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Syntheses of Optically Active 2-(2-Benzothiazolylimino)-heterazolidines

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ABSTRACT: *The syntheses of compounds 4R, 5R-2-[2-benzothiazolylimino]-3-hydro-4-methyl-5-phenylthiazolidine (***5a***), 4S,5S-2-[2-benzothiazolylimino]-3, 4-dimethyl-5-phenylthiazolidine (***5b***), 4R,5S-[2-benzothiazolylimino]-3-hydro-4-methyl-5-phenyloxazolidine (***5c***), 4R,5S-2-[2-benzothiazolyl-imino]-3, 4-dimethyl-5-phenyloxazolidine (***5d***), and 4R,5S-2-[2-benzothiazolylimino]-1,3,4-trimethyl-5-phenylimidazolidine (***5e***) are reported. The stereochemistry of the reaction products and the X-ray diffraction analyses of compounds* **5a–d** *are discussed. Compounds* **5a–d** *present planar structures. We have found short distances between the thiazolidine S1 atom and the benzothiazole N9 atom for* **5a** *and* **5b** *(2.782 and 2.824 Å, respectively) and the distance between the oxazolidine O1 and the benzothiazole S7 for* **5d** *(2.721 Å). These distances are shorter than the sum of the van der Waals radii. These distances, together with the coplanarity of the heterocycles, seem to indicate the existence of a weak coordination bonding from sp^2 oxygen or nitrogen atoms towards the sulfur atom.* © 2001 John Wiley & Sons, Inc. Heteroatom Chem 12:586–593, 2001

INTRODUCTION

We have been investigating the preparation and structure of heterocycles rich in lone pairs that may be used as ligands for main group elements [1–5]. In particular, we are interested in optically active ligands that could afford molecules with a heteroelement being a stereogenic center. Therefore, we studied the preparation of optically active heterazolidines derived from 2-aminobenzothiazole (**1**). Merchan et al. found that the treatment of 2-aminobenzothiazole with CS_2 in an aqueous solution of NaOH, followed by addition of methyl iodide, afforded the dimethylated compound **2** [6–8]. This is an interesting precursor for the preparation of 1,3-heterocycles as is shown in Scheme 1, but to our knowledge, there are no optically active heterocycles that have been prepared in this way.

RESULTS AND DISCUSSION

Herein, we present the syntheses of five new optically active heterazolidines (**5a–e**) derived from 2-aminobenzothiazole (Scheme 2). The use of the disodium salt **4** [9] allowed us to obtain compounds **5a,b** by reaction with chloro-deoxy-*norpseudo* ephedrine or chloro-deoxy-*pseudo*ephedrine in refluxing DMF, while compound **2** was reacted with *nore*phedrine, ephedrine, or *N,N*-dimethyl-2-phenyl-3-methyl-1,3-diaminoethane to give respectively compounds **5c–e**. The structures of compounds **5a–e** were determined by the usual methods, NMR data presented in Tables 1 and 2. Compounds **5a–d** are crystalline solids and their structures in the solid state were determined by X-ray diffraction analyses; **5e** is a liquid.

