# DBU-promoted $Cu(OAc)_2 \cdot H_2O$ -catalysed coupling reactions of aryl iodides and sodium azide

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An efficient and simple protocol for the synthesis of aryl azides by the coupling of aryl iodides with sodium azide, in good to excellent yields in DMSO at 95 °C under catalysis by  $Cu(OAc)_2 \cdot H_2O$  and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), has been established. The optimised loadings of  $Cu(OAc)_2 \cdot H_2O$  and DBU were 10 mol% and 15 mol% respectively.

Keywords: Cu(OAc), H,O, 1,8-diazabicyclo[5.4.0] undec-7-ene, aryl azides, dimethyl sulfoxide

Aryl azides, important intermediates in chemical transformations, have gained much attention for the synthesis of various nitrogen-containing compounds,<sup>1–5</sup> organic and bioorganic materials<sup>6–8</sup> and polymers.<sup>9–12</sup> More interestingly, aryl azides are also well known for their ability to be made with high specific radioactivities as photoaffinity labels.<sup>13,14</sup>

Due to the widespread use of aryl azides in various fields, great efforts have been made to establish synthetic protocols.<sup>15</sup> The starting materials for the synthesis of aryl azides have generally included aryl amines,16-21 activated aryl halides22 or aryl boronic acids.<sup>23–27</sup> The synthesis of aryl azides by the reaction of unactivated aryl halides with sodium azides is still a challenge. In 2004, Ma and co-workers first reported the coupling reaction of aryl halides (I, Br) with sodium azide catalysed by CuI/L-proline, which could work at a relatively low temperature to provide aryl azides in good to excellent yields in dimethyl sulfoxide (DMSO).28,29 After this, a rapid synthesis of aryl azides from the corresponding aryl halides catalysed by CuI/diamine was reported by Liang and coworkers.<sup>30</sup> In 2009, Molander and co-workers established a protocol using CuBr as catalyst in the presence of different amines like L-proline or N, N'-dimethylethylenediamine as a ligand for the synthesis of aryl azides.<sup>31</sup> Other Cu(I) complexes have also been used as catalysts. In 2014, an efficient catalytic system based on Cu<sub>2</sub>O/tetraethylammonium prolinate was used for the reaction of aryl halides with sodium azide in the mixed solvent EtOH/ethylene glycol (7:3 in volume), which provided a variety of aromatic azides.<sup>32</sup> In the same year, Hajipour and coworkers reported a CuFeO<sub>2</sub>/L-proline mixture system for the azidation of aryl halides.<sup>33</sup> Besides a cuprous catalytic system, heterogeneous metallic Cu(0) was also used as the catalyst. In 2014, Cui and co-workers reported the synthesis of aryl azides by a coupling reaction of aryl iodides with sodium azide catalysed by heterogeneous porous Cu(0) under mild conditions in the presence of L-proline and diisopropylamine.<sup>34</sup> Recently, it has been reported that aryl azides can be synthesised using a heterogeneous porous Cu catalyst by a coupling reaction of aryl bromides with sodium azide under mild conditions.<sup>35</sup> Furthermore, in 2014, Lanke and Bhanage reported a method for the coupling of aryl iodides with sodium azide to form aryl azides using the cupric salt of 2,2,6,6-tetramethyl-3,5heptanedione as the catalyst in the absence of base in DMF at 100 °C.36 In our previous work, we reported the synthesis of 1,4-disubstituted 1,2,3-triazoles catalysed by CuI with 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU).37 Due to the

susceptibility to oxidation of Cu(I), finding a Cu(II) system for the coupling of aryl iodides with sodium azide to aryl azides is a worthwhile goal.

In continuation of our interest in Cu(I)- or Cu(II)-catalysed organic reactions,<sup>38,39</sup> we report here an efficient coupling reaction of aryl iodides with sodium azide catalysed by Cu(OAc)<sub>2</sub>·H<sub>2</sub>O in the presence of DBU for the synthesis of aryl azides.

