

Sulfated tungstate: an efficient catalyst for Ritter reaction

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SUPPORTING INFORMATION

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A) Experimental

a) Reagents:

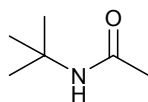
All chemicals were purchased from Spectrochem Pvt. Ltd. India and were used without further purification.

b) General procedure for Ritter reaction:

A mixture of 1-phenylethanol (1g, 8.19 mmol), benzonitrile (0.844g, 8.19 mmol), and sulfated tungstate (0.368 g, 20 wt%) was placed in a round bottom flask. The materials were mixed and heated at 100 °C for the given time. The progress of the reaction was monitored by TLC. After the completion of the reaction, diluted the reaction mixture with EtOAc (15ml) and filtered to recover the catalyst. The organic layer was concentrated under reduced pressure and crude residue obtained was recrystallised with hexane gave *N*-(1-Phenylethyl)benzamide as a white solid (entry 9).

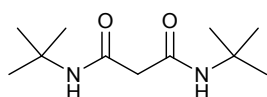
B) Amide Analytical Data

Entry 1) *N*-*tert*-butylacetamide



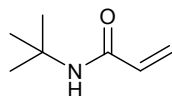
m.p. 95- 96 °C (lit.¹ 97-98 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3445 (NH), 1655 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.34 (9 H, s), 1.98 (3 H, s) and 7.57 (NH, br s); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 23.9, 28.5, 51.0 and 170.3.

Entry 2) *N*¹, *N*³-di-*tert*-butylmalonamide



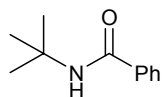
m.p. 114-115 °C (lit.² 112-113 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3420 (NH), 1660 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.30 (18 H, s), 3.07 (2 H, s) and 6.70 (1 H, br s); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 30.5, 44.0, 46.0 and 166.3.

Entry 3) *N*-*tert*-butylacrylamide



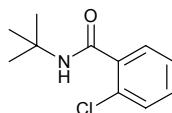
m.p. 125-127 °C (lit.,³ 126-128 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3460 (NH), 1630 (C=C), 1660 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.38 (9 H, s), 5.42 (1 H, dd), 5.57 (1 H, dd), 6.17 (1 H, dd) and 7.24 (1 H, br s); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 28.7, 51.2, 125.2, 131.8 and 165.3.

Entry 4) *N*-*tert*-butylbenzamide



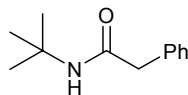
m.p. 129-130 °C (lit.,⁴ 128-129 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3460 (NH), 1660 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.49 (9 H, s), 6.19 (1 H, br s) and 7.44-7.88 (5 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 29.0, 49.5, 126.5, 128.4, 130.7, 135.0 and 166.3.

Entry 5) *N*-*tert*-butyl-2-chlorobenzamide



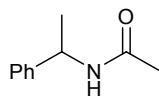
m.p. 106-107 °C (lit.,⁵ 106-108 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3420 (NH), 1640 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.38 (9 H, s), 6.0 (1 H, br s) and 7.25 (4 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 31.0, 47.5, 127.2, 128.4, 128.0, 129.4, 132.0, 133.2, 134.0 and 167.3.

Entry 6) *N*-*tert*-butyl-2-phenylacetamide



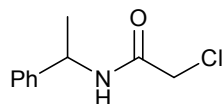
m.p. 113-115 °C (lit.,⁶ 115-116 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3420 (NH), 1648 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.66 (9 H, s), 3.60 (2 H, s), 5.40 (1 H, br s) and 7.33 (5 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 31.0, 41.5, 47.2, 127.2, 129.4, 130.0, 132.4 and 171.3.

Entry 7) *N*-(1-phenylethyl)acetamide



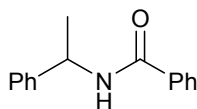
m.p. 75-76 °C (lit.,⁷ 75-77 °C); ν_{\max} (KBr)/cm⁻¹ 3425 (NH), 1650(C=O); δ_{H} (300 MHz; CDCl₃; Me₄Si) 1.42 (3 H, d), 1.76 (3 H, s), 5.34 (1 H, quartet), 6.31 (1 H, br s) and 7.15 (5 H, m); δ_{C} (75 MHz; CDCl₃; Me₄Si) 21.0, 23.0, 49.5, 126.0, 127.2, 128.4, 143.0, and 170.0.

Entry 8) 2-chloro-*N*-(1-phenylethyl)acetamide



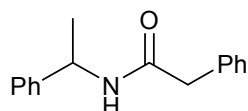
m.p. 69-71 °C (lit.,⁷ 70-72 °C); ν_{\max} (KBr)/cm⁻¹ 3445 (NH), 1655(C=O); δ_{H} (300 MHz; CDCl₃; Me₄Si) 1.64 (3 H, d), 4.06 (2 H, s), 5.34 (1 H, quartet), 6.26 (1 H, br s) and 7.26-7.34 (5 H, m); δ_{C} (75 MHz; CDCl₃; Me₄Si) 21.0, 55.0, 49.5, 126.0, 127.7, 129.4, 143.5, and 171.0.

Entry 9) *N*-(1-Phenylethyl)benzamide



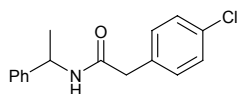
m.p. 103-105 °C (lit.,⁸ 103-104 °C); ν_{\max} (KBr)/cm⁻¹ 3360 (NH), 1640(C=O); δ_{H} (300 MHz; CDCl₃; Me₄Si) 1.58 (3 H, d), 5.20 (1 H, quartet), 6.5 (1 H, br s) and 7.34-7.90 (10 H, m); δ_{C} (75 MHz; CDCl₃; Me₄Si) 21.0, 50.0, 126.8, 127.0, 127.4, 128.6, 128.9, 132.2, 143.5 and 167.5.

Entry 10) 2-phenyl-*N*-(1-phenylethyl)acetamide



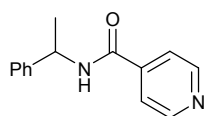
m.p. 100-101 °C (lit.,⁷ 98-100 °C); ν_{\max} (KBr)/cm⁻¹ 3370 (NH), 1650(C=O); δ_{H} (300 MHz; CDCl₃; Me₄Si) 1.59 (3 H, d), 3.52 (2 H, s), 5.23 (1 H, quartet), 7.6 (1 H, br s) and 7.26 (10 H, m); δ_{C} (75 MHz; CDCl₃; Me₄Si) 21.0, 50.0, 54.0, 125.8, 127.0, 127.6, 128.2, 128.9, 131.2, 142.5 and 168.5.

Entry 11) 2-(4-chlorophenyl)-*N*-(1-phenylethyl)acetamide



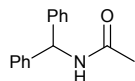
m.p. 108-109 °C (lit.,⁷ 110-111 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3372(NH), 1657(C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.60 (3 H, d), 3.64 (2 H, s), 5.52 (1 H, quartet), 7.40 (1 H, br s) and 7.32-7.37 (9 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 21.0, 41.0, 49.0, 126.8, 127.0, 128.0, 129.4, 131.1, 133.2, 133.8, 142.2 and 166.5.

Entry 12) *N*-(1-phenylethyl)isonicotinamide



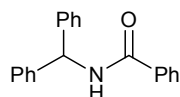
m.p. 119-120 °C (lit.,⁸ 118-120 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3380(NH), 1640(C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.77 (3 H, d), 5.09 (1 H, quartet), 6.21 (1 H, br s), 7.66-7.74 (5 H, m), 8.56-8.89 (4 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 21.0, 49.0, 122.8, 127.0, 126.8, 128.2, 140.0, 143.0, 149.0 and 167.5.

Entry 13) *N*-benzhydrylacetylacetamide



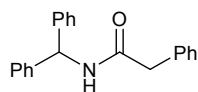
m.p. 144-146 °C (lit.,⁹ 146 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3350(NH), 1645(C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 2.00 (3 H, s), 6.17 (1 H, s), 5.73 (1 H, br s), 7.24 (10 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 23.0, 60.0, 126.8, 128.3, 129.3, 142.2 and 170.5.

Entry 14) *N*-benzhydrylbenzamide



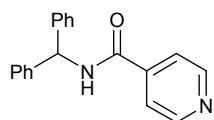
m.p. 171-172 °C (lit.,⁹ 170-172 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3340(NH), 1635(C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 6.36 (1 H, s), 7.68 (1 H, br s), 7.19-7.72 (15 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 60.0, 126.3, 127.5, 128.3, 129.3, 132.2, 134.2, 142.5 and 167.5.

Entry 15) *N*-benzhydryl-2-phenylacetamide



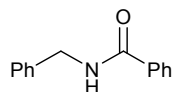
m.p. 138-140 °C (lit.,⁹ 137-140 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3346(NH), 1650(C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 3.65 (2 H, s), 5.49 (1 H, s), 6.25 (1 H, br s), 7.30-7.40 (15 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 42.0, 60.0, 125.3, 126.3, 128.7, 129.8, 131.2, 133.2, 141.5 and 170.5.

Entry 16) *N*-benzhydrylisonicotinamide



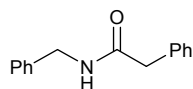
m.p. 213-215 °C (lit.,¹⁰ 213-214 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3385(NH), 1645(C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 5.39 (1 H, quartet), 8.77 (1 H, br s), 7.31-7.60 (14 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 61.0, 122.8, 127.0, 126.3, 127.2, 141.4, 142.6, 149.8 and 167.5.

Entry 17) *N*-Benzylbenzamide



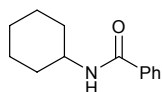
m.p. 129-130 °C (lit.,¹¹ 128-130 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3445 (NH), 1655 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 4.65 (2 H, d, $J=6$), 6.42 (1 H, br s), 7.25-7.56 (8 H, m) and 7.79 (2 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 44.0, 127.1, 127.5, 127.6, 127.8, 128.5, 128.6, 134.3, 138.1 and 167.3.

Entry 18) *N*-Benzyl-2-phenylacetamide



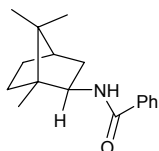
m.p. 117 °C (lit.,¹² 117-119 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3271 (NH) and 1630 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 3.62 (2 H, s), 4.4 (2 H, d, $J=6\text{Hz}$), 5.9 (1 H, br s) and 7.15-7.55 (10 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 43.4, 43.6, 127.2, 127.3, 127.4, 128.5, 128.9, 129.3, 134.8, 138.1 and 170.9.

Entry 19) *N*-cyclohexylbenzamide



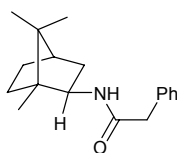
m.p. 137-139 °C (lit.,¹³ 139-141 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3345 (NH), 1645 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 1.18-1.28 (3 H, m), 1.32-1.43 (2 H, m), 1.62-1.78 (3 H, m), 1.96-2.04 (2 H, m), 3.93-4.06 (1 H, m), 5.22 (1 H, br s) and 7.2-7.38 (5 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 24.6, 25.4, 32.9, 48.0, 127.2, 128.9, 129.3, 135.1 and 169.9.

Entry 20) (\pm) *exo*-*N*-bornylbenzamide



m.p. 129-130 °C (lit.,¹⁴ 125-126 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3345 (NH), 1645 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 0.88 (3 H, s), 0.92 (3 H, s), 1.01 (3 H, s), 1.19-1.25 (1 H, m), 1.33-1.42 (1 H, m), 1.59-1.84 (4 H, m), 1.93-2.01 (1 H, m), 4.08-4.15 (1 H, m), 6.08 (1 H, br s) and 7.40-7.72 (5 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 11.8, 20.3, 20.3, 27.1, 35.6, 39.2, 44.7, 47.2, 48.8, 57.3, 126.5, 128.5, 131.0, 135.4 and 166.9.

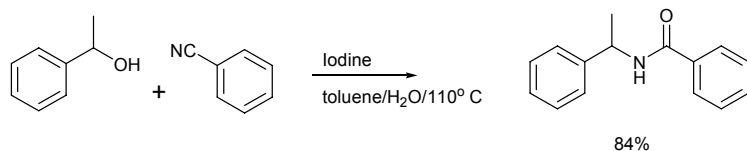
Entry 21) (\pm) *exo*-*N*-bornyl-2-phenylacetamide



m.p. 140-141 °C (lit.,¹⁵ 143 °C); $\nu_{\max}(\text{KBr})/\text{cm}^{-1}$ 3345 (NH), 1645 (C=O); $\delta_{\text{H}}(300 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 0.44 (3 H, s), 0.63 (3 H, s), 0.71 (3 H, s), 1.04-1.12 (1 H, m), 1.18-1.19 (1 H, m), 1.22-1.26 (1 H, m), 1.31-1.40 (1 H, m), 1.43-1.52 (1 H, m), 1.58-1.64 (2 H, m), 1.73-1.80 (2 H, m), 3.49-3.62 (2 H, m), 3.77-3.85 (1 H, m), 5.27 (1 H, br s) and 7.20-7.37 (5 H, m); $\delta_{\text{C}}(75 \text{ MHz}; \text{CDCl}_3; \text{Me}_4\text{Si})$ 14.4, 19.5, 23.5, 31.3, 33.8, 41.4, 46.0, 51.1, 53.4, 127.3, 129.5, 129.7, 135.7 and 170.4.

