

Synthesis and Electron Impact of Mass Spectra of 3-Substituted Chromeno[3,2-*c*]chromen-6,7-diones

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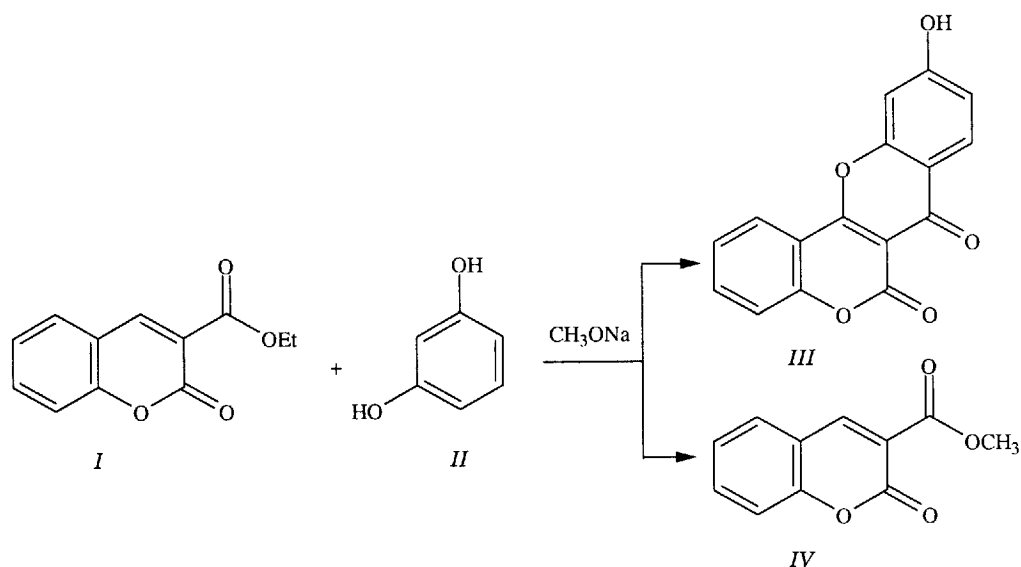
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3-Hydroxychromeno[3,2-*c*]chromen-6,7-dione (*III*) and 3-methoxycarbonylcoumarin (*IV*) were prepared *via* condensation of ethoxycarbonylcoumarin with resorcinol in the presence of sodium methoxide. The chemical behaviour of *III* towards acetic anhydride, alkyl halides, and diazonium chloride is described. EI mass spectrometric behaviour of compounds *IV*, 3-acetyloxy and 3-alkoxy derivatives shows a weak molecular ion peak and a base peak of m/z 89, m/z 280, m/z 91, and m/z 120 resulting from a cleavage fragmentation, respectively. The molecular ion of some chromenochromendiones is a base peak of m/z 280, m/z 366, and m/z 488, respectively. Diphenylazo-hydroxy derivative gives a characteristic fragmentation pattern with two very stable fragments of m/z 383 and m/z 77.

