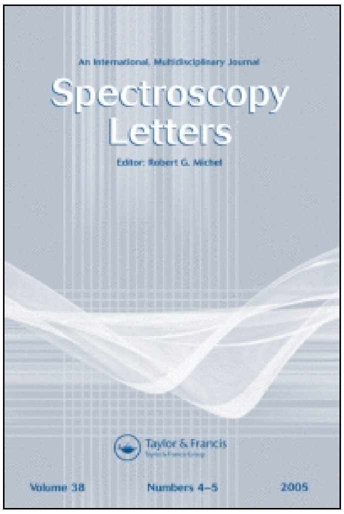

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Vibrational Assignment for Iron (III) Schiff Base Complexes

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VIBRATIONAL ASSIGNMENT FOR IRON (III) SCHIFF BASE COMPLEXES

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Key words: Infrared and Raman spectra, Iron(III) Schiff Base Complexes, Structure.

Abstract

A complete vibrational assignment of the i.r. and Raman bands of several substituted Schiff bases and their Fe(III) complexes has been done. On this basis, a tentative type of structure for complexes is proposed . The influence of the electron donor-acceptor characteristics and size of substituents on the structure of ligands and complexes is discussed.

INTRODUCTION

Few works dealing with spectroscopical and structural studies on iron (III) Schiff base complexes have been performed in the last three decades [1]; to our knowledge no one concerns a complete vibrational assignment. Recently ,we have sinthesized complexes of the type Fe(5-bromosalicylaldehyde-Y-aniline)Cl_2

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with Y= 2,4-diF (1), 2,4-diCl (2), 2,4-diBr (3), 2-CH_3,4-Br (4), 2,5-diCH_3 (5) and 2,4-diOCH_3 (6) and we have decided to accomplish a complete vibrational assignment in order to infer about their structural type; this because our attemps to obtain X-Ray data were unsuccesful. On this basis we have also considered to study the influence that the size and electron donor-acceptor characteristics of substituents have on the structure of ligands and complexes.

EXPERIMENTAL

Ligands and complexes were synthesized by using general methods [2]. The i.r. spectra were registred in KBr mulls by using a Bruker IFS 88 and IFS 113V spectrophotometers in the region 4000 to 400 cm^{-1} and 500 to 100 cm^{-1} respectively. The Raman spectra were scanned from 100 to 3500 cm^{-1} on a Bruker FRA 106 mounted on an IFS 66 FT-IR optical bench.

The vibrational assignment of ligands 1, 4 and 6 and of their corresponding complexes is collected in Table 1. These compounds were selected from the series since they show extreme spectral behaviour arising from the electron donor-acceptor and size characteristics of substituents.

RESULTS AND DISCUSSION

The complex formation manifests itself by the existence of the OH vibrations (ν(OH) and δ(OH)) only in the spectra of ligands, and the ν(Fe-O), ν(Fe-N) and chelate ring modes appearing only in the spectra of complexes. The coordination is also accompained by spectral shifting mainly of the ν(N=C), ν(Ar-N), ν(Ar-C), ν(Ar-O), ν(C-H)azomethine and ν (C-

Table 1. Observed infrared and Raman frequencies (cm^{-1}) and vibrational assignment for ligands (L) 1, 4 and 6 and the corresponding iron(III) Schiff base complexes (C).

