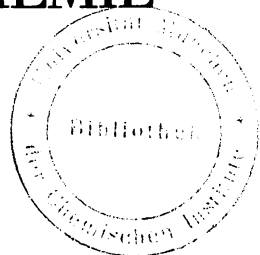


# LIEBIGS ANNALEN DER CHEMIE

HERAUSGEGEBEN VON DER  
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## Benzothiazole durch C – C-Spaltung von $\alpha$ -[(2-Nitrophenyl)thio]ketonen

Klaus Th. Wanner und Fritz Eiden\*

Institut für Pharmazie und Lebensmittelchemie der Universität München,  
Sophienstraße 10, D-8000 München 2

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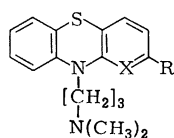
Einwirkung von Ammoniak bzw. Natronlauge auf die  $\alpha$ -[(2-Nitrophenyl)thio]ketone **4**, **11a – c** und **16** führt unter C – C-Spaltung zu den Benzothiazol-*N*-oxid-Derivaten **8**, **12a – c** und **17**; aus dem 3-[(2-Nitrophenyl)thio]-4-piperidon-Derivat **22** entsteht der 2-Benzothiazolcarbaldehyd **23**.

### Benzothiazoles by C – C Cleavage of $\alpha$ -[(2-Nitrophenyl)thio] Ketones

Reaction of  $\alpha$ -[(2-nitrophenyl)thio] ketones **4**, **11a – c**, and **16** with ammonia and sodium hydroxide solution, resp., leads to C – C cleavage and formation of the benzothiazole *N*-oxide derivatives **8**, **12a – c**, and **17**. The 3-[(2-nitrophenyl)thio]-4-piperidone derivative **22** yields 2-benzothiazolecarbaldehyde **23**.

Phenothiazine und Azaphenothiazine mit (Dialkylamino)alkyl-Seitenketten (z. B. **1a** und **c**) spielen als Arzneistoffe mit starker antipsychotischer Aktivität eine gewichtige Rolle<sup>1)</sup>. Die neuroleptische Wirkung kann durch Substitution in 2-Stellung des Phenothiazinmoleküls gesteigert werden (z. B. **1b**).

Schema I



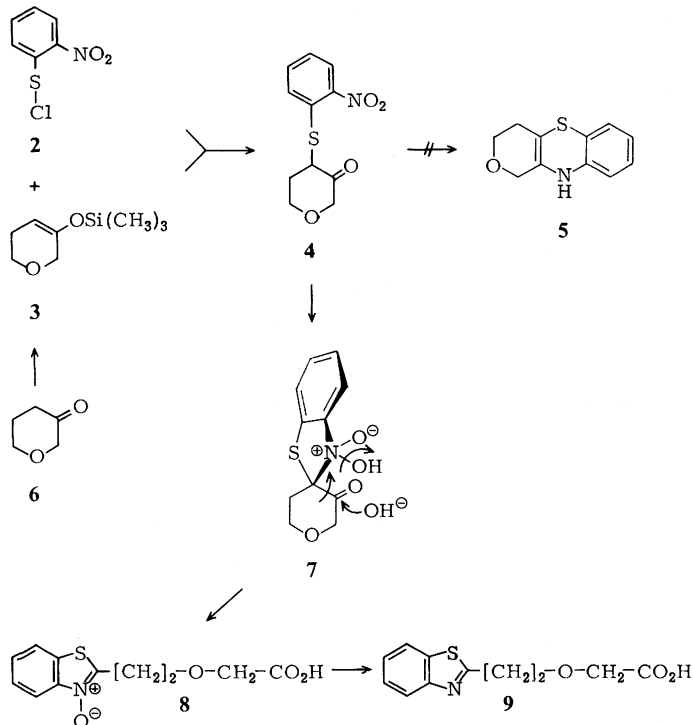
<b>1</b>	X	R	Namen
<b>a</b>	CH	H	Promazin, Protactyl <sup>®</sup>
<b>b</b>	CH	OCH <sub>3</sub>	Methopromazin, Tentone <sup>®</sup>
<b>c</b>	N	H	Prothipendyl, Dominal <sup>®</sup>

Wir versuchten, das Dihydro-1*H*-2-oxaphenothiazin **5** herzustellen, um es nach *N*-Substitution auf zentrale Wirksamkeit zu untersuchen.

Zur Synthese haben wir 5,6-Dihydro-2*H*-pyran-3(4*H*)on (**6**) durch Erhitzen in Tetrahydrofuran mit Trimethylsilylchlorid/Triethylamin zum Silylenolether umgesetzt, wobei in guter Ausbeute ausschließlich das  $\Delta^3$ -Isomer (**3**) entstand<sup>2)</sup>. Dieser durch <sup>1</sup>H-NMR-Spektroskopie gut identifizierbare Enolether reagierte mit (2-Nitrophenyl)sulfonylchlorid (**2**) zum 5,6-Dihydro-4-[(2-nitrophenyl)thio]-2*H*-pyran-3(4*H*)-on (**4**), das nun durch Reduktion mit Natriumdithionit in ammoniakalischer Lösung in das Oxaphenothiazin **5** übergeführt werden sollte. Wir erhielten eine farblose, kristalline Substanz, deren <sup>1</sup>H-NMR-Spektrum zur Formel **5** paßte [ $\delta$ (ppm) = 3.40 (t) und 3.97 (t), je 2H, *J* = 6 Hz (–OCH<sub>2</sub>CH<sub>2</sub>–), 4.10 (s), 2H (–OCH<sub>2</sub>CO–), 7.28 – 7.67

(m) und 7.80 – 8.18 (m), je 2H (Benzolring), 12.5 (breit) 1H, H/D-Austausch]. Die Elementaranalysen dagegen, das Massenspektrum [ $m/e = 237 (M^+)$ ] und das IR-Spektrum (Banden bei 2200 – 3200 und 1730  $\text{cm}^{-1}$ ) sprechen für eine Aufspaltung des Pyranrings und Bildung eines Benzothiazol-Derivates.

