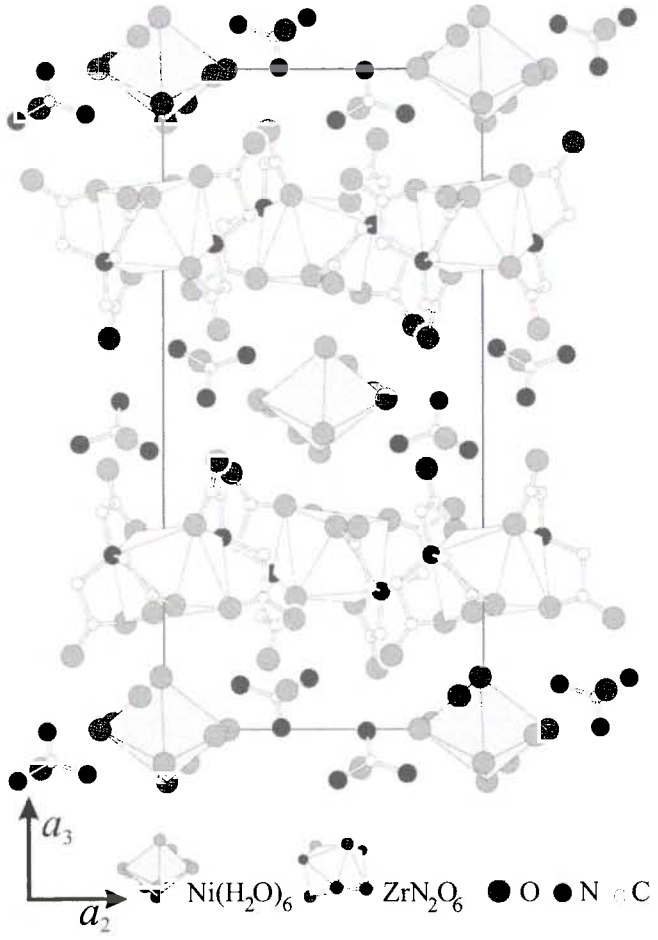
Crystal structure of nickel bis(guanidinium) bis(zirconium) tetra(nitrilotriacetate) dodecahydrate, $Ni(C(NH_2)_3)_2(Zr(N(CH_2COO)_3)_2)_2 \cdot 12H_2O$

E. Haussühl*,[1], G. Giester and E. Tillmanns

Universität Wien - Geozentrum, Institut für Mineralogie und Kristallographie, Althanstr. 14, A-1090 Wien, Austria

Received June 4, 1999, CCDC-No. 1267/205



Abstract

$C_{26}H_{60}N_{10}NiO_{36}Zr_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 11.164(2)$ Å, $b = 10.373(2)$ Å, $c = 21.431(4)$ Å, $\beta = 92.21(3)°$, $V = 2480.0$ Å^3, $Z = 2$, $\rho_m = 1.779(2)$ g·cm^{-3}, $R_{gt}(F) = 0.020$, $wR(F^2) = 0.048$, $T = 293$ K.

Source of material

The raw material was obtained by slow evaporation of an aqueous solution of $(C(NH_2)_3)_2Zr(N(CH_2COO)_3)_2 \cdot H_2O$ and $NiZr(N(CH_2COO)_3)_2 \cdot 8.5H_2O$ in a molar ratio of 2:1 at 300 K.

* Correspondence author (e-mail: eiken@kristall.erdw.ethz.ch)
[1] present address: ETH Zürich, Laboratorium für Kristallographie, Sonneggstr. 5, CH-8092 Zürich, Switzerland

Discussion

The constituents of the structure are arranged in two types of layers parallel (001). The first type is alternately occupied by guanidinium cations, interstitial water and nickel cations which are coordinated by six water molecules. The second type consists of zirconium-nitrilotriacetate anions, in which the Zr anions are coordinated by six oxygen and two nitrogen atoms of the nitrilotriacetate groups. These anions exhibit the well known persistent conformation. The ionic interactions between these layers are reinforced by a 3-dimensional network of hydrogen bonds which are characterized by D···A distances between 2.69 Å and 3.05Å and D–H···A angles of about 170°.

