

SYNTHESIS AND MASS SPECTRA OF AROYLBENZAMIDOXIMES

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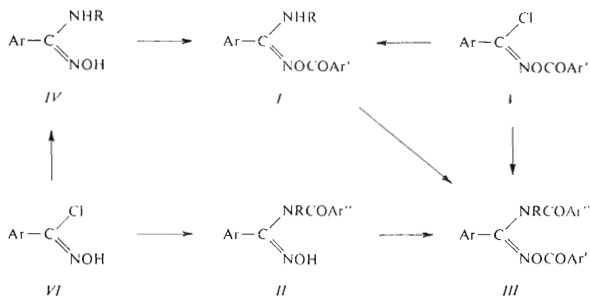
The synthesis of several O-roylbenzamidoximes *I* and of some N-royl (*II*) as well as of bis(royl)-benzamidoximes (*III*) is described and their fragmentation pattern in the mass spectra is discussed. O- and N-Aroylbenzamidoximes are distinguished on the basis of their mass spectra since the O-derivatives afford the ion corresponding to the acid $[\text{ArCOOH}]^+$ whereas the N-derivatives afford the ion corresponding to the amide $[\text{ArCONH}]^+$.

The structure¹⁻³ of benzamidoximes as well as their behavior upon electron impact^{4,5} have been previously investigated. On the other hand, although the acylation of benzamidoximes has been studied by several investigators⁶⁻⁹, the mass spectra of O- or N-roylbenzamidoximes and of bis(royl)benzamidoximes have not received proper attention. Our interest in these compounds was connected with the intention to search for analogies between familiar solution reactions of hydroxylamine derivatives and their fragmentation pattern.

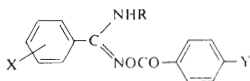
The O-roylbenzamidoximes *I* were prepared by direct aroylation of the corresponding benzamidoximes *IV* with aroyl chlorides, which under neutral conditions or in presence of a small amount of pyridine gave the O-products. The yield in *I* is up to 90% in the reaction with *p*-nitrobenzoyl chloride which reacts even at room temperature, but it drops to ~15% with *p*-methylbenzoyl chloride which reacts only under reflux.

The structure of compounds *I* was confirmed by an independent synthesis⁷ from O-roylbenzohydroximoyl chlorides *V* and an appropriate amine, giving identical products.

When *o*- or *p*-methoxybenzamidoxime was treated with benzoyl or *p*-nitrobenzoyl chloride, mixtures of N- and O-roylated products were obtained. The N-roylbenzamidoximes *II* were better prepared by treating¹⁰ benzohydroximoyl chlorides *VI* with benzamides in the presence of sodium ethoxide. The O-royl derivatives *I* are easily distinguished^{9,11} from their isomeric N-derivatives *II* by means of IR spectra. In particular, the former display a carbonyl absorption, $\nu(\text{CO})$, at 1720–1740



cm^{-1} , whereas the latter at $1660\text{--}1670\text{ cm}^{-1}$. Both O- and N-aryl derivatives *I* and *II* yielded the bis(aryl)benzamidoximes *III* with excess of aroyl chloride. These were also obtained by a reaction of O-arylbenzohydroximoyl chlorides *V* with benzamide in the presence of sodium ethoxide. The products *III* display two carbonyl absorptions at $1740\text{--}1750\text{ cm}^{-1}$ ($\nu(\text{CO})_{\text{ester}}$) and at $1660\text{--}1670\text{ cm}^{-1}$ ($\nu(\text{CO})_{\text{amide}}$).