Schema II



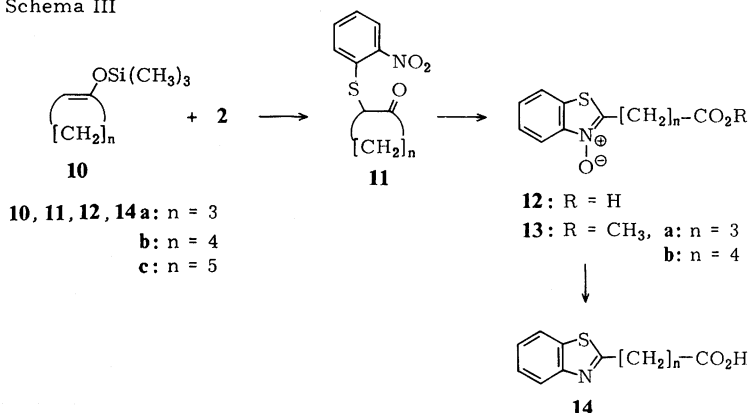
Dieser Reaktionsablauf läßt sich durch Addition der CH-aciden Gruppe in 3-Stellung des Pyranrings (oder des entsprechenden Anions) an die Nitrogruppe unter Bildung von **7** und Pyranringaufspaltung durch OH-Ionen erklären. Das Benzothiazol-*N*-oxid **8** wird dann mit Natriumdithionit zum Benzothiazol-Derivat **9** reduziert. Tatsächlich entstand bei der Wiederholung der Reaktion in Abwesenheit des Reduktionsmittels das *N*-Oxid **8** in guter Ausbeute.

Das Verfahren ließ sich auch mit carbocyclischen Ketonen bzw. ihren Silylenolethern **10a – c** durchführen. Mit **2** entstanden die [(2-Nitrophenyl)thio]cyclohexanone **11a – c**; Einwirkung von Natronlauge führte dann zu den *N*-Oxiden der (2-Benzothiazolyl)alkansäuren **12a – c**. Diese ließen sich mit Natriumdithionit zu den Benzothiazol-Derivaten **14a – c** reduzieren. **12a** und **b** setzten sich mit Diazomethan zu den Estern **13a** und **b** um. Die (2-Benzothiazolyl)alkancarbonsäuren lassen sich auch durch Kondensation von 2-Aminothiophenol mit entsprechenden Dicarbonsäureanhydriden darstellen<sup>3)</sup>.

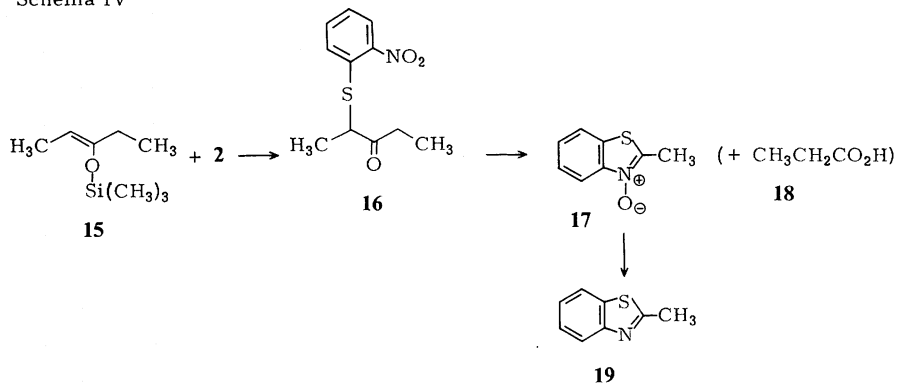
Auch der aus 3-Pentanon gewonnene Silylenolether **15**<sup>4,5)</sup> setzte sich mit **2** zu einem Nitrophenylthio-Derivat (**16**) um, das dann mit Natronlauge erwartungsgemäß unter

Abspaltung von (nicht nachgewiesener) Propansäure **18** das 2-Methylbenzothiazol-*N*-oxid **17** bildete<sup>6)</sup>; durch Reduktion entstand daraus das entsprechende – auch anders herstellbare<sup>7)</sup> – Benzothiazol-Derivat **19**.

