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*Scandium (III) complexes with
2,5-, 2,6- and 3,4-dichlorobenzoic acids*

Kompleksy skandiu (III) z kwasami 2,5-, 2,6- i 3,4-dichlorobenzoesowymi

Rare earth element complexes with dichlorobenzoic acids, except 3,4-dichlorobenzoic acid, are well known. The 2,4-, 2,5- and 2,6-dichlorobenzoates of Y(III) and lanthanides(III) were prepared as neutral salts with various degrees of hydration [1–4]. Their infrared and X-ray spectra were recorded. The thermal decomposition of these complexes in air atmosphere has been studied, too. These complexes are crystalline (without 2,5-dichlorobenzoate of La-Nd and Dy), sparingly soluble in water. During heating they are decomposed in many steps to oxides with intermediate formation of anhydrous compounds and oxo-chlorides.

The scandium (III) ions in aqueous solution have different properties compared to other rare earth element ions. Scandium (III) ions hydrolyze forming hydroxo- or oxosalts and polymerize. These processes depend on concentration and *pH* of the solutions [5,6]. This is why the preparation of neutral scandium (III) complexes from aqueous solution is almost impossible.

In the previous papers we have described complexes of scandium (III) with 2,4-dichlorobenzoic acid in aqueous solution with the formula $\text{Sc}_5\text{O}_6(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$ [7,8] and in ethanol solution with the formula

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$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 0.5 \text{H}_2\text{O}$ [9–11]. The IR spectra and X-ray diffraction patterns of these complexes were recorded, the thermal decomposition was studied and solubilities in water were determined.

The aim of this work was to prepare scandium (III) 2,5-, 2,6- and 3,4-dichlorobenzoates from ethanol and aqueous solutions and to examine and to compare their physico-chemical properties.

EXPERIMENTAL

Sc_2O_3 99.9% (Koch-Light Lab.), 2,5-dichlorobenzoic acid pure, 2,6-dichlorobenzoic acid pure (Merck Schuchard) and 3,4-dichlorobenzoic acid pure (Riedel de Haen) were used.

PREPARATION OF COMPLEXES

Scandium (III) complexes with 2,5-, 2,6- and 3,4-dichlorobenzoic acids were prepared by adding at room temperature a 0.2M ethanol solution of 2,5-, 2,6- or 3,4-dichlorobenzoic acid, respectively, to a suspension of $\text{Sc}(\text{OH})_3$ in aqueous—ethanol solution (with volumetric ratio of 2:1) using a little excess of the precipitant. The precipitates formed were mixed in mother liquor for 1hr, then were filtered off, washed with ethanol and dried at 30°C to constant mass.

Scandium (III) complexes with 2,5-, 2,6- and 3,4-dichlorobenzoic acids in aqueous solution were prepared by adding 0.1M ammonium 2,5-dichlorobenzoate (*pH* 4.7), 0.1M ammonium 2,6-dichlorobenzoate (*pH* 7.0) or 0.1M ammonium 3,4-dichlorobenzoate (*pH* 7.2), respectively, using a little excess of the precipitating agent, in relation to stoichiometric quantities, to a hot solution of 0.1M $\text{Sc}(\text{NO}_3)_3$ (*pH* 3.8). Precipitates formed were heated for 1hr in maternal solution, then were filtered off, washed with water to remove NH_4^+ ions and dried at 30°C to a constant mass.

ELEMENTAL ANALYSES

The contents of carbon and hydrogen in the obtained complexes were determined by elemental analyses using V_2O_5 as oxidizing agent. The content of chlorine was measured by the Schöniger method. The content of scandium (III) was determined by ignition of the complexes to Sc_2O_3 at 900°C and from the TG

curves. The content of water was determined from the TG curves and by isothermal heating at definite temperatures. The obtained results are given in Table 1.

IR SPECTRA AND X-RAY DIFFRACTION PATTERNS

The IR spectra of 2,5-, 2,6- and 3,4-dichlorobenzoic acids and of their complexes with scandium (III) ions were recorded (Tab. 2, 3). Measurements were made with a FTIR 1725X Perkin Elmer Spectrophotometer within the range of 4000–400 cm^{-1} . The samples were prepared as KBr discs.

The X-ray diffraction patterns of the obtained complexes were measured on a DRON-2 diffractometer within $2\Theta = 5\text{--}80^\circ$ by using $\text{CrK}\alpha$ (V-filtered) radiation. Measurements were made according to the Debye-Scherrer method.

