

# Indium triflate as a recyclable catalyst for the nitration of aromatic compounds without a halogenated solvent

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Indium triflate [In(OTf)<sub>3</sub>] was found to be excellent catalyst (0.5 or 2.0 mol%) for the mononitration of aromatic compounds using a single equivalent of nitric acid (60 or 90%, 1.0 equiv.) without using a halogenated solvent. The side product is water and the indium triflate catalyst can be readily recovered from the aqueous phase and reused.

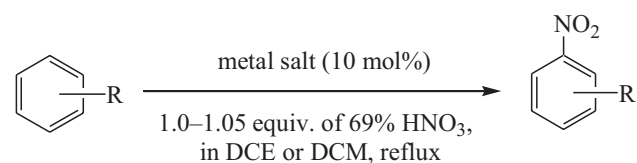
**Keywords:** nitration, aromatic compounds, indium triflate, recyclable catalyst

The nitration of organic compounds has long been a very active and rewarding area of research and is the subject of a large body of literature. Extensive and well documented reviews have been published by Ingold,<sup>1</sup> Olah *et al.*,<sup>2,3,6</sup> Schofield and coworkers,<sup>4,5</sup> and Ione and coworkers,<sup>7</sup> among others. Nitration as a manufacturing process requires the use of potent mixtures of concentrated or fuming nitric acid with sulfuric acid leading to excessive acid waste streams and added expense. Recently, efficient catalytic protocols have emerged including the use of Fe(III)<sup>8</sup> or Cu(II)<sup>9</sup> exchanged montmorillonite clay,<sup>10</sup> zeolites,<sup>11</sup> bismuth(III) salts,<sup>12</sup> group IV metal salts,<sup>13</sup> lanthanide(III) salts<sup>14–17</sup> and an indium complex,<sup>18</sup> and so on. These metal salts can catalyse the nitration of a range of simple aromatic compounds in good to excellent yields using stoichiometric quantities of 69% nitric acid. The only by-product is water and the catalyst can be readily recycled by simple evaporation (Scheme 1).<sup>13–18</sup>

However, in these catalytic systems, 10 mol% catalysts were used and 1,2-dichloroethane (DCE) or dichloromethane (DCM) were used as solvent, which made these methods expensive and environmentally hazardous. Here we wish to report a green nitration process using only 0.5 or 2.0 mol% of indium triflate as catalysts by 90% nitric acid without a halogenated solvent.

## Results and discussion

The use of indium(III) complexes as efficient Lewis acids is becoming increasingly widespread in catalytic organic synthesis, for example acylation,<sup>19</sup> Diels–Alder reactions,<sup>20–23</sup> Michael additions,<sup>24,25</sup> Mukaiyama–Aldol reaction,<sup>26</sup> glycosylation<sup>27</sup> and some reductions.<sup>28–31</sup> Frost and coworkers reported in 2002 that In(NTf<sub>2</sub>)<sub>3</sub> [NTf<sub>2</sub> = (CF<sub>3</sub>SO<sub>2</sub>)<sub>2</sub>N] and In(OTf)<sub>3</sub> (Tf = CF<sub>3</sub>SO<sub>2</sub>) can be used as efficient catalysts for catalytic aromatic electrophilic substitution reactions.<sup>18</sup> In this paper, the nitration of simple aromatic compounds was carried out with 69% nitric acid (1.0 equiv.) and indium salts (10 mol%) as catalysts in 1,2-dichloroethane (5.0 ml)



metal salt: Ln(OTf)<sub>3</sub>, Ln = La–Lu; M(OTf)<sub>4</sub>, M = Hf, Zr;  
M(OTf<sub>3</sub>)<sub>3</sub>, M = Sc, Yb; InX<sub>3</sub>, X = NTf<sub>2</sub>, OTf.

**Scheme 1** Catalytic nitration of aromatic compounds with 69% nitric acid.

under reflux for 6–18 h. Based on this result, we attempted the nitration of aromatic compounds using In(OTf)<sub>3</sub> (0.5–2.0 mol%) as catalyst and 90% HNO<sub>3</sub> (1.0 equiv.) as nitration reagent without a halogenated solvent since we believe that the aromatic compound itself can be used as solvent with higher concentrations of nitric acid. The indium triflate [In(OTf)<sub>3</sub>] was easily prepared by the reaction of indium oxide (In<sub>2</sub>O<sub>3</sub>) with trifluoromethanesulfonic acid (CF<sub>3</sub>SO<sub>3</sub>H).<sup>32</sup>

