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4-Hydroxy(acyloxy)-3-methoxy(ethoxy)phenylmethylidene-(3-carboxyphenyl)amines

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Abstract — Previously unknown Schiff bases were synthesized by reactions of 3-aminobenzoic acid with 4-hydroxy-3-methoxybenzaldehyde, 3-ethoxy-4-hydroxybenzaldehyde, and esters derived therefrom in methanol.

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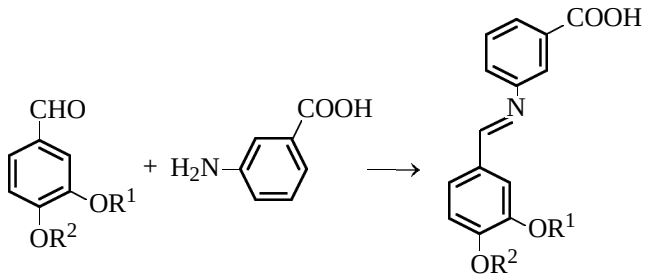
Schiff bases are widely used as biologically active compounds, liquid crystalline substances, dyes, luminophores, and polymer stabilizers [1–4]. Efficient antidepressants, anticonvulsants, and antimicrobial, hypnotic, psychotropic, nematocide, antiinflammatory, antitumor, and other medical agents were designed on the basis of Schiff bases [5, 6]. Due to the presence of a polarized C=N bond, Schiff bases are convenient starting compounds for the synthesis of heterocyclic compounds [7] and β-arylamino ketones which are effective local anaesthetics [8, 9].

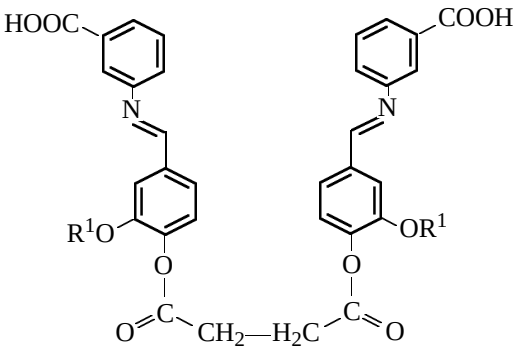
The present article desribes the preparative synthesis of previously unknown derivatives of accessible natural hydroxy aldehydes (4-hydroxy-3-methoxybenzaldehyde and 3-ethoxy-4-hydroxybenzaldehyde) and corresponding esters **I**. By condensation of compounds **I** with 3-aminobenzoic acid (**II**) in boiling anhydrous methanol we obtained 3-[4-hydroxy(or acyloxy)-3-methoxy(or ethoxy)phenylmethylidene-amino]benzoic acids **IIIa**–**IIIz** and **IVa**–**IVk**. The reactions were complete in 1.5–2 h under mild conditions in the absence of a catalyst, so that the labile ester group in the initial aldehydes was preserved. The yields of Schiff bases **IIIa**–**IIIz** and **IVa**–**IVk** were 90–94% (Table 1). Compounds **IIIa**–**IIIz** and **IVa**–**IVk** may be promising from the viewpoints of studying their biological, phototropic, and photochromic properties, synthesis on their basis of valuable products and optical materials [6], and further use in condensations with CH acids with a view to obtain β-arylamino ketones [8, 9].

The structure of Schiff bases **IIIa**–**IIIz** and **IVa**–**IVk** was confirmed by their elemental compositions, molecular weights (determined by titration; Table 1),



I **II** **IIIa**–**IIIy, IVa**–**IVj**

III, R^1 = Me, R^2 = H (**a**), C(O)Me (**b**), C(O)Et (**c**), C(O)Pr (**d**), C(O)Pr-*i* (**e**), $C(O)(CH_2)_6Me$ (**f**), $C(O)(CH_2)_8Me$ (**g**), $C(O)(CH_2)_{11}Me$ (**h**), $C(O)(CH_2)_{16}Me$ (**i**), $C(O)CH{=}CH_2$ (**j**), $C(O)CMe{=}CH_2$ (**k**), $C(O)(CH_2)_7CH{=}CH(CH_2)_7Me$-*cis* (**l**), $C(O)CH_2Ph$ (**m**), $C(O)CH_2CHMePh$ (**n**), $C(O)(CH_2)_2OC_6H_4Me$-4 (**o**), C(O)Ph (**p**), $C(O)C_6H_4Me$-4 (**q**), $C(O)C_6H_4Cl$-2 (**r**), $C(O)C_6H_4Cl$-4 (**s**), $C(O)C_6H_3Cl_2$-2,4 (**t**), $C(O)CH_2OC_6H_3Cl_2$-2,4 (**u**), $C(O)CH_2Br$ (**v**), C(O)CHBrCHBrPh (**w**), $C(O)C_6H_4Br$-4 (**x**), $C(O)C_6H_4NO_2$-3 (**y**); **IV**, R^1 = Et, R^2 = H (**a**), C(O)Me (**b**), C(O)Et (**c**), C(O)Pr (**d**), C(O)Pr-*i* (**e**), $C(O)CH_2CHMe_2$ (**f**), C(O)Ph (**g**), $C(O)C_6H_4Me$-4 (**h**), $C(O)C_6H_4Cl$-2 (**i**), $C(O)C_6H_4Cl$-4 (**j**).



IIIz, IVj

IIIz, R^1 = Me; **IVk**, R^1 = Et.

and ^{1}H NMR, IR, and UV spectra (Table 2). According to the ^{1}H NMR data, all the isolated compounds were almost pure *E* isomers (purity 98±1%). The HC=N proton signal appeared in the ^{1}H NMR spectra of **IIIa**–**IIIz** and **IVa**–**IVk** as a singlet at δ 8.5 ppm. The corresponding signal of the *Z* isomers is usually located in a weaker field (by ~0.5 ppm) due to the deshielding by the benzene ring containing the carboxy group [10].

