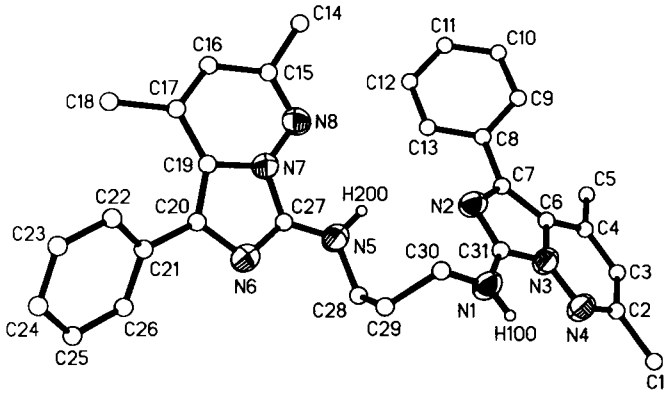
Crystal structure of *N,N'*-bis(2,4-dimethyl-5-phenylimidazo[1,5-*b*]pyridazin-7-yl)-1,3-diaminopropane, $C_{31}H_{32}N_8$

T. Irrgang and R. Kempe*

Universität Bayreuth, Lehrstuhl für Anorganische Chemie II, 95440 Bayreuth, Germany

Received April 26, 2005, accepted and available on-line July 27, 2005; CCDC no. 1267/1539



Abstract

$C_{31}H_{32}N_8$, monoclinic, $P12_1/a1$ (no. 14), $a = 9.581(1)$ Å, $b = 16.381(2)$ Å, $c = 17.803(2)$ Å, $\beta = 100.920(5)°$, $V = 2743.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.111$, $T = 293$ K.

Source of material

The title compound can be synthesized in accordance to a published procedure [1]. Re-crystallization from ethanol gave crystals suitable for X-ray crystal structure analysis.

Experimental details

The H100 and H200 atoms were included in the refinement process due to their importance with regard to the H-bond patterns.

Discussion

We introduced a series of new imidazo[1,5-*b*]pyridazine substituted diamines (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 five membered chelates [1]. The significant shorter N4—C2 (1.30 Å)/N8—C15 (1.31 Å) and C3—C4 (1.36 Å)/C16—C17 (1.33 Å) bonds indicates the localization of the double bonds of the six-membered pyridazine rings. The dihedral angles between the two imidazopyridazine planes are 28.8°. The deviations from the planes are 0.047 Å and 0.028 Å. Both amino N atoms are in the plane with the imidazopyridazines. The deviations of these atoms (N1, N5) out of the imidazopyridazine planes are 0.031 Å and 0.047 Å, respectively. The title compound is predicted to form inter- or intramolecular H bonds, because it contains potential proton donor and acceptor functionalities. The amino H atoms form intramolecular hydrogen bonds to the corresponding pyridazine N atoms (d(H200···N8) = 2.58 Å and d(H100···N4) = 2.50 Å). An intramolecular hydrogen bond N5–H200···N2 with a H200···N2 distance of 2.64 Å forces this molecule into a *transoid* arrangement.

Table 1. Data collection and handling.

Crystal:	red-orange prism, size 0.20 × 0.20 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.78 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, φ
$2\theta_{max}$:	48.72°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8071, 4147
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1966
$N(param)_{refined}$:	360
Programs:	SIR97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.3525	0.2125	−0.0857	0.118
H(1B)	4*e*	0.2290	0.2142	−0.1575	0.118
H(1C)	4*e*	0.3090	0.1323	−0.1324	0.118
H(3)	4*e*	0.0247	0.1017	−0.1418	0.067
H(5A)	4*e*	−0.2384	0.0429	−0.0374	0.099
H(5B)	4*e*	−0.1558	−0.0029	−0.0928	0.099
H(5C)	4*e*	−0.2402	0.0772	−0.1197	0.099
H(9)	4*e*	−0.3419	0.1310	0.0221	0.068
H(10)	4*e*	−0.5518	0.0852	0.0545	0.087
H(11)	4*e*	−0.5479	0.0134	0.1656	0.087
H(12)	4*e*	−0.3361	−0.0123	0.2459	0.079
H(13)	4*e*	−0.1265	0.0321	0.2143	0.063
H(14A)	4*e*	−0.3088	0.1182	0.3529	0.130
H(14B)	4*e*	−0.3476	0.1645	0.4232	0.130
H(14C)	4*e*	−0.3531	0.0689	0.4201	0.130
H(16)	4*e*	−0.1919	0.1076	0.5555	0.072
H(18A)	4*e*	0.1495	0.1016	0.6698	0.094
H(18B)	4*e*	0.0125	0.0481	0.6653	0.094
H(18C)	4*e*	0.0044	0.1429	0.6758	0.094
H(22)	4*e*	0.2752	0.2169	0.6483	0.073
H(23)	4*e*	0.4464	0.2349	0.7576	0.083
H(24)	4*e*	0.6567	0.1637	0.7718	0.084
H(25)	4*e*	0.6971	0.0749	0.6784	0.078
H(26)	4*e*	0.5251	0.0532	0.5718	0.065
H(28A)	4*e*	0.2978	0.0834	0.2484	0.057
H(28B)	4*e*	0.3915	0.0739	0.3301	0.057
H(29A)	4*e*	0.3746	0.2154	0.3484	0.058
H(29B)	4*e*	0.4541	0.1934	0.2819	0.058
H(30A)	4*e*	0.1695	0.2451	0.2590	0.057
H(30B)	4*e*	0.2965	0.3012	0.2476	0.057
H(200)	4*e*	0.106(3)	0.109(2)	0.303(2)	0.06(1)
H(100)	4*e*	0.307(4)	0.236(2)	0.140(2)	0.07(1)

