An Approach to the Synthesis of 7-Amino-6-imino-9-phenyl-6*H*-benzo[*c*]chromene-8-carbonitrile Derivatives *via* a Three-Component Reaction under Ultrasonic Irradiation

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An efficient synthesis of 7-amino-6-imino-9-phenyl-6*H*-benzo[*c*]chromene-8-carbonitrile derivatives **3** by a three-component reaction of salicylaldehydes (=2-hydroxybenzaldehydes) **1**, malononitrile (=propanedinitrile), and 2-(1-arylethylidene)malononitrile **2** under ultrasonic irradiation in EtOH is reported. Good yields, short reaction times, and easy purification are the main advantages of the present method. The structures were confirmed spectroscopically (IR, ^{1}H- and ^{13}C-NMR, and EI-MS) and by elemental analyses. A plausible mechanism for this reaction is proposed (*Scheme 2*).

Introduction. – 'Green chemistry' is an increasingly important aspect of chemical research and engineering that encourages the design of products and processes which minimize the use and generation of hazardous substances, energy, and toxic catalysts. Ultrasound irradiation is one of the important energy sources used as a powerful technique to promote organic reactions. Ultrasonic irradiation is considered a green energy source, because it can enable organic reactions at shorter times, and the reactions have usually higher yields in comparison to those of conventional thermal energy sources. Due to these advantages, recently chemists have focused their attention on this type of energy source, and there are many reports in which ultrasonic irradiation have been used in organic synthesis [1–5].

Polysubstituted benzenes are important compounds in organic chemistry, and they widely occur in natural products and material science [6]. The commonly synthetic methodologies for synthesis of polysubstituted benzenes include coupling reactions [7], *Friedel–Crafts* reactions [8], and electrophilic and nucleophilic substitutions [9][10]. However, these protocols suffer from some disadvantages such as low yields, long reaction times, regioisomeric problems, and low atom economy [11][12]. Moreover, applying these methods for preparation of nitro or cyano derivatives of benzenes is also difficult. These limitation led chemists to try to synthesize the aromatic backbone from acyclic precursors to avoid regioisomeric problem and low atom economy. For this purpose, several methods have been developed such as transition metal-catalyzed [2+2+2] and [4+2] cycloadditions [13], *Dotz* reaction of *Fisher* carbene complexes [14], and [4+2] annulation strategy by the *Baylis–Hillman* reaction [15]. Recently, reaction between 2-(1-arylethylidene)malononitrile **2** (*cf. Scheme 1*) as nucleophile and some electrophilic C=C bonds has been used to synthesize related polysubstitued benzenes [16–20].

Chromenes are among the most important O-containing heterocyclic compounds. They are widely found in a large range of biologically active molecules, plants, and natural products [21]. Over the years, the synthesis of several chromene analogs has been developed, and some of them display pharmaceuticals effects including, antiviral [22], anticoagulant [23], antifungal [24], and molluscidial activities [25]. Due to these properties, there are many reports on the synthesis of new chromene derivatives [26–29].

Results and Discussion. – In continuation of our studies on the synthesis of new polycyclic heterocyclic compounds [30–32], we investigated the reaction of salicylaldehydes (=2-hydroxybenzaldehydes) **1**, malononitrile (=propanedinitrile), and 2-(1-arylethylidene)malononitrile **2** to synthesize new polysubstitued benzene-fused chromene derivatives *via* a one-pot three-component reaction under ultrasound irradiation (*Scheme 1*).

Initially, 2-(1-arylethylidene)malononitrile **2** has been prepared as described in [33]. We selected the reaction of **1a**, malononitrile, and **2a** as a model, and the reaction conditions were investigated for this model (*Table 1*). Performance of the reaction under thermal and ultrasound conditions, in different solvents, and in presence of different and varying amounts of bases has been studied. As can be seen from *Table 1*, the best results were obtained by sonication of the reaction mixture in EtOH in the

Scheme 1. *Synthesis of 7-Amino-6-imino-9-aryl-6*H*-benzo[*c*]chromene-8-carbonitrile Derivatives*

