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Crystal structure of *N,N'*-bis(2-methyl-4,5-diphenyl-imidazo[1,5-*b*]pyridazin-7-yl)-1,3-diaminopropane ethanol solvate, $C_3H_6(NH)_2(C_{19}H_{14}N_3)_2 \cdot C_2H_5OH$

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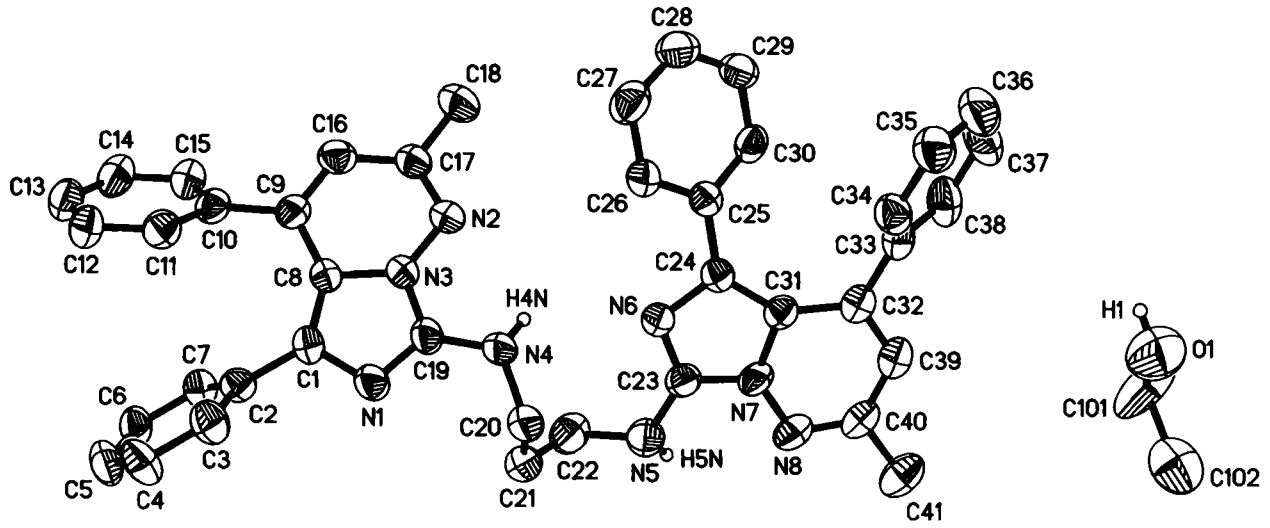

Abstract

$C_{43}H_{42}N_8O$, triclinic, $P\bar{1}$ (no. 2), a = 9.997(2) Å, b = 12.046(2) Å, c = 15.635(3) Å, α = 90.84(3)°, β = 105.18(3)°, γ = 93.43(3)°, V = 1813.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.071, $wR_{ref}(F^2)$ = 0.176, T = 193 K.

Source of material

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

Experimental details

The H4N and H5N atoms were included in the refinement process due to their importance with regard to the hydrogen bond patterns.

Discussion

Deprotonated imidazo[1,5-*b*]pyridazine substituted diamines [2], e.g. the title compound, can act as bisamido ligands and bind early and late transition metals as five-membered chelates [1]. The dihedral angle between the two imidazopyridazine planes is 40.6°. The deviations from the planes are 0.024 Å and 0.028 Å. The significant shorter bonds of N2—C17 (1.290 Å), N8—C40 (1.292 Å), C9—C16 (1.353 Å) and C32—C39 (1.366 Å) indicate the localization of the double bonds of the six-membered pyridazine rings. The phenyl substituents Ph1 (C10 – C15) and Ph2 (C33 – C38) in the 4-positions as well as Ph3 (C2 – C7) and Ph4 (C25 – C30) in the 5-positions of the imidazopyridazines are twisted with regard to the imidazopyridazine planes and the dihedral angles are 44.6° (Ph1), 55.5° (Ph2), 55.7° (Ph3) and 30.5° (Ph4), respectively. Both phenyl substituents of one imidazopyridazines form dihedral angles of 30.1° (Ph1/Ph3) and 42.4° (Ph2/Ph4). 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(H4N···N2) = 2.391 Å and d(H5N···N8) = 2.443 Å). An intramolecular N4–H4N···N6 bond with an H4N···N6 distance of 2.547 Å forces this molecule into a *transoid* arrangement. One ethanol molecule per ligand molecule is coordinated over an intermolecular hydrogen bond O1-H1···N1 (d(H1···N1) = 2.102 Å).

Table 1. Data collection and handling.

