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Proton Magnetic Resonance Spectra of Some Naphthalene Derivatives

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SUMMARY

The chemical shifts of the ring protons of some monosubstituted naphthalenes were assigned from 250 MHz spectra, and the assignments were used to assist in the interpretation of the spectra of a number of naphthalenesulfonic acids which are commonly used as dyestuff intermediates. A table of parameters is presented which would assist in the identification of complex azo dyes that are derived from these naphthalene derivatives.

1 INTRODUCTION

Although naphthalene derivatives have been used extensively for many years in the manufacture of dyestuffs, it has been only relatively recently that papers describing the NMR spectral properties of such compounds have been published. Much of the explanation for the paucity of published work in this area prior to 1970 is probably relatable to the absence of high-field NMR equipment and special computer capabilities prior to that time. The purpose of this paper is to consolidate the spectral data published on this subject and to expand that knowledge to include examples of spectra which would be of a more direct interest to dyestuffs chemists concerned with the structure of naphthalene-based synthetic dyestuffs.

In 1970, Ernsley and coworkers[1] published a paper which outlined the chemical shifts and coupling constants for the ring protons of 10 monosubstituted naphthalenes. The spectra of these compounds were

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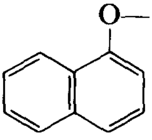
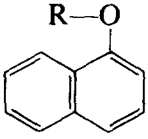
recorded in CCl_4 (5 mol%) using a 100 MHz instrument, and the protons assigned by homonuclear INDOR experiments.[2] Prior to that, only the proton spectra of 1-nitro-[3] and 2-bromonaphthalene[4] had been described in the literature. The ^{1}H-NMR spectral analysis of monosubstituted naphthalenes was later extended[5] to include a series of eight 2-substituted naphthalenes and five additional 1-substituted naphthalenes, also in CCl_4.

It has been shown[6] that the simple theories which may account for the origins of substituent chemical shifts in benzene derivatives cannot be used to predict the chemical shifts for all of the protons in naphthalenes. The authors concluded that attempts to use proton shielding as an indication of molecular structure in naphthalenes must be treated with a degree of caution.

Salman published three interesting papers[7–9] which describe the use of ^{1}H-NMR to determine the conformational preference of certain substituents in the 1- or 2-position of naphthalene. His work showed that a hydroxy or methoxy group, when placed in the α- or β-position of naphthalene, prefers to adopt the conformations **1a/2a** rather than **1b/2b**. In an examination of the preferred conformers of 1- and 2-formylnaphthalene and of 1- and 2-acetylnaphthalene, it was shown that form **3a** predominates in the first pair of isomers, and that the second pair (**4a–4b**) exists as a 1:1 mixture where R = Me, and as predominantly (4:1 ratio) form **4a** where R = H.



1a (*cis*)
R = H, Me



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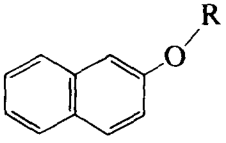

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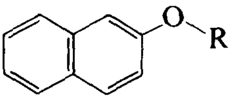
R = H, Me



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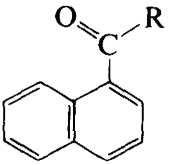
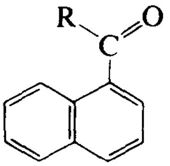
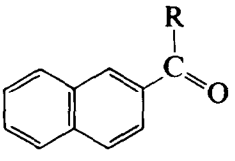


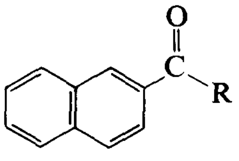
3a
R = H, Me



3b
R = H, Me

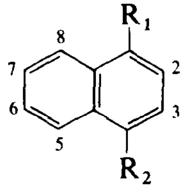


4a
R = H, Me



4b
R = H, Me

The literature contains several papers which describe the ^{1}H-NMR spectra of di- and tri-substituted naphthalenes. Thus, Smith and Chiranjeevi reported[10] the spectra of some symmetrical 1,4-disubstituted naphthalenes (**5**). They noted that an interaction occurs between the α-protons in the 5- and 8-positions and the nitro or hydroxy groups in the 1- and 4-positions, and that variations in chemical shifts for the protons in the naphthalenes studied did not appear to be directly related to the dielectric constant of the NMR solvents employed.



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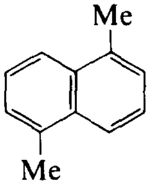
$R_1 = R_2 =$ Me, Cl, Br, OH, NO_2

Lucchini and Wells examined[11] the 100 MHz proton spectra of 3-, 6- and 7-methyl-1-nitronaphthalene in an effort to deduce the nitro-group substituent effect in naphthalene. This work permitted a correction of a previously reported[1] erroneous assignment of protons H-4 and H-5 of 1-nitronaphthalene.

Kim and Anderson studied[12] the 60 MHz proton spectra of 1,5-dimethylnaphthalene (**6**) and its bromo derivatives. An analysis of the spectrum of the parent compound and that of **7** (R = H, Me) permitted the interpretation of the spectra of the bromination products of **6**.



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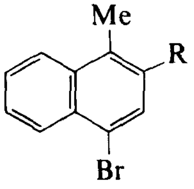

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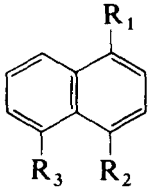
The proton magnetic resonance spectra of some halonitro-substituted naphthalenes (e.g. **8**) have been recorded experimentally and by computer simulation, to generate the chemical shifts and coupling constants for the ring protons.



