Crystal structure of *N,N'*-bis(2-methyl-4-phenyl-5-*tert*-butyl-imidazo[1,5-*b*]pyridazin-7-yl)ethylenediamine, $C_2H_4(NH)_2(C_{17}H_{18}N_3)_2$

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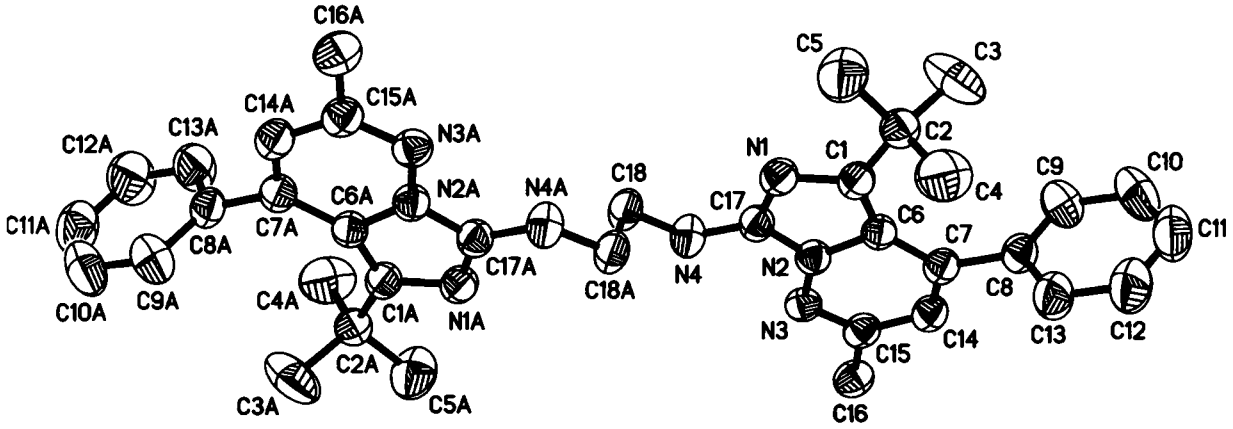

Abstract

$C_{36}H_{42}N_8$, monoclinic, $P12_1/c1$ (no. 14), a = 14.004(3) Å, b = 9.164(2) Å, c = 13.468(3) Å, β = 109.30(3)°, V = 1631.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.060, $wR_{ref}(F^2)$ = 0.158, T = 193 K.

Source of material

The title compound was synthesized in accordance to a published procedure [1]. Re-crystallization from ethanol/chloroform (*v/v* 2:1) gave orange crystals suitable for X-ray structure analysis.

Experimental details

The H4N atom was included in the refinement process due to the importance with regard to the hydrogen bond pattern.

Discussion

We introduced a series of new imidazo[1,5-*b*]pyridazine substituted diamines [1-3] (for instance the title compound) as novel amido ligands. The deprotonated diamines can act as bisamido ligands and bind early and late transition metals as a five-membered chelate [1]. The significant shorter N3—C15 (1.307 Å) and C7—C14 (1.361 Å) bonds indicate the localization of the double bonds of the six-membered pyridazine ring. The deviation from plane of the imidazopyridazine is 0.011 Å. The 7-substituted amido N4 atom is in the plane with the imidazopyridazine (only 0.001 Å deviation above). The phenyl substituent in the 4-position is twisted with regard to the imidazo[1,5-*b*]pyridazine with dihedral angle of 94.7°, respectively. The title compound is predicted to form inter- or intramolecular H bonds, because it contains a potential proton donor and acceptor functionalities. The amino H atoms form intramolecular hydrogen bond to the corresponding pyridazine N3 atoms (d(H4N···N3) = 2.442 Å).

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Table 1. Data collection and handling.

Crystal:	orange prism, size 0.3 × 0.4 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.73 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	51.66°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20975, 3103
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2204
$N(param)_{refined}$:	206
Programs:	SIR97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*e*	0.6491	−0.0598	0.3075	0.149
H(3B)	4*e*	0.5603	0.0311	0.3290	0.149
H(3C)	4*e*	0.6640	0.0023	0.4227	0.149
H(4A)	4*e*	0.6444	0.1148	0.1620	0.137
H(4B)	4*e*	0.6590	0.2857	0.1869	0.137
H(4C)	4*e*	0.5568	0.2063	0.1854	0.137
H(5A)	4*e*	0.5744	0.2941	0.3574	0.130
H(5B)	4*e*	0.6765	0.3778	0.3635	0.130
H(5C)	4*e*	0.6754	0.2672	0.4550	0.130
H(9)	4*e*	0.7558	−0.3006	0.2930	0.079
H(10)	4*e*	0.6160	−0.4183	0.1765	0.097
H(11)	4*e*	0.5433	−0.3320	0.0065	0.100
H(12)	4*e*	0.6126	−0.1312	−0.0491	0.112
H(13)	4*e*	0.7526	−0.0145	0.0659	0.088
H(14)	4*e*	0.9523	−0.2093	0.2288	0.059
H(16A)	4*e*	1.1975	−0.0695	0.3845	0.086
H(16B)	4*e*	1.1478	−0.1263	0.2662	0.086
H(16C)	4*e*	1.1466	−0.2278	0.3625	0.086
H(18A)	4*e*	1.0774	0.4685	0.5977	0.063
H(18B)	4*e*	0.9598	0.4355	0.5746	0.063
H(4N)	4*e*	1.085(2)	0.274(3)	0.498(2)	0.057(8)

