

amine fragments are practically insensitive to the nature of the Li^+ , Na^+ , K^+ , NH_4^+ , and Mg^{2+} cations. The Ni^{2+} cation is an exception.

2. The established differences in the IR spectra of the solid samples and the Raman spectra of solutions of the nickel salt and other salts of N,N'-dinitroethylenediamine can be explained by difference in the coordination of the cations.

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VIBRATIONAL SPECTRA OF FLUORINE-CONTAINING AZOXYBENZENES

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We previously showed on the basis of data from Raman spectra that the introduction of fluorine atoms into the benzene rings of aromatic azomethines and azobenzenes has a weak influence on the frequencies of the stretching vibrations of the bridging groups bonded to them and, at the same time, significantly alters the intensity of the $\nu_{\text{C=N}}$ and ν_{s} vibrations of the benzene rings [1, 2]. The intensity changes were attributed to the difference in the effectiveness of the conjugation of the nonbonding electron pair of the nitrogen atom of the azomethine group with the π -system of the benzene ring [2]. In order to ascertain the generality of the laws revealed, it would be useful to study the influence of the number and arrangement of the fluorine atoms in the benzene ring on the frequencies and intensities of the stretching vibrations of other unsaturated bridging groupings. In the present work we examined the influence of the introduction of fluorine atoms into aromatic fragments on the spectroscopic characteristics of bridging azoxy groups. For this purpose we obtained and analyzed the IR and Raman spectra of azoxybenzene (I) and some of its fluorinated derivatives: 4,4'-difluoro- and 3,3'-difluoroazoxybenzene (II and III), 2,2',4,4',-tetrafluoro- and 2,2',5,5'-tetrafluoroazoxybenzene (IV and V), 2,2', 3,3', 4,4', 5,5'-octafluoroazoxybenzene (VI), and compounds of the type 4-XC₆F₄N=N(O)C₆F₄X-4' (VII) [where X = F (a), H (b), CH₃ (c), OCH₃ (d), CF₃ (e), and Br (f)], as well as samples labeled with ¹⁵N at both nitrogen atoms (compounds I', IV', VIIa', VIIb', and VIId', respectively). The enrichment with ¹⁵N amounted to 94-96%.

The vibrational (as a rule, IR) spectra of azoxybenzenes have already been discussed in [3, 4]. The Raman spectra of azoxybenzene, 3,3', 5,5'-tetrachloro-, 4,4'-dimethoxy-, and 4,4'-dihexyloxyazoxybenzene are presented in catalog [5]. However, the available data do not permit the confident interpretation of the stretching vibrations of the azoxy group. For example, the bands at 1315-1340 and 1450-1480 cm⁻¹ were assigned, respectively, to the symmetric and antisymmetric stretching vibrations of the azoxy group. In [6] the 840-890 cm⁻¹ range was indicated for the vibrations of the N=O group in methylated and halogenated (chlorinated and brominated) azoxybenzenes. An investigation of the IR and Raman spectra of I and its ¹⁵N- (at both nitrogen atoms of the azoxy group) and D-labeled derivatives permitted the assignment of the bands of the stretching vibrations of the azoxy group in the following manner [4]: $\nu_{\text{N=N}} = 1440$ ($\Delta\nu^{15\text{N}} = 30$ cm⁻¹), $\nu_{\text{N=O}} = 1330$ ($\Delta\nu^{15\text{N}} = 11$ cm⁻¹), and $\nu_{\text{C=N}} = 1276$ cm⁻¹ ($\Delta\nu^{15\text{N}} = 18$ cm⁻¹). However, it is noteworthy that, according to the data in [5], the Raman spectrum of I shows another band at 1490 cm⁻¹, which is displaced upon the introduction of

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TABLE 1. Experimental and Calculated Frequencies of Azoxybenzene

