Stereoselective synthesis and stereochemistry of *r*-2-alkoxycarbonyl-*c*-3-*o*-substituted phenyl-1,4-thiazane 1,1-dioxides

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Abstract: *r*-2-Alkoxycarbonyl-*c*-3-aryl-1,4-thiazane 1,1-dioxides were obtained as the stereoselective product, when the aldehyde used was *o*-substituted benzaldehyde while the *p*-substituted benzaldehydes gave a trans product. The relative configuration of the adjacent alkoxycarbonyl and aryl groups was assigned from the vicinal coupling constant, ^3J = 10.6 Hz in the trans isomer and 3.2 Hz in the cis isomer, and from the multiplicity pattern, i.e., a doublet for the H-2 proton of the trans isomer and a triplet for the H-2 proton of the cis isomer. The unusual, large long-range coupling (^4J = 2.8 Hz) because of the "W" arrangement between H-2e and H-6e across the ring type was very useful for confirming the cis configuration and chair conformation of the isomer. The various ^{1}H and ^{13}C NMR assignments were made with the help of ^{1}H–^{1}H COSY, ^{1}H–^{13}C COSY, HMBC, and NOESY spectral analyses.

Key words: ^{1}H and ^{13}C NMR, ^{1}H–^{1}H COSY, ^{1}H–^{13}C COSY, HMBC, NOESY, stereoselectivity, 1,4-thiazane.

Résumé : Les 1,1-dioxydes de *r*-2-alkoxycarbonyl-*c*-3-aryl-1,4-thiazane sont obtenus comme produit stéréosélectif lorsque l'aldéhyde utilisé est un benzaldéhyde ortho substitué alors que les benzaldéhydes substitués en para conduisent au produit trans. Les configurations relatives des groupes alkoxycarbonyle et aryle adjacents ont été attribuées sur la base de la constante de couplage vicinale, ^3J = 10,6 Hz dans l'isomère trans et 3,2 Hz dans l'isomère cis, et du patron de multiplicité, c'est-à-dire la présence d'un doublet pour le proton H-2 de l'isomère trans et d'un triplet pour le proton H-2 de l'isomère cis. La constante de couplage à longue distance relativement importante (^4J = 2,8 Hz) causée par l'arrangement en « W » des protons H-2e et H-6e à travers ce type de cycle a été très utile pour confirmer la configuration cis et la conformation chaise de l'isomère. Les diverses attributions des spectres RMN du ^{1}H et du ^{13}C ont été faites sur la base d'analyses spectrales ^{1}H–^{1}H COSY, ^{1}H–^{13}C COSY, HMBC et NEOSY.

Mots clés : ^{1}H et ^{13}C RMN, ^{1}H–^{1}H COSY, ^{1}H–^{13}C COSY, HMBC, NEOSY, stéréosélectivité, 1,4-thiazane.

[Traduit par la Rédaction]

Introduction

Stereochemistry is an intrinsic universal feature of organic compounds and influences virtually all pharmacodynamic, pharmacokinetic, and biotransformation processes (1). Drug molecules having the proper absolute configuration only react with the receptor sites in the human body (2). Hence, the stereoselectivity (3) gains importance in organic synthesis. Thiazanes are widely used in new drug development (4, 5). Thiazane derivatives stimulate calcium uptake (6) used as antitumour, antiviral (7), bactericidal, parasiticidal (8), antituberculotic (9), and cardiovascular (10) agents, and they are also used as local anesthetics (11).

So far 3,5-disubstituted (12), 2,3,5-trisubstituted (13, 14), and 2,3,5,6-tetrasubstituted (15, 16) 1,4-thiazanes have been reported. Eliel and co-workers (17) reported the synthesis of *cis*-2,3-diphenyl-1,4-thiazane 1,1-dioxide from optically active starting material, and there too after several (more than 10 steps) tedious procedures. There has been no report on the synthesis of 2,3-disubstituted-1,4-thiazane 1,1-dioxides where the two substituents are different and in less stable cis configuration. Here, we report an one-pot synthesis of *cis*-2-alkoxycarbonyl-3-aryl-1,4-thiazane 1,1-dioxides, where the position of a substituent in the aryl ring controls the stereochemistry of the product.

For a 2,3-disubstituted 1,4-thiazane (in the rigid chair form), two configurational isomers, cis and trans, are possible. Generally, the thermal reaction will lead to the thermodynamically more stable trans isomer with the preferred diequatorial orientation of the substituents. The cis isomer with one axial substituent and one equatorial substitutent will be the less stable, kinetic-controlled product. In the present work, the trans isomer was obtained with benzaldehyde and *p*-substituted benzaldehydes, but the cis isomer was the product with *o*-substituted benzaldehydes.

