

A facile one-pot synthesis of ruthenium hydroxide nanoparticles on magnetic silica: Aqueous hydration of nitriles to amides

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Supporting Information

Experimental section

Synthesis of magnetic silica supported ruthenium hydroxide nanoparticles

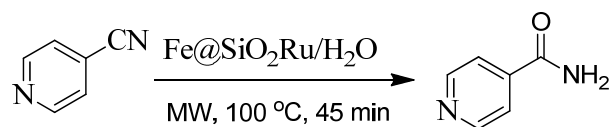
FeSO₄·7H₂O (2.78 g) and Fe₂(SO₄)₃ (4.0 g) were dissolved in 200 mL water in a 500 mL beaker. Ammonium hydroxide (25%) was added slowly to adjust the pH of the solution to 10. The reaction mixture was then continually stirred for 1 h at 50 °C. The reaction mixture was cooled down to room temperature. To this solution, tetraethyl orthosilicate (TEOS, 10 mL) was added and vigorous stirring was continued for 18 h at ambient conditions. To this solution, RuCl₃ (600 mg) was added and the pH of the solution was adjusted to ~10 using ammonium hydroxide solution (25 %) and stirring continued for another 24 h (**Scheme 1**). Magnetic silica supported Ru-hydroxide nano particles were separated using an external magnet, washed with water, followed by acetone, and dried under vacuum at 50 °C for 8 hours. Catalyst characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The weight percentage of Ru was found to be 3.96 % and Si 6.85% by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) analysis.

Hydration of Nitriles

Nitriles (1.0 mmol), and Fe₃O₄@SiO₂Ru (100 mg, 0.0039% of Ru) were placed in a crimp-sealed thick-walled glass tube equipped with a pressure sensor and a magnetic stirrer. Water (6 mL) was added to the reaction mixture. The reaction tube was placed inside the cavity of a CEM Discover focused microwave synthesis system, operated at 100 °C (temperature monitored by a built-in infrared sensor), 100 Watts and 10–60 psi for 45 min-2.5h. After completion of the

reaction, the catalyst was easily removed from reaction mixture using an external magnet. Hydration of nitriles has been monitored using GCMS. The clear liquid was cooled slowly, most of the time analytically pure crystals of corresponding amide were obtained which can be isolated from water medium by simple decantation.

Table 1 Recycling of the catalyst



Entry ^a	Catalyst	Time	Temperature (°C)	Yield ^b
1	Fe@SiO ₂ Ru	45 min	100	>99%
2	Fe@SiO ₂ Ru	45 min	150	>99%
3	Fe@SiO ₂ Ru	45 min	100	>99%

(a) Reactions were carried out with 1 mmol of 4-cyano pyridine, 100 mg nanocatalyst, in water under MW irradiation. (b) Reaction was monitored by GCMS