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8a: $R_1 = Cl, Br; R_2 = NO_2; R_3 = H$
8b: $R_1 = Cl, Br; R_2 = H; R_3 = NO_2$

An INDOR study[13] of the ^1H–^1H inter-ring couplings in 1,2-dichloro-3-nitronaphthalene and in 1,6-dibromo-2-naphthol has permitted the complete analysis of these two naphthalenes. The signs of the inter-ring coupling constants were found to alternate with the number of intervening bonds. In closely related work,[14] methyl substituent effects on ^1H–^1H intra-ring coupling constants in 1,4-, 1,3- and 2,3-dimethylnaphthalene were examined. A negligible effect was found when the Me group occupied positions 2 or 3. Substitution of a Me group in the 1-position caused an increase (+0·22 Hz) in $^3J_{78}$, and a small decrease (−0·12 Hz) in $^3J_{56}$.

Salman has reported[15] the chemical shifts and coupling constants of the ring protons of 11 2,6-disubstituted naphthalenes. Comparisons of the chemical shifts of the protons of these compounds with those observed for 2-methylnaphthalene and naphthalene itself were also made.

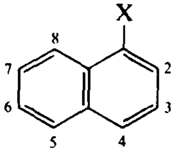
In the present paper, we report expanded tables of the chemical shifts and coupling constants of monosubstituted naphthalenes (in $CDCl_3$) and the proton spectra of nine commonly used naphthalene-based dyestuff intermediates.

2 RESULTS AND DISCUSSION

Tables 1 and 2 give the δ values in ppm for the ring protons of the monosubstituted naphthalenes employed in this investigation. These data resulted from spectra recorded on a 5% solution of each sample in $CDCl_3$ except where indicated. Tetramethylsilane (TMS) was used as the internal reference.

It can be seen from the table of 1-substituted naphthalenes (Table 1) that both ring-activating *and* ring-deactivating substituents cause the *peri* proton (H-8) to lie at lowest field (highest δ value) with the bulky groups (e.g. Cl, Br) and the strongly electron-withdrawing groups (e.g. NO_2, CHO, SO_3H) exhibiting the more pronounced *peri* [X–(H-8)] interactions (cf. Fig. 1).

TABLE 1
Chemical Shifts (δ, ppm) for the Ring Protons of 1-Substituted Naphthalenes



X	δ (*H-2*)	δ (*H-3*)	δ (*H-4*)	δ (*H-5*)	δ (*H-6*)	δ (*H-7*)	δ (*H*-8)
H	7·46	7·46	7·81	7·81	7·46	7·46	7·81
Cl	7·58[a]	7·38	7·77	7·86	7·58	7·58	8·33
Br	7·81[a]	7·32	7·81[a]	7·81[a]	7·57	7·57	8·26
F	7·15	7·40	7·63	7·85	7·54	7·54	8·13
I	8·08[a]	7·19	7·79[a]	7·79[a]	7·54	7·54	8·08[a]
OH	6·78	7·26	7·41	7·79[a]	7·47	7·47	8·19
OMe	6·80	7·38[a]	7·42	7·80	7·48	7·48	8·28
CO_2H	8·42	7·67	8·08	7·91	7·55	7·55	9·10
CHO	8·06	7·59[a]	7·92[a]	7·92[a]	7·59[a]	7·67	9·24
SO_2Cl	8·37	7·59	8·21	8·01	7·69	7·78	8·79
SO_3H[b]	7·96	7·43[a]	8·01[a]	8·01[a]	7·47[a]	7·47[a]	8·80
CH_2OH	7·42[a]	7·42[a]	7·76	7·82	7·42[a]	7·42[a]	8·00
CH_2NH_2	7·41	7·41	7·75	7·86	7·50	7·50	8·05
COCl	8·55[a]	7·54	7·90	7·69	7·67	7·54	8·74
OCOMe	7·23	7·44	7·72	7·85	7·51	7·51	7·85
Me	7·38[a]	7·38[a]	7·68	7·82	7·49	7·49	7·97
Et	7·33	7·38	7·69	7·82	7·48	7·48	8·03
NO_2	8·21	7·52	8·10	7·94	7·60	7·70	8·55
COMe	7·91[a]	7·47	7·91[a]	7·91[a]	7·53[a]	7·53[a]	8·74
CN	8·05	7·50	7·89	7·89	7·58	7·68	8·21
CH_2Cl	7·48	7·39	7·83[a]	7·83[a]	7·57	7·50	8·12
C_5H_6	7·43[a]	7·43[a]	7·87[a]	7·87[a]	7·43[a]	7·43[a]	7·87[a]
$N(CH_3)_2$	7·05	7·37	7·48	7·79	7·45	7·45	8·23
NH_2	6·72	7·29	7·29	7·76	7·41	7·41	7·76
N=NPh	7·85	7·60[a]	8·00	7·94	7·60[a]	7·60[a]	8·96
N=NPh[b]	7·83	7·70[a]	8·18	8·08	7·70[a]	7·70[a]	8·90

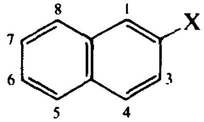
[a] This signal is part of a complex multiplet centered at the δ indicated.
[b] DMSO-d_6 used as solvent.

(Figure 2 depicts the proton spectrum of naphthalene itself as a basis for comparison.) These results are similar to those reported by other workers (in CCl_4 [3,5]) and also suggest that a previous assignment[1] of protons H-2 and H-4 in the spectra of 1-naphthaldehyde and 1-iodonaphthalene was probably in error. We believe that, between the two, the lower-field (higher δ value) proton is correctly assigned as H-2.