C) Green Metrics

1) Calculations for the synthesis of *N*-(1-phenylethyl) benzamide catalysed by Iodine¹⁶.



| Input | | Output | |
|--|--------------|---|---------|
| 1-phenylethanol | 1 g | Crude <i>N</i> -(1-phenylethyl) benzamide | 1.55g |
| Benzonitrile | 0.93g | | |
| Iodine | 0.42 g | Aqueous waste | 30.72 g |
| Water | 0.3 g | | |
| EtOAc | 18 g (20 ml) | | |
| Na ₂ S ₂ O ₃ (aq) | 15 g | Organic Solvent Waste | 1.8 g |
| Brine | 15 g | | |
| Total | 50.65g | Total | 32.52 g |

$$\text{E-Factor, } \left(\frac{32.52 \text{ g of waste produced}}{1.55 \text{ g of crude product}} \right) = 20.98$$

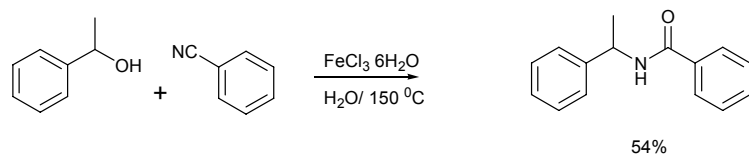
$$\text{Mass Intensity, } \left(\frac{50.65 \text{ g of raw material used}}{1.55 \text{ g of crude product}} \right) = 32.67$$

$$\text{Atom Economy, } \left(\frac{225}{122 + 103} \right) \times 100 = 100\%$$

Assumptions

- 90% of organic solvents are recovered.

2) Calculations for the synthesis of *N*-(1-phenylethyl) benzamide catalysed by FeCl₃ 6H₂O¹⁷.



| Input | | Output | |
|-------------------------------------|-----------------|---|--------|
| 1-phenylethanol | 1 g | Crude <i>N</i> -(1-phenylethyl) benzamide | 0.99 g |
| Benzonitrile | 0.84g | | |
| FeCl ₃ 6H ₂ O | 0.22 g | Aqueous waste | 0.52 g |
| Water | 0.3 g | | |
| EtOAc | 30.49 g (34 ml) | | |
| | | Organic Solvent Waste | 3.04 g |
| Total | 32.85 g | Total | 3.56 g |

$$\text{E-Factor, } \left(\frac{3.56 \text{ g of waste produced}}{0.99 \text{ g of crude product}} \right) = 3.59$$

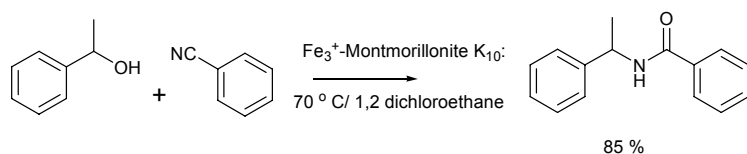
$$\text{Mass Intensity, } \left(\frac{32.85 \text{ g of raw material used}}{0.99 \text{ g of crude product}} \right) = 33.18$$

$$\text{Atom Economy, } \left(\frac{225}{122 + 103} \right) \times 100 = 100\%$$

Assumptions

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

3) Calculation for the synthesis of *N*-(1-phenylethyl) benzamide catalysed by Fe³⁺-Mont. K10¹⁸.



| Input | | Output | |
|-----------------------------|------------------|---|---------|
| 1-phenylethanol | 1 g | Crude <i>N</i> -(1-phenylethyl) benzamide | 1.56g |
| Benzonitrile | 2.11 g | | |
| Fe ³⁺ -Mont. K10 | 1.55 g | Aqueous waste | 00 g |
| 1,2-Dichloroethane | 125.3 g (100 ml) | Organic Solvent Waste | 12.53 g |
| Total | 129.96 g | Total | 12.53 g |

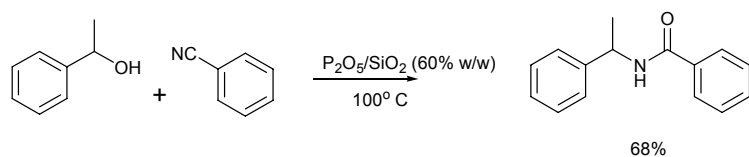
$$\text{E-Factor, } \left(\frac{12.53 \text{ g of waste produced}}{1.56 \text{ g of crude product}} \right) = 8.03$$

$$\text{Mass Intensity, } \left(\frac{129.96 \text{ g of raw material used}}{1.56 \text{ g of crude product}} \right) = 83.30$$

$$\text{Atom Economy, } \left(\frac{225}{122 + 103} \right) \times 100 = 100\%$$

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

4) Calculations for the synthesis of *N*-(1-phenylethyl) benzamide catalysed P₂O₅/SiO₂¹⁹.



| Input | | Output | |
|---|-----------------|---|---------|
| 1-phenylethanol | 1 g | Crude <i>N</i> -(1-phenylethyl) benzamide | 1.25g |
| Benzonitrile | 0.84g | | |
| P ₂ O ₅ /SiO ₂ | 1.1 g | Aqueous waste | 15 g |
| EtOAc | 57.40 g (64 ml) | | |
| NaHCO ₃ (aq) | 15 g | Organic Solvent Waste | 5.74 g |
| Total | 75.34 g | Total | 32.52 g |

$$\text{E-Factor, } \left(\frac{20.74 \text{ g of waste produced}}{1.25 \text{ g of crude product}} \right) = 16.59$$

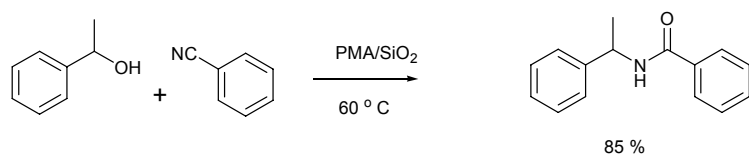
$$\text{Mass Intensity, } \left(\frac{75.34 \text{ g of raw material used}}{1.25 \text{ g of crude product}} \right) = 60.27$$

$$\text{Atom Economy, } \left(\frac{225}{122 + 103} \right) \times 100 = 100\%$$

Assumptions

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

5) Calculations for the synthesis of *N*-(1-phenylethyl) benzamide catalysed by PMA/SiO₂²⁰.



| Input | | Output | |
|----------------------|----------------|---|--------|
| 1-phenylethanol | 0.35 g | Crude <i>N</i> -(1-phenylethyl) benzamide | 0.51 g |
| Benzonitrile | 0.26 g | | |
| PMA/SiO ₂ | 0.25 g | Aqueous waste | 20 g |
| EtOAc | 67.27 g (75ml) | Organic Solvent Waste | 6.72 g |
| Brine | 20g | | |
| Total | 88.13 g | Total | 26.72g |

$$\text{E-Factor, } \left(\frac{26.72 \text{ g of waste produced}}{0.51 \text{ g of crude product}} \right) = 52.39$$

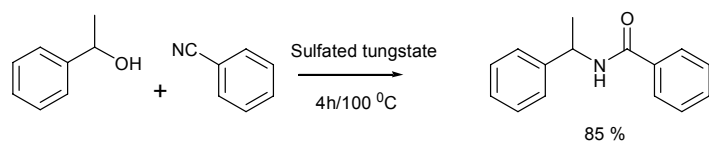
$$\text{Mass Intensity, } \left(\frac{88.13 \text{ g of raw material used}}{0.51 \text{ g of crude product}} \right) = 172.80$$

$$\text{Atom Economy, } \left(\frac{225}{122 + 103} \right) \times 100 = 100\%$$

Assumptions

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

6. Calculations for the synthesis of *N*-(1-phenylethyl) benzamide catalysed by Sulfated tungstate.



| Input | | Output | |
|--------------------|-----------------|---|--------|
| 1-phenylethanol | 1 g | Crude <i>N</i> -(1-phenylethyl) benzamide | 1.56 g |
| Benzonitrile | 0.84g | | |
| Sulfated tungstate | 0.36 g | Aqueous waste | 00 g |
| EtOAc | 13.45 g (15 ml) | | |
| | | Organic Solvent Waste | 1.34 g |
| Total | 15.70 g | Total | 1.34 g |

$$\text{E-Factor, } \left(\frac{1.34 \text{ g of waste produced}}{1.56 \text{ g of crude product}} \right) = 0.85$$

$$\text{Mass Intensity, } \left(\frac{15.70 \text{ g of raw material used}}{1.56 \text{ g of crude product}} \right) = 10.06$$

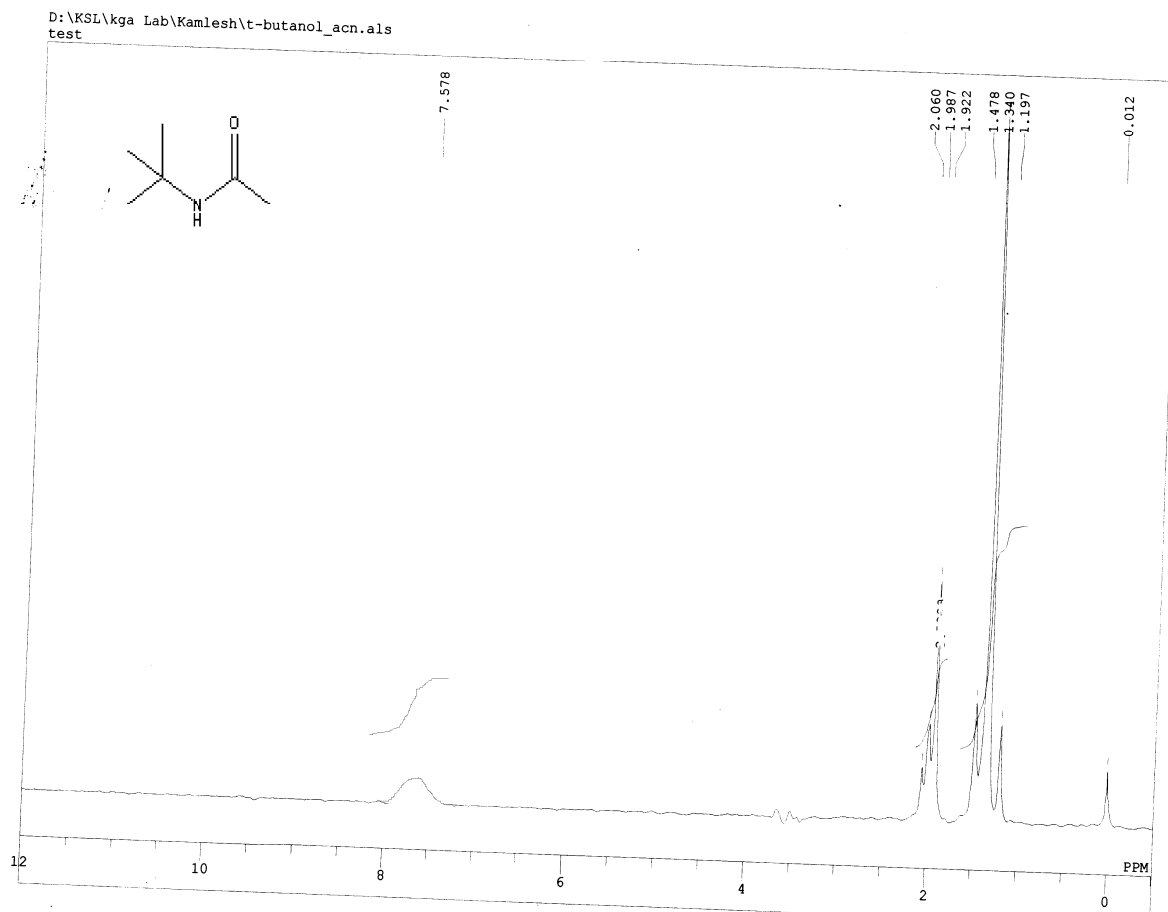
$$\text{Atom Economy, } \left(\frac{225}{122 + 103} \right) \times 100 = 100\%$$

Assumptions

- 1 90% recovery of organic solvents.
- 2 Calculations did not account for the synthesis of the catalyst.
- 3 Calculations did not take into account of recrystallisation of the product.