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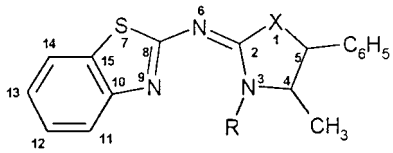
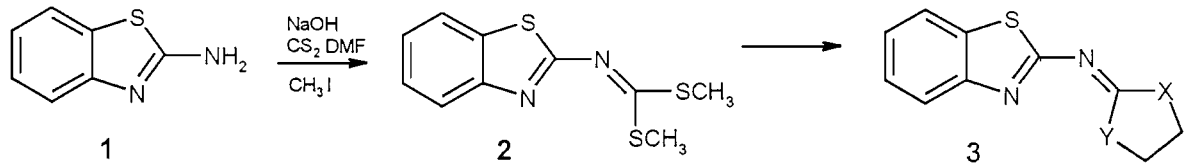
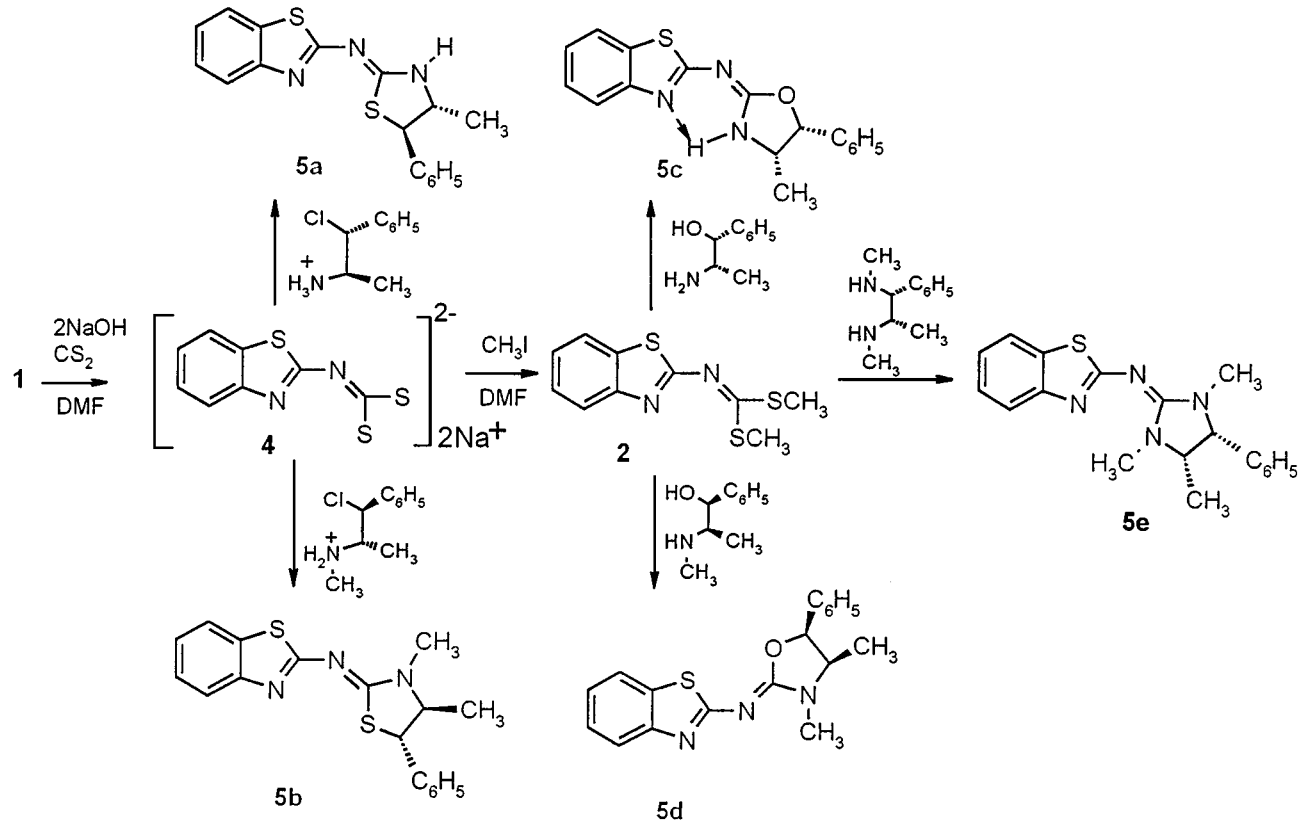
TABLE 1 ^{1}H NMR δ (ppm), J [Hz] of Compounds **5a–e** ($CDCl_3$)



	H5,d	*H4,dq*	*C—CH$_3$, d*	*N—R*	*H11*	*H12*	*H13*	*H14*	*C$_6$H$_5$*
5a	4.52(8.4)	4.30	1.44(6.2)	10.21	7.73	7.32	7.22	7.70	7.30
5b	4.26(6.6)	3.48	1.36(6.6)	3.15	7.92	7.28	7.22	7.67	7.35
5c	5.79(8.0)	4.44	0.87(6.6)	9.60	7.71	7.34	7.20	7.68	7.34
5d	5.72(8.6)	4.00	0.75(6.6)	3.02	7.80	7.22	7.15	7.61	7.30
5e	4.66(9.1)	3.95	0.78(6.6)	2.74, 2.84	7.60	7.22	7.09	7.56	7.30

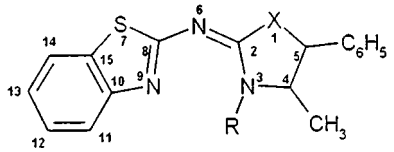


SCHEME 1



SCHEME 2 Syntheses of compounds **5a–e**.

