

Conformations of Unsaturated and Aromatic Alcohols; Intramolecular Hydrogen Bonding in 2-Phenylethanol

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The conformational composition of 2-phenylethanol has been investigated by ¹H NMR and IR spectroscopy of solutions at low concentration in CCl₄ and CFCl₃. The relative energies of the stable conformations have been estimated by molecular mechanics calculations (MMPMI in the dipole–dipole electrostatic mode and with charge–charge interaction) and by semi-empirical (MNDO) and *ab initio* (STO-3G and 4-31G) molecular orbital calculations. Comparison of the results from the calculations with the experimental data shows the MMPMI with charge–charge interaction to represent the best model for the conformational composition. The major conformation of 2-phenylethanol (ca. 45 %) has a *gauche* relationship both around the CH₂CH₂ and the CH₂OH bonds, with the hydroxy proton pointing towards the phenyl ring (conformation C). This conformation is stabilised to the extent of ca. 1.2 kcal mol⁻¹ over the other ones. By comparison with the IR spectra in the OH region and the energetics of other unsaturated alcohols, the conformational stability of C is found to be caused by an intramolecular hydrogen bond between the hydroxy group and the π -electrons of the phenyl ring.

Almost thirty years ago, a series of 2-arylethanols was studied by IR spectroscopy and it was concluded that intramolecular hydrogen bonding to the π -electron system was important in these molecules.¹ This contrasted with earlier reports.² Subsequently, results of several investigations on this system have been published. Studying alcohols with a fixed conformation, Iwamura³ presented a correlation between the rotational angle of the aryl group and the position of the low-frequency OH stretching band. The lowest frequency and presumably strongest hydrogen bond was observed for compounds with an angle of ca. 120°. Further IR studies led to the conclusion that the intramolecular hydrogen bond had an energy of ca. 2.6 kcal mol⁻¹ in the gas phase (in contrast to ca. 1.4 kcal mol⁻¹ in CCl₄).⁴ A study by FT-IR spectroscopy in decane-*d*₂₂ combined with digital resolution of the overlapping bands indicated

three bands to be present in the OH stretching region in this solvent, one corresponding to “free” OH (at 3644 cm⁻¹), one “conformational band” (at 3635 cm⁻¹) and one for the hydrogen-bonded species (at 3609 cm⁻¹).⁵ The energy difference between the species giving rise to the “free” and the “bonded” bands was found to be 1.64 kcal mol⁻¹, and that between the “free” and “conformer” species 0.96 kcal mol⁻¹.⁵

The conformation of 2-phenylethanol has also been studied by NMR spectroscopy.^{6,7} However, these investigations were carried out on solutions at high concentrations and in solvents where interactions between the hydroxy group and the solvent would be expected. We now report the results of an IR and NMR study of 2-phenylethanol at low concentrations in CCl₄ and CFCl₃ (solvents having only very weak interactions with alcohols) carried out in an attempt to gain information on the molecular origin of the two bands in the OH-stretch region of the IR spec-

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Table 1. Thermodynamic parameters for the conformational changes in the alcohols R¹R²CHC(R³R⁴)OH in CCl₄ (C < 10⁻² M). Determined by variable temperature spectroscopy (IR, NMR; -20 to +50 °C). ΔH in cal mol⁻¹, ΔS in cal mol⁻¹ deg⁻¹.

Compound				$\nu_{\text{high}} \rightleftharpoons \nu_{\text{low}}^a$	$\text{CHOH}_{\text{exo}} \rightleftharpoons \text{CHOH}_{\text{endo}}^b$	$(\text{PhC} \cdot \text{C} \cdot \text{OH})_{\text{trans}} \rightleftharpoons (\text{PhC} \cdot \text{C} \cdot \text{OH})_{\text{gauche}}^b$
R ¹	R ²	R ³	R ⁴			
Ph	H	H	H	$\Delta H = -1.12 \pm 0.03$ $\Delta S = -4.3 \pm 0.1$	-0.22 ± 0.11 1.4 ± 0.1	-0.50 ± 0.17 -1.9 ± 1.1
Ph	Ph	Ph		$\Delta H = -0.49 \pm 0.09$ $\Delta S = 1.0 \pm 0.3$	-1.35 ± 0.30 -0.57 ± 1.1	-0.35 ± 0.22 -0.52 ± 0.78
Ph	Ph	Ph	Ph	$\Delta H = -1.40 \pm 0.03$ $\Delta S = -1.90 \pm 0.12$	-	-
CH ₂ =CH	H	H	H	$\Delta H = -0.78 \pm 0.06$ $\Delta S = -3.8 \pm 0.2$	-	-

