



Ain Shams University
Faculty of Education
Department of Chemistry

**Synthesis and chemical reactivity of 6,8-dibromo-
7-hydroxychromone-3-carboxaldehyde towards
some nucleophilic reagents**

Thesis Submitted
By

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Abstract

Synthesis and chemical reactivity of 6,8-dibromo-7-hydroxychromone-3-carboxaldehyde towards some nucleophilic reagents

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Abstract

A novel 6,8-dibromo-7-hydroxychromone-3-carboxaldehyde (**4**) was prepared by the Vilsemier-Haack formylation of 3,5-dibromo-2,4-dihydroxyacetophenone (**3**). The chemical reactivity of carboxaldehyde **4** was studied towards some nitrogen nucleophilic reagents such as amines, 1,2-*N,N*-binucleophiles, 1,2-*N,O*-binucleophiles, 1,3-*N,N*-binucleophiles, 1,4-*N,N*-binucleophiles, 1,4-*N,O*-binucleophiles and 1,4-*N,S*-binucleophiles under different reaction conditions. Also, The chemical reactivity of carboxaldehyde **4** was studied towards some carbon nucleophiles as cyclic and acyclic active methylene nucleophiles and also, 1,3-*C,N*- and 1,3-*C,C*-binucleophiles as a route to achieve ring transformation producing a variety of heterocyclic systems. Structures of the newly synthesized products have been deduced on the basis of elemental analysis and spectral data.

Keywords: Chromone-3-carboxaldehyde, nitrogen nucleophiles, carbon nucleophiles, condensation reactions, ring opening ring closure reactions.

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Amira Mohsen Mohamed

Aim of the work

1. Synthesis of the novel 6,8-dibromo-7-hydroxychromone-3-carboxaldehyde (**4**) as the starting material.
 2. Study the chemical reactivity of the starting compound **4** towards a variety of nitrogen nucleophiles.
 3. Study the chemical behavior of compound **4** towards some of carbon nucleophiles.
 4. Elucidation of the newly synthesized products using elemental analysis and different spectroscopic techniques.
 5. Evaluation the antimicrobial activities of the newly synthesized compounds.
-

ANTIMICROBIAL EVALUATION

The newly synthesized compounds were evaluated for their *in vitro* antibacterial activity against *Staphylococcus aureus* (ATCC 25923) and *Bacillus subtilis* (ATCC 6635) as examples of Gram-positive bacteria and *Escherichia coli* (ATCC 25922) and *Salmonella typhimurium* (ATCC 14028) as examples of Gram-negative bacteria. They were also evaluated against *Candida albicans* (ATCC 10231) as yeast and the fungus *Asperigillus fumigatus*. Agar diffusion technique was used for the determination of the preliminary antibacterial and antifungal activities [204]. The test was performed on medium potato dextrose agar (PDA) which contained infusion of 200 g potatoes, 6 g dextrose and 15 g agar. Uniform size filter paper disks (3 disks per compound) were impregnated by equal volume (10 µl) from the concentrations of 500 and 1000 µg/mL dissolved compounds in dimethylformamide (DMF) and carefully placed on inoculated agar surface. After incubation for 36 h at 27 °C in the case of bacteria and for 48 h at 24°C in the case of fungi. Cephalothin, Chloramphenicol and Cycloheximide were used as reference drugs for Gram-positive bacteria, Gram-negative bacteria and yeast and the fungus, respectively. The obtained results were recorded for each tested compound as average diameter of inhibition zones of the bacteria, yeast and fungus around the disks in mm at the concentrations 500 and 1000 µg/mL. The antimicrobial activities were determined by measuring the inhibition zones (Table 1).

- [1] The results depicted in Table 1 revealed that the most of tested compounds displayed variable inhibitory effects on the growth of the tested Gram-positive and Gram-negative bacterial strains, and also against the yeast and antifungal strain.
- [2] In general, most of the tested compounds revealed low to moderate activities against the microorganism strains.
- [3] Most of the tested compounds recorded good activities against *Bacillus subtilis* and *Candida albicans*.

Antimicrobial evaluation

- [4] Most of the tested compounds recorded no activities against *Asperigillus fumigatus*.
- [5] Only, compounds **22**, **37** and **39** recorded high activities against *Bacillus subtilis*, and compound **23** against *Candida albicans*.
- [6] Presence of the simple functional groups at position 3 of 7-hydroxy-6,8-dibromochromone moiety in compounds **4**, **12**, **13**, **14**, **15** and **24** exhibited good activity profile.
- [7] Conversion of 7-hydroxy-6,8-dibromo-3-formylchromone (**4**) to other heterocycles *via* its reaction with nucleophiles, unfortunately not produce noticeable antimicrobial activities.
- [8] In conclusion, the objective of the present study was to synthesize and investigate the antimicrobial activities of some new functionalized 7-hydroxy-6,8-dibromochromone with the hope of discovering new structure leads serving as antimicrobial agents. Generally, the prepared compounds showed lower to moderate activities. However, none of the tested compounds was nearly or superior activity than the reference drugs.

