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Crystal structure of diaquabis(1,10-phenanthroline-κ^2N,N')sodium chloride, $[Na(H_2O)_2(C_{12}H_8N_2)_2]Cl$

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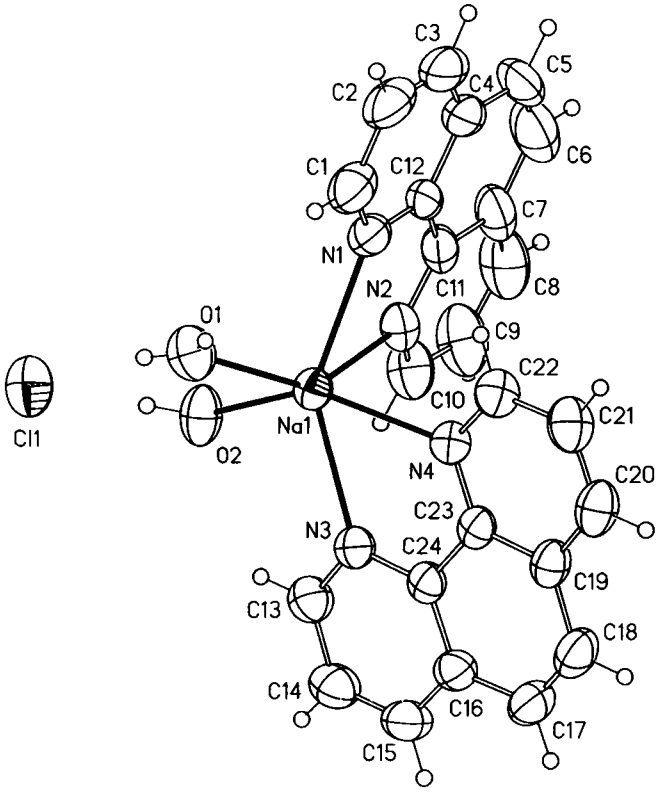

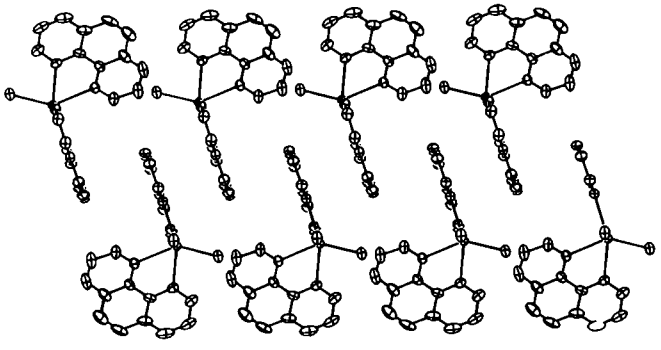

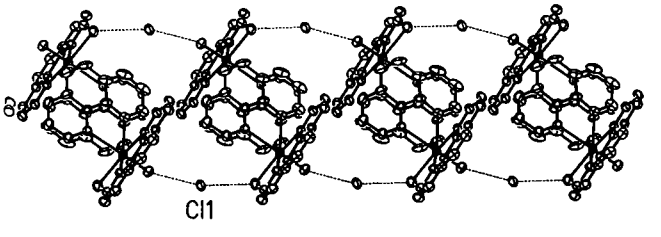

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Abstract

$C_{24}H_{20}ClN_4NaO_2$, monoclinic, $P12_1/c1$ (no. 14), a = 16.352(3) Å, b = 15.579(3) Å, c = 9.132(2) Å, β = 104.54(3)°, V = 2251.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.056, $wR_{ref}(F^2)$ = 0.161, T = 293 K.

Source of material

The title compound was obtained serendipitously from the reaction of manganic dichloride dihydrate (0.40 g, 2.47 mmol), 1,10-phenanthroline monohydrate (0.13 g, 0.66 mmol), phthalic acid (0.43 g, 2.59 mmol) in water (10 mL), and then the pH was raised to about 8.5 with 1.0 M solution of sodium carbonate. The mixture was stirred for ca. 4 h. Subsequently, the resulting suspension was heated in a 25 ml Teflon-lined stainless steel autoclave at 453 K for 5 days. After the autoclave was cooled to room temperature, the solid was filtered off. The resulting yellow filtrate was allowed to stand at room temperature and slow evaporation for two years afforded yellowish block-shaped crystals.

Discussion

Six-coordinated sodium(I) environments are present in the most of the comparatively few sodium(I) complexes, such as in 1,7-bis(2,2'-dihydroxybiphen-3-yl)-4,10-dimethyl-1,4,7,10-tetraazacyclododecane)sodium acetonitrile solvate [1], tetrakis(acetonitrile) bis(tetrahydrofuran)sodium bis(2,6-bis-benzimidazol-2-yl)pyridine)iron [2] and bis(2-(4'-thiazolyl)benzimidazole-N,N') bis(octadeuterofuran)sodium [3]. In the course of studies of phenanthroline complexes, we have synthesized the title mononuclear $[Na(H_2O)_2(phen)_2]Cl$ complex salt and determined its crystal structure.

