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Structures of Triaqua[1,2-benzenedicarboxylato(2-)](1,10-phenanthroline)nickel(II) Monohydrate (I) and Triaqua[1,2-benzenedicarboxylato(2-)](2,2'-dipyridylamine)-nickel(II) Dihydrate (II)*

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(Received 28 March 1989; accepted 27 June 1989)

Abstract. (I): $[\text{Ni}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}$, $M_r = 475.10$, monoclinic, $P2_1/n$, $a = 20.157$ (7), $b = 13.632$ (5), $c = 7.529$ (2) Å, $\beta = 97.36$ (4)°, $V = 2052$ (1) Å³, $Z = 4$, $D_x = 1.54$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.996$ mm⁻¹, $F(000) = 984$, $R = 0.062$ for 3362 observed reflexions. (II): $[\text{Ni}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{10}\text{H}_9\text{N}_3)(\text{H}_2\text{O})_3]\cdot 2\text{H}_2\text{O}$, $M_r = 484.11$, monoclinic, $P2_1/n$, $a = 8.026$ (2), $b = 19.901$ (4), $c = 13.235$ (1) Å, $\beta = 99.61$ (1)°, $V = 2084.3$ (7) Å³, $Z = 4$, $D_x = 1.54$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.986$ mm⁻¹, $F(000) = 1008$, $R = 0.037$ for 3902 observed reflexions. In both compounds the discrete complex molecules are found to be of very similar shape. The Ni atoms are in a distorted octahedral environment with three molecules of coordinated water in the meridional position. The 1,2-benzenedicarboxylate(2-) anions act as monodentate ligands, while the rest of the carboxylate O atoms, together with water of crystallization form a system of hydrogen bonding. On the basis of photographic data $[\text{Co}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}$ is isostructural with the analogous Ni complex.

Introduction. Crystal structures of about fifteen transition metal-phthalate [1,2-benzenedicarboxylate(2-)] complexes have been determined so far. Most of them are Cu^{II} complexes (for a complete list of references see Krstanović, Karanović & Stojaković, 1985), where polydentate, bridging or combined bridging-chelating behaviour of phthalate ion is predominant. No structures of Co^{II} complexes and only two structures of Ni^{II} complexes (Kozlova, Agre, Trunov, Makarevich & Barkhanova, 1982; Endres, 1984) are known. This is not sufficient for a more detailed study of the factors that determine the coordination mode of the phthalate ion.

We have recently prepared some ternary Co^{II} and Ni^{II} complexes with 1,10-phenanthroline (phen), 2,2'-bipyridine (bipy) and 2,2'-dipyridylamine (dipya) as a second ligand. This paper reports the structures of two of them. From the preliminary X-ray data it was found that $[\text{Co}(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}$ is isostructural with (I).

Experimental. Blue (Ni) and pink (Co) monocrystals were prepared by slow evaporation from dilute aqueous solutions containing equimolar amounts of $M(\text{NO}_3)_2$ ($M = \text{Ni}, \text{Co}$), diamine and sodium

* Partially presented at Tenth European Crystallographic Meeting, Wrocław, Poland, 5–9 August 1986.

phthalate. Preliminary crystal data were from oscillation and Weissenberg photographs (Ni-filtered Cu K α radiation). Accurate cell parameters for the Ni complexes were from least-squares refinement of 18 reflexions with $10.10 < \theta < 20.08^\circ$ (I), and 25 reflexions with $14.40 < \theta < 18.02^\circ$ (II). Complex (I): crystal $0.12 \times 0.08 \times 0.07$ mm, Philips PW 1100 diffractometer, $\omega/2\theta$ scan, $(2\theta)_{\min} = 4$, $(2\theta)_{\max} = 60^\circ$ ($0 \leq h \leq 28$, $0 \leq k \leq 19$, $-10 \leq l \leq 10$), two standard reflexions, no significant variations, 6787 collected reflexions, 3362 observed with $I \geq 3\sigma(I)$. Final agreement parameters: $R = 0.062$, $wR = 0.066$, $w = 1/\sigma^2(F_o)$, for 280 independent parameters, $(\Delta/\sigma)_{\max} = 0.15$, $\Delta\rho_{\max} = 0.632 \text{ e } \text{\AA}^{-3}$. Complex (II): crystal $0.071 \times 0.071 \times 0.092$ mm, Enraf-Nonius CAD-4 diffractometer, $\theta/2\theta$ scan, $(2\theta)_{\min} = 4$, $(2\theta)_{\max} = 58^\circ$, ($-10 \leq h \leq 10$, $0 \leq k \leq 26$, $0 \leq l \leq 17$), two standard reflexions, no variations, 5903 collected reflexions, 3902 observed with $I \geq 3\sigma(I)$. Final agreement parameters: $R = 0.037$, $wR = 0.053$, $w = 1/\sigma^2(F_o)$, for 280 independent parameters, $(\Delta/\sigma)_{\max} = 0.10$, $\Delta\rho_{\max} = 0.757 \text{ e } \text{\AA}^{-3}$.

