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## Friedel–Crafts acylation of arenes with carboxylic acids using polystyrene-supported aluminum triflate

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**Abstract:** Cross-linked polystyrene-supported aluminum triflate (Ps–Al(OTf)<sub>3</sub>) has been shown to be a mild, efficient, and chemoselective heterogeneous Lewis acid catalyst for the acylation of aromatic compounds. The catalyst can be easily prepared from cheap starting materials, is stable (as a bench top catalyst) and is reusable.

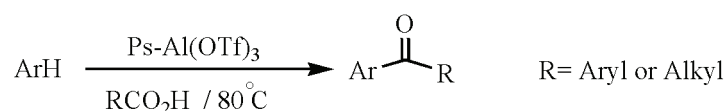
**Keywords:** acylation; ketones; aluminum triflate; polystyrene.

### INTRODUCTION

Friedel–Crafts acylation of arenes is of great importance in both laboratory work and industry processes to synthesize aromatic ketones.<sup>1,2</sup> Usually, this reaction is performed using acid chlorides or anhydrides<sup>3–12</sup> or carboxylic acids<sup>13–25</sup> as acylating agents in the presence of protic acids or Lewis acids. Acylation of arenes using carboxylic acids is preferable to the acylation *via* acid chlorides or anhydrides because the former reaction produces only water as a by-product, which meets recent requirements for environmentally benign chemical processes. Furthermore, carboxylic acids are stable and more available compounds and their handling is much easier than that of the corresponding acid chlorides or anhydrides. However, only a few studies on the use of carboxylic acids as acylating agents have been reported and many of these procedures have serious drawbacks, such as a need for the use of stoichiometric or excess amounts of catalyst in the reactions, tedious work-up, environmental pollution, long reaction times, high reaction temperatures, highly corrosive conditions, the formation of various by-products and the use of moisture-sensitive, non-recyclable or difficult to handle catalysts. In view of this, a reliable method for this useful reaction involving heterogeneous catalysts is demanded.

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Triflic acid (trifluoromethanesulfonic acid), known to be a strong acid, is suitable for use as a catalyst for synthetic applications.<sup>26–28</sup> However, the recovery of the triflic acid from the reaction mixture results in the formation of large amounts of waste, which is environmentally unacceptable. In continuation of our ongoing program to develop environmentally benign methods using heterogeneous Lewis acid catalysts, it was found that Ps–Al(OTf)<sub>3</sub> is a good solid catalyst for highly chemoselective dithioacetalization of carbonyl compounds.<sup>29</sup> Along this line, it is herein reported that Ps–Al(OTf)<sub>3</sub> is also an effective and highly chemoselective catalyst for the acylation of aromatic compounds with carboxylic acids under mild reaction conditions (Scheme 1).



Scheme 1. Acylation of arenes with carboxylic acids using Ps–Al(OTf)<sub>3</sub>.

#### EXPERIMENTAL

The employed chemicals were either self-prepared or were purchased from Merck and Fluka. Polystyrene (8 % divinylbenzene, prepared *via* suspension polymerization, using poly(vinylpyrrolidone) 90 K as the suspension agent, grain size range: 0.25–0.6 mm) was obtained from the Iran Polymer and Petrochemical Institute. Capacity of the catalyst was determined by gravimetric method and atomic absorption technique using a Philips PU9400X atomic absorption spectrophotometer. Reaction monitoring and purity determination of the products were accomplished by GLC or TLC on silica-gel polygram SILG/UV254 plates. Gas chromatography was performed on a Shimadzu GC 14-A. The IR spectra were recorded on a Shimadzu model 8300 FT-IR spectrophotometer. The NMR spectra were recorded on a Bruker Advance DPX-300 spectrometer.

##### Preparation of Ps–Al(OTf)<sub>3</sub>

This catalyst was prepared as reported in the literature.<sup>29</sup> The determined loading of Al(OTf)<sub>3</sub> was 0.41 mmol g<sup>-1</sup>.

##### Typical experimental procedure

To a solution of arene (5 mmol) and carboxylic acid (3.5 mmol) was added 0.35 mmol of Ps–Al(OTf)<sub>3</sub> and the reaction mixture was stirred at 80 °C. After completion of the reaction (monitored by TLC and GC), the catalyst was filtered off and washed with CH<sub>2</sub>Cl<sub>2</sub>. The filtrate was washed with 10 % NaHCO<sub>3</sub> solution (2×10 mL) and water (2×10 mL) and the organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The unreacted anisole was removed by vacuum distillation and the aryl ketone product was isolated in high purity in yields from 87 to 97 %. Whenever required, the products were purified by column chromatography (silica gel) using petroleum ether–ethyl acetate as the eluent, whereby pure ketone was obtained.

