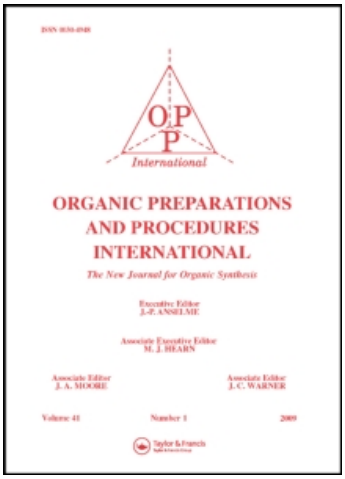
This article was downloaded by:
On: *26 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

A SIMPLE AND EFFICIENT PROCEDURE FOR THE BECKMANN REARRANGEMENT OF OXIME USING *bis*-(TRICHLOROMETHYL) CARBONATE/DMF

W. K. Su[a]; Y. Zhang[a]; J. J. Li[a]; P. Li[a]
[a] Zhejiang Key Laboratory of Pharmaceutical Engineering, College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou, PR CHINA

To cite this Article Su, W. K. , Zhang, Y. , Li, J. J. and Li, P.(2008) 'A SIMPLE AND EFFICIENT PROCEDURE FOR THE BECKMANN REARRANGEMENT OF OXIME USING *bis*-(TRICHLOROMETHYL) CARBONATE/DMF', Organic Preparations and Procedures International, 40: 6, 543 — 550
To link to this Article: DOI: 10.1080/00304940809458118
URL: http://dx.doi.org/10.1080/00304940809458118

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

ORGANIC PREPARATIONS AND PROCEDURES INT., 40 (6), 543-550 (2008)

A SIMPLE AND EFFICIENT PROCEDURE FOR THE BECKMANN REARRANGEMENT OF OXIME USING *bis*-(TRICHLOROMETHYL) CARBONATE/ DMF

W. K. Su,* Y. Zhang, J. J. Li and P. Li

College of Pharmaceutical Sciences, Zhejiang University of Technology
Zhejiang Key Laboratory of Pharmaceutical Engineering
Hangzhou, 310014, P. R. CHINA
E-mail: suweike@zjut.edu.cn

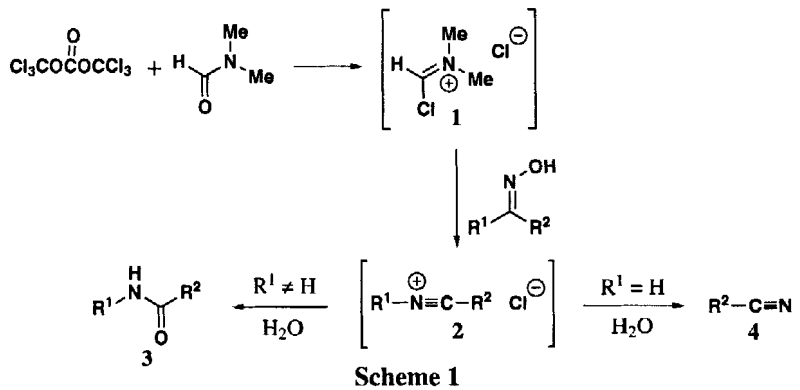
Downloaded At: 17:45 26 January 2011

The Beckmann rearrangement of ketoximes to amides is a powerful method both in organic synthesis and chemical manufacturing;[1] the reaction generally requires high temperature and large amounts of strong Brønsted acid.[1] As a result, mild conditions have been sought and procedures have been reported using supercritical water,[2] vapor-phase processes,[3] and ionic liquids.[4] However, there are many limitations with these methods; for example, supercritical water cannot be widely used and the reaction[5] in vapor-phase generally requires high reaction temperature. Thus the use of sensitive substrates is usually impossible. Although, the reaction conditions are usually mild in ionic liquid, some of the reagents are very expensive. Therefore, the development of a green, simple and cost-effective catalytic system for the Beckmann rearrangement is in great demand.