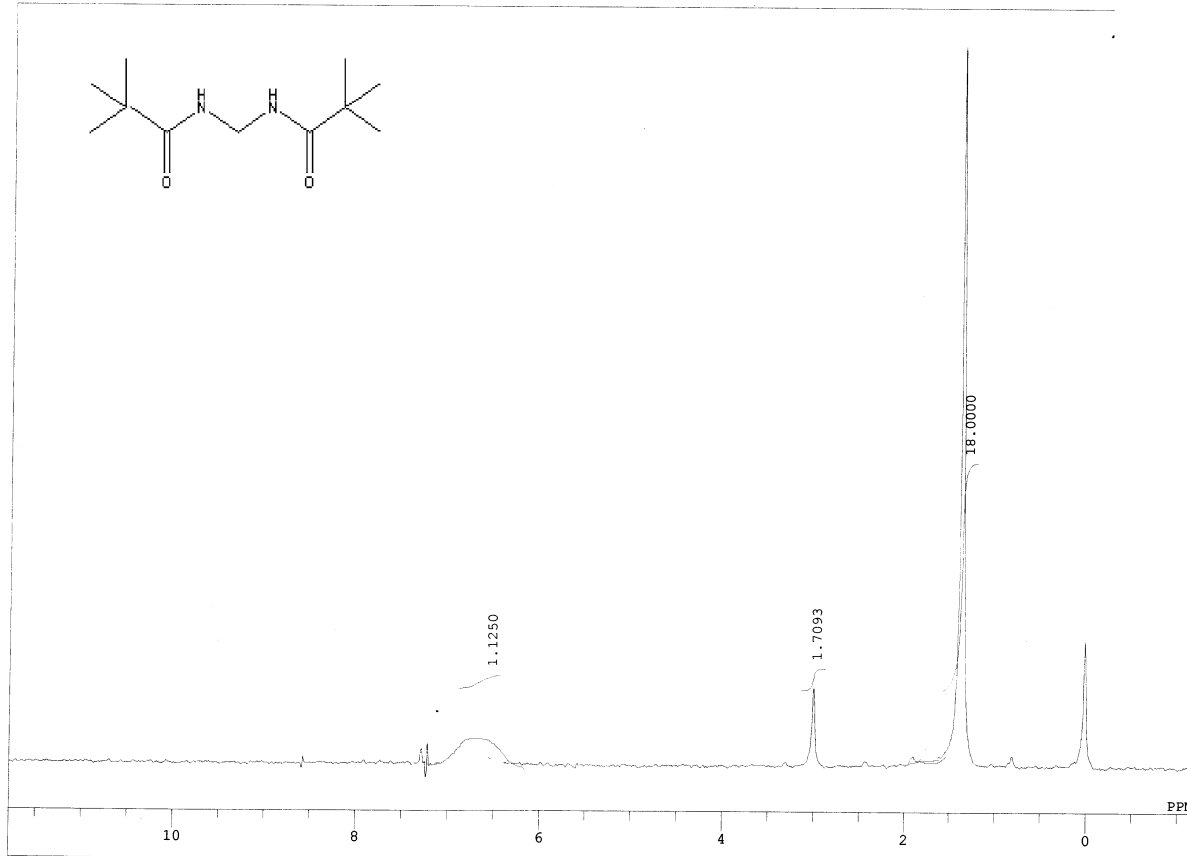
D) NMR Spectra:

Entry 1) *N*-*tert*-butylacetamide



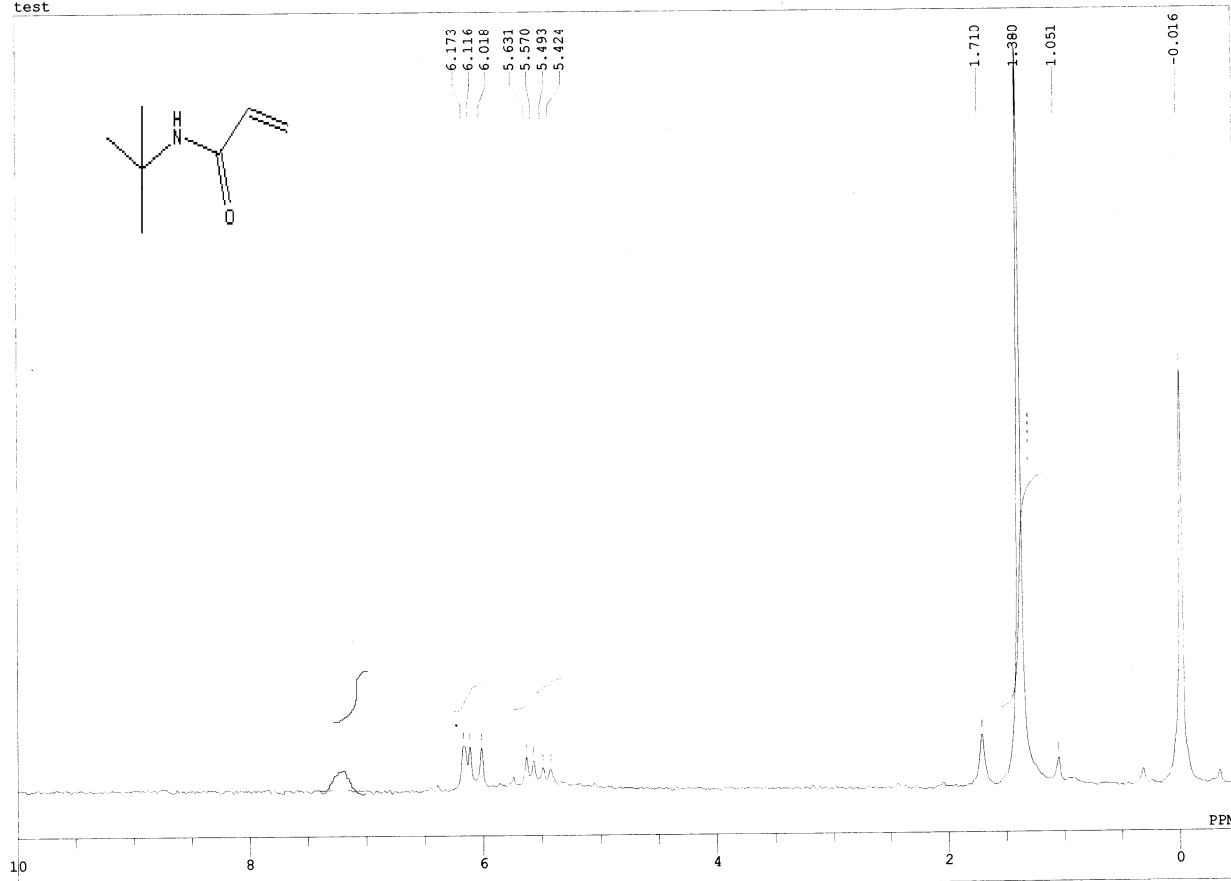
Entry 2) N^1, N^3 -di-*tert*-butylmalonamide

