Crystal structure of (*N*,*N*′-butylenebis(imidazole))(5-amino-isophthalato)zinc(II), $Zn(C_8H_5NO_4)(C_{10}H_{14}N_4)$

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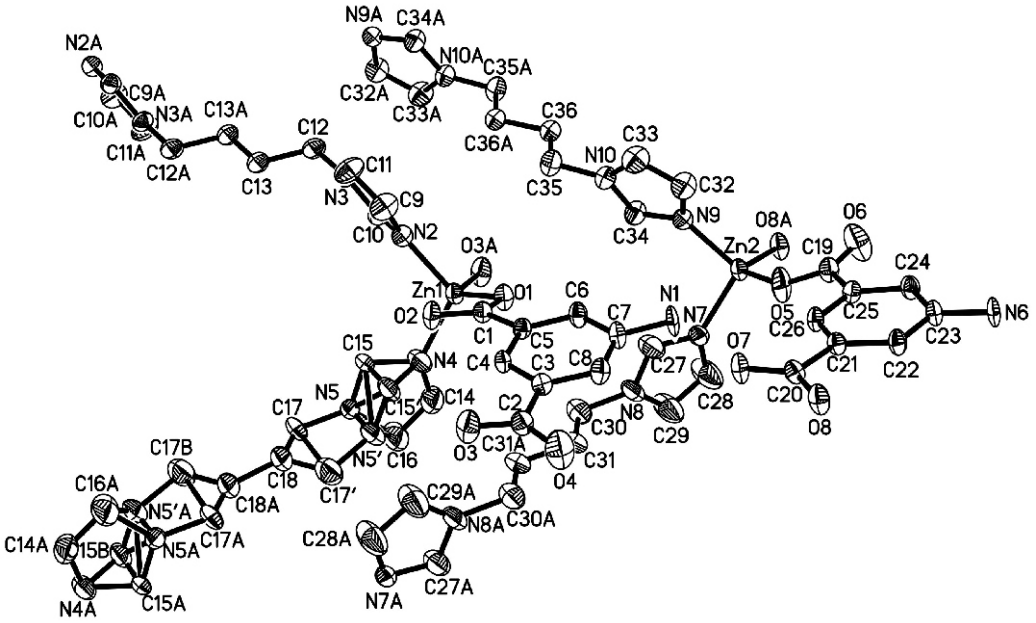

Abstract

$C_{36}H_{38}N_{10}O_8Zn_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.4782(2) Å, b = 13.8067(4) Å, c = 14.8094(5) Å, α = 90.635(2)°, β = 93.462(2)°, γ = 93.506(2)°, V = 1930.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.066, $wR_{ref}(F^2)$ = 0.137, T = 293 K.

Source of material

Starting materials and solvents for the synthesis were purchased commercially. *N*,*N*′-butylenebis(imidazole) (bbi) was synthesized according to [1]. A mixture of 5-aminoisophthalic acid (0.018 g, 0.1 mmol), $Zn(Ac)_2 \cdot 2H_2O$ (0.02 g, 0.1 mmol), bbi (0.02 g, 0.1 mmol) and 10 ml water was sealed in a 15 ml Teflon-lined autoclave. Then the mixture was heated at 160 °C for 3 days. After the mixture was cooled to room temperature at 10 °C/h, colorless crystals of the title compound were obtained (yield 36 %).

Experimental details

All H atoms on C atoms and N atoms were generated at idealized positions and refined as riding with d(C—H) = 0.93 - 0.97 Å, d(N—H) = 0.86 Å and U_{iso}(H) = 1.2 U_{eq}(C,N). C15, C17 and N5 were refined as disordered. The disordered atoms were split over two sites, with a total occupancy of 1. The hydrogen atoms of the disordered atoms were not included in the model. We have checked the structure with PLATON software [2]. There are no additional symmetry elements detected in the structure.

