Robert F. X. Klein,[1] *Ph.D.; Albert R. Sperling,*[1] *Ph.D.; Donald A. Cooper,*[1] *B.S.; and Theodore C. Kram,*[1] *B.S.*

The Stereoisomers of 4-Methylaminorex

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ABSTRACT: Physical constants and instrumental data (melting point [mp], thin-layer chromatography [TLC] [R_f], gas chromatography [GC] [R_t], $[\alpha]_D^{25}$, ^{1}H- and ^{13}C-nuclear magnetic resonance [NMR], infrared (IR), 70-eV, electron impact-mass spectroscopy [EI-MS], color, and microcrystalline tests) are reported for the individual stereoisomers, racemates, and corresponding hydrochloride salts of 4-methylaminorex (2-amino-4-methyl-5-phenyl-Δ^2-oxazoline, 4,5-dihydro-4-methyl-5-phenyl-2-oxazolamine, McN-822, "U4Euh," "ICE"). The data allow identification and differentiation of illicit samples of 4-methylaminorex.

KEYWORDS: forensic science, 4-methylaminorex (U4Euh), 2-amino-4-methyl-5-phenyl-Δ^2-oxazoline, stereoisomers, anorectic agent, CNS stimulant, Controlled Substances Act

4-Methylaminorex (Fig. 1) (2-amino-4-methyl-5-phenyl-Δ^2-oxazoline, 4,5-dihydro-4-methyl-5-phenyl-2-oxazolamine, McN-822, "U4Euh," "ICE") is a potent anorectic [*1-3*][2] and central nervous system stimulant [*2-8*][2] possessing sympathomimetic [*9*],[2] hypertensive [*4*], and norepinephrine potentiating [*10*] properties. It has been observed with increasing frequency on the clandestine market [*11,12*][3,4] and was recently controlled (Schedule I) under the emergency scheduling procedures of the Controlled Substances Act [*13,14*].

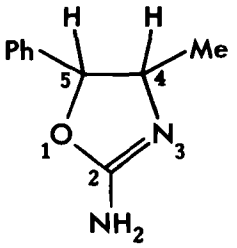

FIG. 1—*4-Methylaminorex.*

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[1]Forensic chemists, respectively, Drug Enforcement Administration, Special Testing and Research Laboratories, McLean, VA.

[2]A. C. Church, Drug Enforcement Administration, Washington, DC, unpublished work, 1987.

[3]F. Sapienza and A. C. Church, Drug Enforcement Administration, Washington, DC, personal communications, 1988.

[4]D. Inaba and L. M. Brewer, personal communication, 1987.

The compound contains two chiral centers (C_4 and C_5); thus, there are four possible stereoisomers (*trans*-(4*R*, 5*R*), *trans*-(4*S*, 5*S*), *cis*-(4*R*, 5*S*) and *cis*-(4*S*, 5*R*)) and two racemates (*dl-cis* and *dl-trans*) (Fig. 2).

The individual stereoisomers and racemates are most conveniently synthesized via condensation of the respective 2-amino-1-phenylpropanols with cyanogen bromide [*1*] (Figs. 3 and 4). The stereochemistry of the starting material is maintained through the course of the reaction; thus, the *erythro* amino-alcohol (norephedrine) gives the corresponding *cis*-4-methylaminorex, the *threo* amino-alcohol (norpseudoephedrine) the corresponding *trans* product (Fig. 4; note: C_1, C_2 in the starting material corresponds to C_5, C_4 in the product).

Pharmacological studies have shown similar anorectic [*1,2*] central nervous system stimulant [*4*], hypertensive [*4*], and norepinephrine potentiating [*10*] activities regardless of stereochemistry. The racemic *dl-cis* free base is currently the most frequently encountered form in illicit samples of 4-methylaminorex[3] and has been fully characterized in several reports [*12*];[4] however, only minimal physical data have been reported for any of the individual stereoisomers, the *dl-trans* racemate, or the corresponding hydrochloride salts.

Experimental Procedures

All precursor chemicals were obtained from Aldrich and used without additional purification. All other chemicals used were reagent grade or better.

Melting points were determined using a Fisher-Johns melting point apparatus and are uncorrected. Thin-layer chromatograms were run on silica gel (Baker-flex Silica Gel IB-F,

