One-pot synthesis of thiocarbamidoalkyl naphthols

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An efficient, inexpensive and mild method for the synthesis of α-thiocarbamidoalkyl-β-naphthols, catalysed by *p*-toluenesulfonic acid at room temperature is reported. A set of thiocarbamidoalkyl naphthols have been synthesised in high yields from aromatic aldehydes, β-naphthol and *N*-phenylthiourea or thiourea. The procedure involves a short reaction time and a simple experimental procedure.

Keywords: multicomponent reactions, thiocarbamidoalkyl naphthols, Betti base

Compounds bearing 1,3-amino oxygenated functional groups are found in biologically important natural products and drugs including a number of nucleoside antibiotics and HIV protease inhibitors, such as ritonavir and lipinavir.[1-6] Aminonaphthols exhibit antihypertensive, adrenoceptor blocking, and Ca^{2+} channel blocking activities.[7-9]

Multicomponent reactions (MCRs) have attracted considerable attention since they do not require the isolation of intermediates savings time, energy and raw materials. Hence the discovery of new MCRs is still in demand.[10-14]

Various methods have been reported for the straightforward synthesis of Betti base derivatives by the aminoalkylation of β-naphthol with aromatic aldehydes and heterocyclic amines,[15,16] pyrrolidine,[17] iminium salts[18,19] and methyl carbamate.[20] Amidoalkyl naphthols can be prepared from the Betti base or by condensation of aromatic aldehydes, β-naphthol and amides as the ammonia source. To our knowledge, relatively few reports have appeared in this field.[21-28] A straightforward synthesis of thiocarbamidoalkyl naphthols from thiourea and its derivatives has not been reported. We report here a mild, simple and efficient method for the one-pot three-component condensation of aromatic aldehydes, β-naphthol and thioureas.

In our initial search for appropriate reaction conditions, we chose the reaction of β-naphthol with benzaldehyde and *N*-phenylthiourea as a model with a variety of catalysts in CH_2Cl_2 at room temperature (Scheme 1 and Table 1, entries 1–7).

As shown in Table 1, the best result was obtained when *p*-toluenesulfonic acid (*p*-TSA) was used as a catalyst (Table 1, entry 7). We then examined the reaction in various solvents in the presence of 10% *p*-TSA (Table 1, entries 7–11). We found that 1,2-dichloroethane and CH_2Cl_2 gave high yields of the product. However, 1,2-dichloroethane is toxic and so we chose CH_2Cl_2 as a solvent for the reactions.

Under the reaction conditions established as above, various thiocarbamidoalkyl naphthols were then synthesised in good to excellent yields by the reaction of different aromatic aldehydes with β-naphthol and *N*-phenylthiourea or thiourea. The results are summarised in Table 2. Among them, **3a** was identified, by R_f value (on TLC), ^1H NMR and m.p., as the same compound as prepared from Betti base and isothiocyanatobenzene in toluene at room temperature. In all cases, aromatic aldehydes bearing either electron-donating or electron-withdrawing groups reacted successfully. As expected aromatic aldehydes with electron-withdrawing groups reacted faster than the aromatic aldehydes with electron-donating groups. When thiourea was used as the amine source, the aminoalkylation of naphthol gave a lower yield and needed a longer reaction time.

Table 1 Effect of catalysts on the reaction of benzaldehyde, β-naphthol and *N*-phenylthiourea[a]

