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1,3,4,6-Tetracarbonyl Compounds: V.* Reaction of 1,6-Disubstituted 3,4-Dihydroxy-2,4-hexadiene-1,6-diones with 2,3-Diaminopyridine

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Abstract—1,6-Disubstituted 3,4-dihydroxy-2,4-hexadiene-1,6-diones having two contiguous β-dicarbonyl fragments readily react with 2,3-diaminopyridine to give (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydropyrido-[2,3-*b*]pyrazines.

Acylpyruvic acids, their esters, and lactones of the γ-enolic form of 5-aryl-2,3-dihydrofuran-2,3-diones are known to react with 2,3-diaminopyridine to give regioisomeric acylmethylene derivatives of pyrido-[2,3-*b*]pyrazinones [2–5]. Apart from our preliminary communications [6–8] where we briefly reported on reactions of 2,3-diaminopyridine with some 1,3,4,6-tetracarbonyl compounds, there are no other published data on the synthesis of 2,3-bis(acylmethylene)pyrido-[2,3-*b*]pyrazines.

We have studied in detail the reaction of 1,6-disubstituted 1,3,4,6-tetraoxo compounds **Ia**–**Ig** [9–11] with 2,3-diaminopyridine. 1,3,4,6-Tetraketones **Ia**–**Ig** in solution exist as at least two tautomers, open-chain keto–enol form **A** and ring form **B** [1, 9–11]. They readily react with 2,3-diaminopyridine on heating in acetic acid or ethanol for a short time to afford (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydropyrido-[2,3-*b*]pyrazines **IIa**–**IIg** [6–8] (Scheme 1). It should be noted that initial 4,5-dihydroxy-3,5-octadiene-2,7-dione (**If**) also gives rise to minor open-chain tetraoxo tautomer **C**.

The yields, melting points, and elemental analyses of compounds **IIa**–**IIg** are collected in Table 1, and their spectral parameters, in Tables 2 and 3. The spectral data of pyrido[2,3-*b*]pyrazines **II** are consistent with their structure and are in a satisfactory agreement with published data for well known structurally related (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydroquinoxalines **III**. The latter are generally available through reaction of 1,3,4,6-tetraoxo derivatives **I** with *o*-phenylenediamine [6, 9, 12–15] and were selected as model compounds (Scheme 1).

The IR spectra of pyrido[2,3-*b*]pyrazines **II** are characterized by the presence of a broad low-frequency band at 1580–1607 cm^{-1} corresponding to stretching vibrations of carbonyl groups of the two aroylmethylene moieties and conjugated exocyclic double bonds. This indicates formation in the *Z* isomers of **II** of NH-chelate ring due to N–H···O=C intramolecular hydrogen bond [5, 13, 15]. Analogous bands are present in the IR spectra of compounds **III** which lack pyridine nitrogen atom in the second ring; they are located in the region 1575–1597 cm^{-1} [12, 13, 15]. It follows that the pyridine nitrogen atom does not affect absorption frequency of the conjugated carbonyl gruops. A similar pattern is observed while comparing the IR spectra of (*Z*)-3-acylmethylene-3,4-dihydropyrido[2,3-*b*]pyrazin-2(1*H*)-ones **IV** and regioisomeric (*Z*)-2-acylmethylene-1,2-dihydropyrido-[2,3-*b*]pyrazin-3(4*H*)-ones **V** with those of structurally related (*Z*)-3-acylmethylene-3,4-dihydroquinoxalin-2(1*H*)-ones **VI** [5] (Scheme 2).

The H^{α} signals in the ^1H NMR spectra of compounds **II** having two aroylmethylene fragments (R = Ar) in DMSO-d_6 are located in the same region (δ 6.41–7.08 ppm; Table 2) as the corresponding signals of 1,2,3,4-tetrahydroquinoxalines **III** (R = Ar; δ 6.87–7.12 ppm [12, 13, 15]). In going to $CDCl_3$, the chemical shift of H^{α} decreases, on the average, by 0.4 ppm. An analogous upfield shift of the H^{α} signal (Δδ 0.6 ppm) is typical of the ^1H NMR spectra

* For communication IV, see [1].

