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## ACS Publications

Supporting Information (R.J. Booth and J.C. Hodges, Polymer-Supported Quenching Reagents for Parallel Purification)

## 1-(3-Isopropoxy-propyl)-3-(2-phenylethyl)-thiourea (9).

A solution of 3-isopropoxypropyl amine ( $7,25 \mathrm{mg}, 0.25 \mathrm{mmol}$ ) and 2-phenylethylisothiocyanate ( $44 \mu \mathrm{l}, 0.3 \mathrm{mmol}$ ) in $\mathrm{DCM}(2 \mathrm{ml})$ was shaken for 1.5 hr and then polymer-supported tris $(2-$ aminoethyl)amine ( $1,50 \mathrm{mg}$ ) was added. After 2 hr the resin was filtered and washed with DCM $(2 \times 1.5 \mathrm{ml})$. The combined organic phases, when concentrated to dryness, gave the title compound ( $64 \mathrm{mg}, 92 \%$ ) as an oil. $\mathrm{R}_{\mathrm{t}}=6.92$; $\mathrm{IR}(\mathrm{KBr}) 1551 ;{ }^{1} \mathrm{H}$ NMR $\partial 1.08(6 \mathrm{H}, \mathrm{d}, J=6)$, $1.77(2 \mathrm{H}, \mathrm{m}), 2.89(2 \mathrm{H}, \mathrm{t}, J=7), 3.5(6 \mathrm{H}, \mathrm{m}), 3.6(1 \mathrm{H}, \mathrm{brs}), 6.08(1 \mathrm{H}, \mathrm{brs}), 6.68(1 \mathrm{H}, \mathrm{brs})$, $7.20(3 \mathrm{H}, \mathrm{m}), 7.28(2 \mathrm{H}, \mathrm{m}) ;{ }^{13} \mathrm{C}$ NMR 22.1 (2C), 28.9, 35.11, 45.37, 67.0, 71.9, 77.0, 126.7, 128.7 (2C), 128.75 (2C), 138.2, 181.22; Predicted mass for $\left(\mathrm{C}_{15} \mathrm{H}_{24} \mathrm{~N}_{2} \mathrm{OS}+\mathrm{H}\right)^{+}, 281.1688$; Found by HRMS (CI), 281.1675.

1-(4-Methoxy-benzenesulfonyl)-4-pyridin-2-yl-piperazine (12).
A suspension of polymer-supported morpholine ( $3,100 \mathrm{mg}$ ), 1-(2-pyridyl)piperazine ( $10,26 \mu \mathrm{l}$, 0.25 mmol ) and 4-methoxybenzenesulphonyl chloride (11, $62 \mathrm{mg}, 0.3 \mathrm{mmol}$ ) in DCM ( 2 ml ) was shaken for 1 hr . Then polymer-supported tris(2-amnioethyl)amine (1, 200 mg) was added. After 24 hr the resin was filtered and washed with $\mathrm{DCM}(2 \times 1.5 \mathrm{ml})$. The combined organic phases, when concentrated to dryness, gave the title compound ( $54 \mathrm{mg}, 65 \%$ ) as a white solid. mp $148-152^{\circ} \mathrm{C} ; \mathrm{R}_{\mathrm{t}}=5.51 ; \operatorname{IR}(\mathrm{KBr}) 1345,1160 ;{ }^{1} \mathrm{H}$ NMR $\partial 3.05(4 \mathrm{H}, \mathrm{t}, J=5), 3.59(4 \mathrm{H}, \mathrm{t}, J=5)$, $3.82(3 \mathrm{H}, \mathrm{s}), 6.55(1 \mathrm{H}, \mathrm{d}, J=9), 6.59(1 \mathrm{H}, \mathrm{dd}, J=7,5), 6.95(2 \mathrm{H}, \mathrm{d}, J=7), 7.43(1 \mathrm{H}, \mathrm{dd}, J=$ $9,7), 7.67(2 \mathrm{H}, \mathrm{d}, J=7), 8.11(1 \mathrm{H}, \mathrm{d}, J=5) ;{ }^{13} \mathrm{C}$ NMR $44.8(2 \mathrm{C}), 45.8(2 \mathrm{C}), 55.65,107.5$, $113.95,114.3$ (2C), 126.88, 129 (2C), 138.1, 147.8, 158.2, 163.2; Predicted mass for
$\left(\mathrm{C}_{16} \mathrm{H}_{19} \mathrm{~N}_{3} \mathrm{O}_{3} \mathrm{~S}+\mathrm{H}\right)^{+}, 334.1255$; Found by HRMS (CI), 334.1220. Recrystallized mp 156$157^{\circ} \mathrm{C}$ (EtOAc/hexanes). Calc'd for $\mathrm{C}_{16} \mathrm{H}_{19} \mathrm{~N}_{3} \mathrm{O}_{3} \mathrm{~S}: \mathrm{C}, 57.64 ; \mathrm{H}, 5.74 ; \mathrm{N}, 12.60$. Found: C , 57.43; H, 5.82; N, 12.34.

