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Short Note

3-Amino-4-(diphenylamino)-1*H*-2-benzopyran-1-one

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Abstract: Various synthetic methodologies to obtain 3,4-diaminoisocumarin nucleus have been reported and described. However, mechanistic analysis based on experimental evidence is lacking. Herein, we report the synthesis of the novel 3-amino-4-(diphenylamino)-1*H*-2-benzopyran-1-one using a two-step methodology with a new mechanistic proposal to explain the formation of the latter based on previously reported precursors and the established conditions. This compound was afforded in 80% yield.

Keywords: phthalides; isobenzofuranones; diaminoisocoumarins; ring contracting rearrangement

1. Introduction

Isocoumarins (1*H*-isochromen-1-ones or 1*H*-2-benzopyran-1-ones) and some of their dihydro derivatives are considered relevant organic cores owing to their significance in medicinal and pharmacological chemistry. A wide variety of biological activities have been tested and demonstrated, such as, for example, antioxidant Penicimarin N (**1**) from mangrove-derived fungus *Penicillium* sp. [1], zoosporocidal 6,8-dimethoxy-3-methylisocoumarin (**2**) from terrestrial *Streptomyces* sp. [2], insecticidal Peniciisocoumarin E (**3**) against *Helicoverpa armiguera* [3], a cytotoxic named Beriticulol (**4**), (12S)-12-hydroxymonocerin (**5**) from *Setosphaeria rostrate* [4,5] and anti-inflammatory species (**6**) from *Homalium paniculiflorum* [6] (Figure 1). As mentioned, isocoumarins have been obtained from different natural sources, where vegetal and fungi species are the main exponents.

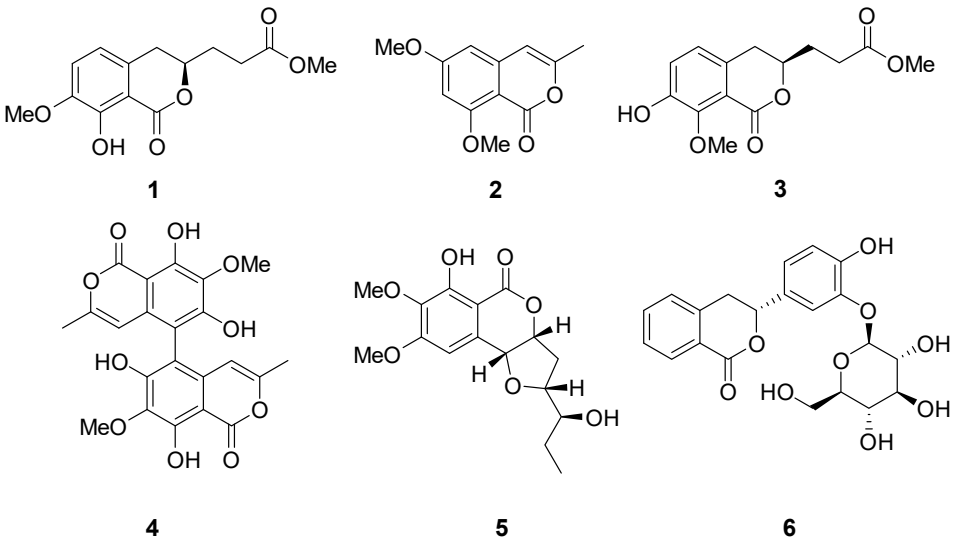

Figure 1. Representative examples of isocoumarin derivatives with demonstrated biological activities.