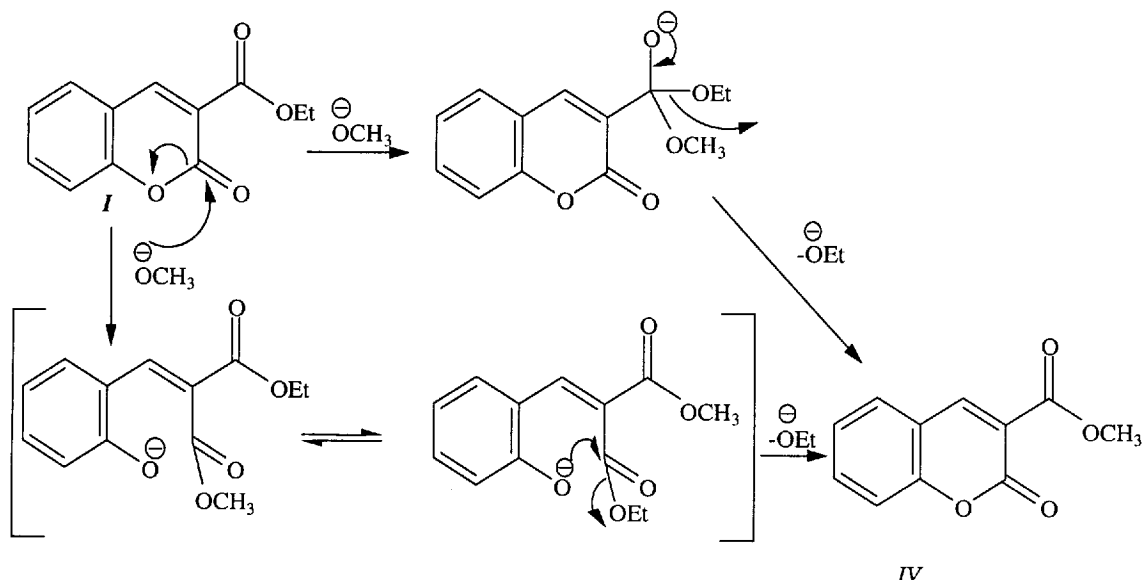
In the previous papers [1–3] the synthesis of benzopyranopyrimidine derivatives from 3-ethoxycarbonylcoumarin (*I*) with thiourea through nucleophilic attack at the position 4 in *I* with ring cyclization with the removal of ethanol has been briefly reported. As an extension of the previous works [4–8] this paper describes the synthesis of 3-substituted chromeno[3,2-*c*]chromen-6,7-diones starting from *I* with resorcinol (*II*) under Michael reaction conditions. The electron impact (EI) ionization mass spectral fragmentation of the prepared compounds was described.

The compound *I* was prepared from salicylaldehyde and dimethyl malonate according to a literature method. Condensation of *I* with *II* in the presence of sodium methoxide under fusion produced the 3-hydroxychromeno[3,2-*c*]chromen-6,7-dione (*III*) and 3-methoxycarbonylcoumarin (*IV*) (Scheme 1).

Compound *IV* may be formed by the nucleophilic attack with methoxide anion at position 2 in *I* with ring opening, followed by ring cyclization *via* the removal of ethanol molecule as shown in Scheme 2.



Scheme 1



Scheme 2

Acylation of *III* with acetic anhydride under reflux gave the corresponding 3-acetyloxychromeno[3,2-*c*]chromen-6,7-dione (*V*) (Scheme 3). The reaction of compound *III* with alkyl halides (such as benzyl chloride, ethyl chloroacetate, and 1,2-dichloroethane) in the presence of anhydrous potassium carbonate in dimethylformamide under reflux produced 3-alkoxychromeno[3,2-*c*]chromen-6,7-diones (*VIa*–*VIc*).

Diazotization [9–11] of aromatic amines (such as aniline and *p*-toluidine) followed by coupling with sodium salt of *III* gave the corresponding 2,4-di(aryloxy)-3-hydroxychromeno[3,2-*c*]chromen-6,7-diones (*VIIa*, *VIIb*).

Table 1 lists the m/z (I_r (relative abundance)/%) values of the principle fragment of some synthesized compounds.

The mass spectra of compounds *V* and *VI* show relatively small molecular ions and peaks typical of a cleavage or elimination type fragmentation. From the study of the mass spectra of compound *III* it was found that the molecular ion is a base peak of m/z 280. This ion of m/z 280 fragmented further involving two various pathways.

The ion of m/z 280 fragmented *via* the pathway A and gave an ionradical of m/z 252 [$M - CO$] $^{*+}$ which further fragmented and gave a fragment ion of m/z 224 [$M - 2CO$] $^{*+}$ by losing CO. Ion of m/z 224 fragmented to give an ion of m/z 196 [$M - 3CO$] $^{*+}$ which lost CO to give a fragment ion of m/z 168 [$M - 4CO$] $^{*+}$. The ion of m/z 168 was broken to give an ion of m/z 139 [$M - 4CO - CHO$] $^{*+}$.

Subsequently, the ion of m/z 280 fragmented *via* the pathway B to a fragment ion of m/z 279 [$M^* - H$] $^+$ by losing hydrogen radical. The ion of m/z 279 was broken to give an ion of m/z 251 [$M^* - H - CO$] $^{*+}$ which lost CO.

