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RING-CHAIN TAUTOMERISM OF 1-HYDROXYPHTHALANS. AN EXAMINATION OF STRUCTURAL EFFECTS.

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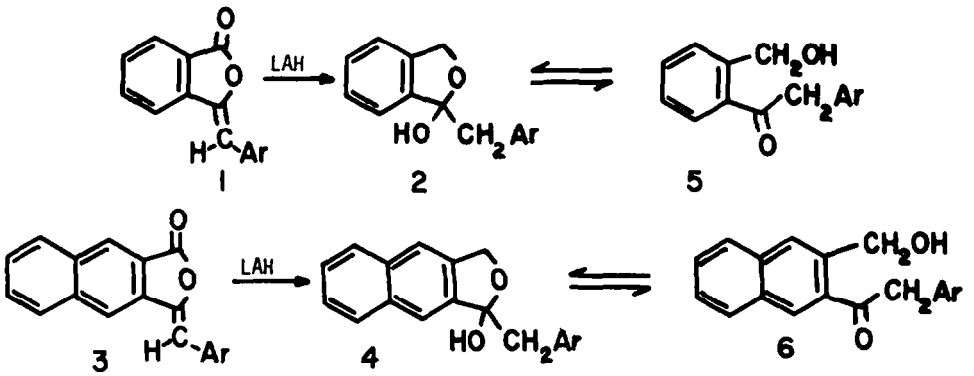
Abstract

1,3-Dihydro-1-isobenzofuranol,(1-hydroxyphthalan) and 2-(hydroxymethyl)-benzaldehyde exist in a tautomeric equilibrium. The effects of molecular structure on this equilibrium was examined using various derivatives of dihydroisobenzofuranol. Two effects of this molecular modification were identified: (i) 1-arylmethyl substituents favored the ring-opened tautomer if steric effects arose in the ring closed form and (ii) extending the conjugated ystem of the dihydroisobenzofuran ring (i.e. dihydronaphtho[2,3-c] furan also favored the ring-opened form. These effects are discussed.

Recently, it was reported[1] that 1,3-dihydro-1-isobenzofuranol (1-hydroxyphthalan) existed in equilibrium with its open-chain tautomer 2-(hydroxymethyl)benzaldehyde. For some time, we have had an interest in ring-chain tautomerism[2] as well as in 1,3-dihydro-1-isobenzofuranols because of their utility as precursors to isobenzofurans[3].

As an extension of our earlier studies[3] of the preparation, via transient isobenzofuran derivatives, of fused ring polycyclic aromatic ring systems, a number of compounds related to 1,3-dihydro-1-isobenzofuranol have been prepared (Scheme **1**, **2** and **4**). These were obtained* by lithium aluminum hydride reduction of the appropriate isobenzofuran-1(3H)-one (phthalide), **1** , or naphtho[2,3-c]furan-1(3H)-one (naphthalide), **3** . The initial products of the reductions were the 1,3-dihydro-1-isobenzofuranols, **2** , and 1,3-dihydro-1-naphtho[2,3-c] furanols, **4** . While these compounds readily dehydrated, they were isolable by using mild non-acidic conditions. Having these compounds (**2** and **4**) in hand, it was possible to assess structural effects on the ring-chain tautomerism.

The infrared spectra of solid samples of **2** and **4** showed no carbonyl absorption band with the exception of **4b** and **4c** . In general, the ring closed form of **2** and **4** was the stable tautomer in the solid state. In contrast, the infrared spectra of chloroform solutions of **2** and **4** all showed a carbonyl band. Clearly, in solution, the equilibria **2**⇌**5** and **4**⇌**6** existed.



a) Ar = C_6H_5
b) Ar = α-$C_{10}H_7$
c) Ar = β-$C_{10}H_7$
d) Ar = o-$C_6H_4CH_3$
e) Ar = m-$C_6H_4CH_3$
f) Ar = p-$C_6H_4CH_3$

Scheme 1. Preparation of Isobenzofuranol Drivatives

The position of these equilibria was evaluated quantitatively by analysis of the NMR spectra of deuterochloroform solutions of the compounds. A typical spectrum of **4c** is shown in Figure 1 and the experimental section describes the specific analysis for each compound with the results summarized in Table 1.

