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N-BORANE ADDUCTS OF OXAZOLIDINES DERIVED FROM EPHEDRINE AND PSEUDOEPHEDRINE. STUDY OF STEREOCHEMISTRY BY NUCLEAR MAGNETIC RESONANCE

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Abstract - A series of stable N-borane-oxazolidine adducts has been prepared in high yield from the reaction of 4-methyl-5-phenyl-oxazolidines with BH_3.DMS. The configurations of all compounds were unambiguously assigned by ^1H, ^{11}B and ^{13}C NMR spectroscopy. Isolation of one pair of N-epimers from ephedrine and another pair from pseudoephedrine let us consider and discuss their stereochemical and spectroscopy relationship.

The stabilization of the configuration for the nitrogen atom by borane addition has been described previously.[1-4] Recently we reported the stereochemical consequences of N-quaternization by borane in several ephedrines.[5] The stable nature of these derivatives permitted us to study one of their more important characteristics which consists in the fixation of the nitrogen configuration, that according to an adequate substitution could generate a new chiral centre.

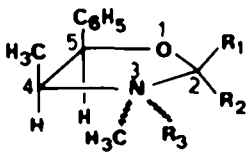
Extending our study on the stereochemistry of the complexes formed by borane addition to cyclic derivatives from ethanolamines, we have now prepared eight oxazolidines derived from optically active ephedrines 1-8 and their corresponding borane adducts 9-16. (Scheme 1).

RESULTS AND DISCUSSION.

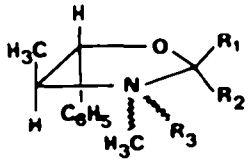
The oxazolidines 3 and 7 derived from acetaldehyde and 4 and 8 derived from benzaldehyde with the corresponding optically active ephedrines were composed mainly of one diastereoisomer (≥95%). The configuration of the oxazolidine 2-carbon in each compound was assigned by ^1H $\{^1H\}$ Nuclear Overhauser Effect (NOE) difference spectra.[6]

Although a wide variety of oxazolidines have been known,[7-9] the synthesis of their borane adducts has not been described. It was reported that the reaction of diborane with oxazolidines afforded the corresponding aminoalcohols.[10] However, there is no mention of isolation or detection of any intermediate N-borane species.

Treatment of 1-8 in equimolar amounts with BH_3.DMS in DMS solution at low temperature (-30°C), resulted in the isolation of the corresponding N-borane adducts 9-16. The addition of borane to the oxazolidines 1-8 should in principle give two N-epimers in each case, one with a configuration in which the N-BH_3 group is *cis* to C-4-Me and another *trans* to it. (Scheme 2).

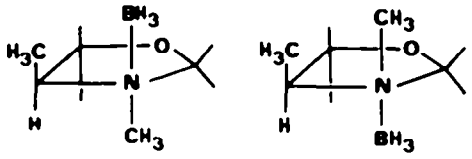


	R_1	R_2	R_3
1	H	H	lone pair
2	CH_3	CH_3	lone pair
3	CH_3	H	lone pair
4	C_6H_5	H	lone pair
9	H	H	BH_3
10	CH_3	CH_3	BH_3
11	CH_3	H	BH_3
12	C_6H_5	H	BH_3



	R_1	R_2	R_3
5	H	H	lone pair
6	CH_3	CH_3	lone pair
7	CH_3	H	lone pair
8	C_6H_5	H	lone pair
13	H	H	BH_3
14	CH_3	CH_3	BH_3
15	CH_3	H	BH_3
16	C_6H_5	H	BH_3

Scheme 1



Scheme 2

Evidence based on NMR spectra of isolated products confirmed the presence of only one N-epimer in each product, indicating that one of the structures is highly favoured. However, ^{11}B-NMR spectra of the reaction mixture showed two ^{11}B-resonances, one of which disappeared within a few minutes. Therefore, it was suggested that the initial product of the reaction is a mixture of two N-epimers. The isomers which have been detected and observed in the final stage of the reaction are denoted as a and those that virtually disappear during the reaction as b. In each case the isomers a have been isolated as pure compounds. If the solvent is evaporated immediately after the addition of borane, or if the reaction carried out at -78°C, a solid product can be isolated, being a mixture of the isomers a and b. The isomers b have only been isolated in pure form from the pseudoephedrine derivatives 7 and 8 and a mixture of isomers 9a and 9b was obtained from the ephedrine derivative 1 (see Figure 1). In other cases isomers b have only been observed in ^{11}B NMR.