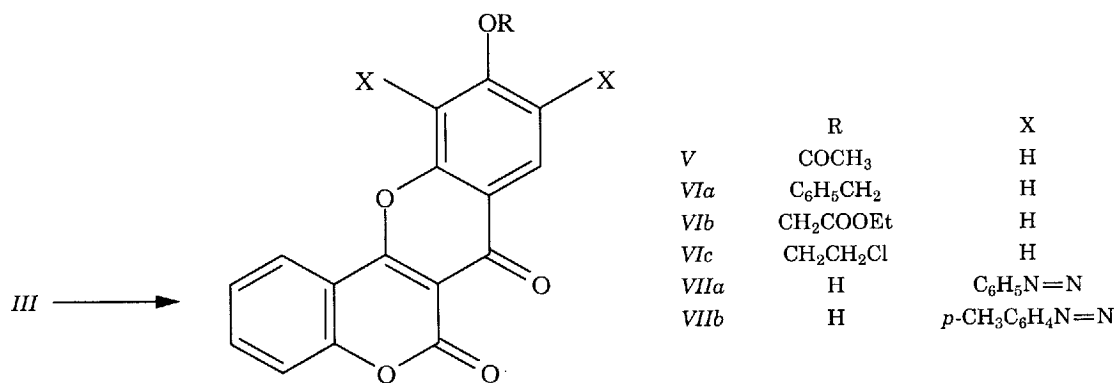
This fragmentation led to m/z 223 [$M^* - H - 2CO$] $^{*+}$, m/z 195 [$M^* - H - 3CO$] $^{*+}$, m/z 167 [$M^* - H - 4CO$] $^{*+}$, and m/z 139 [$M^* - H - 5CO$] $^{*+}$, respectively. The mass spectra of compound *V* gave a characteristic fragmentation pattern with a very stable fragment of m/z 280 which further broke *via* a similar way of the compound *III*.

The electron impact ionization mass spectra of compounds *VIa* and *VIc* show a base peak of m/z 91 and m/z 120, while the base peak of compound *VIIb* is the molecular ion of m/z 366. The ion of m/z 366 fragmented further and involved two pathways as illustrated in Table 1.

The ion of m/z 366 fragmented *via* the pathway A to give a fragment ion of m/z 293 [$M^* - COOEt$] $^{*+}$ by losing ethoxycarbonyl group. Ion of m/z 293 fragmented to give an ion of m/z 263 [$M^* - COOEt - CH_2O$] $^{*+}$ which lost two molecules of carbon monoxide to give a fragment ion of m/z 207 [$M^* - COOEt - CH_2O - 2CO$] $^{*+}$. Finally, the ion of m/z 366 further broke *via* a similar pathway B of compound *III*.

The main fragmentation pathways of compound *IV* are summarized in Table 1. However, pathway A is the predominant one, since fragment ion of m/z 89 [$M - CH_2O - 2CO - CHO$] $^{*+}$ which arises from ion of m/z 174 [$M - CH_2O$] $^{*+}$ is the base peak of the spectrum. Accordingly, the same ion of m/z 204 fragmented *via* the pathway B by losing CH_3O to give an ion of m/z 173 [$M^* - OCH_3$] $^{*+}$ which lost CO to give an ion of m/z 145 [$M^* - OCH_3 - CO$] $^{*+}$.

The electron impact ionization mass spectra of compound *VIIa* show three base peaks of m/z 488, m/z 383, and m/z 77. The main fragmentation pathways of compound *VIIa* are summarized in Table 1. The molecular ion of m/z 488 had fragmented to ion of m/z 411 [$M^* - C_6H_5$] $^{*+}$. The ion of m/z 411 fragmented *via* the path-



Scheme 3

way A and gave a fragment ion of m/z 383 $[M^* - C_6H_5 - N_2]^+$ which further fragmented and gave a fragment ion of m/z 355 $[M^* - C_6H_5 - N_2 - CO]^+$ by losing CO molecule.

Ion of m/z 355 fragmented to give an ion of m/z 299 $[M^* - C_6H_5 - N_2 - 3CO]^+$ which lost two molecules of carbon monoxide to give a fragment ion of m/z 243 $[M^* - C_6H_5 - N_2 - 5CO]^+$. The ion of m/z 243 was broken to give an ion of m/z 138 $[M - 2C_6H_5 - 2N_2 - 5CO]$ which lost nitrogen and phenyl radical group.