## **Results and discussion**

The reaction between iodobenzene and sodium azide was selected as the model reaction for optimising suitable conditions for the synthesis of phenyl azide (see SAFETY CAUTION in Experimental). For screening the cupric salts, the model reaction between iodobenzene (1.0 mmol) and sodium azide (1.2 mmol) was catalysed by a cupric salt (10 mol%) in the presence of DBU (20 mol%) in DMSO (3.0 mL) at 95 °C for 5.0 h. The cupric salts used as the catalysts for screening were Cu(OAc), H<sub>2</sub>O, CuSO, 5H<sub>2</sub>O, CuCl<sub>2</sub>·2H<sub>2</sub>O, CuBr<sub>2</sub> and Cu(acac)<sub>2</sub>. The conversion of the model reaction was determined by HPLC after 30 min. As shown in Fig. 1, the conversion of the model reaction catalysed by  $Cu(OAc)_2 \cdot H_2O$ ,  $CuSO_4 \cdot 5H_2O$ , CuCl<sub>2</sub>·2H<sub>2</sub>O or CuBr<sub>2</sub> showed similar behaviour, which was different from that of the reaction catalysed by Cu(acac)<sub>2</sub>. Cu(acac), gave the lowest catalytic efficiency among the tested cupric salts. As for Cu(OAc), H<sub>2</sub>O, CuSO, 5H<sub>2</sub>O, CuCl, 2H<sub>2</sub>O and CuBr<sub>2</sub>, the conversion reached the highest level at 1.5 h and began to decrease after that. The conversion catalysed by  $Cu(OAc)_{2}$ ·H<sub>2</sub>O decreased more slowly than that catalysed by the other cupric salts. Therefore, Cu(OAc)<sub>2</sub>·H<sub>2</sub>O was selected for further investigation.



Fig. 1 Effects of the different catalysts on the conversion.

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Table 1 Screening solvents for the model reaction<sup>a</sup>

Entry	Solvent	Time (h)	Conversion (%) <sup>b</sup>
1	DMS0	1.5	93
2	DMF	5	80
3	MeOH	8	45
4	EtOH	8	40
5	H <sub>2</sub> 0	9	20
6	EtOH/H <sub>2</sub> 0 (7:3)	10	40
7	1,4-Dioxane	6	No reaction
8	THF	6	No reaction
9	Toluene	10	No reaction

°Reaction conditions: iodobenzene (1.0 mmol), sodium azide (1.2 mmol),  $Cu(OAc)_2 \cdot H_2O$  (10 mol%), DBU (20 mol%), temperature 95 °C.

<sup>b</sup>Conversions were determined by HPLC.

Next, different solvent systems were used for the model reaction in the presence of  $Cu(OAc)_2 \cdot H_2O$  (10 mol%) and DBU (20 mol%). It is noteworthy that the solvent system played an important role in the reaction. As shown in Table 1, among the tested solvents, the model reaction gave the best conversion (93%) in DMSO (Table 1, entry 1). Furthermore, although the model reaction gave a good conversion (80%) in DMF, a low amount of phenylamine was obtained (Table 1, entry 2), which was consistent with the previous reports. Consequently, DMSO was selected as the reaction solvent for further investigation.

The model reaction was then carried out at 95 °C in DMSO (3.0 mL) catalysed by Cu(OAc)<sub>2</sub>·H<sub>2</sub>O (10 mol%) in the presence of different loadings of DBU for 1.5 h. As shown in Table 2, it was found that only a trace of phenyl azide was obtained when the model reaction was carried out in the absence of DBU (Table 2, entry 1), which indicated that DBU was important for the coupling reaction due to its strong basicity and coordination ability.<sup>40,41</sup> Among the tested loadings of DBU, the best isolated yield (92%) was obtained in the presence of 15 mol% DBU (Table 2, entry 4). Furthermore, only 40% phenyl azide was obtained when the model reaction was catalysed by 5 mol% Cu(OAc)<sub>2</sub>·H<sub>2</sub>O in the presence of 15 mol% DBU (Table 2, entry 8).