DERIVATOGRAPHIC MEASUREMENTS

The thermal stability of the obtained complexes was studied. The curves of TG, DTG and DTA were recorded. Measurements were made on a Q 1500D derivatograph at a heating rate of $10^\circ\text{C min}^{-1}$ with a sensitivity: TG–100 mg, DTA–500 μV , DTG–500 μV . Samples (100mg) were heated in air atmosphere in platine crucibles to 1000°C . Al_2O_3 was used as a standard. The results are presented in Table 4.

RESULTS AND DISCUSSION

Scandium (III) 2,5-, 2,6- and 3,4-dichlorobenzoates prepared from ethanol and aqueous solutions are white colored solids. Scandium (III) dichlorobenzoates obtained from ethanol and scandium (III) 3,4-dichlorobenzoate obtained from water are hydrates with a metal to ligand ratio of 1:3. 2,5-dichlorobenzoate of scandium (III) prepared from water is a trihydrated oxosalt with a metal to ligand ratio of 1:1 and 2,6-dichlorobenzoate is a monohydrated basic salt with a metal to ligand ratio of 1:2.

Table 1. Analytical data

Scandium (III) complexes		% Sc		% C		% H		% Cl	
Name	Formula	Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
2,5-dichlorobenzoate et*	$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 2\text{H}_2\text{O}$	6.90	6.20	38.74	39.49	2.01	1.98	32.67	32.05
2,5-dichlorobenzoate w**	$\text{ScO}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO}) \cdot 3\text{H}_2\text{O}$	14.74	15.30	27.56	28.23	2.97	2.94	23.25	22.84
2,6-dichlorobenzoate et*	$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 3\text{H}_2\text{O}$	6.72	6.82	37.70	37.86	2.26	2.22	31.79	31.59
2,6-dichlorobenzoate w**	$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_2(\text{OH}) \cdot \text{H}_2\text{O}$	9.77	10.08	36.55	36.25	1.97	2.08	30.82	30.42
3,4-dichlorobenzoate et*	$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$	6.50	6.78	36.70	36.51	2.49	2.23	30.96	30.92
3,4-dichlorobenzoate w**	$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 2\text{H}_2\text{O}$	6.90	7.20	38.74	39.48	2.01	2.20	32.67	32.18

et* – prepared from ethanol;

w** – prepared from water

Table 2. Frequency of the maxima absorption bands in the IR spectra of scandium dichlorobenzoates, [cm⁻¹]

Scandium dichlorobenzoates						Assignment
(2,5) et	(2,5) w	(2,6) et	(2,6) w	(3,4) et	(3,4) w	
—	—	—	3648 m	—	—	vibration of bounded OH group
3434 wk	3400 m	3432 m	3408 wk	3434 m	3432 wk	stretching of OH group with H ₂ O
—	—	—	—	1619 m	1620 m	deformation OH for hydrated salt
1568 vs	1568 s	1568 vs	1570 vs	1588 s	1588 s	asymmetrical vibrations of COO ⁻ groups
—	—	—	—	—	1580 s	
1543 vs	1544 s	1555 vs	1556 vs	1536 s	1528 vs	vibrations of benzene ring
1464 s	1464 m	1464 s	1464 s	1480 m	1480 m	
—	—	1432 s	1432 s	—	—	symmetrical vibrations of COO ⁻ groups
1430 vs	1420 s	—	—	—	—	
1408 vs	—	1408 vs	1408 vs	1421 vs	1412 vs	vibrations of benzene ring
1376 s	1376 m	—	—	—	1375	
1272 m	1280 wk	1280 vw	—	1280 wk	1280 wk	vibrations of benzene ring
1250 wk	1250vwk	—	—	1248 m	1248 m	
—	—	1200 m	1200 m	—	—	deformation C—H in plane bending vibrations for trisubstituted benzene
1166 vs	1170vwk	1174 m	1176 m	1168 wk	1168 wk	
1134vwk	1130vwk	—	—	1120 m	1128 m	deformation of C—H
1104 s	1100 m	1092 w	1096 wk	—	—	
1055 s	1052 m	—	1060 wk	—	—	deformation C—H out-of-plane bending vibrations for trisubstituted benzene
—	—	—	—	1032 m	1032 m	
956vwk	945 wk	—	945vwk	—	—	deformation C—H out-of-plane bending vibrations for trisubstituted benzene
893 m	888 wk	—	—	888 wk	888 wk	
—	—	850 w	850vwk	842vwk	844vwk	valency vibrations of C—Cl
824 s	820 m	—	—	—	—	
—	—	812 m	808m	—	—	skeletal vibrations metal—oxygen
800 s	800 m	—	—	—	—	
—	—	779 s	776 s	780 s	780 s	metal—oxygen
752 m	756 m	750 m	760 s	756 m	—	
—	—	—	736 m	—	—	skeletal vibrations
706vwk	—	696 m	696 wk	—	—	
—	—	—	—	680 wk	680 wk	skeletal vibrations
668 m	664 m	—	—	—	—	
624 s	624 s	—	620vwk	630 wk	—	skeletal vibrations
—	—	600 w	600vwk	—	596 m	
555 wk	560 m	—	—	564 s	564 s	metal—oxygen
510 wk	—	504 vs	504 vs	—	—	
464 s	476 vs	—	—	470 wk	468 s	metal—oxygen
—	—	448 s	448 s	436 s	440 wk	

et – prepared from ethanol; w – prepared from water;

