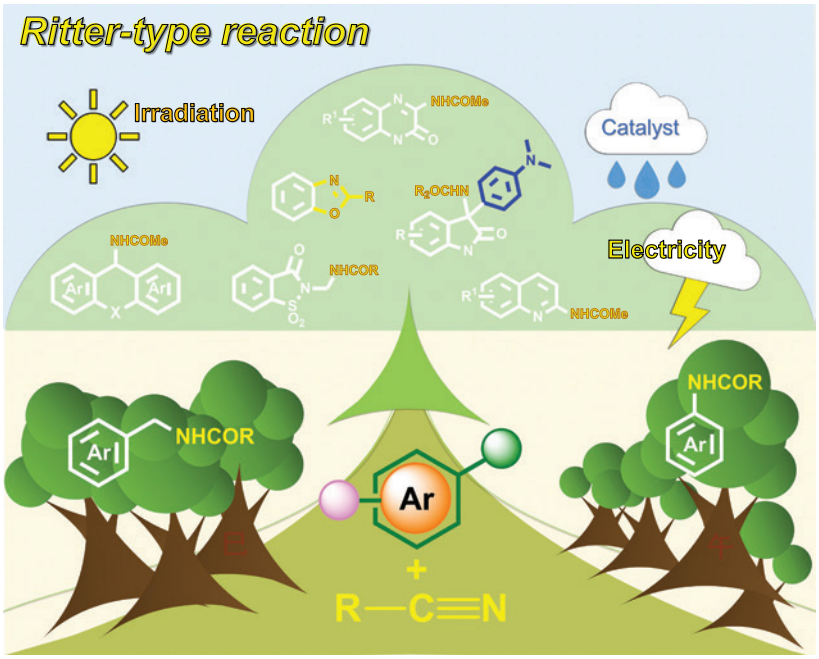
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State-of-the-art and prospects of studying Ritter-type reactions in the synthesis of *N*-(het)arylamides and *N*-benzylamides: from the traditional approach to photo- and electrochemistry

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N-(Het)aryl- and *N*-benzylamides are a practically relevant class of compounds that are widely used in medicinal chemistry, agrochemistry, catalysis, organic synthesis and materials chemistry. The amide group is one of the important structural moieties that form the basis of proteins, peptides, pharmaceuticals, polymeric functional materials, *etc*. To this day, the Ritter reaction, discovered in 1948, is often used in chemical synthesis to obtain amides, especially sterically crowded ones. Nitriles can act as a source of the amide function in the synthesis of *N*-(het)aryl- and *N*-benzylamides. The study of such transformations, which are carried out both under conditions of classical organic synthesis and using photochemistry, electrochemistry and photoelectrochemistry, is a relevant area of research, as evidenced by modern works on this topic. This review summarizes and analyzes the most significant achievements of recent years in the field of synthesis of *N*-(het)aryl- and *N*-benzylamides by reactions following the Ritter reaction pathway. Processes based on traditional methods of organic chemistry are presented, as well as modern of 'green' chemistry approaches using photo- and electrochemical activation of reagents. In addition, known reactions of amidation of heteroaromatic substrates with nitriles, methods of amidation using three-component systems, and amidation of *N*-(hydroxymethyl)saccharin are considered in separate Sections. Proposed mechanisms of transformations that ensure the selectivity of the processes are discussed in detail.
The bibliography includes 122 references.
Keywords: amidation, nitriles, electrooxidation, Ritter-type reactions, photochemistry, C–N bond formation.

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1. Introduction

Aromatic and heteroaromatic amide-substituted compounds are widely used in organic synthesis, medicinal chemistry, materials chemistry, agrochemistry, and organic catalysis.[1–3] The amide functionality is one of the most important moieties that form the basis of proteins, peptides, and many other biologically significant compounds.[4] Currently, about 25% of commercial pharmaceuticals and 75% of drug candidates contain at least one amide or amino group.[5] In addition, agrochemicals with herbicidal and antifungal activity (for example, propanil, boscalid), and many drugs used in practice (paracetamol, niclosamide, imatinib) contain an arylamide moiety (Fig. 1).[6–8]

Aromatic amides are essential precursors for the synthesis of many nitrogen-containing heterocyclic and carbocyclic systems, and also act as raw materials for modern materials used in various industries.[9–12] The practically important properties of nitrogen-containing compounds with an amide group stimulate the search for new reactions leading to compounds of this class, especially under environmentally friendly, so-called 'green', synthesis conditions.[13]

The reaction discovered by John Ritter[14,15] in 1948 and named after him involves the reaction of nitriles with alkenes or tertiary alcohols to give amides under acidic conditions (Scheme 1).[16] Under such conditions, alcohols **1** and alkenes **2** first form active carbocationic intermediates **3** and **4**, which react with nitriles **5** to form key nitrilium ions **6** and **7** (their resonance structures are designated by numbers **8** and **9**). After adding water, the latter furnish intermediates **10** and **11**, which ultimately transform into the corresponding amides **12** and **13**.

In recent decades, not only alcohols and alkenes but also alternative substrates have been used in the Ritter reaction.

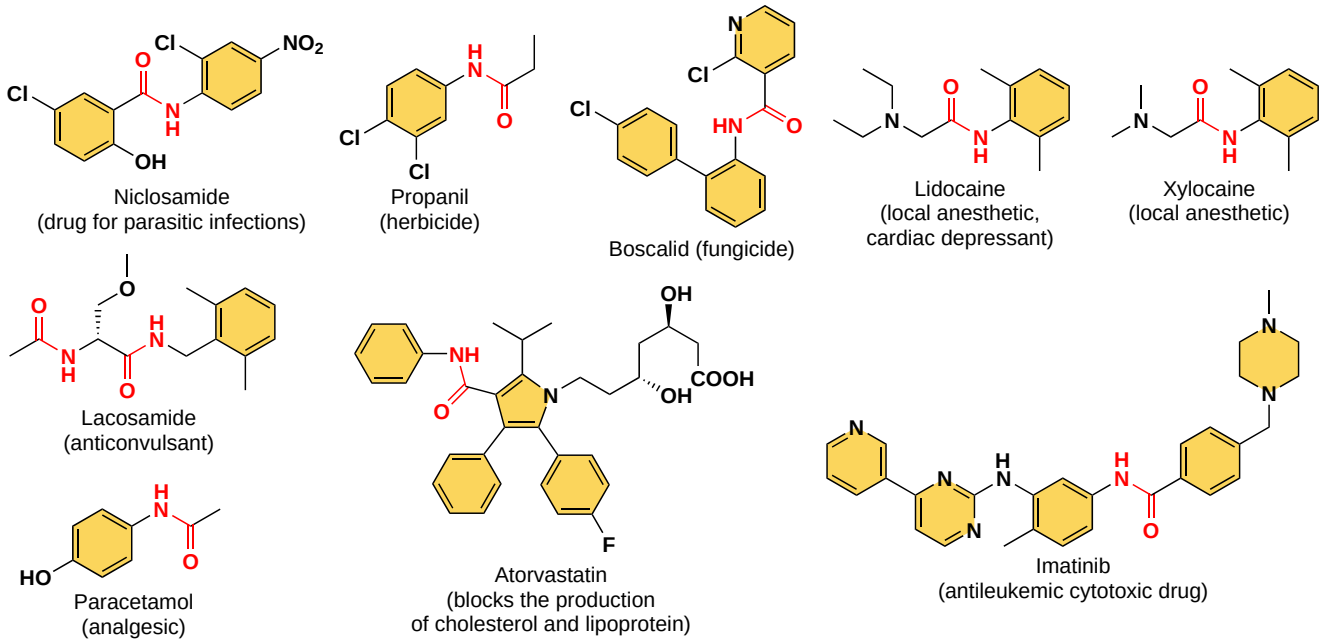
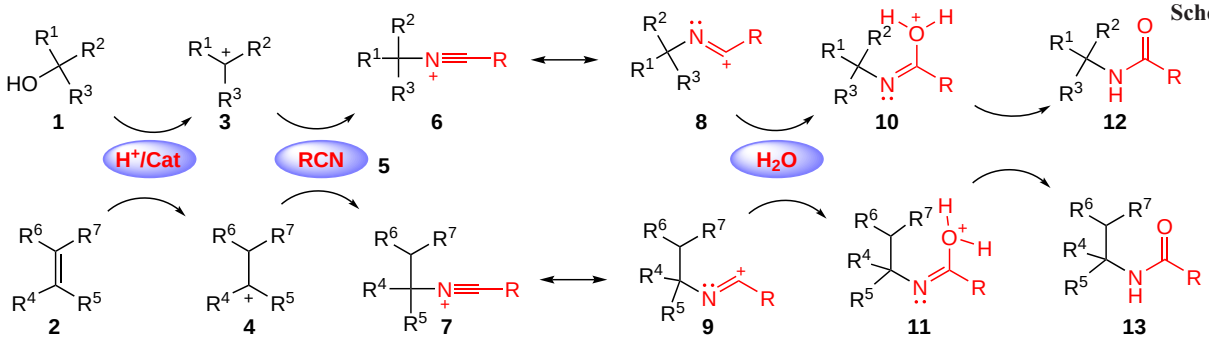


Figure 1. Amide-substituted agrochemicals and pharmaceuticals.

Scheme 1



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Thus, to date, variants of reactions proceeding according to the Ritter reaction type are known involving carboxylic acids,[17] haloalkynes,[18] aldehydes,[19] aryltriazenes,[20] aryldiazonium tetrafluoroborates,[21] cyclic diaryliodonium salts,[22] which open the way to various *N*-substituted amides, in particular *N*-benzylamides and *N*-arylamides. The Ritter reaction is used in the synthesis of biologically active molecules, namely antiallergic,[23] antibacterial,[24] anti-influenza[25] and antimicrobial drugs.[26] Reactions using nitriles as building blocks are one of the most attractive areas of organic synthesis, including electrochemical synthesis. Due to its good conductivity and relatively environmentally friendly properties, acetonitrile has become a sought-after reagent suitable for the production of nitrogenous compounds (*e.g.*, amides and nitrogen heterocycles) or other cyano derivatives.[27]

The preparation of practically significant *N*-aryl- and *N*-benzylamides using nitriles as amidating reagents is considered a relevant area of research, as evidenced by new works that have appeared in the last decade, in which these transformations are carried out both under the conditions of classical organic synthesis and using electrochemical and photoelectrochemical approaches.[28,29] Nevertheless, in the available literature on this topic (see, *e.g.*, Refs 30–35), there is no systematic review devoted to the use of Ritter-type reactions for the synthesis of *N*-arylamides, *N*-benzylamides and *N*-hetarylamides, especially taking into account the latest advances in the field of photochemical and electrochemical methods. The present review aims to fill this gap: it summarizes and systematizes both traditional and modern approaches to the synthesis of *N*-substituted amides using nitriles as amidating reagents. Particular attention is paid to the possibilities of photo- and electrochemistry in the context of sustainable, highly selective and functionally directed organic synthesis.

The review begins with a discussion of Ritter-type reactions that yield *N*-aryl- and *N*-benzylamides under classical or photochemical conditions (Section 2) and with the use of electrochemistry (Section 3). It then presents known amidation reactions of some heteroaromatic substrates with nitriles (Section 4), and concludes with a discussion of amidation methods involving three-component systems and amidation of *N*-(hydroxymethyl)saccharin (Section 5).

2. Amidation of aromatic compounds with nitriles

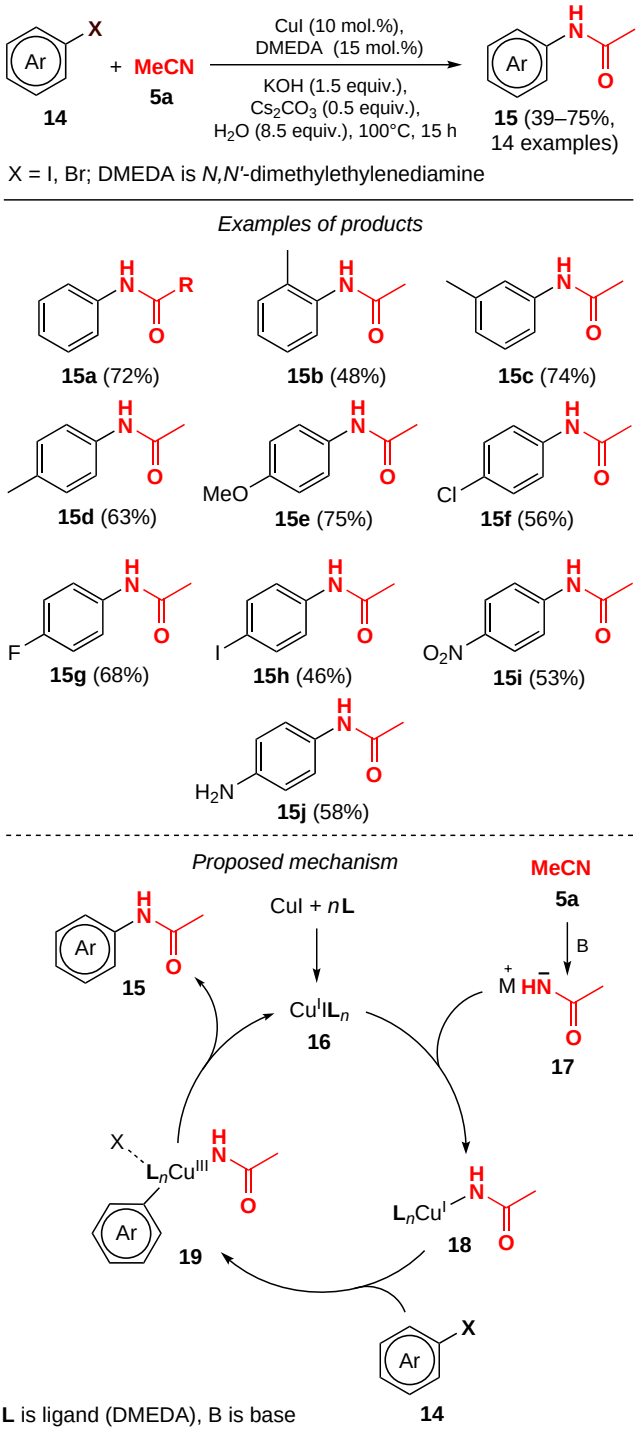
2.1. Amidation of aryl- and benzylhalides

In 2013, Yu and co-workers[36] described Cu-catalyzed amidation of aryl halides **14** with acetonitrile (**5a**). Various *N*-arylamides **15** were obtained in yields from 39% to 75% using monohalogenated benzenes as substrates (Scheme 2). This reaction runs for 15 h on heating to 100°C in the presence of a mixture of bases, KOH (1.5 equiv.) and Cs_2CO_3 (0.5 equiv.).

The plausible pathway of this catalytic process is shown in Scheme 2. The copper catalyst **16** reacts with amide salt **17**, formed in the first step during the hydrolysis of acetonitrile under basic conditions, to give intermediate complex **18**. Aryl halides **14** undergo an oxidative addition to complex **18** to afford intermediate **19**. Next, reductive elimination gives the desired products **15** and catalyst **16** is released to complete the catalytic cycle.

Using 1,2-dihalobenzenes (*e.g.*, iodobenzenes **20**) in this reaction, benzoxazoles **21** were isolated as reaction products rather than *N*-arylamides (Table 1).

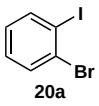
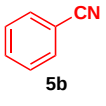
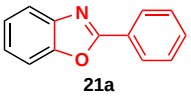
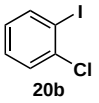
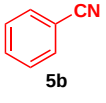
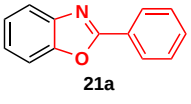
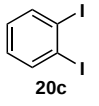
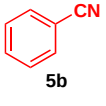
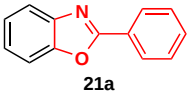
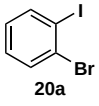
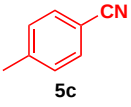
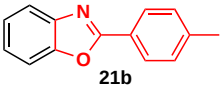
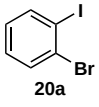
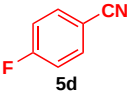
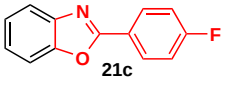
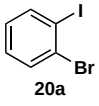
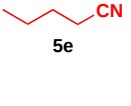
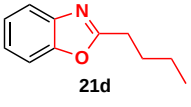
Scheme 2



A simple and efficient method for the synthesis of substituted *N*-benzylamides by coupling benzonitrile (**5b**) with benzyl halides **22** was proposed by Feng *et al.*[37] (Table 2). The reaction runs on heating to 80°C in a solvent-free fashion using $FeCl_2 \cdot 4H_2O$ as a catalyst. This method provides an access to various *N*-benzylbenzamides **23** in high yields of up to 96% in 2 h. Acetonitrile turned out to be an unsuitable reagent for this transformation, since it gives no amidation products.

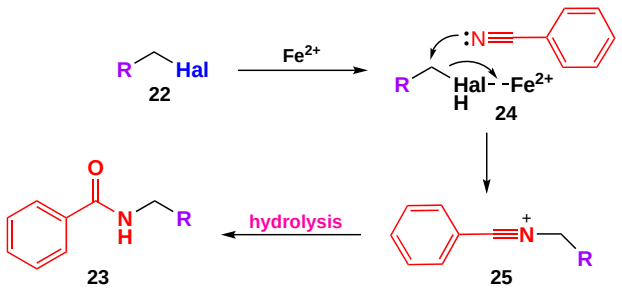
The proposed mechanism of the $FeCl_2 \cdot 4H_2O$-catalyzed coupling of compounds **5b** and **22** is shown in Scheme 3. From a mechanistic point of view, the catalytic efficiency of Fe^{2+} is related to its properties as a Lewis acid, which promotes the amidation process by facilitating the cleavage of the C–Hal

Table 1. Copper-catalyzed annulation of *o*-haloiodobenzenes with nitriles.[36]

Substrate	Nitrile	Product	Yield (%)
 20a	 5b	 21a	87
 20b	 5b	 21a	74
 20c	 5b	 21a	42
 20a	 5c	 21b	89
 20a	 5d	 21c	57
 20a	 5e	 21d	50

bond followed by nucleophilic substitution of the halogen atom in complex **24** with benzonitrile. In the final step, the hydrolysis of intermediate **25** gives *N*-benzylamide **23**.

Scheme 3

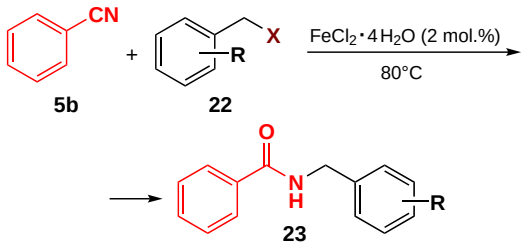


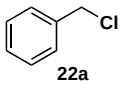
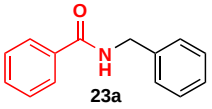
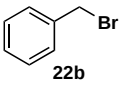
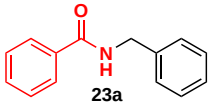
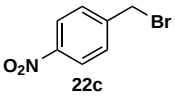
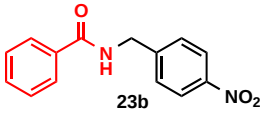
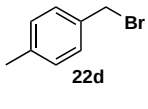
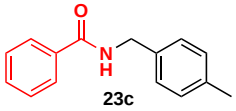
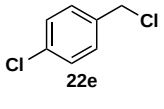
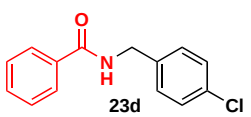
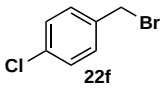
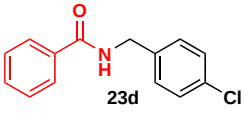
In 2018, Feng *et al.*[38] proposed a similar approach to *N*-benzylamides based on benzyl halides and nitriles using an low-cost and effective catalyst $Zn(ClO_4)_2 \cdot 6H_2O$. The reaction was carried out by heating to 80°C for 3–5 h in the absence of solvent to give the corresponding amides in high yields (up to 98%).

A year later, Perera and Aakeröy[39] reported a Ritter-type reaction of acetonitrile-D3 (**5f**) with bromodiphenylmethane **26**, which led to deuterated *N*-benzhydrylacetamide **27** in the presence of the **Cat1** catalyst (Scheme 4).

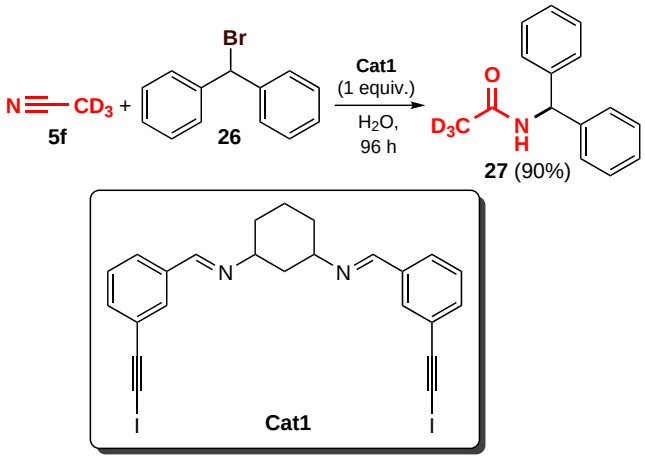
Deuteroacetonitrile acts both as a solvent and a nucleophile in this reaction. In the absence of a catalyst, the amidation product **27** is not formed even after >96 h. According to ^{1}H NMR data, the addition of 1 equiv. of **Cat1** provides 90% conversion of substrate **26** to product after 96 h, with no by-products or decomposition of the catalyst *per se* being observed. When 10% of the same catalyst is added to the

Table 2. Iron(II) chloride-catalyzed coupling of benzonitrile with benzyl halides.[37]



Substrate	Product	Yield (%)
 22a	 23a	96
 22b	 23a	96
 22c	 23b	88
 22d	 23c	94
 22e	 23d	92
 22f	 23d	92

Scheme 4



reaction mixture, the conversion of bromide **26** to amide **27** is 33%, and using 20% of the catalyst, the yield of the product increases to 63%.

2.2. Amidation of diaryliodonium salts

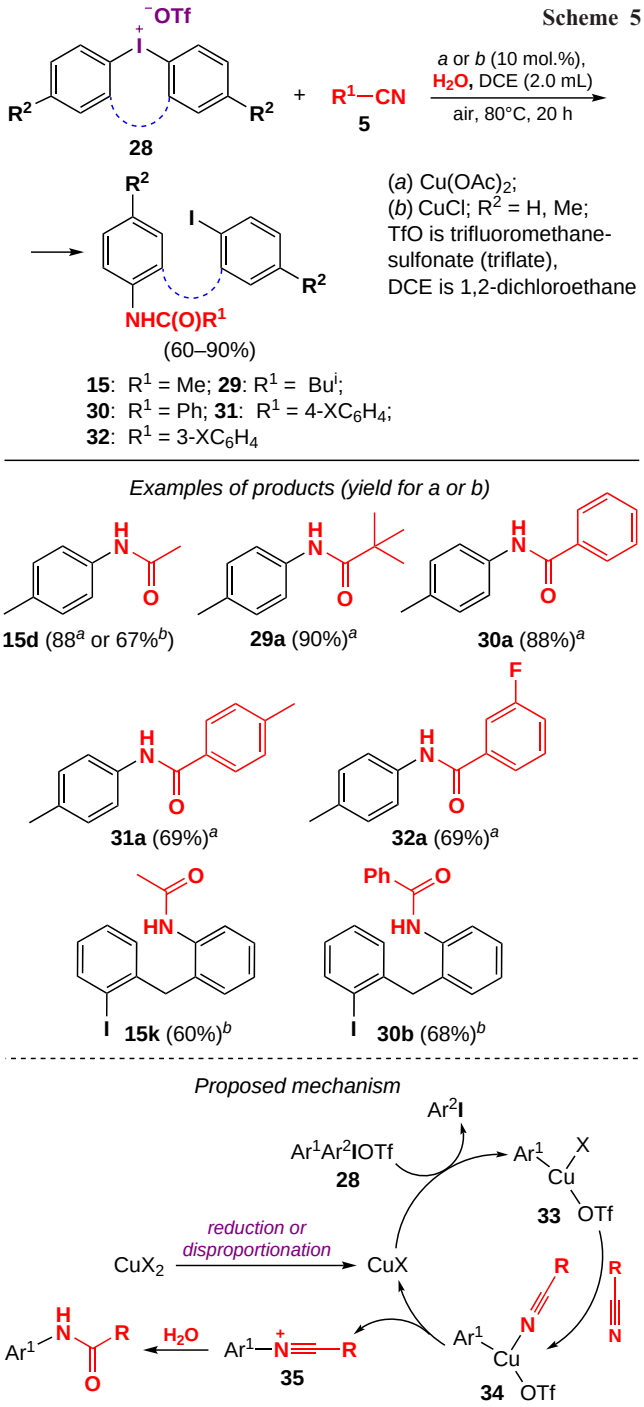
The synthesis of *N*-arylamides *via* copper-catalyzed amidation of diaryliodonium salts **28** with nitriles was proposed by Ji and co-workers[40] in 2021 (Scheme 5). Various substituted aromatic and aliphatic nitriles can be used in this reaction, with *N*-arylamides **15**, **29**–**32** being obtained in moderate to high yields (57–90%).

The plausible reaction pathway is shown in Scheme 5. Presumably, in the first step, the CuX_2 catalyst disproportionates or is reduced to its active form CuX. Then, diaryliodonium salts **28** in the presence of a CuI-based catalyst form a highly active electrophilic copper(III) intermediate **33**. The latter reacts with nitriles, and the reductive elimination of intermediate **34** produces aryl cationic species **35**, which results in the release of the active form of the CuI catalyst and its regeneration in the catalytic cycle. In the final step, cations **35** are hydrolyzed to give the desired reaction products, *N*-arylamides.

