JOURNAL OF PHYSICAL ORGANIC CHEMISTRY
J. Phys. Org. Chem. 2000; **13:** 203–207

Solvolysis of 2-aryl-2-adamantyl *p*-nitrobenzoates and some tertiary benzylic *p*-nitrobenzoates. Y_{BnOPNB} and Y_{xBnOPNB} scales

Kwang-Ting Liu,[1]* Chih-Wei Chang,[1] Hung-I Chen,[1] Chien-Pu Chin[1] and Yeh-Fang Duann[2]

[1]Department of Chemistry, National Taiwan University, Taipei 106, Taiwan
[2]Chemical Engineering Department, National Taipei University of Technology, Taipei 104, Taiwan

Received 23 August 1999; revised 19 November 1999; accepted 22 November 1999

ABSTRACT: Solvolysis rate constants for a number of tertiary benzylic *p*-nitrobenzoate esters in a wide range of solvents were measured. New scales of solvent ionizing power for Grunwald–Winstein-type correlation analyses, Y_{BnOPNB} from 2-phenyl-2-adamantyl *p*-nitrobenzoate and Y_{xBnOPNB} from 2-(6-methoxy-2-naphthyl)-2- adamantyl *p*-nitrobenzoate, were developed for the mechanistic study of solvent effects on the solvolytic reactivity of benzylic *p*-nitrobenzoates. The excellent linear log k –Y_{BnOPNB} plot for 2-(4-methylphenyl)-2-propyl *p*-nitrobenzoate indicated a limiting S_N1 mechanism. The solvolytic transition state for 9-phenyl-9-fluorenyl *p*-nitrobenzoate and that for 1,1-diphenylethyl *p*-nitrobenzoate were considered to involve extended positive charge delocalizations from the observation of linear relationships between log k and Y_{xBnOPNB}. The ambiguity in the case of 2, 2-dimethyl-1-(4-methoxyphenyl)-1-phenyl-1-propyl *p*-nitrobenzoate is discussed. Copyright © 2000 John Wiley & Sons, Ltd.

KEYWORDS: solvolysis; Grunwald–Winstein-type analysis; Y_{BnOPNB} scale; Y_{xBnOPNB} scale; tertiary benzylic *p*-nitrobenzoates

INTRODUCTION

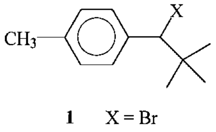
Solvent effects on reactivity have long been considered as an important factor affecting solvolytic mechanisms (for a comprehensive review of solvent effects, see Ref. 1). Correlation analyses of empirical solvent parameters on the reactivity with single- or dual-parameter equations [Eqns (1) and (2)][2,3] are generally employed as a diagnostic tool for identifying S_N1 pathways with or without nucleophilic solvent intervention (for discussion see, e.g., Ref. 4):

$$\log(k/k_0) = mY \tag{1}$$

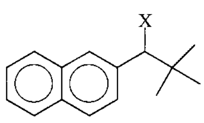
$$\log(k/k_0) = mY + lN \tag{2}$$

Several 1- and 2- substituted adamantanes have been used as reference standards for establishing individual Y_x scales of the solvent ionizing power to correlate the reactivity of a substrate RX containing a specific leaving group X (for compilations of Y_X scales, see Refs 5 and 6). However, no Y scale for *p*-nitrobenzoate has ever been developed, owing to the low reactivity and/or low solubility of 1- and 2-adamantyl derivatives in commonly used solvent systems (the solvolysis rate constant for 1-adamantyl *p*-nitrobenzoate in 60% ethanol at 75 °C was found to be 2.5×10^{-8} s^{-1}.)[7] (T. W. Bentley, personal communication, and unpublished results from this laboratory).