The same ion of m/z 488 has fragmented to ion of m/z 105 via pathway B. Ion of m/z 105 fragmented to give an ion of m/z 77 which lost $CH\equiv CH$ to give a fragment ion at m/z 51.

EXPERIMENTAL

NMR spectra were recorded on a General Electric QE 300 instrument and chemical shifts were given with respect to TMS. IR spectra were recorded on a Perkin—Elmer 1420 spectrometer and a Biorad FTS7 (KBr). Mass spectra were recorded on a GC/MS with CI (chemical ionization) and a Hewlett—Packard MS—Engine Thermo spray and ionization by electron impact at 70 eV. The accelerating voltage was 6 kV, the temperature of the ion source was ≈ 200 °C and the emission current ≈ 100 mA. Microanalyses were conducted using an Elemental analyzer 1106. Melting points were determined on a Reichert hot stage.

3-Hydroxychromeno[3,2-c]chromen-6,7-dione (III) and 3-Methoxycarbonylcoumarin (IV)

A mixture of I (0.01 mol), resorcinol (0.01 mol), and sodium methoxide (0.03 mol) was fused on a hot plate at 70—80 °C for 10—15 min. The reaction mixture was cooled and acidified with hydrochloric acid (6 mol cm^{-3}). The crude product was filtered off, washed with water and dried. The crude product was dissolved in hot ethanol. The insoluble solid in hot ethanol was filtered off and purified by recrystallization with dimethylformamide to give III. The filtrate was cooled and the solid formed

was filtered off, dried and purified by recrystallization with ethanol to give IV. Compound III was obtained as yellow crystals, yield 35 %, m.p. = 369—370 °C.

IR spectrum (KBr), $\bar{\nu}/cm^{-1}$: 3050—3390 (br, OH), 1681—1720 (br, CO), 1605, 1224, 1051, 1032. ¹H NMR spectrum (DMF-*d*₇), δ : 7.02—8.53 (m, 7H, H_{arom}), 8.91 (br, s, 1H, OH). ¹³C NMR spectrum (DMF-*d*₇), δ : 165.28, 163.16 (C=O), 158.09, 156.08, 155.97, 153.41 (C—O), 135.29, 131.96, 130.11, 125.27, 118.16, 116.28, 114.80, 108.27, 103.95, 103.60 (C_{aryl}). For C₁₆H₈O₅ w_i (calc.): 68.57 % C, 2.86 % H; w_i (found): 68.39 % C, 2.67 % H. Compound IV was obtained as colourless crystals, yield 32 %, m.p. = 120—121 °C. IR spectrum (KBr), $\bar{\nu}/cm^{-1}$: 1755, 1721 (CO of ester and α -pyranone), 1610, 1130, 1116, 1030. ¹H NMR spectrum (CDCl₃), δ : 3.95 (s, 3H, OCH₃), 7.08—8.23 (m, 5H, H_{arom}). For C₁₁H₈O₄ w_i (calc.): 64.70 % C, 3.92 % H; w_i (found): 64.53 % C, 3.78 % H.

3-Acetyloxochromeno[3,2-c]chromen-6,7-dione (V)

A solution of III (0.01 mol) in acetic anhydride (30 cm^3) was heated under reflux for 4 h. The product formed after being cooled was filtered off, dried and purified by recrystallization with ethanol to give V as pale yellow crystals, yield 71 %, m.p. = 220—221 °C. IR spectrum (KBr), $\bar{\nu}/cm^{-1}$: 1745 (CO of ester), 1685—1719 (br, CO), 1613, 1212, 1116, 1030, 1012. ¹H NMR spectrum (DMSO-*d*₆), δ : 2.31 (s, 3H, CH₃CO), 7.01—8.51 (m, 7H, H_{arom}). ¹³C NMR spectrum (DMSO-*d*₆), δ : 168.38, 165.23, 163.01 (C=O), 158.01, 156.03, 155.94, 153.39 (C—O), 135.27, 131.97, 130.01, 125.23, 118.13, 116.22, 114.78, 108.25, 103.91, 103.58 (C_{aryl}), 21.32 (CH₃). Mass spectrum, m/z (I_r /%) : 322 [M^+ , 10.00], 282 (4.70), 281 (20.10), 280 (100.00), 279 (32.00), 253 (4.60), 252 (35.60), 251 (2.50), 236 (12.00), 224 (40.30), 223 (6.70), 212 (2.10), 207 (2.50), 196 (49.70), 195 (11.60), 179 (3.20), 168 (18.70), 167 (5.70), 150 (5.70), 139 (43.60), 138 (4.30), 127 (8.30), 126 (9.20), 114 (4.70), 113 (11.70), 101 (5.20), 87 (10.40), 86 (8.30), 75 (15.60), 74 (12.00), 66 (15.00), 63 (20.20), 62 (13.00). For C₁₈H₁₀O₆ w_i (calc.): 67.08 % C, 3.11 % H; w_i (found): 67.00 % C, 3.01 % H.

