Crystal structure of rubidium guanidinium zirconium bis(nitrilotriacetate) dihydrate, $Rb(C(NH_2)_3)Zr(N(CH_2COO)_3)_2 \cdot 2H_2O$

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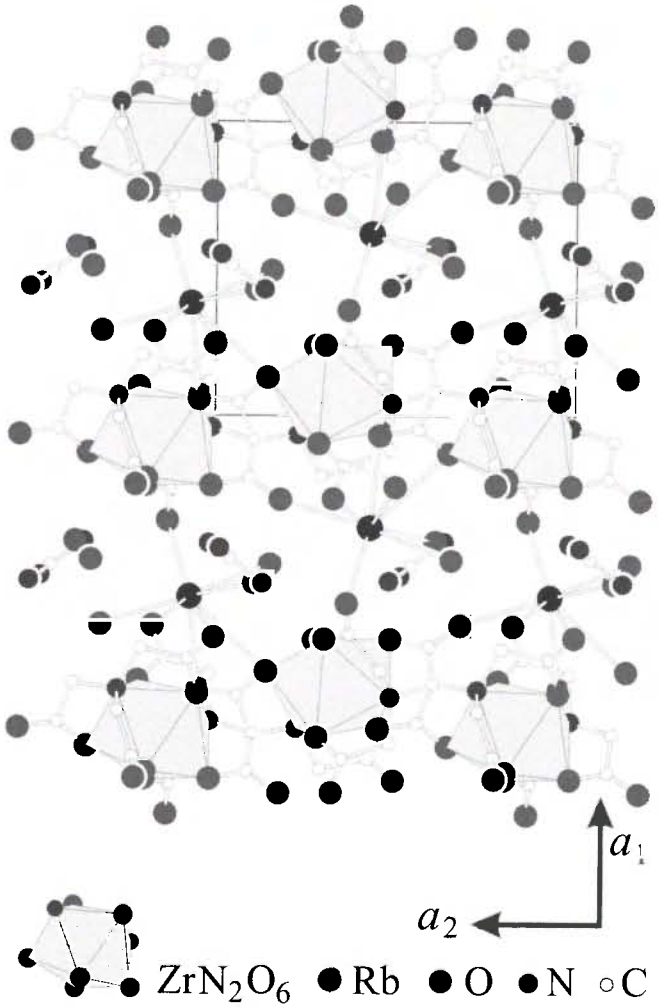
Received December 9, 1999, CCDC-No. 1267/362



Abstract

$C_{13}H_{22}N_5O_{14}RbZr$, monoclinic, $P12_11$ (No. 4), a = 9.956(2) Å, b = 11.208(2) Å, c = 10.615(2) Å, β = 113.53(1)°, V = 1086.0 Å^3, Z = 2, ρ_m = 1.982 g·cm^{-3} at 293 K, $R_{gt}(F)$ = 0.018, $wR_{ref}(F^2)$ = 0.043, T = 298 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 $Rb_2Zr(N(CH_2COO)_3)_2 \cdot 2H_2O$ in a molar ratio of about 1:2 at 300 K.