^aFrom $K = \frac{A_{\text{low}}^{\circ}}{A_{\text{high}}^{\circ}}$ where A_{low}° and A_{high}° are the areas of the low- and high-frequency bands, respectively, in the OH-stretch

region. ^bFrom vicinal proton coupling constants.

trum. To obtain information on the energetics of the conformations we have also performed molecular mechanics calculations, as well as semi-empirical (MNDO) and *ab initio* molecular orbital calculations.^{8,9,10}

Results

The IR and NMR parameters for 2-phenylethanol are given in the Experimental. The thermodynamic parameters (determined by variable temperature spectroscopy) for some substituted ethanols are given in Table 1. For 2-phenylethanol, there is only a relatively small difference between the vicinal coupling constants for the *gauche* and the *anti* rotamers around the CH₂CH₂ fragment. To obtain more accurate estimates of the conformational composition with respect to this bond, both 1- and 2-²H-2-phenylethanol were synthesised. However, even at 500 MHz, only the average ¹H NMR spectrum was obtained for this fragment.

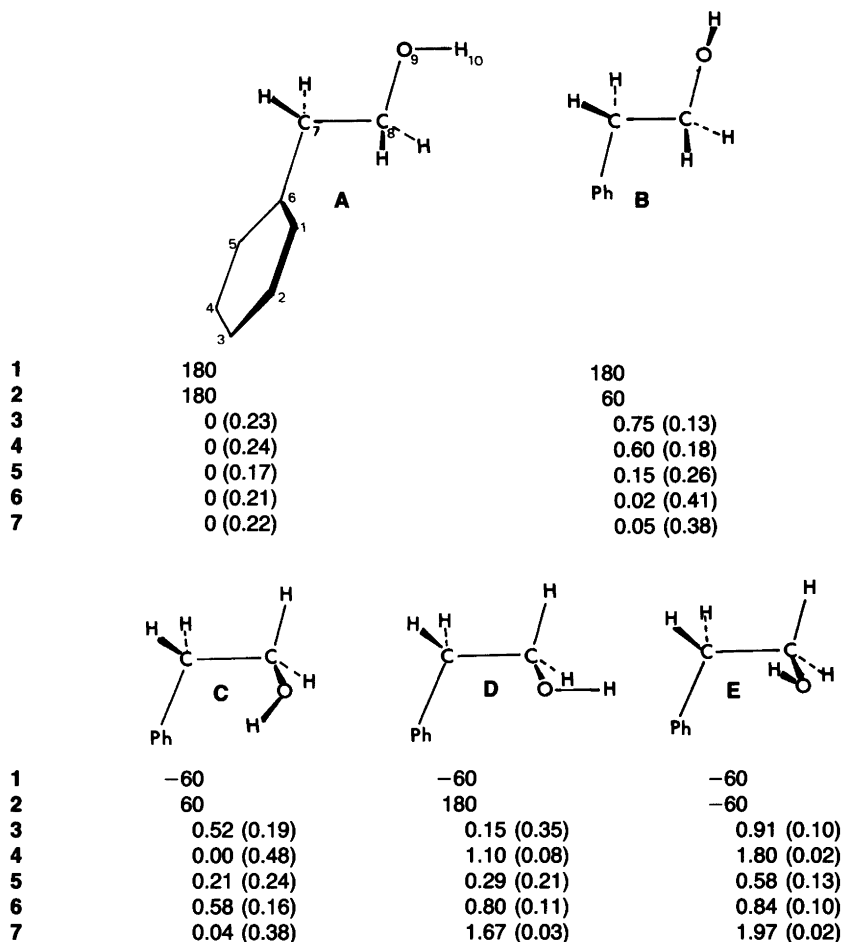
For 2-phenylethanol, we have calculated the relative energies of the stable conformations by molecular mechanics (program MMPMI)⁸ in both the default dipole-dipole electrostatic mode and with charge-charge interactions, the individual atomic charges being calculated according to the Abraham scheme (see Fig. 3 in Experi-

