Crystal structure of bis(4,4′-bipyridinium) bis(imidazolium) hexanitratostrontium(II) bis(imidazolate) — imidazole — water (1:2:1), $[C_{10}H_{10}N_2]_2[C_3H_5N_2]_2[Sr(NO_3)_6][C_3H_3N_2]_2 \cdot 2C_3H_4N_2 \cdot H_2O$

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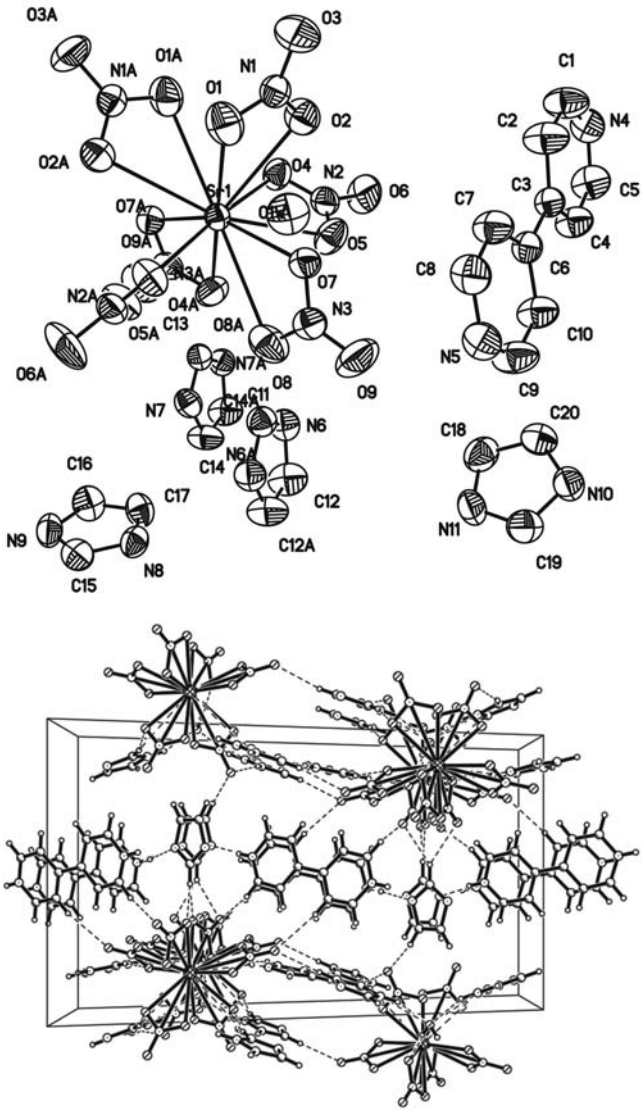

Abstract

$C_{38}H_{46}N_{22}O_{19}Sr$, monoclinic, $P12/c1$ (no. 13), a = 8.957(1) Å, b = 13.055(2) Å, c = 23.670(3) Å, β = 110.267(5)°, V = 2596.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.113, T = 296 K.

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Source of material

All reagents were of analytical grade. The title compound was prepared from imidazole, 4,4′-bipyridine, strontium nitrate, anhydrous ethyl alcohol, and deionized water in the molar ratio 1 : 2 : 3 : 56 : 264 by heating at 130 °C for 8 d. After crystallization, the autoclave was cooled down to room temperature and colourless block-shaped single crystals were separated by sonication, further washed by distilled water and dried on air.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.93 Å, d(N—H) = 0.86 Å and U_{iso}(H) = 1.2 U_{eq}(C,N). The hydrogen atom of water was found in the difference Fourier map and refined.

Discussion

Sr(II) atoms are coordinated by twelve O atoms from six nitrate anions (figure, top). The d(Sr—O) bond lengths are in the range of 2.670(2) - 2.909(2) Å; the d(O—N) bond lengths are in the range of 1.219(3) - 1.256(3) Å; the d(C—N) bond lengths are in the range of 1.305(4) - 1.361(4) Å; and the d(C—C) bond lengths are in the range of 1.325(6) - 1.484(3) Å.

In the crystal structure, there are intermolecular hydrogen bonds (figure, bottom) N5–H5A···N6 (d(N5···N6) = 1.92 Å, ∠N5–H5A···N6 = 171.3°); N4–H4A···N7 (d(N4···N7) = 1.96 Å, ∠N4–H4A···N7 = 174.5°); N11–H11A···O1W (d(N11···O1W) = 2.10 Å, ∠N11–H11A···O1W = 156.7°); N8–H8A···N10 (d(N8···N10) = 1.82 Å, ∠N8–H8A···N10 = 172.9°); N9–H9A···O7 (d(N9···O7) = 1.99 Å, ∠N9–H9A···O7 = 175.0°); O1W–H1W···O7 (d(O1W···O7) = 2.04(1) Å, ∠O1W–H1W···O7 = 174.3°). There are also π-π stacking interactions observed with distance of 3.606 Å between the 4,4′-bipyridine molecules. All the bond lengths and bond angles are within the reasonable ranges.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.21 × 0.23 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.33 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16442, 6247
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4608
$N(param)_{refined}$:	366
Programs:	SHELXS-97, SHELXL-97 [1]