Ia, X = Y = R = H

Ib, X = R = H, Y = CH₃

Ic, X = R = H, Y = Cl

Id, X = R = H, Y = NO₂

Ie, R = Y = H, X = *p*-Cl

If, R = H, Y = CH₃, X = *p*-Cl

Ig, R = H, Y = NO₂, X = *p*-Cl

Ih, R = Y = H, X = *o*-Cl

Ii, R = H, Y = CH₃, X = *o*-Cl

Ij, R = H, Y = NO₂, X = *o*-Cl

Ik, R = Y = H, X = *p*-NO₂

II, R = Y = H, X = *o*-NO₂

Im, R = Y = H, X = *p*-CH₃

In, R = H, Y = NO₂, X = *p*-CH₃

Io, X = Y = H, R = C₂H₅

Ip, Y = H, R = C₂H₅, X = *p*-Cl

Iq, R = C₂H₅, Y = NO₂, X = *p*-Cl

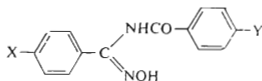
Ir, Y = H, R = C₂H₅, X = *o*-Cl

Is, R = C₂H₅, Y = NO₂, X = *o*-Cl

Iu, X = Y = H, R = C₆H₅

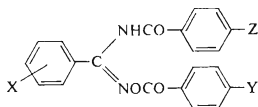
Iv, Y = H, R = C₆H₅, X = *o*-Cl

Iv, Y = NO₂, X = *o*-OCH₃; N(C₂H₅)₂



IIa, Y = H, X = OCH₃

IIb, Y = NO₂, X = Cl



IIIa, Y = Z = NO₂, X = *p*-Cl

IIIb, Y = H, Z = NO₂, X = *p*-Cl

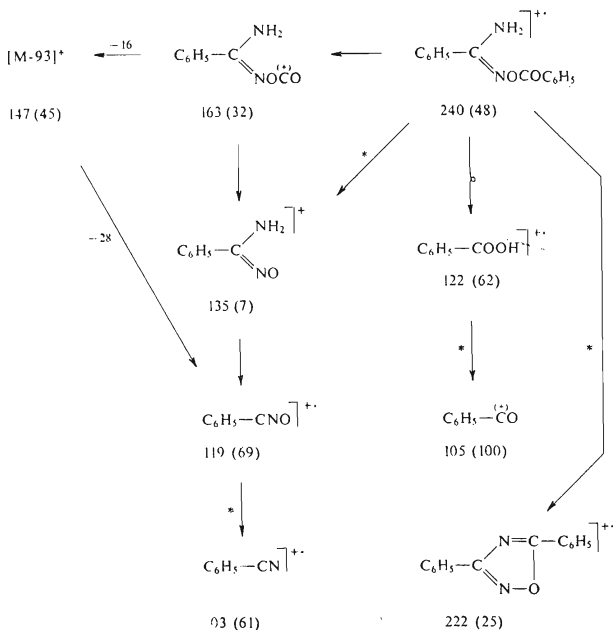
IIIc, Y = Z = NO₂, X = *o*-Cl

IIId, Y = H, Z = NO₂, X = *m*-Cl

IIIe, Y = Z = NO₂, X = *p*-OCH₃

IIIf, Y = Z = NO₂, X = *o*-OCH₃

The analytical data, yields, and some physical characteristics of the products *I–III* are given in Tables I and II, whereas their mass spectra are given in Table III. Exact molecular weights for some ions of the compound *Ic* are given in Table IV.