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Results and discussion

Synthesis of 2-alkoxycarbonyl-3-aryl-1,4-thiazane 1,1-dioxides (Scheme 1) has been carried out by the condensation of dialkyl ethane – 1,2-disulphonylacetate (18) with 1 mol of araldehyde and ammonium acetate. Formation of a six-

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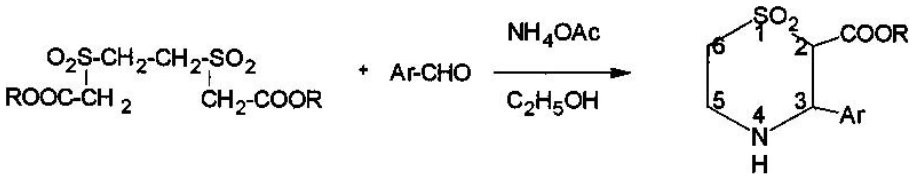
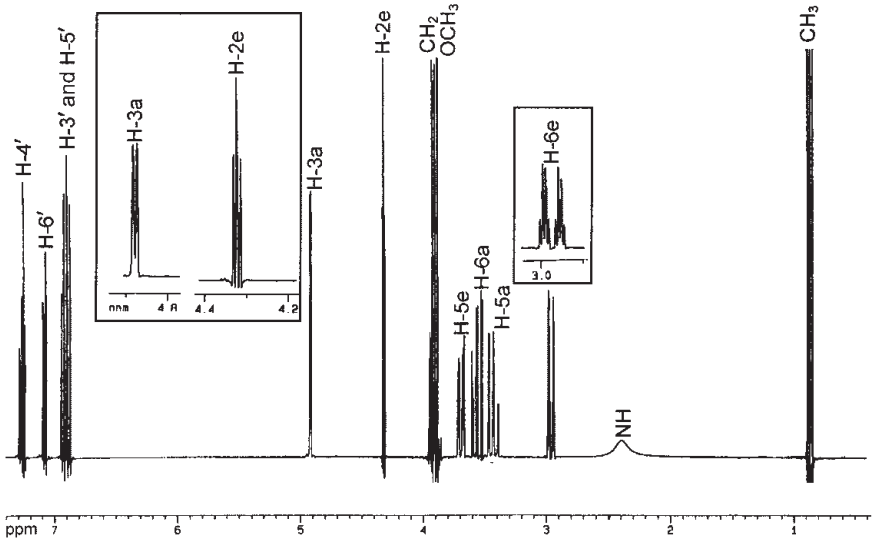
Scheme 1.



	R	Ar
1	C_2H_5	C_6H_5
2	C_2H_5	*p*-$CH_3C_6H_4$
3	CH_3	C_6H_5
4	C_2H_5	*p*-$OCH_3C_6H_4$
5	C_2H_5	*p*-ClC_6H_4
6	C_2H_5	*o*-$OCH_3C_6H_4$
7	C_2H_5	*o*-ClC_6H_4

Spectrum 1. ^{1}H NMR spectrum of **6**.



membered 1,4-thiazane ring was confirmed by microanalysis, IR, and mass spectra. Their stereochemistry was studied through NMR spectra.

The ^{1}H NMR chemical shift values with multiplicity patterns and coupling constants, along with their assignments are given in Table 1.

The diaxial coupling constant (10.6 Hz) between the H-2 and H-3 protons for the compounds (**1**–**3**) gives evidence for the trans (17) configuration with the preferred diequatorial orientation of the two substituents for the *p*-substituted phenyl compounds.

Stereochemistry of *o*-substituted phenyl compounds (6 and 7)

