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DIPOLE MOMENTS OF SUBSTITUTED 1-NAPHTHOIC ACIDS AND METHYL 1-NAPHTHOATES

T. FUJITA, K. KOSHIMIZU and T. MITSUI
Department of Agricultural Chemistry, Kyoto University, Kyoto, Japan

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Abstract—The dipole moments of the 1-naphthoic acids and methyl 1-naphthoates substituted at various positions by a halogen or nitro group were measured in solution. The results are analysed in terms of the conformation of the carboxyl group, in particular, the rotational isomerism between planar *s-cis* and *s-trans* conformation of the carboxyl group in the 3-, 4-, 5- and 6-substituted derivatives.

RECENTLY, we have reported that the UV spectra of variously substituted 1-naphthoic acids can be well accommodated by a simple LCAO-molecular orbital calculation assuming a coplanar structure of the molecule unless a substituent is located at one of the vicinal positions to the carboxyl group (2- and 8-positions).[1] However, in 1-naphthoic acid, which can be regarded as an *ortho* substituted benzoic acid, the *ortho* substituent, i.e., the *peri* methine group in this case, is reasonably considered to exert some steric effects on the 1-carboxyl group. In fact, Trotter has shown by X-ray analysis that the strain in a coplanar model for the molecule is relieved by 11°-twisting of the carboxyl group out of the plane of the naphthalene ring and by valancy-angle distortions.[2] Although these structural deformations could be neglected as a first approximation as far as the MO calculation is concerned, it is still unknown whether the "almost coplanar" 1-naphthoic acid exists as only one structure or as an equilibrium mixture of *s-cis* (I) and *s-trans* (II) conformers in solution due to a steric effect on the internal rotation of the carboxyl group. The purpose of this paper is to make clear this point by analyses of the dipole moments of the substituted 1-naphthoic acids and 1-naphthoates.

The dipole moment $\bar{\mu}$ of the acids and the reference compounds were determined at 25° in dioxan solution as shown in Table 1. The carboxylic acids have been considered to exist as monomer in dioxan solution.[3] The dipole moment values of the halo- and nitro-naphthalenes in dioxan are a little larger than those in benzene (cf. Table 3). That of benzoic acid is in good agreement with the earlier reported value.[3] The other compounds listed have not been measured before. The moment values of the bromo compounds are not much different from the corresponding chloro compounds.

In the evaluation of the moments of the *s-cis* and *s-trans* models (i) the molecule is assumed to be coplanar, (ii) the moment of the carboxyl group is taken as that of benzoic acid, 1·76D, and its moment angle to the carboxyl-C-C_1 axis is assumed as 74° which has been determined from the moments of the halogeno-benzoic acids in dioxan,[4] and (iii) the other component moment is taken as that of the respective

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[1] T. Fujita, K. Koshimizu and T. Mitsui, *Tetrahedron* **22**, 1587 (1966).

[2] J. Trotter, *Acta Cryst.* **13**, 732 (1960).

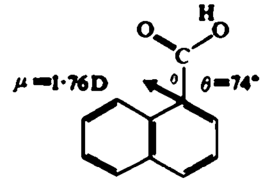
[3] C. S. Brooks and M. E. Hobbs, *J. Am. Chem. Soc.* **62**, 2851 (1940).

[4] M. E. Hobbs and A. J. Weith, Jr., *J. Am. Chem. Soc.* **65**, 967 (1943).

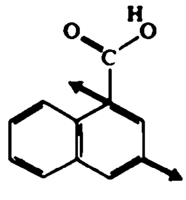
TABLE 1. DIPOLE MOMENTS OF SUBSTITUTED 1-NAPHTHOIC ACIDS AND RELATED COMPOUNDS IN DIOXAN AT 25°