Therefore, 15 mol% DBU and 10 mol% Cu(OAc)<sub>2</sub>·H<sub>2</sub>O were used for further optimisation and the reactions were carried out at different temperatures (105, 95, 80, 75 and 60 °C) in DMSO for 1.5 h. The results are summarised in Fig. 2. When the reaction was carried out at 95 °C, the highest isolated yield (92%) was obtained among the tested temperatures. When elevating the reaction temperature to 105 °C, 80% isolated yield was obtained. The slight decrease of the yield at 105 °C might be ascribed to the decomposition of phenyl azide at a high reaction temperature.<sup>42,43</sup> Decreasing the reaction temperature to 80, 75 or 60 °C, the obtained yields were only 60, 25 and 10% respectively, which indicated that a relatively high reaction temperature could facilitate the coupling reaction. Therefore, 95 °C was selected as the reaction temperature. According to the screening results obtained above, the optimised conditions are as follows: iodobenzene (1.0 mmol), sodium azide (1.2 mmol),  $Cu(OAc)_{2}$ ·H<sub>2</sub>O (10 mol%), DBU (15 mol%), DMSO (3.0 mL) and a reaction temperature of 95 °C.

Having established conditions for the coupling of aryl iodides with sodium azide, we next explored the scope and limitations of this transformation (see Table 3). It was found that both electron-rich and electron-deficient aryl iodides were suitable for this reaction, giving desired coupling products in good to excellent yields. For *para-* and *meta-*substituted iodobenzenes, the reaction also gave the corresponding products in excellent yields (Table 3, entries 2–7 and entries 14 and 15). For *ortho-*substituted iodobenzenes, the reaction was slow and

Table 2 The model reaction for the synthesis of phenyl azide<sup>a</sup>

Entry	DBU (mol %)	Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O	Yield (%) <sup>b</sup>
1	-	10 mol%	Trace
2	5	10 mol%	78
3	10	10 mol%	83
4	15	10 mol%	92
5	20	10 mol%	89
6	25	10 mol%	86
7	30	10 mol%	84
8	15	5 mol%	40

<sup>a</sup>Reaction conditions: iodobenzene (1.0 mmol), sodium azide (1.2 mmol), DMSO (3 mL), reaction time 1.5 h, temperature 95 °C.
<sup>b</sup>Isolated vield.

Fig. 2 Effect of temperature on the yield of the model reaction.

Table 3 The scope of azidation of aryl halides in the reaction with sodium azide  $\ensuremath{^a}$ 

. (^		Cu(OAc) <sub>2</sub> ·H <sub>2</sub> O, DBU	n N <sub>3</sub>
R	INalN <sub>3</sub>	DMSO, 95 °C	R
- Fastaria	Amilhalida	<b>T</b> ime (h)	V:-1-1 (0/)h
Entry	Aryi nalide		
1	R = H	1.5	92
2	$R = 4-CH_3$	3.5	87
3	$R = 4 - CH_{3}^{0}O$	3.5	85
4	R = 4-Br	2.0	88
5	$R = 4 - C_2 H_5 0$	4.0	80
6	$R = 3,4-(CH_3)_2$	2.5	90
7	R = 3-CI	4.5	85
8	R = 2-CH <sub>3</sub>	5.0	75
9	$R = 2,4,6-(CH_3)_3$	2.0	78
10	R = 2-CI	3.5	75
11	R = 2-H0	3.5	71
12	R = 2-F	3.5	71
13	$R = 3 - NO_2 - 4 - CH_3$	4.0	60
14	$R = 4 - NO_2$	2.5	90
15	$R = 3-NO_2$	3.0	85

<sup>a</sup>Reactions conditions: aryl iodides (1.0 mmol), sodium azide (1.2 mmol), Cu(OAc)<sub>2</sub>·H<sub>2</sub>O (10 mol%), DBU (15 mol%), DMSO (3 mL), temperature 95 °C, reaction time 1.5–5.0 h. <sup>b</sup>Isolated yield.

needed more time to be completed (Table 3, entries 8–12). Furthermore, for 4-methyl-3-nitroiodobenzene the reaction afforded a moderate yield of 60% (Table 3, entry 13). To expand further the substrate scope, we also tested the heteroaromatic

substrate 2-iodopyridine, which afforded a yield of 87% of 2-azidopyridine after 4.5 h.