	1		4		6		
	ir. Rel. Int.*	Raman	ir.	Raman	ir.	Raman	Assignment
L	3420 b,w		3420 b,vw		3420 b,vw		ν(OH)
C	3400 b,w		3415 b,vw		3420 b,vw		Water impurity
L	3200-2900 vvw		3200-2800 vw	3200-2800 w	3200-2800 v		ν(CH)
C	3200-2900 vw		3200-2800 vw	3200-2800 w	3200-2800 v		"
L	1620 s		1610 s	1612 s	1617 s	1618 s	ν(C=N)
C	1637 s	1641 s	1628 s	1639 s	1630 s	1638 s	"
L	1596 s	1604 s	1582 w	1580 w	1595 s	1598 s	$\nu(C_1C_6)_{arom.}$
C	1610 s	1599 s	1590 m	1592 s	1600 s	1603 s	"
L					1580 m	1582 s	$\nu(CC)_{arom.}$
C				1572 sh	1582 sh		"
L	1560 m,b	1562 s	1553 m	1556 s	1560 vw	1562 s	$\nu(CC)_{arom.}$
C	1532 s	1535 m	1532 s	1540 m	1530 s	1537 w	"
L	1500 s	1502 m	1475 s		1502 s	1503 m	$\nu(CC)_{arom.}$
C	1500 s	1507 m	1470 s	1483 m	1502 s	1508 w	"
L	1475 s	1472 m	1465 sh	1470 w	1475	1475 m	$\nu(CC)_{arom.}$
C	1475 s		1460 sh		1470	1466 w	"
L			1480 sh		1455 w		δ(Ar-CH$_3$)
C			1480 sh		1455 sh,w	1452 sh.	"
L					1432 w	1435 b,m	ν(O-CH$_3$)
C					1438 w		"
L			1428 vvw	1428 m			$\nu(CC)_{arom.}$
L	1432 b,w	1434 mult.	1450 b,w	1452 w	1414 b,w		δ(OH)
L	1382 b,w		1390 b,w	1398 sh.	1378 b,m	1379 vvw	ν(ArN)
C	1375 m	1381 m	1374 m	1381 s	1375 m	1380 s	"
L	1350 m	1350 w	1347 s	1346 w	1345 s		$\nu(CC)_{arom.}$
C	1330 m	1336 m	1328 b,m	1330 m	1342 s	1346 b,m	"
L		1315 vw	1310 vw	1314 w			δ(CH)
L	1293 sh	1295 w	1277 s,d		1302 s	1308 s	δ(CH$_a$)
C	1300 w	1291 m	1276 sh,m	1284 sh.	1297 s	1303 m	"
L	1268 sh	1260 m	1277 s	1268 vw	1265 m	1265 m	ν(O-Ar)+δ(OH)
C	1280 sh	1260 sh.	1267 s	1271 m	1268 s	1280 b,m	"
L		1236 m	1235 b,vw	1232 vw			Br sensitive
C	1235 vw		1240 w	1237 m			"
L	1217 b,m	1219 vw	1220 w			1230 b,m	ν(C-Ar)
C	1213 s	1215 w	1230 m		1225 s		"
L					1203 s	1212 sh	ν(Ar-OCH$_3$)
C					1205 s		"
L			1200 b,w	1201 m			ν(Ar-CH$_3$)
C			1200 m,d	1203 m			"
L	1171 m	1170 s	1170 s,d		1175 s	1181 m	ν(O-Ar)+δ(OH)
C	1157 s	1160 m,s	1160 s,d	1168 m	1170 s	1173 m	"
L	1143 m	1148 vvw	1170 s	1172 m	1155 m	1174 m	δ(CH)
C	1140 sh		1160 s	1168 m	1165 sh		"
L	1100 m	1096 vw	1120 m	1125 w	1130 m	1128 m	δ(CH)
C	1095 m	1095 w	1110 m	1114 m	1120 m	1123 m	"
L		1078 vw	1080 vw	1086 vw			ν(Ar-Br)
C			1090 vw	1088 m			"
L	1074 w	1074 vw	1070 w		1068 w	1073 vvw	ν(Ar-Br)
C	1074 vw	1072 vw	1080 w		1068 vw		"

(continued)

(Table 1 continued)