D:\kga Lab\Kamlesh\ter butanol_malanonitrile.als
test

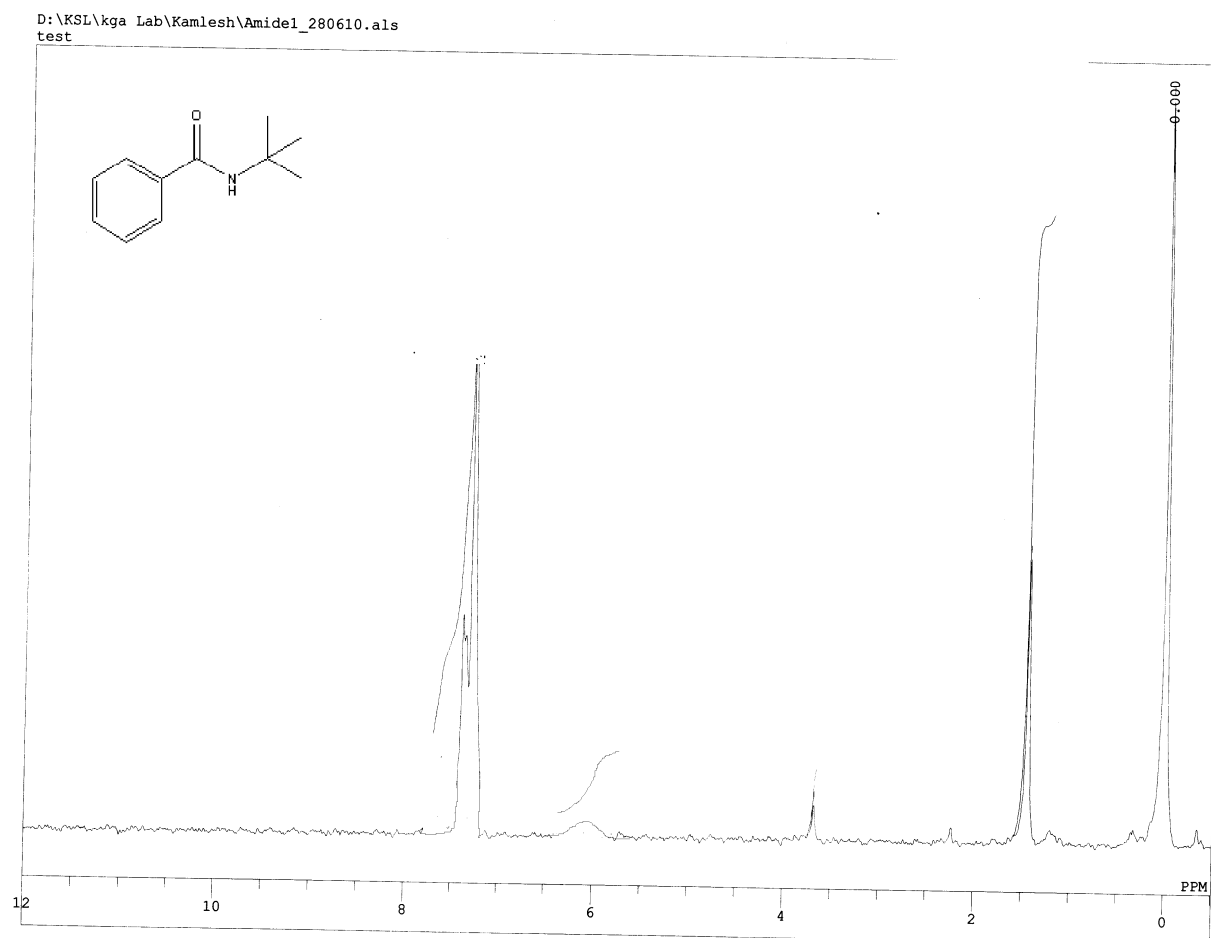


Entry 3) *N-tert*-butylacrylamide

D:\kga Lab\Kamlesh\ter butanol and acrylonitrile.als
test

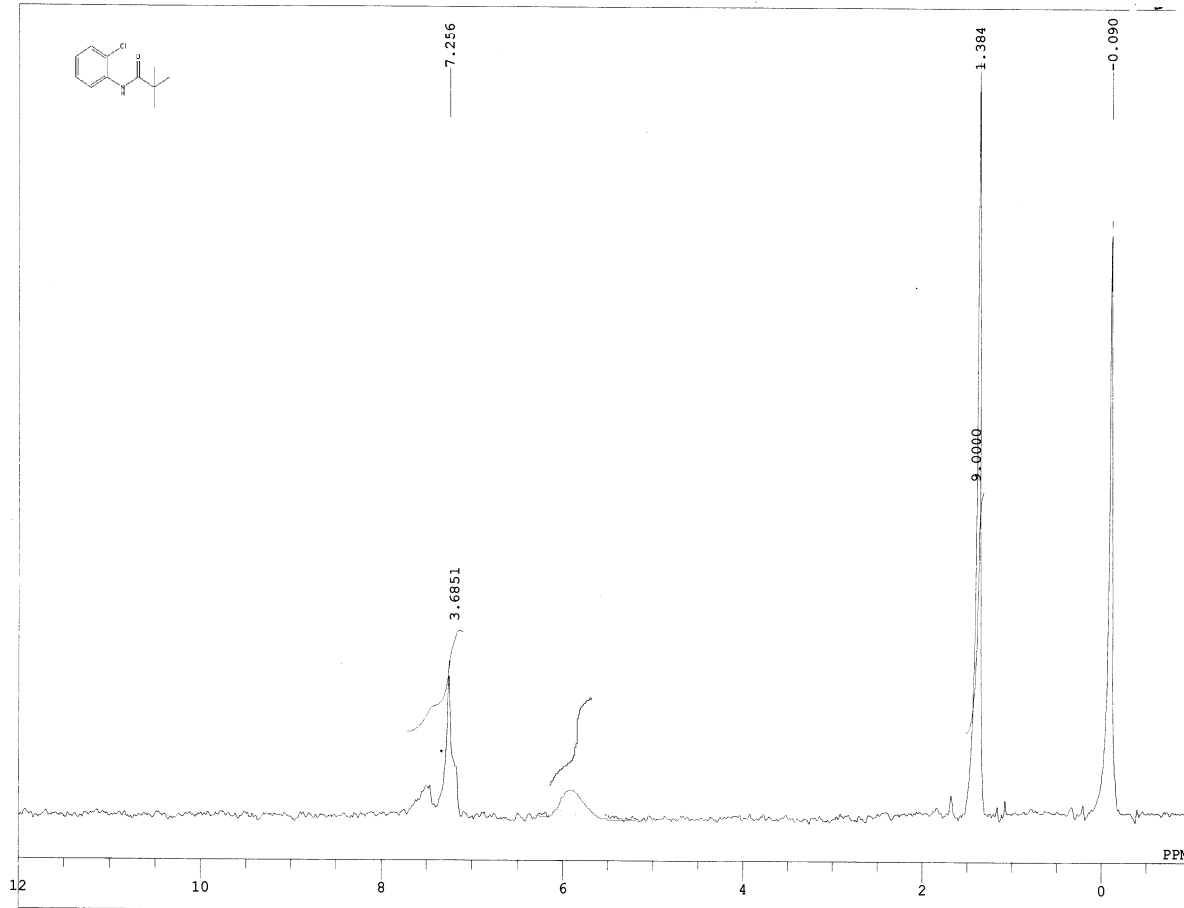


Entry 4) *N-tert*-butylbenzamide



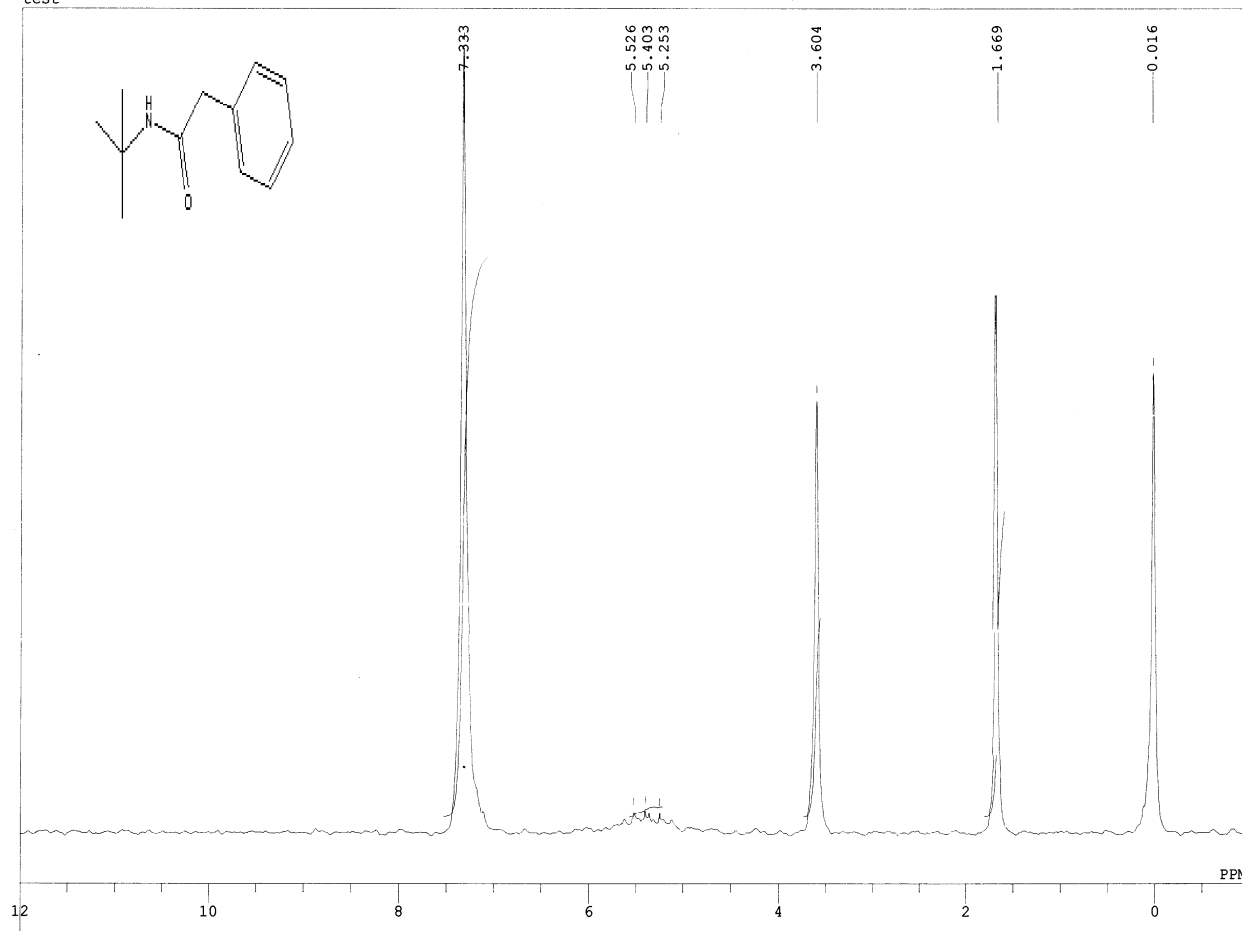
Entry 5) *N-tert-butyl-2-chlorobenzamide*