Figure 1. NMR Spectrum of the 4c, 6c Equilibrium

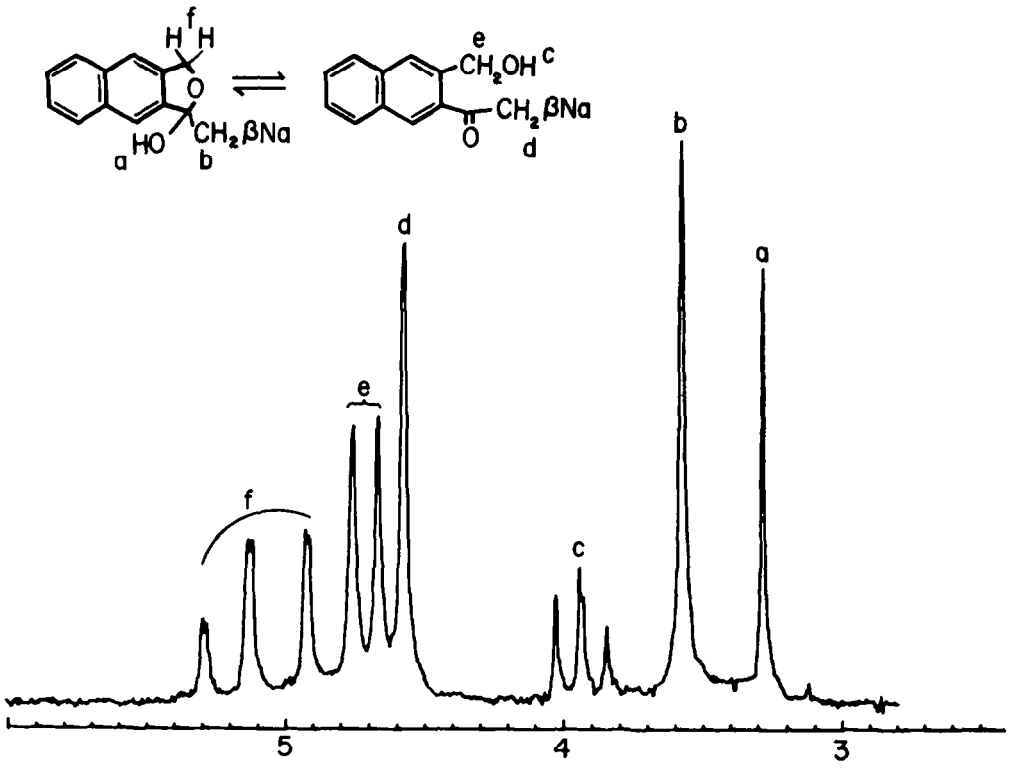
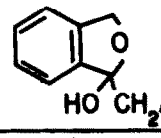
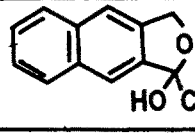
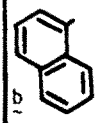
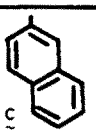
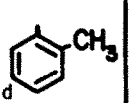
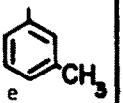
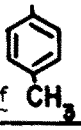


Table 1. Ring-Chain Tautomerism Analyses

Compound %	RO	CY	RO	CY	RO	CY	RO	CY	RO	CY	RO	CY
 2	25	75	48	52	26	74	45	55	27	73	27	73
 4	48	52	76	24	47	53	71	29	48	52	48	52
Ar →	 a		 b		 c		 d		 e		 f	

Two effects of structure on the equilibrium between the RO tautomer (**5,6**) and the CY form (**2,4**) can be seen. Firstly, insofar as the aryl group of either **2** or **4** was concerned, the phenyl and β-naphthyl groups were equivalent. Indeed, in the case of **2a** and **2b** , each equilibrium was almost identical to that reported[1] for 1,3-dihydro-1-isobenzofuranol itself. However, the α-naphthyl group shifted the equilibrium significantly toward the RO tautomer.

Secondly, with a given aryl group, the 1,3-dihydro-1-naphtho[2,3-c] furanols, **4** , definitely favored the RO form relative to the corresponding 1,3-dihydro-1-isobenzofuranols, **2** .

