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Optically Active Amines. Part XIII.[1] Circular Dichroism of the *N*-Salicylidene Derivatives of Alkylamines

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Comparison of the c.d. spectra of (*S*)-(+)-*N*-salicylidene-α-phenylethylamine with those of (*S*)-(+)-*N*-salicylidene-sec-butylamine and (*R*)-(−)-*N*-salicylidene-2,2-dimethyl-3-aminobutane confirms the conclusion that the strong rotational strength shown by *N*-salicylidene-α- and β-phenylalkylamines is due to an interaction of the π-electron system of the phenyl group and the salicylidenimino chromophore. The sign of the Cotton effects near 255 and 315 nm shown by the *N*-salicylidenes of alkylamines and cycloalkylamines, when these are conformationally defined, can be correlated with their absolute configurations using a planar sector rule, similar to that used for the interpretation of the c.d. and o.r.d. spectra of *N*-salicylidene-α- and β-phenylalkylamines.

La comparaison des spectres de d.c. du *S*-(+)-*N*-salicylidène α-phényléthylamine avec ceux du *S*-(+)-*N*-salicylidène sec-butylamine et R-(−)-*N*-salicylidène diméthyl-2,2 amino-3 butane confirme le fait que l'importance de la force de rotation qui se manifeste dans les *N*-salicylidène α et β-phénylalkylamines, est due à une interaction des électrons π du groupe phényle avec le chromophore salicylidenimino. Les signes des effets Cotton entre 255 et 315 nm dans les *N*-salicylidènes d'alkylamines, dans une conformation définie, peuvent être reliés aux configurations absolues en utilisant le même genre de règle que celle utilisée dans l'interprétation des spectres de d.c. et d.o.r. des *N*-salicylidène α et β-phénylalkylamines.

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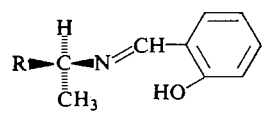
In an earlier paper (2), the optical rotatory dispersion (o.r.d.) curves of the *N*-salicylidenes [(*S*)-**1***b* and (*S*)-**2***b*] of (*S*)-(−)-α-phenylethylamine[3] [(*S*)-**1***a*] and (*S*)-(+)-α-benzylethylamine [(*S*)-**2***a*] were compared with that of the *N*-salicylidene [(*S*)-**3***b*] of (*S*)-(+)-sec-butylamine [(*S*)-**3***a*]. Both (*S*)-**1***b* and (*S*)-**2***b* in ethanol show



(*S*)-**1***b* R = C_6H_5
(*S*)-**2***b* R = $C_6H_5CH_2$
(*S*)-**3***b* R = C_2H_5

positive Cotton effects near 315 and 410 nm. In contrast, (*S*)-**3***b* is much weaker in rotatory power, and in ethanol only one weak peak at 437 nm could be observed. It was concluded that the strong rotational strength shown by *N*-salicylidene-α- and β-phenylalkylamines is due to an interaction of the π-electron system of the

[1]Part XII. H. E. Smith and A. A. Hicks (1).
[2]National Science Foundation Undergraduate Summer Fellow, 1969.
[3]Signs in parentheses refer to rotatory powers observed with sodium D-light for the amines as the neat liquid and for the *N*-salicylidenes as solutions in methanol.

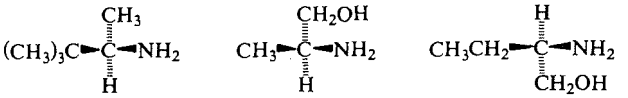
TABLE 1. Spectral data for the *N*-salicylidene derivatives of some open-chain amines