Discussion

There are two similar units in the asymmetric part of the title crystal structure. Each unit contains one Zn(II) ion, one 5-aminoisophthalic acid anion and two halves of *N*,*N*′-butylenebis(imidazole). Each Zn(II) ion is four-coordinated in a tetrahedral arrangement by two O atoms from two different 5-aminoisophthalic acid anions (d(Zn1—O1) = 1.961(3) Å, d(Zn1—O3A) = 1.955(4) Å, d(Zn2—O5) = 1.912(3) Å and d(Zn2—O8A) = 1.931(3) Å) and two N atoms from two different *N*,*N*'-butylenebis(imidazole) molecules (d(Zn1—N2) = 1.996(4) Å, d(Zn1—N4) = 1.990(5) Å, d(Zn2—N7) = 2.010(4) Å and d(Zn2—N9) = 2.015(4) Å). The Zn(II) ions are linked by the 5-aminoisophthalic acid anions to form chains, which are further interconnected by *N*,*N*′-butylenebis(imidazole) to yield layers. The two units form separate layers, which coexist in the structure.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.22 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.07 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14795, 8774
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5739
$N(param)_{refined}$:	532
Programs:	PLATON [2], SHELXS-97, SHELXL-97 [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(6A)	2*i*	1.6849	0.7870	–0.7596	0.051
H(6B)	2*i*	1.8351	0.7715	–0.7311	0.051
H(1A)	2*i*	1.7048	0.7643	–0.2648	0.062
H(1B)	2*i*	1.5587	0.7880	–0.2446	0.062
H(4)	2*i*	1.7665	0.7485	0.1092	0.043
H(8)	2*i*	1.8764	0.7285	–0.1469	0.053
H(26)	2*i*	1.6292	0.7537	–0.3911	0.048
H(24)	2*i*	1.5083	0.7971	–0.6505	0.052
H(6)	2*i*	1.4823	0.7919	–0.0962	0.054
H(9)	2*i*	1.0253	0.8301	0.2652	0.059
H(22)	2*i*	1.9137	0.7510	–0.5803	0.050
H(13A)	2*i*	1.1445	1.0223	0.4830	0.065
H(13B)	2*i*	1.0751	0.9160	0.4836	0.065
H(12A)	2*i*	0.9199	0.9546	0.3600	0.070
H(12B)	2*i*	0.9967	1.0584	0.3580	0.070
H(11)	2*i*	1.2829	1.0537	0.3190	0.088
H(29)	2*i*	1.1787	0.4091	–0.2050	0.159
H(10)	2*i*	1.3902	0.9459	0.2158	0.081
H(30A)	2*i*	1.2944	0.5722	–0.0198	0.099
H(30B)	2*i*	1.2671	0.4600	–0.0365	0.099
H(28)	2*i*	1.1110	0.5038	–0.3356	0.153
H(31A)	2*i*	1.5188	0.5631	–0.0735	0.084
H(31B)	2*i*	1.4911	0.4505	–0.0844	0.084
H(27)	2*i*	1.3112	0.6768	–0.1623	0.097
H(14)	2*i*	1.1588	0.5047	0.1498	0.112
H(34)	2*i*	1.1093	0.8067	–0.1611	0.066
H(35A)	2*i*	1.2351	1.0164	–0.0229	0.077
H(35B)	2*i*	1.1859	0.9079	–0.0064	0.077
H(32)	2*i*	1.3685	0.9489	–0.3137	0.076
H(33)	2*i*	1.3528	1.0452	–0.1763	0.083
H(36A)	2*i*	0.9616	0.9366	–0.0746	0.067
H(36B)	2*i*	1.0118	1.0462	–0.0845	0.067

