

## Studies Related to $\beta$ -Lactam Antibiotics. Part 7.<sup>1</sup> Facile Formation of Oxazolinoazetidinones from Benzothiazolyldithioazetidinones and Related Reactions

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4 $\beta$ -(Benzothiazol-2-ylthio)azetidin-2-one (2a), derived from penicillin G sulphoxide ester (1a), is converted competitively into the oxazolinoazetidinone (3a) and 4 $\alpha$ -(benzothiazol-2-ylthio)azetidinone (4a) upon treatment with triphenylphosphine at room temperature. Conversion of (3a) into (4a) is performed by heating with a large excess of 2-mercaptobenzothiazole in acetonitrile; reaction of (4a) with silver acetate regenerates (3a). The oxazolinoazetidinone (3b) having a 3-methylbut-3-enoic acid side-chain is easily obtained by reaction of the corresponding 4 $\beta$ -(benzothiazol-2-ylthio)azetidinone (2b) with triphenylphosphine. A one-pot synthesis of (3b) from penicillin G sulphoxide (1b) is achieved without isolation and purification of the intermediate dithioazetidinone (2b). 1-Dethia-1-oxa-5-*epi*-anhydropenicillin (9) is prepared by intramolecular thermal cyclisation of (3b) followed by base-catalysed isomerisation of the side-chain. Stereocontrolled substitution at position 5 of the oxazolinoazetidinone (3), leading to (4) and the azetidinone-lactone (8), is notable from the mechanistic point of view.

SINCE the discovery of highly modified penam and cepham skeletons possessing  $\beta$ -lactamase inhibitory activity as well as potent antibacterial activity, much attention has been paid to the synthesis of various fused  $\beta$ -lactam systems.<sup>2</sup> The oxazolinoazetidinone system derived from natural  $\beta$ -lactam can be considered to be one of the desirable intermediates in the synthetic approach to the fused  $\beta$ -lactams, and thus, the synthesis and chemistry of the oxazolinoazetidinones have been extensively investigated.<sup>3-7</sup> This paper describes a new facile synthesis of the oxazolinoazetidinones (3) by means of the reaction of benzothiazolyldithioazetidinones (2) with triphenylphosphine and some related reactions. The latter reactions involve the stereocontrolled inter- and intra-molecular substitution at position 5 of the oxazolinoazetidinones (3) leading to 4 $\alpha$ -(benzothiazol-2-ylthio)azetidinone (4) and the novel azetidinone-lactone (8), which can be converted into 1-dethia-1-oxa-5-*epi*-anhydropenicillin (9).

### RESULTS AND DISCUSSION

It is well documented that the reaction of unsymmetrical disulphides with phosphines or phosphites proceeds to give sulphides *via* an intermediate thiophosphonium thiolate involving the more acidic thiol as a counter anion.<sup>8</sup> Thus, the reaction of 4 $\beta$ -(benzothiazol-2-ylthio)azetidinones (2) with triphenylphosphine must produce an intermediacy of thiophosphonium benzothiazolethiolate (A) (Scheme 1), which subsequently may undergo substitution at the activated 4-position of the azetidinone by either intramolecular partition of the amide side-chain, or attack of the benzothiazolethiolate anion.<sup>†</sup>

When the dithioazetidinone (2a)<sup>9</sup> was treated with a slight excess of triphenylphosphine in acetonitrile at

<sup>†</sup> Barton *et al.* have reported that treatment of 4-isobutylthioazetidinone with trialkyl phosphite gives 4-alkylthioazetidinones in a Michaelis-Arbusov manner (D. H. R. Barton, P. G. Sammes, and M. V. Taylor, *Chem. Commun.*, 1971, 1137).

room temperature, a mixture of the oxazolinoazetidinone (3a) and the 4 $\alpha$ -(benzothiazol-2-ylthio)azetidinone (4a) ‡ (3.4 : 1, by n.m.r.) was obtained. Silica gel chromatography of the reaction mixture allowed isolation of (3a) and (4a) in 61 and 18% yields, respectively. The structure of (3a) was supported by spectral data and chemical conversion into the known isomeric oxazolinoazetidinone (5a)<sup>10</sup> on treatment with triethylamine. The n.m.r. spectrum of (4a) showed that C-4-H and C-3-H are *trans*- ( $J_{H-3, H-4}$  2 Hz). The isopropenyl  $\rightarrow$  isopropylidene isomerisation occurred to give the sulphide (6a) (see Scheme 1) upon treatment of (4a) with triethylamine.