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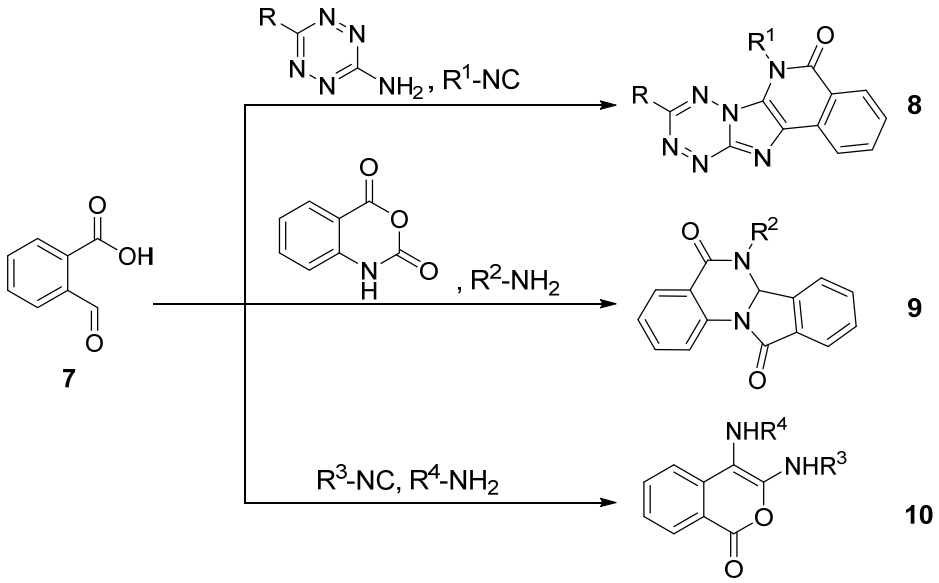
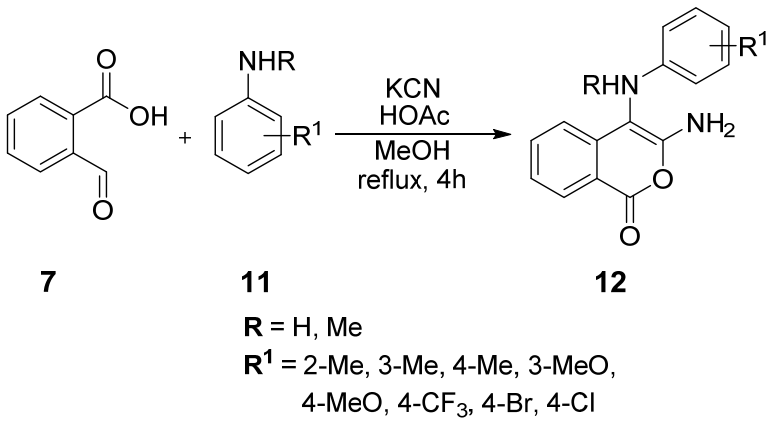
The vast applicability of these types of compounds in areas such as medicine and agriculture [7] has attracted the interest of organic and synthetic chemists, who have designed a considerable diversity of methodologies to afford isocoumarin derivatives [8–12]. Given its structure and extensive derivatization capacity, 2-formylbenzoic acid (**7**) has become a valuable reagent in multicomponent reactions (MCRs). There are many reports in which this molecule is used as a key reagent to achieve high-complexity heterocyclic compounds focusing on atom economy. Recent examples consisted of the synthesis of tetrazinoimidazoisoquinolinones **8** [13], 6,6a-dihydroisoindolo [2,1-*a*]quinazoline-5,11-dione and 5-phenylisoindolo [2,1-*a*]quinazolin-11(6a*H*)-one derivatives **9** [14] as well as isocoumarin ones **10** [15] (Scheme 1).



Scheme 1. Previous reports of MCRs with 2-formylbenzoic acid.

One of the most versatile methods for the synthesis of the 3,4-diaminoisocoumarins derivatives **10** was reported by Opatz and Ferenc [16]. They used KCN, an arylamino-derivative with 2-formylbenzoic acid (**7**), as depicted in Scheme 2.