In view of the facts discussed above it seems that isomer b correspond to the kinetic product of the reaction, which undergoes conversion to the dominant adduct a. It appears that the isomer b could be a product of the approach of BH_3 by the same side of the N-lone pair in the most stable nitrogen configuration of these oxazolidines. (See below).

At this point our attention was turned to the configurational assignment of the isolated isomers, and also to the configuration of the oxazolidine 2-carbon of 3, 4, 7 and 8 which for a long time has been a controversial point in the literature.[9,11,12]

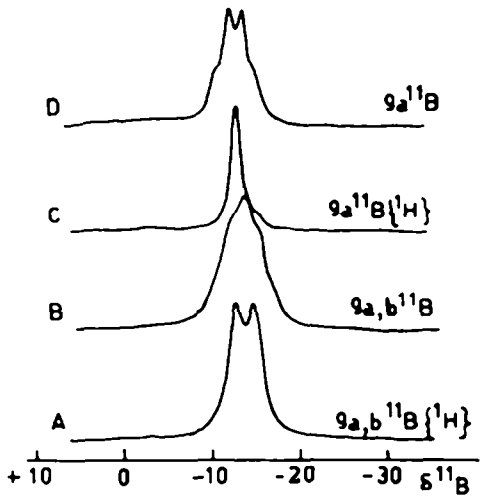
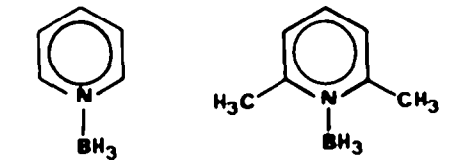


Figure 1. 64.21 MHz ^{11}B-NMR of the borane adducts 9a, 9b (as a mixture) and of pure 9a. A: 9a + 9b, ^{1}H-broad band decoupled; B: 9a + 9b, ^{1}H-coupled; C: 9a, ^{1}H-broad band decoupled; D: 9a, ^{1}H coupled.

NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY STUDY.

^{11}B NMR.- (Table 1). It is known that steric crowding at the boron atom is reflected by ^{11}B chemical shifts (δ ^{11}B).[13,14] The ^{11}B-resonance of 2,6-dimethylpyridine borane (18) is shifted by 6 ppm to lower frequency with respect to pyridine borane (17). (Scheme 3).



17 18

Scheme 3

Therefore, the δ ^{11}B values are useful to distinguish between the N-epimeric oxazolidine-borane adducts, allowing the nitrogen configuration to be determined in the isomers 9a, 10a, 13a and 14a (scheme 4a) which are derivatives from 1, 2, 5, and 6. (Table 1). The comparison with δ ^{11}B values of three oxazolidine borane adducts 19-21 (Scheme 4b) which have been synthesised to serve as structural models corroborates the assignments in scheme 4a.

As illustrated in scheme 4b, the ^{11}B-nuclear shielding increases when one of the hydrogen atoms which are in close spatial contact to the BH_3-group (21) is replaced by a methyl group (19, 20). According to the substituent position on C-4 or C-2, the shielding is 4 and 1.4 ppm respectively. The compounds 9a, 13a and 10a, 14a should have a borane group trans to C-4-Me, in other case the corresponding δ ^{11}B values should be close to -16 or -17 ppm respectively.