bis-(Trichloromethyl) carbonate (BTC, triphosgene) has emerged as a versatile auxiliary for the synthesis of some important class of organic compounds,[5] and its use has proved to be safe and advantageous over that of phosgene. Vilsmeier reagents generated from DMF and $POCl_3$ (PCl_5, P_2O_5) have been widely used in acylation, chlorination, chloroformylation, aromatization, annulation and so on.[6] As a part of our ongoing program directed to the development of Vilsmeier reagent using BTC/DMF,[7] we found this type of Vilsmeier reagent[8] could also be used for the Beckmann rearrangement with satisfactory results under mild conditions (*Scheme 1*). Jochims *et al.*[9] have reported that oximes react with oxalyl chloride in the presence of Lewis acid to give nitrilium salts which then rearrange. Compared with this method, our procedure is much easier to control and more environmentally friendly. Acetophenone oxime (mixture of E/Z isomers) was used as a substrate to test the feasibility of BTC/DMF system as Vilsmeier reagent for the Beckmann rearrangement. The reagent **1** (*Scheme 1*) was easily prepared by dissolving BTC in acetonitrile followed by the dropwise addition of DMF to the mixture at 0°C. Then,

© 2008 by Organic Preparations and Procedures Inc.

acetophenone oxime was added-in one portion and the mixture was refluxed for 1 hour. After completion of the reaction as monitored by TLC, acetanilide (**3a**) was purified by column chromatography in 90% yield.



Scheme 1

Various reaction conditions were investigated and the results are summarized in *Table 1*. To determine the most appropriate solvent, the reaction was examined using toluene, CH_2Cl_2, $CHCl_3$, THF, CH_3CN and DMF; CH_3CN was the best solvent (*Table 1, Entry 6*) in terms of yields. As shown in *Table 1*, it was also found that the reaction was facilitated by increasing the

Table 1. Beckmann Rearrangement of Acetophenone Oxime using BTC/DMF under Different Conditions.[a]

Entry	Solvent	BTC/DMF (mmol/mmol)	Reaction temp (°C)	Reaction time (h)	Yield[b,c] of **3a** (%)
1	toluene	1/3	reflux	1	45
2	CH_2Cl_2	1/3	reflux	1	60
3	$CHCl_3$	1/3	reflux	1	78
4	THF	1/3	reflux	1	53
5	DMF	1/3	reflux	1	0
6	CH_3CN	1/3	reflux	1	90
7	CH_3CN	1/3	60	1	66
8	CH_3CN	1/3	40	1	45
9	CH_3CN	1/3	20	1	0
10	CH_3CN	1/3	reflux	0.5	73
11	CH_3CN	1/3	reflux	2	91
12	CH_3CN	1/1	reflux	2	48
13	CH_3CN	1/6	reflux	2	20
14	CH_3CN	0.5/1.5	reflux	2	63

a) Reaction conditions: acetophenone oxime (3 mmol); solvent (2 mL). b) **3a**: acetanilide. c) Isolated yield.

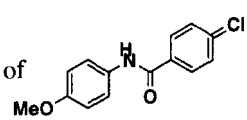
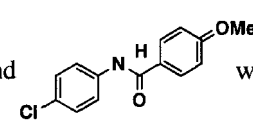
Downloaded At: 17:45 26 January 2011

temperature; under reflux in CH_3CN a high yield was obtained in a relative short time. The reaction did not take place at room temperature (*Table 1, Entry 9*). Furthermore the ratio effect of BTC/DMF was studied (*Table 1, Entries 11-14*). It seemed that either an excess of DMF or the absence of DMF reduce the yields, the probable reason being that a less reactive adduct was formed in excess of DMF[8] and there was no Vilsmeier reagent formed in the absence of DMF. Therefore, the best results were obtained by using 3: 1: 3 mole ratio of oxime: BTC: DMF in acetonitrile as solvent under reflux temperature.