## Conclusions

In summary, aryl azides were efficiently synthesised in moderate to excellent yields in short times by the coupling reaction of aryl iodides with sodium azide using a  $Cu(OAc)_2 \cdot H_2O$  (10 mol%)/DBU (15 mol%) catalytic system. The established method was suitable for both electron-rich and electron-deficient aryl iodides.

## Experimental

All reagents were purchased from commercial sources and used without further treatment, unless otherwise indicated. The products were characterised using <sup>1</sup>H NMR and <sup>13</sup>C NMR (Bruker Avance 400 MHz and Bruker Avance III HD 600 MHz) which used CDCl<sub>3</sub> as the solvent. Data are represented as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, dd = double of doublets, t = triplet, q = quartet, m = multiplet) and coupling constants (*J*) in Hertz (Hz). <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra of the products are given in the Electronic Supplementary Information.

#### Synthesis of aryl azides; general procedure

**CAUTION**: Azides are potentially explosive and so appropriate precautions against blast must be taken when preparing, handling and heating them. Reactions must be carried out on a small scale.

A mixture of the aryl iodide (1.0 mmol), sodium azide (1.2 mmol), DBU (0.15 mmol) and Cu(OAc)<sub>2</sub>·H<sub>2</sub>O (0.1 mmol) in DMSO (3.0 mL) in a 10 mL flask was heated to 95 °C (the temperature in the reaction flask was monitored) for 1.5–5.0 h. After the reaction was completed as judged by TLC, the cooled mixture was poured into water (30 mL) containing several drops of ammonia. The resulting aqueous phase was extracted with ethyl acetate (3 × 30 mL). The combined organic phases were washed with brine (50 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The residue was loaded on a silica gel column and eluted with petroleum ether (boiling range 60–90 °C)/ethyl acetate to afford the product.

*Phenyl azide* (Table 3, entry 1):<sup>28</sup> Light yellow liquid; yield 92%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.38–7.34 (m, 2H), 7.14 (t, *J* = 7.4 Hz, 1H), 7.03 (d, *J* = 7.6 Hz, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  140.0, 129.8, 124.9, 119.0.

4-*Methylphenyl azide* (Table 3, entry 2):<sup>28</sup> Light yellow liquid; yield 87%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.16 (d, J = 8.2 Hz, 2H), 6.93 (d, J = 8.4 Hz, 2H), 2.34 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 137.1, 134.6, 130.4, 118.9, 20.9.

4-Methoxyphenyl azide (Table 3, entry 3):<sup>28</sup> Yellow liquid; yield 85%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 6.97–6.95 (m, 2H), 6.90–6.88 (m, 2H), 3.80 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 157.0, 132.3, 120.0, 115.1, 55.6.

4-Bromophenyl azide (Table 3, entry 4):<sup>28</sup> White solid; m.p. 24–25 °C (lit.<sup>44</sup> 20 °C); yield 88%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.46–7.44 (m, 2H), 6.91–6.89 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 139.2, 132.8, 120.7, 117.8.

4-*Ethoxyphenyl azide* (Table 3, entry 5):<sup>45</sup> Light yellow liquid; yield 80%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 6.96–6.94 (m, 2H), 6.89–6.87 (m, 2H), 4.01 (q, J = 7.0 Hz, 2H), 1.42 (t, J = 7.0 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 156.3, 132.1, 119.9, 115.7, 63.8, 14.8.

3,4-Dimethylphenyl azide (Table 3, entry 6):<sup>45</sup> Greenish yellow liquid; yield 90%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.10 (d, J = 8.0 Hz, 1H), 6.81 (s, 1H), 6.77 (d, J = 8.0 Hz, 1H), 2.25 (s, 3H), 2.24 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  138.3, 137.3, 133.4, 130.8, 120.0, 116.3, 19.9, 19.2.

*3-Chlorophenyl azide* (Table 3, entry 7):<sup>24</sup> Light yellow liquid; yield 85%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.30–7.26 (m, 1H), 7.12 (d, J = 8.0 Hz, 1H), 7.03 (t, J = 2.0 Hz, 1H), 6.93 (dd, J = 8.1, 2.1 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  141.5, 135.4, 130.7, 125.1, 119.3, 117.2.