Table 1. EI Mass Spectra (70 eV) of Compounds III, IV, VIb, and VIIa

Compound	M ⁺ m/z (I _r %)	Pathway A		Pathway B		Other important ions m/z (I _r %)
		Symbol	M ⁺ m/z (I _r %)	Symbol	M ⁺ m/z (I _r %)	
III	280 (100)	[M - CO] ⁺⁺	252 (22.5)	[M - H] ⁺	279 (45.9)	281 (M ⁺ + 1, 2.3), 263 (5.9), 253 (6.0), 236 (10.4), 225 (6.5), 212 (6.0), 207 (3.4), 197 (10.3), 179 (3.9), 140 (20.0), 138 (3.8), 127 (10.5), 126 (14.0), 113 (12.2), 112 (6.9), 89 (10.3), 87 (12.7), 86 (12.2), 84 (14.5), 76 (10.3), 75 (16.0), 69 (23.4), 63 (34.8), 190 (28.5), 147 (16.0), 144 (17.0), 119 (17.5), 105 (9.5), 102 (20.5), 101 (17.5), 90 (19.00), 87 (11.5), 78 (23.5), 7 (17.5), 63 (58.5), 62 (43.5)
		[M - 2CO] ⁺⁺	224 (33.2)	[M - H - CO] ⁺	251 (5.0)	
		[M - 3CO] ⁺⁺	196 (67.0)	[M - H - 2CO] ⁺	223 (10.8)	
		[M - 4CO] ⁺⁺	168 (25.6)	[M - H - 3CO] ⁺	195 (17.6)	
		[M - 4CO - CHO] ⁺	139 (42.6)	[M - H - 4CO] ⁺	167 (4.5)	
IV	204 (36.5)	[M - OCH ₃] ⁺	173 (72.0)	[M - H - 5CO] ⁺	139 (42.6)	190 (28.5), 147 (16.0), 144 (17.0), 119 (17.5), 105 (9.5), 102 (20.5), 101 (17.5), 90 (19.00), 87 (11.5), 78 (23.5), 7 (17.5), 63 (58.5), 62 (43.5)
		[M - OCH ₃ - CO] ⁺	145 (42.0)	[M - CH ₂ O] ⁺⁺	174 (22.5)	
		[M - OCH ₃ - CO - CHO] ⁺	116 (13.5)	[M - CH ₂ O - CO] ⁺⁺	146 (92.5)	
				[M - CH ₂ O - 2CO] ⁺⁺	118 (37.5)	
VIb	366 (100)	[M - COOEt] ⁺	293 (18.3)	[M - CH ₂ O - 2CO - CHO] ⁺	89 (10.0)	367 (M ⁺ + 1, 25.1), 206 (9.3), 179 (25.3), 173 (35.2), 168 (2.5), 151 (24.2), 150 (22.3), 139 (24.36), 101 (18.2), 75 (27.2)
		[M - COOEt - CH ₂ O] ⁺	263 (19.3)	[M - CH ₂ COOEt] ⁺	279 (63.2)	
		[M - COOEt - CH ₂ O - CO] ⁺	235 (9.3)	[M - CH ₂ COOEt - CO] ⁺	251 (10.3)	
		[M - COOEt - CH ₂ O - 2CO] ⁺	207 (13.3)	[M - CH ₂ COOEt - 2CO] ⁺	223 (18.2)	
				[M - CH ₂ COOEt - 3CO] ⁺	195 (10.3)	
VIIa	488 (100)	[M - Ph] ⁺	411 (13.3)			489 (M ⁺ + 1, 32.2), 462 (15.2), 460 (13.3), 384 (35.6), 357 (7.8), 327 (5.3), 271 (20.4), 256 (2.1), 255 (6.3), 250 (7.3), 226 (25.0), 194 (18.3), 169 (50.2), 166 (46.2), 139 (36.3), 138 (63.1), 127 (26.2), 105 (17.2), 93 (33.9), 77 (100), 65 (14.7), 51 (23.2)
		[M - Ph - N ₂] ⁺	383 (100)			
		[M - Ph - N ₂ - CO] ⁺	355 (11.3)			
		[M - Ph - N ₂ - 3CO] ⁺	299 (11.3)			
		[M - Ph - N ₂ - 5CO] ⁺	243 (10.3)			