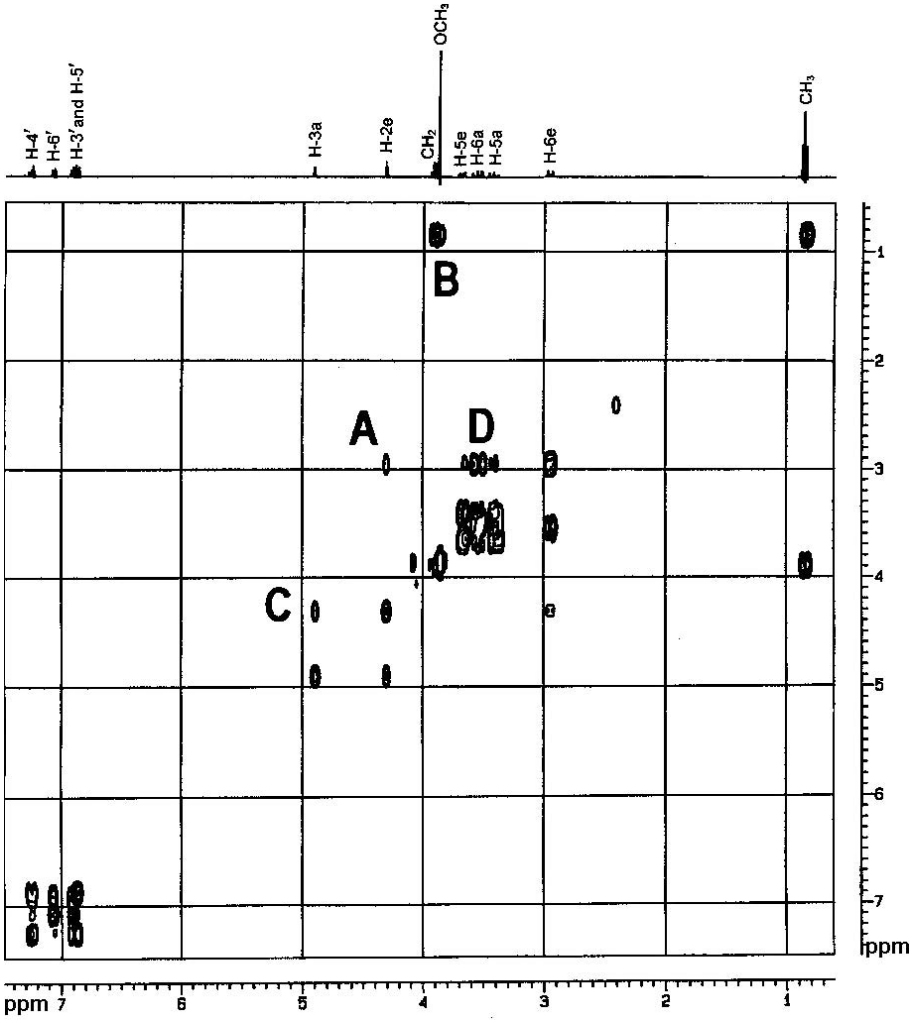
The H-2 and H-3 protons do not appear as two simple doublets with diaxial coupling constants. From the small coupling (17) constant (Table 1) between H-2 and H-3 protons, it is inferred that there is a change in configuration from *e*, *e* to *a*, *e*. For the cis configuration of the substituents with an *a*, *e* conformation, there are two possibilities, i.e., aryl group equatorial and ethoxycarbonyl group axial or aryl group axial and ethoxycarbonyl group equatorial. The benzylic proton signal appears as a doublet ($^3J_{a,e}$ = 3.2 Hz) so it has only one coupling partner. But the triplet (Spectrum 1) for the H-2 proton indicates that it has one more coupling partner, and that should be necessarily a long-range coupling partner (19). Long-range coupling is possible only with a "W" arrangement (20), and hence, the H-2 proton must be equatorial and ester group axial; consequently, the aryl group should be equatorial. In the ^{1}H–^{1}H COSY (21) spectrum (Spectrum 2), there is a cross peak (A) between the H-2e triplet and the doublet of the quartet signal at 2.96 ppm (i.e., H-6e), and this is an added evidence for the long-range coupling. The large cross peak (B) is due to the coupling be-

Table 1. ^{1}H NMR spectral data of 2-alkoxycarbonyl-3-aryl-1,4–thiazane 1,1-dioxides (**1**–**3**, **6**, **7**).[a]

Compound	NH	H-6a	H-6e	H-5a	H-5e	H-2[b]	H-3[c]
1	1.88 (s)	3.22 (m, 13.7, 10.3, 3.8)	3.15 (m, 13.7, 3.0)	3.47[d] (m)	3.47[d] (m)	4.06 (d, 10.6)	4.46 (d, 10.6)
2	2.05 (s)	3.12 (m)	3.12 (m)	3.40[d] (m)	3.40[d] (m)	4.04 (d, 10.6)	4.38 (d, 10.6)
3	2.05 (s)	3.22 (m)	3.22 (m)	3.48[d] (m)	3.48[d] (m)	4.09 (d, 10.6)	4.48 (d, 10.6)
6	2.39 (s)	3.56 (td, 13.4 12.5, 2.2)	2.96 (dq, 13.4, 2.4)	3.42 (td, 14.1 12.5, 2.2)	3.68 (m, 14.1, 2.2)	4.32 (t, 3.0)[e]	4.38 (d, 3.2)
7	2.32 (s)	3.58 (ddd,13.3, 12.1, 3.8)	2.99 (dq, 13.3 2.4)	3.44 (ddd, 14.1, 12.1, 2.4)	3.68 (m, 14.1, 3.8)	4.26 (t, 3.0)	4.98 (d, 3.2)

[a]NMR could not be recorded for compounds **4** and **5** because of their low solubility in $CDCl_3$ and DMSO-d_6.
[b]H-2 is axial for **1**–**3** and equatorial for **6** and **7**.
[c]H-3 is axial for **1**–**3**, **6**, and **7**.
[d]Overlapped signals correspond to 2H.
[e]$^4J_{e,e}$ for H-2 is calculated as the total width (6 Hz) – $^3J_{a,e}$ for H-3a (3.2 Hz) = 2.8 Hz.