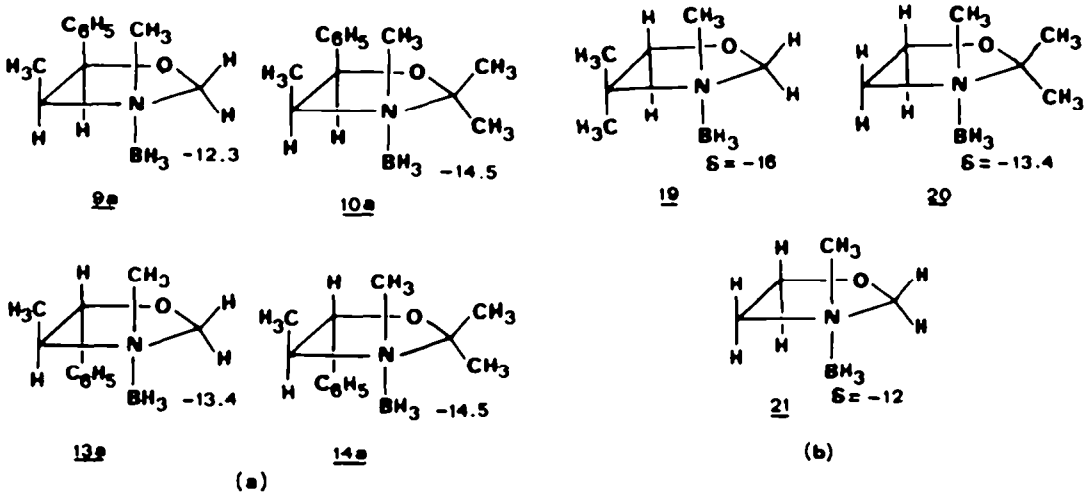
The δ ^{11}B values for the substituted compounds with a methyl (11a) or a phenyl group at C-2 (12a) are very similar to those of 9a (scheme 5); this suggests that the

TABLE 1. ^{1}H and ^{11}B NMR shifts[a] of oxazolidines and the corresponding borane adducts.

COMPOUNDS	H-5	H-4	C-4-CH_3	N-CH_3	C-2-H	C-2-R	C-5-ϕ	^{11}B[c]
1	5.09(d) 7.0Hz	2.84(m)	0.63(d) 6.7Hz	2.32(s)	4.05(d) 4.86(d) 3.0Hz		7.30(s)	
2	5.0(d) 9Hz	3.11(m)	0.61(d) 7.5Hz	2.26(s)		1.21(s) 1.51(s)	7.30(m)	
3[b]	4.92(d) 7.9Hz	2.70(dc)	0.60(d) 6.4Hz	2.20(s)	3.88(c) 5.2Hz	1.42(d) 5.2Hz	7.31(s)	
4[b]	5.12(d) 8.2Hz	2.94(dc)	0.76(d) 6.4Hz	2.15(s)	4.67(s)	7.18-7.70(m)		
5	4.49(d) 8.3Hz	2.44(m)	1.14(d) 6.1Hz	2.32(s)	4.29(d) 4.75(d) 3.0Hz		7.31(s)	
6	4.45(d) 8.7Hz	2.58(m)	1.08(d) 6.0Hz	2.28(s)		1.32(s) 1.42(s)	7.38(s)	
7[b]	4.52(d) 8.6Hz	2.33(dc)	1.12(d) 6.1Hz	2.24(s)	4.22(c) 5.2Hz	1.38(d) 5.2Hz	7.2-7.4(m)	
8[b]	4.75(d) 8.5Hz	2.50(dc)	1.19(d) 6.1Hz	2.16(s)	4.92(s)	7.1-7.6(m)		
9a[b,d]	5.30(d) 9.3Hz	3.85(m) 9.3,7.3Hz	0.88(d) 7.3Hz	2.58(s)	4.60(d) 4.70(d) 6.6Hz		7.25-7.43(m)	-12.3
9b[d]	5.56(d) 7.2Hz	3.50(m)	0.90(d) 6.8Hz	2.98(s)	4.48(d) 4.83(d) 6Hz		7.20-7.5(m)	-14.8
10a[b,d]	5.24(d) 9.0Hz	4.17(m)	0.94(d) 6.7Hz	2.42(s)		1.70(s)	7.17-7.5(m)	-14.5
11a[b]	5.16(d) 9.5Hz	3.88(dc)	0.94(d) 7.3Hz	2.29(s)	4.71(c) 5.8Hz	1.64(d) 5.8Hz	7.2-7.44(m)	-12.8
12a[b]	5.40(d) 9.5Hz	4.08(dc)	0.98(d) 7.3Hz	2.11(s)	5.68(s)	7.3-7.98(m)		-12.3
13a	4.68(d) 9.3Hz	3.24(m)	1.33(d) 7.2Hz	2.66(s)	4.60(d) 4.72(d) 7.0Hz		7.30(m)	-13.4
14a	4.66(d) 10.5Hz	3.62(m)	1.38(d) 6.0Hz	2.55(s)		1.62(s) 1.72(s)	7.40(m)	-14.5
15a[b]	4.56(d) 9.7Hz	3.40(dc)	1.35(d) 6.8Hz	2.38(s)	4.90(c) 5.7Hz	1.54(d) 5.7Hz	7.25-7.42 (m)	-13.8
15b[b]	4.93(d) 9.9Hz	2.87(dc)	1.35(d) 6.8Hz	2.54(s)	4.60(c) 5.7Hz	1.62(d) 5.7Hz	7.24-7.42 (m)	-19.1
16a[b]	4.83(d) 9.5Hz	3.62(dc)	1.41(d) 6.1Hz	2.16(s)	5.89(s)	7.25-7.80(m)		-13.3
16b[b]	5.27(d) 8.0Hz	3.10(dc)	1.44(d) 6.4Hz	2.60(s)	5.42(s)	7.30-7.70(m)		-18.4