mental for the calculated charges).¹¹ The results from these calculations are shown in Scheme 1 for the five stable conformations. For comparison, we have also included the relative energies estimated by semi-empirical (MNDO) and *ab initio* (STO-3G and 4-31G) molecular orbital calculations.^{9,10} For all five conformations, the MM calculations indicate the phenyl ring to be disposed at an angle of ca. 90° with respect to the CH₂CH₂ chain (favoured by ca. 1.5–3 kcal mol⁻¹ over conformations in which this angle is ca. 0°). The calculated angles for the totally relaxed conformations are given in Table 3.

From the relative energies, the molecular compositions have been calculated. From these and the experimental coupling constants discussed below, the vicinal coupling constants for the CH₂CH₂ and CH₂OH fragments were calculated. These calculated coupling constants, together with the experimental ones, are given in Table 2.

Discussion

We will first discuss the conformational composition of 2-phenylethanol as manifested by the ¹H NMR spectrum. There are two stable conformational situations with respect to the CH₂CH₂ bond, one with the OH and the phenyl groups *anti* (conformers A and B, Scheme 1), and one



Scheme 1. Stable conformations of 2-phenylethanol (A–E). Row 1: Dihedral angle 6–7–8–9. Row 2: Dihedral angle 7–8–9–10. Relative energies (kcal mol⁻¹), Row 3: from MMPMI dipole–dipole, Row 4: from MMPMI atomic charges, Row 5: from MNDO, Row 6: from *ab initio* STO-3G, Row 7: from *ab initio* 4-31G calculations. Composition (mol fractions) in parentheses.

with these two groups *gauche* to each other (conformers C, D and E). There are also two such situations in relation to the CH₂OH bond, one *exo* (conformers A and D), the other *endo* (conformers B, C and E) with respect to the carbon–carbon chain. For the CH₂OH vicinal coupling constants, we use 2.2 Hz for the *gauche* and 12.5 Hz for the *anti* coupling.¹² This then gives 2.2 Hz for the *exo* and 7.4 Hz for the *endo* rotamer.

The spectrum of the CH₂CH₂ fragment shows only the averaged AA'BB' coupling and only limited information is therefore obtainable. We

will use the coupling constant values in Fig. 1 in order to estimate the composition of this fragment. The individual *gauche* coupling constants have been calculated from the equations of Abraham *et al.*¹³ The *anti* coupling constant is an estimate based on the *anti* coupling constant in 1-(*t*-butyl)-2-phenylethanol (10.6 Hz),¹⁴ the J_{aa} for cyclohexanol (11.4 Hz),¹² and those for 4,4-*d*₂-tetrahydropyran (12.8 and 12.2 Hz).^{15,16} These values then give $^3J_{\text{CH}_2\text{CH}_2} = 8.6$ Hz for the *anti* and 5.1 Hz for the *gauche* rotamer.

From these values and the observed coupling

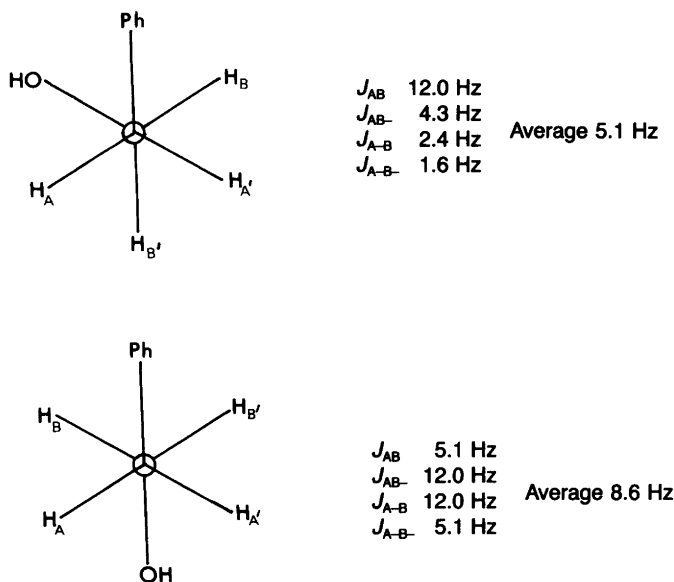


Fig. 1. $^3J^1H$ coupling constants used for estimation of the conformational composition around the CH_2CH_2 bond of 2-phenylethanol.