#### RESULTS AND DISCUSSION

Ps–Al(OTf)<sub>3</sub> was prepared by the exchange reaction between cross-linked polystyrene supported AlCl<sub>3</sub> (Ps–AlCl<sub>3</sub>) and triflic acid in Freon-113 under reflux conditions. Using catalytic amounts of this catalyst in the Friedel–Crafts

acylation of arenes with carboxylic acids as acylating agents under solvent-free conditions, high to excellent yields of product were obtained (Table I). The optimum molar ratio of Ps-Al(OTf)<sub>3</sub> to carboxylic acid was 0.1:1. From the results, it is clear that Ps-Al(OTf)<sub>3</sub> is capable of catalyzing not only the acylation of activated arenes, but also of deactivated arenes (Table I, entries 1–23). The methodology showed the excellent positional selectivity as the *para* substituted product was formed almost exclusively. Naphthalene, 2-methoxynaphthalene and anthracene underwent acylation with high regioselectivity in 87–93 % yields (Table I, entries 24–30). It was satisfying to observe that even heterocyclic compounds, such as furan, thiophene and pyrrole, were smoothly converted into the corresponding ketones, a conversion which is otherwise problematic in the presence of strong acid catalysts (Table I, entries 31–36). These reactions were regioselective, affording only the 2-acyl product in high to excellent yields. The acylation of indole with benzoic acid in the presence of Ps-Al(OTf)<sub>3</sub> was also studied and the corresponding indolyl aryl ketone was obtained with high regioselectivity in 90 % yield (Table I, entry 37). No *N*-substituted products were observed under these reaction conditions. Acylation of highly deactivated arenes, such as nitrobenzene and 1,2-dichlorobenzene failed.

TABLE I. Acylation of arenes with carboxylic acids catalyzed by Ps-Al(OTf)<sub>3</sub> (all reactions performed at 80 °C in the absence of solvent, unless otherwise indicated. The molar ratio of Ps-Al(OTf)<sub>3</sub>:carboxylic acid is 0.1:1)


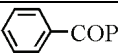

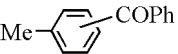

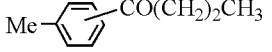
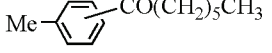
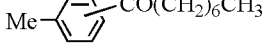
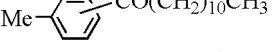
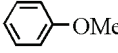
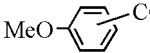
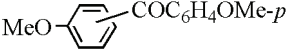

Entry	Arene	Carboxylic acid	Product	Time h	Yield, % ( <i>o:m:p</i> ) <sup>a</sup>	Ref.
1		PhCO <sub>2</sub> H		3.3	90	20,30
2		PhCO <sub>2</sub> H		3	92 (6:3:91)	25
3		<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H		3.1	90 (3:0:97)	25
4		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>2</sub> CO <sub>2</sub> H		2.9	95 (4:3:93)	13
5		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>5</sub> CO <sub>2</sub> H		2.9	94 (5:3:92)	13
6		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>6</sub> CO <sub>2</sub> H		3	88 (4:0:96)	13
7		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>10</sub> CO <sub>2</sub> H		3.1	85 (4:0:96)	13
8		PhCO <sub>2</sub> H		3	94 (4:0:96)	25
9		<i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H		2.9	95 (5:3:92)	15,25
10		MeCO <sub>2</sub> H		2.9	94 (6:4:90)	31–33

TABLE I. Continued

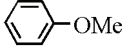
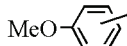
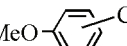
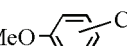
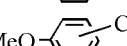
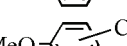
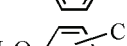
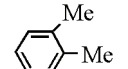
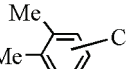
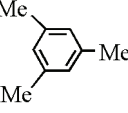
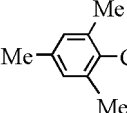
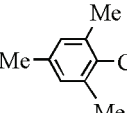
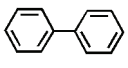
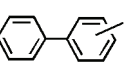
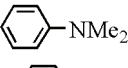
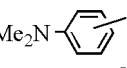
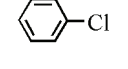
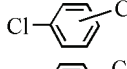
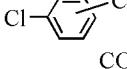
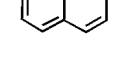
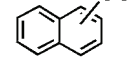
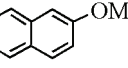
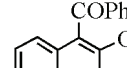
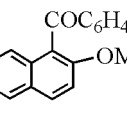
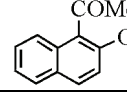
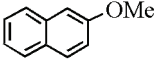
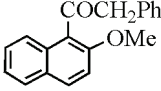
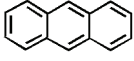
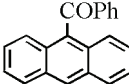
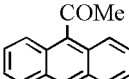