We examined the nitration of aromatic compounds with In(OTf)<sub>3</sub> (2.0 mol%) as catalyst and 90% HNO<sub>3</sub> (1.0 equiv.) as nitration reagent and the results are summarised in Table 1. As can be seen from Table 1, without using halogenated solvent, the nitration of various aromatic compounds indeed took place at 60°C to give the corresponding nitrated products in good yields (73–88%) (Table 1, entries 1, 3, 5–11 in column 3). Also the In(OTf)<sub>3</sub> catalyst was easily recovered from the aqueous layer just by heating the aqueous solution in an oven after extraction of the organic products with ether, and the recovered In(OTf)<sub>3</sub> catalyst can be reused for the next reaction. In our experiments, more than 95% of the catalyst was recovered after reaction. In addition, we found that similar results were given when recovered catalyst was used (Table 1, entries 2 and 4 in column 3). It should be emphasised here that the nitrated product was obtained

**Table 1** Nitration of aromatic compounds with In(OTf)<sub>3</sub>\*

Entry	R	Yield/% <sup>a</sup>	
		In (OTf) <sub>2</sub> (2.0 mol%)	In (OTf) <sub>2</sub> (0.5 mol%)
1	H	87	78
2	H	83 <sup>b</sup>	
3	F	88 <sup>c</sup>	74 <sup>c</sup>
4	F	81 <sup>b,c</sup>	
5	Cl	81 <sup>d</sup>	77 <sup>d</sup>
6	Br	78 <sup>e</sup>	79 <sup>e</sup>
7	Me	83 <sup>f</sup>	69 <sup>f</sup>
8	Et	79 <sup>g</sup>	71 <sup>g</sup>
9	<i>i</i> -Pr	76 <sup>h</sup>	64 <sup>h</sup>
10	Ph*	84	86
11 <sup>i</sup>		73	56

<sup>a</sup>Isolated yields. <sup>b</sup>Recycled catalyst was used. <sup>c</sup>*p:m:o* = 83:0:17. <sup>d</sup>*p:m:o* = 69:0:31. <sup>e</sup>*p:m:o* = 60:0:40. <sup>f</sup>*p:m:o* = 54:3:43. <sup>g</sup>*p:m:o* = 56:0:44. <sup>h</sup>*p:m:o* = 73:0:27. <sup>i</sup>Mixture of dimethylbenzenes.

\*See CAUTION in Experimental Section

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in < 10% yield if the  $\text{In}(\text{OTf})_3$  was absent under otherwise identical conditions. We next examined the nitration of aromatic compounds with  $\text{In}(\text{OTf})_3$  (0.5 mol%) and 90%  $\text{HNO}_3$  (1.0 equiv.) under identical conditions. The results are again summarised in Table 1. The yields of the corresponding nitration products in general can still reach 56–86% (Table 1, entries 1, 3 and 5–11 in column 4). Therefore, with 90%  $\text{HNO}_3$  as nitration reagent, an  $\text{In}(\text{OTf})_3$  catalyst loading of only 0.5 mol% is also fairly effective for the nitration of aromatic compounds.

For solid aromatic compounds such as naphthalene and 2-methylnaphthalene, the nitration can also be carried out under similar conditions at 60°C with 60% nitric acid (1.0 equiv.) as nitration reagent because they are more reactive to electrophilic substitution (Scheme 2). The corresponding nitrated products were obtained in 98% and 97% yields, respectively.

In conclusion, we have discovered a very efficient nitration process in the presence of indium triflate under mild reaction conditions. This new nitration process employs 0.5–2.0 mol% of indium triflate as catalyst without using a halogenated organic solvent. Thus, it is a much eco-safer protocol of nitration process. Efforts are underway to elucidate the mechanistic details for the this reaction and to identify systems enabling analogous reactions of other aromatic compounds, and the subsequent transformations thereof.

