Supplementary Material (ESI) for Green Chemistry


# Sulfated Tungstate : A New Solid Heterogeneous Catalyst For Amide Synthesis

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Supplementary Material (ESI) for Green Chemistry


## A) Experimental

### a) Regents:

$Na_2WO_4.2H_2O$ was purchased from West Coast Laboratories, India. Other chemicals were purchased from Spectrochem Pvt. Ltd. India and were used without further purification.

### b) Catalyst preparation:

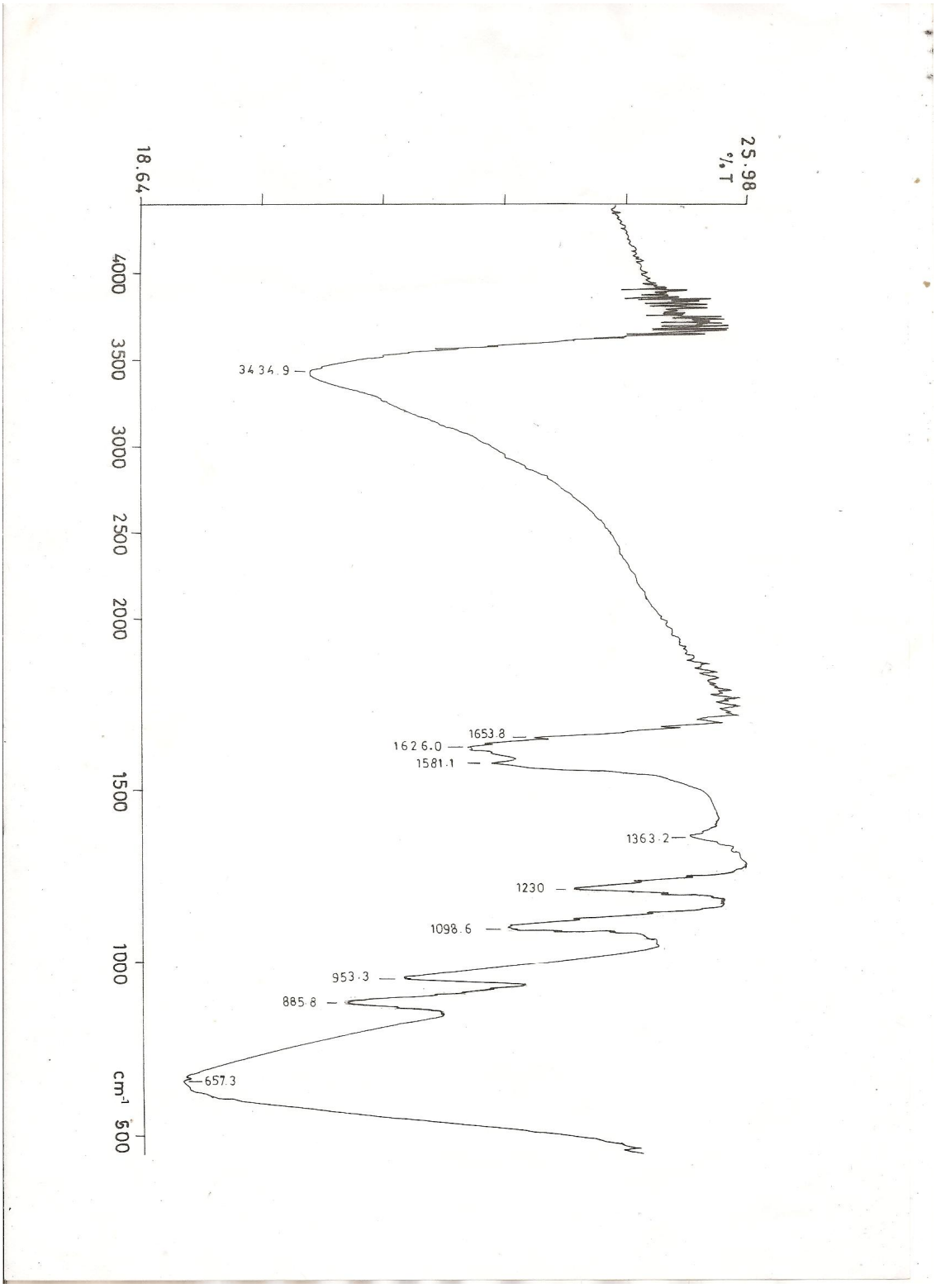
The catalyst was prepared by adding anhydrous sodium tungstate (0.1 mol) gradually to a stirred solution of chlorosulfonic acid (0.2 mol) in chloroform (150ml) contained in a 250ml round bottom flask placed in an ice bath. After completion of addition the mixture was stirred further for 1 h. A yellowish-white solid was obtained. It was filtered and repeatedly washed with deionized water until a neutral filtrate was obtained. The absence of chlorine ion was detected by $AgNO_3$ test. Then catalyst was dried in an oven for 2 h at 100 $^0$C.

