Monatshefte für Chemie 131, 711–719 (2000)

**Monatshefte für Chemie**
**Chemical Monthly**

Printed in Austria

# Preparation, Structure, and Thermal Decomposition of Co(II) Complexes with 2,3- and 2,5-Dichlorobenzoic Acid and Imidazole

**Wiesława Wołodkiewicz**[1,*] and **Tadeusz Głowiak**[2]

[1] Faculty of Chemistry, Maria Curie-Skłodowska University, PL-20031 Lublin, Poland
[2] Faculty of Chemistry, University of Wrocław, PL-50383 Wrocław, Poland

**Summary.** The reaction products of Co(II)-2,3- and -2,5-dichlorobenzoate with imidazole (**1**, **2**; $CoL_2 \cdot \cdot 2imd \cdot 2H_2O$, $L = C_7H_3Cl_2O_2^-$, $imd$ = imidazole) were characterized by their spectroscopic and thermochemical properties. The compounds crystallize in the monoclonic system with space group = $P2_1/c$, $a = 13.848(3)$, $b = 12.841(3)$ Å, $c = 7.064(2)$ Å, $\beta = 98.12°$, $V = 1243.5(4)$ Å$^3$, $Z = 2$ for **1** and space group = $P2_1/n$, $a = 13.293(3)$, $b = 6.964(2)$, $c = 13.800(3)$ Å, $\beta = 108.92(3)°$, $V = 1208.6(4)$ Å$^3$, $Z = 2$ for **2**. The complexes lose their crystal water in one step at 333 K and subsequently decompose to CoO with intermediate formation of $Co_3O_4$.

**Keywords.** Cobalt; Dichlorobenzoic acid; Complex; X-Ray analysis; Thermal analysis.

## Introduction

A general survey of the literature on complexes of Co(II) with nitrogen donors reveals that a considerable amount of work has been devoted to the preparation and characterization of complexes of Co(II) carboxylates with nitrogen donors. Some adducts of Co(II) benzoate [1, 2] and nitrobenzoate [2] with pyridine and 2-methylpyridine have been described. Carboxylates of divalent metals have been noted to give complexes of three different stoichiometries (1:1, 1:2, and 1:4) with various donor ligands [3–7]. In some cases, however, metal carboxylates afford adducts of two different stoichiometries with the same ligand under different experimental conditions [8, 9].

The complexes of bridged *bis*(imidazole) and *bis*(benzimidazole) ligands of various bridge types with transition metals have been examined as potential models of structure and mobility of biological metal binding sites in metalloproteins like haemerythrin, hemocyanin, tyrosinase, or azurin [10–16]. One of these N-donors ligands is imidazole. The present investigation is a continuation of our study on the structure and physico-chemical properties of metal complexes with dichlorobenzoic acids and N-donor ligands.

---

[*] Corresponding author

## Results and Discussion

The addition complexes of Co(II)-2,3-dichlorobenzoate and Co(II)-2,5-dichlorobenzoate with imidazole (**1**, **2**) with the general formula $CoL_2 \cdot 2imd \cdot 2H_2O$ ($L = C_7H_3Cl_2O_2^-$, *imd* = imidazole) were prepared as pink crystalline solids.