TABLE 2 ^{13}C NMR δ (ppm), of **5a–e** ($CDCl_3$)

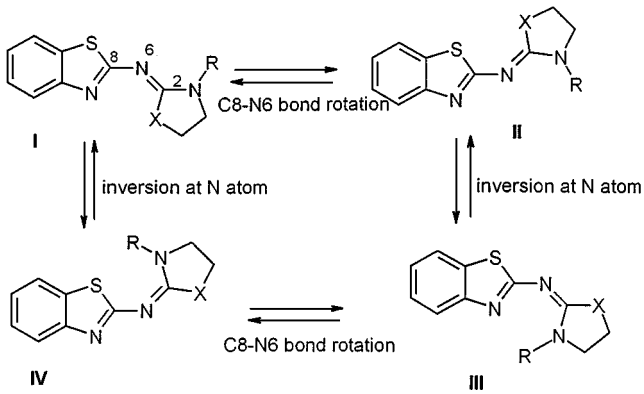


Comp	*C2*	*C4*	*C5*	*C—CH_3*	*N—CH_3*	*Ci*	*Co*	*Cm*	*Cp*	*C8*	*C10*	*C11*	*C12*	*C13*	*C14*	*C15*
5a	168.5	56.6	64.3	19.3		137.1	128.2	129.1	128.7	172.2	151.2	125.8	121.3	120.6	123.4	132.4
5b	163.8	54.5	66.2	17.3	32.5	138.7	127.8	129.0	128.3	171.3	151.7	125.5	121.0	120.9	122.8	133.7
5c	161.3	54.5	82.2	17.5		134.2	126.1	128.6	128.7	172.7	151.3	125.7	121.2	120.2	123.2	132.6
5d	156.9	58.0	82.6	13.9	29.9	134.1	126.0	128.6	128.8	166.4	150.8	125.3	121.0	120.7		128.7
5e	159.0	58.7	67.9	15.0	32.1, 33.5	135.5	128.3	128.8	128.5	170.5	152.7	125.3	120.7	119.5		134.0

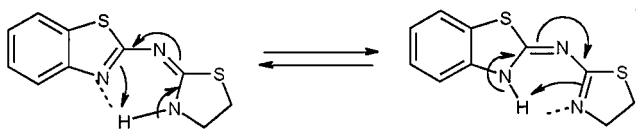
The syntheses of compounds **5a,b** were completely stereoselective. It could be assumed that for these molecules an aziridine is an intermediate, which in turn is attacked by the sulfide. These reactions have two inversion steps at C1 giving overall retention of the configuration. Similar behavior have been observed before in other substitution reactions at ephedrine derivatives [10]. On the other hand, the formation of compounds **5c,d** occurs by substitution of *S*-methyl groups by nitrogen and oxygen atoms of the ephedrines without modifying the stererochemistry and with the elimination of CH_3SH.

Compounds **5a–d** are planar molecules (vide infra); two configurations are expected for the double bond C2=N6, which can undergo exchange by isomerization at the exocyclic nitrogen atom as shown in Scheme 3 (this barrier amounts to about 13–25 kcal/mol [11]). Also, two conformers are possible by rotation of the bond C8—N6; for analogous systems, the energy barrier of this rotation is found between 10 and 18 kcal/mol [12–14]. Examination of all the possible conformers and isomers for each compound **5** suggests that conformers **I** and **II** could be favored for N—CH_3 compounds because **III** and **IV** could have a steric tension in their planar structures because of the interaction of the methyl group with the neighboring ring, whereas for N—H compounds (**5a** and **5c**) conformers **III** and **IV** could be the more stable by formation of a hydrogen bond.

In addition, for compounds **5a** and **5c** an exchange of the N—H group is expected between N3 and N9 (Scheme 4). In the ^{13}C NMR spectra of compounds **5a** and **5c,** the effect of this exchange in solution is shown through the strong broadening of the signals of C2 and C4. This phenomenon is not observed in compounds bearing the N—CH_3 group. The latter fact suggests that the preferred conformation for compounds **5a** and **5c** in solution is **III** (Scheme 3).



SCHEME 3 Conformers and isomers for [benzothiazolylimino]heterazolidines.



SCHEME 4 N—H exchange between nitrogen atoms N9 and N3.