Schema III

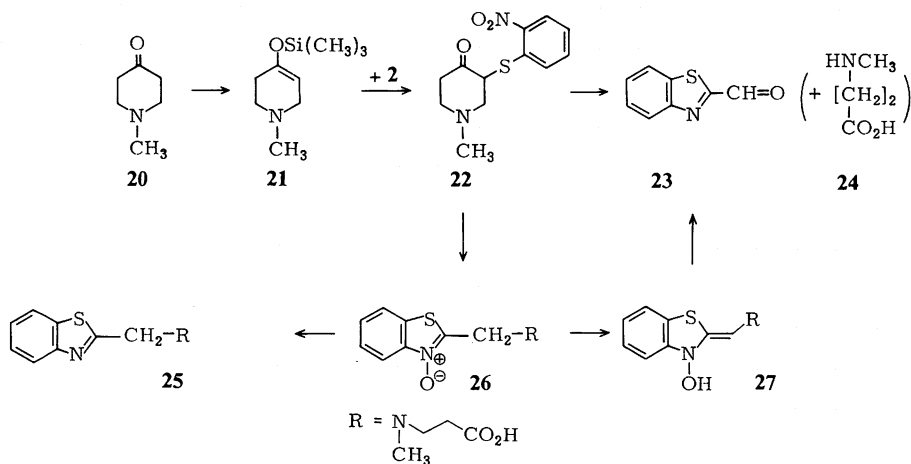


Schema IV



Nicht ohne weiteres zu erwarten war das Ergebnis der Umsetzung von *N*-Methyl-4-piperidon (**20**). Das aus **20** über **21** hergestellte 1-Methyl-3-[(2-nitrophenyl)thio]-4-piperidon (**22**) reagierte zwar mit Natronlauge, isoliert wurde jedoch nicht die Carbonsäure **26**, sondern der 2-Benzothiazolcarbaldehyd (**23**), der bereits auf anderem Wege dargestellt worden ist<sup>8)</sup>. Dabei ist anzunehmen, daß auch hier – in Analogie zur Bildung von **8**, **12** und **17** – zuerst das *N*-Oxid **26** entstanden ist, da sich durch Behandeln von **22** mit 0.01 *N* Ba(OH)<sub>2</sub> eine instabile Verbindung isolieren ließ, deren <sup>1</sup>H-NMR-Spektrum zur Struktur **26** paßt; das Spektrum veränderte sich jedoch nach kurzer Zeit. Wir schlagen als Weg zur Bildung von **23** die Folge **22** → **26** → **27** → **23** vor, wenn es uns bisher auch nicht gelungen ist, **26** rein zu isolieren. Gut paßt zu dieser Vorstellung, daß wir nach dem Zufügen von Natriumdithionit zur neutralisierten Lösung von **26** ein Produkt isolieren konnten, bei dem es sich nach Analysendaten und Spektren um die *N*-(2-Benzothiazolylmethyl)aminopropansäure **25** handelt.

Schema V



Wir danken dem *Fonds der Chemischen Industrie* für die finanzielle Unterstützung unserer Arbeit, Frau *A. Kärtner* für ihre engagierte Mitarbeit beim Experimentieren.

## Experimenteller Teil

Schmelz- und Siedepunkte sind nicht korrigiert. – IR-Spektren: Accu Lab 6 (Beckman). –  $^1\text{H-NMR}$ -Spektren: A 60 bzw. T 60 (Varian). – Massenspektren: CH7 (Varian).

Darstellung von **3** wie in Lit.<sup>2)</sup> beschrieben, von **10a** – **c** und **15** nach dem von *House*<sup>4)</sup> angegebenen Verfahren (Erhitzen des Ketons mit Chlortrimethylsilan und Triethylamin und Triethylamin in Dimethylformamid). **10a** und **b** sind in Lit.<sup>4)</sup> beschrieben, **10c** und **15** in Lit.<sup>5)</sup>

*1,2,3,6-Tetrahydro-1-methyl-4-(trimethylsiloxy)pyridin (21)*: Eine Mischung aus 43.13 g (396 mmol) Chlortrimethylsilan, 80.10 g (792 mmol) Triethylamin und 33.90 g (300 mmol) **20** wurde in 120 ml absol. Dioxan unter Stickstoff 72 h rückfließend erhitzt. Nach Abdestillieren des Lösungsmittels i. Vak. wurde mit Pentan versetzt, filtriert und das Filtrat destilliert. Sdp. 80 – 85°C/12 – 15 Torr, Ausb. 25.2 g (45%). – IR (Film): 1675, 1460, 1365  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta = 0.18$  (s; 9H), 1.97 – 2.33 (m; 2H), 2.37 (s; 3H), 2.47 – 2.75 (m; 2H), 2.85 – 3.04 (m; 2H), 4.71 – 4.92 (m; 1H).

$\text{C}_9\text{H}_{19}\text{NOSi}$  (185.3) Ber. C 58.32 H 10.33 N 7.56

Gef. C 58.20 H 10.07 N 7.58 Molmasse 185 (MS)

### A) Allgemeine Vorschrift zur Darstellung der $\alpha$ -[(2-Nitrophenyl)thio]ketone **4**, **11a** – **c**, **16** und **22**

Der (Trimethylsilyl)enoether wurde unter Stickstoff in absol. Dichlormethan gelöst, auf die angegebene Temp. abgekühlt und unter Rühren tropfenweise mit einer Lösung von **2** in absol. Dichlormethan versetzt. Nach der angegebenen Reaktionszeit und -temp. wurde i. Vak. vom Lösungsmittel befreit und das Rohprodukt wie angegeben gereinigt.

*5,6-Dihydro-4-[(2-nitrophenyl)thio]-2H-pyran-3(4H)-on (4)*: 2.58 g (15.0 mmol) **3** in 15 ml absol. Dichlormethan und 2.84 g (15.0 mmol) **2** in 15 ml absol. Dichlormethan wurden bei – 50 bis – 40°C nach Vorschrift A umgesetzt und anschließend bei Raumtemp. 12 h gerührt. Aus Toluol gelbe Kristalle, Schmp. 118 – 118.5°C, Ausb. 1.1 g (29%). – IR (KBr): 2910, 1723,

1510  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  = 1.97–2.90 (m; 2H), 3.78–4.53 (m; 5H), 6.97–7.70 (m; 3H), 7.88–8.13 (m; 1H).