### *Infrared spectra*

The IR spectroscopic data of the compounds under investigation are summarized in Table 1. The IR spectra of **1** and **2** shows three strong bands at 1328, 1068, and 944 cm$^{-1}$ due to valence C-N vibrations of imidazole. They are shifted towards higher energy compared with the corresponding C-N vibrations of the uncoordinated ligand. The $\nu$(Co-N) band is observed at 696 cm$^{-1}$ for **1** and 670 cm$^{-1}$ for **2** These bands are observed neither in the IR spectra of free imidazole nor in Co(II)-2,3-dichlorobenzoate [25] and Co(II)-2,5-dichlorobenzoate [26]. In the IR spectrum of the free acid there is a single strong absorption band at 1688 cm$^{-1}$ for **1** and at 1704 cm$^{-1}$ for **2** (COOH group). In the IR spectrum of the title compounds, this absorption band disappears, and a split band of asymmetrical (COO$^-$) vibrations ($\nu_{as}$) at 1544 cm$^{-1}$ as well as band of symmetrical vibrations ($\nu_s$) at 1392 cm$^{-1}$ for **1** and 1548 cm$^{-1}$ and 1408 cm$^{-1}$ for **2**, respectively appear. The broad absorption band of $\nu$(OH) at 3420 cm$^{-1}$ and a narrow band of $\delta(H_2O)$ at 1580 cm$^{-1}$ for **1** and 3440 cm$^{-1}$ and 1568 cm$^{-1}$ for **2** confirm the presence of crystal water. The split band of $\nu_{as}$ (COO$^-$) and $\nu_s$ (COO$^-$) the as well as the separation ($\Delta\nu$) of $\nu_{as}$(COO$^-$) and $\nu_s$(COO$^-$) of 152 cm$^{-1}$ for **1** and 140 cm$^{-1}$ for **2** suggest that the carboxylate groups act as monodentate ligands [17–19]. This was confirm by X-ray analysis. The C-C

**Table 1.** Frequencies for characteristic absorption bands in the IR spectra of **1** and **2** and their precursors (cm$^{-1}$)

| 2,3-Co$L_2$.3H$_2$O | 2,5-Co$L_2 \cdot 5H_2O$ | Imidazole | **1** | **2** | Assignment |
|---|---|---|---|---|---|
| 3424 | 3400 | | 3420 | 3440 | $\nu$ (OH) |
| | | 3200–2624 | 3240 | 3256 | $\nu$ (N–H) |
| | | 1668 | | | $\nu$ (C=N) |
| 1568 | 1584 | | 1588 | 1568 | $\delta$ (H$_2$O) |
| 1536 | 1552 | | 1544 | 1548 | $\nu_{as}$ (OCO$^-$) |
| | | | | | $\nu$(C–C) |
| 1404 | 1412 | | 1392 | 1408 | $\nu$s (OCO$^-$) |
| | | 1326 | 1328 | 1328 | $\nu$ (C–N) |
| 1264 | 1280 | 1272 | | | $\nu$ (C–C) |
| 1152 | 1096 | | 1158 | 1156 | $\beta$ (C–H) |
| | | 1056 | 1068 | 1068 | $\nu$(C–N) |
| 1048 | 1048 | 1048 | 1048 | 1048 | $\nu$(C–C) |
| | | 936, 840 | 944, 840 | 944, 840 | $\nu$(C–N) |
| | | 756 | 772 | 760 | $\nu$(C–H) |
| 768 | 816 | | 736 | 816 | $\nu$(C–Cl) |
| | | | 696 | 670 | $\nu$(Co–N) |
| 584 | 608, 540 | 660 | 656 | 656 | $\nu$(C–H) |

and C-H vibrational bands of the Co(II) complexes change their frequencies only insignificantly compared to the corresponding bands of imidazole and the free acids, thus indicating that the Co(II) ion in the complexes does not deform the rings of phenyl and imidazole to a noticeable extent, an assumption which was confirmed by the crystal structure determination.

### *Crystal structure*

The structures of title compounds and the numbering of atoms are shown in Figs. 1 and 2. Table 2 contains selected bonds lengths and bonds angles. The final atomic parameters are listed in Table 3. The carboxyl groups are bonded as a monodentate ligands with metal-oxygen bond lengths of 2.113(1) Å for **1** and 2.104(1) Å for **2** which is longer than the corresponding distances in Co(II) complexes with 3,4-dichlorobenzoic acid (2.050(2) and 2.066(2) Å, [20]) and 3,5-dichlorobenzoic (2.044(3) and 2.044(3) Å, [21]). The central ion is connected to two imidazole molecules and to two water molecules; its coordination number is therefore 6 which is typical for compounds of Co(II).

