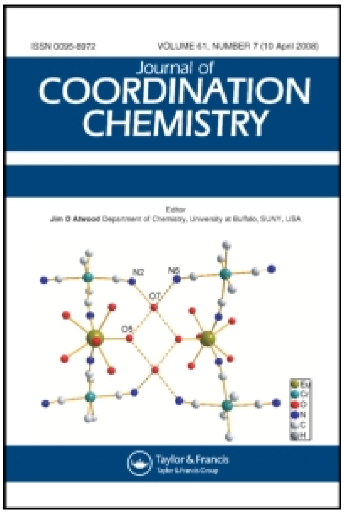

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Crystal structure, spectroscopic and thermal studies of 3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid sodium salt

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To cite this Article Kula, Alina , Mazur, Liliana and RzĄczyńska, Zofia(2007) 'Crystal structure, spectroscopic and thermal studies of 3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid sodium salt', Journal of Coordination Chemistry, 60: 8, 843 — 850

To link to this Article: DOI: 10.1080/00958970600936765

URL: http://dx.doi.org/10.1080/00958970600936765

Journal of Coordination Chemistry
Vol. 60, No. 8, 20 May 2007, 843–850

Taylor & Francis
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Crystal structure, spectroscopic and thermal studies of 3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid sodium salt

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(*Received 21 June 2006; in final form 18 July 2006*)

The crystal structure, infrared spectrum and thermal stability of 3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid sodium salt have been studied. The compound of general formula $\{[Na_2L_2 \cdot 3H_2O] \cdot H_2O\}_n$ is a two-dimensional polymer, in which Na ions are bridged by one or two water molecules and additionally coordinated to oxygen atoms of carboxylate, hydroxy and methoxy groups. The structure is stabilized by a hydrogen bond network. The compound dehydrates at 75°C and then decomposes at 150°C. The IR spectra of the salt and free acid are discussed.

Keywords: Sodium 3-(4-hydroxy-3-methoxyphenyl)-2-propenoate; Sodium ferulate; Sodium 4-hydroxy-3-methoxycinnamate; X-ray structure; IR spectra

1. Introduction

Pure 3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid (ferulic acid), yellowish powder, crystallizes in the monoclinic system, with unit cell dimensions of: $a = 4.6405(4)$ Å, $b = 16.824(5)$ Å, $c = 12.019(4)$ Å, $\beta = 90.15(1)^\circ$, $V = 938.4(1)$ Å^3 [1]. It is found in seeds and leaves of many plants, such as brown rice, wheat, oat, as well as in many fruits. It is a biologically active substance of low toxicity, which can be absorbed and readily metabolized in the human body. It is reported to have influence on many physiological functions as an antioxidant [2–6] and as a substance, which reduces blood pressure [7]. Similar biological properties have been observed for sodium ferulate, which may inhibit artherosclerogenesis and prevent neurotoxicity [8, 9]. This work is a continuation of studies of the structural, spectral and thermal properties of the complexes of benzoic acid derivatives, which contain hydroxy and methoxy functional groups. Particularly interesting is the metal–ligand coordination in the above mentioned complexes. In 2-hydroxybenzoates of Ag(I), Zn(II), Cu(II), Ni(II), Mg(II) [10–14], 3-hydroxybenzoate of Nd(III) [15], 2,6-dihydroxybenzoates of Tb(III) and Ho(III) [16] only a

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online
DOI: 10.1080/00958970600936765

carboxylate group takes part in the metal ion coordination. Hydroxy groups of ligands do not take part in the coordination at all. Coordination of metal ion through oxygen atoms of hydroxyl groups has been described only for 2-hydroxybenzoates of Sm(III) and Am(III) [17]. Studies of metal complexes with benzenecarboxylic acids, containing additional methoxyl groups, showed that the metal–ligand coordination is possible only through oxygen atoms from carboxylate groups [18–24].

