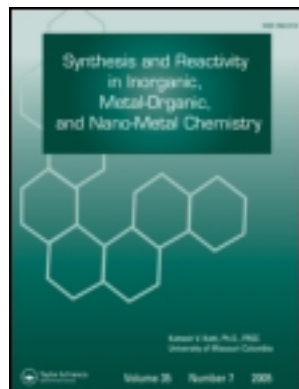


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3,5-Dimethylpyrazolium Chlorochromate(VI): An Efficient Reagent for Solvent-Free Oxidation of Organic Substrates

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A new chromium(VI) reagent 3,5-dimethylpyrazolium chlorochromate, $C_5H_8N_2H[CrO_3Cl]$ (DmpzHCC), was synthesized and used for the selective oxidation of various organic compounds under solvent-free conditions with high efficiency. This new compound has certain advantages over its companion analogues in terms of controlled acidity, amount of oxidant, lack of solvent, short reaction times, and high yields.

Keywords 3, 5-dimethylpyrazolium chlorochromate, alcohols, organic substrates, oxidation, oximes

INTRODUCTION

Chromium(VI)-based oxidizing reagents are the most widely used transition metal oxidants for the effective and selective oxidation of various organic compounds. Since the appearance of Collins reagent in 1968,^[1] the development of new chromium(VI) oxidizing agents for the effective and selective oxidation of organic substrates, in particular alcohols, under mild conditions has attracted a great deal of continued interest in organic synthesis.

Extensive work has led to the development of a good number of these oxidants such as pyridinium chlorochromate (PCC),^[2,3] pyridinium fluorochromate (PFC),^[4,5] pyridinium dichromate (PDC),^[6] quinolinium fluorochromate (QFC),^[7] quinolinium chlorochromate (QCC),^[8] 3,5-dimethylpyrazolium fluorochromate (DmpzHFC),^[9] and pyridinium sulfonate chlorochromate (PSCC).^[10]

Although many chromium(VI) reagents are available for the oxidation of organic substrates, they have certain limitations such as high acidity, instability of the reagents, the need of an excess amount of the reagent, long reaction times, and poor selectivity to substrates. Therefore, there still exists need for highly efficient and mild oxidizing agents.

There is an increasing interest in the use of environmentally benign conditions^[11,12] and particularly solvent-free procedures.^[13–15] These reactions are not only of interest from an ecological point of view but avoiding organic solvents during the reactions in organic synthesis leads to a clean, efficient, and economical technology. Recently we have reported the oxidizing properties of two new chromium (VI) reagents, 4-benzylpyridinium fluorochromate^[16] and quinoxalinium fluorochromate.^[17] In extension of our studies on development of new reagents based on chromium(VI) we present here the synthesis and characterization of 3,5-dimethylpyrazolium chlorochromate and its use for the oxidative transformation of various alcohols, oximes, and polycyclic aromatic hydrocarbons into the corresponding carbonyl compounds selectively under solvent-free conditions.

Experimental

General

All reagents and solvents were obtained from Aldrich and used without further purification. ¹H-NMR spectra were recorded on Bruker 300 MHz high performance FT-NMR spectrometer (Germany). UV spectra was performed using Analytica Jena Specord 200 instrument (Germany), elemental analyses were performed using Elementar Micro Vario CHNS elemental analyzer (Germany), and found to be in accord with calculated values. Melting points were determined with Barnstead Electrothermal 9200 digital melting point apparatus (United Kingdom). IR spectra were recorded on Mathson 1000 FT-IR spectrometer. Chromium analysis was performed using Perkin-Elmer Optima 5300DV ICP-OES (USA), and cyclic voltammogram was recorded with CHI 660 B CH Instrument Electrochemical Workstation (USA).

Preparation of 3,5-Dimethylpyrazolium Chlorochromate

To a solution of 1.0 g (10 mmol) CrO_3 in 0.5 mL H_2O , 2 mL (23 mmol) 12 M HCl was added with stirring at room temperature. The orange red solution thus obtained was cooled in an ice-bath (0–5°C). To this solution 0.96 g (10 mmol) 3,5-dimethylpyrazole was added portion-wise in 20 min. During stirring the reaction mixture for 1 h, a bright-orange crystalline compound separated out. This was filtered under

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TABLE 1
Oxidation of alcohols, polycyclic arenes, and oximes with DmpzHCC under solvent-free conditions at room temperature^a