In the complexes described, the Co(II) ions occupy the inversion centre. Four oxygen atoms (two from the carboxylate groups and two from the water molecules) and two nitrogen atoms form a distored octahedron. The bond length from the metal to the oxygen of water are 2.151(2) Å for **1** and 2.167(1) Å for **2**; the Co-N bond lengths amount to 2.096(2) Å for **1** and 2.092(2) Å for **2**. Deformation of the coordination sphere is significant both with regard to the Co-ligand distance and the angles. The angles in the coordination octahedrons of the complexes are in the range of 92.12(7)-87.88(7)° for **1** and of 92.59(6)–87.41(6)° for **2**.

**1** and **2** are stabilized by hydrogen bonds of the O-H···O and the N-H···O types (Table 4). There are three hydrogen bonds. For **1** two of them (O(3)-H(2)···O(2): 2.805(2) Å, 149(2)°; N(2)-H(7)···O(2): 2.876(3) Å, 174(3)°) are intermolecular, the third (O(3)-H(1)···O(2):2.717(2) Å, 167.(3)°) is intramolecular. The same holds for complex **2** (O(3)-H(2)···O(2): 2.867(2) Å 152(3)° and

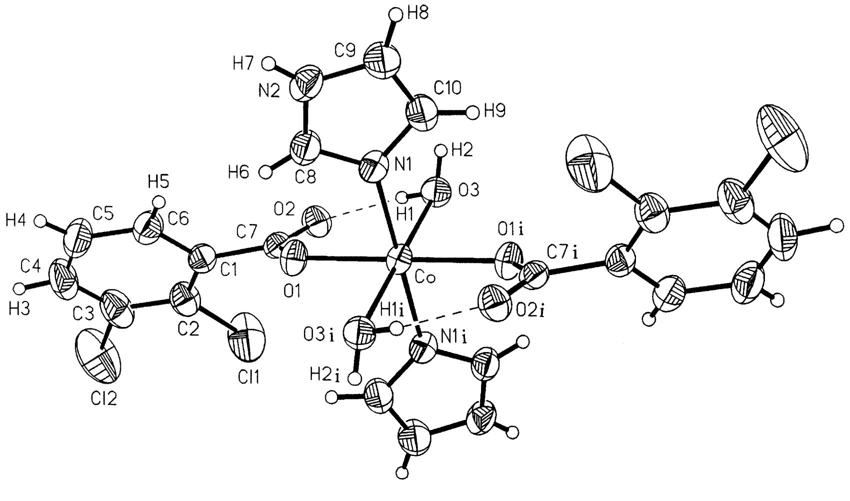

**Fig. 1.** Molecular structure of [Co($C_7H_3Cl_2O_2$)$_2$ · 2*imd* · 2$H_2O$] (**1**)

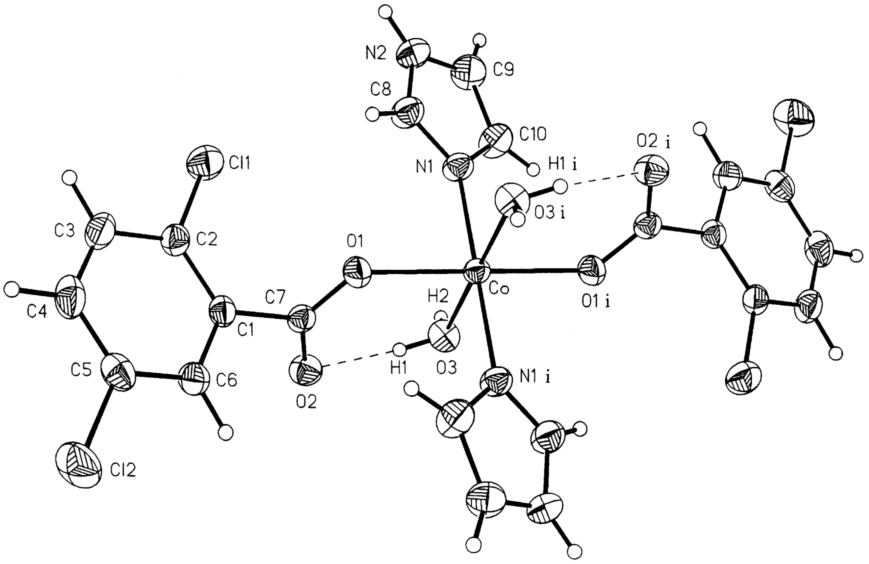