Experiments at different temperatures (0–60 °C) indicated that the product distribution of (3a) and (4a) was almost temperature-independent. Interconversion between (3a) and (4a) required more drastic conditions: the sulphide (4a) was produced quantitatively when a solution of (3a) and a large excess of 2-mercaptobenzothiazole in acetonitrile was refluxed. No formation of the 4 $\beta$ -epimer of (4a) occurred. The oxazolinoazetidinone (3a) was obtained in 70% yield upon treatment of (4a) with silver acetate in methanol at room temperature.

On the basis of above facts, it can be concluded that formation of (3a) and (4a) occurs competitively *via* an intermediate (A) produced by the reaction of (2a) with triphenylphosphine.

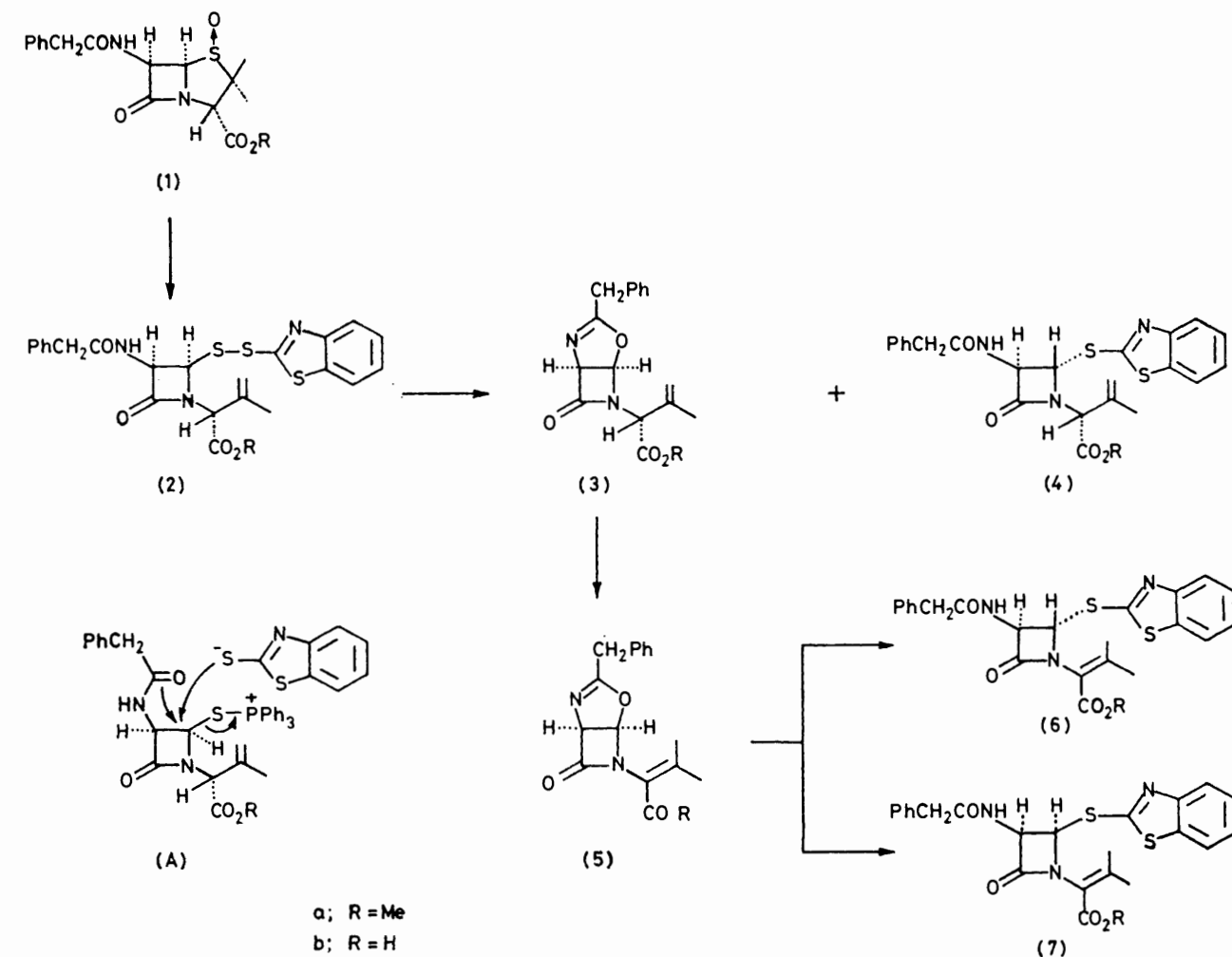
When the disulphide (2b), having a 3-methylbut-3-enoic acid side-chain, was treated with triphenylphosphine in acetonitrile at room temperature, the reaction mixture deposited the oxazolinoazetidinone (3b) as a crystalline mass in 60% yield. Thin-layer chromatographic analysis of the mother-liquor showed the presence of a small amount of (3b) and an undetermined product.