The steric structure of compounds **IIIa**–**IIIz** and **IVa**–**IVk** was also confirmed by quantum-chemical calculations of the heat of formation H_f of the *E* and *Z* isomers of **IIIa**, **IIIb**, **IIIp**, **IVa**, **IVb**, and **IVg** interms of MNDO PM3 semiempirical approximation

Table 1. Yields, melting points, elemental analyses, and molecular weights of Schiff bases **IIIa**–**IIIz** and **IVa**–**IVk**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %			*M*	
			C	H	N		C	H	N	found	calculated
IIIa	90	136–137	66.74	5.07	4.96	$C_{15}H_{13}NO_4$	66.41	4.83	5.16	266.8	271.3
IIIb	91	165–166	65.48	5.07	4.21	$C_{17}H_{15}NO_5$	65.17	4.83	4.47	307.5	313.3
IIIc	92	143–144	66.36	5.43	4.10	$C_{18}H_{17}NO_5$	66.05	5.23	4.28	314.7	327.3
IIId	90	117–118	67.08	5.74	3.88	$C_{19}H_{19}NO_5$	66.85	5.61	4.10	337.6	341.4
IIIe	93	128–129	67.14	5.80	3.92	$C_{19}H_{19}NO_5$	66.85	5.61	4.10	339.1	341.4
IIIf	90	77–78	69.73	6.98	3.42	$C_{23}H_{27}NO_5$	69.50	6.85	3.52	388.0	397.5
IIIg	92	93–94	70.84	7.52	3.04	$C_{25}H_{31}NO_5$	70.57	7.34	3.29	415.3	425.5
IIIh	90	63–64	72.14	8.11	2.70	$C_{28}H_{37}NO_5$	71.92	7.98	3.00	455.4	467.6
IIIi	91	72–73	74.10	9.05	2.34	$C_{33}H_{47}NO_5$	73.71	8.81	2.60	522.3	537.7
IIIj	90	194–105	66.72	4.80	4.09	$C_{18}H_{15}NO_5$	66.46	4.65	4.31	318.2	325.3
IIIk	92	174–175	67.68	5.19	3.79	$C_{19}H_{17}NO_5$	67.25	5.05	4.13	337.5	339.3
IIIl	90	57–58	74.32	8.70	2.31	$C_{33}H_{45}NO_5$	73.99	8.47	2.61	529.7	535.7
IIIm	93	156–157	71.25	5.16	3.27	$C_{23}H_{19}NO_5$	70.94	4.92	3.60	384.0	389.4
IIIn	91	68–69	72.16	5.70	3.11	$C_{25}H_{23}NO_5$	71.93	5.55	3.36	408.9	417.5
IIIo	94	154–155	69.60	5.48	2.98	$C_{25}H_{23}NO_6$	69.27	5.35	3.23	425.6	433.5
IIIp	94	172–173	70.62	4.63	3.58	$C_{22}H_{17}NO_5$	70.39	4.56	3.73	368.7	375.4
IIIq	93	180–181	71.16	5.08	3.29	$C_{23}H_{19}NO_5$	70.94	4.92	3.60	381.8	389.4
IIIr[a]	92	157–158	64.77	4.09	3.17	$C_{22}H_{16}ClNO_5$	64.48	3.93	3.42	404.1	409.8
IIIs[b]	93	178–179	64.71	4.20	3.22	$C_{22}H_{16}ClNO_5$	64.48	3.93	3.42	402.4	409.8
IIIt[c]	91	210–211	59.76	3.61	2.90	$C_{22}H_{15}Cl_2NO_5$	59.48	3.40	3.15	437.7	444.3
IIIu[d]	92	197–198	58.45	3.82	2.71	$C_{23}H_{17}Cl_2NO_6$	58.24	3.61	2.95	462.2	474.3
IIIv[e]	90	147–148	52.34	3.65	3.29	$C_{17}H_{14}BrNO_5$	52.06	3.60	3.57	384.3	387.0
IIIw[f]	90	89–90	51.62	3.52	2.33	$C_{24}H_{19}Br_2NO_5$	51.36	3.41	2.50	553.0	261.2
IIIx[g]	92	182–183	58.49	3.67	2.90	$C_{22}H_{16}BrNO_5$	58.17	3.55	3.08	447.5	454.3
IIIy	92	210–211	63.11	4.05	6.34	$C_{22}H_{16}N_2O_7$	62.86	3.84	6.66	412.9	417.6
IIIz	94	241–242	65.70	4.71	4.18	$C_{34}H_{28}N_2O_{10}$	65.38	4.52	4.49	610.3	624.6
IVa	90	205–206	67.61	5.48	4.65	$C_{16}H_{15}NO_4$	67.36	5.30	4.91	277.6	285.3
IVb	91	135–136	66.42	5.28	3.90	$C_{18}H_{17}NO_5$	66.05	5.23	4.28	319.4	327.3
IVc	92	148–149	66.96	5.74	3.76	$C_{19}H_{19}NO_5$	66.85	5.61	4.10	336.3	341.4
IVd	90	104–105	67.80	6.11	3.75	$C_{20}H_{21}NO_5$	67.59	5.96	3.94	347.8	355.4
IVe	92	142–143	67.84	6.12	3.70	$C_{20}H_{21}NO_5$	67.59	5.96	3.94	349.2	355.4
IVf	92	92–93	68.57	6.46	3.62	$C_{21}H_{23}NO_5$	68.28	6.28	3.79	360.1	369.4

Table 1. (Contd.)

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %			*M*	
			C	H	N		C	H	N	found	calculated
IVg	93	172–173	71.32	5.20	3.47	$C_{23}H_{19}NO_5$	70.94	4.92	3.60	382.6	389.4
IVh	92	159–160	71.72	5.34	3.19	$C_{24}H_{21}NO_5$	71.45	5.25	3.47	394.7	403.4
IVi[h]	92	136–137	65.42	4.53	3.17	$C_{23}H_{18}ClNO_5$	65.18	4.28	3.30	414.8	423.9
IVj[i]	91	156–157	65.24	4.43	3.09	$C_{23}H_{18}ClNO_5$	65.18	4.28	3.30	419.0	423.9
IVk	94	199–200	66.52	5.12	4.08	$C_{36}H_{32}N_2O_{10}$	66.25	4.94	4.29	644.5	652.7

[a] Found Cl, %: 8.34. Calculated Cl, %: 8.65. [b] Found Cl, %: 8.26. Calculated Cl, %: 8.65. [c] Found Cl, %: 15.70. Calculated Cl, %: 15.96. [d] Found Cl, %: 14.68. Calculated Cl, %: 14.95. [e] Found Br, %: 20.04. Calculated Br, %: 20.37. [f] Found Br, %: 28.08. Calculated Br, %: 28.47. [g] Found Br, %: 17.29. Calculated Br, %: 17.59. [h] Found Cl, %: 8.12. Calculated Cl, %: 8.36. [i] Found Cl, %: 8.19. Calculated Cl, %: 8.36.

