Influence of Substituent Positions in Benzene Ring on the Properties of Co(II) 2,3- , 3,5- and 2,6-Dimethoxybenzoates

Wiesława Ferenc[1] *, Agnieszka Walków-Dziewulska[1], Jan Sarzyński[2]

[1] *Department of General and Coordination Chemistry, Maria Curie-Skłodowska University, Pl 20-031, Lublin, Poland*

[2] *Institute of Physics, Maria Curie-Skłodowska University, Pl 20-031, Lublin, Poland*

* *Corresponding author: e-mail: wetafer @hermes.umcs.lublin.pl*

Abstract

The physico-chemical properties and thermal stability in air of Co(II) 2,3-, 3,5- and 2,6-dimethoxybenzoates were compared and the influence of the position of –OCH_3 substituent on their thermal stability was investigated. The complexes are crystalline, hydrated and anhydrous salts with pink colour. The carboxylate ions are a monodentate, bidentate chelating or bridging and tridentate groups. The thermal stability of analysed Co(II) dimethoxybenzoates was studied in the temperature range of 293–1173 K. The positions of methoxy groups in benzene ring influence the thermal properties of studied complexes. Their different thermal properties are markedly connected with the various influences of inductive, mesomeric and steric effects of the OCH_3 substituent on the electron density in benzene ring. The magnetic susceptibilities of the complexes were measured over the range of 76–300 K and the magnetic moments were calculated. The results show that 2,3–, 3,5- and 2,6-dimethoxybenzoates of Co(II) are high-spin complexes with weak ligand fields.

Keywords: 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II), magnetic moments of Co(II) complexes, thermal stability, FTIR spectra of Co(II) compounds

1. Introduction

2,3-, 3,5- and 2,6-Dimethoxybenzoic acids are crystalline solids sparingly soluble in cold water.[1–5] The literature survey shows that, in principle, there is no information about the complexes of these acid anions with various cations. Papers exist only on their compounds with rare earth elements in solution and solid state.[6–8]

According to the literature review it follows that there are reports about resembling aspects of studies on the properties of similar complexes, as we presented here.[9–20] The compounds of Co(II), Ni(II) and Cu(II) with various carboxylic acid anions, different organic ligands and even with mixed ligands were synthesized in solid state and their characterizations were based on the elemental analysis, spectroscopic (UV, VIS, IR, and EPR) data, and magnetic investigations. As it is presented in the above mentioned papers the magnetic measurements and EPR spectra estimate the character of coordination geometry of central ions,[18] estimate the molecular structures of analysed complexes[12] and indicate the presence of the exchange coupling between magnetic centres in the compounds.[14] In

some papers we also discussed the comparison of some physico-chemical properties of 3d and 4f metal ion complexes with various carboxylic acid anion.[18, 19]

In the present paper we prepared 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II) as solids, to examine some of their properties and to compare them.

In our previous papers[20–23] their characterizations were discussed on the basis of elemental analysis, IR spectral data, thermogravimetric studies, X-ray diffraction and magnetic susceptibility measurements. The results revealed them to have various properties. Therefore, taking into the account the different positions of methoxy groups in benzene ring, we decided to compare them in order to study the influence of the substituent positions on the properties of the analysed compounds.

2. Experimental

2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II) were prepared by the addition of equivalent quantities of 0.1 M ammonium 2,3-, 3,5- and 2,6-dimethoxybenzoates

(pH ≈ 5) to hot 0.1 M aqueous solutions containing the nitrates (V) of Co(II) ions and crystallizing at 293 K. The solids were filtered off, washed several times with hot water and methanol to remove the ammonium ions and dried at 303 K.[20–23]

Elemental analysis was performed using a Perkin-Elmer CHN 2400 analyser. The contents of Co^{2+} metal ions were established gravimetrically, and by ASA method with the use of ASA 880 spectrophotometer (Varian).

The FTIR spectra of the complexes were recorded in the range of 4000–400 cm^{-1} using an FTIR 1725X Perkin – Elmer spectrometer. The samples for the FTIR spectroscopy were prepared as KBr discs. Some of the results are presented in Table 1 and Figure 1.