Compound	Methanol		*n*-Hexane	
	The e.a. maxima* λ, nm (log ε)	The c.d. maxima† λ, nm ([θ])‡	The e.a. maxima* λ, nm (log ε)	The c.d. maxima† λ, nm ([θ])‡
(*S*)-**1***b*	404 (2.78)§‖	405 (+1100)§¶		
	315 (3.61)	315 (+18000)	320 (3.70)§	319 (+40000)§**
	283 (3.35)††			
		274 (−3000)		274 (−4000)
			262 (4.13)††	
	256 (4.14)	253 (+34000)	255 (4.18)	253 (+58000)
(*S*)-**3***b*	400 (3.22)	395 (+630)		
	314 (3.52)	313 (+1600)	318 (3.68)	315 (+2200)
	277 (3.60)			
	258 (4.00)††	253 (+4900)	260 (4.06)	255 (+3000)
	252 (4.04)		254 (4.09)	
(*R*)-**4***b*	399 (3.26)	400 (−1100)		
	314 (3.53)	315 (−2500)	317 (3.70)	314 (−2600)
	276 (3.64)			
	258 (4.02)††	261 (−6200)	261 (4.09)	261 (−6700)
	253 (4.05)		254 (4.14)	
(*S*)-**5***b*	400 (3.10)	396 (+710)	400 (1.25)††‡‡	420 (−21)§§
	314 (3.56)	312 (+2700)	317 (3.66)	315 (+1800)
	275 (3.51)††			
		269 (−1900)		267 (−3800)
	257 (4.02)††		259 (4.06)††	
	254 (4.05)	248 (+6300)	255 (4.11)	251 (+8300)
(*R*)-**6***b*	400 (3.10)	388 (−160)	404 (1.47)‖‖	415 (+120)¶¶
	314 (3.58)	315 (−450)	319 (3.67)	316 (+1100)
	277 (3.54)††			
		267 (+6000)		267 (+6500)
	258 (4.04)††		260 (3.06)††	
	254 (4.08)	248 (−2300)	256 (4.11)	248 (−2900)

*For data not previously reported, 4.0 × 10^{-5} to 9.9 × 10^{-4} mol/l except for those maxima noted otherwise.
†For data not previously reported, temperature 25–28°; *c* 1.6 × 10^{-5} to 4.3 × 10^{-2} g/100 ml except for those maxima noted otherwise.
‡Molecular ellipticity.
§Data from ref. 4.
‖95% Ethanol as the solvent for this spectrum.
¶Absolute ethanol as the solvent for this spectrum.
**Isooctane as the solvent for this spectrum.
††Shoulder.
‡‡1.15 × 10^{-2} mol/l.
§§*c* 0.206 g/100 ml.
‖‖1.69 × 10^{-2} mol/l.
¶¶*c* 0.655 g/100 ml.

phenyl group and the salicylidenimino chromophore, the chirality of which gives the sign to the observed Cotton effects. This conclusion has been confirmed by subsequent o.r.d. and circular dichroism (c.d.) measurements with a substantial number of *N*-salicylidenes of α- and β-phenylalkylamines (3–5) and comparison of these measurements with similar ones made with the *N*-salicylidenes of steroidal amines (6–8) and of amino sugars (9). However, direct comparison of c.d. measurements with open-chain α- and β-phenylalkylamine derivatives with those of open-chain alkylamine derivatives has been lacking.

We now report the comparison of the electronic absorption (e.a.) and c.d. spectra of (*S*)-**1***b* with those of (*S*)-**3***b* as well as the *N*-salicylidene [(*R*)-**4***b*] of (*R*)-(−)-2,2-dimethyl-3-aminobutane (10) [(*R*)-(−)-pinacolylamine] [(*R*)-**4***a*]. (*S*)-**4***a* has been prepared earlier with a low rotatory power from (*R*)-tert-leucine (10). The complete resolution of (±)-**4***a* is now reported. In addition, the e.a. and c.d. spectra for the *N*-salicylidene [(*S*)-**5***b* and (*R*)-**6***b*] of (*S*)-(+)-2-amino-1-propanol (11) [(*S*)-**5***a*] and (*R*)-(−)-2-amino-1-butanol (12) [(*R*)-**6***a*] are also reported. The c.d. measurements with these latter two *N*-salicylidenes are pertinent to the interpretation of the c.d. spectra of salicylidenimino sugars (9).

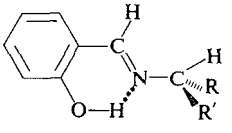


(*R*)-**4***a* (*S*)-**5***a* (*R*)-**6***a*

Results and Discussion

As seen in Table 1, the e.a. spectra of (*S*)-(+)-*N*-salicylidene-α-phenylethylamine [(*S*)-**1***b*], (*S*)-(+)-*N*-salicylidene-sec-butylamine [(*S*)-**3***b*], and (*R*)-(−)-*N*-salicylidene-2,2-dimethyl-3-aminobutane [(*R*)-**4***b*] in a particular solvent are the same. The absorption bands near 255 and 315 nm are assigned to electronic transitions of the intramolecularly hydrogen-bonded salicylidenimino group. The absorption band near 400 nm has been assigned, for the alkylamine *N*-salicylidenes in ethanol, to hydrogen-bonded complexes with the solvent (13) or to a tautomeric form (14). At high concentration, however, an absorption band near 400 nm is observed in the spectrum of *N*-salicylideneisopropylamine in carbon tetrachloride (13). This band disappears on dilution, and it is attributed to a small concentration of a dimeric species (13).