L	1025 vvw		1025 w		1033 s	1032 vvw	δ (CH)
C	1014 m	1013 w	1018 w	1017 m	1030 s		"
L			1005 vw,sh	1000 vvw			breathing
C			995 w				"
L	965 w	967 vw	957 w	953 vvw	960 m	965 vw	$\rho(CH_a)$
C	1003 m		1015 m		1008 s		"
L	945 vvw		950 sh,vvw		955 vw		ρ(CH)
C	938 vvw		940 vw		945 vw		"
L	910 w	916 vw	912 w	912 vw	920 b,vw	910 b,v	ρ(CH)
C	962 s		912 w	920 vw	913 m	921 vw	"
L					905 sh		breathing
C		888 vvw		880 vw			"
L	870 m		870 s	871 vvw	890 b,m		ρ(CH)
C	868 sh		872 m				"
L	850 sh		868 sh		865 w		ρ(CH)
C	862 s,asym.		868 m		863 m		"
L	844 m	846 w	850 sh		843 m	842 m	ρ(CH)
C	850 m		852 m	855 w	845 m		"
L	816 m		818 m	815 w	815 s		ρ(CH)
C	828 m	832 w	820 d,m	821 vvw	825 m	834 vw	"
L	802 s		818 s		789 s	782 w	ρ(CH)
C	808 m		800 m	793 vvw	805 m	805 vw	"
L	775 w	777 w	773 w	774 vw	773 w	777 w	ρ(CH)
C	798 m	797 vw	785 m		780 m		"
L	726 w	725 w			721 w	724 w	ρ(CH)
C	726 w	726 w		727 vvw	719 w		"
L		668 vw	677 sh			679 w	Br sensit.
C			677 m				"
C	668 m	671 w	682 d,m	683 w	669 m	667 vw	Metal chelate ring
L	638 w	630 vvw	631 m	628 m	631 m	629 vvw	Br$_{ortho}$ sensit.
C	637 w	631 w	637 m	642 m	630 m	633 vw	"
L	609 w		618 vw		605 w	602 w	δ (ring)
C	609 w		615 vw	616 sh	598 w		"
L	595 vvw	599 vvw					F sensitive
C	595 vvw	596 vvw					"
L				565 vw		562 w	ring def.
C	548 m,asym.		560 m	560 w	560 b,m		"
L	550 w		542 m	555 vvw	548 w	560 w	χ (ring)
C	543 asym.,b,m		542 m	531 vvw	535 m		"
L	512 m	511 vvw					F sensitive
C	508 w						"
C	510 b,m		491 m		515 b,m		Metal chelate ring
L	500 m			510 vw	500 b,vw		γ (ring)
C	496 sh				505 b,vw		"
L	484 w		470 m	482 m	480 m	480 b,w	γ (ring)
C	481 m	473 m	472 m	472 sh	492 m		"
C	470 sh.		445 w,sh	447 b,m	475 m		ν (Fe-O)
L	462 w		458 vvw	461b,vw	462 vw		χ (ring)
C	445 vw	446 vw	437 w		441 vvw		"
L		432 vw	435 vvw		417 w	420 d,w	δ (skelet.)
L			421 vvw		443 d,vw		χ (ring)
C					424 vvw		"
C	402 s	410 vw	401 m	405 vw	388 m	**387 b,w	ν (Fe-N)
L	400 vw	400 vw					δ (Ar-F)
C	381 m	382 vw	383 m	375 sh	383 m	387 b,w	ν (Fe-Cl)$_t$

(continued)

(Table 1 continued)

L			366 w				CH_3 sensit.
C	359 m		350 m		359 m		ν_s (Fe-Cl)$_t$
L	345 m	341 m					ρ (Ar-F)
C	346 m	340 b,m					"
L			340 m	340 m	342 vvw	335 vw	Br sensit.
C	320 m	319 w	328 m	335 d,m	335 m	336 w	"
L	294 vvw	296 vw			291 vvw	300 vw	skelet. def.
C	288 sh		292 w		302 vvw		"
L					295 b,vw	295 b,w	ρ (Ar-OCH_3)
C	279 d,w	283 b,w	281 d,w	280 b,m	277 w		ν (Fe-Cl)$_b$
L					270 w		δ skel.
L	260 w	262 w	264 b,w	264 b,w	262 sh,vw	262 w	τ (Ar-N)
L	239 vw	235 b,vw			225 vw		i.p. skel. def.
C	240 vw		242 w	240 w	235 vw	245 v,w	"
L	225 w		216 b,w	218 d,w	215 vw		skel. def.
C	225 vvw	226 b,w	216 vw		208 vw		"
L	199 w			197 vw		200 b,w	skel. def.
C	191 w	184 d,w					"
C	176 w		192 d,w	196 w			chelat. ring def.
L			169 d,w	166 vw	171 vw	160 vw	skel. def.
C			175 m	170 b,m	167 b,m		"
L	142 vw	144 m	140 vvw	139 m	142 w		δ (Ar-N)
L					132 w	130 m	skel. def.
C	138 vvw	149 w	130 vvw	140 m	131 d,w		"
C	119 m		121 b,m		121 w		bridge ring def.
L	109 vw				114 vw		skel. def.
C	105 vw		108 w				"

* s: strong, w : weak, vw: very weak, sh: shoulder,
b: broad, d : double.

** used twice.

C)aromatic modes. Almost all the ligands and the complexes vibrations are shifted by the substitution effect.

Vibrational assignment and structural type for complexes

The broad and weak band observed in the ligands at about 3400 cm^{-1}, has been assigned to an hydrogen bonded ν(O-H) mode of an intramolecular bond between the hydroxilic hydrogen and the N atom. The ν (CC) aromatic modes are currently placed between 1600 and 1300 cm^{-1}; at least 8 of them were assigned. Only the band at about 1600 cm^{-1} shows a frequency shift to higher energy by complexation; this band could be ascribed to the C_1- C_6 bond of the benzylidene ring.