It is clear from Table 2 that ring-activating groups cause an upfield (to a

TABLE 2

Chemical Shifts (δ, ppm) for the Ring Protons of 2-Substituted Naphthalenes

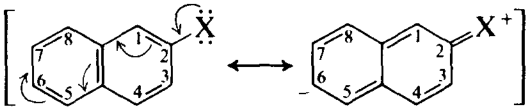


X	δ (*H-1*)	δ (*H-3*)	δ (*H-4*)	δ (*H-5*)	δ (*H-6*)	δ (*H-7*)	δ (*H-8*)
H	7·81	7·46	7·81	7·81	7·46	7·46	7·81
Br	8·00	7·54	7·70	7·78^{a}	7·48	7·48	7·78^{a}
OH	7·12	7·11	7·69^{a}	7·69^{a}	7·35	7·35	7·69^{a}
OMe	7·13^{a}	7·14^{a}	7·74^{a}	7·74^{a}	7·33^{a}	7·43^{a}	7·74^{a}
CO_2H	8·66	8·09	7·92^{a}	7·92^{a}	7·56	7·56	7·96^{a}
CHO	8·31	7·98	7·88	7·92^{a}	7·60	7·60	7·92^{a}
SO_2Cl	8·60	8·01^{a}	8·01^{a}	8·01^{a}	7·71	7·71	8·01^{a}
SO_3H^b	8·19	7·74	7·88^{a}	7·88^{a}	7·51	7·51	7·95
CH_2OH	7·76^{a}	7·41^{a}	7·76^{a}	7·76^{a}	7·41^{a}	7·41^{a}	7·76^{a}
SH	7·68^{a}	7·29	7·68^{a}	7·68^{a}	7·41^{a}	7·41^{a}	7·68^{a}
COCl	8·69	8·06	7·88	7·88	7·62	7·62	7·97
OCOMe	7·54	7·22	7·80^{a}	7·80^{a}	7·45	7·45	7·80^{a}
Me	7·70	7·29	7·75^{a}	7·75^{a}	7·40	7·40	7·75^{a}
Et	7·61	7·33	7·76^{a}	7·76^{a}	7·41	7·41	7·76^{a}
NO_2	8·76	8·21	7·93	7·93	7·65	7·65	8·00
COMe	8·43	8·01	7·86	7·86	7·55	7·55	7·94^{a}
NH_2	6·91	6·88	7·62^{a}	7·56	7·21	7·34	7·67^{a}
N=N—Ph	8·45	8·01^{a}	8·01^{a}	8·01^{a}	7·55^{a}	7·55^{a}	8·01^{a}
N=N—Ph^{b}	8·61	7·98	8·05	8·02	7·65	7·65	8·19

[a] This signal is part of a complex multiplet centered at the δ indicated.

[b] DMSO-d_6 used as solvent.

lower δ value) shift in the position for protons H-1 and H-3 in the 2-substituted system (cf. Fig. 3), as would be anticipated. A ring-deactivating group causes the opposite effect on these two protons. Interestingly, an electron-donating group in the 2-position enhances the magnetic differences between protons H-6 and H-7. This effect is probably explained in terms of the greater negative character of carbon-6 versus carbon-7 (cf. **9**). The net result is an upfield shift (to lower δ) in the position of H-6 relative to H-7 in the spectra of such naphthalenes (cf. Fig. 3).



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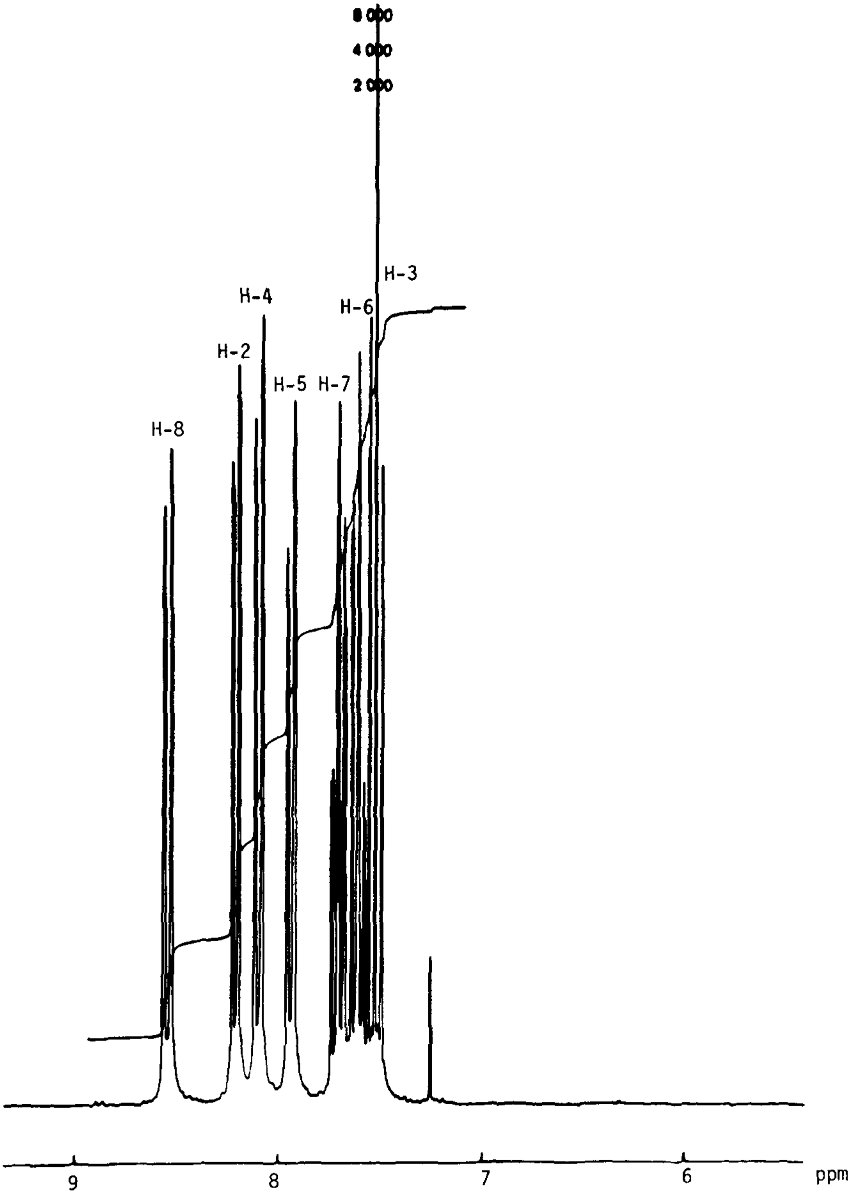