### c) General procedure for amide synthesis:

To a solution of benzoic acid (2g, 16.39 mmol) in toluene (20ml) was added the catalyst (0.64g, 18 %( w/w)). To this mixture was added benzylamine (1.59g, 14.9 mmol) in one portion. The reaction mixture was refluxed for 12 hour and water was collected azeotropically in the Dean-Stark trap. After 12 hour the reaction mixture was allowed to cool at about 50-60 $^0$C and filtered through a Buchner funnel and catalyst was recovered by washing with acetone and water. The filtrate was distilled off under reduced pressure to remove toluene. The residue obtained was dissolved in ethyl acetate (30ml) and wash with 10% (w/v) $NaHCO_3$ (10ml) and 5% (w/v) HCl (10ml). The organic layer was dried over $Na_2SO_4$ and concentrated under reduced pressure to afford amide as a white solid with purity of 98% (HPLC)

**B) Characterization data of Catalyst:**

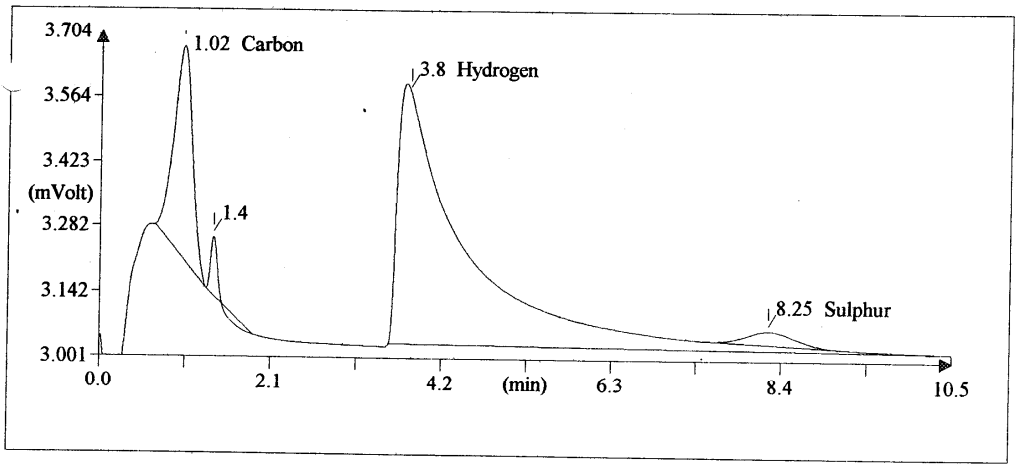
1. IR of Catalyst of the catalyst :