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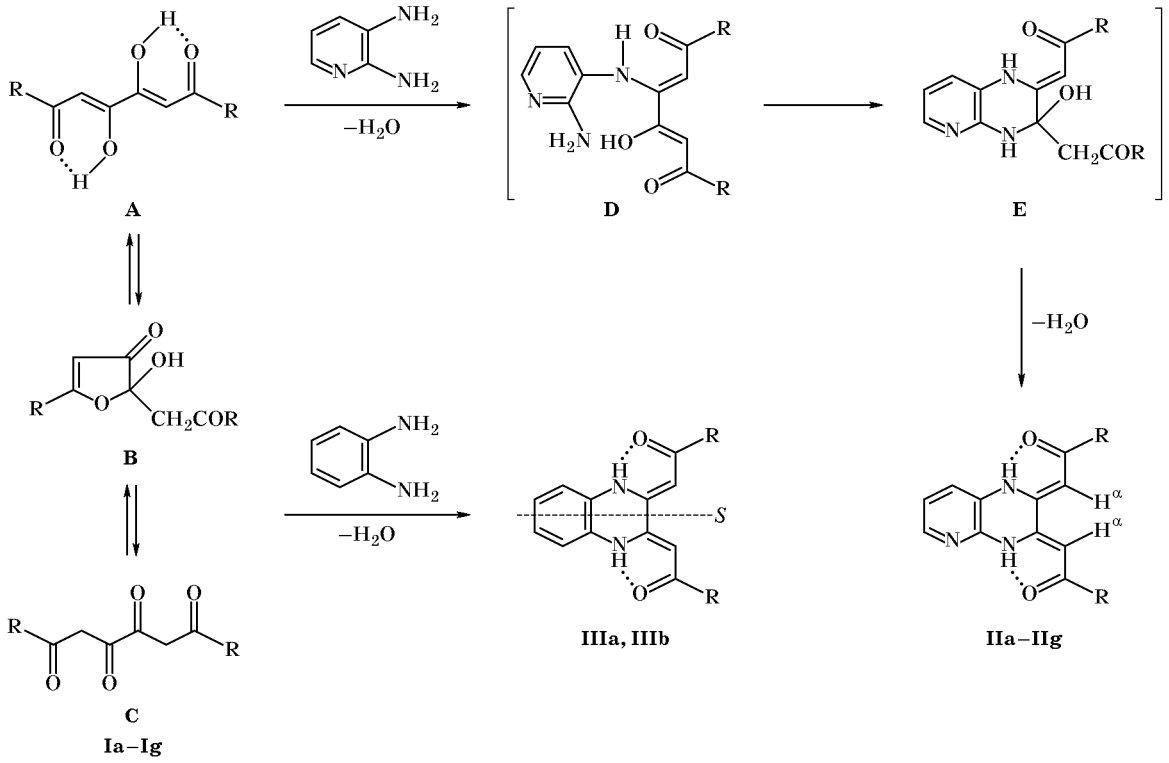
Scheme 1.



