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Journal of Molecular Catalysis A: Chemical 270 (2007) 289–294
JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL
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Catalytic oxidative cleavage of terminal olefins by chromium(III) stearate

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Received 28 July 2006; received in revised form 8 February 2007; accepted 8 February 2007
Available online 16 February 2007

Abstract

A new synthetic methodology for the preparation of carbonyl compounds from the oxidative cleavage of terminal olefins has been developed. With the use of TBHP in combination with chromium(III) stearate, selective oxidation of double bonds conjugated with aromatic ring or carbonyl group could be achieved at ambient temperature in moderate to excellent yield. The oxidative cleavage of electron rich α-methylstyrene derivatives proceeded in good to excellent yield whereas lower yields were observed in α-methylstyrene derivatives containing an electron withdrawing group. This developed oxidation reaction was believed to undergo *via* free radical process and high valent chromium oxo species.
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Keywords: Oxidative cleavage; Terminal olefins; Chromium(III) stearate; Homogeneous catalysis

1. Introduction

The C=C double bond cleavage is one of synthetically useful reactions to degrade large compounds or to introduce oxygen functionality into molecules [1,2] such as to ketones [3,4], aldehydes [5–7] or carboxylic acids [8,9]. Numerous efforts have been addressed to convert olefins to versatile and valuable raw materials for other chemical industries, for example, production of acetophenone and α-keto acid [10–12]. Acetophenone is used in fragrance industries as a perfume head, while α-keto acids, especially those analogues to the naturally occurring amino acids, are of major importance in intermediary metabolism. In addition, they have been used in the therapy of certain conditions, *e.g.*, uremia and nitrogen accumulation disorders and have been reported continually as intermediates in chemical syntheses, in the development of enzyme inhibitors and drugs and as model substrates of enzymes [13,14].

Many synthetic methodologies for oxidizing double bonds have been widely reported, including ozonolysis [15,16], oxidation with $KMnO_4$ [17], with OsO_4 [18], RuO_4 [19,20] and combination of oxidant and a catalyst [8,21,22]. In 1999, Brooks and colleagues achieved rapid and complete conversion in the oxidative cleavage of terminal olefins to carbonyl compounds using aqueous H_2O_2 as an oxidant and 6-molybdo-6-tungstophosphoric acid (PMWA) on either magnesium or aluminium or zinc oxide as a catalyst; 1-octene was stoichiometrically converted to heptanoic acid [23]. In 2001, Yang and colleagues utilized $RuCl_3$-oxone-$NaHCO_3$ to oxidize α-methylstyrene to acetophenone in 78% yield. More recently, $RuCl_3/NaIO_4$ was shown to cleave cyclohexene to adipaldehyde (70%) and could oxidize 1-dodecene smoothly to undecenal with 95% yield [24].

With the ability of being solubilized in organic media, metal ester complexes have been extensively investigated to use in conjunction with oxygen [25]. For ester part in these aforementioned metal complexes, a cheap fatty acid has been generally employed. Metal stearates and metal acetates [26,27] have nonetheless been less used. Additionally, in particular metal trifluoroacetates, metal naphthenates, and metal octanoates [28] have been rarely utilized.

The most popular reagent for oxidative cleavage of double bonds was Jones' reagent which is the combination of CrO_3, H_2SO_4 and acetone [29]. A major drawback of this reagent was however poor solubility in organic solvents. In this paper, we aim to increase the solubility of chromium salts by using different hydrophobic ligands. Thus, the scope of this work is to develop of a new synthetic methodology for the preparation of carbonyl compounds from the oxidative cleavage of terminal olefins by the use of transition metal stearate complexes.

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doi:10.1016/j.molcata.2007.02.007

2. Experimental

2.1. General

The ^{1}H NMR spectra were obtained in $CDCl_3$ with Varian model Mercury 400 spectrometers. The Shimadzu gas chromatograph GC-14A (capillary column: DB-WAX, 30 m, i.d. 0.25 mm) was employed for both qualitative and quantitative analysis of the target product. All organic substrates were purchased from Fluka.

2.2. Preparation of metal stearate complexes [30]

Stearic acid (22 mmol) was dissolved in 1 M NaOH solution at 80 °C. After the mixture was heated and transmuted homogeneously, a solution of metal (III) chloride (7.3 mmol) dissolved in distilled water 10 mL was added dropwise. The mixture was continued heating at about 80 °C for about 30 min, the precipitate was collected and dried *in vacuo*.