Table 2. IR, UV, and ^{1}H NMR spectra of Schiff bases **IIIa**–**IIIz** and **IVa**–**IVk**

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)	^{1}H NMR spectrum, δ, ppm
IIIa	1800–3670 (OH); 3087, 3065, 3010 (CH_{Ar} and =CH); 2961, 2922, 2853, 2819 (CH_{Alk}); 1658 (C=O); 1629 (C=N); 1584, 1518, 1591, 1481, 1457, 1418, 1364 (Ar); 1311, 1267, 1250, 1220, 1168, 1129, 1118, 1034, 1023 (C–O); 863, 837, 819, 787, 754, 738, 674, 640, 600 (CH_{Ar})	205 (20000), 220 (30000), 250 (12000), 307 (4000)	3.95 s (3H, CH_3O), 6.50 br.s (1H, OH), 6.90–7.90 m (7H, C_6H_3 and C_6H_4), 8.54 s (1H, HC=N), 9.90 s (1H, CO_2H)
IIIb	2050–3640 (OH); 3100, 3085, 3070, 3040, 3005 (CH_{Ar} and =CH); 2980, 2940, 2905, 2885, 2845, 2820 (CH_{Alk}); 1740, 1697 (C=O); 1635 (C=N); 1597, 1578, 1511, 1460, 1450, 1416, 1367 (Ar); 1309, 1277, 1235, 1218, 1200, 1153, 1123, 1080, 1035, 921 (C–O); 860, 840, 815, 790, 762, 743, 692, 660, 630, 615, 600 (CH_{Ar})	204 (17000), 221 (29000), 250 (10000), 306 (3000)	2.27 s (3H, CH_3COO), 3.88 s (3H, CH_3O), 6.90–8.00 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.96 s (1H, CO_2H)
IIIc	2150–3640 (OH); 3090, 3080, 3060, 3035, 3005 (CH_{Ar} and =CH); 2980, 2935, 2880, 2855, 2820 (CH_{Alk}); 1744, 1698 (C=O); 1631 (C=N); 1595, 1584, 1508, 1486, 1459, 1443, 1416, 1372 (Ar); 1307, 1275, 1225, 1201, 1152, 1127, 1075, 1033, 987 (C–O); 863, 838, 815, 789, 760, 744, 720, 688, 624 (CH_{Ar})	204 (18000), 221 (28000), 252 (11000), 306 (3000)	1.27 t (3H, CH_3CH_2), 2.55 q (2H, CH_2), 3.88 s (3H, CH_3O), 6.90–8.02 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.97 s (1H, CO_2H)
IIId	2140–3630 (OH); 3077, 3056, 2999 (CH_{Ar} and =CH); 2965, 2936, 2922, 2876, 2852, 2835 (CH_{Alk}); 1743, 1688 (C=O); 1632 (C=N); 1582, 1508, 1490, 1458, 1444, 1417, 1380, 1372, 1349 (Ar); 1311, 1272, 1250, 1215, 1199, 1153, 1118, 1082, 1035, 973 (C–O); 906, 866, 847, 818, 800, 785, 761, 743, 685, 621 (CH_{Ar})	205 (19000), 220 (28000), 252 (12000), 306 (3000)	1.04 t (3H, CH_3), 1.66 m (2H, CH_2), 2.55 t (2H, CH_2), 3.87 s (3H, CH_3O), 6.90–8.04 m (7H, C_6H_3 and C_6H_4), 8.58 s (1H, HC=N), 9.97 s (1H, CO_2H)
IIIe	2100–3650 (OH); 3100, 3085, 3070, 3005 (CH_{Ar} and =CH); 2980, 2940, 2920, 2880, 2845, 2830 (CH_{Alk}); 1746, 1688 (C=O); 1630 (C=N); 1596, 1575, 1504, 1460, 1452, 1416, 1385, 1375, 1355 (Ar); 1297, 1274, 1220, 1198, 1181, 1152, 1133, 1117, 1102, 1032, 998, 969 (C–O); 907, 866, 860, 818, 808, 782, 762, 743, 688, 669, 617 (CH_{Ar})	204 (17000), 221 (29000), 250 (10000), 306 (3000)	1.37 d [6H, $(CH_3)_2C$], 2.90 kvintet (1H, CH), 3.87 s (3H, CH_3O), 6.90–8.04 m (7H, C_6H_3 and C_6H_4), 8.58 s (1H, HC=N), 9.97 s (1H, CO_2H)