Table 1. Data collection and handling.

Crystal:	green prism, size 0.24 × 0.25 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.05 cm^{-1}
Diffractometer, scan mode:	Stoe-Siemens AED, 2θ/ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12012, 5699
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4880
$N(param)_{refined}$:	461
Programs:	SHELXS-97 [1], SHELXL-97 [2], ATOMS [3]

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

Atom	Site	x	y	z	U_{iso}
H(11A)	4e	–0.298(2)	0.220(2)	0.2932(8)	0.031(5)
H(11B)	4e	–0.262(2)	0.084(2)	0.3120(8)	0.026(4)
H(12A)	4e	–0.088(2)	0.260(2)	0.3774(8)	0.033(5)
H(12B)	4e	–0.110(2)	0.109(2)	0.3768(8)	0.033(5)
H(13A)	4e	–0.041(2)	0.360(2)	0.2894(9)	0.037(5)
H(13B)	4e	–0.164(2)	0.364(2)	0.2649(8)	0.031(5)
H(21A)	4e	0.301(2)	–0.195(2)	0.2554(9)	0.036(5)
H(21B)	4e	0.274(2)	–0.071(2)	0.2952(9)	0.033(5)
H(22A)	4e	0.103(2)	–0.283(2)	0.3373(8)	0.033(5)
H(22B)	4e	0.147(2)	–0.146(2)	0.3554(8)	0.025(4)
H(23A)	4e	0.158(2)	–0.322(2)	0.2151(9)	0.036(5)
H(23B)	4e	0.035(2)	–0.324(2)	0.2388(8)	0.033(5)
H(31A)	4e	0.149(3)	–0.823(3)	0.044(2)	0.11(1)
H(31B)	4e	0.096(2)	–0.769(3)	0.101(1)	0.068(8)
H(32A)	4e	0.158(2)	–0.475(3)	0.068(1)	0.074(9)
H(32B)	4e	0.072(2)	–0.550(3)	0.105(1)	0.076(8)
H(33A)	4e	0.247(3)	–0.558(3)	–0.015(1)	0.09(1)
H(33B)	4e	0.259(3)	–0.700(4)	–0.014(2)	0.12(1)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
HW(1A)	4*e*	0.124(2)	0.088(3)	0.922(1)	0.074(8)
HW(1B)	4*e*	0.141(3)	–0.006(3)	0.964(2)	0.10(1)
HW(2A)	4*e*	–0.325(3)	0.718(3)	0.489(1)	0.09(1)
HW(2B)	4*e*	–0.384(2)	0.691(2)	0.537(1)	0.065(8)
HW(3A)	4*e*	0.339(3)	0.103(3)	0.586(1)	0.09(1)
HW(3B)	4*e*	0.220(3)	0.141(3)	0.578(1)	0.081(9)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
HW(4A)	4*e*	–0.143(2)	0.503(2)	0.589(1)	0.062(8)
HW(4B)	4*e*	–0.200(2)	0.553(2)	0.539(1)	0.046(6)
HW(5A)	4*e*	0.177(2)	0.530(2)	0.573(1)	0.046(6)
HW(5B)	4*e*	0.107(2)	0.468(2)	0.612(1)	0.046(6)
HW(6A)	4*e*	0.040(2)	0.267(2)	0.474(1)	0.051(6)
HW(6B)	4*e*	0.018(2)	0.261(2)	0.535(1)	0.049(6)