Data corrected for Lp, not for absorption. Both structures solved by Patterson method. Full-matrix least-squares refinement on F with anisotropic thermal parameters for all non-H atoms. Most water H atoms from ΔF maps, remaining H atoms placed at calculated positions. All H atoms introduced in the final structure-factor calculation at fixed positions with fixed thermal parameters ($B = 4.5 \text{ \AA}^2$). Scattering factors for neutral atoms and anomalous dispersion for Ni from *International Tables for X-ray Crystallography* (1974). The main programs used on the CDC-3600 computer were *FORDAP*, Zalkin's (1965) Fourier program, *NUCLS*, a modification by Ibers & Doedens of the full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962) and *GEOM* (Mallinson & Muir, 1985) for molecular geometry calculations.

Photographic data for Co complex, C₂₀H₂₀N₂-CoO₈, $M_r = 475.32$, are: monoclinic, $P2_1/n$, $a = 20.36$ (3), $b = 13.72$ (2), $c = 7.54$ (4) \AA , $\beta = 98$ (1) $^\circ$.

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Packing of the molecules and atomic numbering are shown in Fig. 1 (I) and Fig. 2 (II). Selected bond distances and angles are listed in Table 2.

The crystal structures consist of discrete complex molecules that are mutually very similar in shape and geometry, and of water of hydration.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, hydrogen bonding and least-square planes, and a fuller list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52380 (89 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional ($\times 10^4$) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
(I)				
Ni	388 (1)	1937 (1)	7829 (1)	2.18 (2)
O(1)	1098 (2)	856 (2)	8303 (4)	2.4 (1)
O(2)	1540 (2)	1168 (2)	11107 (5)	3.4 (1)
O(3)	1183 (2)	-869 (2)	5474 (5)	3.0 (1)
O(4)	389 (2)	-1013 (2)	7265 (4)	2.9 (1)
OW(1)	543 (2)	1899 (2)	5177 (4)	2.7 (1)
OW(2)	318 (2)	2049 (2)	10533 (4)	3.1 (1)
OW(3)	-288 (2)	789 (2)	7496 (4)	2.9 (1)
OW(4)	1688 (2)	926 (3)	4685 (5)	3.8 (1)
N(1)	-334 (2)	3023 (3)	7301 (5)	2.5 (1)
N(2)	1000 (2)	3172 (3)	8136 (5)	2.3 (1)
C(1)	-990 (2)	2935 (4)	6910 (8)	3.6 (2)
C(2)	-1416 (3)	3758 (5)	6583 (9)	5.0 (2)
C(3)	-1144 (3)	4674 (4)	6687 (9)	5.2 (2)
C(4)	-452 (3)	4786 (4)	7150 (8)	4.6 (2)
C(5)	-70 (2)	3933 (4)	7446 (6)	3.0 (2)
C(6)	650 (2)	4021 (4)	7903 (6)	2.5 (2)
C(7)	947 (3)	4943 (4)	8108 (8)	3.6 (2)