* Correspondence author (e-mail: kempe@uni-bayreuth.de)

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}
N(1)	4*e*	0.2450(3)	0.2182(2)	0.1648(1)	0.040(2)	0.069(2)	0.041(2)	–0.015(2)	0.010(1)	0.003(1)
N(2)	4*e*	0.0196(3)	0.1531(2)	0.1583(1)	0.036(2)	0.054(2)	0.044(2)	–0.005(1)	0.010(1)	–0.000(1)
N(3)	4*e*	0.1063(3)	0.1666(1)	0.0521(1)	0.042(2)	0.050(2)	0.036(2)	–0.004(1)	0.012(1)	0.002(1)
N(4)	4*e*	0.2044(3)	0.1872(1)	0.0072(1)	0.048(2)	0.049(2)	0.049(2)	–0.000(1)	0.020(1)	0.006(1)
N(5)	4*e*	0.1865(3)	0.0933(2)	0.3322(1)	0.040(2)	0.075(2)	0.037(2)	–0.005(2)	0.009(1)	0.005(1)
N(6)	4*e*	0.3141(3)	0.1083(2)	0.4605(1)	0.042(2)	0.065(2)	0.043(2)	–0.001(1)	0.013(1)	0.005(1)
N(7)	4*e*	0.0789(3)	0.1099(2)	0.4422(1)	0.033(2)	0.064(2)	0.040(2)	–0.001(1)	0.008(1)	0.008(1)
N(8)	4*e*	–0.0575(3)	0.1105(2)	0.4026(1)	0.032(2)	0.070(2)	0.051(2)	–0.003(1)	0.006(1)	0.014(1)
C(1)	4*e*	0.2744(4)	0.1823(2)	–0.1145(2)	0.097(4)	0.087(3)	0.062(2)	–0.013(2)	0.040(2)	0.003(2)
C(2)	4*e*	0.1680(4)	0.1630(2)	–0.0638(2)	0.067(3)	0.048(2)	0.047(2)	0.005(2)	0.019(2)	0.006(2)
C(3)	4*e*	0.0424(4)	0.1186(2)	–0.0910(2)	0.062(3)	0.062(2)	0.044(2)	0.004(2)	0.010(2)	–0.001(2)
C(4)	4*e*	–0.0543(4)	0.0992(2)	–0.0469(2)	0.049(2)	0.043(2)	0.047(2)	0.005(2)	0.001(2)	–0.001(2)
C(5)	4*e*	–0.1837(4)	0.0497(2)	–0.0769(2)	0.064(3)	0.070(3)	0.058(2)	0.001(2)	–0.002(2)	–0.015(2)
C(6)	4*e*	–0.0239(3)	0.1259(2)	0.0305(2)	0.039(2)	0.041(2)	0.042(2)	–0.001(2)	0.005(1)	–0.000(2)
C(7)	4*e*	–0.0769(3)	0.1192(2)	0.0981(2)	0.034(2)	0.045(2)	0.043(2)	0.003(2)	0.004(1)	0.000(2)
C(8)	4*e*	–0.2109(3)	0.0865(2)	0.1144(2)	0.035(2)	0.047(2)	0.052(2)	–0.000(2)	0.008(2)	–0.010(2)
C(9)	4*e*	–0.3393(4)	0.1020(2)	0.0673(2)	0.041(3)	0.066(2)	0.061(2)	–0.001(2)	0.005(2)	–0.002(2)
C(10)	4*e*	–0.4655(4)	0.0745(2)	0.0868(2)	0.034(3)	0.095(3)	0.086(3)	–0.002(2)	0.005(2)	–0.016(2)
C(11)	4*e*	–0.4633(4)	0.0318(2)	0.1531(2)	0.047(3)	0.094(3)	0.082(3)	–0.016(2)	0.029(2)	–0.020(2)
C(12)	4*e*	–0.3377(4)	0.0163(2)	0.2007(2)	0.056(3)	0.076(3)	0.067(2)	–0.019(2)	0.021(2)	–0.006(2)
C(13)	4*e*	–0.2121(4)	0.0432(2)	0.1815(2)	0.045(3)	0.059(2)	0.055(2)	–0.007(2)	0.009(2)	–0.004(2)
C(14)	4*e*	–0.3039(4)	0.1164(3)	0.4072(2)	0.046(3)	0.140(4)	0.072(2)	0.000(2)	0.006(2)	0.022(2)