The X-ray diffraction patterns were taken on a HZG-4 (Carl Zeiss, Jena) diffractometer using Ni filtered CuK_α radiation. The measurements were made within the range of 2Θ = 4–80° by means of the Debye-Scherrer-Hull method. The relationships between I/I_0 and 2Θ for these complexes are presented in Figure 2.

The thermal stability and decomposition of the complexes were studied in air using a Q-1500 D derivatograph with a Derill converter, which simultaneously records TG, DTG and DTA curves. The measurements were made at a heating rate of 10 Kmin^{-1}. The 100 mg samples were heated in platinum crucibles in static air to 1173 K with a TG sensitivity of 100 mg (i.e., the whole scale of the balance was equal to 100 mg). The DTG and DTA sensitivities were regulated by the Derill computer program. The paper speed was 2.5 mm min^{-1} and Al_2O_3 was used as a standard. The decomposition products were calculated from the TG curve and verified by powder diffraction analysis.

Magnetic susceptibilities of analyzed polycrystalline samples were investigated at 76–303 K. The measurements were carried out using the Gouy method. Weight changes were obtained from Cahn RM-2 electrobalance. The calibrant employed was $Hg[Co(SCN)_4]$ for which the magnetic susceptibility was assumed to be $1.6441 0^{-5}$ cm^{3}g^{-1}. Correction for diamagnetism of the calibrant

atoms was calculated by the use of Pascal's constants.[24, 25] Magnetic moments were calculated from Eq.(1).

$$\mu = 2.83 \, (\chi_M \cdot T)^{1/2} \qquad (1)$$

The solubility of 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II) in water was measured at 293 K. Saturated solution of the obtained compounds were prepared under isothermal conditions. The contents of Co(II) were determined by using ASA 880 spectrophotometer (Varian). The values of solubility are presented in Table 1.

3. Results and Discussion

The complexes of the 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II) were obtained as polycrystalline pink solids with a metal to ligand ratio of 1:2 and the general formula $Co(C_9H_9O_4)_2 nH_2O$, where n=0 for 2,3-dimethoxybenzoate, n = 2 for 3,5-dimethoxybenzoate and n=4 for 2,6-dimethoxybenzoate.[20–23] The details connected with their identification by elemental and spectral analyses were extensively presented in our pervious papers.[20–23] Therefore, in this article only some selected results of the FTIR investigations are presented in Table 1.

There are two bands arising from asymmetric and symmetric vibrations of the COO$^-$ groups at 1629–1579 cm^{-1} and 1444–1392 cm^{-1}, respectively for analysed complexes.[26–31] The bands due to ν(M–O) appear in the range of 425–408 cm^{-1}. The magnitudes of the separation ΔνOCO$^-$ (where ΔνOCO$^-$ = v_{as}OCO$^-$ – v_sOCO$^-$), which characterize the type of metal ion-oxygen bond change from 220 cm^{-1} to 135 cm^{-1}. According to the spectroscopic criteria and especially with regard to Nakamoto,[28,31,32] the carboxylate groups show different modes of coordination. In 2,3- and 3,5-dimethoxybenzoates of Co(II) they may function as bidentate chelating and bidentate bridging or tridentate groups and in 2,6-dimethoxybenzoate only as monodentate.

Table 1: Frequencies of the OCO$^-$ absorption bands for analysed complexes of Co(II) and Na(I) and those of CO for non-coordinated 2,3-, 3,5- and 2,6-dimethoxybenzoic acids (cm^{-1}).