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(2)	2*i*		1.20005(5)	0.73803(4)	–0.35113(4)	0.0291(3)	0.0623(4)	0.0319(3)	0.0092(3)	0.0019(2)	0.0046(3)
Zn(1)	2*i*		1.22454(5)	0.73956(4)	0.13984(4)	0.0321(3)	0.0591(4)	0.0355(3)	0.0096(3)	0.0012(2)	0.0028(3)
O(2)	2*i*		1.5253(3)	0.7835(3)	0.1653(2)	0.042(2)	0.094(3)	0.032(2)	0.010(2)	0.002(2)	–0.008(2)
N(6)	2*i*		1.7492(3)	0.7780(3)	–0.7175(2)	0.022(2)	0.092(3)	0.015(2)	0.008(2)	0.003(1)	0.008(2)
O(7)	2*i*		1.8825(3)	0.7508(3)	–0.3204(2)	0.043(2)	0.091(3)	0.027(2)	0.001(2)	0.003(2)	0.006(2)
N(1)	2*i*		1.6424(4)	0.7733(3)	–0.2262(2)	0.035(2)	0.105(4)	0.014(2)	0.010(2)	–0.000(2)	0.007(2)
O(1)	2*i*		1.3660(3)	0.7635(3)	0.0500(2)	0.030(2)	0.085(3)	0.039(2)	0.009(2)	0.003(2)	–0.002(2)
O(8)	2*i*		2.0291(3)	0.7325(3)	–0.4294(2)	0.029(2)	0.097(3)	0.037(2)	0.009(2)	–0.001(1)	0.008(2)
O(5)	2*i*		1.3712(3)	0.7496(4)	–0.4132(3)	0.034(2)	0.155(4)	0.041(2)	0.014(2)	0.011(2)	0.011(3)
C(21)	2*i*		1.7905(4)	0.7543(3)	–0.4740(3)	0.029(2)	0.047(3)	0.032(3)	–0.001(2)	–0.002(2)	0.002(2)
N(3)	2*i*		1.1134(4)	0.9540(3)	0.3117(3)	0.054(3)	0.048(3)	0.051(3)	0.008(2)	0.015(2)	–0.004(2)
O(3)	2*i*		2.0243(4)	0.7254(3)	0.0988(3)	0.039(2)	0.128(4)	0.053(3)	0.024(2)	–0.010(2)	–0.017(2)
C(23)	2*i*		1.7153(5)	0.7740(4)	–0.6306(3)	0.036(2)	0.062(3)	0.025(2)	–0.001(2)	0.001(2)	0.005(2)
C(3)	2*i*		1.8441(4)	0.7352(3)	–0.0141(3)	0.030(2)	0.041(3)	0.038(3)	–0.004(2)	0.003(2)	0.002(2)
C(4)	2*i*		1.7430(4)	0.7507(3)	0.0474(3)	0.036(2)	0.046(3)	0.026(2)	–0.001(2)	–0.002(2)	0.000(2)
C(8)	2*i*		1.8091(5)	0.7408(4)	–0.1058(3)	0.033(2)	0.063(4)	0.036(3)	–0.005(2)	0.005(2)	–0.005(2)
C(26)	2*i*		1.6514(4)	0.7599(4)	–0.4512(3)	0.034(2)	0.060(3)	0.026(2)	0.000(2)	0.006(2)	0.001(2)
C(5)	2*i*		1.6065(5)	0.7693(3)	0.0162(3)	0.033(2)	0.045(3)	0.033(3)	0.002(2)	0.001(2)	–0.002(2)
N(2)	2*i*		1.2115(4)	0.8555(3)	0.2199(3)	0.039(2)	0.058(3)	0.048(3)	0.010(2)	0.012(2)	0.003(2)
C(25)	2*i*		1.5458(5)	0.7748(4)	–0.5168(3)	0.033(2)	0.052(3)	0.032(3)	0.003(2)	0.002(2)	–0.003(2)
O(6)	2*i*		1.3027(4)	0.7995(4)	–0.5464(3)	0.045(2)	0.190(6)	0.080(3)	0.039(3)	0.014(2)	0.053(3)
O(4)	2*i*		2.0761(4)	0.6913(4)	–0.0379(3)	0.053(3)	0.186(6)	0.070(3)	0.042(3)	0.013(2)	0.004(3)
N(7)	2*i*		1.2141(4)	0.6199(3)	–0.2738(3)	0.048(2)	0.050(3)	0.046(3)	0.007(2)	–0.006(2)	0.001(2)
C(24)	2*i*		1.5786(5)	0.7841(4)	–0.6065(3)	0.035(2)	0.061(3)	0.033(3)	0.006(2)	–0.002(2)	0.003(2)
C(6)	2*i*		1.5732(5)	0.7775(4)	–0.0757(3)	0.032(2)	0.067(4)	0.033(3)	–0.002(2)	–0.005(2)	–0.002(2)
N(8)	2*i*		1.2665(6)	0.5358(4)	–0.1530(4)	0.111(4)	0.052(3)	0.059(3)	0.015(3)	–0.017(3)	0.016(3)
C(9)	2*i*		1.1048(5)	0.8731(4)	0.2653(3)	0.052(3)	0.048(3)	0.048(3)	0.006(3)	0.008(2)	0.001(3)
C(22)	2*i*		1.8217(5)	0.7585(4)	–0.5641(3)	0.033(2)	0.063(3)	0.030(3)	0.000(2)	0.006(2)	0.004(2)
C(1)	2*i*		1.4927(5)	0.7736(4)	0.0827(3)	0.033(2)	0.053(3)	0.037(3)	0.007(2)	0.002(2)	–0.001(2)
C(7)	2*i*		1.6758(5)	0.7643(4)	–0.1369(3)	0.046(3)	0.068(4)	0.027(3)	–0.004(3)	0.001(2)	0.003(2)