2.3. Syntheses of starting materials

2.3.1. Preparation of terminal olefins [31]

Into a 250 mL three-necked round bottom flask was placed methyltriphenylphosphonium iodide (4.85 g, 12 mmol) suspended in 25 mL dry THF. The suspension was cooled to 0 °C with an ice bath. The reaction mixture was charged slowly with *n*-BuLi (1.6 M in hexanes, 7.5 mL, 12.1 mmol) to give an orange solution. After an hour, the solution of a carbonyl compound (10 mmol) in 15 mL dry THF was added slowly to the flask from the additional funnel, the ice bath was removed and the flask was gradually warmed to room temperature. After the reaction mixture was stirred overnight, TLC showed complete consumption of the starting material. HCl 1N was added to the reaction mixture, and the aqueous layer (pH 5) was extracted with EtOAc, washed with brine, dried, filtered and concentrated *in vacuo*. The crude residue was purified by silica gel column and analyzed by ^{1}H NMR.

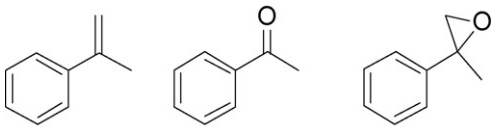
2.4. The general procedure for the oxidative cleavage of terminal olefins

A solution of substrate (5 mmol) in isooctane (5 mL) containing chromium(III) stearate (0.2 mmol) in a round bottom flask and 70% TBHP (9 mmol) was added. The mixture was stirred at 70 °C for 24 h. After the reaction finished, 1 mL of the reaction mixture was taken and extracted with Et_2O. The combined extracts were washed with 25% H_2SO_4 and saturated $NaHCO_3$, respectively. The organic layer was dried over anhydrous Na_2SO_4 and analyzed by GC with the addition of an exact amount of appropriate internal standard.

3. Results and discussion

Table 1 reveals the oxidative cleavage of α-methylstyrene (**1**) performed in isooctane in the presence of different metal stearates and TBHP. Chromium(III) stearate emerged as a viable catalyst affording the best yield of acetophenone (**2**) (85%, entry 1). In all cases, the only by-product was the corresponding epoxide (**3**) and some unreacted α-methylstyrene (**1**) was observed.

Table 1
The effect of metal stearate complexes on the oxidative cleavage of **1**[a]



1 **2** **3**

Entry	Metal stearate	**1** recovery (%)	% Yield		Mass balance
			2	**3**	
1	Chromium(III)	9	85	2	96
2	Manganese(II)	40	57	2	99
3	Iron(III)	37	59	4	100
4	Cobalt(II)	9	71	18	98
5	Nickel(II)	19	66	15	100
6	Copper(II)	31	63	3	97
7	Zinc(III)	46	42	10	98

[a] Reactions conditions: **1** (5 mmol), metal stearate (0.2 mmol), isooctane (5 mL) and TBHP (9 mmol) at 70 °C for 24 h.

A series of controlled experiments was conducted and observed that in the absence of chromium(III) stearate, the oxidation with only TBHP gave a small amount of **2**. Addition of 4 mol% (0.2 mmol) of chromium(III) stearate raised the yield to 85%. This observation strongly supported the concept of metal catalysis in the oxidation reaction as well as TBHP was no doubt essential for the reaction as the oxidant.

At low catalyst loading (0.4–1 mol%), though the reaction gave a fair yield of **2** (69% and 61%, respectively), incomplete conversion was observed along with the detection of a small amount of **3**. The optimum catalyst loading was 4 mol%. Accounting on turnover number (TON), using the catalyst 0.01 mmol provided interesting result to gain over 200 TON. From all above results, it was observed that not only type of catalyst but also amount of catalyst affected the production of the desired product.

The effect of solvent on the oxidative cleavage of **1** was also carefully scrutinized as accumulated in Table 2. Isooctane and acetonitrile (entries 1 and 3) were found to be ideal solvents. Reactions in acetone, acetonitrile/pyridine 3:1, butanol and 1,2-DCE all gave **2** in good yield (entries 8, 10, 14 and 15). It was very interesting to note that the reaction carried out in pyridine/acetic acid 3:1 resulted in almost quantitative amount of product with excellent selectivity. However, the workup of reaction was quite complicated and highly exothermic. Thus, isooctane is the solvent of choice because of the availability, cheapness and being able to solubilize chromium(III) stearate catalyst.