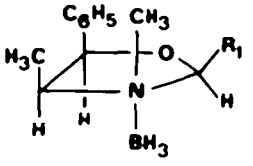
a) In $CDCl_3$ relative to TMS (δ=0). 90 MHz. b) 200 MHz. c) In $CDCl_3$ relative to BF_3OEt_2 (δ=0). All signals are quartets. d) δ ^{1}H values of the BH_3 groups: 9a, 1.66; 9b, 1.59; 10a, 1.55 ppm (decoupled in ^{11}B).

borane group is trans to C-4-Me and that the configuration at C-2 must be that in which the substituent group R_1 is cis to C-4-Me.



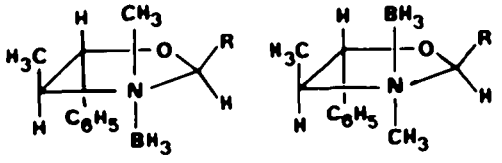
Scheme 4. ^{11}B NMR chemical shifts of compounds 9a, 10a, 13a, 14a, and 19-21.

In the borane adducts from oxazolidine 7 and 8 for which both isomers were isolated, ^{11}B-resonances (scheme 6) for the isomers 15a, 16a and for 15b, 16b are very similar. This indicates that the configuration in the nitrogen and in the C-2 must be identical, which also applies to the two isomers 15b, 16b. A notable difference of ~ 5 ppm is observed between the shifts of the isomeric species a and b. The δ ^{11}B-resonances which are found at lower frequency for the isomers b are clearly indicating the presence of a steric interaction between the BH_3-group and the methyl, phenyl at C-2 and C-4. It is suggested from the similarity of the δ ^{11}B values for 15a, 16a and 13a that the borane must be trans to C-4-Me and also to C-2-R_1 whereas the ^{11}B-resonances for 15b and 16b suggest that the borane should be cis to C-4-Me and to C-2-R_1.



9a R_1 = H δ = -12.3
11a R_1 = CH_3 δ = -12.8
12a R_1 = C_6H_5 δ = -12.3

Scheme 5



15a R_1=CH_3 δ = -13.8 15b R_1=CH_3 δ = -19.1
16a R_1=C_6H_5 δ = -13.3 16b R_1=C_6H_5 δ = -18.4

Scheme 6

^1H NMR.- (Table 1). The analysis of the ^1H chemical shifts is showing interesting features:

a) Correlation of Δδ ^1H for the different hydrogens between the oxazolidines and the oxazolidine-borane adducts a derived from the pseudoephedrine series 13-16 or from the ephedrine series 9-12 show the same trend through all the chemical shifts as illustrated in Figure 2 and 3. (Δδ= δ(^1H) oxazolidine-δ(^1H) oxazolidine borane). This suggests that in each series of compounds the configuration does not change.

