Crystal structure of *N,N'*-bis(2-methyl-4-phenyl-5-*tert*-butyl-imidazo[1,5-*b*]pyridazin-7-yl)ethylenediamine, $C_{26}H_{42}N_8$

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Abstract

$C_{26}H_{42}N_8$, monoclinic, $P12_1/c1$ (no. 14), $a = 14.004(3)$ Å, $b = 9.164(2)$ Å, $c = 13.468(3)$ Å, $\beta = 109.30(3)^\circ$, $V = 1631.3$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.060$, $wR_{\text{ref}}(F^2) = 0.158$, $T = 193$ 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 ($d(\text{H4N} \cdots \text{N3}) = 2.442$ Å).

Table 1. Data collection and handling.

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

Table 2. Atomic coordinates and displacement parameters (in Å²).

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

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> 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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