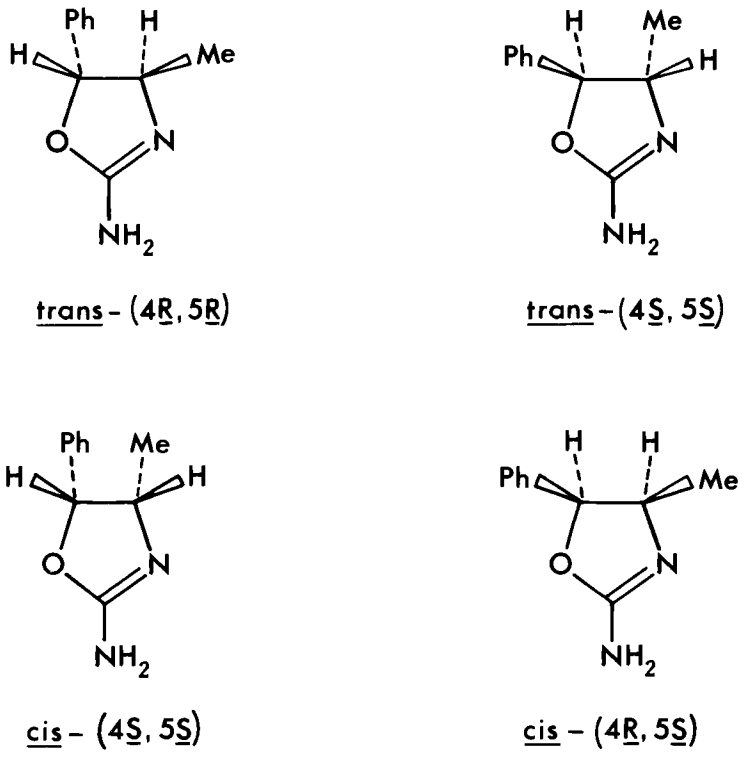

FIG. 2—*Stereoisomers of 4-methylaminorex.*

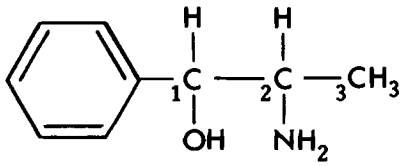

FIG. 3—*2-Amino-1-phenylpropanol.*

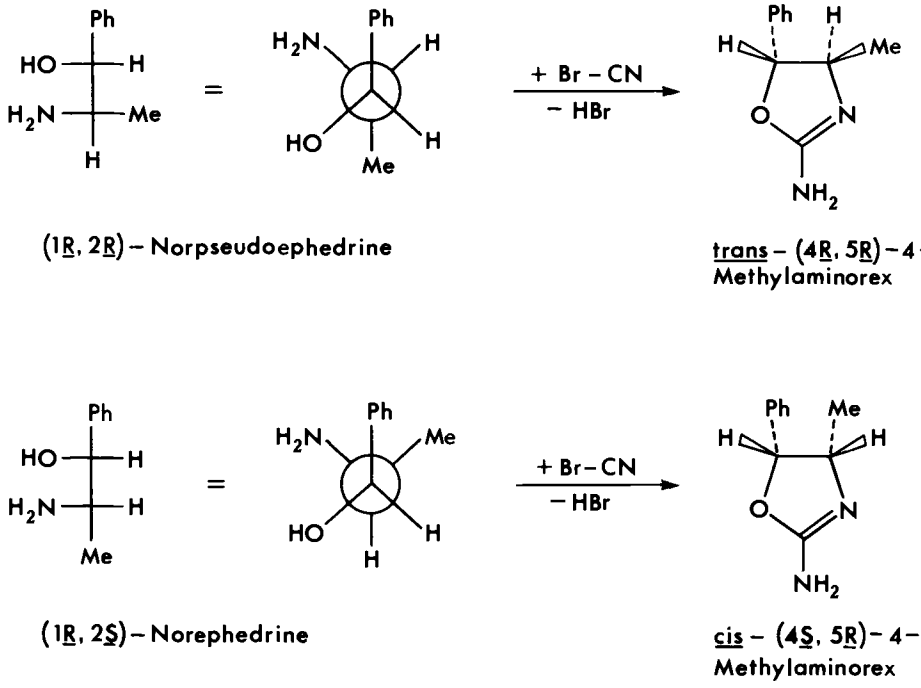

FIG. 4—*Stereochemical relationships between precursor 2-amino-1-phenylpropanols (shown in Fisher and Newman projections) and product 4-methylaminorexes.*

J. T. Baker 4-4463), alumina (Baker-flex Alumina IB-F, J. T. Baker 4-4467), and C_{18}-reverse phase (Whatman $MKC_{18}F$, 4803-110) thin-layer plates. Gas chromatographic (GC) analyses were performed on a Perkin-Elmer Sigma 2000 Capillary Chromatograph equipped with a DB-1 30-m by 0.25-mm capillary column coated with a 0.25-μm film thickness (J&W Scientific) and a flame ionization detector. Hydrogen was used as the carrier gas (linear velocity 4.65 cm/s). Specific rotations were determined using a Perkin-Elmer 241 MC Polarimeter; all rotations were measured in ethanol (EtOH) at 25°C using a 1-dm cell and the sodium D line (589 nm). ^{1}H- and ^{13}C-nuclear magnetic resonance (NMR) spectra were obtained in deuterochloroform ($CDCl_3$) or deuterium oxide (D_2O) using a Bruker AM-300 WB NMR spectrometer (operating at 300 MHz for ^{1}H and 75.5 MHz for ^{13}C); tetramethylsilane (Me_4Si) and dioxane were used as internal calibration standards (for samples in $CDCl_3$ and D_2O, respectively). ^{13}C-NMR spectra were obtained in auto-recur mode (that is, a 35° pulse was utilized with a zero relaxation delay). Infrared (IR) spectra were obtained as KBr pellets using a Perkin-Elmer 1750 Infrared Fourier Transform Spectrometer; polystyrene was uti-

lized as a calibration standard. High-resolution mass spectra (70-eV electron impact [EI]) were obtained using a Finnigan-MAT 8230 Mass Spectrometer (resolution = 7500; 5% valley). All samples were run using the solid-probe inlet.

Results and Discussion

Chemistry

Two of the six precursor 2-amino-1-phenylpropanols, *dl*-norpseudoephedrine and (1*S*, 2*S*)-norpseudoephedrine, are (currently) commercially unavailable and were synthesized via a benzylic inversion route from racemic *dl*-norephedrine and (1*R*, 2*S*)-norephedrine, respectively (Fig. 5) [*1*].

The individual enantiomers and racemates of 4-methylaminorex were synthesized via condensation of the respective 2-amino-1-phenylpropanols with cyanogen bromide [*1*]. Recrystallization (chloroform/carbon tetrachloride [$CHCl_3/CCl_4$] 1 : 1) gave the free bases as white, crystalline powders. The corresponding hydrochloride (HCl) salts were precipitated from chloroform/diethyl ether ($CHCl_3/Et_2O$) 1 : 1 solutions of the free bases with anhydrous HCl gas. Recrystallization (*i*PrOH/$CHCl_3/Et_2O$ 1 : 1 : 1) gave five of the six hydrochloride salts as white crystalline powders; the racemic *dl-trans* hydrochloride salt is hygroscopic, however, and rapidly "melted" following isolation from the reaction mixture. The resulting oil slowly crystallized over four weeks and was eventually isolated as an amorphous solid.

The free bases are readily liberated from the hydrochloride salts by solution in the minimum volume of distilled water (H_2O) followed by dropwise addition of excess saturated aqueous sodium carbonate (Na_2CO_3).