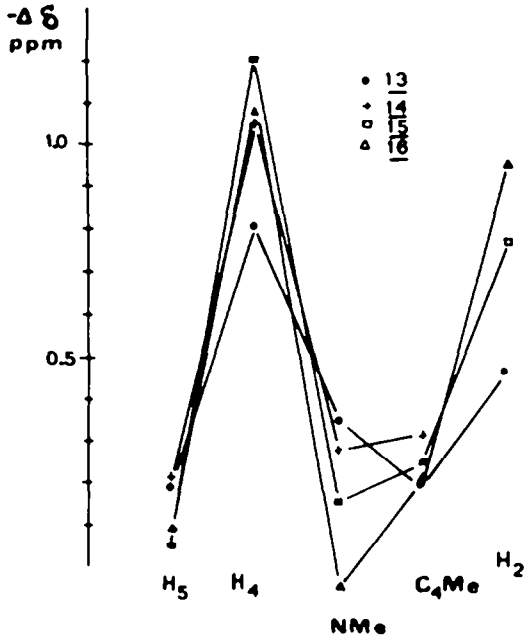


Figure 2. Correlation of $\Delta\delta$ ^1H between oxazolidines 5-8 and oxazolidine borane adducts a 13-16.

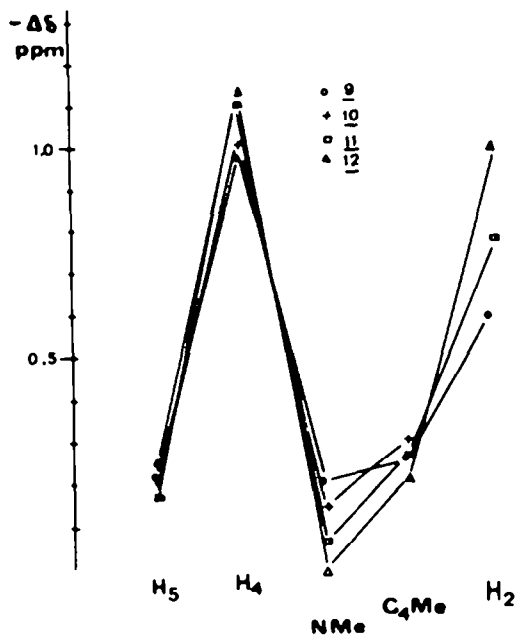


Figure 3. Correlation of $\Delta\delta$ ^1H between oxazolidines 1-4 and oxazolidine borane adducts a 9-12.

A plot of the $\Delta\delta$ ^1H of the pair of compounds 15a and 15b, along with 16a and 16b, is showing that 15a and 16a have similar trends and analogous situation is observed from 15b and 16b. (Figure 4). The complete set of data seems to be reasonably consistent and we assume that the nitrogen configuration for each pair 15a and 16a or 15b and 16b should be the same.

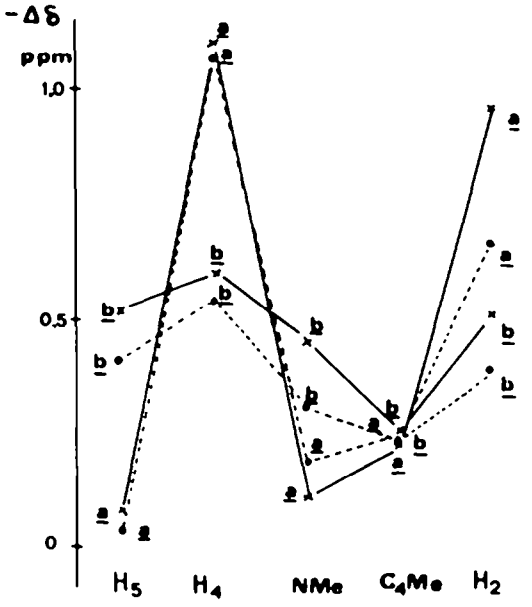


Figure 4. Relationship between variation in chemical shifts of the pair of isomers 15a, 15b (dashed line) and 16a, 16b, (solid line) with respect to oxazolidines 7 and 8.