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

Atom	Site	x	y	z	U_{iso}
H(5A)	4*g*	1.1850	0.4514	0.1568	0.064
H(4A)	4*g*	0.3193	0.5689	–0.1526	0.070
H(1)	4*g*	0.4421	0.6981	–0.0957	0.084
H(10)	4*g*	0.8547	0.3528	0.0122	0.068
H(8)	4*g*	1.0813	0.6064	0.1446	0.069
H(7)	4*g*	0.8538	0.6404	0.0657	0.068
H(4)	4*g*	0.6376	0.3772	–0.0554	0.067
H(9)	4*g*	1.0774	0.3262	0.0934	0.079
H(2)	4*g*	0.6654	0.6725	–0.0143	0.079
H(5)	4*g*	0.4121	0.4121	–0.1342	0.076
H(11)	2*f*	½	0.5406	¼	0.059
H(12)	4*g*	0.3676	0.255	0.2124	0.083
H(18)	4*g*	0.7841	0.1028	0.1137	0.078
H(19)	4*g*	1.2323	0.0560	0.1363	0.072
H(11A)	4*g*	1.0362	0.0471	0.1811	0.077
H(20)	4*g*	0.8259	0.1481	0.0207	0.069
H(8A)	4*g*	0.8385	0.1243	0.4907	0.059
H(16)	4*g*	0.4716	0.1227	0.5422	0.076
H(9A)	4*g*	0.7116	0.1595	0.6255	0.066
H(15)	4*g*	0.9356	0.1597	0.5947	0.062
H(17)	4*g*	0.5549	0.0998	0.4554	0.068
H(13)	2*e*	0	0.5281	¼	0.057
H(14)	4*g*	0.1323	0.2421	0.2877	0.081
H(1W)	4*g*	1.079(3)	0.921(2)	0.263(2)	0.078