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Discussion

The constituents of the structure are arranged in two types of layers parallel to (100). The first type is occupied by rubidium cations coordinated by eight oxygen atoms forming irregular RbO_8-polyhedra, guanidinium cations and interstitial water. The second type consists of zirconium-nitrilotriacetate anions, in which the Zr-cation, coordinated by six oxygen and two nitrogen atoms of the nitrilotriacetate groups, possess an almost 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 2.77 Å and 3.19 Å and D–H···A angles of about 160°. The static piezoelectric constant d_{222} is about 5 times larger than d_{111} of α-quartz. The pyroelectric effect surmounts the corresponding value of tourmaline by a factor 10.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.30 × 0.32 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	28.15 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, ω/2θ
$2\theta_{max}$:	51.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4480, 4251
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4144
$N(param)_{refined}$:	394
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)	2a	1.107(3)	0.448(3)	0.966(3)	0.046(8)
H(11B)	2a	1.166(3)	0.397(3)	0.864(3)	0.040(8)
H(12A)	2a	1.259(3)	0.230(2)	1.084(3)	0.033(7)
H(12B)	2a	1.260(4)	0.223(3)	0.939(3)	0.051(9)
H(13A)	2a	1.041(3)	0.213(3)	1.106(3)	0.045(8)
H(13B)	2a	0.998(3)	0.344(3)	1.065(3)	0.037(7)
H(21A)	2a	1.000(3)	−0.137(2)	0.692(3)	0.021(6)
H(21B)	2a	0.901(3)	−0.161(3)	0.555(3)	0.038(8)
H(22A)	2a	1.140(3)	−0.015(2)	0.539(3)	0.026(6)
H(22B)	2a	1.171(3)	−0.028(2)	0.694(3)	0.023(6)
H(23A)	2a	0.835(4)	−0.035(3)	0.380(3)	0.050(9)
H(23B)	2a	0.937(3)	0.081(3)	0.394(3)	0.043(8)
H(31A)	2a	0.427(4)	0.841(3)	0.810(3)	0.05(1)
H(31B)	2a	0.390(4)	0.832(3)	0.926(3)	0.05(1)
H(32A)	2a	0.606(6)	1.055(5)	1.087(6)	0.13(2)
H(32B)	2a	0.498(6)	0.984(6)	1.108(6)	0.13(2)
H(33A)	2a	0.541(6)	0.979(5)	0.780(5)	0.11(1)
H(33B)	2a	0.615(5)	1.068(4)	0.887(5)	0.11(1)
HW(1A)	2a	0.362(5)	−0.173(4)	0.552(5)	0.10(2)
HW(1B)	2a	0.512(6)	−0.146(5)	0.601(5)	0.10(2)
HW(2A)	2a	1.586(6)	0.302(5)	0.736(6)	0.13(1)
HW(2B)	2a	1.427(6)	0.285(5)	0.673(6)	0.13(1)

