Crystal structure of 1,3,5-tris[(3,5-dimethylpyrazol)-1-ylmethyl]-2,4,6-trimethylbenzene, $C_{27}H_{36}N_6$

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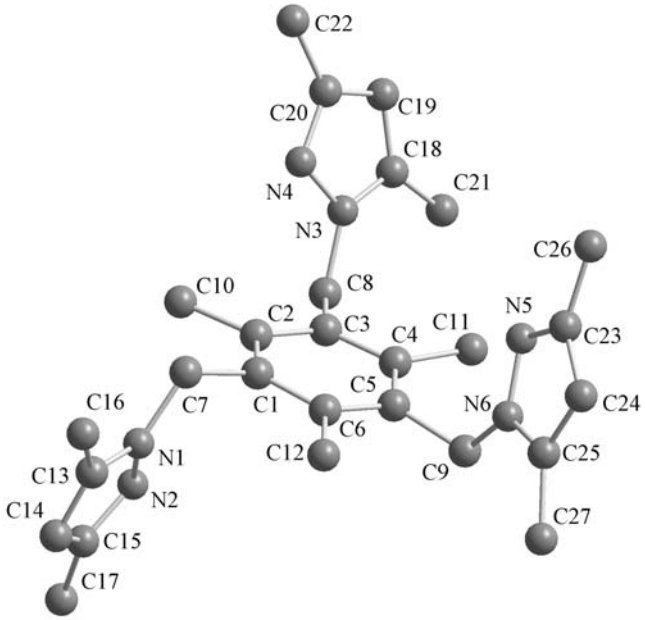
Received December 6, 2010, accepted and available on-line January 24, 2011; CCDC no. 1267/3319



Abstract

$C_{27}H_{36}N_6$, triclinic, $P\bar{1}$ (no. 2), a = 9.043(1) Å, b = 12.676(1) Å, c = 12.756(2) Å, α = 119.756(1)°, β = 99.166(2)°, γ = 91.012(1)°, V = 1245.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.130, T = 296 K.

Source of material

The title compound was prepared as described in literature [1], using 3,5-dimethylpyrazole instead of piperidin and recrystallized from methanol solution at room temperature to give the desired crystals suitable for single crystal X-ray diffraction.

Discussion

Tripodal ligands have been widely employed in many areas of inorganic chemistry [2]. The most common trigonal building blocks are rigid aryl-heterocyclic tripodal ligands [3,4]. Only few examples of flexible tripodal ligands are reported [5-7]. Compared with the rigid tripodal ligands, the flexible tripodal ligands have much more possible coordination modes due to the flexibility and low symmetry, and can adopt different conformations according to the geometric requirements of metal atoms.

In the title flexible tripodal ligand, the C atoms (methyl and (3,5-dimethyl-pyrazol)-1-ylmethyl) and benzene ring are approximately coplanar. The three pyrazol groups are arranged in an asymmetrical fashion with respect to the anchoring mesitylene ring, two pyrazol moieties located on one side of the central benzene ring and the other pyrazol moieties located on the opposite side of central benzene ring. The H atoms of all the methyl groups on the pyrazol and benzene rings are disordered. The dihedral angles between each pyrazol moiety are 121.1°, 101.1° and 38.5°, and the corresponding angles about benzene ring and pyrazol moiety are 100.4°, 80.2°, and 117.0°.