D:\kga Lab\Kamlesh\2 chlorobezonitrile_terbytanol.als
test

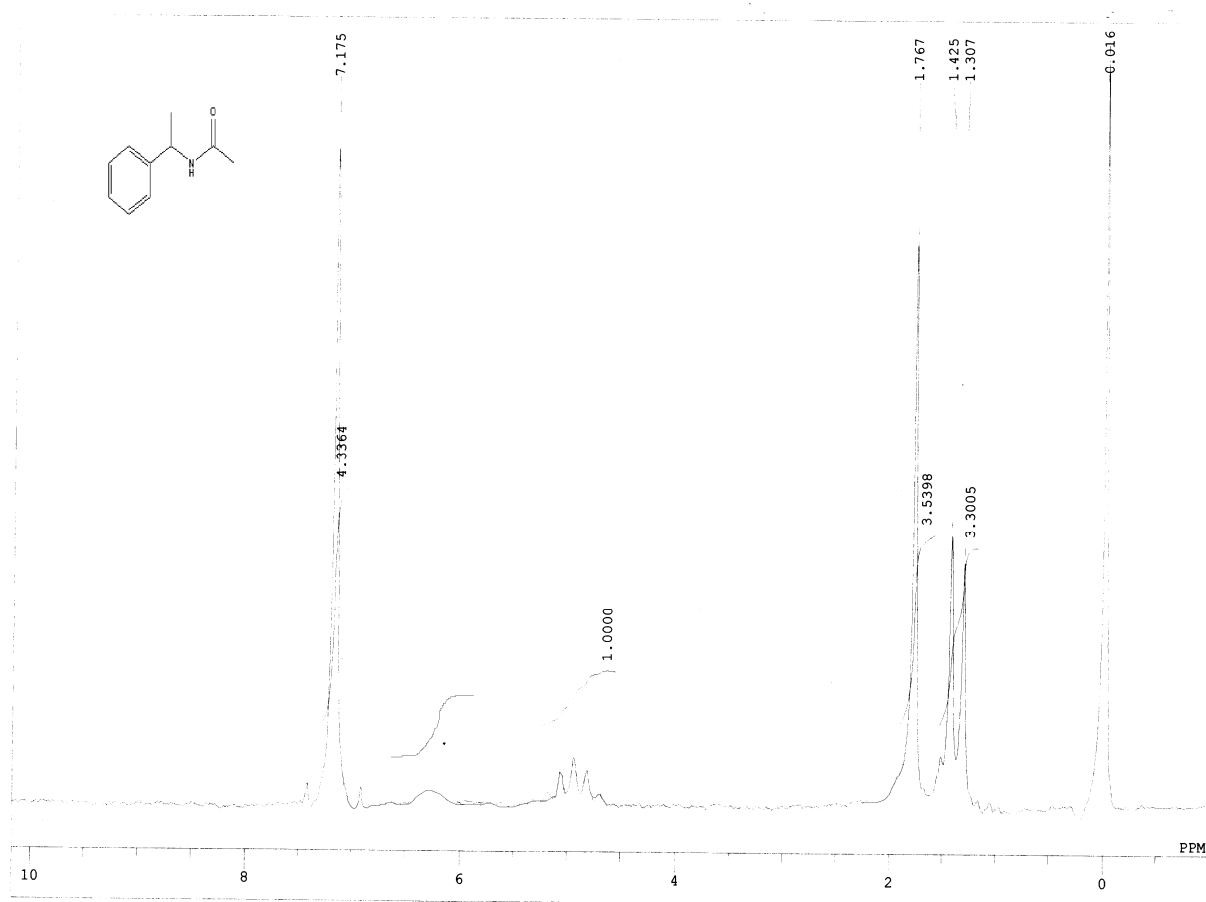


Entry 6) *N-tert-butyl-2-phenylacetamide*

D:\kga Lab\Kamlesh\terbutabol_benzyl cyanide.als
test

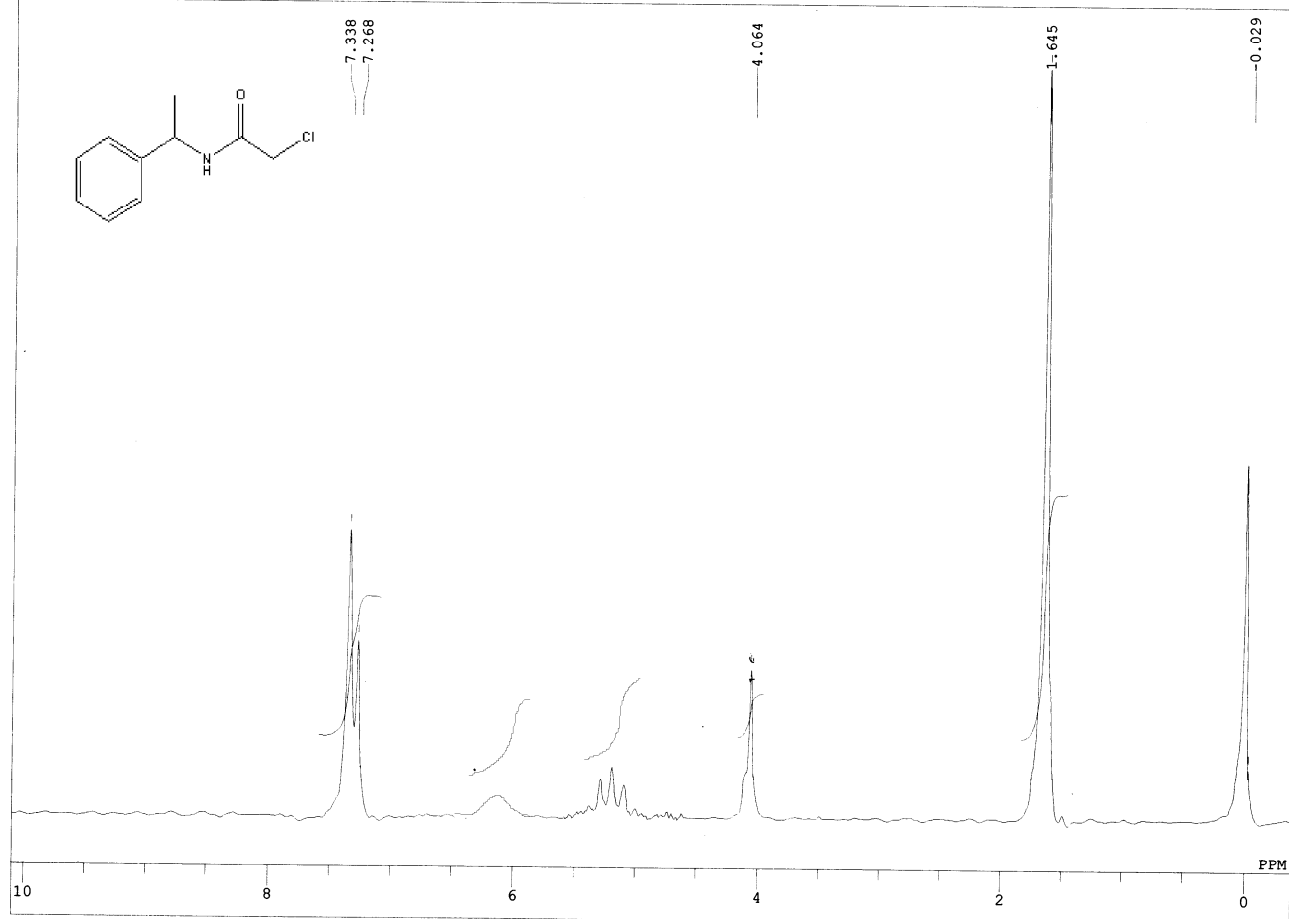


Entry 7) *N*-(1-phenylethyl)acetamide

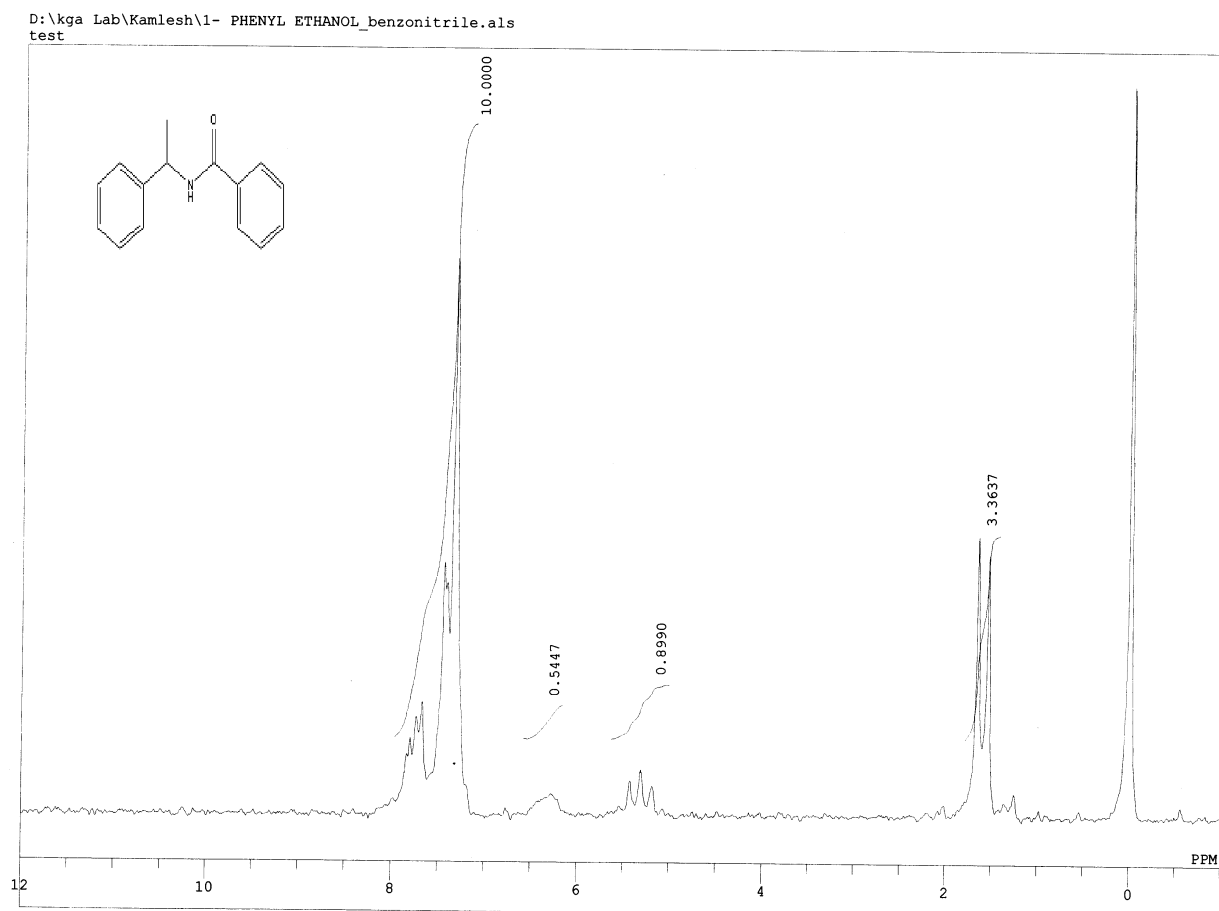


Entry 8) 2-chloro-N-(1-phenylethyl)acetamide

D:\KSL\kga Lab\Kamlesh\1-phenyl ethanol_chloro ACN.als
test

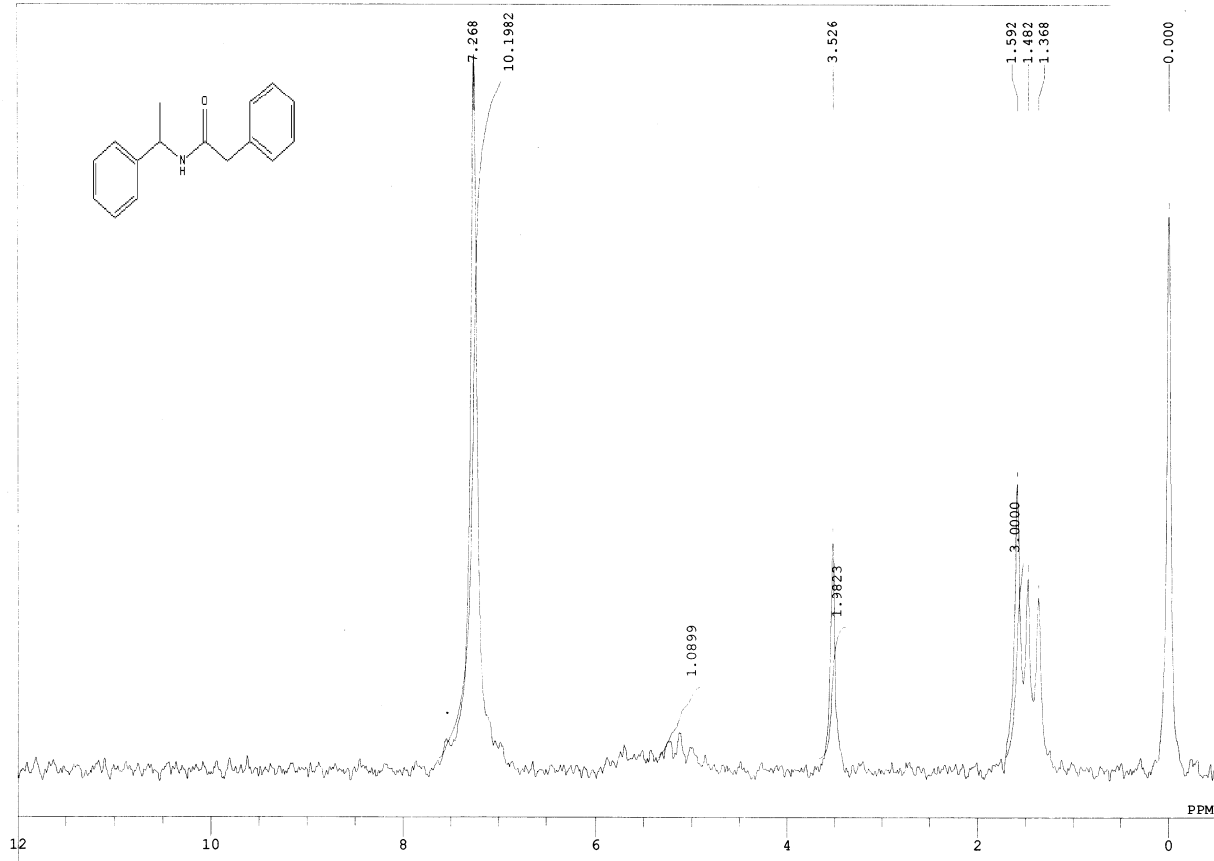


Entry 9) *N*-(1-Phenylethyl)benzamide

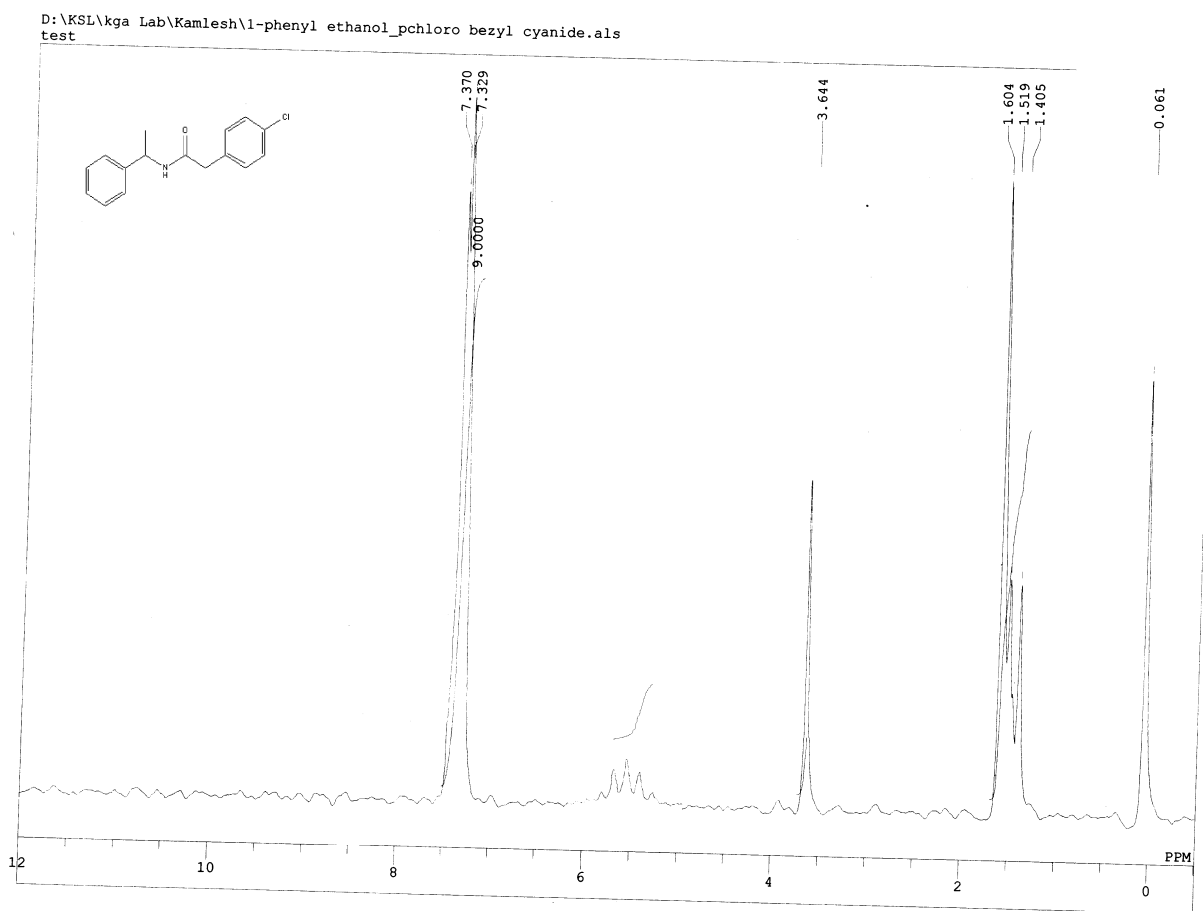