Antimicrobial evaluation

Table 1: In *vitro* antimicrobial activities of the synthesized compounds 4-48 at 500 and 1000 µg/mL by disc diffusion assay.

Compd. No.	Conc. (µg/mL)	Zone of inhibition (mm)*					
		Bacteria		Bacteria		Yeast	Fungi
		Gram (+) ve		Gram (+) ve			
		S. aureus	B. subtilis	EE. coli	S. S. typhimurium	C. C. albicans	AA. fumigatus
26	500	9	12	-10	- 10	- 15	-7
26	1000	12	15	-14	- 12	- 20	-12
27	500	10	12	78	11 8	8 8	-10
27	1000	12	13	101	1210	9 10	-15
28	500	8	10	916	7 8	1211	--
28	1000	11	16	138	1015	1520	--
29	500	-	9	-11	7 6	- -	--
29	1000	-	12	-13	8 10	- -	--
31	500	9	17	77	1110	- 12	-14
31	1000	13	19	101	1414	- 18	-20
32	500	11	8	-11	- 12	7 14	-15
32	1000	16	12	-14	- 15	1019	-18
33	500	12	16	-10	7 11	8 15	-7
33	1000	15	18	-13	8 13	1018	-12
35	500	8	11	-10	- 7	- -	-11
35	1000	14	15	-15	- 11	- -	-15
36	500	8	16	-7	7 -	16 -	102
36	1000	11	22	-11	10 -	21 -	167
37	500	-	18	17-	- -	16 -	--
37	1000	-	23	20-	10 -	23 -	--
38	500	9	8	--	- -	8 12	--
38	1000	12	14	--	- -	1016	--
39	500	8	20	79	7 7	1812	19
39	1000	10	22	103	8 9	2015	22
40	500	-	20	-7	- -	1117	--
40	1000	-	23	-11	- -	1617	--
41	500	-	12	-14	- -	7 20	--
41	1000	-	17	-20	- -	8 25	--
43	500	7	12	-7	- 11	11 8	--
43	1000	12	16	-10	- 12	8 9	--
45	500	8	18	--	7 -	8 13	--
45	1000	9	18	--	8 -	1016	--

* Low active: 6–12 mm; moderately active: 13–19 mm; highly active: 20–30 mm; --: No inhibition or inhibition less than 5 mm.

Continued Table 1: In *vitro* antimicrobial activities of the synthesized compounds 4-48 at 500 and 1000 µg/mL by disc diffusion assay.

* Low active: 6–12 mm; moderately active: 13–19 mm; highly active: 20–30 mm; --: No inhibition or inhibition less than 5 mm.

Antimicrobial evaluation

Continued Table 1: In *vitro* antimicrobial activities of the synthesized compounds **4-48** at 500 and 1000 µg/mL by disc diffusion assay.

Compd. No.	Conc. (µg/ml)	Zone of inhibition (mm)*					
		Bacteria		Bacteria		Yeast	Fungi
		Gram (+) ve		Gram (-) ve			
		<i>S. aureus</i>	<i>B. subtilis</i>	<i>E. coli</i>	<i>S. typhimurium</i>	<i>C. albicans</i>	<i>A. fumigatus</i>
46	500	-	7	-	-	15	-
	1000	-	10	-	-	19	-
47	500	7	10	-	7	10	-
	1000	10	15	-	9	17	-
48	500	-	9	-	5	7	-
	1000	-	14	-	8	10	-
Standard	500	26	25	28	27	28	26
drug	1000	35	35	36	38	35	37

* Low active: 6–12 mm; moderately active: 13–19 mm; highly active: 20–30 mm; –: No inhibition or inhibition less than 5 mm.



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Approval Sheet

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Higher studies:

The thesis was approved

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Experimental

Experimental

Melting points were determined on a digital Stuart SMP3 apparatus. Infrared spectra were measured on FTIR Nicolet IS10 spectrophotometer (cm^{-1}), using KBr disks. ^1H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were measured on Mercury-300BB, using $\text{DMSO-}d_6$ as a solvent and TMS (δ) as the internal standard. Mass spectra were obtained using GC-2010 Shimadzu Gas chromatography instrument mass spectrometer (70 eV). Elemental microanalyses were performed Perkin-Elmer 2400II at the Chemical War department, Ministry of Defense, Egypt.

