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ENVIROCAT EPIC[R] AS A NOVEL CATALYST FOR ACYLATION OF ANISOLE USING BENZOIC ACIDS[1]

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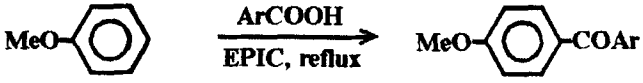
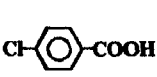
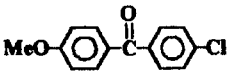
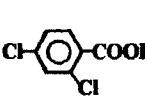
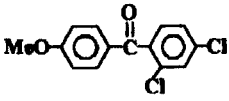
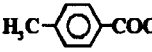
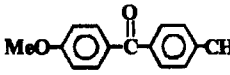
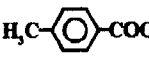
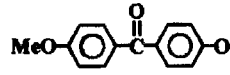
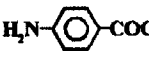
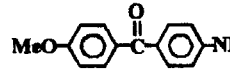
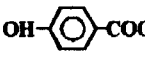
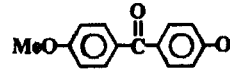
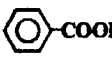
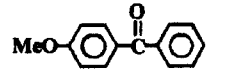
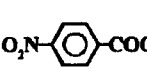
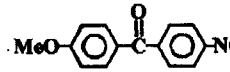
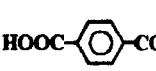
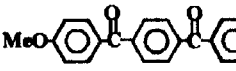
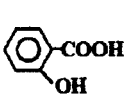
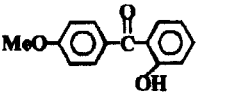
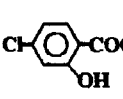
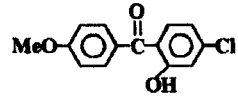
ABSTRACT : Friedel-Crafts acylation of anisole has been carried out using Envirocat EPIC[R] as a novel heterogenous catalyst and benzoic acids under reflux condition. This method is suitable for regioselective acylation of activated aromatics with benzoic acids having electron-donating substituents.

Envirocat[R] a new family of supported reagents, is a significant break-through in environmentally-friendly chemistry. These solid supported reagents are non-toxic powders which can be filtered easily from the process and may be reused. Envirocat EPIC[R] is one of the solid supported acid catalyst. Recently we have used Envirocat EPZG[R] for acetalization of carbonyl compounds[2], synthesis of conjugated nitroolefins[3] and chemoselective silylation of alcohols[4]. We report herein acylation of anisole with benzoic acids using Envirocat EPIC[R] as a solid supported acid catalyst (scheme).



*To whom correspondence should be addressed.

Table 1 : Friedel - Crafts acylation of anisole with aromatic acids and Envirocat EPIC®

Entry	Aromatic acid	Product	Reaction Time(min)	Yield (%)	MP (°C)
1			10	69	113-114
2			8.5	55	120
3			05	55	90-92
4			8.5	38	140-142
5			20	25	138-141
06			08	49	149-151
7			21	20	60-61
8			20	No reaction	
9			12	No reaction	
10			12.5	No reaction	
11			12.5	No reaction	

Results for the Friedel-Crafts acylation of anisole with aromatic acids and Envirocat EPIC[R] are summarized in table 1. Benzoic acids with electron donating groups undergo complexation easily with the catalyst resulting in the formation of the electrophilic intermediate (entries 1-7). Therefore, activated aromatic compound, anisole undergoes acylation with these benzoic acids at 4-position only (entries 1-7). But in case of benzoic acids with electron-withdrawing groups (entries 8-9) and 2-hydroxy group (entries 10-11), complexation with the catalyst may be difficult. Therefore, acylation of anisole with these acids (entries 8-11) is diffcult

In conclusion, regioselective acylation of anisole with substituted benzoic acids could be successfully carried out using EPIC[R] as a non-polluting heterogenous catalyst. Considering importance of substituted benzophenones as fine chemicals, we believe that this new acylation methodology could be useful addition to synthetic organic chemistry.

Experimental :

Envirocat EPIC[R] was procured from Contract Chemicals, England and it was used as obtained. All chemicals were of analytical grade. Products were characterized by their physical constants and spectral characteristics (IR and ^{1}H NMR)

General procedure for acylation of anisole using benzoic acids and Envirocat EPIC[R] :

A mixture aromatic carboxylic acid (4 mmol) and Envirocat EPIC[R] (100 mg) in anisole (15 ml) was refluxed for the time specified in table 1. The reaction was monitored by TLC. After completion of reaction, mixture was filtered off and catalyst was washed with ether (3 x 10 ml). Removal of the solvent under reduced pressure afforded almost pure product.

Acknowledgement :

We thank Contract Chemicals, England for the generous gift of Envirocat EPIC[R].

References and footnotes :

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3. Bandgar, B. P.; Zirange, M. B. and Wadgaonkar, P. P. *Synlett.* **1996**, 149.
4. Bandgar, B. P. and Wadgaonkar, P. P. *Synth. Commun.* **1997**, 27, 2069.

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