Complex L = $C_9H_9O_4^-$		ν C=O	v_{as}OCO	v_sOCO	ΔνOCO	νM–O	Solubility (mol dm^{-3})
2,3-	CoL$_2$	–	1629	1409	220	425	2.8 10^{-3}
			1579	1444	135		
	NaL	–	1604	1396	208		
	HL	1686	–	–	–		
3,5-	CoL$_2$ 2H$_2$O	–	1598	1401	197	408	1.5 10^{-3}
				1428	170		
	NaL	–	1580	1385	195		
	HL	1684	–	–	–		
2,6-	CoL$_2$ 4H$_2$O	–	1600	1392	208	424	2.8 10^{-2}
	NaL	–	1608	1408	200		
	HL	1712	–	–	–		
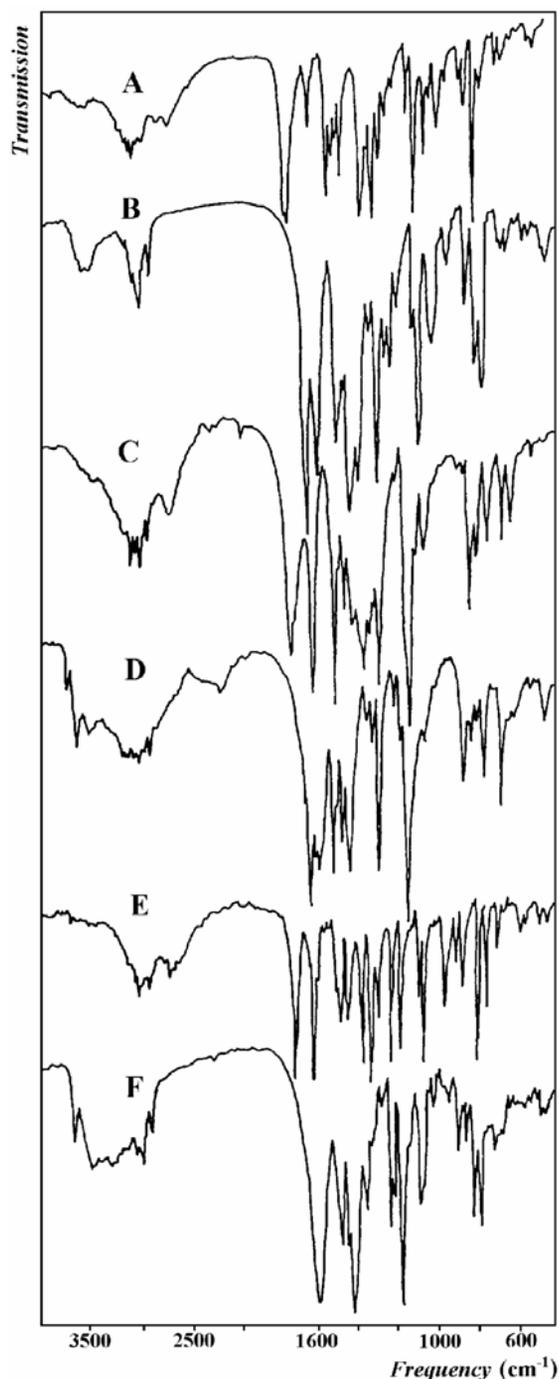
Figure 1. IR spectra of 2,3-dimethoxybenzoic acid (A), 2,3-dimethoxybenzoate of Co(II) (B), 2,6-dimethoxybenzoic acid (C), 2,6-dimethoxybenzoate of Co(II) (D), 3,5-dimethoxybenzoic acid (E), 3,5-dimethoxy-benzoate of Co(II) (F)