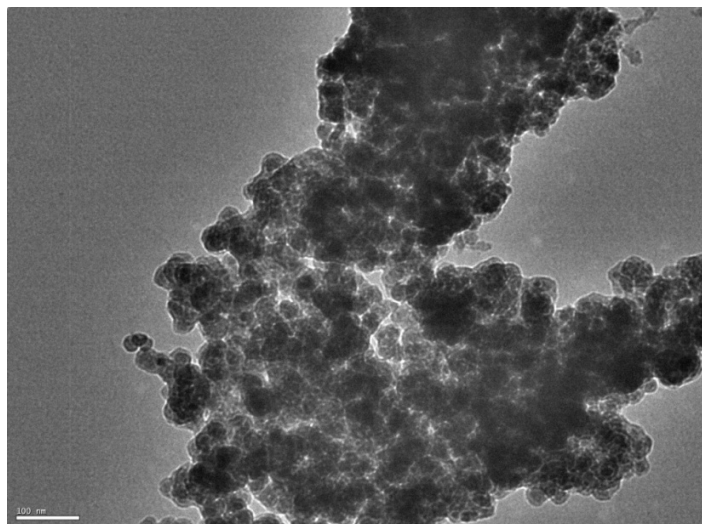
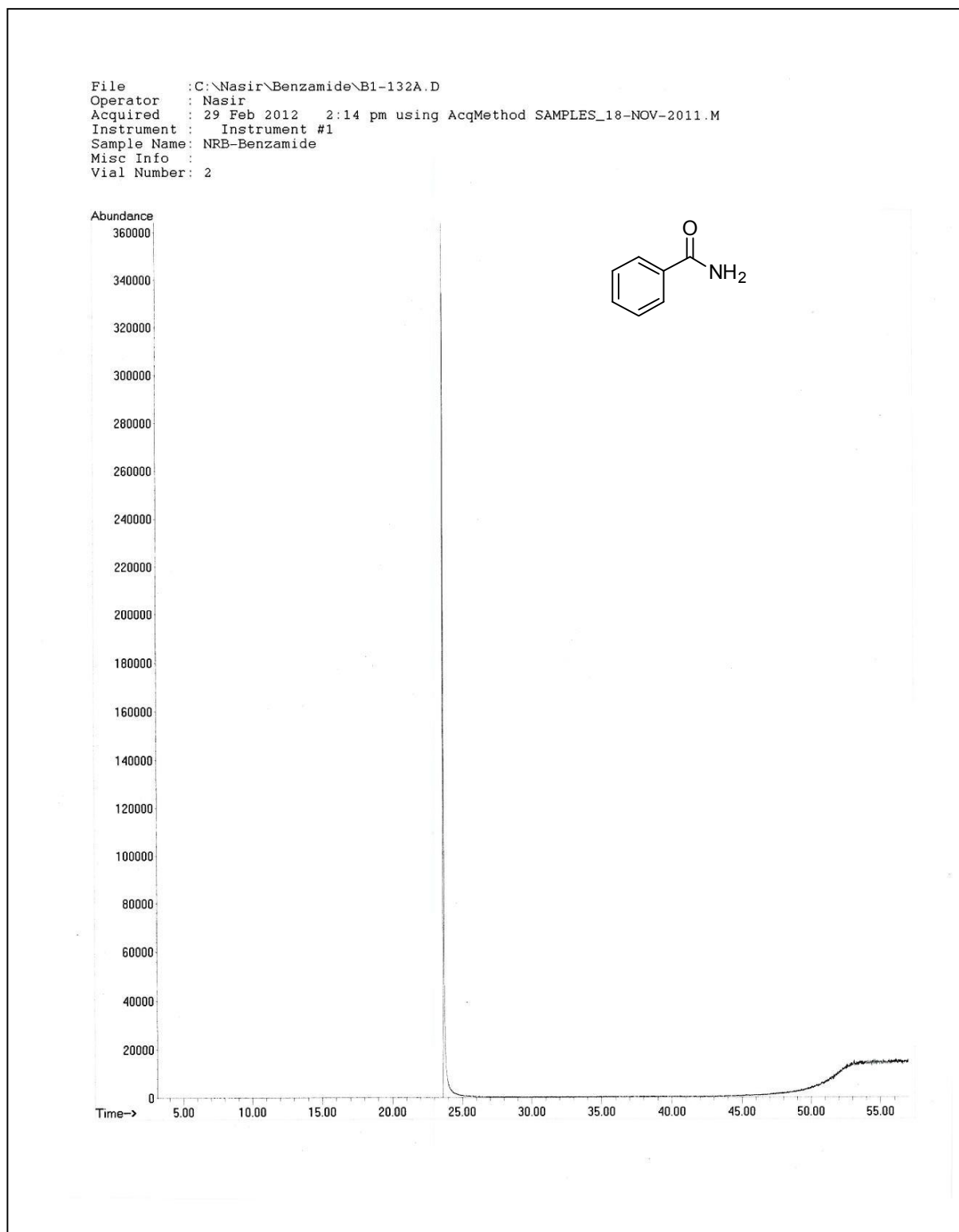