Our previous work dealt with lanthanide(III) ferulates in which the IR spectra, thermal stability and solubility in water at 293 K were studied. As shown by the IR spectra, metal coordination is possible only through oxygen atoms of carboxylate groups [25–27].

2. Experimental

2.1. *Preparation of sodium salt*

Sodium ferulate was prepared by the reaction of equivalent quantities of ferulic acid and 0.5 M sodium hydroxide. The aqueous solution was evaporated at room temperature. Single crystals were obtained after few weeks.

2.2. *X-ray crystallography*

Single-crystal diffraction data were measured at room temperature in the $\omega/2\theta$ mode on the Oxford Diffraction Xcalibur diffractometer using graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). The stability of intensities was monitored by measurement of three standards every 100 reflections. Crystal structure was solved by direct methods using SHELXS97 [28] and refined by full-matrix least-squares on F^2 using the SHELXL97 [29] program. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms of water molecules and hydroxy groups were located from a difference Fourier map and refined. The remaining hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Details of the crystal data, X-ray data collection and refinement are given in table 1. Selected bond parameters are listed in table 2.

2.3. *The IR spectra*

The IR spectra of ferulic acid and its sodium salt were recorded in the range of 4000–400 cm^{-1}, using a Perkin Elmer 1725X FTIR spectrometer. The samples were prepared as KBr discs.

2.4. *Thermal analysis*

The thermal stability of the sodium salt in air was determined using a Setsys 16/18 TG, DTG, DTA instrument. The sample (7.9 mg) was heated in the Al_2O_3 crucible at 293–973 K in flowing air with a heating rate of 5 K min^{-1}. The products of decomposition were calculated from the TG curve.

Table 1. Crystal data and experimental parameters.

Empirical formula	$C_{20}H_{26}Na_2O_{12}$
Formula weight	504.39
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	12.666(4)
b (Å	7.293(3)
c (Å)	25.035(7)
β (°)	98.98(4)
V (Å^3)	2284(1)
Z	4
D_c (g cm^{-3})	1.467
Crystal size (mm^3)	$0.48 \times 0.40 \times 0.12$
μ (mm^{-1})	0.152
F(000)	1056
θ range (°)	1.6–30.1
Index ranges	$-17 \le h \le 17$; $0 \le k \le 10$; $0 \le l \le 35$
Reflections collected	6841
Crystal decay (%)	1.2
Data/parameters in refinement	6699/337
GOF	0.973
R_1	0.0695
wR_2	0.1537
$\Delta\rho_{max}$; $\Delta\rho_{min}$ (e Å^{-3})	0.36; −0.29

3. Results and discussion

3.1. *Crystal structure*

The crystal structure of the sodium salt of 3-(4-hydroxy-3-methoxyphenyl)-2-propenoic acid (ferulic acid), of the formula $\{[Na_2(C_6H_3OHOCH_3C_2H_2COO)_2 \cdot 3H_2O] \cdot H_2O\}_n$ was determined by single-crystal X-ray diffraction. The compound in the crystalline state is a two-dimensional polymer where centrosymmetric tetramers can be distinguished (figures 1 and 2). Sodium ions are coordinated by oxygen atoms of carboxylate, hydroxy and methoxy groups as well as water molecules. The asymmetric unit of the complex consists of two sodium cations, two 3-(4-hydroxy-3-methoxyphenyl)-2-propenoate anions (ferac$^-$) making the complex neutral, and four water molecules. There are two types of metal ion environments: Na(1) is coordinated by five oxygen atoms, three from ferac$^-$ anions and two from water molecules, while Na(2) is surrounded by six oxygen atoms, three from ferac$^-$ anions and three from water molecules. The coordination polyhedron of Na(1) ion takes the form of a distorted square-pyramid whereas that of Na(2) ion can best be described as a significantly distorted octahedron. The polyhedra in the tetramer are connected by water molecules which form their vertices.