**3) Elemental Analysis the catalyst :**

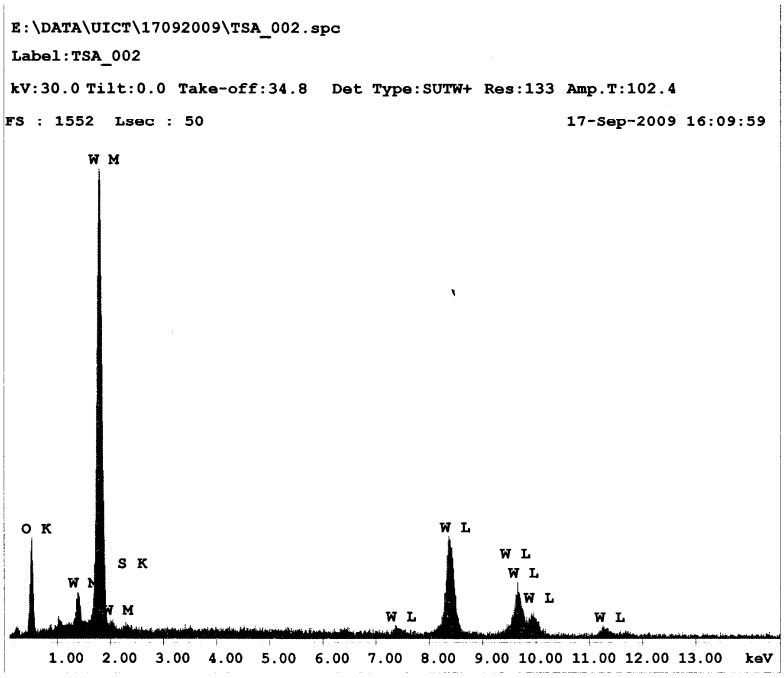
# Title: SAIF-IIT, Powai, Mumbai

| | |
|---|---|
| Operator ID: | SMD |
| Company name: | ThermoFinnigan |
| Method filename: | C:\Eager 300 for EA1112\data\Sys_data_example\chn-2009\14-09-2009-CHNS.mth |
| Method name: | NCHS |
| Analysed: | 09/14/2009 19:04 |
| Printed: | 09-16-2009 11:16 |
| Elemental Analyser method: | |
| Sampler method: | |
| Sample ID: | 14-09-2009-035-P-12-CHN-97 (# 35) |
| Analysis type: | UnkNown |
| Chromatogram filename: | 14-09-2009-035-P-12-CHN-97.dat |
| Calibration method: | K Factors |
| Sample weight: | 2.281 |
| Protein factor: | 6.25 |




| Peak Number (#) | Retention Time (min) | Area (.1*uV*sec) | Element % | Compon |
|---|---|---|---|---|
| 1 | 1.017 | 73336 | 0.000 | Carbon |
| 2 | 1.400 | 3723 | 0.000 | |
| 3 | 3.800 | 335642 | 0.825 | Hydrog |
| 4 | 8.250 | 11806 | 0.294 | Sulphu |
| | | 424507 | 1.119 | |

## 4) EDAX Report of the catalyst




**EDAX ZAF Quantification (Standardless)**
**Element Normalized**
**SEC Table : Default**

| Element | Wt % | At % | K-Ratio | Z | A | F |
|---|---|---|---|---|---|---|
| O K | 30.55 | 83.19 | 0.0708 | 1.1688 | 0.1983 | 1.0000 |
| S K | 0.31 | 0.43 | 0.0009 | 1.1173 | 0.2626 | 1.0000 |
| W L | 69.13 | 16.38 | 0.6245 | 0.8674 | 1.0415 | 1.0000 |
| Total | 100.00 | 100.00 | | | | |

| Element | Net Inte. | Bkgd Inte. | Inte. Error | P/B |
|---|---|---|---|---|
| O K | 34.60 | 3.50 | 2.64 | 9.89 |
| S K | 0.88 | 7.32 | 63.31 | 0.12 |
| W L | 78.00 | 6.08 | 1.72 | 12.83 |

## 5) Particle size analysis report

# Size Statistics Report by Intensity

*v2.0*

Malvern

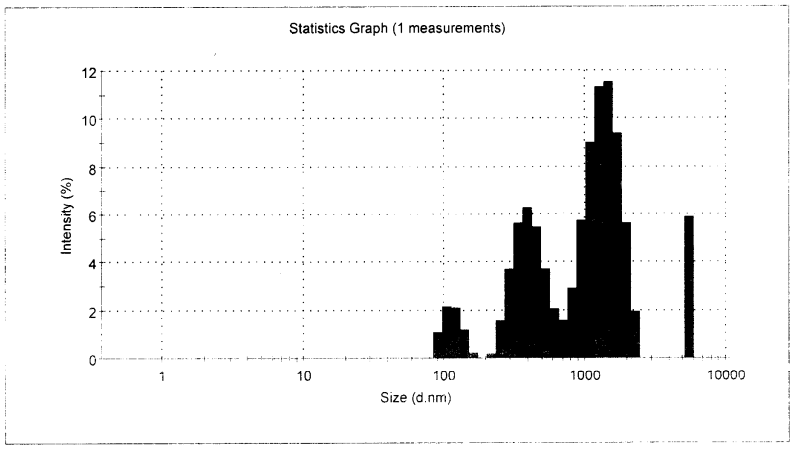


**Sample Details**

**Sample Name:** Sawant Sir 1
**File Name:** AATV_Nanosuspension fine tuned.dts
**SOP Name:** Atovaquone Nanosuspension.sop
**Measurement Date and Time:** Friday, September 18, 2009 3:39:24 PM

| | | | |
|---|---|---|---|
| **Z-Average (nm):** | 1428.832 | **Derived Count Rate (kcps):** | 2663791.63214... |
| **Standard Deviation:** | 0 | **Standard Deviation:** | 0 |
| **%Std Deviation:** | 0 | **%Std Deviation:** | 0 |
| **Variance:** | 0 | **Variance:** | 0 |