SCHEME 1

TABLE I
Analytical data of the compounds I

Compound	M.p., °C ^a yield, %	IR spectrum (cm ⁻¹) ^b ν(C=O)	Formula (m.w.)	Calculated/Found		
				%C	%H	%N
<i>Ia</i>	146–148 ^c 50	1 730	C ₁₄ H ₁₂ N ₂ O ₂ (240·3)	—	—	—
<i>Ib</i>	163–165 16	1 730	C ₁₅ H ₁₄ N ₂ O ₂ (254·3)	70·85 70·76	5·55 5·31	11·02 11·28
<i>Ic</i>	198–199 ^d 40	1 725	C ₁₄ H ₁₁ ClN ₂ O ₂ (274·7)	—	—	—
<i>Id</i>	202–203 90	1 725	C ₁₄ H ₁₁ N ₃ O ₄ (285·3)	58·94 58·92	3·89 4·07	14·73 14·39
<i>Ie</i>	173–174 ^{e,f} 70	1 725	C ₁₄ H ₁₁ ClN ₂ O ₂ (274·7)	—	—	—
<i>If</i>	195–197 15	1 735	C ₁₅ H ₁₃ ClN ₂ O ₂ (288·7)	62·50 62·88	4·51 4·52	9·72 9·94
<i>Ig</i>	184–185 90	1 720	C ₁₄ H ₁₀ ClN ₃ O ₄ (319·7)	52·66 52·83	3·13 3·19	13·17 13·28
<i>Ih</i>	182–183 87	1 740	C ₁₄ H ₁₁ ClN ₂ O ₂ (274·7)	61·31 61·48	4·01 3·79	10·22 9·83
<i>Ii</i>	160–162 ^e 23	1 725	C ₁₅ H ₁₃ ClN ₂ O ₂ (288·7)	62·50 62·43	4·51 4·46	9·72 9·34
<i>Ij</i>	217–218 70	1 725	C ₁₄ H ₁₀ ClN ₃ O ₄ (319·7)	52·66 52·72	3·13 3·13	13·17 13·45
<i>Ik</i>	164–166 57	1 740	C ₁₄ H ₁₁ N ₃ O ₄ (285·3)	58·94 58·48	3·89 3·51	14·73 14·68
<i>Il</i>	206–208 12	1 735	C ₁₄ H ₁₁ N ₃ O ₄ (285·3)	58·94 58·61	3·89 3·54	14·73 14·62
<i>Im</i>	184–185 53	1 725	C ₁₅ H ₁₄ N ₂ O ₂ (254·3)	70·85 70·49	5·55 5·24	11·02 11·15
<i>In</i>	180–181 47	1 725	C ₁₅ H ₁₃ N ₃ O ₄ (299·8)	60·19 60·50	4·38 4·42	14·04 13·87
<i>Io</i>	156–158 34	1 725	C ₁₆ H ₁₆ N ₂ O ₂ (268·3)	71·62 72·03	6·01 5·78	10·44 10·28
<i>Ip</i>	138–139 64	1 730	C ₁₆ H ₁₅ ClN ₂ O ₂ (302·8)	63·58 63·42	4·97 4·87	9·27 8·99
<i>Iq</i>	170–171 67	1 740	C ₁₆ H ₁₄ ClN ₃ O ₄ (347·8)	55·33 55·36	4·03 4·09	12·10 11·98

TABLE I
 (Continued)

Compound	M.p., °C ^a yield, %	IR spectrum (cm ⁻¹) ^b ν(C=O)	Formula m.w.) ^c	Calculated/Found		
				% C	% H	% N
<i>Ir</i>	142–144	1 735	C ₁₆ H ₁₅ ClN ₂ O ₂ (302.8)	63.58	4.97	9.27
	24			63.94	5.06	9.16
<i>Is</i>	186–188	1 735	C ₁₆ H ₁₄ ClN ₃ O ₄ (347.8)	55.33	4.03	12.10
	40			55.38	4.02	12.28
<i>It</i>	171–173	1 730	C ₂₀ H ₁₆ N ₂ O ₂ (316.3)	75.93	5.10	8.86
	22			75.14	5.16	8.76
<i>Iu</i>	191–193	1 730	C ₂₀ H ₁₅ ClN ₂ O ₂ (350.8)	68.57	4.29	8.00
	20			68.52	4.54	8.02
<i>Iv</i>	165–167	1 730	C ₁₉ H ₂₁ N ₃ O ₅ (371.4)	61.44	5.70	11.32
	10			61.28	5.84	11.14

^a Recrystallization solvent ethanol; ^b nujol; ^c lit.^{1,2} 148°C; ^d lit.³ 200°C; ^e recrystallization solvent benzene; ^f lit.³ 175°C.

All the mass spectra of compounds *I*, *II*, *III* display the presence of the molecular ion with moderate relative intensity. The spectra of the compounds *Ja–In* show a peak $[M-18]^+$ with variable intensity and this loss of water from the molecular ion is usually followed by a metastable ion peak. The cyclisation reaction to yield 1,2,4-oxadiazoles is well known from the synthetic chemistry of these compounds.