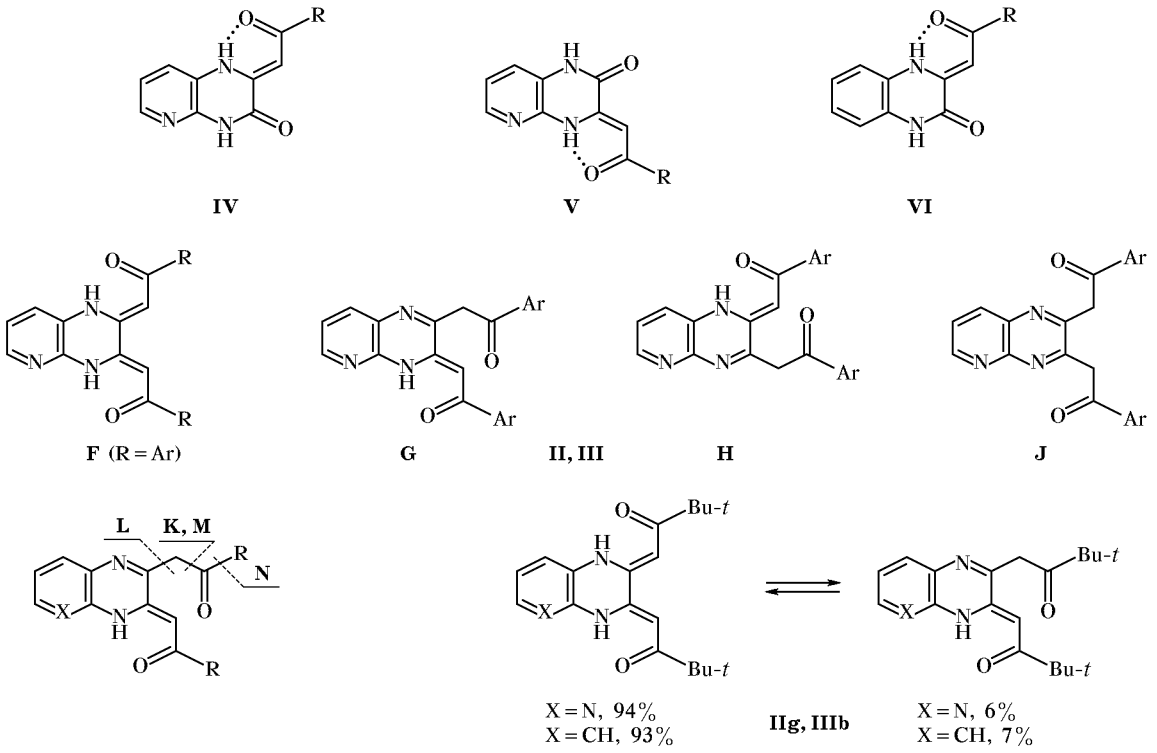
I, **II**, R = C_6H_5 (**a**), 4-$CH_3C_6H_4$ (**b**), 2,4-$(CH_3)_2C_6H_3$ (**c**), 4-$CH_3OC_6H_4$ (**d**), 4-ClC_6H_4 (**e**), CH_3 (**f**), $(CH_3)_3C$ (**g**).
III, R = C_6H_5 (**a**), $(CH_3)_3C$ (**b**).

of quinoxalines **III** in going from DMSO-d_6 to $CDCl_3$ [12, 13]. This may be due to the lack of specific solvation in deuterated chloroform [15]. The position of the H^{α} signal almost does not change on addition of trifluoroacetic acid to solutions of **II** in DMSO-d_6, but it shifts upfield in pure CF_3COOH.

The H^{α} protons in compound **IIc** in DMSO-d_6 are nonequivalent, but the difference in their chemical shifts is very small, δ 6.41 and 6.46 ppm. Probably, an analogous pattern could be expected for the other pyrido[2,3-*b*]pyrazines **II** provided that the operating frequency of NMR spectrometer was sufficiently high. The H^{α} signals in the ^1H NMR spectra of acetylmethylene and pivaloylmethylene derivatives **IIf** (R = CH_3) and **IIg** (R = *tert*-C_4H_9) appear at δ 6.09–6.16 ppm, i.e., about 0.8 ppm upfield relative to the corresponding signals of aroylmethylene-substituted compounds **II** (R = Ar). Analogous signal of model (2*Z*,3*Z*)-bis(pivaloylmethylene)-1,2,3,4-tetrahydroquinoxaline (**IIIb**) is located at δ 6.00 ppm. The H^{α} protons in compound **IIg** dissolved in DMSO-d_6 are nonequivalent: δ 6.12 and 6.16 ppm; obviously, this is the result of a weak effect of the nitrogen atom in the pyridine moiety.