$\text{C}_{11}\text{H}_{11}\text{NO}_4\text{S}$  (253.3) Ber. C 52.17 H 4.38 N 5.53 S 12.66  
Gef. C 52.03 H 4.41 N 5.60 S 12.65 Molmasse 253 (MS)

*2-[(2-Nitrophenyl)thio]-1-cyclopentanon (11a)*: 1.56 g (10.0 mmol) **10a** in 15 ml absol. Dichlormethan wurden mit 1.70 g (9.00 mmol) **2** in 15 ml absol. Dichlormethan bei  $-70^\circ\text{C}$  nach Vorschrift A umgesetzt. Dann wurde 1 h bei Raumtemp. gerührt und nach dem Abdestillieren des Dichlormethans zweimal aus Essigester umkristallisiert. Zitronengelbe Kristalle, Schmp.  $70-72^\circ\text{C}$ , Ausb. 1.2 g (56%). – IR (KBr): 1735, 1515, 1335  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  = 1.8–2.8 (s; 6H), 3.9 (t,  $J$  = 6.5 Hz; 1H), 7.20–8.05 (m; 3H), 8.23 (dd,  $J$  = 8/1.5 Hz; 1H).

$\text{C}_{11}\text{H}_{11}\text{NO}_3\text{S}$  (237.3) Ber. C 55.68 H 4.67 N 5.90 S 13.51  
Gef. C 55.51 H 4.60 N 5.99 S 13.57 Molmasse 237 (MS)

*2-[(2-Nitrophenyl)thio]-1-cyclohexanon (11b)*: Nach Vorschrift A wurden 11.07 g (65.0 mmol) **10b** in 65 ml absol. Dichlormethan mit 12.35 g (65.0 mmol) **2** in 65 ml absol. Dichlormethan bei  $-70^\circ\text{C}$  versetzt. Man ließ dann 1 h bei  $-70^\circ\text{C}$  und 3 h bei Raumtemp. reagieren. Aus Essigester gelbe Kristalle, Schmp.  $108-109^\circ\text{C}$  (Lit.<sup>9</sup> Schmp.  $113-114^\circ\text{C}$ ), Ausb. 8.59 g (53%). – IR (KBr): 2920, 1705, 1590  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  = 1.52–2.63 (m; 7H), 2.63–3.23 (m; 1H), 4.02 (t,  $J$  = 5 Hz; 1H), 7.0–7.6 (m; 3H), 8.08 (dd,  $J$  = 8/1 Hz; 1H).

$\text{C}_{12}\text{H}_{13}\text{NO}_3\text{S}$  (251.3) Ber. C 57.35 H 5.21 N 5.57 S 12.76  
Gef. C 57.12 H 5.22 N 5.58 S 12.70 Molmasse 251 (MS)

*2-[(2-Nitrophenyl)thio]-1-cycloheptanon (11c)*: Nach Vorschrift A wurden 9.22 g (50 mmol) **10c** in 50 ml absol. Dichlormethan mit 8.53 g (45 mmol) **2** in 45 ml absol. Dichlormethan bei  $-70^\circ\text{C}$  umgesetzt. Dann wurde auf Raumtemp. erwärmt. Nach wiederholtem Umkristallisieren aus Methanol und Essigester gelbe Kristalle, Schmp.  $87-91^\circ\text{C}$ , Ausb. 4.02 g (34%). – IR (KBr): 2940, 1710, 1510  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  = 1.15–3.05 (m; 10H), 4.07 (dd,  $J$  = 9.5/5.5 Hz; 1H), 7.15–7.95 (m; 3H), 8.2 (dd,  $J$  = 8/1.5 Hz; 1H).

$\text{C}_{13}\text{H}_{15}\text{NO}_3\text{S}$  (265.3) Ber. C 58.85 H 5.70 N 5.28 S 12.08  
Gef. C 58.70 H 5.63 N 5.37 S 12.13 Molmasse 265 (MS)

*2-[(2-Nitrophenyl)thio]-3-pentanon (16)*: Nach Vorschrift A wurden 1.58 g (10.0 mmol) **15** in 15 ml absol. Dichlormethan bei  $-70^\circ\text{C}$  mit 1.7 g (9.0 mmol) **2** in 15 ml absol. Dichlormethan umgesetzt. Man erwärmte anschließend auf Raumtemp. Der Rückstand wurde mit Methanol versetzt, Unlösliches abfiltriert und das Filtrat i. Vak. eingengt. Die bei  $5^\circ\text{C}$  sich abscheidenden Kristalle wurden aus Methanol umkristallisiert, Schmp.  $34.5-36^\circ\text{C}$ , Ausb. 1.18 g (55%). – IR (KBr): 1710, 1520  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  = 1.05 (t,  $J$  = 7.5 Hz; 3H), 1.58 (d,  $J$  = 7.5 Hz; 3H), 2.68 (q,  $J$  = 7.5 Hz; 2H), 4.07 (q,  $J$  = 7.5 Hz; 1H), 7.22–7.72 (m; 3H), 8.25 (d,  $J$  = 8 Hz; 1H).