C(13)	2*i*		1.0572(6)	0.9829(4)	0.4701(4)	0.057(3)	0.054(4)	0.053(3)	0.014(3)	0.010(3)	–0.005(3)
C(12)	2*i*		1.0102(6)	0.9909(4)	0.3716(4)	0.058(3)	0.058(4)	0.062(4)	0.021(3)	0.013(3)	–0.004(3)
C(19)	2*i*		1.3942(5)	0.7771(4)	–0.4912(4)	0.036(3)	0.075(4)	0.039(3)	0.012(3)	0.003(2)	0.002(3)
C(20)	2*i*		1.9074(5)	0.7447(4)	–0.4014(3)	0.031(2)	0.049(3)	0.036(3)	–0.002(2)	0.002(2)	0.008(2)
C(11)	2*i*		1.2426(7)	0.9950(5)	0.2954(5)	0.069(4)	0.055(4)	0.097(5)	–0.013(3)	0.026(4)	–0.019(4)
C(2)	2*i*		1.9935(5)	0.7156(4)	0.0173(4)	0.040(3)	0.060(4)	0.044(3)	0.006(2)	0.006(2)	0.004(3)
C(29)	2*i*		1.199(1)	0.4753(6)	–0.2121(6)	0.23(1)	0.057(5)	0.100(7)	–0.022(6)	–0.073(7)	0.012(5)
C(10)	2*i*		1.3015(6)	0.9350(5)	0.2387(4)	0.049(3)	0.072(4)	0.083(5)	–0.006(3)	0.028(3)	–0.005(4)
C(30)	2*i*		1.3168(7)	0.5181(5)	–0.0577(4)	0.099(5)	0.086(5)	0.066(5)	0.026(4)	0.007(4)	0.018(4)
C(28)	2*i*		1.164(1)	0.5282(6)	–0.2844(6)	0.19(1)	0.075(6)	0.106(7)	–0.025(6)	–0.080(7)	0.017(5)
C(31)	2*i*		1.4703(7)	0.5065(5)	–0.0486(4)	0.086(5)	0.059(4)	0.067(4)	0.025(3)	–0.002(3)	0.016(3)
C(27)	2*i*		1.2710(8)	0.6208(5)	–0.1913(5)	0.108(6)	0.053(4)	0.075(5)	–0.001(4)	–0.032(4)	0.007(4)
N(4)	2*i*		1.2551(6)	0.6211(4)	0.2120(3)	0.083(4)	0.052(3)	0.067(4)	–0.004(3)	–0.025(3)	0.014(3)
C(14)	2*i*		1.2119(9)	0.5287(5)	0.2010(5)	0.143(7)	0.069(5)	0.065(5)	–0.005(5)	–0.009(5)	–0.006(4)
C(15)	2*i*	0.50	1.274(1)	0.6307(7)	0.3071(7)	0.055(6)	0.040(6)	0.039(6)	0.010(5)	–0.001(5)	0.012(5)
C(15')	2*i*	0.50	1.385(2)	0.6086(9)	0.2617(9)	0.12(1)	0.057(8)	0.055(8)	0.008(8)	–0.026(8)	0.014(7)
C(16)	2*i*		1.252(1)	0.4726(6)	0.2705(6)	0.167(9)	0.075(6)	0.076(6)	–0.008(6)	–0.003(6)	0.019(5)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(18)	2*i*		1.458(1)	0.5094(5)	0.4559(5)	0.185(9)	0.066(5)	0.053(5)	0.029(5)	–0.018(5)	0.008(4)
N(5)	2*i*	0.50	1.280(1)	0.5426(7)	0.3427(6)	0.106(8)	0.042(6)	0.040(6)	0.013(6)	0.006(6)	0.004(5)
N(5')	2*i*	0.50	1.377(1)	0.5202(9)	0.2999(8)	0.105(9)	0.063(8)	0.065(8)	0.017(7)	–0.011(7)	0.021(6)
C(17)	2*i*	0.50	1.317(2)	0.522(1)	0.4380(8)	0.12(1)	0.073(9)	0.046(8)	0.026(9)	0.000(7)	0.029(7)
C(17')	2*i*	0.50	1.488(2)	0.488(1)	0.374(1)	0.10(1)	0.12(1)	0.08(1)	0.04(1)	0.001(9)	0.02(1)
N(9)	2*i*		1.2277(4)	0.8493(3)	–0.2613(3)	0.037(2)	0.049(3)	0.046(3)	0.004(2)	0.008(2)	0.003(2)
N(10)	2*i*		1.2148(5)	0.9355(3)	–0.1369(3)	0.051(3)	0.056(3)	0.048(3)	0.002(2)	0.006(2)	–0.005(2)
C(34)	2*i*		1.1718(6)	0.8543(4)	–0.1825(4)	0.058(3)	0.055(4)	0.053(4)	–0.010(3)	0.014(3)	–0.003(3)
C(35)	2*i*		1.1735(6)	0.9621(5)	–0.0465(4)	0.064(4)	0.077(4)	0.051(4)	0.006(3)	0.003(3)	–0.011(3)
C(32)	2*i*		1.3134(6)	0.9320(4)	–0.2658(4)	0.055(3)	0.067(4)	0.067(4)	–0.012(3)	0.016(3)	–0.001(3)
C(33)	2*i*		1.3053(6)	0.9854(5)	–0.1899(5)	0.061(4)	0.064(4)	0.082(5)	–0.015(3)	0.009(3)	–0.005(4)
C(36)	2*i*		1.0228(6)	0.9893(4)	–0.0475(3)	0.067(4)	0.054(4)	0.049(3)	0.012(3)	0.007(3)	–0.002(3)

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