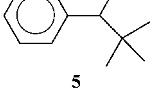
On the other hand, in our recent studies on the solvolysis of benzylic substrates, new Y_{BnX} scales for benzylic bromides,[8] chlorides[9] and *p*-nitrobenzoates[10] were established based on the solvolytic rate constants of corresponding 2-aryl-2-adamantyl derivatives. Later work yielded more Y_{BnBr}[11] and Y_{BnCl}[12] values from the solvolysis of 1-(4-methylphenyl)-1-*tert*-butylmethyl bromide (**1**) and chloride (**2**), respectively. Moreover, we found it necessary to develop new $Y_{x\mathrm{BnBr}}$[13] and Y_{xBnCl},[14] which were derived from the logarithms of solvolytic rate constants (log k) for α-*tert*-butyl(2-naphthyl)methyl bromide (**3**) and chloride (**4**), respectively, for substrates with extended positive charge delocalization in the



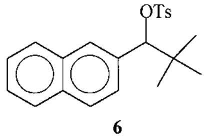
1 X = Br
2 X = Cl



3 X = Br
4 X = Cl



5

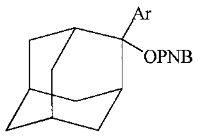


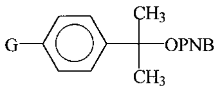
6

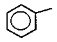
**Correspondence to:* K.-T. Liu, Department of Chemistry, National Taiwan University, Taipei 106, Taiwan, Republic of China.

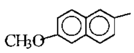
transition state. The corresponding Y_{BnOTs} values for tosylates were determined from 1-*tert*-butyl-1-phenylmethyl tosylate (**5**).[15] For tosylates which might give a cationic transition state having charge delocalization over more than one phenyl ring, such as 9-fluorenyl tosylate,[16] the log k for the solvolysis of α-*tert*-butyl(2-naphthyl)-methyl tosylate (**6**)[17] could be considered as the reference standard in the correlation analysis.

p-Nitrobenzoate esters belong to a category of substrate attracting continuous interest for solvolytic studies.[18] In a preliminary communication,[10] a number of Y_{BnOPNB} values were established from rate data for 2-phenyl-2-adamantyl *p*-nitrobenzoate (**7**). Its advantage could be demonstrated by the detection of a significant nucleophilic solvent intervention in the solvolysis of 2-phenyl-2-propyl *p*-nitrobenzoate (**8**) but not in the case of the more reactive 2-(4-methylphenyl)-2-propyl *p*-nitrobenzoate (**9**), from the observed non-linear log k – Y_{BnOPNB} relationship and other supporting evidence.[19] It is therefore desirable to explore the possibility of developing a new $Y_{xBnOPNB}$ scale for studying solvent effect on the reactivity of *p*-nitrobenzoate esters with extended positive charge delocalization in the solvolytic transition state. In this paper, we present the results of a study employing 2-(6-methoxy-2-naphthyl)-2-adamantyl *p*-nitrobenzoate (**10**) in this concern. More Y_{BnOPNB} values are also be reported.





7 Ar = 

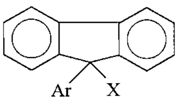
10 Ar = 

8 G = H

9 G = CH_3

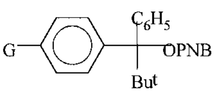
RESULTS

2-Phenyl-2-adamantyl *p*-nitrobenzoate (**7**), 2-(6-methoxy-2-naphthyl)-2-adamantyl *p*-nitrobenzoate (**10**), 9-phenyl-9-fluorenyl *p*-nitrobenzoate (**11**) and 2, 2-dimethyl-1-(4-methoxyphenyl)-1-phenyl-1-propyl *p*-nitrobenzoate (**12**) were prepared from the reaction of *p*-nitrobenzoyl chloride with the lithium salt of the corresponding alcohols, which had been synthesized by the Grignard addition of an arylmagnesium halide to the appropriate ketone. Elemental analyses and spectral data were found to be in harmony with the individual structures. 2-(4-Methylphenyl)-2-propyl *p*-nitrobenzoate (**9**)[20] and 1,1-diphenylethyl *p*-nitrobenzoate (**13**)[20] were prepared according to the literature method. Rate constants of the solvolysis for *p*-nitrobenzoates **7** and **10–13** were measured conductimetrically, and in some cases titrimetrically. Pertinent results and the literature data for **9**[19] are listed in Table 1.