C(8)	1647 (3)	4967 (4)	8557 (8)	3.8 (2)
C(9)	1999 (3)	4118 (4)	8764 (8)	3.5 (2)
C(10)	1654 (2)	3220 (4)	8534 (7)	3.1 (2)
C(11)	540 (3)	5799 (4)	7856 (10)	5.7 (3)
C(12)	-122 (4)	5736 (4)	7391 (10)	6.0 (3)
C(13)	1430 (2)	615 (3)	9758 (6)	2.5 (2)
C(14)	1717 (3)	-408 (4)	9891 (8)	2.2 (2)
C(15)	2192 (3)	-651 (5)	11308 (8)	3.3 (2)
C(16)	2476 (3)	-1575 (5)	11418 (9)	4.3 (2)
C(17)	2297 (3)	-2267 (5)	10106 (9)	3.9 (2)
C(18)	1823 (3)	-2036 (4)	8686 (9)	3.7 (2)
C(19)	1527 (3)	-1109 (4)	8557 (8)	2.8 (2)
C(20)	988 (2)	-959 (3)	6985 (6)	2.1 (1)
(II)				
Ni	8217 (1)	277 (1)	2033 (1)	1.99 (1)
O(1)	8115 (2)	1067 (1)	991 (2)	2.50 (6)
O(2)	10819 (3)	1365 (1)	1108 (2)	2.96 (7)
O(3)	4852 (3)	1146 (1)	-814 (2)	3.13 (8)
O(4)	6875 (3)	437 (1)	-1099 (2)	2.77 (7)
OW(1)	10846 (3)	218 (1)	2058 (2)	2.74 (7)
OW(2)	5600 (3)	424 (1)	2021 (2)	2.57 (7)
OW(3)	7695 (3)	-293 (1)	711 (2)	2.45 (6)
OW(4)	4259 (3)	1584 (1)	1075 (2)	4.19 (9)
OW(5)	3496 (3)	274 (2)	3729 (2)	5.27 (11)
N(1)	8214 (3)	-579 (1)	2911 (2)	2.65 (7)
N(2)	8564 (3)	837 (1)	3365 (2)	2.26 (7)
N(3)	7383 (3)	-4 (1)	4309 (2)	3.05 (8)
C(1)	8523 (4)	-1181 (2)	2494 (3)	3.04 (10)
C(2)	8177 (5)	-1786 (2)	2890 (3)	3.78 (12)
C(3)	7461 (5)	-1788 (2)	3776 (3)	4.53 (13)
C(4)	7162 (5)	-1195 (2)	4226 (3)	4.12 (11)
C(5)	7609 (4)	-585 (2)	3786 (2)	2.76 (9)
C(6)	8009 (4)	639 (2)	4215 (2)	2.46 (9)
C(7)	7996 (4)	1070 (2)	5053 (2)	3.27 (11)
C(8)	8576 (4)	1711 (2)	5008 (3)	3.18 (11)
C(9)	9170 (5)	1929 (2)	4134 (3)	3.36 (12)
C(10)	9154 (4)	1478 (2)	3343 (2)	2.78 (10)
C(11)	9304 (4)	1368 (1)	662 (2)	2.09 (8)
C(12)	8857 (4)	1765 (1)	-307 (2)	2.43 (8)
C(13)	9852 (4)	2319 (2)	-458 (3)	3.40 (11)
C(14)	9423 (5)	2723 (2)	-1317 (3)	4.70 (13)
C(15)	8040 (5)	2573 (2)	-2033 (3)	5.76 (14)
C(16)	7058 (4)	2013 (3)	-1907 (3)	4.01 (12)
C(17)	7456 (4)	1612 (2)	-1039 (2)	2.67 (9)
C(18)	6312 (4)	1019 (2)	-960 (2)	2.19 (8)

The complex (II) contains more water than (I) and this can be attributed to non-planarity of dipya and to the presence of the amine N atom as an H donor. The similar unit-cell contents result in the close cell volumes, but the molecules are differently oriented relative to crystallographic axes and symmetry elements. In spite of this difference, the molecular packing, governed by van der Waals contacts and the system of hydrogen bonding, are similar. In both

Table 2. Selected bond distances (Å) and angles (°) with *e.s.d.*'s in parentheses