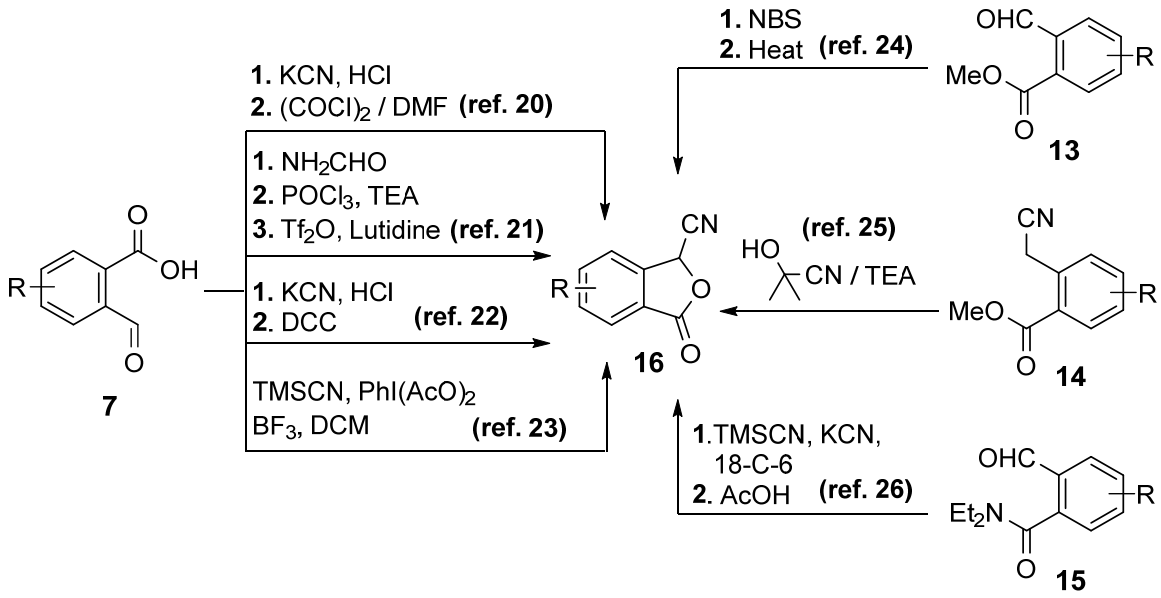


Scheme 2. Multicomponent synthesis of 3,4-diaminoisocoumarins derivatives **12**.

Following this methodology, our research group has reported the synthesis of several derivatives which have been applied for the obtention of more complex structures [17]. Overall, 3,4-diaminoisocoumarin derivatives have been achieved using two main procedures, which consist of 2-formylbenzoic acid, a primary amine derivative and a cyanide

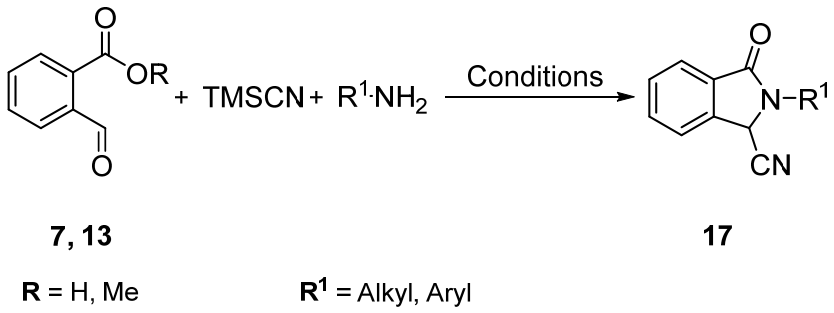
source (inorganic or organic) or an isonitrile derivative. Different mechanisms have been postulated to explain the formation of the final product **12** [16,18,19].

However, when a cyanide source (KCN, trimethylsilyl cyanide (TMSCN) or acetone cyanohydrin) is mixed with 2-formylbenzoic acid or its derivatives under the described conditions, the major or unique product is 3-oxo-1,3-dihydro-2-benzofuran-1-carbonitrile **16** [20–26] (Scheme 3).



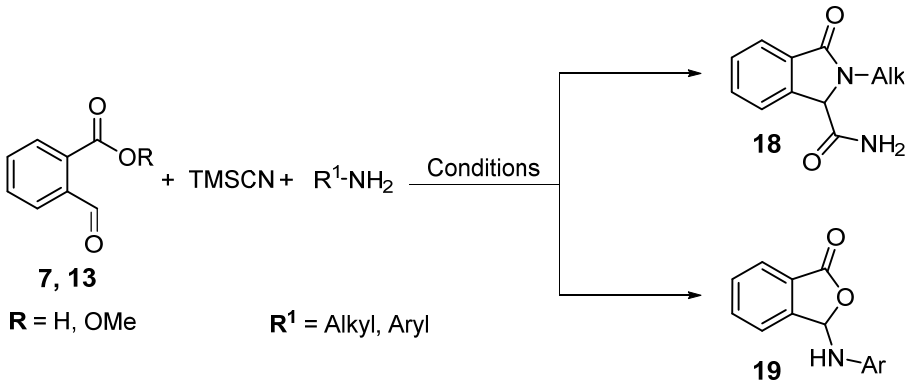
Scheme 3. Previous reports of 3-oxo-1,3-dihydro-2-benzofuran-1-carbonitrile **16** synthesis from **7** derivatives and cyanide sources [20–26].