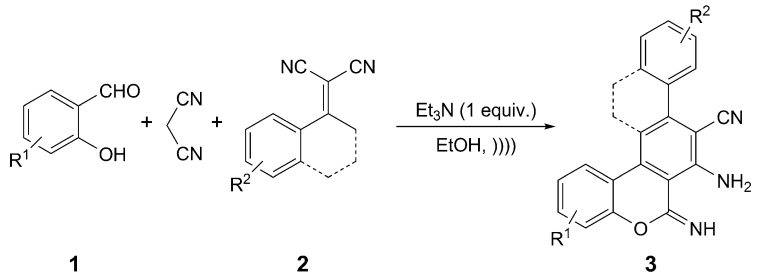


Table 1. *Optimization of the Reaction Conditions*

Entry	Solvent	Base [equiv.]	Yield [%][a])	Yield [%][b])
1	EtOH	Et_3N (1)	50	82
2	MeCN	Et_3N (1)	45	74
3	THF	Et_3N (1)	30	47
4	CH_2Cl_2	Et_3N (1)	18	31
5	EtOH	Et_3N (0.05)	–	–
6	EtOH	Et_3N (0.1)	–	–
7	EtOH	Piperidine (1)	–	–
8	EtOH	K_2CO_3 (1)	35	60

[a]) Reflux; 6 h. [b]) Ultrasonic irradiation; 40 min.

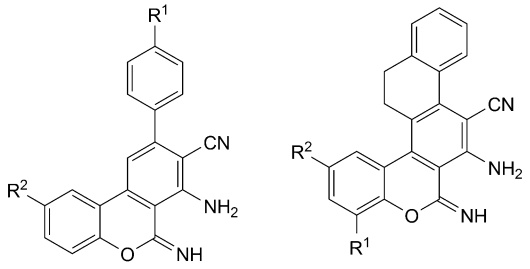
presence of 1 equiv. Et_3N to afford 7-amino-6-imino-9-phenyl-6*H*-benzo[c]chromen-8-carbonitrile (**3a**) in good yield (*Table 1*, *Entry 1*).

Further experiments were conducted by using catalytic amounts of Et_3N to compare their catalytic activities under similar conditions but this attempt failed to provide the desired product, and the intermediate **7** (*cf. Scheme 2*, below; *Table 1*, *Entries 5* and *6*). Although the *Knovenagel* condensation between salicyclaldehyde (**1a**) and malononitrile has been performed in the presence of catalytic amounts of Et_3N in aromatization step by losing HCN, the equivalent amount of the base was needed. In another attempt, we performed the model reaction in the presence of piperidine, and a complex mixture was obtained (*Table 1*, *Entry 7*). Having established the optimal conditions, we then examined the scope of the reaction for the synthesis of compounds **3**, and the results are compiled in *Table 2*.

The structures of compounds **3a**–**3f** were deduced from their IR, and high-field ^{1}H- and ^{13}C-NMR spectra, and elemental analyses. The mass spectrum of **3a** displayed the molecular-ion peak at *m*/*z* 311, in agreement with the proposed structure. In the IR spectrum of **3a**, two sharp absorption bands at 3438 and 3349, a sharp band at 2206, a sharp band at 1607, three bands at 1559, 1471, and 1424, and a sharp band at 1261 cm^{-1}, were observed, and they were assigned to NH_2, NH, CN, C=N, C=C, and C–O stretching frequencies, respectively, clearly indicating the most significant functional groups of the product. The ^{1}H-NMR spectrum of **3a** exhibited one *multiplet*, one *triplet*, and two *doublet*s for aromatic H-atoms (δ(H) 7.45–7.53, 7.7 ($J = 8.0$), 8.41 ($J = 7.6$), and 8.58 ($J = 8.0$)), and four *singlet*s for H-atoms of polysubstituted benzene ring, NH_2, and NH, respectively (7.60, 7.58, and 8.34 ppm, resp.). The ^{1}H-decoupled ^{13}C-NMR spectrum of **3a** showed 18 distinct signals in agreement with the suggested structure.

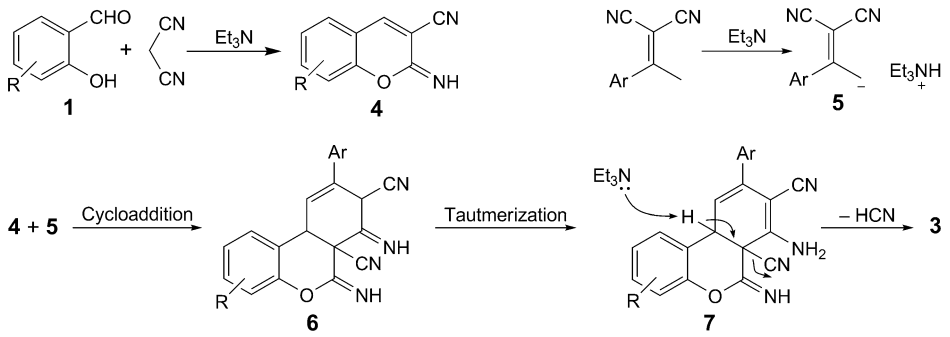
According to results, a plausible mechanism for the sequential three-component reaction is proposed (*Scheme 2*).

