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Rapid oxidation of alcohols and trimethylsilyl and tetrahydropyranyl ethers with CrO₃ in the presence of sulfonic acid functionalized ordered nanoporous Na⁺-montmorillonite

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ABSTRACT

A mild, efficient and fast method for the oxidation of alcohols and trimethylsilyl and tetrahydropyranyl ethers to their corresponding carbonyl compounds using CrO₃ in the presence of sulfonic acid functionalized ordered nanoporous Na⁺-montmorillonite (SANM) and under solvent-free conditions is reported. All reactions were performed at room temperature in high to excellent yields. Ease of the preparation and handling of the catalyst, heterogeneous conditions, easy work-up of the products and high reaction rates are the main advantages of this method.

Keywords: Na⁺-montmorillonite, CrO₃, Alcohols, Trimethylsilyl ethers, Tetrahydropyranyl ethers, Oxidation reactions.

1. Introduction

Aldehydes and ketones are considered as important precursors for the preparation of many of the organic compounds, including valuable fine chemicals, such as fragrances, vitamins and drugs [1]. Oxidation of alcohols and oxidative deprotection of trimethylsilyl and tetrahydropyranyl ethers is one of the main methods for the preparation of these compounds and several methods have been explored to such conversions [2-14]. Several reagents for the oxidation of the above mentioned compounds have been reported of which chromium (VI)-based oxidants impregnated on solid supports have been extensively used in organic synthesis [15-31].

However, some of the reported methods suffer from disadvantages, such as long reaction times, low yields of the products, use of expensive reagents, requirement of the aqueous reaction conditions and tedious work-up. Therefore, an inexpensive and efficient reagent for this reaction under milder conditions is still in demand. In recent years, clays as nanostructured materials have been widely used in organic transformations as solid acid catalysts [32].

These compounds have some advantages such as accessibility, cheapness, easy modification, non-toxicity and recyclability. The montmorillonite minerals are one of the most widely used clays which have very small micron sized particles and are extremely fine-grained and thin-layered [33].

2. Experimental

2.1. General

Chemicals were purchased from Fluka, Merck and Aldrich Chemical Companies. All the products were separated and characterized using spectral analysis (IR and NMR). All yields refer to the isolated products. The purity determination of the substrate and reaction monitoring were accompanied by TLC on silica-gel polygram SILG/UV 254 plates.

2.2. General procedure

A mixture of SANM (0.1 g) and CrO₃ (0.125 g, 1.25 mmol, 125 mol%) was grounded in a mortar for 1 min. Then, the substrate (1 mmol) was added and the mixture was grounded at room temperature for the specified time (Tables 2 and 3). The progress of the reaction was monitored by TLC (*n*-hexane/EtOAc = 10:2). After completion of the reaction, the mixture was diluted with 5 mL CH₂Cl₂ and filtered. The solid

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residue was washed with CH_2Cl_2 (3×5 mL). Evaporation of the solvent followed by column chromatography on silica gel (*n*-hexane/ EtOAc = 10:2) gave the corresponding carbonyl compounds in good to high yields.

Selected spectral data:

Table 2, entry 1: IR (KBr) υ: 3069, 2817, 2735, 1699, 1660, 1586, 831, 745, 682 cm^{-1}; ^{1}H NMR (CDCl$_3$, ppm) δ: 7.2–8.0 (m, 5 H), 9.95 (s, 1 H).

Table 3, entry 5: IR (KBr) υ: 3050, 2905, 2880, 2775, 1705, 1595, 850 cm^{-1}; ^{1}H NMR (CDCl$_3$, ppm) δ: 7.40 (d, 2 H, *J* = 6.2 Hz), 7.70 (d, 2 H, *J* = 6.2 Hz), 9.91 (s, 1 H).

Table 3, entry 34: IR (KBr) υ: 3090, 2910, 1685, 1610, 1155 cm^{-1}; ^{1}H NMR (CDCl$_3$, ppm) δ: 2.45 (s, 3 H), 7.45–7.90 (m, 5 H).

