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One-pot synthesis of azo compounds in the absence of acidic or alkaline additives

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Abstract

A one-pot method for the synthesis of azo compounds by the reaction of β-naphthol with aryl amines using *t*-BuONO as the nitrosonium source in DCM at room temperature was developed. This method features mild reaction conditions, a simple experimental procedure, and is free of acidic or alkaline additives.

Keywords

One-pot method, azo compounds, acidic–alkaline additives free

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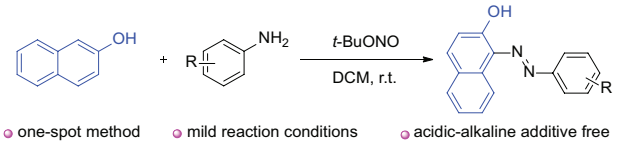

Introduction

Azo compounds, some of the most important synthetic highly colored dyes, are widely distributed.[1–3] At present, about 3000 azo dyes are known to be in use in various fields around the world.[4] For instance, azo dyes are used in textile dyeing, histology dyeing, drug colorimetric analysis, cosmetics, food, digital printing, photographic colorants, and so on.[5–12] However, azo compounds also have excellent optical and photoelectric properties[13,14] and can be used to diagnose Alzheimer's disease.[15] In addition, some azo compounds have been reported to possess antibacterial and antifungal activities.[16,17] Due to their broad applications, many reports have focused on the preparation of synthetic azo compounds.[18–20]

The traditional approach for the preparation of azo compounds is usually divided into two steps: first, aromatic primary amines are diazotized to form diazonium salts using mineral acids, and then the so formed diazonium salts are coupled with active aromatic compounds under alkaline conditions.[21] This classical synthetic method is limited by the need for low reaction temperatures and, especially, the usage of acidic or alkaline reagents[22,23] which violate the principles of green chemistry and cause permanent pollution to the environment.[24] Therefore, in recent years, many researchers have devoted themselves to exploring mild and green synthetic strategies, especially on developing novel catalysts for diazotization–diazo coupling. For example, a nanomagnetic-supported sulfonic acid was developed by Kolvari to catalyze diazotization–diazo coupling reactions in the absence of solvents.[25] Chermahini reported the synthesis of azo compounds using modified montmorillonite K-10.[26] According to Bagherzade's research achievement, diethylamine functionalized polyethylene glycol could be used as a catalyst in the process of preparing azo dyes.[27] In addition, many investigations demonstrated that $Fe(HSO_4)_3$,[28] magnetic Fe_3O_4 nanoparticles,[29] granular PTFE (polytetrafluoroethylene),[30] and graphene quantum dots[4] were also effective catalysts for the synthesis of azo compounds. However, the above-mentioned methodologies still suffer from some disadvantages, such as

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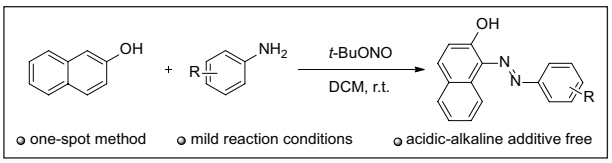
tedious processes, high manufacturing costs, and the use of metals or mineral acids. In this study, we report a one-pot method for the synthesis of azo compounds at room temperature in the absence of any acidic or alkaline additive using *tert*-butyl nitrite as the nitrosonium source (Scheme 1).

Results and discussion

Initially, we carried out the reaction using β-naphthol (**1**) and *p*-toluidine (**2**) as model substrates, *t*-BuONO as the nitrosonium source, $CuSO_4.5H_2O$ as the catalyst, and AcOH as the additive in DCM at room temperature for 24 h. This provided the desired azo products in 73% yield (Table 1, entry 1). Next, various acidic additives were screened. However, they all showed inferior results to AcOH (Table 1, entries 2–5). To our delight, when the loading of *p*-toluidine, *t*-BuONO, and AcOH increased to 1.5 equiv., the product could be obtained in 92% yield (Table 1, entry 6). Subsequently, several other solvents such as acetone, CH_3CN, and CH_3OH were investigated. Compared with DCM, they were less effective for this transformation (Table 1, entries 7–9). Control experiments demonstrated that *t*-BuONO was indispensable for the reaction (Table 1, entry 10), and the reaction could proceed efficiently in the absence of the catalyst $CuSO_4.5H_2O$ (Table 1, entry 11). To our surprise, when AcOH was excluded from the reaction, the azo products were obtained in an 88% yield (Table 1, entry 12).