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}
Zr	4*e*	–0.00271(1)	0.02566(1)	0.231282(6)	0.01648(7)	0.01385(7)	0.01558(7)	0.00188	0.00061(4)	0.00025(4)
Ni	2*d*	0	1/2	1/2	0.0269(1)	0.0234(1)	0.0163(1)	0.0020(1)	0.0000(1)	0.00110(9)
N(1)	4*e*	–0.1166(1)	0.1831(1)	0.29062(5)	0.0191(5)	0.0185(5)	0.0208(6)	0.0001(4)	0.0000(4)	–0.0033(4)
N(2)	4*e*	0.1218(1)	–0.1603(1)	0.26417(5)	0.0201(6)	0.0205(5)	0.0203(6)	0.0013(4)	0.0019(4)	0.0035(4)
N(31)	4*e*	0.1390(2)	–0.7613(2)	0.0673(1)	0.105(2)	0.0379(9)	0.057(1)	–0.012(1)	0.038(1)	–0.0116(8)
N(32)	4*e*	0.1311(2)	–0.5443(2)	0.0771(1)	0.077(1)	0.0389(9)	0.064(1)	0.0141(9)	0.026(1)	0.0017(8)
N(33)	4*e*	0.2442(3)	–0.6346(2)	0.0026(1)	0.124(2)	0.051(1)	0.075(1)	0.023(1)	0.062(2)	0.017(1)
C(11)	4*e*	–0.2452(1)	0.1502(1)	0.28361(7)	0.0181(6)	0.0249(7)	0.0269(7)	0.0004(5)	0.0022(5)	–0.0046(6)
C(12)	4*e*	–0.0722(1)	0.1785(2)	0.35646(7)	0.0284(8)	0.0323(7)	0.0202(7)	0.0046(6)	–0.0004(6)	–0.0069(6)
C(13)	4*e*	–0.0961(2)	0.3134(1)	0.26433(8)	0.0290(8)	0.0161(6)	0.0367(8)	0.0003(6)	0.0037(6)	–0.0030(6)
C(14)	4*e*	–0.2720(1)	0.0995(1)	0.21836(7)	0.0216(6)	0.0146(6)	0.0254(7)	–0.0002(5)	0.0007(5)	0.0006(5)
C(15)	4*e*	0.0618(1)	0.1491(1)	0.35955(7)	0.0291(7)	0.0251(7)	0.0233(7)	0.0006(6)	–0.0031(6)	–0.0031(6)
C(16)	4*e*	–0.0490(1)	0.3109(1)	0.19937(7)	0.0225(7)	0.0199(6)	0.0343(8)	0.0003(5)	–0.0005(6)	0.0034(6)
C(21)	4*e*	0.2484(1)	–0.1229(2)	0.25877(7)	0.0178(6)	0.0274(7)	0.0301(8)	0.0026(5)	–0.0005(5)	0.0070(6)
C(22)	4*e*	0.0940(1)	–0.1927(2)	0.32931(7)	0.0272(7)	0.0291(7)	0.0218(7)	0.0050(6)	0.0005(6)	0.0081(6)
C(23)	4*e*	0.0925(1)	–0.2696(1)	0.22117(7)	0.0277(7)	0.0168(6)	0.0322(8)	0.0038(5)	0.0020(6)	0.0006(6)
C(24)	4*e*	0.2621(1)	–0.0358(1)	0.20265(6)	0.0212(6)	0.0223(6)	0.0222(6)	–0.0004(5)	0.0003(5)	–0.0018(5)
C(25)	4*e*	–0.0319(1)	–0.1512(1)	0.34291(7)	0.0286(7)	0.0193(6)	0.0225(7)	–0.0010(5)	0.0024(6)	0.0021(5)
C(26)	4*e*	0.0398(1)	–0.2247(1)	0.15919(7)	0.0244(7)	0.0200(6)	0.0283(7)	0.0012(5)	0.0026(6)	–0.0037(6)
C(30)	4*e*	0.1689(2)	–0.6470(2)	0.04821(9)	0.050(1)	0.0400(9)	0.0360(9)	0.0074(8)	0.0090(8)	0.0018(8)
O(14A)	4*e*	–0.37307(9)	0.1091(1)	0.19422(5)	0.0209(5)	0.0285(5)	0.0348(6)	0.0047(4)	–0.0051(4)	–0.0051(5)
O(14B)	4*e*	–0.18439(9)	0.0413(1)	0.19323(5)	0.0207(5)	0.0261(5)	0.0254(5)	0.0047(4)	–0.0023(4)	–0.0056(4)