Table 2. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)	^1H NMR spectrum, δ, ppm
IIIf	2000–3630 (OH); 3100, 3080, 3075, 3050, 3002 (CH_{Ar} and =CH); 2956, 2927, 2870, 2855 (CH_{Alk}); 1762, 1697 (C=O); 1629 (C=N); 1601, 1586, 1578, 1512, 1455, 1416, 1380, 1355 (Ar); 1316, 1289, 1276, 1268, 1215, 1200, 1160, 1132, 1110, 1030, 980 (C–O); 920, 905, 866, 826, 808, 794, 762, 729, 688, 669, 617 (CH_{Ar})	205 (17000), 220 (29000), 250 (11000), 305 (3000)	0.94 t (3H, CH_3), 1.35 m [8H, $(CH_2)_4$], 1.80 m (2H, CH_2), 2.60 m (2H, CH_2), 3.88 s (3H, CH_3O), 6.95–8.05 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIg	2100–3620 (OH); 3100, 3080, 3070, 3012 (CH_{Ar} and =CH); 2951, 2924, 2870, 2855 (CH_{Alk}); 1766, 1693, 1682 (C=O); 1630 (C=N); 1600, 1580, 1511, 1455, 1416, 1380 (Ar); 1321, 1286, 1266, 1220, 1200, 1175, 1132, 1110, 1080, 1060, 1034, 1000, 980 (C–O); 905, 865, 840, 810, 795, 780, 762, 755, 720, 685, 660, 615 (CH_{Ar})	204 (18000), 220 (29000), 250 (10000), 306 (3000)	0.92 t (3H, CH_3), 1.20–1.50 m [12H, $(CH_2)_6$], 1.87 t (2H, CH_2), 2.67 t (2H, CH_2), 3.88 s (3H, CH_3O), 6.90–8.05 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.97 s (1H, CO_2H)
IIIh	2150–3650 (OH); 3090, 3070, 3040, 3008 (CH_{Ar} and =CH); 2954, 2921, 2851 (CH_{Alk}); 1762, 1698, 1683 (C=O); 1628 (C=N); 1600, 1582, 1509, 1486, 1465, 1454, 1418, 1376 (Ar); 1315, 1273, 1198, 1150, 1119, 1108, 1076, 1060, 1031, 1000, 970 (C–O); 896, 868, 825, 818, 800, 783, 759, 720, 680, 619 (CH_{Ar})	204 (17000), 221 (29000), 250 (11000), 305 (3000)	0.90 t (3H, CH_3), 1.34 m [18H, $(CH_2)_9$], 1.78 m (2H, CH_2), 2.66 t (2H, CH_2), 3.87 s (3H, CH_3O), 6.95–8.10 m (7H, C_6H_3 and C_6H_4), 8.58 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIi	2150–3650 (OH); 3080, 3065, 3010 (CH_{Ar} and =CH); 2955, 2917, 2849 (CH_{Alk}); 1767, 1700, 1683 (C=O); 1631 (C=N); 1600, 1581, 1510, 1466, 1453, 1417, 1379, 1370 (Ar); 1314, 1290, 1273, 1245, 1210, 1201, 1148, 1113, 1057, 1033 (C–O); 917, 890, 875, 855, 840, 815, 795, 785, 756, 720, 685, 670, 620 (CH_{Ar})	204 (18000), 220 (28000), 250 (10000), 307 (3000)	0.89 t (3H, CH_3), 1.04–1.90 m [30H, $(CH_2)_{15}$], 2.55 t (2H, CH_2), 3.90 s (3H, CH_3O), 6.95–7.95 m (7H, C_6H_3 and C_6H_4), 8.61 s (1H, HC=N), 9.94 s (1H, CO_2H)
IIIj	2130–3640 (OH); 3090, 3072, 3010 (CH_{Ar} and =CH); 2958, 2924, 2870, 2853 (CH_{Alk}); 1762, 1691 (C=O); 1657 (C=C); 1630 (C=N); 1605, 1592, 1547, 1511, 1452, 1419, 1366 (Ar); 1220, 1270, 1202, 1155, 1123, 1031, 980 (C–O); 935, 900, 880, 860, 820, 755, 680, 665, 650 (CH_{Ar} and =CH)	205 (23000), 220 (32000), 252 (12000), 306 (4000)	3.88 s (3H, CH_3O), 5.82–6.86 m ($CH=CH_2$), 6.95–8.00 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIk	2050–3640 (OH); 3100, 3080, 3060, 3040, 3005 (CH_{Ar} and =CH); 2980, 2970, 2960, 2940, 2880, 2840, 2820 (CH_{Alk}); 1726, 1696 (C=O); 1632 1595, 1577, 1510, 1480, 1461, 1450, 1415, 1375 (Ar); 1325, 1305, 1285, 1277, 1146, 1080, 1033, 970 (C–O); 909, 870, 855, 820, 810, 790, 755, 740, 690, 675, 645, 635, 620, 605 (CH_{Ar} and =CH)	205 (24000), 221 (32000), 250 (12000), 305 (4000)	2.08 t (3H, CH_3), 3.89 s (3H, CH_3O), 5.82 t (1H, =CH), 6.42 t (C=N); (1H, =CH), 7.00–8.05 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIl	2350–3700 (OH); 3070, 3006 (CH_{Ar} and =CH); 2955, 2925, 2854 (CH_{Alk}); 1765, 1697 (C=O); 1658 (C=C); 1630 (C=N); 1600, 1584, 1508, 1465, 1418, 1376 (Ar); 1320, 1273, 1199, 1152, 1120, 1035, 980 (C–O); 920, 870, 820, 780, 759, 740, 723, 688, 665, 630 (CH_{Ar} and =CH)	206 (23000), 220 (32000), 250 (12000), 306 (4000)	0.89 t (3H, CH_3), 1.10–2.20 m [22H, $(CH_2)_5$ and $(CH_2)_6$], 2.62 t (2H, CH_2), 3.88 s (3H, CH_3O), 5.42 t [2H, 2(=CH)], 6.95–8.10 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)