Scheme 1. One-pot synthesis of azo compounds.

With the optimized conditions in hand, the scope of amines as the substrates was examined (Scheme 2). Initially, various substituted amines were reacted with β-naphthol in the presence of *t*-BuONO in DCM at room temperature. Delightfully, both electron-donating and electron-withdrawing functional groups such as alkyl (**3b**, **3c**, **3m**), methoxy (**3h**), fluoro (**3d**), chloro (**3e**, **3n**), bromo (**3f**, **3o**), iodo (**3g**), ester (**3i**), nitro (**3j**, **3l**), and trifluoromethyl (**3k**) were well tolerated, affording the azo products in moderate to excellent yields. For instance, Sudan I (**3a**) and *Para* Red (**3j**) could be synthesized under the standard conditions in 87% and 86% yields, respectively. In addition, the substituent at the *para-, ortho-*, and *meta*-positions of the aromatic ring did not affect the reaction efficiency. Unfortunately, no desired azo product was detected when 4-aminopyridine or an alkyl amine was used as the substrate (**3p**, **3q**).

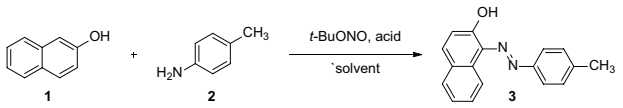
Conclusion

In summary, we have developed a one-pot approach for the synthesis of azo compounds by the reaction of naphthol with aromatic amines at room temperature. This protocol was characterized by easily accessible raw materials, mild reaction conditions, and a simple operational procedure. Most importantly, the azo compounds could be synthesized in the absence of any acidic or alkaline additive, which is in line with the theme of green chemistry. We expect this method to find considerable potential for application in industry.

Experimental procedure

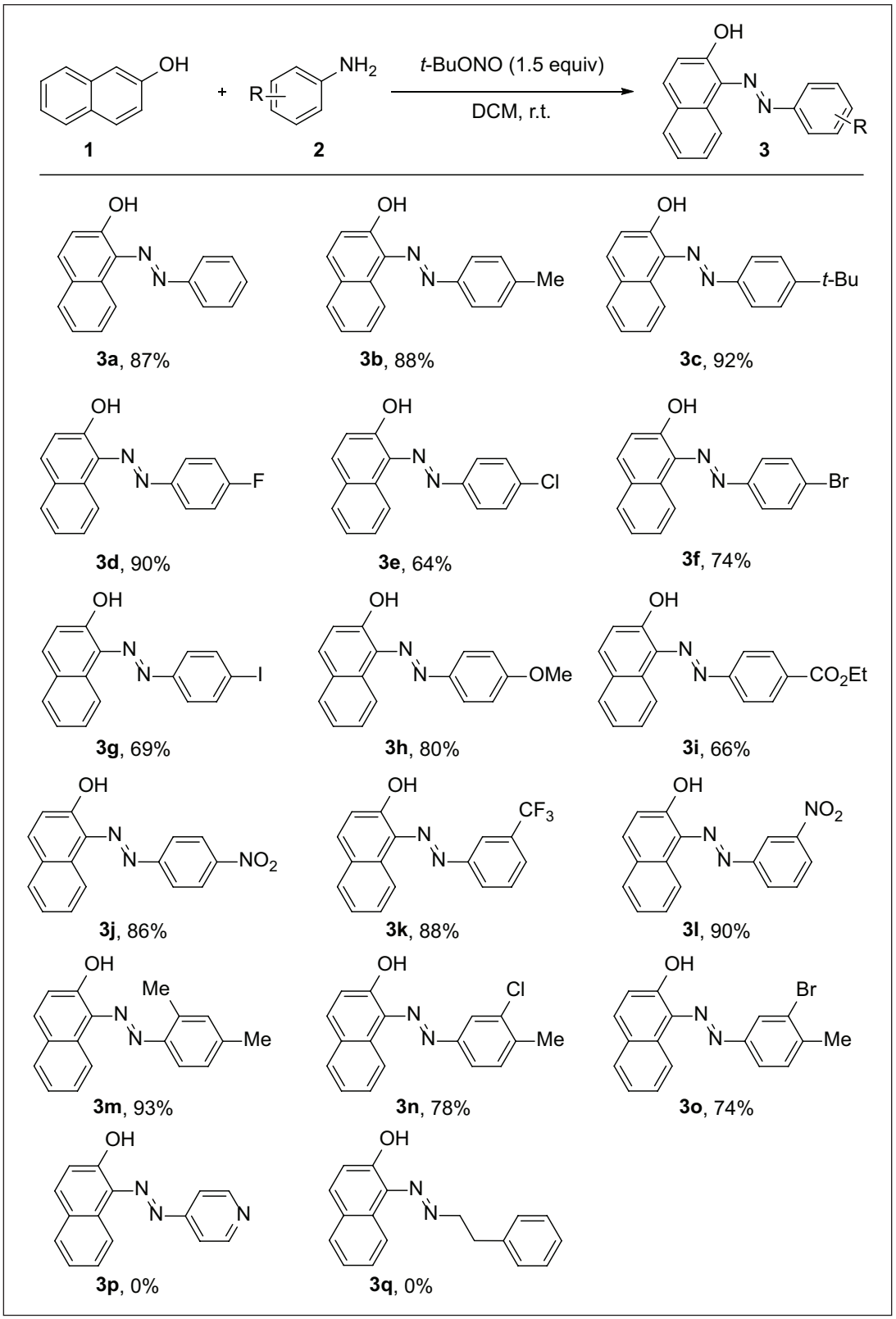
Commercially available chemicals were obtained from commercial suppliers and used without further purification unless otherwise stated. Proton NMR (^{1}H) and carbon