Among the water molecules one (O1w) is a monodentate ligand and two others are bidentates. The water molecules O(2w) and O(2wa) create a binary centrosymmetric bridge between Na(2) and Na(2a) ions (figure 1), whereas O(3w) bridges Na(1) and Na(2) ions. The fourth water molecule (O4w) occurs in the external coordination sphere. Adjacent Na(1) and Na(2) ions are additionally linked *via* a bidentate carboxylate C(1)O(1)O(2) group of the *syn–syn* type. The C–O–Na angles are 136.1(2)°

Table 2. Selected bond lengths (Å) and angles (°) for sodium ferulate.

Na(1)–O(3)	2.468(2)	Na(2)–O(13)	2.415(2)
Na(1)–O(4)	2.348(3)	Na(2)–O(14)	2.408(3)
Na(1)–O(1w)	2.442(3)	Na(2)–O(2w)	2.377(3)
Na(1)–O(3w)	2.315(3)	Na(2)–O(3w)	2.426(3)
Na(1)–O(1)$^{(i)}$	2.306(2)	Na(2)–O(2)$^{(i)}$	2.302(2)
		Na(2)–O(2w)$^{(ii)}$	2.469(3)
C(1)–O(1)	1.257(4)	C(11)–O(11)	1.268(4)
C(1)–O(2)	1.260(4)	C(11)–O(12)	1.244(4)
C(1)–C(2)	1.491(4)	C(11)–C(12)	1.499(4)
C(2)–C(3)	1.323(4)	C(12)–C(13)	1.310(4)
C(3)–C(1f)	1.466(4)	C(13)–C(11f)	1.461(4)
C(3f)–O(3)	1.375(4)	C(13f)–O(13)	1.368(4)
O(3)–C(3m)	1.419(3)	O(13)–C(13m)	1.416(3)
C(4f)–O(4)	1.368(3)	C(14f)–O(14)	1.364(3)
O(1)–Na(1)–O(3w)$^{(i)}$	90.6(1)	O(2w)–Na(2)–O(2)$^{(i)}$	105.5(1)
O(4)–Na(1)–O(1)$^{(i)}$	109.6(1)	O(14)–Na(2)–O(2)$^{(i)}$	89.8(1)
O(3w)–Na(1)–O(4)	153.4(1)	O(2w)–Na(2)–O(14)	100.9(1)
O(1w)–Na(1)–O(1)$^{(i)}$	93.4(1)	O(13)–Na(2)–O(2)$^{(i)}$	155.0(1)
O(3w)–Na(1)–O(1w)	100.9(1)	O(2w)–Na(2)–O(13)	85.0(1)
O(4)–Na(1)–O(1w)	95.1(1)	O(13)–Na(2)–O(14)	65.7(1)
O(3)–Na(1)–O(1)$^{(i)}$	172.5(1)	O(3w)–Na(2)–O(2)$^{(i)}$	87.4(1)
O(3w)–Na(1)–O(3)	93.0(1)	O(2w)–Na(2)–O(3w)	160.2(1)
O(3)–Na(1)–O(4)	65.0(1)	O(14)–Na(2)–O(3w)	94.0(1)
O(1w)–Na(1)–O(3)	92.4(1)	O(13)–Na(2)–O(3w)	89.4(1)
		O(2w)–Na(2)–O(2)$^{(i)}$	111.5(1)
		O(2w)$^{(ii)}$–Na(2)–O(2)$^{(i)}$	111.5(1)
		O(2w)–Na(2)–O(2w)$^{(ii)}$	78.6(1)
		O(14)–Na(2)–O(2w)$^{(ii)}$	158.2(2)
		O(13)–Na(2)–O(2w)$^{(ii)}$	92.6(1)
		O(3w)–Na(2)–O(2w)$^{(ii)}$	82.8(1)
O(1)–C(1)–O(2)	124.6(3)	O(11)–C(11)–O(12)	124.6(3)
O(1)–C(1)–C(2)	117.3(3)	O(11)–C(11)–C(12)	116.8(3)
O(2)–C(1)–C(2)	118.2(3)	O(12)–C(11)–C(12)	118.6(3)
C(3)–C(2)–C(1)	122.8(3)	C(13)–C(12)–C(11)	121.8(3)
C(2)–C(3)–C(1f)	129.6(3)	C(12)–C(13)–C(11f)	129.0(3)
C(6f)–C(1f)–C(3)	122.9(3)	C(16f)–C(11f)–C(13)	123.2(3)
C(2f)–C(1f)–C(3)	119.0(2)	C(12f)–C(11f)–C(13)	118.7(3)
O(4)–C(4f)–C(5f)	123.8(2)	O(14)–C(14f)–C(15f)	123.6(3)
O(4)–C(4f)–C(3f)	116.7(3)	O(14)–C(14f)–C(13f)	116.7(3)
O(3)–C(3f)–C(2f)	125.5(2)	O(13)–C(13f)–C(12f)	124.6(2)
O(3)–C(3f)–C(4f)	115.1(2)	O(13)–C(13f)–C(14f)	116.4(2)
C(3f)–O(3)–C(3m)	117.2(2)	C(13f)–O(13)–C(13m)	117.6(2)