**Fig. 2.** Molecular structure of $[Co(C_7H_3Cl_2O_2)_2 \cdot 2imd \cdot 2H_2O]$ (**2**)

**Table 2.** Selected bond lengths (Å) and angles (°)

| | **1** | **2** |
|---|---|---|
| Co–O (1) | 2.113(1) | 2.103(1) |
| Co–O (3) | 2.151(2) | 2.167(2) |
| Co–N (1) | 2.096(2) | 2.092(2) |
| O(1)–C(7) | 1.249(2) | 1.249(2) |
| O(2)–C(7) | 1.260(3) | 1.258(2) |
| C(1)–C(7) | 1.512(3) | 1.514(2) |
| Cl(1)–C(2) | 1.728(3) | 1.735(2) |
| Cl(2)–C(3) | 1.732(3) | 1.737(2) |
| N(1)–Co–O(1) | 91.05(7) | 91.18(6) |
| N(1)–Co–O(1)$^{i}$ | 88.95(7) | 88.82(6) |
| N(1)–Co–O(3) | 92.12(7) | 92.59(6) |
| O(3)–Co–N(1)$^{i}$ | 87.88(7) | 87.41(6) |
| O(1)–Co–O (3) | 90.71(7) | 90.76(6) |
| O(3)–Co–O(1)$^{i}$ | 89.29(7) | 89.24(6) |
| O(1)–C(7)–O(2) | 125.8(2) | 125.9(2) |
| C(6)–C(1)–C(7) | 118.4(2) | 122.8(2) |
| C(2)–C(1)–C(7) | 122.5(2) | 118.7(2) |
| O(1)–C(7)–C(1) | 115.8(2) | 116.0(2) |

Symmetry code: i $(-x, -y, -z)$

N(2)-H(7)···O(2): 2.894(2) Å, 166(3)° intermolecular, O(3)-H(1)···O(2): 2.752(2) Å, 160(3)° intramolecular). Different length of hydrogen bonds and different bonding strengths can cause deformations of the octahedral surrounding of central ions [22].

**Table 3.** Atomic coordinates and equivalent isotropic displacement parameters for **1** and **2**; *U*(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor

| | *x* | *y* | *z* | *U*(eq) |
|---|---|---|---|---|
| **1** | | | | |
| Co | 0.0000 | 0.0000 | 0.0000 | 0.03021(14) |
| Cl (1) | −0.35685(5) | 0.03113(7) | 0.27058(17) | 0.0831(3) |
| Cl(2) | −0.52694(6) | −0.12361(10) | 0.30539(18) | 0.1066(4) |
| O(1) | −0.11244(11) | −0.08676(12) | 0.0992(2) | 0.0383(4) |
| O(2) | −0.13448(11) | −0.00272(12) | 0.3666(2) | 0.0400(4) |
| O(3) | 0.03809(14) | 0.06919(13) | 0.2778(2) | 0.0393(4) |
| N(1) | 0.09893(13) | −0.12259(14) | 0.0667(3) | 0.0373(4) |
| N(2) | 0.15636(16) | −0.28055(17) | 0.1098(3) | 0.0483(5) |
| C(1) | −0.24344(15) | −0.1399(2) | 0.2528(3) | 0.0349(5) |
| C(2) | −0.33533(16) | −0.1012(2) | 0.2706(3) | 0.0453(6) |
| C(3) | −0.41191(18) | −0.1706(3) | 0.2824(4) | 0.0572(7) |
| C(4) | −0.3970(2) | −0.2759(3) | 0.2786(4) | 0.0630(8) |
| C(5) | −0.3062(2) | −0.3137(2) | 0.2614(4) | 0.0592(7) |
| C(6) | −0.22984(19) | −0.2463(2) | 0.2474(4) | 0.0450(6) |
| C(7) | −0.15718(14) | −0.0698(2) | 0.2377(3) | 0.0316(4) |
| C(8) | 0.07733(18) | −0.2219(2) | 0.0709(4) | 0.0428(5) |
| C(9) | 0.2341920 | −0.2161(2) | 0.1320(5) | 0.0605(8) |
| C(10) | 0.19876(18) | −0.1197(2) | 0.1054(4) | 0.0534(7) |
| **2** | | | | |
| Co | 0.0000 | 0.0000 | 0.0000 | 0.02122(12) |
| Cl(1) | −0.04467(4) | 0.32430(9) | 0.29933(94) | 0.04254(16) |
| Cl(2) | −0.53252(4) | 0.30957(10) | 0.07896(5) | 0.05061(18) |
| O(1) | −0.09192(11) | 0.1239(2) | 0.08224(10) | 0.0299(3) |
| O(2) | −0.14948(11) | 0.3984(2) | −0.00074(10) | 0.0308(3) |
| O(3) | 0.01335(12) | 0.2755(2) | −0.06782(11) | 0.0313(3) |
| N(1) | 0.13967(12) | 0.0440(2) | 0.12293(12) | 0.0280(3) |
| N(2) | 0.24660(14) | 0.0704(3) | 0.28037(13) | 0.0366(4) |
| C(1) | −0.21897(14) | 0.2996(2) | 0.13006(13) | 0.0243(3) |
| C(2) | −0.18061(15) | 0.3133(2) | 0.23621(14) | 0.0275(4) |
| C(3) | −0.24879(19) | 0.3252(3) | 0.29373(15) | 0.0350(4) |
| C(4) | −0.35730(19) | 0.3223(3) | 0.24521(17) | 0.0372(4) |
| C(5) | −0.39587(15) | 0.3106(3) | 0.13994(16) | 0.0326(4) |
| C(6) | −0.32851(15) | 0.3013(3) | 0.08151(15) | 0.0297(4) |
| C(7) | −0.14698(13) | 0.2731(2) | 0.06564(13) | 0.0235(3) |
| C(8) | 0.14596(16) | 0.0503(3) | 0.21990(15) | 0.0335(4) |
| C(9) | 0.30930(16) | 0.0792(4) | 0.21940(18) | 0.0401(5) |
| C(10) | 0.24271(16) | 0.0624(3) | 0.12198(16) | 0.0363(4) |

In the structure of these complexes, the cobalt(II) cations are not located in the carboxylate group planes or in the planes of the phenyl or imidazole rings. The distance of the metal ion from the carboxylate group plane is 0.236(3) and 0.299(3) Å, respectively. The angles between the planes of the carboxylate groups and phenyl rings are 53.7 (1) and 62.2(1) ° for **1** and **2**. The chlorine atoms in positions

