# $CuBr/FeCl_3$ Catalysis: a Novel and Efficient Method for the Preparation of New Aryl (Iminomethyl)propargyl Ether Derivatives *via* C–H Activation of Aryl Propargyl Ethers

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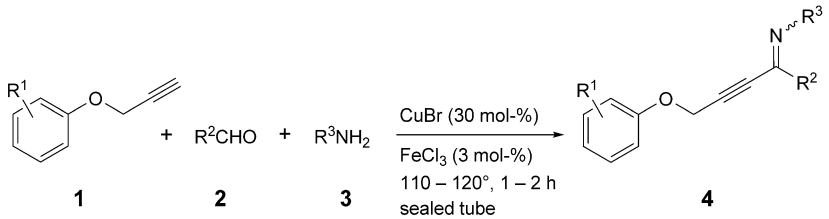
$CuBr/FeCl_3$ Catalysis as a novel and efficient method has been developed for the preparation of new aryl propargyl imine ether derivatives *via* C–H activation of aryl propargyl ethers, followed by reaction with imines generated from aldehydes and amines.

**Introduction.** – The modern trend in synthetic organic chemistry is directed to multicomponent reactions (MCRs) because of their added advantage over linear reactions in synthesis of highly complex and medicinally important compounds [1]. Notably, the propargylamine (= prop-2-yn-1-ylamine) derivatives are considered as attractive targets for their functionality and derivatization. $A^3$-Coupling of alkyne, aldehyde, and amine is one of the synthetic ways widely used to prepare propargylamine derivatives. It is mainly prepared by C–H activation of alkyne with metal, followed by reaction with imine generated *in situ* from aldehydes and amines. In former reports, $A^3$-couplings were catalyzed by Au in $H_2O$ [2], CuBr in $H_2O$ [3], $Zn(OTf)_2/R_3N$ [4], Au nanoparticles [5], *etc.* Enantioselective synthesis of propargylamine derivatives was also accomplished by using CuBr [6]. Similarly, propargylamine derivatives were prepared by Cu/Ru-catalyzed coupling of alkynes with imines [7][8]. The same concept was further exploited by *Roy et al.* [9] to prepare (iminomethyl)-propargyl ether derivatives by coupling of propargyl glycoside ethers with different aldehydes and amines using a Cu/Ru catalyst. Based on the supporting supplementary data of [9], the products are found to be 'glycoside propargyl imine ether' derivatives. However, the authors claim the products as 'glycoside propargyl amine ether derivatives'. Some of the propargylimine derivatives such as 1-azabut-1-en-3-ynes were prepared from arylsulfonyloxime [10], imidoyl chlorides [11][12], nitrones [13], alkynyl imines [14], and arylacetylene. To the best of our knowledge, except [9], no report was available for the preparation of aryl (iminomethyl)propargyl ether derivatives, and thus attracted our attention. As a sequel to our endeavor in developing new catalysts and methodologies, we report here the development of a simple, efficient and environmentally benign approach for new aryl (iminomethyl)propargyl ether derivatives using $CuBr/FeCl_3$ as catalyst.

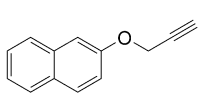
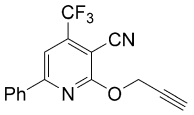
**Aryl (Iminomethyl)propargyl Ethers.** – Aryl propargyl ethers **1** were prepared under *Claisen* conditions ($K_2CO_3$, propargyl bromide, acetone). The multicomponent

reaction of aryl aldehydes **2**, aryl amines **3**, and propargyl ethers **1** in presence of CuBr/$FeCl_3$ catalyst gave the new aryl (iminomethyl)propargyl ether derivatives **4** (*Table 4*). The synthetic sequence involves formation of imine at 60°, followed by reaction with activated alkyne at 120°. To test the versatility of the reaction, diverse substituted aryl aldehydes and aryl amines were reacted with the activated alkyne leading to the corresponding products in high yields. Further, the described reaction was also attempted with heptanal, however, with no success. Aliphatic aldimines are less stable and show low reactivity towards carbon nucleophiles [3]. It is noteworthy to mention that the reaction without the catalytically active couple, *i.e.*, only with CuBr or $FeCl_3$, did not lead to the formation of product. Moreover, the ether **1a**, on reaction with different aldehydes and aniline using CuBr/$FeCl_3$ couple at room temperature or at reflux temperature in MeCN, gave also no reaction. At high temperature, *i.e.*, 150°, ether **1** was cleaved to phenol. In the *Table*, the products formed in each reaction are compiled.