Table 1. Optimization of the reaction conditions.[a]



Entry	2/1	Acid (equiv.)	Catalyst (equiv.)	Solvent	Yield[b] (%)
1	1.2	AcOH (1.2)	$CuSO_4.5H_2O$ (0.2)	DCM	73
2	1.2	CH_3SO_3H (1.2)	$CuSO_4.5H_2O$ (0.2)	DCM	60
3	1.2	TfOH (1.2)	$CuSO_4.5H_2O$ (0.2)	DCM	39
4	1.2	CF_3CO_2H (1.2)	$CuSO_4.5H_2O$ (0.2)	DCM	62
5	1.2	$TsOH.H_2O$ (1.2)	$CuSO_4.5H_2O$ (0.2)	DCM	32
6	1.5	AcOH (1.5)	$CuSO_4.5H_2O$ (0.2)	DCM	92
7	1.5	AcOH (1.5)	$CuSO_4.5H_2O$ (0.2)	Acetone	8
8	1.5	AcOH (1.5)	$CuSO_4.5H_2O$ (0.2)	CH_3CN	61
9	1.5	AcOH (1.5)	$CuSO_4.5H_2O$ (0.2)	CH_3OH	62
10[c]	1.5	AcOH (1.5)	$CuSO_4.5H_2O$ (0.2)	DCM	0
11	1.5	AcOH (1.5)	–	DCM	90
12	1.5	–	–	DCM	88

[a]Reaction conditions: **1** (0.5 mmol), **2** (0.6 mmol, 1.2 equiv.), *t*-BuONO (0.6 mmol, 1.2 equiv.), solvent (3.0 mL), acid, $CuSO_4.5H_2O$ (0.1 mmol, 0.2 equiv.), r.t., 24 h.
[b]Isolated yield.
[c]Without *t*-BuONO.



Scheme 2. The scope of amines as substrates. Reagents and conditions: **1** (0.5 mmol, 1.0 equiv.), **2** (0.75 mmol, 1.5 equiv.), *t*-BuONO (0.75 mmol, 1.5 equiv.), DCM (5.0 mL), r.t., 24 h, in air.