							Dipole Moment		
						$P_E + P_A$	obsd.	calcd. for	
Compounds[a]	ε_1[b]	d_1[b]	a[b]	b[b]	$P_{2\infty}$(cc)[b]	(cc)[b]	$\bar{\mu}$(D)	$\mu_{s\text{-}cis}$	$\mu_{s\text{-}trans}$
1-NA	2·2152	1·0274	2·67	0·203	114·20	50·80	1·76 ± 0·02		
2-Cl–1-NA	2·2167	1·0274	4·62	0·262	199·88	55·63	2·66 ± 0·02	2·65[c]	
3-Cl–1-NA	2·2153	1·0274	2·29	0·248	121·66	55·63	1·80 ± 0·02	0·43	3·23
4-Cl–1-NA	2·2158	1·0274	2·61	0·260	131·84	55·63	1·93 ± 0·02	2·04	2·04
5-Cl–1-NA	2·2149	1·0275	3·26	0·252	154·36	55·63	2·20 ± 0·02	2·04	2·04
6-Cl–1-NA	2·2150	1·0274	4·81	0·255	206·80	55·63	2·72 ± 0·01	3·23	0·43
8-Cl–1-NA	2·2161	1·0275	4·70	0·266	202·38	55·63	2·68 ± 0·01	2·70[c]	
3-Br–1-NA	2·2147	1·0266	1·88	0·387	121·53	58·51	1·76 ± 0·03	0·43	3·22
4-Br–1-NA	2·2133	1·0271	2·22	0·390	135·33	58·51	1·94 ± 0·02	2·04	2·04
5-Br–1-NA	2·2138	1·0268	2·74	0·390	156·83	58·51	2·19 ± 0·03	2·04	2·04
8-Br–1-NA	2·2133	1·0272	3·64	0·400	193·28	58·51	2·57 ± 0·02	2·71[c]	
3-NO_2–1-NA	2·2078	1·0269	8·55	0·310	348·48	57·25	3·77 ± 0·01	2·80	5·83
4-NO_2–1-NA	2·2089	1·0269	9·23	0·290	373·86	57·25	3·94 ± 0·01	3·78	3·78
5-NO_2–1-NA	2·2088	1·0268	8·62	0·305	351·19	57·25	3·79 ± 0·01	3·78	3·78
6-NO_2–1-NA	2·2082	1·0269	15·00	0·308	579·45	57·25	5·05 ± 0·01	5·83	2·80
8-NO_2–1-NA	2·2088	1·0269	11·33	0·302	448·32	57·25	4·37 ± 0·01	4·67[c]	
1-Cl–N	2·2139	1·0266	2·40	0·145	103·34	49·43	1·62 ± 0·02		
2-Cl–N	2·2137	1·0266	2·73	0·141	112·35	49·43	1·75 ± 0·02		
1-Br–N	2·2139	1·0267	1·92	0·304	106·28	52·31	1·63 ± 0·03		
2-Br–N	2·2147	1·0267	2·14	0·300	113·99	52·31	1·74 ± 0·03		
1-NO_2–N	2·2075	1·0267	11·17	0·203	357·75	51·05	3·87 ± 0·02		
2-NO_2–N	2·2080	1·0268	14·57	0·201	459·93	51·05	4·47 ± 0·01		
BA	2·2181	1·0272	3·34	0·155	96·00	32·65	1·76 ± 0·02		

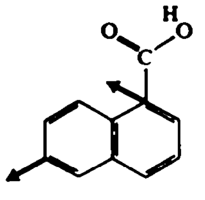
[a] Abbreviations: NA: naphthoic acid, N: naphthalene, BA: benzoic acid. [b] See experimental part. [c] Calcd. value as of 90° conformer.



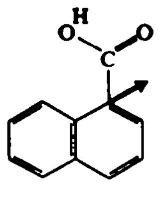
I: *s-cis*



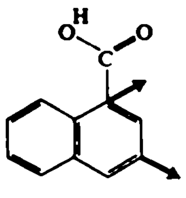
III: 3-*cis*



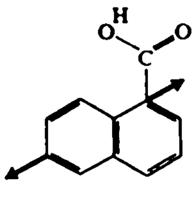
V: 6-*cis*



II: *s-trans*



IV: 3-*trans*



VI: 6-*trans*

reference substituted naphthalene directing at regular hexagonal angle. The calculated moment values are also listed in Table 1. If a model where the carboxyl group is twisted by 11° from the ring plane is assumed, the calculated values are not markedly different.