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)	4e	0.7917(2)	0.1519(2)	0.3522(2)	0.054(1)	0.041(1)	0.042(1)	−0.001(1)	0.017(1)	0.001(1)
C(2)	4e	0.6771(2)	0.1615(3)	0.3199(2)	0.052(1)	0.049(1)	0.055(1)	−0.001(1)	0.021(1)	−0.003(1)
C(3)	4e	0.6338(2)	0.0214(4)	0.3472(4)	0.070(2)	0.073(2)	0.175(4)	−0.004(2)	0.067(2)	0.018(2)
C(4)	4e	0.6302(2)	0.1951(5)	0.2032(2)	0.055(2)	0.143(3)	0.069(2)	0.005(2)	0.011(1)	0.003(2)
C(5)	4e	0.6483(2)	0.2863(4)	0.3792(3)	0.061(2)	0.092(2)	0.108(3)	0.012(2)	0.027(2)	−0.028(2)
C(6)	4e	0.8565(2)	0.0546(2)	0.3266(2)	0.050(1)	0.041(1)	0.038(1)	−0.003(1)	0.0149(9)	0.002(1)
C(7)	4e	0.8585(2)	−0.0703(2)	0.2640(2)	0.056(1)	0.040(1)	0.040(1)	−0.001(1)	0.017(1)	0.003(1)
C(8)	4e	0.7668(2)	−0.1431(3)	0.1920(2)	0.054(1)	0.042(1)	0.054(1)	−0.001(1)	0.019(1)	−0.009(1)
C(9)	4e	0.7265(2)	−0.2646(3)	0.2233(2)	0.059(2)	0.055(2)	0.080(2)	−0.004(1)	0.016(1)	0.008(1)
C(10)	4e	0.6435(2)	−0.3348(3)	0.1540(3)	0.060(2)	0.054(2)	0.125(3)	−0.013(1)	0.027(2)	−0.007(2)
C(11)	4e	0.6009(2)	−0.2846(4)	0.0535(3)	0.064(2)	0.087(2)	0.091(2)	−0.012(2)	0.013(2)	−0.034(2)
C(12)	4e	0.6415(3)	−0.1658(4)	0.0209(3)	0.089(2)	0.114(3)	0.062(2)	−0.029(2)	0.006(2)	−0.012(2)
C(13)	4e	0.7243(2)	−0.0962(4)	0.0896(2)	0.080(2)	0.081(2)	0.052(2)	−0.024(2)	0.012(1)	−0.002(1)
C(14)	4e	0.9504(2)	−0.1253(3)	0.2694(2)	0.057(2)	0.045(1)	0.047(1)	0.001(1)	0.018(1)	−0.003(1)
C(15)	4e	1.0435(2)	−0.0630(3)	0.3329(2)	0.056(1)	0.046(1)	0.042(1)	0.003(1)	0.018(1)	0.004(1)
C(16)	4e	1.1423(2)	−0.1271(3)	0.3369(2)	0.057(2)	0.063(2)	0.054(1)	0.009(1)	0.020(1)	0.000(1)
C(17)	4e	0.9438(2)	0.2270(2)	0.4374(2)	0.051(1)	0.041(1)	0.042(1)	−0.002(1)	0.015(1)	0.000(1)
C(18)	4e	1.0136(2)	0.4411(3)	0.5421(2)	0.065(2)	0.046(1)	0.045(1)	−0.005(1)	0.014(1)	−0.009(1)
N(1)	4e	0.8477(2)	0.2573(2)	0.4198(2)	0.056(1)	0.045(1)	0.047(1)	0.0008(9)	0.0198(9)	−0.0012(9)
N(2)	4e	0.9544(1)	0.1072(2)	0.3838(1)	0.047(1)	0.042(1)	0.041(1)	−0.0007(8)	0.0151(8)	0.0006(8)
N(3)	4e	1.0476(1)	0.0533(2)	0.3902(1)	0.049(1)	0.046(1)	0.043(1)	0.0019(9)	0.0177(8)	0.0031(9)
N(4)	4e	1.0259(2)	0.2990(2)	0.5012(2)	0.054(1)	0.048(1)	0.056(1)	−0.001(1)	0.012(1)	−0.009(1)

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