

Addendum zu

Einige neue Thiaspirane

Über Spirane, 14. Mitt.¹

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Spektroskopische Daten und Elementaranalysen

3,3'-Spirobi(benzo[1,5]dithiepan) (1)

IR (KBr): 650, 700 (C—S—C), 1160, 1200, 1214, 1253, 2910 (C—H aliph.),
755, 3052 (C—H arom.), 1413, 1455 cm⁻¹ (C=C arom.).

UV (Dioxan): λ (lg ϵ) = 226 (4,33), 254 nm (4,39).

NMR (CS₂): 2,7—3,4 (s, 8 H —CH₂—), 7,4—7,6 ppm (m, 8 H arom.).

MS: M^+ m/e = 348.

C₁₇H₁₆S₄ (348,5). Ber. C 58,58, H 4,63, S 36,79.

Gef. C 58,55, H 4,94, S 37,03.

3,3'-Spirobi(naphtho[1.8-bc]-1,5-dithiocan) (2)

IR (KBr): 600—700 (C—S—C), 770, 820, 3060 (C—H arom.),
2900—2950 cm⁻¹ (—CH₂— aliph.).

UV (Ethanol): λ (lg ϵ) = 246 (4,38), 250 (4,29), 270 nm (4,00).

NMR (CDCl₃): 3,25 (s, 8 H, —CH₂—), 7,4—8,5 ppm (m, 12 H, arom.).

MS: M^+ m/e = 448.

C₂₅H₂₀S₄ (448,6). Ber. C 66,92, H 4,49, S 28,58.

Gef. C 66,86, H 4,61, S 28,28.

3,3'-Spirobi(trans-1,5-dithiabicyclo[5.3.0]decan) (3)

IR (KBr): 694 (C—S—C), 830 (C—C), 1414, 1448, 2845, 2910, 2951,
2970 cm⁻¹ (C—H aliph.).

UV (CH₂Cl₂): λ (lg ϵ) = 232 nm (3,05).

¹ Mh. Chem. **108**, 885 (1977).

NMR (C_4Cl_6): 1,20—2,25 (m, 12 H, „e“), 2,25—3,10 ppm (m, 12 H, „a“ und „b“).

MS: $M^+ m/e = 332$.

$C_{15}H_{24}S_4$ (332,6). Ber. C 54,16, H 7,27, S 38,57.
Gef. C 54,16, H 7,15, S 38,32.

3,3'-Bis(hydroxymethyl)-trans-1,5-dithiabicyclo[5.4.0]undecan (4)

IR (KBr): 667 (C—S—C), 1033 (—C—OH), 1340 (prim. OH), 1410, 1449, 1465, 2859, 2838 (C—H aliph.), 3390 cm^{-1} (O—H).

NMR ($CDCl_3$): 1,20—2,20 (m, 8 H, „e“), 2,35 (s, 4 H, „b“), 2,50—2,75 und 2,80 (m und s, 4 H, „c, d“), 3,62 ppm (s, 4 H, „a“).

$C_{11}H_{20}O_3S_2$ (248,4). Ber. C 53,18, H 8,12, S 25,82.
Gef. C 53,21, H 7,78, S 25,92.

Ditosylat des 3,3'-Bis(hydroxymethyl)-trans-1,5-dithiabicyclo[5.4.0]undecans (5)

IR (KBr): 670 (C—S—C), 1100, 1115, 1125, 3036, 3080 (C—H arom.), 1356 (—O—SO₂), 2860, 2940 cm^{-1} (C—H aliph.).

$C_{25}H_{32}O_6S_4$ (556,8). Ber. C 53,93, H 5,79, S 23,03.
Gef. C 53,94, H 5,72, S 23,31.

3,6'-Spiro(trans-1,5-dithiabicyclo[5.4.0]undecan-1,4-dithiepan) (6)

IR (KBr): 677 (C—S—C), 1419, 1452, 2800, 2860, 2911, 2932 cm^{-1} (C—H aliph.).

UV (CH_2Cl_2): λ ($lg \epsilon$) = 232 nm (2,99).

NMR ($CDCl_3$): 0,9—2,2 (m, 8 H, „b“), 2,2—3,0 ppm (m, 14 H, „a“).

MS: $M^+ m/e = 306$.

$C_{13}H_{22}S_4$ (306,5). Ber. C 50,93, H 7,23, S 41,84.
Gef. C 51,00, H 7,03, S 42,18.

3,3'-Spiro(cis-1,5-dithiabicyclo[5.4.0]undecan-trans-1,5-dithiabicyclo[5.4.0]undecan) (7)

IR (KBr): 697 (C—S—C), 831 (C—C), 1410, 1445, 2850, 2932 cm^{-1} (C—H aliph.).

UV (CH_2Cl_2): λ ($lg \epsilon$) = 232 nm (2,98).

NMR ($CDCl_3$): 0,8—2,1 (m, 16 H, „a“), 2,1—3,25 ppm (m, 12 H, „b“).

MS: $M^+ m/e = 360$.

$C_{17}H_{28}S_4$ (360,7). Ber. C 56,61, H 7,83, S 35,56.
Gef. C 56,80, H 7,35, S 35,57.

6,6-Bis(hydroxymethyl)-1,4-dithiepan (8)

IR (KBr): 645 (C—S—C), 745 (C—C), 1030, 1040, 1055 (C—OH), 1312, 1418 (prim. OH), 1460, 2880, 2905, 2918, 2932, 2945 (—CH₂—), 3390 cm^{-1} (—OH).

NMR ($CDCl_3$): 2,83 (s, 4 H, „b“), 2,97 (s, 4 H, „e“), 2,89 (s, 2 H, „d“), 3,68 ppm (s, 4 H, „c“).

MS: $M^+ m/e = 194$.

$C_7H_{14}O_2S_2$ (194,3). Ber. C 43,27, H 7,26, S 33,01.
Gef. C 43,22, H 7,17, S 32,94.

Ditosylat des 6,6-Bis(hydroxymethyl)-1,4-dithiepan (**9**)

IR (KBr): 645 (C—S—C), 1100, 1120, 3040, 3080, 3100 (aromat.), 1360 (—O—SO₂), 1417, 2895, 2980 cm⁻¹ (—CH₂—).

C₂₁H₂₆O₆S₄ (502,7). Ber. C 50,17, H 5,21, S 25,52.
Gef. C 50,81, H 5,21, S 25,17.

3,6'-Spiro(cis-1,5-dithiabicyclo[5.4.0]undecan-1,4-dithiepan) (**10**)

IR (KBr): 680 (C—S—C), 825, 1414, 1445, 2880, 2895, 2925, 2940 cm⁻¹ (—CH₂—).

UV (CH₂Cl₂): λ (lg ε) = 232 nm (3,07).

NMR (CDCl₃): 1,3—2,35 (m, 8 H, „e“), 2,35—3,0 (gem. ABq., J_{AB} = 15 Hz, „b“), 2,76 (s, 4 H, „d“), 2,9 (s, 4 H, „c“), 3,0—3,25 ppm (m, 2 H, „a“).

MS: M⁺ m/e = 306.

C₁₃H₂₂S₄ (306,5). Ber. C 50,93, H 7,23, S 41,84.
Gef. C 51,23, H 6,94, S 42,18.