| Size d.nm | Mean Intensity % | Std Dev Intensity % | Size d.nm | Mean Intensity % | Std Dev Intensity % | Size d.nm | Mean Intensity % | Std Dev Intensity % | Size d.nm | Mean Intensity % | Std Dev Intensity % |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.4000 | 0.0 | | 5.615 | 0.0 | | 78.82 | 0.0 | | 1106 | 9.0 | |
| 0.4632 | 0.0 | | 6.503 | 0.0 | | 91.28 | 1.1 | | 1281 | 11.3 | |
| 0.5365 | 0.0 | | 7.531 | 0.0 | | 105.7 | 2.1 | | 1484 | 11.5 | |
| 0.6213 | 0.0 | | 8.721 | 0.0 | | 122.4 | 2.1 | | 1718 | 9.4 | |
| 0.7195 | 0.0 | | 10.10 | 0.0 | | 141.8 | 1.2 | | 1990 | 5.6 | |
| 0.8332 | 0.0 | | 11.70 | 0.0 | | 164.2 | 0.2 | | 2305 | 2.0 | |
| 0.9649 | 0.0 | | 13.54 | 0.0 | | 190.1 | 0.0 | | 2669 | 0.0 | |
| 1.117 | 0.0 | | 15.69 | 0.0 | | 220.2 | 0.2 | | 3091 | 0.0 | |
| 1.294 | 0.0 | | 18.17 | 0.0 | | 255.0 | 1.5 | | 3580 | 0.0 | |
| 1.499 | 0.0 | | 21.04 | 0.0 | | 295.3 | 3.7 | | 4145 | 0.0 | |
| 1.736 | 0.0 | | 24.36 | 0.0 | | 342.0 | 5.6 | | 4801 | 0.0 | |
| 2.010 | 0.0 | | 28.21 | 0.0 | | 396.1 | 6.3 | | 5560 | 5.9 | |
| 2.328 | 0.0 | | 32.67 | 0.0 | | 458.7 | 5.5 | | 6439 | 0.0 | |
| 2.696 | 0.0 | | 37.84 | 0.0 | | 531.2 | 3.7 | | 7456 | 0.0 | |
| 3.122 | 0.0 | | 43.82 | 0.0 | | 615.1 | 2.0 | | 8635 | 0.0 | |
| 3.615 | 0.0 | | 50.75 | 0.0 | | 712.4 | 1.6 | | 1.000e4 | 0.0 | |
| 4.187 | 0.0 | | 58.77 | 0.0 | | 825.0 | 2.9 | | | | |
| 4.849 | 0.0 | | 68.06 | 0.0 | | 955.4 | 5.7 | | | | |



# Size Distribution Report by Intensity

*v2.0*

Malvern



**Sample Details**

**Sample Name:** Sawant Sir 1
**SOP Name:** Atovaquone Nanosuspension.sop
**General Notes:**

| | | | |
|---|---|---|---|
| **File Name:** | AATV_Nanosuspension fi... | **Dispersant Name:** | Water |
| **Record Number:** | 2895 | **Dispersant RI:** | 1.330 |
| **Material RI:** | 1.53 | **Viscosity (cP):** | 0.8872 |
| **Material Absorbtion:** | 0.01 | **Measurement Date and Time:** | Friday, September 18, 2009 3... |

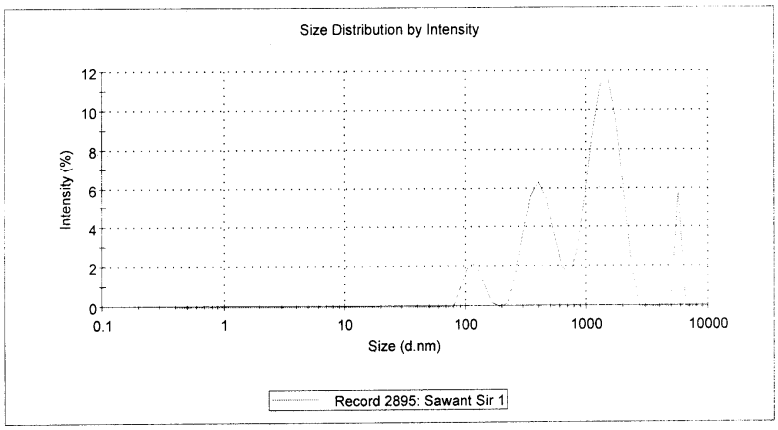
**System**

| | | | |
|---|---|---|---|
| **Temperature (°C):** | 25.0 | **Duration Used (s):** | 50 |
| **Count Rate (kcps):** | 326.4 | **Measurement Position (mm):** | 0.65 |
| **Cell Description:** | Disposable sizing cuvette | **Attenuator:** | 3 |