Table 2. *Scope of the Reaction*



Entry	Product	Structure	R^1	R^2	Yield [%]
1	**3a**	**A**	H	H	82
2	**3b**	**A**	Me	H	86
3	**3c**	**A**	H	MeO	83
4	**3d**	**B**	H	H	80
5	**3e**	**B**	MeO	H	73
6	**3f**	**B**	H	MeO	85

Scheme 2. *A Plausible Mechanism for the Formation of Products* **3a**–**3f**



The formation of chromene-bearing polysubstituted benzenes **3** can be rationalized by the initial *Knoevenagel* condensation between salicylaldehydes **1** and malononitrile in the presence of Et_3N leading to 2-iminochromene-3-carbonitriles **4**. Then, cycloaddition of 2-(1-arylethylidene)malononitrile ion pair **5** to the intermediate **4** leads to intermediate **6** and, after imine–enamine tatumerization, to intermediate **7**. Finally, dehydrocyanation of **7** affords the desired products **3**. To support the proposed mechanism, 2-iminochromene-3-carbonitrile **4** was synthesized separately [34], and it was added to 2-(1-arylethylidene)malononitrile ion-pair solution, and the same result was observed (*Scheme 1*). This reaction has been further performed in the presence of catalytic amount of Et_3N and the intermediate **7** was separated, and its structure was established by IR and MS data. This observation clearly evidenced that, for dehydrocyanation step, 1 equiv. of base was required.

In summary, we described a novel approach to the synthesis of new polysubstituted benzene derivatives by one-pot reaction between salicylaldehydes, malononitrile, and 2-(1-arylethylidene)malononitrile under ultrasound irradiation. The present method has some advantages such as excellent yields of the products, fairly fast reaction times, and simple purification. The simplicity of the present procedure renders it an interesting alternative to complex multistep approaches.

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Experimental Part

General. All starting materials were obtained from *Merck* (Germany) and *Fluka* (Switzerland), and were used without further purification. M.p.: *Electrothermal 9100* apparatus. IR Spectra: in KBr on a *Shimadzu IR-460* spectrometer. ^{1}H- and ^{13}C-NMR spectra: at 400 and 100 MHz, resp., on a *BRUKER DRX 400-AVANCE* FT-NMR instrument, in $CDCl_3$, if not otherwise stated. MS: *FINNIGAN-MAT* 8430 mass spectrometer operating at an ionization potential of 70 eV. Elemental analyses for C, H, and N: *Heraeus CHN-O-Rapid* analyzer.

Representative General Procedure (for **3a**). A soln. of *salicylaldeyde* (=2-hydroxybenzaldehyde; **1a**; 0.122 g, 1 mmol), *2-(1-phenylethylidene)propanedinitrile* (**2a**; 0.168 g, 1 mmol), propanedinitrile (0.066 g, 1 mmol), and Et_3N (0.101 g, 1 mmol) in EtOH (3 ml) was sonicated by a 6-mm ultrasonic probe for 40 min. After completion, the mixture was filtered, and the precipitate was washed with hot MeCN to afford the pure product **3a**.

*7-Amino-6-imino-9-phenyl-6*H-*benzo[*c*]chromene-8-carbonitrile* (**3a**). Yield: 255 mg (82%). Yellow powder. M.p. 300°. IR: 3438, 3349 (NH_2, NH), 2206 (CN), 1607 (C=N), 1559, 1471, 1424 (Ar), 1261 (C–O). ^{1}H-NMR: 7.47 (*t*, *J* = 7.6, 1 H); 7.51 (*t*, *J* = 8.8, 1 H); 7.54 (*s*, 2 H); 7.57 (*s*, 1 H); 7.59 – 7.60 (*m*, 2 H); 7.70 (*t*, *J* = 8.0, 1 H); 7.78 (*t*, *J* = 8.0, 1 H); 8.34 (*s*, 1 H); 8.41 (*d*, *J* = 7.6, 2 H); 8.58 (*d*, *J* = 8.0, 1 H). ^{13}C-NMR: 102.5; 104.7; 116. 5; 117.3; 117.9; 124.8; 125.2; 125.3; 127.7; 128.7; 130.7; 133.1; 137.9; 138.6; 151.7; 155.4; 159.9; 162.1. EI-MS (70 eV): 311.34 (17, M^+), 272 (12), 236 (10), 171 (25), 149 (25), 105 (100), 97 (33), 83 (62), 69 (96). Anal. calc. for $C_{20}H_{13}N_3O$ (311.34): C 77.16, H 4.21, N 13.50; found: C 77.09, H 4.15, N 13.46.

