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THE SO_2 STRETCHING VIBRATIONS IN SOME METAL SACCHARINATES: SPECTRA–STRUCTURE CORRELATIONS

Key words: Metal saccharinates, SO_2 stretching vibrations,
spectra–strucure correlations

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ABSTRACT

The infrared spectra of a series of metal saccharinates (those of Na, Mg, Mn Fe, Co, Ni, Zn, Cd, Pb as well as mercury(II) saccharinate and chloromercury(II) saccharinate*) were studied in the region of the antisymmetric and symmetric SO_2 stretching vibrations [$\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ hereafter]. The results concerning the number of bands attributable to the abovementioned vibrations, their frequencies and intensities were correlated with the crystallographically determined type of metal–to–ligand bonding, on the one hand, and the number of the structurally different SO_2 groups, on the other.

It was shown that, irrespective on whether the sulphonyl oxygen atoms do or do not participate in bonding to the metal atom and/or in hydrogen bonding and irrespective on the type of the metal–to–saccharin bond (from covalent to purely ionic), the frequencies of both $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ modes are lower for the metal saccharinates than for saccharin itself, the effect being more pronounced in the case of the $\nu_{as}(SO_2)$ bands.

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*The last of the above mentioned compounds will be referred to as the saccharinate of ClHg.

As expected, the appearance of the spectra in the SO_2 stretching regions is rather strongly dependent on the values of the O–S–O angles. Thus, two rather different types of O–S–O angles exist in the structure of the *covalently bonded* saccharinate of mercury and of the *ionic* lead saccharinate (the values of these angles are 118.7 and 111.8 ° and 120.4 and 111.8 ° respectively) and two pairs of $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ bands are present in the spectra of these two compounds. On the other hand, the existence of only one type of SO_2 groups in the saccharinates of Mn and ClHg or the presence of SO_2 groups with very close O–S–O angles in the saccharinates of Na and Mg is manifested by the appearance of only a single pair of intense $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ bands in their spectra.

INTRODUCTION

Saccharin* [1,2-benzisothiazole-3(2*H*)-one 1,1-dioxide] reacts with various metals forming several types of metal saccharinates. Their structural characteristics are rather extensively investigated in the last few years [1–16].

From the comparison of the observed metal–to–ligand distances with the sum of the van der Waals radii it was shown that the bonding in the saccharinates of Hg(II) and ClHg(II) is mostly covalent [2,7], that in those of Na, K_2Na, Mg and Pb(II) it is purely ionic [8–10], whereas the M–N bond in the series of saccharinates with the general formula $[M(sac)_2(H_2O)_4]\cdot 2H_2O$ [M = Mn(II), Fe(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), V(II) and Cr(II)] is of the intermediate type [1,3–6]. It was also shown that the saccharinato ligands in the complexes of Mn(II), Fe(II), Co(II), Ni(II), Zn(II), Cu(II), V(II), Cr(II), Cd(II), Hg(II) and ClHg(II) coordinate to the central metal atom solely through their nitrogen atoms [1–7], while the carbonyl and sulphonyl oxygen atoms as well as nitrogen atoms [8–10] take part in the metal–to–saccharinate contacts in the saccharinates of Na, K_2Na and Pb(II). In Mg(II) saccharinate, however, the anions contact the central metal atom solely through their carbonyl oxygen atoms [8].

The analysis of the infrared spectra has shown that the carbonyl stretching frequencies are lower in the saccharinates than in saccharin itself

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* In what follows, saccharin will be abbreviated as H(sac), so that sac will denote either a saccharinate anion or a saccharinato ligand

[11]. The extent of this lowering depends on the type of metal–to–saccharinate contacts and is more pronounced in the case of the ionic metal saccharinates.

In order to get more information about the spectroscopic characteristics of various metal saccharinates (those of Na, Mg, Mn, Fe, Co, Ni, Zn, Cd, Pb, Hg and ClHg), we studied their infrared spectra in the region of the SO_2 stretching vibrations. The results of the correlation between the number of bands attributable to the antisymmetric and symmetric SO_2 stretchings as well as their frequencies and intensities, on the one hand, and the crystallographically determined type of metal–to–ligand bonding and the number of different SO_2 groups in the structure, on the other, are presented below.