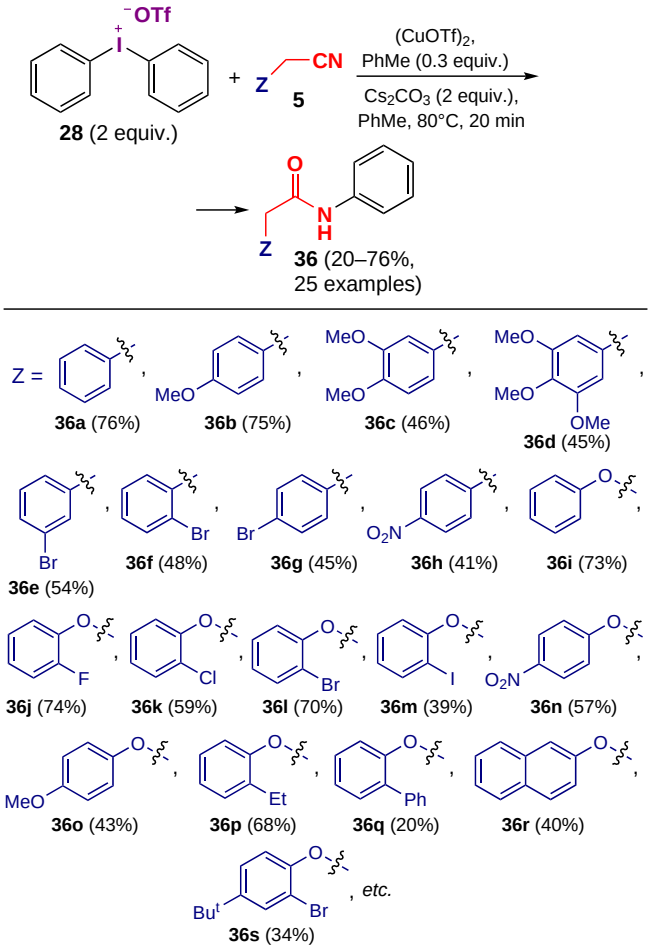
When acyclic diaryliodonium salts are used in a similar reaction, *N*-arylamides are formed, and the coupling of cyclic diaryliodonium salts with nitriles leads to diarylmethanamides through the iodonium ring-opening with retention of the iodine atom in one of the aryl moieties. The catalyst $Cu(OAc)_2$ turned out to be more suitable for the amidation of acyclic diaryliodonium salts, while the authors preferred CuCl for the formation of diarylmethanamides. A similar method for the preparation of diarylmethanamides (yields up to 82%) by CuCl-catalyzed reaction between cyclic diaryliodonium salts and nitriles was developed by Chen and co-workers.[22]

Another simple and practical synthetic approach to *N*-arylamides **36** using readily available benzyl nitriles and diaryliodonium salts **28** as electrophilic coupling partners was proposed in 2021 by Gillaizeau and co-workers[41] (Scheme 6). Benzyl nitriles with electron-donating and electron-withdrawing groups give the desired products in moderate and good yields (up to 76%) in this reaction. This method is characterized by high efficiency, the possibility of using a wide range of substrates and good tolerance to the functional groups therein.

Scheme 5



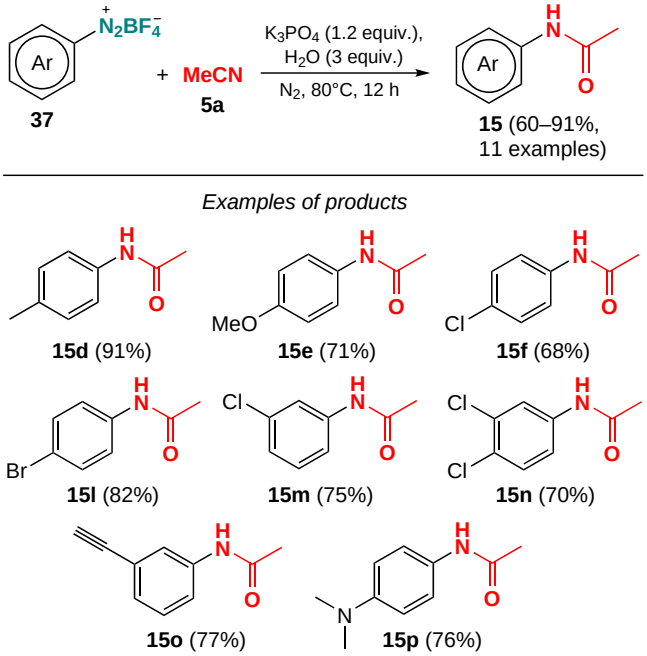
Scheme 6



2.3. Amidation of aryldiazonium tetrafluoroborates

In 2018, Tang and co-workers[42] proposed a direct synthesis of *N*-arylacetamides **15**, which involved coupling of aryldiazonium tetrafluoroborates **37** with acetonitrile (**5a**) without the use of metal catalysts. The reaction proceeded under nitrogen

Scheme 7



atmosphere in the presence of a stoichiometric amount of K_3PO_4 base at 80°C for 12 h (Scheme 7).

A number of *N*-arylacetamides **15** containing electron-donor or electron-withdrawing groups were obtained in high yields (up to 91%). The proposed approach allows avoiding the use of strong acids and air-sensitive reagents.

The authors believe that the amidation proceeds *via* the decomposition of aryldiazonium tetrafluoroborate to generate an aryl radical, as confirmed by experiments with a spin trap, (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO).[42,43] The resulting radical then undergoes nucleophilic attack by nitrile and water to give the amidation product. However, another mechanism of the process is also possible, which involves the direct reaction of nitriles with aryldiazonium cations without the involvement of free radicals.[44,45]

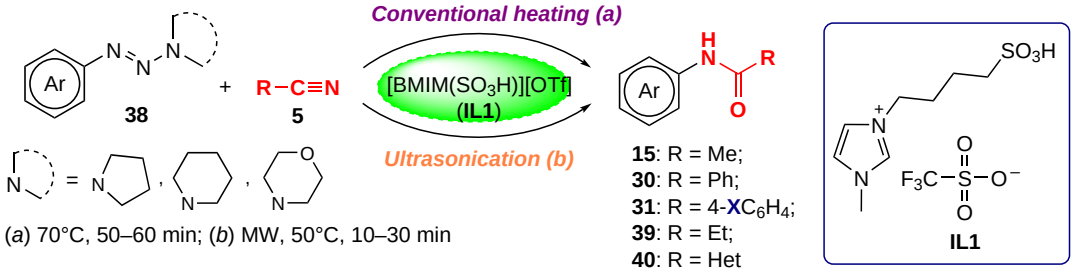
2.4. Amidation of aryltriazenes

In 2021, Sutar and Kalkhambkar[46] proposed to obtain *N*-arylamides under mild conditions by coupling aliphatic, aromatic or heterocyclic nitriles with aryltriazenes **38** using the Brønsted ionic liquid [BMIM(SO_3H)][OTf] (**IL1**) as a convenient promoter. The reaction was carried out in two ways (Scheme 8): on heating to 70°C for 1 h or under ultrasound (US) assistance at a lower temperature. The second method proved to be more efficient, providing the corresponding *N*-arylamides **15**, **30**, **31**, **39** and **40** in high yields (up to 92%), while upon heating, the yields of amidation products did not exceed 83%.

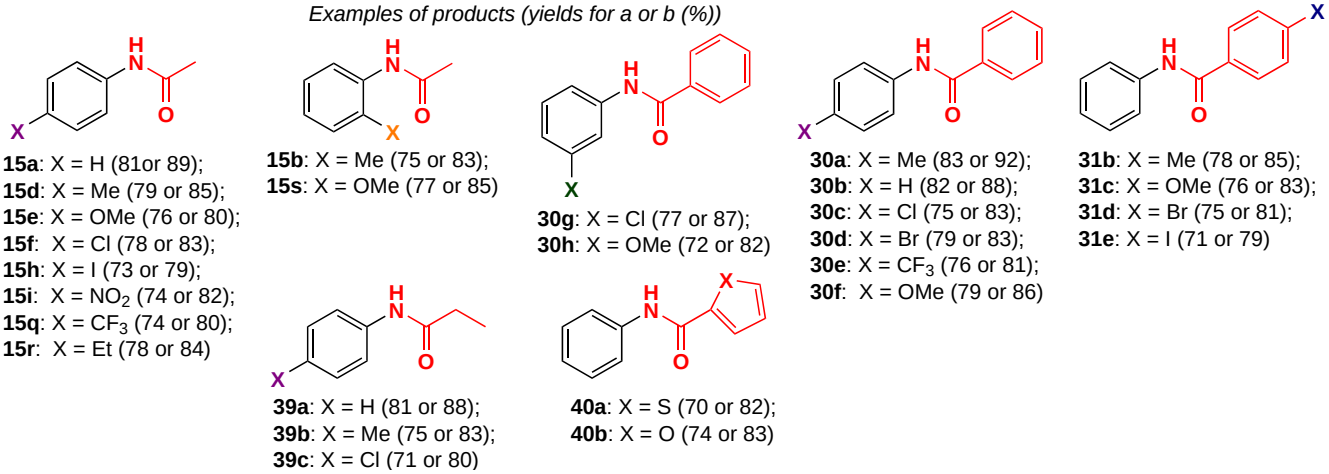
The probable mechanism of the process, presented in Scheme 9 on the example of pyrrolidine substrates **38**, is as follows: in the first step, protonation of the aryltriazene at the pyrrolidine nitrogen atom occurs to give ammonium salt **41**. Further elimination of pyrrolidine and molecular nitrogen leads to aryl cation **42**, which in turn undergoes nucleophilic attack by the nitrile to afford cation **43**. Subsequent hydrolysis and deprotonation of this cation yield the desired products, *N*-arylamides.

A method for the synthesis of *N*-arylacetamides **15** from aryltriazenes **38** and acetonitrile (**5a**) at room temperature was developed in 2021 by Liu and co-workers.[47] The catalyst-free reaction is carried out using the protic Brønsted ionic liquid **IL2** as a potential promoter (Scheme 10). Triazenes with electron-donating and electron-withdrawing groups in the benzene ring proved to be suitable substrates for this reaction, affording the

Scheme 8



Examples of products (yields for a or b (%))



15a: X = H (81or 89);
15d: X = Me (79 or 85);
15e: X = OMe (76 or 80);
15f: X = Cl (78 or 83);
15h: X = I (73 or 79);
15i: X = NO_2 (74 or 82);
15q: X = CF_3 (74 or 80);
15r: X = Et (78 or 84)

15b: X = Me (75 or 83);
15s: X = OMe (77 or 85)

30g: X = Cl (77 or 87);
30h: X = OMe (72 or 82)

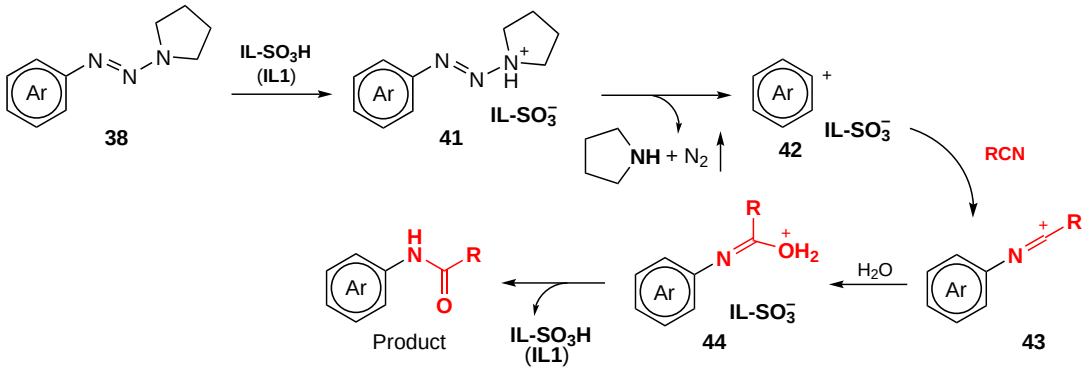
30a: X = Me (83 or 92);
30b: X = H (82 or 88);
30c: X = Cl (75 or 83);
30d: X = Br (79 or 83);
30e: X = CF_3 (76 or 81);
30f: X = OMe (79 or 86)

31b: X = Me (78 or 85);
31c: X = OMe (76 or 83);
31d: X = Br (75 or 81);
31e: X = I (71 or 79)

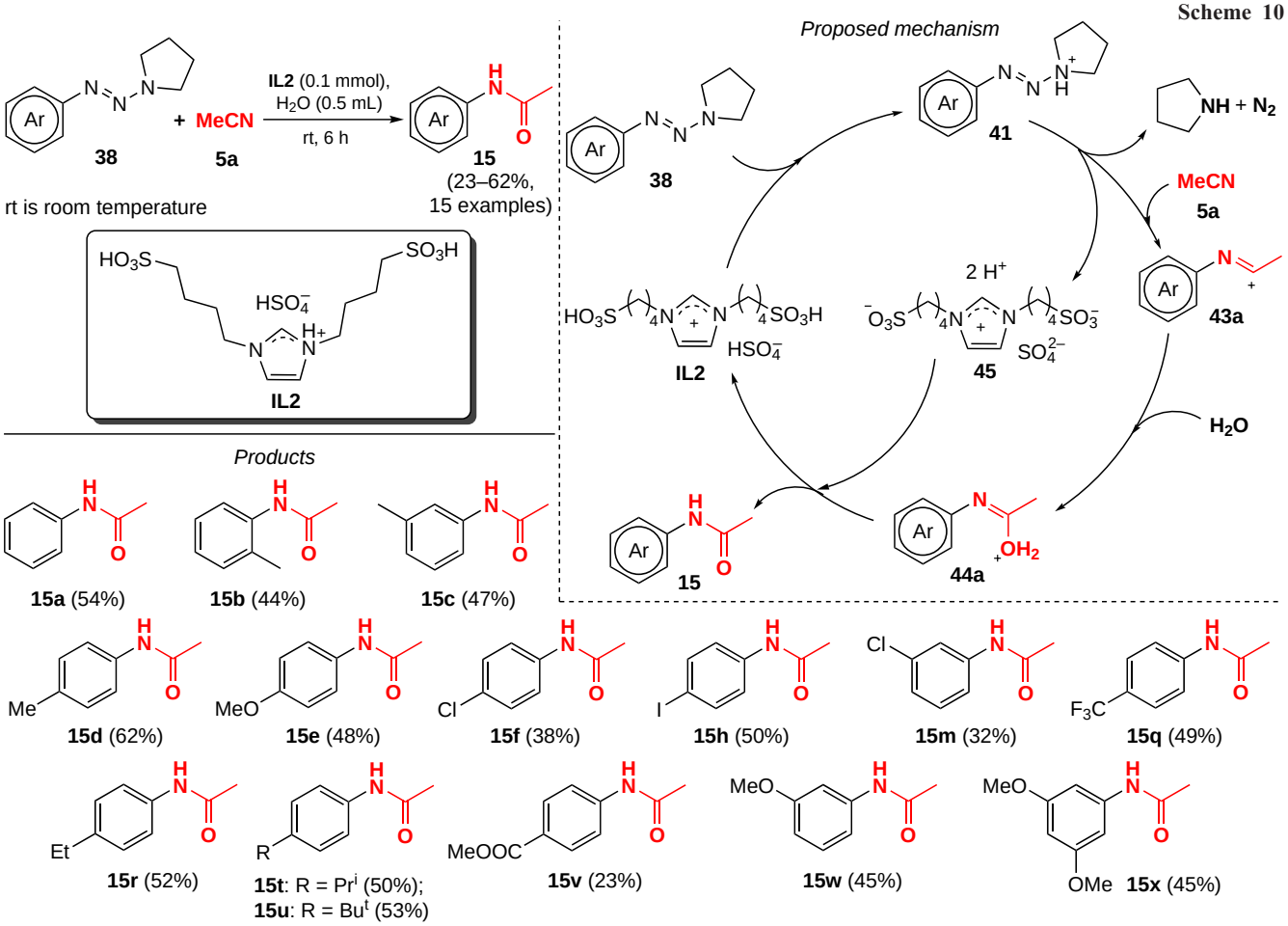
39a: X = H (81 or 88);
39b: X = Me (75 or 83);
39c: X = Cl (71 or 80)

40a: X = S (70 or 82);
40b: X = O (74 or 83)

Scheme 9



Scheme 10



corresponding *N*-arylacetamides **15** in moderate yields (from 23% to 62%).

The plausible mechanism of the process is shown in Scheme 10. First, aryltriazene **38** is protonated to form ammonium salt **41**. Next, the pyrrolidine molecule and molecular nitrogen (N_2) are eliminated, and subsequent nucleophilic attack of nitrile **5a** leads to cation **43a**, which undergoes hydrolysis to generate intermediate **44a**. In the final step, deprotonation and regeneration of **IL1** from dication **45** yield product **15**.

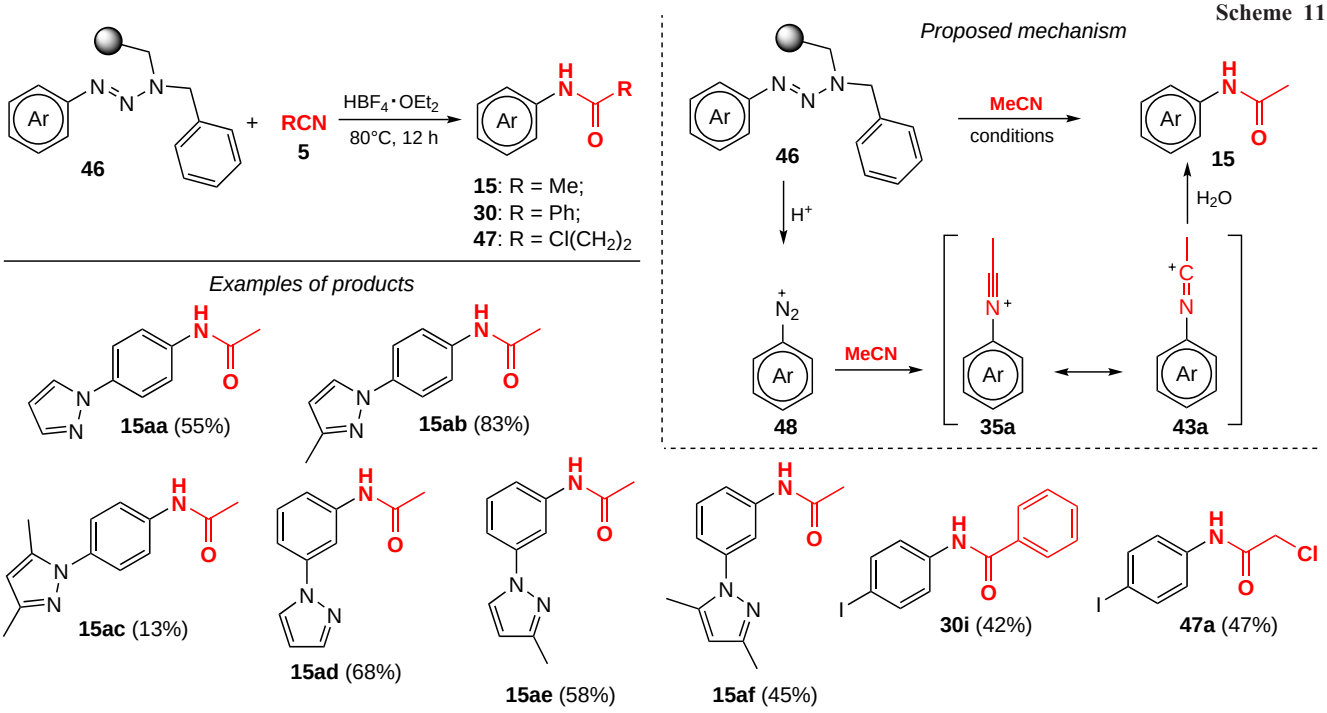
In 2019, Bräse and co-workers[20] obtained *N*-arylacetamides **15** (including pyrazolylbenzene derivatives), **30** and **47** from *ortho*-, *meta*- and *para*-substituted aryltriazenes **46** using acetonitrile as a solvent and reagent (Scheme 11; the black ball denotes a polymer support).

The corresponding arylamides are formed by heating the reagents to 80°C for 12 h (yields up to 83%) *via* cleavage of immobilized aryltriazenes in the presence of alkyl or aryl nitriles. Cations **48**, **35a** and **43a** are the key intermediates of this transformation (see Scheme 11).

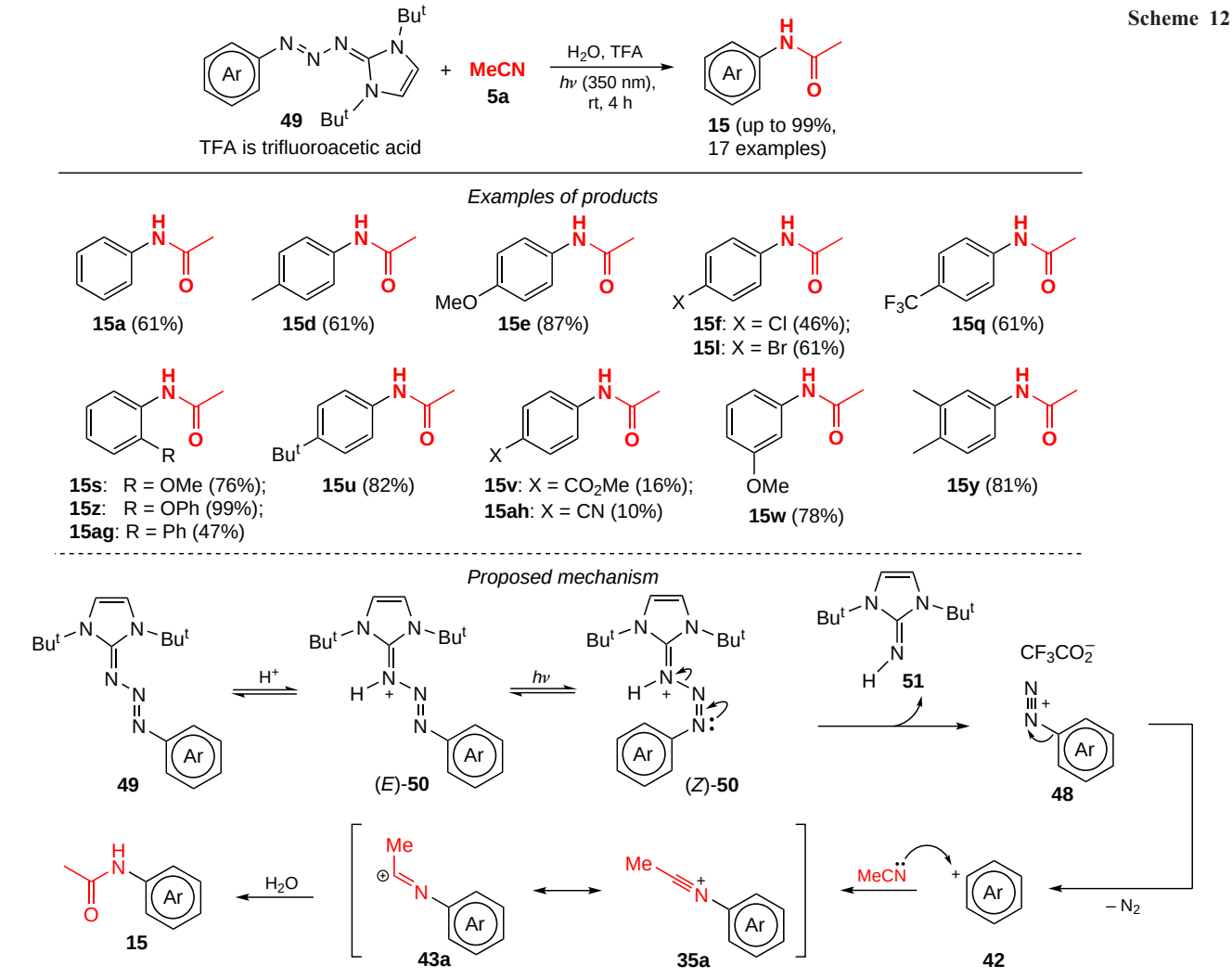
In 2020, Bugarin and co-workers[48] developed a photochemical approach to the synthesis of anilides by coupling π-conjugated aryltriazenes **49** with acetonitrile (Scheme 12). The reaction products are obtained in moderate to high yields (up to 99%) in the absence of metal catalysts under UV irradiation within 4 h.

The proposed reaction pathway is shown in Scheme 12. In the initial step, triazene **49** is protonated with trifluoroacetic acid, then the protonated form of triazene undergoes photoisomerization from the *E*-isomer (*E*)-**50** to the *Z*-form. Then, the protonated triazene (*Z*)-**50** gives guanidine **51** and an unstable aryldiazonium salt **48**, which decomposes to give the aryl cation **42** and molecular nitrogen. The subsequent

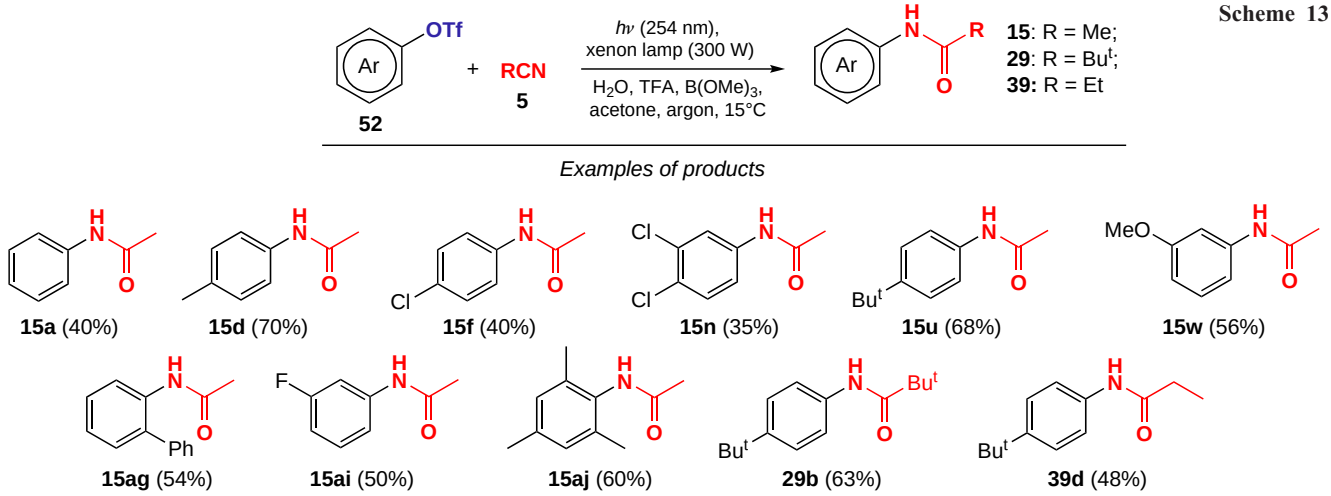
Scheme 11



Scheme 12



Scheme 13



nucleophilic attack of acetonitrile on carbocation **42** affords the intermediate nitrilium ion **35a**, which tautomerizes to the iminium cation **43a**. Subsequent hydrolysis of this cation leads to anilide **15**.