Table 2. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)	^1H NMR spectrum, δ, ppm
IIIm	2100–3650 (OH); 3090, 3058, 3029, 3015, 2998 (CH_{Ar} and =CH); 2975, 2947, 2930, 2920, 2880, 2852, 2827 (CH_{Alk}); 1764, 1687 (C=O); 1632 (C=N); 1595, 1577, 1505, 1460, 1452, 1416, 1380, 1340 (Ar); 1303, 1274, 1232, 1219, 1198, 1151, 1134, 1117, 1077, 1031, 980, 946 (C–O); 921, 906, 866, 840, 820, 790, 761, 745, 726, 715, 691, 680, 640, 620, 605 (CH_{Ar})	207 (24000), 220 (32000), 250 (11000), 305 (3000)	3.80 s (2H, CH_2), 3.90 s (3H, CH_3O), 7.00–8.10 m (12H, C_6H_3, C_6H_4 and C_6H_5), 8.61 s (1H, HC=N), 10.02 s (1H, CO_2H)
IIIn	2300–3700 (OH); 2980, 2965, 3029, 3002 (CH_{Ar} and =CH); 2964, 2940, 2920, 2875, 2831 (CH_{Alk}); 1758, 1695 (C=O); 1631 (C=N); 1600, 1581, 1505, 1455, 1440, 1418, 1369, 1355 (Ar); 1315, 1273, 1245, 1199, 1151, 1122, 1082, 1033, 980 (C–O); 920, 870, 840, 825, 810, 780, 762, 740, 701, 689, 660, 620 (CH_{Ar})	206 (25000), 220 (31000), 250 (11000), 305 (4000)	1.43 d (3H, CH_3), 2.90 d (2H, CH_2), 3.42 q (1H, CH), 3.90 s (3H, CH_3O), 6.98–8.08 m (12H, C_6H_3, C_6H_4 and C_6H_5), 8.60 s (1H, HC=N), 10.00 s (1H, CO_2H)
IIIo	2030–3650 (OH); 3090, 3075, 3045, 3020, 3008 (CH_{Ar} and =CH); 3080, 2935, 2920, 2882, 2856, 2830 (CH_{Alk}); 1767, 1700, 1679 (C=O); 1625 (C=N); 1609, 1582, 1463, 1447, 1417, 1391, 1370 (Ar); 1315, 1294, 1273, 1249, 1222, 1197, 1159, 1147, 1116, 1075, 1045, 1026, 1012, 971 (C–O); 912, 893, 871, 839, 827, 814, 783, 760, 739, 684, 674, 640, 621 (CH_{Ar})	205 (25000), 222 (35000), 250 (14000), 305 (5000)	2.30 s (3H, CH_3), 3.12 t (2H, CH_2O), 3.89 s (3H, CH_3O), 4.42 t (2H, CH_2C=O), 6.98–8.08 m (11H, C_6H_3 and $2C_6H_4$), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIp	2200–3630 (OH); 3080, 3040, 3007 (CH_{Ar} and =CH); 2960, 2935, 2920, 2880, 2840, 2830 (CH_{Alk}); 1728, 1707 (C=O); 1633 (C=N); 1596, 1580, 1509, 1454, 1417, 1380 (Ar); 1295, 1270, 1260, 1205, 1185, 1157, 1125, 1081, 1066, 1040, 1025, 980 (C–O); 920, 905, 870, 855, 830, 805, 780, 760, 740, 702, 680, 660, 640, 625 (CH_{Ar})	242 (22000), 250 (12000), 207 (22000), 220 (32000), 305 (5000)	3.90 s (3H, CH_3O), 6.90–8.10 m (12H, C_6H_3, C_6H_4 and C_6H_5), 8.64 s (1H, HC=N), 10.04 s (1H, CO_2H)
IIIq	2200–3650 (OH); 3085, 3070, 3008 (CH_{Ar} and =CH); 2970, 2935, 2905, 2880, 2830 (CH_{Alk}); 1727, 1706 (C=O); 1623 (C=N); 1608, 1602, 1579, 1515, 1464, 1442, 1420, 1380, 1360 (Ar); 1321, 1298, 1263, 1202, 1175, 1162, 1121, 1104, 1074, 1029, 1015, 1002, 985 (C–O); 931, 901, 874, 858, 840, 825, 800, 760, 748, 687, 661, 640, 620, 603 (CH_{Ar})	208 (19000), 221 (36000), 252 (30000), 306 (6000)	2.46 s (3H, CH_3), 3.89 s (3H, CH_3O), 7.00–8.10 m (11H, C_6H_3 and $2C_6H_4$), 8.62 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIr	2050–3650 (OH); 3100, 3075, 3005 (CH_{Ar} and =CH); 2965, 2925, 2880, 2820 (CH_{Alk}); 1752, 1694 (C=O); 1627 (C=N); 1590, 1582, 1506, 1472, 1439, 1417, 1370 (Ar); 1290, 1271, 1244, 1208, 1155, 1140, 1122, 1097, 1036, 980 (C–O); 931, 900, 875, 860, 820, 780, 755, 739, 710, 681, 670, 650, 620 (CH_{Ar}); 550 (C–Cl)	207 (37000), 221 (37000), 255 (20000), 305 (5000)	3.90 s (3H, CH_3O), 6.95–8.26 m 2860, (11H, C_6H_3 and $2C_6H_4$), 8.64 s (1H, HC=N), 10.04 s (1H, CO_2H)
IIIs	2200–3650 (OH); 3098, 3080, 3055, 3006 (CH_{Ar} and =CH); 2966, 2936, 2900, 2880, 2855, 2823 (CH_{Alk}); 1738, 1703 (C=O); 1624 (C=N); 1600, 1591, 1580, 1514, 1485, 1464, 1442, 1417, 1400, 1361 (Ar); 1319, 1298, 1262, 1202, 1163, 1120, 1091, 1074, 1030, 1013, 985 (C–O); 932, 900, 872, 859, 848, 824, 799, 760, 754, 708, 687, 680, 643, 613 (CH_{Ar}); 550 (C–Cl)	206 (36000), 221 (37000), 255 (19000), 305 (5000)	3.92 s (3H, CH_3O), 6.90–8.24 m (11H, C_6H_3 and $2C_6H_4$), 8.63 s (1H, HC=N), 10.04 s (1H, CO_2H)