3,5-Dibromo-2,4-dihydroxyacetophenone (3)

To a solution of 2,4-dihydroxyacetophenone (**2**) (15.2 g, 0.1 mol) in acetic acid (80%, 20 mL), bromine (32 g, 10.4 mL, 0.2 mol) in acetic acid (10 mL), was added dropwise with continuous stirring for 30 minutes. The resulting solid was filtered off and crystallized from benzene to give compound **3** as white crystals, yield (14.4 g, 46%), m.p. 173–174 °C (lit. 172–173 °C) [190]. IR (KBr, cm^{-1}): 3399 (br, OH), 1624 ($\text{C}=\text{O}_{\text{Hydrogen bonded}}$), 1559 ($\text{C}=\text{C}$).

6,8-Dibromo-7-hydroxychromone-3-carboxaldehyde (4).

Method A: Phosphoryl chloride (3 mL) was added dropwise with continuous stirring to a pre-cooled DMF (10 mL) and the mixture was further stirred at room temperature for 30 minutes. Then 3,5-dibromo-2,4-dihydroxyacetophenone (**3**) (0.93 g, 3 mmol) in DMF (10 mL) was added dropwise with continuous stirring. The mixture was stirred at room temperature for 2 h, left overnight and poured onto crushed ice (50 g). The resulting solid was filtered off, air dried and crystallized from ethanol to give compound **4** as yellow crystals, yield (0.80 g, 77%), m.p. 250–251 °C.

Experimental

Method B: A mixture of 7-hydroxychromone-3-carboxaldehyde (**5**) (0.57 g, 3 mmol) and bromine (0.96 g, 0.32 mL, 6 mmol) in acetic acid (80%, 5 mL) was stirred at room temperature for 1 h. The resulting solid was filtered off and crystallized from ethanol to give compound **4** as yellow crystals, yield (0.61 g, 59%), m.p. 250–251 °C. IR (KBr, cm^{-1}): 3235 (OH), 3058 (CH_{arom}), 1685 ($\text{C}=\text{O}_{\text{formyl}}$), 1667 ($\text{C}=\text{O}_{\gamma\text{-pyrone}}$), 1599 ($\text{C}=\text{C}$). ^1H -NMR ($\text{DMSO-}d_6$): 8.16 (s, 1H, H-5_{chromone}), 8.89 (s, 1H, H-2_{chromone}), 10.07 (s, 1H, CHO). ^{13}C -NMR ($\text{DMSO-}d_6$): 100.4 (C-8), 110.8 (C-6), 118.9 (C-3), 119.7 (C-4a), 127.6 (C-5), 153.1 (C-8a), 156.9 (C-7), 163.2 (C-2), 172.8 (C=O), 187.9 (CHO). MS (m/z , I%): 350 (M+4, 2), 348 (M+2, 5), 346 (M^+ , 2), 322 (52), 320 (100), 318 (50), 296 (8), 294 (16), 292 (8), 280 (3), 278 (7), 276 (3), 215 (2), 213 (2), 199 (3), 197 (3), 187 (3), 185 (4), 175 (2), 173 (2), 159 (2), 157 (2), 133 (3), 119 (2), 91 (2). Anal. Calcd for $\text{C}_{10}\text{H}_4\text{Br}_2\text{O}_4$ (347.94): C, 34.52; H, 1.16%. Found: C, 34.17; H, 1.16%.

Synthesis of Schiff bases 9-11; General procedure for reaction of carboxaldehyde **4** with heterocyclic amines:

To a hot solution of carboxaldehyde **4** (0.35 g, 1 mmol) in absolute ethanol (15 mL), each one of the heterocyclic amines namely: 4-amino-5-phenyl-2,4-dihydro-[1,2,4]triazole-3-thione (**6**), 4-amino-6-methyl-3-thioxo-3,4-dihydro-1,2,4-triazin-5(2*H*)-one (**7**) and 3-amino-2-phenylquinazoline-4(3*H*)-one (**8**) (1 mmol) was added with continuous stirring. The mixture was heated under reflux for 30 minutes. The resulting solids after cooling were filtered off and crystallized from ethanol to give compounds **9-11**, respectively, as yellow crystals.