It was hypothesized that the first effect resulted from an increased steric interaction in the cyclic tautomer between the aryl substituent and the hydrogen of the methylene group opposing it. In the cyclic form of **2a** (i.e. **7**), this steric interaction

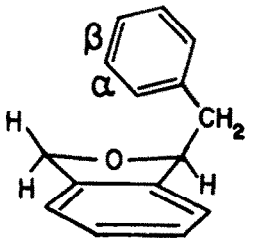


Figure 2 Steric Interaction of Cyclic Phthalanol

would not be significantly increased if a benzo-ring were added to the β side of the phenyl ring (i.e. as in **2c**). In contrast, increased steric interaction would occur if an α-benzo ring were added (i.e. as in **2b**). In this case, the equlibrium would shift in favour of the RO tautomer in order to relieve the increased interaction.

It is interesting to note that the NMR spectra of **2c** and **4c** show the $ArCH_2$ as an AB quartet, the only such compounds to do so. While the H's of the methylene would be expected to be diastereotopic, the chemical shift difference must normally be small. Only when the

increased steric interactions present in **2c** and **4c** disturb the "normal" distribution of conformations does the chemical shift difference become large enough to be observed.

To test this steric hypothesis, a second series of 1,3-dihydro-1-isobenzofuranols and 1,3-dihydro-1-naphtho[2,3-c] furanols, **2d** , **e** , **f** and **4d** , **e** , **f** were prepared with the aryl group being varied from o-, to m- and p-tolyl.

As expected the m- and p-tolyl derivatives **2e** , **2f** , and **4e** , **4f** showed equilibria closely resembling that of the phenyl and β-naphthyl analogs. However, the o-tolyl derivatives **2d** and **4d** showed a shift towards the RO tautomer and the equilibria resembled that of the α-naphthyl analogs **2c** and **4c** . The obvious increased steric interaction of the o-methyl group (relative to m- and p-methyl) with the ring methylene of the CY tautomer would explain these shifts in equilibria. Additionally, the AB quartet pattern for $ArCH_2$ was observed in the NMR spectra of **2d** and **4d** but not in the other tolyl derivatives.

The second structural effect mentioned earlier, that those compounds containing a naphtho ring (the **4** series) showed a greater amount of RO tautomer in the equilibrium than the corresponding derivatives with a benzo ring (the **2** series), is puzzling. Initially, it was thought that the carbonyl group of the RO tautomer conjugated with the more extended aromatic system of the naphtho ring (relative to the benzo ring) would lend additional stability to the RO form. However, the position of the carbonyl absorption bands of both the **2** (i.e. **5**) and the **4** (i.e. **6**) series showed little difference.

While the above hypothesis has not been discarded, the possibility is also being considered that the naphthalene derivatives **4** (and **6**) deviate sufficiently from the idealized hexagonal shape of the aromatic moiety that angular strains are introduced into the molecules which affect the equilibria and the strains are absent in the "benzene" series **2** (and **5**). Efforts are underway to synthesize appropriate compounds to test this hypothesis.

Experimental

Melting points were determined in open capillaries with a Mel-Temp apparatus and are uncorrected. Infrared (IR) spectra were recorded on a Beckman Acculab 10 spectrometer and NMR spectra were determined on a Bruker WP-80 spectrometer with tetramethylsilane (TMS) as an internal standard. Chemical analyses were determined by NHW Laboratories, Phoenix, Arizona and the Uniroyal Research Laboratories, Guelph, Ontario.

3-(Phenylmethylene)isobenzofuran-1(3H)-one, **1a** ,(3-benzalphthalide or benzylidene phthalide) was obtained commercially. Its analogs were prepared by a standard method[5]. The individual compounds are described below.

3-(1-Naphthalenylmethylene)isobenzofuran-1(3H)-one, **1b** , was obtained in 50% yield, from acetone mp 168-170°C (lit.[6] 179°C). NMR ($CDCl_3$) 7.16 (s, 1H), 7.3-8.4 (m, 11H); IR (nujol) 1770, 1242, 1064, 963, 788, 769, 752 cm^{-1}.

3-(2-Naphthalenylmethylene)isobenzofuran-1(3H)-one, **1c** , was obtained in 70% yield, from acetone mp 175-176°C (lit.[7] 170-171°C); NMR ($CDCl_3$) 6.57 (s, 1H), 7.3-8.3 (m, 11H); IR (nujol) 1768, 1075, 979, 953, 752, 741 cm^{-1}.