Crystal:	red prism, size 0.3 × 0.5 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.78 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	51.68°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	24191, 6857
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2637
$N(param)_{refined}$:	477
Programs:	SIR97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.6680	−0.6111	1.4688	0.066
H(4)	2*i*	0.7764	−0.7710	1.5241	0.077
H(5)	2*i*	0.6630	−0.9455	1.4827	0.073
H(6)	2*i*	0.4390	−0.9609	1.3921	0.065
H(7)	2*i*	0.3264	−0.8015	1.3391	0.058
H(11)	2*i*	0.3712	−0.6453	1.5408	0.064
H(12)	2*i*	0.3702	−0.8037	1.6247	0.071
H(13)	2*i*	0.1705	−0.9240	1.5954	0.077
H(14)	2*i*	−0.0252	−0.8841	1.4878	0.077
H(15)	2*i*	−0.0224	−0.7288	1.4016	0.064
H(16)	2*i*	−0.0043	−0.5213	1.4080	0.061
H(18A)	2*i*	−0.0320	−0.2841	1.2664	0.098
H(18B)	2*i*	−0.0666	−0.3122	1.3583	0.098
H(18C)	2*i*	−0.1320	−0.3909	1.2724	0.098
H(20A)	2*i*	0.6247	−0.3655	1.2478	0.071
H(20B)	2*i*	0.5576	−0.2679	1.1860	0.071
H(21A)	2*i*	0.4930	−0.4984	1.1435	0.082
H(21B)	2*i*	0.5938	−0.4209	1.1021	0.082
H(22A)	2*i*	0.3689	−0.4516	1.0027	0.080
H(22B)	2*i*	0.3033	−0.3921	1.0730	0.080
H(26)	2*i*	0.2750	−0.1263	1.2590	0.060
H(27)	2*i*	0.1536	−0.0712	1.3592	0.074
H(28)	2*i*	−0.0163	0.0540	1.3188	0.072
H(29)	2*i*	−0.0727	0.1220	1.1763	0.064
H(30)	2*i*	0.0460	0.0668	1.0758	0.056
H(34)	2*i*	0.3619	0.1873	1.1329	0.061
H(35)	2*i*	0.2936	0.3472	1.1889	0.077
H(36)	2*i*	0.1082	0.4391	1.1037	0.098
H(37)	2*i*	−0.0055	0.3717	0.9621	0.104
H(38)	2*i*	0.0674	0.2145	0.9016	0.076
H(39)	2*i*	0.2959	0.1531	0.8664	0.062
H(41A)	2*i*	0.4344	−0.0708	0.7640	0.109
H(41B)	2*i*	0.4696	0.0604	0.7805	0.109
H(41C)	2*i*	0.3157	0.0138	0.7294	0.109
H(10A)	2*i*	0.6281	0.3535	0.1865	0.138
H(10B)	2*i*	0.7131	0.2735	0.2602	0.138
H(10C)	2*i*	0.6688	0.4327	0.3247	0.075
H(10D)	2*i*	0.7474	0.5043	0.2650	0.075
H(10E)	2*i*	0.8322	0.4250	0.3381	0.075
H(1)	2*i*	0.8243	0.3227	0.1399	0.278
H(4N)	2*i*	0.348(5)	−0.318(4)	1.225(3)	0.07(2)
H(5N)	2*i*	0.412(6)	−0.288(4)	0.959(4)	0.09(2)