Unlike quinoxalines **III**, molecules of pyrido[2,3-*b*]pyrazines **II** have no symmetry axis *S* which is orthogonal to the C^2-C^3 bond (Scheme 1); however, in most cases, the H^{α} protons of **II** give only one singlet in the ^1H NMR spectra. As noted above, exceptions are compounds **IIc** and **IIg** for which the difference in the chemical shifts is 0.05 and 0.04 ppm, respectively. Such a small difference seems to be surprising, and it indicates once more either the lack or the presence of a very weak effect of the pyridine nitrogen atom on the H^{α} protons. Its effect on the chemical shifts of the NH protons in the pyrazine fragment is also insignificant. The NH signals appear separately only in the spectra of compounds **IIc**, **IId**, and **IIg** in DMSO-d_6: Δδ 0.09–0.23 ppm (Table 2). The NH signals of the other compounds of this series resemble those of symmetric quinoxalines **III**. The only difference is that the NH protons in **II** are shielded more strongly. The Δδ(NH) value for 2,3-bis(pivaloylmethylene)pyrido[2,3-*b*]pyrazine (**IIg**) increases to 0.51 ppm in going from DMSO-d_6 to $CDCl_3$, δ(NH) 13.68 and 14.19 ppm. As expected, the NH signals in the spectra of all compounds **II** disappear on addition of trifluoroacetic acid.

Scheme 2.



By analogy with pyrido[2,3-*b*]pyrazin-2-ones **IV** studied previously [5], in the ^{1}H NMR spectrum of pyrido[2,3-*b*]pyrazine **IIg** in DMSO-d_6, apart from signals of the major tautomer (**F**), we clearly observed a minor signal from CH_2 group (δ 4.22 ppm) belonging to isomer **G** (Table 2, Scheme 2); its fraction was estimated at 6%. In solutions of the other compounds **II** we detected no appreciable amounts of tautomeric form **G**, **H**, or **J**. For comparison, quinoxaline **IIIb** in DMSO-d_6 also gives rise to 7% of tautomer **G**, δ(CH_2) 4.26 ppm.

The main fragmentation pathways of pyrido[2,3-*b*]-pyrazines **II** and quinoxaline **IIIa** under electron impact include elimination of the R substituent and acyl(or aroyl)methyl group with formation of ions **K**–**N** (Table 3, Scheme 2). The molecular ions of compounds **II** are low-abundant.

According to quantum-chemical calculations, nucleophilic attack on 1,6-disubstituted 3,4-dihydroxy-2,4-hexadiene-1,6-diones **I** should be directed at C^3 or C^4 of the β-diketone moiety of open-chain tautomer **A** or at C^5 of cyclic tautomer **B** [15]. Presumably, in the reactions of **I** with 2,3-diaminopyridine, the latter adds through its 3-amino group at C^3 or C^4 of substrate **A** to give intermediate enamine **D** which undergoes cyclization with participation of the second amino group. Elimination of water molecule from cyclic structure **E** yields the final product, (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydropyrido[2,3-*b*]-pyrazine **II**.

Preliminary tests showed that some pyrido[2,3-*b*]-pyrazines **II** exhibit an appreciable bacteriostatic activity against standard strains of *Staphylococcus aureus* P-209 and *Escherichia coli* M17 [7, 8].

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrometer from samples dispersed in mineral oil. The ^{1}H NMR spectra were obtained on RYa-2310 (60 MHz), Bruker AC-300 (300.13 MHz), and Bruker DRX-500 (500.13 MHz) instruments in $CDCl_3$, $CDCl_3$–CF_3COOH (10 : 1), DMSO-d_6, DMSO-d_6–CF_3COOH (10 : 1), or CF_3COOH (for compounds poorly soluble in DMSO); tetramethylsilane or hexamethyldisiloxane was used as internal reference. The mass spectra (electron impact, 70 eV) were run on a Kratos MS-30 instrument (United Kingdom) with direct sample admission into the ion source; emission current 1000 mA, vaporizer temperature 100–130°C. The purity of the products was checked by TLC on Silufol UV-254 plates using benzene–ether–acetone (10 : 9 : 1) as eluent and iodine vapor as developer. The initial compounds, 1,6-diaryl-3,4-dihydroxy-2,4-