**Table 4.** Hydrogen bonds for compounds **1** and **2** (Å and °)

| *D*–H···*A*, | | *d*(*D*–H), | *d*(H···*A*,) | *d*(D···*A*), | *D*–H···*A* |
|---|---|---|---|---|---|
| | **1** | | | | |
| O(3)–H(1)···O(2) | | 0.82(3) | 1.91(3) | 2.717(2) | 167(3) |
| O(3)–H(2)···O(2) | $(-x, -y, -z+1)$ | 0.80(3) | 2.09(3) | 2.805(2) | 149(2) |
| N(2)–H(7)···O(2) | $(-x, y-1/2, -z+1/2)$ | 0.78(3) | 2.10(3) | 2.876(3) | 174(3) |
| | **2** | | | | |
| O(3)–H(1)···O(2) | | 0.85(3) | 1.94(3) | 2.752(2) | 160(3) |
| O(3)–H(2)···O(2) | $(-x, -y+1, -z)$ | 0.82(4) | 2.12(4) | 2.867(2) | 152(3) |
| N(2)–H(7)···O(2) | $(x+1/2, -y+1/2, z+1/2)$ | 0.86(4) | 2.05(4) | 2.894(2) | 166(3) |

2 and 3 for **1** and in positions 2 and 5 for **2** cause a significant twist of the carboxylate group from the phenyl ring plane and contribute to the distortion of the Co(II) octahedron [21].

## *Thermochemical properties*

**1** and **2** are stable in air at room temperature. With a heating rate of 10 K/min, the dihydrated complexes are stable up to 333 K and then lose two crystal water molecules, forming the anhydrous complexes which are stable up to 513 K (**1**) and 493 K (**2**), respectively. Next they decomposes to CoO at 1223 K with intermediate formation of $Co_3O_4$ (Fig. 3, Table 5).

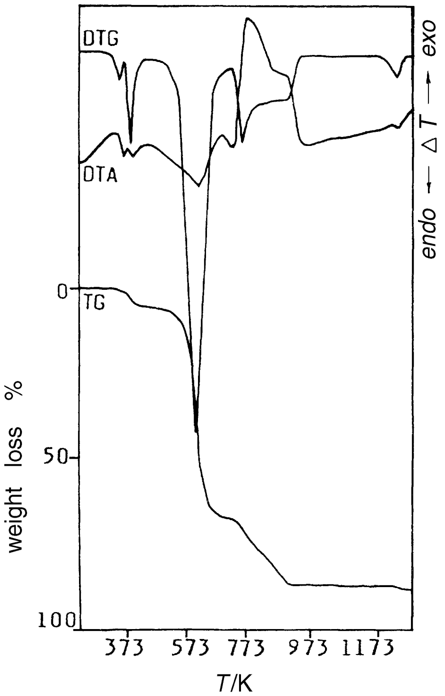

**Fig. 3.** TG, DTG, and DTA curves of $[Co(C_7H_3Cl_2O_2)_2 \cdot 2\mathit{imd} \cdot 2H_2O]$ (**1**)

**Table 5.** Thermal decomposition of **1** and **2**

| | Range of dehydration (K) | $T_1^a$ (K) | Loss of weight (%) | | Temp. of decomp. | Temp. of oxide formation | Residue (%) | |
|---|---|---|---|---|---|---|---|---|
| | | | Calcd. | Found | (K) | (K) | Calcd. | Found |
| **1** | 333–433 | 393 | 5.88 | 6.0 | 513 | 1186[b] | 13.11 | 13.2 |
| | | | | | | 1223[c] | 12.24 | 12.2 |
| **2** | 333–393 | 373 | 5.88 | 5.8 | 493 | 993[b] | 13.11 | 13.0 |
| | | | | | | 1223[c] | 12.24 | 12.5 |

[a]Temperature of endothermic peak on DTA; [b]$Co_3O_4$; [c]CoO

## Experimental

2,3- and 2,5-dichlorobenzoates of Co(II) were obtained by the modified method given in Refs. [25, 26]. 0.01 mol of basic Co(II) carbonate ($CoCO_3 \cdot Co(OH)_2 \cdot xH_2O$) was added to a hot solution of 2,3-dichlorobenzoic acid (0.02 mol in 0.2 $dm^3$ of water), and the suspension was heated while stirring for about 4 h. The same procedure was applied for 2,5-dichlorobenzoate of Co(II). After filtering the saturated solutions, the Co(II) complexes were subjected to slow crystallization. The precipitates formed were filtered, washed with water, and dried at 303 K to constant mass.