Entry	Substrates	Substrate: Time		Product ^a	Yield % ^b	Mp (°C) of DNPH Found Reported ^[26]	
		oxidant	(min)				
1	Benzyl alcohol	1:1	3	Benzaldehyde	86	236–237	237
2	4-Methylbenzyl alcohol	1:1	2	4-Methylbenzaldehyde	90	234–235	233
3	4-Chlorobenzyl alcohol	1:1	2	4-Chlorobenzaldehyde	96	264–265	265
4	4-Methoxybenzyl alcohol	1:1	1	4-Methoxybenzaldehyde	99	253–254	254
5	4-Nitrobenzyl alcohol	1:1	4	4-Nitrobenzaldehyde	87	319–320	320
6	Cinnamyl alcohol	1:1	1	Cinnamaldehyde	100	253–254 ^d	255 ^d
7	Citronellol	1:1	5	Citronellal	94	76–77	78
8	1-Octanol	1:1	2	1-Octanal	85	104–105	106
9	Cyclohexanol	1:1	5	Cyclohexanone	79	161–162	162
10	4-tert-Butylcyclohexanol	1:1	2	4-tert-Butylcyclohexanone	99	154–155	156
11	Menthol	1:1	5	Menthone	100	147–148	146
12	Cyclohexanone oxime	1:1	1.5	Cyclohexanone	84	160–161	162
13	Cyclopentanone oxime	1:1	1	Cyclopentanone	85	146–147	146
14	Benzaldoxime	1:1	2	Benzaldehyde	89	236–237	237
15	Benzhydrol	1:1	8	Benzophenone	75	237–239	238
16	Isoborneol	1:1	5	Camphor ^c	92	176–177 ^d	179
17	Benzoin	1:1	15	Benzil ^c	85	95–96 ^e	97
18	Anthracene	1:2	2 h	9,10-Anthraquinone ^c	72	284–285 ^f	286
19	Phenanthrene	1:2	2 h	Phenanthrene-9,10-quinone ^c	83	208–209 ^g	209
20	Triphenylphosphine	1:1	3	Triphenylphosphine oxide ^c	82	155–156 ^h	157

^aAll the aldehydes and ketones have been described previously in the literature and were identified by their IR, ¹H-NMR spectra, melting points, or by the IR spectra and melting points of their 2,4-dinitrophenylhydrazones. ^bIsolated yields as the dinitrophenylhydrazones. ^cYield of isolated carbonyl compound. ^dMp of camphor. ^eMp of benzil. ^fMp of 9,10-anthraquinone. ^gMp of phenanthrene-9,10-quinone. ^hMp of triphenylphosphine oxide.

vacuum, washed with hexane (3 × 10 mL), dried, and finally stored in a sealed bag in a freezer. Yield (2.28 g; 98%), m.p 55–56°C; ¹H-NMR (300 MHz, CDCl₃): δ = 2.2 (s, 6H), 5.77 (s, 1H); IR (KBr): $\bar{\nu}$ = 954 cm⁻¹ (Cr = O), 870 cm⁻¹ (Cr = O), 460 cm⁻¹ (Cr-Cl); UV-vis (acetonitrile, 2.10⁻² mol/L): λ_{\max} (ϵ) = 361.5–400 nm (Cr-Cl), 220.8 nm (originating from the 3,5-dimethylpyrazolium cation). Found C, 25.13%; H, 3.64%; N, 11.65%; Cr, 22.79%. C₅H₈N₂H[CrO₃Cl] Anal. Calcd.: C, 25.8%; H, 3.87%; N, 12.04%; Cr, 22.36%.

General Procedure for Oxidation of Organic Substrates with DmpzHCC Under Solvent-Free Conditions

DmpzHCC (1.0–2.0 mmol) was added to the substrate (1.0 mmol) in a mortar. The homogeneous mixture thus obtained was kept for appropriate period of time at room temperature with grinding. During the reactions, the color of the oxidant changes from orange to brown, providing visual means for ascertaining the progress of the oxidation. The progress of the reaction was monitored by using TLC on silica gel (n-hexane-ethylacetate = 2:1). After completion of the reaction, the reaction mixture was worked up by dilution with diethylether (3 × 15 mL) and filtration. The solvent was removed by simple distillation and the product was isolated with acceptable purity. Yields were based on either to isolation of the carbonyl product or to isolation of the 2,4-dinitrophenylhydrazones (DNPH).