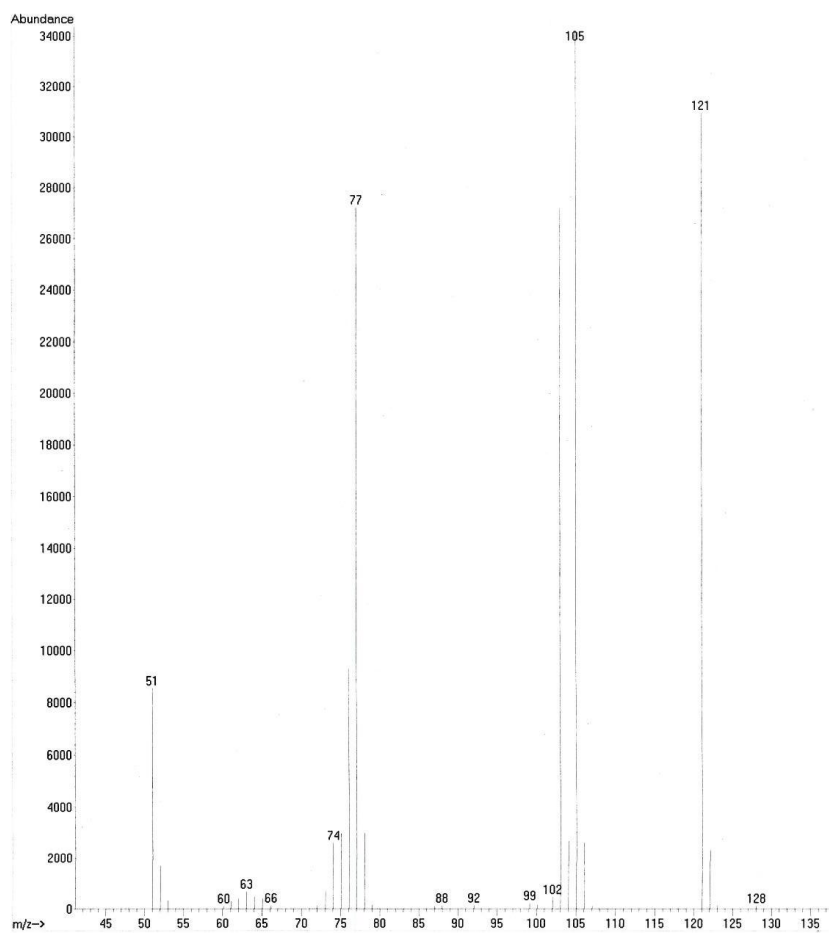
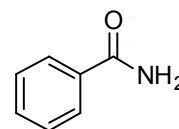