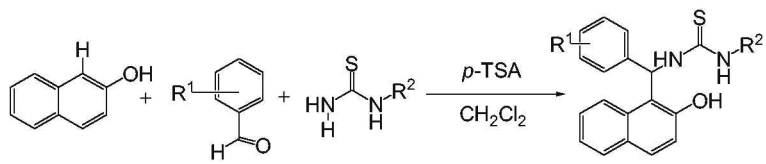
Entry	Catalyst	Solvent	Yield/%[b]
1	$Yb(OTf)_3$	CH_2Cl_2	<30
2	$La(OTf)_3$	CH_2Cl_2	<30
3	$Cu(OTf)_2$	CH_2Cl_2	73
4	$Zn(OTf)_2$	CH_2Cl_2	Trace
5	HOTf	CH_2Cl_2	52
6	CH_3SO_3H	CH_2Cl_2	<30
7	CF_3COOH	CH_2Cl_2	48
8	*p*-TSA	CH_2Cl_2	90
9	*p*-TSA	$ClCH_2CH_2Cl$	93
10	*p*-TSA	Toluene	78
11	*p*-TSA	CH_3OH	85
12	*p*-TSA	C_2H_5OH	80

[a]Reaction conditions: solvent (5 mL), benzaldehyde (1 mmol), β-naphthol (1 mmol), *N*-phenylthiourea (1.1 mmol), cat. amount 10%, room temperature, 12 h. [b]Isolated yield.

Table 2 One-pot synthesis of thiocarbamidoalkyl naphthols in the presence of *p*-TSA[a]

No.	R^1	R^2	Yield[b]/%	t/h
3a	H	Ph	90	12
3b	*p*-Cl	Ph	91	8
3c	*o*-Cl	Ph	88	10
3d	2,4-dichloro	Ph	86	10
3e	*m*-NO_2	Ph	93	8
3f	*p*-F	Ph	92	9
3g	*p*-OH	Ph	78	24
3h	*p*-CH_3O	Ph	81	24
3i	3,4-dimethyl	Ph	74	24
3j	H	H	72	24
3k	*m*-NO_2	H	78	18
3l	*o*-Cl	H	77	20
3m	3,4-dimethyl	H	58	24

[a]Reaction condition: CH_2Cl_2 as solvent (5 mL), aromatic aldehyde (1 mmol), β-naphthol (1 mmol), *N*-phenylthiourea or thiourea (1.1 mmol), cat. amount 10%, room temperature. [b]Isolated yield.



Scheme 1

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In conclusion, we have developed an efficient and convenient synthesis of 1-thiocarbamidoalkyl-2-naphthol derivatives by a three-component condensation of aryl aldehydes, 2-naphthol and *N*-phenylthiourea or thiourea in the presence of *p*-TSA. This method offers several significant advantages: high conversions, mild reaction conditions and easy handling, which makes it a useful and attractive process for the convenient synthesis of substituted thiocarbamidoalkyl naphthols.

Experimental

Melting points were determined on a Büchi B-540 melting apparatus and are uncorrected. The NMR spectra were measured on a Bruker Advance III 500 instrument using DMSO-d_6 as the solvent with TMS as the internal standard. IR spectra were recorded using KBr pellets on a Nicolet 6700 FI-IR spectrophotometer. Mass spectra were measured with GCT Premier CAB 075. Elemental analyses were performed on Thermo Finnigan EA1112 elemental analyser.

General procedure for preparation of **3a–m**

A mixture of aromatic aldehyde (1 mmol), β-naphthol (1 mmol), *N*-phenylthiourea or thiourea (1.1 mmol) and TsOH·H_2O (0.1 mmol) in CH_2Cl_2 (5 mL) was stirred at room temperature for the appropriate time (Table 2). The progress of the reaction was monitored by TLC (2:1 hexane: ethyl acetate). After completion of the reaction, as indicated by TLC, the reaction mixture was filtered and the precipitate washed throughly with CH_2Cl_2 and then with water. The crystalline product was collected without further purification. The physical and spectroscopic data of the compounds **3a–m** are as follows.