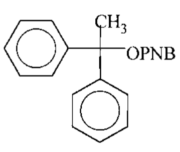
11 X = OPNB , Ar = C_6H_5

16 X = Cl

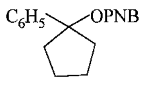


12 G = OCH_3

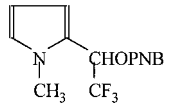
17 G = CH_3



13



14



15

Similarly to the original Y_{BnBr}[8] and Y_{BnCl}[9] scales, the solvent ionizing power for the solvolysis of benzylic *p*-nitrobenzoates, the Y_{BnOPNB} scale (Table 2), could be defined from the log k of **7** using Eqn. (1). Table 3 gives the results of regression analyses of log k values for **9**[19] and **10–13** against Y_{BnOPNB}. Since the corresponding *p*-nitrobenzoate to **1** and **2** would have too low a reactivity to be used practically as a reference standard to define the analogous Y scale for an extended delocalized system, a substrate resembling **7** would be suitable to serve as a reference standard for the new $Y_{xBnOPNB}$ scale. 2-(6-Methoxy-2-naphthyl)-2-adamantyl *p*-nitrobenzoate (**10**) was the choice because the presence of a 6-methoxy group would exhibit a higher reactivity and would increase its solubility in a binary solvent system with a relatively high water content. Indeed, all of the rate constants for **10** were determined directly at 25 °C (Table 1). The calculated $Y_{xBnOPNB}$ values are also listed in Table 2. The correlation analysis for **9** and **11–13** using Eqn. (1) against $Y_{xBnOPNB}$ was carried out and the results are given in Table 4.

DISCUSSION

Although scales of solvent ionizing power have been developed for aliphatic (Y_X), benzylic (Y_{BnX}) and extended benzylic (Y_{xBnX}) systems for common leaving groups, including bromide (X = Br), chloride (X = Cl) and tosylate (X = OTs), the corresponding scales for *p*-nitrobenzoate ester (X = OPNB) were established only for benzylic and extended benzylic systems in this work. Non-benzylic *p*-nitrobenzoates of the adamantane system were found to be inconvenient for the kinetic determination of Y values due to their low solubility in binary solvents of high water content and low reactivity in other nucleophilic solvents[7] (T. W. Bentley, personal communication, and unpublished results from this laboratory). Other carboxylate esters, such as 1-adamantyl trifluoroacetate, had been studied[21] and the corresponding Y_x values were calculated.[5] However, the variation of

Table 1. Pertinent rate constants for solvolysis of *p*-nitrobenzoates

Solvent[a]	k (s^{-1} (25 °C)					
	7	9[d]	10	11	12	13
100E	1.63×10^{-7b}	7.23×10^{-7}	7.76×10^{-7b}	3.93×10^{-7b}	1.15×10^{-4}	1.29×10^{-6b}
90E	9.96×10^{-7b}	5.09×10^{-6}	7.71×10^{-6b}	2.55×10^{-6b}	3.49×10^{-4}	5.25×10^{-6b}
80E	3.73×10^{-6b}	1.68×10^{-5}	3.04×10^{-5}	5.95×10^{-6b}	1.39×10^{-3}	1.25×10^{-5}
70E	9.66×10^{-6b}		7.89×10^{-5}	1.25×10^{-5}		2.84×10^{-5}
60E	2.26×10^{-5}		1.82×10^{-4}	2.78×10^{-5}		
90A			1.36×10^{-7b}	6.04×10^{-8b}		1.00×10^{-7b}
80A	3.05×10^{-7b}	1.54×10^{-6}	1.08×10^{-6b}	3.46×10^{-7b}	1.40×10^{-4}	7.64×10^{-7b}
70A	1.74×10^{-6b}	6.58×10^{-6}	8.02×10^{-6b}	1.29×10^{-6b}	7.80×10^{-4}	3.18×10^{-6b}
60A	8.56×10^{-6b}	3.10×10^{-5}	3.64×10^{-5}	3.91×10^{-6b}		1.37×10^{-5}
50A	2.16×10^{-5}		1.67×10^{-4}	1.30×10^{-5}		
100M	1.51×10^{-6b}	5.18×10^{-6}	1.65×10^{-5b}	3.60×10^{-6b}		
90M	6.69×10^{-6b}	2.11×10^{-5}	5.96×10^{-5}	1.19×10^{-5}	1.88×10^{-3}	
80M	2.02×10^{-5}	5.46×10^{-5}	1.87×10^{-4}	3.02×10^{-5}	2.84×10^{-3}	3.59×10^{-5}
70M	6.36×10^{-5}		7.03×10^{-4}	7.24×10^{-5}		
100T	1.55×10^{-3}	1.01×10^{-3}	1.16×10^{-2}	1.66×10^{-3}	3.00×10^{-2}	1.14×10^{-3}
80T20E	1.77×10^{-4}	2.03×10^{-4}	1.39×10^{-3}	3.25×10^{-4}	1.15×10^{-2}	2.72×10^{-4}
60T40E	3.81×10^{-5}		2.41×10^{-4}	6.39×10^{-5}		
40T60E	4.74×10^{-6b}	1.16×10^{-5}	3.41×10^{-5}	1.05×10^{-5}		
70Tw[c]	1.07×10^{-3}	1.18×10^{-3}	9.59×10^{-3}	1.69×10^{-3}	1.61×10^{-2}	8.07×10^{-4}