(I)		(II)	
<i>(a) In the coordination polyhedron</i>			
Ni—O(1)	2.053 (3)	Ni—O(1)	2.083 (2)
Ni—OW(1)	2.061 (3)	Ni—OW(1)	2.108 (2)
Ni—OW(2)	2.065 (3)	Ni—OW(2)	2.119 (2)
Ni—OW(3)	2.070 (3)	Ni—OW(3)	2.068 (3)
Ni—N(1)	2.080 (4)	Ni—N(1)	2.063 (3)
Ni—N(2)	2.082 (4)	Ni—N(2)	2.065 (2)
O(1)—Ni—OW(1)	87.8 (1)	O(1)—Ni—OW(1)	88.94 (8)
O(1)—Ni—OW(2)	90.9 (1)	O(1)—Ni—OW(2)	87.80 (8)
O(1)—Ni—OW(3)	84.9 (1)	O(1)—Ni—OW(3)	82.79 (8)
O(1)—Ni—N(1)	179.0 (1)	O(1)—Ni—N(1)	172.92 (9)
O(1)—Ni—N(2)	99.9 (1)	O(1)—Ni—N(2)	98.82 (9)
OW(1)—Ni—OW(2)	174.6 (1)	OW(1)—Ni—OW(2)	175.23 (9)
OW(1)—Ni—OW(3)	92.4 (1)	OW(1)—Ni—OW(3)	92.52 (9)
OW(1)—Ni—N(1)	91.2 (1)	OW(1)—Ni—N(1)	92.30 (9)
OW(1)—Ni—N(2)	88.1 (1)	OW(1)—Ni—N(2)	91.39 (9)
OW(2)—Ni—OW(3)	92.7 (1)	OW(2)—Ni—OW(3)	90.53 (9)
OW(2)—Ni—N(1)	90.0 (1)	OW(2)—Ni—N(1)	91.36 (9)
OW(2)—Ni—N(2)	86.9 (1)	OW(2)—Ni—N(2)	85.63 (9)
OW(3)—Ni—N(1)	94.7 (1)	OW(3)—Ni—N(1)	90.21 (9)
OW(3)—Ni—N(2)	175.2 (1)	OW(3)—Ni—N(2)	175.98 (9)
N(1)—Ni—N(2)	80.6 (1)	N(1)—Ni—N(2)	88.7 (1)
<i>(b) In the carboxylate groups</i>			
O(1)—C(13)	1.252 (5)	O(1)—C(11)	1.264 (4)
O(2)—C(13)	1.261 (6)	O(2)—C(11)	1.260 (3)
O(3)—C(20)	1.257 (6)	O(3)—C(18)	1.244 (4)
O(4)—C(20)	1.254 (6)	O(4)—C(18)	1.268 (6)
O(1)—C(13)—O(2)	124.6 (4)	O(1)—C(11)—O(2)	124.2 (3)
O(3)—C(20)—O(4)	125.3 (4)	O(3)—C(18)—O(4)	125.2 (3)

structures hydrogen bonds connect molecules related by a center of symmetry as well as molecules that are translated by one unit along the *z* (I) or the *x* axis (II), thereby forming stacks that run parallel to these axes. An intramolecular hydrogen bond [OW(2)⋯O(2)] is found in both complexes [(I): 2.725 (5) Å; (II): 2.605 (3) Å] and appears to be typical of the phthalato-nickel complexes (see below).

The Ni atoms have a distorted octahedral environment with three H₂O molecules coordinated in meridional positions. The Ni—ligand bond lengths differ less in (I) than in (II) (Table 2). No dependence of the bond lengths on donor-atom type is observed.

In the diamine ligands bond lengths and bond angles are as expected, and are in accordance with those found for the free ligands (phen: Nishigaki, Yoshioka & Nakatsu, 1978; dipya: Johnson & Jacobson, 1973). In (II), the angle between the two pyridine rings is 18.1° which is smaller than that in the free ligand (23°; Johnson & Jacobson, 1973), but larger than found in some Cu complexes (average value for three studied compounds being 12.6°; Ray, Hulett, Sheahan & Hathaway, 1981). The angle between the basal plane [Ni, N(1), N(2), O(1), OW(3)] and the plane of the phen ligand is only 1.4 (2)° in (I).

The phthalate ions (pht) are monodentately coordinated through the O(1) atom. The remaining carboxylate O atoms participate in the formation of two [O(2) and O(3)] or even three [O(4)] hydrogen bonds. Both carboxylate groups have their plane tilted to the same side of the benzene ring plane

[uncoordinated: 75.6 (2)° in (I), 70.4 (1)° in (II); coordinated: 11.7 (2)° in (I), 27.0 (1)° in (II)], which is quite usual for monodentate or bridging phthalate coordination (Poleti, 1988). It should be pointed out that all four C—O bond lengths in a given pht ion are very similar [averages: 1.256 (6) Å in (I), 1.259 (4) Å in (II)], which is rather surprising in the case of coordinated COO⁻ groups.