C(15)	4*e*	–0.1533(4)	0.1128(2)	0.4465(2)	0.035(2)	0.073(2)	0.056(2)	–0.001(2)	0.008(2)	0.016(2)
C(16)	4*e*	–0.1184(4)	0.1099(2)	0.5281(2)	0.048(3)	0.083(3)	0.054(2)	0.001(2)	0.020(2)	0.013(2)
C(17)	4*e*	0.0156(3)	0.1105(2)	0.5667(2)	0.040(2)	0.056(2)	0.049(2)	0.006(2)	0.012(2)	0.011(2)
C(18)	4*e*	0.0485(4)	0.0998(2)	0.6521(2)	0.057(3)	0.087(3)	0.048(2)	–0.002(2)	0.017(2)	0.007(2)
C(19)	4*e*	0.1257(3)	0.1161(2)	0.5220(2)	0.037(2)	0.052(2)	0.041(2)	0.003(2)	0.011(1)	0.009(2)
C(20)	4*e*	0.2708(4)	0.1172(2)	0.5307(2)	0.041(2)	0.059(2)	0.043(2)	0.003(2)	0.010(2)	0.007(2)
C(21)	4*e*	0.3796(3)	0.1304(2)	0.5998(2)	0.041(2)	0.056(2)	0.044(2)	–0.000(2)	0.013(2)	0.005(2)
C(22)	4*e*	0.3590(4)	0.1868(2)	0.6551(2)	0.058(3)	0.064(2)	0.062(2)	–0.007(2)	0.019(2)	–0.002(2)
C(23)	4*e*	0.4620(5)	0.1985(2)	0.7199(2)	0.079(3)	0.079(3)	0.052(2)	–0.023(2)	0.016(2)	–0.012(2)
C(24)	4*e*	0.5872(5)	0.1561(2)	0.7282(2)	0.065(3)	0.086(3)	0.052(2)	–0.024(2)	–0.007(2)	0.013(2)
C(25)	4*e*	0.6110(4)	0.1025(2)	0.6727(2)	0.052(3)	0.076(3)	0.064(2)	–0.005(2)	0.002(2)	0.012(2)
C(26)	4*e*	0.5083(4)	0.0897(2)	0.6091(2)	0.046(3)	0.069(2)	0.046(2)	–0.002(2)	0.009(2)	0.005(2)
C(27)	4*e*	0.1963(3)	0.1042(2)	0.4095(2)	0.033(2)	0.052(2)	0.045(2)	–0.002(2)	0.008(2)	0.006(2)
C(28)	4*e*	0.3148(3)	0.1052(2)	0.3000(2)	0.040(2)	0.062(2)	0.043(2)	0.005(2)	0.014(1)	0.005(2)
C(29)	4*e*	0.3623(3)	0.1925(2)	0.2973(2)	0.037(2)	0.064(2)	0.044(2)	–0.007(2)	0.010(1)	0.000(2)
C(30)	4*e*	0.2615(3)	0.2455(2)	0.2438(2)	0.044(2)	0.052(2)	0.046(2)	–0.008(2)	0.009(1)	–0.004(2)
C(31)	4*e*	0.1271(3)	0.1812(2)	0.1284(2)	0.037(2)	0.046(2)	0.043(2)	0.000(2)	0.007(2)	0.002(2)

Acknowledgments. We thank the Fonds der Chemischen Industrie for financial support and Dr. Anke Spannenberg for assistance in the X-ray laboratory.

References

1. Irrgang, T.; Kempe, R.: Transition Metal Complexes stabilised by Imidazopyridazine-substituted Bisamido Ligands. Eur. J. Inorg. Chem. 2005, accepted.
2. Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R.: SIR97: a new tool for crystal structure determination and refinement. J. Appl. Crystallogr. **32** (1999) 115-119.
3. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.