‡ Woodward and co-workers have reported that the reaction of 3-unsubstituted-4-benzothiazolyldithioazetidin-2-one with triphenylphosphine in acetic acid-acetic anhydride (with subsequent addition of pyridine) gives partially racemized 4-(benzothiazol-2-ylthio)azetidinone in 10% yield (I. Ernst, J. Gostali, and R. B. Woodward, *J. Am. Chem. Soc.*, 1979, **101**, 6301).

The oxazolinoazetidinone (3b) was also obtained in a one-pot reaction from the penicillin G sulphoxide (1b) without isolation of the disulphide (2b) (yield 35%). This one-pot procedure is convenient for the preparation of (3b) because purification of (2b) is fairly troublesome.

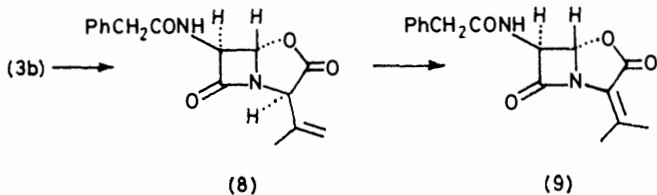
penicillin (9) <sup>4,11</sup> (Scheme 2). The present results provide a convenient preparative method for the 5-*epi*-oxa-anhydro-penicillin (9), as well as the oxazolinoazetidinone (3b).

Stoodley and Corbett <sup>12</sup> have reported that the oxazo-



Methylation of (3b) with diazomethane gave the corresponding ester (3a).

The oxazolinoazetidinone (3b) thus obtained cyclised easily to give the azetidinone-lactone (8) quantitatively upon heating in acetonitrile. The n.m.r. spectrum of the



lactone (8) showed that the protons in the  $\beta$ -lactam are *trans*-oriented, on the basis of the insignificant coupling constant between them. The isopropenyl  $\rightarrow$  isopropylidene isomerisation of (8), catalysed by base, resulted in the formation of the 1-dethia-1-oxa-5-*epi*-anhydro-

linoazetidinone (5a) with a 3-methylbut-2-enoate side-chain undergoes nucleophilic attack from various thiols at position 5 in the presence of toluene-*p*-sulphonic acid or a Lewis acid, leading to the formation of a mixture of 4 $\alpha$ - and 4 $\beta$ -azetidinone sulphides. Thus the reaction of (5a) with 2-mercaptobenzothiazole in the presence of boron trifluoride gave a mixture of the 4 $\alpha$ - and 4 $\beta$ -sulphides (6a) and (7a) (1:1 by n.m.r.), although no reaction occurred in the absence of the Lewis acid.

