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bis[(*N*-Benzyloxycarbonyl)-(2*S*)-amino-4-methylpentyl]disulfide

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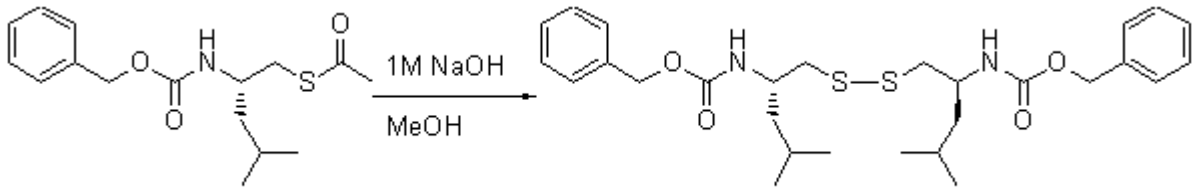

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Disulfides that mimic the pentapeptide Phe-Leu-Gly-Leu-Phe can be obtained in high yield from the corresponding thioacetate *via* the intermediate mercaptan by prolonged standing in alkali [1]. To a stirred solution of **1** (0.370 g, 1.20 mmol) in methanol (5 mL) was added dropwise a 1M solution of sodium methanoate in methanol (1.2 mL). The reaction mixture was stirred overnight and neutralised by dropwise addition of a 1M solution of HCl (1.2 mL). Methanol was evaporated under reduced pressure and the residue taken up with ethyl acetate (50 ml), washed with water (2 x 10 mL) and dried over anhydrous Na_2SO_4. Removal of the solvent under reduced pressure afforded the title compound as a white solid in 97% yield (0.310 g, 0.583 mmol).

M.p. 85-86 °C.

$[a]_D^{20}$ = -48.9° (c 1.38, CH_2Cl_2)

TLC (hexane/ethyl acetate 80:20): Rf 0.71.

IR (KBr, cm^{-1}): 3355 (br, s, NH), 1695 (s, C=O), 1535 (s, C=C), 1010 (m, C-O).

^{1}H-NMR (400 MHz, $CDCl_3$): 7.30 (m, 10H, Ph), 5.18 (d, J=8.4 Hz, 2H, NH), 3.96 (m, 2H, Ha), 2.96 (dd, J=14 Hz and J=5.2 Hz, 2H, CHS), 2.71 (dd, J=13.6 Hz and J=5.6 Hz, 2H, CHS), 1.65 (m, 2H, Hg), 1.19 (m, 4H, Hb), 0.90 (d, J=6.4 Hz, 12H, Hd).

^{13}C-NMR (100 MHz, $CDCl_3$): 156.0 (C=O), 136.5 (quat. arom.), 128.4 and 128.0 (CH arom.), 66.5 (CH_2-Ph), 49.0 (CH_2S), 45.4 (Ca), 42.4 (Cb), 24.8 (Cg), 23.0 and 21.0 (Cd).

ES-MS (m/z): 533 (M+H, 100%), 425 (25%), 266 (93%), 234 (76%), 91 (23%).

Anal. calc. for ($C_{28}H_{40}O_4N_2S_2$): C 63.12, H 7.57, N 5.26; found: C 63.06, H 7.89, N 5.05.

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Reference

1. Corelli, F.; Crescenza, A.; Dei, D.; Taddei, M.; Botta, M. *Tetrahedron Asymmetry* **1994**, 5, 1469-1472.

Sample availability : Available from the authors and from MPDI.