The assignment of the ν(C=N) (1630 cm^{-1}), ν(Ar-N) (1374 cm^{-1}) and ν(Ar-C) (1215 cm^{-1}) has been performed in agreement with similar propositions for several related Schiff bases [3].

The strong and broad bands at $\sim$1260 cm^{-1} and at 1180 cm^{-1} in the spectra of ligands were assigned to a combination of δ(OH) and ν(Ar-O) modes in agreement with that given by Socrates [4] in phenols. Unfortunately, because of the extreme coupling with other modes in this region we have not observed simplification of these bands by complexation; although for the band at 1180 cm^{-1} we verify a frequency shift to lower energy by chelation.

No band dissapears by complexation about 1280 cm^{-1} as described by Bailar et al [5] in metal complexes with N, N'-bis(salicylidene)-1,1-(dimethyl)ethylene-diamine. The band at 1440 cm^{-1} which dissapears by chelation is assigned to the δ(OH) mode following Socrates [4].

The chelate ring deformations are expected near 1250 cm^{-1}; we have not distinguish these modes because of a strong overlapping of several bands observed in that region. Moreover these modes generally display very low relative intensities [6].

We assign the bands at 1290 and 960 cm^{-1} to the in- and out-of-plane deformations of the azometine hydrogen ($\delta(CH)_a$ and $\rho(CH)_a$) respectively, following Meic et al. [7] in stilbene. The $\delta(CH)_a$ mode shows a strong frequency shift by substitution and its energy is practically unmodified by chelation. The $\rho(CH)_a$ band displays a perfect opposite behaviour.

Following Bailar et al. [5] we assign the bands at $\sim$670 and 500 cm^{-1}, appearing only in the spectrum of complexes, to the metal chelate ring vibrations.

The assignment of the ν(Fe-O) and ν(Fe-N) modes is based on reported data of related molecules [8-10] and on the current observation that the ν(M-O) > ν(M-N) [5,11].

The (Fe-Cl)$_{terminal}$ vibrations were ascribed to the strong absorptions at $\sim$350 and 380 cm^{-1} following reported data [12,13]. We assign the former band to the symmetric mode while the second one should correspond to the asymmetric mode. The band at about 280 cm^{-1} has been ascribed to a ν(Fe-Cl)$_{bridge}$ mode; this assignment is supported by the trends observed by Nakamoto [12] where the ν(M-X)$_b$ < ν(M-X)$_t$ and by the observed difference between the relative intensities of their ir. and Raman bands.

The chelate and bridge ring deformations should be actives below 300 cm^{-1} [14].

Other bands were attributed following current assignments.

The identification of the ν(Fe-N), ν(Fe-Cl)$_t$, ν(Fe-O) and chelate ring vibrations, and the fact that we have not observed the Fe-O-Fe vibrations between 850-780 cm^{-1} [15] suggests that the coordination does not involve the donor center N_2O_2 as in related complexes [5,16], but it goes through a coordination sphere composed by N, O and Cl. The ν(Fe-Cl)$_b$ and the bridge ring deformation modes suggest a dimeric structure for the complexes. This analysis and sterical considerations allow us to propose for the complexes the structural type shown in Fig. 1.

Substituent effect on the structure of ligands and complexes

It has been observed in related Schiff bases that the tendency to a whole co-planarity is accompained by an increasing

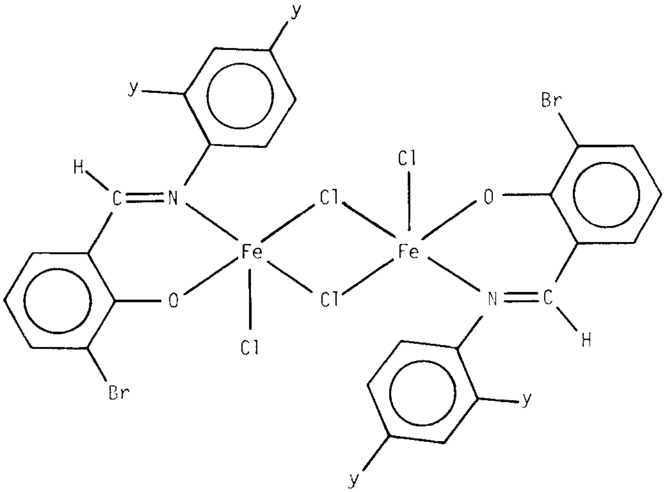

Fig. 1. Type of structure for the Fe(5-Bromosalicylaldehyde-Y-aniline)Cl_2 complexes 1-6.