Solid State Structures

X-ray diffraction structures of **5a–5d** are shown in Figs. 1–4 and selected data are provided in Tables 3 and 4. Compounds **5a–5d** are planar, the two five membered rings being connected by an sp^2 nitrogen atom (Figs. 1–4). The five sp^2 heteroatoms (N9=C8—N6=C2—N3) are in a planar delocalized π system. Examination of Tables 3 and 4 shows that, for all compounds, C8—N9 (1.29–1.31 Å) and C2—N6 (1.29–1.31 Å) have a double bond character (reported C=N bond length is 1.28 Å [15]), whereas bonds C8—N6 (1.34–1.36 Å) and C2—N3 (1.33–1.35 Å) have

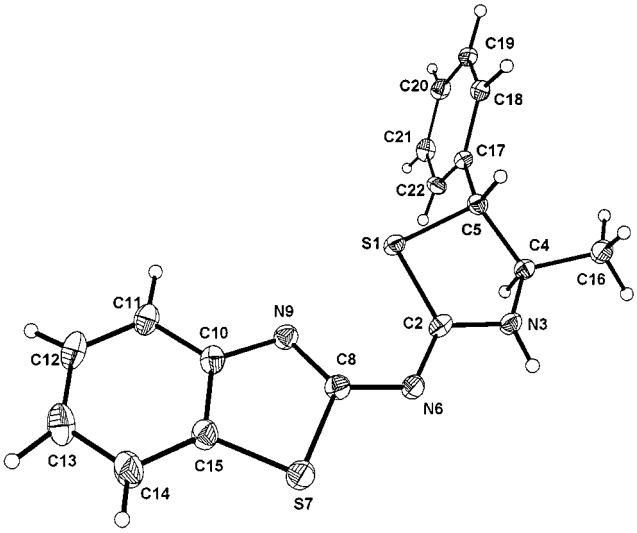


FIGURE 1 X-ray diffraction structure of compound **5a**.

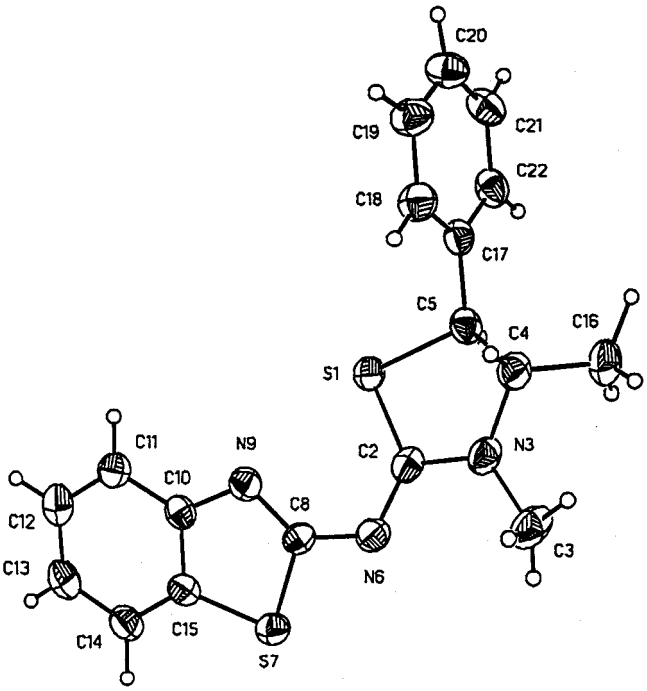


FIGURE 2 X-ray diffraction structure of compound **5b**.

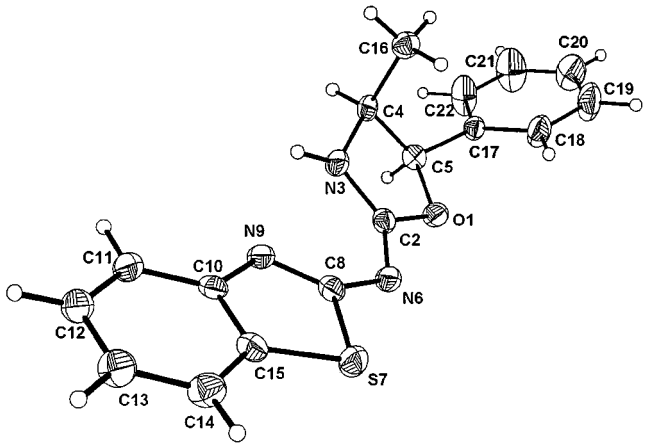


FIGURE 3 X-ray diffraction structure of compound **5c**.

single bond character (Csp^2—Nsp^2 1.35 Å). The conformation of compounds **5a–5d** makes them suitable ligands for metallic coordination.