The title complexes were obtained by dissolving the above Co(II) complexes (0.01 mol in 0.2 $dm^3$ of a 0.1 *M* solution of imidazole in water (cobalt:imidazole ratio = 1:2). The solutions were subjected to slow crystallization, and pink crystals suitable for X-ray data collection were deposited after three weeks. The products were filtered off, washed with water, and dried at 303 K to constant mass.

The carbon, hydrogen, and nitrogen content in the complexes was determined by elemental analysis using a Perkin Elmer CAN 2000 analyzer. The chlorine content was measured by the Schöniger method. The Co(II) content was determined by AAS using an Atomic Absorption Spectrophotometer AAS-3 (Carl Zeiss-Jena). The crystal water content was determined from TG curves and by heating the complex isothermally to 433 K. The experimental results are concordant with the calculated data.

IR spectra were recorded over the range of 4000-400 $cm^{-1}$ using a FT-IR 1725X Perkin Elmer spectrophotometer. The thermal stability of the Co(II) complex was determined using a Paulik-Paulik-Erdey Q 1500 D derivatograph with a *Derill* converter; TG, DTG and DTA curves were recorded. Measurements were made at a sensitivity of 100 mg (TG). Samples (100 mg) were heated in platinum crucibles to 1273 K in static air at a heating rate of 10 $K/min^{-1}$. The solubility of the complexes in water was determined at 295 K. Saturated solution was prepared under isothermal conditions.

X-Ray measurements were made on a Kuma KM4 ℵ-axis for four-circle diffractometer with a graphite monochromator and Mo$K_\alpha$ radiation(**1**) and a Kuma KM4CCD ℵ-axis diffractometer with graphite-monochromator and Mo$K_\alpha$ radiation(2). The crystal was positioned at 65 mm from the KM4CCD camera. 612 frames were measured at 0.75° intervals with a counting time of 30 sec. The data were corrected for *Lorentz* and polarization effects. No absorption correction was applied. Data reduction and analysis were carried out with the Kuma Diffraction (Wroław) programs.

The crystallographic data and the refinement procedure details are given in Table 6. The structure were solved by direct methods with SHELXS97 [25] and refined by the full-matrix least-square method on all $F^2$ data using the SHELXL97 [26] program. Non-hydrogen atoms were refined with anisotropic thermal parameters; hydrogen atoms were included from the difference *Fourier* maps and refined with isotropic thermal parameters resulting in $R_1 = 0.0320$ for **1** and $R_1 = 0.0336$ for **2**. Fractional atomic coordinates and equivalent isotropic thermal parameters are presented in Table 3

**Table 6.** Crystal data and structure refinement for compounds **1** and **2**

| | **1** | **2** |
|---|---|---|
| Empirical formula | $C_{20}H_{18}O_6Cl_4N_4Co$ | $C_{20}H_{18}O_6Cl_4N_4Co$ |
| Temperature (K) | 293(2) | 293(2) |
| Molecular weight | 611.11 | 611.11 |
| Wavelength (Å) | 0.71073 | 0.71073 |
| Crystal system | monoclinic | monoclinic |
| Space group | $P2_1/c$ | $P2_1/n$ |
| $a$ (Å) | 13.848(3) | 13.293(3) |
| $b$ (Å) | 12.841(3) | 6.964(2) |
| $c$ (Å) | 7.064(2) | 13.800(3) |
| $\beta$(°) | 98.12(3) | 108.92(3) |
| $V$ (Å$^3$) | 1243.5(4) | 1208.6(4) |
| $Z$ | 2 | 2 |
| $F$ (000) | 618 | 618 |
| $D_m$ flotation (Mg/m$^3$) | 1.63 | 1.68 |
| $D_c$ (Mg/m$^3$) | 1.632 | 1.679 |
| Crystal size (mm) | 0.20×0.20 ×0.25 | 0.20×0.20 ×0.20 |
| Diffractometer | Kuma KM4 | Kuma KM4CCD |
| $2\theta$ limit (°) | 50.2 | 57.4 |
| Ranges $h$ | $-16$ to 16 | $-17$ to 17 |
| $k$ | $-15$ to 0 | $-9$ to 9 |
| $l$ | $-8$ to 8 | $-18$ to 18 |
| Reflections collected | 7392 | 7553 |
| Reflections unique | 2215 | 2805 |
| $R$ (int) | 0.0286 | 0.0220 |
| Data ($I > 2\sigma(I)$)/parameters | 1872/196 | 2689/196 |
| GOF on $F^2$ | 1.059 | 1.174 |
| $R_1$, $wR_2(I) > 2\sigma(I)$) | 0.0320, 0.0809 | 0.0336, 0.1041 |
| $R_1$, $wR_2$ indices (all data) | 0.0407, 0.0849 | 0.0350, 0.1063 |
| $\Delta\ \rho$/e°Å$^{-3}$ | 0.431 and $-0.381$ | 0.298 and $-0.514$ |