Spectrum 2. ^{1}H–^{1}H COSY spectrum of **6**.



tween ester methylene and methyl protons. The cross peak (C) (between H-2e and H-3a) and a group of cross peaks (D) (between H-6e and H-5a, H-6a, H-5e) are obvious since they are coupling partners.

If the aryl group were to be axial, there would be steric interaction (from the Drieding model) between the *o*-substituent in the aryl group and the axial S=O bond of the sulphonyl group (Fig. 1).

^{13}C NMR assignments are given in Table 2. The carbon signals, especially for the aromatic and ipso carbons, are assigned only with the help of the HMBC (21) spectrum (Spectrum 3). For example, between the two high frequency

Spectrum 3. HMBC spectrum of **6**.

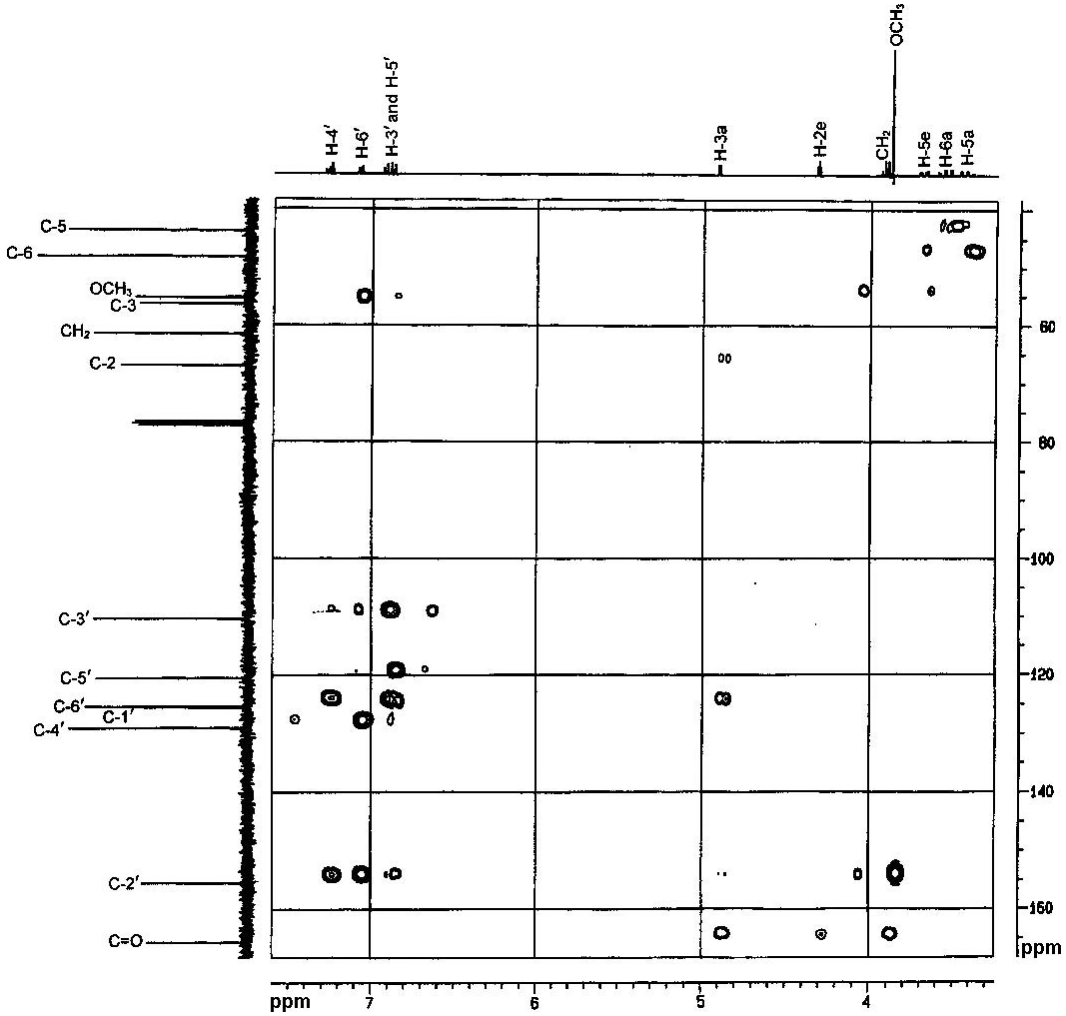


Fig. 1. Steric interaction between the *o*-OCH_3 group and the axial S=O group.