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*	0.4187(5)	−0.5873(3)	1.3658(3)	0.049(3)	0.035(2)	0.045(3)	0.003(2)	0.011(2)	0.007(2)
C(2)	2*i*	0.4864(5)	−0.6901(4)	1.3996(3)	0.039(3)	0.046(3)	0.047(3)	0.003(2)	0.009(2)	0.001(2)
C(3)	2*i*	0.6202(5)	−0.6821(4)	1.4536(3)	0.057(3)	0.042(3)	0.062(3)	0.006(2)	0.007(3)	0.002(2)
C(4)	2*i*	0.6851(5)	−0.7771(4)	1.4856(3)	0.052(3)	0.061(4)	0.070(3)	0.019(3)	−0.002(3)	0.010(3)
C(5)	2*i*	0.6175(6)	−0.8803(4)	1.4617(3)	0.076(4)	0.048(3)	0.058(3)	0.019(3)	0.012(3)	0.013(2)
C(6)	2*i*	0.4853(5)	−0.8894(4)	1.4079(3)	0.068(4)	0.040(3)	0.055(3)	0.006(3)	0.019(3)	0.011(2)
C(7)	2*i*	0.4185(5)	−0.7948(4)	1.3764(3)	0.049(3)	0.043(3)	0.049(3)	0.003(2)	0.008(2)	0.008(2)
C(8)	2*i*	0.2935(5)	−0.5498(3)	1.3683(3)	0.043(3)	0.039(3)	0.042(3)	0.007(2)	0.011(2)	0.006(2)
C(9)	2*i*	0.1786(5)	−0.5749(3)	1.4036(3)	0.044(3)	0.041(3)	0.041(3)	0.001(2)	0.012(2)	0.001(2)
C(10)	2*i*	0.1770(5)	−0.6731(3)	1.4613(3)	0.056(3)	0.041(3)	0.041(3)	0.000(2)	0.018(2)	−0.001(2)
C(11)	2*i*	0.2908(5)	−0.6952(4)	1.5286(3)	0.062(4)	0.056(3)	0.044(3)	0.008(3)	0.015(3)	0.001(2)
C(12)	2*i*	0.2904(6)	−0.7885(4)	1.5791(3)	0.071(4)	0.063(3)	0.044(3)	0.013(3)	0.014(3)	0.016(3)
C(13)	2*i*	0.1718(6)	−0.8589(4)	1.5620(3)	0.087(4)	0.056(3)	0.058(3)	0.006(3)	0.034(3)	0.014(3)
C(14)	2*i*	0.0567(6)	−0.8361(4)	1.4976(3)	0.085(4)	0.058(3)	0.053(3)	−0.005(3)	0.026(3)	0.005(3)
C(15)	2*i*	0.0581(5)	−0.7439(4)	1.4467(3)	0.059(3)	0.050(3)	0.051(3)	−0.001(3)	0.015(2)	0.004(2)
C(16)	2*i*	0.0728(5)	−0.5067(4)	1.3841(3)	0.046(3)	0.051(3)	0.058(3)	0.002(2)	0.020(2)	0.001(2)
C(17)	2*i*	0.0730(5)	−0.4133(4)	1.3286(3)	0.045(3)	0.048(3)	0.053(3)	0.006(2)	0.015(2)	0.003(2)
C(18)	2*i*	−0.0500(5)	−0.3442(4)	1.3043(3)	0.054(3)	0.064(3)	0.083(4)	0.024(3)	0.020(3)	0.011(3)
C(19)	2*i*	0.4020(5)	−0.4353(4)	1.2934(3)	0.043(3)	0.037(3)	0.053(3)	0.004(2)	0.014(2)	0.008(2)
C(20)	2*i*	0.5394(5)	−0.3444(4)	1.2045(3)	0.060(3)	0.043(3)	0.083(4)	0.011(2)	0.034(3)	0.015(3)
C(21)	2*i*	0.5103(6)	−0.4218(4)	1.1250(4)	0.082(4)	0.052(3)	0.089(4)	0.015(3)	0.051(4)	0.016(3)
C(22)	2*i*	0.3868(6)	−0.3932(4)	1.0503(3)	0.092(4)	0.045(3)	0.072(4)	−0.007(3)	0.043(3)	−0.009(3)
C(23)	2*i*	0.3509(5)	−0.1933(4)	1.0325(3)	0.058(3)	0.044(3)	0.056(3)	0.001(2)	0.026(3)	−0.001(2)
C(24)	2*i*	0.2496(4)	−0.0716(3)	1.0928(3)	0.044(3)	0.036(2)	0.040(2)	0.004(2)	0.012(2)	0.001(2)
C(25)	2*i*	0.1753(4)	−0.0341(3)	1.1561(3)	0.035(3)	0.034(2)	0.043(3)	−0.002(2)	0.006(2)	−0.002(2)
C(26)	2*i*	0.2047(5)	−0.0752(4)	1.2416(3)	0.063(3)	0.046(3)	0.047(3)	0.014(2)	0.020(3)	0.011(2)
C(27)	2*i*	0.1326(5)	−0.0425(4)	1.3013(3)	0.074(4)	0.069(4)	0.043(3)	0.004(3)	0.020(3)	0.009(3)
C(28)	2*i*	0.0315(5)	0.0309(4)	1.2772(3)	0.063(4)	0.055(3)	0.071(4)	0.002(3)	0.034(3)	−0.007(3)
C(29)	2*i*	−0.0015(5)	0.0717(4)	1.1933(3)	0.050(3)	0.045(3)	0.069(3)	0.004(2)	0.025(3)	0.000(3)