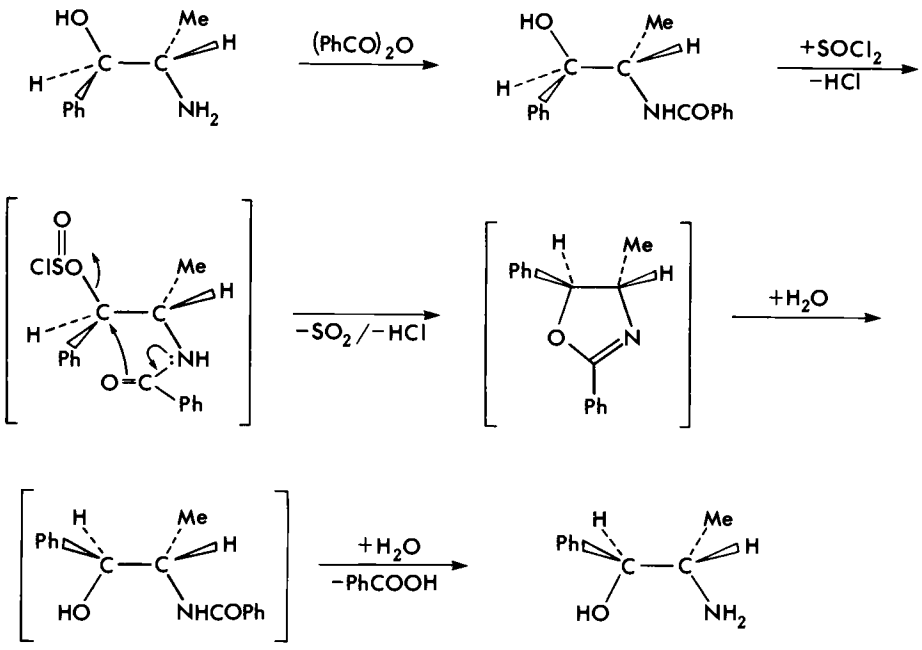

FIG. 5—*Benzylic inversion of (1R, 2S)-norephedrine to (1S, 2S)-norpseudoephedrine. Brackets indicate nonisolated intermediates.*

Melting Points

Melting points are presented in Table 1. In all cases, the melting ranges are unusually broad; that is, a gradual breakdown of crystal structure is observed over the final 15 to 20°C (sublimation evident), with complete dissolution occurring at the reported value. The close similarities between the diastereomeric *cis* and *trans* free bases ($\Delta = 3$°C) precludes differentiation via melting point except by mixed melting-point techniques [*15*].

The reported melting point for the racemic *dl-cis* free base (139 to 142°C, Table 1) does not agree with the original literature (154.5 to 156°C [*1*]). The racemic *dl-cis-*d_2 analogue was reported to be hygroscopic [*16*]; however, recrystallization from refluxing toluene with azeotropic removal of H_2O gave crystals with the same melting point (indicating that the prepared compound does not contain water of hydration). Recrystallization from boiling water, however, gave crystals with a melting point of 154 to 155°C. Chromatographic and spectroscopic evidence confirmed the structure as the hydrate. The individual *cis* enantiomers also gave hydrates with slightly higher melting points (Table 1) from boiling water; however, the *trans* isomers did not give hydrates.

Interestingly, the crude free bases (precipitated from aqueous solutions by addition of aqueous base) are the anhydrous form—only *cis* products recrystallized from boiling water form hydrates. Several illicit samples of racemic *cis*-4-methyl-aminorex analyzed in this lab all gave melting points of approximately 140°C.

Thin-Layer Chromatography

The diastereomeric *cis* and *trans* free bases are marginally resolved on silica gel and alumina and unresolved on C_{18}-reverse-phase thin-layer plates (the hydrochloride salts give multiple spots and streaking on all three stationary phases). R_f values for three different solvent systems giving optimal development (0.4 to 0.7) are presented in Table 2. The more symmetrical (less polar) *trans* isomer migrates slightly faster on both silica gel and alumina; the most favorable resolution was observed on silica gel using an ammonium hydroxide (NH_4OH)-saturated-$CHCl_3$/methanol(MeOH) 9:1 eluting solvent.

Gas Chromatography

The diastereomeric *cis* and *trans* free bases are sufficiently resolved for differentiation by capillary GC (Fig. 6). Retention times (R_t) are presented in Table 1. The more symmetrical (less polar) *trans* isomers elute prior to the *cis* isomers.

The hydrochloride salts undergo extensive thermally induced decomposition and give a more complex chromatogram; the free bases are, however, still apparent in the chromatogram (Fig. 6). Programmed injector port temperatures (from 75 to 280°C) only marginally improved performance.

Specific Rotation

The enantiomeric free bases and hydrochloride salts can be differentiated via specific rotation. Values are reported in Table 1. All compounds were determined in EtOH at 25°C using a 1-dm cell and the sodium D line (589 nm). The reported specific rotation for the *cis*-(4*S*, 5*R*) free base ($[\alpha]_D^{25} = -244.7°$, Table 1) does not agree with the original literature ($[\alpha]_D^{20} = -9.5°$ [*4*]); however, the latter value is incorrect. The reported rotations were scaled up from measurement (to three decimal places) of dilute solutions of differing concentrations—as the magnitudes of rotation for the enantiomers are internally self-consistent, that is, equal and opposite (+240.9 versus −244.7°, Table 1), the values are accurate. (The error in the original article is most likely due to measurement off-scale, that is, not −9.5 but rather

TABLE 1—*Melting points, retention times, and specific rotations.*

Product Stereochemistry	Precursor Stereochemistry	mp, °C[a]				R_t, min[b]		$[\alpha]_D^{25}$, °C[c]		
		Free Base	Hydrate	Lit.	HCl Salt	Free Base	HCl Salt[d]	Free Base	Lit.	HCl Salt
trans-(4*R*, 5*R*)	*threo*-(1*R*, 2*R*)	181–182	...	...	164–166	5.68	5.64	−6.0	...	+50.2
trans-(4*S*, 5*S*)	*threo*-(1*S*, 2*S*)[e]	182–183	...	177.5–179.5[f] 181[g]	163–166	5.62	5.62	+6.0	(+)[f] +9.7[g]	−51.8
dl-trans	*dl-threo*[e]	150–152	...	148–150[f]	(132–136)[h]	5.67	5.62	...	...	...
cis-(4*S*, 5*R*)	*erythro*-(1*R*, 2*S*)	177–179	182–184	181[g]	190–192	6.08	6.03	−244.7	−9.5[g,i]	−116.8
cis-(4*R*, 5*S*)	*erythro*-(1*S*, 2*R*)	177–180	180–183	...	188–190	6.04	6.05	+240.9	...	+114.8
dl-cis	*dl-erythro*	139–142	155–156	154.5–156[f]	178–180	6.10	6.04	...	...	...