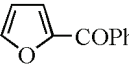
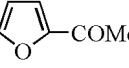


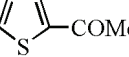
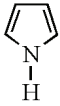
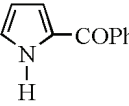
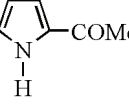

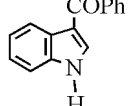
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11		PhCH <sub>2</sub> CO <sub>2</sub> H		2.8	94 (6:4:90)	34
12		PhCH=CHCO <sub>2</sub> H		3.1	90 (3:0:97)	25,35
13		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>2</sub> CO <sub>2</sub> H		2.7	97 (5:4:91)	16,18
14		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>5</sub> CO <sub>2</sub> H		2.8	95 (5:3:92)	16,18
15		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>6</sub> CO <sub>2</sub> H		3	93 (3:0:97)	16,18
16		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>10</sub> CO <sub>2</sub> H		3	92 (3:0:97)	16,18
17		CH <sub>3</sub> (CH <sub>2</sub> ) <sub>5</sub> CO <sub>2</sub> H		3	90 (95:5) <sup>b</sup>	21
18		PhCO <sub>2</sub> H		2.9	93 -	25
19		MeCO <sub>2</sub> H		2.8	94 -	18,36
20		PhCO <sub>2</sub> H		3.1	88 (3:4:97) <sup>c</sup>	30
21		PhCO <sub>2</sub> H		3	91 (7:3:90)	7,30
22		PhCO <sub>2</sub> H		3.5	87 (4:3:93)	30
23		MeCO <sub>2</sub> H		3.3	89 (5:3:92)	30
24		MeCO <sub>2</sub> H		3.3	88 (3:97) <sup>c,d</sup>	18
25		PhCO <sub>2</sub> H		3.1	93 <sup>c</sup> -	25
26		<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H		3	90 <sup>c</sup> -	25
27		MeCO <sub>2</sub> H		3.1	90 <sup>c</sup> -	18,37

TABLE I. Continued

Entry	Arene	Carboxylic acid	Product	Time h	Yield, % ( <i>o:m:p</i> ) <sup>a</sup>	Ref.
28		PhCH <sub>2</sub> CO <sub>2</sub> H		3	90 <sup>c</sup> –	18,38
29		PhCO <sub>2</sub> H		3.4	87 <sup>c</sup> –	25
30		MeCO <sub>2</sub> H		3.3	89 <sup>c</sup> –	18,37
31		PhCO <sub>2</sub> H		3.5	89 <sup>e</sup> –	20,39
32		MeCO <sub>2</sub> H		3.3	90 <sup>e</sup>	5,40
33		<i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CO <sub>2</sub> H		3.6	89 <sup>e</sup> –	5
34		MeCO <sub>2</sub> H		3.6	92 <sup>e</sup>	5,41
35		PhCO <sub>2</sub> H		3.4	87 <sup>e</sup> –	5,42
36		MeCO <sub>2</sub> H		3.3	89 <sup>e</sup> –	18,42
37		PhCO <sub>2</sub> H		4	90 <sup>c</sup> –	43–45

<sup>a</sup>Isolated yields. Isomer distribution based on <sup>1</sup>H-NMR spectroscopy and GC. All products are known compounds and were identified by comparison of their physical and spectral data with those of authentic samples;

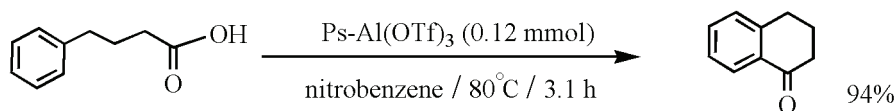
<sup>b</sup>isomer distribution of the 3,4-dimethylphenyl isomer to the 2,3-dimethylphenyl isomer; <sup>c</sup>the reaction was performed in 1,2-dichloroethane; <sup>d</sup> $\alpha$ : $\beta$  ratio; <sup>e</sup>the reaction was performed at 60 °C.