The peak $[M-18]^+$ is absent in the case of N-ethyl derivatives *Io–Is*, but it is present in N-phenyl derivatives *It–Iu*, a fact suggesting a participation of the phenyl *o*-hydrogens in water elimination (Scheme 1).

Very characteristic is the ion corresponding to the acid, $[\text{ArCOOH}]^{+}$, which is found with a high relative intensity (45–90%) in all spectra of the compounds *I*, except *If* (m/z 136) where its relative intensity drops to 33%. In order to find out which hydrogen is eliminated to give the acid ion, attempts were made with deuterium labelling in the N–H group for a number of the compound *I*, but unambiguous evidence in favor of a certain pathway was not obtained. The elimination of the substituent X* from the nitrile ion $\text{X}-\text{C}_6\text{H}_4-\text{CN}^{+}$ yields the ion m/z 102. The transition is in all cases followed by a metastable ion peak. The ions m/z 290/288 are due to the loss of CH₂ from the molecular ion of compounds *Ip*, *Ir*. An analogous elimination is observed in all compounds where R = –CH₂CH₃ (*Io–Is*). Typical fragmentation pathways for the compound *Ia* and *Ic* are given in Schemes 1 and 2.

Compounds *II* do not reveal the presence of the acid ion, but instead a peak corresponding to the amide fragment, $[\text{ArCONH}]^+$. These two ions could be used for distinguishing the two isomeric compounds *I* and *II*. As an example, the mass spectra of two isomers *Ig* and *IIf* are given in Figs 1 and 2, respectively. On the other hand, the compounds *III* show peaks of both the acid and the amide ion, of which the latter appears with higher relative intensity (Fig. 3).

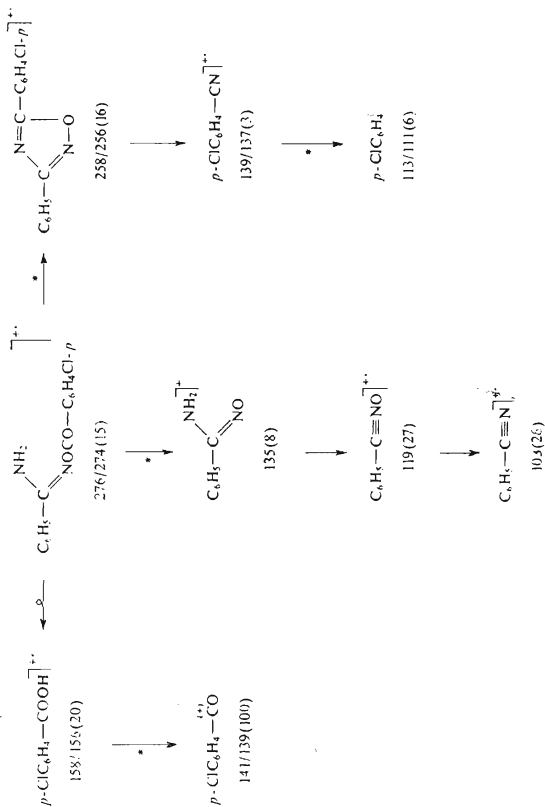
The aroyl ion $[\text{ArCO}]^+$ is the base peak in almost all the spectra studied. Also the ion $[\text{ArCN}]^{+}$ appears with a very high intensity; in the case of *p*-chloro substituted derivatives the ion of this type competes in intensity with the aroyl ion. With much lower intensity appears the ion corresponding to the nitrile oxide $[\text{ArCNO}]^{+}$. It must be noticed that there was no significant difference in the fragmentation pattern between the *ortho*- and *para*-substituted derivatives *I*, except the relative intensities of some ions.