Table 2. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)	^1H NMR spectrum, δ, ppm
IIIt	2100–3640 (OH); 3097, 3073, 3027, 3008 (CH_{Ar} and =CH); 2970, 2940, 2923, 2875, 2852, 2830 (CH_{Alk}); 1751, 1705 (C=O); 1627 (C=N); 1584, 1560, 1504, 1475, 1463, 1440, 1417, 1374 (Ar); 1305, 1270, 1235, 1215, 1204, 1149, 1120, 1093, 1041, 1032, 980 (C–O); 930, 895, 880, 850, 837, 815, 780, 756, 755, 680, 670, 640, 620 (CH_{Ar}); 545 (C–Cl)	210 (43000), 221 (41000), 257 (24000), 306 (6000)	3.90 s (3H, CH_3O), 6.95–8.50 m (10H, $2C_6H_3$ and C_6H_4), 8.62 s (1H, HC=N), 10.04 s (1H, CO_2H)
IIIu	2200–3630 (OH); 3100, 3072, 3040, 3000 (CH_{Ar} and =CH); 2983, 2970, 2931, 2855 (CH_{Alk}); 1786, 1717 (C=O); 1623 (C=N); 1604, 1597, 1580, 1509, 1479, 1470, 1443, 1422, 1393, 1375 (Ar); 1323, 1287, 1270, 1258, 1237, 1210, 1181, 1157, 1120, 1110, 1089, 1047, 1032, 1000, 986 (C–O); 910, 866, 843, 821, 808, 780, 759, 741, 719, 690, 665, 646, 623 (CH_{Ar}); 541, 560 (C–Cl)	206 (40000), 220 (34000), 251 (14000), 305 (4000)	3.90 s (3H, CH_3O), 5.02 s (2H, CH_2), 6.95–8.05 m (10H, $2C_6H_3$ and C_6H_4), 8.64 s (1H, HC=N), 10.02 s (1H, CO_2H)
IIIv	2030–3630 (OH); 3100, 3061, 3012 (CH_{Ar} and =CH); 2966, 2931, 2895, 2876, 2851 (CH_{Alk}); 1761, 1693 (C=O); 1633 (C=N); 1597, 1578, 1509, 1480, 1462, 1447, 1415, 1405, 1369 (Ar); 1308, 1274, 1214, 1194, 1172, 1153, 1123, 1085, 1033, 980, 970 (C–O); 925, 907, 857, 813, 795, 760, 743, 688, 667, 640, 620 (CH_{Ar}); 540 (C–Br)	207 (20000), 221 (28000), 252 (12000), 306 (4000)	3.90 s (3H, CH_3O), 4.42 s (2H, CH_2), 7.04–8.00 m (7H, C_6H_3 and C_6H_4), 8.59 s (1H, HC=N), 9.98 s (1H, CO_2H)
IIIw	2350–3700 (OH); 3065, 3040, 3006 (CH_{Ar} and =CH); 2960, 2939, 2920, 2880, 2833 (CH_{Alk}); 1740, 1693 (C=O); 1631 (C=N); 1601, 1504, 1463, 1448, 1418, 1377 (Ar); 1315, 1275, 1238, 1194, 1181, 1151, 1120, 1077, 1050, 1031, 969 (C–O); 920, 863, 840, 805, 780, 762, 740, 691, 605 (CH_{Ar}); 540 (C–Br)	207 (25000), 221 (32000), 250 (12000), 306 (3000)	3.91 s (3H, CH_3O), 4.32 d (1H, CH), 5.15 d (1H, CH), 7.00–8.04 m (12H, C_6H_3, C_6H_4 and C_6H_5), 8.60 s (1H, HC=N), 10.02 s (1H, CO_2H)
IIIx	2200–3600 (OH); 3096, 3080, 3070, 3040, 3004 (CH_{Ar} and =CH); 2967, 2937, 2905, 2870, 2814 (CH_{Alk}); 1736, 1703 (C=O); 1624 (C=N); 1600, 1580, 1514, 1481, 1464, 1442, 1417, 1397, 1362 (Ar); 1320, 1299, 1262, 1203, 1163, 1120, 1074, 1029, 1009, 987 (C–O); 900, 872, 859, 845, 823, 798, 762, 750, 700, 686, 678, 661, 642, 627, 612 (CH_{Ar}); 550 (C–Br)	206 (35000), 220 (40000), 250 (20000), 306 (5000)	3.90 s (3H, CH_3O), 6.90–8.10 m (11H, C_6H_3 and $2C_6H_4$), 8.63 s (1H, HC=N), 10.04 s (1H, CO_2H)
IIIy	2050–3610 (OH); 3088, 3055, 3004 (CH_{Ar} and =CH); 2968, 2932, 2861, 2822 (CH_{Alk}); 1743, 1692 (C=O); 1631 (C=N); 1620, 1595, 1582, 1507, 1446, 1415 (Ar); 1540, 1348 (NO_2); 1310, 1294, 1270, 1256, 1205, 1152, 1125, 1090, 1080, 1063, 1031, 1000 (C–O); 920, 910, 890, 865, 820, 800, 785, 770, 715, 690, 680, 650, 630, 615 (CH_{Ar})	205 (24000), 220 (54000), 260 (30000), 305 (5000)	3.92 s (3H, CH_3O), 6.95–9.10 m (11H, C_6H_3 and $2C_6H_4$), 8.65 s (1H, HC=N), 10.06 s (1H, CO_2H)
IIIz	2150–3650 (OH); 3100, 3081, 3005 (CH_{Ar} and =CH); 2973, 2941, 2927, 2881, 2849, 2830 (CH_{Alk}); 1745, 1697 (C=O); 1628 (C=N); 1603, 1579, 1505, 1480, 1473, 1456, 1419, 1369 (Ar); 1310, 1276, 1259, 1212, 1197, 1153, 1139, 1117, 1091, 1031, 980 (C–O); 915, 870, 840, 820, 790, 761, 748, 687, 660, 620, 611 (CH_{Ar})	205 (34000), 220 (55000), 251 (20000), 306 (5000)	3.06 s [4H, $(CH_2)_2$], 3.89 s (6H, $2CH_3O$), 7.00–8.00 m (14H, $2C_6H_3$ and $2C_6H_4$), 8.59 s (2H, 2HC=N), 9.99 s (2H, CO_2H)