3. Results and Discussion

Over the past decade, a considerable part of our research program has focused on the development of new methods and use of new reagents for the oxidation of organic compounds [34-41].

Herein and in continuation of these studies, we wish to report the applicability of sulfonic acid functionalized ordered nanoporous sodium montmorillonite (SANM) as newly reported solid acid catalyst [42], in the promotion of the oxidation of alcohols and trimethylsilyl and tetrahydropyranyl ethers with CrO$_3$.

At the first step and in order to optimize the reaction conditions, oxidation of 2-chlorobenzyl alcohol by CrO$_3$ in the presence of SANM was studied as a model reaction. The reaction was performed in different solvents and also under solvent-free conditions, using different amounts of SANM and CrO$_3$ at room temperature. The results are shown in Table 1.

The obtained results showed that the reaction using 0.1 g of SANM and 1.25 mmol of CrO$_3$ in the absence of solvent proceeded in highest yield in very short reaction times (Table 1, entry 2).

The selected condition is shown in Scheme 1.

After optimization of the reaction conditions and in order to show the general applicability of this method, different types of alcohols were subjected to the same reaction under the determined conditions. The results are summarized in Table 2.

Different types of benzylic alcohols (including electron-donating or electron-withdrawing groups) are efficiently converted to their corresponding aldehydes and ketones in high yields in very short times (Table 2, entries 1-16). Although oxidation of benzylic alcohols containing electron-donating groups is faster than electron-withdrawing groups, this method is very effective for the oxidation of primary and secondary aliphatic alcohols (Table 2, entries 17-21). No traces of by-products were detected in all the cases studied.

After the successful oxidation of alcohols, we were interested in extending the applicability of the above reagent system to direct oxidation of trimethylsilyl and tetrahydropyranyl ethers to their corresponding carbonyl compounds (Scheme 1).

The obtained results showed that CrO$_3$ in the presence of SANM can also be efficiently used for the oxidation of various trimethylsilyl and tetrahydropyranyl ethers including different substituents (Cl, Br, NO$_2$, CH$_3$ and OCH$_3$) in high to excellent yields in very short times as well as alcohols (Table 3).

Under the same reaction conditions, the direct oxidation of methoxymethyl ethers was not successful and the starting material was recovered unchanged after 0.5 h. Therefore, this method can be useful for the selective oxidation of alcohols and trimethylsilyl and tetrahydropyranyl ethers in the presence of the above-mentioned substrates (Table 3, entries 42-44).
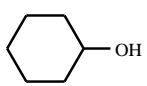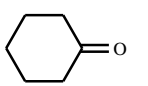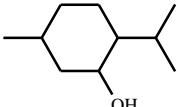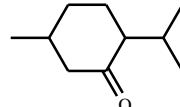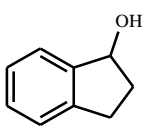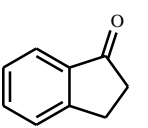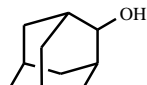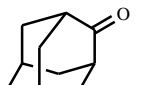
Table 1. The effect of different conditions on the oxidation of 2-chlorobenzyl alcohol with CrO$_3$/SANM.

Entry	SANM (g)	CrO$_3$ (mmol)	Solvent	Time (min)	Conversion (%)
1	0.15	1.25	Solvent-free	4	100
2	0.1	1.25	Solvent-free	6	100
3	0.05	1.25	Solvent-free	5	90
4	0.1	0.08	Solvent-free	5	80
5	0.1	1.5	Solvent-free	3	100
6	0.1	1.25	CH$_3$CN	10	100
7	0.1	1.25	*n*-Hexane	25	100
8	0.1	1.25	CH$_2$Cl$_2$	30	70
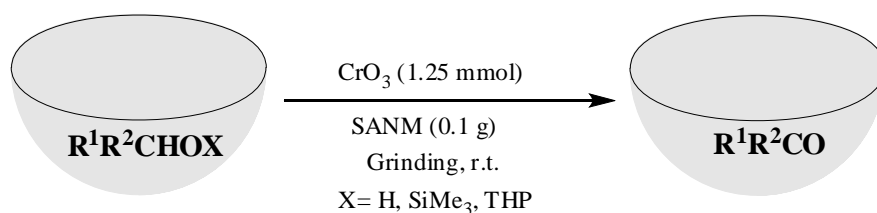
Scheme 1. Oxidation of alcohols and trimethylsilyl and tetrahydropyranyl ethers to their corresponding carbonyl compounds using CrO_3 in the presence of SANM.