**Results**

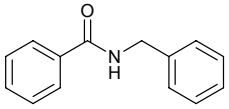
| | | | Diam. (nm) | % Intensity | Width (nm) |
|---|---|---|---|---|---|
| **Z-Average (d.nm):** | 1429 | **Peak 1:** | 1396 | 58.1 | 376.4 |
| **PdI:** | 0.877 | **Peak 2:** | 424.4 | 29.6 | 116.5 |
| **Intercept:** | 0.807 | **Peak 3:** | 116.8 | 6.6 | 18.24 |

**Result quality :** Refer to quality report



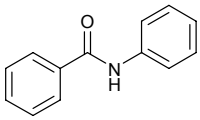
**C) Amide Analytical Data**

*Entry 1) N*-Benzylbenzamide



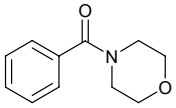
m.p. 129-130 $^0$C (lit.,[1] 128-130 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3445 (NH), 1655 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 4.65 (2 H, d, J=6), 6.42 (1 H, br s), 7.25-7.56 (8 H, m) and 7.79 (2 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 44.0, 127.1, 127.5, 127.6, 127.8, 128.5, 128.6, 134.3, 138.1 and 167.3.

*Entry 2) N*-Phenylbenzamide



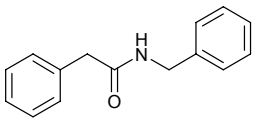
m.p. 165-166 $^0$C (lit.,[2] 162 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3343 (NH) and 1653 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 7.13-7.86 (10 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 165.79, 137.88, 134.95, 131.79, 129.05, 128.73, 127.00, 124.53 and 120.20.

*Entry 3)* Morpholino (phenyl)methanone



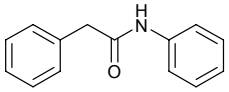
m.p. 72 $^0$C (lit.,[3] 72-74 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 1660 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 3.34 (4 H, t), 1.5 (6 H, m), 7.95 (2 H, m), 7.44 (2 H, m) and 7.51 (1 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 46.1, 66.8, 127.2, 128.6, 129.2, 135.5 and 168.9.

*Entry 4) N*-Benzyl-2-phenylacetamide



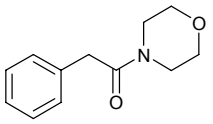
m.p. 117 $^0$C (lit.,[4] 117-119 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3271 (NH) and 1630 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 3.62 (2 H, s), 4.4 (2 H, d, *J* =6Hz), 5.9 (1 H, br s) and 7.15-7.55 (10 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 43.4, 43.6, 127.2, 127.3, 127.4, 128.5, 128.9, 129.3, 134.8, 138.1 and 170.9.

*Entry 5)* 2,*N*-Diphenylacetamide



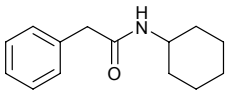
m.p. 118-119 $^0$C (lit.,[5] 118 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3254 (NH) and 1655 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 7.25-7.39 (10 H, m), 3.49 (2 H, s) and 6.2 (1 H, br s); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 169.12, 137.85, 134.41, 129.47, 129.16, 128.89, 127.61, 124.41, 119.81 and 44.76.

*Entry 6)* 4-(phenylacetyl)morpholine



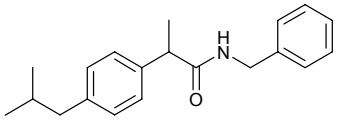
m.p. 62-64 $^0$C (lit.,[6] 64 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 1655 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 7.31 (2 H, t), 7.21-7.25 (3H, m), 3.72 (2 H, m), 3.63 (4 H, m) and 3.40-3.47 (4 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 45.6, 66.3, 38.6, 135.7, 129.7, 129.3, 127.7 and 160.5.