EXPERIMENTAL

The studied compounds were prepared by standard methods. The infrared spectra were recorded on a Perkin–Elmer 580 spectrophotometer, using KBr pellets and Nujol mulls. The X–ray data were collected on a Philips PW 1100 four–circle diffractometer. Standard crystallographic programs were used for structure determination. Model normal–coordinate calculations were carried out using the programme KRAFT15 written in FORTRAN by Eriksson [17].

RESULTS AND DISCUSSION

Spectral and structural data

The infrared spectra of saccharin and the studied metal saccharinates in the region of the SO_2 stretching vibrations are shown in Figs. 1–4.

The approximate assignment of the ring stretching vibrations in the infrared spectrum of the $Pb(sac)_2 \cdot H_2O$ (and, accordingly, in the spectra of the rest of the studied metal saccharinates) based on the normal–coordinate analysis for potassium phtalimidate (PIMK) [18] and potassium tetrachlorophtalimidate (TCPIMK) [19] in the region from 1375 to 1130 cm^{-1} is given in Table 1.

The data concerning the frequencies of the antisymmetric and symmetric SO_2 stretching vibrations are given in Table 2 together with some

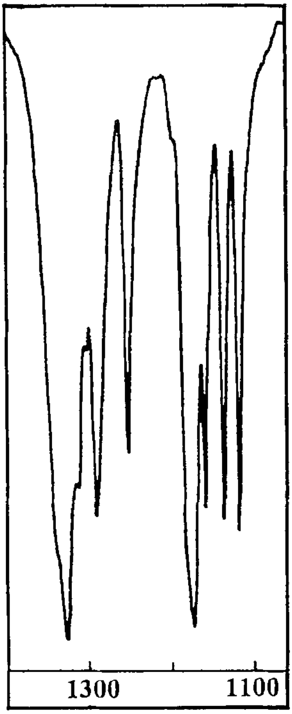

FIG. 1. Infrared spectrum of saccharin in the SO_2 stretching region.

pertinent crystallographic data. In fact, in Table 2, only the data concerning the S–O distances and the O–S–O angles within the SO_2 groups are summed up. Additional structural data concerning the metal–to–saccharin bond type and the participation of the sulphonyl oxygen atoms in bonding to the metal atom and/or in hydrogen bonding are given in Table 3 whereas full details about the structural characteristics of saccharin and the studied metal saccharinates (the compounds of Na, Mg, Mn, Pb, Hg and ClHg) are given in references 1, 2, 7, 8, 10, 12 and 13.

The predicted values for the frequencies of the stretching SO_2 modes based on the model normal–coordinate analysis for the $NS(O_2)C$ fragment of the saccharinate anion in $Pb(sac)_2 \cdot H_2O$ as well as the experimentally obtained frequencies for these modes are shown in Table 4.