3.1. Kinetic study on the oxidative cleavage of *1* catalyzed by chromium(III) stearate

Fig. 1 exhibits the kinetic study on the oxidative cleavage of **1** catalyzed by chromium(III) stearate. Fifty percentage conversion was observed at 3 h and the reaction reached completion

Table 2
The effect of solvents on the oxidative cleavage of **1** catalyzed by chromium(III) stearate[a]

Entry	Solvents	**1** recovery (%)	% Yield		Mass balance
			2	**3**	
1	Isooctane	9	85	2	96
2	Dichloromethane[b]	67	31	4	102
3	CAN	18	80	6	104
4	Chloroform	81	17	Trace	98
5	Methanol	90	7	Trace	97
6	Ethanol	83	10	Trace	93
7	Pyridine	51	34	Trace	85
8	Acetone[b]	12	59	2	73
9	CCl_4	97	12	Trace	109
10	Acetonitrile:pyridine (3:1)	13	61	Trace	74
11	Pyridine:acetic acid (3:1)	Trace	97	Trace	97
12	DMSO	68	31	4	103
13	DMF	95	3	0	98
14	*n*-Butanol	26	70	2	98
15	1,2-DCE	16	77	4	97
16	THF	99	Trace	0	99

[a] Reactions conditions: **1** (5 mmol), chromium(III) stearate (0.2 mmol), solvent (5 mL) and TBHP (9 mmol) at 70 °C for 24 h.
[b] Refluxing temperature of solvent.

after 24 h. If the reaction was left on for 48 h, the yield deteriorated.

3.2. Oxidative cleavage of selected terminal olefins catalyzed by chromium(III) stearate

3.2.1. Oxidative cleavage of 1,3-diisopropenylbenzene (*4*)

1,3-Diisopropenylbenzene (**4**) was selected as another chemical model. The results are tabulated in Table 3.

When **4** was treated with 0.1 mmol chromium(III) stearate and 9 mmol TBHP in isooctane 5 mL at 70 °C for 24 h, **5** was formed in 71% yield. In entries 1–4, the effect of reaction time was explored in order to improve the yield. It was found that the longer the reaction time, the more **6** was produced. This was indicative of the further oxidation of **5** took place. On the other hand, **6** could be obtained as a major product when the reaction was carried out with 0.25 mmol chromium(III) stearate and 18 mmol TBHP in isooctane 5 mL at 70 °C for 48 h (entry 11). Therefore, the oxidative cleavage conditions could be controlled by appropriate reaction time and amount of TBHP for the production of either **5** or **6**.

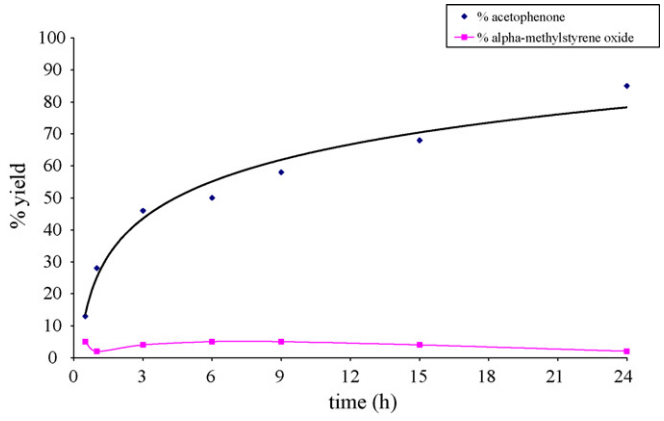


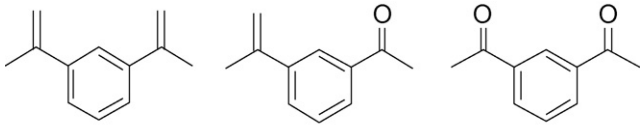
Fig. 1. The kinetic study of the oxidative cleavage of **1**.

3.2.2. Oxidative cleavage of α-methylstyrene derivatives

The oxidative cleavage of various α-methylstyrene derivatives was performed to observe the structure–reactivity relationship of this developed reaction. The outcomes are tabulated in Table 4.

Apparently, the oxidative cleavage of monosubstituted α-methylstyrene gave a good mass balance. α-Methylstyrene containing electron donating groups cleanly produced good

Table 3
The oxidative cleavage of **4**[a]

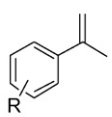


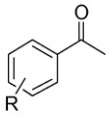
4 **5** **6**

Entry	Chromium(III) stearate (mmol)	Time (h)	% Yield		Mass balance
			5	**6**	
1	0.1	12	21	Trace	97
2	0.1	18	39	Trace	95
3	0.1	24	71	12	101
4	0.1	48	60	19	103
5[b]	0.1	24	47	35	96
6	0.2	12	32	Trace	100
7	0.2	18	44	16	101
8	0.2	24	46	29	99
9[b]	0.2	24	41	48	97
10[b]	0.2	48	23	57	95
11[b]	0.25	48	13	62	99

[a] Reactions conditions: **4** substrate (5 mmol), chromium(III) stearate (0.1–0.25 mmol), isooctane (5 mL) and TBHP (9 mmol) at 70 °C for 12–48 h.
[b] TBHP (18 mmol).