3a: Colourless crystals; m.p. 178–180 °C; IR (KBr) cm^{-1}: 3447, 3285, 3172, 3066, 1626, 1605, 1546, 1515, 1436, 1360, 1259, 1144, 811, 747; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.20 (s, 1H), 10.16 (s, 1H), 8.71–8.70 (ss, br, 1H), 8.16 (s, br, 1H), 8.00–7.98 (ss, 1H), 7.86 (d, J = 8.0 Hz, 1H), 7.82 (d, J = 8.5 Hz, 1H), 7.54–7.48 (m, 3H), 7.34 (q, J = 8 Hz, 3H), 7.28–7.13 (m, 7H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 180.7, 153.6, 142.8, 139.4, 133.0, 130.0, 129.3, 129.1, 128.6, 127.5, 126.8, 126.3, 125.0, 123.8, 123.2, 122.9, 119.1, 119.0, 53.8; MS (EI) *m/z* (%): 231 (100), 202 (20), 144 (75), 115 (29); Anal. Calcd for $C_{24}H_{20}N_2OS$: C, 74.97; H, 5.24; N, 7.29. Found: C, 74.87; H, 5.46; N, 7.47%.

3b: Colourless crystals; m.p. 164–166 °C; IR (KBr) cm^{-1}: 3363, 3191, 3018, 1629, 1543, 1518, 1489, 1328, 1267, 812, 747; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.23 (s, 2H), 8.68–8.67 (ss, br, 1H), 8.14 (s, br, 1H), 7.97–7.95 (ss, 1H), 7.87 (d, J = 8.0 MHz, 1H), 7.83 (d, J = 9.0 Hz, 1H), 7.54–7.33 (m, 8H), 7.24–7.22 (ss, 1H), 7.19–7.13 (m, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 180.4, 153.2, 141.5, 139.0, 132.4, 130.9, 129.8, 128.8, 128.7, 128.2, 128.1, 127.7, 127.1, 125.5, 124.6, 123.3, 122.8, 122.3, 118.5, 118.2, 52.8; MS (EI) *m/z* (%): 384 (M-34, 1), 267 (28), 231 (100), 202 (30), 144(60), 115 (23); Anal. Calcd for $C_{24}H_{19}ClN_2OS$: C, 68.81; H, 4.57; N, 6.69. Found: C, 68.70; H, 4.56; N, 6.54%.

3c: Colourless crystals; m.p. 174–176 °C; IR (KBr) cm^{-1}: 3382, 3186, 3009, 1629, 1592, 1540, 1515, 1435, 1371, 1331, 1266, 811, 747; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.04 (s, 1H), 9.99 (s, 1H), 8.47 (d, J = 8.5 Hz, 1H), 8.27 (d, J = 8.5 Hz, 1H), 7.82 (d, J = 8 Hz, 2H), 7.78 (d, J = 8.0 Hz, 1H), 7.65–7.64 (ss, 1H), 7.63–7.48 (m, 3H), 7.34–7.22 (m, 6H), 7.14 (d, J = 9.0 Hz, 1H), 7.08 (d, J = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 179.7, 153.7, 139.5 × 2, 132.7, 132.1, 130.0, 129.8, 129.2, 128.6, 128.5, 128.2, 128.1, 126.6, 126.2, 124.0, 123.1, 122.6, 122.5, 118.5, 117.2, 52.4; MS (EI) *m/z* (%): 384 (M-34, 1), 231 (100), 202 (15), 144(60), 115 (36); Anal. Calcd for $C_{24}H_{19}ClN_2OS$: C, 68.81; H, 4.57; N, 6.69. Found: C, 68.84; H, 4.59; N, 6.62%.

3d: Colourless crystals; m.p. 152–154 °C; IR (KBr) cm^{-1}: 3380, 3160, 1628, 1541, 1517, 1266, 1195, 814, 744; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.06 (s, 1H), 9.98 (s, 1H), 8.48 (d, J = 8.5 Hz, 1H), 8.24 (d, J = 9.0 Hz, 1H), 7.82 (d, J = 7.5 Hz, 1H), 7.79 (d, J = 8.5 Hz, 1H), 7.74–7.73 (ss, 1H), 7.67–7.65 (m, 3H), 7.56 (d, J = 8.0 Hz, 1H), 7.47–7.45 (m, 2H), 7.33–7.29 (m, 3H), 7.13–7.17 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 180.3, 154.3, 139.9, 139.5, 133.1, 133.0, 132.1, 131.8, 130.6, 129.1, 129.0, 128.9, 128.6, 127.2, 126.8, 124.5, 123.3, 123.1, 123.0, 118.9, 117.0, 52.5; MS (EI) *m/z* (%): 418 (M-34, 2), 265 (100), 202 (13), 144(42), 115 (15); Anal. Calcd for $C_{24}H_{18}Cl_2N_2OS$: C, 63.58; H, 4.00; N, 6.18. Found: C, 63.64; H, 4.15; N, 6.22%.