Based on the above results, a series of ketoximes were examined to evaluate the scope of this procedure (*Table 2*). In each case, ketoximes were smoothly transformed to the corresponding amides thin 1 to 6 hours in moderate to high yields. It was shown that aryl ketoximes

Table 2. Conversion of Ketoximes into Amides under BTC/DMF System.[a]

Entry	R^1	R^2	Time (h)	Yield (%)	mp. (°C)	*lit.* mp. (°C)
3a	C_6H_5	CH_3	1	90	100.3-100.6	114[10a]
3b	3-$O_2NC_6H_4$	CH_3	4	70	147.3-147.5	155[10a]
3c	4-$CH_3OC_6H_4$	CH_3	1	95	124.0-125.1	130[10a]
3d	3-O_2N-4-CH_3-C_6H_3	CH_3	4	85	126.2-127.4	----
3e	2,5-Cl_2-4-F- C_6H_2	CH_3	3	83	115.6-116.2	----
3f	3-$MeCONHC_6H_4$	CH_3	4	82	187.7-188.0	188[10b]
3g	C_6H_5	C_6H_5	1	99	163.6-163.7	163[10c]
3h	3-ClC_6H_4	C_2H_5	4	81	89.2-89.3	86[10d]
3i	$(CH_2)_6$		1	50	62.0-63.5	67[10e]
3j	1,2,3,4-tetrahydronaphthalen-1-yl		2	80	92.8-93.7	142.5-143[10f]
3k	2-thienyl	CH_3	2	83	183.3-183.7	161-162[10g]
3l	2-furyl	CH_3	6	72	113.4-115.8	92-94[10h]
3m	4-$CH_3OC_6H_4$	4-ClC_6H_4	5	84[b]	209.3-210.0(**3m-1**) 206.2-208.5(**3m-2**)	210[10i] 206[10j]
3n	isobutyl	CH_3	24	--[c]		
3o	CH_3CH_2	CH_3	24	--[c]		

a) Reaction conditions: ketoxime (3 mmol), BTC (1 mmol), DMF (3 mmol), MeCN (2 mL), reflux; the ketoximes were prepared by standard procedures.[11] b) Overall yield of isomeric mixtures, the ratio of  and  was 72/28. c) Not detected.

Downloaded At: 17:45 26 January 2011

are more reactive than alkyl ketoximes, the best result being obtained with benzophenone oxime (*Table 2, Entry 3g*). Furthermore, the asymmetrical aryl ketoxime (4-chlorophenyl)(4-methoxyphenyl)methanone oxime gave a mixture of isomeric amides 4-chloro-*N*-(4-methoxyphenyl)benzamide (**3m-1**) and *N*-(4-chlorophenyl)-4-methoxybenzamide (**3m-2**) in the ratio of 72/28. The conversion cyclohexanone oxime is low in comparison with aryl ketoximes (*Table 2, Entry 3i*) and the transformation of sensitive oximes, such as 1-(2-furyl)ethanone oxime (*Table 2, Entry 3l*) and 1-(2-thienyl) ethanone oxime (*Table 2, Entry 3k*), into the corresponding amides also proceeded smoothly. As expected, electron-donating groups on the aromatic rings facilitate the reaction while electron-withdrawing groups retard it (*Table 2, Entries 3a-3e*). In addition, some aliphatic ketoximes, such as 4-methly-3-pentanone oxime and 2-butanone oxime were not suitiable for this reaction under the described conditions (*Table 2, Entries 3n,3o*).

Moreover, the aldoximes were also treated with the BTC/DMF system in acetonitrile at room temperature (*Scheme 1*). From the experimental results, we found that if the substrates were aromatic aldoximes, the corresponding nitriles were obtained in excellent yields (*Table 3*) and the reactions took place rapidly and most were complete within 10 minutes. The electron-withdrawing group of **4b** slows the reaction (*Table 3, Entry 4b*). Unfortunately, no desired rearranged products were detected in our experiments when the substrates were aliphatic aldoximes (*Table 3, Entries 4g-4h*). Then, we attempted to optimize the reaction conditions, such as elevating the reaction temperatures, prolonging the reaction times and changing the solvent, but there were no improvement was observed.

Downloaded At: 17:45 26 January 2011

Table 3. Conversion of Aldoximes into Nitriles under BTC/DMF System.[a]

Entry	R	Time	Yield[b] (%)	mp. (°C)	*lit.* mp. (°C)
4a	C_6H_5	10 min	97	oil	----
4b	3-$O_2NC_6H_4$	30 min	76	117.9-118.0	116[10k]
4c	4-$MeOC_6H_4$	10 min	98	59.9-60.5	57[10k]
4d	4-ClC_6H_4	10 min	96	89.3-89.6	91[10k]
4e	4-$Me_2NC_6H_4$	10 min	99	73.9-74.0	76[10l]
4f	2-Cl-6-F-C_6H_3	10 min	94	59.1-60.2	56-59[10m]
4g	tert-amyl	24 h	--[c]		
4h	cyclohexyl	24 h	--[c]		

a) Reaction conditions: aldoxime (3 mmol), BTC (1 mmol), DMF (3 mmol), MeCN (2 mL), room temperature; the aldoximes were prepared by standard procedures.[11] b) Isolated yield. c) Not detected.