constants a rotamer composition of 61% *gauche* for the CH_2CH_2 fragment and 73% *endo* for the CH_2OH fragment is obtained. Given the virtually unlimited number of combinations of the five conformers in Scheme 1 that would reproduce these values, this result is in itself of limited value. However, with careful interpretation of the calculated compositions in Scheme 1 and, additionally, the guidance afforded by the IR spectrum of 2-phenylethanol, useful information may be gleaned.

In Table 2, calculated vicinal coupling con-

Table 2. Vicinal proton coupling constants for 2-phenylethanol, calculated on the basis of the calculated molecular composition in Scheme 1 and coupling constants in Fig. 1. Observed coupling constants: $J_{CH_2CH_2} = 6.47 \pm 0.05$ Hz; $J_{CH_2CH} = 5.99 \pm 0.05$ Hz.

Computational Method	Calculated vicinal coupling constants	
	$J_{CH_2CH_2}$	J_{CH_2CH}
MMPMI dipole mode	6.36	4.38
MMPMI charge mode	6.57	5.74
MNDO	6.66	5.50
<i>Ab initio</i> STO-3G	7.22	5.66
<i>Ab initio</i> 4-31G	7.18	6.28

stants are given for each of the five methods of calculation. A comparison with the experimental values shows the MMPMI method in the dipole-dipole mode to underestimate the population of the conformations with an *endo* conformation around the CH_2OH bond. The MMPMI technique with individual atomic charges gives a better result in this respect. The *ab initio* calculations overestimate the populations of the conformations with an *anti* orientation around the CH_2CH_2 bond. The MNDO calculation on the other hand underestimates the importance of conformations with an *endo* conformation around the CH_2OH bond. Nevertheless, the differences between the results from the MNDO calculations and those from the MM2 technique with individual atomic charges are almost within the experimental error. In comparing the results of the *ab initio* calculations at the STO-3G and 4-31G levels, perhaps most striking is the stabilisation of conformer C (the conformer geometrically most likely to possess an $OH \cdots \pi$ hydrogen bond) in the latter treatment compared with the former. Close inspection of the atomic charges from the two levels of calculation for all the conformers in Scheme 1 provides a possible rationale for this result. The 4-31G technique places significantly more electron density on oxygen (-0.743 e) and the ring carbons (average per C for the *o*-, *m*- and

p -atoms = $-0.192 e$) and correspondingly less on the OH hydrogen ($+0.405 e$) than does the STO-3G approach (corresponding quantities are -0.327 , -0.065 and $+0.201 e$). Such increased charge separation in the 4-31G treatment presumably corresponds to relative stabilisation of a coiled conformation such as C through advantageous electrostatic interactions between juxtaposed centres of positive and negative charge. In this connection, it is not without interest to note that the charges produced by the Abraham formalism (Fig. 3) lie between those predicted by the two *ab initio* treatments.

Additional information is available from the IR spectrum. 2-Phenylethanol shows two bands in the OH-stretch region, at 3629 and 3602 cm^{-1} , the low frequency band having 43 % of the combined band area. Only conformer C in Scheme 1 has the possibility of an interaction between the OH hydrogen and the phenyl ring. It appears reasonable to assume that such an interaction

would result in a lengthening and weakening of the O-H bond and therefore in a shift to lower frequency of the stretching vibration of the OH group in this conformation. We therefore assign the low frequency band to conformer C. This also implies that the OH bands for the other conformations in Scheme 1 are not separated under the present conditions. We now see that the MMPMI charge-charge calculation reproduces rather well both the observed ^1H NMR and IR spectra, this calculation predicting 48 % of the conformation having the possibility of an OH-phenyl ring interaction (conformer C) and the IR spectrum indicating 42 % of such a conformation. We have previously argued that for IR bands as close together as those here, the molecular integrated absorption coefficients for the bands will be of the same magnitude.¹⁷ This is also supported by studies on systems with intermolecular hydrogen bonds.^{18,19}