*Entry 7) N*-Cyclohexyl-2-phenylacetamide



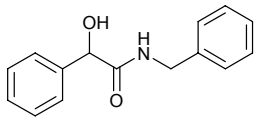
m.p. 124-128 $^0$C (lit.,[7] 125-126 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3345 (NH), 1645 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 0.92-1.14 (3 H, m), 1.23-1.36 (2 H. m), 1.48-1.63 (3 H, m), 1.78-1.84 (2 H, m), 3.52 (2 H, s), 3.65-3.76 (1 H, m), 5.22 (1 H, br s) and 7.2-7.38 (5 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 24.6, 25.4, 32.9, 44.0, 48.1, 127.2, 128.9, 129.3, 135.1 and 169.9.

*Entry 8) N*-Benzyl-2-(4-isobutylphenyl)propanamide



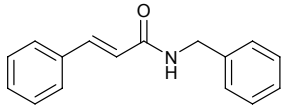
m.p. Yellow Oil (lit.,[8] Yellow Oil); $\nu_{max}$(Neat)/cm$^{-1}$ 3280 (NH) and 1646 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 0.89 (6 H, d, *J*=6Hz), 1.53 (3 H, d, *J*=6Hz), 1.84 (1 H, m), 2.5 (3 H, d, *J*=8Hz), 3.64 (1 H, quartet, *J*=8Hz), 4.34 (2 H, d, *J*=6Hz), 6.4 (1 H, br s) and 7.05-7.4 (9 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 18.27, 22.24, 30.01, 43.26, 44.86, 46.61, 127.28, 127.4, 127.43, 128.59, 129.64, 138.54, 140.69 and 174.77.

*Entry 9) N*-Benzyl-2-hydroxy-2-phenylethanamide



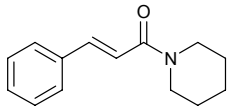

m.p. 96-99 $^0$C (lit.,[9] 96 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3250 (NH), 1641 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 3.6-3.65 (1 H, br s, OH exchangeable with $D_2O$), 4.43 (2 H, d, J=5.78), 5.07 (1 H, d, J=3.49), 6.52 (1 H, br s) and 7.16-7.43 (10 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 43.06, 43.14, 73.93, 74.0, 126.59, 127.44, 127.48, 128.33, 128.55, 128.59, 137.63, 139.52 and 172.54.

*Entry 10)* (*E*)-*N*-Benzylcinnamamide



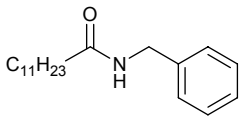

m.p. 106-108 $^0$C (lit.,[10] 108 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3263 (NH), 1652 (C=O) and 1615 (C=C); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 7.67 (1 H, d, *J*=15.3), 7.47 (2 H, m), 7.32 (8 H, m), 6.44 (1 H, d, *J*=15.3), 6.21 (1 H, s) and 4.54 (2 H, d, *J*=4.8); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 165.8, 141.3, 138.2, 134.7, 129.6, 128.7, 128.6, 127.9, 127.8, 127.5, 120.4 and 43.8.

*Entry 11)* (*E*)-3-phenyl-1-(piperidine-1-yl)prop-2-en-1-one




m.p. 110-112 $^0$C (lit.,[11] 114 $^0$C); $\nu_{max}$(KBr)/cm$^{-1}$ 1620 (C=C), 1645 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 1.30-1.78 (6 H, m), 3.32-3.65 (4 H, m), 6.74 (1 H, d, J=16), 7.01-7.4 (5 H, m) and 7.48 (1 H, d, J=16); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 44.8, 25.4, 25.6, 166.4, 144, 118.9, 135.2, 126,4, 128.7 and 128.

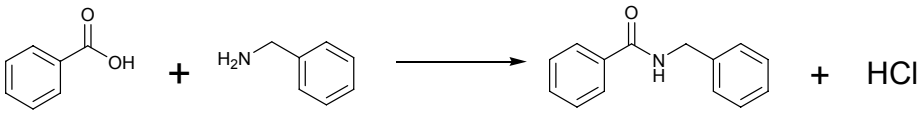
*Entry 12) N*-Benzyl laurylamide




m.p. 82-83 $^{0}$C (lit.,[12] 84 $^{0}$C); $\nu_{max}$(KBr)/cm$^{-1}$ 3250 (NH), 1641 (C=O); $\delta_H$(300 MHz; $CDCl_3$; $Me_4Si$) 0.88 (3 H, t), 1.51 (2 H, sextet), 1.21 (16 H, m), 1.41 (2 H, t), 3.85(2 H, d) and 7.15-7.33 (5 H, m); $\delta_C$(75 MHz; $CDCl_3$; $Me_4Si$) 14.13, 22.69, 25.78, 29.24, 29.62, 25.9, 35.8, 175.0, 57.4, 142.10, 128.2, 128.3 and 126.7.