[a]Recorded at 0.5°C/min increase. Note: Except for the *dl-cis*-free base, capillary melting points are 5 to 10°C lower.
[b]GC conditions: injector, 280°C; column, 140°C initial (held 1 min), programmed increase 4°C/min.
[c]Solvent: EtOH (all compounds). Reported values are the average of ten measurements; literature values are at 20°C. Hydrates were not measured.
[d]HCl salts undergo extensive thermally induced decomposition; see text. Minor variations in R_t versus the free bases are within experimental error.
[e]Currently commercially unavailable; see text.
[f]From Poos, et al. [*1*].
[g]From Wollweber, et al. [*4*].
[h]Reported value is of the amorphous hydrated salt; see text.
[i]Literature value is incorrect; see text.

TABLE 2—*Thin-layer chromatography.* R_f *values.*

Stationary Phase	Solvent System[a]	Free Bases	
		trans-(4*R*, 5*R*)	*cis*-(4*S*, 5*R*)
Silica gel IB-F	I	0.42	0.40
	II	0.57	0.50
Alumina IB-F	I	0.55	0.51
	II	0.66	0.64
C_{18}-reverse phase	III	0.59	0.59

[a]Solvent systems:
I EtOAc/EtOH/NH_4OH 86:10:4
II NH_4OH sat'd $CHCl_3$/MeOH 9:1
III *i*PrOH/H_2O/NH_4OH 72:24:4

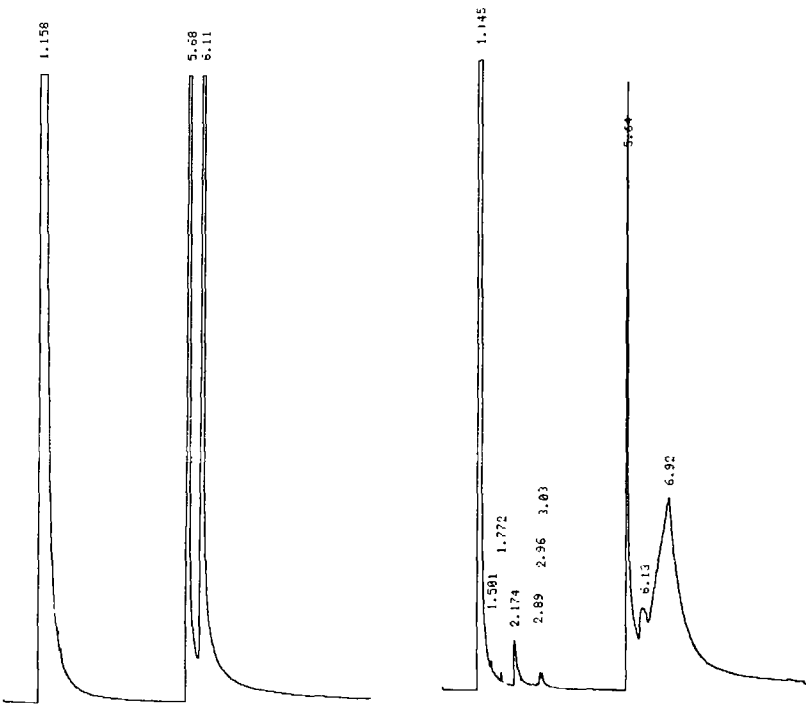

FIG. 6—Left-hand trace: *capillary GC of* cis-*(4S, 5R)- and* trans-*(4R, 5R)-4-methylaminorex 1:1 (first eluting compound is the* trans *isomer).* Right-hand trace: trans-*(4R, 5R)-4-methylaminorex hydrochloride (the* cis *isomer exhibits similar decomposition). For conditions, see Table 1.*

$-369.5°$, a reasonable explanation in view of the high concentration (1 g/mL) used for the measurement [*4*].)

NMR Spectroscopy

The ^{1}H-NMRs of the diastereomeric *cis* and *trans* free bases display significant variations in both chemical shifts (δ) and scalar coupling constants (J) (Fig. 7, Table 3). The most

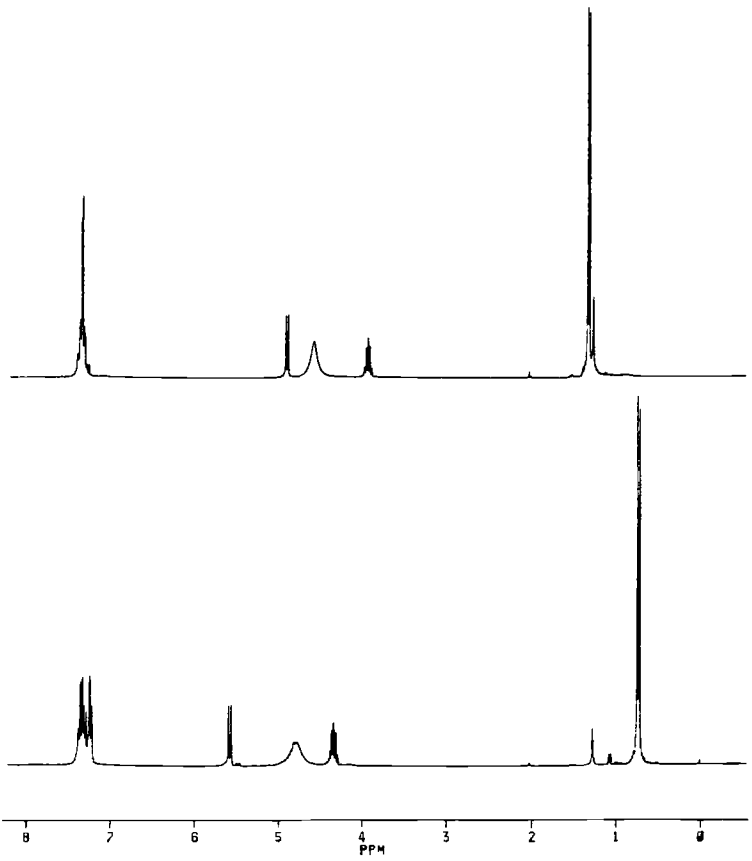

FIG. 7—*^{1}H-NMR, 16 scans.* Top trace: trans-*(4*R, 5R*)-4-methylaminorex;* bottom trace: cis-*(4*S, 5R*)-4-methylaminorex. For conditions, see Table 3.*

marked difference in chemical shift is observed with the 4-methyl substituent (δ_{cis} = 0.73 ppm versus δ_{trans} = 1.33 ppm); in the *cis* isomers, the methyl group lies within the shielding zone of the phenyl ring and is shifted 0.60 ppm upfield relative to the *trans* isomers. In addition, the aromatic ring protons display more fine structure in the *cis* isomer as a result of hindered rotation about and shielding from the methyl substituent. The largest variance in coupling constant is observed with the benzylic proton ($J_{H_5-H_4}$ (*trans*): 7.36 Hz; (*cis*): 8.80 Hz); the observed values are in agreement with other 4-alkyl-5-aryl-substituted-Δ^2-oxazolines (typical $J_{H_5-H_4}$ (*trans*): 7 to 8 Hz, (*cis*): 9 to 10 Hz [*17*]).