Table. *Synthesis of Aryl (Iminomethyl)propargyl Ether Derivatives*




| *Entry* | **1** | $R^1$ | **2** | $R^2$ | **3** | $R^3$ | **4** | Yield [%] |
|---|---|---|---|---|---|---|---|---|
| *1* | **1a** | H | **2a** | Ph | **3a** | Ph | **4a** | 78 |
| *2* | **1a** | H | **2b** | 4-Cl–$C_6H_4$ | **3a** | Ph | **4b** | 72 |
| *3* | **1a** | H | **2c** | 4-Br–$C_6H_4$ | **3a** | Ph | **4c** | 75 |
| *4* | **1a** | H | **2d** | 4-MeO–$C_6H_4$ | **3a** | Ph | **4d** | 70 |
| *5* | **1a** | H | **2e** | 4-Me–$C_6H_4$ | **3a** | Ph | **4e** | 70 |
| *6* | **1a** | H | **2f** | 4-F–$C_6H_4$ | **3a** | Ph | **4f** | 75 |
| *7* | **1a** | H | **2a** | Ph | **3b** | 4-Me–$C_6H_4$ | **4g** | 78 |
| *8* | **1a** | H | **2a** | Ph | **3c** | 4-F–$C_6H_4$ | **4h** | 75 |
| *9* | **1a** | H | **2a** | Ph | **3d** | 2-F–$C_6H_4$ | **4i** | 75 |
| *10* | **1a** | 4-Me | **2a** | Ph | **3a** | Ph | **4j** | 72 |
| *11* | **1b**[a]) | – | **2a** | Ph | **3a** | Ph | **4k** | 78 |
| *12* | **1a** | H | **2g** | Hexyl | **3a** | Ph | No reaction | |
| *13* | **1c**[b]) | – | **2a** | Ph | **3a** | Ph | **4l** | 42 |
| *14* | **1c**[b]) | – | **2e** | 4-Me–$C_6H_4$ | **3a** | Ph | **4m** | 40 |

[a])  . [b]) 

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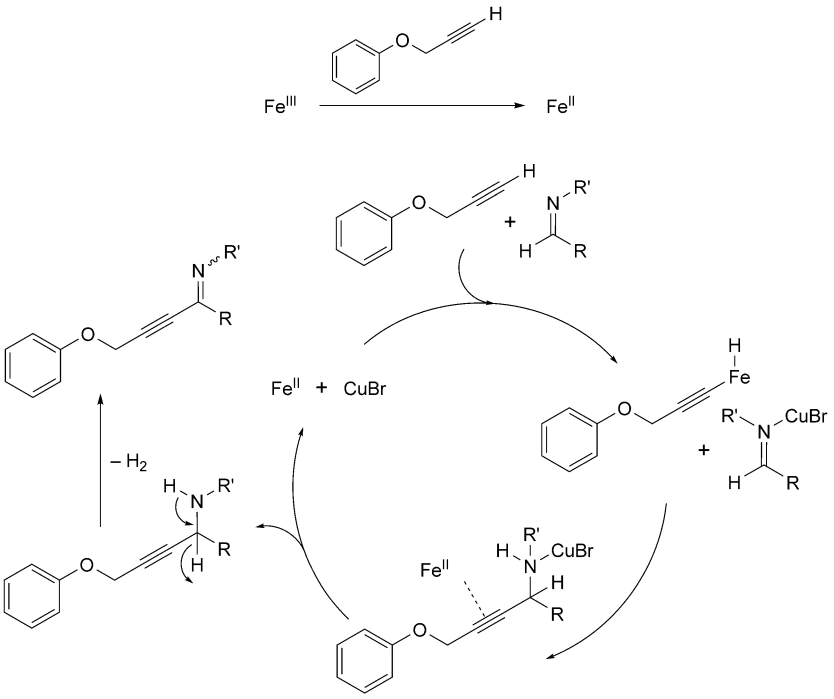
A possible mechanism is proposed which involves the activation of the terminal C–H bond of the alkyne by $FeCl_3$, possibly generated *in situ* by reaction with $Fe^{III}$ and the activation of the imine with copper. The iron-activated intermediate immediately

reacts with the activated imine to yield the nucleophilic addition product of the aryl (iminomethyl)propargyl ether derivative. It seems to be quite unstable in the reaction medium used, and thus reductive elimination of two H-atoms takes place resulting in the product, *i.e.*, the aryl (iminomethyl)propargyl ether derivative. The proposed pathway for imine addition *via* C–H activation is outlined in the *Scheme.*