3-(Phenylmethylene)naphtho[2,3-c]furan-1-(3H)-one, **3a** , was obtained in 75% yield, from acetone - DMF mp 195-197°C (lit.[8] 191-193°C); NMR ($CDCl_3$) 6.56 (s, 1H), 7.2-8.3 (m, 10H), 8.52 (s, 1H); IR (nujol) 1778, 1150, 1090, 962, 742 cm^{-1}.

3-(1-Naphthalenylmethylene)naphtho[2,3-c]furan-1(3H)-one, **3b** , was obtained in 35% yield, from DMF mp 251-253°C; NMR ($CDCl_3$) 7.36 (s, 1H), 7.3-8.4 (m, 12H), 8.57 (s, 1H); IR (nujol) 1775, 1157, 1104, 966, 867, 776 cm^{-1}. Anal. Calc'd. for $C_{19}H_{12}O_2$: C, 83.81; H, 4.44. Found: C, 83.63; H, 4.58.

3-(2-Naphthalenylmethylene)naphtho[2,3-c]furan-1(3H)-one, **3c** , was obtained in 45% yield, from DMF mp 215-216°C (lit.[9] 207°C); NMR ($CDCl_3$) 6.73 (s, 1H), 7.4-8.3 (m, 12H), 8.56 (s, 1H); IR (nujol) 1768, 1109, 990, 901, 746 cm^{-1}.

3-[(2-Methylphenyl)methylene]isobenzofuran-1(3H)-one, **1d** , was obtained in 52% yield, from ethanol mp 130-131°C (lit.[10] 136.5°C); NMR ($CDCl_3$) 2.50 (s, 3H), 6.7 (s, 1H), 7.1-8.3 (m, 8H); IR (nujol) 1760, 1267, 1088, 974, 751 cm^{-1}.

3-[(3-Methylphenyl)methylene]isobenzofuran-1(3H)-one, **1e** , was obtained in 50% yield, from ethanol-acetone mp 153-154°C (lit.[11] 152-153°C); NMR ($CDCl_3$) 2.39 (s, 3H), 6.37 (s, 1H), 7.0-8.0 (m, 8H); IR (nujol) 1762, 1660, 1278, 1086, 976, 688 cm^{-1}.

3-[(4-Methylphenyl)methylene]isobenzofuran-1(3H)-one, **1f** , was obtained in 63% yield, from ethanol-acetone mp 151-154°C (lit.[12] 151°C); NMR ($CDCl_3$) 2.37 (s, 3H), 6.39 (s, 1H), 7.1-8.0 (m, 8H); IR (nujol) 1780, 1660, 1353, 1077, 973, 764 cm^{-1}.

3-[(2-Methylphenyl)methylene]naphtho[2,3-c]furan-1(3H)-one, **3d** , was obtained in 13% yield, from DMF mp 218-222°C; NMR ($CDCl_3$) 2.52 (s, 3H), 6.76 (s, 1H), 7.2-8.2 (m, 8H), 8.50 (s, 1H), 8.74 (s, 1H); IR (nujol) 1761, 1658, 1158, 1099, 974, 749 cm^{-1}. Anal. Calc'd. for $C_{20}H_{14}O_2$: C, 83.90; H, 4.93. Found: C, 84.10; H, 5.00.

3-[(3-Methylphenyl)methylene]naphtho[2,3-c]furan-1(3H)-one, **3e** , was obtained in 33% yield, from acetone mp 185-188°C; NMR ($CDCl_3$) 2.40 (s, 3H), 6.50 (s, 1H), 7.0-8.1 (m, 8H), 8.20 (s, 1H), 8.40 (s, 1H); IR (nujol) 1767, 1665, 1155, 1105, 975, 892 cm^{-1}. Anal. Calc'd. for $C_{20}H_{14}O_2$: C, 83.90; H, 4.93. Found: C, 83.85; H, 4.87.

3-[(4-Methylphenyl)methylene]naphtho[2,3-c]furan-1(3H)-one, **3f** , was obtained in 15% yield, from acetone mp 211-215°C; NMR ($CDCl_3$) 2.39 (s, 3H), 6.53 (s, 1H), 7.1-8.1 (m, 8H), 8.18 (s, 1H), 8.49 (s, 1H); IR (nujol) 1775, 1659, 1155, 1096, 1078, 971 cm^{-1}. Anal. Calc'd. for $C_{20}H_{14}O_2$: C, 83.90; H, 4.93. Found: C, 84.09; H, 5.07.