O(15A)	4*e*	0.1215(1)	0.1686(1)	0.40732(6)	0.0373(7)	0.0578(8)	0.0295(6)	0.0068(6)	–0.0100(5)	–0.0146(6)
O(15B)	4*e*	0.10282(9)	0.0982(1)	0.30929(5)	0.0224(5)	0.0271(5)	0.0244(5)	0.0007(4)	–0.0015(4)	–0.0050(4)
O(16A)	4*e*	–0.0578(1)	0.4080(1)	0.16677(6)	0.0522(8)	0.0246(6)	0.0465(7)	0.0099(5)	0.0078(6)	0.0125(5)
O(16B)	4*e*	0.0050(1)	0.2076(1)	0.18334(5)	0.0309(6)	0.0192(5)	0.0312(6)	0.0053(4)	0.0077(4)	0.0058(4)
O(24A)	4*e*	0.35796(9)	–0.0326(1)	0.17649(5)	0.0207(5)	0.0426(6)	0.0302(6)	0.0026(5)	0.0056(4)	0.0055(5)
O(24B)	4*e*	0.17038(9)	0.0336(1)	0.18781(5)	0.0241(5)	0.0281(5)	0.0298(5)	0.0065(4)	0.0072(4)	0.0091(4)
O(25A)	4*e*	–0.0713(1)	–0.1738(1)	0.39449(5)	0.0400(7)	0.0452(7)	0.0245(5)	0.0070(5)	0.0096(5)	0.0099(5)
O(25B)	4*e*	–0.08938(9)	–0.0867(1)	0.30025(5)	0.0219(5)	0.0221(5)	0.0249(5)	0.0021(4)	0.0029(4)	0.0050(4)
O(26A)	4*e*	0.0525(1)	–0.2863(1)	0.11163(6)	0.0638(9)	0.0333(6)	0.0317(6)	0.0161(6)	0.0027(6)	–0.0105(5)
O(26B)	4*e*	–0.0237(1)	–0.1201(1)	0.16162(5)	0.0323(6)	0.0238(5)	0.0245(5)	0.0099(4)	–0.0054(4)	–0.0060(4)
OW(1)	4*e*	0.1621(1)	0.0754(2)	0.94959(7)	0.0524(8)	0.0552(9)	0.0380(7)	0.0148(7)	–0.0171(6)	–0.0062(6)
OW(2)	4*e*	–0.3412(2)	0.6597(2)	0.50950(8)	0.0576(9)	0.0541(9)	0.0492(8)	0.0087(7)	0.0080(7)	–0.0026(7)
OW(3)	4*e*	0.2859(2)	0.1156(2)	0.55896(7)	0.0463(8)	0.071(1)	0.0474(8)	0.0092(7)	0.0067(7)	–0.0095(7)
OW(4)	4*e*	0.1082(1)	0.5096(1)	0.57940(6)	0.0354(6)	0.0424(6)	0.0230(5)	–0.0016(5)	–0.0054(5)	0.0070(5)
OW(5)	4*e*	–0.1513(1)	0.4986(1)	0.55177(6)	0.0345(6)	0.0442(7)	0.0232(6)	0.0045(5)	0.0030(5)	–0.0002(5)
OW(6)	4*e*	–0.0023(1)	0.2996(1)	0.49945(6)	0.0474(7)	0.0287(5)	0.0247(5)	0.0068(5)	0.0003(5)	0.0012(4)

Acknowledgments. The authors wish to thank the Austrian Fonds zur Förderung der wissenschaftlichen Forschung FWF (P11684-GEO) and the International Center for Diffraction Data for financial assistance of this work (Grant 90-03 ET).

References

1. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Large Structures. Acta Crystallogr. A**46** (1990) 467-473.
2. Sheldrick, G. M.: SHELXL-97. Program for Refinement of Crystal Structures. University of Göttingen, Germany 1997.
3. Dowty, E.: Atoms 4.1, A Complete Program for Displaying Atomic Structures. By Shape software, 521 Hidden Valley Road, Kingsport, TN 37663, USA 1998.