and 131.8(2)°. Carboxylate group C(11)O(11)O(12) of the other ligand does not participate in metal coordination. The C–O distances in both COO^{-} systems are almost the same, in the range 1.244(4)–1.268(4) Å which confirms deprotonation of both carboxylic groups. The values of O–C–O angles in both organic molecules are also the same [124.6(3)°], so engagement of carboxylate group in the metal ion coordination does not change significantly their geometry. The Na–O$_{\text{carboxyl}}$ distances are 2.306(2)–2.302(2) Å being shorter than the remaining Na–O bonds. The Na–O$_{\text{hydroxy}}$ distances are 2.348(3) and 2.408(3) Å, while Na–O$_{\text{methoxy}}$ distances are 2.468(3) and 2.415(2) Å. The Na–O$_{\text{water}}$ bond lengths vary in the range 2.315(3)–2.469(3) Å. The differences in bond lengths are connected with a significant distortion of the coordination polyhedra of Na ions. The angles, that are 90° in a

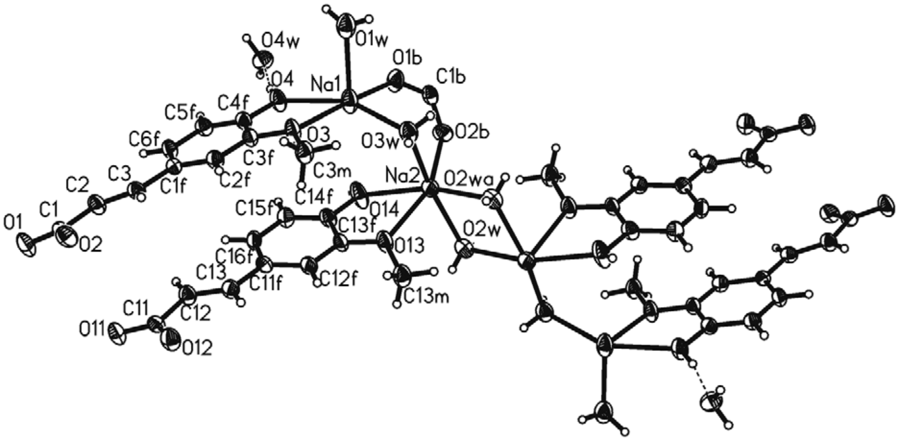

Figure 1. The molecular structure of the title complex with the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (a) $-x$, $1-y$, $1-z$, (b) x, $1.5-y$, $0.5+z$.