Entry 10) 2-phenyl-N-(1-phenylethyl)acetamide

D:\kga Lab\Kamlesh\1-phenyl ethanol_benzyl cyanide.als
test

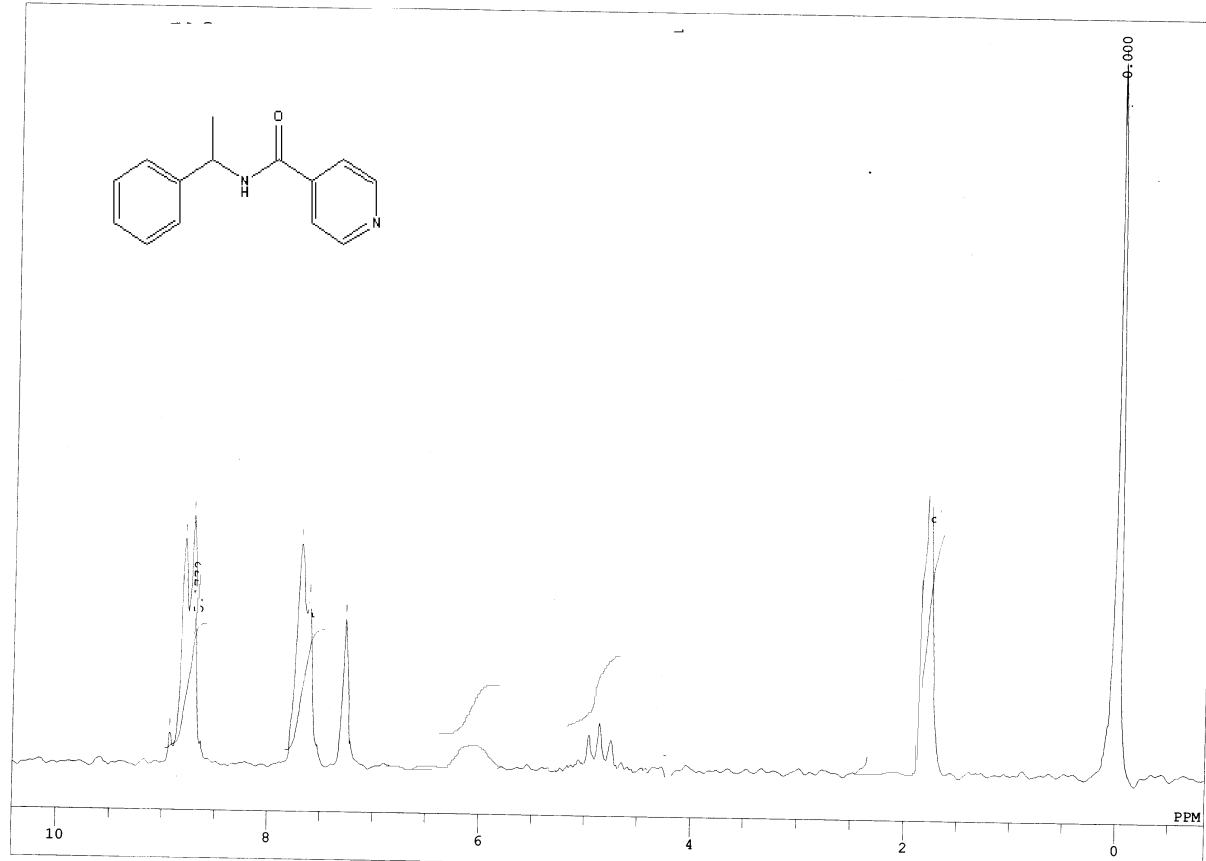


Entry 11) 2-(4-chlorophenyl)-*N*-(1-phenylethyl)acetamide



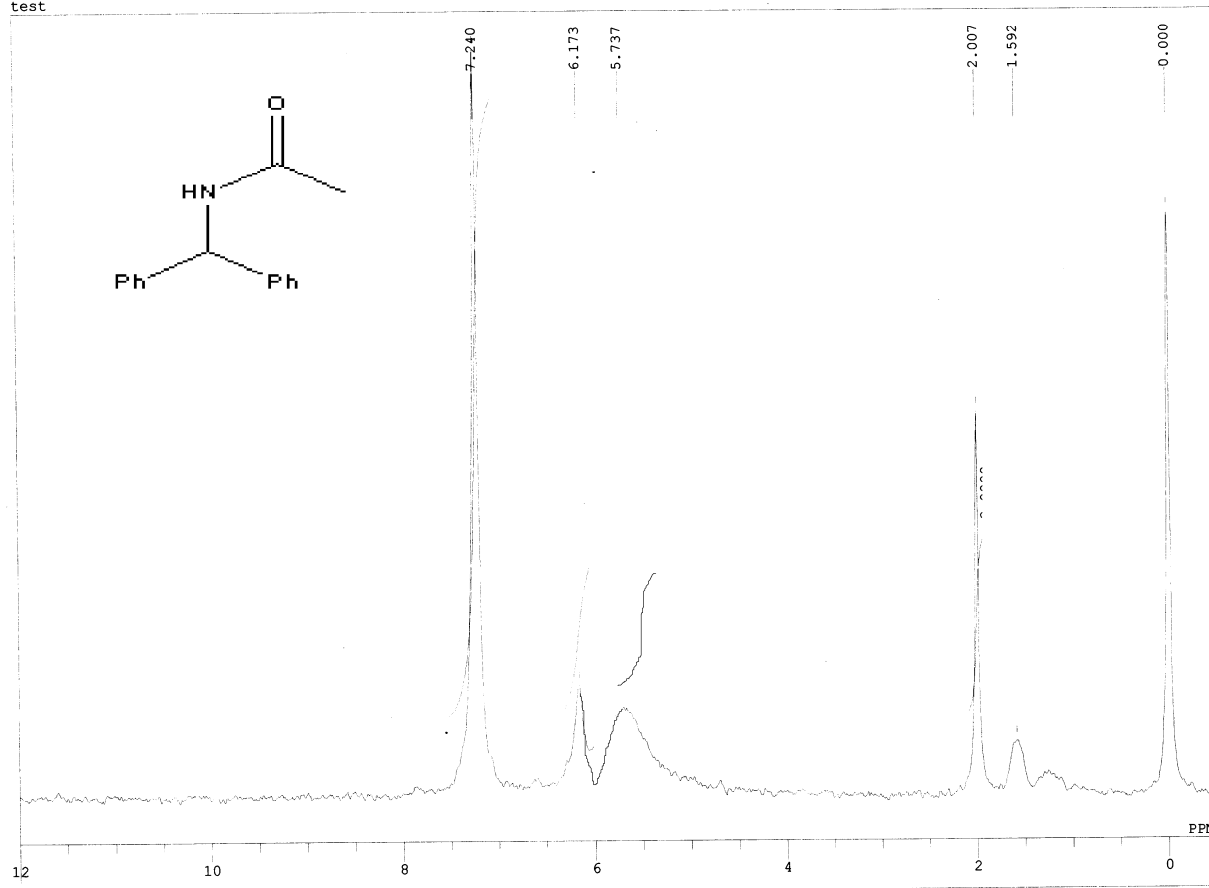
Entry 12) *N*-(1-phenylethyl)isonicotinamide

D:\KSL\kga Lab\Kamlesh\1-phenyl ethanol_pcyano pyridine.als
test

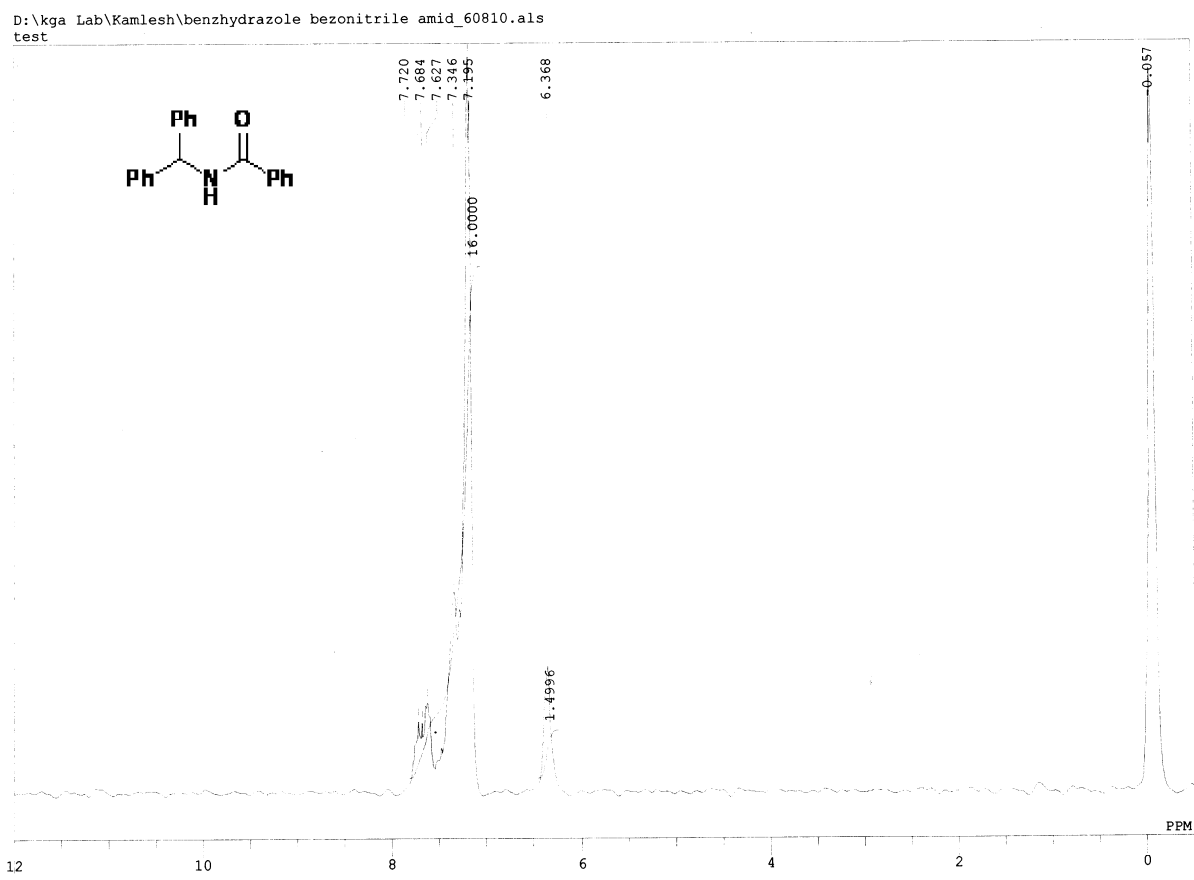


Entry 13) *N*-benzhydrylacetamide

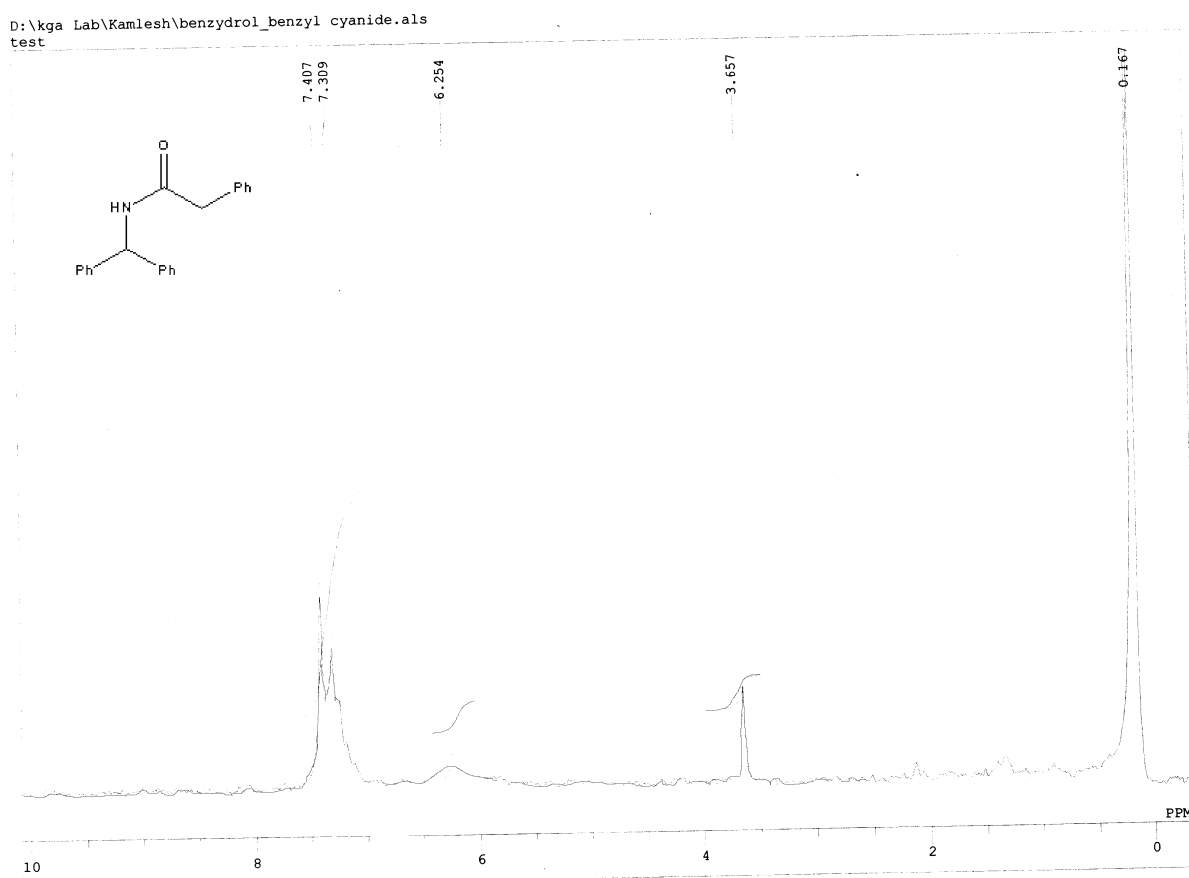
D:\kga Lab\Kamlesh\benzhydrol_acetonitrile.als
test