2.5. Amidation of aryl triflates

In 2019, Li and co-workers[49] hypothesized that aryl triflates **52** can react with nitriles under the action of light in the presence of water to construct an amide bond (Scheme 13). The corresponding *N*-arylamides are obtained in moderate to good yields (35–70%). The reaction was monitored by gas chromatography with mass spectrometry (GC-MS). This reaction serves as an intermediate step for the further conversion of *N*-arylamides to anilines during alkaline hydrolysis.

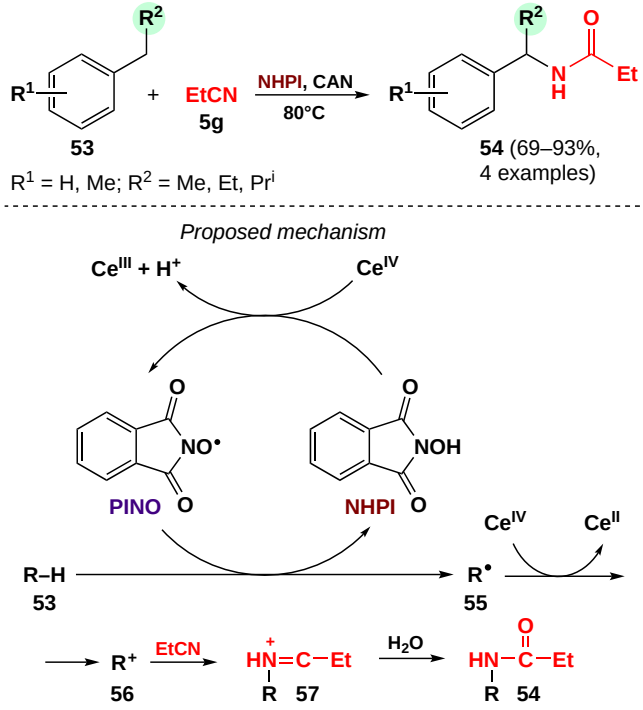
Aliphatic nitriles (MeCN, EtCN, Bu^{t}CN) were found to be suitable reagents for this process, while the use of aromatic nitriles did not give amidation products under the above conditions. This is probably due to the fact that aromatic nitriles contain chromophore groups capable of absorbing energy, which, in turn, negatively affects the photoinitiation of the desired reaction.

2.6. Amidation of alkylbenzenes

In 2002, Ishii and co-workers[50] first reported a Ritter-type reaction of alkylbenzenes **53** with propionitrile (**5g**) using *N*-hydroxyphthalimide (NHPI) as the catalyst in combination with cerium(IV) ammonium nitrate (CAN) as an oxidizing agent. As a result, *N*-benzylpropionamides **54** were obtained from toluene derivatives with good selectivity in 69–93% yield (Scheme 14).

This reaction is presumably initiated by the reaction of NHPI with CAN to form phthalimide *N*-oxyl (PINO), which is considered a key intermediate for the generation of alkyl radicals (see Scheme 14).[51,52] The PINO radical abstracts a hydrogen atom from the substrate to generate a benzyl radical **55**, which then undergoes single-electron oxidation in the presence of a cerium(IV) compound to form carbocation **56**. The latter reacts with propionitrile to afford intermediate **57**. Further nucleophilic attack by water contained in the solvent on intermediate **57** yields *N*-benzylpropionamide **54**. If the reaction is carried out in an oxygen atmosphere, benzyl radicals **55** will react with O_2 rather than with Ce^{IV}, giving rise to aromatic aldehydes.

Scheme 14

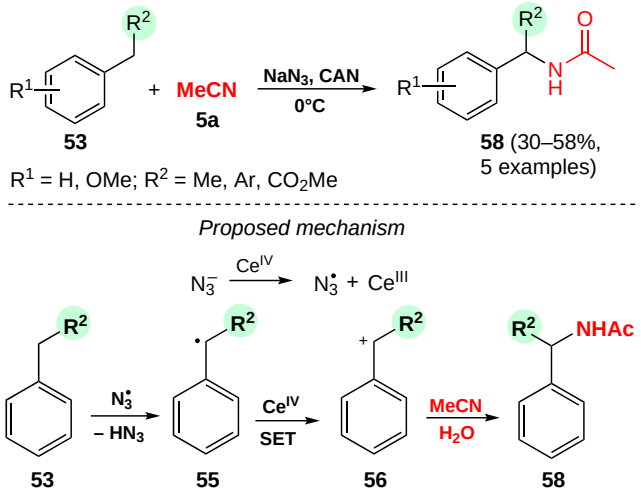


A similar method for the oxidative amidation of alkylbenzenes **53** with acetonitrile (**5a**) mediated by sodium azide NaN_3 was developed by Mohanan and co-workers[53] (Scheme 15). Most likely, the azide ion is first oxidized to an azide radical, which can activate the C–H bond by cleaving it to generate the benzyl radical **55**. Further single-electron oxidation of the radical **55** affords cation **56**. Subsequent reaction of the benzyl carbocation **56** with acetonitrile in the presence of water yields the corresponding *N*-benzylacetamides **58** (see Scheme 15).

This approach is characterized by milder conditions compared to reactions carried out by Ishii and co-workers[50,51] and a broader range of substrates. However, *N*-benzylacetamides **58** are obtained in moderate yields (30–58%) due to the inevitable formation of benzaldehyde as a by-product.

In 2025, Huang, Xiao and co-workers[54] developed a photocatalytic strategy for the C–H amination of alkylarenes **53** with acetonitrile (Scheme 16). Under blue light irradiation and using CeI_3 as a catalyst, bromination with tribromoacetic acid at the benzyl C(sp^3)–H bond first occurs. The resulting benzyl

Scheme 15



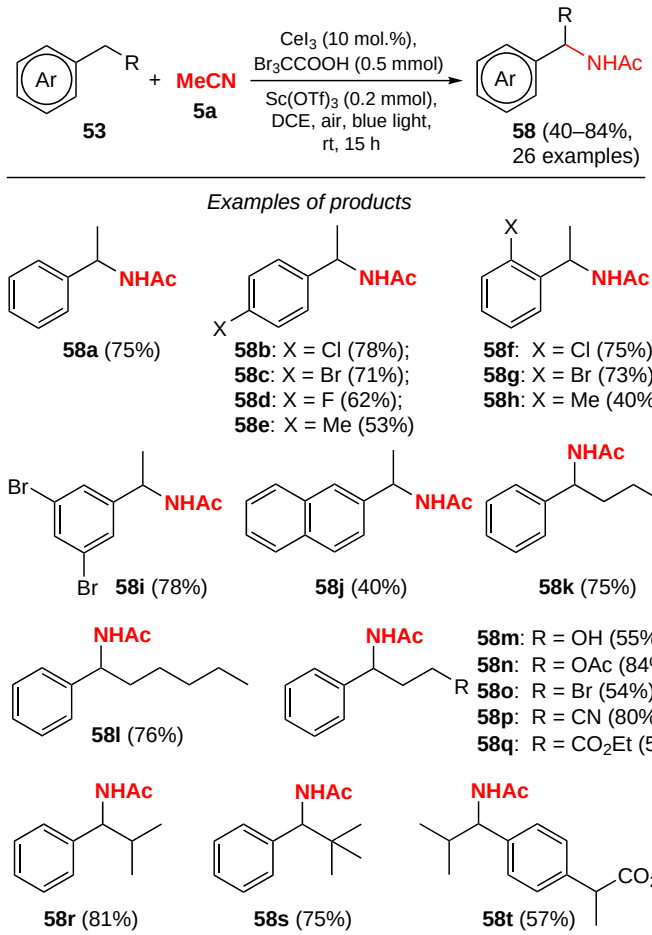
bromide then reacts with the nitrile to give the corresponding *N*-benzylamides in good yields (up to 84%).

In the initial step of the process, the active form of the catalyst [Ce^{III}]* is generated under photoinduction conditions. Then, the interaction of [Ce^{III}]* with tribromoacetic acid initiates two redox transformations: the reduction of Br_3CCOOH to the corresponding anion-radical $[Br_3CCOOH]^-$ and the oxidation of [Ce^{III}]* to [Ce^{IV}]. The unstable intermediate $[Br_3CCOOH]$ undergoes decomposition to the radical $^{\bullet}CBr_2COOH$ and the bromide anion (Br^-). Then, the proton abstraction from alkylbenzene **53** takes place involving the unstable radical $^{\bullet}CBr_2COOH$ to give $CHBr_2COOH$ and benzyl radical **55**. The reaction of the latter with [Ce^{IV}]–Br leads to intermediate **59** and the regeneration of cerium(III) in the catalytic cycle. In the presence of Lewis acid $Sc(OTf)_3$, intermediate **59** undergoes heterolysis to carbocation **56**, which reacts with MeCN. The resulting nitrilium cation **60a** is then hydrolyzed to the final product, *N*-benzylamide **58**.

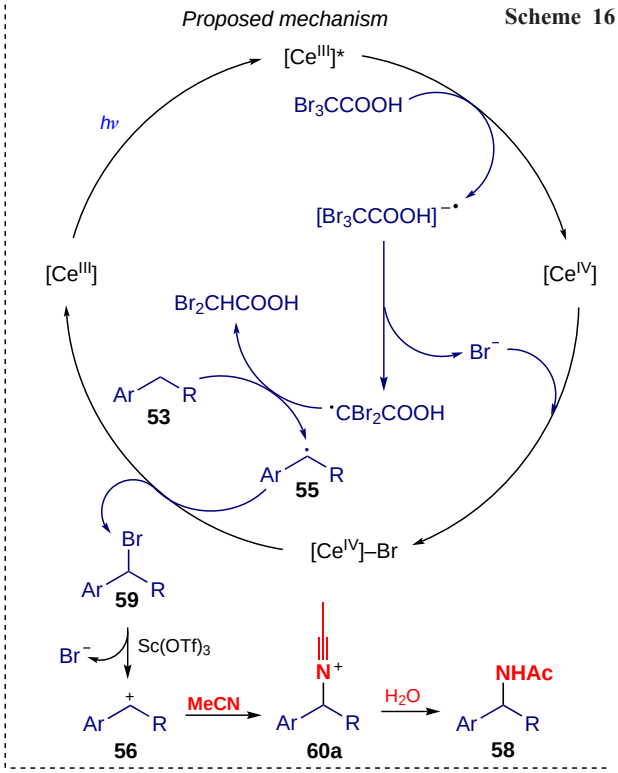
König and co-workers[55] proposed a method for the amidation of alkylbenzenes with acetonitrile under photochemical conditions in the presence of the iodonium complex iodine(III)–BF_3, obtained from iodobenzene, a carboxylate ligand, the oxidizing agent (Selectfluor) and Lewis acid (BF_3). Such a catalytic system allows the isolation of *N*-benzylamide derivatives in a yield of up to 91% without the use of a metal catalyst or a photocatalyst.

Doyle and co-workers[56] developed a photocatalytic method for the selective amidation of primary and secondary benzyl C(sp^3)–H bonds with nitriles and water using sulfonyloxyamide as a hydrogen atom transfer (HAT) reagent in the presence of the organic photocatalyst **Cat2** (Scheme 17).

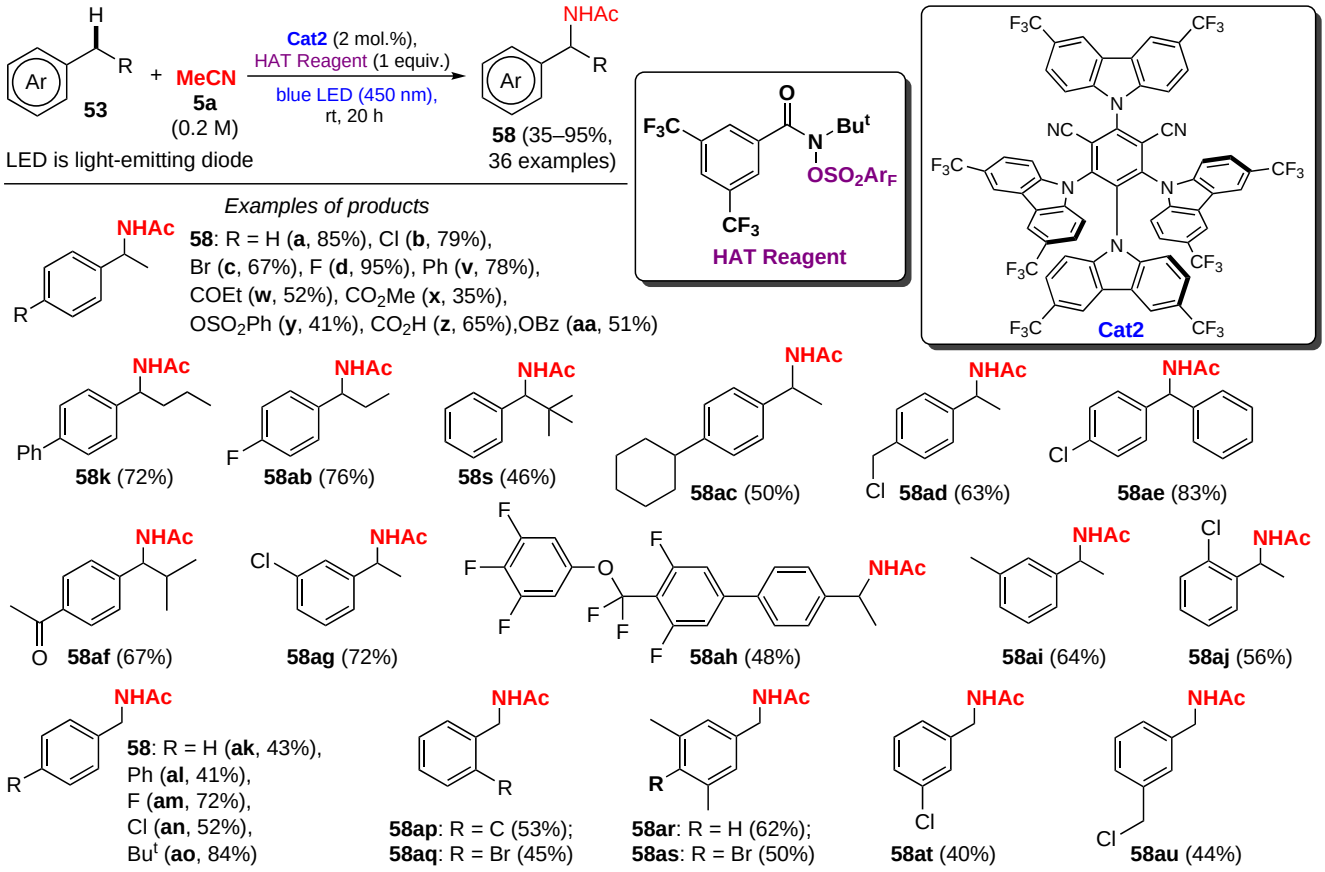
The amidation is carried out in several steps. At the initial step, homolytic cleavage of the N–O bond in the sulfonyloxyamide molecule occurs with the generation of an amidyl radical, which promotes the formation of a benzyl radical. When irradiated, the photocatalyst **Cat2** undergoes conversion to the oxidized form, which reacts with the benzyl radical and gives a carbocation. The latter reacts with the nitrile to furnish the resulting *N*-benzylamides. The reduced form of the photocatalyst mediates the single-electron homolytic cleavage of the N–O bond in the sulfonyloxyamide molecule and is regenerated in the catalytic cycle. Other nucleophiles,



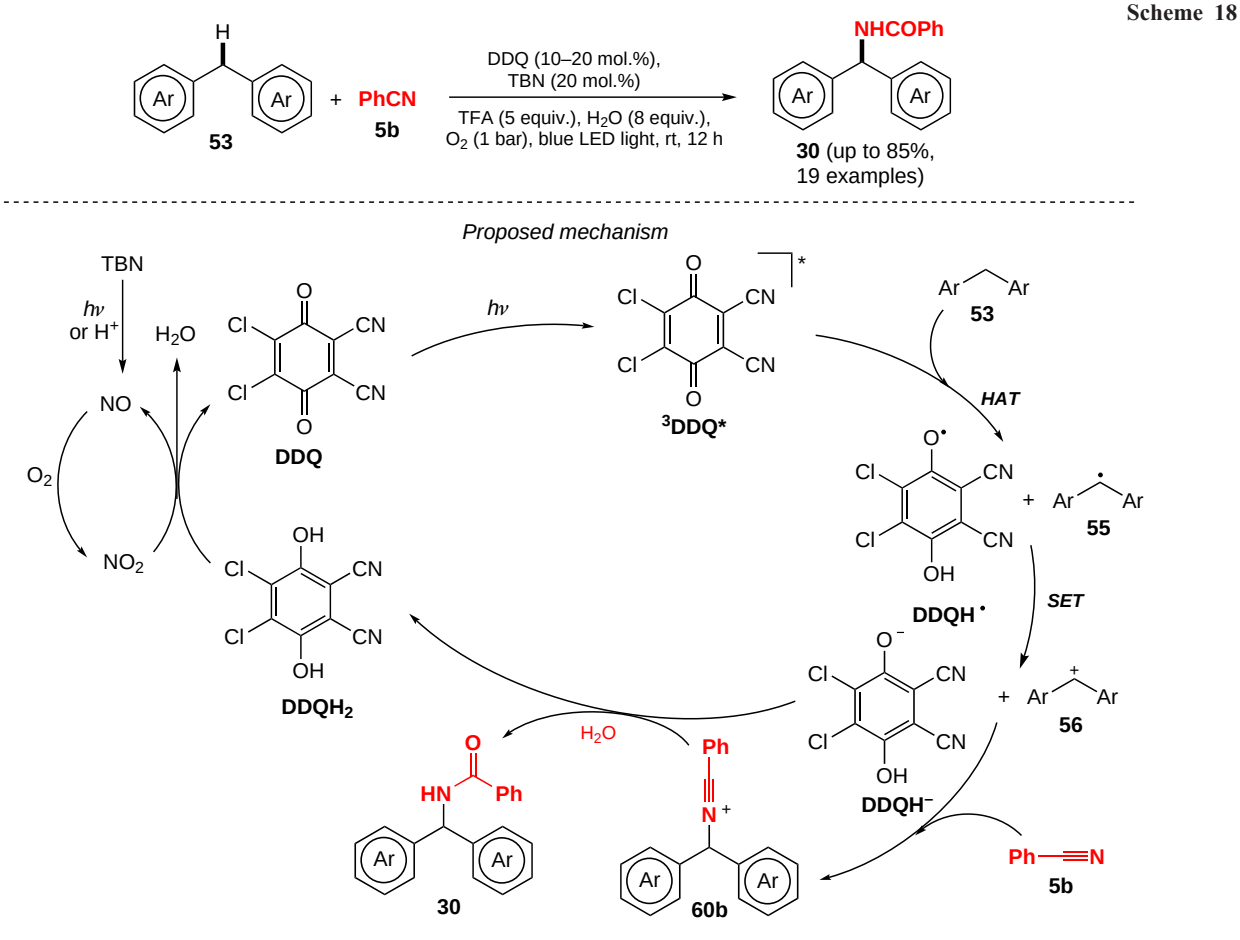
Scheme 16



Scheme 17



Scheme 18



such as amides, sulfonamides, carbamates and azoles, can also undergo this reaction.

Li *et al.*[57] developed a method for photocatalytic Ritter-type amidation of non-activated C(sp^3)–H bonds in diarylmethanes without using metal catalysts and in the presence of molecular oxygen as a 'green' oxidizing agent (Scheme 18). Using 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) and *tert*-butyl nitrite (TBN) cocatalysts, amides were obtained in moderate to excellent yields (up to 86%) under mild conditions.

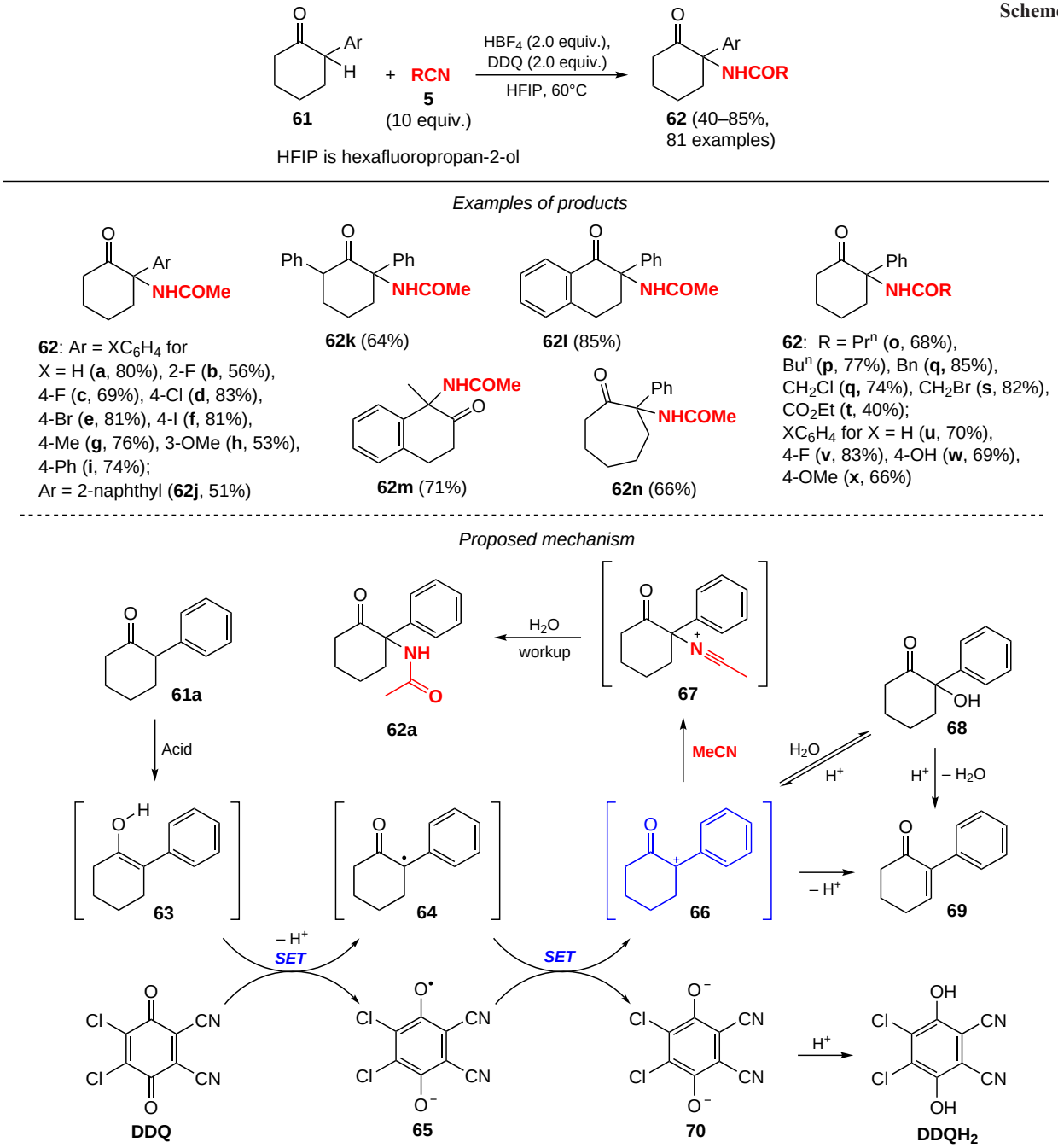
Most likely, the first step involves photolysis or acidolysis of TBN with the release of NO, which can be readily oxidized by oxygen (O_2) to NO_2 (see Scheme 18). Under visible light irradiation, DDQ is excited to the triplet state (^{3}DDQ*). Hydrogen atom transfer between substrate **53** and ^{3}DDQ* generates DDQH$^{\bullet}$ and an alkyl radical **55**, which undergoes subsequent electron transfer to afford the diphenylmethylium cation **56** and $DDQH^-$. The carbocation **56** reacts with benzonitrile (**5b**) to form the nitrilium ion **60b**, which is hydrolyzed to the amide **30**. At the same time, the $DDQH^-$ anion is protonated to the corresponding hydroquinone ($DDQH_2$), which can be reoxidized to DDQ with NO_2.

Zhang *et al.*[58] proposed oxidative amidation of benzyl C(sp^3)–H bonds with nitriles using $Mn(OAc)_3$ as a catalyst and a Lewis acid. Primary, secondary, and tertiary alkyl, alkenyl, benzyl and aryl nitriles can be used in this reaction. *N*-Benzylamides are obtained in yields from 20 to 96% by heating to 90°C under nitrogen for 12 h in the presence of DDQ as an oxidizing agent and trifluoroacetic acid.

2.7. Amidation of arylketones

A large series of *N*-(2-oxo-1-arylcyclohexyl)amides **62** were synthesized *via* oxidative coupling of 2-arylcyclohexan-1-ones **61** with nitriles (Scheme 19).[59] The proposed approach features

Scheme 19



the use of 10 equiv. of nitrile, a wide range of substrates, and functional group tolerance. The reaction runs for 1.5 h in the presence of tetrafluoroboric acid and DDQ oxidizing agent to give the amidation products in yields of up to 85%.

A plausible reaction pathway is shown in Scheme 19. Under acidic conditions, substrate **61a** is enolized to give intermediate **63**, which, when treated with DDQ, undergoes a single-electron oxidation to radical **64**, and DDQ is converted into the reduced radical form **65**. In the next step, carbocation **66** is formed through a second sequential step of a single-electron oxidation of radical **64** by intermediate **65**. In the next step, carbocation **66** is formed through the second successive step of a single-electron oxidation of radical **64** by intermediate **65**. Then the process follows the classical route: carbocation **66** undergoes nucleophilic attack by acetonitrile to generate nitrilium cation **67**, which is hydrolyzed to the desired amidation product **62a**. If carbocation **66** has no time to interact with acetonitrile, then alcohol **68** is formed as a result of a side nucleophilic attack by water.