Experiment			Calculation		Assignment
IR	Raman	$\Delta\nu^{15}\text{N}$	ν	$\Delta\nu^{15}\text{N}$	
1594	1595	—	1606	4	Ar(ν_{8a})
	1580	—	1589	—	
1572		—	1557	—	Ar(ν_{8b})
1556		—	1556	—	
1484	1484	—	1491	2	Ar(ν_{19a})
			1488	1	
1472	1480	10	1476	6	Ar(ν_{19b}); q_{14} ; q_{16} ; q_{13}
1450		—	1441	—	
1440	1444	30	1413	27	q_{14} ; q_{16} ; q_{13} ; q_{15}
1330	1335	9	1349	16	
1311	1307	—	1330	—	Ar(ν_2)
1300		4	1314	9	
1276	1282	16	1298	12	q_{15} ; q_{15}
1216	1220	—	1225	—	
			1223	—	Ar(ν_{14})
	1184	—	1178	—	
			1178	—	Ar(ν_{15})
			1149	—	
1165	1165	—	1146	—	Ar(ν_{15})
1155		—	1106	9	
	1107	3	1084	2	α ; q_{16} ; q_{14} ; $\gamma_{6,13}$; $\gamma_{13,14}$; $\gamma_{15,16}$
1075	1084	—	1072	1	
1067		—	1034	1	Ar(ν_{18b})
1023	1025	—	1020	—	
			1013	—	Ar(ν_{12})
998	1006	—	1011	—	
			909	8	Ar(ν_1); $\gamma_{14,15}$; q_{13} ; q_{16}
906	912	16	856	7	
	830	6	671	6	Ar(ν_{6a}); $\gamma_{14,15}$; $\gamma_{15,16}$
675	682	10	621	—	
615	620	—	620	—	Ar(ν_{6b})
			596	6	
582		5	415	4	Ar(ν_{6a}); $\gamma_{14,16}$; $\gamma_{14,15}$
425		5			
			287	—	$\gamma_{6,13}$ $\gamma_{6,13}$; $\gamma_{7,15}$; $\gamma_{15,16}$; $\gamma_{12,15}$
			239	—	
			143	—	$\gamma_{13,14}$ $\gamma_{15,16}$; $\gamma_{14,16}$ α ; $\gamma_{15,16}$; $\gamma_{14,16}$

*The participation of the aromatic rings in a vibration is indicated by the symbol Ar; the Wilson numbers of vibrations are given in parentheses; the coordinates participating in the vibrations are listed in order of decreasing contributions to the mode of vibration.

^{15}N into the azoxy group ($\Delta\nu^{15}\text{N} = 12 \text{ cm}^{-1}$). An assignment was not given for it in that work. In addition, in our opinion, the assignment of the band to $\nu\text{N}\rightarrow\text{O}$ proposed in that work (at 1330 cm^{-1}) seems questionable in view of the fact that this band is the most intense band in the Raman spectrum of I.

In order to interpret the stretching vibrations of the $\text{N}\rightarrow\text{O}$ group in I, we utilized the shifts of the absorption bands in solvents of different nature. For this purpose, we verified the dependence of the positions of the absorption bands in the IR spectrum of I on the polarity of the following solvents: CCl_4 , CDCl_3 , CD_3OD , $(\text{CD}_3)_2\text{CO}$, and $(\text{CD}_3)_2\text{SO}$. It turned out that there are several bands which experience small (up to 5 cm^{-1}) in the $1200\text{--}1500\text{-cm}^{-1}$ region, which is accessible to observation in all the solvents indicated. These data do not permit the rigorous interpretation of the stretching vibrations of the azoxy group.

We obtained and analyzed the IR and Raman spectra of ^{15}N -labeled trans-azoxybenzene (see [7]) and calculated the frequencies and modes of the in-plane vibrations of azoxybenzene (I and I'). As is seen from the data in Table 1, the introduction of ^{15}N results in changes in the positions of a number of bands in the $800\text{--}1500\text{-cm}^{-1}$ region.

The calculation of the vibrational spectrum of trans-I was performed in the intramolecular force field approximation according to the programs in [8]. The geometric parameters published in [9] were used in the calculation, and the force constants for the benzene fragments were taken from [10]. Figure 1 and Table 2 present, respectively, the numbering of the atoms and the bonds and the force constants of I used for the calculation (the force constants of I refined during the solution of the inverse spectroscopic problem are presented). The results

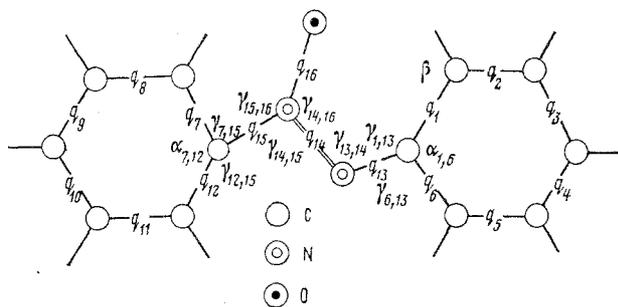


Fig. 1. Natural vibrational coordinates of the azoxybenzene molecule.