Entry 14) *N*-benzhydrylbenzamide

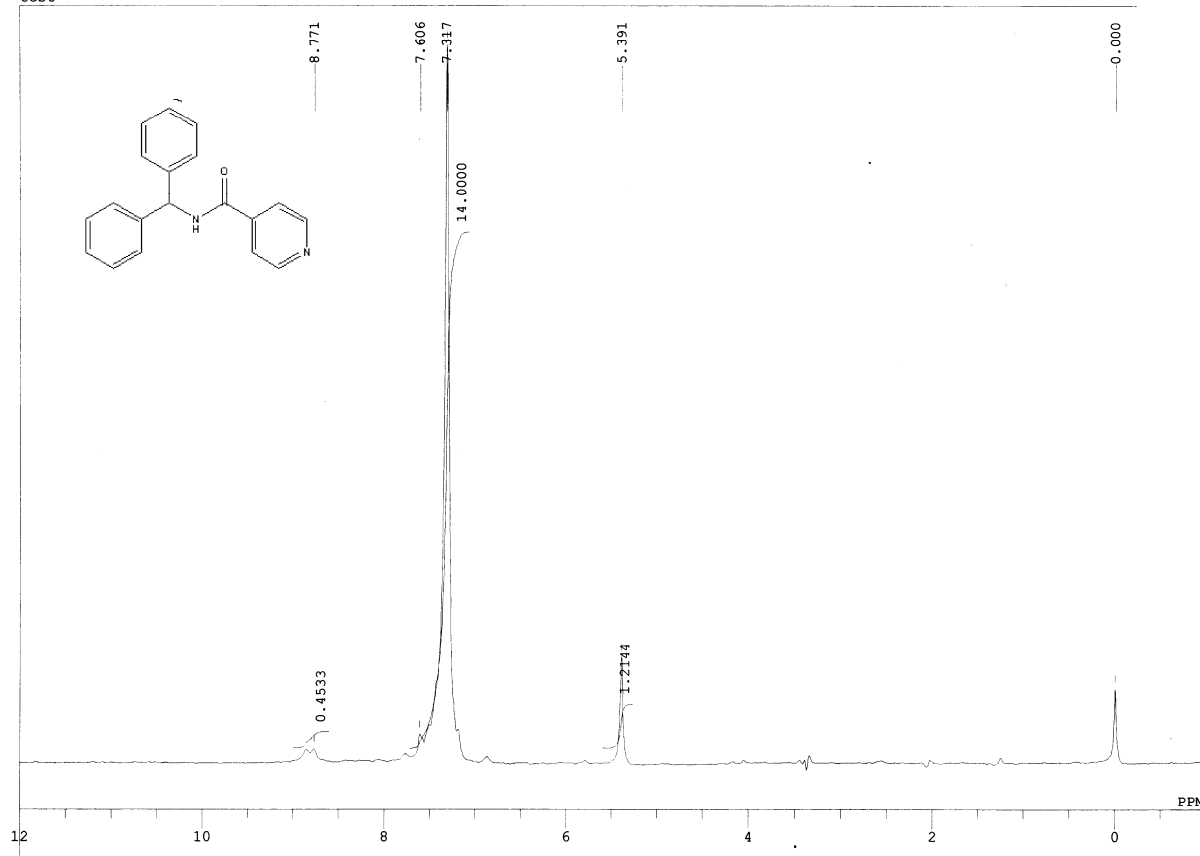


Entry 15) *N*-benzhydryl-2-phenylacetamide

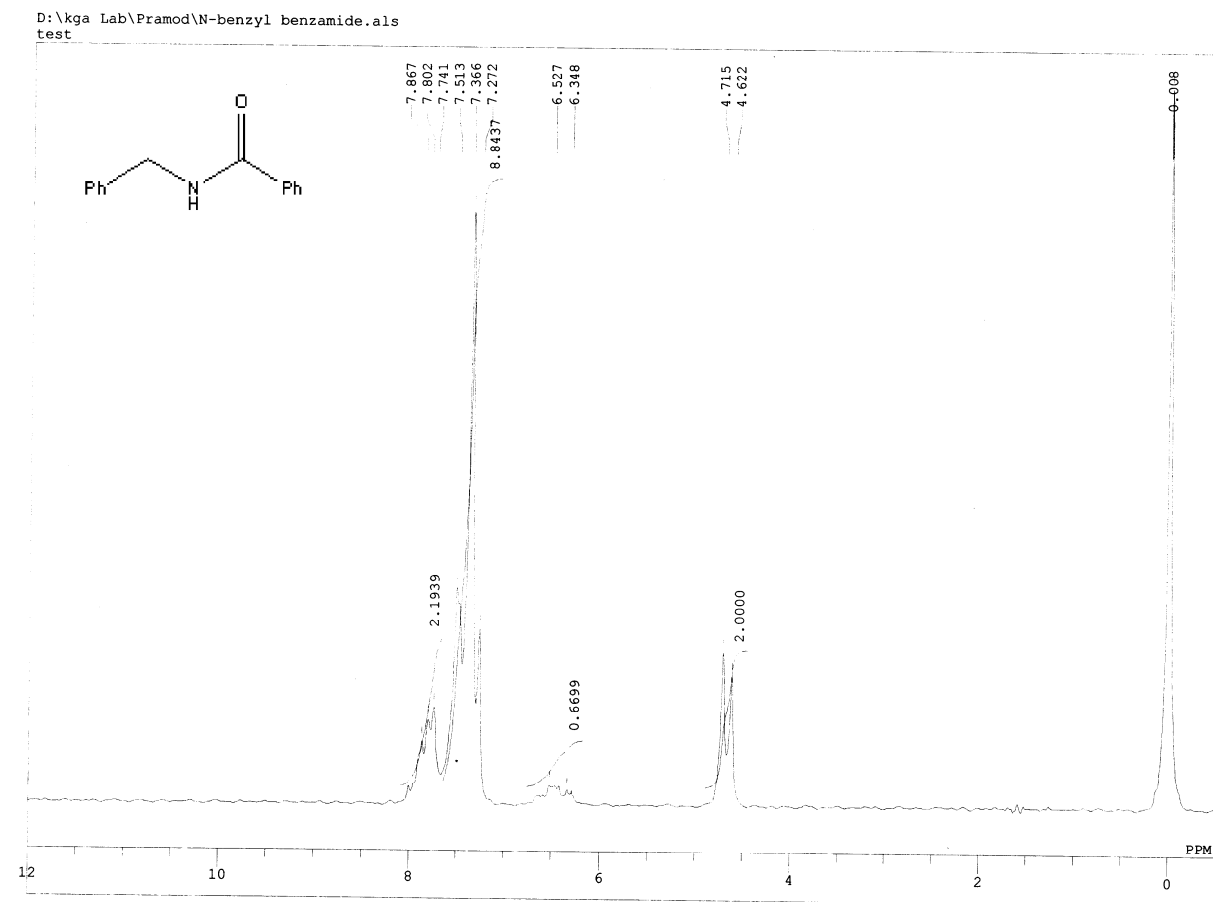


Entry 16) *N*-benzhydrylisonicotinamide

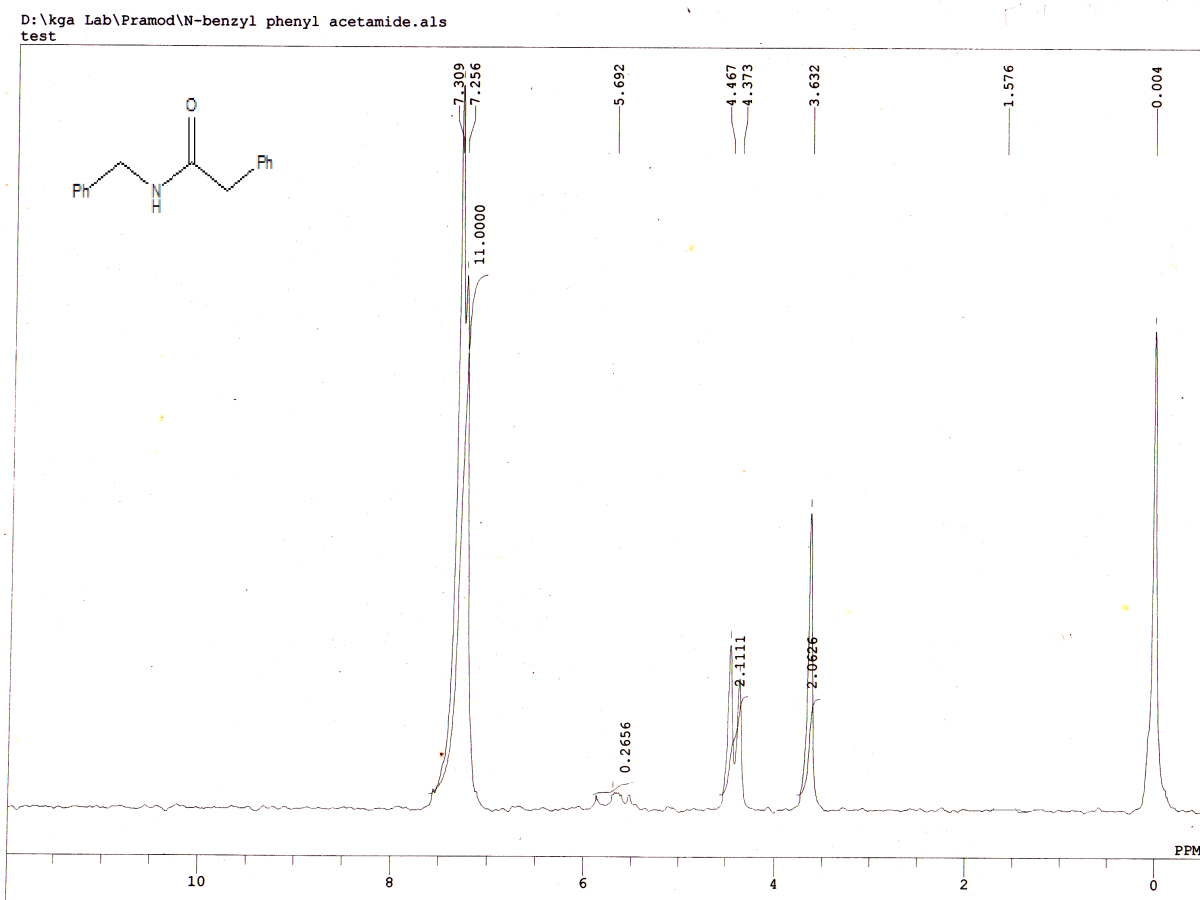
D:\KSL\kga Lab\Kamlesh\benzhydrol_pcyano pyrdine.als
test