TABLE II
Analytical data of the compounds *II* and *III*

Compound	M.p., °C ^a yield, %	IR spectrum (cm ⁻¹) ^b ν(C=O)	Formula (m.w.)	Calculated/Found		
				% C	% H	% N
<i>IIf</i>	124–126 ^c	1 670	C ₁₅ H ₁₄ N ₂ O ₃ (270.3)	66.65	5.22	10.37
	20			66.37	5.49	10.24
<i>IIf</i>	147–148 ^c	1 665	C ₁₄ H ₁₀ ClN ₃ O ₄ (319.7)	52.66	3.13	13.17
	50			52.87	3.17	13.27
<i>IIIa</i>	174–175	1 725	C ₂₁ H ₁₃ ClN ₄ O ₇ (468.8)	53.85	2.77	11.97
	70	1 670		53.64	3.02	11.78
<i>IIIb</i>	113–114	1 725	C ₂₁ H ₁₄ ClN ₃ O ₅ (423.8)	59.57	3.31	13.24
	55	1 660		59.69	3.18	13.42
<i>IIIc</i>	220–221	1 730	C ₂₁ H ₁₃ ClN ₄ O ₇ (468.8)	53.85	2.77	11.97
	82	1 675		54.08	2.87	11.75
<i>IIIId</i>	196–198	1 740	C ₂₁ H ₁₄ ClN ₃ O ₅ (423.8)	59.57	3.31	13.24
	34	1 665		59.34	3.47	13.27
<i>IIIe</i>	218–220	1 730	C ₂₂ H ₁₆ N ₄ O ₈ (464.4)	56.90	3.47	12.07
	22	1 670		56.85	3.76	11.89
<i>IIIIf</i>	202–204	1 730	C ₂₂ H ₁₆ N ₄ O ₈ (464.4)	56.90	3.47	12.07
	24	1 660		56.68	3.17	12.31

^a Recrystallization solvent ethanol; ^b nujol; ^c recrystallization solvent ethanol–water.

Substitution in compounds *I* offered the opportunity to examine the dependence¹³ of relative intensities, related to the intensity of the molecular ion, on substituent constants. However, no correlation was obtained with the ions $[M-YC_6H_4]^+$ (substitution in X), or $[C_6H_5COOH]^+$ (substitution in Y) which satisfy the pre-conditions pointed out by Bursey¹³, as well as with some other ions (Scheme 1).



SCHEME 2

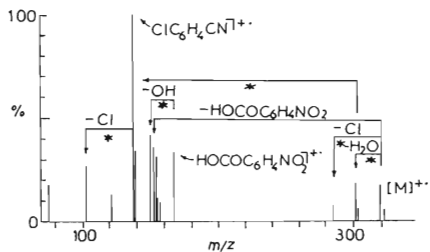


FIG. 1

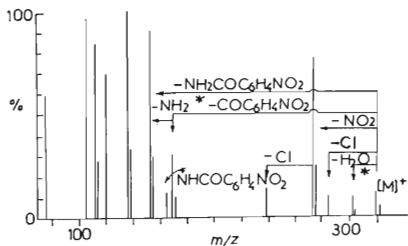
Mass spectrum of O-(4'-nitrobenzoyl)-4-chlorobenzamidoxime (*Ia*)

FIG. 2

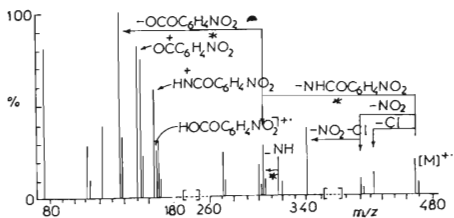
Mass spectrum of N-(4'-nitrobenzoyl)-4-chlorobenzamidoxime (*IIb*)

FIG. 3

Mass spectrum of N,O-bis(4'-nitrobenzoyl)-4-chlorobenzamidoxime (*IIIa*)