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	2*a*	0.91040(2)	0.16416(2)	0.72238(2)	0.02234(9)	0.02009(9)	0.02008(9)	–0.00128(9)	0.00961(7)	–0.00372(9)
Rb	2*a*	0.38100(3)	0.06902(2)	0.38450(3)	0.0454(2)	0.0416(2)	0.0593(2)	0.0097(1)	0.0257(1)	0.0117(1)
N(1)	2*a*	1.0655(2)	0.2708(2)	0.9334(2)	0.028(1)	0.0188(9)	0.024(1)	–0.0015(8)	0.0108(8)	–0.0016(8)
N(2)	2*a*	0.9592(2)	0.0101(2)	0.5799(2)	0.028(1)	0.0257(9)	0.026(1)	–0.0007(8)	0.0138(8)	–0.0032(8)
N(31)	2*a*	0.4291(3)	0.8653(3)	0.8859(3)	0.053(2)	0.056(2)	0.054(2)	–0.024(1)	0.030(1)	–0.010(1)
N(32)	2*a*	0.5490(4)	0.9860(5)	1.0680(4)	0.054(2)	0.132(3)	0.069(2)	–0.001(2)	0.020(2)	–0.058(2)
N(33)	2*a*	0.5654(4)	1.0167(4)	0.8624(5)	0.059(2)	0.086(2)	0.096(3)	–0.028(2)	0.021(2)	0.030(2)
C(11)	2*a*	1.0857(3)	0.3940(2)	0.8950(3)	0.041(1)	0.020(1)	0.034(1)	–0.008(1)	0.016(1)	–0.0018(9)
C(12)	2*a*	1.2049(3)	0.2040(2)	0.9948(3)	0.025(1)	0.028(1)	0.031(1)	–0.0003(9)	0.006(1)	–0.0020(9)
C(13)	2*a*	0.9913(3)	0.2695(2)	1.0306(2)	0.037(1)	0.024(1)	0.023(1)	0.000(1)	0.014(1)	–0.0026(9)
C(14)	2*a*	0.9531(3)	0.4363(2)	0.7735(2)	0.036(1)	0.026(1)	0.031(1)	0.001(1)	0.022(1)	0.001(1)
C(15)	2*a*	1.1693(2)	0.0720(2)	0.9816(2)	0.027(1)	0.028(1)	0.026(1)	0.005(1)	0.014(1)	0.000(1)
C(16)	2*a*	0.8349(3)	0.2269(2)	0.9687(3)	0.040(1)	0.022(1)	0.037(1)	0.001(1)	0.025(1)	–0.001(1)
C(21)	2*a*	0.9166(3)	–0.1066(2)	0.6163(3)	0.041(1)	0.022(1)	0.035(1)	–0.004(1)	0.019(1)	–0.006(1)
C(22)	2*a*	1.1176(3)	0.0145(2)	0.6086(3)	0.030(1)	0.032(1)	0.036(1)	0.003(1)	0.017(1)	–0.003(1)
C(23)	2*a*	0.8696(3)	0.0392(2)	0.4332(3)	0.038(1)	0.037(1)	0.026(1)	0.001(1)	0.013(1)	–0.007(1)
C(24)	2*a*	0.7866(3)	–0.0940(2)	0.6534(3)	0.034(1)	0.030(1)	0.027(1)	–0.006(1)	0.011(1)	–0.004(1)
C(25)	2*a*	1.1718(3)	0.1421(2)	0.6360(2)	0.028(1)	0.035(2)	0.027(1)	0.0010(9)	0.0111(9)	0.0027(9)
C(26)	2*a*	0.7454(3)	0.1247(2)	0.4120(2)	0.033(1)	0.038(1)	0.024(1)	–0.004(1)	0.010(1)	–0.0046(9)
C(30)	2*a*	0.5134(3)	0.9573(3)	0.9381(3)	0.024(1)	0.042(2)	0.042(2)	0.001(1)	0.011(1)	–0.005(1)
O(14A)	2*a*	0.9323(2)	0.5438(2)	0.7492(2)	0.052(1)	0.0222(9)	0.048(1)	0.0029(8)	0.0256(9)	0.0046(8)
O(14B)	2*a*	0.8711(2)	0.3543(2)	0.6967(2)	0.037(1)	0.0239(8)	0.0298(9)	0.0024(7)	0.0056(8)	–0.0040(7)
O(15A)	2*a*	1.2434(2)	0.0003(2)	1.0667(2)	0.043(1)	0.037(1)	0.038(1)	0.0116(8)	0.0088(9)	0.0081(8)
O(15B)	2*a*	1.0542(2)	0.0447(1)	0.8714(2)	0.0359(9)	0.0231(8)	0.0292(9)	–0.0029(7)	0.0122(8)	–0.0003(7)
O(16A)	2*a*	0.7636(2)	0.2223(2)	1.0397(2)	0.051(1)	0.047(1)	0.052(1)	–0.0031(9)	0.039(1)	–0.0071(9)
O(16B)	2*a*	0.7848(2)	0.1911(2)	0.8431(2)	0.0328(9)	0.044(1)	0.0367(9)	–0.0094(7)	0.0203(7)	–0.0159(8)
O(24A)	2*a*	0.7143(2)	–0.1821(2)	0.6552(2)	0.041(1)	0.031(1)	0.061(1)	–0.0141(8)	0.022(1)	–0.0025(9)
O(24B)	2*a*	0.7612(2)	0.0112(2)	0.6849(2)	0.039(1)	0.0329(9)	0.051(1)	–0.0127(8)	0.0273(9)	–0.0170(8)
O(25A)	2*a*	1.2830(2)	0.1710(2)	0.6211(2)	0.0360(9)	0.047(1)	0.064(1)	–0.004(1)	0.0309(9)	0.000(1)
O(25B)	2*a*	1.0957(2)	0.2126(2)	0.6786(2)	0.0352(9)	0.0264(8)	0.0338(9)	–0.0037(7)	0.0201(8)	–0.0024(7)
O(26A)	2*a*	0.6415(2)	0.1316(2)	0.3018(2)	0.044(1)	0.067(2)	0.031(1)	0.0071(9)	0.0013(9)	–0.0157(9)
O(26B)	2*a*	0.7615(2)	0.1930(2)	0.5147(2)	0.044(1)	0.037(1)	0.0254(8)	0.0141(8)	0.0032(8)	–0.0064(7)
OW(1)	2*a*	0.4288(3)	–0.1110(2)	0.5946(2)	0.043(1)	0.044(1)	0.057(1)	–0.005(1)	0.013(1)	–0.004(1)
OW(2)	2*a*	1.5066(3)	0.3455(3)	0.7119(4)	0.050(2)	0.063(2)	0.106(2)	–0.012(1)	0.010(2)	0.002(2)

References

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