Because of the possibility to have hydrogen bonds for compounds **5a** and **5c,** we were expecting that both compounds would present conformation **III** (Scheme 3) in the solid state, but they presented different structures. Compound **5a** has both C=N bonds in a S-*cis* configuration and N9 and S1 in a U conformation (Fig. 1). For compound **5a** (structure **I,** Scheme 3) to reach a conformation analogous to that of compound **5c** (structure **III**), a rotation around the C8—N6 bond is necessary, together with an inversion of the N6 configuration.

Compound **5a** has a similar configuration to that of compound **5b,** which indicates that probably both are kinetically controlled compounds. In both compounds, the ephedrinic fragment forms a ring without inversion of the configuration at C5; the thiazolidine rings have an envelope conformation with C(4) out of the ring plane (dihedral angles for **5a**: C5—S1—C2—N3, 8.7° and C2—S1—C5—C4, 27.0°; for **5b**: N3—C2—S1—C5, 10.6° and S1—C5—C4—N3, 34.3°). The phenyl and C—CH_3 groups are in *pseudo*equatorial positions and the bonds around N(3) are planar. In compound **5a**, there are intermolecular hydrogen bonds between N3—H proton and N6 (atoms distance 2.385 Å) forming a polymer. Compound **5b** also presents intermolecular short contacts between H21 and S1 (2.937 Å).

Compound **5c** has an intramolecular hydrogen bond with the nitrogen atom of the benzothiazole (N9···H3) 2.185 Å; both rings are not completely coplanar (torsional angles N3—C2—N6—C8 3.60°; N9—C8—N6—C2 18.56°) and both the nitrogen atoms, N3 and N9, are in a U conformation. In compound **5c**, C4 is out of the plane of the oxazolidine ring. The torsional angles of the oxazolidine ring are N3—C2—O1—C5 8.6°, C4—N3—C2—O1 12.8°, and C2—O1—C5—C4 25°. The C—CH_3 group occupies a pseudoaxial position. (Fig. 3).

It is interesting that in compound **5d**, the N9=C8—N6=C2 fragment has an *S-trans*

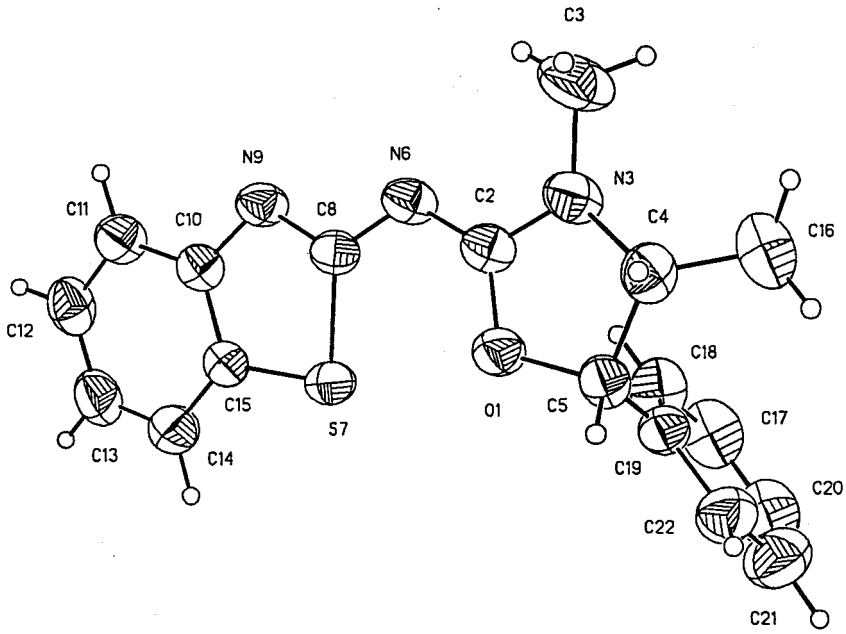


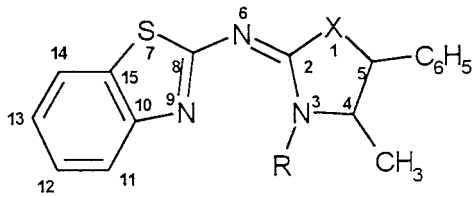
FIGURE 4 Solid state structure of compound **5d**.