Table 1. Yields, melting points, and analytical data of (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydropyrido[2,3-*b*]-pyrazines **IIa**–**IIg**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IIa	67	185–186[a]	74.86	4.80	11.59	$C_{23}H_{17}N_3O_2$	75.19	4.66	11.44
IIb	62	194–195[a]	75.71	5.54	10.38	$C_{25}H_{21}N_3O_2$	75.93	5.35	10.63
IIc	58	156–157[a]	76.42	6.17	10.23	$C_{27}H_{25}N_3O_2$	76.57	5.95	9.92
IId	64	208–209[a]	70.46	4.84	9.78	$C_{25}H_{21}N_3O_4$	70.25	4.95	9.83
IIe	65	215–216[a]	63.56	3.63	9.79	$C_{23}H_{15}Cl_2N_3O_2$[b]	63.32	3.47	9.63
IIf	51	204–205[a]	63.87	5.64	17.51	$C_{13}H_{13}N_3O_2$	64.19	5.39	17.27
IIg	61	171–172	70.02	7.85	12.37	$C_{19}H_{25}N_3O_2$	69.70	7.69	12.83

[a] With decomposition.
[b] Found Cl, %: 16.44. Calculated Cl, %: 16.25.

Table 2. ^{1}H NMR spectra of (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydropyrido[2,3-*b*]pyrazines **IIa**–**IIg** and (2*Z*,3*Z*)-bis(acylmethylene)-1,2,3,4-tetrahydroquinoxalines **IIIa** and **IIIb**

Comp. no.	Solvent	Chemical shifts δ, ppm
IIa	$CDCl_3$	6.55 s (2H, CH), 6.95–8.05 m (13H, H_{arom}, 5-H–7-H), 14.24 s (2H, NH)
	DMSO-d_6	6.81 s (2H, CH), 7.15–8.18 m (13H, H_{arom}, 5-H–7-H), 13.68 s (2H, NH)
	DMSO-d_6–CF_3COOH, 10 : 1	6.78 s (2H, CH), 7.20–8.15 m (13H, H_{arom}, 5-H–7-H)
IIb	DMSO-d_6	2.42 s (6H, CH_3), 7.08 s (2H, CH), 7.26–8.05 m (11H, H_{arom}, 5-H–7-H)
	DMSO-d_6–CF_3COOH, 10 : 1	2.40 s (6H, CH_3), 7.08 s (2H, CH), 7.22–8.01 m (11H, H_{arom}, 5-H–7-H)
IIc	DMSO-d_6	2.27 s, 2.35 s (12H, CH_3), 6.41 s (1H, CH), 6.46 s (1H, CH), 7.01–7.82 m (9H, H_{arom}, 5-H–7-H), 14.19 s (1H, NH), 14.28 s (1H, NH)
	DMSO-d_6–CF_3COOH, 10 : 1	2.25 s, 2.38 s (12H, CH_3), 6.48 s (2H, CH), 7.03–7.92 m (9H, H_{arom}, 5-H–7-H)
IId	DMSO-d_6	3.84 s (6H, CH_3O), 6.87 s (2H, CH), 6.98–7.90 m (11H, H_{arom}, 5-H–7-H), 14.45 s (1H, NH), 14.68 s (1H, NH)
	CF_3COOH	3.58 s (6H, CH_3O), 6.76 s (2H, CH), 7.10–7.90 m (11H, H_{arom}, 5-H–7-H)
IIe[a]	DMSO-d_6	6.90 s (2H, CH), 7.15–8.03 m (11H, H_{arom}, 5-H–7-H)
IIf	DMSO-d_6	2.17 s (6H, CH_3), 6.09 s (2H, CH), 7.08–7.97 m (3H, 5-H–7-H), 13.51 s (2H, NH)
IIg	$CDCl_3$	1.19 s (18H, CH_3), 5.91 s (2H, CH), 6.89 t, 7.22 d, 7.95 d (3H, 5-H–7-H), 13.68 s (1H, NH), 14.19 s (1H, NH)
	DMSO-d_6	1.20 s (18H, CH_3), 4.22 s (2H, CH_2, **G**, 6%), 5.61 s (1H, CH, **G**), 6.12 s (1H, CH, **F**, 94%), 6.16 s (1H, CH, **E**), 7.05 t, 7.67 d, 8.00 d (3H, 5-H–7-H), 13.82 s (1H, NH), 13.98 s (1H, NH)
	DMSO-d_6–CF_3COOH, 10 : 1	1.22 s (18H, CH_3), 6.03 s (1H, CH), 6.12 s (1H, CH), 7.17 t, 7.61 d, 8.06 d (3H, 5-H–7-H)
IIIa	DMSO-d_6	6.87 s (2H, CH) {6.44 ($CDCl_3$) [13]}, 7.21–8.17 m (14H, H_{arom}), 15.18 s (2H, NH)
IIIb	$CDCl_3$	1.22 s (18H, CH_3), 5.90 s (2H, CH), 7.04 s (4H, H_{arom})
	DMSO-d_6	1.21 s (18H, CH_3), 4.26 s (2H, CH_2, **G**, 7%), 5.51 s (1H, CH, **G**), 6.00 s (2H, CH, **E**, 93%), 7.10, 7.26 m (4H, H_{arom}), 14.32 s (2H, NH)