Table 4
The oxidative cleavage of α-methylstyrene derivatives[a]





1 R = H
7 R = *p*-CH_3O
8 R = *p*-Br
9 R = *p*-Cl
10 R = *p*-CH_3CH_2
11 R = *p*-CH_3
12 R = *o*-NO_2
13 R = *m*-NO_2
14 R = *p*-NO_2

2 R = H
15 R = *p*-CH_3O
16 R = *p*-Br
17 R = *p*-Cl
18 R = *p*-CH_3CH_2
19 R = *p*-CH_3
20 R = *o*-NO_2
21 R = *m*-NO_2
22 R = *p*-NO_2

Entry	Substrate	Acetophenone derivative (% yield)	Substrate recovery (%)	Mass balance
1	**1**	**2** (85)	9	96
2	**7**	**15** (89)	9	98
3	**8**	**16** (85)	11	96
4	**9**	**17** (81)	11	92
5	**10**	**18** (67)	30	97
6	**11**	**19** (65)	36	101
7	**12**	**20** (32)	67	99
8	**13**	**21** (46)	50	96
9	**14**	**22** (41)	57	98

[a] Reactions conditions: substrate (5 mmol), chromium(III) stearate (0.2 mmol), isooctane (5 mL) and TBHP (9 mmol) at 70 °C for 24 h.

yield of the corresponding acetophenone slightly increasing with the electron donating ability of the substituent (81% for *p*-Cl < 85% for *p*-Br < 89% for *p*-OMe). These derivatives showed similar reactivity to the unsubstituted α-methylstyrene.

For alkyl substituted α-methylstyrenes, the oxidative cleavage surprisingly gave a somewhat lower yield (65% for *p*-Me and 67% for *p*-Et). This lower product yield was not the direct result of the alkyl group on the electronic property of α-methylstyrene. Instead, it should be from the presence of a new benzylic carbon which could be readily oxidized, giving out other products from a competitive reaction.

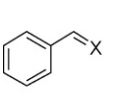
To investigate the effect of electron-withdrawing substituents, *o*-, *m*-, and *p*-NO_2 substituted α-methylstyrenes were separately subjected to the oxidative cleavage. The lowest yield in this series (32%) was observed for *o*-NO_2. The *p*-NO_2 substituted substrate gave a slightly higher yield (41%). This observed difference may be the result of the *ortho* electronic effect. Oxidative cleavage of *m*-NO_2-α-methylstyrene gave the desired product in 46% yield. The *m*-substituent has obviously less deactivating effect than *o*- and *p*-ones, probably derived from only inductive effect.

3.2.3. *Oxidative cleavage of other selected terminal olefins*

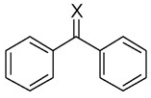
To shed some light on the applicability of this reaction, further study on the oxidative cleavage reaction of other terminal olefins were examined. The results are shown in Table 5.

The oxidative cleavage of styrene **23** gave benzaldehyde **24** in 69% yield. It is slightly lower than the reaction of α-methylstyrene of which methyl group helped stabilization of the intermediate. The reaction of 1,1-diphenylethylene **25** gave the benzophenone **26** in good isolated yield (76%). For the oxidative cleavage of 1,1-diphenylethylene **25** a drop in the product yield is probably the result from steric congestion in the cyclization of Cr^V=O species into the proposed intermediate. The reaction of MMA **27** proceeded to give methyl pyruvate **28** in 55% yield. Surprisingly, diethyl itaconate **29** did not give the corresponding oxidative cleavage product, diethyl oxalacetate **30** and the starting material **29** was recovered quantitatively (entry 4). Camphene **31** and 1-dodecene **32** were also unreactive towards this oxidation. The oxidation of 4-vinyl-1-cyclohexene **33** resulted in an unexpected allylic oxidation to give a diketone product **34** in 17% yield. This obtained product **34** could be gained from the direct isolation from the reaction mixture.