*7-Amino-6-imino-9-(4-methylphenyl)-6*H-*benzo[*c*]chromene-8-carbonitrile* (**3b**). Yield: 279 mg (86%). Yellow powder. M.p. 300 – 302°. IR: 3441, 3347, 3170 (NH_2, NH), 2206 (CN), 1603 (C=N), 1479, 1424 (Ar), 1265 (C–O). ^{1}H-NMR: 2.41 (*s*, 3 H); 7.39 (*d*, *J* = 7.6, 2 H); 7.40 (*t*, *J* = 7.2, 1 H); 7.50 (*s*, 2 H); 7.52 (s, 1 H); 7.54 (*d*, *J* = 7.2, 1 H); 7.71 (*t*, *J* = 7.2, 1 H); 8.33 (*d*, *J* = 7.6, 2 H); 8.34 (*s*, 1 H); 8.60 (*d*, *J* = 7.2, 1 H). ^{13}C-NMR: 21.0; 102.4; 104.3; 116.5; 117.3; 117.9; 124.8; 125.2; 125.3; 127.7; 129.4; 133.1; 135.0; 138.4; 140.7; 151.7; 155.5; 159.1; 162.1. EI-MS (70 eV): 326 (29, $[M+1]^+$, 29), 325 (100, M^+), 324 (7), 311 (5), 149 (25), 118 (16), 105 (35), 95 (16), 81 (45), 69 (71), 55 (25). Anal. calc. for $C_{21}H_{15}N_3O$ (325.37): C 77.52, H 4.65, N 12.91; found: C 77.49, H 4.72, N 12.81.

*7-Amino-6-imino-2-methoxy-9-phenyl-6*H-*benzo[*c*]chromene-8-carbonitrile* (**3c**). Yield: 283 mg (83%). Yellow powder. M.p. 300°. IR: 3303, 3177 (NH_2 and NH), 2203 (CN), 1596 (C=N), 1480, 1420 (Ar), 1261 (C–O). ^{1}H-NMR: 3.91 (*s*, 3 H); 7.25 (*d*, *J* = 7.6, 1 H); 7.44 (*d*, *J* = 7.6, 1 H); 7.47 (*s*, 2 H); 7.55 – 7.70 (*m*, 4 H); 8.00 (*s*, 1 H); 8.34 (*s*, 1 H); 8.42 (*d*, *J* = 7.2, 1 H). ^{13}C-NMR: 57.7; 103.8; 106.3; 109.0; 118.1; 119.2; 120.6; 122.1; 129.4; 130.3; 132.2; 139.5; 140.1; 147.4; 157.1; 158.0; 160.6; 163.1; 163.3. EI-MS (70 eV): 341 (M^+), 239 (3), 200 (83), 185 (12), 168 (100), 140 (43), 128 (35), 102 (35), 77 (25). Anal. calc. for $C_{21}H_{15}N_3O_2$ (341.37): C 73.89, H 4.43, N 12.31; found: C 73.97, H 4.34, N 12.20.

*6-Amino-13,14-dihydro-7-imino-7*H-*phenanthro[2,1-*c*]chromene-5-carbonitrile* (**3d**). Yield: 269 mg (80%). Yellow powder. M.p. > 300°. IR: 3464, 3354, 3184 (NH_2, NH), 2209 (CN), 1605 (C=N), 1548, 1478 (Ar), 1226 (C–O). ^{1}H-NMR: 2.84 – 2.90 (*m*, 2 H); 3.00 – 3.07 (*m*, 2 H); 7.18 (*s*, 2 H); 7.37 (*d*, *J* = 7.6, 1 H); 7.42 (*t*, *J* = 7.6, 1 H); 7.45 (*d*, *J* = 7.6, 1 H); 7.50 (*t*, *J* = 7.6, 1 H); 7.57 (*t*, *J* = 7.6, 1 H); 7.64 (*t*, *J* = 7.6, 1 H); 7.95 (*d*, *J* = 7.6, 1 H); 7.98 (*d*, *J* = 7.6, 1 H); 8.12 (*s*, 1 H). ^{13}C-NMR: 27.6; 30.6; 101.6; 115.7; 117.1; 119.3; 122.5; 124.4; 125.1; 126.1; 126.5; 127.0; 128.4; 128.9; 130.7; 131.3; 131.9; 132.7; 151.3; 154.8; 160.1; 163.1. EI-MS (70 eV): 336 ($[M-1]^+$), 300 (13), 236 (100), 209 (48), 181 (75), 171 (58), 140 (30), 127 (38), 115 (37), 91 (36), 77 (16). Anal. calc. for $C_{22}H_{15}N_3O$ (337.38): C 78.32, H 4.48, N 12.45; found: C 78.45, H 4.61, N 12.34.