The ^{13}C-NMRs display analogous variations in chemical shifts (Fig. 8, Table 3); in the *cis* isomers, the methyl substituent and the aliphatic oxazoline carbons (C_4 and C_5) are all shifted upfield relative to the *trans* isomers.

The hydrochloride salts display similar behavior (Figs. 9 and 10, Table 4); the principal differences observed between the free bases and the hydrochloride salts is the general downfield shift of all protons in the hydrochloride salts and specifically the marked downfield shift and integration of the ammonium substituent ($-NH_2$ versus $-NH_3^+$).

TABLE 3—*^{1}H- and ^{13}C-NMR parameters for 4-methylaminorex.*[a]

	δ[b]		Multiplicities[c]		J[d]	
Protons[e]	4*R*, 5*R*	4*S*, 5*R*	4*R*, 5*R*	4*S*, 5*R*	4*R*, 5*R*	4*S*, 5*R*
$-CH_3$	1.33	0.73	d	d	6.41	6.82
C_4-H	3.94	4.35	dq	dq	6.51,7.25	6.82,8.75
$-NH_2$[f]	4.83	5.03	s, br	s, br	...	...
C_5-H	4.91	5.59	d	d	7.36	8.80
Aryl−H	7.27-7.41	7.23-7.39	m	m	...	...
Carbons[g]	δ[b]					
$-CH_3$	21.56	18.46				
C_4	68.38	62.89				
C_5	88.95	84.49				
$C_{3'}$,$C_{5'}$	125.76	126.03				
$C_{4'}$	128.27	127.78				
$C_{2'}$,$C_{6'}$	128.71	128.22				
$C_{1'}$	140.02	137.20				
C_2	159.59	160.14				

[a]In $CDCl_3$, at 300 MHz for ^{1}H, 75.5 HMz for ^{13}C.
[b]In ppm, relative to Me_4Si.
[c]s = singlet, d = doublet, q = quartet, m = multiplet.
[d]In Hertz.
[e]Refer to Fig. 7.
[f]δ varies with concentration.
[g]Refer to Fig. 8.

IR Spectroscopy

The diastereomeric *cis* and *trans* free bases display similar but distinct IR spectra (Fig. 11). The principal absorbtions include the amine stretch (ν_{N-H} = 3450 cm^{-1}), which surprisingly is a singlet, the imine stretch ($\nu_{C=N}$ = 1700 cm^{-1}), quite intense and shifted to higher than usual frequency by the amine substituent, and the mono-substituted phenyl ring stretching bands (ν_{C-C} = 1500, 1600 cm^{-1}). Similar results are obtained for the hydrochloride salts (Fig. 12); the principal differences are the broadening of the amine stretch and the higher frequency of the imine stretch.

Mass Spectroscopy

The diastereomeric *cis* and *trans* free bases display virtually identical mass spectra under 70-eV EI conditions (Fig. 13, Table 5). The first two fragment ions (*m/z* = 161.0715 and 132.0813) correspond to loss of methyl and carbonylamine, respectively. The base ion (*m/z* = 70.0531) corresponds to loss of benzaldehyde. Other ions include a C_7H_7 ion (*m/z* = 91.0548, possibly tropylium), a C_6H_5 ion (*m/z* = 77.0391, probably phenyl), and a C_2H_5N ion (*m/z* = 43.0422, possibly aziridine).

Somewhat unusually, the total ion current due to $M^{+\cdot}$ (a measure of the relative stability of the parent molecules [*18*] is greater for the *cis* isomer (the thermodynamically *less* stable isomer). This implies an alternate, more accessible fragmentation pathway for $M^{+\cdot}$ in the case of the *trans* isomer; the $M^{+\cdot}$/total ion current ratios (Table 5) may thus be used (with caution) to differentiate diastereomers.

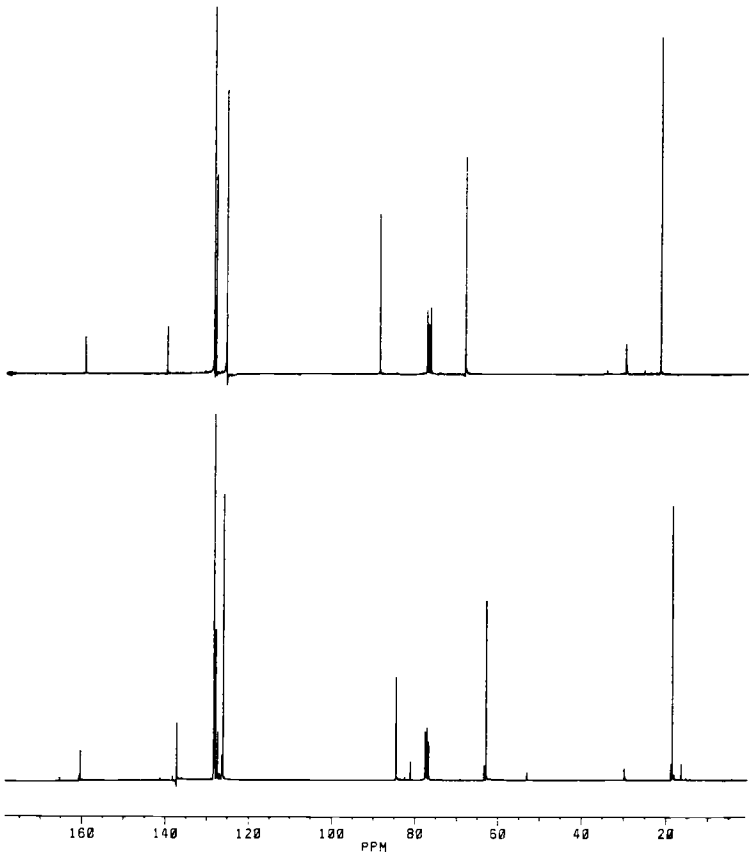

FIG. 8—*^{13}C-NMR, 20 000 scans (auto-recur).* Top trace: trans-*(4*R, *5*R*)-4-methylaminorex;* bottom trace: cis-*(4*S, *5*R*)-4-methylaminorex. For conditions, see Table 3.*