Contrastingly, when 2-formylbenzoic acid or its methyl ester reacts with TMSCN and an aliphatic or aryl primary amine, the afforded product is 2-alkyl(aryl)-3-oxo-2,3-dihydro-1*H*-isoindole-1-carbonitrile **17** [27–30] (Scheme 4).



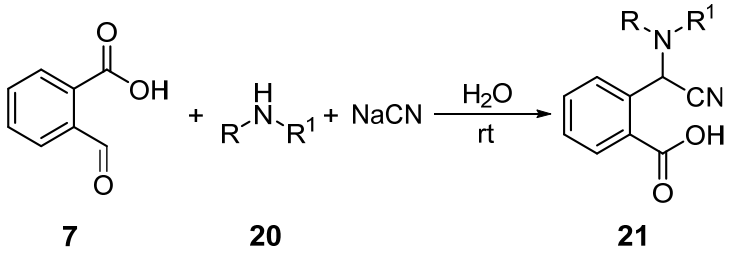
Scheme 4. Synthesis of 2-alkyl(aryl)-3-oxo-2,3-dihydro-1H-isoindole-1-carbonitrile (**17**) from **7** and ester derivative, TMSCN and primary amines.

It is important to note that when Bunce's group reacted 2-formylbenzoic acid, primary alkyl or arylamines and TMSCN under the same conditions, two different nuclei were formed [29] (Scheme 5).



Scheme 5. Bunce's synthesis of **18** and **19** from **7**, **13**, TMSCN and primary amines.

Another experimental observation is provided by Soleimani's group [31]. They carried out the tricomponent reaction of 2-formylbenzoic acid, sodium cyanide and secondary aliphatic amines, obtaining Strecker product **21** (Scheme 6).

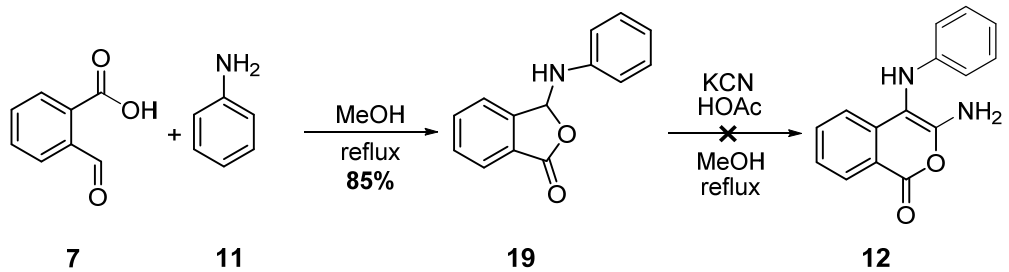


Scheme 6. Synthesis of α-aminonitriles from 2-formylbenzoic acid via Strecker reaction.

These findings caught our attention because we expected products **19** and **21** would be the same as those obtained using the Opatz and Ferenc methodology [16], i.e., derivatives of 3,4-diaminoisocoumarins **12**. Considering all these established findings, in the present work, we report the first synthesis of a new derivative of 3,4-diaminoisocoumarin, which has two phenyl substituents attached in the nitrogen atom at C-4, by using a two-step pathway.