*6-Amino-13,14-dihydro-7-imino-9-methoxy-7*H-*phenanthro[2,1-*c*]chromene-5-carbonitrile* (**3e**). Yield: 267 mg (73%). Green-yellow powder. M.p. > 300°. IR: 3450, 3344, (NH_2, NH), 2212 (CN), 1632 (C=N), 1552, 1474 (Ar), 1275, 1207 (C–O). ^{1}H-NMR: 2.94 – 2.97 (*m*, 2 H); 3.02 – 3.17 (*m*, 2 H); 3.94 (*s*, 3 H); 7.03 (*d*, *J* = 7.6, 1 H); 7.13 (*s*, 2 H); 7.18 (*d*, *J* = 7.6, 1 H); 7.24 (*t*, *J* = 7.6, 1 H); 7.32 (*t*, *J* = 7.6, 1 H); 7.42 (*t*, *J* = 7.2, 1 H); 7.90 (*d*, *J* = 7.2, 1 H); 8.14 (*d*, *J* = 7.2, 1 H); 8.36 (br. *s*, 1 H). ^{13}C-NMR: 28.4; 30.7; 55.9; 103.3; 111.5; 113.7; 114.5; 119.5; 120.7; 126.5; 127.0; 127.4; 128.9; 129.4; 129.7; 130.7; 132.6; 133.8; 148.1; 151.5; 153.8; 158.5; 162.5. EI-MS (70 eV): 367 (100, M^+), 350 (30), 334 (16), 304 (20), 267 (37), 243 (16), 201 (16), 167 (15), 126 (15). Anal. calc. for $C_{23}H_{17}N_3O_2$ (367.40): C 75.19, H 4.66, N 11.44; found: C 75.33, H 4.71, N 11.35.

*6-Amino-13,14-dihydro-7-imino-11-methoxy-7*H-*phenanthro[2,1-*c*]chromene-5-carbonitrile* (**3f**). Yield: 321 mg (85%). Yellow powder. M.p. > 300°. IR: 3394, 3336, 3221 (NH_2, NH), 2221, 2185 (CN), 1655 (C=N), 1582, 1498 (Ar), 1208 (C–O). ^{1}H-NMR: 2.82 – 2.90 (*m*, 2 H); 3.00 – 3.04 (*m*, 2 H); 3.73 (*s*, 3 H); 7.27 (*d*, *J* = 8.00, 1 H); 7.36 (*d*, *J* = 8.00, 1 H); 7.39 (*s*, 2 H); 7.44 (*t*, *J* = 7.2, 1 H); 7.55 (*t*, *J* = 7.2, 1 H); 7.64 (*s*, 1 H); 7.72 (*d*, *J* = 7.6, 1 H); 7.89 (*d*, *J* = 7.6, 1 H); 8.30 (*s*, 1 H). ^{13}C-NMR: 23.3; 25.2; 55.5; 102.5; 111.4; 113.4; 114.4; 120.9; 122.9; 126.2; 126.7; 128.2; 128.6; 129.1; 129.4; 131.6; 133.3; 133.8; 149.8; 151.5; 155.6; 158.7; 163.5. EI-MS (70 eV): 367 (100, M^+), 350 (30), 336 (20), 304 (20), 279 (20), 267 (37), 167 (6), 139 (13), 126 (15). Anal. calc. for $C_{23}H_{17}N_3O_2$ (367.40): C 75.19, H 4.66, N 11.44; found: C 75.33, H 4.78, N 11.33.

*7-Amino-6-imino-9-phenyl-6*H-*benzo[*c*]chromene-6a,8(10a*H*)-dicarbonitrile* (**7a**). Yellow powder. M.p. 284°. IR: 3444, 3216 (NH_2, NH), 2360, 2207 (CN), 1602 (C=N), 1525, 1414, 1424 (Ar), 1255 (C–O). EI-MS (70 eV): 338 (27, M^+), 336 (100), 311 (10), 301 (20), 272 (30), 168 (12), 104 (10), 91 (11), 77 (22).

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