3e: (Yellow crystals); m.p. 142–144 °C; IR (KBr) cm^{-1}: 3422, 3334, 3165, 1628, 1522, 1475, 1352, 1319, 1245, 1190, 807, 744; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.37 (s, 1H), 10.31 (s, 1H), 8.77–8.76 (ss, 1H), 8.21 (s, br, 1H), 8.11–8.06 (m, 3H), 7.89 (t, J = 9.0 Hz, 2H), 7.63–7.55 (m, 5H), 7.39–7.35 (m, 3H), 7.26 (d, J = 8.5 Hz, 1H), 7.16 (t, J = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 181.2, 153.8, 148.2, 145.6, 139.5, 133.2, 132.7, 130.7, 130.2, 129.3, 129.2, 128.7, 127.9, 125.1, 123.8, 123.5, 122.7, 122.0, 120.7, 118.9, 118.1, 53.3; MS (EI) *m/z* (%): 348 (3), 231 (50), 202 (25), 144 (100), 115 (17); Anal. Calcd for $C_{24}H_{19}N_3O_3S$: C, 67.12; H, 4.46; N, 9.78. Found: C, 67.15; H, 4.45; N, 9.81%.

3f: Colourless crystals; m.p. 166–168 °C; IR (KBr) cm^{-1}: 3368, 3170, 3016, 1630, 1544, 1520, 1506, 1439, 1330, 1269, 1186, 835, 751; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.22 (s, 1H), 10.20 (s, 1H), 8.70 (s, br, 1H), 8.13 (s, br, 1H), 7.96–7.95 (ss, 1H), 7.86 (d, J = 8.0 Hz, 1H), 7.83 (d, J = 8.5 Hz, 1H), 7.55–7.33 (m, 6H), 7.24–7.09 (m, 6H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 180.3, 161.9, 160.0, 153.2, 139.0, 138.4, 132.3, 129.7, 128.8, 128.7, 128.2, 127.8, 127.7, 127.1, 125.5, 124.6, 123.3, 122.8, 122.3, 118.5, 118.3, 114.9, 114.8, 52.8; MS (EI) *m/z* (%): 251 (7), 249 (100), 231 (6), 144 (50), 115 (20); Anal. Calcd for $C_{24}H_{19}FN_2OS$: C, 71.62; H, 4.76; N, 6.96. Found: C, 71.53; H, 4.85; N, 6.83%.

3g: Pale yellow crystals; m.p. 162–164 °C; IR (KBr) cm^{-1}: 3527, 3369, 3190, 3016, 1629, 1546, 1513, 1437, 1331, 1258, 1176, 832, 747; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.14 (s, 1H), 10.11 (s, 1H), 9.21 (br, 1H), 8.71 (s, br, 1H), 8.12 (s, br, 1H), 7.89–7.79 (m, 3H), 7.52–7.31 (m, 6H), 7.22 (d, J = 9 Hz, 1H), 7.21–7.10 (m, 1H), 7.01 (d, J = 8.5 Hz, 2H), 6.66 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 180.2, 156.5, 153.5, 139.4, 132.9, 132.7, 129.7, 129.3, 129.0, 128.6 × 2, 127.7, 127.3, 125.0, 123.8, 123.2, 119.2, 119.0, 115.3, 53.6; MS (EI) *m/z* (%): 249 (4), 247 (42), 231 (16), 144 (100), 115 (35); Anal. Calcd for $C_{24}H_{20}N_2O_2S$: C, 71.98; H, 5.03; N, 6.99. Found: C, 71.94; H, 4.92; N, 6.81%.