Table 2. Oxidation of alcohols using CrO_3/SANM reagent system in the absence of solvent. [a, b]

Entry	Substrate	Product	Time (min)	Yield (%)[c]
1	$C_6H_5CH_2OH$	C_6H_5CHO	2	98
2	$2\text{-}ClC_6H_4CH_2OH$	$2\text{-}ClC_6H_4CHO$	4	90
3	$4\text{-}ClC_6H_4CH_2OH$	$4\text{-}ClC_6H_4CHO$	5	98
4	$2\text{-}BrC_6H_4CH_2OH$	$2\text{-}BrC_6H_4CHO$	6	95
5	$4\text{-}BrC_6H_4CH_2OH$	$4\text{-}BrC_6H_4CHO$	7	90
6	$3,4\text{-}Cl_2C_6H_3CH_2OH$	$3,4\text{-}Cl_2C_6H_3CHO$	5	98
7	$2\text{-}MeC_6H_4CH_2OH$	$2\text{-}MeC_6H_4CHO$	6	95
8	$3\text{-}MeOC_6H_4CH_2OH$	$3\text{-}MeOC_6H_4CHO$	4	98
9	$4\text{-}MeOC_6H_4CH_2OH$	$4\text{-}MeOC_6H_4CHO$	4	98
10	$4\text{-}Me_3CC_6H_4CH_2OH$	$4\text{-}Me_3CC_6H_4CHO$	3	98
11	$4\text{-}Me_2CHC_6H_4CH_2OH$	$4\text{-}Me_2CHC_6H_4CHO$	3	98
12	$3\text{-}NO_2C_6H_4CH_2OH$	$3\text{-}NO_2C_6H_4CHO$	14	98
13	$4\text{-}NO_2C_6H_4CH_2OH$	$4\text{-}NO_2C_6H_4\ CHO$	18	90
14	Ph_2CHOH	Ph_2CO	6	95
15	$PhCH(OH)Me$	$PhCOMe$	4	98
16	$PhCH(OH)COPh$	$PhCOCOPh$	31	80
17	$C_6H_5CH_2CH_2OH$	$C_6H_5CH_2CHO$	4	98
18			10	98
19			7	98
20			3	98
21			8	98

[a] Products were characterized by their physical constants, comparison with authentic samples and IR and NMR spectroscopy.
[b] Reactions were performed using 225 mg of CrO_3/SANM (CrO_3 content 1.25 mmol).
[c] Yields are based on isolated products.