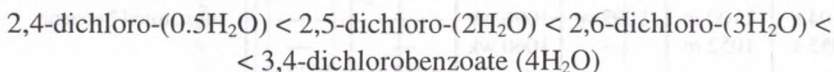
vs – very strong; s – strong; m – medium; wk – weak; vwk – very weak

Table 3. Frequency of the absorption bands of COO^- group and metal—oxygen bond in scandium (III) and sodium dichlorobenzoates, $[\text{cm}^{-1}]$

Compound	$\nu_{as}\text{COO}^-$	$\nu_{sym}\text{COO}^-$	$\Delta\nu$	$\nu \text{M—O}$
$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 2\text{H}_2\text{O}$ (2,5) et*	1556	1419	137	464
$\text{ScO}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 3\text{H}_2\text{O}$ (2,5) w**	1556	1420	136	476
$\text{NaCl}_2\text{C}_6\text{H}_3\text{COO}$	1591	1408	183	
$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 3\text{H}_2\text{O}$ (2,6) et*	1562	1408	154	448
$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_2(\text{OH}) \cdot \text{H}_2\text{O}$ (2,6) w**	1563	1408	155	448
$\text{NaCl}_2\text{C}_6\text{H}_3\text{COO}$	1607	1391	216	
$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 4\text{H}_2\text{O}$ (3,4) et*	1562	1412	150	453
$\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 2\text{H}_2\text{O}$ (3,4) w**	1565	1412	153	454
$\text{NaCl}_2\text{C}_6\text{H}_3\text{COO}$	1573	1405	168	

et* – prepared from ethanol; w** – prepared from water

The hydration degree of neutral dichlorobenzoates of scandium (III) prepared from ethanol changes with the change of the chlorine atom position in the benzene ring in the following order:



2,4- and 2,5-dichlorobenzoates prepared from aqueous solution are oxosalts with a ratio of metal to ligand of 5:3 [7] and 1:1, respectively.

The IR spectra of the obtained complexes and, for comparison, of the parent acids (Tab. 2, 3) were recorded. Analyses of the IR spectra confirmed the composition of the complexes.

The IR spectra of the prepared scandium (III) dichlorobenzoates exhibit broad absorption bands with max. at $3434\text{--}3400 \text{ cm}^{-1}$, confirming the presence of crystallization water molecules. In the IR spectrum of scandium (III) 2,6-dichlorobenzoate (prepared from aqueous solution) a sharp absorption band of the OH group appears at 3648 cm^{-1} which confirms the basic character of the complex. This band does not occur in the IR spectra of the other complexes.

In the IR spectra of scandium (III) dichlorobenzoates appear two bands arising from asymmetric and symmetric vibrations of COO^- group at $1588\text{--}1528 \text{ cm}^{-1}$ and $1430\text{--}1408 \text{ cm}^{-1}$, respectively, and the absorption band of metal—oxygen bond at $476\text{--}436 \text{ cm}^{-1}$. The band of metal—oxygen bond in the IR spec-

trum of scandium (III) 2,5-dichlorobenzoate, prepared from aqueous solution, has lower intensity compared to those bands in the spectra of other complexes, which confirms that it was prepared as oxosalt.

In the IR spectra of scandium (III) dichlorobenzoates several strong absorption bands characteristic of the C—Cl bond in the range of low frequency at 780–560 cm^{-1} are present as well as the absorption bands of the deformation in plane vibration of the C—H in benzene ring in the trisubstituted position at 1176–1032 cm^{-1} and the out-of plane vibration of the C—H at 893–680 cm^{-1} . In the range 779–680 cm^{-1} the absorption bands of valency vibrations of the C—Cl bond and of out-of plane vibration of the C—H characteristic of trisubstituted benzene coincide.