A likely mechanism (*Scheme 1*) for the above transformation involves addition of the Vilsmeier reagent **1** the hydroxy group of the oxime to form the intermediate, which then rearranges to adduct **2**. The adduct **2** should then afford directly the nitrile **4** in the case of aldoxime, or afford the amides **3** upon hydrolytic workup in the case of ketoxime.

In conclusion, we have developed a novel and convenient method for the Beckmann rearrangement of oximes to afford amides or nitriles. This procedure offers several advantages, such as mild reaction conditions, good yields and is more environmentally friendly than traditional approaches. Hence, it is a useful addition to the exiting methods.

EXPERIMENTAL SECTION

Melting points were obtained on an electrothermal melting-point apparatus and are uncorrected. Infrared spectra were recorded on a Thermo Nicolet Avatar 370 spectrophotometer. ^{1}H NMR spectra were measured on a Varian Mercur plus-400 spectrometer with tetramethylsilan (TMS) as an internal standard and $CDCl_3$ or DMSO as the solvent. Mass spectra were obtained with a Finnigan Trace DSQ spectrometer. Elemental analysis was performed on a VarioEL-3 instrument. All chemicals are from commercial sources.

Downloaded At: 17:45 26 January 2011

Typical Procedure for the Preparation of Amides.- *bis*-(Trichloromethyl) carbonate (0.30 g, 1 mmol) was dissolved in 2 mL MeCN, cooled in ice bath for 20 min, then DMF (0.25 mL, 3 mmol) was added dropwise to the mixture, stirred in the ice bath for 1 hour. Then the ketoxime (3 mmol) was added in one portion and the mixture was heated to reflux for the time given in *Table 2*. After completion of the reaction as monitored by TLC, the reaction was quenched with saturated aqueous $NaHCO_3$ (10 mL) and the product was extracted with ethyl acetate (10 mL x 3). The combined organic layer washed with water (10 mL), dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The crude product was purified by column chromatography using a mixture of petroleum ether/ ethyl acetate (v:v = 8:1) as the eluent.

Typical Procedure for the Preparation of Nitriles.- *bis*-(Trichloromethyl) carbonate (0.30 g, 1 mmol) was dissolved in 2 mL MeCN, cooled in ice bath for 20 min, then DMF (0.25 mL, 3 mmol) was added dropwise to the mixture, stirred in the ice bath for 1 hour. Then the aldoxime (3 mmol) was added in one portion and the mixture stirred at room temperature for the time given in *Table 3*. After completion of reaction as monitored by TLC, saturated aqueous $NaHCO_3$ was added to neutralize the mixture. The product was filtered, washed with cold water (20 mL x 2) then the solid product was purified by recrystallization in CH_2Cl_2. For the liquid product, it was purified by column chromatography with a petroleum ether/ ethyl acetate (v:v = 8:1) eluent.

Table 4. Physical Data for Compounds **3** and **4**.