The second by-product is enone **69**, which is formed by deprotonation of carbocation **66** or by elimination of water from **68**. Enone **69** is obtained only in trace amounts, while alcohol **68** is generated in the initial reaction step and is completely converted to the desired amidation product **62a**. It should be noted that SET from radical **64** is accompanied by the formation of dianion **70**, which, upon acidification, yields the corresponding hydroquinone.

In 2023, the same research group[60] reported the synthesis of 3-acyl-3-pyrrolin-2-ones by coupling α-aryl ketones with substituted propionitriles using a similar oxidation system based on DDQ and HBF_4. The process was carried out in two steps. In the initial step, the starting substrates were reacted to afford an intermediate under Ritter-type oxidative amidation conditions. The following intramolecular aldol condensation yielded the desired 3-acyl-3-pyrrolin-2-ones.

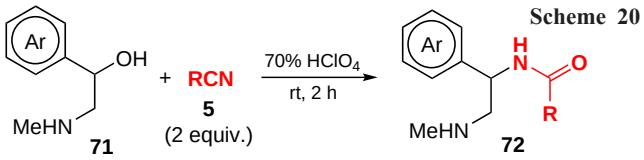
2.8. Amidation of benzyl alcohol derivatives

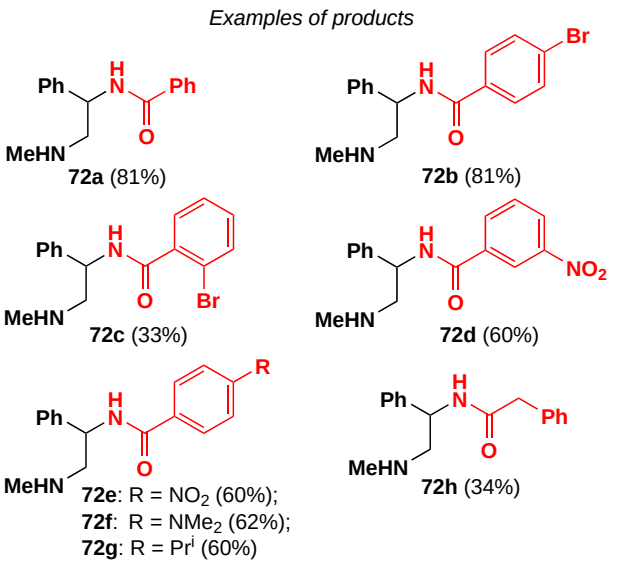
Sosnovskikh and co-workers[61] proposed the synthetic approach to 2-aminoethyl-1-benzamides from arylethanolamines **71**. The reaction runs in the presence of excess perchloric acid and 2 equiv. of nitrile **5** at room temperature for 2 h (Scheme 20). Benzonitriles containing electron-withdrawing substituents (such as a nitro group or a halogen atom) in the *para*- and *meta*-positions readily reacted with substrates **71** at room temperature, affording products **72** in 60–81% yields. Benzonitriles with electron-donating groups (OMe, Alk) promoted competitive Friedel–Crafts alkylation to afford mixtures of products. 2-Aminoethyl-1-benzamides **72** were then used in a cyclization reaction to give imidazolines.

In 2023, Marques and co-workers[62] reported the synthesis of *N*-benzylamides using a heterogeneous MWCNT-CSP (multi-walled carbon nanotubes modified with *p*-toluenesulfonic acid) catalyst (Scheme 21). Heating benzyl alcohols **73** and nitriles to 100°C for 24–120 h yielded *N*-benzylamides **23**, **54**, **58** and **74** in moderate to good yields (up to 78%). According to the process pathway shown in Scheme 21, the authors suggest that substrate **73** is first protonated by acid, releasing a water molecule, followed by the formation of carbocation **56**, which is captured by the nitrile and affords nitrilium cation **60**. Subsequent hydrolysis of cation **60** leads to amidation products.

Wang and co-workers[63] developed a simple and efficient method for the synthesis of *N*-benzylamides and other amides by the reaction of secondary alcohols with nitriles mediated by

Scheme 20





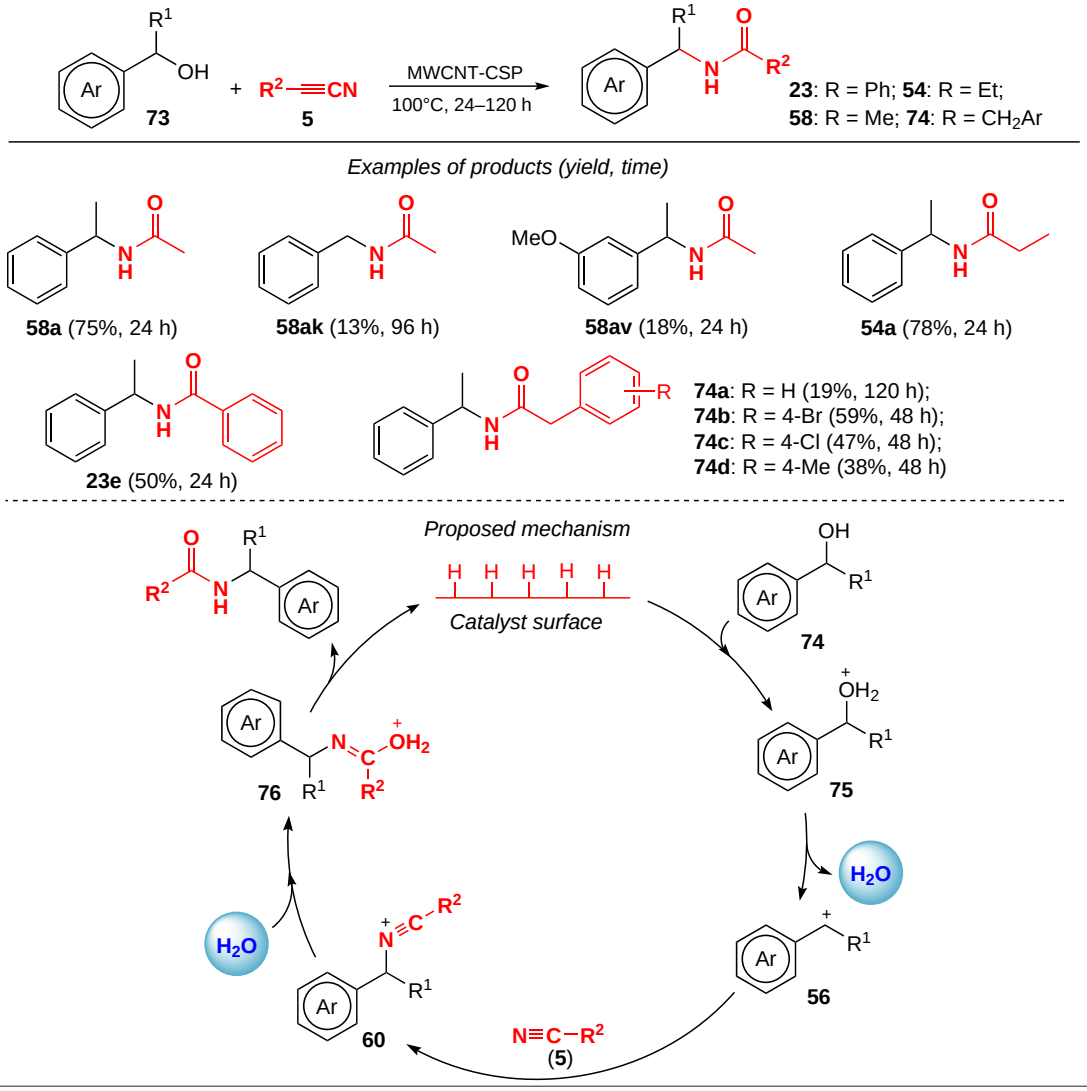
$Fe(NO_3)_3 \cdot 9H_2O$ (Scheme 22, reaction (*1*)). Substrates with electron-donating groups gave the corresponding amides in good or high yields (up to 97%), while in the case of electron-withdrawing groups the yields of the products were significantly lower (up to 61%).

Ueno *et al.*[64] carried out an environmentally friendly reaction of benzyl alcohols with nitriles using commercially available bismuth salt $BiBr_3$ as a catalyst (see Scheme 22, reaction (*2*)). The amidation products were formed in the presence of the bismuth salt at 120°C within 48 h. The yield of the corresponding desired *N*-benzylamides was improved by adding a small amount of water, and no deactivation of the bismuth salts was observed under these conditions.

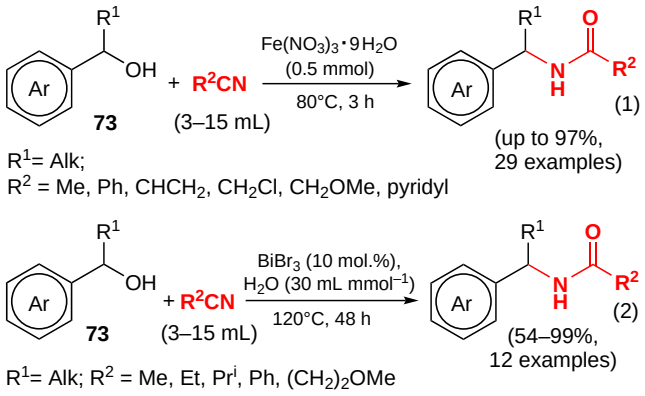
Chen and co-workers[65] developed a method for the photocatalytic dehydration of benzyl alcohols suitable for their efficient direct conversion to the corresponding *N*-benzylamides using 2,4,6-triphenylpyrylium tetrafluoroborate (**Cat3**) as a photocatalyst (Scheme 23). The reaction of benzyl alcohols **73** and nitriles afforded the corresponding amides after 16 h of blue light irradiation in a nitrogen atmosphere. Benzyl alcohols containing electron-donating groups react quite readily (product yields range from 50% to 63%), while their analogues with electron-withdrawing groups give *N*-benzylamides in lower yields. The method can be applied to various alkyl nitriles, but with lengthening of the alkyl chain the activity of the nitriles decreases, which leads to a decrease in the yields of amidation products.

The probable mechanism of this process using the substituents R^1=H, R^2=Me as an example is shown in Scheme 23. At the initial step, under irradiation conditions, the photocatalyst (its cation is designated as TP) passes into an excited state to form a cation-radical. The interaction of benzyl alcohol with the active form of the photocatalyst and further transformation steps lead to the intermediate cationic form **75** with the release of radical **76**. Then, cation **75** is dehydrated to the corresponding benzyl carbocation **56**, which further reacts with acetonitrile and water to give successively cation **77**, enol **78** and the amidation product.

Scheme 21



Scheme 22



2.9. Amidation of arylacetic acid derivatives

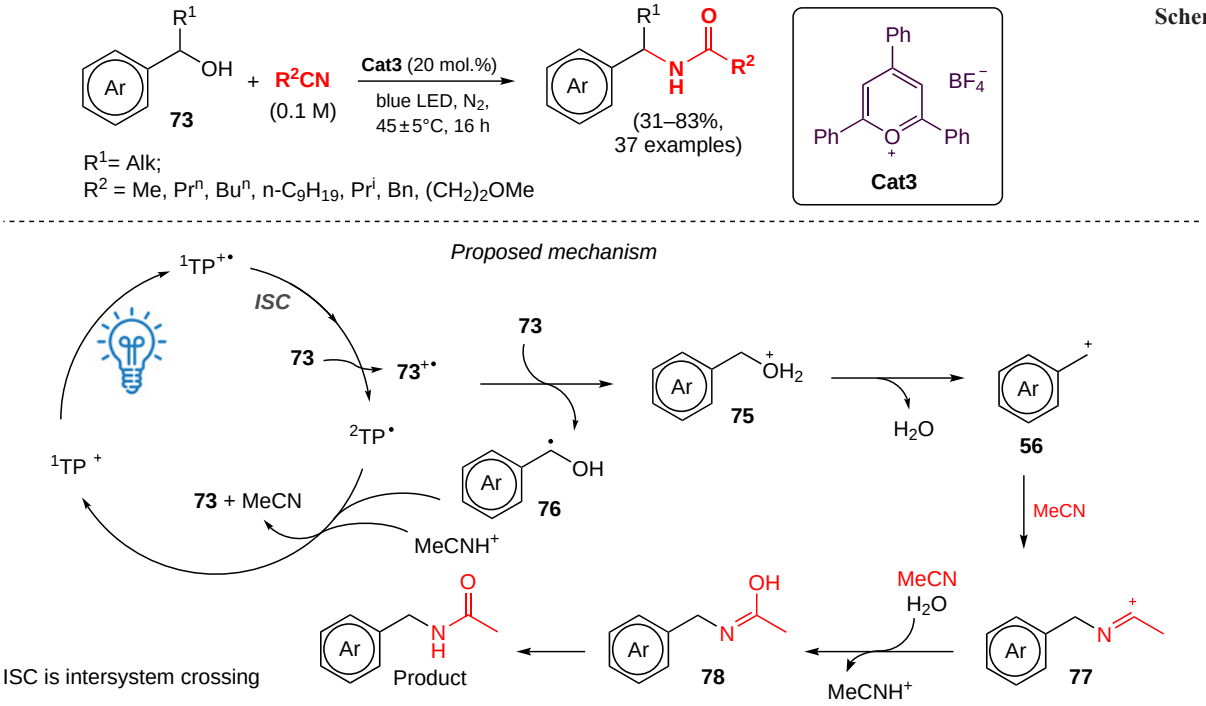
In 2022, König and co-workers[66] reported the decarboxylative amidation of carboxylic acids by a Ritter-type reaction using the iodine(III)–BF_3 complex obtained from iodobenzene, Selectfluor as an oxidizing agent and BF_3 as the Lewis acid. Carboxylic acids **79**, irradiated with light in the presence of catalytic amounts of iodobenzene and stoichiometric amounts of Selectfluor in the presence of water and BF_3, undergo an amidation to give *N*-benzylamides **58**, **80** and **81** (Scheme 24). This reaction is applicable to both benzylic and aliphatic carboxylic acids.

Based on the data obtained, the authors proposed two reaction pathways, shown in Scheme 24. Pathway *a* involves homolysis of the I–O bond of the initially formed iodonium complex **82**, which gives rise to radicals **83** and **84**. Next, decarboxylation of intermediate **83** occurs yielding benzyl radical **55** and further oxidation to carbocation **56**. The next step is the nucleophilic addition of the nitrile to carbocation **56**, followed by hydrolysis to the desired *N*-benzylamide.

The amidation process can be accomplished by an alternative mechanism when mediated by I_2 (see Scheme 24, pathway *b*). In the first step, molecular iodine reacts with carboxylic acid **79** to give acyl hypoiodides RCO_2I (**85**). Blue light irradiation of compounds **85** is accompanied by their decarboxylation to the corresponding alkyl iodides **86**. Oxidation of the latter occurs either due to the action of Selectfluor or in a reaction with the fluoroborate complex PhI^{III}, in which case iodonium complex **82** is formed. Intermediate **82** undergoes rapid nucleophilic substitution in the presence of a nitrile with subsequent hydrolysis to the target amide.

In 2022, Yoon and co-workers[67] developed a method for oxidative decarboxylative coupling of carboxylic acids **79** with nitriles under visible light irradiation in the presence of $Cu(OTf)_2$ and Na_2CO_3. The authors hypothesized that the photoactive copper(II) carboxylate chromophore, obtained *in situ* by the

Scheme 23



reaction of the carboxylic acid with $Cu(OTf)_2$ in the presence of a base, leads to the dissociative formation of a carboxyl radical, which undergoes decarboxylation under blue light (427 nm) irradiation and generates a benzyl radical. The latter can be oxidized by copper(II) salts to form a carbocation, which then reacts with acetonitrile to give amidation products, *N*-benzylamides.

Ji and co-workers[68] proposed an effective and low-cost method for the synthesis of *N*-substituted amides based on the reaction of nitriles with (methoxymethyl)benzene derivatives, catalyzed by $Fe(ClO_4)_3 \cdot H_2O$. This catalyst proved to be quite effective for this reaction in the solvent-free protocol providing *N*-benzylamides in yields of up to 93%.

2.10. Functionalization of C(sp²)–H bonds in amidation of benzene derivatives

In 2015, Redshaw and co-workers[69] developed a copper-catalyzed amidation of benzene derivatives **87** with electron-donating groups using benzonitrile (**5b**) as an amide source. Interestingly, the reaction occurs selectively at the *para*-position (Scheme 25). The process runs in the presence of a catalyst, copper(II) trifluoromethanesulfonate, the iodine-centered electrophile 2-(diacetoxyiodo)mesitylene ($MesI(OAc)_2$) and water (3 equiv.) in boiling dichloroethane for 15 h. The resulting *N*-arylbenzamides **30** are obtained in yields from 35% to 93%. The regioselectivity of this amidation is controlled by both steric and electronic factors, which leads to the selective formation of only *para*-amidation products. No other isomers were detected in the reaction mixture by ^1H NMR spectroscopy. Aromatic compounds with electron-withdrawing groups, such as chlorobenzene and nitrobenzene, are unsuitable for this reaction, and no *N*-arylbenzamides were detected in this case.

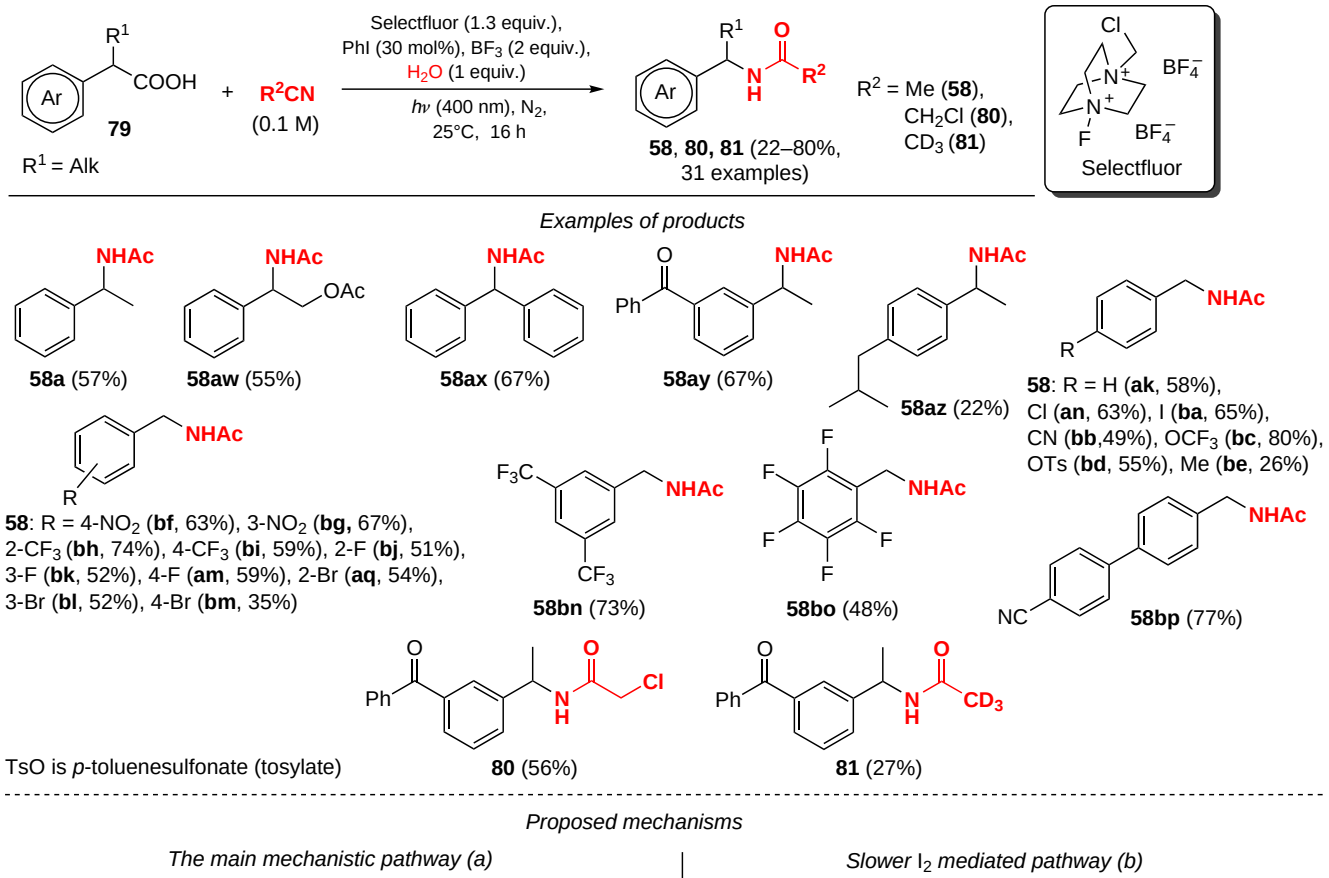
The probable mechanism of this catalytic transformation is shown in Scheme 25. In the first step, electrophilic substitution in the aromatic ring initiated by trimethylsilyl trifluoromethanesulfonate leads to diaryliodonium salts **88**. The active form of the catalyst CuOTf, formed from $Cu(OTf)_2$ *via* disproportionation or reduction, is oxidized by the diaryliodonium salt **88** to intermediate **89**, which undergoes ligand exchange with benzonitrile (**5b**) to furnish complex **90**. The proposed Cu^{III} species promote the hydrolysis of the coordinated benzonitrile, forming intermediate **91**, which then undergoes reductive elimination to *N*-arylbenzamides **30**. In this case, the active form of the catalyst CuOTf is regenerated to participate in the catalytic cycle.

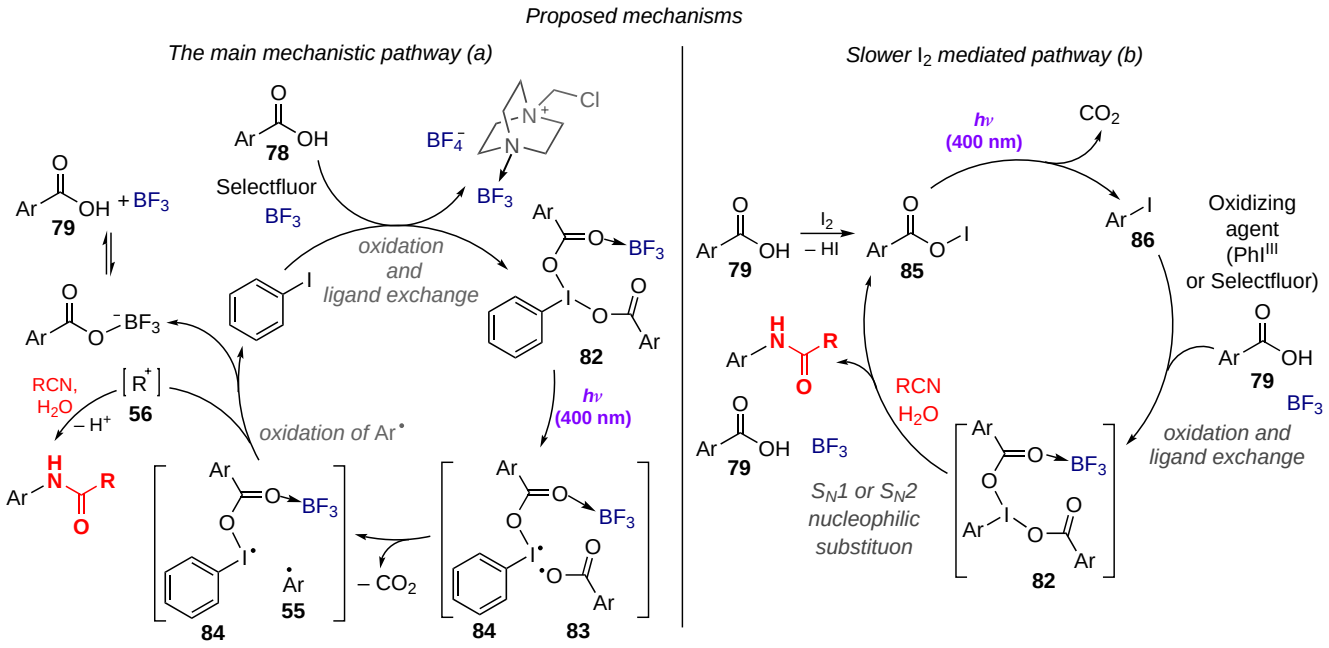
With an increase in the amount of aromatic substrate to 4 equiv., further arylation of the obtained *N*-arylamides occurs selectively in the *meta* position, which was shown by the example of the reaction between *o*-xylene **87a** with various benzonitriles (Scheme 26). Benzonitriles containing both electron-donating and electron-withdrawing substituents undergo arylation to give the corresponding products **92** in moderate yields (36–51%).

3. Electrooxidative amidation of aromatic compounds with nitriles

Organic electrosynthesis is an environmentally friendly alternative to traditional chemical reactions and allows valuable *N*-containing compounds to be obtained in an atom-efficient manner under mild conditions with a minimum number of steps.[70–74] Electrochemical approaches in which electrons are used as a 'traceless' reagent are also of interest for dehydrogenating C–H/N–H cross-coupling reactions.[75–77] Ritter-type reactions of electrochemical amidation of alkanes,[78,79] esters[80] and ketones[81] were described as early as the 1970s. Since then, the study of electrochemical processes following the Ritter-type reaction has slowed down somewhat. However, in the last few years, new electrochemical approaches have been proposed to the amidation of aromatic compounds by functionalizing the C(sp³)–H bonds at benzyl carbon atoms[82–91] and the C(sp²)–H bonds in aromatic centres.[89–95] Also worth mentioning are studies[96–102] on electrooxidative C–H/N–H coupling, implemented with the involvement of compounds of various classes.