[a] Solvents: A = acetone, E = ethanol, M = methanol, T = 2,2,2-trifluoroethanol. The numbers denote the volume percentage of the specific solvent.
[b] Extrapolated from data obtained at other temperatures.
[c] Weight percent.
[d] Ref. 19.

difference between Y values of individual solvent for various sulfonate esters[5] suggested that the extrapolation of the Y scale for trifluoroacetate to *p*-nitrobenzoate would be unsuitable. Although a linear relationship between Y_{OTs} and log k [Eqn. (1)] for 1-methyl-1-cyclopentyl *p*-nitrobenzoate (**14**) was observed,[10] the correlation analysis for other aliphatic *p*-nitrobenzoates was found to be in less satisfactory (unpublished results from this laboratory). The observed linear relationship for 1-(1-methyl-2-pyrrolyl)-2,2,2-trifluoroethyl *p*-nitrobenzoate (**15**), $\log k = 0.56Y_{\mathrm{OTs}}–3.36$ ($r = 0.990$),[22] is probably due to the limited variety of solvents, lacking aqueous acetone and trifluoroethanol–ethanol, employed in the solvolytic study. For solvolyses of diphenylmethyl (benzhydryl) *p*-nitrobenzoate in aqueous ethanol and trifluoroethanol, the observed linear log k –Y_{OTs} plot ($r = 0.998$)[23] might also be interpreted similarly. In addition, log k –Y_{OTs} plots for **9–13** gave only a poor correlation ($r = 0.87–0.94$).

Table 2 gives two sets of Y values, the revised and expanded Y_{BnOPNB} scale based on the rate constants of 2-phenyl-2-adamantyl *p*-nitrobenzoate (**7**) and the new Y_{xBnOPNB} scale derived from those of 2-(6-methoxy-2-naphthyl)-2-adamantyl *p*-nitrobenzoate (**10**). The usefulness of the former scale can be illustrated by the results of regression analyses shown in Table 3, in which the excellent linear log k –Y_{BnOPNB} relationship for 2-(4-methylphenyl)-2-propyl *p*-nitrobenzoate (**9**) is in line

Table 2. Y_{BnOPNB} and Y_{xBnOPNB} scales

Solvent[a]	Y_{BnOPNB}[b]	Y_{xBnOPNB}[c]
100E	−1.36	−1.18
90E	−0.573	−0.421
80E	0.00	0.00
70E	0.413	0.306
60E	0.782	0.605
90A		−1.99
80A	−1.09	−1.21
70A	−0.331	−0.606
60A	0.361	−0.095
50A	0.763	0.427
100M	−0.393	−0.275
90M	0.254	0.189
80M	0.734	0.645
70M	1.23	1.02
100T	2.62	2.05
80T20E	1.68	1.44
60T40E	1.01	0.818
40T60E	0.104	0.173
70T*w*	2.46	1.84

[a] For abbreviation of solvents, see Table 1.
[b] Based on log k for **7**.
[c] Based on log k for **10**.