## Experimental

### General

M.P.s were obtained with a Yanagimoto micro melting point apparatus and are uncorrected.  $^1\text{H}$  NMR spectra were recorded on a Bruker AM-300 spectrometer for solutions in  $\text{CDCl}_3$  with tetramethylsilane (TMS) as internal standard;  $J$ -values are in Hz and for AA'XX' systems  $J^* = J_{23} + J_{25}$ . Mass spectra were recorded with a HP-5989 instrument and HRMS measurement were made on a Finnigan MA + mass spectrometer. Organic solvents were dried by standard methods when necessary. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with Huanghai  $\text{GF}_{254}$  silica gel coated plates. The orientation of nitration was determined by GC analysis. Flash column chromatography was carried out using 300–400 mesh silica gel.  $\text{In}(\text{CF}_3\text{SO}_3)_3$  was prepared from the reaction of  $\text{CF}_3\text{SO}_3\text{H}$  with  $\text{In}_2\text{O}_3$ . An excess amount of  $\text{In}(\text{III})$  oxide was added to an aqueous solution of  $\text{CF}_3\text{SO}_3\text{H}$  (50% v/v) and heated under reflux for 12 h. The mixture was filtered to remove the unreacted oxide. The water was then removed from the filtrate under reduced pressure. The resulting hydrate was dried by heating under vacuum at 180–200°C for 4 h.<sup>32</sup>

### General procedure for the nitration of aromatic compounds catalysed by $\text{In}(\text{OTf})_3$ (example used toluene)

$\text{In}(\text{OTf})_3$  (168 mg, 0.3 mmol, 2.0 mol%) was put into a glass vessel and then toluene (1.38 g, 15 mmol) was added to the glass vessel. Nitric acid (90%, 0.72 ml, 15 mmol, 1.0 equiv.) was slowly added dropwise, the reaction mixture was stirred for 4 h at room temperature and then kept stirring for 20 h at 60°C. The reaction mixture was washed with water (10 ml) and extracted with ethyl acetate (AcOEt). The solvent

was removed under reduced pressure and the residue was purified by a silica gel column chromatograph (eluent: petroleum ether/EtOAc = 20/1) to give nitrotoluene as a yellow liquid. 1.70 g, 83% yield; *p*: *m*: *o* = 54: 3: 43; 2-nitrotoluene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$  2.61 (3H, s,  $\text{CH}_3$ ), 7.20–7.45 (2H, m, Ar), 7.48 (1H, d,  $J = 7.5$  Hz, Ar), 7.96 (1H, d,  $J = 7.5$  Hz, Ar); 3-nitrotoluene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$  2.48 (3H, s,  $\text{CH}_3$ ), 7.40–7.51 (2H, m, Ar), 7.92–8.09 (2H, m, Ar); 4-nitrotoluene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$  2.48 (3H, s,  $\text{CH}_3$ ), 7.33 (2H, m,  $J^* = 8.4$  Hz, Ar), 8.13 (2H, m,  $J^* = 8.4$  Hz, Ar). MS (EI)  $m/z$  137 ( $\text{M}^+$ ) [Calc. for  $\text{C}_7\text{H}_7\text{NO}_2$  (137.1360): requires M, 137.0477. Found:  $\text{M}^+$  137.0464].

**CAUTION:** The isolation of nitrated products must be carried out carefully with due precautions as some such compounds are known human carcinogens. The manufacture of 4-nitrobiphenyl and its use for all purposes is prohibited in the UK.

*Nitration of fluorobenzene:* A yellow oil, 1.85 g, 88% yield; *p*: *m*: *o* = 83: 0: 17; 4-fluoronitrobenzene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$  7.20–7.25 (2H, m, Ar), 8.26–8.31 (2H, m, Ar); 2-fluoronitrobenzene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$  7.27–7.34 (2H, m, Ar), 7.62–7.70 (1H, m, Ar), 8.05–8.10 (1H, m, Ar). MS (EI)  $m/z$  141 ( $\text{M}^+$ ) [Calc. for  $\text{C}_6\text{H}_4\text{FNO}_2$  (141.0999): requires M, 141.0226. Found:  $\text{M}^+$  141.0239].