This observation is in a sharp contrast to the case of the oxazolinoazetidinone (3a); the reaction of (3a) with 2-mercaptobenzothiazole in the presence of boron trifluoride gave only the 4 $\alpha$ -sulphide (4a). As mentioned previously, the reaction also took place without any catalyst to produce (4a). The marked difference of the ease and stereospecificity of substitution at position 5 in the oxazolinoazetidinones (3a) and (5a), which have isomeric 3-methylbutenoate side-chains, is of interest

from the mechanistic point of view, as is the stereocontrolled formation of the azetidinone-lactone (8).

Studies on the synthetic application of the present observation to a fused azetidinone system are now in progress.\*

#### EXPERIMENTAL

Melting points were determined with a Yanagimoto micro-hot-stage apparatus and are uncorrected. I.r. spectra were recorded with a Hitachi 215 spectrometer for neat films.  $^1\text{H}$  N.m.r. spectra were obtained on a Hitachi R-24 B (60 MHz) spectrometer, using deuteriochloroform or hexadeuteriodimethyl sulphoxide as solvent and tetramethylsilane as internal standard. Mass spectra were measured at 75 eV with a JEOL JMS-01SG spectrometer. Column chromatography was performed on silica gel (Wako gel C-300) using diethyl ether or benzene-ethyl acetate as eluant.

**Reaction of Methyl (2R)-2-[(3S,4R)-4-(Benzothiazol-2-ylthio)-2-oxo-3-phenylacetamidoazetidin-1-yl]-3-methylbut-3-enoate (2a) with Triphenylphosphine.**—A mixture of the benzothiazolyldithioazetidinone (2a) (500 mg) and triphenylphosphine (560 mg) in acetonitrile (30 ml) was stirred at room temperature for 30 min. After removal of the solvent under reduced pressure, the residual oil was chromatographed (diethyl ether) to give methyl (2R)-2-[(1S,5R)-3-benzyl-7-oxo-4-oxa-2,6-diazabicyclo[3.2.0]hept-2-en-6-yl]-3-methylbut-3-enoate (3a) (186.7 mg) and methyl (2R)-2-[(3S,4S)-4-(benzothiazol-2-ylthio)-2-oxo-3-phenylacetamidoazetidin-1-yl]-3-methylbut-3-enoate (4a) (84.4 mg), respectively. Compound (3a): m.p. 58–60 °C (Found: C, 64.7; H, 5.75; N, 8.85.  $\text{C}_{17}\text{H}_{19}\text{N}_2\text{O}_4$  requires C, 64.95; H, 5.77; N, 8.91%),  $m/e$  314 ( $M^+$ );  $\nu_{\text{max}}$  1780 ( $\beta$ -lactam), 1740 ( $\text{CO}_2\text{Me}$ ), and 1690  $\text{cm}^{-1}$  (CONH);  $\delta(\text{CDCl}_3)$  1.53 (3 H, s, C-Me), 3.67 (5 H, s,  $\text{CO}_2\text{Me}$  and  $\text{PhCH}_2$ ), 4.82 (1 H, s,  $\text{CHCO}_2\text{Me}$ ), 4.90 (2 H, m,  $\text{C}=\text{CH}_2$ ), 5.18 (1 H, d,  $J$  2.6 Hz, lactam proton), and 6.12 (1 H, d,  $J$  2.6 Hz, lactam proton), and 7.23 (5 H, s, Ph);  $[\alpha]_{\text{D}}^{20}$  -75.1 ( $c = 1.0$ , MeOH). Compound (4a):  $m/e$  315 ( $M^+ - 167$ );  $\nu_{\text{max}}$  1780 ( $\beta$ -lactam), 1740 ( $\text{CO}_2\text{Me}$ ), and 1650  $\text{cm}^{-1}$  (CONH);  $\delta(\text{CDCl}_3)$  1.93 (3 H, s, C-Me), 3.60 (2 H, s,  $\text{PhCH}_2$ ), 3.74 (3 H, s,  $\text{CO}_2\text{Me}$ ), 4.82 (1 H, dd,  $J$  2 and 8 Hz, C-3-H), 4.88 (1 H, s,  $\text{CHCO}_2\text{Me}$ ), 4.90 and 5.07 (each 1 H, each br s,  $\text{C}=\text{CH}_2$ ), 5.67 (1 H, d,  $J$  2 Hz, C-4-H), 6.58 (1 H, d,  $J$  8 Hz, NH), 7.03–7.83 (4 H, m, aromatic protons), and 7.30 (5 H, s, Ph).