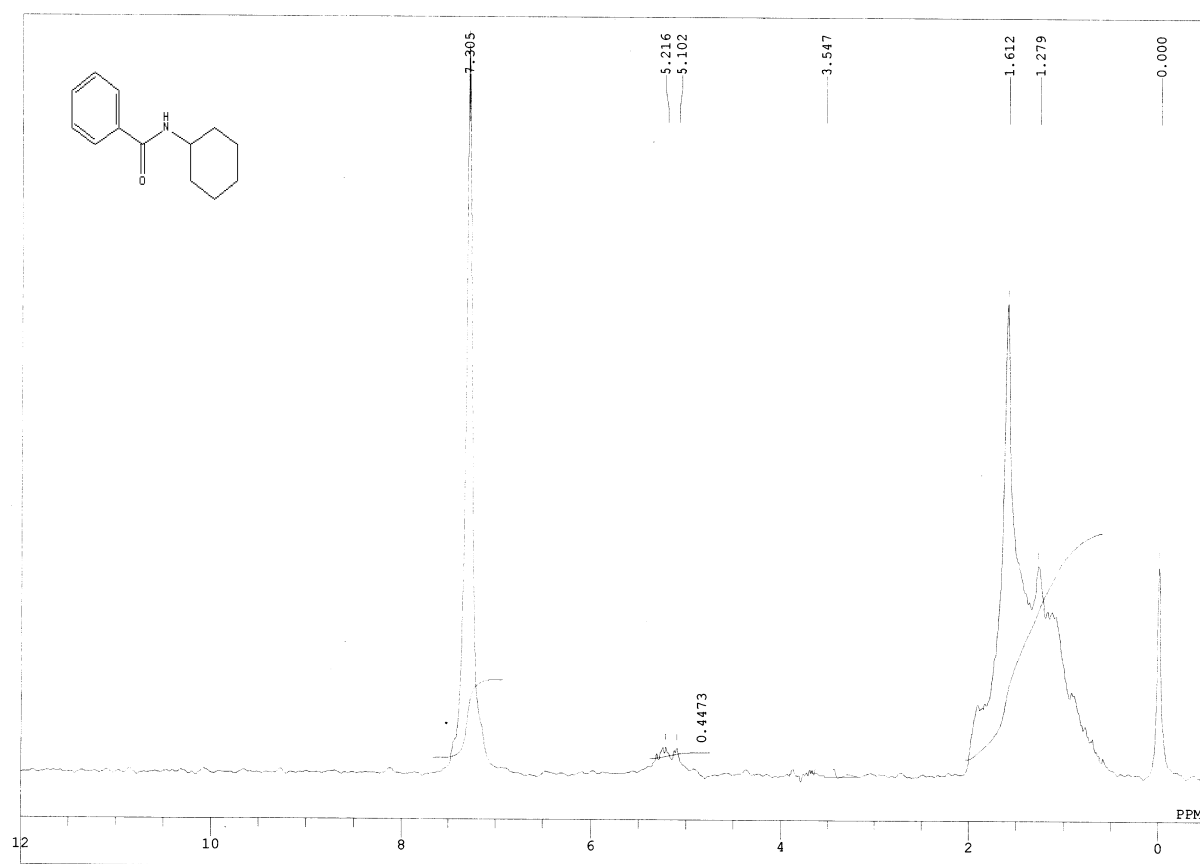
Entry 17) *N*-Benzylbenzamide



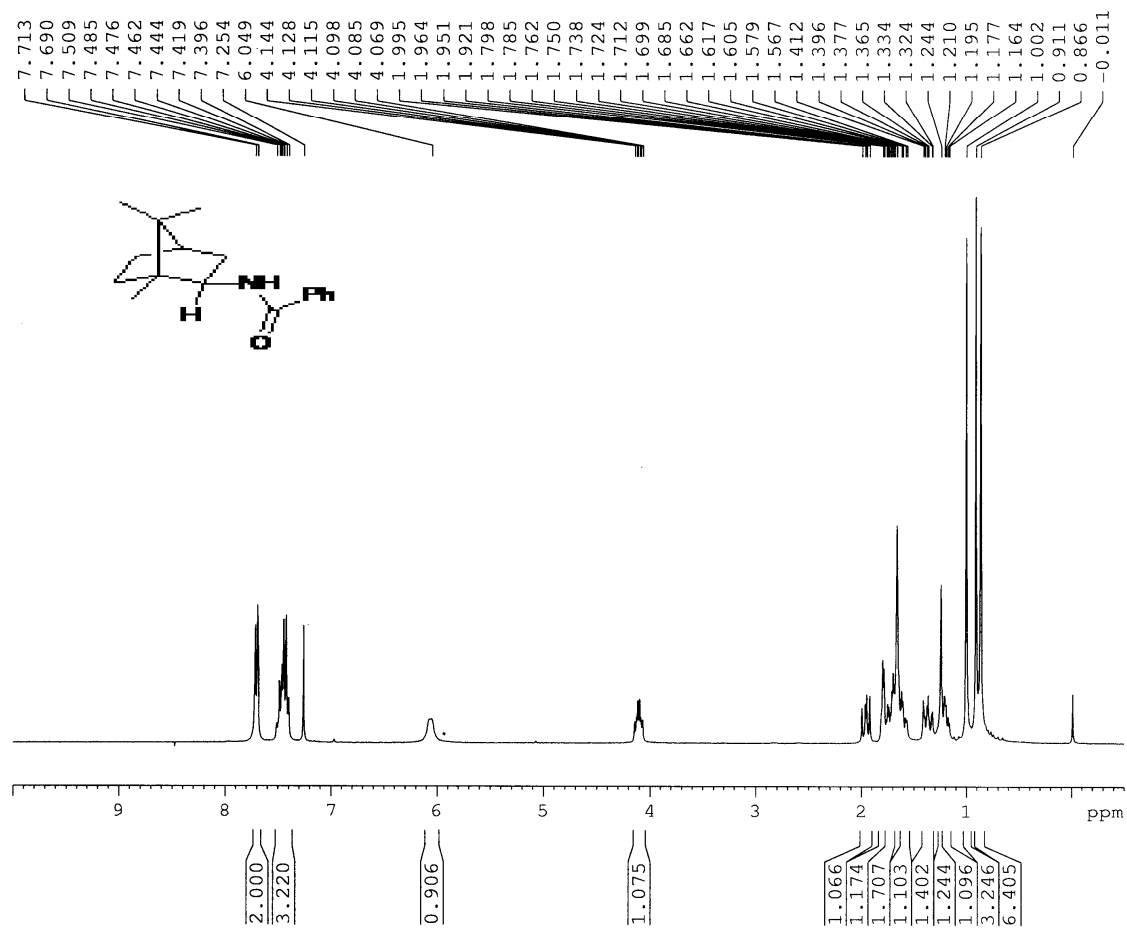
Entry 18) *N*-Benzyl-2-phenylacetamide



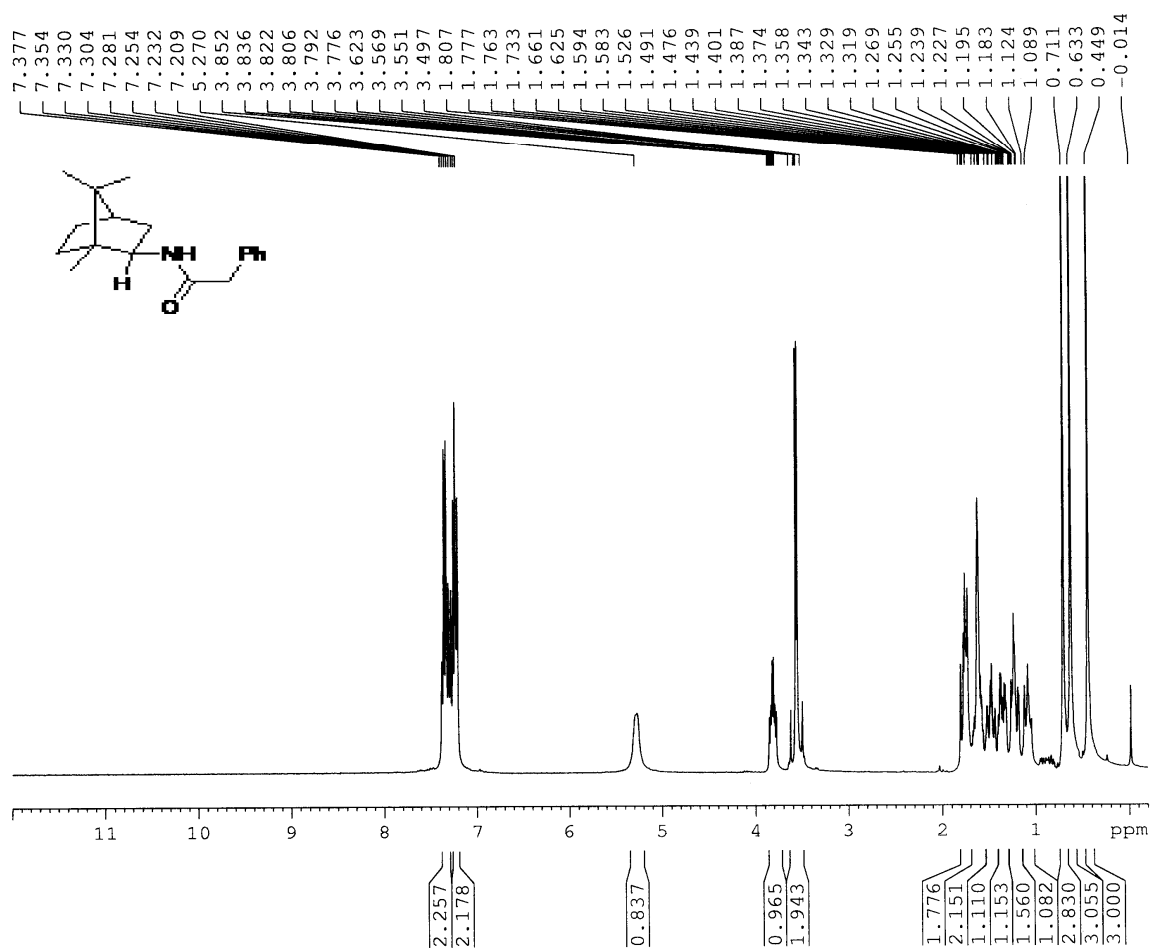
Entry 19) *N*-cyclohexylbenzamide



Entry 20) (\pm) *exo*-N- bornylbenzamide



Entry 21) (\pm) *exo*-N-bornyl-2-phenylacetamide



E) References:

1. a) J. R. Bowser, *J. Org. Chem.*, 1983, **48**, 4111; b) R. Nomura, *J. Org. Chem.*, 1991, **56**, 4076; c) L. Mamani, A. Heydari and M. Sheykhan, *Applied Catalysis A: General*, 2010, **39**, 122.
2. K. Niknam, M. A. Zolfigol, T. Sadabadia; *J. Iran. Chem. Soc.* 2007, **4**, 199.
3. T. Okuhara, X. Chen, *Microporous and Mesoporous Materials*. 2001, **48**, 293.
4. B. B. F. Mirjalilia and B. Sadeghi, *Iran. J. Org. Chem.* 2009, **2**, 76.
5. a) P. Theerthagiri, A. Lalitha, P. N. Arunachalam, *Tetrahedron Lett.* 2010, **51**, 2813. b) X. Wan, Z. Ma, B. Li, K. Zang, S. Cao, S. Zhang, Z. Shi, *J. Am. Chem. Soc.* 2006, **128**, 7416.
6. (a) K. L. Reddy, *Tetrahedron Lett.* 2003, **44**, 1453; (b) J. C. Baum, J. E. Milne, J. A. Murry, O. R. Thiel, *J. Org. Chem.* 2009, **74**, 2207.
7. a) B. Kumar, H. Kumar, and N. Singh, *Indian J. Chem.*, 1991, **30B**, 460; b) H. Firouzabadi, A. R. Sardarian. and H. Badparva, *Synth. Commun.*, 1994, **24**, 601.
8. R. Allmann, A. Frankowski and J. Streith, *Tetrahedron*, 1972, **28**, 581.
9. P. Salehi and A. R. Motlagh, *Synth. Commun.* 2000, 671.
10. A. A. Bakibaev, *Khimiko-Farmatsevticheskii Zhurnal*, 1989, **23**, 1455.
11. B. Adele; D. M. Vittoria, M., Orazio and G. Federico, *Tetrahedron*, 1991, **47**, 7417.
12. A. R. Katritzky, C. Cai and S. K. Singh, *J. Org. Chem.*, 2006, **71**, 3375.
13. J. Youngshin, *J. Org. Chem.*, 2009, **74**, 6358.
14. Y. Kasashima, A. Uzawa, K. Hashimoto, Y. Yokoyama, T. Mino, M. Sakamoto, and T. Fujita, *J. Oleo Science*, 2010, **59**, 607.
15. M. Mousseron, *Bull. Soc. Chim. Fr.* 1947, 868.
16. P. Theerthagiri, A. Lalitha, P. N. Arunachalam, *Tetrahedron Lett.* 2010, **51**, 2813.
17. B. Anxionnat, A. Guérinot, S. Reymond and J. Cossy, *Tetrahedron Lett*, 2009, **50**, 3470.
18. M. M. Lakourag, B. Movassagh and J. Fasihi, *Synth. Commun.* 2000, **30**, 821.
19. F. Tamaddon, M. Khoobi and E. Keshavarz, *Tetrahedron Lett*, 2007, **48**, 3643.
20. J. S. Yadav, B. V. S. Reddy, T. Pandurangam, Y. J. Reddy and M. K. Gupta. *Catal. Commun.* 2008, **9**, 1297.