Table 2. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)	^1H NMR spectrum, δ, ppm
IVa	2000–3620 (OH); 3090, 3071, 3045, 3010 (CH_{Ar} and =CH); 2987, 2951, 2903, 2880, 2853, 2822 (CH_{Alk}); 1686 (C=O); 1628 (C=N); 1604, 1590, 1583, 1509, 1448, 1400, 1385 (Ar); 1308, 1290, 1269, 1249, 1215, 1195, 1181, 1155, 1118, 1075, 1032, 960, 935 (C–O); 900, 880, 826, 783, 762, 750, 826, 783, 762, 750, 688, 670 (CH_{Ar})	204 (20000), 220 (30000), 250 (12000), 306 (4000)	1.32 t (3H, CH_3), 4.18 q (2H, CH_2), 6.50 br.s (1H, OH), 6.98–7.95 m (7H, C_6H_3 and C_6H_4), 8.56 s (1H, HC=N), 9.90 s (1H, CO_2H)
IVb	2000–3600 (OH); 3090, 3060, 3020, 3000 (CH_{Ar} and =CH); 2980, 2935, 2900, 2880, 2850, 2820 (CH_{Alk}); 1748, 1700 (C=O); 1633 (C=N); 1594, 1585, 1510, 1444, 1396, 1368 (Ar); 1297, 1274, 1232, 1199, 1161, 1129, 1054, 1014, 1005, 983 (C–O); 933, 915, 909, 859, 834, 792, 761, 748, 721, 679, 663, 625, 598 (CH_{Ar})	204 (18000), 220 (30000), 250 (10000), 306 (3000)	1.24 t (3H, CH_3), 2.30 s (3H, CH_3COO), 4.12 q (2H, CH_2), 7.00–8.02 m (7H, C_6H_3 and C_6H_4), 8.59 s (1H, HC=N), 9.98 s (1H, CO_2H)
IVc	2100–3620 (OH); 3080, 3059, 3010 (CH_{Ar} and =CH); 2976, 2928, 2900, 2880, 2853, 2820 (CH_{Alk}); 1748, 1690 (C=O); 1629 (C=N); 1584, 1506, 1441, 1432, 1393, 1380 (Ar); 1295, 1272, 1230, 1196, 1160, 1122, 1085, 1060, 1044, 985, 974 (C–O); 931, 909, 890, 875, 840, 815, 790, 760, 750, 682, 620, 602 (CH_{Ar})	205 (19000), 220 (30000), 250 (11000), 305 (3000)	1.28 t (3H, CH_3), 1.42 t (3H, CH_3), 2.70 q (2H, CH_2), 4.15 q (2H, CH_2), 6.98–8.00 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)
IVd	2130–3640 (OH); 3075, 3055, 3012 (CH_{Ar} and =CH); 2976, 2935, 2900, 2879, 2850, 2830 (CH_{Alk}); 1747, 1692 (C=O); 1629 (C=N); 1583, 1505, 1438, 1431, 1393, 1379, 1367 (Ar); 1310, 1271, 1215, 1190, 1157, 1118, 1076, 1043, 1000, 972 (C–O); 926, 908, 867, 842, 812, 798, 785, 1, 752, 683, 671, 618 (CH_{Ar})	204 (18000), 220 (29000), 250 (10000), 305 (3000)	1.02 t (3H, CH_3), 1.24 t (3H, CH_3), 1.64 m (2H, CH_2), 2.55 t (2H, CH_2), 4.15 q (2H, CH_2), 7.02–8.04 m (7H, C_6H_3 and C_6H_4), 8.59 s (1H, HC=N), 9.98 s (1H, CO_2H)
IVe	2030–3640 (OH); 3100, 3071, 3040, 3008 (CH_{Ar} and =CH); 2978, 2934, 2883, 2845, 2830 (CH_{Alk}); 1747, 1690 (C=O); 1630 (C=N); 1591, 1575, 1504, 1471, 1448, 1428, 1418, 1396, 1384, 1367, 1357 (Ar); 1308, 1296, 1272, 1212, 1196, 1180, 1158, 1132, 1116, 1102, 1039, 997, 967 (C–O); 920, 910, 868, 818, 787, 762, 751, 687, 669, 656, 616 (CH_{Ar})	205 (18000), 222 (32000), 248 (11000), 304 (4000)	1.24 t (3H, CH_3), 2.90 quintet (1H, CH), 1.34 d [6H, $(CH_3)_2C$], 4.16 q (2H, CH_2), 6.98–8.02 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.98 s (1H, CO_2H)
IVf	2000–3700 (OH); 3075, 3010 (CH_{Ar} and =CH); 2990, 2968, 2950, 2931, 2872, 2850, 2820 (CH_{Alk}); 1746, 1687 (C=O); 1627 (C=N); 1600, 1575, 1511, 1475, 1450, 1420, 1396, 1370, 1350 (Ar); 1307, 1291, 1263, 1245, 1210, 1195, 1163, 1118, 1105, 1044, 981, 960 (C–O); 920, 911, 880, 870, 850, 825, 760, 700, 682, 665, 640, 620 (CH_{Ar})	204 (18000), 221 (30000), 250 (10000), 305 (3000)	1.10 d [6H, $(CH_3)_2C$], 1.24 t (3H, CH_3), 1.42–2.90 m (3H, CH and CH_2), 4.10 q (2H, CH_2), 7.00– 8.04 m (7H, C_6H_3 and C_6H_4), 8.60 s (1H, HC=N), 9.99 s (1H, CO_2H)
IVg	2130–3630 (OH); 3095, 3080, 3061, 3031, 3005 (CH_{Ar} and =CH); 2983, 2955, 2916, 2900, 2866 (CH_{Alk}); 1728, 1702 (C=O); 1633 (C=N); 1597, 1578, 1509, 1480, 1437, 1424, 1395, 1377, 1365 (Ar); 1313, 1275, 1216, 1202, 1164, 1131, 1082, 1065, 1047, 1026, 995 (C–O); 926, 909, 865, 858, 830, 805, 790, 764, 750, 720, 700, 691, 667, 630, 620 (CH_{Ar})	208 (22000), 220 (31000), 241 (23000), 250 (12000), 305 (5000)	1.26 t (3H, CH_3), 4.20 q (2H, CH_2), 7.04–8.05 m (12H, C_6H_3, C_6H_4 and C_6H_5), 8.64 s (1H, HC=N), 10.04 s (1H, CO_2H)
IVh	2030–2680 (OH); 3100, 3075, 3040, 3007 (CH_{Ar} and =CH); 2990, 2970, 2950, 2935, 2900, 2880, 2840, 2820 (CH_{Alk}); 1734, 1682 (C=O); 1627 (C=N); 1610, 1601, 1574, 1510, 1475, 1449, 1422, 1397, 1360 (Ar); 1315, 1293, 1261, 1115, 1199, 1178, 1162, 1117, 1104, 1071, 1043, 1020, 972 (C–O); 921, 910, 880, 870, 840, 830, 815, 805, 761, 745, 682, 660, 625, 605 (CH_{Ar})	208 (20000), 221 (35000), 252 (30000), 306 (6000)	1.28 t (3H, CH_3), 2.48 s (3H, CH_3), 4.12 q (2H, CH_2), 6.98–8.12 m (11H, C_6H_3 and $2C_6H_4$), 8.62 s (1H, HC=N), 9.98 s (1H, CO_2H)

