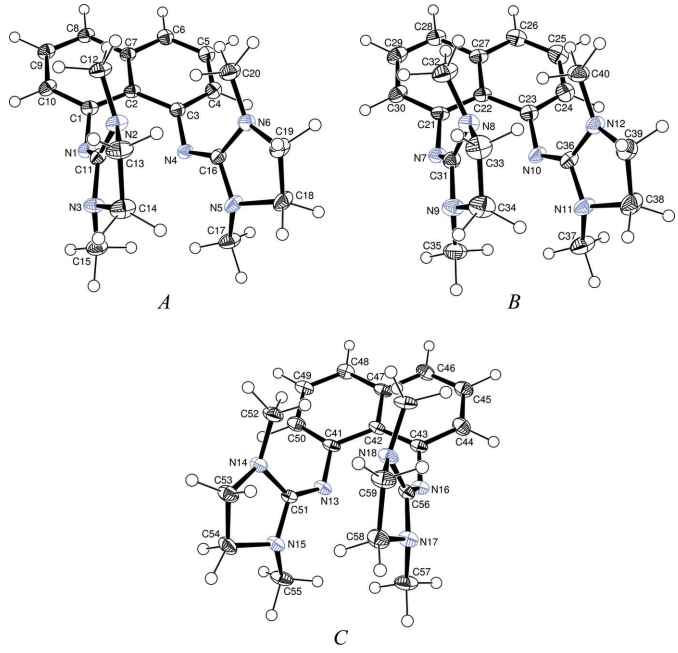


Figure 1
The three independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level.

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