In the title crystal structure there exist four types of intermolecular C–H···N hydrogen bonds (d(H12F···N4) = 2.747 Å and ∠C12–H12F···N4=140.26°, d(H16F···N4) = 2.615 Å and ∠C16–H16F···N4=172.70°, d(H7B···N5) = 2.485 Å and ∠C7–H7B···N5=146.15°, d(H9B···N2) = 2.487 Å and ∠C9–H9B···N2 = 142.85°), which are constructing the chain structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.36 × 0.37 × 0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.72 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9559, 4594
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3477
$N(param)_{refined}$:	298
Programs:	SHELXS-97, SHELXL-97 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(7A)	2*i*		0.5596	0.5086	0.6525	0.062
H(7B)	2*i*		0.5101	0.3675	0.5751	0.062
H(8A)	2*i*		0.7767	0.1922	0.1405	0.061
H(8B)	2*i*		0.6879	0.1247	0.1900	0.061
H(9A)	2*i*		0.9915	0.6547	0.5321	0.057
H(9B)	2*i*		0.9742	0.6021	0.3902	0.057
H(10A)	2*i*	0.50	0.5585	0.1329	0.2958	0.090
H(10B)	2*i*	0.50	0.4646	0.2146	0.3945	0.090
H(10C)	2*i*	0.50	0.6179	0.1769	0.4351	0.090
H(10D)	2*i*	0.50	0.5355	0.2167	0.4545	0.090
H(10E)	2*i*	0.50	0.6294	0.1350	0.3558	0.090
H(10F)	2*i*	0.50	0.4761	0.1727	0.3152	0.090
H(11A)	2*i*	0.50	0.7977	0.3398	0.1337	0.086
H(11B)	2*i*	0.50	0.9359	0.4362	0.2224	0.086
H(11C)	2*i*	0.50	0.7810	0.4800	0.1950	0.086
H(11D)	2*i*	0.50	0.8787	0.4975	0.2337	0.086
H(11E)	2*i*	0.50	0.7405	0.4011	0.1450	0.086
H(11F)	2*i*	0.50	0.8955	0.3574	0.1724	0.086
H(12A)	2*i*	0.50	0.7415	0.6237	0.7102	0.088

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(12B)	2*i*	0.50	0.7495	0.7048	0.6498	0.088
H(12C)	2*i*	0.50	0.8957	0.6557	0.6854	0.088
H(12D)	2*i*	0.50	0.8496	0.6991	0.6534	0.088
H(12E)	2*i*	0.50	0.8416	0.6180	0.7138	0.088
H(12F)	2*i*	0.50	0.6954	0.6671	0.6782	0.088
H(14)	2*i*		0.8198	0.4290	0.9504	0.075
H(16A)	2*i*	0.50	0.5086	0.5516	0.8176	0.120
H(16B)	2*i*	0.50	0.6131	0.6125	0.9481	0.120
H(16C)	2*i*	0.50	0.5029	0.4946	0.9016	0.120
H(16D)	2*i*	0.50	0.5745	0.5542	0.9606	0.120
H(16E)	2*i*	0.50	0.4700	0.4933	0.8301	0.120
H(16F)	2*i*	0.50	0.5802	0.6112	0.8766	0.120
H(17A)	2*i*	0.50	1.0199	0.2222	0.6937	0.122
H(17B)	2*i*	0.50	0.9658	0.2065	0.7965	0.122
H(17C)	2*i*	0.50	1.0816	0.3182	0.8328	0.122
H(17D)	2*i*	0.50	1.0250	0.2757	0.8550	0.122
H(17E)	2*i*	0.50	1.0790	0.2915	0.7522	0.122
H(17F)	2*i*	0.50	0.9632	0.1798	0.7159	0.122
H(19)	2*i*		0.3477	0.0982	–0.1632	0.072
H(21A)	2*i*	0.50	0.7668	0.1343	–0.0385	0.108
H(21B)	2*i*	0.50	0.6783	0.1655	–0.1339	0.108
H(21C)	2*i*	0.50	0.6674	0.0303	–0.1612	0.108