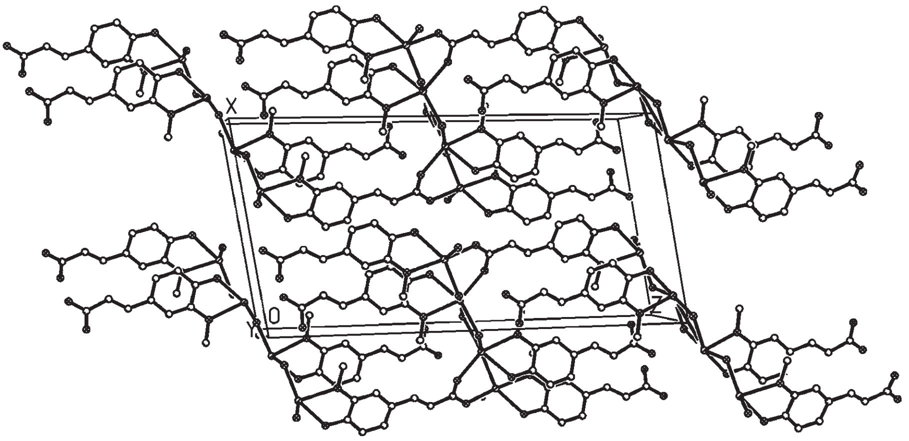

Figure 2. Crystal packing along the *y* axis. Hydrogen atoms and guest water molecules are omitted for clarity.

regular square-pyramidal or octahedral arrangement, in this case range from 65.0(1)° to 111.5(1)°. The coordination polyhedra geometry is given in table 2.

Coordination of metal ions is connected with changes in conformation of 3-(4-hydroxy-3-methoxyphenyl)-2-propenoate anions in comparison with free acid molecules [1]. The main difference is in orientation of the phenyl ring. In ferulic acid the C(2) atom is *syn* oriented with respect to the C(2f) atom, whereas in ferac$^-$ anions the atoms C(2) and C(2f) are *anti* to each other. In this respect, they resemble 3,4-dihydroxycinnamic acid [30].

In the crystal structure of sodium ferulate, as in free ferulic acid, the 4-hydroxy-3-methoxyphenyl fragments of both organic ligands are almost planar, only the carbon atoms C(3m), C(13m) of methoxy groups are deflected by 0.23(1) Å and 0.39(1) Å

Table 3. Hydrogen bond geometry.

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (°)
O(4)–H(4o)···O(4w)	0.87(4)	1.75	2.615(3)	170
O(14)–H(14o)···O(1w)$^{(i)}$	0.90(4)	1.82	2.692(4)	163
O(1w)–H(1w1)···O(1)$^{(ii)}$	0.99(4)	1.72	2.698(4)	175
O(1w)–H(2w1)···O(4w)$^{(iii)}$	0.72(4)	2.12	2.841(4)	177
O(2w)–H(1w2)···O(12)$^{(iv)}$	0.76(4)	2.02	2.780(3)	171
O(2w)–H(2w2)···O(11)$^{(v)}$	0.86(4)	1.98	2.835(3)	172
O(3w)–H(1w3)···O(12)$^{(vi)}$	0.85(4)	1.99	2.827(3)	168
O(3w)–H(2w3)···O(11)$^{(vii)}$	0.83(4)	1.98	2.799(3)	173
O(4w)–H(1w4)···O(2)$^{(viii)}$	0.88(4)	1.86	2.728(4)	170
O(4w)–H(2w4)···O(11)$^{(ii)}$	0.88(4)	1.81	2.667(3)	167

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, y+1/2, -z+1/2$; (iii) $-x+1, -y+2, -z+1$; (iv) $-x, y-1/2, -z+1/2$; (v) $x, -y+1/2, z+1/2$; (vi) $-x, y+1/2, -z+1/2$; (vii) $x, -y+3/2, z+1/2$; (viii) $-x+1, y-1/2, -z+1/2$.

from the phenyl ring planes. However, the 4-hydroxy-3-methoxyphenyl systems are not coplanar with aliphatic fragments; the carboxylate groups C(1)O(1)O(2) and C(11)O(11)O(12) form dihedral angles of 13.3(4) and 13.5(4)° with appropriate phenyl ring planes. In ferulic acid the corresponding angle is equal to 4.0(1)° [1].