Table 3. Correlation analyses of log k against Y_{BnOPNB}

Parameter	9	10	11	12	13
n	13	18	18	10	11
m	0.784	0.833	0.967	0.589	0.793
σ[a]	0.019	0.034	0.046	0.035	0.039
r	0.996	0.987	0.982	0.986	0.988

[a] Standard deviation.

Table 4. Correlation analyses of log *k* against Y_{xBnOPNB}

Parameter	9	11	12	13
n	13	19	10	12
m	0.919	1.13	0.698	0.970
σ^a	0.043	0.028	0.042	0.025
r	0.979	0.995	0.986	0.998

[a] Standard deviation.

with other evidence to demonstrate the absence of nucleophilic solvent intervention in the solvolysis.[19] In the case of 9-phenyl-9-fluorenyl *p*-nitrobenzoate (**11**), the solvolytic transition state might involve benzylic or a more extended positive charge delocalization. The observation of a less satisfactory ($r = 0.982$) correlation against Y_{BnOPNB} than that against Y_{xBnOPNB} ($R = 0.995$ in Table 4) suggested a more effective delocalization within the 9-fluorenyl ring. The difference is also depicted in Figs 1 and 2. A similar conclusion has already been reached in the solvolysis of 9-aryl-9-chlorofluorenes (**16**), from correlation analysis of log *k* versus Y_{xBnCl} using Eqn. (1), and *ab initio* calculation of atomic charges for the corresponding cation.[24] Moreover, in Fig. 1 the data points measured in aqueous acetone (closed circles) are in one line ($r = 0.996$) below another line defined by all other points (open circles) ($r = 0.997$). It is similar to the log k–Y_{BnX} plots for **3**[13] and **4**.[14] Consequently, a recent proposal on the steric and hydrophobic perturbation of solvation in the solvolysis of aliphatic and alicyclic systems[25] would not be applicable in the present work.

The solvolysis of 2, 2-dimethyl-1-(4-methoxyphenyl)-1-phenyl-1-propyl *p*-nitrobenzoate (**12**) is more interesting. In spite of the presence of two aryl rings, a phenyl and a 4-methoxyphenyl, the data in Tables 3 and 4 gave nearly the same correlation for log $k - Y_{\text{BnOPNB}}$ and log $k - Y_{\text{xBnOPNB}}$ plots. Probably in this highly congested system the two aryl rings in the cationic transition state are unlikely to be coplanar, and in consequence the delocalization of positive charge over both rings would be less effective. The results of Grunwald–Winstein-type analyses might not be conclusive. Furthermore, the behavior of the 4-methylphenyl analogue (**17**) was found likewise.[10,26]

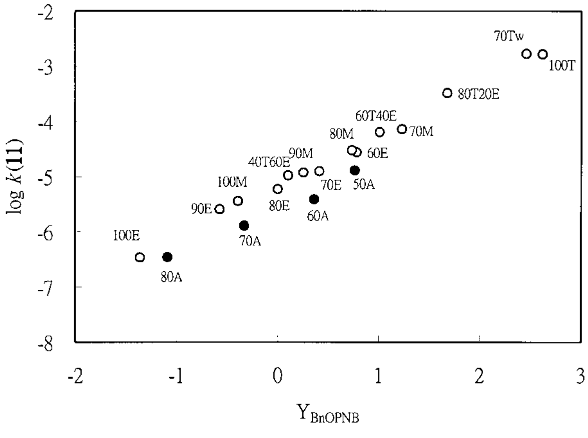


Figure 1. Correlation of log *k* for **11** against Y_{BnOPNB}. Abbreviations for solvents as in Table 1

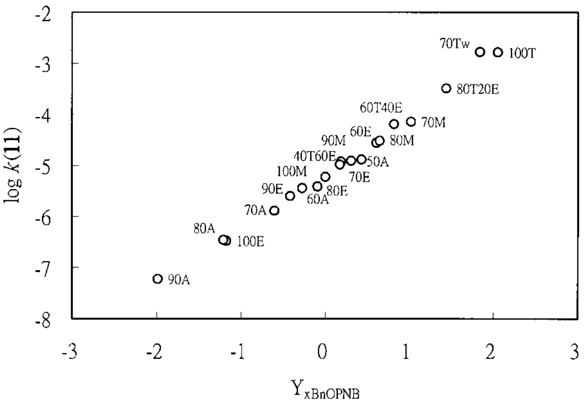


Figure 2. Correlation of log *k* for **11** against Y_{xBnOPNB}. Abbreviations for solvents as in Table 1