2-Methylphenyl azide (Table 3, entry 8):<sup>28</sup> Light yellow liquid; yield 75%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.26–7.22 (m, 1H), 7.18–7.11 (m,

2H), 7.07–7.03 (m, 1H), 2.22 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 138.4, 131.2, 129.6, 127.1, 124.6, 117.9, 17.3.

2,4,6-*Trimethylphenyl azide* (Table 3, entry 9):<sup>26</sup> Light yellow liquid; yield 78%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 6.86 (s, 2H), 2.35 (s, 6H), 2.28 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 135.3, 134.4, 131.9, 129.5, 20.7, 18.1.

2-*Chlorophenyl azide* (Table 3, entry 10):<sup>46</sup> Colourless oil; yield 75%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.37 (d, J = 8.0 Hz, 1H), 7.31–7.27 (m, 1H), 7.17 (d, J = 8.1 Hz, 1H), 7.08 (t, J = 8.0 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 137.2, 130.8, 127.9, 125.6, 125.0, 119.7.

2-Hydroxyphenyl azide (Table 3, entry 11):<sup>47</sup> Brown liquid; yield 71%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.08–7.03 (m, 2H), 6.95–6.91 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  147.4, 126.0, 126.0, 121.2, 118.4, 116.0.

2-*Fluorophenyl azide* (Table 3, entry 12):<sup>28</sup> Brown liquid; yield 71%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.14–7.05 (m, 4H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 154.9 (d, J = 165 Hz, 1C), 128.0 (d, J = 8 Hz, 1C), 125.8 (d, J = 5 Hz, 1C), 124.9 (d, J = 3 Hz, 1C), 121.1, 116.7 (d, J = 6 Hz, 1C).

*3-Nitro-4-methylphenyl azide* (Table 3, entry 13):<sup>48</sup> Light yellow solid; m.p. 70–71 °C (lit.<sup>48</sup> 67–68 °C); yield 60%; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.63 (s, 1H), 7.33 (d, *J* = 8.2 Hz, 1H), 7.16 (d, *J* = 8.4 Hz, 1H), 2.57 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  149.7, 139.3, 134.1, 130.0, 123.4, 115.2, 20.0.

4-*Nitrophenyl azide* (Table 3, entry 14):<sup>25</sup> Yellow solid; m.p. 69–70 °C (lit.<sup>25</sup> 63–66 °C); yield 90%; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>): δ 8.25 (d, J = 9.0 Hz, 2H), 7.14 (d, J = 9.0 Hz, 2H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>): δ 146.9, 144.6, 125.6, 119.4.

*3-Nitrophenyl azide* (Table 3, entry 15):<sup>49</sup> Yellow solid; m.p. 56–57 °C (lit.<sup>49</sup> 53.1–54.5 °C); yield 85%; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  8.01–8.00 (m, 1H), 7.89 (t, J = 2.2 Hz, 1H), 7.54 (t, J = 8.1 Hz, 1H), 7.36–7.34 (m, 1H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>):  $\delta$  149.2, 142.0, 130.6, 124.9, 119.7, 114.1.

2-*Azidopyridine*:<sup>50</sup> Yellow liquid; yield 87%; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  8.40 (dd, J = 4.8, 1.7 Hz, 1H), 7.65 (td, J = 7.7, 2.0 Hz, 1H), 7.33 (d, J = 8.0 Hz, 1H), 7.23–7.21 (m, 1H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>):  $\delta$  151.6, 149.8, 138.7, 124.5, 122.2.

#### Acknowledgements

This work was supported financially by the Innovative Talents Programme of Henan Province (Nos 164100510015 and 174100510025), the Foundation of Henan Educational Committee (Nos 18A150030, 15A150054 and 16A350015), the Scientific Research Foundation for Doctors (No. qd16106) and the Youth Foundation (No. 2016QK09) of Henan Normal University.

## **Electronic Supplementary Information**

The ESI (<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra of the products) is available through

http://ingentaconnect.com/content/stl/jcr/2018/00000042/00000005/art00005

Received 13 January 2018; accepted 16 April 2018 Paper 1805192 https://doi.org/10.3184/174751918X15260507766192 Published online: 30 May 2018

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