Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

# Catalytic Esterifications of Carboxylic Acids and Alcohols by Sodium Bisulfate Monohydrate

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Published online: 17 Sep 2007.

To cite this article: Yi-Qun Li (1999): Catalytic Esterifications of Carboxylic Acids and Alcohols by Sodium Bisulfate Monohydrate, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 29:22, 3901-3903

To link to this article: http://dx.doi.org/10.1080/00397919908085911

SYNTHETIC COMMUNICATIONS, 29(22), 3901–3903 (1999)

# CATALYTIC ESTERIFICATIONS OF CARBOXYLIC ACIDS AND ALCOHOLS BY SODIUM BISULFATE MONOHYDRATE

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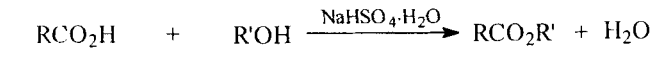
**Abstract:** The efficient esterification of primary and secondary alcohols with aliphatic carboxylic acids in the presence of a catalytic amount of sodium bisulfate monohydrate to afford the corresponding esters in high yields.

Esterification of hydroxyl groups is an important and well-established reaction in organic synthesis. The traditional methods use acid and alcohol in the presence of classic mineral acids such as sulfuric acid[1], which are highly corrosive in nature and usually accompanying side reactions such as carbonization, oxidation and dehydration, etc. Several esterification catalysts, which have been found some advantages over the classic acid catalysts, have been reported in literature[2-8]. Some of these catalysts are also applicable for secondary alcohols, but most require long reaction times and tedious work up.

Recently, the transition metal salts such as $Cu(NO_3)_2{\cdot}3H_2O$[9], $FeSO_4{\cdot}XH_2O$[10]and $Fe(ClO_4)_3(ROH)_6/SiO_2$[11] etc. catalyzed esterification have gained wide interest. Herein we wish to report a alkali metal salts, sodium bisulfate monohydrate, catalyzed rapid, convenient and general synthesis of esters from aliphatic carboxylic acids and primary and secondary alcohols (scheme 1).




Scheme 1

**Table 1. Esterification of acid with primary and secondary alcohol[a]**

| Entry | Acid | Alcohol | Time(h) | b.p. (°C) | Yields(%)[b] |
|---|---|---|---|---|---|
| 1 | Acetic acid | *n*-butanol | 0.5 | 124~126 | 96.2 |
| 2 | Acetic acid | *sec*- butanol | 1.5 | 110~112 | 84.5 |
| 3 | Acetic acid | *iso*- butanol | 1.0 | 111~113 | 92.5 |
| 4 | Acetic acid | *n*-pentanol | 0.5 | 146~149 | 93.6 |
| 5 | Acetic acid | *iso*- pentanol | 0.5 | 138~142 | 85.9 |
| 6 | Acetic acid | cyclohexanol | 1.5[c] | 168~174 | 84.9 |
| 7 | Acetic acid | benzyl alcohol | 2.0[c] | 202~206 | 73.7 |
| 8 | Propanoic acid | *n*- butanol | 0.5 | 143~145 | 95.8 |
| 9 | Propanoic acid | *sec*- butanol | 1.5 | 130~132 | 91.5 |
| 10 | Propanoic acid | *iso*- butanol | 0.5 | 134~136 | 95.1 |
| 11 | Propanoic acid | *n*- pentanol | 0.7 | 165~169 | 88.5 |
| 12 | Propanoic acid | *iso*- pentanol | 0.6 | 157~161 | 95.0 |
| 13 | Chloroacetic acid | ethanol | 2.0[c] | 144~146 | 90.4 |
| 14 | Chloroacetic acid | *iso*-propanol | 1.5[c] | 146~150 | 82.9 |
| 15 | Trichloroacetic acid | ethanol | 2.0[c] | 164~168 | 84.5 |

**a The molar ratio of acid and alcohol is 1:1, unless otherwise noted**

**b Isolated yields**

**c Use 30ml cyclohexane as azeotropic entrainer and the molar ratio of acid and alcohol is 1.5:1.0**

Esterification of acids and alcohols in the presence of a catalytic amount of $NaHSO_4 H_2O$ is performed under reflux and produces the desired esters in excellent yields (Table 1).

The experimental results show that the esters can be synthesized in large quantities, the reaction condition is mild, the reaction time is short, the yields are excellent and the operation is simple.

## Experimental

### The esterification of acetic acid with *n*-butanol; Typical Procedure

Place a mixture of 0.4mol of acetic acid, 0.4mol of alcohol, 1.0g of $NaHSO_4 H_2O$ and several pieces of boiling chips in a flask, equipped with an automatic water separator carrying a reflux condenser at its upper end. Reflux the mixture on a heating mantle till no more water collects in appreciable amount in the water separator. Filter off the catalyst. The filtrate is washed with 10% sodium bicarbonate solution, brine and dried over anhydrate sodium sulfate, then distilled under the normal pressure to afford the corresponding ester.

## Acknowledgement

This paper was supported by the Nature Science Foundation of Guangdong province.

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accepted 17-3-99