TABLE 2. Values of the Force Constants Used for the Calculation of the Vibrational Spectrum of Azoxybenzene ($\cdot 10^6$, cm^{-2}) *

$K_1 = 10,39$	$H_1^2 = 1,16$	$A_{14}^{13,44} = 0,40$
$K_2 = 18,56$	$H_1^3 = -0,73$	$A_{14}^{14,15} = 0,32$
$K_{13} = 9,36$	$H_1^4 = 0,83$	$A_{14}^{14,16} = 9,80$
$K_{14} = 12,75$	$H_{13}^{14} = 1,30$	$A_{14}^{15,16} = -0,11$
$K_{15} = 10,20$	$H_{14}^{15} = 1,40$	$A_{15}^{14,15} = 1,07$
$K_{16} = 9,56$	$H_{14}^{16} = 1,20$	$A_{15}^{14,16} = 0,21$
$K_{1,13} = 1,30$	$H_{15}^{13} = 0,95$	$A_{15}^{15,16} = -0,40$
$K_{6,13} = 1,30$	$A_1^{6,13} = -0,04$	$A_{16}^{14,15} = -0,09$
$K_{13,14} = 0,70$	$A_1^{1,13} = 0,01$	$A_{16}^{14,16} = 0,16$
$K_{14,15} = 1,10$	$A_{13}^{13,14} = -0,63$	$A_{16}^{15,16} = 0,46$
$K_{14,16} = 0,70$	$A_{13}^{1,13} = 1,20$	$L_{1,13}^{6,13} = -0,04$
$K_{15,16} = 0,40$	$A_{13}^{6,13} = 0,77$	$L_{6,13}^{13,14} = 0,07$
		$L_{13,14}^{14,15} = 0,03$

*The notation follows [11]: K_i is the force constant of the i -th bond; $K_{i,j}$ is the force constant of the angle formed by the i -th and j -th bonds; H_j^i is the force constant of the interaction of the stretching coordinates of the i -th and j -th bonds; $A_i^{n,m}$ is the force constant of the interaction of the stretching coordinate of the i -th bond and the variation of the bond angle formed by the n -th and m -th bonds; $L_{i,j}^{n,m}$ is the force constant of the interaction of the two angular coordinates with indices corresponding to the i -th and j -th bonds and the n -th and m -th bonds.

of the calculation of the frequencies and modes of vibration of I are presented in Table 1 (the frequencies of the stretching vibrations of the C-N bonds were not included in the table).

An analysis of the data in Table 1 allows us to draw the following conclusions. The vibrations of the aromatic fragments mainly do not interact with the vibrations of the azoxy group. The calculation confirms that a vibration with predominant participation of the N=N bond in I is observed at 1444 cm^{-1} ; however, the N=N bond makes appreciable contributions to a number of vibrations ($1480, 1335, \text{ and } 1107 \text{ cm}^{-1}$). The $\nu_{\text{N}=\text{O}}$ stretching vibration is not characteristic; the N=O bond makes significant contributions to a whole series of vibrations (see Table 1), the greatest contribution being made to the vibrations with frequencies equal to $830 \text{ and } 1107 \text{ cm}^{-1}$. According to the calculation, two vibrations, viz., those with $\nu_{\text{cal}} = 1349 \text{ and } 1298 \text{ cm}^{-1}$, have predominant contributions from the C-N bonds. The greatest contribution to the potential energy of the vibrations are ~ 40 from q_{15} , ~ 11 from q_{13} , and $\sim 8\%$ from q_{14} , and the greatest contribution to the latter is made by the q_{13} coordinate (the contributions to the energy are ~ 50 from q_{13} and $\sim 8\%$ from q_{15}). Since the C-N bonds vibrate in phase in the former vibrations and in antiphase in the latter vibration, these vibrations may