The significant difference between observed and calculated values, especially those of the 3- and 6-substituted acids, suggests that the 1-naphthoic acids are equilibrium mixtures of two conformers. If the proportion of the *s-cis* conformer is taken to be x, the apparent moment values of the 3- and 6-substituted acids ($\bar{\mu}_3$ and $\bar{\mu}_6$) can be calculated by Eqs 1 and 2.

$$\bar{\mu}_3^2 = \mu_{3\text{-}cis}^2\, x + \mu_{3\text{-}trans}^2(1 - x) \tag{1}$$

$$\bar{\mu}_6^2 = \mu_{6\text{-}cis}^2\, x + \mu_{6\text{-}trans}^2(1 - x) \tag{2}$$

Summing up Eqs 1 and 2 and substitutions of $\mu_{3\text{-}cis} = \mu_{6\text{-}trans}$ and $\mu_{3\text{-}trans} = \mu_{6\text{-}cis}$ into the resultant equation yield Eq. 3,

$$\bar{\mu}_3^2 + \bar{\mu}_6^2 = \mu_{3\text{-}trans}^2 + \mu_{6\text{-}trans}^2 \tag{3}$$

where suffixes, 3-*cis*, etc, refer to the conformers, III to VI. In the chloro acids, $\bar{\mu}_3 = 1{\cdot}80$D, $\bar{\mu}_6 = 2{\cdot}72$D and $\mu_{6\text{-}trans} = 0{\cdot}43$D. In the nitro acids, $\bar{\mu}_3 = 3{\cdot}77$D, $\bar{\mu}_6 = 5{\cdot}05$D and $\mu_{6\text{-}trans} = 2{\cdot}80$D.

From these equations and values, the proportion of *s-cis* conformer, x, and the moment of *s-trans* confromer of 3-substituted acids, $\mu_{3\text{-}trans}$, can be calculated. If the potential energy of the *s-trans* form is assumed to be higher than that of *s-cis* form, the energy difference, ΔE, between the two conformers can be estimated according to Eq. 4.

$$\exp(-\Delta E/RT) = (1 - x)/x \tag{4}$$

The moment angle, θ, of the carboxyl group with respect to the short axis of naphthalene can be determined from Eq. 5,

$$\mu_{3\text{-}trans}^2 = \mu_{6\text{-}cis}^2 = \mu^2 + \mu_s^2 + 2\mu\mu_s \cos(120° - \theta) \tag{5}$$

where μ is taken to be 1·76D and μ_s as that of 2-chloro- or 2-nitronaphthalene. The results are recorded in Table 2. The moment values thus estimated for $\mu_{3\text{-}trans}$ (or $\mu_{6\text{-}cis}$) isomers conform to those calculated from the model by vector addition and the moment angle is quite consistent with that of the benzoic acids, especially in the chloro

TABLE 2. RESULTS OF CALCULATIONS BY EQS 1–5

	Proportion of *s-cis* form; x	$\mu_{3\text{-}trans}$ (= $\mu_{6\text{-}cis}$)		Energy difference ΔE; $E_{s\text{-}trans} - E_{s\text{-}cis}$	Moment angle with respect to the short axis of naphthalene; θ calcd. by Eq. 5.
		from Eq. 3.	from vector addition		
From the 3- and 6-Cl-acids	0·70	3·23D	3·23D	507 cal/mole	73°45′
From the 3- and 6-NO_2-acids	0·74	5·65D	5·83D	605 cal/mole	64°05′

acids. The proportion of *s-cis* conformer and the potential energy difference evaluated from the chloro acids are in very good agreement with those from the nitro acids.

The value of $\mu_{s\text{-}trans}$ derived from the nitro acids is somewhat smaller than that from a simple vector addition, and consequently the moment angle θ is calculated as a smaller value than that from the chloro acids. These are probably because the observed moment values of the nitro acids are smaller than those expected from Eqs 1 and 2. The carboxyl group moment would be affected by a strong electron attracting power of the nitro group and *vice versa* (mostly inductive effect), expecially in the 3-nitro acid. The direct resonance effect of the substituents would not be significant in these compounds because the 3- and 6-substituents cannot resonate directly with the 1-carboxyl group.