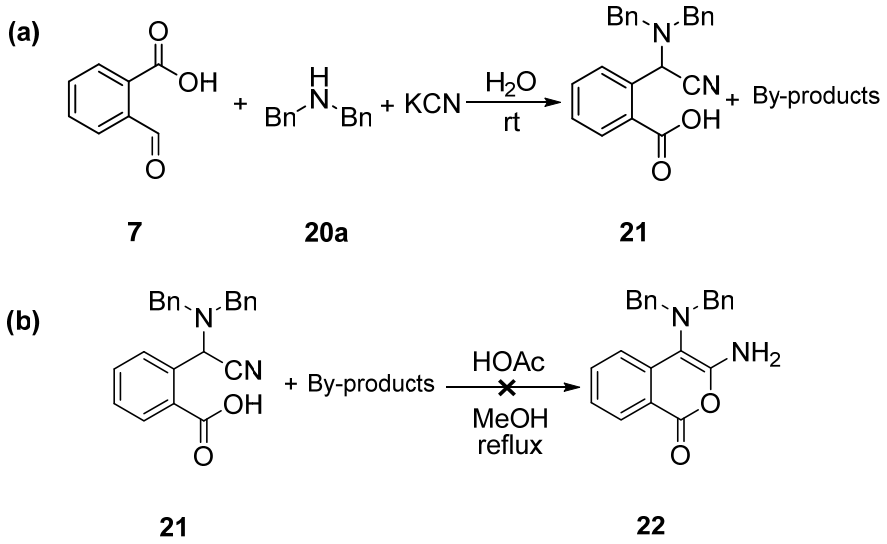
2. Results and Discussion

During our ongoing research on the synthesis of new heterocyclic compounds containing the isocoumarin nucleus, we wanted to understand the mechanism by which the 3,4-diaminoiscoumarin derivatives are formed. To do this, we carried out two experiments. In the first one, we submitted compound **19** (obtained as shown in Scheme 7), to react with KCN and acetic acid in boiling methanol as a modification of the Opatz and Ferenc methodology [16]. TLC control did not show the formation of compound **12**, although the reaction mixture turned slightly yellow.



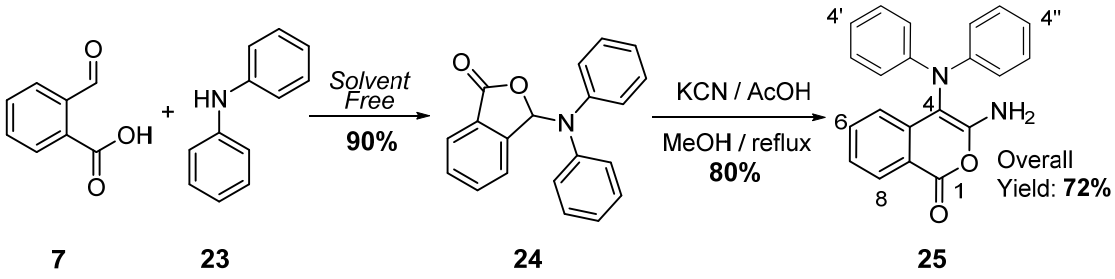
Scheme 7. Attempted synthesis of **12** using Opatz and Ferenc methodology modification.

In the second experiment, we tried to develop the protocol proposed by Soleimani's group, but we were unable to isolate a pure product in the high reported yields [31]. We assumed that α-aminonitrile **21** was in the reaction mixture, along with other by-products (Scheme 8a) (see Supplementary Materials, Figure S1). Expecting to obtain compound **22**, we reacted this mixture with acetic acid in boiling methanol. However, as in the previous experiment (Scheme 7) it was not possible to isolate any product, although the solution developed the characteristic yellowish color of 3,4-diaminoisocoumarin derivatives (Scheme 8b).



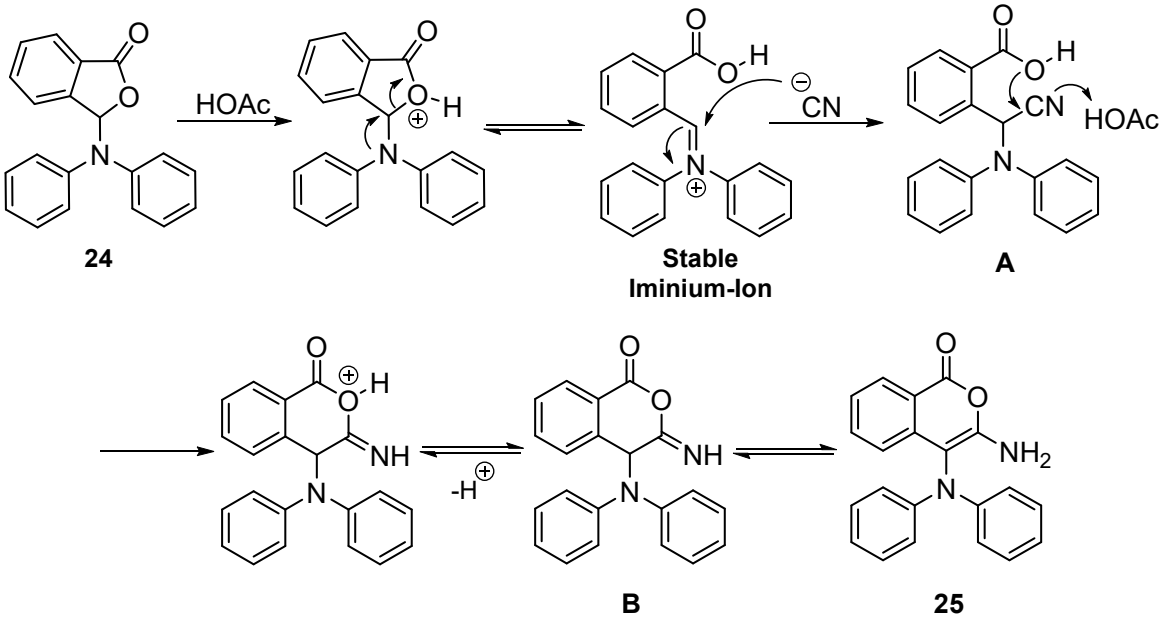
Scheme 8. (**a**). Synthesis of α-aminonitrile **21** using Soleimani's methodology. (**b**). Attempted synthesis of diaminoisocoumarin **22** from compound **21**.