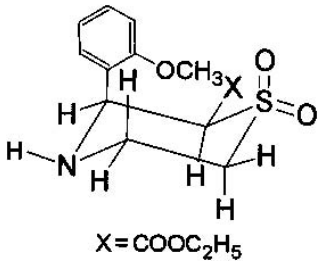


Table 2. ^{13}C NMR chemical shift values (ppm) of the ring carbons of 2-alkoxycarbonyl-3-aryl-1,4-thiazane 1,1-dioxides (**1**–**3**, **6**, **7**).[a]

Compound	C-2	C-3	C-5	C-6
1	72.65	62.78	43.71	52.89
2	72.58	62.42	43.62	52.77
3	72.44	62.67	43.58	52.73
6	67.04	56.39	43.90	48.32
7	66.72	58.56	43.92	48.35

[a]NMR could not be recorded for compounds **4** and **5** because of their low solubility in $CDCl_3$ and DMSO-d_6.

signals, 155.41 and 165.67 ppm, the former is assigned to the methoxy group bearing the ipso carbon (C-2′) since it gives multiple bond correlations with aromatic protons and methoxy methyl protons, and the latter is assigned to the carbonyl carbon since it gives multiple bond correlations with H-3a, H-2e, and ester methylene protons.

The orientation of the NH proton, whether axial or equatorial, is derived only from the NOE (21) spectrum (Spectrum 4). The NH proton gives a NOE with the H-6′ proton (A) and a strong NOE with the H-5a proton (B); the latter excludes the axial orientation of the NH proton and there is no NOE cross peak between the NH proton and the methoxy methyl protons. The various NOE cross peaks confirm the equatorial orientation of the NH proton and the perpendicular orientation of the *o*-methoxyphenyl ring to the plane of the thiazane ring (Fig. 2). The absence of NOE between the NH and ethoxycarbonyl methyl and methylene protons (which are relatively in the 1,3-diaxial position, if N-H is axial) excludes the possibility of an equilibrium between axial NH and equatorial NH.

Thus, the axial orientation of the ester group at the C-2 position, the equatorial and perpendicular orientation of the

Spectrum 4. NOESY spectrum of **6**.