If we now turn to the thermodynamic para-

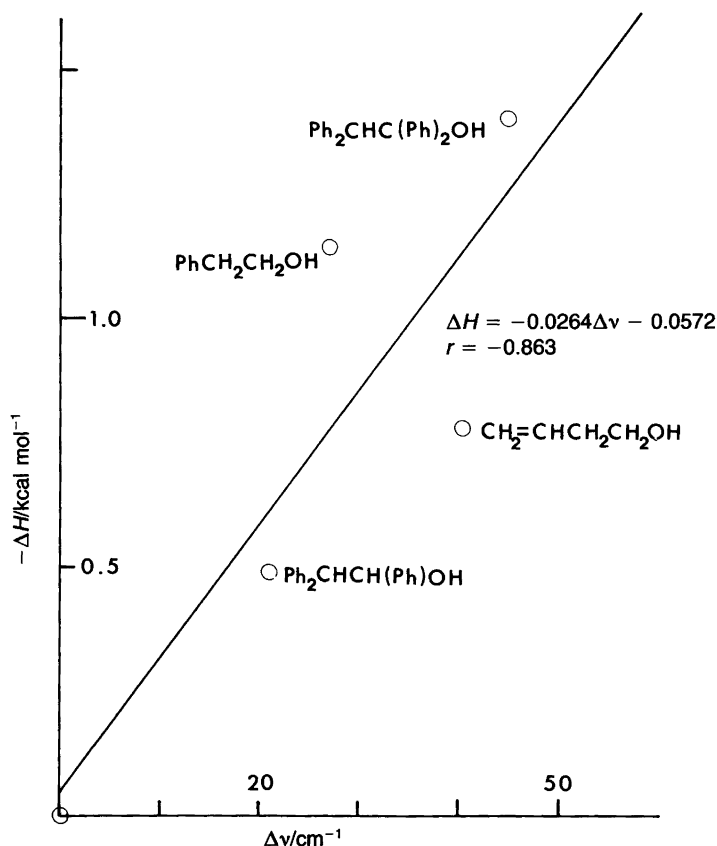


Fig. 2. Correlation of ΔH for the OH... π -bond interactions (from variable temperature IR spectroscopy) with frequency shifts of the low-frequency OH bands.

meters for 2-phenylethanol (Table 1), we apparently find a contradiction between the results from the IR and NMR investigations. The IR investigation indicates an energy difference of 1.12 ± 0.03 kcal mol⁻¹ in favour of the conformation with the low-frequency absorption. The NMR investigation does not reveal any molecular changes entailing such a high expenditure of energy: ΔH for the *gauche/anti* equilibrium of the CH₂CH₂ rotation is only 0.5 ± 0.2 kcal mol⁻¹, and that for the *endo/exo* equilibrium of the CH₂OH fragment almost zero to within the experimental error (0.2 ± 0.1 kcal mol⁻¹).

This apparent contradiction is resolved if we assume the model from the MMPMI charge-charge calculation to be valid. According to this (Scheme 1) there are only three conformations of any importance: **A**, *anti* with respect to the CH₂CH₂ bond and *exo* with respect to the CH₂OH bond (ca. 27%), **B**, with *anti* and *endo* orientations, respectively (ca. 20%), and **C** with *gauche* and *endo* conformations (ca. 53%). Thus, with almost equal populations of conformers **A** and **B**, the *observable* molecular change in going from **A/B** to **C** will be the rotation of the CH₂CH₂ fragment with concomitant change of the corresponding vicinal coupling constant. The ΔH for this rotation was found to be 0.5 kcal mol⁻¹. The change in the CH₂OH fragment will show a smaller energy, as this will represent the change from **A** to **B**; the change from **B** to **C** will not be observable on the basis of the vicinal coupling constant for this fragment, both conformers having *endo* rotamers around the C–O bond.