Color Test

The addition of one drop of 2% aqueous cobalt (II) thiocyanate to 1 mg of the free base or hydrochloride salt dissolved in 0.5 mL of EtOH gives an instantaneous deep blue color; the color is slightly less intense for the hydrochloride salt. The reagent is nonspecific with respect to stereochemistry.

No color change is observed upon addition of the reagent to an aqueous solution of the hydrochloride salt(s); however, subsequent addition of one drop of saturated aqueous Na_2CO_3 instantly precipitates a blue solid.

Microcrystalline Tests

Microcrystalline tests were performed on the hydrochloride salts with 5% aqueous platinous chloride ($PtCl_2$) and 5% aqueous gold trichloride ($AuCl_3$). Results are reported in Table 6. In all tests with $PtCl_2$, the addition of one drop of the reagent to one drop of an aqueous solution of the hydrochloride salt (2 mg/mL) gives microcrystals within 30s. Similar

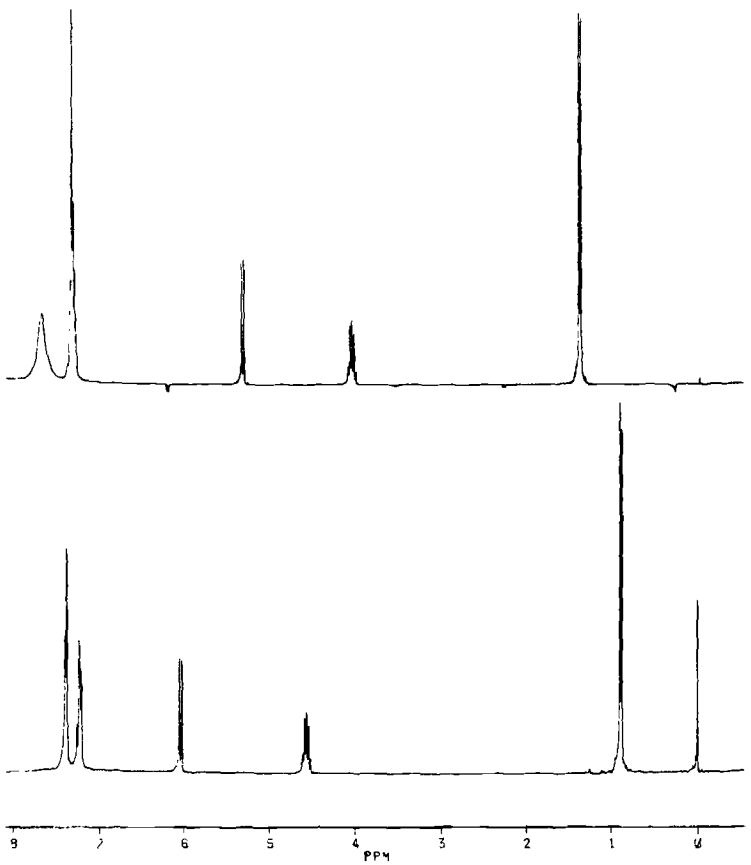

FIG. 9—*^{1}H-NMR, 16 scans.* Top trace: trans-*(4R, 5R)-4-methylaminorex hydrochloride;* bottom trace: cis-*(4S, 5R)-4-methylaminorex hydrochloride (*$-NH_3^+$ *off-scale at* $\delta = 9.12$ *ppm). For conditions, see Table 4.*

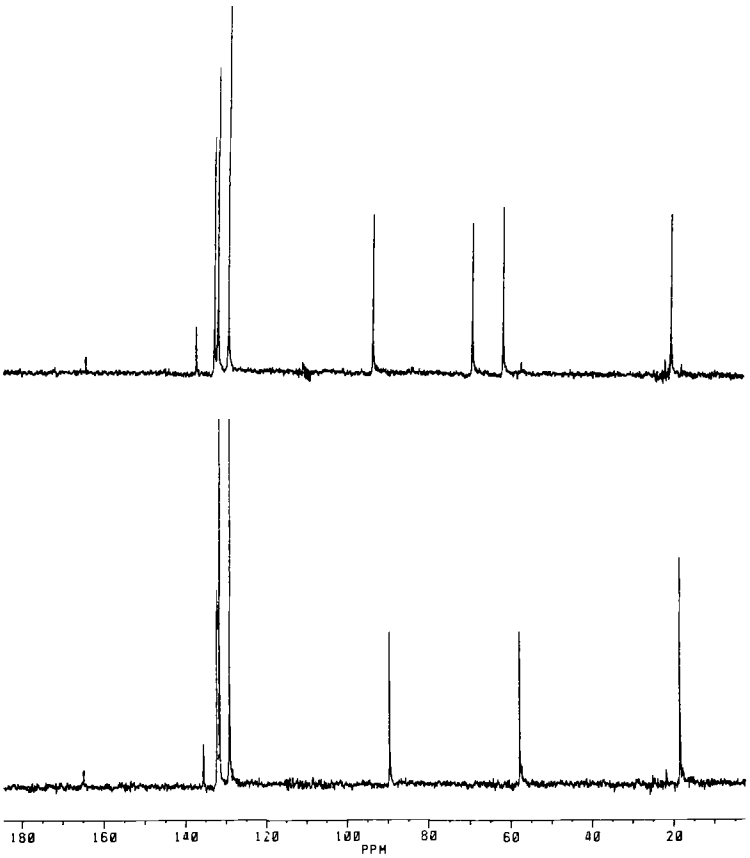

FIG. 10—*^{13}C-NMR, 20 000 scans (auto-recur).* Top trace: trans-*(4*R, 5R*)-4-methylaminorex hydrochloride (additional peak at δ = 66.4 ppm is internal dioxane standard);* bottom trace: cis-*(4*S, 5R*)-4-methylaminorex hydrochloride. For conditions, see Table 4.*