In the c.d. spectrum of (*S*)-**1***b*, the sign of the Cotton effects near 255 and 315 nm is correlated with the absolute configuration of (*S*)-**1***b* using a planar sector rule (5). Thus, in the preferred conformation of (*S*)-**1***b* (**7***a*), the phenyl group is behind the plane of the salicylidenimino chromophore, and in the spectrum, the two Cotton effects are positive. The c.d. maximum at 274 nm, not seen in the spectra of *N*-salicylidene-β-phenylalkylamines (3–5), has been assigned to a $\pi \rightarrow \pi^*$ transition of the phenyl ring of the amine moiety (4).



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a R = C_6H_5; R′ = CH_3
b R = C_2H_5; R′ = CH_3
c R = CH_3; R′ = $C(CH_3)_3$
d R = CH_2OH; R′ = CH_3

For (*S*)-(+)-*N*-salicylidene-sec-butylamine [(*S*)-**3***b*] and (*R*)-(−)-*N*-salicylidene-2,2-dimethyl-3-aminobutane [(*R*)-**4***b*] corresponding c.d. maxima near 255 and 315 nm are observed. The intensities of these maxima are reduced since the interaction of a phenyl group with the salicylidenimino chromophore is no longer present. Both (*S*)-**3***b* and (*R*)-**4***b* have a preferred conformation (**7***b* and *c*) similar to that of (*S*)-**1***b*. As has been shown for alkyl-substituted cyclohexanones, the rotatory perturbation of the carbonyl chromophore by a tert-butyl group is larger than that by a methyl group (15). The rotatory perturbation of the salicylidenimino chromophore by an ethyl group may also be taken as larger than that by a methyl group. Using the same sector rule, (*S*)-**3***b* and (*R*)-**4***b* are predicted to show Cotton effects near 255 and 315 nm with signs as shown in Table 1.

Assuming a similar preferred conformation for the salicylidenimino group about its attachment bond and this sector rule, the sign of the Cotton effect near 315 nm shown by the *N*-salicylidenes of a number of 20-aminopregnanes can be predicted (6, 7). Much the same is true for the *N*-salicylidenes of cyclic amines, especially 17α-, 17β-, and 3α-amino steroids (8). For other cyclic amines, including other amino steroids, contributions from other salicylidenimino conformations may be important and perturbations by several remote groups may be significant (8). In these latter cases, predictions are sometimes unreliable.

The e.a. spectra of (*S*)-(+)-*N*-salicylidene-2-amino-1-propanol [(*S*)-**5***b*] and (*R*)-(+)-*N*-salicylidene-2-amino-1-butanol [(*R*)-**6***b*] in methanol are essentially the same as those of the others. In hexane, however, a weak absorption band near 400 nm is detected in each spectrum. The molecular absorptivity of the band decreases on dilution. The band may be due to a small concentration of the intermolecularly hydrogen-bonded dimer (13) in *n*-hexane.

The c.d. spectra of (*S*)-**5***b* and (*R*)-**6***b* also show an additional maximum near 268 nm. A maximum near this same wavelength is frequently present in the c.d. spectra of the *N*-salicylidene derivatives of α-hydroxy steroidal amines (8).

The rotatory perturbation of the salicylidenimino chromophore by a hydroxymethylene group is taken as greater than that by a methyl group. Application of the sector rule to a conformation of (*S*)-**5***b* (**7***d*) similar to the preferred conformation of (*R*)-**4***b* (**7***c*), predicts, as are observed, positive maxima near 255 and 315 nm

in the c.d. spectrum of (*S*)-**5***b*. The rotatory perturbation by a hydroxymethylene group is taken to be about the same as that by an ethyl group, and unusually low molecular ellipticities are found for the maxima near 255 and 315 nm in the c.d. spectrum of (*R*)-**6***b*. Also the sign of the maximum near 315 nm is different in the two solvents used.