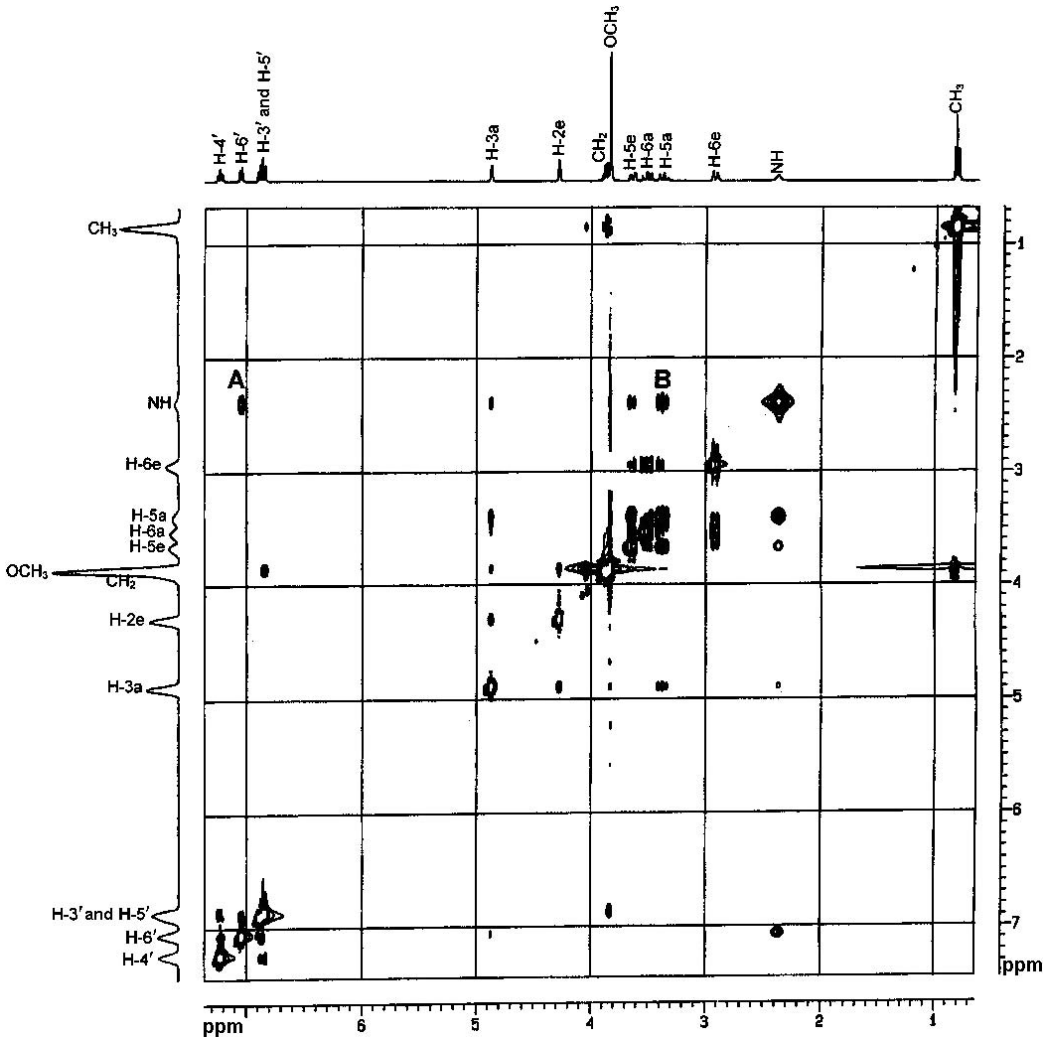


Fig. 2. Relative configuration and conformation of compound **6**.

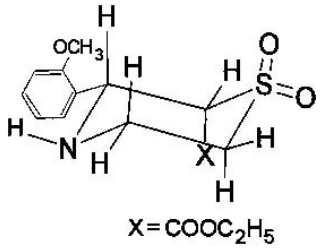
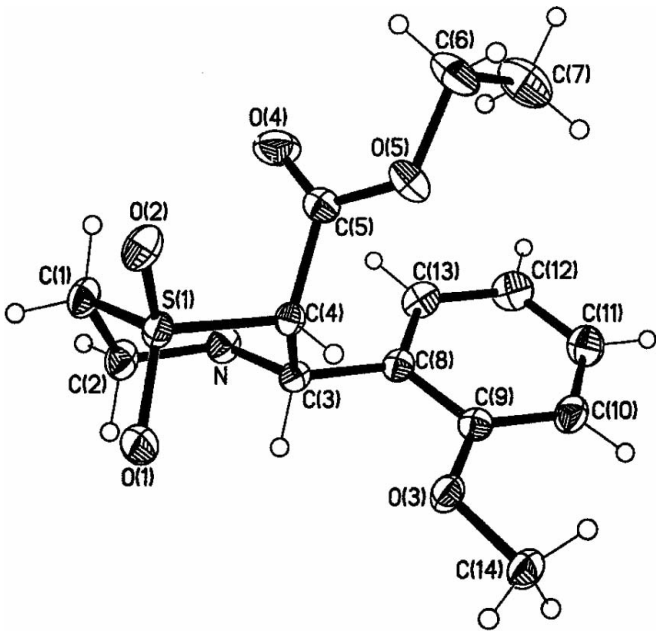


Fig. 3. ORTEP diagram of **6**.



o-substituted phenyl group at the C-3 position, and the chair conformation of the thiazane ring are derived from NMR spectra and confirmed by single crystal X-ray diffraction study. The ORTEP diagram is given in Fig. 3.

Conclusion

o-Substituted benzaldehydes give *r*-2a-alkoxycarbonyl-*c*-3-*o*-substituted phenyl-1,4-thiazane 1,1-dioxides when treated with dialkyl ethane – 1,2–disulphonylacetate and ammonium acetate as the stereoselective cis product. On the other hand, benzaldehyde and *p*-substituted benzaldehydes

give stereoselectively the trans product. The cis configuration with the axial ethoxycarbonyl group and equatorial aryl group is a consequence of the steric hindrance between the *o*-substituent in the phenyl ring and the ethoxycarbonyl group if it were in an equatorial orientation.

Experimental

Melting points are uncorrected. Microanalytical data were obtained from IISc, Bangalore-12, India. IR spectra were recorded on a JASCO-IR-700 IR spectrophotometer as potassium bromide discs of the sample. Mass spectra were obtained from RSIC, IIT, Chennai, India.

NMR spectra were recorded at 27 °C on a Bruker AMX 400 instrument operating at 400 MHz at a concentration of 20 mg/mL in $CDCl_3$ for ^1H NMR and 100 MHz at a concentration of 40 mg/mL in $CDCl_3$ for ^{13}C NMR, and chemical shifts were referenced internally to TMS in all cases.