## R-N-(2-Hydroxy-1-phenyl-ethyl)-4-methyl-benzamide (15).

A suspension of polymer-supported morpholine ( $\mathbf{3}, 85 \mathrm{mg}$ ), R-(-)-2-phenylglycinol ( $14,41 \mathrm{mg}$, 0.3 mmol ) and 4-methylbenzoyl chloride ( $13,33 \mu \mathrm{l}, 0.25 \mathrm{mmol}$ ) in DCM ( 2 ml ) was shaken for 1 hr. Polymer-supported isocyanate ( $2,150 \mathrm{mg}$ ) and polymer-supported tris(2-aminoethyl)amine (1, 50 mg ) were added and the reaction mixture was shaken for 2 hr . The resin was filtered and washed with DCM ( $2 \times 1.5 \mathrm{ml}$ ). The combined organic phases, when concentrated to dryness, gave the title compound ( $52 \mathrm{mg}, 81 \%$ ) as a white solid. $\mathrm{mp} 154-155^{\circ} \mathrm{C} ; \mathrm{R}_{\mathrm{t}}=5.65$; $\mathrm{IR}(\mathrm{KBr}) 1631$; ${ }^{1} \mathrm{H}$ NMR $\partial 2.38(3 \mathrm{H}, \mathrm{s}), 3.00(1 \mathrm{H}, \mathrm{brs}), 3.96(2 \mathrm{H}, \mathrm{d}, J=5), 5.23(1 \mathrm{H}, \mathrm{m}), 6.9(1 \mathrm{H}, \mathrm{d}, J=6)$, $7.0(2 \mathrm{H}, \mathrm{d}, J=8), 7.2-7.4(5 \mathrm{H}, \mathrm{m}), 7.69(2 \mathrm{H}, \mathrm{d}, J=8),{ }^{13} \mathrm{C}$ NMR $21.5,56.2,66.6,126.7$, 127.1 (2C), 127.9 (2C), 128.9, 129.3 (2C), 131.2 (2C), 139.1, 142.3, 167.9; Predicted mass for $\left(\mathrm{C}_{16} \mathrm{H}_{17} \mathrm{NO}_{2}+\mathrm{H}\right)^{+}, 256.1338$; Found by HRMS (CI), 256.1336. Recrystallized mp 157-158 ${ }^{\circ} \mathrm{C}$ (EtOAc/hexanes). Cald'd for $\mathrm{C}_{16} \mathrm{H}_{17} \mathrm{NO}_{2} \cdot 0.3 \mathrm{H}_{2} \mathrm{O}: \mathrm{C}, 73.71 ; \mathrm{H}, 6.80 ; \mathrm{N}, 5.37$. Found: C , 73.80; H, 6.60; N, 5.32.

Amide Mixture Synthesis (24, 25 and 26).
A mixture of $20(18.7 \mathrm{mg}, 0.066 \mathrm{mmol}), 21(19.5 \mathrm{mg}, 0.066 \mathrm{mmol})$ and $22(19.7 \mathrm{mg}, 0.066$ mmol ) was dissolved in DCM ( 2.2 mL ). An aliquot of this solution ( $1.0 \mathrm{~mL}, 0.09 \mathrm{mmol}$ ) was transferred to a vial containing $3(180 \mathrm{mg}, 0.6 \mathrm{mmol})$ and a miniature magnetic spinbar. Isobutyl chloroformate ( $15 \mu \mathrm{~L}, 0.11 \mathrm{mmol}$ ) was added, the vial was capped and the reaction was stirred at

RT for 30 min . A solution of $23(27 \mathrm{mg}, 0.15 \mathrm{mmol})$ in DCM ( 1.0 mL ) was added, and the reaction was stirred for $2.5 \mathrm{~h} .1(100 \mathrm{mg})$ and $\mathbf{2}(100 \mathrm{mg})$ were added and the resulting mixture was allowed to stand for 16 h at RT. Resin was collected by filtration and rinsed with DCM (5 mL ). Combined filtrate and washings were evaporated and dried at $0.25 \mathrm{mmHg}, \mathrm{RT}$ for 24 h to afford a mixture of $\mathbf{2 4}, 25$ and 26 as a gum ( $46 \mathrm{mg}, 112 \%$ ). TLC (Si gel, $\mathrm{CHCl}_{3}-\mathrm{EtOAc}^{2}$ 10:1) shows 3 major spots under UV light, $\mathrm{Rf}=0.9,0.5$ and 0.4 (staining gives no ninhydrin positive spots); ${ }^{1} \mathrm{H}$ NMR $\partial 4.93(1 \mathrm{H}, \mathrm{t}), 5.02(1 \mathrm{H}, \mathrm{t}), 5.19(1 \mathrm{H}, \mathrm{t})$ indicating 3 equal amide $\mathrm{NH}, 0.89$ ( $\sim 2 \mathrm{H}, \mathrm{d}$ ) indicating contamination by the isobutyl carbamate of 23 (see accompanying spectrum); Predicted masses for $\left(\mathrm{C}_{26} \mathrm{H}_{26} \mathrm{~N}_{3} \mathrm{FOS}+\mathrm{H}\right)^{+}, 448.1859,\left(\mathrm{C}_{24} \mathrm{H}_{29} \mathrm{NO}_{2} \mathrm{~S}_{3}+\mathrm{H}\right)^{+}, 460.1439$, and $\left(\mathrm{C}_{29} \mathrm{H}_{35} \mathrm{NO}_{2} \mathrm{~S}+\mathrm{H}\right)^{+}, 462.2467$; Found by HRMS (ES), 448.1868, 460.1445, and 462.2486. See also the HPLC chromatogram in text.

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