Scheme. *Proposed Mechanism for Imine Addition* via *C–H Activation*




In conclusion, we have developed an efficient way for the synthesis of new aryl (iminomethyl)propargyl ether derivatives by CuBr/$FeCl_3$-catalyzed $A^3$-coupling of aryl propargyl ethers, aldehydes, and amines. The reaction was established with diverse aldehydes and amines for general application.

Authors are grateful to Dr. *J. S. Yadav*, Director, IICT for his constant encouragement, and *G. S. K.*, *C. K.*, *P. S. R.*, and *B. V.* thank the *Council of Scientific and Industrial Research* (*CSIR*), India, for the financial support in the form of JRF fellowship.

## Experimental Part

*General.* All of the reactions were monitored by TLC on precoated silica gel *60* $F_{254}$; spots were visualized with UV light. Column chromatography (CC): *Merck* silica gel (60–120 mesh). M.p.: *Casia-Siamia* (*VMP-AM*) melting-point apparatus; uncorrected. IR Spectra: *Perkin-Elmer FT-IR 240-C* spectrophotometer; KBr discs; in $cm^{-1}$. $^1H$- and $^{13}C$-NMR spectra: *Bruker AV* (300 and 75 MHz, resp.), in $CDCl_3$, TMS as internal standard; $\delta$ in ppm, $J$ in Hz. Electron impact (EI) and chemical ionization (CI) MS: *VG 7070 H* instrument; at 70 eV; *m*/*z*. CHN Analyses: *Vario EL* analyzer.

*Typical Procedure for the A³-Coupling Reaction.* A mixture of aldehyde (1 mmol) and arylamine (1.1 mmol) was stirred at 60° for 1 h. Then, aryl propargyl ether (1.1 mmol) in MeCN (2 ml) was added, followed by CuBr (30 mol-%) and $FeCl_3$ (3 mol-%) in a sealed tube. It was externally heated at 110–120° for 1–2 h, and after completion of the reaction, the mixture was diluted with $CH_2Cl_2$ (15 ml) and washed with $H_2O$ (30 ml). The org. layer was separated, dried ($Na_2SO_4$), and concentrated under vacuum. The residue thus obtained was purified by CC (hexane/AcOEt 3:1).

N-*(4-Phenoxy-1-phenylbut-2-yn-1-ylidene)benzenamine* (**4a**; *Table*, *Entry 1*). M.p. 120–122°. IR: 2154 (–C≡C–), 1601 (C=N), 1245 (C–O–C). $^1$H-NMR: 5.53 (*s*, $CH_2O$); 6.95–7.04 (*m*, 3 arom. H); 7.26–7.32 (*m*, 2 arom. H); 7.44–7.56 (*m*, 4 arom. H); 7.71 (*t*, *J* = 8.3, 1 arom. H); 7.98 (*t*, *J* = 8.3, 2 arom. H); 8.14–8.21 (*m*, 3 arom. H). $^{13}$C-NMR: 66.9; 74.3; 79.1; 114.8; 117.2; 121.4; 122.6; 124.8 126.4; 127.5; 127.7; 128.7; 129.1; 129.3; 129.4; 129.6; 130.4; 139.4; 142.5; 148.2; 157.1; 158.3. MS: 311 ($M^{+\bullet}$), 218 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{22}H_{17}NO$ (311.38): C 84.86, H 5.50, N 4.50; found: C 84.57, H 5.42, N 4.41.

N-*[1-(4-Chlorophenyl)-4-phenoxybut-2-yn-1-ylidene]benzenamine* (**4b**; *Table*, *Entry 2*). M.p. 108–110°. IR: 2164 (–C≡C–), 1600 (C=N), 1240 (C–O–C). $^1$H-NMR: 5.53 (*s*, $CH_2O$); 6.94–7.05 (*m*, 3 arom. H); 7.28–7.32 (*m*, 2 arom. H); 7.45 (*t*, *J* = 7.6, 1 arom. H); 7.72 (*d*, *J* = 8.3, 2 arom. H); 7.78 (*t*, *J* = 6.8, 1 arom. H); 7.83 (*t*, *J* = 7.6, 2 arom. H); 8.12 (*d*, *J* = 8.3, 1 arom. H); 8.21 (*d*, *J* = 8.3, 1 arom. H). MS: 345 ($M^{+\bullet}$), 252 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{22}H_{16}ClNO$ (345.82): C 76.41, H 4.66, N 4.05; found: C 76.04, H 4.60, N 3.93.