The X-ray diffraction patterns of analyzed complexes were recorded. The analysis of the diffractograms suggests them to be polycrystalline compounds with various degrees of crystallinity and different structures.[33] However their structures have not been determined as attempts to obtain single crystals failed.
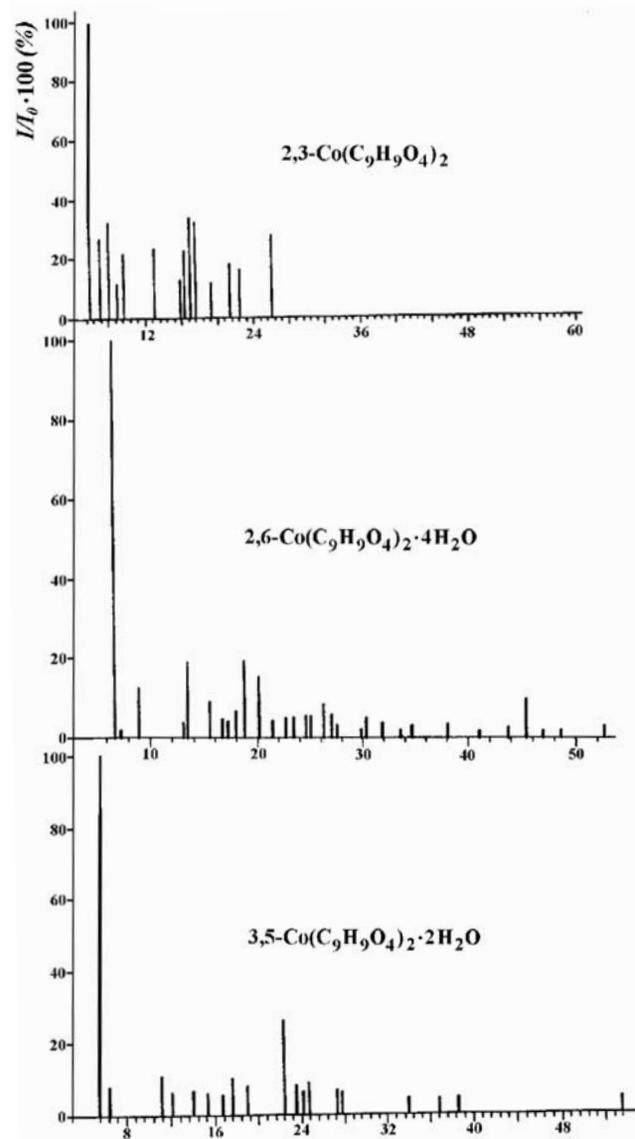
Figure 2. Dependence of I/I_0 vs 2Θ for Co(II) complexes.

The thermal stability of Co(II) dimethoxybenzoates was studied in air. The details concerning their thermal decomposition are presented in our previous articles.[8–12] In the present paper we report some selected results obtained from their thermal stability investigation (Table 2). The complexes are stable up to 331–376 K. The hydrates lose water molecules in one step and form the anhydrous compounds. The dehydration processes are connected with an endothermic effect seen on the DTA curves. All dimethoxybenzoates decompose to CoO, which is the final product of their decompositions. The thermal stability of anhydrous compounds increases in order: 2,3- < 2,6- < 3,5-dimethoxybenzoate, while that of hydrated ones changes in the sequence: 2,6- < 3,5- dimethoxybenzoate. From the comparison of the decomposition results it follows that the various position of OCH_3 substituents in benzene ring influences the decomposition process being

Table 2: Temperature ranges of the thermal stability of analysed complexes in air

Complex		Temperature decomp. range (K)	Mass loss (%)		Water molecule	Intermediate and final
			Calcd.	Found	n	products of decomp.
2,3-	CoL_2	369–809	86.00	85.60	–	Co
	Co	907–974	80.93	80.70	–	Co_3O_4
	Co3O4	1158–1206	82.19	81.90	–	CoO
3,5-	$CoL_2 \, 2H_2O$	376–468	7.88	8.00	2	CoL_2
	CoL_2	546–924	87.10	87.00	–	Co
	Co	942–973	82.43	82.60	–	Co_3O_4
	Co_3O_4	1161–1221	83.60	84.10	–	CoO
2,6-	$CoL_2 \, 4H_2O$	331–426	14.61	14.40	4	CoL_2
	CoL_2	467–858	88.05	87.80	–	Co
	Co	871–905	83.72	83.10	–	Co_3O_4
	Co_3O_4	1146–1188	84.97	85.80	–	CoO

connected with the various participations of the inductive and mesomeric effects of OCH_3 groups in the electron density of the system.