The induced polarization effect of a substituent on the unsubstituted ring of the two mutually condensed rings of naphthalene has been suggested to be a source of difference between the observed moments of 1- and 2-substituted naphthalene.[5,6] Since the induced moment may vary with the direction of the substituent group moment, the dipole moment and the moment angle of the carboxyl group in 1-naphthoic acid would be different for the two conformers. However, even if this effect is taken into account, the above discussions about the conformational equilibrium of the 3- and 6-substituted 1-naphthoic acids would not be seriously affected, since the induced moment values of the carboxyl group are expected to be small and not much different for the two conformers and the angles of β-halogen–C and β-NO_2–C moments to the short axis of naphthalene are not markedly varied from 60°.[5,7]

The fact that the moment of 4-halo acid is smaller than that of 5-halo acid probably follows from the fact that the induced polarization effect of α-halogen on the unsubstituted ring cannot be neglected in this case, i.e., the C-halogen moment at the α-position is inclined to the short axis of naphthalene.[5] Using the observed moment values of the 4- and 5-chloro acids and assuming that the proportion of *s-cis* conformer is the same as that in the 3- and 6-chloro acids and that the moment angle of the C–Cl dipole is inclined at 9° as suggested for 1-chloronaphthalene,[8] the moment angle of the carboxyl group is estimated to be 76° which is in good agreement with that calculated from the 3- and 6-chloro acids.

In the 4- and 5-nitro acids, it could be reasonably assumed that C–NO_2 moment at the α-position is inclined similarly to the C–halogen moment in the 4- and 5-halo acids.[7] However the same procedure as above cannot be applied in this case, because the apparent moment of the 4-nitro acid is larger than that of the 5-nitro acid of which cause is uncertain. The electron attracting effect of the 4-nitro group on the 1-carboxyl has been suggested to be much stronger than that of the 5-nitro group by pK_A studies.[9] In this situation, however, the moment of the 4-nitro acid should be smaller since the groups are competing for electrons.

Although the dipole moment values of 2- and 8-chloro acids are in agreement with those calculated as the twisted conformer at 90°, and those of the 8-bromo and 8-nitro acids are less than those of the 90° conformer, one cannot conclude from the

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[5] G. C. Hampson and A. Weissberger, *J. Chem. Soc.* 393 (1936).

[6] T. Shimozawa and Y. Morino, *J. Chem. Soc. Japan* (Pure Chem. Section) **81**, 20 (1960).

[7] Ref. 6, recalculated.

[8] Ref. 5, recalculated.

[9] E. Berliner and E. H. Winicov, *J. Am. Chem. Soc.* **81**, 1630 (1959).

moment values more than that the carboxyl group is twisted considerably out of the ring plane as indicated previously by UV spectra and MO calculations because of the strong induced polarization effect between two vicinal substituents and the reduced mesomeric moment of the carboxyl and nitro group caused by the twisting.

A quite similar conclusion can be obtained from the moment values of the chloro-1-naphthoates in benzene solution at 25° recorded in Table 3. Thus, if $\mu_{3\text{-}cis}$ ($= \mu_{6\text{-}trans}$) is taken as the difference between two moments, those of methyl 1-naphthoate and 2-chloronaphthalene in nearly opposite directions, 0·1D, the proportion of *s-cis* conformer, x, the moment of *s-trans* conformer of the 3-chloroester, the energy difference ΔE and moment angle of carbomethoxy group, θ, can be calculated according to Eqs 1–5; $x = 0{\cdot}76$, $\mu_{3\text{-}trans} = \mu_{6\text{-}cis} = 2{\cdot}97$D, $\Delta E = 685$ cal/mole and $\theta = 54°05'$. From this x-value and the moment values of the 4-chloro and the 5-chloro esters, the moment angle of carbomethoxy group, θ, is estimated as 60°55′ when the C–Cl dipole is inclined at 9° with respect to the short axis of naphthalene. From these figures, the moment angle of the carbomethoxy group is considered to be around 60°.[10]

TABLE 3. DIPOLE MOMENTS OF SUBSTITUTED 1-NAPHTHOATES AND RELATED COMPOUNDS IN BENZENE[a]