(^{13}C) NMR spectra were recorded on an Ascend TM 500 MHz NMR spectrometer. The following abbreviations are used for the multiplicities: s: singlet, d: doublet, t: triplet, q: quartet, m: multiplet, and br s: broad singlet for proton spectra. Coupling constants (*J*) are reported in Hertz (Hz). Melting points (uncorrected) were determined on an automatic melting point apparatus (ZRD-1) from Tianjin optical instrument factory. Analytical thin-layer

chromatography was performed on Polygram SIL G/UV_{254} plates. Visualization was accomplished with shortwave UV light, or by $KMnO_4$ staining followed by heating. Flash column chromatography was performed using silica gel (200300 mesh) from Qingdao Haiyang Chemical Co., Ltd with solvents distilled prior to use.

General procedure for the synthesis of azo compounds

To a solution of β-naphthol (1.0 mmol, 1.0 equiv.) in DCM (5.0 mL) was added the aryl amine (1.5 mmol, 1.5 equiv.) and *t*-BuONO (1.5 mmol, 1.5 equiv.). The reaction mixture was stirred in air at room temperature for 24 h and then concentrated in vacuum. The crude mixture was purified by column chromatography on silica gel with a mixture of petroleum ether and ethyl acetate as eluent to afford the target compounds.

1-(phenyldiazenyl)naphthalen-2-ol (**3a**):[26] Red powder; yield 87%; m.p. 130–132 °C (lit.[26]128–130 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 8.56 (d, *J*=8.2 Hz, 1H), 7.76-7.70 (m, 3H), 7.60 (d, *J*=7.8 Hz, 1H), 7.55 (t, *J*=7.7 Hz, 1H), 7.48 (t, *J*=7.9 Hz, 2H), 7.39 (t, *J*=7.5 Hz, 1H), 7.30 (t, *J*=7.4 Hz, 1H), 6.87 (d, *J*=9.4 Hz, 1H).

1-(p-tolyldiazenyl)naphthalen-2-ol (**3b**):[29] Red powder; yield 88%; m.p. 132–133 °C (lit.[29]130–132 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 8.62 (d, *J*=8.3 Hz, 1H), 7.73 (d, *J*=9.3 Hz, 1H), 7.68 (d, *J*=8.3 Hz, 2H), 7.63 (d, *J*=7.9 Hz, 1H), 7.56 (t, *J*=7.6 Hz, 1H), 7.40 (t, *J*=7.5 Hz, 1H), 7.29 (d, *J*=8.1 Hz, 2H), 6.93 (d, *J*=9.3 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 168.6, 143.7, 139.0, 138.5, 133.7, 130.3, 129.9, 128.7, 128.6, 128.2, 125.5, 124.1, 121.8, 119.3, 21.4.

1-({4-[tert-butyl]phenyl}diazenyl)naphthalen-2-ol (**3c**): Red solid; yield 92%; m.p. 87–89 °C (lit.[31]86–88 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 8.60 (d, *J*=8.3 Hz, 1H), 7.73-7.70 (m, 3H), 7.62 (d, *J*=7.8 Hz, 1H), 7.56 (t, *J*=7.7 Hz, 1H), 7.51 (d, *J*=8.7 Hz, 2H), 7.39 (t, *J*=6.9 Hz, 1H), 6.92 (d, *J*=9.4 Hz, 1H), 1.37 (s, 9H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 169.4, 151.6, 143.3, 139.2, 133.7, 130.0,128.8, 128.7, 128.2, 126.7, 125.5, 124.4, 121.8, 119.0, 34.9, 31.5.

1-({4-fluorophenyl}diazenyl)naphthalen-2-ol (**3d**): Red floccule; yield 90%; m.p. 144–146 °C (lit.[28]143–145 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 15.80 (s, 1H), 8.57 (d, *J*=8.2 Hz, 1H), 7.77-7.70 (m, 3H), 7.62 (d, *J*=7.9 Hz, 1H), 7.54 (t, *J*=7.7 Hz, 1H), 7.38 (t, *J*=6.9 Hz, 1H), 7.16 (t, *J*=8.6 Hz, 2H), 6.93 (d, *J*=9.3 Hz, 1H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 166.5, 162.5 (d, *J*=249.3 Hz), 143.0 (d, *J*=2.9 Hz), 138.9, 133.5, 129.9, 128.7, 128.6, 128.3, 125.5, 123.4, 121.7, 121.3 (d, *J*=8.5 Hz), 116.6 (d, *J*=23.2 Hz).