3h: Colourless crystals; m.p. 174–176 °C; IR (KBr) cm^{-1}: 3364, 3195, 30156, 1628, 1543, 1511, 1327, 1246, 1177, 830, 747; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.16 (s, 1H), 10.14 (s, 1H), 8.71 (s, br, 1H), 8.12 (s, br, 1H), 7.92–7.90 (ss, 1H), 7.85 (d, J = 8.0 Hz, 1H), 7.80 (d, J = 9.0 Hz, 1H), 7.52–7.31 (m, 6H), 7.22 (d, J = 9.0 Hz, 1H), 7.14 (t, J = 7.5 Hz, 1H), 7.10 (d, J = 8.5 Hz, 2H), 6.85–6.82 (dd, J = 9.0 Hz, J = 2.0 Hz, 2H), 3.68 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 158.4, 153.5, 139.4, 134.6, 132.8, 129.8, 129.3, 129.1, 128.6, 128.5, 127.6, 127.4, 125.0, 123.8, 123.2, 119.0 × 2, 114.0, 55.5, 53.5; MS (EI) *m/z* (%): 380 (M-34, 2), 263 (10), 261 (100), 231 (78), 144 (73) 115 (28); Anal. Calcd for $C_{25}H_{22}N_2O_2S$: C, 72.44; H, 5.35; N, 6.76. Found: C, 72.24; H, 5.33; N, 6.72%.

3i: Colourless crystals; m.p. 168–170 °C; IR (KBr) cm^{-1}: 3363, 3186, 3012, 1630, 1544, 1518, 1438, 1265, 822, 745; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.14 (s, 1H), 10.11 (s, 1H), 8.67 (s, br, 1H), 8.15 (s, br, 1H), 7.92–7.90 (ss, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.80 (d, J = 9.0 Hz, 1H), 7.51–7.31 (m, 6H), 7.20 (d, J = 8.0 Hz, 1H), 7.14 (t, J = 7.5 Hz, 1H), 7.01 (d, J = 8.0 Hz, 1H), 6.96 (s, 1H), 6.89 (d, J = 8.0 Hz, 1H), 2.14 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 180.4, 153.6, 140.2, 139.5, 136.1, 134.7, 132.9, 129.8, 129.7, 129.3, 129.1, 128.6, 128.5, 127.4, 127.3, 126.0, 125.0, 123.9, 123.7, 123.2, 123.0, 119.3, 119.0, 53.6, 20.1, 19.4; MS (EI) *m/z* (%): 412 (M, 2), 262 (2), 245 (100), 144 (31); Anal. Calcd for $C_{26}H_{24}N_2OS$: C, 75.70; H, 5.86; N, 6.79. Found: C, 75.64; H, 5.85; N, 6.75%.

3j: Colourless crystals; m.p. 180–182 °C; IR (KBr) cm^{-1}: 3417, 3302, 3191, 1614, 1514, 1435, 1350, 1262, 1210, 1173, 811, 697; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.02 (s, 1H), 8.41–8.39 (ss, 1H), 8.13 (s, br, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.80 (d, J = 9.0 Hz, 1H), 7.76–7.74 (ss, 1H), 7.50–7.47 (m, 3H), 7.32 (t, J = 7.5 Hz, 1H), 7.25–7.20 (m, 4H), 7.16 (t, J = 7.5 Hz, 1H), 7.11 (d, J = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 183.7, 153.7, 143.4, 133.0, 129.8, 129.0, 128.6, 128.4, 127.2, 126.6, 126.2, 123.2, 123.0, 119.7, 119.0, 54.3; MS (EI) *m/z* (%): 274 (M-34, 2), 231 (55), 202 (14), 144 (100), 115 (33); Anal. Calcd for $C_{18}H_{16}N_2OS$: C, 70.10; H, 5.23; N, 9.08. Found: C, 69.97; H, 5.25; N, 9.13%.