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(21D)	2*i*	0.50	0.6415	0.0858	–0.1839	0.108
H(21E)	2*i*	0.50	0.7300	0.0545	–0.0885	0.108
H(21F)	2*i*	0.50	0.7409	0.1897	–0.0612	0.108
H(22A)	2*i*	0.50	0.1365	0.1694	0.0946	0.126
H(22B)	2*i*	0.50	0.1038	0.0659	–0.0442	0.126
H(22C)	2*i*	0.50	0.1129	0.2035	–0.0090	0.126
H(22D)	2*i*	0.50	0.0990	0.1231	–0.0670	0.126
H(22E)	2*i*	0.50	0.1317	0.2267	0.0718	0.126
H(22F)	2*i*	0.50	0.1226	0.0890	0.0365	0.126
H(24)	2*i*		0.7886	1.0002	0.5498	0.081
H(26A)	2*i*	0.50	0.4877	0.7671	0.2879	0.106
H(26B)	2*i*	0.50	0.5668	0.8905	0.3124	0.106
H(26C)	2*i*	0.50	0.4758	0.8901	0.4071	0.106
H(26D)	2*i*	0.50	0.5325	0.9313	0.3837	0.106
H(26E)	2*i*	0.50	0.4534	0.8080	0.3592	0.106
H(26F)	2*i*	0.50	0.5444	0.8084	0.2645	0.106
H(27A)	2*i*	0.50	1.0798	0.8286	0.6351	0.163
H(27B)	2*i*	0.50	1.0131	0.9494	0.7181	0.163
H(27C)	2*i*	0.50	1.1022	0.9426	0.6195	0.163
H(27D)	2*i*	0.50	1.0502	0.9852	0.6800	0.163
H(27E)	2*i*	0.50	1.1170	0.8643	0.5970	0.163
H(27F)	2*i*	0.50	1.0278	0.8711	0.6957	0.163