	Product (°C)	mp. m/z	MS (CI⁺) (cm⁻¹)	IR	^{1}H NMR (δ)
3a	100.3-100.6	135 (M^+)	3295 1664 1599		7.64 (s, 1 H, NH), 7.50 (d, 2 H, J = 7.6 Hz, ArH), 7.30 (t, 2 H, J = 7.6 Hz, ArH), 7.10 (t, 2 H, J = 7.6 Hz, ArH), 2.16 (s, 3 H, CH_3)
3b	147.3-147.5	181 (M^+)	3355 1675 1601		8.35 (s, 1 H, ArH), 7.96 (d, 2 H, J = 8.4 Hz, ArH), 7.52 (s, 1 H, NH), 7.50 (t, 1 H, J = 8.4 Hz, ArH), 2.24 (s, 3 H, CH_3)
3c	124.0-125.1	165 (M^+)	3243 1648 1605		7.61 (s, 1 H, NH), 7.38 (d, 2 H, J = 8.8 Hz, ArH), 6.83 (d, 2 H, J = 8.8 Hz, ArH), 3.78 (s, 3 H, OCH_3), 2.13 (s, 3 H, CH_3)
3d [a]	126.2-127.4	194 (M^+)	3354 1675 1602		8.12 (s, 1 H, ArH), 7.90 (s, 1 H, NH), 7.75 (d, 1 H, J = 8.4 Hz, ArH), 7.27 (d, 1 H, J = 8.0 Hz, ArH), 2.54 (s, 3 H, $ArCH_3$), 2.22 (s, 3 H, CH_3)
3e [b]	115.6-116.2	223 (M^+) 221 (M^+)	3255 1675 1518		8.38 (d, 1 H, J = 11.2 Hz, ArH), 7.62 (s, 1 H, NH), 7.41 (d, 1 H, J = 7.2 Hz, ArH), 2.25 (s, 3 H, CH_3)
3f [c]	187.7-188.0	192 (M^+)	3296 1654 1600		9.93 (s, 2 H, NH), 7.89 (s, 1 H, ArH), 7.27 (d, 2 H, J = 8.0 Hz, ArH), 7.18 (t, 1 H, J = 8.0 Hz, ArH), 2.03 (s, 6 H, 2 x CH_3)
3g	163.6-163.7	197 (M^+)	3344 1655 1599		7.91 (s, 1 H, NH), 7.86 (d, 2 H, J = 7.6 Hz, ArH), 7.64 (d, 2 H, J = 8.0 Hz, ArH), 7.54 (t, 1 H, J = 7.6 Hz, ArH), 7.46 (t, 2 H, J = 7.6 Hz, ArH), 7.36 (t, 2 H, J = 8.0 Hz, ArH), 7.15 (t, 1 H, J = 8.0 Hz, ArH)
3h	89.2-89.3	187 (M^+) 185 (M^+)	3249 1667 1596		7.65 (s, 1 H, NH), 7.43 (s, 1 H, NH), 7.36 (d, 1 H, J = 8.0 Hz, ArH), 7.22 (t, 1 H, J = 8.0 Hz, ArH), 7.07 (d, 1 H, J = 8.0 Hz, ArH), 2.39 (q, 2 H, J = 7.6 Hz, CH_2), 1.24 (t, 3 H, J = 7.6 Hz, CH_3)
3i	62.0-63.5	113 (M^+)	3299 1661 1481		6.91 (s, 1 H, NH), 3.21 (q, 2 H, J = 5.2 Hz, CH_2), 2.46 (t, 2 H, J = 4.8 Hz, CH_2), 1.64~1.76 (m, 6 H, CH_2)
3j	92.8-93.7	161 (M^+)	2943 1709 1677		9.54 (s, 1 H, NH), 7.76 (d, 1 H, J = 7.2 Hz, ArH), 7.52 (t, 1 H, J = 7.2 Hz, ArH), 7.40 (t, 1 H, J = 7.2 Hz, ArH), 7.24 (d, 1 H, J = 7.2 Hz, ArH), 3.67 (t, 2 H, J = 6.4 Hz, CH_2), 2.84 (t, 2 H, J = 6.8 Hz, CH_2), 2.04 (m, 2 H, CH_2)
3k	183.3-183.7	141 (M^+)	3252 1653 1578		8.32 (s, 1 H, NH), 6.87 (d, 1 H, J = 4.8 Hz, C=CH), 6.84 (t, 1 H, J = 3.6 Hz, C=CH), 6.65 (d, 1 H, J = 3.6 Hz, C=CH)

Downloaded At: 17:45 26 January 2011

Table 4. Continued...