[a] Poorly soluble in DMSO-d_6.

Table 3. Mass spectra of pyrido[2,3-*b*]pyrazines **IIa**, **IIc**, **IId**, and **IIg** and (2*Z*,3*Z*)-bis(benzoylmethylene)-1,2,3,4-tetrahydroquinoxaline (**IIIa**)

Comp. no.	*m*/*z* (I_{rel}, %)[a]
IIa	368 (6) $[M+1]^+$, 367 (14) $M^{+\cdot}$, 365 (5) $[M-2H]^{+\cdot}$, 349 (7) $[M-H_2O]^{+\cdot}$, 272 (7), 265 (18) $[M-C_5H_2NCN]^+$, 264 (9), 263 (34), 262 (100) $[M-C_6H_5CO]^+$ (**K**), 248 (23) $[M-C_6H_5COCH_2]^+$ (**L**), 245 (12), 244 (7), 234 (6), 233 (5), 188 (6), 147 (6), 131 (7), 114 (5), 106 (13), 105 (95) $[C_6H_5C\equiv O]^+$ (**M**), 104 (6), 103 (7), 102 (6), 91 (6), 78 (20), 77 (92) $[C_6H_5]^+$ (**N**), 76 (8)
IIc	423 (9) $M^{+\cdot}$, 291 (18), 290 (100) $[M-2,4\text{-}(CH_3)_2C_6H_3CO]^+$ (**K**), 247 (5), 217 (22), 176 (8), 175 (69), 148 (5), 134 (7), 133 (79) $[2,4\text{-}(CH_3)_2C_6H_3C\equiv O]^+$ (**M**), 115 (5), 107 (11), 105 (32) $[2,4\text{-}(CH_3)_2C_6H_3]^+$ (**N**), 103 (14), 91 (9), 79 (15), 78 (6), 77 (17), 69 (37), 44 (7), 40 (18)
IId	427 (3) $M^{+\cdot}$, 293 (10), 292 (67) $[M-4\text{-}CH_3OC_6H_4CO]^+$ (**K**), 278 (3) $[M-4\text{-}CH_3OC_6H_4COCH_2]^+$ (**L**), 187 (7), 150 (11), 136 (7), 135 (100) $[4\text{-}CH_3OC_6H_4C\equiv O]^+$ (**M**), 109 (8), 108 (7), 107 (11) $[4\text{-}CH_3OC_6H_4]^+$ (**N**), 92 (21), 78 (6), 77 (29), 69 (17)
IIg	327 (7) $M^{+\cdot}$, 270 (15) $[M-(CH_3)_3C]^+$, 243 (17), 242 (100) $[M-(CH_3)_3CCO]^+$ (**K**), 186 (5), 185 (57) $[M-(CH_3)_3C-(CH_3)_3CCO]^+$, 184 (18), 158 (5), 157 (10) $[M-2(CH_3)_3CCO]^+$, 104 (5), 57 (32) $[(CH_3)_3C]^+$, 41 (12)
IIIa	367 (8) $[M+1]^+$, 366 (31) $M^{+\cdot}$, 338 (7) $[M-CO]^{+\cdot}$, 264 (9) $[M-C_6H_4CN]^+$, 263 (5), 262 (24), 261 (100) $[M-C_6H_5CO]^+$ (**K**), 247 (7) $[M-C_6H_5COCH_2]^+$ (**L**), 106 (8), 105 (91) $[C_6H_5C\equiv O]^+$ (**M**), 97 (7), 85 (5), 83 (9), 81 (7), 78 (7), 77 (18) $[C_6H_5]^+$ (**N**), 73 (7), 71 (9), 69 (16), 60 (14), 57 (18), 55 (16), 51 (9), 45 (17), 43 (22), 41 (16)