$\text{C}_{11}\text{H}_{13}\text{NO}_3\text{S}$  (239.3) Ber. C 55.21 H 5.48 N 5.85 S 13.40  
Gef. C 55.12 H 5.38 N 5.53 S 13.41 Molmasse 239 (MS)

*1-Methyl-3-[(2-nitrophenyl)thio]-4-piperidon (22)*: Nach Vorschrift A wurden 7.41 g (40.0 mmol) **21** in 50 ml absol. Dichlormethan bei  $-45$  bis  $-30^\circ\text{C}$  mit 6.82 g (36.0 mmol) **2** in 50 ml absol. Dichlormethan umgesetzt. Nach 30 min bei  $-40^\circ\text{C}$  wurde mit 10 ml Wasser und nach Erwärmen auf Raumtemp. mit Natriumhydrogencarbonat-Lösung versetzt. Die organische Phase wurde i. Vak. eingengt und auf eine Kieselgelsäule gebracht (Korngröße 0.063–0.200 mm, Merck,  $\varnothing$  2.7 cm, H = 27 cm). Nach Spülen mit 250 ml Essigester/Dichlormethan (2: 8) wurde **22** mit Essigester eluiert. Aus Essigester gelbe Kristalle, Schmp.  $107-109^\circ\text{C}$ , Ausb. 3.58 g



(37%). – IR (KBr): 1710, 1510  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta = 2.20\text{--}3.45$  (m; 6H), 2.45 (s; 3H), 3.86–4.13 (m; 1H), 7.20–7.93 (m; 3H), 8.11–8.33 (m; 1H).

$\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$  (266.3) Ber. C 54.12 H 5.30 N 10.52 S 12.04

Gef. C 53.98 H 5.27 N 10.51 S 11.93 Molmasse 266 (MS)

*5-(2-Benzothiazolyl)-3-oxapentansäure-3'-oxid* (**8**): 0.18 g (0.70 mmol) **4** wurden bei Raumtemp. in 15 ml Ethanol suspendiert und mit 1.5 ml konz. Ammoniak versetzt (die sofort eintretende Rotfärbung verblaßte nach 10 min). Nach 2 h wurde das Lösungsmittel i. Vak. entfernt, der Rückstand in Wasser gelöst, die Lösung mit 6 N HCl auf pH 2–3 eingestellt und mit Chloroform extrahiert. Die mit  $\text{Na}_2\text{SO}_4$  getrocknete Chloroformphase wurde i. Vak. eingengt und der Rückstand aus Methanol umkristallisiert. Farblose Kristalle, Schmp. 126–128°C (Zers.), Ausb. 0.12 g (68%). – IR (KBr): 1735, 1210  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $[\text{D}_6]\text{DMSO}$ ):  $\delta = 3.43$  (t,  $J = 5.5$  Hz; 2H), 3.93 (t,  $J = 5.5$  Hz; 2H), 4.13 (s; 2H), 7.45–7.83 (m; 2H), 7.83–8.27 (m; 2H), 11.6–13.3 (1H,  $\text{D}_2\text{O}$ -Austausch). – MS:  $m/e = 237$  ( $\text{M}^+ - \text{O}$ ).

$\text{C}_{11}\text{H}_{11}\text{NO}_4\text{S}$  (253.3) Ber. C 52.17 H 4.38 N 5.53 S 12.66

Gef. C 51.80 H 4.42 N 5.37 S 12.45

**B) Allgemeine Vorschrift zur Darstellung der Benzothiazol-N-oxide 12a–c, 17 und des 2-Benzothiazolcarbaldehyds 23**

Eine Mischung aus 10 ml 1 N NaOH, 90 ml Tetrahydrofuran und 45 ml Wasser wurde unter Rühren tropfenweise mit einer Lösung des [(Nitrophenyl)thio]ketons (5.0 mmol) in 15–30 ml Tetrahydrofuran (je nach Löslichkeit) versetzt, wobei sich die Reaktionslösung anfangs violett und dann allmählich braun färbte. Anschließend wurde mit 16 ml 1 N HCl versetzt bzw. bei **17** und **23** auf pH = 7 eingestellt und i. Vak. weitgehend eingengt. Wenn sich dabei ein Niederschlag bildete, wurde dieser isoliert und mit Wasser gewaschen. Sonst wurde mehrfach mit Dichlormethan extrahiert, der Dichlormethan-Extrakt mit Wasser gewaschen, mit  $\text{Na}_2\text{SO}_4$  getrocknet und i. Vak. eingengt. Der Rückstand wurde wie angegeben umkristallisiert.

*4-(2-Benzothiazolyl)butansäure-3'-oxid* (**12a**): Nach Vorschrift B schwach gelbe Kristalle; Schmp. 172–174°C (Methanol), Ausb. 0.53 g (44%). – IR (KBr): 3100–1750, 1680  $\text{cm}^{-1}$  (br). –  $^1\text{H-NMR}$  ( $[\text{D}_6]\text{DMSO}$ ):  $\delta = 1.7\text{--}2.53$  (m; 4H), 3.18 (t,  $J = 7.5$  Hz; 2H), 7.57–7.92 (m; 2H), 7.97–8.43 (m; 2H), 11.7–12.7 (1H,  $\text{D}_2\text{O}$ -Austausch).