Preparation of 1,3-dihydro-1-phenylmethyl-1-isobenzofuranol, **2a** and its analogs.

The preparation followed the published procedure[4,13] which involved a LAH reduction of the appropriate **1** or **3** . The product was isolated by removing the solvent (diethyl ether) and, when possible, recrystallising from diethyl ether.

NMR spectra were obtained immediately after isolation. The spectra are reported for RO and CY tautomers. For consistency, the products are named as their ring-closed forms. Attempts to obtain chemical analyses were frequently unsuccessful due to facile dehydration of these compounds.

1,3-Dihydro-1-phenylmethyl-1-isobenzofuranol, **2a** .

NMR ($CDCl_3$)(CY) 3.09 (s, 1H, exchanges with D_2O), 3.31 (s, 2H), 4.70 and 5.06 (ABq, 2H, J=13 Hz); (RO) 3.72 (t, 1H, J=7.5 Hz, exchanges with D_2O), 4.55 (d, 2H, J=7.5 Hz, s with D_2O; 7.07-7.47 (m, aromatics). The peak area of resonances at 3.31 and 4.90 were used to determine the tautomeric ratio. IR (nujol) 3340, 1112, 1050, 1010, 979, 772, 723, 708 cm^{-1}. ($CHCl_3$) 3550, 3360 (broad), 1660, 1010, 690 cm^{-1}.

1,3-Dihydro-1-(1-naphthalenylmethyl)-1-isobenzofuranol, **2b** .

NMR ($CDCl_3$) (CY) 3.61 and 3.82 (ABq, 2H, J=14 Hz), 3.89 (s, 1H, exchanges with D_2O), 4.42 and 4.79 (ABq, 2H, J=13 Hz); (RO) 3.84 (t, 1H, exchanges with D_2O), 4.42 (d, 2H, J= 8Hz, s with D_2O), 4.53 (s, 2H); 6.82-8.26 (m, aromatics). The peak area of all resonances between 4.9 and 4.3 δ (CY plus RO) was corrected for the contribution due to CY tautomer as determined by the peak area of the ABq at 3.61 and 3.82 δ. The relative amounts of the two tautomers were then calculated. IR (neat) 3440, 1685, 1104, 1030, 775 cm^{-1}.

1,3-Dihydro-1-(2-naphthalenylmethyl)-1-isobenzofuranol, **2c** .

NMR ($CDCl_3$) (CY) 3.06 (s, 1H, exchanges with D_2O), 3.48 (s, 2H), 4.71 and 5.08 (ABq, J=13 Hz, 2H); (RO) 3.72 (t, 1H, exchanges with D_2O), 4.45 (s, 2H), 4.57 (d, J=7 Hz, 2H, singlet with D_2O); 7.07-7.86 (m, aromatics). The same procedure was used to calculate the relative amounts of tautomers as with **2b** using the area of the signal at 3.48 δ (CY) and the total area of the signals between 4.45 and 5.1 (CY plus RO). IR (nujol) 3340, 1070, 1000, 980, 760, 740, 720 cm^{-1}; ($CHCl_3$) 3600, 3400 (Broad), 1675, 1510, 1470, 1020, 860cm^{-1}.

1,3-Dihydro-1-(phenylmethyl)-1-naphtho[2,3-c]furanol, **4a** .

NMR ($CDCl_3$) (CY) 3.38 (s, 2H), 3.46 (s, 1H, exchanges with D_2O), 4.81 and 5.16 (ABq of doublets, 2H, J_1=13 Hz, J_2=1 Hz); (RO) 3.93 (t, 1H, exchanges with D_2O), 4.38 (s, 2H), 4.68 (d, 2H, J=7 Hz, s with D_2O); 7.1-8.4 (m, aromatics). Integration of the signals at 3.38 and 4.38 δ was used to determine the concentration of the tautomers. IR (nujol) 3350, 1110, 1040, 1020, 880, 760, 710 cm^{-1}; ($CHCl_3$) 3580, 1670, 1490, 1460, 1020, 870 cm^{-1}. This compound proved stable enough to recrystallize from diethyl ether, mp 113-115°C. Anal. Calc'd. for $C_{19}H_{16}O_2$: C, 82.58; H, 5.84. Found: C, 82.30; H, 6.09.

1,3-Dihydro-1-(1-naphthalenylmethyl)-1-naphtho[2,3-c]furanol, **4b** .