Product	mp. (°C)	MS (CI^+) m/z	IR (cm^{-1})	^1H NMR (δ)
3l	113.4-115.8	125 (M^+)	3200 3033 1660	9.54 (s, 1 H, NH), 7.20 (d, 1 H, J = 0.8 Hz, furyl 5-H), 6.34 (m, 1 H, furyl 4-H), 6.07 (d, 1 H, J = 3.2 Hz, furyl 3-H), 1.96 (s, 3H, CH_3)
3m-1	209.3-210.0	261 (M^+)	3346 1648 1535	10.19 (s, 1 H, NH), 7.97 (m, 2 H, ArH), 7.66 (m, 2 H, ArH), 7.61 (m, 2 H, ArH), 6.93 (m, 2 H, ArH), 3.75 (s, 3H, CH_3)
3m-2	206.2-208.5	261 (M^+)	3357 1655 1531	7.84 (m, 2 H, ArH), 7.72 (s, 1 H, NH), 7.59 (m, 2 H, ArH), 7.34 (m, 2 H, ArH), 6.99 (m, 2 H, ArH), 3.88 (s, 3H, CH_3)
4a	oil	103 (M^+)	2229	7.58~7.65 (m, 3 H, ArH), 7.47 (t, 2 H, J = 8.0 Hz, ArH)
4b	117.9-118.0	102 (M^+)	2237	8.55 (s, 1 H, ArH), 8.49 (d, 1 H, J = 8.0 Hz, ArH), 8.01 (d, 1 H, J = 8.0 Hz, ArH), 7.75 (t, 1 H, J = 8.0 Hz, ArH)
4c	59.9-60.5	133 (M^+)	2219	7.58 (d, 2 H, J = 8.4 Hz, ArH), 6.95 (d, 2 H, J = 8.4 Hz, ArH), 3.86 (s, 3 H, OCH_3)
4d	89.3-89.6	139 (M^+)	2226	7.61 (d, 2 H, J = 8.4 Hz, ArH), 7.47 (d, 2 H, J = 8.4 Hz, ArH)
4e	73.9-74.0	146 (M^+)	2211	7.46 (d, 2 H, J = 8.8 Hz, ArH), 6.64 (d, 2 H, J = 8.8 Hz, ArH), 3.03 (s, 6 H, $N(CH_3)_2$)
4f	59.1-60.2	155 (M^+)	2242	7.55 (q, 1 H, ArH), 7.34 (d, 1 H, J = 8.0 Hz, ArH), 7.16 (t, 1 H, J = 8.4 Hz, ArH)

a) Elemental analysis (Found): C, 55.65; H, 5.20; N, 14.43 (C, 55.67; H, 5.19; N, 14.43).

b) Elemental analysis (Found): C, 43.23; H, 2.72; N, 6.33 (C, 43.27; H, 2.72; N, 6.31).

c) DMSO as solvent.

Acknowledgement.- We are grateful to the National Key Technology R&D Program (No. 2007 BAI34B04) and National Natural Science Foundation of China (No. 20676123) for financial support.

REFERENCES

1. a) R. E. Gawley, *Org. React.*, **35**, 1 (1988); b) Y. Izumi, S. Sato and K. Urabe, *Chem. Lett.*, 1649 (1983); c) K. Narasaka, H. Kusama, Y. Yamashita and H. Sato, *Chem. Lett.*, 489 (1993); d) J. S. Yadav, B. V. S. Reddy, A. V. Madhavi and Y. S. S. Ganesh, *J. Chem. Res. (S)*, 236 (2002); e) K. V. N. S. Srinavas, E. B. Reddy and B. Das, *Synlett.*, 625 (2002); f) Y. Ikushima, O. Sato, M. Sato, K. Hatakeda and M. Arai, *Chem. Eng. Sci.*, **58**, 935 (2003); g) M. Arisawa and M. Yamaguchi, *Org. Lett.*, **3**, 311 (2001).

2. a) O. Sato, Y. Ikushima and T. Yokoyama, *J. Org. Chem.*, **63**, 9100 (1998); b) Y. Yamaguchi, N. Yasutake and M. Nagaoka, *J. Mol. Struct. (Theochem)*, **639**, 137 (2003).

Downloaded At: 17:45 26 January 2011

3. a) Y. Shinohara, S. Mae, D. Shouro and T. Nakajima, *J. Mol. Struct. (Theochem)*, **497**, 1 (2000); b) D. Mao, G. Lu and Q. Chen, *Appl. Catal. A: Gen.*, **279**, 145 (2005); c) M. Ghiaci, A. Abbaspur and R. J. Kalbasi, *Appl. Catal. A: Gen.*, **287**, 83 (2005); d) R. Raja, G. Sankar, J. M. Thomas, *J. Am. Chem. Soc.*, **123**, 8153 (2001).