However, the performance of the less crowded 1,1-diphenylethyl *p*-nitrobenzoate (**13**) is different. The excellent linear correlation of log *k* against Y_{xBnOPNB} ($r = 0.997$) and the observed high *m* value (0.970) in Table 4 indicated a limiting S_N1 process and an extended delocalization in the cationic transition state was possible. In other words, the replacement of the *tert*-butyl group by a significantly less bulky methyl group makes the formation of a planar or nearly planar transition state more likely. For the solvolysis of diphenylmethyl *p*-nitrobenzoate,[22,27] only five rate constants in aqueous ethanol and aqueous trifluoroethanol were available for the correlation analysis with Y_{xBnOPNB}, and a linear relationship ($r = 0.970$) was found. Unfortunately, the available data points are too few for a meaningful correlation using the dual-parameter Eqn. (2).

In conclusion, the two new scales of solvent ionizing power developed in the present work provided suitable solvent parameters for studying the solvolytic mechanism of tertiary *p*-nitrobenzoate esters. An excellent linear log $k - Y_{\text{BnOPNB}}$ plot suggested solvolysis involving a transition state having a delocalized positive charge within a simple benzylic system, while extended charge delocalization could be proposed from the observation of a linear log $k - Y_{\text{xBnOPNB}}$ relationship.

EXPERIMENTAL

Capillary melting points were uncorrected. Proton and carbon-13 NMR spectra were measured on a Bruker Model AC-200 or AM-300 instrument using tetramethylsilane as internal standard. IR spectra were taken using a Perkin-Elmer Model 983G spectrometer. Elemental analyses were performed by the Microanalytical Laboratory at the National Taiwan University.

Materials. Reagent-grade solvents and chemicals were used for general purposes. The *p*-nitrobenzoate esters were prepared by the use of conventional methods, and their NMR and IR spectra were in agreement with the assigned structures. Elemental analyses and melting points are as follows. 2-Phenyl-2-adamantyl *p*-nitrobenzoate (**7**), m.p. 183–184 °C. Analysis: calculated for $C_{23}H_{23}NO_4$, C 74.37, H 6.24; found, C 74.25, H 6.08%. 2-(4-Methylphenyl)-2-propyl *p*-nitrobenzoate (**9**), m.p. 95.5–96.5 °C (lit.[20] 96–97 °C). 2-(6-Methoxy-2-naphthyl)-2-adamantyl *p*-nitrobenzoate (**10**), m.p. 185–186 °C. Analysis: calculated for $C_{28}H_{27}NO_5$, C 72.51, H 5.95, N 3.06; found C 72.42, H 6.01, N 3.04%. 9-Phenyl-9-fluorenyl *p*-nitrobenzoate (**11**), m.p. 218–219 °C. Analysis: calculated for $C_{26}H_{17}NO_4$, C 76.65, H 4.21, N 3.24; found, C 76.60, H 4.40, N 3.26%. 2,2-Dimethyl-1-(4-methoxyphenyl)-1-phenyl-1-propyl *p*-nitrobenzoate (**12**), m.p. 180 °C (decomp.). Analysis calculated for $C_{25}H_{25}NO_4$, C 71.23, H 6.23, N 3.32; found, C 71.25, H 6.05, N 3.31%. 1,1-Diphenylethyl *p*-nitrobenzoate (**13**), m.p. 135 °C (decomp.) (lit.[20] 135 °C, decomp.).

Spectral- or reagent-grade solvents were purified according to the standard procedure[28] for kinetic studies. Doubly deionized water was used to prepare aqueous solvent systems for solvolysis.

Kinetic measurements.[9b] Conductimetric rate constants were measured at least in duplicate for general solvolytic studies. The conductivity cells containing a solution of 1×10^{-4}–1×10^{-5} M were placed in a thermostat with a temperature variation of about ± 0.02 °C. The maximum error of k is $\pm 3\%$. The potentiometric titration method was used for measurements at 85 °C or higher temperatures. Rate constants monitored at other temperatures were extrapolated to those at 25 °C by using an Arrhenius plot. The conventional method[29] was employed for statistical analysis of the rate data.