N-*[1-(4-Bromophenyl)-4-phenoxybut-2-yn-1-ylidene]benzenamine* (**4c**; *Table*, *Entry 3*). M.p. 103–105°. IR: 2163 (–C≡C–), 1603 (C=N), 1255 (C–O–C). $^1$H-NMR: 5.52 (*s*, $CH_2O$); 6.98–7.09 (*m*, 3 arom. H); 7.28–7.31 (*m*, 2 arom. H); 7.54 (*t*, *J* = 7.6, 1 arom. H); 7.62 (*d*, *J* = 8.3, 2 arom. H); 7.73 (*t*, *J* = 6.8, 1 arom. H); 7.96 (*t*, *J* = 7.6, 2 arom. H); 8.23 (*d*, *J* = 8.3, 2 arom. H); 8.31 (*d*, *J* = 8.3, 1 arom. H). MS: 390 ($M^{+\bullet}$), 297 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{22}H_{16}BrNO$ (390.27): C 67.71, H 4.13, N 3.59; found: C 68.10, H 4.04, N 3.47.

N-*[1-(4-Methoxyphenyl)-4-phenoxybut-2-yn-1-ylidene]benzenamine* (**4d**; *Table*, *Entry 4*). M.p. 106–108°. IR: 2153 (–C≡C–), 1601 (C=N), 1264 (C–O–C). $^1$H-NMR: 2.61 (*s*, MeO); 5.53 (*s*, $CH_2O$); 6.95 (*t*, *J* = 6.7, 1 arom. H); 7.01–7.12 (*m*, 2 arom. H); 7.25–7.30 (*m*, 2 arom. H); 7.41 (*t*, *J* = 6.7, 1 arom. H); 7.48–7.53 (*m*, 2 arom. H); 7.56 (*d*, *J* = 8.7, 1 arom. H); 7.81 (*d*, *J* = 8.7, 2 arom. H); 8.08 (*d*, *J* = 8.7, 1 arom. H); 8.15 (*d*, *J* = 8.7, 2 arom. H). MS: 341 ($M^{+\bullet}$), 248 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{23}H_{19}NO_2$ (341.4): C 80.92, H 5.61, N 4.10; found: C 80.76, H 5.52, N 3.98.

N-*[1-(4-Methylphenyl)-4-phenoxybut-2-yn-1-ylidene]benzenamine* (**4e**; *Table*, *Entry 5*). M.p. 103–105°. IR: 2142 (–C≡C–), 1608 (C=N), 1252 (C–O–C). $^1$H-NMR: 2.46 (*s*, Me); 5.57 (*s*, $CH_2O$); 6.91–7.12 (*m*, 3 arom. H); 7.29–7.34 (*m*, 4 arom. H); 7.54 (*t*, *J* = 7.6, 1 arom. H); 7.77 (*t*, *J* = 7.6, 1 arom. H); 7.92 (*d*, *J* = 8.3, 2 arom. H); 8.08 (*d*, *J* = 8.3, 2 arom. H); 8.23 (*d*, *J* = 9.1, 1 arom. H). MS: 325 ($M^{+\bullet}$), 232 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{23}H_{19}NO$ (325.4): C 84.89, H 5.89, N 4.30; found: C 85.04, H 5.80, N 4.28.

N-*[1-(4-Fluorophenyl)-4-phenoxybut-2-yn-1-ylidene]benzenamine* (**4f**; *Table*, *Entry 6*). M.p. 102–104°. IR: 2158 (–C≡C–), 1603 (C=N), 1252 (C–O–C). $^1$H-NMR: 5.54 (*s*, $CH_2O$); 6.98–7.23 (*m*, 4 arom. H); 7.31–7.46 (*m*, 2 arom. H); 7.58 (*d*, *J* = 7.6, 1 arom. H); 7.66 (*d*, *J* = 8.3, 2 arom. H); 7.74 (*t*, *J* = 6.8, 1 arom. H); 7.92 (*d*, *J* = 8.3, 1 arom. H); 8.03 (*d*, *J* = 8.3, 2 arom. H); 8.21 (*d*, *J* = 8.3, 1 arom. H). MS: 329 ($M^{+\bullet}$), 236 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{22}H_{16}FNO$ (329.37): C 80.23, H 4.90, N 4.25; found: C 80.39, H 4.81, N 4.14.