Table 3. Oxidation of trimethylsilyl and tetrahydropyranyl ethers using CrO_3/SANM reagent system in the absence of solvent.[a, b]
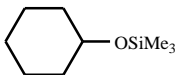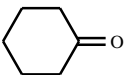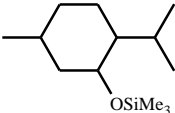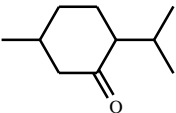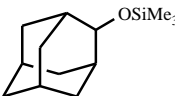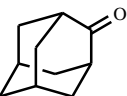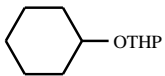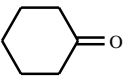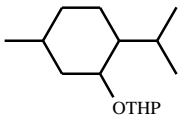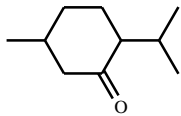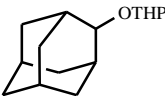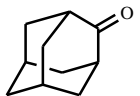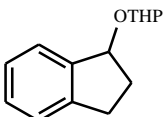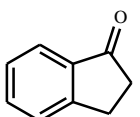
Entry	Substrate	Product	Time (min)	Yield (%)[c]
1	$C_6H_5CH_2OSiMe_3$	C_6H_5CHO	1	98
2	$2\text{-}ClC_6H_4CH_2OSiMe_3$	$2\text{-}ClC_6H_4CHO$	1	98
3	$4\text{-}ClC_6H_4CH_2OSiMe_3$	$4\text{-}ClC_6H_4CHO$	1	98
4	$2\text{-}BrC_6H_4CH_2OSiMe_3$	$2\text{-}BrC_6H_4CHO$	1	98
5	$4\text{-}BrC_6H_4CH_2OSiMe_3$	$4\text{-}BrC_6H_4CHO$	1	98
6	$3,4\text{-}Cl_2C_6H_3CH_2OSiMe_3$	$3,4\text{-}Cl_2C_6H_3CHO$	1	98
7	$2\text{-}MeC_6H_4CH_2OSiMe_3$	$2\text{-}MeC_6H_4CHO$	1	98
8	$3\text{-}MeOC_6H_4CH_2OSiMe_3$	$3\text{-}MeOC_6H_4CHO$	0.5	98
9	$4\text{-}MeOC_6H_4CH_2OSiMe_3$	$4\text{-}MeOC_6H_4CHO$	0.5	98
10	$4\text{-}Me_3CC_6H_4CH_2OSiMe_3$	$4\text{-}Me_3CC_6H_4CHO$	0.5	98
11	$4\text{-}Me_2CHC_6H_4CH_2OSiMe_3$	$4\text{-}Me_2CHC_6H_4CHO$	0.5	98
12	$2\text{-}NO_2C_6H_4CH_2OSiMe_3$	$2\text{-}NO_2C_6H_4CHO$	5	95
13	$4\text{-}NO_2C_6H_4CH_2OSiMe_3$	$4\text{-}NO_2C_6H_4 CHO$	4	95
14	$Ph_2CHOSiMe_3$	Ph_2CO	2	98
15	$PhCH(OSiMe_3)Me$	$PhCOMe$	1	98
16	$PhCH_2CH_2OSiMe_3$	$PhCH_2CHO$	2	98
17			4	98
18			3	98
19			3	98
20	$C_6H_5CH_2OTHP$	C_6H_5CHO	3	98
21	$2\text{-}ClC_6H_4CH_2OTHP$	$2\text{-}ClC_6H_4CHO$	4	98
22	$4\text{-}ClC_6H_4CH_2OTHP$	$4\text{-}ClC_6H_4CHO$	4	98
23	$2\text{-}BrC_6H_4CH_2OTHP$	$2\text{-}BrC_6H_4CHO$	5	98
24	$4\text{-}BrC_6H_4CH_2OTHP$	$4\text{-}BrC_6H_4CHO$	4	98
25	$3,4\text{-}Cl_2C_6H_3CH_2OTHP$	$3,4\text{-}Cl_2C_6H_3CHO$	5	98
26	$2\text{-}MeC_6H_4CH_2OTHP$	$2\text{-}MeC_6H_4CHO$	4	98
27	$3\text{-}MeOC_6H_4CH_2OTHP$	$3\text{-}MeOC_6H_4CHO$	4	98
28	$4\text{-}MeOC_6H_4CH_2OTHP$	$4\text{-}MeOC_6H_4CHO$	4	98
29	$4\text{-}Me_3CC_6H_4CH_2OTHP$	$4\text{-}Me_3CC_6H_4CHO$	4	98