**Isomerisation of the Oxazolinoazetidinone (3a) to Methyl 2-[(1S,5R)-3-Benzyl-7-oxo-4-oxa-2,6-diazabicyclo[3.2.0]hept-2-en-6-yl]-3-methylbut-2-enoate (5a).**—A solution of the oxazolinoazetidinone (3a) (100 mg) and triethylamine (0.03 ml) in chloroform (10 ml) was stirred at room temperature for 30 min. The reaction mixture was washed with 0.5N-HCl, dried ( $\text{Na}_2\text{SO}_4$ ), and evaporated under reduced pressure. The residue was triturated with n-hexane to give the isomeric oxazolinoazetidinone (5a) in quantitative yield. The product (5a) was identical in every respect with an authentic sample.<sup>10</sup>

**Isomerisation of the Benzothiazolyldithioazetidinone (4a) to Methyl 2-[(3S,4S)-4-(Benzothiazol-2-ylthio)-2-oxo-3-phenylacetamidoazetidin-1-yl]-3-methylbut-2-enoate (6a).**—A solution of the benzothiazolyldithioazetidinone (4a) (100 mg) and triethylamine (0.03 ml) in chloroform (10 ml) was stirred

at room temperature for 30 min. The mixture was washed with 0.5N-HCl, dried ( $\text{Na}_2\text{SO}_4$ ), and evaporated under reduced pressure. The residue was purified by column chromatography (benzene-ethyl acetate) to give the isomeric benzothiazolyldithioazetidinone (6a) in quantitative yield,  $m/e$  315 ( $M^+ - 167$ );  $\nu_{\text{max}}$  (Nujol) 1775 ( $\beta$ -lactam), 1740 ( $\text{CO}_2\text{Me}$ ), and 1670  $\text{cm}^{-1}$  (CONH);  $\delta(\text{CDCl}_3)$  1.96 (3 H, s, C-Me), 2.13 (3 H, s, C-Me), 3.63 (2 H, s,  $\text{PhCH}_2$ ), 3.70 (3 H, s,  $\text{CO}_2\text{Me}$ ), 5.17 (1 H, dd,  $J$  2 and 8 Hz, C-3-H), 6.00 (1 H, d,  $J$  2 Hz, C-4-H), 6.78 (1 H, d,  $J$  8 Hz, NH), and 7.30 (5 H, s, Ph).

**Conversion of the Oxazolinoazetidinone (3a) into Benzothiazolyldithioazetidinone (4a).**—A mixture of the oxazolinoazetidinone (3a) (314 mg) and 2-mercaptobenzothiazole (3.34 g) in acetonitrile (20 ml) was refluxed for 2 h. After removal of the solvent under reduced pressure, the residue was purified by column chromatography [benzene-ethyl acetate (10 : 1)] to give the benzothiazolyldithioazetidinone (4a) (450 mg).

**Conversion of the Benzothiazolyldithioazetidinone (4a) into Oxazolinoazetidinone (3a).**—To a solution of the benzothiazolyldithioazetidinone (4a) (100 mg) in methanol (10 ml), silver acetate (37 mg) was added, and the mixture was stirred at room temperature for 30 min. After removal of the precipitate by filtration, the filtrate was evaporated under reduced pressure and purified by column chromatography (diethyl ether) to give the oxazolinoazetidinone (3a) (46 mg).