The experimental parameters for the ^1H–^1H COSY spectra were: number of scans 16, number of data points 1 K, relaxation delay 1 s, acquisition time 0.1348 s, and spectral width 3817 Hz. The ^1H–^{13}C COSY spectra were obtained using the following parameters: number of scans 64, number of data points 2 K, acquisition time 0.666 s, relaxation delay 1 s, and spectral width 9981 Hz in F_1 and 15 576 Hz in F_2.

Crystallogrpahic data (22)

Compound **6** forms monoclinic crystals, a = 25.706 Å (5), b = 9.7180 Å (19), c = 12.245 Å (2), β = 93.44(3)°, Z = 8, space group $C2/c$. The structure was solved from 3132 observed reflections (Mo Kα radiation), and refined to R = 0.093 and R_w = 0.100.

Compounds

r-2e-Ethoxycarbonyl-*t*-3-phenyl-1,4-thiazane 1,1-dioxide (1)

A mixture of diethyl ethane – 1,2-disulphonylacetate (0.05 mol) (18), benzaldehyde (0.05 mol), and ammonium acetate (0.06 mol) in ethanol (100 mL) was heated under reflux for 6 h. The excess of solvent was removed by distillation and the mixture was kept overnight. The separated solid was filtered, washed with water, dried and recrystallized from ethanol. Yield: 55%; mp 164 to 165 °C. IR (cm^{-1}): 3400 (NH), 1722 (C=O), 1292 (asy. SO_2), 1126 (sym. SO_2). ^1H NMR (ppm): 1.00 (3H, t, CH_3), 1.88 (1H, br.s, NH), 3.19 (2H, m, H-6a, H-6e), 3.47 (2H, m, H-5a, H-5e), 4.01 (2H, q, CH_2), 4.06 (1H, d, $^3J_{a,a}$ = 10.6 Hz, H-2a), 4.46 (1H, d, $^3J_{a,a}$ = 10.6 Hz, H-3a), 7.32–7.38 (5H, m, aromatic). ^{13}C NMR (ppm): 13.69 (CH_3), 43.71 (C-5), 52.89 (C-6), 62.20 (CH_2), 62.78 (C-3), 72.65 (C-2), 127.71–128.92 (aromatic carbon), and 138.05 (ipso carbon). MS (*m*/*z*): 283, 238, 211, 196, 191, 172, 146, 133, 131, 130, 119, 118, 105, 104. Anal. calcd. for $C_{13}H_{17}NO_4S$: C 55.12, H 6.01, N 4.94; found: C 55.45, H 6.10, N 4.81.

r-2e-Ethoxycarbonyl-*t*-3-*p*-tolyl-l,4-thiazane 1,1-dioxide (2)

r-2e-Ethoxycarbonyl-*t*-3-*p*-tolyl-l,4-thiazane 1,1-dioxide (**2**) was obtained following the procedure of compound **1**. Yield: 50%; mp 145 to 146 °C. IR (cm^{-1}): 3420 (NH), 1724 (C=0), 1290 (asy. SO_2), 1125 (sym. SO_2). ^1H NMR (ppm): 0.99 (3H, t, CH_3), 2.05 (1H, br.s, NH), 2.29 (3H, s, tolyl), 3.12 (2H, m, H-6a, H-6e), 3.40 (2H, m, H-5a, H-5e), 3.99 (2H, q, CH_2), 4.04 (1H, d, $^3J_{a,a}$ = 10.6 Hz, H-2a), 4.38 (1H, d, $^3J_{a,a}$ = 10.6 Hz, H-3a), 7.10 (2H, d, aromatic), 7.23 (2H, d, aromatic). ^{13}C NMR (ppm): 13.66 (CH_3), 21.03 (tolyl), 43.62 (C-5), 52.77 (C-6), 62.08 (CH_2), 62.42 (C-3), 72.58 (C-2), 125.49–138.62 (aromatic), 161.71 (C=O). Anal. calcd. for $C_{14}H_{19}NO_4S$: C 56.56, H 6.40, N 4.71; found: C 56.43, H 6.46, N 4.63.

r-2e-Methoxycarbonyl-*t*-3-phenyl-1,4-thiazane 1,1-dioxide (3)

r-2e-Methoxycarbonyl-*t*-3-phenyl-1,4-thiazane 1,1-dioxide (**3**) was obtained following the procedure of compound **1**. Yield: 45%; mp 144–146 °C. IR (cm^{-1}), 3425 (NH), 1715 (C=0), 1295 (asy. SO_2), 1130 (sym. SO_2). ^1H NMR (ppm): 2.05 (1H, s, NH), 3.22 (2H, m, H-6a, H-6e), 3.48 (2H, m, H-5a, H-5e), 3.59 (3H, s, CH_3), 4.09 (1H, d, $^3J_{a,a}$ = 10.6 Hz, H-2a), 4.48 (1H, d, $^3J_{a,a}$ = 10.6 Hz, H-3a), 7.31 (5H, m, aromatic). ^{13}C NMR (ppm): 43.58 (C-5), 52.73 (C-6), 52.92 (CH_3), 62.67 (C-3), 72.44 (C-2), 127.48–128.83 (aromatic), 138.07 (ipso), 162.28 (C=O). Anal. calcd. for $C_{12}H_{15}NO_4S$: C 53.53, H 5.58, N 5.20; found: C 53.38, H 5.62, N 5.14.