Scheme 24



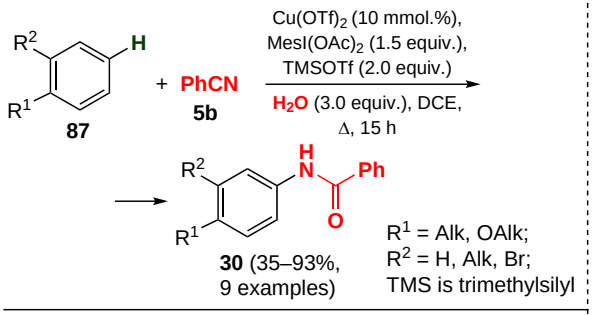


3.1. Electrooxidative amidation of alkylbenzenes

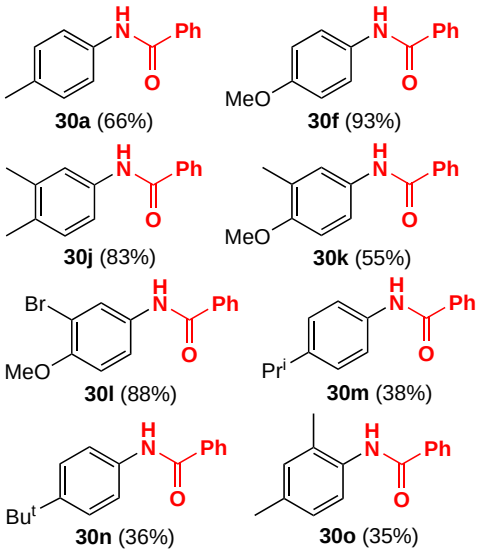
In 2017, Ley and co-workers[82] reported the synthesis of *N*-benzylacetamides **58** from toluene derivatives by anodic oxidation in a flow reactor in the presence of methanesulfonic acid (MSA) as the Brønsted acid (Scheme 27).

The amidation products of *para*-, *meta*- and *ortho*-substituted toluenes **53** were isolated in moderate yields (53–64%). In all cases, unreacted starting compounds were detected in the reaction mixture, indicating incomplete conversion of the substrates. By-products (<2%) of this reaction are benzaldehydes and benzyl alcohols. The process does not proceed without the addition of acid.

In 2021, Shen and Lambert[83] developed an electrophotocatalytic method for the synthesis of *N*-benzylacetamides **58** using tris(*cis*-2,6-dimethylpiperidin-1-yl)cyclopropenium (TAC) ion as a catalyst (Scheme 28). Aromatic compounds containing an ester, alcohol, chloroalkyl, or tosylate moiety were used in the amidation reaction. The target products **58** were obtained in moderate to good yields (31–78%), and the reaction did not proceed without application of the cell potential and in the absence of a catalyst. When the reaction was carried out in the absence of compact fluorescent lamp (CFL) irradiation, the amidation products were formed only in small quantities (yield of about 9%).

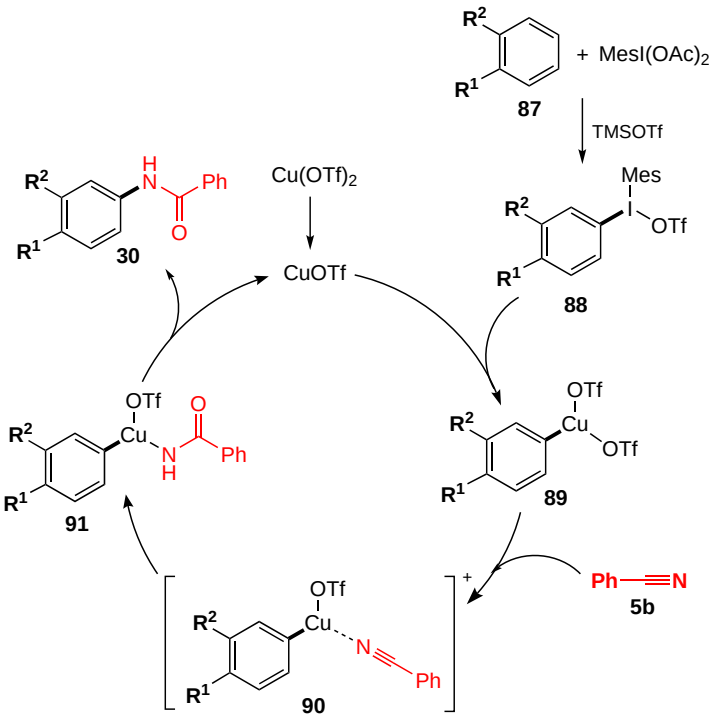


Examples of products

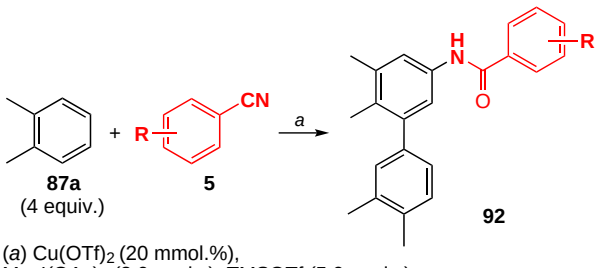


Scheme 25

Proposed mechanism

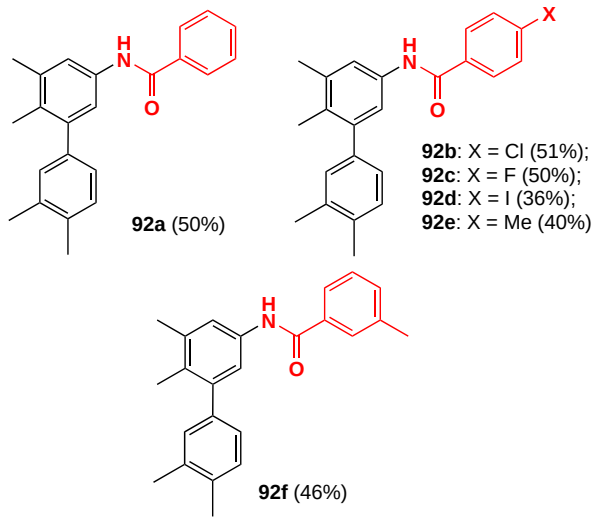


Scheme 26

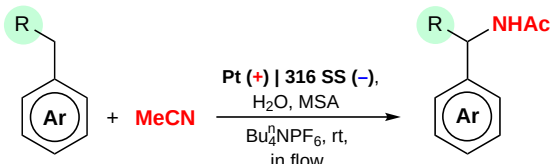


(*a*) $Cu(OTf)_2$ (20 mmol.%), $MesI(OAc)_2$ (3.0 equiv.), TMSOTf (5.0 equiv.), H_2O (3.0 equiv.), DCE, Δ, 24 h

Examples of products

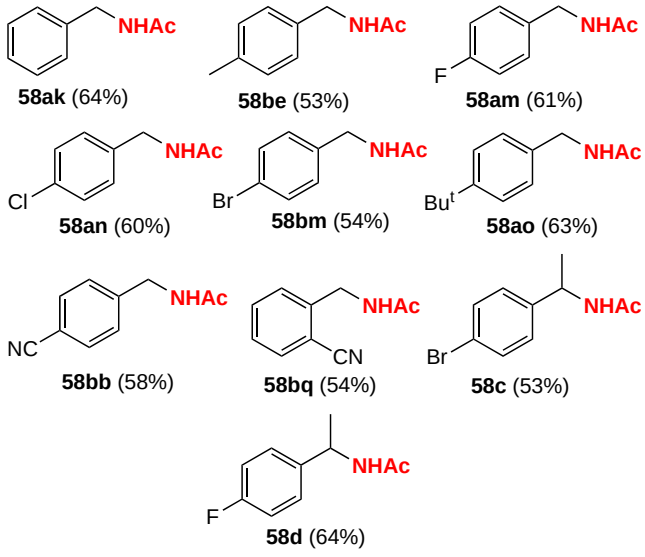


Scheme 27

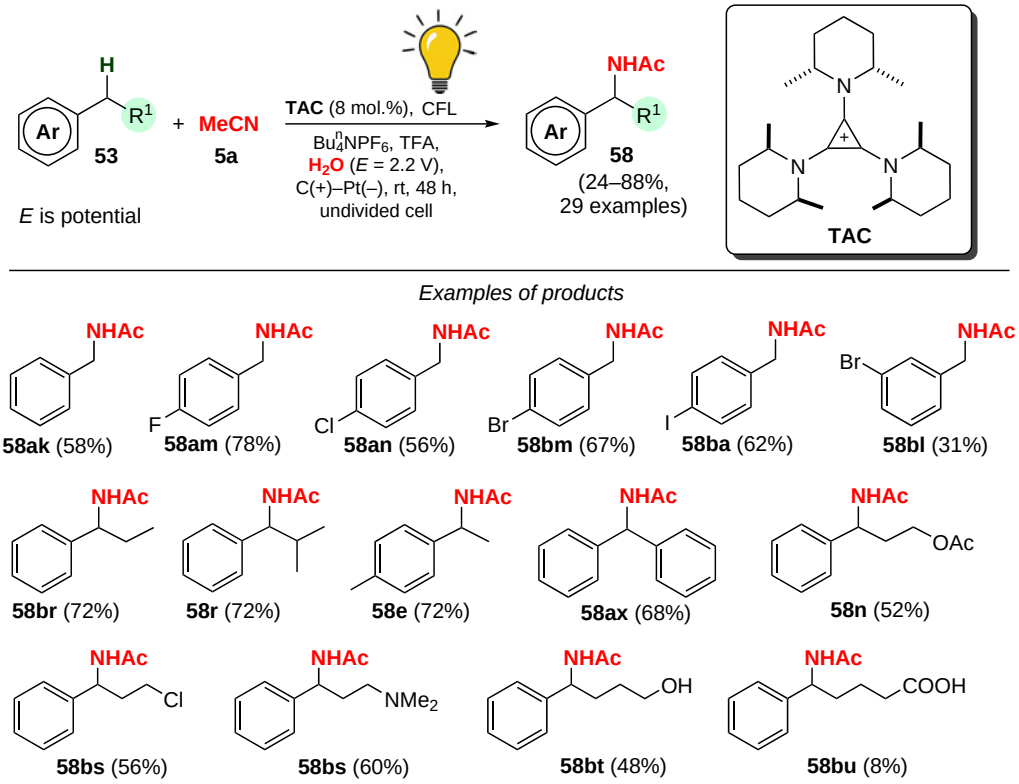


316 SS is stainless steel cathode

Examples of products



Scheme 28



The mechanism of this electrophotocatalytic reaction is shown in Scheme 29. The TAC catalyst undergoes single-electron oxidation to generate a stable dication radical **93**. Irradiation of the latter affords photoexcited intermediate **94**, which is involved in the single-electron oxidation of substrate **53** to form cation radical **95**.

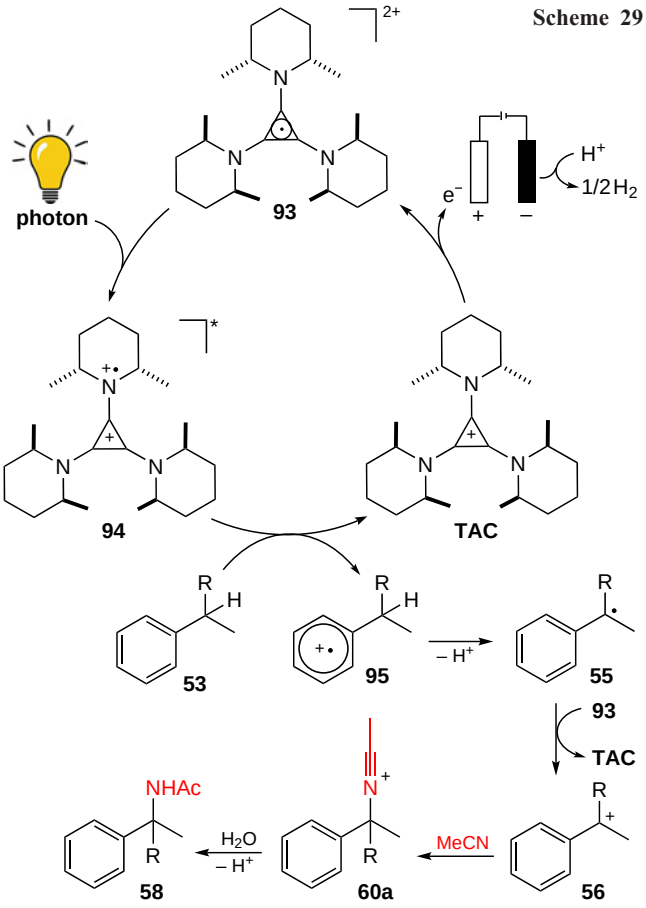
Deprotonation of this radical cation generates benzyl radical **55**, which is then reoxidized either by TAC dication radical **93** or directly at the anode to yield carbocation **56**. Nucleophilic addition of acetonitrile to cation **56** yields intermediate **60a**. In the final step, cation **60a** is hydrolyzed to the corresponding *N*-benzylacetamide **58**. The key point of this approach is that the reaction occurs at the oxidation potential of the TAC electrophotocatalyst rather than the aromatic substrates, thereby minimizing the risk of side processes.

In 2022, Ye and co-workers[84] proposed another example of electrochemical amidation. The authors carried out the reaction using sulfuric acid in an undivided cell at room temperature (Scheme 30). The amidation products of toluene and ethylbenzene derivatives containing various functional groups (F, Cl, Br, I, CN, NO_2, NH_2, Alk, Ac, Ph, OMe) in the benzene ring are obtained in high yields (up to 85%). This method is characterized by high regioselectivity; substrates with several alkyl groups in the aromatic ring undergo amidation exclusively at one of them.

In the absence of sulfuric acid, there is virtually no reaction. Presumably, the sulfate anion SO_4^{2-} undergoes single-electron oxidation at the anode to form $SO_4^{\cdot-}$. Being an active acceptor of the hydrogen atom, $SO_4^{\cdot-}$ abstracts the hydrogen atom from the aromatic substrate to generate a benzyl radical, which is oxidized to a carbocation at the anode. The latter then undergoes a nucleophilic addition to the nitrile to give the corresponding *N*-benzylacetamides **58**.

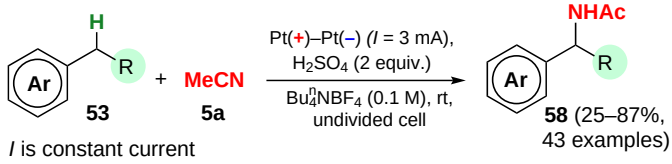
In 2023, Zhou and co-workers[85] reported a method for the amidation of alkylbenzenes under electrochemical conditions. The reaction was carried out under mild conditions at room

Scheme 29

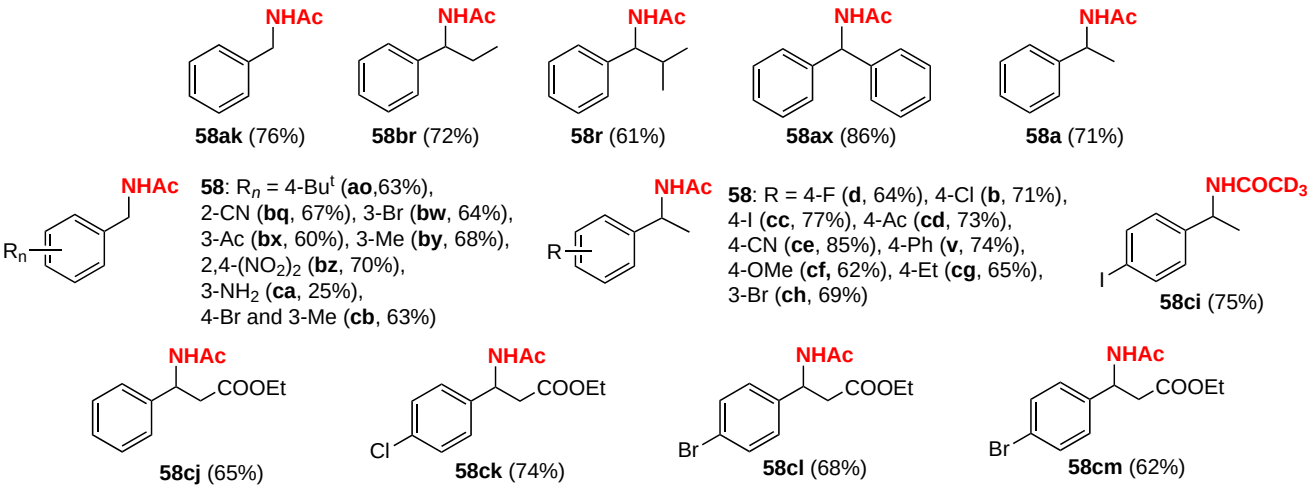


temperature in an undivided cell in the presence of trifluoromethanesulfonic acid to furnish the corresponding *N*-benzylacetamides **58** in yields from 20 to 91% (Scheme 31). Substrates containing electron-donating substituents in the

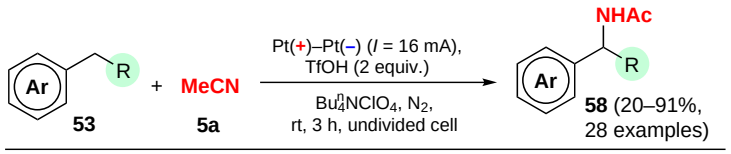
Scheme 30



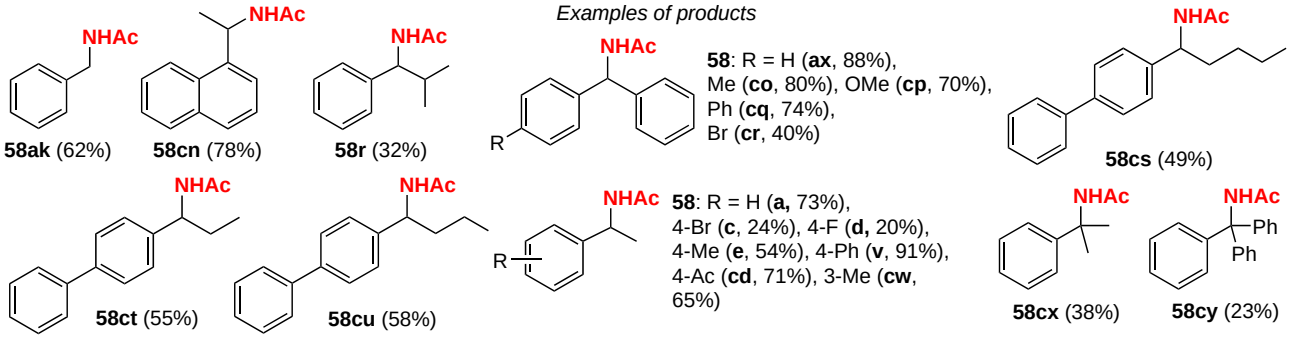
Examples of products



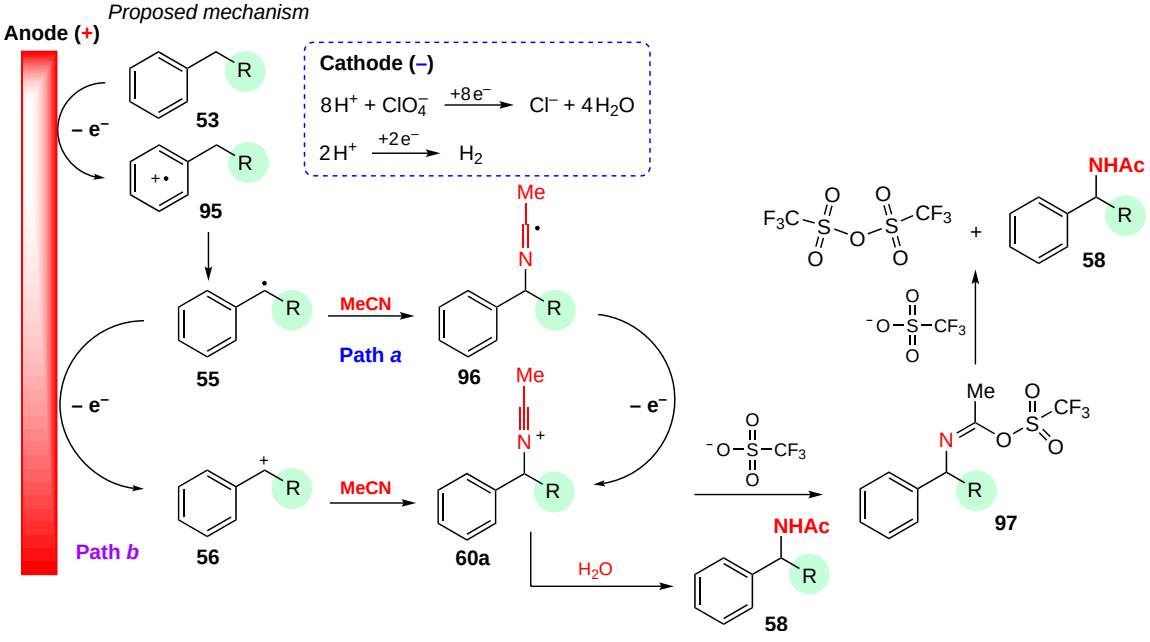
Scheme 31



Examples of products



Proposed mechanism



aromatic ring were more active in this reaction than their analogues with electron-withdrawing groups. This is apparently due to the higher electron density, which facilitates the activation of benzyl C–H bonds under direct current electrolysis conditions. The presence of several alkyl substituents in the ring has little effect on the yield of the target product. The yields of amidation products are significantly reduced when substrates with long alkyl chains or bulkier substituents are used.

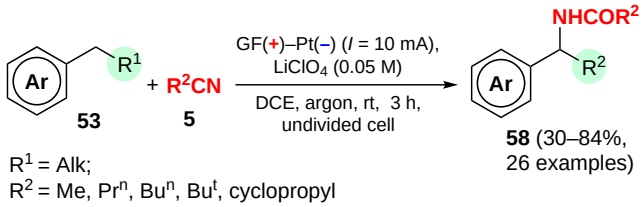
Based on the obtained experimental and literature data,[103–106] a mechanism for this process has been proposed (see Scheme 31). First, single-electron oxidation of substrate **53** occurs at the anode to form benzyl radical **55**. Then, two reaction paths are possible. In path *a*, radical **55** reacts with MeCN to generate first radical **96** and then nitrilium cation **60a** after another single-electron oxidation. In path *b*, radical **55** is oxidized to carbocation **56**, which then reacts with MeCN to afford nitrilium cation **60a**. The latter reacts with TfO^- to form intermediate **97**, which then undergoes detrifluoromethanesulfonylation to yield amidation product **58** and trifluoromethanesulfonic anhydride. The nitrilium cation **60a** can be hydrolyzed directly to *N*-benzylamide **58**. At the cathode, ClO_4^- is reduced to Cl^- and H_2O in the presence of H^+.

Yin and co-workers[86] developed a new electrocatalytic protocol for the conversion of toluene to *N*-benzylacetamide using MeCN as an amidating reagent. The proposed approach involves the use of a hydrophobic electrode material based on polytetrafluoroethylene (PTFE)-coated carbon paper (CP) and a conductive membrane instead of the background electrolyte. Based on the experimental results and quantum chemical calculations, it was demonstrated that coating the electrode with such a polymer promotes the adsorption of toluene and its efficient dehydrogenation. In addition, the hydrophobic material prevents the adsorption of water onto the electrode, inhibiting the competitive reaction of water oxidation.

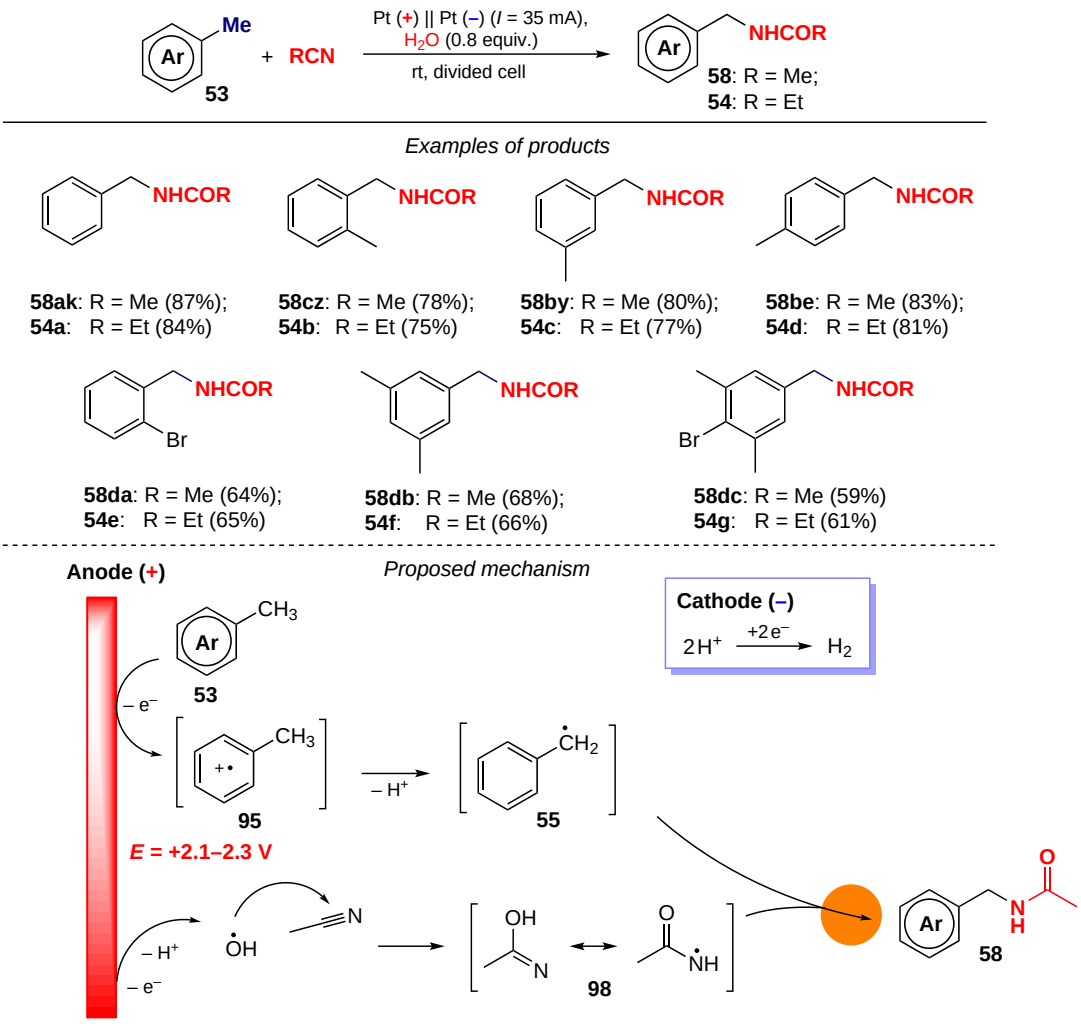
Sun and co-workers[87] described a method for the electrochemical amidation of alkylarenes with nitriles and water in the absence of an oxidizing agent and mediator, which provides an access to various *N*-benzylamides in moderate and good yields (Scheme 32).