Compounds	Temp	ε_1	d_1	a	b	$P_{2\infty}$ (cc)	$P_E + P_A$ (cc)	$\bar{\mu}$(D)
1-NA–CH_3 ester	10°	2·3032	0·8883	2·31	0·220	126·07	55·57	1·81 ± 0·02
1-NA–CH_3 ester	25°	2·2737	0·8724	2·19	0·224	123·92	55·57	1·83 ± 0·02
1-NA–CH_3 ester	40°	2·2435	0·8564	2·04	0·227	120·59	55·57	1·83 ± 0·02
2-Cl-CH_3 ester	10°	2·3044	0·8890	3·44	0·271	190·40	60·40	2·46 ± 0·01
2-Cl-CH_3 ester	25°	2·2744	0·8731	3·17	0·269	183·55	60·40	2·45 ± 0·02
2-Cl–CH_3 ester	40°	2·2440	0·8571	2·96	0·274	178·98	60·40	2·47 ± 0·02
3-Cl–CH_3 ester	10°	2·3032	0·8883	1·31	0·289	103·41	60·40	1·41 ± 0·02
3-Cl-CH_3 ester	25°	2·2742	0·8725	1·27	0·288	103·18	60·40	1·45 ± 0·02
3-Cl–CH_3 ester	40°	2·2434	0·8563	1·27	0·287	104·65	60·40	1·51 ± 0·02
3-Br–CH_3 ester	10°	2·3037	0·8883	1·115	0·369	106·70	63·28	1·42 ± 0·02
3-Br–CH_3 ester	25°	2·2742	0·8723	1·091	0·375	106·06	63·28	1·45 ± 0·01
3-Br–CH_3 ester	40°	2·2439	0·8564	1·077	0·369	107·12	63·28	1·50 ± 0·02
3-NO_2–CH_3 ester	10°	2·3035	0·8889	6·17	0·315	310·56	62·02	3·40 ± 0·01
3-NO_2–CH_3 ester	25°	2·2745	0·8731	5·78	0·305	302·41	62·02	3·43 ± 0·01
3-NO_2–CH_3 ester	40°	2·2435	0·8570	5·63	0·313	302·95	62·02	3·52 ± 0·01
4-Cl–CH_3 ester	25°	2·2741	0·8731	1·48	0·281	112·23	60·40	1·59 ± 0·02
5-Cl–CH_3 ester	25°	2·2743	0·8730	1·91	0·275	130·80	60·40	1·86 ± 0·02
6-Cl–CH_3 ester	25°	2·2743	0·8731	3·54	0·285	197·52	60·40	2·59 ± 0·01
1-Cl–N	25°	2·2791	0·8770	1·96	0·240	99·72	49·43	1·57 ± 0·02
2-Cl–N	25°	2·2793	0·8769	2·21	0·234	107·71	49·43	1·69 ± 0·01
1-Br–N	25°	2·2794	0·8769	1·56	0·363	101·68	52·31	1·55 ± 0·02
2-Br–N	25°	2·2797	0·8769	1·80	0·360	111·21	52·31	1·70 ± 0·02
1-NO_2–N	25°					358·99[b]	51·05	3·88 ± 0·01
2-NO_2–N	25°					445·80[b]	51·05	4·39 ± 0·01

[a] Abbreviations and notations are the same as Table 1.

[b] Ref. 6.

Furthermore, the temperature dependence of dipole moment of the 3-substituted

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[10] cf. C. P. Smyth; *Dielectric Behavior and Structure* p. 330. McGraw-Hill, New York (1955) and A. R. Katritzky, E. W. Randall and L. E. Sutton, *J. Chem. Soc.* 1773 (1957).

esters supports the above conclusion. As shown in Table 3, the apparent moments of the 3-substituted esters increase with temperature. Because the moment value of *s-cis* conformer is clearly smaller than that of *s-trans* conformer, the temperature dependence, $d\bar{\mu}/dT > 0$, means that the increasing temperature shifts the equilibrium in favor of the *s-trans* conformer which is less stable than the *s-cis* conformer.

The apparent dipole moment $\bar{\mu}$ can be calculated by Eq. 6, which is derived from Eqs 1 and 4, or Eq. 7.

$$\bar{\mu}^2 = x\mu^2_{3\text{-}cis} + (1 - x)\mu^2_{3\text{-}trans} = \frac{\mu^2_{3\text{-}cis} + \mu^2_{3\text{-}trans}\exp(-\Delta E/RT)}{1 + \exp(-\Delta E/RT)} \tag{6}$$

$$\bar{\mu}^2 - \mu^2_{3\text{-}cis} = \frac{\mu^2_{3\text{-}trans} - \mu^2_{3\text{-}cis}}{1 + \exp(\Delta E/RT)} \tag{7}$$

In Eq. 7, $\mu_{3\text{-}cis}$ is taken as 0·1D (3-halo-1-naphthoate) and 2·6D (3-nitro-1-naphthoate). Using these values for the *s-cis* conformers and the apparent moment values $\bar{\mu}$ observed at various temperatures, the energy differences ΔE and the moments, $\mu_{3\text{-}trans}$, of the *s-trans* conformers are calculated according to the method proposed.[11] The results are shown in Table 4. The moments, $\mu_{3\text{-}trans}$, agree within experimental error with those calculated from the *s-trans* model. The fact that the energy differences ΔE are larger than those estimated for the acids may be attributable to difference of bulkiness between OH and OMe groups.