6,8-Dibromo-7-hydroxy-3-[(5-phenyl-3-thioxo-1,3-dihydro-4*H*-1, 2, 4-triazol-4-yl)imino] methyl}-4*H*-chromone (9**):** Yield (0.31 g, 59%), m.p. 291–292 °C. IR (KBr, cm^{-1}): 3311 (OH, NH), 1654 ($\text{C}=\text{O}_{\gamma\text{-pyrone}}$), 1610 ($\text{C}=\text{N}$), 1595 ($\text{C}=\text{C}$). ^1H -NMR ($\text{DMSO-}d_6$): 7.59–7.89 (m, 5H,

Experimental

Ph-H), 8.15 (s, 1H, H-5_{chromone}), 8.75 (s, 1H, CH=N), 9.13 (s, 1H, H-2_{chromone}), 12.00 (brs, 1H, OH exchangeable with D₂O), 13.63 (brs, 1H, NH_{triazole} exchangeable with D₂O). Anal. Calcd for C₁₈H₁₀Br₂N₄O₃S (522.17): C, 41.40; H, 1.93; N, 10.73; S, 6.14%. Found: C, 41.32; H, 1.74; N, 11.02; S, 5.84%.

6,8-Dibromo-7-hydroxy-3-[[(6-methyl-3-thioxo-3,4-dihydro-5-oxo-1,2,4-triazin-4-yl)imino]methyl]-4H-chromone (10): Yield (0.33 g, 68%), m.p. 229–230 °C. IR (KBr, cm⁻¹): 3207 (OH), 3080 (CH_{arom}), 2982 (CH_{aliph}), 1697 (C=O_{triazine}), 1641 (C=O_{γ-pyrone}), 1596 (C=N), 1580 (C=C). ¹H-NMR (DMSO-*d*₆): 2.18 (s, 3H, CH₃ triazine), 8.20 (s, 1H, H-5_{chromone}), 8.70 (s, 1H, CH=N), 9.16 (s, 1H, H-2_{chromone}), 13.75 (brs, 1H, NH_{triazine} exchangeable with D₂O). Anal. Calcd for C₁₄H₈Br₂N₄O₄S (488.11): C, 34.45; H, 1.65; N, 11.48; S, 6.57%. Found: 34.23; H, 1.60; N, 11.16; S, 6.42%.

6,8-Dibromo-7-hydroxy-3-[[(2-phenyl-4-oxo-quinazolin-3-yl)imino]methyl]-4H-chromone (11): Yield (0.39 g, 69%), m.p. 248–250 °C. IR (KBr, cm⁻¹): 3249 (OH), 3073 (CH_{arom}), 1661 (C=O_{quinazolinone} and C=O_{γ-pyrone}), 1597 (C=N). ¹H-NMR (DMSO-*d*₆): 7.47–7.89 (m, 8H, Ar-H), 8.18 (s, 1H, H-5_{chromone}), 8.23 (d, 1H, *J*=6.9 Hz, Ar-H), 8.70 (s, 1H, CH=N), 9.11 (s, 1H, H-2_{chromone}). MS (*m/z*, I^o): 565 (M⁺, 3), 548 (4), 537 (4), 519 (5), 318 (3), 293 (2), 236 (100), 222 (6), 208 (9), 180 (17), 152 (13), 103 (14), 90 (13), 77 (16). Anal. Calcd for C₂₄H₁₃Br₂N₃O₄ (567.49): C, 50.82; H, 2.31; N, 7.41%. Found: C, 50.76; H, 2.24; N, 7.77%.

6,8-Dibromo-7-hydroxy-4H-chromone-3-carboxaldehyd-oxime (12).

To a solution of carboxaldehyde **4** (0.70 g, 2 mmol) in 95% ethanol (15 mL), hydroxylamine hydrochloride (0.15 g, 2.2 mmol) in

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water (10 mL) was added. The reaction mixture was heated under reflux for 10 minutes. The resulting solid after cooling was filtered off and crystallized from DMF/H₂O to give compound **12** as yellow crystals, yield (0.51 g, 70%), m.p. > 300 °C. IR (KBr, cm⁻¹): 3274 (2 OH), 1660 (C=O_{γ-pyrone}), 1620 (C=N), 1590 (C=C). ¹H-NMR (DMSO-*d*₆): δ 8.14 (s, 1H, H-5_{chromone}), 8.32 (s, 1H, CH=N), 8.78 (s, 1H, H-2_{chromone}), 11.87 (brs, 1H, OH exchangeable with D₂O), 12.44 (brs, 1H, OH exchangeable with D₂O). Anal. Calcd for C₁₀H₅Br₂NO₄ (362.96): C, 33.09; H, 1.39; N, 3.86%. Found: C, 32.78; H, 1.31; N, 3.59%.

6,8-Dibromo-7-hydroxy-4H-chromone-3-carbonitrile (13).