Fig. 1. ^{1}H-NMR spectrum of 1-nitronaphthalene.

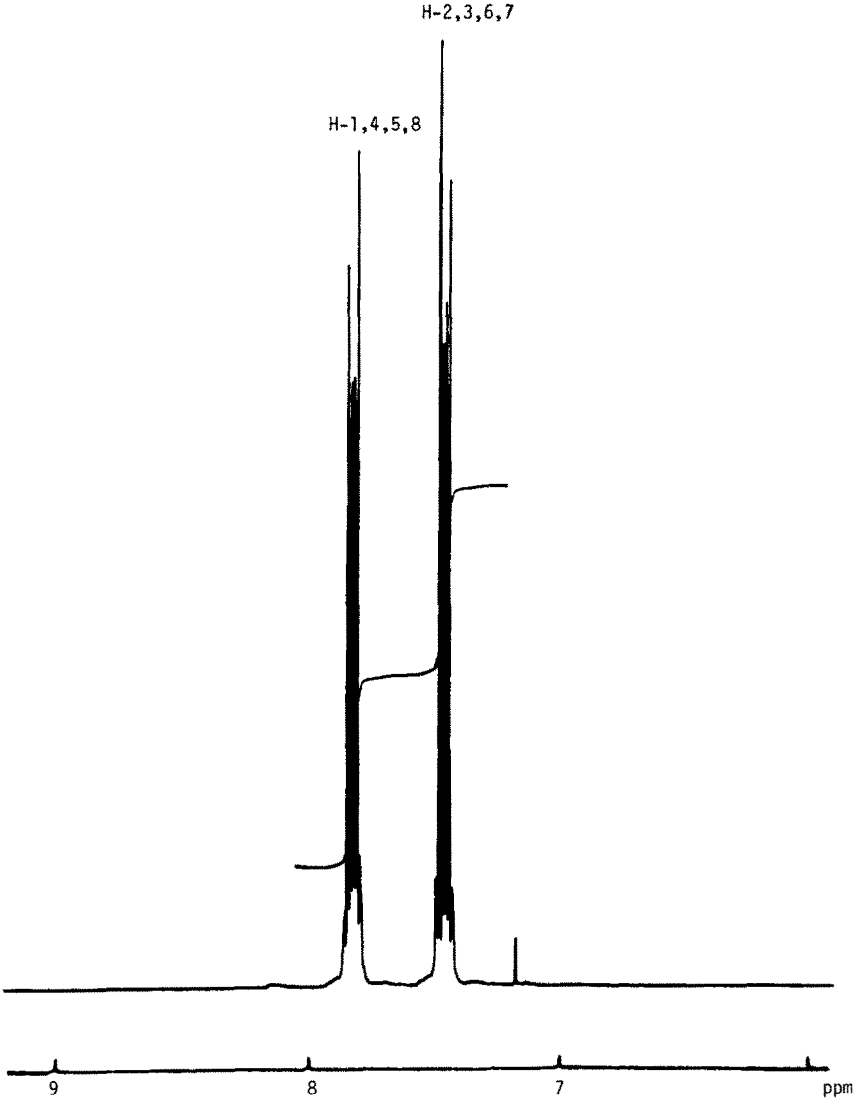


Fig. 2. ^{1}H-NMR spectrum of naphthalene.

Tables 3 and 4 show the changes in the δ values ($\Delta\delta$) of the protons of some monosubstituted naphthalenes relative to the protons of naphthalene itself. Table 5 summarizes briefly the coupling constants observed for the ring protons of the 1- and 2-substituted naphthalenes examined in this study. The range of values reported for each of the various proton couplings is in good agreement with the values in previous reports.[1,5]