Figure 1 TEM image after recycling of the catalyst

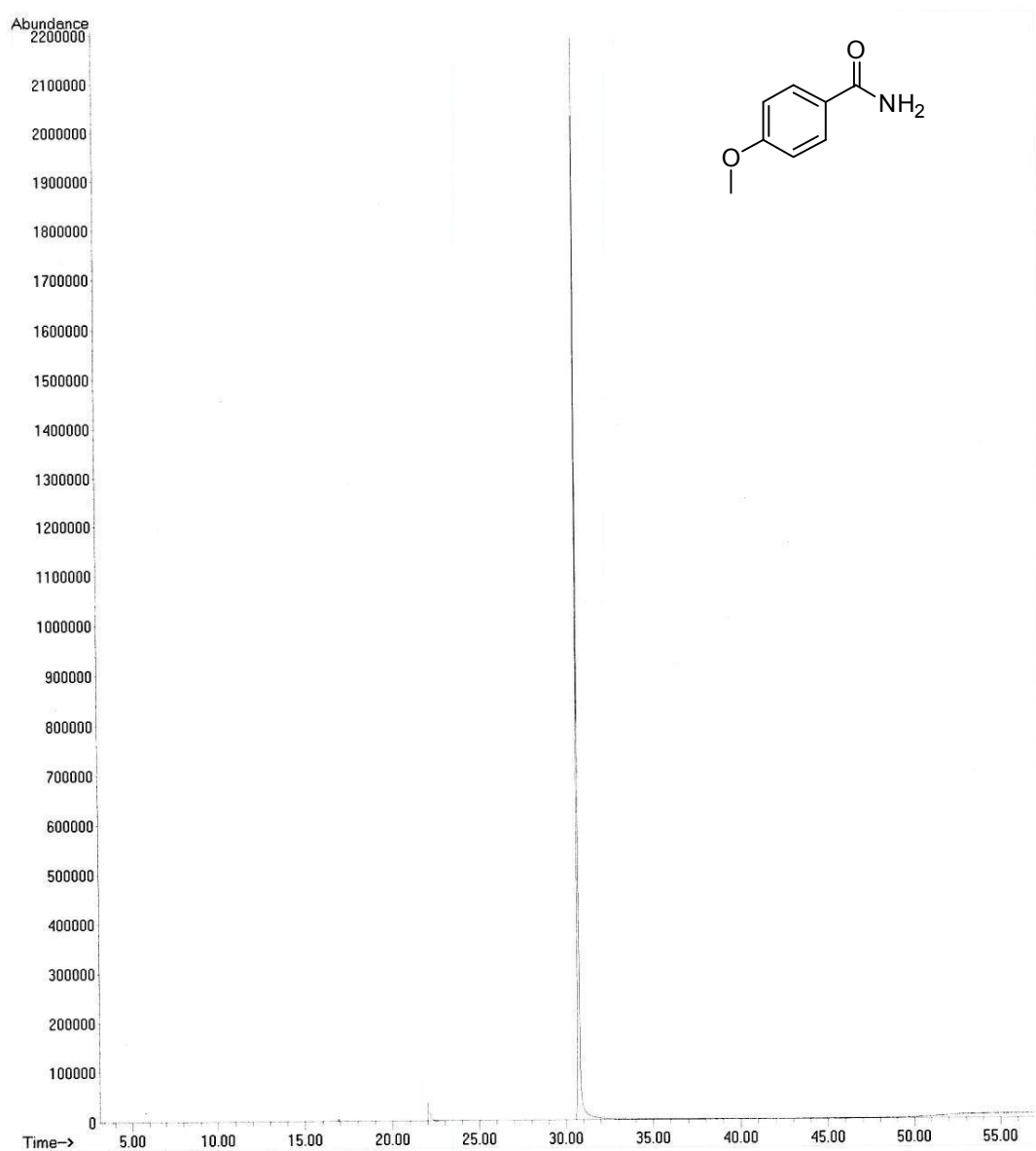
GC pattern and Mass spectra for the representative compounds:



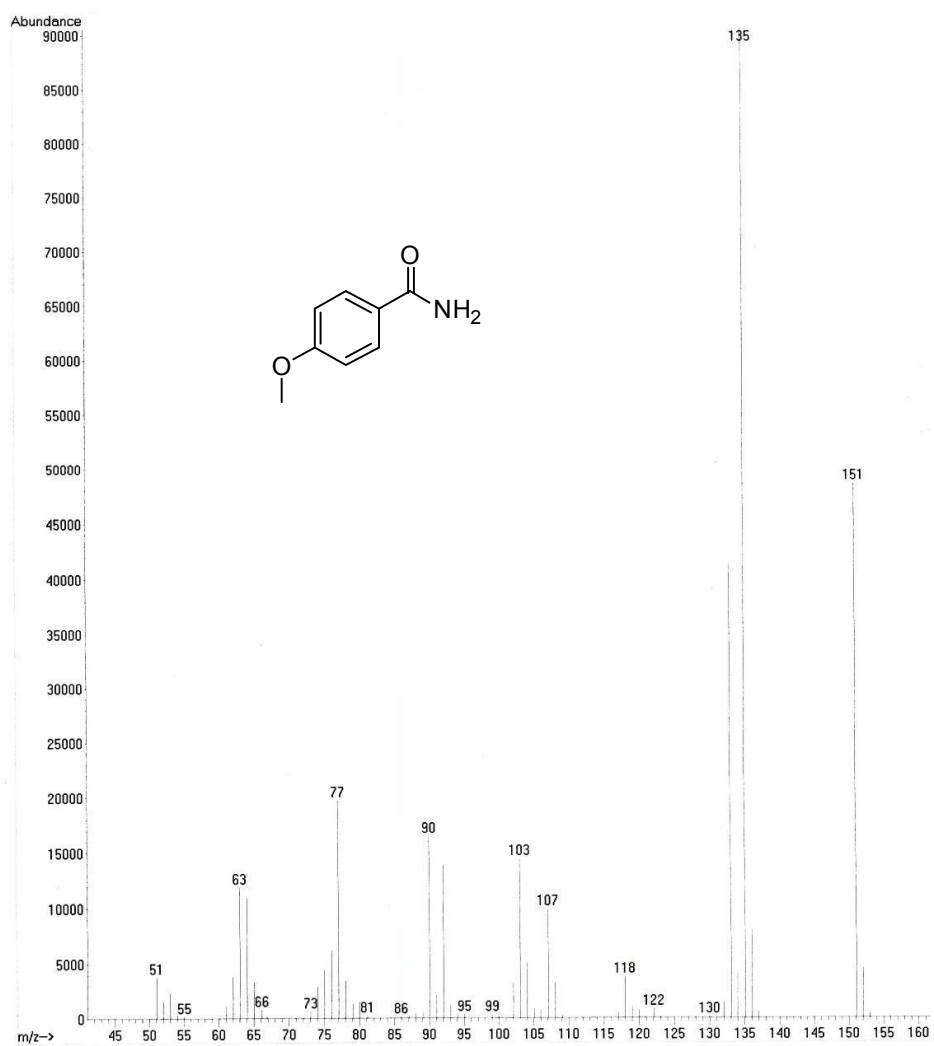
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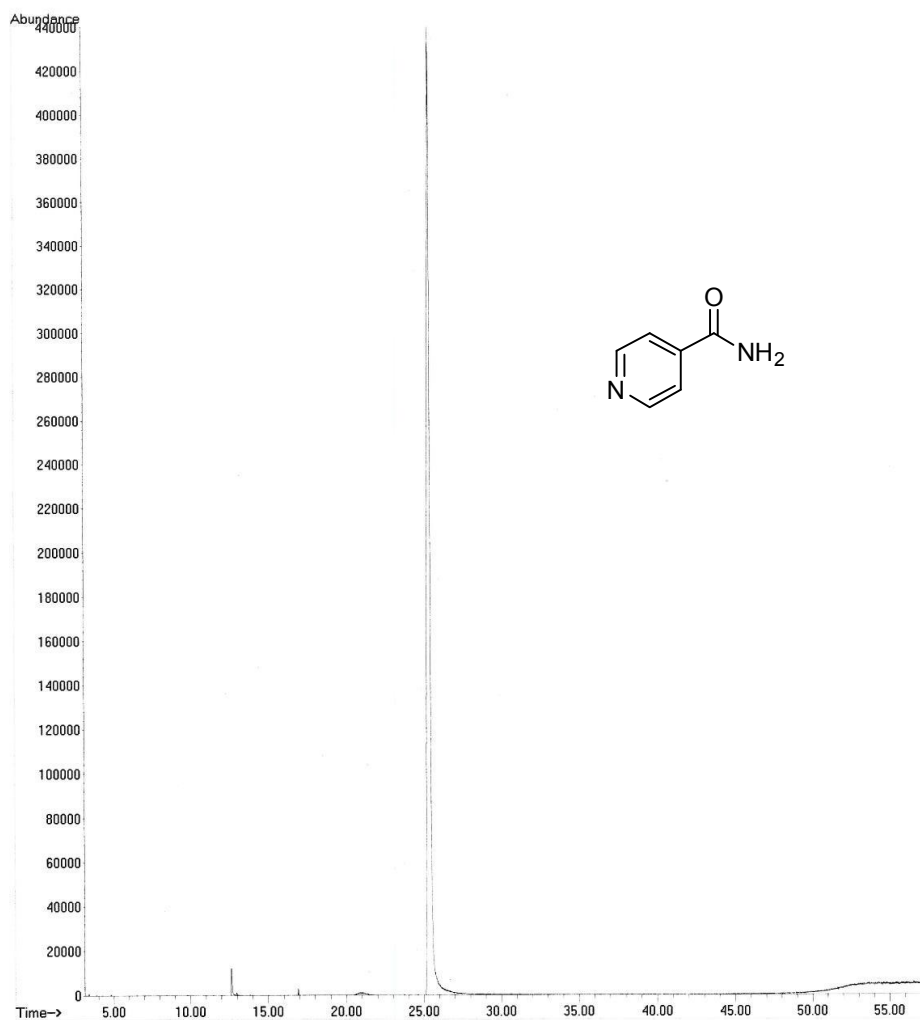