3k: Yellow crystals; m.p. 168–170 °C; IR (KBr) cm^{-1}: 3422, 3356, 3096, 1626, 1529, 1437, 1350, 1267, 1210, 1171, 813, 749; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.09 (s, 1H), 9.17 (s, br, 1H), 8.13 (s, 2H), 8.12 (s, 1H), 8.03–8.02 (ss, 1H), 7.84–7.80 (dd, J = 11 Hz, J = 2.5 Hz, 2H), 7.63–7.49 (m, 3H), 7.31 (t, J = 7.5 Hz, 1H), 7.19 (d, J = 9.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 184.3, 153.9, 148.2, 146.3, 133.0, 132.8, 130.6, 130.1, 129.2, 128.7, 127.5, 123.2, 121.7, 120.7, 118.8, 118.3, 53.6; MS (EI) *m/z* (%): 319 (M-34, 3), 276 (20), 202 (3), 144 (100), 115 (19); Anal. Calcd for $C_{18}H_{15}N_3O_3S$: C, 61.18; H, 4.28; N, 11.89. Found: C, 61.26; H, 4.30; N, 11.97%.

3l: Colourless crystals; m.p. 166–168 °C; IR (KBr) cm^{-1}: 3447, 3285, 3172, 3066, 1605, 1546, 1515, 1436, 1360, 1260, 1144, 811, 747; ^{1}H NMR (500 MHz, DMSO-d_6): δ 9.85 (s, 1H), 8.33 (d, J = 5 Hz, 1H), 8.31 (d, J = 4.5 Hz, 1H), 7.79 (d, J = 8.0 Hz, 1H), 7.74 (d,

J = 9.0 Hz, 1H), 7.60 (t, J = 9.0 Hz, 2H), 7.48 (t, J = 7.5 Hz, 1H), 7.33–7.20 (m, 6H), 7.09 (d, J = 8.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 183.1, 154.0, 140.8, 133.3, 132.3, 130.4, 130.1, 129.4, 128.9, 128.6, 128.3, 126.9, 126.4, 123.8, 122.9, 119.0, 118.1, 53.5; MS (EI) *m/z* (%): 308 (M-34, 3), 231 (100), 202 (20), 144 (53), 115 (20); Anal. Calcd for $C_{18}H_{15}ClN_2OS$: C, 63.06; H, 4.41; N, 8.17. Found: C, 63.14; H, 4.45; N, 8. 26%.

3m: Colourless crystals; m.p. 164°C; IR (KBr) cm^{-1}: 3439, 3230, 3145, 1600, 1541, 1514, 1438, 1351, 1267, 1211, 1145, 813, 732; ^{1}H NMR (500 MHz, DMSO-d_6):δ 9.97 (s, 1H), 8.39–8.37 (ss, 1H), 8.15 (s, br, 1H), 7.82 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 9 Hz, 1H), 7.71–7.69 (ss, 1H), 7.47 (br, 3H), 7.30 (t, J = 7.5 Hz, 1H), 7.21 (d, J = 9.0 Hz, 1H), 6.98 (d, J = 8.0 Hz, 1H), 6.92 (s, 1H), 6.83 (d, J = 7.5 Hz, 1H), 2.13 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 183.5, 153.6, 140.8, 135.8, 134.3, 133.1, 129.6, 129.5, 128.6, 127.3, 127.1, 123.8, 123.3, 123.0, 120.0, 119.1, 54.1, 20.1, 19.4; MS (EI) *m/z* (%): 302 (M-34, 3), 259 (19), 245 (100), 202 (5), 144 (100) 115 (41); Anal. Calcd for $C_{20}H_{20}N_2OS$: C, 71.40; H, 5.99; N, 8.33. Found: C, 71.51; H, 6.05; N, 8.32%.

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