If the two compounds described in the present work and the mentioned Co complex, isostructural

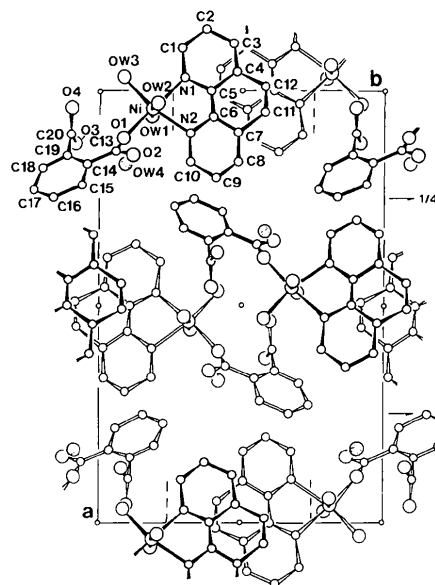


Fig. 1. Projection of the structure (I) on the *ab* plane. H atoms are omitted for clarity.

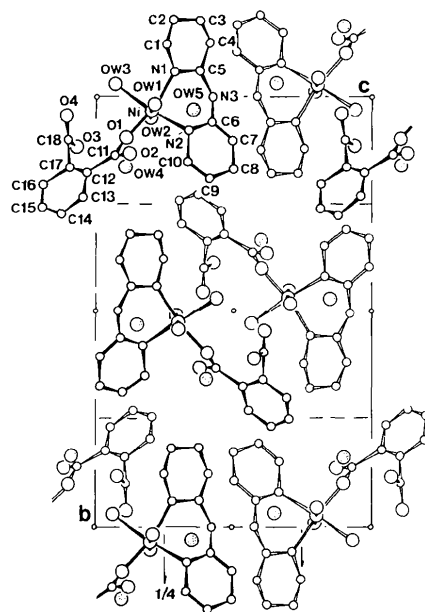


Fig. 2. Projection of the structure (II) on the *bc* plane. H atoms are omitted for clarity.

with (I), are taken into account a total of five Co^{II} and Ni^{II} phthalate complexes are known so far. In four of these complexes only monodentate-type coordination is encountered; in all the cases the intramolecular hydrogen bond is also found (as noted above). As pointed out earlier (Poleti, Stojaković, Prelesnik & Herak, 1988), the existence of such hydrogen bonding should markedly diminish the ability of the pht ion to act as a chelate or as a bridging ligand. Further work on this subject is in progress.

This work was supported by the Research Fund of SR Serbia.

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Acta Cryst. (1990). **C46**, 402–404

Structure of Hexa- μ -chloro- μ_4 -oxo-tetrakis[1,2-dimethylimidazolecopper(II)]

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(Received 2 December 1988; accepted 6 July 1989)

Abstract. [Cu₄Cl₆(O)(C₅H₈N₂)₄].0.25H₂O, $M_r = 871.90$, monoclinic, $P2_1/c$, $a = 22.582(5)$, $b = 11.829(2)$, $c = 16.114(4)$ Å, $\beta = 128.46(2)^\circ$, $V = 3370.5$ Å³, $Z = 4$, $D_m = 1.744$, $D_x = 1.718$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 30.15$ cm⁻¹, $F(000) = 1746$, $T = 293$ K, final $wR = 0.055$ for 1319 significant reflections. The compound contains a central O atom coordinated tetrahedrally to four Cu atoms, with the metal atoms being bridged in pairs by six Cl atoms. A 1,2-dimethylimidazole (dmim) ligand completes a trigonal-bipyramidal coordination about each of the four Cu atoms. Disordered water molecules which are not bonded to the copper compound are also present in the unit cell.

Introduction. In order to study magnetic superexchange interactions we decided to synthesize compounds with chain structures by established procedures (Van Ooijen, 1979; Van Ooijen, Reedijk

& Spek, 1979). During one of these attempts a tetrameric complex, analysing as [Cu₄Cl₆OL₄].0.25H₂O, where $L = 1,2$ -dimethylimidazole, was obtained instead of a chain compound. The structure determination of this complex was undertaken because of the increasing interest in tetrameric compounds and because such compounds have not previously been reported for imidazole ligands.

Experimental. Crystals were prepared by mixing ethanolic and methanolic solutions of the hydrated copper(II) chloride and ligand in a ratio less than 1:2. An excess of triethyl orthoformate was then added to the solution as a dehydrating agent. Plate-shaped single crystals so obtained were washed with ethanol and diethyl ether and dried at room temperature.

Crystal dimensions approximately $0.3 \times 0.2 \times 0.07$ mm. Density measured by flotation method. Intensities were measured on an Enraf-Nonius CAD-4 four-circle diffractometer (graphite-monochromated Mo $K\alpha$ X-rays). Accurate unit-cell parameters and the orientation matrix determined by

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