**D) Green Metrics**

1) Calculations for the synthesis of *N*-Benzylbenzamide via thionyl chloride, producing an acid chloride.[13]




72%

| Input | | Output | |
|---|---|---|---|
| Benzoyl Chloride | 3.5g | Crude *N*-benzylbenzamide | 3.8g |
| Benzyl amine | 2.7g | | |
| Acetone | 26.4 (33ml) | Aqueous waste | 62g |
| $NaHCO_3$ (aq) | 12g | | |
| Brine (aq) | 50g | Organic Solvent Waste | 2.7g |
| Total | 94.6g | Total | 64.7 |

$$\text{E-Factor,} \quad \left( \frac{64.7\text{g of waste produced}}{3.8\text{g of crude product}} \right) = 17$$

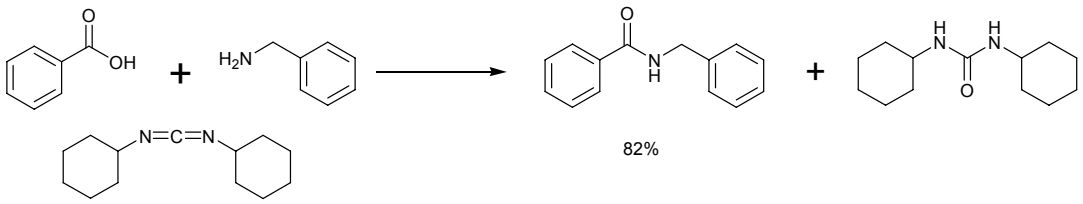
$$\text{Mass Intensity,} \quad \left( \frac{94.6\text{g of raw material used}}{3.8\text{g of crude product}} \right) = 25$$

$$\text{Atom Economy,} \quad \left( \frac{211}{140.5 + 107} \right) 100 = 85.2\%$$

Assumptions

1. 90% of organic solvents are recovered.
2. The formation of acyl chloride and use of thionyl chloride is not accounted for in calculations.

2) Calculations for the synthesis of *N*-Benzylbenzamide using DCC as activating agent.[14]




| Input | | Output | |
|---|---|---|---|
| Benzoic acid | 1.5g | Crude *N*-benzylbenzamide | 2g |
| Benzyl amine | 1.3g | | |
| DCC | 2.7g | Aqueous waste | 36g |
| DCM | 92.7g (70ml) | | |
| HCl (aq) | 18g | Organic Solvent Waste | 9.3g |
| $KHCO_3$ (aq) | 18g | | |

| Total | 134.2g | Total | 45.3g |
|---|---|---|---|

$$\text{E-Factor,} \quad \left( \frac{\text{45.3g of waste produced}}{\text{2g of crude product}} \right) = 22.6$$

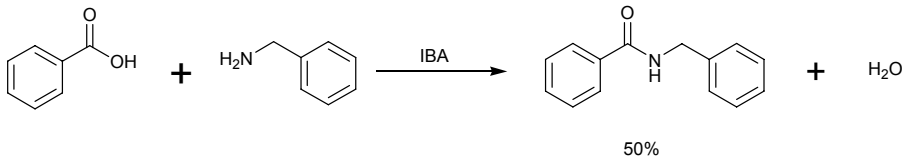
$$\text{Mass Intensity,} \quad \left( \frac{\text{134.2g of raw material used}}{\text{2g of crude product}} \right) = 67$$

$$\text{Atom Economy,} \quad \left( \frac{211}{206 + 107 + 122} \right) 100 = 48.5$$

Assumptions

1. 90% of organic solvents were recovered.
2. Calculations did not take into account of recrystallisation of the product.
3. Calculations did not account for the synthesis of DCC.

3) Calculation for the synthesis of *N*-Benzylbenzamide using ortho-*N,N*-Di-isopropylbenzylaminoboronic acid (IBA).[15]




| Input | | Output | |
|---|---|---|---|
| Benzoic acid | 0.6g | Crude *N*-benzylbenzamide | 0.53g |
| Benzyl amine | 0.54g | | |
| IBA | 0.12g | | |
| Flurobenzene | 51.2g (50ml) | | |

| | | | |
|---|---|---|---|
| DCM | 33.1g (25ml) | Aqueous waste | 125g |
| Brine (aq) | 25g | | |
| HCl (aq) | 25g | | |
| Brine (aq) | 25g | | |
| NaOH (aq) | 25g | Organic Solvent Waste | 8.4g |
| Brine (aq) | 25g | | |
| Total | 210.6g | Total | 133.4g |