The crystal structure of the complex is stabilized by a network of strong O–H···O hydrogen bonds. In these interactions the water molecules and hydroxy groups of ligands are proton donors while oxygen atoms of carboxylate systems as well as some water molecules are proton acceptors. Each coordinated water O(2w) and O(3w) forms two hydrogen bonds, whereas the third coordination water molecule O(1w) is involved in four hydrogen bonds. Deprotonated carboxylate C(11)O(11)O(12) does not take part in the metal ion coordination, but participates as a proton acceptor in five almost linear, short hydrogen bonds. The complete list of hydrogen bonds and their geometric parameters are given in table 3.

3.2. *IR spectra*

The IR spectrum of free ferulic acid shows a sharp absorption band of carboxylic group –COOH at 1690 and 1664 cm^{-1}, sharp absorption band of hydroxylic group –OH (free) at 3435 cm^{-1} and ν(COH) at 1277 cm^{-1}. The characteristic bands of $\nu_{as}(CH_3)$ and $\nu_s(CH_3)$ appear at 3016, 2969 and 2841 $\mathrm{cm}^{-1}$and the bands of $\delta_{as}(CH_3)$, $\delta_s(CH_3)$ and ν_s(C–O–C) are at 1466, 1433, 1035 cm^{-1}, respectively [31, 32].

In the IR spectrum of the sodium salt, the bands of carboxylic group disappear whereas the asymmetric absorption band of $\nu_{as}(OCO^-)$ at 1548 cm^{-1} and the symmetric absorption band $\nu_{as}(OCO^-)$ at 1382 cm^{-1} can be observed. The symmetric absorption band ν_s is sensitive to the coordination mode. In the structure there are two types of carboxylate groups. Almost identical geometry of both groups may account for the fact that the symmetric absorption band ν_s is not split. Nevertheless, the IR spectrum is not enough to determine that only one carboxylic group participates in the metal–ligand coordination, while the other group is deprotonated and does not participate directly in coordination.

The absorption bands $\delta_{as}(CH_3)$, $\delta_s(CH_3)$, band ν_s(C–O–C) and ν(COH) in sodium ferulate are insignificantly shifted to lower frequencies (6–14 cm^{-1}), compared to those in the free acid. The shifting of these bands is due to the fact that metal–ligand coordination occurs through both functional groups.

In the IR spectrum the characteristic, broad band at $3401\,\mathrm{cm}^{-1}$ for hydrogen-bonded water and a sharp deformation band $\delta(H_2O)$ at $1641\,\mathrm{cm}^{-1}$ are observed. The vibration bands at 781, 699 and $623\,\mathrm{cm}^{-1}$ suggest coordinated water. The band corresponding to Na–O bonds is probably below $400\,\mathrm{cm}^{-1}$ [33, 34].

3.3. *Thermal analysis*

Sodium ferulate is stable at room temperature. Upon heating, the dehydration process begins at about 75°C and the complex loses two water molecules. The TG and DTA curves show a loss of mass at 75 to 153°C accompanied by one endothermic effect on the DTA curve at 140°C. The anhydride compound is unstable and just after dehydration the decomposition process occurs. The TG curve shows the mass loss of 27.7% at 240°C which is connected with partial decarboxylation of organic ligand. At 300°C further decomposition of compound takes place. In the temperature range of 550–700°C the mass loss is 50.1% and the TG curve shows a plateau. The final product of decomposition is black and contains a mixture of sodium carbonate, oxide and peroxide as well as carbon, similar to sodium aminosalicylates thermal decomposition [35]. It is impossible to identify the intermediate solids obtained because they are very unstable. The gaseous products of thermal decomposition are probably water, CO_2, gaseous alcohols and hydrocarbons as in the case of thermal decomposition of lanthanide(III) vanillates [36].

Supplementary material

Crystallographic data for the structure reported in this article have been deposited with the Cambridge Crystallographic Data Center as supplementary publication No. CCDC 611099. Copies of the data can be obtained free of charge from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336 033; Email: deposit@ccdc.cam.ac.uk).

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