Oxidation of isoborneol to camphor:

FT-IR(KBr): 2955 (Aliph. C-H), 1720 (C = O)

¹H-NMR (CDCl₃): 0.90–2.40 (m, 16H, Aliph. C-H)

Oxidation of benzoin to benzil:

FT-IR(KBr): 1679 (C = O), 1593 (C = C)

¹H-NMR (DMSO-d₆): 7.65 (t, 4H, Ar-H), 7.80(t, 2H, Ar-H), 7.90 (d, 4H, Ar-H)

Oxidation of anthracene to 9,10-anthraquinone:

FT-IR(KBr): 1680(C = O), 1590 (C = C)

¹H-NMR (DMSO-d₆): 7.85 (m, 4H, Ar-H), 8.35(m, 4H, Ar-H)

Oxidation of phenanthrene to phenanthrene-9,10-quinone:

FT-IR(KBr): 1680 (C = O), 1590(C = C)

¹H-NMR (CDCl₃): 8.20 (dd, 2H, Ar-H), 8.0(d, 2H, Ar-H), 7.7 (t, 2H, Ar-H), 7.5 (t, 2H, Ar-H)

Oxidation of triphenylphosphine to triphenylphosphine oxide:

FT-IR(KBr): 1100 (P = O), 1490 (C = C)

¹H-NMR (CDCl₃): 7.70–7.50 (m, 15H, Ar-H)

Qualitative identification of the rest of the carbonyl products was made by comparison of the FT-IR spectra (C = N band varying between 1614 cm⁻¹ and 1621 cm⁻¹) and melting points of their 2,4-dinitrophenylhydrazones with derivatives of known compounds (Table 1).

TABLE 2
Comparison of oxidation of various organic substrates by DmpzHCC, PCC, PFC, BAAOC, and DmpzHFC^a

Substrate	Yield (%) (time [min])				
	DmpzHCC	PCC ^{20,21}	PFC ^{22,23}	BAAOC ²⁴	DmpzHFC ²⁵
4-Methoxybenzyl alcohol	99 (1)	94 (5)	89 (10)	99 (2)	92 (2)
4-Chlorobenzyl alcohol	96 (2)	93 (10)	78 (10)	95 (7)	—
Benzyl alcohol	86 (3)	96 (15)	93 (10)	98 (1)	85 (1)
Cinnamyl alcohol	100 (1)	95 (15)	85 (10)	85 (20)	89 (2)
Benzaldoxime	89 (2)	80 (2h) ^b	87 (15)	—	—
Anthracene	72 (2h) ^b	<i>N.R.</i> (5h) ^c	75 (2h) ^d	—	84 (20) ^f
Phenanthrene	83 (2h) ^b	<i>N.R.</i> (5h) ^c	79 (2h) ^e	—	78 (20) ^f

^aSubstrate/reagent molar ratio is 1:1 unless otherwise stated. ^bSubstrate/reagent (1:2). ^cSubstrate/reagent (1:10) reaction does not proceed. ^dSubstrate/reagent (1:2.2) reaction carried out at 50°C. ^eSubstrate/reagent (1:2.2) reaction carried out at 70°C. ^fSubstrate/reagent (1:2.1).

RESULTS and DISCUSSION

The reagent 3,5-dimethylpyrazolium chlorochromate, has been prepared in excellent yield (98%) from CrO₃, aqueous hydrochloric acid (12 N) and 3,5-dimethylpyrazole in the molar ratio of 1:2:1. The bright-orange crystalline reagent can be stored in a sealed bag in a freezer for long periods without decomposition. The structure of DmpzHCC was confirmed by elemental analysis, IR (KBr), UV-vis, and ¹H-NMR spectra. DmpzHCC is soluble in dichloromethane, water, acetonitrile, chloroform, ethanol, methanol, acetone, DMSO, and DMF, and insoluble in carbon tetrachloride, benzen, and hexane. These results are indicative of the ionic nature of DmpzHCC.

The chromium content of the reagent was determined by inductively coupled plasma (ICP). The compound is diamagnetic as tested by means of magnetic susceptibility measurement. DmpzHCC is a 1:1 electrolyte ($\Lambda_M = 140 \text{ ohm}^{-1}\text{cm}^2\text{mol}^{-1}$, in CH₃CN). The acidity of DmpzHCC (pH of a 0.01 molar solution: 2.55) is less pronounced than that of pyridinium chlorochromate (pH of a 0.01 molar solution: 1.75) and pyridinium fluorochromate (pH of a 0.01 molar solution: 2.45).

Figure 1 shows the cyclic voltammogram of the oxidant. The oxidant, DmpzHCC, shows irreversible behavior. This is