Acknowledgements

We are indebted to the National Science Council for financial support of this research.

REFERENCES

1. Reichard C. *Solvents and Solvent Effects in Organic Chemistry* (2nd edn). VCH: Weinheim, 1988.
2. Grunwald E, Winstein S. *J. Am. Chem. Soc.* 1948; **70**: 846.
3. Winstein S, Grunwald E, Jones HW. *J. Am. Chem. Soc.* 1951; **73**: 2700.
4. Lowry TH, Richardson KS. *Mechanism and Theory in Organic Chemistry* (3rd edn). Harper and Row: New York, 1987; 335–349.
5. Bentley TW, Llewellyn G. *Prog. Phys. Org. Chem.* 1990; **17**: 121.
6. (a) Kevill DN and Hawkinson DC. *J. Org. Chem.* 1990; **55**: 4304; (b) Kevill DN, Kyong JB, Weitl FL. *J. Org. Chem.* 1990; **55**: 5394.
7. Bentley TW, Christl M, Norman SJ. *J. Org. Chem.* 1991; **56**: 6238.
8. Liu K-T, Sheu H-C. *J. Chin. Chem. Soc. (Taipei)* 1991; **38**: 29.
9. (a) Liu K-T, Sheu H-C, Chen H-I, Chiu P-F, Hu C-R. *Tetrahedron Lett.* 1990; **31**: 3611; (b) Liu K-T, Sheu H-C. *J. Org. Chem.* 1991; **56**: 3021.
10. Liu K-T, Chen H-I, Chin C-P. *J. Phys. Org. Chem.* 1991; **4**: 463.
11. Liu K-T, Chin C-P, Yu D-G, Duann Y-F. *J. Chin. Chem. Soc. (Taipei)* 1999; **46**: 789.
12. Liu K-T, Chang L-W, Yu D-G, Chen P-S, Fan J-T. *J. Phys. Org. Chem.* 1997; **10**: 879.
13. Liu K-T, Chin C-P, Lin Y-S, Tsao M-L. *J. Chem. Res. (S)* 1997; 18.
14. Liu K-T, Lin Y-S, Tsao M-L. *J. Phys. Org. Chem.* 1998; **11**: 223.
15. Liu K-T, Yang J-S, Chang S-M, Lin Y-S, Sheu H-C, Tsao M-L. *J. Org. Chem.* 1992; **57**: 3041.
16. Liu K-T, Lin Y-S. *Tetrahedron Lett.* 1995; **38**: 1419.
17. Liu K-T, Hsu H-Y, Yang J-S. *Tetrahedron Lett.* 1992; **33**: 3327.
18. Kusuyama Y. *Bull. Chem. Soc. Jpn.* 1998; **71**: 685.
19. Liu K-T, Chang L-W, Chen P-S. *J. Org. Chem.* 1992; **57**: 4791.
20. Brown HC, Ravindranathan M, Peters ED. *J. Org. Chem.* 1977; **42**: 1073.
21. Bentley TW, Roberts K. *J. Chem. Soc. Perkin Trans. 2* 1989; 1055.
22. Allen AD, Kwong-Chip J-M, Lin WC, Nguyen P, Tidwell TT. *Can. J. Chem.* 1990; **68**: 1709.
23. McLennan DJ, Martin PL. *J. Chem. Soc. Perkin Trans. 2* 1982; 1091.
24. Liu K-T, Lin Y-S, Tsao M-L. *Org. React.* 1997; **31**: 59.
25. Takeuchi K. *Pure Appl. Chem.*, 1998; **70**: 2023.
26. Chin C-P. PhD Thesis. National Taiwan University, 1996.
27. McLennan DJ, Martin PL. *Aust. J. Chem.* 1979; **32**: 2361.
28. Perrin DD, Armarego WLF. *Purification of Laboratory Chemicals* (3rd edn). Pergamon Press: New York, 1988.
29. Miller JC, Miller JN. *Statistics for Analytical Chemistry* (2nd edn). Wiley: New York, 1988.