TABLE III

Principal fragment ions in the mass spectra of the compounds *I*, *II* and *III*

Compound	<i>m/z</i> (% Relative intensity)
<i>Ia</i>	240 (48, M ⁺), 222 (25), 163 (32), 147 (45), 135 (7), 122 (62), 121 (6), 119 (69), 105 (100), 103 (61), 91 (32), 77 (26), 51 (20).
<i>Ib</i>	254 (24, M ⁺), 239 (7), 236 (14), 223 (4), 163 (16), 136 (43), 119 (100), 105 (57), 103 (46), 91 (69), 77 (29), 51 (44)
<i>Ic</i>	276/274 (15, M ⁺), 258/256 (16), 245/243 (3), 223 (1), 158/156 (20), 141/139 (100), 137 (3), 135 (8), 119 (27), 113/111 (6), 104 (5), 103 (26), 91 (7), 76 (12), 50 (8)
<i>Id</i>	285 (53, M ⁺), 267 (29), 255 (5), 239 (38), 167 (91), 163 (27), 150 (85), 119 (100), 104 (93), 103 (78), 91 (40), 77 (41), 50 (28)
<i>Ie</i>	276/274 (35, M ⁺), 258/256 (3), 223 (1), 171/169 (3), 155/153 (7), 154/152 (14), 139/137 (71), 127/125 (8), 122 (21), 113/111 (8), 105 (100), 102 (17), 77 (58), 51 (18)
<i>If</i>	290/288 (14, M ⁺), 270 (2), 223 (3), 205 (<1), 171/169 (4), 155/153 (21), 139/137 (100), 136 (33), 127/125 (11), 122 (5), 119 (8), 113/111 (27), 102 (57), 75 (28), 50 (19)
<i>Ig</i>	321/319 (18, M ⁺), 303/301 (19), 284 (7), 223 (5), 167 (34), 155/153 (31), 154/152 (37), 150 (42), 139/137 (100), 127/125 (15), 122 (13), 113/111 (9), 104 (18), 102 (27), 75 (18), 50 (24)
<i>Ih</i>	276/274 (51, M ⁺), 258/256 (7), 239 (4), 171/169 (14), 155/153 (100), 139/137 (92), 127/125 (9), 122 (49), 113/111 (9), 105 (66), 102 (78), 90 (39), 50 (25)
<i>Ii</i>	290/288 (50, M ⁺), 270 (<1), 171/169 (18), 155/153 (100), 139/137 (56), 136 (50), 127/125 (27), 122 (9), 119 (27), 113/111 (49), 102 (86), 90 (47), 50 (59)
<i>Ij</i>	321/319 (4, M ⁺), 303/301 (13), 284 (2), 268 (1), 223 (<1), 167 (24), 155/153 (73), 150 (56), 139/137 (81), 138 (25), 127/125 (39), 122 (26), 113/111 (32), 104 (100), 102 (92), 90 (60), 76 (59), 50 (44)
<i>Ik</i>	285 (21, M ⁺), 267 (31), 255 (12), 239 (15), 223 (2), 208 (43), 180 (33), 164 (100), 148 (83), 136 (4), 122 (88), 118 (1), 105 (80), 102 (32), 77 (46), 51 (28)
<i>Il</i>	285 (14, M ⁺), 267 (28), 255 (4), 239 (11), 208 (36), 180 (47), 164 (100), 148 (96), 136 (2), 122 (73), 118 (1), 105 (89), 102 (35), 77 (30), 51 (36)
<i>Im</i>	254 (31, M ⁺), 239 (24), 236 (20), 223 (2), 177 (43), 149 (15), 133 (68), 122 (41), 118 (13), 117 (100), 105 (97), 102 (61), 91 (97), 77 (77), 51 (32)
<i>In</i>	299 (24, M ⁺), 284 (16), 281 (11), 269 (19), 253 (8), 222 (7), 177 (39), 167 (76), 150 (86), 149 (32), 133 (80), 122 (39), 117 (100), 105 (2), 104 (91), 102 (14), 91 (92), 77 (35), 51 (42)
<i>Io</i>	268 (13, M ⁺), 254 (19), 240 (3), 224 (8), 191 (31), 163 (6), 122 (70), 119 (100), 105 (96), 103 (93), 91 (74), 75 (46), 50 (39)
<i>Ip</i>	304/302 (9, M ⁺), 290/288 (3), 274/272 (13), 258/256 (6), 223 (2), 155/153 (25), 154/152 (6), 139/137 (100), 127/125 (26), 122 (35), 113/111 (37), 105 (90), 102 (47), 75 (29), 51 (37)

TABLE III
(Continued)