Experimental

General

Boiling points are not corrected. The *N*-salicylidenes were prepared as previously reported (2). They had n.m.r. spectra compatible with their assigned structures. Optical rotations at the sodium D-line were obtained using a 1-dm sample tube. Elemental analyses were done by Galbraith Laboratories, Inc., Knoxville, Tennessee. The e.a. spectra were measured with a Cary Model 14 spectrophotometer using 1-cm sample cells. The c.d. spectra were measured at 25–28° with a Cary Model 60 spectropolarimeter with a Model 6001 accessory. The slit was programmed for a spectral band width of 1.5 nm, and a path length of 1 cm was used. Cut-off was indicated when the dynode voltage reached 400 V.

(S)-(+)-N-Salicylidene-sec-butylamine [(S)-3b][4]

This compound had $[\alpha]_D^{25}$ +73° (*c* 1.15, MeOH).

Resolution of (±)-2,2-Dimethyl-3-aminobutane [(±)-4a]

To 113.0 g (0.753 mol) of (+)-tartaric acid in 1000 ml of boiling 95% EtOH was added 75.0 g (0.741 mol) of (±)-**4***a*. On cooling the mixture to room temperature, there was deposited 141.2 g (152%) of solid, $[\alpha]_D^{23}$ +16° (*c* 2.78, H_2O). Four recrystallizations of this solid from 95% EtOH gave 59.1 g (63%) of the acid tartrate salt of (*R*)-**4***a*, $[\alpha]_D^{26}$ +16° (*c* 2.64, H_2O). Fifty grams of this salt was decomposed with 25% KOH, and the amine extracted into ether. The ethereal solution was dried (KOH), and the ether evaporated. Distillation of the residue gave 15.5 g (49%) of (*R*)-**4***a*, b.p. 100–103°, d_4^{20} 0.758, $[\alpha]_D^{23}$ −5.6° (neat), $[\alpha]_D^{25}$ −2.6° (*c* 5.10, MeOH) (lit. (10) $[\alpha]_D^{24}$ +0.70° (neat, *l* = 0.2 dm) for (*S*)-**4***a*).

The mother liquors from the above resolution were combined, and the solvent was removed. Decomposition of the residual salt gave 36.6 g (0.362 mol) of the partially racemic amine, $[\alpha]_D^{23}$ +2.02° (neat), which was combined with 54.4 g (0.362 mol) of (−)-tartaric acid in 500 ml of boiling 95% EtOH. On cooling this solution to room temperature, there was deposited 54.4 g (58%) of solid, $[\alpha]_D^{26}$ −16° (*c* 5.70, H_2O). After three recrystallizations of this solid from 95% EtOH, there was obtained 10.5 g (11%) of the acid tartrate salt of (*S*)-**4***a*, $[\alpha]_D^{25}$ −16° (*c* 4.69, H_2O).

The salt was decomposed and the amine purified as before. There was thus obtained 2.40 g (6.4%) of (*S*)-**4***a*, b.p. 100–103°, d_4^{20} 0.760, $[\alpha]_D^{24}$ +5.4° (neat), $[\alpha]_D^{24}$ +2.5° (*c* 5.10, MeOH).

[4]The preparation and characterization of this sample were reported earlier (2).

(±)-N-Salicylidene-2,2-dimethyl-3-aminobutane [(±)-4b]

From 5.06 g (50.0 mmol) of (±)-2,2-dimethyl-3-aminobutane [(±)-**4***a*] and 5.50 g (45.0 mmol) of salicylaldehyde in 15 ml of MeOH there was obtained 3.03 g (33%) of (±)-**4***b*, yellow oil, b.p. 82–84° (0.1 mm), n_D^{25} 1.5323, d_4^{20} 0.965.

Anal. Calcd. for $C_{13}H_{19}NO$: C, 76.05; H, 9.33; N, 6.82. Found: C, 76.21; H, 9.39; N, 7.05.

(R)-(−)-N-Salicylidene-2,2-dimethyl-3-aminobutane [(R)-4b]

From 4.10 g (40.5 mmol) of (*R*)-(−)-2,2-dimethyl-3-aminobutane [(*R*)-**4***a*] and 4.45 g (36.4 mmol) of salicylaldehyde in 50 ml of benzene there was obtained 3.54 g (47%) of (*R*)-**4***b*, yellow oil, b.p. 89–90° (0.2 mm), which on standing crystallized as yellow needles, m.p. *ca.* 30°, $[\alpha]_D^{24}$ −133° (*c* 2.04, MeOH).

Anal. Calcd. for $C_{13}H_{19}NO$: C, 76.05; H, 9.33; N, 6.82. Found: C, 76.21; H, 9.30; N, 7.04.