Aiming to understand the behavior of this type of reaction, we decided to synthesize compound **24** following the methodology reported by Moreno-Fuquen [32]. Then, we reacted phthalide **24** with KCN and acetic acid in refluxing methanol, as shown in Scheme 7. Surprisingly, the solution slowly began to turn yellowish, and a TLC control showed the formation of a single bright-yellow product (Scheme 9).



Scheme 9. Two-step sequence for the synthesis of **25**.

Derived from the experimental evidence, we postulate the following reaction mechanism (Scheme 10). The first step is the protonation of an oxygen atom of the phthalide ring, which leads to the formation of the stable iminium ion. Then, cyanide anion attacks the iminium ion, forming **A**. finally, intermediate **A** cyclizes to form **B**. The latter tautomerizes to product **25**. We consider the formation of the iminium ion stabilized by the two aromatic phenyl rings as the key step of the reaction.



Scheme 10. Proposed mechanism for the formation of **25**.

Compound **25** was characterized based on FT-IR, ^{1}H and ^{13}C-NMR, and its melting point (see Supplementary Materials). In the IR spectrum, there were two absorption bands at 3479 and 3288 cm^{-1}, which were assigned to the N-H bond of the primary amine group on the benzopyran ring. A strong absorption band at 1734 cm^{-1} corresponding to the carbonyl of the lactone group was observed. The C-O and C(sp^{2})-N bands appeared at 1270 and 1297 cm^{-1}, respectively. Additionally, two strong absorption bands appeared at 757 and 692 cm^{-1}, indicating the monosubstitution of the two phenyl rings observed for compound **25**. The HR-MS featured a peak at m/z = 329.12873, which is in accordance with the [M + H]$^{+}$ molecular ion.

The ^{1}H-NMR spectrum signals of the purified product were in accordance with the proposed structure. The first signal at 6.81 ppm was assigned to the NH_2 protons. There was a triplet at 6.90 ppm corresponding to the protected H-4′,4″ protons. A doublet at 7.02 ppm was attributed to H-5 proton on the benzopyran system, while a multiplet centered at 7.11 ppm represented the H-7 proton and both couples of H-2′,6′ and H-2″,6″ protons at the diphenyl amino moiety. An unresolved doublet of doublets at 7.22 ppm was attributed to H-3′,5′ and H-3″,5″. The resting aromatic proton signals were at 7.51 ppm as a doublet of doublets of doublets for H-6 and at 7.96 as doublet of doublets for H-8.

Finally, the ^{13}C-NMR spectrum exhibited a total of 13 signals. The APT experiment showed that six of them were corresponding to quaternary carbons, where the signal resonating at 160.2 ppm was assigned to C-1 (the carbonyl group of the lactone). The rest of the quaternary and tertiary signals were attributed to aromatic carbons on the diphenylamino rings and the rest of the benzopyran system. All the described findings are supported by ^{1}H- ^{1}H-COSY, ^{1}H-^{13}C-HSQC, and ^{1}H-^{13}C-HMBC spectra (see Supplementary Materials), in strong agreement with the proposed structure of compound **25**. Numbering can be seen in Scheme 9.