*4-Methyl*-N-*(4-phenoxy-1-phenylbut-2-yn-1-ylidene)benzenamine* (**4g**; *Table*, *Entry 7*). M.p. 101–103°. IR: 2162 (–C≡C–), 1605 (C=N), 1265 (C–O–C). $^1$H-NMR: 2.54 (*s*, Me) 5.56 (*s*, $CH_2O$); 6.93 (*t*, *J* = 7.5, 1 arom. H); 7.09 (*d*, *J* = 8.4, 2 arom. H); 7.18–7.32 (*m*, 2 arom. H); 7.43 (*t*, *J* = 7.5, 1 arom. H); 7.42–7.56 (*m*, 3 arom. H); 7.78 (*d*, *J* = 8.4, 2 arom. H); 8.19 (*d*, *J* = 8.4, 1 arom. H); 8.26 (*d*, *J* = 7.5, 2 arom. H). MS: 325 ($M^{+\bullet}$), 232 ([*M* – OPh]$^{+\bullet}$). Anal. calc. for $C_{23}H_{19}NO$ (325.4): C 84.89, H 5.89, N 4.30; found: C 85.01, H 5.81, N 4.18.

*4-Fluoro*-N-*(4-phenoxy-1-phenylbut-2-yn-1-ylidene)benzenamine* (**4h**; *Table*, *Entry 8*). M.p. 105–107°. IR: 2155 (–C≡C–), 1600 (C=N), 1262 (C–O–C). $^1$H-NMR: 5.52 (*s*, $CH_2O$); 6.96 (*t*, *J* = 7.5, 1 arom. H); 7.09 (*s*, *J* = 8.4, 1 arom. H); 7.11–7.31 (*m*, 2 arom. H); 7.43 (*t*, *J* = 7.5, 1 arom. H); 7.48–7.54 (*m*, 4 arom. H); 7.82 (*d*, *J* = 8.4, 2 arom. H); 8.18 (*d*, *J* = 8.4, 1 arom. H); 8.25 (*d*, *J* = 7.5, 2 arom. H). MS: 329

($M^{+\bullet}$), 236 ($[M - OPh]^{+\bullet}$). Anal. calc. for $C_{22}H_{16}FNO$ (329.37): C 80.23, H 4.90, N 4.25; found: C 80.37, H 4.81, N 4.14.

*2-Fluoro*-N-*(4-phenoxy-1-phenylbut-2-yn-1-ylidene)benzenamine* (**4i**; *Table*, *Entry 9*). M.p. 108–110°. IR: 2153 (–C≡C–), 1600 (C=N), 1258 (C–O–C). $^1$H-NMR: 5.53 (*s*, $CH_2O$); 6.95 (*t*, *J* = 7.5, 1 arom. H); 7.06 (*d*, *J* = 8.4, 2 arom. H); 7.18–7.30 (*m*, 2 arom. H); 7.43 (*t*, *J* = 7.5, 1 arom. H); 7.48–7.52 (*m*, 3 arom. H); 7.88 (*d*, *J* = 7.5, 2 arom. H); 8.18 (*d*, *J* = 8.4, 1 arom. H); 8.29 (*d*, *J* = 7.5, 2 arom. H). MS: 329 ($M^{+\bullet}$), 236 ($[M - OPh]^{+\bullet}$). Anal. calc. for $C_{22}H_{16}FNO$ (329.37): C 80.23, H 4.90, N 4.25; found: C 80.07, H 4.82, N 4.13.

N-*[4-(4-Methylphenoxy)-1-phenylbut-2-yn-1-ylidene]benzenamine* (**4j**; *Table*, *Entry 10*). M.p. 106–108°. IR: 2156 (–C≡C–), 1608 (C=N), 1252 (C–O–C). $^1$H-NMR: 2.56 (*s*, Me); 5.53 (*s*, $CH_2O$); 6.92–7.20 (*m*, 3 arom. H); 7.29–7.31 (*m*, 2 arom. H); 7.42 (*t*, *J* = 7.6, 1 arom. H); 7.49–7.60 (*m*, 3 arom. H); 7.74 (*d*, *J* = 8.3, 2 arom. H); 8.12 (*d*, *J* = 8.3, 2 arom. H); 8.28 (*d*, *J* = 7.6, 1 arom. H). MS: 325 ($M^{+\bullet}$), 218 ($[M - OPh]^{+\bullet}$). Anal. calc. for $C_{23}H_{19}NO$ (325.4): C 84.89, H 5.89, N 4.30; found: C 84.73, H 5.80, N 4.18.