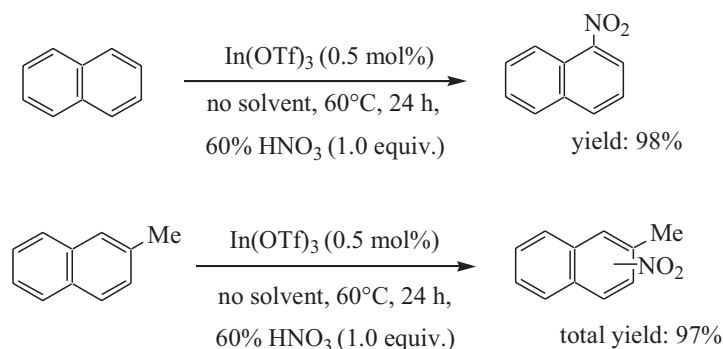
*Nitration of chlorobenzene:* Total 1.92 g, 81% yield; *p*: *m*: *o* = 69: 0: 31; 4-chloronitrobenzene: a white solid, m.p. 84–86°C,  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 7.52 (2H, d,  $J = 7.8$  Hz, Ar), 8.20 (2H, d,  $J = 7.8$  Hz, Ar); 2-chloronitrobenzene: a white solid, m.p. 32–34°C,  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 7.43 (1H, t,  $J = 7.5$  Hz, Ar), 7.5–7.58 (2H, m, Ar), 7.89 (1H, d,  $J = 7.5$  Hz, Ar). MS (EI)  $m/z$  158 ( $\text{M}^+$ ) [HRMS calcd for  $\text{C}_6\text{H}_4\text{ClNO}_2$  (157.5542): requires M, 156.9931. Found:  $\text{M}^+$  156.9915].

*Nitration of bromobenzene:* Total 2.36 g, 78% yield; *p*: *m*: *o* = 60: 0: 40; 4-bromonitrobenzene: a white solid, m.p. 126–128°C,  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 7.69 (2H, m,  $J^* = 9.0$  Hz, Ar), 8.11 (2H, m,  $J^* = 9.0$  Hz, Ar); 2-bromonitrobenzene: a white solid, m.p. 39–41°C,  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 7.44–7.48 (2H, m, Ar), 7.75 (1H, d,  $J = 9.6$  Hz, Ar), 7.85 (1H, d,  $J = 9.6$  Hz, Ar). MS (EI)  $m/z$  202 ( $\text{M}^+$ ) [HRMS calcd for  $\text{C}_6\text{H}_4\text{BrNO}_2$  (202.0055): requires M, 200.9425. Found:  $\text{M}^+$  200.9410].

*Nitration of benzene:* Nitrobenzene: a yellow oil, 1.62 g, 87% yield;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 7.31 (2H, t,  $J = 7.7$  Hz, Ar), 7.52–7.58 (1H, m, Ar), 8.24 (2H, d,  $J = 7.8$  Hz, Ar); MS (EI)  $m/z$  123 ( $\text{M}^+$ ) [HRMS calcd for  $\text{C}_6\text{H}_5\text{NO}_2$  (123.1094): requires M, 123.0320. Found:  $\text{M}^+$  123.0333].

*Nitration of ethylbenzene:* A yellow oil, 1.79 g, 79% yield; *p*: *m*: *o* = 56: 0: 44; 1-ethyl-2-nitrobenzene  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 1.26 (3H, t,  $J = 7.2$  Hz,  $\text{CH}_3$ ) 2.88 (2H, q,  $J = 7.2$  Hz,  $\text{CH}_2$ ) 7.10–7.60 (3H, m, Ar) 7.75 (1H, d,  $J = 8.0$  Hz, Ar). 1-ethyl-4-nitrobenzene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS)  $\delta$ : 1.27 (3H, t,  $J = 7.2$  Hz,  $\text{CH}_3$ ), 2.74 (2H, q,  $J = 7.2$  Hz,  $\text{CH}_2$ ), 7.30 (2H, m,  $J^* = 8.0$  Hz, Ar), 8.03 (2H, m,  $J^* = 8.0$  Hz, Ar). MS (EI)  $m/z$  151 ( $\text{M}^+$ ) [HRMS calcd for  $\text{C}_8\text{H}_9\text{NO}_2$  (151.1626): requires M, 151.0633. Found:  $\text{M}^+$  151.0623].

*Nitration of isopropylbenzene:* A yellow oil, 1.87 g, 76% yield; *p*: *m*: *o* = 73: 0: 27; 1-isopropyl-2-nitrobenzene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 1.28 (6H, d,  $J = 7.8$  Hz,  $\text{CH}_3$ ), 3.36–3.44 (1H, m, CH), 7.28–7.31 (1H, m, Ar), 7.45–7.53 (2H, m, Ar), 7.69 (1H, d,  $J = 8.1$  Hz, Ar). 1-isopropyl-4-nitrobenzene:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , TMS, 300 MHz)  $\delta$ : 1.31 (6H, d,  $J = 7.8$  Hz,  $\text{CH}_3$ ), 2.96–3.08 (1H, m, CH), 7.37 (2H, m,  $J^* = 8.2$  Hz, Ar), 8.15 (2H, m,  $J^* = 8.2$  Hz, Ar). MS (EI)



Scheme 2.