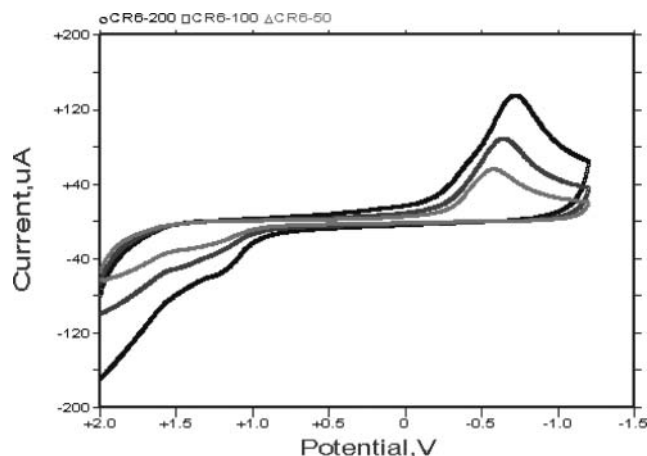
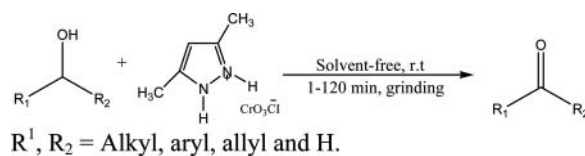


FIG. 1. Cyclic voltammogram of C₅H₈N₂H[CrO₃Cl].

because of highest oxidation state of chromium is six, Cr(VI); for this reason it is expected that the Cr(VI) compounds such as this oxidant decomposed by increasing the potential and does not show any reversible behavior, as seen in Figure 1.

The reagent, DmpzHCC, provided a homogeneous melt under solvent-free conditions because of its relatively low melting point (55–56°C). During the course of the reaction the homogeneous mixture turned more viscous. The reactions were carried out smoothly with good to excellent isolated yields in short reaction times. For the brown product isolated after the oxidation reaction the magnetic susceptibility was measured to amount 2.84 BM at room temperature. Thus, the oxidation level of the metal is 3.8–4.1. This suggests clearly that isolated solid product is C₅H₈N₂H[CrO₂Cl] and DmpzHCC serves as a 2-electron transfer reagent.

In order to ascertain the efficiency of the reagent as an oxidant, a wide variety of alcohols such as benzylic, allylic, and aliphatic were treated with one equivalent of DmpzHCC, under solvent-free conditions at room temperature into the corresponding carbonyl compounds (Scheme 1).



SCH. 1.

Various primary benzylic alcohols and 1-octanol were converted into their corresponding aldehydes with high to excellent yields (Table 1, entries 1–5 and 8). Overoxidation of products to the corresponding carboxylic acids was not observed at all. It is noteworthy that cinnamyl alcohol (entry 6) was converted to cinnamaldehyde without the cleavage of the benzylic double bond and the reaction is essentially chemoselective. Benzoin (entry 17) was oxidized to benzil with no bond cleavage with quantitative yields. Isoborneol (entry 16) was oxidized to camphor with excellent yield.

3,5-DmpzHCC, owing to its controlled acidity, was successful in oxidizing acid-sensitive substrates. Thus, treatment of citronellol (entry 7) with DmpzHCC yielded citronellal in 94% isolated yield. No isopulegone arising from acid promoted cationic cyclization and further oxidation as in the case of unbuffered pyridinium chlorochromate (PCC) was observed.^[18] This result is believed to be a direct consequence of the far less pronounced acidic character of the new reagent.

Significantly, oxidative deoxygenation of various oximes gave corresponding carbonyl compounds with high yields (entries 12–14), thus providing an alternative pathway to the regeneration of carbonyl compounds from non-carbonyl substrates. The selective protection and deprotection of carbonyl groups frequently represent a key step in synthetic organic chemistry and many reagents have been introduced over the years^[19] for the purpose.

Finally oxidation of fused ring hydrocarbons such as anthracene (entry 18) and phenanthrene (entry 19) gave 9,10-anthraquinone and phenanthrene-9,10-quinone, respectively, with good yields in reasonably short reaction times.

The reaction of triphenylphosphine (entry 20) with DmpzHCC gave triphenylphosphine oxide in quantitative yields. This provides clear-cut example of oxygen transfer reaction involving DmpzHCC and the result may also be useful in defining other related reactions.

In order to show the oxidative ability of this reagent (DmpzHCC), we compared some of our results with those of PCC,^[20,21] PFC,^[22,23] 1-butyl-4-aza-1-azoniabicyclo[2.2.2]octane chlorochromate (BAAOC),^[24] DmpzHFC,^[25] carried out under solvent-free conditions (Table 2).

It has been found that this reagent has certain advantages over similar oxidizing agents in terms of its controlled acidity, chemoselectivity, amounts of oxidant, short reaction times required, higher product yields, and milder conditions.

In conclusion, we have developed an efficient, solvent-free method for the oxidation of alcohols, polycyclic aromatic hydrocarbons, and oximes that possesses significant advantages over the existing methods such as simple procedure, easy reaction workup, efficiency, high yields, and lack of solvent. Based on all the results heretofore obtained it may be stated that DmpzHCC is an important addition to the species of oxidizing agents for the oxidation of organic substrates.

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