	1	2	3	4	5	6
(Y)	2,4-diF	2,4-diCl	2,4-diBr	2-CH_3,4-Br	2,4-diCH_3	2,4-diOCH_3

of the π-character on the Ar-N bond and a weakness of the N=C bond [3a]; on the other hand, the benzylidene ring has essentially free rotation (barrier ca. 0.4 Kcal/mol) [17]. The observed trend for the ν(C=N) and ν(Ar -N) frequencies of ligands ($\nu L_1 \sim \nu L_6 < \nu L_4$), see Table 1, could be explained in terms of two competive effects: the steric hindrance and the electron donor-acceptor characteristics of substituents. In fact, the F atom having a high electron acceptor tendency and a small size in comparison to the -OCH_3 group, will produce a decreasing of the π-character of the Ar-N bond; as a consequence, it will favour a lesser non-coplanarity of the molecule. The steric hindrance caused by the metoxi group constraints the anilinic ring to adopt also a non-coplanar structure for the molecule. Extreme frequencies of the ν (CN) and ν (Ar-N) modes in ligand 4 are readily explained in terms of the intermediate characteristics of size and electron donor-acceptor tendency of the CH_3 group relative to the -F and $-OCH_3$ substituents. Hence this molecule will display a lesser non-planar structure in comparison to 1 and 6.

The high value of the ν (Ar-C) frequency (~1220 cm^{-1}) in all ligands in comparison to the one in the non-planar molecule N-benzylideneaniline (1190 cm^{-1}), suggests a reinforcement of the π-character of the Ar-C bond. On the basis of this fact and in terms of the strong interaction between the hydroxilic hydrogen and the N atom [18], allow us to infer that in our ligands a lesser non-planar structure of the benzylidene ring should be favoured.

The trends to the co-planarity of the benzilidene ring and the opposite tendency of the aniline ring in ligands is accompained by a redistribution of the π-electron around the benzilidene ring and by a sp^2 hybridization of the N atom. Thus, the ν(Ar-O) frequency should be higher than in a free Ar-OH (~1240 cm^{-1}) [4] and the $\delta(CH)_a$, $\rho(CH)_a$, ν(Ar-O) and $\nu(C_1C_6)$ modes should be sensitive to the substitution, as it is observed.

The complex formation is accompained by an increase of the ν(C=N) and $\nu(C_1C_6)$ frequencies, and a decrease of the ν(Ar-N) and ν(Ar-O) frequencies. The ν(Ar-C) shows a decreasing in energy in complexes 1 and 6 but an increasing in 4. The observed increase in frequency when passing from complex 4 to 1 and 6, could be interpreted in term of a major trends to the non-coplanarity of the aniline ring conferred by substituents -F and $-OCH_3$. This set of results suggests that the complexation is accompained by the adoption of a lesser coplanar structure for the aniline ring, a better sp^2 hybridization of the N atom and a decreasing of the π-electron delocalization on the chelate ring.

Since the substituents -F and $-OCH_3$ confer to the systems a bigger non coplanarity in relation to the $-CH_3$ group, we should expect the formation of complexes 1 and 6 to be more favoured than the formation of complex 4 . This proposition is supported by the trends observed in the ν(Fe-O) values for the distinct complexes. In these terms and on the basis of the ν(Fe-N) and ν(Fe-O) frequencies we infer that the chelation goes preferently through the Fe-O bond.

CONCLUSIONS

The substituent effect on the stretching modes of the bridge and chelate rings support the proposed assignment for the corresponding deformations. In particular, it is possible to infer that the bridge ring vibrations are practically unaffected by substitution.

The ligand structures are characterized by a non-coplanarity of the aniline ring arising from the influence of the electron donor-acceptor properties and the size of substituents in para position. The trend to the coplanarity of the benzylidene ring is conferred by the intra-molecular interaction between the acidic hydrogen of the OH group and the availability of the lone pair electrons of nitrogen.

The complexation is accompained by an increasing of the tendency to a lesser co-planarity of the aniline ring and an increasing of the coplanarity of the benzylidene ring.

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