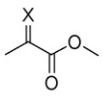
Table 5
The oxidative cleavage of selected terminal olefins[a]



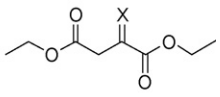
23 X = CH_2
24 X = O



25 X = CH_2
26 X = O



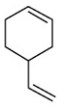
27 X = CH_2
28 X = O



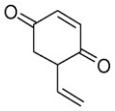
29 X = CH_2
30 X = O

31

32



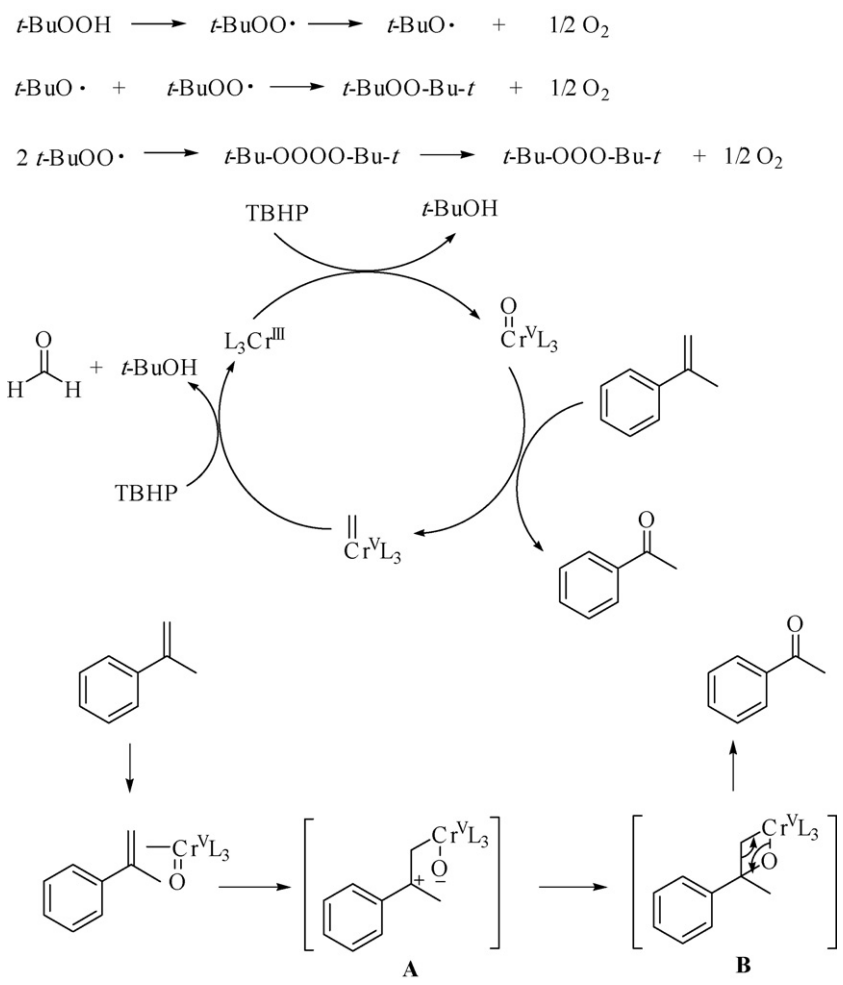
33



34

Entry	Substrate	Product	% Yield	Substrate recovery (%)	Mass balance
1	**23**	**24**	69	28	97
2	**25**	**26**	76	24	100
3	**27**	**28**	55	14	69
4	**29**	No reaction	–	100	100
5	**31**	No reaction	–	100	100
6	**32**	No reaction	–	100	100
7	**33**	**34**	17	43	60

[a] Reactions conditions: substrate (5 mmol), chromium(III) stearate (0.2 mmol), isooctane (5 mL) and TBHP (9 mmol) at 70 °C for 24 h.



Scheme 1. Proposed mechanism for the oxidative cleavage of terminal olefins catalyzed by chromium(III) stearate.

Therefore, based upon the results attained this developed oxidative cleavage was selective for conjugated double bonds and could not use with the substrates containing a nonconjugated methyl or methylene protons, respectively.

3.3. Proposed mechanism

The mechanism for the oxidative cleavage of activated double bonds by TBHP catalyzed by Cr(III) stearate is proposed in Scheme 1.

The chromium(III) complex was transformed to the corresponding high valent (formally) Cr^{V}=O species by interaction with TBHP. In similar fashion to Barton's theory for the formation of high valent iron oxo species [32], the addition of the Cr^{V}=O species to the double bond followed by cleavage to the ketone and a methylene carbene. There are experiments which confirmed the detection of formaldehyde [32].

4. Conclusions

Chromium(III) stearate/TBHP was disclosed to be a new catalytic oxidative cleavage of terminal olefins. Aromatic substituents have a large influence on the reactivity of the α-methylstyrene derivatives. Compounds bearing electron donating substituents on the aromatic ring are more reactive towards oxidative cleavage than those containing electron withdrawing substituents.

Acknowledgement

This work was supported in part by the Graduate School, Chulalongkorn University.

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