In 2023, Budnikova and co-workers[90] carried out electrooxidative amidation of toluene derivatives in aceto- and propionitriles, leading to *N*-benzylamides in yields of up to 87% (Scheme 33). The reaction occurs under mild conditions (at room temperature) in the absence of metal catalysts, oxidizing agents and acid additives. For the substrates having several methyl groups in the aromatic ring (xylenes, mesitylene), the

Схема 32



Scheme 33



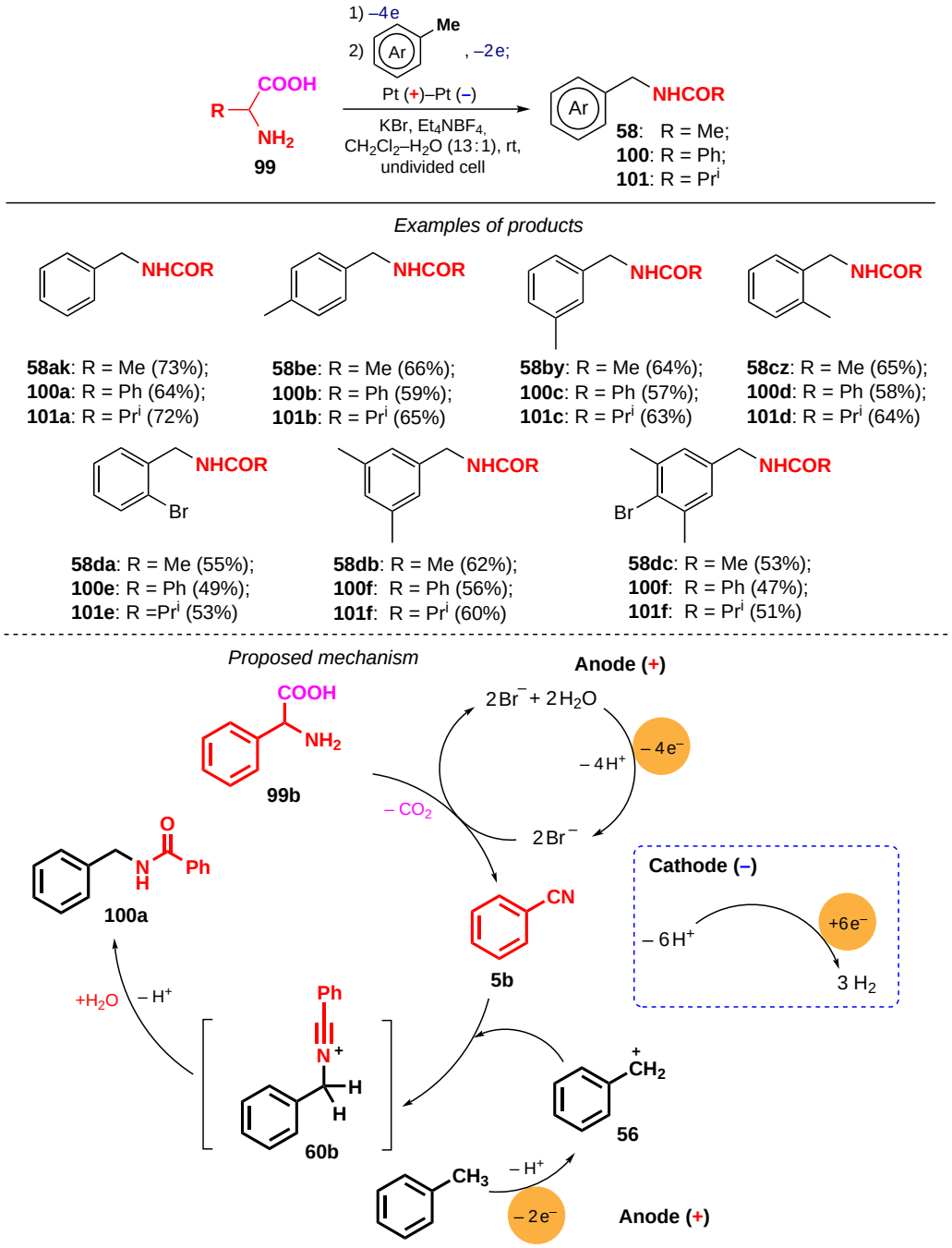
main product is amidation products with only one substituted methyl group, even when passing a larger amount of electricity and increasing the synthesis time. The presence of a bromine atom as a substituent in the ring reduces the yield of the target amidation product, since by-products, *viz*., aldehydes and dimers, are also formed.[91] The oxidation of *ortho*-, *meta*- and *para*-xylenes, in addition to *N*-benzylamides, produces by-products–aromatic aldehydes (yields of 8–12%). In all cases, products with only one amide group are formed during the reaction.

The mechanism of electrooxidative amidation of toluene derivatives with nitriles is shown in Scheme 33. It is assumed that the key step is the anodic oxidation of water generating hydroxyl radicals, which add to the nitrile to give spin adduct **98**. Aromatic substrates with a methyl group (toluene, xylenes, mesitylene) are oxidized in the same potential range as water (+2.1÷+2.3 V).[80–81] Probably, in this case, one electron is spent on the formation of the benzyl radical **55**, and the second electron is spent on the generation of hydroxyl radicals followed by the formation of adduct **98**. Further interaction of the two radicals affords amidation products **58**. At the same time, the amidation can be realized by another route, where at the initial step, successive two-electron oxidation and deprotonation of methylarene to benzyl carbocation takes place.[83,86,87] Further nucleophilic addition of acetonitrile to the carbocation gives a nitrilium cation, which undergoes hydrolysis to the corresponding *N*-benzylamide.

In 2022, the same research group[88] described a one-pot protocol for the synthesis of *N*-benzylamides using amino acids **99** (2-phenylglycine, valine, alanine) as an amide source (Scheme 34).

In the first step, amino acids undergo decarboxylation to nitriles in the presence of a mediator, potassium bromide, under anodic oxidation conditions. Subsequent addition of an aromatic

Scheme 34



substrate to the reaction mixture and passing electricity in the amount of 2 F leads to *N*-benzylamides **58**, **100** and **101** in moderate yields (up to 73%).

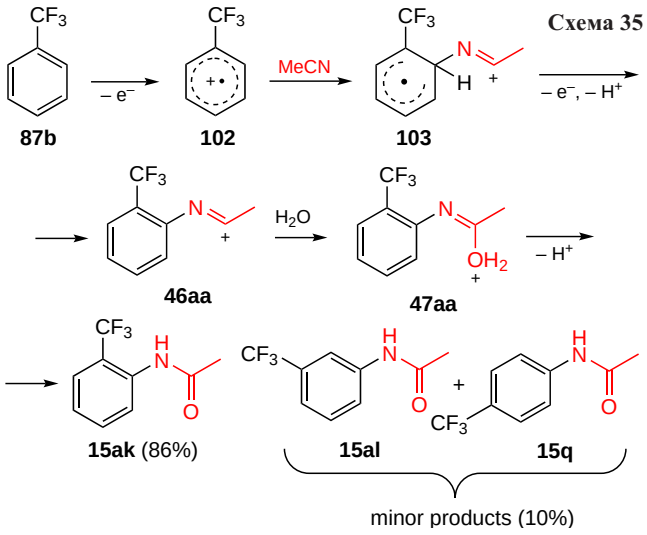
The plausible mechanism of the process is shown in Scheme 34. Under conditions of anodic oxidation of the KBr mediator, hypobromite ions (BrO^-) are generated, which selectively oxidize 2-phenylglycine (**99b**) to benzonitrile (**5b**). Addition of an aromatic substrate to the reaction mixture and subsequent two-electron oxidation in the presence of benzonitrile (**5b**) gives intermediate **60b**. In the final step, hydrolysis of the nitrilium cation **60b** affords *N*-benzylbenzamide **100a** in 73% yield.

3.2. Electrooxidative functionalization of C(sp^2)–H bonds in amidation of benzene derivatives

The first example of electrochemical amidation of trifluoromethylbenzene (**87b**) with acetonitrile was described in 2014 by Barba *et al.*[92] The reaction was carried out under anodic oxidation conditions in a potentiostatic mode at a potential of +2.8 V (*vs*. Ag/AgCl (sat.)) in absolute acetonitrile using tetra-*n*-butylammonium tetrafluoroborate as a background electrolyte (Scheme 35). The plausible mechanism of the process includes the formation of cation radicals **102** and **103**.

The reaction produces 2-(trifluoromethyl)acetanilide (**15ak**) as the major product (yield 86%), the by-product is a 10% mixture of 3-(trifluoromethyl)acetanilide (**15al**) and 4-(trifluoromethyl)acetanilide (**15q**) in a 3 : 1 ratio, as determined by gas chromatography (GC) and ^{1}H NMR spectroscopy. No other substrates or nitriles were used in this reaction. The formation of 2-(trifluoromethyl)acetanilide (**15ak**) as the predominant isomer can be explained by the strong inductive effect of the trifluoromethyl group, which provokes a charge

Схема 35

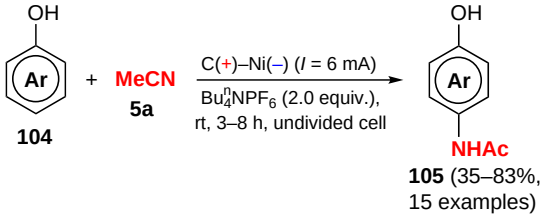


deficit in the adjacent positions, thereby facilitating the nucleophilic attack of the nitrile.

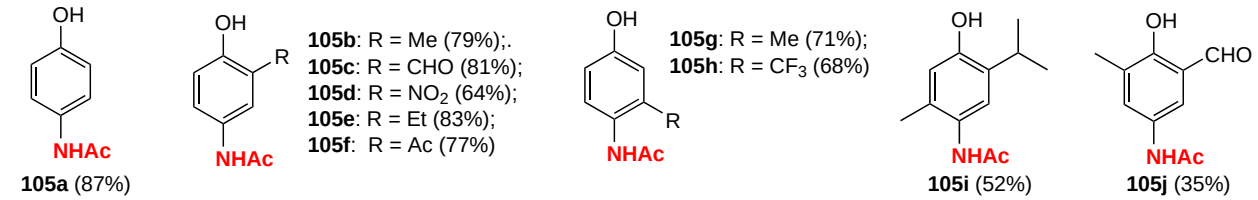
In 2022, Banerjee and co-workers[93] proposed an approach for the electrochemical amidation of phenols **104** with alkyl, aldehyde and ketone groups, allowing the preparation of various *N*-(4-hydroxyphenyl)acetamides **105** (Scheme 36).

The reaction proceeds regioselectively in the *para*-position relative to the hydroxyl group in the absence of external oxidizing agents and catalysts, but this method is limited to phenolic substrates only. Substituents in the benzene ring affect the yield of the target products and the reaction time. The yields of *N*-arylacetamides **105** are lower for phenols with electron-withdrawing groups in the benzene ring (NO_2, CF_3, CHO, Ac),

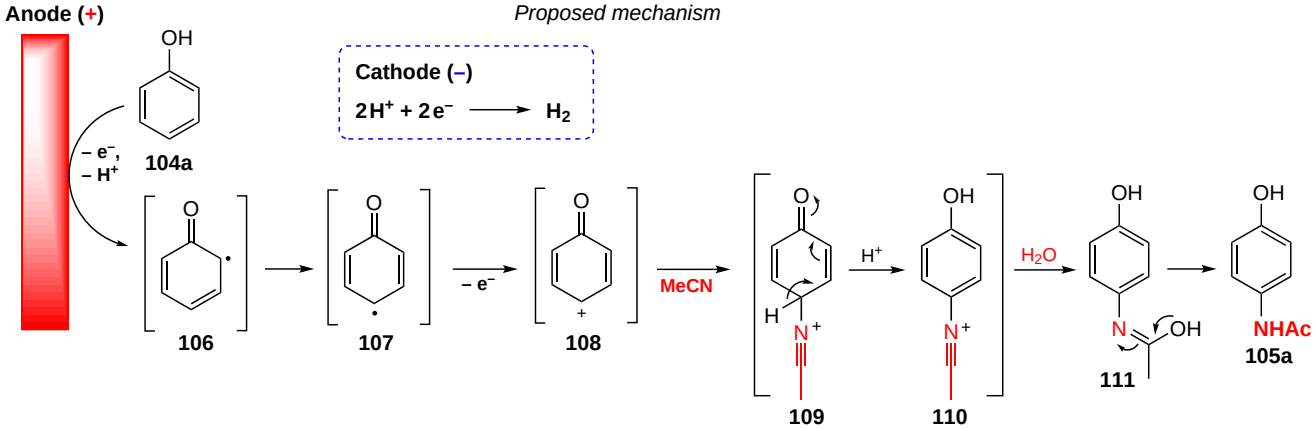
Scheme 36



Examples of products



Proposed mechanism



and more time is required for the reaction to be completed. Electrooxidation of phenols with electron-donating substituents (Me, Et) proceeds comparatively faster.

The probable mechanism of the process is shown in Scheme 36. The reaction is initiated by single-electron oxidation of phenol **104a** to generate radicals **106** and **107**, further single-electron oxidation of which gives carbocation **108**. Carbocation **108** then undergoes classical Ritter reaction steps in the presence of acetonitrile to form nitrilium ions **109** and **110**. The latter is then hydrolyzed and *via* the formation of hydroxyimine **111** yields the target product, acetaminophen **105a**.

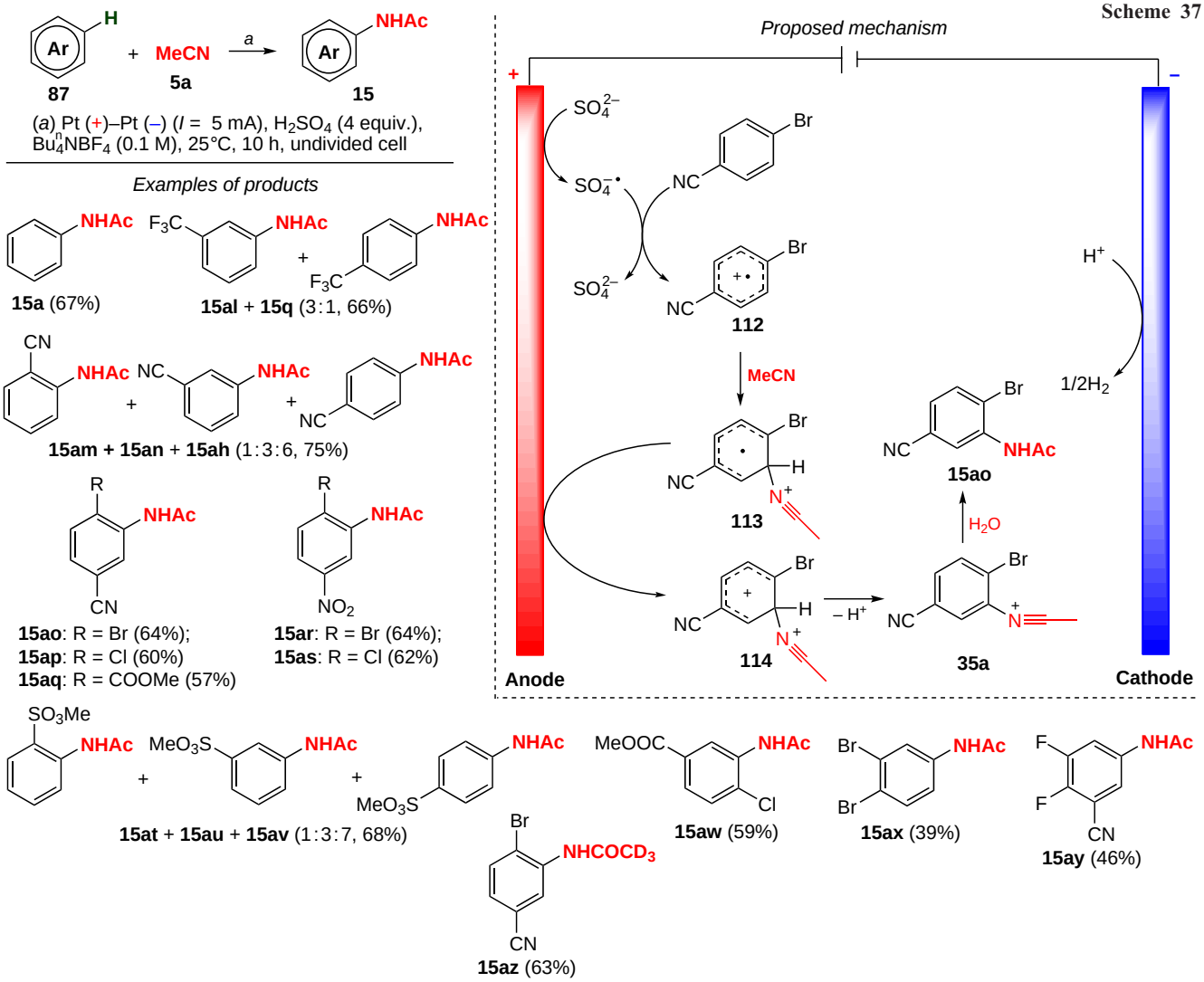
In 2023, Ye and co-workers[94] carried out amidation of benzene derivatives containing electron-withdrawing substituents in the aromatic ring in an undivided electrochemical cell (Scheme 37). Mono-, di-, and trisubstituted benzenes with CO_2Me, SO_3Me, CN, CF_3 groups or Br, Cl, F atoms were used in this reaction, affording the corresponding *N*-arylacetamides **15** in moderate and good yields (from 39 to 75%). The process takes 10 h and requires the presence of 4 equiv. sulfuric acid. The method is atom-economical, since it utilizes cost-effective and readily available reagents.

The proposed mechanism of amidation is shown in Scheme 37. In the first step, as a result of anodic oxidation of the sulfate ion, a sulfate anion radical is formed, which is involved in the single-electron oxidation of 4-bromobenzonitrile to generate cation radical **112**. This cation radical then undergoes nucleophilic attack by acetonitrile to give radical **113**, after which it undergoes further oxidation at the anode, affording cation **114**. Deprotonation of the latter gives the nitrilium cation **35a**, further hydrolysis of which leads to the target amidation product. Another possible reaction mechanism involves two-electron oxidation and deprotonation of the aromatic substrate **87** to furnish the corresponding cation, which then adds acetonitrile.

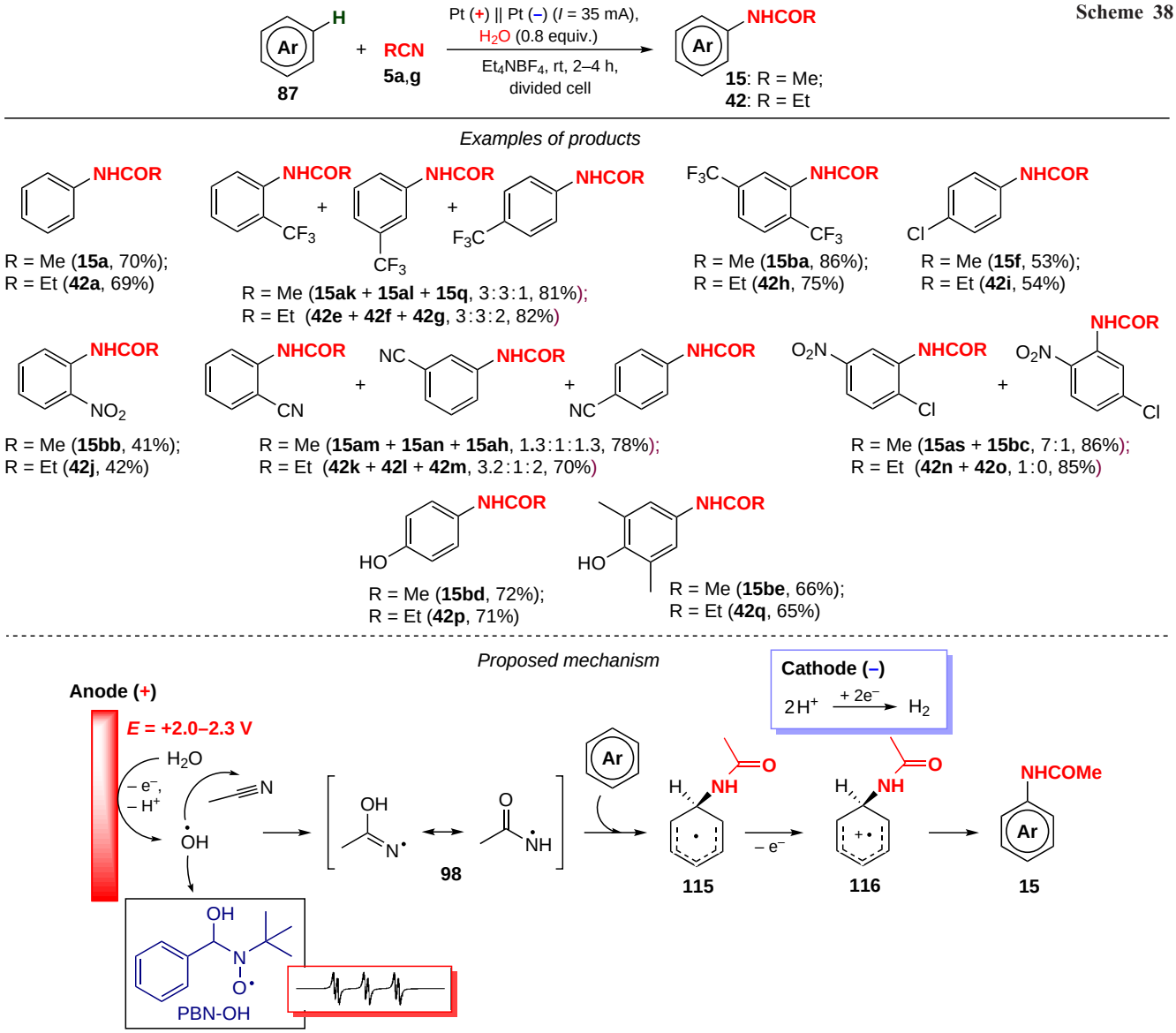
Yu.H.Budnikova and co-workers[90,95] developed an electrochemical protocol for the synthesis of anilides **15** and **42** in moderate to high yields (41–86%) using aceto- (**5a**) and propionitriles (**5g**) as amide sources (Scheme 38). This approach features mild conditions (room temperature, normal pressure); the reaction is carried out in an electrochemical cell with separation of the cathode and anodic spaces without the use of additives.

The proposed mechanism of the transformation is shown in Scheme 38. One of its key steps is the anodic oxidation of water to generate hydroxyl radicals (•OH), capable of adding to both the nitrile and the aromatic substrate. The formation of radicals during the electrochemical oxidation of aromatic substrates in acetonitrile was detected using electron paramagnetic resonance (EPR). When a benzene solution was oxidized in a MeCN–water (0.8 equiv.) mixture in the presence of *N-tert*-butyl-α-phenylnitrone (PBN) as a spin trap, the EPR spectrum of the spin adduct PBN-OH• was recorded. It is likely that the hydroxyl

Scheme 37



Scheme 38



radical adds to the nitrile to afford the spin adduct **98**, which then reacts with the aromatic substrate, which gives the amidation product **15** *via* intermediates **115**, **116**.

In 2022, Song and co-workers[107] reported electrophilic amidomethylation of aromatic compounds to give *N*-benzylacetamides **58**. The authors used dimethyl sulfoxide and acetonitrile as sources of methyl (CH_2) and amide (NHAc) units, respectively, and the process was carried out in a strongly acidic medium with prolonged heating to 60–80°C for 20 h (Scheme 39). The corresponding *N*-benzylacetamides **58** were isolated in moderate and good yields (31–82%). This method provides efficient practical synthesis of deuterated benzylamides from readily available deuterated solvents, DMSO-D_6 and MeCN-D_3, and also allows functionalization of known drugs (gemfibrozil, naproxen, *etc*.).

The proposed mechanism of the process is shown in Scheme 39. In the initial steps, dimethyl sulfoxide is protonated in an acidic medium to give an intermediate cationic form **117**; its further dehydration leads to methyl(methylene)sulfonium **118**. At the same time, acetonitrile undergoes acidic hydrolysis to acetamide, which reacts with the sulfonium cation **118** to afford intermediate **119**. In an acidic medium, intermediate **119** decomposes to methanethiol and the ammonium cation *N*-methyleneacetamide **120**. The latter reacts reversibly with acetamide and yields a by-product, *N*,*N*′-methylenediacetamide **121**, which was isolated in an individual state.