Table 3. *(Continued).*

30	$4\text{-Me}_2\text{CHC}_6\text{H}_4\text{CH}_2\text{OTHP}$	$4\text{-Me}_2\text{CHC}_6\text{H}_4\text{CHO}$	3	98
31	$3\text{-NO}_2\text{C}_6\text{H}_4\text{CH}_2\text{OTHP}$	$3\text{-NO}_2\text{C}_6\text{H}_4\text{CHO}$	6	98
32	$4\text{-NO}_2\text{C}_6\text{H}_4\text{CH}_2\text{OTHP}$	$4\text{-NO}_2\text{C}_6\text{H}_4\text{CHO}$	9	90
33	Ph_2CHOTHP	Ph_2CO	4	98
34	PhCH(OTHP)Me	PhCOMe	3	98
35	$\text{PhCH}_2\text{CH}_2\text{OTHP}$	PhCH_2CHO	2	98
36			2	98
37			2	98
38			3	98
39			3	98
40	$4\text{-MeOC}_6\text{H}_4\text{CH}_2\text{OCH}_2\text{OMe}$	$4\text{-MeOC}_6\text{H}_4\text{CHO}$	30	0^d
41	$4\text{-ClC}_6\text{H}_4\text{CH}_2\text{OCH}_2\text{OMe}$	$4\text{-ClC}_6\text{H}_4\text{CHO}$	30	0^d
42	$4\text{-MeOC}_6\text{H}_4\text{CH}_2\text{OH}$ + $4\text{-MeOC}_6\text{H}_4\text{CH}_2\text{OCH}_2\text{OMe}$	$4\text{-MeOC}_6\text{H}_4\text{CHO}$	8	100^d + 0^d
43	$4\text{-Me}_2\text{CHC}_6\text{H}_4\text{CH}_2\text{OSiMe}_3$ + $4\text{-MeOC}_6\text{H}_4\text{CH}_2\text{OCH}_2\text{OMe}$	$4\text{-Me}_2\text{CHC}_6\text{H}_4\text{CHO}$	2	100^d + 0^d
44	$4\text{-ClC}_6\text{H}_4\text{CH}_2\text{OTHP}$ + $4\text{-ClC}_6\text{H}_4\text{CH}_2\text{OCH}_2\text{OMe}$	$4\text{-ClC}_6\text{H}_4\text{CHO}$	5	100^d + 0^d

[a] Products were characterized by their physical constants, comparison with authentic samples and IR and NMR spectroscopy.
[b] Reactions were performed using 225 mg of CrO_3/SANM (CrO_3 content 1.25 mmol).
[c] Yields are based on isolated products.
[d] Conversion.

A plausible mechanism for this reaction is shown in Scheme 2. On the basis of this mechanism SANM can act as source of the proton for the acceleration of the reaction.

To show the efficiency of the selected method, Table 4 compares the results obtained in the present study with some of those reported in the literature.

It is important to note that, this oxidizing reagent system is more effective than the other oxidizing systems with respect to the amounts of the oxidizing agent, reaction times and yields. In addition, in some of the reported methods larger amounts of the catalyst and oxidant are needed while the present method is performed using much less amounts of both of CrO_3 and SANM under the grinding conditions.

Table 4. Comparison of our results with some of those reported in the literature.