TABLE 4—*^{1}H- and ^{13}C-NMR parameters for 4-methylaminorex-hydrochloride.*[a]

	δ[b]		Multiplicities[c]		J[d]	
Protons[e]	4*R*, 5*R*	4*S*, 5*R*	4*R*, 5*R*	4*S*, 5*R*	4*R*, 5*R*	4*S*, 5*R*
			Protons			
$-CH_3$	1.47	0.89	d	d	6.13	6.65
C_4-H	4.16	4.57	dq	dq	6.32,8.12	6.72,8.48
$-NH_3^+$[f]	7.67	9.12	s	s	...	...
C_5-H	5.36	6.05	d	d	8.17	8.53
Aryl−H	7.35-7.43	7.22-7.43	m	m	...	...
Carbons[g]	δ[h]					
$-CH_3$	17.57	15.63				
C_4	58.84	54.85				
C_5	90.69	86.67				
$C_{3'}$,$C_{5'}$	126.60	126.21				
$C_{2'}$,$C_{6'}$	129.13	128.66				
$C_{4'}$	130.03	129.24				
$C_{1'}$	134.49	132.60				
C_2	162.10	162.35				

[a] ^{1}H in $CDCl_3$, at 300 MHz, ^{13}C in D_2O, at 75.5 MHz.
[b] In ppm, relative to Me_4Si.
[c] s = singlet, d = doublet, q = quartet, m = multiplet.
[d] In Hertz.
[e] Refer to Fig. 9.
[f] δ varies with concentration.
[g] Refer to Fig. 10.
[h] In ppm, relative to dioxane ($\delta_{dioxane}$ = 66.42 ppm).

results are obtained from the free bases by dissolving them in the minimum volume of 5% HCl. Different microcrystalline precipitates are observed for each of the racemates and enantiomeric pairs (Table 6). In the case of $AuCl_3$, only the racemic *dl-cis* hydrochloride salt gives a microcrystalline precipitate; thus, $AuCl_3$ is a specific reagent for the racemic *dl-cis* hydrochloride salt.

It must be emphasized that microcrystal tests are often sensitive to concentration, pH, impurities, and technique; thus, positive identification should be attempted only via direct comparison with the known standards [*19,20*].

Acknowledgment

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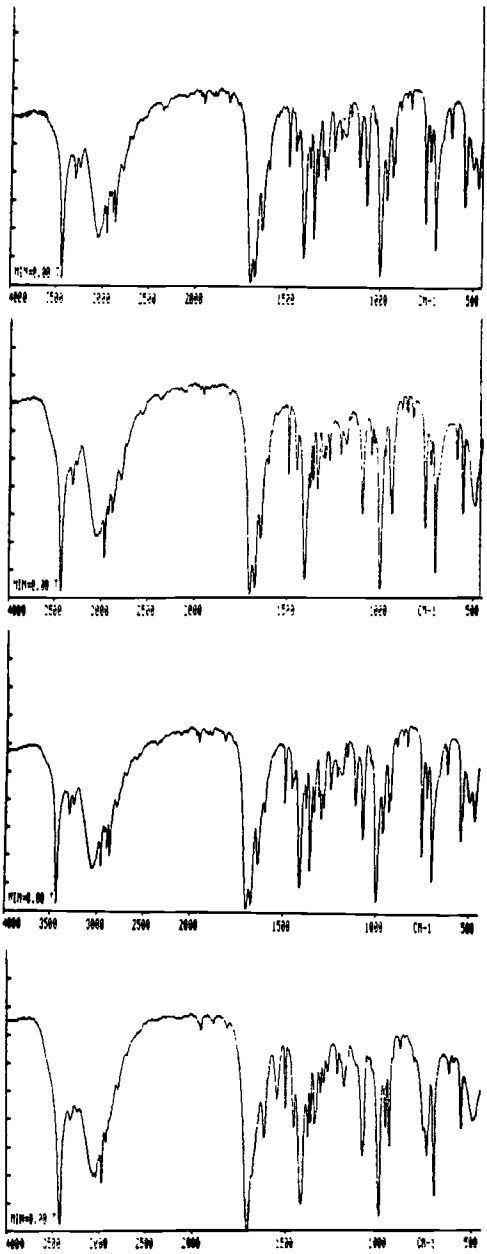

FIG. 11—*IR*. Top trace: trans-*(4*R, *5R)-4-methylaminorex;* second trace: cis-*(4*S, *5*R*)-4-methylaminorex;* third trace: *racemic*-dl-trans-*4-methylaminorex;* bottom trace: *racemic*-dl-cis-*4-methylaminorex.*