TABLE 2. ^{13}C NMR shifts[a] of oxazolidines and their corresponding borane adducts.

COMPOUND	C-2	C-4	C-5	C-2-R	C-4-Me	N-Me	C_1'(i)	C_2'(o)	C_3'(m)	C_4'(p)
1[f]	87.6	62.7	81.4		13.6	36.9	139.6	126.5	127.3	126.7
2[f]	95.0	60.5	80.8	18.2(e) 26.6(d)	15.4	33.4	140.5	127.7 or	127.8[g]	127.3
3	93.8	64.2	81.9	19.0	14.7	35.9	140.3	127.8	127.8	127.5
4	99.0	64.1	82.6	138.4(i) 128.1(o) 128.5(m) 129.2(p)	15.1	35.8	140.0	128.0	128.50	127.7
5	89.0[b]	68.30	86.20		14.60	37.30	140.90	126.40	128.60	127.90
	88.22[c]	67.79	85.26		14.02	36.36	140.85	125.89	127.95	127.23
6[c]	94.49	64.58	84.03	27.47(d) 21.21(e)	14.27	32.19	139.98	126.21	127.87	127.30
7	94.7	69.1	85.4	19.8	14.4	35.6	140.90	126.60	128.40	127.80
	93.96[c]	68.67	84.62	19.62	14.10	35.14	140.66	126.06	127.80	127.19
8	99.6	68.8	86.5	139.7(i) 128.1(o) 128.3(m) 129.0(p)	14.4	35.1	140.6	126.7	128.4	127.90
9a[f]	91.3	65.8	81.7		10.9	42.6	136.6	125.9	128.2	127.9
9b[f]	89.9	67.9	81.2		13.9	50.9	136.9	125.8	128.2	127.7
10a[f]	100.6	64.9	77.8	21.9 22.9	12.0	40.1	136.9	126.1	128.1	127.7
11a	95.6	67.5	79.6	13.6	11.6	35.7	137.2	126.5	128.6	128.2
12a	99.1	67.3	79.8	131.4(i) 128.0(o) 129.2(m) 130.2(p)	11.9	37.6	137.3	126.5	128.7	128.3
13a[c]	92.35	71.36	82.99		9.14	41.10	138.74	125.85	128.54	128.21
14a[c]	101.42	68.33	81.46	22.52(d) 27.03(e)	10.29	38.87	138.60	125.74	128.25	127.75
15a	96.7	72.5	82.3	13.90	10.4	34.1	138.80	125.80	128.80	128.50
	96.22[c]	72.38	80.86	13.71	9.83	34.0	139.17	125.79	128.49	128.06
15b	98.5	74.2	83.3	15.5	10.8	45.7	139.0	126.10	128.70	128.50
16a	100.6	72.5	82.8	131.4(i) 130.2(p) 127.8 129.3(o,m)[d]	10.7	35.7	138.8	126.0	129.0	128.70
16b[h]	101.3	72.8	82.2	130.6(i)	9.6	44.2	137.6			

a) In $CDCl_3$ relative to TMS (δ=0) 25.2 MHz. b) In $CDCl_3$ relative to TMS (δ=0). Ref. 8. c) In $(CD_3)_2$ SO relative to TMS (δ=0) 25.2 MHZ. Ref. 7 d) Me cis to C-4-Me. e) Me trans to C-4-Me. f) In $CDCl_3$ relative to TMS (δ=0) 50.3 MHz. g) Assignment may be reversed. h) Assignment in aromatic region unreliable.

b) The presence of borane in all compounds 9-16 affects the C-4 hydrogen deshielding; this indicates the mutual trans position of BH_3 and C-4-Me. In compounds 11-12 and 15-16 the C-2-H is suffering a deshielding in a similar manner than C-4-H, suggesting in these compounds that the C-2 configuration is that in which the substituent R_1 is cis to C-4-Me. Then, the oxazolidines 3-4 and 7-8 have the configuration illustrated in scheme 7.

c) It is of interest to note the small sensitivity of the C-Me groups to the nitrogen configuration.