TABLE 3. Frequencies (ν , cm^{-1}) and Intensities (I*) of Stretching Vibrations in the IR and Raman Spectra of Fluorine-Containing Azoxybenzenes[†]

Compound	$\nu_{\text{C}_6\text{H}_5}$, $\nu_{\text{C}_6\text{F}_5}$ Raman (I)	$\nu_{\text{N}=\text{N}}$		$\nu_{\text{cophas C-N}}$		$\nu_{\text{antiphase C-N}}$	
		IR	Raman	IR	Raman (I)	IR	Raman (I)
(I)	1595 1592(2,8)	1440 m	1444 m	1330 w	1335 vs (5,7)	1276 m	1282 s (5,4)
(I')	1595	1410 s	1414 s	1320 w	1326 vs	1258 m	1266 s
(II)	1604(2,0)	1480 [3]	1479 s	1330 w	1334 vs (5,6)	1282 s	1282 s(4,2)
		1410 m	1413 m				
(III)	1608	1470 s	1471 m	1320 m	1320 vs	1265 s	1267 s
	1583	1440 m	1438 s				
(IV)	1616(1,2)	1480 s	1482 s	1335 w	1338 vs (4,6)	1285 vs	1286br(3,8)
	1595	1439 m	1432 m				
(IV')	1614	1460 m	1462 m	1315 w	1321 vs	1275 vs	1271 s
	1596	1412 m	1409 m				
(V)	1616	1470 s	1468 s	1330 m	1335 vs	1280 m	1293 s
		1430 w	1428 m				
(VI)	1637	1463 m	1466 s	1340 m	1345 vs	1260 m	1263 s
			1427 w				
(VIIa)	1660(0,6)	1480 s	1481 m	1356 m	1359 vs (1,5)	1287 w	1291 s(0,3)
		1432 m					
(VIIa')	1661	1455 m	1460 m	1340 m	1342 vs	1274 s	1279 s
		1420 s					
(VIIb)	1646(0,6)	1482 s	1486 m	1348 m	1356 vs (1,3)	1250 vw	1247 m
		1454 m	1452 w				
(VIIb')	1646	1468 m	1471 m	1321 m	1334 vs	1235 vw	1241 m
		1435 m	1436 m				
(VIIc)	1661(1,1)	1490 vs	1489 s	1350 m	1356 vs (2,8)	1268 vw	1274 w (0,5)
		1420 m	1426 m				
(VII d)	1654	1480 m	1478 s	1352 m	1360 vs (3,5)	1280 w	1285 w (0,9)
		1430 m	1432 m				
(VII d')	1653	1455 vs	1460 m	1335 m	1342 vs	1270 w	1275 w
		1412 s					
(VIIe)	1666	1505 s	1501 s	1360 s	1365 vs (1,1)		1272 vw
		1420 s	1426 s				(0,04)
(VII f)	1637		1482 m	1340 m	1349 vs	1250 vw	1253 w
		1413 m	1411 s				

*In relative units in comparison to the band of acetone at 1710 cm^{-1} .

†The designations vs, s, m, w, vw, and br refer to the distribution of the intensity within the particular spectrum.

be considered approximately as cophasal and antiphase stretching vibrations of the C-N bonds. It should be noted that the introduction of ^{15}N into the azoxy group results in the observation of some redistribution of the intensities of the bands at 1335 and 1282 cm^{-1} in the Raman spectrum of I and a number of fluorinated derivatives of azoxybenzenes (for examples see [12]). This is possibly due to some alteration of the modes of the corresponding vibrations, which is indicated by the calculation.