3-Alkoxychromeno[3,2-*c*]chromen-6,7-diones (VIa—VIc)

A mixture of III (0.01 mol), alkyl halides (such as benzyl chloride, ethyl chloroacetate, and 1,2-dichloroethane) (0.01 mol), and anhydrous potassium carbonate (0.02 mol) in dimethylformamide (30 cm³) was heated under reflux for 3 h. The reaction mixture was cooled and poured into water. The product formed was collected by filtration, washed with water, dried, and purified by recrystallization with ethanol.

3-(Phenylmethoxy)chromeno[3,2-*c*]chromen-6,7-dione (VIa) as pale yellow crystals, yield 73 %, m.p. = 225—226 °C. IR spectrum (KBr), $\bar{\nu}/\text{cm}^{-1}$: 1685—1721 (br, CO), 1615, 1580, 1250, 1031, 1010. ¹H NMR spectrum (CDCl₃), δ : 4.65 (s, 2H, OCH₂), 7.01—8.51 (m, 12, H_{arom}). ¹³C NMR spectrum (CDCl₃), δ : 165.27, 163.13 (C=O), 158.07, 156.05, 155.96, 153.40 (C—O), 141.30, 135.26, 131.97, 130.10, 129.02, 128.07, 126.22, 125.25, 118.165, 116.26, 114.81, 108.26, 103.92, 103.58 (C_{aryl}), 53.25 (OCH₂). Mass spectrum, m/z (I_r%) : 371 [M⁺ + 1, 1.40], 370 [M⁺, 2.40], 280 (0.40), 279 (0.30), 251 (0.70), 195 (0.60), 167 (0.50), 138 (0.50), 127 (0.40), 127 (0.5), 126 (0.80), 101 (0.60), 92 (11.30), 91(100.00), 65 (11.70). For C₂₃H₁₄O₅ w_i(calc.): 74.59 % C, 3.78 % H; w_i(found): 74.46 % C, 3.58 % H.

3-(Ethoxycarbonylmethoxy)chromeno[3,2-*c*]chromen-6,7-dione (VIb) as pale yellow crystals, yield 72 %, m.p. = 218—219 °C. IR spectrum (KBr), $\bar{\nu}/\text{cm}^{-1}$: 1745 (CO of ester), 1683—1719 (br, CO), 1615, 1585, 1235, 1030, 1015. ¹H NMR spectrum (DMSO-*d*₆), δ : 1.35 (t, 3H, CH₃), 4.25 (q, 2H, OCH₂), 5.15 (s, 2H, OCH₂CO), 7.10—8.43 (m, 7H, H_{arom}). ¹³C NMR spectrum (DMSO-*d*₆), δ : 171.23, 165.29, 163.17 (C=O), 158.10, 156.11, 155.98, 153.40 (C—O), 135.27, 131.97, 130.10, 125.26, 118.14, 116.27, 114.78, 108.25, 103.93, 103.57 (C_{aryl}), 71.21 (OCH₂), 61.31 (OCH₂), 14.23 (CH₃). For C₂₀H₁₄O₇ w_i(calc.): 65.57 % C, 3.82 % H; w_i(found): 65.46 % C, 3.66 % H.