The second part of this NMR study has involved the interpretation of the

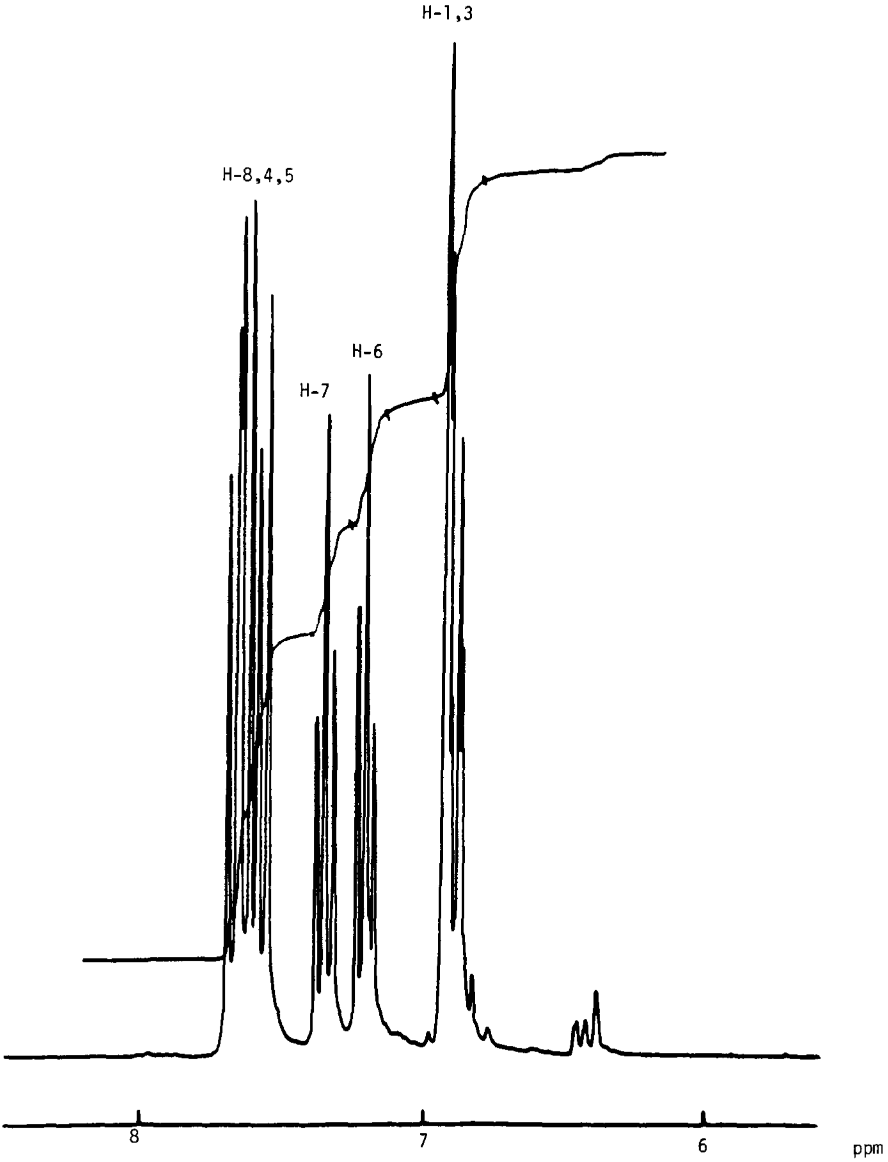
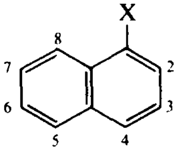


Fig. 3. ^{1}H-NMR spectrum of 2-aminonaphthalene.

proton spectra of some important naphthalene-based dyestuffs intermediates. The 250 MHz spectra of compounds **10**–**18** (cf. Fig. 4) were recorded in DMSO-d_6. The δ values for the ring protons, and the observed J values, are listed in Table 6. As expected, the SO_3H group of S-acid (**16**) shifts the *peri* proton H-5 to a low-field position. The 1,4-substituted naphthalenes **14** and **17** show a 'double' *peri* effect, as both protons H-5 and H-8 are downfield

TABLE 3

Change in δ Value (ppm) of the Ring Protons Produced by Introducing a Substituent into the 1-Position of Naphthalene[a]



X	$\Delta\delta$ (*H-2*)	$\Delta\delta$ (*H-3*)	$\Delta\delta$ (*H-4*)	$\Delta\delta$ (*H-5*)	$\Delta\delta$ (*H-6*)	$\Delta\delta$ (*H-7*)	$\Delta\delta$ (*H-8*)
Cl	0·12	−0·08	−0·04	0·05	0·12	0·12	0·52
Br	0·35	−0·14	0·00	0·00	0·11	0·11	0·45
F	−0·31	−0·06	−0·18	0·04	0·08	0·08	0·33
I	0·62	−0·27	−0·02	−0·02	0·08	0·08	0·29
OH	−0·68	−0·20	−0·40	−0·02	0·01	0·01	0·38
OMe	−0·66	−0·08	−0·39	−0·01	0·02	0·02	0·47
CO_2H	0·96	0·21	0·27	0·10	0·09	0·09	1·29
CHO	0·60	0·13	0·11	0·11	0·13	0·21	1·43
SO_2Cl	0·91	0·13	0·40	0·20	0·23	0·32	0·98
SO_3H[b]	0·50	−0·03	0·20	0·20	0·01	0·01	0·99
CH_2OH	−0·04	−0·04	−0·05	+0·01	−0·04	−0·04	0·19
CH_2NH_2	−0·05	−0·05	−0·06	0·05	0·04	0·04	0·24
COCl	1·09	0·08	0·09	−0·12	0·21	0·08	0·97
OCOMe	−0·23	−0·02	−0·09	0·04	0·05	0·05	0·04
Me	−0·08	−0·08	−0·13	0·01	0·03	0·03	0·16
Et	−0·13	−0·08	−0·12	0·01	0·02	0·02	0·22
NO_2	0·75	0·06	0·29	0·13	0·14	0·14	0·74
COMe	0·45	0·01	0·10	0·10	0·07	0·07	0·93
CN	0·59	0·04	0·08	0·08	0·12	0·12	0·40
CH_2Cl	0·02	−0·07	0·02	0·02	0·11	0·04	0·31
C_6H_5	−0·02	−0·03	0·06	0·06	−0·03	−0·03	0·06
NMe_2	−0·41	−0·09	−0·33	−0·02	−0·01	−0·01	0·42
NH_2	−0·74	−0·17	−0·52	−0·04	−0·05	−0·05	−0·05
N=N—Ph	0·39	0·14	0·19	0·13	0·14	0·14	1·15
N=N—Ph[b]	0·37	0·24	0·33	0·27	0·24	0·24	1·09

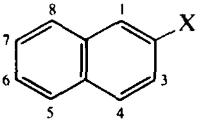
[a] A positive value indicates a downfield shift relative to the TMS peak.