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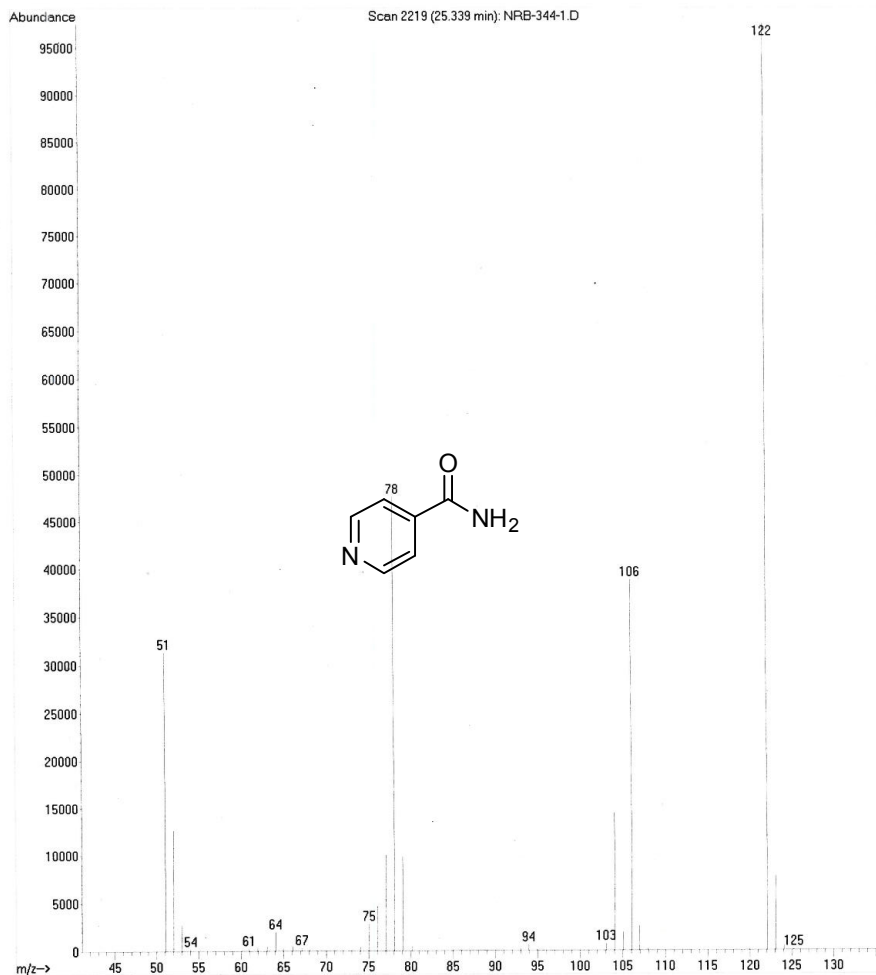