3. Materials and Methods

3.1. General Information

The reagents and solvents used were obtained from commercial sources and were used without previous purification. The reaction progress was monitored via TLC with 0.2 mm precoated plates of silica gel 60 F254 (Merck). The melting points were measured using a Stuart SMP3 melting point apparatus (Cole-Parmer, Staffordshire, UK) and were corrected. The IR spectrum was recorded on a Shimadzu IR Affinity (Shimadzu, Kyoto, Japan) with

ATR probe. The ^{1}H, ^{13}C-NMR, ^{1}H- ^{1}H-COSY, ^{1}H-^{13}C-HSQC, and ^{1}H-^{13}C-HMBC spectra were recorded in a BRUKER DPX 400 spectrophotometer (Bruker, Bruker BioSpin GmbH, Rheinstetten, Germany) operating at 400 and 100 MHz, respectively, using DMSO-d6 as the solvent. Chemical shifts (δ) are given in ppm and coupling constants (*J*) are given in *Hz*. The following abbreviations are used for multiplicities: s—singlet, d—doublet, t—triplet, dd—doublet of doublets, and m—multiplet. High-resolution mass spectra (HRMS) were recorded using an Agilent Technologies Q-TOF 6520 spectrometer via electrospray ionization (ESI).

*3.2. Preparation of 3-Amino-4-(diphenylamino)-1H-isochromen-1-one (***25***)*

To a stirred solution of **24** (1.50 g, 5mmol) in methanol (12 mL), acetic acid (0.43 mL, 7.5 mmol) and potassium cyanide (0.39 g, 6 mmol) were added. The reaction mixture was refluxed for 8 h (the reaction progress was controlled by TLC). Once cooled, the yellow precipitate was filtered and washed with methanol, yielding **25** as bright-yellow crystals.

Yield: 1.31 g, 80%. M.p. 175 °C (dec). FT-IR (KBr disk) $\bar{\upsilon}$ (cm^{-1}): 3479, 3288, 3166, 1734, 1632, 1602, 1582, 1483, 1297, 1270, 757, 692. ^{1}H RMN (400 MHz, DMSO-d_6) δ (ppm) 6.81 (s, 2H, NH_2), 6.90 (t, *J* = 7.2 Hz, 2H, 4′,4″-H), 7.02 (d, *J* = 8.0 Hz, 1H, 5-H), 7.09–7.14 (m, 5H, 7-H, 2′,6′-H, 2″,6″-H), 7.22 (dd, *J* = 8.8, 7.2 Hz, 4H, 3′,5′-H, 3″,5″-H), 7.51 (ddd, *J* = 8.4, 7.2, 1.4 Hz, 1H, 6-H), 7.96 (dd, *J* = 8.1, 1.5 Hz, 1H, 8-H). ^{13}C NMR (100 MHz, DMSO-d_6) δ (ppm) 95.6 (C-4), 114.2 (C-8a), 119.1 (C-5), 119.4 (C-2′,6′, C-2″,6″), 121.3 (C-4′, C-4″), 122.6 (C-7), 129.1 (C-3′,5′, C-3″,5″), 129.7 (C-8), 135.3 (C-6), 141.4 (C-4a), 145.4 (C-1′, C-1″), 156.6 (C-3), 160.2 (C-1). HR-MS (ESI): *m*/*z* calculated for $[M + H]^+$: 329.12900, found: 329.12873.

4. Conclusions

We have developed a new methodology to afford compound **25** from previously reported **24** as a modification of the Opatz and Ferenc procedure. A mechanistic proposal was designed and explained in order to guide the comprehension of the diaminoisocoumarins formation and describe how this reaction is influenced by the *N*-substitution of the arylamino moiety. Compound **25** was successfully characterized by its spectroscopic properties.

Supplementary Materials: Figure S1. ^{1}H-NMR spectrum of product mixture (Scheme 8a); Figure S2. 1H-NMR spectrum of compound **25**; Figure S3. ^{13}C-NMR spectrum of compound **25**; Figure S4. APT spectrum of compound **25**; Figure S5. ^{1}H- ^{1}H-COSY spectrum of compound **25**; Figure S6. ^{1}H- ^{13}C-HSQC spectrum of compound **25**; Figure S7. ^{1}H- ^{13}C-HMBC spectrum of compound **25**; Figure S8. FT-IR spectrum of compound **25**; Figure S9. HR-MS of compound **25**.

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Conflicts of Interest: The authors declare no conflict of interest.

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