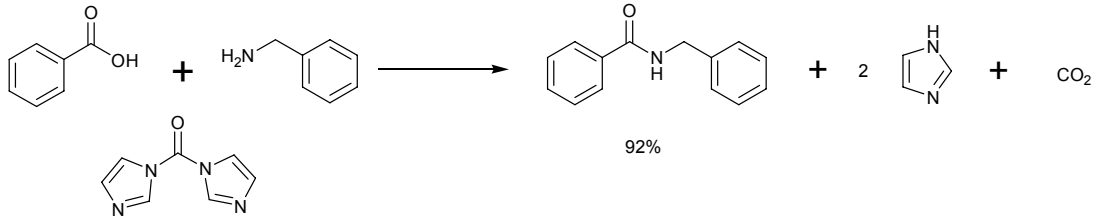
$$\text{E-Factor,} \quad \left( \frac{\text{133.4g of waste produced}}{\text{0.5g of crude product}} \right) = 251.7$$

$$\text{Mass Intensity,} \quad \left( \frac{\text{210.5g of raw material used}}{\text{0.53g of crude product}} \right) = 397.3$$

$$\text{Atom Economy,} \quad \left( \frac{211}{107 + 122} \right) 100 = 92.13\%$$

1. Calculations did not account for the synthesis of catalyst.
2. 90%recovery of organic solvents.

4) Calculations for the synthesis of *N*-Benzylbenzamide using *N,N'*-Carbonyldiimidazole as an activating agent.[16]




| Input | | Output | |
|---|---|---|---|
| Benzoic acid | 1.2g | Crude *N*- | 1.9g |

| | | | |
|---|---|---|---|
| Benzyl amine | 1g | benzylbenzamide | |
| THF | 17.7g (20ml) | Aqueous waste | 60g |
| CDI | 1.62g | | |
| NaOH (aq) | 10g | Organic Solvent Waste | 1.7g |
| HCl (aq) | 50g | | |
| Total | 63.9g | Total | 61.7g |

$$\text{E-Factor,} \quad \left( \frac{\text{61.7g of waste produced}}{\text{1.9g of crude product}} \right) = 31.8$$

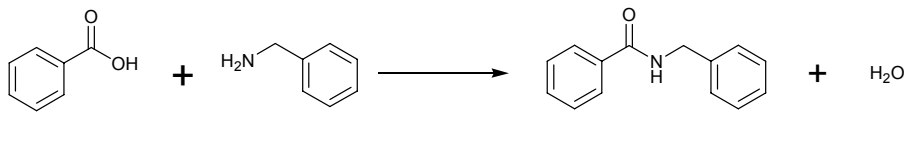
$$\text{Mass Intensity,} \quad \left( \frac{\text{63.9g of raw material used}}{\text{1.9g of crude product}} \right) = 32.9$$

$$\text{Atom Economy,} \quad \left( \frac{211}{122 + 107 + 162} \right) 100 = 53.9\%$$

Assumptions

1. 90% of organic solvents were recovered.
2. Calculations did not take into account of recrystallisation of the product.
3. Calculations did not account for the synthesis of CDI.

5) Calculations for the synthesis of *N*-Benzylbenzamide using Sulfated Tungstate (Catalyst).




81%

| Input | | Output | |
|---|---|---|---|
| Benzoic acid | 2g | Crude *N*- | 2.8g |

| | | | |
|---|---|---|---|
| Benzyl amine | 1.7g | benzylbenzamide | |
| Catalyst | 0.64g | Aqueous waste | 4.4g |
| Toluene | 17.2g (20ml) | | |
| EtOAc | 27g (30ml) | Organic Solvent Waste | 20 |
| $NaHCO_3$ (aq) | 10g | | |
| HCl (aq) | 10g | | |
| Total | 68.5g | Total | 24.4g |

$$\text{E-Factor,} \quad \left( \frac{\text{24.4g of waste produced}}{\text{2.8g of crude product}} \right) = 8.7$$

$$\text{Mass Intensity,} \quad \left( \frac{\text{68.5g of raw material used}}{\text{2.8g of crude product}} \right) = 24.4$$

$$\text{Atom Economy,} \quad \left( \frac{211}{122 + 107} \right) 100 = 92.13\%$$

Assumptions

1. 90% recovery of organic solvents.
2. Calculations did not account for the synthesis of the catalyst.

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