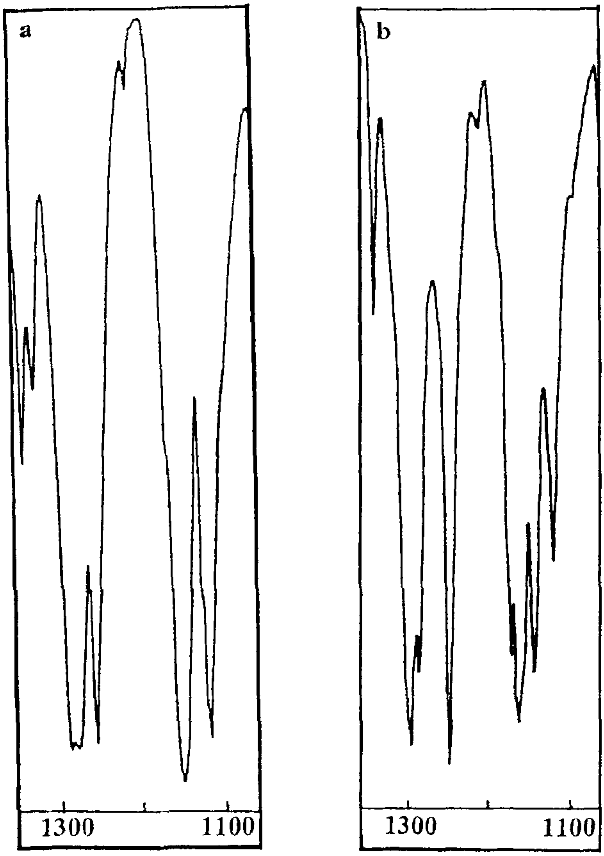

FIG. 2. Infrared spectra of $Mn[(sac)_2(H_2O)_4]\cdot 2H_2O$ (a) and $ClHg(sac)$ (b) in the SO_2 stretching region.

Spectra–structure correlations

The assignment of the bands originating from the SO_2 stretching vibrations in the studied metal saccharinates as well as in saccharin itself was accompanied by some difficulties concerning the presence of additional bands in the same spectral region. More precisely, according to the normal-coordinate analysis for phtalimide, potassium phtalimidate and its tetrachloro analogue, at least four to five [18,19] bands due to the ring stretches should be present in the region 1375 - 1130 cm^{-1}, where the bands origina-

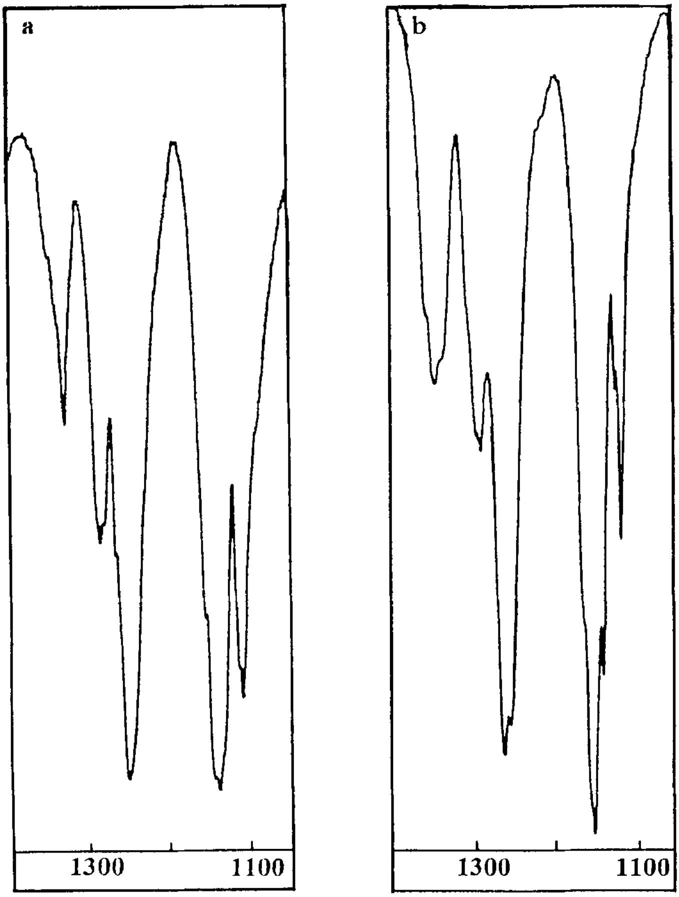

FIG. 3. Infrared spectra of $Na_3(sac)_3 \cdot 2H_2O$ (a) and $Mg(sac)_2 \cdot 7H_2O$ (b) in the SO_2 stretching region.