The title compound consists of monovalent $[Na(H_2O)_2(phen)_2]^+$ complex cations and Cl^- anions. The Na^1 ion is coordinated by four N atoms from two bidentate chelating o-phenanthroline ligands and two O atoms from two coordinated water molecule ligands, to complete a distorted NaN_4O_2 octahedral environment (figure, top). The Na—N bond distances are in the range of 2.460(2) Å to 2.563(2) Å. The Na—O2 and Na—O1 bond lengths are 2.358(2) Å and 2.401(2) Å, respectively. The monovalent $[Na(H_2O)_2(phen)_2]^+$ complex cations are interlinked via hydrogen bonds between the Cl^- anions and O atoms of the coordinated water molecules. The chelating phen ligands exhibit nearly perfect co-planarity, each phen ligand is sandwiched by two phen ligands from two neighboring mononuclear complex cations. The mean interplanar distances are 3.469 Å and 3.566 Å, alternatively, indicating π-π stacking interactions along [010] direction (figure, bottom). Moreover, there are weak hydrogen bonds between Cl^- anions and o-phenanthroline H3 atoms (d(C3···Cl1) = 3.706 Å, ∠C3–H3···Cl1 = 146.5°). Through the hydrogen bonds and π-π stacking interactions, a three-dimensional supramolecular network is constructed.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.18 × 0.26 × 0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.18 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	21760, 5131
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2597
$N(param)_{refined}$:	290
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
HO(1A)	4e	0.2923	0.1082	0.2886	0.133
HO(1B)	4e	0.2803	0.1786	0.1601	0.133
HO(2A)	4e	0.2368	0.2002	−0.1194	0.130
HO(2B)	4e	0.2370	0.1542	−0.2410	0.130
H(1)	4e	0.4113	−0.0027	0.3104	0.111
H(2)	4e	0.5414	−0.0711	0.3948	0.136
H(3)	4e	0.5949	−0.1472	0.2266	0.144
H(5)	4e	0.5802	−0.2029	−0.0427	0.140
H(6)	4e	0.5049	−0.2051	−0.2864	0.155
H(8)	4e	0.3796	−0.1569	−0.4882	0.140
H(9)	4e	0.2581	−0.0789	−0.5554	0.142
H(10)	4e	0.2124	−0.0088	−0.3704	0.106
H(13)	4e	0.0686	0.1003	−0.2552	0.089
H(14)	4e	−0.0685	0.0714	−0.3823	0.101
H(15)	4e	−0.1305	−0.0531	−0.3337	0.099
H(17)	4e	−0.1207	−0.1903	−0.1834	0.101
H(18)	4e	−0.0455	−0.2788	−0.0067	0.099
H(20)	4e	0.0923	−0.3156	0.1793	0.099
H(21)	4e	0.2301	−0.2810	0.2893	0.098
H(22)	4e	0.2868	−0.1572	0.2148	0.084