[b] DMSO-d_6 used as solvent.

shifted (cf. Fig. 5). The spectrum of compound **10** (J-acid), by comparison, shows only the expected one signal in the region where the *peri* protons are normally found (cf. Fig. 6). The chemical shifts of the *peri* protons in the spectra of these naphthalenesulfonic acids appear in the positions that would be expected from the δ values of the *peri* protons of the corresponding 1-substituted naphthalenes. For instance, the δ values of the *peri* protons (H-8) of 1-naphthol (δ 8·19) and of 1-naphthalenesulfonic acid (δ 8·80) compare well with protons H-8 (δ 8·15) and H-5 (δ 8·70) of Nevile–Winther acid (**14**).

TABLE 4

Changes in δ Value (ppm) of the Ring Protons Produced by Introducing a Substituent into the 2-Position of Naphthalene[a]



X	Δδ (*H-1*)	Δδ (*H-3*)	Δδ (*H-4*)	Δδ (*H-5*)	Δδ (*H-6*)	Δδ (*H-7*)	Δδ (*H-8*)
Br	0·19	0·08	−0·11	−0·03	0·02	0·02	−0·03
OH	−0·69	−0·35	−0·12	−0·12	−0·11	−0·11	−0·12
OMe	−0·68	−0·32	−0·07	−0·07	−0·13	−0·03	−0·07
CO_2H	0·85	0·63	0·11	0·11	0·10	0·10	0·15
CHO	0·50	0·52	0·07	0·11	0·14	0·14	0·11
SO_2Cl	0·79	0·55	0·20	0·20	0·25	0·25	0·20
SO_3H[b]	0·38	0·28	0·07	0·07	0·05	0·05	0·14
CH_2OH	−0·05	−0·05	−0·05	−0·05	−0·05	−0·05	−0·05
SH	−0·13	−0·17	−0·13	−0·13	−0·05	−0·05	−0·13
COCl	0·88	0·60	0·07	0·07	0·16	0·16	0·16
OCOMe	−0·27	−0·24	−0·01	−0·01	−0·01	−0·01	−0·01
Me	−0·11	−0·17	−0·06	−0·06	−0·05	−0·05	−0·06
Et	−0·20	−0·13	−0·05	−0·05	−0·05	−0·05	−0·05
NO_2	0·95	0·75	0·12	0·12	0·19	0·19	0·19
COMe	0·62	0·55	0·05	0·05	0·09	0·09	0·13
NH_2	−0·90	−0·58	−0·19	−0·25	−0·25	−0·12	−0·14
N=N—Ph	0·64	0·55	0·20	0·20	0·09	0·09	0·20
N=N—Ph[b]	0·80	0·52	0·24	0·21	0·19	0·19	0·38

[a] A positive value indicates a downfield shift relative to the TMS peak.
[b] DMSO-d_6 used as solvent.

TABLE 5

Coupling Constant Recorded for Some Monosubstituted Naphthalenes

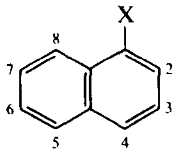
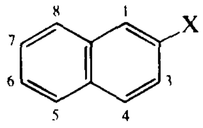
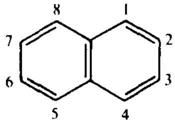
Compound	*Coupling constants* (*Hz*)
	$^3J_{23}$ = 7·0–7·8 $^3J_{34}$ = 7·9–8·3 $^4J_{24}$ = 0·9–1·1 $^3J_{56}$ = 7·2–8·1 $^3J_{67}$ = 7·0 $^3J_{78}$ = 7·6–8·6
	$^4J_{13}$ = 1·6–2·6 $^3J_{34}$ = 8·0–9·1 $^3J_{56}$ = 7·3–8·2 $^3J_{67}$ = 6·9–7·3 $^3J_{78}$ = 7·7–8·3

TABLE 6

Chemical Shifts (ppm) and Coupling Constants (Hz) for the Ring Protons of Some Naphthalene-Based Dyestuffs Intermediates