Finally, the configurations of oxazolidines 3, 4, 7 and 8 and also of the corresponding borane adducts 11a, 12a, 15a, 15b, 16a and 16b were obtained by differential NOEs[6], and these results are supporting the conclusions obtained through out this work. The experimenal details are given in this paper.

^{13}C-NMR.- (Table 2). As can be observed, the N-Me ^{13}C-resonances in adducts b are shifted to higher frequency with respect to adducts a; there is less steric compression of the N-Me group in the adduct b than in adduct a, confirming the proposed structure for these adducts.

A comparative study for the steric compression effects between a N-BH_3 and N-Me group in the borane oxazolidine compounds and the oxazolidinium salts, both derived from pseudoephedrine confirm that the configurations are assigned correctly.[15]

CONCLUSIONS.

1) Ten adducts of oxazolidine borane derivatives from ephedrines and pseudoephedrines were synthesised. Their stability encouraged us to carry out a thorough stereochemical study.

2) The configuration of all compounds was unambiguously assigned by ^{11}B, ^{1}H and ^{13}C NMR spectroscopy.

3) The configurations of the major isomers obtained from the syntheses of the oxazolidines 3-4 and 7-8 were determined, and it has been demonstrated that all these compounds have the same configuration with the substituent in C-2 cis to C-4. The recent report of the X-ray structure redetermination of (2S, 4S, 5R)-2-(p-bromophenyl)-3,4-dimethyl-5-phenyloxazolidine[16] is also in agreement with our assignments.

4) One isomer of the borane addition products a is thermodynamically much more stable than the other b. This is remarkable, since the BH_3 and CH_3 groups are very similar, as far as their steric requirement is concerned.

EXPERIMENTAL.

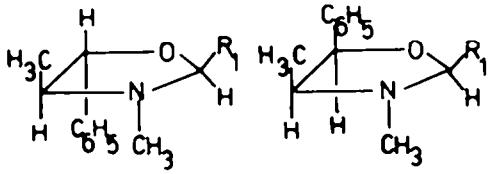
^{1}H NMR spectra were recorded at 90 MHz on a Varian EM-390 spectrometer, and at 200 MHz on a Bruker WP200 instrument in $CDCl_3$ solution with TMS as internal reference. Chemical shifts are expressed in units of δ (ppm downfield from TMS) and coupling constants (J) are given in Hertz (Hz). For the NOE difference spectra[17] (Bruker WP 200) a presaturation time of 10 s with an r.f. power setting of 40-50L (low power for selective irradiation) was used to build up the NOE. Difference for off resonance and on resonance spectra were generated after any eight transients. 16 x 16 transients were sufficient to observe NOEs > 1%. Simultaneous ^{11}B-decoupling was used to assign the BH_3- resonances and for some of the NOE difference experiments.

^{11}B NMR spectra were recorded on a Varian XL-100A (32.1 MHz) or a Bruker WP200 (64.2 MHz) spectrometer using $BF_3.Et_2O$ as external reference.

TABLE 3. Analytical and infrared data for N-borane oxazolidine compounds.

Compound	M.W.[a]	m.p.[b] (°C)	Yield (%)	IR(cm^{-1})[d] B-H	B←N
9a	191	93	98	2373(s) 2316(m) 2276(w)	1162(s)
10a	219	75	90	2389(m) 2367(m) 2346(w)	1160(m)
11a	205	83	76	2374(vs) 2317(m) 2277(m)	1164(s)
12a	267	97	90	2374(s) 2370(m) 2275(m)	1164(s)
13a	191	98	99	2377(s) 2336(m) 2272(w)	1167(s)
14a	219	79	83	2379(s) 2335(m) 2281(w)	1161(m)
15a	205	95	50	2385(s) 2328(m) 2280(m)	1161(m)
15b	205	75	50	2384(s) 2373(m) 2275(m)	1160(m)
16a	267	100	99	2382(s) 2332(m) 2275(m)	1166(s)
16b	267	85	34	2383(s)	1168(m)

a) Determined from the mass spectra, based on ^{11}B isotope only. b) Melting points are uncorrected. c) Crude product. d) KBr pellets.