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}
Na(1)	4e	0.24427(6)	0.02643(6)	−0.00190(9)	0.0605(6)	0.0547(5)	0.0516(5)	−0.0026(4)	0.0163(4)	−0.0025(4)
N(1)	4e	0.3882(2)	−0.0385(1)	0.0995(3)	0.063(2)	0.064(1)	0.076(2)	−0.007(1)	0.005(1)	0.002(1)
N(2)	4e	0.3017(2)	−0.0428(1)	−0.1968(2)	0.081(2)	0.054(1)	0.069(1)	0.002(1)	0.027(1)	−0.003(1)
N(3)	4e	0.0911(1)	−0.0028(1)	−0.1308(2)	0.061(1)	0.056(1)	0.062(1)	0.000(1)	0.015(1)	0.003(1)
N(4)	4e	0.1857(1)	−0.1150(1)	0.0731(2)	0.066(1)	0.057(1)	0.051(1)	−0.001(1)	0.013(1)	0.001(1)
Cl(1)	4e	0.25958(6)	0.32207(5)	0.02343(8)	0.1297(8)	0.0747(5)	0.0539(4)	−0.0012(5)	0.0170(4)	−0.0046(3)
O(1)	4e	0.2820(1)	0.1291(1)	0.1999(2)	0.107(2)	0.088(1)	0.076(1)	0.006(1)	0.033(1)	−0.018(1)
O(2)	4e	0.2254(2)	0.1501(1)	−0.1556(2)	0.130(2)	0.062(1)	0.077(1)	−0.010(1)	0.042(1)	0.002(1)
C(1)	4e	0.4328(2)	−0.0341(2)	0.2419(4)	0.090(3)	0.081(2)	0.091(2)	−0.017(2)	−0.005(2)	0.008(2)
C(2)	4e	0.5116(3)	−0.0750(3)	0.2939(5)	0.089(3)	0.091(3)	0.127(3)	−0.025(2)	−0.035(3)	0.023(3)
C(3)	4e	0.5428(3)	−0.1201(3)	0.1938(7)	0.068(3)	0.071(2)	0.203(5)	−0.009(2)	0.002(3)	0.043(3)
C(4)	4e	0.4989(2)	−0.1269(2)	0.0433(5)	0.059(2)	0.059(2)	0.158(4)	−0.006(2)	0.024(2)	0.025(2)
C(5)	4e	0.5292(3)	−0.1735(2)	−0.0705(7)	0.080(3)	0.068(2)	0.224(5)	0.023(2)	0.079(3)	0.020(3)
C(6)	4e	0.4841(4)	−0.1747(3)	−0.2159(7)	0.142(5)	0.081(3)	0.193(5)	0.022(3)	0.097(4)	0.002(3)
C(7)	4e	0.4073(3)	−0.1317(2)	−0.2640(5)	0.110(3)	0.058(2)	0.141(3)	0.007(2)	0.075(3)	−0.002(2)
C(8)	4e	0.3605(4)	−0.1276(3)	−0.4145(5)	0.183(5)	0.091(3)	0.098(3)	0.006(3)	0.078(3)	−0.017(2)
C(9)	4e	0.2887(4)	−0.0822(3)	−0.4549(4)	0.184(5)	0.098(3)	0.082(2)	0.016(3)	0.050(3)	−0.017(2)
C(10)	4e	0.2620(2)	−0.0407(2)	−0.3423(3)	0.127(3)	0.078(2)	0.063(2)	0.004(2)	0.028(2)	−0.007(2)
C(11)	4e	0.3746(2)	−0.0859(2)	−0.1553(4)	0.081(2)	0.046(1)	0.089(2)	−0.001(1)	0.042(2)	−0.002(1)
C(12)	4e	0.4208(2)	−0.0840(2)	−0.0001(4)	0.054(2)	0.041(1)	0.116(2)	−0.001(1)	0.032(2)	0.011(2)
C(13)	4e	0.0443(2)	0.0494(2)	−0.2338(3)	0.074(2)	0.070(2)	0.078(2)	0.003(2)	0.017(2)	0.008(2)
C(14)	4e	−0.0387(2)	0.0326(2)	−0.3113(4)	0.071(2)	0.090(2)	0.085(2)	0.012(2)	0.007(2)	0.010(2)
C(15)	4e	−0.0752(2)	−0.0406(2)	−0.2820(4)	0.056(2)	0.095(2)	0.090(2)	0.005(2)	0.009(2)	−0.008(2)
C(16)	4e	−0.0303(2)	−0.0978(2)	−0.1743(3)	0.057(2)	0.071(2)	0.072(2)	−0.001(1)	0.020(1)	−0.015(1)
C(17)	4e	−0.0653(2)	−0.1760(2)	−0.1351(4)	0.067(2)	0.081(2)	0.107(3)	−0.018(2)	0.026(2)	−0.016(2)
C(18)	4e	−0.0205(2)	−0.2289(2)	−0.0310(4)	0.084(2)	0.070(2)	0.101(2)	−0.024(2)	0.036(2)	−0.009(2)
C(19)	4e	0.0656(2)	−0.2105(2)	0.0443(3)	0.080(2)	0.056(1)	0.066(2)	−0.009(1)	0.030(2)	−0.002(1)
C(20)	4e	0.1153(2)	−0.2653(2)	0.1521(3)	0.109(3)	0.063(2)	0.080(2)	−0.016(2)	0.035(2)	0.007(2)
C(21)	4e	0.1968(2)	−0.2453(2)	0.2167(3)	0.108(3)	0.069(2)	0.068(2)	0.003(2)	0.021(2)	0.015(2)
C(22)	4e	0.2302(2)	−0.1696(2)	0.1722(3)	0.080(2)	0.069(2)	0.058(2)	−0.000(2)	0.014(1)	0.007(1)
C(23)	4e	0.1037(2)	−0.1347(2)	0.0092(3)	0.063(2)	0.054(1)	0.051(1)	−0.005(1)	0.023(1)	−0.011(1)
C(24)	4e	0.0541(2)	−0.0761(2)	−0.1008(3)	0.060(2)	0.053(1)	0.054(1)	−0.001(1)	0.022(1)	−0.009(1)

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