NMR ($CDCl_3$) (CY) 3.10 (s, 1H, exchanges with D_2O), 3.82 and 4.02 (ABq, J=14 Hz, 2H), 5.21 (half of ABq of doublets, J_1=13 Hz, J_2=1 Hz); (RO) 3.79 (t, 1H, J=7 Hz, exchanges with D_2O), 4.71 (d, 2H, s with D_2O), 4.91 (s, 2H); 7.4-8.6 (m, aromatics). The total area of the signals at 4.7-5.2 δ (CY plus RO) was corrected for the presence of the CY tautomer using the peak area of the 3.8/4.0 δ ABq. This latter area together with the corrected area was used to determine the relative amounts of the tautomers. IR (nujol) 3300, 1660, 1100, 1030, 780 cm^{-1}; ($CHCl_3$) 3500, 1670, 1110, 1020, 780 cm^{-1}. This material was recrystallized from diethyl ether, mp 133-134°C. Anal. Calc'd for $C_{13}H_{18}O_2$: C, 84.64; H, 5.56. Found: C, 84.60; H, 5.41.

1,3-Dihydro-1-(2-naphthalenylmethyl)-1-naphtho[2,3-c]furanol, **4c** .

NMR ($CDCl_3$) (CY) 3.28 (s, 1H, exchanges with D_2O), 3.56 (s, 2H), 4.85 and 5.19 (ABq of doublets, 2H, J_1=13 Hz, J_2=1 Hz); (RO) 3.90 (t, 1H, J= 7Hz, exchanges with D_2O), 4.56 (d, 2H, s with D_2O); 7.2-8.5 (m, aromatics H). Relative amount of the tautomers was determined by peak areas of the 3.56 and 4.56 δ signals. IR (nujol) 3530, 1690, 1000, 880, 740 cm^{-1}; ($CHCl_3$) 3550, 1670, 1010, 880 cm^{-1}. The compound, **4c** , was recrystallised from diethyl ether, mp 133-136°C dec. Anal. Calc'd. for $C_{23}H_{18}O_2$: C, 84.64; H, 5.56. Found: C, 84.56; H, 5.72.

