Tris(pyrazol-1-yl)methane (Tpm)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.125
Data-to-parameter ratio = 14.5

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

The title compound, $C_{10}H_{10}N_6$, was crystallized from a methylene dichloride solution. Tpm has gained much attention of late as a neutral scorpionate ligand analogue of hydrotris(pyrazol-1-yl)borate. We report here the structure of the free Tpm ligand, which shows a structure typical of a tripodal neutral ligand. The compound crystallizes in a chiral space group despite the fact that it is achiral, ideally C_{3v}.

Comment

The ligand tris(pyrazol-1-yl)methane (Tpm), (I), was first reported by Trofimenko in 1970. Tpm has gained much attention of late as an isoelectronic neutral scorpionate ligand analogue of hydrotris(pyrazol-1-yl)borate (Tp^-) (Reger, 1999; Reger & Grattan, 2003). We report here the structure of the free Tpm ligand. The molecule is achiral in solution with idealized C_{3v} ($3m$) symmetry but crystallizes in the space group $P2_12_12_1$ with four molecules in the asymmetric unit. Examination of the atomic coordinates with the program *ADDSYM* of the *PLATON* suite of programs (Spek, 2002) did not suggest the presence of any additional symmetry. The loss of symmetry in the solid state is evident in the varying torsion of the pyrazole rings of the four crystallographically unique Tpm molecules (Table 1).

(I)
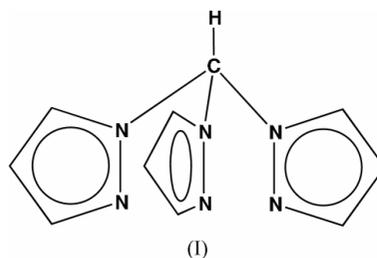
Experimental

The title compound was prepared according to the procedure of Reger *et al.* (2000). Crystals suitable for study (m.p. 371–375 K) were obtained by slow evaporation of a methylene dichloride solution. Spectroscopic analysis, ^{1}H NMR (CDCl$_3$, p.p.m.): 8.437 (*s*, 1H), 7.673 (*d*, 3H), 7.579 (*d*, 3H), 6.370 (*t*, 3H); ^{13}C NMR (CDCl$_3$, p.p.m.): 142.08, 129.77, 107.57, 83.45.

Crystal data

$C_{10}H_{10}N_6$	Mo $K\alpha$ radiation
M_r = 214.24	Cell parameters from 13354
Orthorhombic, $P2_12_12_1$	reflections
a = 10.7882 (5) Å	θ = 2.1–24.7°
b = 15.3791 (9) Å	μ = 0.09 mm^{-1}
c = 25.4634 (13) Å	T = 293 (2) K
V = 4224.7 (4) Å^3	Prism, colorless
Z = 16	0.24 × 0.10 × 0.05 mm
D_x = 1.347 Mg m^{-3}	

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Table 1
Selected torsion angles (°).

C10—N1—N4—C1	170.2 (2)	C30—N13—N16—C21	175.7 (2)
C10—N2—N5—C4	−179.8 (2)	C30—N14—N17—C24	172.31 (19)
C10—N3—N6—C7	178.15 (19)	C30—N15—N18—C27	−173.08 (19)
C20—N7—N10—C13	−178.4 (2)	C40—N19—N22—C31	176.53 (19)
C20—N8—N11—C14	−172.51 (19)	C40—N20—N23—C34	−175.1 (2)
C20—N9—N12—C17	−174.6 (2)	C40—N21—N24—C37	−174.26 (19)

Figure 1
Displacement ellipsoid plot of the asymmetric unit of (I), showing the four crystallographically unique molecules of Tpm.

The value of the absolute structure parameter (Flack, 1983) is meaningless for the title compound as Mo Kα radiation was employed and no atoms heavier than N are present. Friedel pair reflections were merged before the final refinement. H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined using a riding model, with $U_{iso}(H) = 1.2U_{iso}(C)$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).
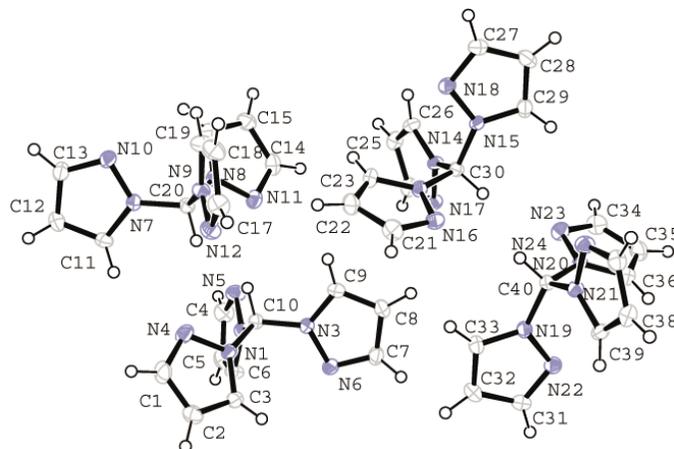
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