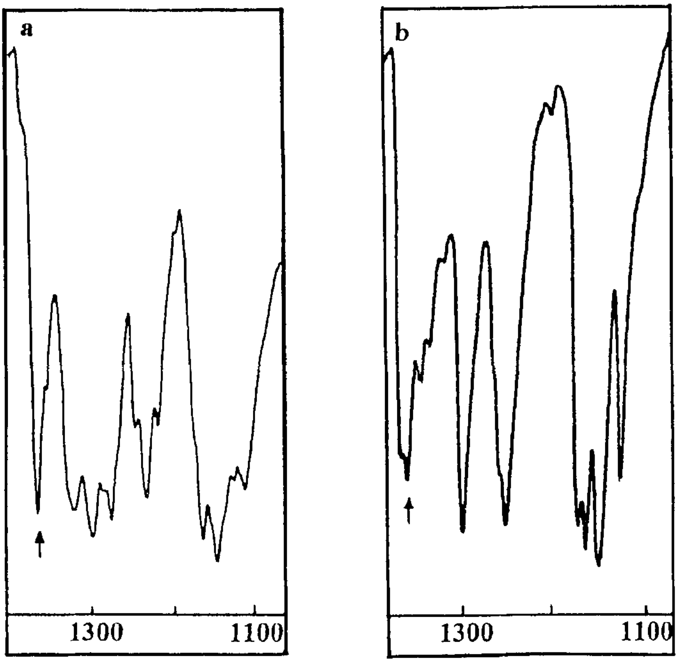

FIG. 4. Infrared spectra of $Hg(sac)_2$ (a) and $Pb(sac)_2{\cdot}H_2O$ (b) in the SO_2 stretching region (the arrows denote Nujol bands).

ting from the SO_2 stretching vibrations are also expected to appear [20–23] (see Table 1) *. The benzenoid ring stretching bands, however, are found to be much sharper compared to those arising from the sulphonyl stretches.

On the other hand, the normal–coordinate analysis for dimethyl sulphone and dimethyl sulphone–d_6 [22] has shown that the $\nu(SO_2)$ modes are rather good group vibrations. Similar conclusions could be drawn from our model calculations for the N–$S(O_2)$–C fragment of the saccharin anions in

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* Although the five-membered rings of phtalimide and saccharin are not identical, we believe that the results of the normal-coordinate analysis for potassium phtalimidate and potassium tetrachlorophtalimidate could be used as a guide for an approximate assignment of the ring stretching vibrations not only in the two above-mentioned compounds but also in the infrared spectra of the metal saccharinates.

TABLE 1

Assignment of the ring stretching vibrations in the 1375–1130 cm^{-1} region of the infrared spectra of Pb(sac)$_2$·H$_2$O*, potassium phtalimidate (PIMK) [18] and potassium tetrachlorophtalimidate (TCPIMK) [19]

PIMK [18]	TCPIMK [19]	Pb(sac)$_2$ · H$_2$O	Assignment
1375 s	1353 vs	1363 s 1351 s	ring stretch
	1318 vs	1334 w	ring stretch
1300 s	1262 m	1258 sh	ring stretch
1185 s	1192 m	1204 w	ring stretch
1135 s	1131 s	1159 s	ring stretch

s - strong; vs - very strong; m - medium; w - weak; sh - shoulder

* In addition to the bands originating from the ν(SO$_2$) modes, about 5 to 8 bands with similar frequencies also appear in the infrared spectra of all other studied metal saccharinates in the region of the benzenoid ring stretching vibrations.

the structure of Pb(sac)$_2$·H$_2$O (see below). Therefore, the strongest bands in the mentioned spectral region of the studied metal saccharinates were attributed to the corresponding antisymmetric and symmetric SO$_2$ stretching vibrations (see references 14–16 and Table 2).

The analysis of the structural and spectroscopic data given in Table 2 and Table 3 shows that, irrespective on the type of the metal–to–saccharin bond (from covalent to purely ionic) and irrespective on whether or not the sulphonyl oxygenatoms participate in bonding to the metal atom and/or in hydrogen bonding, the frequencies of the antisymmetric and symmetric SO$_2$ stretching vibrations in metal saccharinates are lower than in saccharin itself. Thus, the frequencies of the ν_s(SO$_2$) modes in the studied metal saccharinates range from 1173 to 1142 cm^{-1} and are from 7 to 38 cm^{-1} lower than the corresponding mode in saccharin (1180 cm^{-1}), while the lowering of the ν_{as}(SO$_2$) frequencies (1310–1255 cm^{-1} as compared to the value of 1335 cm^{-1} in saccharin) in metal complexes is more pronounced and range from 25 to 80 cm^{-1}. Such a behaviour of the ν(SO$_2$) modes is in agreement