$\text{C}_{11}\text{H}_{11}\text{NO}_3\text{S}$  (237.3) Ber. C 55.68 H 4.67 N 5.90 S 13.51

Gef. C 55.57 H 4.64 N 5.91 S 13.37 Molmasse 237 (MS)

*5-(2-Benzothiazolyl)pentansäure-3'-oxid* (**12b**): Nach Vorschrift B farblose Kristalle; Schmp. 145–147°C (Methanol), Ausb. 0.85 g (68%). – IR (KBr): 3200–2300, 1710  $\text{cm}^{-1}$ . –  $^1\text{H-NMR}$  ( $[\text{D}_6]\text{DMSO}$ ):  $\delta = 1.43\text{--}2.10$  (m; 4H), 2.37 (t,  $J = 6$  Hz; 2H), 3.20 (t,  $J = 6$  Hz; 2H), 7.50–7.93 (m; 2H), 7.93–8.45 (m; 2H), 11.6–12.5 (1H,  $\text{D}_2\text{O}$ -Austausch).

$\text{C}_{12}\text{H}_{13}\text{NO}_3\text{S}$  (251.3) Ber. C 57.35 H 5.21 N 5.57 S 12.76

Gef. C 57.25 H 5.15 N 5.46 S 12.94 Molmasse 251 (MS)

*6-(2-Benzothiazolyl)hexansäure-3'-oxid* (**12c**): Nach Vorschrift B schwach gelbe Kristalle; Schmp. 137–138°C (Methanol), Ausb. 0.9 g (68%). – IR (KBr): 3100–1750, 1690  $\text{cm}^{-1}$  (br). –  $^1\text{H-NMR}$  ( $[\text{D}_6]\text{DMSO}$ ):  $\delta = 1.1\text{--}1.9$  (m; 6H), 2.08–2.43 (m; 2H), 3.13 (t,  $J = 7$  Hz; 2H), 7.6–7.9 (m; 2H), 7.97–8.37 (m; 2H), 12.0 (br.; 1H,  $\text{D}_2\text{O}$ -Austausch).

$\text{C}_{13}\text{H}_{15}\text{NO}_3\text{S}$  (265.3) Ber. C 58.85 H 5.70 N 5.28 S 12.08

Gef. C 58.78 H 5.67 N 5.29 S 11.95 Molmasse 265 (MS)

*2-Methylbenzothiazol-3-oxid* (**17**): Nach Vorschrift B hellgelbe Kristalle (Dihydrat); Schmp. 45–47°C (wasserhaltiges Acetonitril) (Lit.<sup>6</sup>) Schmp. 46–48°C, Ausb. 0.49 g (49%, ber. auf Dihydrat). – IR (KBr): 1660, 1495  $\text{cm}^{-1}$ . – MS:  $m/e = 165$  ( $\text{C}_8\text{H}_7\text{NOS}$ ). –  $^1\text{H-NMR}$

([D<sub>6</sub>]DMSO):  $\delta$  = 2.67 (s; 3H), 3.37 (s; 4H, D<sub>2</sub>O-Austausch), 7.52–7.88 (m; 2H), 7.93–8.33 (m; 2H).

C<sub>8</sub>H<sub>7</sub>NOS · 2H<sub>2</sub>O (201.2) Ber. C 47.75 H 5.51 N 6.96 S 15.93  
Gef. C 48.16 H 5.23 N 6.97 S 16.03

**2-Benzothiazolcarbaldehyd (23):** Nach Vorschrift B farblose Kristalle; Schmp. 70–73 °C (Petrolether) (Lit.<sup>8</sup>) Schmp. 72–74 °C, Ausb. 0.38 g (47%). – IR (KBr): 1695, 1485 cm<sup>-1</sup>. – <sup>1</sup>H-NMR (CDCl<sub>3</sub>):  $\delta$  = 7.57–7.90 (m; 2H), 7.97–8.5 (m; 2H), 10.33 (s; 1H).

C<sub>8</sub>H<sub>5</sub>NOS (163.2) Ber. C 58.88 H 3.09 N 8.58 S 19.65  
Gef. C 58.76 H 3.22 N 8.46 S 19.38 Molmasse 163 (MS)

**C) Allgemeine Vorschrift zur Darstellung der Benzothiazole 9 und 14a–c**

Das N-Oxid wurde in Wasser suspendiert, bei 70–80 °C (Badtemp.) tropfenweise mit einer wäßrigen Natriumdithionit-Lösung versetzt und die Mischung bei dieser Temp. die angegebene Zeit gerührt. Wenn nach dem Abkühlen ein Niederschlag auftrat, wurde dieser isoliert, mit Wasser gewaschen und mit dem Rückstand vereinigt, der anschließend durch mehrfache Dichlormethan-Extraktion gewonnen wurde. Dann wurde wie angegeben gereinigt.

**5-(2-Benzothiazolyl)-3-oxapentansäure (9)**

a) Nach Vorschrift C: 0.05 g (0.2 mmol) **8** in 2 ml Wasser; 0.29 g (1.6 mmol) Natriumdithionit in 5 ml Wasser; Reaktionszeit 30 min; Schmp. 136–138 °C; Ausb. 0.04 g (84% nicht umkristallisiert).

b) 1.27 g (5.00 mmol) **4** in 100 ml Ethanol wurden in der Siedehitze unter Rühren mit 10 ml konz. Ammoniak und anschließend mit 2.61 g (15.0 mmol) Natriumdithionit in 15 ml Wasser versetzt. Es wurde 1 h rückfließend erhitzt, nach dem Abkühlen filtriert und das Filtrat i. Vak. eingengt. Der Rückstand wurde mit Diethylether gewaschen und in Wasser gelöst. Die Lösung wurde mit 2 N HCl auf pH = 2–3 eingestellt und mehrfach mit Chloroform extrahiert. Nach dem Einengen der Chloroform-Extrakte wurde aus Ethanol umkristallisiert. Farblose Kristalle, Schmp. 135–137 °C, Ausb. 0.25 g (21%). – IR (KBr): 3200–2200, 1730, 1440 cm<sup>-1</sup>. – <sup>1</sup>H-NMR ([D<sub>6</sub>]DMSO):  $\delta$  = 3.40 (t,  $J$  = 6.0 Hz; 2H), 3.97 (t,  $J$  = 6 Hz; 2H), 4.10 (s; 2H), 7.28–7.67 (m; 2H), 7.80–8.18 (m; 2H), 12.5 (br.; 1H, D<sub>2</sub>O-Austausch).