configuration, the oxygen and sulfur being in U conformation, and both rings being coplanar (dihedral angles: C8—N6—C2—O1, 3.07°; S7—C8—N6—C2, 3.87°). The ephedrine ring is quite planar (torsional angle O1—C5—C4—N3, 15°). The phenyl group is *pseudo*axial (torsional angle C2—O1—C5—C17, 114.1°) whereas the methyl group is *pseudo* equatorial (Fig. 4). There are intermolecular hydrogen bonds between the C5—H proton and N6 (2.476 Å).

The liquid nature of compound **5e** could be attributed to a nonplanar structure because of the repulsion of the *N*-methyl groups by the benzothiazole ring.

One important result was the short distance found between the thiazolidine S1 atom and the benzothiazole N9 atom for **5a** and **5b** (2.782 and 2.824 Å, respectively) and also the short distance between the oxazolidine O1 and the benzothiazole S7 for **5d** (2.721 Å). These distances are shorter than the sum of the van der Waals radii (S—N 3.35 Å and S—O 3.30 Å) [15]. The distances, together with the coplanarity of the heterocycles, seem to indicate a weak coordination bonding from sp^2 oxygen or nitrogen atoms towards the sulfur atom. The angles of the sulfur rings are close to 90°; they correspond to a nonhybridized sulfur atom. Therefore, one lone electron pair of the sulfur is in a central 3s orbital and the second in a 3p orbital perpendicular to the ring plane. In consequence, the sulfur does not present a repulsion to the sp^2 lone pair orbitals

TABLE 3 Some Selected Bond Lengths of Compounds **5a–d** (Å)



	X1—C2	*C2—N3*	*N3—C4*	*C4—C5*	*C5—X1*	*C2—N6*	*N6—C8*	*C8—N9*	*N9—C10*	*C10—C15*	*C15—S7*	*S7—C8*
5a	1.762(2)	1.340(4)	1.467(4)	1.536(4)	1.841(4)	1.312(4)	1.361(4)	1.302(4)	1.381(4)	1.403(4)	1.737(4)	1.773(3)
5b	1.759(4)	1.350(5)	1.471(5)	1.535(6)	1.824(4)	1.296(5)	1.359(5)	1.296(5)	1.389(5)	1.411(5)	1.743(4)	1.767(4)
5c	1.342(5)	1.343(6)	1.462(6)	1.528(6)	1.477(5)	1.299(6)	1.361(5)	1.309(6)	1.381(5)	1.406(7)	1.725(5)	1.758(5)
5d	1.326(5)	1.335(5)	1.451(5)	1.546(6)	1.467(4)	1.298(5)	1.343(5)	1.296(4)	1.385(5)	1.375(5)	1.728(4)	1.771(4)

TABLE 4 Some Selected Bond Angles of Compounds **5a–d** (°)

	X1–C2–N3	*C2–N3–C4*	*N3–C4–C5*	*C4–C5-X1*	*C5–X1–C2*	*C2–N6–C8*	*C8–N9–C10*	*N9–C10–C15*	*C10–C15–S7*	*C15–S7–C8*	*S7–C8-N9*
5a	110.8(2)	116.7(2)	103.5(2)	104.2(2)	91.05(16)	119.3(2)	111.4(3)	115.8(3)	108.9(3)	89.3(2)	114.5(3)
5b	111.1(3)	116.4(3)	104.6(3)	104.9(3)	91.56(18)	119.0(3)	111.2(3)	115.8(3)	108.4(3)	89.5(2)	115.1(3)
5c	110.9(4)	110.4(4)	98.7(4)	103.0(3)	107.1(3)	118.3(4)	110.2(4)	116.0(4)	109.1(3)	89.3(2)	115.4(3)
5d	111.1(4)	111.6(3)	101.7(3)	102.7(3)	110.5(3)	122.7(3)	112.2(3)	115.1(4)	109.9(3)	89.3(2)	113.5(3)

of nitrogen or oxygen atom, which in turn can donate density to the 3d orbitals of the sulfur. Coordination to sulfur atom is depicted in the literature (ref. 17). A theoretical study is in progress to give evidence of this observation.