7 $R_1 = CH_3$ 3 $R_1 = CH_3$

8 $R_1 = C_6H_5$ 4 $R_1 = C_6H_5$

Scheme 7

^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO (δ, ppm, TMS) on either a Bruker WP200 (50.3 Hz) or Varian XL-100A (25.2 MHz).

IR spectra were recorded KBr pellets on a Nicolet MX-1-FT spectrophotometer. Mass spectromety was performed on a Hewlett-Packard 5985-A instrument with a ionizing potential of 15 eV.

Melting points were determined on a Gallempak apparatus and are uncorrected.

All solvents were dried and purified by standard techniques prior to use, and the reactions were carried out under nitrogen atmosphere. The preparation of the oxazolidines have been reported elsewhere.[12,18,19]

General procedure for the preparation of N-borane oxazolidine adducts: The syntheses of the compounds 9-16 have been conducted in DMS, with equimolar amounts

of borane in dimethylsulphide (BH_3.DMS) and the respective oxazolidine. (An excess of borane in some reactions produce open-ring products).

The borane was added dropwise under cooling at ≈30°C. After 15 min at room temperature, the solvent was evaporated under reduced pressure to give the corresponding a adducts. The isolation of b was carried out in exactly the same manner, but with an immediate removal of the solvent after the addition of borane, or by addition of the borane at -78°C.

The crude products, as required, were purified by chromatography on a short column of silica with a mixture of hexane and methylene chloride, giving white crystalline solids. An alternative method used was recrystallization from chloroform and hexane. Analytical and IR data is given in Table 3. All oxazolidine-borane adducts were stable in air on standing for several days. Compounds 10a and 14a were the most unstable species, showing decomposition after a few weeks.

^{13}C-NMR spectra of solutions (DMSO) of 14a showed decomposition into the oxazolidine product on standing for 1 h. Also, this sample decomposed in chloroform.

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REFERENCES.

1. J.C. Fraud and H.B. Kagan, Bull. Soc. Chim. Fr., 2742 (1969).
2. R.E. Lyle, E.W. Southwick and J.J. Kaminsky, J. Amer. Chem. Soc. 94, 1413 (1972).
3. A. Picot and X. Lusinchi, Bull. Soc. Chim. Fr., 1227 (1977).
4. M.F. Grundon, D.G. McCleery and J.W. Wilson, J. Chem. Soc. Perkin I, 231 (1981).
5. F. Santiesteban, T. Mancilla, A. Klaébé and R. Contreras, Tetrahedron Letters, 24, 759 (1983).
6. F. Santiesteban, C. Grimaldo, R. Contreras and B. Wrackmeyer, J. Chem. Soc. Chem. Comm. 1486 (1983).
7. L. Knorr and H. Matthes, Chem. Ber. 3484 (1901).
8. A.C. Cope and E.M. Hancock, J. Amer. Chem. Soc. 64, 1503 (1942).
9. L. Neelakantan, J. Org. Chem. 36, 2256 (1971).
10. C. Fuganti, D. Ghiringhelli, P. Graselli and M. Mazza, Tetrahedron Letters, 26, 2261 (1974).
11. G. Just, C. Luthe and P. Potvin, Tetrahedron Letters, 2285 (1982).
12. A.H. Beckett and G.R. Jones, Tetrahedron 33, 3313 (1977).
13. H. Nöth and B. Wrackmeyer, "NMR Spectroscopy of Boron Compounds" in NMR-Basic Principles and Progress, eds. P. Diehl, E. Fluck, R. Kosfeld, Vol. 14 Springer Verlag, Berlin, 1978.
14. E.F. Mooney and M.A. Anaseem, J. Inorg. Nucl. Chem. 30, 1439 (1968).
15. M.A. Paz-Sandoval, F. Santiesteban and R. Contreras, Submitted for publication.
16. G. Just, P. Potvin, P. Uggowitzer and P. Bird, J. Org. Chem. 48, 2923 (1983).
17. R. Benn, A. Rufińska and G. Schroth, J. Organometal. Chem. 217, 91, (1981).
18. M. Baudet and M. Gelbcke, Analytical Lett. A12, 641 (1979).
19. M. Baudet and M. Gelbcke, Analytical Lett., 12(B4), 325 (1979).