**(2R)-2-[(1S,5R)-3-Benzyl-7-oxo-4-oxa-2,6-diazabicyclo[3.2.0]hept-2-en-6-yl]-3-methylbut-3-enoic acid (3b).**—A solution of the benzothiazolyldithioazetidinone (2b) (500 mg) and triphenylphosphine (290 mg) in acetonitrile (30 ml) was stirred at room temperature for 30 min. The separated solid was collected and recrystallised from tetrahydrofuran to give the oxazolinoazetidinone (3b) (180 mg), m.p. 148–149 °C (Found: C, 63.7; H, 5.3; N, 9.2.  $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_4$  requires C, 63.99; H, 5.37; N, 9.33%),  $m/e$  300 ( $M^+$ );  $\nu_{\text{max}}$  (Nujol) 1780 ( $\beta$ -lactam), 1710 ( $\text{CO}_2\text{H}$ ), and 1640  $\text{cm}^{-1}$  (CONH);  $\delta([\text{C}_6\text{H}_6]\text{DMSO})$  1.52 (3 H, s, C-Me), 3.63 (2 H, s,  $\text{PhCH}_2$ ), 4.65 (1 H, s,  $\text{CHCO}_2\text{H}$ ), 4.90 (2 H, s,  $\text{C}=\text{CH}_2$ ), 5.17 (1 H, d,  $J$  2.6 Hz, lactam proton), 6.10 (1 H, d,  $J$  2.6 Hz, lactam proton), and 7.24 (5 H, s, Ph).

**One-pot Synthesis of the Oxazolinoazetidinone (3b) from Penicillin G Sulphoxide (1b).**—A mixture of penicillin G sulphoxide (1b) (20 g) and 2-mercaptobenzothiazole (14.3 g) in t-butyl alcohol (400 ml) was refluxed for 12 h. After removal of the solvent under reduced pressure, the residual oil was dissolved in acetone (200 ml) and treated with triphenylphosphine (9 g) at room temperature for 30 min. The resulting crystalline mass was collected and recrystallised from tetrahydrofuran to give the oxazolinoazetidinone (3b) (6.0 g).

**Esterification of the Oxazolinoazetidinone (3b).**—To a suspension of the oxazolinoazetidinone (3b) (100 mg) in ethyl acetate (20 ml), diazomethane-diethyl ether was added at room temperature until the starting material could no longer be detected by t.l.c. The mixture was stirred for a further 30 min. After removal of the solvent under reduced pressure, the residue was triturated with n-hexane to give the oxazolinoazetidinone (3a) (95 mg), which was identical in every respect with the compound obtained by the reaction of the benzothiazolyldithioazetidinone (2a) with triphenylphosphine.

**(2R,5S,6S)-2-Isopropenyl-3,7-dioxo-6-phenylacetamido-4-oxa-1-azabicyclo[3.2.0]heptane (8).**—A solution of the oxazo-

\* After completion of the present work, an example of the oxapenam synthesis by intramolecular cyclisation of 4-dithioazetidinoylhydroxycrotonates with triphenylphosphine was patented (Japanese P. 130981/1980).

linoazetidinone (3b) (526 mg) in acetonitrile (30 ml) was refluxed for 4 h. After removal of the solvent under reduced pressure, the residue was triturated with n-hexane and recrystallised from ethanol to give the *azetidinone-lactone* (8) (452 mg), m.p. 117–118 °C (Found: C, 63.95; H, 5.2; N, 9.15.  $C_{16}H_{16}N_2O_4$  requires C, 63.99; H, 5.37; N, 9.33%);  $\nu_{\max}$  (Nujol) 3 250 (NH), 1 790 ( $\beta$ -lactam), and 1 660  $cm^{-1}$  (CONH);  $m/e$  300 ( $M^+$ );  $\delta$ ( $[^2H_6]$ DMSO) 1.79 (3 H, s, C-Me), 3.51 (2 H, s,  $PhCH_2$ ), 4.83 (1 H, s,  $CHCO_2$ ), 4.93–5.20 (3 H, m,  $C=CH_2$  and C-6-H), 5.79 (1 H, br s, C-5-H), 7.25 (5 H, s, Ph), and 8.95 (1 H, d,  $J$  7.5 Hz, NH);  $[\alpha]_D^{20} - 139.5^\circ$  ( $c = 1.0$ , MeOH).