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}
Sr(1)	2*f*	½	0.85618(2)	¼	0.0312(2)	0.0349(2)	0.0278(2)	0	0.0089(1)	0
O(2)	4*g*	0.4975(2)	0.9920(1)	0.16039(8)	0.055(1)	0.055(1)	0.047(1)	–0.0070(9)	0.0101(9)	–0.0028(9)
O(4)	4*g*	0.2201(2)	0.8474(1)	0.15651(8)	0.049(1)	0.056(1)	0.040(1)	0.0066(8)	0.0091(8)	–0.0078(9)
O(7)	4*g*	0.7411(2)	0.8155(1)	0.21165(7)	0.0404(9)	0.0404(9)	0.042(1)	0.0037(7)	0.0148(8)	0.0023(8)
O(1)	4*g*	0.6660(2)	1.0347(2)	0.24528(9)	0.050(1)	0.078(1)	0.044(1)	–0.001(1)	0.0047(9)	0.002(1)
O(3)	4*g*	0.6174(3)	1.1359(2)	0.1687(1)	0.107(2)	0.047(1)	0.088(2)	–0.012(1)	0.051(2)	0.012(1)
O(8)	4*g*	0.6921(3)	0.6768(2)	0.24942(9)	0.076(1)	0.054(1)	0.059(1)	–0.017(1)	0.015(1)	0.009(1)
O(5)	4*g*	0.4099(2)	0.7649(2)	0.14164(9)	0.044(1)	0.067(1)	0.055(1)	0.0068(9)	0.0086(9)	–0.019(1)
O(6)	4*g*	0.1806(3)	0.7736(2)	0.07157(9)	0.083(2)	0.094(2)	0.037(1)	0.017(1)	–0.013(1)	–0.020(1)
O(9)	4*g*	0.8459(3)	0.6755(2)	0.1965(1)	0.070(2)	0.078(2)	0.083(2)	0.031(1)	0.024(1)	–0.016(1)
N(1)	4*g*	0.5952(2)	1.0556(2)	0.1915(1)	0.038(1)	0.043(1)	0.042(1)	0.0040(9)	0.020(1)	0.001(1)
N(3)	4*g*	0.7625(2)	0.7207(2)	0.21959(9)	0.036(1)	0.045(1)	0.040(1)	0.0008(9)	0.0022(9)	–0.005(1)
N(2)	4*g*	0.2699(3)	0.7952(2)	0.12273(9)	0.054(1)	0.042(1)	0.033(1)	–0.002(1)	0.005(1)	–0.0027(9)
N(5)	4*g*	1.1000(3)	0.4633(2)	0.1265(1)	0.041(1)	0.065(2)	0.044(1)	–0.001(1)	0.001(1)	–0.002(1)
N(4)	4*g*	0.4047(3)	0.5577(2)	–0.1224(1)	0.044(1)	0.075(2)	0.042(1)	0.007(1)	–0.001(1)	0.001(1)
C(3)	4*g*	0.6796(3)	0.5205(2)	–0.0239(1)	0.041(1)	0.041(1)	0.037(1)	0.000(1)	0.009(1)	–0.004(1)
C(6)	4*g*	0.8254(3)	0.5006(2)	0.0288(1)	0.043(1)	0.040(1)	0.037(1)	–0.003(1)	0.011(1)	–0.006(1)
C(1)	4*g*	0.4814(4)	0.6318(2)	–0.0871(2)	0.061(2)	0.055(2)	0.069(2)	0.011(2)	–0.008(2)	–0.001(2)
C(10)	4*g*	0.8975(3)	0.4064(2)	0.0388(1)	0.055(2)	0.044(2)	0.056(2)	0.005(1)	–0.001(1)	–0.013(1)
C(8)	4*g*	1.0340(3)	0.5541(2)	0.1177(1)	0.059(2)	0.056(2)	0.043(2)	–0.007(1)	0.001(1)	–0.011(1)
C(7)	4*g*	0.8976(3)	0.5751(2)	0.0701(1)	0.063(2)	0.044(2)	0.049(2)	0.005(1)	0.002(1)	–0.010(1)
C(4)	4*g*	0.5999(3)	0.4441(2)	–0.0619(1)	0.055(2)	0.047(2)	0.054(2)	0.003(1)	0.002(1)	–0.013(1)
C(9)	4*g*	1.0317(4)	0.3911(2)	0.0875(2)	0.055(2)	0.055(2)	0.067(2)	0.014(1)	–0.005(2)	–0.008(2)
C(2)	4*g*	0.6162(4)	0.6170(2)	–0.0381(2)	0.062(2)	0.045(2)	0.066(2)	0.001(1)	–0.009(2)	–0.011(1)
C(5)	4*g*	0.4646(3)	0.4659(2)	–0.1096(1)	0.057(2)	0.062(2)	0.053(2)	–0.001(2)	–0.005(1)	–0.017(2)
C(11)	2*f*	½	0.4694(3)	¼	0.048(2)	0.050(2)	0.050(2)	0	0.018(2)	0
C(12)	4*g*	0.4276(4)	0.3125(3)	0.2293(2)	0.056(2)	0.056(2)	0.087(3)	–0.008(1)	0.013(2)	–0.006(2)
N(6)	4*g*	0.3830(3)	0.4113(2)	0.2162(1)	0.040(1)	0.062(2)	0.050(1)	–0.003(1)	0.012(1)	–0.001(1)
N(10)	4*g*	1.0589(3)	0.1077(2)	0.0632(1)	0.060(2)	0.052(1)	0.047(1)	0.000(1)	0.022(1)	0.003(1)
C(18)	4*g*	0.8796(4)	0.0990(2)	0.1065(2)	0.067(2)	0.057(2)	0.084(2)	0.003(2)	0.042(2)	0.005(2)
C(19)	4*g*	1.1256(4)	0.0735(2)	0.1185(1)	0.062(2)	0.057(2)	0.059(2)	0.008(1)	0.018(2)	0.003(2)
N(11)	4*g*	1.0188(3)	0.0678(2)	0.1449(1)	0.095(2)	0.059(2)	0.046(1)	0.011(1)	0.035(2)	0.010(1)
C(20)	4*g*	0.9031(4)	0.1238(2)	0.0556(2)	0.053(2)	0.057(2)	0.057(2)	0.002(1)	0.011(1)	0.005(1)
N(8)	4*g*	0.7804(3)	0.1281(2)	0.5129(1)	0.060(1)	0.047(1)	0.050(1)	–0.002(1)	0.030(1)	–0.002(1)
C(16)	4*g*	0.5748(4)	0.1274(2)	0.5419(2)	0.054(2)	0.076(2)	0.064(2)	–0.009(2)	0.026(2)	0.000(2)
N(9)	4*g*	0.7077(3)	0.1481(2)	0.5892(1)	0.077(2)	0.052(1)	0.042(1)	–0.003(1)	0.026(1)	–0.004(1)
C(15)	4*g*	0.8304(4)	0.1478(2)	0.5706(1)	0.053(2)	0.044(2)	0.054(2)	–0.006(1)	0.014(1)	0.000(1)
C(17)	4*g*	0.6210(4)	0.1149(2)	0.4944(1)	0.061(2)	0.059(2)	0.045(2)	–0.012(1)	0.012(1)	–0.005(1)
C(13)	2*e*	0	0.4569(3)	¼	0.054(2)	0.046(2)	0.042(2)	0	0.017(2)	0
C(14)	4*g*	0.0722(4)	0.2996(2)	0.2707(2)	0.058(2)	0.051(2)	0.081(2)	0.009(1)	0.006(2)	0.008(2)
N(7)	4*g*	0.1166(3)	0.3989(2)	0.2839(1)	0.043(1)	0.060(1)	0.045(1)	–0.003(1)	0.009(1)	0.003(1)
O(1W)	2*e*	0	0.9598(2)	¼	0.040(2)	0.057(2)	0.058(2)	0	0.016(1)	0

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References

1. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.