TABLE 2

Some Structural and Spectroscopic Characteristics Concerning the SO_2 Groups in Saccharin and Various Metal Saccharinates

Compound	*R*(S–O)/pm	∠(O–S–O)/°	Frequency data/cm^{-1}					
			ν_{as}	ν_s	Δ	Δ'	Δ''	ν_s/ν_{as}
Saccharin Ref. 14			1335	1180	155			0.884
Ref. 12	142.7 142.8	117.4						
Ref. 13	142.9 140.9	117.7						
Metal saccharinates								
Hg(II) Refs. 2,14,15	143.5 144.3 142.2 143.1 141.2	116.7 117.2 118.7	1310	1155	155	25	25	0.882
	141.8 142.2 143.4	111.8	1286	1173	113	49	7	0.912
ClHg(II) Refs. 7,14,15	143.6 143.3	117.2	1300	1163	137	35	17	0.895
Pb(II) Refs. 10,16	143.9 143.3	111.8	1255	1167	88	80	13	0.930
	146.3 142.9	120.4	1308	1142	166	27	38	0.873
Mn(II) Refs. 1,14,15	143.7 144.5	116.1	1288	1155	133	47	25	0.897
Mg(II) Refs. 8,14,15	144.5 144.9 142.2 144.5	114.4 114.4	1265	1155	110	70	25	0.913
Na Refs. 8,14–16	144.3 145.0 144.6 145.6 144.5 145.5	112.9 113.9 114.0	1260	1150	110	75	30	0.913

$\Delta = \nu_{as} - \nu_s$; $\Delta' = \nu_{as}[H(sac)] - \nu_{as}[M\text{-}sac]$; $\Delta'' = \nu_s[H(sac)] - \nu_s[M\text{-}sac]$

TABLE 3

Structural Data Concerning the Type of Metal–to–Saccharin Bonds and the Participation of Sulphonyl Oxygen Atoms in Bonding to the Metal Atom and /or in Hydrogen Bonding in the Studied Metal Saccharinates [1,2,7,8,10]

Compound	Type of metal–to–saccharin bond	Participation of O_{SO_2} in the	
		metal coordination sphere	hydrogen bonding
$Hg(sac)_2$	covalent	no	no
$ClHg(sac)$	covalent	no	no
$[Mn(sac)_2(H_2O)_4] \cdot 2H_2O$	intermediate	no	yes
$[Mg(sac)(H_2O)_5](sac) \cdot 2H_2O$	ionic	no	yes
$Pb(sac)_2 \cdot H_2O$	ionic	yes	yes
$Na_3(sac)_3 \cdot 3H_2O$	ionic	yes	yes

TABLE 4

The predicted and experimentally found values for the frequencies of the stretching SO_2 modes in $Pb(sac)_2 \cdot H_2O$

∠(O–S–O) / °	Frequency data /cm^{-1}				Reference
	ν_{as}	ν_s	$\nu_{as} - \nu_s$	ν_{as}/ν_s	
120.4	1297.1	1126.3	170.8	0.87	This work
	1308	1142	166	0.87	Ref. 16
111.8	1270.8	1169.4	101.4	0.92	This work
	1255	1167	88	0.93	Ref. 16

with the observations [24] that the frequency of the antisymmetric SO_2 stretching vibration is more sensitive to external influences.

As seen from Table 2 and Figs. 1–4, however, the shift of the $\nu(SO_2)$ modes in metal saccharinates to lower values varies on going from one compound to another. The lowering mainly depends on the type of the metal–to–saccharinate bonding and is more pronounced in the case of the ionic compounds. The influence of the participation of the sulphonyl oxygens in the hydrogen bonding as well as in the coordination sphere around the metal atoms, however, should not be excluded.