The suggested thermal decomposition paths of Co(II) dimethoxybenzoates are as follows:

$$CoL_2 \rightarrow Co \rightarrow Co_3O_4 \rightarrow CoO \text{ for 2,3-dimethoxybenzoate}$$
$$CoL_2 \cdot nH_2O \rightarrow CoL_2 \rightarrow Co \cdot \rightarrow Co_3O_4 \rightarrow CoO \; n = 2$$
$$\text{for 3,5-dimethoxybenzoate}$$
$$n = 4 \text{ for 2,6-dimethoxybenzoate}$$
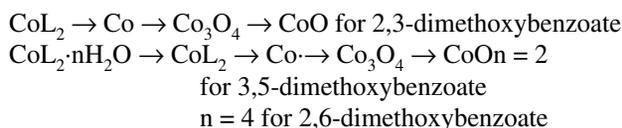
During the decomposition of the anhydrous complexes of Co(II), the Co and Co_3O_4 are formed as intermediate products.

Considering the temperature at which the dehydration processes occur and the ways in which they proceed, one can assume that the water molecules are in the outer or inner coordination spheres of the complexes.[34–36]

The solubility of the analyzed dimethoxybenzoates in water at room temperature was determined (Table 1). 2,6-Dimethoxybenzoate of Co(II) is the best soluble salt while that of 3,5-dimethoxybenzoate the least one. The values of solubilities increase in the order: 3,5- < 2,3- < 2,6-dimethoxybenzoate. Their changes are connected with various influences of inductive, mesomeric and steric effects of methoxy groups on the electron density of the system depending on their position in benzene ring. The inductive effects of each OCH_3 group cause the delocalization of the electrons in the molecule and the change of its energy state brought about the conjugation of electrons. It leads to the stabilization of the system.[37–40]

Magnetic susceptibility of the analysed compounds was measured in the range of 76–303 K (Table 3). Their values decrease with rising temperature. The effective magnetic moment values of the 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II) change from 4.61 BM (at 77 K) to 5.08 BM (at 269 K), from 4.33 BM (at 77 K) to 4.86 BM (at 221 K), and from 3.76 BM (at 76 K) to 4.51 BM (at 303 K), respectively for analysed dimethoxybenzoates.

The complexes show paramagnetic properties and they obey the Curie-Weiss law. The values of the Weiss constant (Θ) for all the complexes were found to be negative, which probably arises from a crystal field splitting of the paramagnetic spin state.[40–43] The paramagnetic dependence of values of the magnetic susceptibility as a function of temperatures are given in Table 3. They give information about the magnetic interaction between paramagnetic centers. As a rule, if χ_M values increase with increasing temperatures, this indicates an antiferromagnetic interaction but when the χ_M values decrease with increasing temperature, the magnetic interaction is ferromagnetic. The χ_M values for analyzed complexes show a gradual decrease with raising temperature. This indicates a tendency of ferromagnetic interaction between the metal ions. In the case of the 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II), the effective magnetic moments are equal to 4.61–5.08 BM, 4.33–4.86 BM and 3.76–4.51 BM (Table 3), while those measured at room temperature for the Co(II) complexes are 5.08, 4.86 and 4.51 BM. These values differ from that of the spin-only moment, which amounts to 3.88 BM. This relatively large difference between the measured and calculated values results from spin-orbital coupling.[44] The experimental data may suggest that analysed dimethoxybenzoates of Co(II) are high-spin complexes with weak ligand fields and probably with octahedral coordination of Co ions in which there are oxygen atoms of carboxylate groups and oxygen atoms of water molecules in the case of hydrated complexes. They may crystallize in the rhombohedral systems. The coordination of central ions could be established on the basis of the complete crystal structure determinations of monocrystals but they have not been obtained.

However, the EPR spectra could help to estimate the character of the coordination geometry of central ions but they were not registered. We presented the interpretation of the ways of coordination in the complexes on the basis of the experimental data and the literature survey concerning the similar investigations[14,16,45,47]. In the case of 3,5-

Table 3: Values of χ_M and μ_{eff}, for analyzed complexes.