$m/z$  165 ( $M^+$ ) [HRMS calcd for  $C_9H_{11}NO_2$  (165.1892): requires  $M$ , 165.0790, Found:  $M^+$  165.0786].

**Nitration of xylene** [Mixture of dimethylbenzenes (1,2-, 1,3-, and 1,4-dimethylbenzene)]: A yellow oil, 1.64 g, 73% yield; 1,2-dimethyl-4-nitrobenzene:  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.38 (6H, s,  $CH_3$ ), 7.25 (1H, d,  $J = 8.0$  Hz, Ar), 7.94–8.0 (2H, m, Ar); 1,3-dimethyl-5-nitrobenzene:  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.42 (6H, s,  $CH_3$ ), 7.30 (1H, s, Ar), 7.83 (2H, s, Ar); 1,4-dimethyl-2-nitrobenzene:  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.40 (3H, s,  $CH_3$ ), 2.55 (3H, s,  $CH_3$ ), 7.20–7.33 (2H, m, Ar), 7.78 (1H, s, Ar); 1,2-dimethyl-6-nitrobenzene:  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.37 (6H, s,  $CH_3$ ), 7.08–7.45 (2H, m, Ar), 7.58 (1H, d,  $J = 7.8$  Hz, Ar); 1,3-dimethyl-6-nitrobenzene:  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.40 (3H, s,  $CH_3$ ), 2.60 (3H, s,  $CH_3$ ), 7.11–7.16 (2H, m, Ar), 7.92 (1H, d,  $J = 7.9$  Hz, Ar); 1,3-dimethyl-2-nitrobenzene:  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.30 (6H, s,  $CH_3$ ), 7.11–7.28 (3H, m, Ar). MS (EI)  $m/z$  151 ( $M^+$ ) [HRMS calcd for  $C_8H_9NO_2$  (151.1626): requires  $M$ , 151.0633, Found:  $M^+$  151.0630].

**Nitration of biphenyl** (see CAUTION above): 4-Nitrobiphenyl: a yellow solid, 1.66 g, 84% yield; m.p. 110–113°C;  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 7.44–7.53 (3H, m, Ar), 7.62–7.65 (2H, m, Ar), 7.74 (2H, m,  $J^* = 6.6$  Hz, Ar), 8.31 (2H, m,  $J^* = 6.6$  Hz, Ar); MS (EI)  $m/z$  199 ( $M^+$ ) [Calc. for  $C_{12}H_9NO_2$  (199.2054): requires  $M$ , 199.0633, Found:  $M^+$  199.0630].

**Nitration of naphthalene**: 1-Nitronaphthalene: a yellow solid, 1.70 g, 98% yield; m.p. 56–58°C;  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 7.53 (1H, t,  $J = 7.8$  Hz, Ar), 7.63 (1H, t,  $J = 7.8$  Hz, Ar), 7.73 (1H, t,  $J = 7.8$  Hz, Ar), 7.97 (1H, d,  $J = 7.8$  Hz, Ar), 8.12 (1H, t,  $J = 7.8$  Hz, Ar), 8.24 (1H, t,  $J = 7.8$  Hz, Ar), 8.57 (1H, d,  $J = 7.8$  Hz, Ar); MS (EI)  $m/z$  173 ( $M^+$ ). Found: C, 69.2; H, 4.2; N, 8.0%.  $C_{10}H_7NO_2$  requires C, 69.4; H, 4.1; N, 8.1%.

**Nitration of 2-methylnaphthalene**: A yellow solid, m.p. 38–42°C, 1.82 g, 97% yield, a mixture of isomers.  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.51–2.58 (3H,  $CH_3$ ), 7.35–8.51 (6H, Ar); 2-methyl-1-nitronaphthalene: a yellow solid, m.p. 54–56°C;  $^1H$  NMR ( $CDCl_3$ , TMS, 300 MHz)  $\delta$ : 2.51 (1H, s,  $CH_3$ ), 7.37 (1H, d,  $J = 8.7$  Hz, Ar), 7.54–7.64 (2H, m, Ar), 7.72 (1H, d,  $J = 8.7$  Hz, Ar), 7.88 (2H, d,  $J = 8.7$  Hz, Ar); MS (EI)  $m/z$  187 ( $M^+$ ) [Calc. for  $C_{11}H_9NO_2$  (187.1947): requires  $M$ , 187.0633, Found:  $M^+$  187.0629].

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