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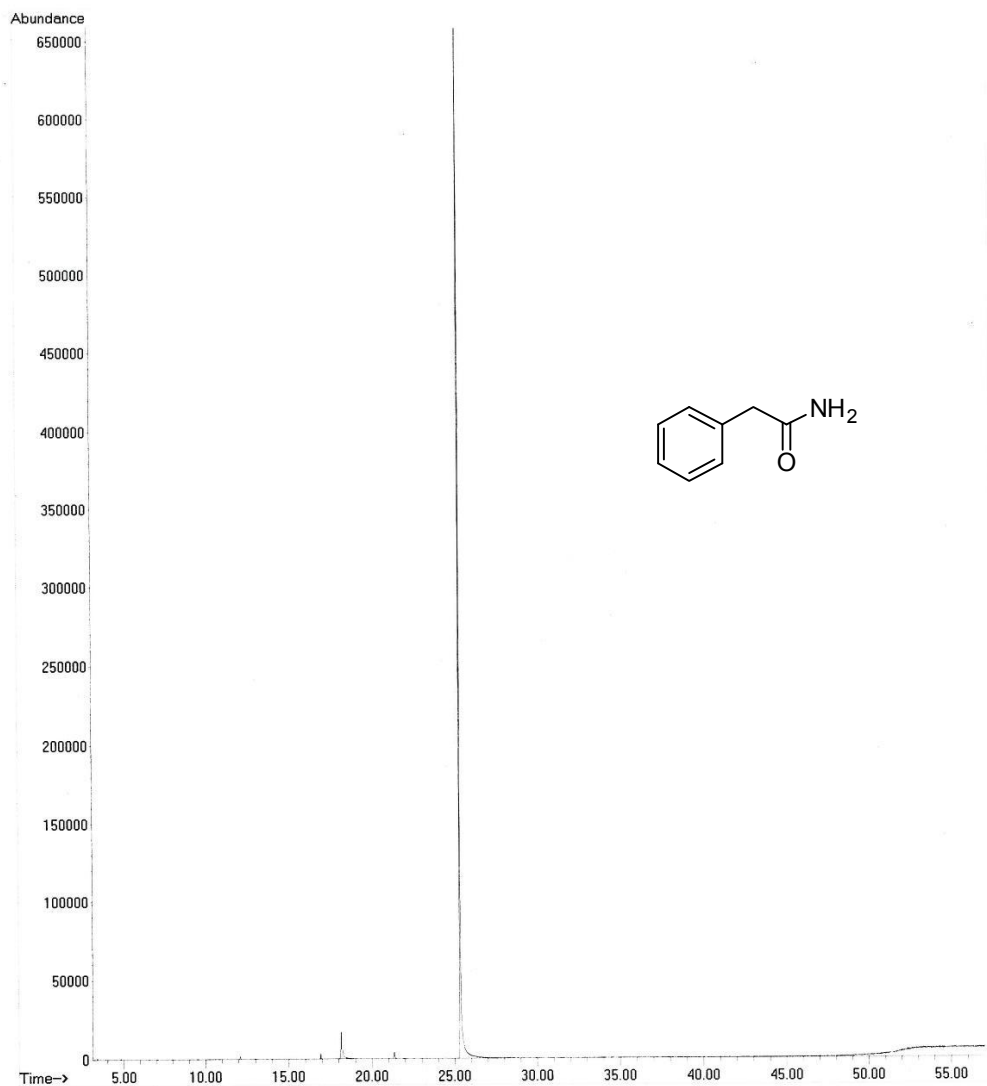
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