*Isomerisation of the Azetidinone-lactone (8) into (5S,6S)-2-Isopropylidene-3,7-dioxo-6-phenylacetamido-4-oxa-1-azabicyclo[3.2.0]heptane (9).*—A mixture of the azetidinone-lactone (8) (100 mg) and triethylamine (0.05 ml) in chloroform (20 ml) was stirred at room temperature for 30 min. The resulting crystalline mass was collected and recrystallised from chloroform to give the isomeric *azetidinone-lactone* (9) (95 mg), m.p. 151–152 °C (Found: C, 63.3; H, 5.25; N, 9.3.  $C_{16}H_{16}N_2O_4$  requires C, 63.99; H, 5.37; N, 9.33%);  $m/e$  300 ( $M^+$ );  $\nu_{\max}$  (Nujol) 3 225 (NH), 1 800 (lactone), 1 775 ( $\beta$ -lactam), and 1 650  $cm^{-1}$  (CONH);  $\delta$ ( $[^2H_6]$ DMSO) 2.06 (3 H, s, C-Me), 2.11 (3 H, s, C-Me), 3.50 (2 H, s,  $PhCH_2$ ), 5.01 (1 H, d,  $J$  8 Hz, C-6-H), 5.74 (1 H, br s, C-5-H), 7.26 (5 H, s, Ph), and 8.95 (1 H, d,  $J$  8 Hz, NH);  $[\alpha]_D^{20} - 65.0^\circ$  ( $c = 1.0$ , MeOH).

*Reaction of Oxazolinoazetidinone (5a) with 2-Mercaptobenzothiazole in the Presence of Boron Trifluoride.*—To a stirred solution of the oxazolinoazetidinone (5a) (157 mg) and 2-mercaptobenzothiazole (500 mg) in acetonitrile (5 ml), boron trifluoride-ether (3 drops) was added at 0 °C, and the mixture was stirred for a further 1 h. The resulting solution was diluted with chloroform (20 ml), washed with saturated aqueous sodium hydrogencarbonate, dried ( $Na_2SO_4$ ), and evaporated under reduced pressure. The residue was purified by column chromatography [benzene-ethyl acetate (10 : 1)] to give an inseparable mixture of the epimeric benzothiazolythioazetidinones (6a) and (7a) (24 mg, 1 : 1 by n.m.r.). The n.m.r. spectrum of the mixture showed signals at  $\delta$  1.57 (3 H, s, C-Me), 2.08 (3 H, s, C-Me), 5.83 (1 H, dd,  $J$  5 and 8 Hz, C-3-H), 5.91 (1 H, d,  $J$  5 Hz, C-4-H), and 6.62 (1 H, d,  $J$  8 Hz, NH), indicating the formation

of methyl 2-[(3S,4R)-4-(benzothiazol-2-ylthio)-2-oxo-3-phenylacetamidoazetidin-1-yl]-3-methylbut-2-enoate (7a), together with those of the 4 $\alpha$ -epimer (6a).

*Reaction of the Oxazolinoazetidinone (3a) with 2-Mercaptobenzothiazole in the Presence of Boron Trifluoride.*—To a solution of the oxazolinoazetidinone (3a) (157 mg) and 2-mercaptobenzothiazole (167 mg) in acetonitrile (4 ml), boron trifluoride-ether (3 drops) was added at 0 °C, and the mixture was stirred for a further 30 min. The resulting solution was diluted with chloroform (20 ml), washed with saturated aqueous sodium hydrogencarbonate, dried ( $Na_2SO_4$ ), and evaporated under reduced pressure. The residue was purified by column chromatography [benzene-ethyl acetate (5 : 1)] to give the 4 $\alpha$ -benzothiazolythioazetidinone (4a) (63.7 mg).

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