(S)-(+)-N-Salicylidene-2-amino-1-propanol [(S)-5b]

From 1.90 g (25.3 mmol) of (*S*)-(+)-2-amino-1-propanol [(*S*)-**5***a*], d_4^{20} 0.955, $[\alpha]_D^{24}$ +15.1° (neat), $[\alpha]_D^{24}$ +16° (*c* 4.99, MeOH) (lit. (16) $[\alpha]_D^{20}$ +15.8° (neat)), and 3.20 g (26.2 mmol) of salicylaldehyde in 10 ml of MeOH, there was obtained 3.67 g (81%) of (*S*)-**5***b*, yellow oil, b.p. 114–115° (0.3 mm), n_D^{25} 1.5815, $[\alpha]_D^{24}$ +76.2° (*c* 2.23, MeOH).

Anal. Calcd. for $C_{10}H_{13}NO_2$: C, 67.02; H, 7.31; N, 7.82. Found: C, 66.86; H, 7.15; N, 7.80.

(R)-(+)-N-Salicylidene-2-amino-1-butanol [(R)-6b]

From 4.68 g (52.5 mmol) of (*R*)-(−)-2-amino-1-butanol [(*R*)-**6***a*], d_4^{20} 0.936, $[\alpha]_D^{25}$ −10.2° (neat), $[\alpha]_D^{24}$ −10° (*c* 5.00, MeOH) (lit. (17, 18) $[\alpha]_D^{26}$ −9.92° (neat), $[\alpha]_D^{22}$ −10.00° (*c* 2, EtOH)), and 6.70 g (54.9 mmol) of salicylaldehyde in 20 ml of MeOH, there was obtained 7.15 g (70%) of (*R*)-**6***b*, recrystallized from *n*-hexane, yellow needles, m.p. 55–56° (corrected), $[\alpha]_D^{24}$ +8.1° (*c* 3.50, MeOH).

Anal. Calcd. for $C_{11}H_{15}NO_2$: C, 68.37; H, 7.82; N, 7.25. Found: C, 68.53; H, 7.67; N, 7.25.

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A Differential Scanning Calorimeter Study of Phase Transitions in Alkali Metal Stearates

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Differential scanning calorimeter thermograms have been obtained for the alkali metal stearates from 25 to 200 °C. Four different samples of different thermal history were studied for each soap, and differences in temperature and relative enthalpy of transitions were found to depend on the various sample histories. For lithium stearate good correlation is found between DSC and nuclear magnetic resonance (n.m.r.) results. Rubidium and caesium stearates seem to be less sensitive to thermal history than the sodium and potassium salts.

Des thermogrammes par calorimétrie différentielle de précision ont été obtenus pour des stéarates de métaux alcalins de 25 à 200 °C. Quatre échantillons différents avec une histoire thermique différente ont été étudiés pour chaque savon et les différences trouvées dans la température et l'enthalpie relative de transitions dépendent des différentes histoires. Pour le stéarate de lithium, il existe une bonne corrélation entre les résultats en DSC et en r.m.n. Les stéarates de rubidium et de caesium paraissent moins sensibles à l'histoire thermique que les sels de sodium et potassium.

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In the course of a broad line nuclear magnetic resonance study of alkali metal oleates and stearates (1), we examined a number of these soaps by the differential scanning calorimeter technique, using a Perkin–Elmer DSC 1-B instrument. In particular, DSC curves were run on different samples of the same substance of which the various samples had different thermal histories. As might be expected from previous work (2, 3), the thermal history had significant influence on the thermogram obtained for the sample. The temperatures of the various phase transitions as observed from the thermograms are also compared with transition temperatures observed by other techniques.

It may be worth noting at this point that in differential thermal analysis (DTA) the difference in temperature between the sample and a standard reference substance is measured, whereas in the differential scanning calorimeter (DSC) the difference in the energy required to keep sample and reference at the same temperature under constant rate of heating is recorded. In the DTA method, peaks are normally observed for both first and second order transitions, but in the DSC method peaks should accompany only first order transitions; the second order transitions should cause only a shift in the baseline of the thermogram. In this report only distinct peaks in the DSC thermograms are considered, so that all transitions listed for the DSC method should be first order.

Samples of alkali metal stearates were prepared from Eastman Kodak white label grade stearic acid and reagent grade alkali metal hydroxide by precipitation from hot ethanol and aqueous

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