r-2e-Ethoxycarbonyl-*t*-3-(*p*-methoxyphenyl)-1,4–thiazane 1,1-dioxide (4)

Diethylethane-1,2-disulphonyl acetate (0.05 mol), *p*-methoxybenzaldehyde (0.05 mol), and ammonium acetate (0.06 mol) in ethanol (100 mL) was refluxed for 15 h. The excess solvent was removed by distillation and the residue was poured into water. It solidified after 15 days. The solid was filtered, washed with water, then with ether, dried, and recrystallized from an ethanol–acetone mixture. Yield: 25%; mp 165–167 °C. IR (cm^{-1}): 3450 (NH), 1735 (C=O), 1290 (asy. SO_2), 1115 (sym. SO_2). Anal. calcd. for $C_{14}H_{19}NO_5S$: C 53.67, H 6.07, N 4.47; found: C 53.60, H 6.03, N 4.38. Very low solubility in $CDCl_3$.

r-2e-Ethoxycarbonyl-*t*-3-(*p*-chlorophenyl)-1,4-thiazane 1,1-dioxide (5)

r-2e-Ethoxycarbonyl-*t*-3-(*p*-chlorophenyl)-1,4-thiazane 1,1-dioxide (**5**) was obtained following the procedure of compound **4**. Yield: 30%; mp 178–180 °C. IR (cm^{-1}): 3420 (NH), 1687 (C=O), 1290 (asy. SO_2), 1120 (sym. SO_2). Anal. calcd. for $C_{13}H_{16}NO_4SCl$: C 49.21, H 5.05, N 4.42; found: C 49.24, H 5.02, N 4.38. Very low solubility in $CDCl_3$.

r-2a-Ethoxycarbonyl-*c*-3-(*o*-methoxyphenyl)-1,4-thiazane 1,1-dioxide (6)

r-2a-Ethoxycarbonyl-*c*-3-(*o*-methoxyphenyl)-1,4-thiazane 1,1-dioxide (**6**) was obtained following the procedure of compound **4**; reaction time 20 h. Yield: 20%; mp 143–145 °C. IR (cm^{-1}): 3475 (NH), 1740 (C=O), 1295 (asy. SO_2), 1114 (sym. SO_2). ^1H NMR (ppm): 0.86 (3H, s, CH_3), 2.39 (1H, br.s, NH), 2.96 (1H, dt, J = 13.4, 2.2 Hz, H-6e), 3.42 (1H, td, J = 14.1, 12.5, 2.2 Hz, H-5a), 3.56 (1H, td, J = 13.4, 12.5, 2.2 Hz, H-6a), 3.68 (1H, m, J = 14.1, 2.2 Hz, H-5e), 3.87 (3H, s, methoxy), 3.90 (2H, Dq, CH_2), 4.32 (1H, t, J = 3.2, 2.8 Hz, H-2e), 4.91 (1H, d, J = 3.2 Hz, H-3a), 7.07 (4H, m, aromatic). ^{13}C NMR (ppm): 13.44 (CH_3), 43.90 (C-

5), 48.32 (C-6), 55.31 (methoxy), 56.39 (C-3), 61.57 (CH_2), 67.04 (C-2), 110.28 (C-3′), 120.51 (C-5′), 125.37 (C-6′), 125.57 (C-1′), 129.03 (C-4′), 155.41 (C-2′), 165.67 (C=O). Anal. calcd. for $C_{14}H_{19}NO_5S$: C 53.67, H 6.07, N 4.47; found: C 53.58, H 5.59, N 4.38.

r-2a-Ethoxycarbonyl-*c*-3-(*o*-chlorophenyl)-1,4-thiazane 1,1-dioxide (7)

r-2a-Ethoxycarbonyl-*c*-3-(*o*-chlorophenyl)-1,4-thiazane 1,1-dioxide (**7**) was obtained following the procedure of compound **4**; reaction time 20 h. Yield: 20%; mp 134–136 °C. IR (cm^{-1}): 3410 (NH) 1682 (C=0), 1303 (asy. SO_2), 1115 (sym. SO_2). Anal. calcd. for $C_{13}H_{16}NO_4SCl$: C 49.21, H 5.05, N 4.42; found: C 49.13, H 5.09, N 4.36.

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