C<sub>11</sub>H<sub>11</sub>NO<sub>3</sub>S (237.3) Ber. C 55.68 H 4.67 N 5.90 S 13.51  
Gef. C 55.62 H 4.75 N 5.98 S 13.43 Molmasse 237 (MS)

**4-(2-Benzothiazolyl)butansäure (14a):** Nach Vorschrift C: 0.84 g (3.5 mmol) **12a** in 40 ml Wasser; 3.15 g (17.5 mmol) Natriumdithionit in 30 ml Wasser; Reaktionszeit 14 h. Farblose Kristalle, Schmp. 145–147 °C (Methanol) (Lit.<sup>3</sup>) Schmp. 145–146 °C, Ausb. 0.49 g (64%). – IR (KBr): 3200–2200, 1705 cm<sup>-1</sup>. – <sup>1</sup>H-NMR ([D<sub>6</sub>]DMSO):  $\delta$  = 1.8–2.67 (m; 4H), 3.2 (t,  $J$  = 7 Hz; 2H), 7.33–7.77 (m; 2H), 7.9–8.3 (m; 2H), 12.0 (br.; 1H, D<sub>2</sub>O-Austausch).

C<sub>11</sub>H<sub>11</sub>NO<sub>2</sub>S (221.3) Ber. C 59.71 H 5.01 N 6.33 S 14.49  
Gef. C 59.54 H 5.09 N 6.26 S 14.37 Molmasse 221 (MS)

**5-(2-Benzothiazolyl)pentansäure (14b):** Nach Vorschrift C: 0.50 g (2.0 mmol) **12b** in 10 ml Wasser; 1.39 g (8.00 mmol) Natriumdithionit in 25 ml Wasser; Reaktionszeit 30 min. Farblose Kristalle, Schmp. 130–132 °C (Essigester) (Lit.<sup>3</sup>) Schmp. 134 °C, Ausb. 0.28 g (59%). – IR (KBr): 3200–2300, 1710 cm<sup>-1</sup>. – <sup>1</sup>H-NMR (CDCl<sub>3</sub>):  $\delta$  = 1.43–2.07 (m; 4H), 2.32 (t,  $J$  = 6.5 Hz; 2H), 3.15 (t,  $J$  = 6.5 Hz; 2H), 7.21–7.77 (m; 2H), 7.77–8.18 (m; 2H), 12.1 (br.; 1H, D<sub>2</sub>O-Austausch).

C<sub>12</sub>H<sub>13</sub>NO<sub>2</sub>S (235.3) Ber. C 61.25 H 5.57 N 5.95 S 13.63  
Gef. C 60.77 H 5.58 N 5.99 S 13.71 Molmasse 235 (MS)

**6-(2-Benzothiazolyl)hexansäure (14c):** Nach Vorschrift C: 0.81 g (3.0 mmol) **12c** in 15 ml Wasser; 4.27 g (24.0 mmol) Natriumdithionit in 20 ml Wasser; Reaktionszeit 1.5 h. Farblose Kristalle,

Schmp. 72–73 °C (Benzol/Cyclohexan) (Lit.<sup>3</sup>) Schmp. 70–72 °C, Ausb. 0.37 g (50%). – IR (KBr): 3300–2300, 1710  $\text{cm}^{-1}$ . – <sup>1</sup>H-NMR ( $\text{CDCl}_3$ ):  $\delta$  = 1.25–2.23 (m; 6H), 2.23–2.62 (m; 2H), 3.17 (t,  $J$  = 7.5 Hz; 2H), 7.22–7.72 (m; 2H), 7.72–8.20 (m; 2H), 10.03 (1H,  $\text{D}_2\text{O}$ -Austausch).

$\text{C}_{13}\text{H}_{15}\text{NO}_2\text{S}$  (249.3) Ber. C 62.62 H 6.06 N 5.62 S 12.86

Gef. C 62.60 H 6.11 N 5.68 S 13.01 Molmasse 249 (MS)

*2-Methylbenzothiazol* (**19**): 1.4 g (7.0 mmol) **17** in 20 ml Ethanol wurden unter Rühren bei 70–80 °C tropfenweise mit 9.97 g (56.0 mmol) Natriumdithionit in 50 ml Wasser versetzt. Nach 1.5 h ließ man abkühlen und versetzte mit Cyclohexan. Die organische Phase wurde abgetrennt und die wäßrige Lösung mehrfach mit Cyclohexan extrahiert. Die vereinigten Extrakte wurden mit  $\text{Na}_2\text{SO}_4$  getrocknet und destilliert. Farblose Flüssigkeit, Sdp. 72 °C/1.8 Torr (Lit.<sup>7</sup>) Sdp. 238 °C/760 Torr, Ausb. 0.71 g (68%).