bond lengths and bond angles in Table 2. The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre under the deposition number CCDC 138839.

## References

[1] Drew J, Hursthouse MB, Welch AJ (1973) JCS Chem Comm 52

[2] Catterick J, Thorton P (1976) JCS Dalton 1634

[3] Kato M, Jonassen HB, Fanning JC (1964) Chem Rev **64**: 99

[4] Oldham C (1968) Prog Inorg Chem **10**: 223

[5] Kettle SFA, Pioli AJP (1968) J Chem Soc (A) 1243

[6] Agamber CA, Orrell KG (1969) J Chem Soc (A) 897

[7] Lever ABP, Ogden D (1967) J Chem Soc (A) 2041

[8] Kumar N (1974) J Indian Chem Soc **51**: 768

[9] Amasa S, Brown DH, Sharp DWA (1969) J Chem Soc (A) 2892

[10] Adams H, Neil AB, Crane JD, Fenton DE, Latour JM, Wiliams JM (1990) J Chem Soc Dalton Trans 1727

[11] Sorrell TN, Garrity ML (1991) Inorg Chem **30**: 210

[12] Chen S, Richardson J (1994) Inorg Chem **33**: 2376

[13] Tandon S, Thomson L, Bridson J, Dewan J (1992) Can J Chem **70**: 2771

[14] Sessler J, Hugdhl J, Lynch B (1991) Inorg Chem **30**: 334

[15] van Steenberger A, Bouwman E, de Graaf A, Driessen W, Reedijk J, Zanello P (1990) J Chem Soc Dalton Trans 3175

[16] Lockhart J, Clegg W, Hill M, Rushton D (1990) J Chem Dalton Trans 3541

[17] Gross A, Jonews R (1969) An Introduction to Practical Infrared Spectroscopy. Butterworhs, London

[18] Manhas B, Trikha A (1982) J Indian Chem **59**: 315

[19] Nakamoto K (1978) Infrared and Raman Spectra of Inorganic Compounds. Wiley, NY

[20] Wołodkiewicz W, Głowiak T (1998) Polish J Chem **72**: 1249

[21] Wołodkiewicz W, Brzyska W, Głowiak T (1996) Polish J Chem **70**: 409

[22] Wołodkiewicz W, Głowiak T, Brzyska W (1998) Monatsh Chem **129**: 111

[23] Wołodkiewicz W, Brzyska W (1998) Polish J Chem **72**: 2366

[24] Wołodkiewicz W, Brzyska W (1999) J Thermal Anal Calorimetry **55**: 639

[25] Sheldrick GM (1997) SHELXS-97. Program for solution of crystal structure (structures), University of Göttingen, Germany

[26] Sheldrick GM (1997) SHELXLP-97. Program for crystal structure refinement, University of Göttingen, Germany

*Received August 9, 1999. Accepted (revised) February 11, 2000*