C(30)	2*i*	0.0698(4)	0.0387(3)	1.1340(3)	0.044(3)	0.044(3)	0.053(3)	−0.003(2)	0.015(2)	0.010(2)
C(31)	2*i*	0.2791(4)	−0.0200(3)	1.0204(3)	0.042(3)	0.043(3)	0.037(2)	0.002(2)	0.012(2)	0.002(2)
C(32)	2*i*	0.2677(4)	0.0824(4)	0.9764(3)	0.040(3)	0.052(3)	0.039(3)	−0.004(2)	0.004(2)	0.005(2)
C(33)	2*i*	0.2225(5)	0.1835(4)	1.0126(3)	0.048(3)	0.040(3)	0.050(3)	−0.005(2)	0.010(2)	0.006(2)
C(34)	2*i*	0.2874(5)	0.2250(4)	1.0972(3)	0.046(3)	0.041(3)	0.066(3)	−0.001(2)	0.013(3)	0.002(2)
C(35)	2*i*	0.2466(6)	0.3196(4)	1.1310(4)	0.066(4)	0.050(3)	0.076(4)	−0.002(3)	0.021(3)	0.000(3)
C(36)	2*i*	0.1373(7)	0.3741(5)	1.0805(5)	0.091(5)	0.046(3)	0.112(5)	0.005(3)	0.034(4)	0.004(4)
C(37)	2*i*	0.0706(6)	0.3343(5)	0.9964(5)	0.069(4)	0.055(4)	0.133(6)	0.021(3)	0.016(4)	0.029(4)
C(38)	2*i*	0.1125(5)	0.2401(4)	0.9604(3)	0.053(3)	0.053(3)	0.073(4)	0.002(3)	−0.003(3)	0.023(3)
C(39)	2*i*	0.3057(5)	0.0865(4)	0.8987(3)	0.052(3)	0.054(3)	0.047(3)	−0.006(2)	0.009(2)	0.012(2)
C(40)	2*i*	0.3601(5)	−0.0068(4)	0.8644(3)	0.046(3)	0.067(3)	0.040(3)	−0.008(2)	0.014(2)	0.003(3)
C(41)	2*i*	0.3983(5)	−0.0003(5)	0.7769(3)	0.073(4)	0.098(4)	0.049(3)	−0.007(3)	0.023(3)	0.010(3)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(101)	2*i*	0.7169(8)	0.3461(4)	0.2318(4)	0.213(8)	0.054(4)	0.119(5)	–0.049(4)	0.135(6)	–0.043(4)
C(102)	2*i*	0.7433(4)	0.4335(4)	0.2946(3)	0.032(3)	0.056(3)	0.062(3)	–0.001(2)	0.015(2)	0.008(2)
N(1)	2*i*	0.4860(4)	–0.5157(3)	1.3202(2)	0.044(2)	0.039(2)	0.067(3)	0.004(2)	0.020(2)	0.008(2)
N(2)	2*i*	0.1761(4)	–0.3836(3)	1.2969(2)	0.047(2)	0.044(2)	0.055(2)	0.012(2)	0.019(2)	0.006(2)
N(3)	2*i*	0.2850(4)	–0.4513(3)	1.3193(2)	0.043(2)	0.043(2)	0.047(2)	0.011(2)	0.016(2)	0.008(2)
N(4)	2*i*	0.4277(4)	–0.3440(3)	1.2478(3)	0.040(3)	0.044(2)	0.073(3)	0.012(2)	0.025(2)	0.016(2)
N(5)	2*i*	0.4107(5)	–0.2844(3)	1.0130(3)	0.095(4)	0.049(3)	0.061(3)	0.012(2)	0.044(3)	0.001(2)
N(6)	2*i*	0.2975(4)	–0.1773(3)	1.1001(2)	0.056(3)	0.040(2)	0.046(2)	0.005(2)	0.022(2)	0.003(2)
N(7)	2*i*	0.3417(4)	–0.1022(3)	0.9818(2)	0.053(2)	0.049(2)	0.036(2)	–0.001(2)	0.017(2)	0.002(2)
N(8)	2*i*	0.3820(4)	–0.0997(3)	0.9044(2)	0.056(3)	0.064(3)	0.043(2)	–0.005(2)	0.022(2)	0.000(2)
O(1)	2*i*	0.8449(7)	0.3576(5)	0.1890(4)	0.220(7)	0.172(6)	0.161(5)	0.014(5)	0.046(5)	–0.035(4)

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References

1. Irrgang, T.; Kempe, R.: Early and Late Transition Metal Complexes Stabilised by Imidazopyridazine-Substituted Bisamido Ligands. Eur. J. Inorg. Chem. (2005) 4382-4392.
2. Irrgang, T.; Kempe, R.: Crystal structure of *N,N'*-bis(2,4-dimethyl-5-phenylimidazo[1,5-*b*]pyridazin-7-yl)-1,3-diaminopropane, $C_{31}H_{32}N_8$. Z. Kristallogr. NCS **220** (2005) 403-404.
3. 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.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.