4. a) S. Guo and Y. Deng, *Catal. Commun.*, **6**, 225 (2005); b) R. X. Ren, L. D. Zueva and W. Ou, *Tetrahedron Lett.*, **42**, 8441 (2001); c) J. Peng and Y. Deng, *Tetrahedron Lett.*, **42**, 403 (2001); d) D. Li, F. Shi, S. Guo and Y. Deng, *Tetrahedron Lett.*, **46**, 671 (2005).

5. a) L. Cotarca, P. Delogu, A. Nardelli and V. Sunjic, *Synthesis*, 553 (1996); b) D. Krishnaswamy, B. M. Bhawal and A. R. A. S. Deshmukh, *Tetrahedron Lett.*, **41**, 417 (2000); c) L. Pasquato, G. Modena, L. Cotarca, P. Delogu and S. Mantovani, *J. Org. Chem.*, **65**, 8224 (2000); d) D. Krishnaswamy, V. V. Govande, V. K. Gumaste, B. M. Bhawal and A. R. A. S. Deshmukh, *Tetrahedron*, **58**, 2215 (2002).

6. a) D. L. Comins and Y. C. Myoung, *J. Org. Chem.*, **55**, 292 (1990); b) R. S. Rogers, *Tetrahedron Lett.*, **33**, 7473 (1992); c) A. R. Katritzky, C. M. Marson and Z. Wang, *J. Org. Chem.*, **52**, 2730 (1987); d) P. R. Giles and C. M. Marson, *Tetrahedron*, **47**, 1303 (1991).

7. a) W. G. Shan, X. J. Shi and W. K. Su, *Org. Prep. Proced. Int.*, **36**, 337 (2004); b) X. J. Shi, W. K. Su and W. G. Shan, *J. India Chem. Soc.*, **82**, 1019 (2005); c) W. K. Su, W. H. Zhong, G. F. Bian, X. J. Shi and J. P. Zhang, *Org. Prep. Proced. Int.*, **36**, 499 (2004); d) W. K. Su and J. P. Zhang, *Org. Prep. Proced. Int.*, **38**, 404 (2006).

8. I. A. Rivero, K. A. Espinoza and A. Ochoa, *J. Comb. Chem.*, **6**, 270 (2004).

9. J. C. Jochims, S. Hehl and S. Herzberger, *Synthesis*, 1128 (1990).

10. a) A. I. Vogel, In Text Book of *Practical Organic Chemistry*; Addition Wesely Longman Limited: UK, 1363 (1997); b) K. J. Morgan and A. M. Turner, *Tetrahedron* **22**, 1175 (1966); c) S. Ito, Y. Tanaka and A. Kakehi. *Bull. Chem. Soc. Jpn*, **55**, 895 (1982); d) X. H. Du and Z. Y. Xu, *Synlett*, **12**, 1953 (2006); e) E. Hossein, H. Asadollah and M. Elaheh, *Journal of Chemical Research*, **4**, 218 (2006); f) E. C. Horning, V. L. Stromberg and A. H. Lloyd, *J. Am. Chem. Soc.*, **74**, 5153 (1952); g) B. Dieter, H. Georg and N. Christian R., *Synthesis*, **4**, 255 (1977); h) P. Albert, C. Kenneth R., R. Paitoon and R. Mickea, *Journal of Organic Chemistry*, **68**, 2609 (2003); i) C. I. Chiriac, *Rev. Roum. Chim.*, **27**, 747 (1982); j) H. Ki-Jong, T. Beom Seok and K. Misoo, *Organic Preparations and Procedures International*, **37**, 198 (2005); k) "*Aldrich Handbook of Fine Chemicals and Laboratory Equipment*," Bangalore, India, 2003–2004; l) F. Sachs and P. Steinert, *Chem. Ber.* **37**, 1733 (1904); m) X. Y. Li *et al. Chinese Chemical Letters*, **64**, 895 (2004).

11. "*Organic Syntheses*," Coll. Vol .**7**, p. 149 (1990); Vol. **64**, p.19 (1986).

(Received February 8, 2008; in final form August 29, 2008)

Downloaded At: 17:45 26 January 2011