The small shift (5 cm^{-1}) of the absorption bands of valency vibrations of the C—Cl bond in the IR spectra of these complexes compared to the spectra of dichlorobenzoic acids suggests that the chlorine atoms do not take part in metal-ligand coordination. The absorption bands of aromatic ring vibrations in the IR spectra of scandium (III) dichlorobenzoates at 1376–1200 cm^{-1} do not change compared to those bands in the spectra of suitable dichlorobenzoic acids. It was found that scandium ion does not influence the change of electron density in the aromatic ring [12].

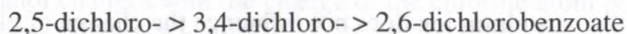
The magnitudes of separation ($\Delta\nu$) between frequencies due to asymmetrical and symmetrical vibration of the COO^- group (Tab. 3) of scandium (III) 2,6- and 3,4-dichlorobenzoates are almost identical and less for scandium 2,5-dichlorobenzoate which indicates the similar degree of ionic bond in scandium (III) 2,6-, and 3,4-dichlorobenzoates and a similar way of bonding ligands with scandium (III) ion. The participation of ionic bond in scandium (III) 2,5-dichlorobenzoate is smaller in comparison with remaining [9] dichlorobenzoates. For 2,4-dichlorobenzoate of scandium (III) the participation of ionic bond is much smaller. The influence of substituent on the carboxylate ion is different and depends on their position. The presence of two chlorine atoms near the carboxylate ion in scandium (III) 2,6-dichlorobenzoate or close to each other but maximally distant from the carboxylate ion in scandium (III) 3,4-dichlorobenzoate increases the degree of the ionization of the $\text{COO}-\text{Sc}/3$ bond in comparison with the scandium (III) 2,5- and 2,4-dichlorobenzoates, where the position of two chlorine atoms is irregular. The magnitudes of separation ($\Delta\nu$) between frequencies due to asymmetrical vibration of the COO^- group of scandium (III) dichlorobenzoates prepared from aqueous solution and from ethanol are almost identical, respectively, which indicates that the degree of ionization

of COO—Sc/3 bond depends on organic anion but is independent of the kind of salt.

The separations of absorption bands of COO⁻ group ($\Delta\nu$) for scandium (III) dichlorobenzoates are far smaller than those for the respective salts of sodium (Tab. 3). This is the result of a stronger action of the Sc³⁺ ion than of the Na⁺ one on the ligand, greater deformation and smaller degree of ionic bond in studied complexes than in the sodium salt.

The bands of asymmetrical vibration $\nu_{as}\text{COO}^-$ of scandium (III) complexes with described acids are shifted to lower frequencies and the bands of symmetrical vibrations $\nu_{sym}\text{COO}^-$ to higher ones in relation to absorption bands of sodium salts. Splitting these bands in relation to the absorption bands of sodium salts indicates that the carboxylic ion in studied complexes is probably a bidentate chelating ligand [13].

The frequency of the absorption band of the scandium-oxygen bond (Tab. 3) for dichlorobenzoates prepared from water and for those prepared from ethanol changed in the order:



It suggests that the 2,6-dichlorobenzoate of scandium (III) is the least stable [14]. The difference of degree of hydration in 3,4-dichlorobenzoates from ethanol and from aqueous solution does not influence complex stability. The presence of hydroxylate group in 2,6-dichlorobenzoate of scandium (III) prepared from aqueous solution does not influence the complex stability in comparison with neutral 2,6-dichlorobenzoate of scandium (III), too. The stability of 2,5-dichlorobenzoate from ethanol (neutral salt) and from water (oxosalt) is variable.

In order to confirm crystalline structure of the prepared complexes X-ray spectra were recorded. On the basis of these diffractograms it was found that these complexes are roentgenographically amorphous compounds.

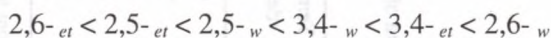
The prepared complexes heated in air decompose in two steps. In the first step they are dehydrated endothermically within the range of 30–280 °C losing some of their crystallization water molecules and then fewer hydrated complexes decompose immediately after the dehydration, simultaneously with decomposition of the organic anions. The water lost at the lower temperature is probably outer-sphere water and crystallization water lost at the higher one is inner-sphere water. These results suggest that the hydrates of scandium 2,5-, 2,6- and 3,4-dichlorobenzoates prepared from ethanol exist, probably, as com-

plexes with formula $[\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot \text{H}_2\text{O}] \cdot \text{H}_2\text{O}$, $[\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot \text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}$ and $[\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot 2\text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}$, respectively and 2,5- and 3,4-dichlorobenzoates of scandium (III) prepared from water as salts with formula: $[\text{ScO}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO}) \cdot 2\text{H}_2\text{O}] \cdot \text{H}_2\text{O}$ and $[\text{Sc}(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_3 \cdot \text{H}_2\text{O}] \cdot \text{H}_2\text{O}$ respectively. Only the 2,6-dichlorobenzoate of scandium (III) prepared from water loses 0.5 molecule of crystallization water at 30–200 °C and as hemihydrated salt is stable up to 260°C and next decomposes to Sc_2O_3 . In this case the salt exists, probably, as dimer with formula $[\text{Sc}_2(\text{OH})_2(\text{Cl}_2\text{C}_6\text{H}_3\text{COO})_4 \cdot \text{H}_2\text{O}] \cdot \text{H}_2\text{O}$.