TABLE 4. ENERGY DIFFERENCE BETWEEN TWO CONFORMERS

	ΔE (cal/mole)	$\mu_{3\text{-}trans}$	$\mu_{3\text{-}trans}$ calcd. by vector addition*
3-Halo ester	850 ± 300	3·3 ± 0·4D	3·05D
3-Nitro ester	1000 ± 300	5·3 ± 0·9D	5·54D

* The moment angle of carbomethoxy group to the short axis of the naphthalene ring is taken to be 60°.

The moment of methyl 2-chloronaphthoate is little affected by temperature. This is not unexpected because, in this compound, the carbomethoxy group is twisted considerably from the ring plane and cannot take part in the equilibrium between two conformers whose dipole moments are greatly different from each other.

Thus, the steric effect of *peri*-CH group on the internal rotation of the carboxyl group is revealed as a difference of potential energy between two nearly coplanar conformers in solution and the carboxyl group is not appreciably twisted until a second group is introduced into a vicinal position.

EXPERIMENTAL

Measurement. The dielectric constant ε was measured with a heterodyne beat apparatus[12] and density d with a Lipkin-type pycnometer.[13] From these values the dipole moment $\bar{\mu}$ was calculated

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[11] J. E. Lennard-Jones and H. M. Pike, *Trans. Faraday Soc.* **30**, 845 (1934).

[12] Y. Morino and I. Miyagawa, *Kagaku-no-Ryoiki* (J. Japan. Chemistry), Extra No. 8 (1953).

[13] M. R. Lipkin, J. A. Davison, W. T. Harvey and S. S. Kurtz, Jr., *Ind. Eng. Chem., Anal. Ed.* **16**, 55 (1944).

TABLE 5. SUBSTITUTED METHYL 1-NAPHTHOATE

Esters	M.p. from petr. ether and benzene[a]	Analysis[b] Calcd. C%	H%	Found C%	H%
Methyl-2-Cl-1-naphthoate	50–51° (50°)[c]		—		
Methyl-3-Cl-1-naphthoate	54–55°	65·32	4·11	65·49	4·14
Methyl-4-Cl-1-naphthoate	47–48°	65·32	4·11	65·85	4·38
Methyl-5-Cl-1-naphthoate	65–66°	65·32	4·11	65·44	3·94
Methyl-6-Cl-1-naphthoate	68–69°	65·32	4·11	65·56	4·49
Methyl-3-Br-1-naphthoate	62–63° (59°)[d]		—		
Methyl-3-NO_2-1-naphthoate	139-140°	62·34	3·92	62·38	4·48

[a] All m.ps are uncorrected. Values in parentheses are literature values.

[b] The analyses were by Miss Y. Oishi.

[c] E. Bergmann and J. Hirshberg, *J. Chem. Soc.* 331 (1936).

[d] H. G. Rule and S. B. Thompson, *J. Chem. Soc.* 1764 (1937).

according to a modified Hedestrand method similar to that of Halverstadt and Kumler,[14] differing in that densities were used rather than specific volumes. The notations in Tables 1 and 3 are the same as those used in a previous paper.[15] $P_E + P_A$ was taken to be equal to the molecular refraction calculated from the bond refractivities[16] and refraction exaltation.[17] The value of the exaltation which should be added to the sum of the bond refractivities in the naphthalene system was taken as 2·55 cc which is the value reported for naphthalene itself,[17] and the group refractivity of the nitro group as 8·13 cc which is estimated from the molecular refractivities of benzene and nitrobenzene and toluene and *p*-nitrotoluene.[18]

Compounds. The acids in this work are the same samples described in a previous paper.[1] The esters were made using diazomethane in ether or dioxan soln. Their m.ps are recorded in Table 5. Benzoic acid and halo- and nitro-naphthalenes were purified by recrystallization or distillation of commercial samples.

Acknowledgment—The authors wish to express their sincere thanks to Dr. Takashi Shimozawa for suggestive discussions.

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