1-({4-chlorophenyl}diazenyl)naphthalen-2-ol (**3e**):[26] Red floccule; yield 64%; m.p. 153–155 °C (lit.[26]158–160 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 16.03 (s, 1H), 8.53 (d, *J*=8.2 Hz, 1H), 7.72 (d, *J*=9.4 Hz, 1H), 7.67 (d, *J*=8.9 Hz, 2H), 7.60 (d, *J*=7.8 Hz, 1H), 7.55 (t, *J*=7.7 Hz, 1H), 7.45-7.38 (m, 3H), 6.87 (d, *J*=9.4 Hz, 1H).

1-([4-bromophenyl]diazenyl)naphthalen-2-ol (**3f**):[29] Red powder; yield 74%; m.p. 168–170 °C (lit.[32]169–170 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 16.06 (s, 1H), 8.53 (d, *J*=8.2 Hz, 1H), 7.73 (d, *J*=9.4 Hz, 1H), 7.62-7.54 (m, 6H), 7.41 (t, *J*=7.0 Hz, 1H), 6.86 (d, *J*=9.4 Hz, 1H).

1-({4-iodophenyl}diazenyl)naphthalen-2-ol (**3g**):[33] Dark red powder; yield 69%; m.p. 164–166 °C; ^{1}H NMR (500 MHz, Chloroform-*d*): δ 16.05 (s, 1H), 8.46 (d, *J*=8.1 Hz, 1H), 7.74 (d, *J*=8.7 Hz, 2H), 7.68 (d, *J*=9.4 Hz, 1H), 7.56 (d, *J*=7.8 Hz, 1H), 7.54-7.50 (m, 1H), 7.41 (d, *J*=8.7 Hz, 2H), 7.38 (t, *J*=7.5 Hz, 1H), 6.81 (d, *J*=9.4 Hz, 1H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 172.6, 144.5, 140.7, 138.7, 133.5, 130.4, 129.1, 128.8, 128.3, 126.1, 124.9, 121.9, 120.2, 91.8.

1-({4-methoxyphenyl}diazenyl)naphthalen-2-ol (**3h**):[29] Red powder; yield 80%; m.p. 136–138 °C (lit.[29]137–138 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 15.72 (s, 1H), 8.70 (d, *J*=8.3 Hz, 1H), 7.82 (d, *J*=9.0 Hz, 2H), 7.76 (d, *J*=9.2 Hz, 1H), 7.69 (d, *J*=7.9 Hz, 1H), 7.57 (t, *J*=7.7 Hz, 1H), 7.40 (t, *J*=7.5 Hz, 1H), 7.03 (dd, *J*=10.4, 9.1 Hz, 3H), 3.88 (s, 3H).

Ethyl-4-({2-hydroxynaphthalen-1-yl}diazenyl)benzoate (**3i**): Red powder; yield 66%; m.p. 139–141 °C (lit.[34]149 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 8.43 (d, *J*=8.1 Hz, 1H), 8.12 (d, *J*=8.6 Hz, 2H), 7.67-7.64 (m, 3H), 7.58-7.51 (m, 2H), 7.40 (t, *J*=7.4 Hz, 1H), 6.73 (d, *J*=9.6 Hz, 1H), 4.41 (q, *J*=7.2 Hz, 2H), 1.44 (t, *J*=7.1 Hz, 3H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 178.0, 166.1, 146.8, 142.3, 133.5, 131.4, 131.1, 129.5, 129.0, 128.4, 127.8, 126.8, 126.2, 122.2, 116.9, 61.2, 14.5.

1-({4-nitrophenyl}diazenyl)naphthalen-2-ol (**3j**):[4] Red powder; yield 86%; m.p. 250–252 °C (lit.[4]249–250 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 16.13 (s, 1H), 8.42 (d, *J*=8.0 Hz, 1H), 8.32 (d, *J*=9.1 Hz, 2H), 7.71-7.68 (m, 3H), 7.60-7.51 (m, 2H), 7.44 (t, *J*=7.4 Hz, 1H), 6.70 (d, *J*=9.6 Hz, 1H).