[a] Given are ion peaks with I_{rel} > 5%. Boldface characters **K**–**N** denote common fragment ions (Scheme 2).

hexadiene-1,6-diones **Ia**–**Ie** were synthesized by the Claisen condensation of excess aryl methyl ketone with diethyl oxalate in the presence of sodium methoxide or ethoxide [1, 9, 11, 16]. 4,5-Dihydroxy-3,5-octadiene-2,7-dione (2,4,5,7-tetraoxooctane, **If**) was prepared as described in [1, 17]. 5,6-Dihydroxy-2,2,9,9-tetramethyl-4,6-decadiene-3,8-dione (2,2,9,9-tetramethyl-3,5,6,8-tetraoxodecane, **Ig**) was obtained by the Claisen condensation of excess pinacolone with diethyl oxalate in the presence of sodium methoxide [18, 19]. Model (2*Z*,3*Z*)-bis(benzoylmethylene)-1,2,3,4-tetrahydroquinoxaline (**IIIa**) was synthesized by reaction of 3,4-dihydroxy-1,6-diphenyl-2,4-hexadiene-1,6-dione (**Ia**) with *o*-phenylenediamine [13]; its spectral parameters were reported in [12–14]. 2,3-Diaminopyridine was purchased from Maybridge Chemical Company, Tintagel, Cornwall (United Kingdom), mp 111–112°C; it was used without additional purification.

(2*Z*,3*Z*)-Bis(aroylmethylene)-1,2,3,4-tetrahydropyrido[2,3-*b*]pyrazines IIa–IIe (*general procedure*). A mixture of 0.002 mol of tetraoxo compound **Ia**–**Ie** and 0.22 g (0.002 mol) of 2,3-diaminopyridine in 15–20 ml of acetic acid was heated until it became homogeneous. The mixture was cooled, and the precipitate was filtered off and recrystallized from ethanol or ethyl acetate.

(2*Z*,3*Z*)-Bis(*p*-methoxybenzoylmethylene)-1,2,3,4-tetrahydropyrido[2,3-*b*]pyrazine (IId). IR spectrum, ν, cm^{-1}: 1590–1607 ($C=O_{chelat}$, C=C), 1517, 1463.

(2*Z*,3*Z*-Bis(acetonylidene)-1,2,3,4-tetrahydropyrido[2,3-*b*]pyrazine (IIf). A mixture of 0.85 g (0.005 mol) of tetraketone **If** and 0.55 g (0.005 mol) of 2,3-diaminopyridine in 80 ml of ethanol was refluxed for 2–3 min. After cooling, the precipitate was filtered off and recrystallized from ethanol.

(2*Z*,3*Z*)-Bis(pivaloylmethylene)-1,2,3,4-tetrahydropyrido[2,3-*b*]pyrazine (IIg). A mixture of 0.51 g (0.002 mol) of tetraketone **Ig** and 0.22 g (0.002 mol) of 2,3-diaminopyridine in 40 ml of ethanol was refluxed for 5 min. The precipitate was filtered off and recrystallized from ethanol.

(2*Z*,3*Z*)-Bis(pivaloylmethylene)-1,2,3,4-tetrahydroquinoxaline (IIIb). A mixture of 0.51 g (0.002 mol) of tetraketone **If** and 0.21 g (0.002 mol) of *o*-phenylenediamine in 40 ml of ethanol was heated until it became homogeneous, and the resulting solution was refluxed for 5 min and cooled. The precipitate was filtered off, and recrystallized from ethanol.

Yield 0.54 g (83%). mp 155–156°C. Found, %: C 73.34; H 7.86; N 8.80. $C_{20}H_{26}N_2O_2$. Calculated, %: C 73.59; H 8.03; N 8.58.

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