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*i*	0.6700(2)	0.4211(2)	0.5092(2)	0.0343(8)	0.057(1)	0.0482(9)	0.0090(7)	0.0051(7)	0.0354(8)
C(2)	2*i*	0.6472(2)	0.3126(2)	0.3951(2)	0.0374(8)	0.0514(9)	0.0509(9)	0.0037(7)	0.0011(7)	0.0337(8)
C(3)	2*i*	0.7034(2)	0.3108(1)	0.2976(1)	0.0392(8)	0.0467(9)	0.0451(9)	0.0067(7)	0.0019(7)	0.0268(8)
C(4)	2*i*	0.7820(2)	0.4169(2)	0.3150(1)	0.0365(8)	0.0509(9)	0.0477(9)	0.0102(7)	0.0074(7)	0.0314(8)
C(5)	2*i*	0.8141(2)	0.5212(1)	0.4321(2)	0.0329(8)	0.0457(9)	0.0509(9)	0.0080(7)	0.0054(7)	0.0299(8)
C(6)	2*i*	0.7568(2)	0.5240(1)	0.5292(1)	0.0352(8)	0.0496(9)	0.0457(9)	0.0098(7)	0.0037(7)	0.0276(8)
C(7)	2*i*	0.5965(2)	0.4293(2)	0.6113(2)	0.0411(9)	0.074(1)	0.055(1)	0.0089(8)	0.0092(8)	0.043(1)
C(8)	2*i*	0.6885(2)	0.1927(2)	0.1751(2)	0.052(1)	0.049(1)	0.051(1)	0.0079(8)	0.0038(8)	0.0268(8)
C(9)	2*i*	0.9189(2)	0.6288(2)	0.4567(2)	0.0394(9)	0.0475(9)	0.060(1)	0.0058(7)	0.0074(7)	0.0310(8)
C(10)	2*i*	0.5645(2)	0.1988(2)	0.3786(2)	0.060(1)	0.062(1)	0.067(1)	–0.0056(9)	0.0024(9)	0.043(1)
C(11)	2*i*	0.8284(2)	0.4184(2)	0.2066(2)	0.060(1)	0.067(1)	0.056(1)	0.0106(9)	0.0173(9)	0.038(1)
C(12)	2*i*	0.7888(2)	0.6374(2)	0.6551(2)	0.061(1)	0.060(1)	0.051(1)	0.0098(9)	0.0086(9)	0.0262(9)
C(13)	2*i*	0.6863(2)	0.4591(2)	0.8228(2)	0.068(1)	0.054(1)	0.048(1)	0.0048(9)	0.0131(9)	0.0307(8)
C(14)	2*i*	0.7967(2)	0.4132(2)	0.8699(2)	0.079(1)	0.068(1)	0.049(1)	0.003(1)	0.004(1)	0.039(1)
C(15)	2*i*	0.8679(2)	0.3382(2)	0.7734(2)	0.053(1)	0.069(1)	0.061(1)	0.0037(9)	0.0038(9)	0.046(1)
C(16)	2*i*	0.5672(3)	0.5363(2)	0.8774(2)	0.114(2)	0.076(1)	0.067(1)	0.035(1)	0.039(1)	0.042(1)
C(17)	2*i*	0.9952(3)	0.2647(2)	0.7742(2)	0.071(1)	0.105(2)	0.099(2)	0.025(1)	0.014(1)	0.074(2)
C(18)	2*i*	0.5434(2)	0.1364(2)	–0.0362(2)	0.067(1)	0.048(1)	0.044(1)	0.0048(8)	0.0079(8)	0.0227(8)
C(19)	2*i*	0.3923(2)	0.1215(2)	–0.0828(2)	0.067(1)	0.061(1)	0.044(1)	0.0028(9)	–0.0019(9)	0.0235(9)
C(20)	2*i*	0.3188(2)	0.1483(2)	0.0142(2)	0.058(1)	0.057(1)	0.051(1)	0.0039(9)	0.0001(9)	0.0244(9)
C(21)	2*i*	0.6758(2)	0.1147(2)	–0.0980(2)	0.075(1)	0.083(2)	0.055(1)	0.008(1)	0.016(1)	0.032(1)
C(22)	2*i*	0.1532(2)	0.1466(2)	0.0138(2)	0.061(1)	0.105(2)	0.067(1)	0.004(1)	–0.001(1)	0.033(1)
C(23)	2*i*	0.6796(2)	0.8280(2)	0.4175(2)	0.0456(9)	0.058(1)	0.070(1)	0.0069(8)	0.0098(8)	0.044(1)
C(24)	2*i*	0.7871(2)	0.9158(2)	0.5135(2)	0.065(1)	0.047(1)	0.084(1)	0.0066(9)	–0.001(1)	0.032(1)
C(25)	2*i*	0.8912(2)	0.8536(2)	0.5445(2)	0.057(1)	0.048(1)	0.073(1)	0.0032(9)	–0.0070(9)	0.029(1)
C(26)	2*i*	0.5400(2)	0.8455(2)	0.3502(2)	0.060(1)	0.079(1)	0.093(2)	0.010(1)	0.002(1)	0.062(1)
C(27)	2*i*	1.0343(3)	0.8975(2)	0.6376(3)	0.096(2)	0.058(1)	0.125(2)	–0.006(1)	–0.049(2)	0.031(1)
N(1)	2*i*	0.6967(2)	0.4129(1)	0.7033(1)	0.0469(8)	0.0592(9)	0.0495(8)	0.0079(7)	0.0102(6)	0.0360(7)
N(2)	2*i*	0.8062(2)	0.3368(1)	0.6709(1)	0.0455(8)	0.072(1)	0.0601(9)	0.0131(7)	0.0129(7)	0.0432(8)
N(3)	2*i*	0.5547(2)	0.1728(1)	0.0846(1)	0.0522(8)	0.0510(8)	0.0453(8)	0.0054(6)	0.0047(6)	0.0237(7)
N(4)	2*i*	0.4174(2)	0.1792(1)	0.1167(1)	0.0528(9)	0.0613(9)	0.0490(8)	0.0044(7)	0.0041(7)	0.0255(7)
N(5)	2*i*	0.7138(2)	0.7164(1)	0.3880(1)	0.0411(8)	0.0544(8)	0.0608(9)	0.0010(6)	0.0015(6)	0.0371(7)
N(6)	2*i*	0.8426(2)	0.7339(1)	0.4683(1)	0.0403(7)	0.0465(8)	0.0582(8)	0.0035(6)	0.0022(6)	0.0311(7)

Acknowledgment. This project was supported by the Fund for Doctorates of Henan University of Science and Technology.

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