1,3-Dihydro[(2-methylphenyl)methyl]-1-isobenzofuranol, **2d** .
NMR ($CDCl_3$)(CY) 2.34 (s, 3H), 3.21 (s, 1H, exchanges with D_2O), 3.29 and 3.41 (ABq, 2H, J= 14 Hz), 4.71 and 5.03 (ABq, 2H, J=14 Hz); (RO) 2.26 (s, 3H), 3.76 (t, 1H, J= 7 Hz, exchanges with D_2O),4.33 (s, 2H), 4.55 (d, 2H, J= 7 Hz, s with D_2O); 7.0-8.1 (m, aromatic H). The areas of the Abq at 3.3 δ (CY) and the singlet at 4.3 δ (RO) as well as the singlets at 2.34 (CY) and 2.26 (RO) were used to evaluate the ratio of the tautomers. IR (neat) 3400, 1675, 1463, 1020, 737 cm^{-1}.
1,3-Dihydro-[(3-methylphenyl)methyl-1-isobenzofuranol, **2e** .
NMR ($CDCl_3$)(CY) 2.25 (s, 3H), 3.25 (s, 2H), 4.62 and 4.96 (ABq, 2H, J= 13 Hz); (RO) 2.32 (s, 3H), 4.20 (s, 2H), 4.52 (s, 2H); 3.73 (s, exchanges with D_2O); 6.8-8.0 (m, aromatic H). Peak areas at 3.25 δ (CY), 4.20 δ (RO), 2.25 (CY) and 2.32 (RO) were used to calculate the ratio of tautomers. IR (neat) 3400, 1675, 1460, 1010,
1,3-Dihydro-1-[(4-methylphenyl)methyl]-1-isobenzofuranol, **2f** .
NMR ($CDCl_3$) (CY) 2.28 (s, 3H), 2.97 (s, 1H, exchanges with D_2O), 3.27 (s, 2H), 4.75 and 5.09 (ABq, 2H, J= 13 Hz); (RO) 2.32 (s, 3H) 760, 732 cm^{-1},3.74 (t, J= 7 Hz, 1H, exchanges with D_2O), 4.25 (s, 2H), 4.55 (d, J= 7 Hz, 2H, s with D_2O). The areas of the signals at 3.27 δ (CY) and 4.25 δ (RO) were used to estimate the tautomeric ratio. Mp 93-94°C after recrystallisation from diethyl ether; Anal. calc'd. for $C_{16}H_{16}O_2$: C, 79.97; H, 6.71. Found: C, 79.85; H, 6.95.
1,3-Dihydro-1-[(2-methyphenyl)methyl]-1-naphtho[2,3-c]furanol, **4d** .
NMR ($CDCl_3$) (CY) 2.34 (s, 3H), 3.32 and 3.50 (ABq, J= 14 Hz, 2H), 4.85 and 5.20 (ABq, J= 12 Hz, 2H); (RO) 2.28 (s, 3H), 3.95 (t, J=7 Hz,1H, exchanges with D_2O), 4.45 (s, 2H), 4.72 (d, J= 7 Hz, 2H); 7.1-8.5 (m, aromatic H). The areas of the signals at 2.28 and 2.34 were used to calculate the tautomeric ratio. IR (neat) 3400, 1685, 1030, 890, 751 cm^{-1}.
1,3-Dihydro-1-[(3-methylphenyl)methyl]-1-naphtho-[2,3-c]furanol, **4e** .
NMR ($CDCl_3$) (CY) 2.19 (s, 3H), 3.32 (s, 2H), 4.77 and 5.08 (ABq, 2H, J= 13 Hz); (RO) 2.28 (s, 3H), 4.26 (s, 2H), 4.65 (s, 2H); 3.94 (broad s, exchanges with D_2O), 6.8-8.3 (m, aromatic H). The areas of the signals at 4.26 δ (RO) and 3.32 δ (CY) were used to calculate the tautomeric ratio. IR (neat) 3400, 1675, 1010, 760 cm^{-1}.
1,3-Dihydro-1-[(4-methylphenyl)methyl-1-naphtho-[2,3-c]furanol, **4f** .
NMR ($CDCl_3$) (CY) 2.32 (s, 3H), 3.01 (s, 1H, exchanges with D_2O), 3.38 (s, 2H), 4.92 and 5.24 (ABq, 2H, J= 13 Hz); (RO) 2.29 (s, 3H), 3.91 (t, 1H, J= 7 Hz, exchanges with D_2O), 4.41 (s, 2H), 4.72 (d, 2H, J= 7 Hz, s with D_2O); 6.9-8.5 (m, aromatic H's). Peak areas at 3.38 δ(CY) and 4.41 (RO) were used to estimate the tautomeric ratio, IR (KBr) 3400, 1416, 1397, 1094, 1020, 996 cm^{-1}; ($CHCl_3$) 3550, 1666, 1508, 1458, 1017 cm^{-1}. Recrystallisation from diethyl ether provided a sample of 4f, mp 112-114°C.

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References

1. Harron, J.; McClelland, R.A.; Thankachan, C; Tidwell, T.T. J. Org. Chem., 1981, 46, 903.
2. Smith,, J.G.; Wikman, R.T. Tetrahedron, 1974, 30, 2603.
3. (a) Smith, J.G.; Welankiwar, S.S.; Shantz, B.S.; Lai, E.H.; Chu, N.C. J. Org. Chem., 1980, 45, 1817.
(b) Smith, J.G.; Welankiwar, S.S.; Chu, N.G.; Lai, E.H.; Sondheimer, S. ibid., 1981, 46, 4083.
4. Smith, J.G.; Wikman, R.T. ibid. 1974, 39, 3648.
5. Weiss, R. "Organic Syntheses", Collect. Vol. 2; Blatt, A.H., Ed.; Wiley: New York, 1943.
6. Bermann, E.D.; J. Org. Chem., 1956, 21, 461.
7. Blank, O.; Berichte, 1896, 29, 2373.
8. Koelsch, C.F.; J. Org. Chem., 1945, 10, 366.
9. Agranat, I.; Shih, Y.; Syn. Comm., 1974, 4, 119.
10. Bethmann, F.; Berichte, 1899, 32, 1104.
11. Heilmann, E.; Berichte, 1890, 23, 3158.
12. Rukemann, A.; Berichte, 1891, 24, 3965.
13. Schnekenburger, J.; Kaufmann, R. Archiv der Pharmazie, 1970, 303, 760.