When Ps–Al(OTf)<sub>3</sub> was used as the catalyst in the acylation reactions, no band corresponding to –CO stretching in the IR spectrum of Ps–Al(OTf)<sub>3</sub> was observed after the reactions in either the presence or absence of substrate, indicating that polystyrene itself did not undergo acylation under the employed experimental conditions. Probably acylation reactions are not favored with Ps–Al(OTf)<sub>3</sub> as a  $\pi$  complex is formed between the polystyrene and Al(OTf)<sub>3</sub>.<sup>29,46</sup> By-product formation was not observed in the studied reactions. Steric crowding

of the supported catalyst influences the positional selectivity (isomer distribution) observed in the acylation of aromatic compounds.

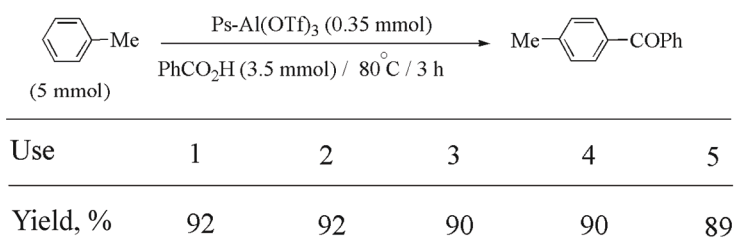
To determine whether the reaction occurs in the solid matrix of  $\text{Ps-Al(OTf)}_3$  or whether  $\text{Al(OTf)}_3$  simply released into the reaction medium is responsible for the acylation reaction,  $\text{Ps-Al(OTf)}_3$  was added to toluene and the mixture was stirred at  $80^\circ\text{C}$  for 2 h. Then, the catalyst was filtered off and the filtrate was analyzed for its aluminum content, which showed a negligible release of  $\text{Al(OTf)}_3$ . The filtrate was found to be inactive for acylation reaction. These observations indicate that  $\text{Ps-Al(OTf)}_3$  is stable under the employed reaction conditions and there was no leaching of acid moieties during the studied reactions.

One notable achievement of this solid acid catalyst was intramolecular Friedel-Crafts acylation. For example, 4-phenylbutanoic acid cyclized in nitrobenzene at  $80^\circ\text{C}$  in the presence of  $\text{Ps-Al(OTf)}_3$  to afford the desired 1-tetralone in 94 % yield (Scheme 2).



Scheme 2. Intramolecular Friedel-Crafts acylation using  $\text{Ps-Al(OTf)}_3$ .

$\text{Ps-Al(OTf)}_3$  recovered after a reaction can be washed with dichloromethane and used again at least five times without any noticeable loss of catalytic activity (Scheme 3). The capacity of the catalyst after five uses was 0.40 mmol  $\text{Al(OTf)}_3$  per gram of polymeric catalyst.



Scheme 3. Acylation of toluene using recovered  $\text{Ps-Al(OTf)}_3$  (recovered catalyst was successfully reused).

A comparison of the efficiency of  $\text{Ps-Al(OTf)}_3$  catalyst with some of those reported in the literature is given in Table II. As can be seen, in addition to having the general advantages attributed to solid supported catalysts,  $\text{Ps-Al(OTf)}_3$  also has good efficiency compared to other recently reported catalysts.

Representative spectral data of some of the obtained compounds are given below.

TABLE II. Comparison of the catalytic activity of Ps-Al(OTf)<sub>3</sub> against other reported catalysts for the acylation of anisole and toluene with carboxylic acids

Arene	Carboxylic acid	Catalyst	Temperature, °C	Time, h	Yield, % (o:p)
Anisole	Benzoic acid	Ps-Al(OTf) <sub>3</sub>	80	3	94(4:96)
		AIPW <sub>12</sub> O <sub>40</sub>	120	10	92 <sup>18</sup>
		AIPW <sub>12</sub> O <sub>40</sub> /TFAA <sup>a</sup>	25	2.5	96 <sup>18</sup>
		P <sub>2</sub> O <sub>5</sub> /Al <sub>2</sub> O <sub>3</sub>	85	5	65 <sup>25</sup>
Anisole	Heptanoic acid	Ps-Al(OTf) <sub>3</sub>	80	2.8	95(5:92)
		HZSM-5 zeolite	150	48	5 <sup>14</sup>
		Eu(NTf <sub>2</sub> ) <sub>3</sub>	250	6	87(3:97) <sup>21</sup>
Anisole	Octanoic acid	Ps-Al(OTf) <sub>3</sub>	80	3	93(3:97)
		Cs <sub>2.5</sub> H <sub>0.5</sub> PW <sub>12</sub> O <sub>40</sub>	110	5	45(1:73) <sup>16</sup>
		Beta zeolite	155	6	42.6(1:38) <sup>22</sup>
Anisole	Dodecanoic acid	Ps-Al(OTf) <sub>3</sub>	80	3	92(3:97)
		FePW <sub>12</sub> O <sub>40</sub>	160	1	97(2:98) <sup>24</sup>
Toluene	Octanoic acid	Ps-Al(OTf) <sub>3</sub>	80	3	88(4:96)
		CeNaY zeolite	150	48	75(3:94) <sup>13</sup>