Table 2. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)	^1H NMR spectrum, δ, ppm
IVi	2000–2650 (OH); 3095, 3068, 3045, 3005 (CH_{Ar} and =CH); 2983, 2970, 2940, 2890, 2863, 2820 (CH_{Alk}); 1757, 1699 (C=O); 1630 (C=N); 1592, 1578, 1505, 1475, 1432, 1413, 1387, 1360 (Ar); 1295, 1271, 1240, 1198, 1159, 1121, 1094, 1031, 980 (C–O); 923, 908, 860, 825, 792, 760, 741, 688, 665, 645, 630, 620 (CH_{Ar}); 552 (C–Cl)	207 (36000), 221 (37000), 255 (21000), 305 (5000)	1.29 t (3H, CH_3), 4.19 q (2H, CH_2), 7.00–8.26 m (11H, C_6H_3 and $2C_6H_4$), 8.65 s (1H, HC=N), 10.02 s (1H, CO_2H)
IVj	2000–3670 (OH); 3071, 3004 (CH_{Ar} and =CH); 2987, 2970, 2950, 2929, 2900, 2880, 2875, 2819 (CH_{Alk}); 1734, 1682 (C=O); 1627, 1610, 1600, 1580, 1574, 1511, 1480, 1447, 1422, 1397, 1360 (Ar); 1318, 1293, 1261, 1230, 1200, 1178, 1162, 1117, 1105, 1071, 1043, 1020, 977 (C–O); 925, 910, 875, 825, 810, 760, 745, 683, 670, 645, 630, 605 (CH_{Ar}); 560 (C–Cl)	206 (36000), 221 (37000), 255 (20000), 305 (5000)	1.29 t (3H, CH_3), 4.20 q (2H, CH_2), 7.02–8.28 m (11H, C_6H_3 and $2C_6H_4$), 8.64 s (1H, HC=N), 10.04 s (1H, CO_2H)
IVk	2130–3650 (OH); 3100, 3080, 3040, 3006 (CH_{Ar} and =CH); 2983, 2970, 2940, 2909, 2870, 2840, 2835 (CH_{Alk}); 1744, 1693 (C=O); 1629 (C=N); 1602, 1590, 1587, 1504, 1485, 1455, 1431, 1419, 1375, 1355 (Ar); 1310, 1271, 1260, 1215, 1190, 1150, 1137, 1115, 1084, 1038, 985 (C–O); 918, 990, 980, 940, 915, 790, 761, 750, 690, 660, 625, 612 (CH_{Ar})	205 (35000), 220 (54000), 250 (20000), 306 (5000)	1.27 t (3H, CH_3), 3.05 s [4H, $(CH_2)_2$], 4.18 q (2H, CH_2), 6.98– 8.00 m (14H, $2C_6H_3$ and $2C_6H_4$), 8.60 s (2H, 2HC=N), 9.98 s (2H, CO_2H)

[11] using the GAMESS program [12]. The calculations were performed with full optimization of all bond lengths and bond and dihedral angles. The following data were obtained, H_f, kcal mol^{-1}: *E* isomers: –101.1 (**IIIa**), –136.3 (**IIIb**), –100.4 (**IIIp**), –107.6 (**IVa**), –141.8 (**IVb**), –106.4 (**IVg**); *Z* isomers: –100.7 (**IIIa**), –135.6 (**IIIb**), –99.5 (**IIIp**), –106.1 (**IVa**), –141.3 (**IVb**), –105.5 (**IVg**). It is seen that the *E* configuration is more favorable than *Z* by 0.4–1.5 kcal mol^{-1}. The results of calculations are in a good agreement with the X-ray diffraction data for structurally related compounds [13–15].

EXPERIMENTAL

The IR spectra were recorded in KBr on a Nicolet Protégé-460 Fourier spectrometer. The UV spectra were measured on a Specord UV-Vis spectrophotometer from 10^{-4} M solutions in methanol. The ^1H NMR spectra were obtained on a Tesla BS-587A instrument (100 MHz) from 5% solutions in DMSO-d_6 using octamethylcyclotetrasiloxane as internal reference. The molecular weights were determined by alkalimetric titration of the carboxy group with a 0.1 N solution of sodium hydroxide in the presence of phenolphthalein as indicator. Initial esters **I** were synthesized by the procedure described in [16].

3-[4-Hydroxy(or acyloxy)-3-methoxy(or ethoxy)phenylmethylideneamino]benzoic acids IIIa–IIIz and IVa–IVk *(general procedure).* 4-Hydroxy-3-methoxybenzaldehyde, 3-ethoxy-4-hydroxybenzaldehyde, or the corresponding ester **I**, 0.01 mol, was dissolved in 50–100 ml of anhydrous methanol, 0.01 mol of 3-aminobenzoic acid (**II**) (0.02 mol in the synthesis of **IIIz** and **IVk**) was added, and the mixture was heated for 1.5–2 h under reflux and left to stand for 20–30 h at 20–23°C. The precipitate of Schiff base **IIIa–IIIz** or **IVa–IVk** was filtered through a glass filter, washed with a small amount of methanol, and dried under reduced pressure. There was no need of additionally purifying the products by recrystallization. The solvent (methanol) can be reused after distillation through a Vigreaux column.

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