3-(Chloroethoxy)chromeno[3,2-*c*]chromen-6,7-dione (VIc) as pale yellow crystals, yield 73 %, m.p. = 226—227 °C. IR spectrum (KBr), $\bar{\nu}/\text{cm}^{-1}$: 2995, 1689—1721 (br, C=O), 1614, 1588, 1310, 1223, 1030, 1015. ¹H NMR spectrum (DMF-*d*₇), δ : 4.21 (t, 2H, CH₂Cl), 4.83 (t, 2H, OCH₂), 7.10—8.43 (m, 7H, H_{arom}). ¹³C NMR spectrum (DMF-*d*₇), δ : 164.44, 163.15 (C=O), 157.93, 155.85, 155.44, 153.25 (C—O), 135.56, 131.70, 130.07, 125.40, 118.21, 116.23, 114.28,

109.75, 103.27, 102.52 (C_{aryl}), 69.94 (OCH_2), 43.35 (CH_2Cl). Mass spectrum, m/z ($I_r/\%$): 344 [$M^+ + 2$, 2.35], 342 [M^+ , 10.33], 243 (1.51), 280 (20.35), 279 (35.36), 263 (14.21), 251 (6.39), 235 (8.10), 223 (12.78), 206 (8.31), 179 (23.51), 173 (20.11), 168 (3.61), 151 (23.50), 150 (18.35), 139 (20.63), 120 (100), 91 (14.80). For $C_{18}H_{11}ClO_5$ w_i (calc.): 63.06 % C, 3.21 % H, 10.36 % Cl; w_i (found): 63.01 % C, 3.04 % H, 10.18 % Cl.

2,4-Di(arylazo)-3-hydroxychromeno[3,2-c]-chromen-6,7-diones (VIIa, VIIb)

A solution of III (0.01 mol) in aqueous sodium hydroxide (50 cm³, 10 %) was chilled in ice to 0–5 °C. A cold aqueous solution (0–5 °C) of the diazonium salt (0.02 mol) was added dropwise with stirring during 45 min. After addition the mixture was stirred for further 30 min and then left for 2 h in a refrigerator. The precipitated product was collected, washed with water, dried, and purified by recrystallization with acetic acid.

2,4-Di(phenylazo)-3-hydroxychromeno[3,2-c]-chromen-6,7-dione (VIIa) as red crystals, yield 78 %, m.p. = 269–270 °C. IR spectrum (KBr), $\tilde{\nu}/\text{cm}^{-1}$: 2860–3305 (br, OH), 1685–1720 (br, CO), 1615, 1595, 1310, 1030, 1100. ¹H NMR spectrum (CD_3COOD), δ : 6.98–8.45 (m, 15H, H_{arom}), 9.35 (br, s, 1H, OH). For $C_{28}H_{16}N_4O_5$ w_i (calc.): 68.85 % C, 3.28 % H, 11.47 % N; w_i (found): 68.63 % C, 3.08 % H, 11.29 % N.

2,4-Di(p-tolylazo)-3-hydroxychromeno[3,2-c]-chromen-6,7-dione (VIIb) as brown crystals, yield 81 %, m.p. = 235–237 °C. IR spectrum (KBr), $\tilde{\nu}/\text{cm}^{-1}$: 2860–3310 (br, OH), 1687–1721 (br, CO), 1610, 1603, 1585, 1305, 1215, 1030, 1010. ¹H NMR spectrum (CD_3COOD),

δ : 2.21 (s, 6H, $2 \times \text{CH}_3$), 6.79–8.41 (m, 13H, H_{arom}), 9.31 (br, s, 1H, OH). Mass spectrum, m/z ($I_r/\%$): 516 [M^+ , 3.20], 197 (5.01), 196 (2.50), 122 (1.50), 121 (1.70), 120 (1.21), 119 (13.10), 118 (3.40), 108 (19.10), 107 (73.40), 106 (100), 105 (3.00), 104 (8.50), 98 (3.20), 92 (12.70), 91 (76.00), 90 (8.70), 78 (9.20), 77 (26.60), 76 (5.70), 65 (23.50), 53 (14.90), 52 (13.20), 51 (16.40). For $C_{30}H_{20}N_4O_5$ w_i (calc.): 69.77 % C, 3.87 % H, 10.85 % N; w_i (found): 69.52 % C, 3.65 % H, 10.61 % N.

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