*5-(2-Benzothiazolyl)-4-methyl-4-azapentansäure-3'-oxid* (**26**) und *5-(2-Benzothiazolyl)-4-methyl-4-azapentansäure* (**25**): 650 ml 0.01 N  $\text{Ba}(\text{OH})_2$  wurden unter Stickstoff und starkem Rühren tropfenweise mit 1.35 g (5.00 mmol) **22** in 50 ml Tetrahydrofuran versetzt. Nach Zugabe von 65 ml 0.1 N  $\text{H}_2\text{SO}_4$  wurde gekühlt und das entstandene Bariumsulfat abfiltriert. Das Filtrat wurde mit Dichlormethan geschüttelt (= Ausgangslösung zur Darstellung von **25**). Zur Messung des Spektrums wurde 1/5 dieser Lösung möglichst rasch bei Raumtemp. i. Vak. eingeengt und der Rückstand mit wenig Aceton versetzt. Aus der bei –5 °C aufbewahrten Lösung schied sich **26** in farblosen Kristallen (als Hydrat) ab. – <sup>1</sup>H-NMR ( $\text{CD}_3\text{OD}$ ):  $\delta$  = 2.27–2.73 (m; 5H), 2.73–3.17 (m; 2H), 4.13 (s, br.; 2H), 7.43–7.77 (m; 2H), 7.77–8.12 (m; 2H).

4/5 der Ausgangslösung (4.0 mmol) von **26** wurden mit 4.27 g (24.0 mmol) Natriumdithionit in 40 ml Wasser versetzt und 2.5 h bei 70 °C gehalten. Nach dem Abkühlen wurde filtriert, das Filtrat mit Diethylether und dann mehrfach mit Dichlormethan geschüttelt. Die vereinigten Dichlormethan-Extrakte wurden getrocknet und eingeengt. Dann wurde mit Diisopropylether versetzt und gekühlt. Farblose Kristalle, Schmp. 25–30 °C, Ausb. 0.1 g (10%). – MS:  $m/e$  = 250 ( $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$ ). – <sup>1</sup>H-NMR ( $\text{CDCl}_3$ ):  $\delta$  = 2.53 (s; 3H), 2.57–2.80 (m; 2H), 2.88–3.17 (m; 2H), 4.17 (s; 2H), 7.30–7.77 (m; 2H), 7.87–8.23 (m; 2H). Dem <sup>1</sup>H-NMR-Spektrum in [ $\text{D}_6$ ]DMSO zufolge liegt **25** als Oligohydrat vor.

$\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2\text{S} \cdot 4.5 \text{H}_2\text{O}$  (331.4) Ber. C 43.49 H 6.99 N 8.45 S 9.67

Gef. C 43.85 H 6.59 N 8.55 S 9.81

*4-(2-Benzothiazolyl)butansäure-methylester-3'-oxid* (**13a**): 0.31 g (1.3 mmol) **12a** wurden bei Raumtemp. unter Stickstoff in 12 ml absol. Methanol suspendiert. Diese Suspension wurde bis zur vollständigen Auflösung tropfenweise mit etherischer Diazomethanlösung versetzt (Kontrolle der quantitativen Umsetzung durch DC). Dann wurde i. Vak. eingeengt, Dichlormethan zugegeben, mit Wasser geschüttelt, mit  $\text{Na}_2\text{SO}_4$  getrocknet und erneut vom Lösungsmittel befreit. Der Rückstand wurde auf eine Kieselgelsäule (Merck, Korngröße 0.063–0.200 mm;  $\varnothing$  2.7 cm, H = 8 cm) aufgetragen und mit 150 ml Essigester gespült. **13a** wurde dann mit Essigester/Ethanol (1:1) eluiert und aus Diisopropylether umkristallisiert. Gelbliche Kristalle, Schmp. 65–68.5 °C, Ausb. 0.1 g (31%). – IR (KBr): 1730  $\text{cm}^{-1}$ . – <sup>1</sup>H-NMR ( $\text{CDCl}_3$ ):  $\delta$  = 1.92–2.67 (m; 4H), 3.23 (t,  $J$  = 7 Hz; 2H), 3.67 (s; 3H), 7.27–7.87 (m; 3H), 7.97–8.23 (m; 1H).

$\text{C}_{12}\text{H}_{13}\text{NO}_3\text{S}$  (251.3) Ber. C 57.35 H 5.21 N 5.57 S 12.76

Gef. C 57.27 H 5.16 N 5.47 S 12.74 Molmasse 251 (MS)

*5-(2-Benzothiazolyl)pentansäure-methylester-3'-oxid* (**13b**): Wie bei **13a** wurden 0.75 g (3.0 mmol) **12b** in 25 ml Methanol mit etherischer Diazomethanlösung umgesetzt. Nach dem Abdampfen des Lösungsmittels wurde zweimal aus Essigester umkristallisiert. Farblose Kristalle, Schmp. 83–85 °C, Ausb. 0.34 g (43%). – IR (KBr): 1735  $\text{cm}^{-1}$ . – <sup>1</sup>H-NMR ( $\text{CDCl}_3$ ):  $\delta$  =

1.63–2.1 (m; 4H), 2.23–2.6 (m; 2H), 3.03–3.43 (m; 2H), 3.67 (s; 3H), 7.42–7.93 (m; 3H), 8.07–8.37 (m; 1H).

$C_{13}H_{15}NO_3S$  (265.3) Ber. C 58.85 H 5.70 N 5.28 S 12.08

Gef. C 58.67 H 5.70 N 5.36 S 12.09 Molmasse 265 (MS)

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