N-*[4-(Naphthalen-2-yloxy)-1-phenylbut-2-yn-1-ylidene]benzenamine* (**4k**; *Table*, *Entry 11*). M.p. 130–132°. IR: 2151 (–C≡C–), 1607 (C=N), 1242 (C–O–C). $^1$H-NMR: 5.68 (*s*, $CH_2O$); 7.10–7.29 (*m*, 3 arom. H); 7.32–7.49 (*m*, 2 arom. H); 7.52–7.67 (*m*, 2 arom. H); 7.69 (*t*, *J* = 7.9, 1 arom. H); 7.76–7.96 (*m*, 4 arom. H); 8.11 (*t*, *J* = 8.8, 2 arom. H); 8.21–8.34 (*m*, 2 arom. H); 8.38 (*d*, *J* = 8.8, 1 arom. H). $^{13}$C-NMR : 67.1; 74.6; 78.4; 107.4; 117.5; 118.9; 122.9; 124.2; 125.0; 126.7; 127.0; 127.6; 127.7; 127.8; 129.0; 129.1; 129.5; 129.7; 129.9; 130.7; 134.5; 139.6; 142.6; 148.4; 156.4; 157.4. MS: 361 ($M^{+\bullet}$), 218 ($[M - OPh]^{+\bullet}$). Anal. calc. for $C_{26}H_{19}NO$ (361.44): C 86.40, H 5.30, N 3.88; found: C 86.24, H 5.21, N 3.76.

*6-Phenyl-2-{[4-phenyl-4-(phenylimino)but-2-yn-1-yl]oxy}-4-(trifluoromethyl)-3-pyridinecarbonitrile* (**4l**; *Table*, *Entry 13*). M.p. 230–236°. IR: 2153 (–C≡C–), 2224 (–C≡N), 1605 (C=N), 1245 (C–O–C). $^1$H-NMR: 6.01 (*s*, $CH_2O$); 7.31 (*d*, *J* = 7.4, 2 arom. H); 7.39–7.46 (*m*, 4 arom. H); 7.54 (*d*, *J* = 7.4, 2 arom. H); 7.61 (*t*, *J* = 7.4, 1 arom. H); 7.69 (*s*, 1 arom. H); 7.78 (*d*, *J* = 7.4, 2 arom. H); 8.12 (*m*, 2 arom. H); 8.36 (*d*, *J* = 7.4, 2 arom. H). MS: 481 ($M^{+\bullet}$), 218 ($[M - OPh]^{+\bullet}$). Anal. calc. for $C_{29}H_{18}F_3N_3O$ (481.47): C 72.34, H 3.77, N 8.73; found: C 72.44, H 3.69, N 8.61.

*2-{[4-(4-Methylphenyl)-4-(phenylimino)but-2-yn-1-yl]oxy}-6-phenyl-4-(trifluoromethyl)-3-pyridinecarbonitrile* (**4m**; *Table*, *Entry 14*). M.p. 241–246°. IR: 2151 (–C≡C–), 2224 (–C≡N), 1607 (C=N), 1242 (C–O–C). $^1$H-NMR: 2.46 (*s*, Me); 6.09 (*s*, $CH_2O$); 7.32 (*d*, *J* = 7.4, 2 arom. H); 7.42–7.50 (*m*, 4 arom. H); 7.56 (*d*, *J* = 7.4, 2 arom. H); 7.69 (*s*, 1 arom. H); 7.77 (*d*, *J* = 7.4, 2 arom. H); 8.15 (*t*, *J* = 7.6, 1 arom. H); 8.20–8.32 (*m*, 3 arom. H). MS: 495 ($M^{+\bullet}$), 232 ($[M - OPh]^{+\bullet}$). Anal. calc. for $C_{30}H_{20}F_3N_3O$ (495.49): C 72.72, H 4.07, N 8.48; found: C 72.80, H 4.01, N 8.36.

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*Received February 17, 2011*