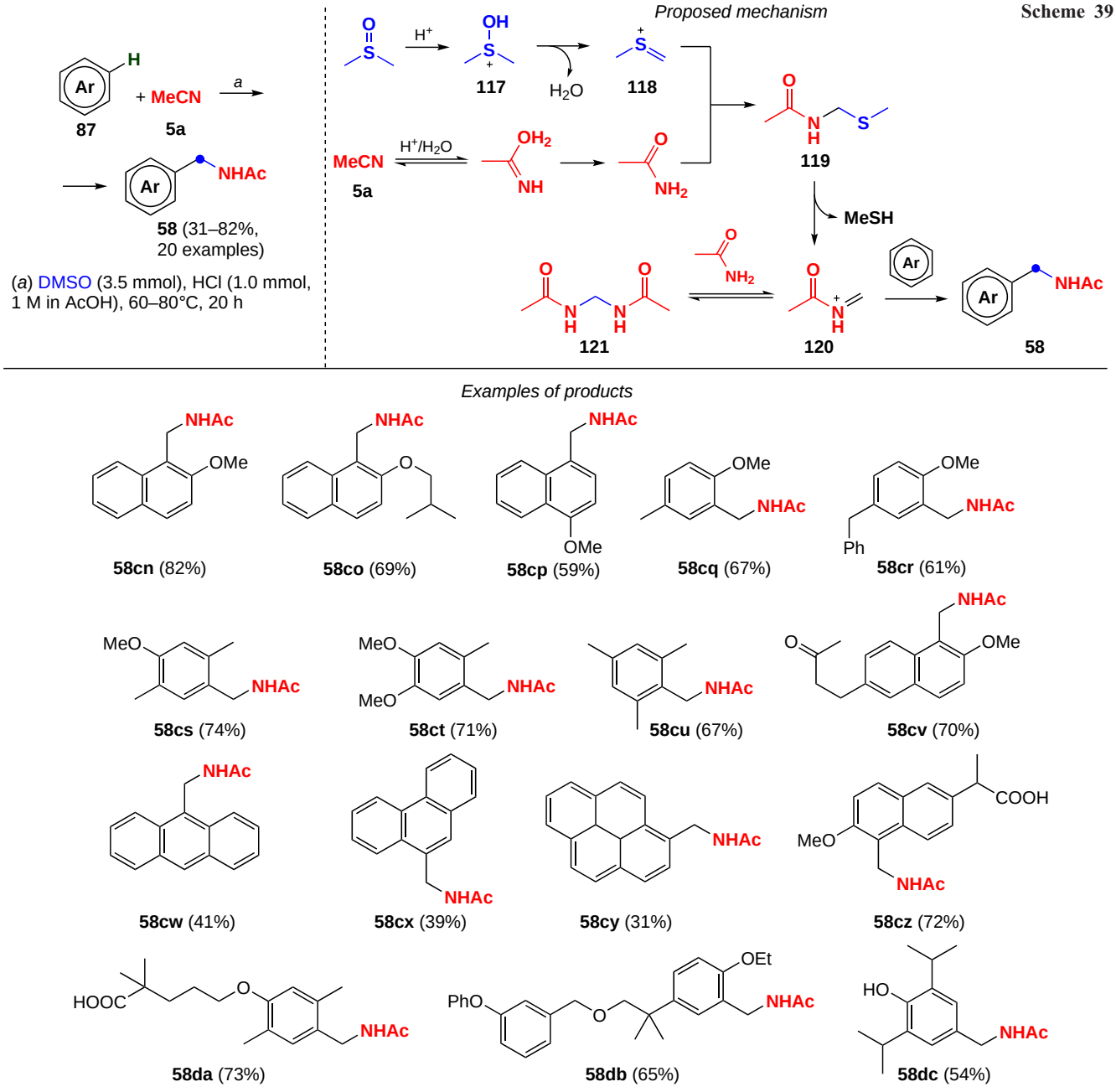
Upon addition of an aromatic substrate to cation **120**, the target amidomethylation occurs to afford *N*-benzylacetamide **58**. The use of acetamide instead of acetonitrile also promotes the reaction, which could be evidence of the acidic hydrolysis of acetonitrile to acetamide as one of the key steps.

4. Amidation of heteroaromatic substrates

4.1. Amidation of quinoxalin-2(1*H*)-ones

In 2019, Qu and co-workers[108] developed a method for direct oxidative palladium-catalyzed amidation of quinoxalin-2(1*H*)-ones **122** with acetonitrile (**5a**). The reaction proceeds at 100°C in the presence of an oxidizing agent, potassium persulfate ($K_2S_2O_8$), for 12 h (Scheme 40). This method makes available various 3-acetaminoquinoxalin-2(1*H*)-ones **123** in good and

Scheme 39



high yields (up to 90%). Substrates containing electron-donating groups in the heterocycle (*e.g.* methyl) provide higher yields of products compared to quinoxalin-2(1*H*)-ones with electron-withdrawing substituents (F, Cl, Br). Nitroquinoxalin-2(1*H*)-ones were found to be unsuitable for this reaction, as was quinoxalin-2(1*H*)-one with a free nitrogen atom.

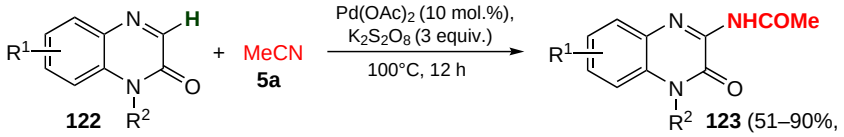
The proposed catalytic mechanism of the process using 1-methylquinoxalin-2(1*H*)-one (**122a**) as an example is shown in Scheme 41. Acetonitrile (**5a**) reacts with H_2O under the action of $K_2S_2O_8$ to give acetamide **124**.[109] Promoted by $K_2S_2O_8$ and the catalyst $Pd(OAc)_2$, homolytic cleavage of the N–H bond occurs in acetamide **124**, forming radical **98**. Metallation of 1-methylquinoxalin-2(1*H*)-one (**122a**) with palladium acetate affords intermediate **125**, which undergoes oxidative addition to radical **98** to yield palladium(III) complex **126**. Under dual palladium catalysis, Pd^{III} intermediate **126** is oxidized to palladium(IV) compound **127** in the presence of a photoredox catalyst and/or an oxidizing agent.[110–112] Product **123a** is formed by reductive elimination to release the Pd^{II} catalyst to be involved in the next catalytic cycle.

4.2. Amidation of xanthenes

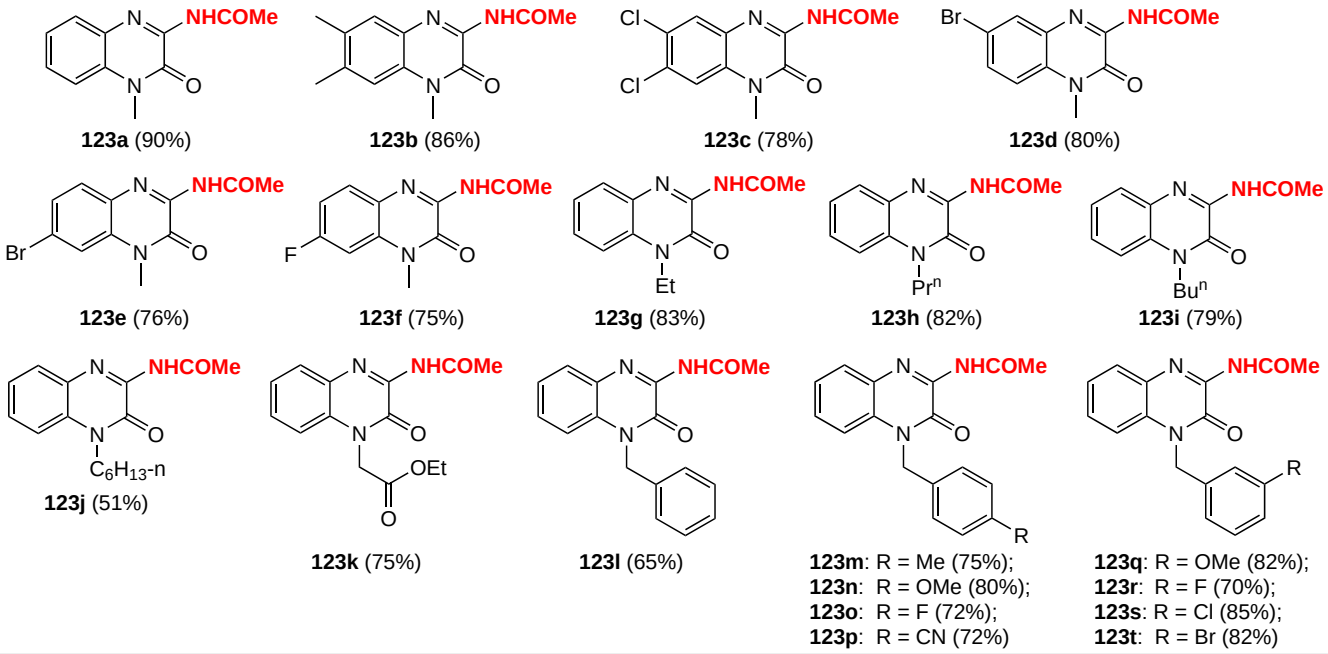
In 2023, Li and co-workers[113] developed electrochemical amidation of xanthenes **128** with acetonitrile. The process takes place over 5 h in an undivided electrochemical cell equipped with graphite electrodes (Scheme 42). Xanthenes bearing Me, Ph, OMe and CF_3 substituents were suitable substrates for this reaction, giving amidation products **129** in 56–75% yields.

The plausible pathway of the electrochemical reaction with acetonitrile on the example of xanthene **128a** is shown in Scheme 42. In the first step, single-electron oxidation of the substrate to cation-radical **130** takes place, followed by proton elimination and generation of radical **131**. Next, anodic oxidation

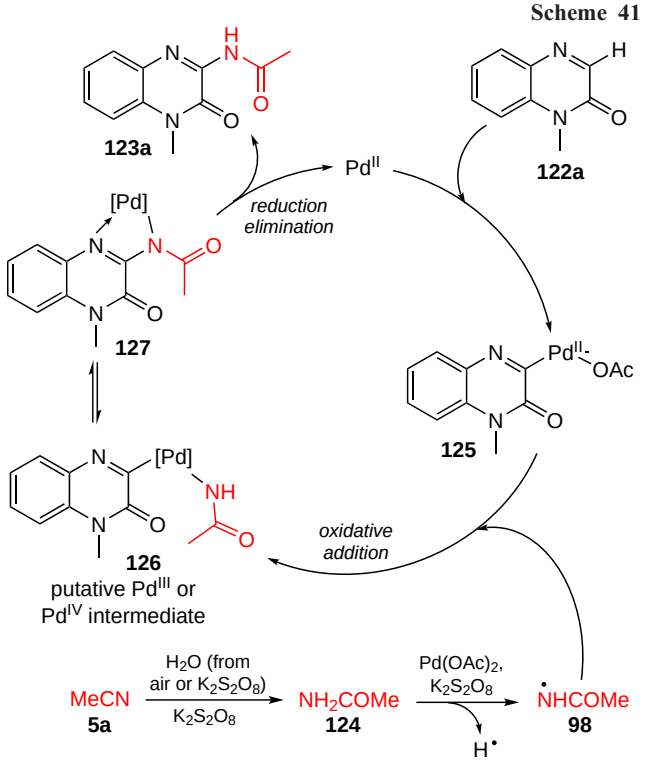
Scheme 40



Examples of products



Scheme 41



of radical **131** occurs to give cation **132**, with reacts with acetonitrile *via* a nucleophilic addition mechanism to generate an intermediate nitrilium cation **133**. At the final step, cation **133** is hydrolyzed to the desired amide **129a**.

4.3. Amidation of quinoline *N*-oxides

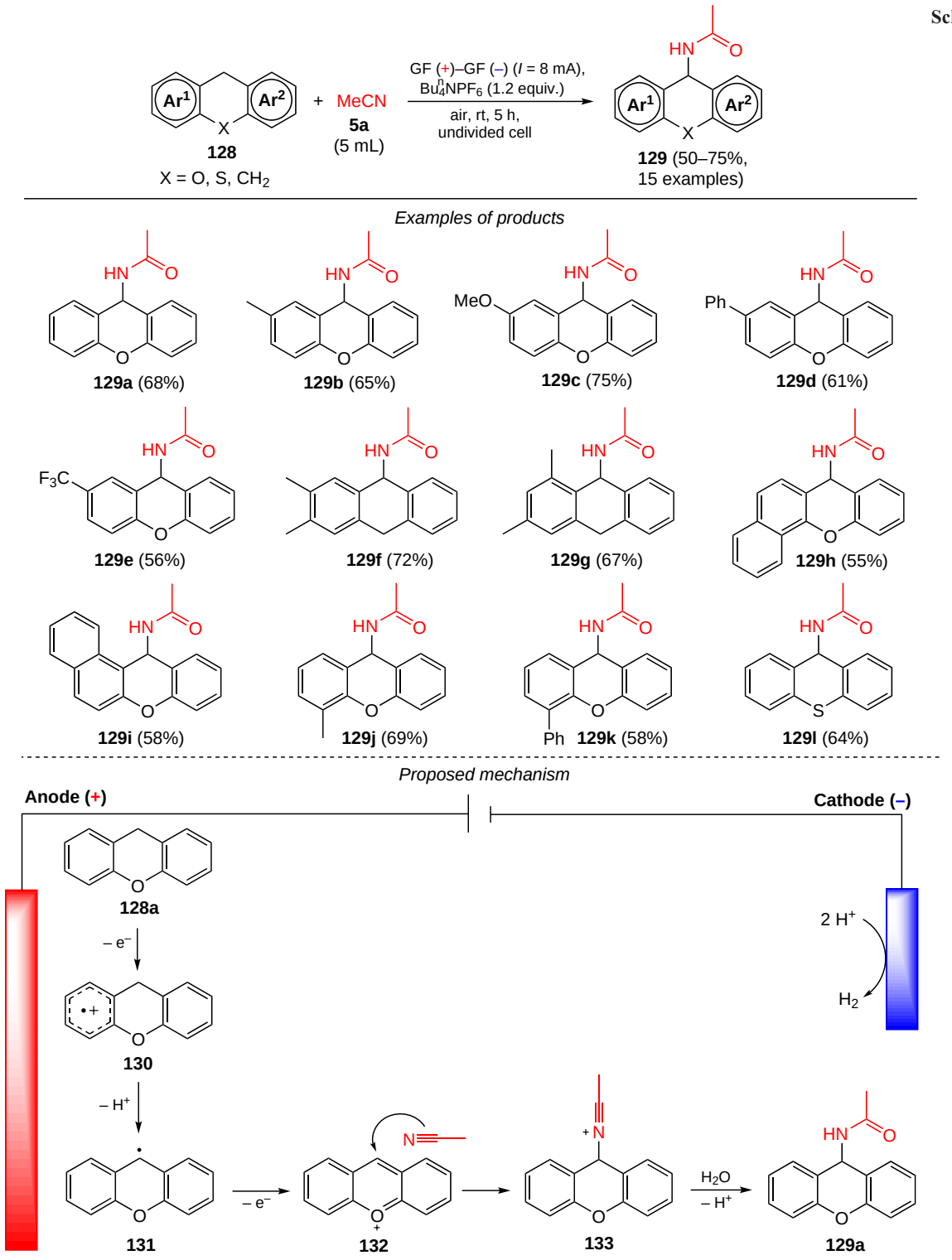
In 2018, He and co-workers[114] developed an approach to *N*-acylated 2-aminoquinolines **135**, **136** based on quinoline *N*-oxides **134** using the [BMIM(SO_3H)][OTf] (**IL1**) ionic liquid as a Brønsted acid (Scheme 43).[†] The reaction proceeds with 100% atom economy and high regioselectivity, and can involve aliphatic and aromatic nitriles, as well as diversely functionalized quinoline *N*-oxides. The advantage of this method is that the ionic liquid can be reused in subsequent reactions.

Based on these data and previous studies,[115,116] a probable mechanism for the process was proposed (Scheme 44). First, nitrile **5** is protonated by ionic liquid **IL1** to generate cation **137**. Then, the nucleophilic oxygen atom of quinoline *N*-oxide **134a** attacks carbocation **137** to form intermediate **138**, which undergoes intramolecular cyclization to yield cation **139**. Finally, heterolytic cleavage of the N–O bond in this cation leads to intermediate **140**, which undergoes deprotonation to afford the amidation product. The total amidation process involves cleavage of C–H and N–O bonds, formation of new C–N and C=O bonds, and a proton exchange between **IL1** and quinoline *N*-oxide.

An alternative method for the high-temperature amidation of quinoline *N*-oxides **134** using *p*-toluenesulfonic acid monohydrate as a promoter and commercially available benzonitriles as amidating reagents was developed by Xiao and co-workers[117] (Scheme 45). This methodology does not require large amounts of organic solvent, is highly atom-economical

[†] In Schemes 43, 45, and 47, double letter numbering is used for some products: the first letter refers to substituents in the heterocycle, the second to those in the aroyl moiety.

Scheme 42

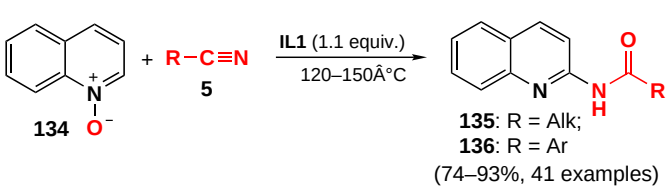


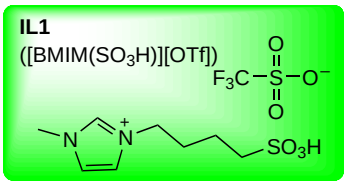
(100% with respect to quinoline *N*-oxide), has simple reaction conditions, and yields of amidation products that range from good to excellent (75–94%).

A probable mechanism of the process on the example of amidation of quinoline *N*-oxide **134a** with benzonitrile (**5b**) is shown in Scheme 46. The amidation apparently occurs *via* 1,3-dipolar cycloaddition of quinoline *N*-oxide to cation **141**, generated *in situ* under the conditions of the reaction of benzonitrile with $TsOH \cdot H_2O$,[118] affording a five-membered oxadiazolidine ring **139b**. Then, intermediate **139b** undergoes ring opening to give intermediate cationic form **140b**, which is followed by a deprotonation/aromatization, which leads to the target product **136c** and the regeneration of TsOH in the catalytic cycle.

He and co-workers[119] proposed an effective alternative to amidation of quinoline *N*-oxides in the absence of metal catalysts, bases, and reducing agents using nitriles as the amide source and methyl carbazate as the radical activator and oxygen

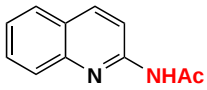
Scheme 43

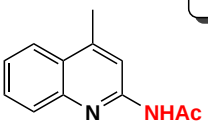


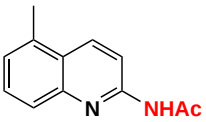


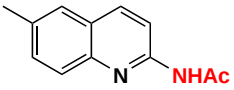
Examples of products (yield, time)

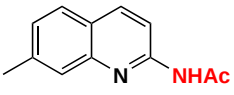
R = Me

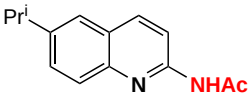

135a (88%, 14 h)

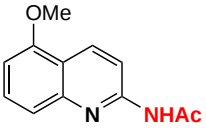

135b (90%, 12 h)

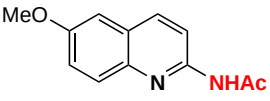

135c (87%, 12 h)

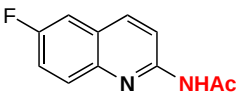

135d (78%, 12 h)

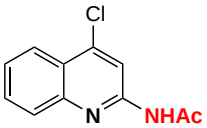

135e (83%, 12 h)

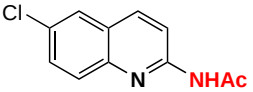

135f (91%, 12 h)

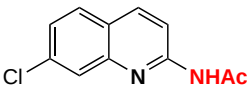

135g (87%, 10 h)

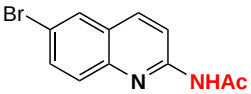

135h (82%, 9 h)

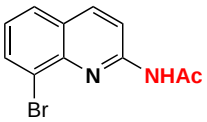

135i (86%, 10 h)

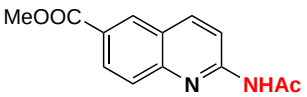

135j (90%, 14 h)

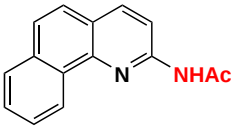

135k (92%, 12 h)


135l (89%, 12 h)

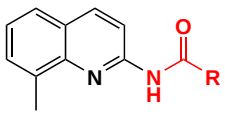

135m (85%, 10 h)


135n (86%, 12 h)


135o (93%, 8 h)

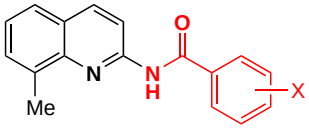

135p (75%, 15 h)

R = Alk



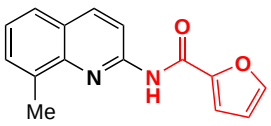
R = Me (**135q**, 91%, 8 h),
Pr^n (**135qa**, 84%, 10 h),
n-undecyl (**135qb**, 93%, 10 h),
Pr^i (**135qc**, 84%, 8 h),
$(CH_2)_2OMe$ (**135qd**, 78%, 12 h),
$(CH_2)_2Ph$ (**135qe**, 77%, 12 h),
$(CH_2)_3Cl$ (**135qf**, 82%, 12 h),
cinnamyl (**135qg**, 84%, 10 h),
cyclopropyl (**135qh**, 87%, 8 h)
cyclohexyl (**135qi**, 88%, 8 h)

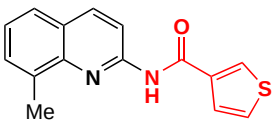
R = Ar

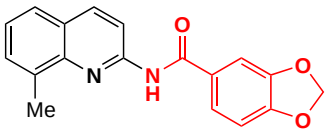


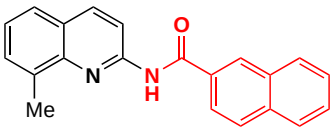
R = H (**136a**, 86%, 9 h),
2-Me (**136ab**, 84%, 9 h),
2-F (**136ac**, 93%, 12 h),
3-Me (**136ad**, 84%, 12 h),
4-Me (**136ae**, 78%, 12 h),
4-Bu^t (**136af**, 77%, 12 h),
4-F (**136ag**, 82%, 12 h),
4-Cl (**136ah**, 74%, 12 h),
4-Br (**136ai**, 88%, 12 h),
4-CF_3 (**136aj**, 81%, 12 h)

R = Het

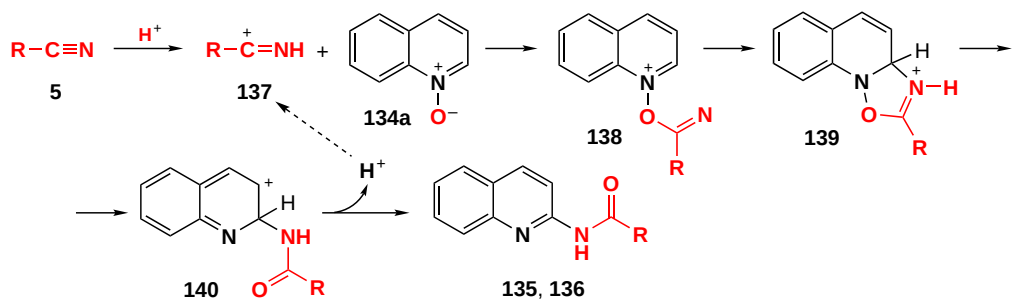

136ak (88%, 8 h)


136al (88%, 8 h)

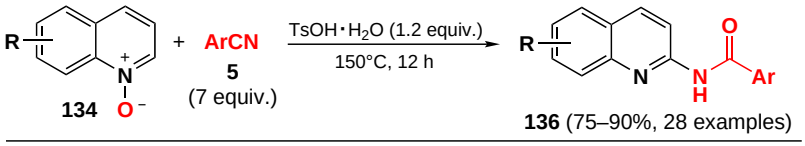

136am (86%, 12 h)


136an (87%, 12 h)

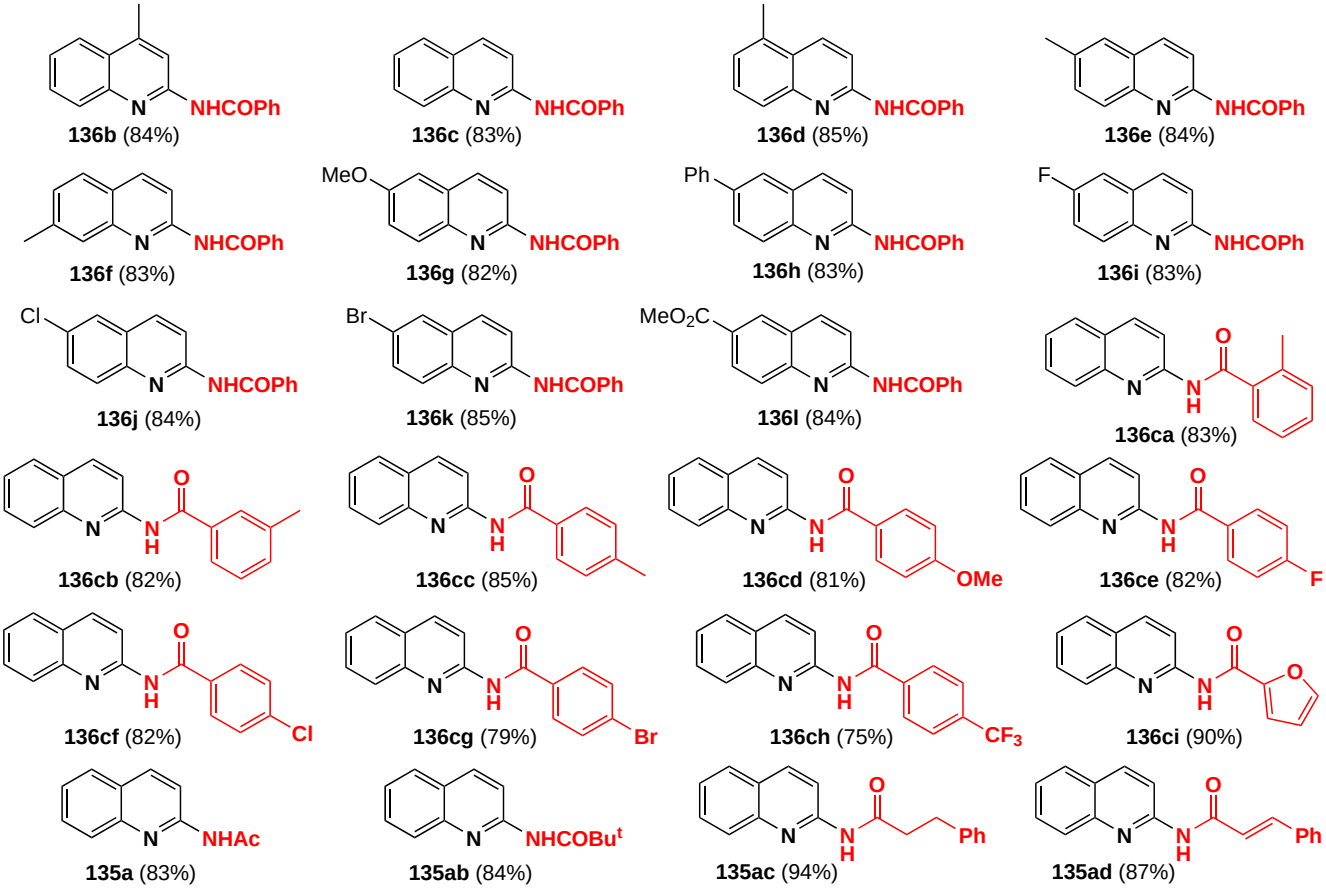
Scheme 44



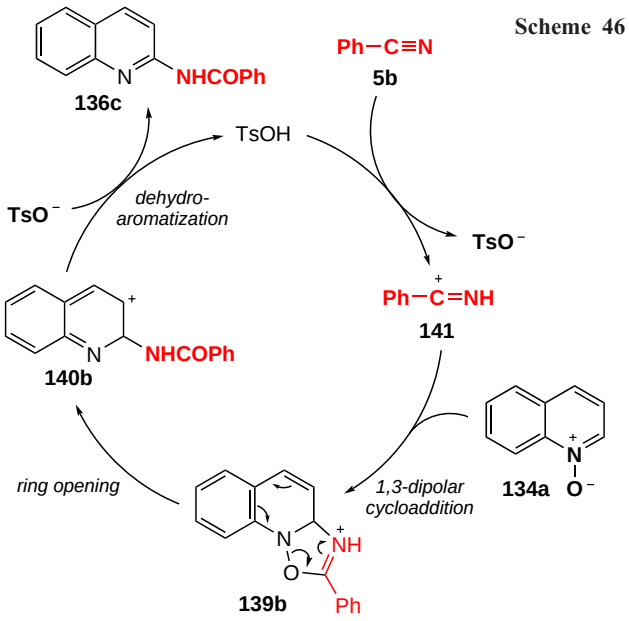
Scheme 45



Examples of products



Scheme 46



source (Scheme 47). Quinoline *N*-oxides and various functionalized nitriles readily react to furnish *N*-acylated 2-aminoquinolines in yields of up to 93%.