The frequencies of the stretching vibrations of the N=N and C-N bonds of the polyfluorinated azoxybenzenes presented in Table 3, as well as the frequencies of the ν_s vibrations of the aryl ($\nu_{\text{C}_6\text{H}_5}$) and polyfluoroaryl ($\nu_{\text{C}_6\text{F}_5}$) rings, were revealed by comparing the IR and Raman spectra of the samples labeled with ^{15}N (the complete IR and Raman spectra of the azoxybenzenes investigated were published in [12]). The spectra of all the fluorinated azoxybenzenes display two bands in the 1410 - 1490-cm^{-1} region, which are displayed upon the introduction of ^{15}N into the azoxy group (see Table 3). In analogy to I, we assigned the bands in the region just indicated to vibrations of the N=N bond. It should, however, be noted that it is not always possible to single out a vibration with a predominant contribution from the N=N bond on the basis of the magnitude of the isotope shift of these bands in the spectra of polyfluorinated azobenzenes, as was done in the case of I. For example, in the case of decafluoroazoxybenzene (VIIa) labeled with the ^{15}N isotope, there are shifts of the band at 1481 cm^{-1} in the Raman spectrum and of the band at 1432 cm^{-1} in the IR spectrum by 21 and 12 cm^{-1} , respectively, whereas in the case of VIIb, the band at 1490 cm^{-1} in the Raman spectrum and the band at 1454 cm^{-1} in the IR spectrum are displaced by 15 and 16 cm^{-1} , respectively.

The band in the 1330 - 1350-cm^{-1} region is characteristic of polyfluorinated azoxybenzenes. As a rule, it is the most intense band in the Raman spectrum, is weak in the IR spectrum, is

TABLE 4. Physicochemical Characteristics of Fluorine-Containing Azoxybenzenes

Com- pound	Yield	mp, °C	Found, %				Empirical Formula	Calculated, %				UV spectrum λ_{max} , nm (log ϵ)
			C	H	F	N		C	H	F	N	
(IV)	85	104-105	53,10	1,98	27,90	9,95	$\text{C}_{12}\text{H}_6\text{F}_4\text{N}_2\text{O}$	53,33	2,22	28,14	10,37	230 (3,95) 316 (4,03) 228 (3,87) 248 (3,88)
(V)	80	95-96	53,20	2,10	27,84	10,05						
(VI)	79	64-65	42,22	0,53	44,47	7,86	$\text{C}_{12}\text{H}_2\text{F}_8\text{N}_2\text{O}$	42,11	0,58	44,44	8,19	230 (3,97) 253 (3,90) 316 (4,43) 234 (4,00) 288 (3,78)
(VIIb)	79	62-63 52 [15]	42,22		44,30	7,86						
(VIIc)	75	113-115	45,23	1,57	40,89	7,45	$\text{C}_{11}\text{H}_6\text{F}_8\text{N}_2\text{O}$	45,41	1,62	41,08	7,57	236 (4,09) 300 (3,86)
(VIIId)	80	130-131	41,64		38,28	6,50	$\text{C}_{11}\text{H}_6\text{F}_8\text{N}_2\text{O}_2$	41,79		37,81	6,96	232 (3,53) 314 (3,28)
(VIIe)	80	84-85	35,23		55,70	5,64	$\text{C}_{11}\text{F}_{11}\text{N}_2\text{O}$	35,15		55,65	5,86	234 (3,83) 282 (3,64)
(VIIf)	82	94-96	28,72		30,37	5,53	$\text{C}_{12}\text{BrF}_8\text{N}_2\text{O}$	28,80		30,40	5,60	239 (4,36) 262 (4,23) 299 (3,98)

displaced by 15-20 cm^{-1} upon the introduction of the ^{15}N isotope, and shows little sensitivity to the nature of the solvent. It should apparently be assigned to the cophasal vibration of the C-N bonds. The band in the 1250-1290- cm^{-1} region is assigned to the antiphasal vibration of the C-N bonds. The IR spectrum of VIIb shows an isotope shift $\Delta\nu^{15}\text{N} = 8 \text{ cm}^{-1}$ for the absorption band at 830 cm^{-1} , which, in analogy to I and the data in [6], may be assigned to a vibration with participation of the N \rightarrow O bond.

Thus, the introduction of fluorine atoms into the benzene ring in I is not accompanied by significant changes in the frequency ranges for the appearance of the stretching vibrations of the azoxy group.