Compound	*Chemical Shifts*								*Coupling constants*
	H-1	*H-2*	*H-3*	*H-4*	*H-5*	*H-6*	*H-7*	*H-8*	
10 (J-acid)	8·18	7·38	—	7·67	7·83	—	7·22	—	$J_{12} = 8{\cdot}9$
11 (H-acid)	—	7·60	—	7·94	7·64	—	7·25	—	—
12 (Chromotropic acid)	—	7·03	—	7·57	7·57	—	7·03	—	$J_{24} = 1{\cdot}4$
13 (BON acid)	7·36	—	—	8·58	7·98	7·37	7·53	7·78	$J_{56} = 8{\cdot}2$ $J_{56} = 7{\cdot}1$ $J_{78} = 8{\cdot}3$
14 (Nevile–Winther acid)	—	6·82	7·81	—	8·76	7·45	7·45	8·15	$J_{23} = 8{\cdot}0$ $J_{56} = 7{\cdot}6$ $J_{78} = 7{\cdot}9$
15 (γ-acid)	7·96	—	7·36	7·92	7·62	—	7·22	—	$J_{57} = 1{\cdot}3$ $J_{13} = 2{\cdot}0$ $J_{34} = 8{\cdot}7$
16 (S-acid)	—	7·25	7·90	—	8·37	7·37	6·96	—	$J_{23} = 7{\cdot}7$ $J_{56} = 8{\cdot}0$ $J_{67} = 8{\cdot}6$
17 (Naphthionic acid)	—	7·33	7·93	—	8·90	7·60	7·60	7·98	$J_{23} = 7{\cdot}7$
18 (Broenner's acid)	6·95	—	7·03	7·62	7·97	—	7·47	7·62	$J_{34} = 8{\cdot}7$ $J_{78} = 8{\cdot}3$

The chemical shift of proton H-2 of compounds **16** and **17** were found at a higher field than would be anticipated, based on the results of Table 1. The reasons for this shift are not clear at this time, especially since the signal for proton H-3 does appear in the expected position.

It does not appear that the δ values of protons which are under the influence of two groups in naphthalenes can always be predicted by adding the effects of the individual groups. For example, the use of Tables 1 and 2 to determine the expected chemical shift of H-2, H-4, H-5 and H-7 of compound **11** gave the δ values 7·00, 7·67, 7·79 and 7·06, respectively. However, the observed values were 7·60, 7·94, 7·64 and 7·25. By comparison, the observed chemical shift of proton H-3 (δ 7·03) of compound **12** agrees well with the value based on the tables (δ 7·06), but the observed chemical shift of proton H-4 (7·54) is lower than the value (δ 7·79) which would result from using the tables. However, in no case did such deviations from the

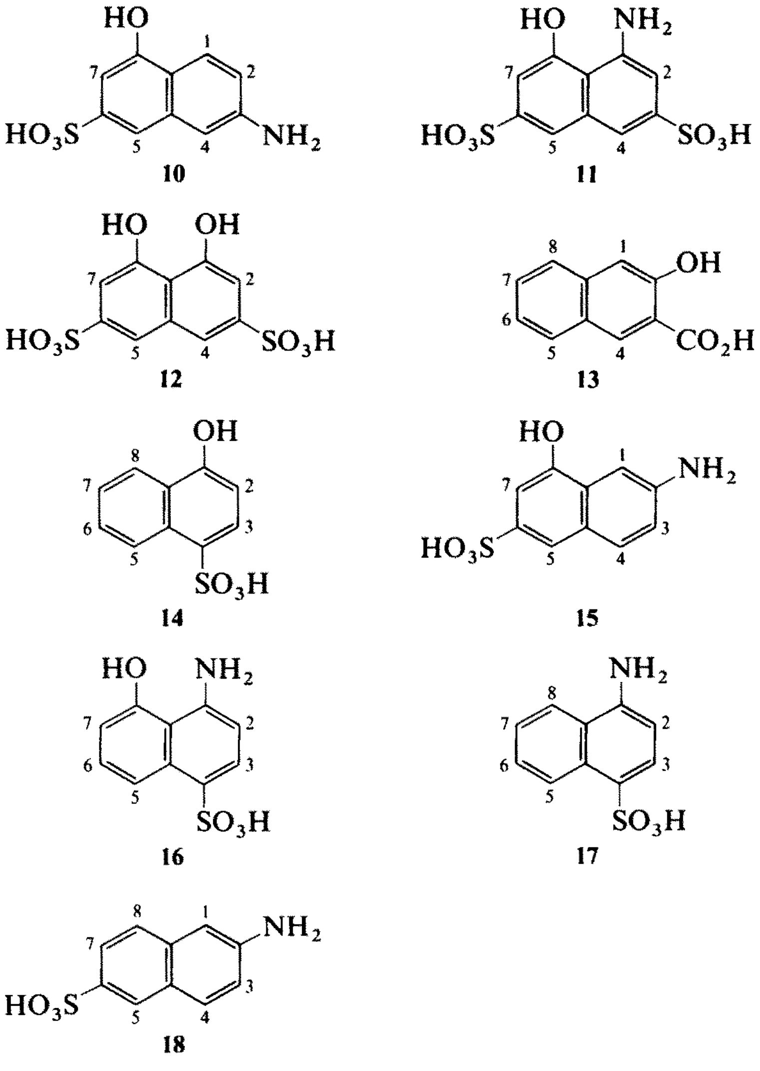


Fig. 4. Naphthalene-based dyestuffs intermediates examined in this investigation.

expected values cause problems in the interpretation of a spectrum, once peak multiplicities and coupling constants were taken into consideration.