Entry	Reaction conditions	Time (min)	Yield (%)	[Ref.]
1	HIO_4/ Poly(1,4-phenylene-2,5-pyridinedicarboxyamide) (1.05 mmol: 0.6 g), CH_3CN, reflux	1-6 h	5-98	[43][a]
2	CrO_3/Al_2O_3 (2 mmol: 2 g), Solvent-free, Grinding	5-10	68-95	[28][a]
3	$(NH_4)_2Cr_2O_7$/Silica chloride (1 mmol: 0.3 g), wet SiO_2 (0.2 g), Solvent-free, 80 °C	3-30	80-92	[34][a]
4	Trinitratocerium(IV) bromate/ $NaHSO_4.H_2O$ (0.5 mmol: 0.3 g), Solvent-free, r.t.	2-20	82-95	[40][b]
5	HIO_3/ $NaHSO_4.H_2O$ (1.5 mmol: 0.035 g), CH_3CN, reflux	0.17-6.3 h	70-95	[39][b]
6	$Fe(NO_3)_3.9 H_2O$/ KBr (2 mmol: 0.042 g), CH_3CN, reflux	0.67-3.3 h	30-95	[38][a]
7	$Fe(NO_3)_3.9 H_2O$/ KBr (2 mmol: 0.042 g), CH_3CN, reflux	0.25-3.3 h	30-92	[38][b]
8	CrO_3/ Silica sulfuric acid (1.25 mmol: 0.25 g), wet SiO_2 (0.5 g), CH_3CN, r.t.	1-30	85-90	[44][a]
9	CrO_3/ Silica sulfuric acid (1.25 mmol: 0.25 g), wet SiO_2 (0.5 g), CH_3CN, r.t.	1-90	82-95	[44][b]
10	CrO_3/ SANM (1.25 mmol: 0.1 g), Solvent-free, Grinding	2-31	80-98	This work[a]
11	CrO_3/ SANM (1.25 mmol: 0.1 g), Solvent-free, Grinding	0.5-9	95-98	This work[b]

[a] Oxidation of alcohols.
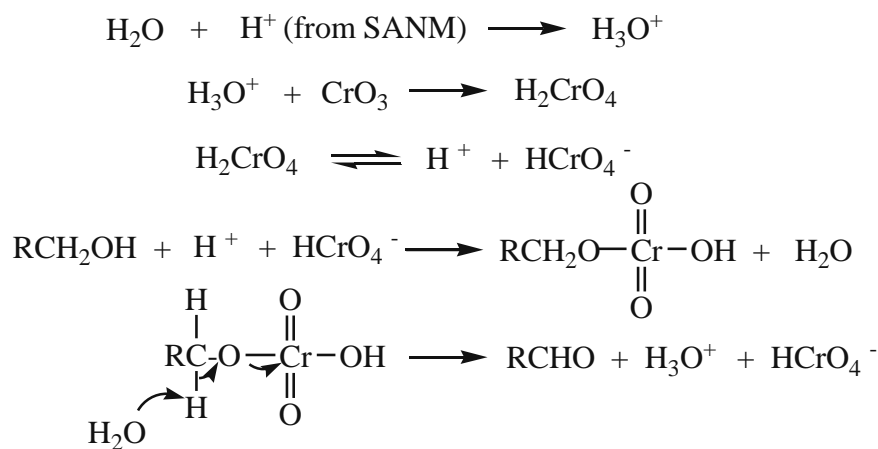[b] Oxidation of silyl and THP ethers.

4. Conclusions

In conclusion, we have introduced a mild, efficient and high yielding method for the oxidation of alcohols and trimethylsilyl and tetrahydropyranyl ethers using CrO_3 in the presence of SANM as an efficient oxidizing reagent system. Ease of preparation and handling of the catalyst, simple procedure and easy work-up, high reaction rates and excellent yields of the products are among the other advantages of this method, which make this procedure a useful and attractive addition to the available methods.

$$H_2O \ + \ H^+ \text{(from SANM)} \longrightarrow H_3O^+$$

$$H_3O^+ \ + \ CrO_3 \longrightarrow H_2CrO_4$$

$$H_2CrO_4 \rightleftharpoons H^+ \ + \ HCrO_4^-$$

$$RCH_2OH + H^+ + HCrO_4^- \longrightarrow RCH_2O{-}\overset{\overset{O}{\|}}{\underset{\underset{O}{\|}}{Cr}}{-}OH \ + \ H_2O$$

$$\underset{\underset{H_2O}{\nwarrow}}{RC}\overset{H}{\underset{H}{|}}{-}O{=}\overset{\overset{O}{\|}}{\underset{\underset{O}{\|}}{Cr}}{-}OH \longrightarrow RCHO + H_3O^+ + HCrO_4^-$$

Scheme 2. Proposed mechanism of this reaction.

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