<sup>a</sup>Trifluoroacetic anhydride

*4-Methylbenzophenone*. IR (KBr, cm<sup>-1</sup>): 1660 (C=O). <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 2.51 (3H, s), 7.51 (2H, d, J = 8.31 Hz), 7.87–8.15 (7H, m).

*1-(4-Methoxyphenyl)-3-phenyl-2-propen-1-one*. IR (KBr, cm<sup>-1</sup>): 1650 (C=O). <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 3.91 (3H, s), 6.99 (2H, d, J = 8.71 Hz), 7.47 (3H, m), 7.59 (1H, d, J = 15.15 Hz), 7.69 (2H, m), 7.86 (1H, d, J = 15.15 Hz), 8.11 (2H, d, J = 8.71 Hz).

*1-(3,4-Dimethylphenyl)-1-heptanone*. IR (KBr, cm<sup>-1</sup>): 1685 (C=O). <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 0.82–0.91 (3H, m), 1.23–1.41 (6H, m), 1.63–1.74 (2H, m), 2.26–2.33 (6H, m), 2.80 (0.1H, t, J = 7.2 Hz, 2,3-dimethyl isomer), 2.90 (1.9H, t, J = 7.2 Hz, 3,4-dimethyl isomer), 7.11 (0.05H, t, J = 7.2 Hz, 2,3-dimethyl isomer), 7.13–7.20 (1H, m), 7.25 (0.05H, d, J = 7.2 Hz, 2,3-dimethyl isomer), 7.65 (0.95H, d, J = 7.2 Hz, 3,4-dimethyl isomer), 7.70 (0.95H, d, J = 7.2 Hz, 3,4-dimethyl isomer).

*2,4,6-Trimethylbenzophenone*. IR (KBr, cm<sup>-1</sup>): 1669 (C=O). <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 2.2 (6H, s, 2- and 6-Me), 2.4 (3H, s, 4-Me), 6.91 (2H, s, aromatic protons of mesityl group), 7.45 (2H, m), 7.55 (1H, m), 7.81 (2H, m).

*1-Benzoyl-2-methoxynaphthalene*. IR (KBr, cm<sup>-1</sup>): 1665 (C=O). <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>, δ / ppm): 3.88 (3H, s), 7.31–7.52 (7H, m), 7.55–8.12 (4H, m).

#### CONCLUSIONS

In conclusion, a convenient and chemoselective method of the acylation of aromatic compounds has been devised. The significant advantages of this methodology are mild reaction conditions, high to excellent yields, solvent-free conditions and easy preparation and handling of the catalyst. In addition, the use

of  $\text{Ps-Al}(\text{OTf})_3$  resulted in a reduction in the unwanted and hazardous waste that is produced during conventional homogeneous processes.

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## ИЗВОД

ФРИДЕЛ–КРАФТСОВО АЦИЛОВАЊЕ АРЕНА КАРБОКСИЛНИМ КИСЕЛИНАМА  
ПОМОЋУ АЛУМИНИЈУМ-ТРИФЛАТА НА ПОЛИСТИРЕНУКАВЕН PARVANAK BOROUJENI<sup>1</sup> и КАМРАН PARVANAK<sup>2</sup><sup>1</sup>Department of Chemistry, Shahrekord University, Shahrekord 115 u <sup>2</sup>Islamic Azad University, Shahr-e-Ray Branch, Iran

Показано је да је Луисова киселина алуминијум-трифлат на унакрсно повезаном полистирену благ, ефикасан и хемоселективан катализатор за ациловање ароматичних једињења. Катализатор се лако припрема из јефтених полазних једињења, стабилан је и може се више пута користити.

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