EXPERIMENTAL

The NMR spectra were obtained with JEOL GXS-270, JEOL-400, and Bruker-300 spectrometers in $CDCl_3$ solutions. Melting pints were measured on a Gallenkamp apparatus and are uncorrected. Elemental analyses were performed by Oneida Research Services, Whitesboro, New York. The MS spectra were obtained by direct insertion at 15 eV in an HP 5989 spectrometer. An IR spectrometer (Perkin Elmer 1600) was used for obtaining IR spectra in KBr pellets and in $CHCl_3$ solutions. X-ray diffraction analysis for compound **5a** was performed with a Siemens P4 instrument equipped with a CCD area detector and with a low temperature device LPT2. The unit cell was determined from data on 75 frames and data collection performed in the hemisphere mode with $\Delta\varphi = 0.3^\circ$ and a selected set of 10 s/frame exposures. Data reduction was implemented with use of the program SAINT. For compounds **5b–d,** we used a CAD4-Enraf-Nonius Diffractometer, radiation Mo Kα ($1 = 0.71079$ Å, with scan type $\omega/2\theta$. The SHELXL-97 software package was used for structures solution, refinement and data output. See Table 5 for crystal data and data collection. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre: Compound **5a** CCDC 161786, Compound **5b** CCDC 161789, Compound **5c** CCDC 161787, and Compound **5d** CCDC 161788.

The 1*S*,2*R*-ephedrine and 1*R*,2*S*-*nor*ephedrine were commercial products. Disodium *N*-(2-benzothiazolyl)dithiocarbonimidato [8] (**4**), dimethyl *N*-(2-benzothiazolyl)dithiocarbonimidato [8] (**2**), 1*S*, 2*S*-chloro-deoxy-ephedrine [18], 1*R*,2*R*-chloro-deoxy-*nor*ephedrine [18], and (1*R*,2*S*)-*N*,*N*′-dimethyl-2-phenyl-3-methyl-1,3-diaminoethane [10] were prepared according to the literature.

*2-[2-Benzothiazolylimino]thiazolidines (***5a,b***). General Procedure*

To a solution of 1 g (3.7 mmol) of disodium compound **4** in 5 ml of DMF, one equivalent of the chloro-deoxy-ephedrine hydrochloride or chloro-deoxy-*nor*ephedrine was added and the solution was refluxed for 8 h. The DMF was evaporated under

TABLE 5 Crystal Data and Data Collection Parameters for Compounds **5a–d**

	5a	**5b**	**5c**	**5d**
Chem. formula	$C_{17}H_{15}N_3S_2$	$C_{18}H_{17}N_3S_2$	$C_{17}H_{15}N_3OS$	$C_{18}H_{17}N_3OS$
Form. weight.	325.44	339.47	309.38	323.41
Cryst. size [mm]	$0.2 \times 0.2 \times 0.45$	$0.2 \times 0.2 \times 0.2$	$0.12 \times 0.22 \times 0.50$	$0.20 \times 0.20 \times 0.20$
Cryst. system	Monoclinic	Orthorhombic	Orthorhombic	Orthorhombic
Space group	C2	P 21 21 21	P 21 21 21	P 21 21 21
a (Å)	20.531(6)	7.371(3)	5.7712(5)	10.546(3)
b (Å)	5.3968(14)	13.093(3)	14.0701(12)	11.010(2)
c (Å)	14.319(4)	17.609(4)	19.2842(18)	14.883(3)
α (°)	90.00	90.00	90.00	90.00
β (°)	100.010(7)	90.00	90.00	90.00
γ (°)	90.00	90.00	90.00	90.00
V (Å^3)	1562.4(7)	1699.4(9)	1565.9(2)	1728.0(7)
Z	4	4	4	4
ρ (calcd.) (Mg/m^3)	1.384	1.327	1.312	1.243
μ (mm^{-1})	0.340	0.315	0.212	0.195
F(000)	680	712	648	680
Index range	$-25 \le h \le 25$ $-5 \le k \le 5$ $-18 \le l \le 18$	$-8 \le h \le 0$ $0 \le k \le 15$ $0 \le l \le 20$	$-7 \le h \le 0$ $0 \le k \le 17$ $0 \le l \le 23$	$0 \le h \le 13$ $0 \le k \le 14$ $0 \le l \le 18$
2θ(°)	55.20	49.90	51.92	53.94
Temp (K)	193(2)	293(2)	293(2)	273
Refl. collected	4329	3466	1849	2203
Refl. unique	2752	1733	1712	2147
Refl. observed (4σ)	2251	1382	901	1027
R (int.)	0.0411	0.0012	0.01100	0.0102
No. variables	211	277	214	226
Weighting scheme[a] x/y	0.0520/0.8594	0.0677/0.1965	0.0609/0.0000	0.0676/0.000
GOOF	1.035	0.968	0.907	0.992
Final $R(4\sigma)$	0.0411	0.0346	0.0397	0.0368
Final $wR2$	0.0891	0.0876	0.1028	0.1039
Larg. Res. Peak [e/Å^3]	0.189	0.188	0.206	0.136
Absolute Structure[b]	0.00(13)	0.00(13)	0.0(3)	0.00(11)