1-({3-(trifluoromethyl)phenyl}diazenyl)naphthalen-2-ol (**3k**): Bright red powder; yield 88%; m.p. 159–160 °C (lit.[35]193–195 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 16.06 (s, 1H), 8.47 (d, *J*=8.5 Hz, 1H), 7.93 (s, 1H), 7.79 (d, *J*=8.0 Hz, 1H), 7.69 (d, *J*=9.5 Hz, 1H), 7.61-7.49 (m, 4H), 7.39 (t, *J*=7.4 Hz, 1H), 6.80 (d, *J*=9.5 Hz, 1H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 173.9, 145.1, 141.4, 133.4, 132.3 (q, *J*=32.9 Hz), 130.7, 130.3, 129.4, 128.9, 128.4, 126.5, 125.1, 123.9 (q, *J*=272.6 Hz), 123.3 (q, *J*=3.6 Hz), 122.1, 121.4, 114.9 (q, *J*=4.0 Hz).

1-({3-nitrophenyl}diazenyl)naphthalen-2-ol (**3l**):[32] Red powder; yield 90%; m.p. 194–196 °C (lit.[32]194–196 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 16.03 (s, 1H), 8.54 (s, 1H), 8.50 (d, *J*=8.2 Hz, 1H), 8.08 (d, *J*=8.1 Hz, 1H), 7.91 (d, *J*=8.0 Hz, 1H), 7.73 (d, *J*=9.5 Hz, 1H), 7.65-7.53 (m, 3H), 7.43 (t, *J*=7.4 Hz, 1H), 6.80 (d, *J*=9.5 Hz, 1H).

1-({2,4-dimethylphenyl}diazenyl)naphthalen-2-ol (**3m**): Dark red solid; yield 93%; m.p. 149–150 °C (lit.[36]159–160 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 8.59 (d, *J*=8.2 Hz, 1H), 7.94 (d, *J*=8.3 Hz, 1H), 7.70 (d, *J*=9.4 Hz,

1H), 7.59 (d, J=7.8 Hz, 1H), 7.54 (t, J=8.2 Hz, 1H), 7.37 (t, J=7.4 Hz, 1H), 7.14 (d, J=8.3 Hz, 1H), 7.07 (s, 1H), 6.90 (d, J=9.4 Hz, 1H), 2.50 (s, 3H), 2.36 (s, 3H).

1-({3-chloro-4-methylphenyl}diazenyl)naphthalen-2-ol (**3n**):[37] Red powder; yield 78%; m.p. 133–135 °C; ^{1}H NMR (500 MHz, Chloroform-*d*): δ 15.92 (s, 1H), 8.52 (d, J=8.2 Hz, 1H), 7.75 (d, J=2.0 Hz, 1H), 7.69 (d, J=9.4 Hz, 1H), 7.58 (d, J=7.8 Hz, 1H), 7.54 (t, J=8.2 Hz, 1H), 7.45 (dd, J=8.2, 2.0 Hz, 1H), 7.38 (t, J=7.4 Hz, 1H), 7.28 (d, J=8.2 Hz, 1H), 6.86 (d, J=9.4 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 169.8, 144.6, 139.9, 135.7, 133.5, 131.7, 130.2, 129.0, 128.7, 128.2, 125.8, 124.2, 121.9, 118.9, 117.8, 20.0.

1-({3-bromo-4-methylphenyl}diazenyl)naphthalen-2-ol (**3o**):[38] Dark red powder; yield 74%; m.p. 217–218 °C (lit.[38]217–218 °C); ^{1}H NMR (500 MHz, Chloroform-*d*): δ 15.91 (s, 1H), 8.53 (d, J=8.2 Hz, 1H), 7.94 (d, J=2.1 Hz, 1H), 7.70 (d, J=9.4 Hz, 1H), 7.59 (d, J=7.8 Hz, 1H), 7.55 (t, J=7.7 Hz, 1H), 7.51 (dd, J=8.1, 1.9 Hz, 1H), 7.38 (t, J=7.4 Hz, 1H), 7.29 (d, J=8.2 Hz, 1H), 6.87 (d, J=9.4 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR (126 MHz, Chloroform-*d*): δ 169.7, 144.7, 139.9, 137.5, 133.5, 131.5, 130.2, 129.0, 128.7, 128.2, 125.9, 125.9, 124.2, 122.2, 121.9, 118.4, 22.9.

Declaration of conflicting interests

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Supplemental material

Supplemental material for this article is available online.

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