Compound	<i>m/z</i> (% Relative intensity)
<i>Iq</i>	349/347 (7, M ⁺), 333 (1), 319 (13), 303/301 (3), 275/273 (5), 197 (26), 181 (58), 167 (97), 155/153 (95), 154/152 (37), 150 (33), 139/137 (100), 127/125 (21), 122 (28), 113/111 (85), 102 (68), 75 (61), 50 (64).
<i>Ir</i>	304/302 (6, M ⁺), 290/288 (3), 274/272 (12), 258/256 (8), 223 (1), 155/153 (100), 139/137 (96), 127/125 (28), 122 (19), 113/111 (10), 105 (87), 90 (62), 51 (60)
<i>Is</i>	349/347 (9, M ⁺), 333 (3), 319 (7), 303/301 (3), 289/287 (9), 273 (4), 197 (24), 181 (49), 167 (99), 155/153 (100), 150 (40), 139/137 (90), 127/125 (20), 122 (37), 113/111 (8), 104 (86), 90 (39), 50 (79)
<i>It</i>	316 (18, M ⁺), 298 (13), 239 (22), 224 (54), 135 (10), 122 (33), 119 (100), 105 (99), 103 (81), 91 (37), 77 (43), 51 (20)
<i>Iu</i>	352/350 (13, M ⁺), 334/332 (11), 315 (3), 275/273 (17), 260/258 (36), 171/169 (3), 155/153 (77), 139/137 (75), 127/125 (2), 122 (36), 113/111 (9), 105 (100), 90 (28), 51 (37)
<i>Iv</i>	371 (4, M ⁺), 356 (9), 325 (4), 221 (70), 207 (10), 193 (7), 179 (11), 167 (16), 165 (28), 150 (54), 149 (47), 133 (100), 121 (2), 118 (18), 107 (89), 102 (64), 75 (41)
<i>IIa</i>	270 (18, M ⁺), 255 (5), 252 (13), 239 (19), 165 (64), 150 (9), 149 (90), 133 (100), 120 (37), 118 (3), 107 (49), 105 (40), 102 (21), 76 (52)
<i>IIb</i>	321/319 (12, M ⁺), 303/301 (9), 284 (10), 275/273 (78), 238 (14), 171/169 (31), 165 (12), 155/153 (90), 150 (17), 139/137 (100), 120 (79), 119 (4), 113/111 (84), 104 (97), 102 (46), 76 (59)
<i>IIIa</i>	470/468 (17, M ⁺), 433 (10), 424/422 (7), 341 (36), 320/318 (20), 305/303 (27), 302/300 (16), 274/272 (23), 222 (8), 170/168 (30), 167 (25), 165 (58), 155/153 (75), 150 (82), 139/137 (100), 127/125 (38), 122 (39), 119 (15), 113/111 (29), 91 (76), 76 (82)
<i>IIIb</i>	425/423 (22, M ⁺), 388 (20), 379/377 (12), 342 (28), 320/318 (17), 303/301 (9), 275/273 (25), 260/258 (46), 223 (7), 170/168 (39), 165 (29), 155/153 (66), 150 (35), 139/137 (100), 127/125 (54), 122 (32), 119 (9), 113/111 (25), 105 (89), 90 (70), 76 (40).
<i>IIIc</i>	470/468 (14, M ⁺), 433 (5), 424/422 (9), 341 (25), 320/318 (12), 305/303 (21), 302/300 (28), 274/272 (21), 222 (6), 170/168 (33), 167 (32), 165 (48), 155/153 (69), 150 (36), 139/137 (100), 127/125 (20), 122 (26), 119 (12), 113/111 (20), 91 (78), 75 (60).
<i>IIId</i>	425/423 (16, M ⁺), 388 (6), 379/377 (9), 342 (22), 320/318 (11), 303/301 (12), 275/273 (28), 260/258 (31), 223 (3), 170/168 (27), 165 (49), 155/153 (55), 150 (39), 139/137 (100), 127/125 (58), 122 (26), 119 (9), 113/111 (35), 105 (85), 90 (79), 76 (62)
<i>IIIe</i>	464 (17, M ⁺), 449 (22), 433 (10), 418 (11), 341 (15), 314 (7), 299 (27), 296 (14), 268 (37), 222 (6), 167 (15), 165 (32), 164 (30), 150 (89), 149 (79), 133 (100), 122 (29), 121 (57), 120 (10), 107 (86), 91 (62), 76 (52)
<i>IIIf</i>	464 (11, M ⁺), 449 (16), 433 (15), 418 (9), 341 (7), 314 (14), 299 (31), 296 (23), 268 (55), 222 (11), 167 (23), 165 (65), 164 (40), 150 (75), 149 (50), 133 (100), 122 (31), 121 (47), 120 (12), 107 (80), 90 (68), 76 (83)