[a] $W^{-1} = \sigma^2 F_0^2 + (xP)^2 + yP$; $P = (F_0^2 + 2F_c^2)/3$,
[b] Ref. [16].

vacuum and **5a** was crystallized from acetone and **5b** from methanol.

*4R,5R-2-[2-Benzothiazolylimino]-3-hydro-4-methyl-5-phenylthiazolidine (**5a**).* White crystalline compound, 60% yield. mp 148–150°C. $[\alpha]_D = -176.6°$ (1.186 g/100 ml, THF), IR $\nu = 1567.9$ cm^{-1} (C=N). Mass: *m/z* (%) [M$^+$] 325, (100). Anal. cald for $C_{17}H_{15}N_3S_2$: C, 62.74; H, 4,65; N, 12.91. Found: C, 62.70; H, 4.52; N, 12.88.

*4R,5R-2-[2-Benzothiazolylimino]-3,4-dimethyl-5-phenylthiazolidine (**5b**).* Crystalline yellow solid, 50% yield. mp 155°C. $[\alpha]_D = +221.3°$ (15 mg/10 ml, $CHCl_3$). IR $\nu = 1546$ cm^{-1} (C=N). Mass: *m/z* (%), [M$^+$] 339, (100). Anal. cald for $C_{18}H_{17}N_3S_2$: C, 63.69; H, 5.05; N, 12.38. Found: C, 63.81; H, 5.08; N, 12.36.

*2-[2-Benzothiazolylimino]heterazolidines (**5c–e**). General Procedure*

To a solution of 1.0 g (3.94 mmol) of compound **2** in 5 ml of DMF, one equivalent of 1*S*, 2*R*-ephedrine, 1*R*, 2*S*-*nor*ephedrine or (1*R*,2*S*-*N*,*N*-dimethyl-2-phenyl-3-methyl-1,3-diaminoethane was added and the reaction mixture was refluxed for 8 h. The DMF was evaporated under vacuum, **5c** was crystallized from methanol and **5d** from ethyl acetate. Compound **5e** was purified by chromatography using a column of silica gel with $CHCl_3$ as eluent.

*4S,5R-2-[2-Benzothiazolylimino]-3-hydro-4-methyl-5-phenyloxazolidine (**5c**).* mp 130–131°C. $[\alpha]_D = -54.54°$ (17.6 mg/10 ml, $CHCl_3$), IR $\nu = 1622.6$ cm^{-1} (C=N). Anal. cald for $C_{17}H_{15}ON_3S$: C, 66.0; H, 4,89; N, 13.58. Found: C, 65.91; H, 4.77; N, 13.43.

*4S,5R-2-[2-Benzothiazolylimino]-3,4-dimethyl-5-phenyloxazolidine (**5d**).* mp 233–235°C. $[\alpha]_D = +50.4^\circ$ (18 mg/10 ml, $CHCl_3$), IR $\nu = 1644$ cm^{-1} (C=N).

*4R,5S-2-[2-Benzothiazolylimino]-1,3,4-trimethyl-5-phenyl-imidazolidine (**5e**).* Brown reddish liquid. $[\alpha]_D = -54.3^\circ$ (20.0 mg/10 ml, $CHCl_3$), IR $\nu = 1598$ cm^{-1} (C=N). Anal. cald for $C_{19}H_{20}N_4S \cdot 3/4CHCl_3$: C, 55.69; H, 4,91; N, 13.15. Found: C, 56.97; H, 5.15; N, 13.0.

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