The intensities of the Raman lines corresponding to vibrations of groups with multiple bonds are known to be sensitive to conjugation effects and to be relatively insensitive to induction effects [13, 14]. Unfortunately, we are not able to compare the intensities of the $\nu\text{N}=\text{N}$ bands in the Raman spectra of fluorinated and unfluorinated azoxybenzenes, since changes in the relative intensities of the bands in the 1400-1500- cm^{-1} range in the Raman spectra of acetone solutions in comparison to the solid state occur for many fluorinated compounds. The reason for these changes is still not clear to us. Table 3 presents the intensities for the $\nu_{\text{a,a,b}}\text{C}_6\text{H}_5$, $\nu_{\text{a,a,b}}\text{C}_6\text{F}_5$, and $\nu\text{C}-\text{N}$ bands in the Raman spectra, which were measured relative to the band of acetone at 1710 cm^{-1} . No correction for the spectral sensitivity of the instrument was introduced, since we were only interested in a qualitative comparison of the intensities of the same vibrations upon the introduction of fluorine atoms and other substituents into the benzene rings. From the data in Table 3 it follows that the introduction of fluorine atoms into the benzene rings lowers the intensity of the measured bands to a greater extent, the greater is the number of fluorine atoms in each ring.

The intensity of the C-N bands for the polyfluorinated compounds (see Table 3) displays a clear dependence on the nature of the substituent in positions 4 and 4' of the benzene rings, their intensity increasing along the series $\text{CF}_3 < \text{H} < \text{F} < \text{CH}_3 < \text{OCH}_3$. Increases in intensity by hypsochromic shifts of the long-wavelength maximum in the UV spectra of these compounds are observed along the same series (Table 4). This is qualitatively consistent with the conclusions which we drew in [2] that the intensity of the stretching vibrations of the bridging group depends on the effectiveness of the conjugation of the nonbonding electron pair of one nitrogen atom of this group with the π system of the benzene ring.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrophotometer in KBr (1:800), CCl_4 , CDCl_3 , CD_2OD , $(\text{CD}_3)_2\text{SO}$, and $(\text{CD}_3)_2\text{CO}$ (C = 5%, d = 0.1 mm), and the Raman spectra of the solid samples and solutions in acetone (C = 0.39 M) were recorded on a Coderg PH-1 spectrometer with an He-Ne laser ($\lambda_{\text{exc}} = 6328 \text{ \AA}$). The integrated intensities in the Raman spectra were evaluated with respect to the acetone line at 1710 cm^{-1} , which served as an internal standard, and the bands corresponding to the $\nu_{\text{a,a}}$ and $\nu_{\text{s,b}}$ vibrations of the aromatic fragments were integrated together. The UV absorption spectra were recorded on a Specord UV-VIS spectrophotometer. The calculations were performed on an ES-1035 computer. The fluorine-containing azoxybenzenes were obtained by oxidizing the corresponding anilines by peracetic acid according to the method in [15]. Compound I was synthesized according to [16], ^{15}N -labeled azoxybenzene was obtained from ^{15}N -labeled nitrobenzene, compound II was obtained according to [17], compound VIIa was obtained according to [18], and compound III had mp 82-83°C and the following UV spectrum $\lambda_{\text{max}} = 226 \text{ nm}$ ($\log \epsilon = 3.96$), 260 nm ($\log \epsilon = 3.89$), and 322 nm ($\log \epsilon = 4.21$).

Synthesis of Fluorine-Containing Azoxybenzenes. A mixture of 0.1 mole of polyfluorinated aniline, 30 ml of 30% H_2O_2 , and 150 ml of glacial CH_3COOH was held for 50 h at 20°C. The reaction mixture was poured into water and extracted by ether. The ethereal extract was dried over MgSO_4 . After the solvents were distilled off in a rotary vacuum evaporator, the residue was passed through a column with silica gel (the eluent was a 1:10 mixture of diethyl ether and petroleum ether, 40-60°C). After the solvents were distilled off, the residue was recrystallized from hexane. The yields and the characteristics of the products are presented in Table 4.

The synthesis of the ^{15}N -labeled azoxybenzenes containing fluorine atoms was carried out in a similar manner.

CONCLUSIONS

The vibrations of the azoxy group in azoxybenzene have been assigned on the basis of calculations of the frequencies and normal modes of vibrations for it. The introduction of fluorine atoms into the benzene rings has practically no influence on the frequencies of the stretching vibrations of the azoxy group. An increase in the number of fluorine atoms in the benzene rings of azoxybenzenes lowers the intensities of the stretching vibrations of the azoxy and phenyl groups in the IR spectra.

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