EXPERIMENTAL

All melting points are uncorrected and were obtained with a Kofler hot stage apparatus. The mass spectra were run at 70 eV on a RMU-6L Hitachi-Perkin-Elmer single focusing mass spectrometer using the direct insertion probe of the samples. The analyses were performed with a Perkin-Elmer Analyser, Model 240. The IR spectra were measured in Nujol with a Perkin-Elmer 297 infrared spectrophotometer.

O-Aroylbenzamidoximes *I*

An ether solution containing equivalent amounts of the amidoxime *IV* and the appropriate aroyl chloride in presence of pyridine was stirred for 2 h, eventually under reflux. Addition of water yielded the crude O-arylbendamidoxime *I*, which was recrystallized from the proper solvent. For the analytical data of the compounds *I* see Table I.

N-Aroylbendamidoximes *II*

Compounds *II* were obtained by refluxing for 3 h in pyridine one equivalent of benzohydroximoyl chloride *VI* with two equivalents⁹ of the appropriate benzamide in presence of sodium ethoxide. By adding water the crude N-arylbendamidoxime *II* was obtained, which was recrystallized from water-ethanol. For the analytical data of the compounds *II* see Table II.

Bis(arylo)benzamidoximes *III*

a) From O-arylbendamidoximes *I* by refluxing for 2 h in pyridine one equivalent of *I* with two equivalents of the appropriate aroyl chloride. Addition of water yielded the crude *III*, which was recrystallized from ethanol.

TABLE IV

Exact molecular weights and elemental composition of the ions of compound *Ic*

<i>m/z</i> found	Elemental composition	Ion
274-0511	C ₁₄ H ₁₁ ³⁵ ClN ₂ O ₂	$\left[\begin{array}{c} \text{C}_6\text{H}_5-\text{C} \begin{array}{l} \nearrow \text{NH}_2 \\ \searrow \text{NOCO}-\text{C}_6\text{H}_4\text{Cl-}p \end{array} \end{array} \right]^{++}$
256-0413	C ₁₄ H ₉ ³⁵ ClN ₂ O	$\left[\begin{array}{c} \text{C}_6\text{H}_5-\text{C} \begin{array}{l} \nearrow \text{N} \\ \searrow \text{N}-\text{O} \end{array} \text{C}=\text{C}-\text{C}_6\text{H}_4\text{Cl-}p \end{array} \right]^{++}$
155-9968	C ₇ H ₅ ³⁵ ClO ₂	$p\text{-ClC}_6\text{H}_4\text{COOH}]^{++}$
138-9956	C ₇ H ₄ ³⁵ ClO	$p\text{-ClC}_6\text{H}_4\text{-CO}^{(+)}$
119-0371	C ₇ H ₅ NO	$\text{C}_6\text{H}_5\text{-CNO}]^{++}$
111-0030	C ₆ H ₄ ³⁵ Cl	$p\text{-ClC}_6\text{H}_4^+$
104-0496	C ₇ H ₆ N	$\text{C}_6\text{H}_5\text{-C=NH}^{(+)}$

b) From N-aroylbenzamidoximes *II* by refluxing for 5 h in pyridine equivalent amounts of *II* and of aroyl chloride. By adding water the crude *III* was obtained.

c) From O-aroylbenzohydroximoyl chlorides *V* by refluxing for 2 h in pyridine one equivalent of *V* with two equivalents of the appropriate benzamide and with sodium ethoxide. Addition of water yielded the crude *III*. For the analytical data of the compounds *III* see Table II.

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