The conclusion from the NMR and IR investigations is therefore that conformer **C** is favoured over **A** and **B** to the extent of ca. 1.2 kcal mol⁻¹ and that in this conformation the hydroxy group proton points towards the π -electron system of

the phenyl ring. Whether this conformational preference stems from the existence of a hydrogen bond or is a consequence of repulsion between the π -electrons and the lone-pairs of the hydroxy oxygen²⁰ is best inferred from the IR spectroscopic evidence.

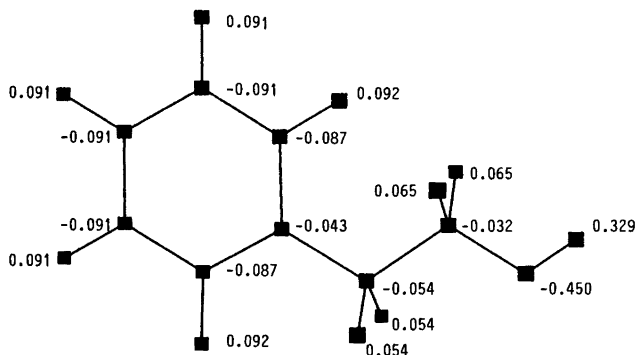
It is accepted that there exists a linear relationship between ΔH for formation a hydrogen bond and the shift in the OH-stretch band for the hydrogen-bonded species (to lower frequency) as compared to that of the "free" hydroxy group.²¹ Data are almost non-existent for the weak putative hydrogen bonds possible here, be they intramolecular or intermolecular. This is especially so with π -electron systems as the base (acceptor). We have therefore determined thermodynamic parameters for a series of unsaturated and aromatic alcohols (Fig. 2) and have correlated ΔH with the shift in the OH-stretching band frequency ($\Delta\nu$). The latter has been a somewhat uncertain quantity as the exact frequency of the "free" band has been difficult to determine. By the combined use of NMR and IR it has, however, proved possible to obtain a reasonable estimate of the two frequencies.¹⁷ Fig. 2 shows the correlation between ΔH and $\Delta\nu$ for a series of alcohols. For some of these, e.g. 3-buten-1-ol and 1,1,2,2-tetraphenylethanol, the shift is so large that it is reasonable to assume that intramolecular hydrogen bonds exist. Assuming that zero, zero is also part of the plot, we obtain a slope of 0.026 ± 0.008 kcal mol⁻¹ $\Delta\nu^{-1}$. This is of the same magnitude as found for several intermolecularly hydrogen-bonded systems.²¹ 2-Phenylethanol shows a somewhat high value in Fig. 2. It might be argued that this molecule experiences both an attraction due to a hydrogen bond between the OH and the phenyl ring, and a repulsion between the ring and the lone-pair electrons of the hydroxy oxygen. How-

Table 3. Calculated dihedral angles for 2-phenylethanol for the relaxed molecule. See Scheme 1 for designation of conformations and atom-numbers.

Computational method	A			B		
	Angles			Angles		
	1-6-7-8	6-7-8-9	7-8-9-10	1-6-7-8	6-7-8-9	7-8-9-10
MMPMI dipole-dipole	89.6	180.0	180.0	88.0	179.9	66.4
MMPMI atomic charges	89.6	180.0	180.0	88.7	178.7	64.2
MNDO	90.3	179.3	-179.9	89.1	178.0	72.7

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Fig. 3. Individual atomic charges in 2-phenylethanol calculated by the Abraham scheme.¹¹



ever, the variation is not larger than for some other molecules in Fig. 2 (e.g. 3-buten-1-ol) and we can only conclude from Fig. 2 that the preference for conformer C in 2-phenylethanol is caused, at least in part, by an intramolecular hydrogen bond.

This conclusion is further reinforced by observations concerning the IR spectra of ring-substituted 2-phenylethanol.¹ Here, it is found that the $\Delta\nu$ value is dependent on the ring substituent, being largest for electron-donating substituents (with the strongest hydrogen bonds) and smallest for electron-withdrawing substituents.¹ This is not the case for substituted benzyl alcohols, where the shift is independent of the substituent.²² We have taken this as evidence against intramolecular hydrogen bonding in benzyl alcohols.²⁰

We will return to the conformational composition of the other alcohols in Fig. 2 in later publications.