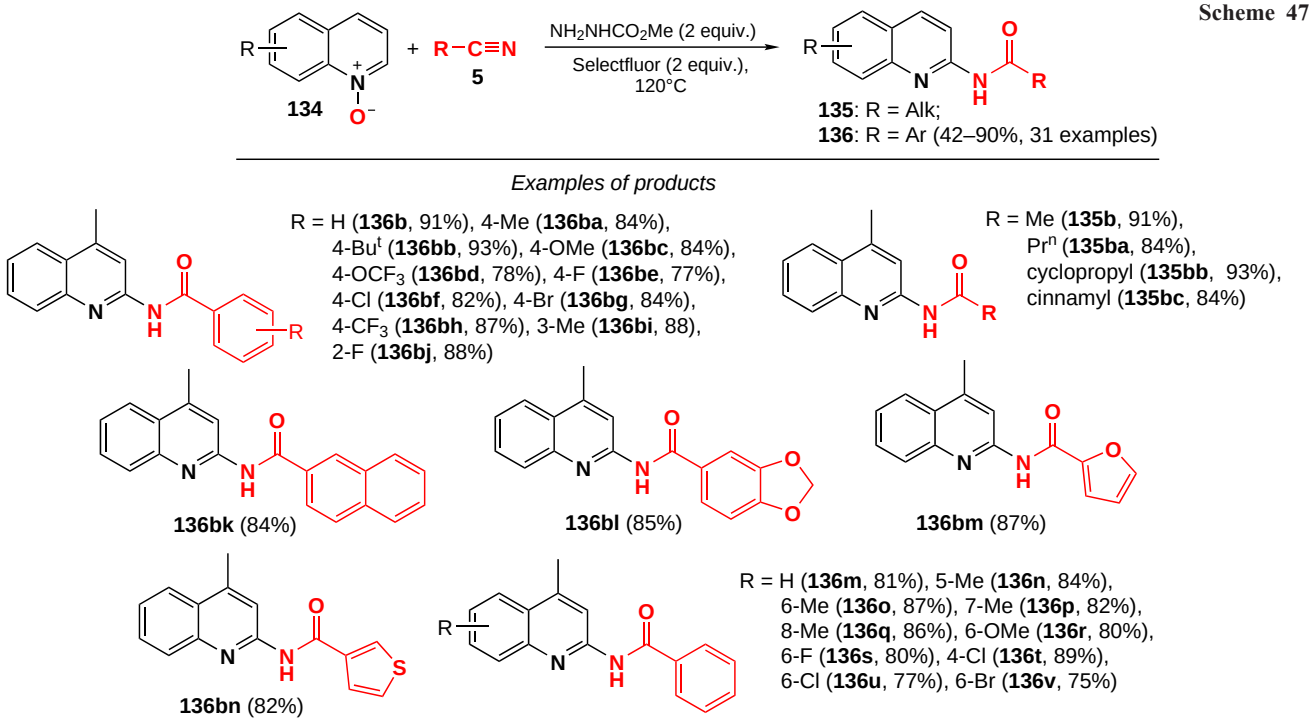
5. Other examples of Ritter-type reactions

In 2024, de Oliveira and de Mattos [120] developed an approach to the amidation of *N*-(hydroxymethyl)saccharin (**141**) with nitriles, which allowed the synthesis of various *N*-[(saccharinyl)-methyl]amides **142**, which are of interest for medicinal chemistry. Amidation products **142** are obtained in yields of up to 92% within 24 h in the presence of 9 equiv. H_2SO_4 in the absence of solvent (Scheme 48). The rate-limiting step of this reaction is the formation of the relatively unstable intermediate carbocation **143**, therefore, only a strongly acidic medium is effective for the amidation.

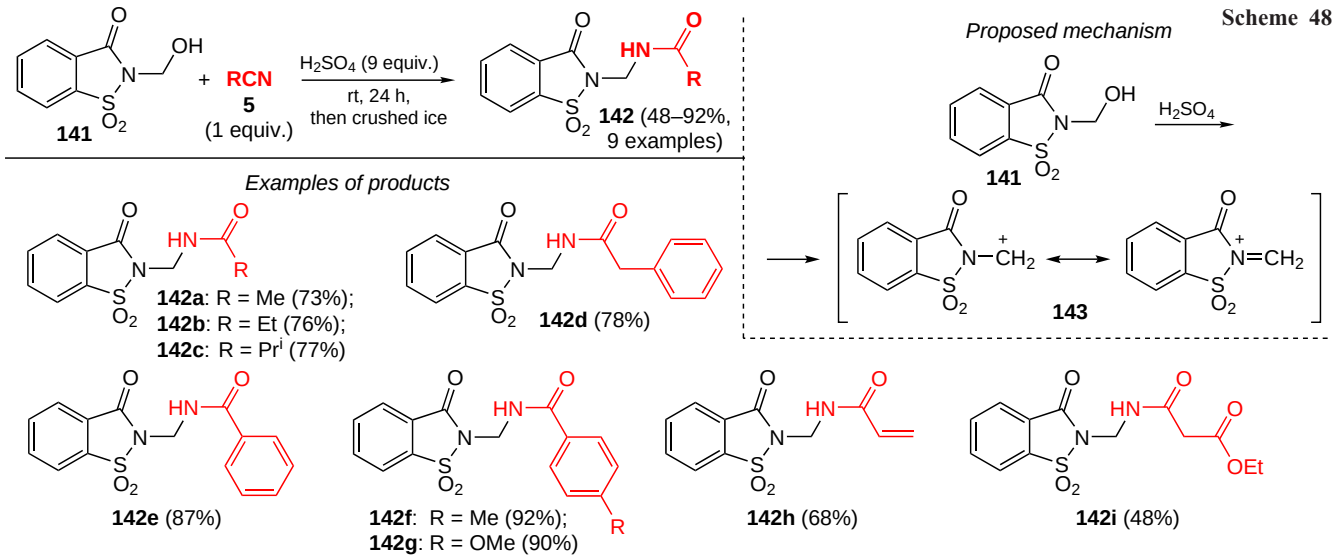
Malapaka and co-workers [121] described an acid-catalyzed three-component one-pot protocol to obtain 3-aryl-3-amidooxindoles directly from commercially available isatin derivatives **144**, nitriles, and C-nucleophiles, tertiary arylamines **145** (Scheme 49).

Various C-nucleophiles such as *N,N*-dimethylaniline (DMA), *N,N*-diethylaniline (DEA), 1-methyl-1,2,3,4-tetrahydroquinoline and 1-phenylpiperidine were used in this reaction. Tertiary amines reacted smoothly with isatin and acetonitrile upon heating to 85°C for 12–16 h in the presence of TfOH to give the amidation products **146** in high yields (up to 87%). The nucleophilic 3-chlorophenol was also subjected to a tandem Ritter reaction with isatin, furnishing the amide **146g** in low yield (52%).

Scheme 47



Scheme 48



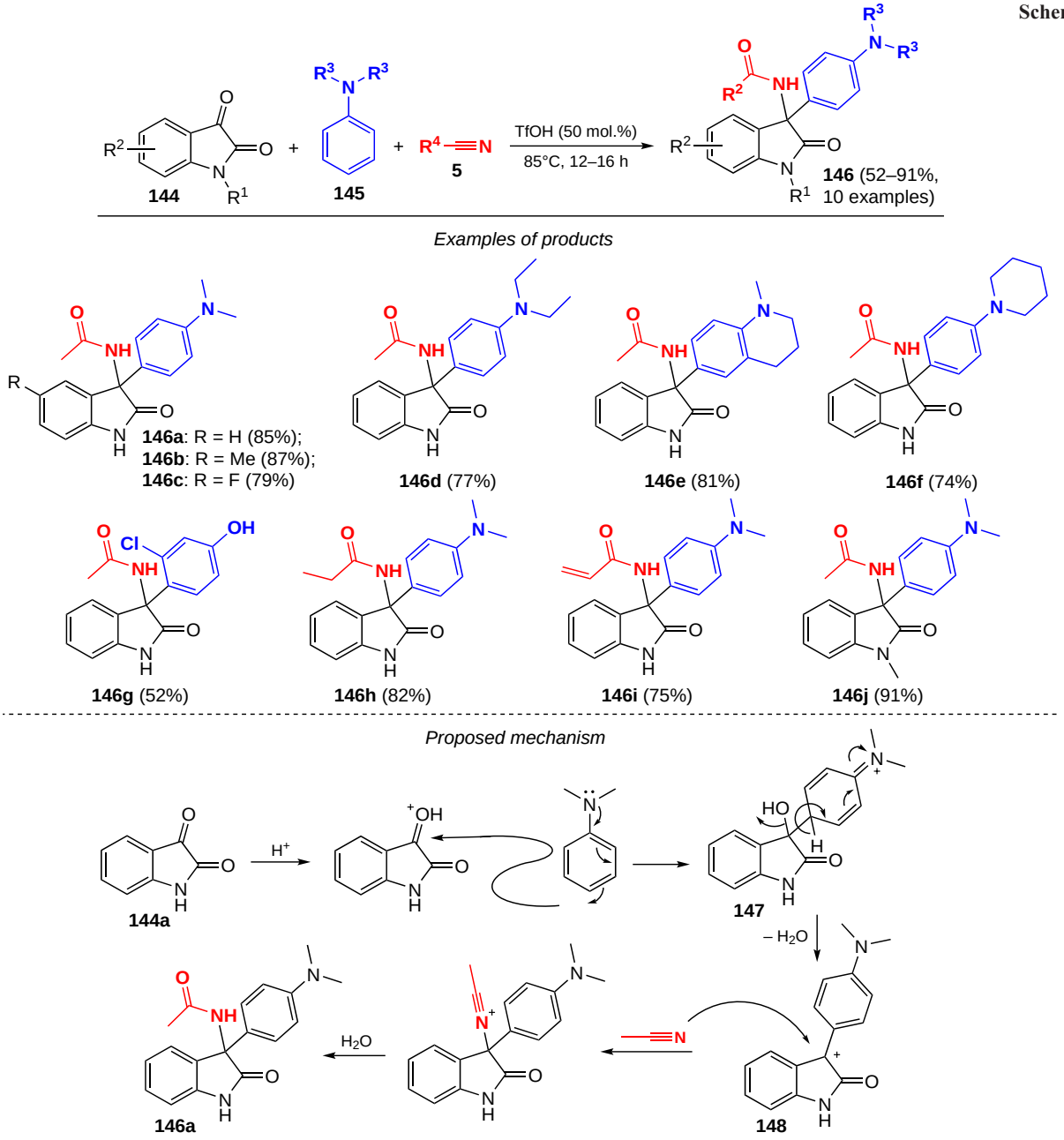
The use of propionitrile and acrylonitrile in the reaction with isatin and DMA led to the formation of amidation products in good yields (75–82%), but aromatic nitriles in this case were unreactive. *N*-Substituted isatins also undergo this reaction, as demonstrated by the example of the coupling of *N*-methylisatin with DMA and MeCN. As a result, *N*-{[3-(4-(dimethylamino)-phenyl]-1-methyl-2-oxoindolin-3-yl}acetamide (**146j**) was obtained in 91% yield.

A probable mechanism of the process is illustrated by the example of the formation of *N*-{[3-(4-(dimethylamino)phenyl]-2-oxoindolin-3-yl}acetamide (**146a**) (see Scheme 49). In the first step, the carbonyl group of isatin **144a** is protonated at position C(3). Next, the protonated form of isatin undergoes nucleophilic attack by DMA, which leads to intermediate **147**. In turn, this intermediate undergoes rearomatization under dehydrating conditions to give intermediate carbocation **148**. In the final step, nucleophilic attack of the nitrile at position C(3) followed by hydrolysis yields the amidation product.

The same research group[122] developed an acid-catalyzed multicomponent procedure for the synthesis of *N*-benzylamide derivatives from aldehydes **149**, *N*,*N*-disubstituted arylamines **145** and nitriles (Scheme 50). Aromatic aldehydes with electron-withdrawing and electron-donating groups, aliphatic and aromatic nitriles, and C-nucleophiles (DMA, DEA, 1-methyl-1,2,3,4-tetrahydroquinoline, 1-methylindoline) were used in this reaction; the yields of the amidation products **150** reached 93%. The proposed method also allows the use of β-naphthols, 1,3-dicarbonyl compounds and 1,3,5-trimethoxybenzene as C-nucleophiles.

The plausible mechanism of the reaction is presented on the example of the preparation of *N*-{[4-(dimethylamino)phenyl](phenyl)methyl}acetamide (**150a**) (Scheme 51). In the first step,

Scheme 49



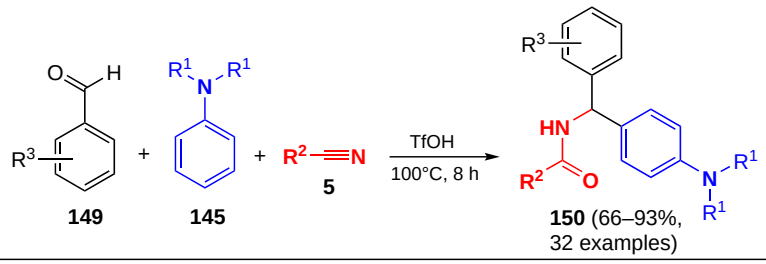
benzaldehyde undergoes protonation at the formyl oxygen atom. Next, nucleophilic addition of DMA to the protonated form of benzaldehyde occurs to give intermediate **151**. The subsequent rearomatization of **151** furnishes intermediate **152**. Intermediate **152** after dehydration undergoes nucleophilic attack by acetonitrile and water to the iminol form **153**. The resulting iminol **153** ultimately tautomerizes to the target amide **150a**.

6. Conclusion

Organic compounds containing *N*-arylamide moieties are versatile components of pharmaceuticals, agrochemicals and organic functional materials. The search for new and improvement of known approaches to their synthesis have been developing rapidly in recent years. This review summarizes various transformations that follow the Ritter-type reaction pathway and are used in the synthesis of *N*-(het)arylamides and *N*-benzylamides both under the conditions of traditional organic synthesis and in their photo-, electro- and photoelectrochemical modifications (Scheme 52). Over the last 5 years, many significant works have appeared on the synthesis of *N*-arylamides and *N*-benzylamides involving nitriles, especially under electrochemical conditions. In this case, the latest publications have considered the functionalization of not only alkyl and benzyl C(sp^3)–H bonds, but also aromatic C(sp^2)–H bonds, which previously could not be involved in such transformations. This opens up new possibilities for improving this simple and atom-efficient method, which does not require preliminary functionalization of the substrates. There are very few known examples of obtaining *N*-arylamides by direct functionalization of aromatic C(sp^2)–H bonds with nitriles, and some of them were developed only in 2023–2024.

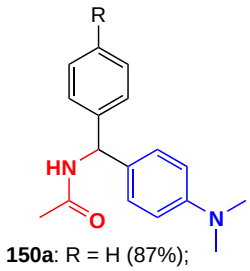
The reactions considered in this review are used, *inter alia*, for the synthesis of biologically active compounds and

Scheme 50

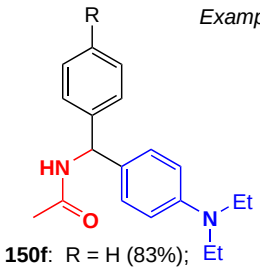


149 **145** **5** **150** (66–93%, 32 examples)

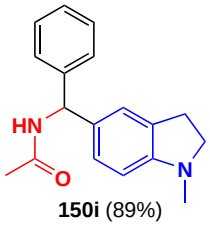
Examples of products



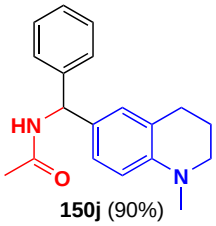
150a: R = H (87%);
150b: R = NO_2 (70%);
150c: R = Me (76%);
150d: R = OMe (93%);
150e: R = Br (76%)



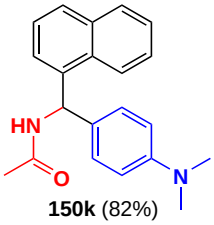
150f: R = H (83%); **150g**: R = NO_2 (89%);
150h: R = CF_3 (77%)



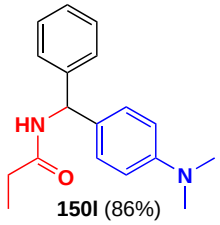
150i (89%)



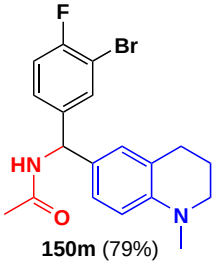
150j (90%)



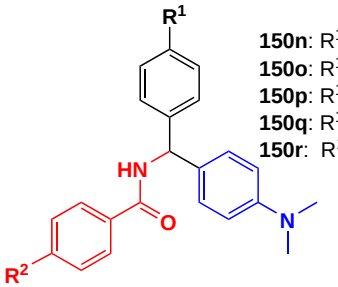
150k (82%)



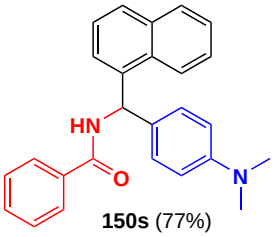
150l (86%)



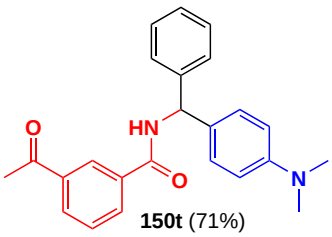
150m (79%)



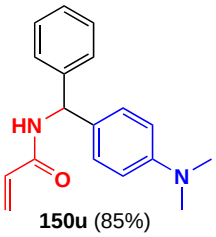
150n: R^1 = R^2 = H (88%);
150o: R^1 = R^2 = Me (90%);
150p: R^1 = Br, R^2 = Me (74%);
150q: R^1 = OMe, R^2 = NO_2 (92%);
150r: R^1 = H, R^2 = OMe (73%)



150s (77%)

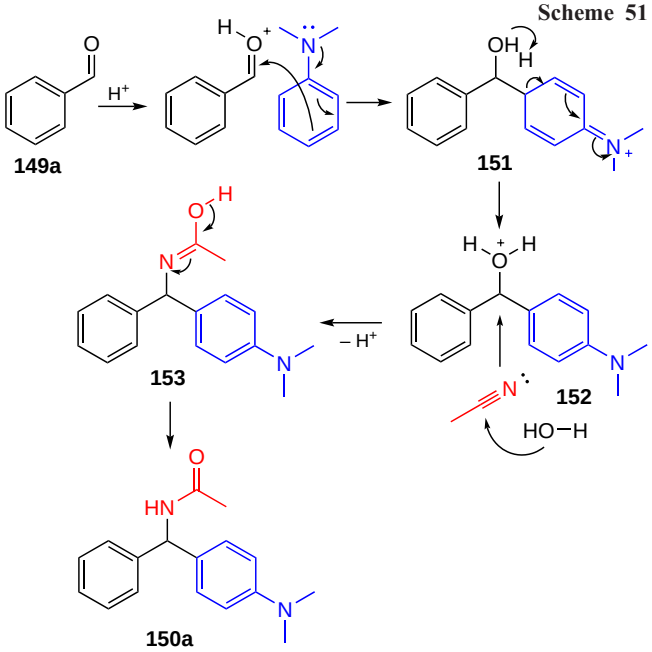


150t (71%)



150u (85%)

Scheme 51

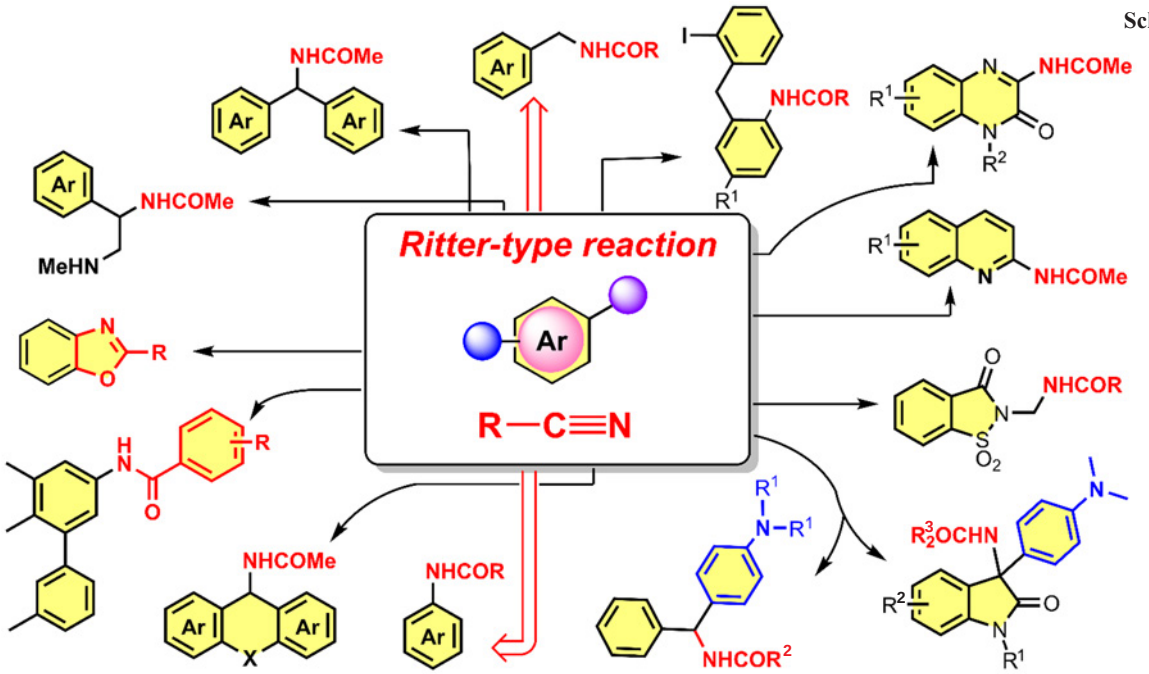


149a **151** **152** **153** **150a**

compounds with potential activity. The authors not only developed new synthetic approaches to *N*-(het)arylamides and *N*-benzylamides, but also proposed mechanisms for these processes, which undoubtedly contributes to the understanding of the essence of known reactions and the discovery of new ones and approaches intended for obtaining nitrogenous compounds of other classes.

In recent years, there has been an increase in the number of studies in which the Ritter amidation reaction is a key step, indicating the expansion of the capabilities of this methodology. The involvement of more complex and multifunctional substrates, as well as the production of practically relevant biologically active molecules and natural products, emphasizes the promise of this synthetic approach. The authors hope that this review will inspire researchers to search for and develop new methods of Ritter amidation under classical conditions, as well as using electrochemistry and photoelectrochemistry. Such transformations should involve heteroaromatic substrates, examples of which are currently virtually absent, as well as complex molecules of practical value. Expanding the scope of application of the Ritter reaction and its modern versions can open new horizons in the field of organic synthesis and contribute to the development of effective pharmaceuticals and innovative materials.

Scheme 52



7. List of abbreviations and designations

The following abbreviations and designations are used in the review:

B — base,
E — potential,
I — current,
L — ligand,
CAN — cerium(IV)–ammonium nitrate,
Cat — catalyst,
CFL — compact fluorescent lamp,
CP — carbon paper,
DCE — 1,2-dichloroethane,
DDQ — 2,3-dichloro-5,6-dicyano-1,4-benzoquinone,
DEA — *N*,*N*-diethylanilin,
DMA — *N*,*N*-dimethylanilin,
DMEDA — *N*,*N*′-dimethylethylenediamine,
EPR — electron paramagnetic resonance,
GC-MS — gas chromatography–mass spectrometry,
HAT — hydrogen atom transfer,
HFIP — hexafluoropropan-2-ol,
IL — ionic liquid,
LED — light-emitting diode,
Mes — mesityl,
MSA — methanesulfonic acid,
MWCNT-CSP — multi-walled carbon nanotubes modified with *p*-toluenesulfonic acid,
NHPI — *N*-hydroxyphthalimide,
PBN — *N*-*tert*-butyl-α-phenylnitrone,
PINO — phthalimide *N*-oxyl,
PTFE — polytetrafluoroethylene,
rt — room temperature,
SET — single-electron transfer,
SS — stainless steel (electrode),
TAC — tris(*cis*-2,6-dimethylpiperidin-1-yl)cyclopropenium,
TBN — *tert*-butyl nitrite,
TEMPO — (2,2,6,6-tetramethylpiperidin-1-yl)oxyl,
TFA — trifluoroacetic acid,
TfO — trifluoromethanesulfonate (triflate),
TMS — trimethylsilyl,
TsO — *p*-toluenesulfonate (tosylate),
US — ultrasound.

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