CoL$_2$ 2,3-dimethoxybenzoate			CoL$_2$ 2H$_2$O 3,5-dimethoxybenzoate			CoL$_2$ 4H$_2$O 2,6-dimethoxybenzoate		
T (K)	χ_M 10^6	μ_{eff} (BM)	T (K)	χ_M 10^6	μ_{eff} (BM)	T (K)	χ_M 10^6	μ_{eff} (BM)
77	34389	4.61	77	30294	4.33	76	23208	3.76
103	28033	4.82	101	26273	4.63	123	16999	4.09
114	26097	4.90	104	25405	4.62	133	15552	4.07
119	24498	4.85	109	24308	4.60	143	14617	4.09
124	23572	4.85	113	23349	4.61	153	13826	4.12
129	22940	4.88	119	22480	4.65	163	13142	4.14
134	21762	4.85	123	21932	4.67	173	12554	4.17
139	21551	4.92	128	21430	4.71	183	12004	4.19
149	20246	4.93	133	20881	4.74	193	11531	4.22
159	18899	4.93	138	20104	4.74	203	11204	4.27
169	17805	4.93	143	19647	4.77	213	10799	4.29
179	17384	5.02	148	19328	4.81	223	10394	4.31
190	16247	5.00	153	19053	4.86	233	10075	4.34
200	15363	4.99	158	18505	4.86	243	9709	4.35
211	14943	5.06	163	18003	4.87	253	9449	4.38
220	13806	4.96	173	17089	4.89	263	9246	4.41
230	13469	5.01	183	15992	4.87	273	8986	4.43
240	12880	5.01	193	15306	4.90	283	8774	4.46
250	12417	5.02	203	14666	4.92	293	8581	4.49
254	12206	5.02	214	13936	4.92	303	8369	4.51
269	11786	5.08	221	13159	4.86			

L = C$_9$H$_9$O$_4^-$

dimethoxybenzoate the Co(II) ion is presumably in octahedral coordination in which there are four oxygen atoms of two bidentate carboxylate groups, and two oxygen atoms of water molecules.[45–47]

In the 2,6-dimethoxybenzoate the Co(II) ion is presumably also in the octahedral coordination in which there are two oxygen atoms of two monodentate carboxylate groups and four oxygen atoms of water molecules.[14–16,45–47] For Co(II) coordination in the 2,3-dimethoxybenzoate we can only suppose that there may be two tridentate carboxylate groups.[31,32] As it was indicated by thermal analysis data the water molecules in complexes were supposed to be lattice water because they were released below 423 K,[34, 35] but their position in the complex coordination sphere was not precisely determined. However, taking into account the dentates of carboxylate groups, we could suggest being coordination water, which is released at the temperature typical for lattice water.

4. Conclusions

On the basis of the results it appears that 2,3-, 3,5- and 2,6-dimethoxybenzoates of Co(II) were synthesized as anhydrous or hydrated pink complexes. They are crystalline compounds. On heating in air to 1173 K they decompose in three or four steps and form CoO as the final residue of the decompositions. Their solubility in water is

of the order of 10^{-2}–10^{-3} moldm^{-3}. The values of μ_{eff}, calculated for compounds in the range of 76–303 K, reveal them to be probably high-spin complexes with octahedral coordination and weak ligand field.

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Povzetek

Prispevek obravnava fizikalno-kemijske lastnosti in termično stabilnosti na zraku za komplekse 2,3-, 3,5- in 2,6-dimetoksibenzoatov z Cu(II) ionom ter vpliv položaja OCH_3 substituent na termično stabilnost. Kompleksi so kristalinični, kot hidratirani ali brezvodne soli rožnate barve. Termična stabilnost študiranih Co(II) dimetoksibenzoatov je bila študirana v temperaturnem območju od 293–1173 K. Položaj metoksi skupin na benzenovem obroču vpliva na termične lastnosti študiranih kompleksov. Njihova različna termična obstojnost je izrazito povezana z vplivom induktivnega, mezomernega in steričnega vpliva OCH_3 substituent na elektronsko gostoto v benzenovem obroču. Izmerjena je bila magnetna susceptibilnost kompleksov v območju 76–300 K in izračunani magnetni momenti. Rezultati kažejo, da so kompleksi 2,3-, 3,5- in 2,6-dimetoksibenzoatov z Cu(II) ionom visokospinski komleksi s šibkim poljem ligandov.