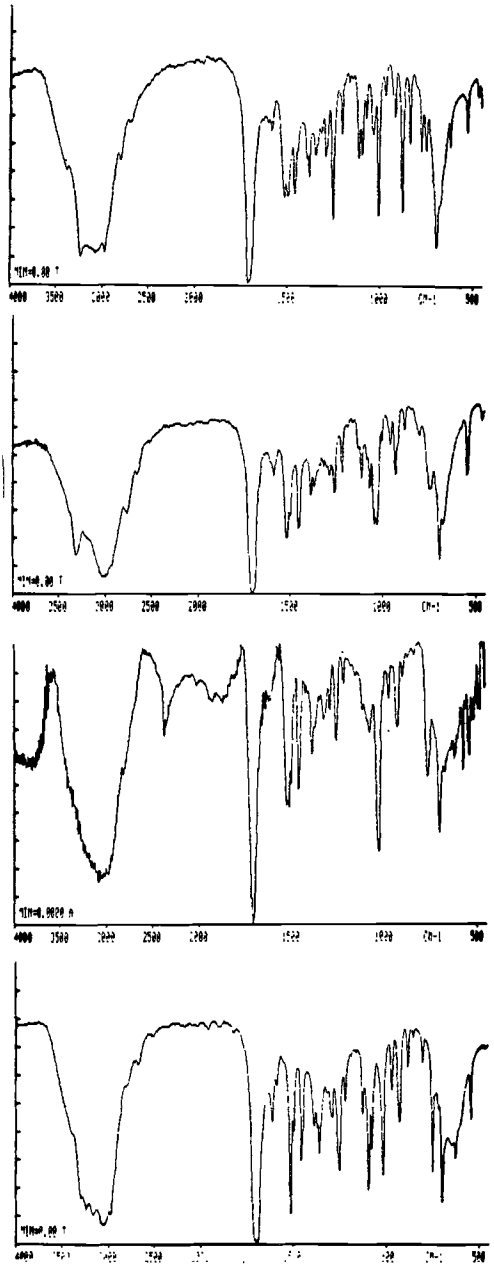

FIG. 12—*IR.* Top trace: trans-*(4R, 5R)-4-methylaminorex hydrochloride;* second trace: cis-*(4S, 5R)-4-methylaminorex hydrochloride;* third trace: *racemic*-dl-trans-*4-methylaminorex hydrochloride;* bottom trace: *racemic*-dl-cis-*4-methylaminorex hydrochloride. (Note: the* trans *enantiomers are polymorphic and may alternately closely resemble the* trans *racemate.)*

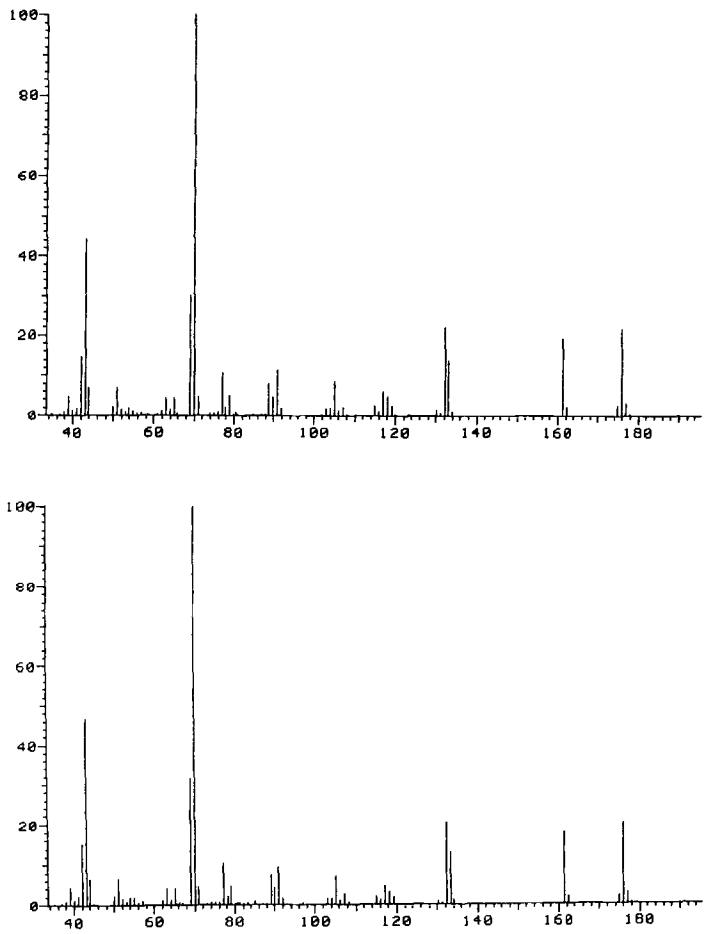

FIG. 13—*70-eV EI-MS.* Top trace: trans-*(4*R, *5*R*)-4-methylaminorex;* bottom trace: cis-*(4*S, *5*R*)-4-methylaminorex.*

TABLE 5—*High resolution 70-eV EI mass spectral parameters for 4-methylaminorex.*[a]

Empirical Composition	Theoretical	*trans*-(4*R*, 5*R*) Found[b]	*trans*-(4*R*, 5*R*) Relative Abundance	*cis*-(4*S*, 5*R*) Found[b]	*cis*-(4*S*, 5*R*) Relative Abundance
$C_1H_3N_2$	43.0296	43.0260	1750	43.0302	1700
C_2H_5N	43.0422	43.0385	3550	43.0428	2975
$C_3H_5N_2$	69.0453	69.0450	3450	69.0459	3170
$C_3H_6N_2$	70.0531	70.0530	10000	70.0539	10000
C_6H_5	77.0391	77.0328	1250	77.0396	1055
C_7H_5	89.0391	89.0437	910	89.0408	740
C_7H_7	91.0548	91.0572	1290	91.0546	960
C_8H_9	105.0704	105.0713	940	105.0710	740
C_9H_7	115.0548	115.0577	300	115.0559	220
C_8H_7N	117.0579	117.0582	640	117.0590	340
$C_9H_{10}N$	132.0813	132.0807	2240	132.0802	2090
$C_9H_{11}N$	133.0891	133.0888	1350	133.0876	1300
$C_9H_9N_2O$	161.0715	161.0710	1860	161.0704	1790
$C_{10}H_{12}N_2O$	176.0950	176.0947	1960	176.0944	2040
$M^{+\cdot}$/total ion current (×100):		3.43		4.81	

[a] Refer to Fig. 13.
[b] Reported values are the average of nine measurements.

TABLE 6—*Microcrystalline tests.*[a]

HCl Salts	$PtCl_2^b$	Birefringence	$AuCl_3^b$	Birefringence
trans-(4*R*, 5*R*)	(+)[c]	strong	(−)	...
dl-trans[d]	(+)[e]	strong	(−)	...
cis-(4*S*, 5*R*)	(+)[f]	moderate	(−)	...
dl-cis	(+)[g]	strong	(+)[h]	weak

[a] All crystals viewed at ×120 magnification.
[b] Five percent aqueous solution.
[c] Elongated prisms, some with slanted ends.
[d] Solution prepared by dissolving the free base in 10% HCl.
[e] Long, thin needles, some in feather-combs.
[f] Long, thin irregular rods, no aggregation.
[g] Rods in rosettes, sheaves, and bundles.
[h] Large irregular plates and blades.

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Address requests for reprints or additional information to
Robert F. X. Klein
Drug Enforcement Administration
Special Testing and Research Laboratories
7704 Old Springhouse Rd.
McLean, VA 22102-3494