A mixture of carboxaldehyde **4** (0.70 g, 2 mmol) and hydroxylamine hydrochloride (0.15 g, 2.2 mmol) in 95% ethanol (20 mL) and concentrated hydrochloric acid (5 mL) was heated under reflux for 4 h. After cooling, the reaction mixture was poured onto crushed ice. The resulting solid was filtered off and crystallized from ethanol to give compound **13** as pale yellow crystals, yield (0.45 g, 65%), m.p. 274–275 °C. IR (KBr, cm⁻¹): 3308 (OH), 3020 (CH_{arom}), 2216 (C≡N), 1618 (C=O), 1540 (C=C). ¹H-NMR (DMSO-*d*₆): 8.19 (s, 1H, H-5_{chromone}), 8.96 (s, 1H, H-2_{chromone}), 12.44 (brs, 1H, OH exchangeable with D₂O). MS (*m/z*, I%): 347 (M+4, 24), 345 (M+2, 48), 343 (M⁺, 23), 321 (16), 319 (36), 317 (18), 296 (48), 294 (100), 292 (49), 280 (14), 278 (29), 276 (15), 215 (33), 213 (34), 199 (12), 197 (13), 159 (45), 157 (26), 129 (78), 89 (72). Anal. Calcd for C₁₀H₃Br₂NO₃ (344.94): C, 34.82; H, 0.88; N, 4.06%. Found: C, 34.65; H, 0.85; N, 4.02%.

2-Amino-6,8-dibromo-7-hydroxy-4H-chromone-3-carboxamide (14).

A mixture of carboxaldehyde **4** (0.70 g, 2 mmol) and hydroxylamine hydrochloride (0.15 g, 2.2 mmol) in ethanol (10 mL) and sodium hydroxide solution (1%, 10 mL), was heating on a water

Experimental

bath for 2 h. After cooling, the reaction mixture was neutralized with diluted hydrochloric acid. The resulting solid was filtered off and crystallized from acetic acid to give compound **14** as yellow crystals, yield (0.36 g, 48%), m.p > 300 °C. IR (KBr, cm⁻¹): 3446, 3308, 3253, 3142 (OH, 2 NH₂), 1640 (2 C=O), 1593 (C=C). ¹H-NMR (DMSO-*d*₆): 7.45 (brs, 1H, NH exchangeable with D₂O), 8.05 (s, 1H, H-5_{chromone}), 9.31 (brs, 1H, NH exchangeable with D₂O), 9.46 (brs, 1H, NH exchangeable with D₂O), 10.52 (brs, 1H, NH exchangeable with D₂O). MS (*m/z*, I%): 380 (M+4, 34), 378 (M+2, 67), 376 (M⁺, 34), 363 (24), 361 (43), 359 (23), 336 (2), 334 (5), 332 (3), 296 (24), 294 (49), 292 (25), 268 (2), 266 (5), 264 (3), 250 (5), 248 (4), 159 (13), 157 (13), 77 (38), 68 (100). Anal. Calcd for C₁₀H₆Br₂N₂O₄ (377.97): C, 31.78; H, 1.60; N, 7.41%. Found: C, 31.66; H, 1.52; N, 7.19%.

2-Amino-6,8-dibromo-7-hydroxy-4*H*-chromone-3-carboxaldehyde (15).

A mixture of carboxaldehyde **4** (0.70 g, 2 mmol) and hydroxylamine hydrochloride (0.15 g, 2.2 mmol) in 95% ethanol (15 mL) was refluxed for 5 minutes. Ammonium hydroxide (5 mL) was added into the reaction mixture and heated further for 30 minutes. The resulting solid during heating was filtered off and crystallized from acetic acid to give compound **15** as yellow crystals, yield (0.43 g, 59%), m.p. > 300 °C. IR: (KBr, cm⁻¹): 3214 (OH, NH₂), 3072 (CH_{arom}), 1675 (C=O_{formyl}), 1656 (C=O_{γ-pyrone}), 1594 (C=C). ¹H-NMR (DMSO-*d*₆): 8.05 (s, 1H, H-5), 9.65 (s, 1H, NH exchangeable with D₂O), 9.83 (s, 1H, NH exchangeable with D₂O), 10.03 (s, 1H, CHO). Anal. Calcd for C₁₀H₅Br₂NO₄ (362.96): C, 33.09; H, 1.39; N, 3.86%. Found: C, 32.82; H, 1.28; N, 3.69%.

6,8-Dibromo-3-{[2-(7-chloroquinolin-4-yl)hydrazinylidene]methyl}-7-hydroxy-4*H*-chromone (17).