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# 1,2-Bis(*p*-phenyl-thiocarbamic Acid-*S*-benzothiazol-2-yl-ester)ethane and 1-(*o*-Phenyl-thiocarbamic Acid-*S*-benzothiazol-2-yl-ester)-2-(*p*-phenyl-thiocarbamic Acid-*S*-benzothiazol-2-yl-ester)ethane

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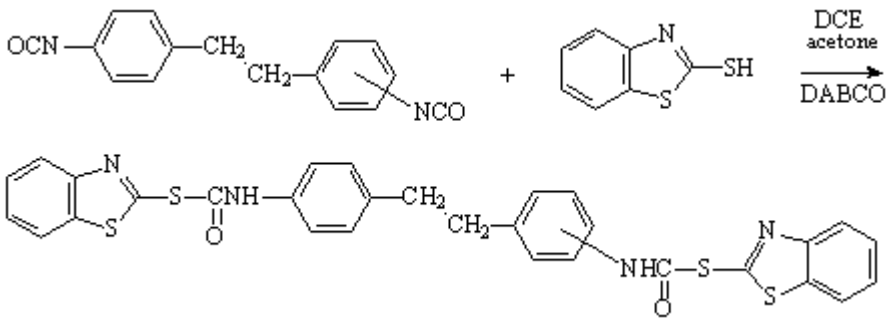


The experimental procedure follows the general synthesis of blocked isocyanates [1, 2]. A solution of mercaptobenzothiazole (MBT) was obtained by mixing 23 g (0.1373 mol) with 250 mL 1,2-dichloroethane (DCE) and 50 mL acetone, then heating gradually to 35-50°C for 0.5 h until the mixture became homogeneous. A solution of 1,2-bis(*p*-isocyanato-phenyl)ethane (4,4'-dibenzyl diisocyanate) [3, 4] (17 g, 0.0644 mol) in 100 mL DCE was added to this mixture under stirring during 0.25 h and then 0.2 g diazabicyclo[2.2.2]octane (DABCO) also was added. The mixture was stirred at 68-72°C for 6 - 8 h. After cooling, the light yellow solid was isolated by filtration, washed with DCE and acetone (to remove the excess of MBT) and dried at 90-100°C (22.5 g, 57.73% yield).

The synthesis of the nonsymmetrical isomer is described as follows. To a solution of MBT (5 g, 0.029895 mol) in 100 mL DCE and 15 mL acetone, 1-(*o*-isocyanato-phenyl)-2-(p-isocyanato-phenyl)ethane (2,4'-dibenzyl diisocyanate) (3.42 mL, 3.8 g, 0.01439 mol) and 0.1 g DABCO was added. The mixture was stirred for 10 h at 70-72°C. After cooling, the solid product was isolated by filtration, washed several times with acetone and dried (3 g, 34.84% yield).

## 1,2-Bis(*p*-phenyl-thiocarbamic acid-*S*-benzothiazol-2-yl-ester)ethane

Thermal behavior: M.p. >328°C (decomposition), (thermo-optical method)

IR (KBr, $cm^{-1}$): 3320 m (NH), 3030 mw (C-H), 2945 mw ($CH_2$), 2850 mw ($CH_2$), 1720-1700 m (NHCOS), 1670-1650 m (CONH), 1600 ms (*p*-Ar), 1550 s, 1530 s, 1510 s (SCONH), 1500 m (sh, *p*-Ar), 1410 m (C-S, MBT), 1320 m (MBT, *p*-Ar), 1240 ms (C-S, C-O), 1200 m (sh, C-N, C-O), 1080 m (C-O, C-N), 835 m (*p*-Ar), 750 mw (C-N, MBT), 660 mw (C-S).

$^1$H NMR (60 MHz, d, DMSO-$d_6$, 80°C): 8.95, 8.2 (NH, 2H), 7.2, 7.08, 6.95, 6.8 (m, Ar, 16H), 3.62 (s, $CH_2$-Ar, 4H) ppm.

Elemental analysis: Calculated for: $C_{30}H_{22}N_4O_2S_4$: C%= 60.18; H%= 3.37; N%= 9.36; S%= 21.42. Found: C%= 60.58; H%= 3.73; N%= 9.87.

## 1-(*o*-phenyl-thiocarbamic acid-*S*-benzothiazol-2-yl-ester)-2-(p-phenyl-thiocarbamic acid-*S*-

**benzothiazol-2-yl-ester)ethane**

Thermal behavior: M.p.> 228°C (decomposition), (thermo-optical method).

IR (KBr, $cm^{-1}$): 3300 ms (NH), 3040 w (C-H), 2940 w ($CH_2$), 2865 w ($CH_2$), 1700 m (sh, NHCOS), 1680-1655 ms (CONH), 1610-1590 ms (Ar), 1550 s, 1515 s (SCONH), 1500 ms (sh, Ar), 1450 ms, 1410 m (C-S, MBT), 1330-1300 m (MBT), 1260-1240 ms (C-S, C-O), 1180 m (sh, C-N, C-O), 1090 mw (C-O), 830 mw (*p*-Ar), 760 m (*o*-Ar, NNCOS), 700 w, 670 w (C-S).

$^1$H NMR (60 MHz, d, DMSO-$d_6$, 60°C): 8.56, 8.2, 7.95 (t, NH, 2H), 7.65, 7.45, 7.3, 7.2, 7.07, 6.95, 6.85 (m, Ar, 16H), 3.56 (m, $CH_2$-Ar, 4H) ppm.

Elemental analysis: Calculated for: $C_{30}H_{22}N_4O_2S_4$: C%= 60.18; H%= 3.37; N%= 9.36; S%= 21.42. Found: C%= 60.47; H%= 3.85; N%= 9.65.

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