3 EXPERIMENTAL

The ^{1}H-NMR spectra were recorded on a Bruker 250 MHz instrument using 5% solutions in $CDCl_3$ or DMSO-d_6. Where available the naphthalene derivatives were obtained from Aldrich Chemicals Co., and were used

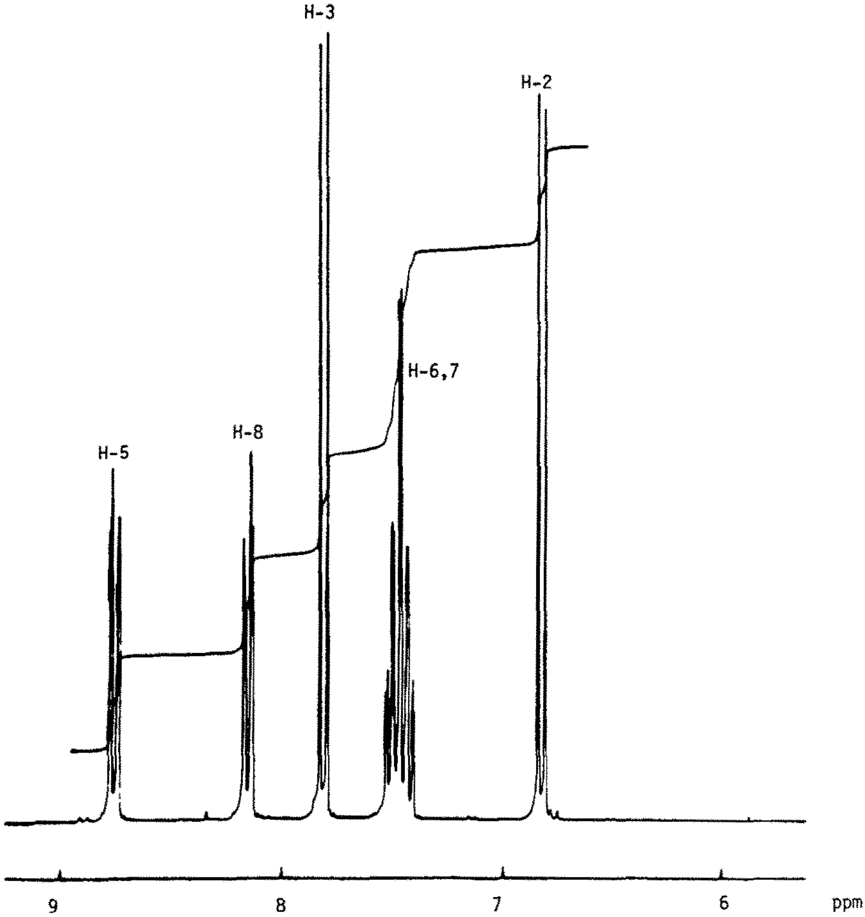


Fig. 5. ^{1}H-NMR spectrum of Nevile–Winther acid.

without further purification. The aminonaphthalenes were prepared in these laboratories by catalytic reduction of the corresponding nitro derivatives. The aminonaphthalenes were, in turn, used to prepare 1- and 2-phenylazonaphthalene via a previously reported route.[16] The two isomers were purified by flash chromatography,[17] followed by recrystallization from 70% EtOH/H_2O.

4 CONCLUSION

It has been shown that whilst data on the proton magnetic resonance spectra of monosubstituted naphthalenes serve as a useful guide for the interpretation of the spectra of more complex naphthalene derivatives, it is

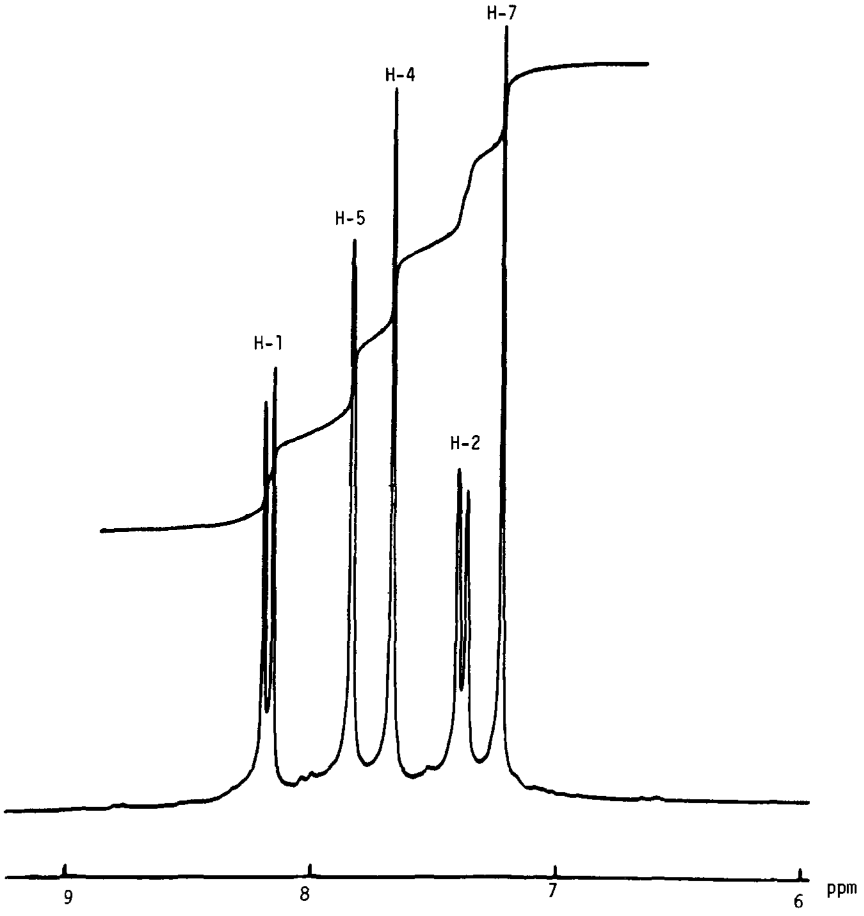


Fig. 6. ^{1}H-NMR spectrum of J-acid.

not always possible to use that information to predict the chemical shifts of the naphthalene ring protons which are under the influence of two or more ring substituents. We believe, however, that the tables presented provide general trends which should permit the assignment of the protons of typical naphthalene-based dyestuffs.

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