The spectra of the covalently bonded $Hg(sac)_2$ [2] and the ionic $Pb(sac)_2 \cdot H_2O$ [10] in the region of the SO_2 stretches, however, do not follow the above mentioned pattern. Namely, contrary to the appearance of single strong bands originating from the $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ modes in the spectra of the saccharinates of the ClHg, Mn(II), Fe(II), Co(II), Ni(II), Zn(II), Cd(II), Mg and Na, the infrared spectra of the saccharinates of Hg(II) and Pb in the region of the $\nu(SO_2)$ modes behave in a very similar fashion and are characterized by the presence of *two pairs* of bands (see Figs. 2–4, Table 2 and references 14, 15 and 16). It should be noted that such a behaviour is consistent with the crystallographic data for the studied metal saccharinates. As can be seen from Table 2, namely, the SO_2 groups in the structures of the saccharinates of the manganese [1] (and its isomorphous analogues) as well as in the chloromercury compound [7] are equivalent and the values of the structurally different O–S–O angles in sodium saccharinate [8] (112.9, 113.9 and 114.0 °) and magnesium saccharinate [8] (114.4 and 114.7 °) are very close to each other. On the other hand, two rather different values for the O–S–O angles are present in the structure of lead saccharinate [10] (120.4 and 111.8 °) and, similarly, the O–S–O angles present in the structure of mercury(II) saccharinate [2] (116.7, 117.2, 118.7 and 111.8 °) can be classed into two different groups. From the data presented above it is evident that the $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ frequencies in the spectra of the studied metal saccharinates are considerably influenced by the values of the O–S–O angles in the respective structures.

In order to test (at least semiquantitatively) the effect of the O–S–O angle on the frequency of the antisymmetric and symmetric SO_2 vibrations, we carried out a model normal–coordinate analysis for the $N{-}S(O_2){-}C$ fragment of the saccharinate anions in the structure of $Pb(sac)_2 \cdot H_2O$. In the employed model, only the sulfur and oxygen atoms were allowed to vibrate whereas the position of the carbon and nitrogen atoms was held fixed. The initial values for the S–O force constants were taken from reference 22 but were adjusted to obtain a reasonable agreement between the observed and calculated wavenumbers. The experimentally determined values for the relevant distances and angles were also taken into account. The final values for the force constants used in the calculation were: *F*(S,C) = 7.128, *F*(S,N) = 5.385, *F*(S,O_1) = 9.0500, *F*(S,O_2) = 9.0520 mdyn Å^{-1} for the fragment with an O–S–O angle of 111.8 ° and *F*(S,C) = 7.100, *F*(S,N) = 5.178, *F*(S,O_1) = 9.0420, *F*(S,O_2) = 9.0548 mdyn Å^{-1} for the N–S(O_2)–C group with an O–S–O angle of 120.4 °. As mentioned before, the calculated PED values indicated (at least in the framework of the employed model) that the SO_2 vibrations are good group frequencies.

The agreement between the experimentally determined and the estimated frequencies could be considered as satisfactory although it is far from being perfect (cf. Table 4). Furthermore, the calculations lead to the conclusion that the presence of two SO_2 groups in the structure of $Pb(sac)_2 \cdot H_2O$ having rather different O–S–O angles (120.4 and 111.8 °) should be accompanied by the appearance of two pairs of bands originating from the stretching SO_2 vibrations. The $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ modes of the saccharinate with a larger O–S–O angle (120.4 °) are expected to give rise to bands with the highest and the lowest frequency whereas the corresponding vibrations of the saccharinate characterized by a smaller O–S–O angle should manifest themselves with bands at intermediate frequencies. Hence, it is reasonable to assume that the bands at 1308 and 1142 cm^{-1} in the spectrum of $Pb(sac)_2 \cdot H_2O$ should be assigned to the $\nu_{as}(SO_2)$ and $\nu_s(SO_2)$ modes of the former and those at 1255 cm^{-1} and 1167 cm^{-1} to the corresponding modes of the latter type of saccharinate anions (see Fig. 4b).

Keeping in mind the pronounced effect of the differences in the values of the O–S–O angles on the SO_2 stretching frequencies, the general conclusion from this work could be that, contrary to the lowering of the carbonyl stretching vibrations the frequencies of which mainly depend on the type of the metal–to–saccharin bond, the position of the bands arising from the SO_2 stretching vibrations can not be used to make far–reaching conclusions about the type of metal–to–ligand bonding in metal saccharinates.

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