Experimental

The NMR spectra were recorded on Bruker 400 or Bruker 500 MHz spectrometers with a numerical resolution of 0.1 Hz. The IR spectrometer

used was a Beckman IR 4250. The procedures for obtaining the spectra have been described previously.²³ The ¹H NMR and IR spectra of 2-phenylethanol (Koch-Light, purified by distillation) were obtained for solutions in CFC₁₃ or CCl₄ (for variable temperature experiments) at concentrations lower than 10⁻² M. The reported coupling constants are the averaged values for the triplets of the PhCH₂ and the OH fragments. NMR (3.2 mM, CFC₁₃): δ 7.1–7.0 (5H, m), 3.660 (2H, q, *J* 6.2 Hz), 2.724 (2H, t, *J* 6.47±0.05 Hz), 0.813 (extrapolated to infinite dilution) (t, *J* 5.99±0.06 Hz). IR (2.5 mM, CCl₄): 3629 cm⁻¹ (*A*₀ = 3196 M⁻¹ cm⁻²), 3602 cm⁻¹ (*A*₀ = 2459 M⁻¹ cm⁻²). The methods used for obtaining the thermodynamic parameters by variable temperature spectroscopy have been described previously.¹²

2-Phenyl-2-²H-ethanol. α -²H-Benzyl chloride²⁴ (5 g) in dry diethyl ether (50 ml) was reacted with Mg (1 g). After heating under reflux (30 min) the resulting Grignard solution was poured into a mixture of finely crushed solid CO₂ and ether (25 ml). After reaching room temperature, the aqueous phase was extracted twice with ether, acidified with hydrochloric acid and extracted three times with ether. The ether phase was dried

Table 3. (contd)

C			D			E		
Angles			Angles			Angles		
1-6-7-8	6-7-8-9	7-8-9-10	1-6-7-8	6-7-8-9	7-8-9-10	1-6-7-8	6-7-8-9	7-8-9-10
94.0	-63.6	70.2	88.5	-61.2	-178.9	88.0	-60.2	-61.1
93.8	-62.5	64.8	88.3	-65.1	177.8	86.4	-62.2	-57.2
95.9	-65.7	79.6	95.7	-62.1	177.0	94.6	-60.0	-70.9

over MgSO_4 and evaporated to give α - ^2H -phenylacetic acid (4 g; m.p. 76 – 77°C). This (0.6 g) was reduced with LiAlH_4 (0.27 g) in tetrahydrofuran under reflux. Aqueous NaOH (10%, 0.5 ml) was added to the cooled solution; the ether was then removed by evaporation and the product distilled (Kuegelrohr) to give 2- ^2H -2-phenylethanol. NMR (3.3 mM, CCl_4): δ 7.1–7.0 (5H, m), 3.681 (2H, t, J 6.2 Hz), 2.713 (1H, t of t, $J_{\text{H}^2\text{H}}$ 2.1 ± 0.1 Hz, J_{vic} 6.57 ± 0.19 Hz), 0.850 (1H, t, J 6.00 ± 0.1 Hz). In variable temperature experiments the two J_{vic} 's varied in the same way as for the undeuterated 2-phenylethanol; $J_{\text{H}^2\text{H}}$ was constant within the experimental error.

1- ^2H -2-Phenylethanol. This was prepared by lithium aluminium deuteride reduction of phenylacetaldehyde in diethyl ether. NMR (4.3 mM, CFCl_3): δ 7.1–7.0 (5H, m), 3.644 (1H, q, J 6.4 Hz), 2.679 (2H, d, J 6.40), 0.842 (1H, d, J 5.95 Hz).

Theoretical Investigations. Calculations were performed on either MicroVax II or Olivetti M24 computers. Molecular mechanics calculations utilised the MMPMI program,⁸ and semi-empirical molecular orbital calculations the program MNDO,⁹ both as implemented by Serena Software, Bloomington, Indiana, USA. Total geometry optimisation was permitted throughout. Atomic charges evaluated according to the Abraham scheme¹¹ for input to MMPMI are given in Fig. 3. *Ab initio* calculations at the STO-3G and 4-31G levels were carried out on MNDO-relaxed geometries with GAUSSIAN 80¹⁰ via the Chem-X molecular modelling package, developed and distributed by Chemical Design Ltd., Oxford, England.

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