The partially dehydrated 3,4-dichlorobenzoate of scandium (III) prepared from ethanol begins to decompose at the lower temperature than that prepared from aqueous solution. This fact suggests that the presence of two molecules of crystallization water in inner-sphere increases the thermal stability of scandium (III) 3,4-dichlorobenzoate.

The neutral dichlorobenzoates (2,5-, 2,6- and 3,4-), which have one molecule of crystallization water in inner-sphere begin to decompose nearly at the same temperature. This fact suggests that the position of chlorine atoms in the benzene ring does not influence the thermal stability of the scandium (III) dichlorobenzoates but the stability of the neutral compounds depends on the amount of crystallization water in inner-sphere.

Dichlorobenzoates of scandium (III) decompose directly to Sc_2O_3 (not through oxochlorides like dichlorobenzoates of other rare earth elements) [1–4], which formed at 575–750 °C. The temperature of Sc_2O_3 formation increases in the order:



The temperature of Sc_2O_3 formation is the greatest for scandium (III) 2,6-dichlorobenzoate prepared from water which, probably, exists as dimer.

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Table 4. Temperature data of dehydration and decomposition of scandium (III) dichlorobenzoates

Complex	Temp. range of dehydration [°C]	Loss of weight [%]		Loss of H ₂ O molecules [n]	Temp. range of decomposition [°C]	Loss of weight [%]	
		Calcd.	Found			Calcd.	Found
Sc(Cl ₂ C ₆ H ₃ COO) ₃ ·2H ₂ O (2,5) et*	40–270	2.73	2.7	1	270–625	89.41	90.5
ScO(Cl ₂ C ₆ H ₃ COO) ₃ ·3H ₂ O (2,5) w**	40–280	5.91	6.0	1	280–640	77.36	76.5
Sc(Cl ₂ C ₆ H ₃ COO) ₃ ·3H ₂ O (2,6) et*	30–280	5.38	5.2	2	280–575	89.70	89.6
Sc(Cl ₂ C ₆ H ₃ COO) ₂ (OH)·H ₂ O (2,6) w**	30–200	1.96	2.0	0.5	260–750	85.00	84.5
Sc(Cl ₂ C ₆ H ₃ COO) ₃ ·4H ₂ O (3,4) et*	30–210	5.24	5.5	2	210–700	90.00	89.6
Sc(Cl ₂ C ₆ H ₃ COO) ₃ ·2H ₂ O (3,4) w**	40–265	2.76	3.0	1	265–660	89.41	88.9

et* – prepared from ethanol, w** – prepared from water

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STRESZCZENIE

2,5-, 2,6- i 3,4-dichlorobenzoesany skandu (III) otrzymano z roztworów wodnych lub etanolowych i wyznaczono ich skład ilościowy. Z etanolowych roztworów otrzymano kompleksy o stosunku molowym metal–ligand organiczny 1:3 o różnym stopniu uwodnienia. 2,5-Dichlorobenzoesan skandu (III) jest dihydratem, 2,6-dichlorobenzoesan – trihydratem, a 3,4-dichlorobenzoesan – tetrahydratem. Kompleksy otrzymane z roztworów wodnych są związkami o różnym składzie. 2,5-Dichlorobenzoesan skandu (III) jest trójwodną oksosolą o stosunku molowym metal – ligand organiczny 1:1; 2,6-dichlorobenzoesan jest jednowodną hydroksysołą o stosunku molowym metal–ligand organiczny 1:2; 3,4-dichlorobenzoesan skandu (III) jest dwuwodną solą obojętną o stosunku molowym metal – ligand 1:3. Na podstawie analizy dyfraktogramów stwierdzono, że otrzymane kompleksy są amorficzne. Na podstawie wyników analiz widm IR można przypuszczać, że w koordynacji metal–ligand organiczny biorą udział tylko atomy tlenu grup COO^- . W czasie ogrzewania kompleksy tracą cząsteczki wody krystalizacyjnej, a następnie rozkładają się do Sc_2O_3 .