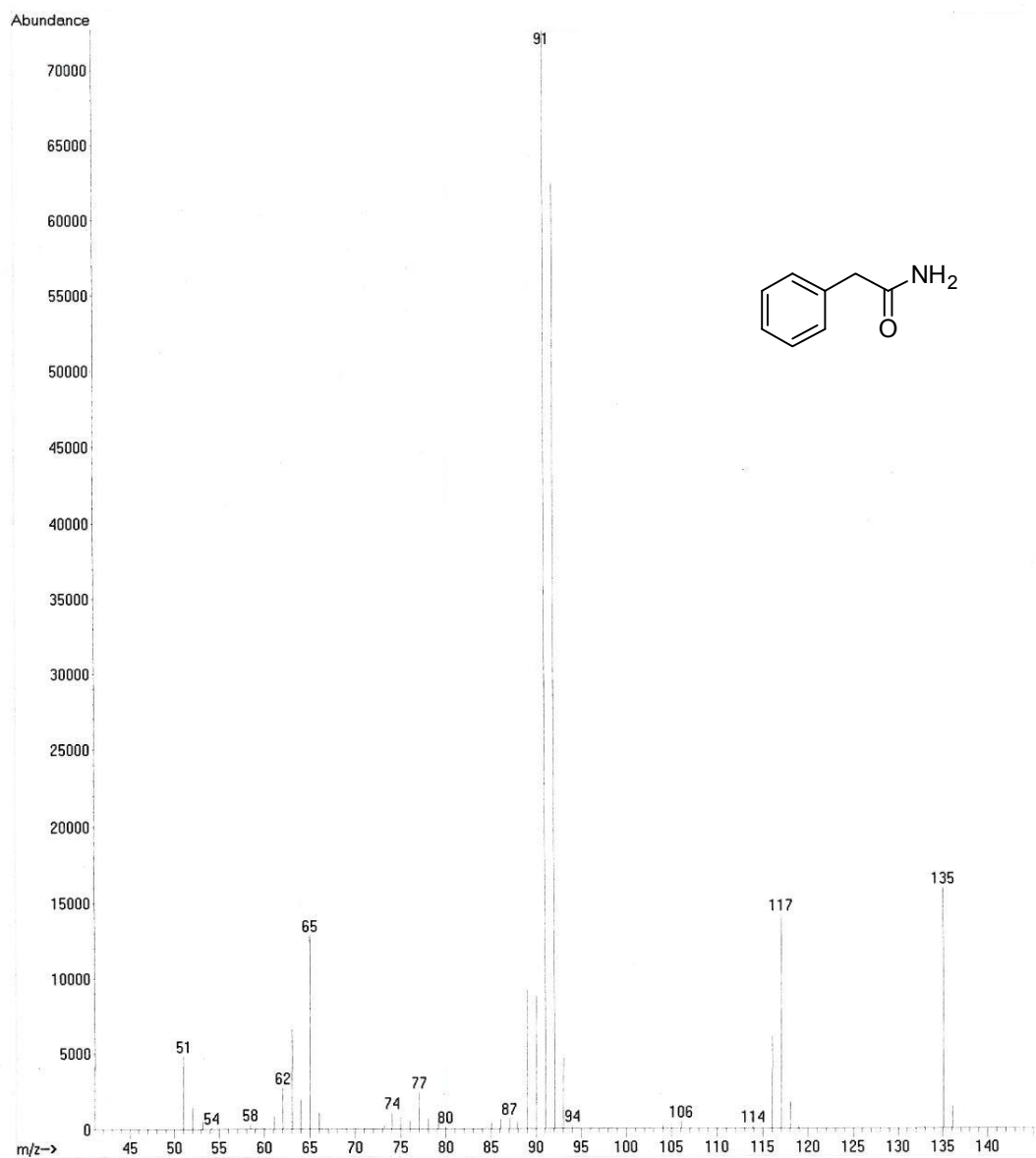
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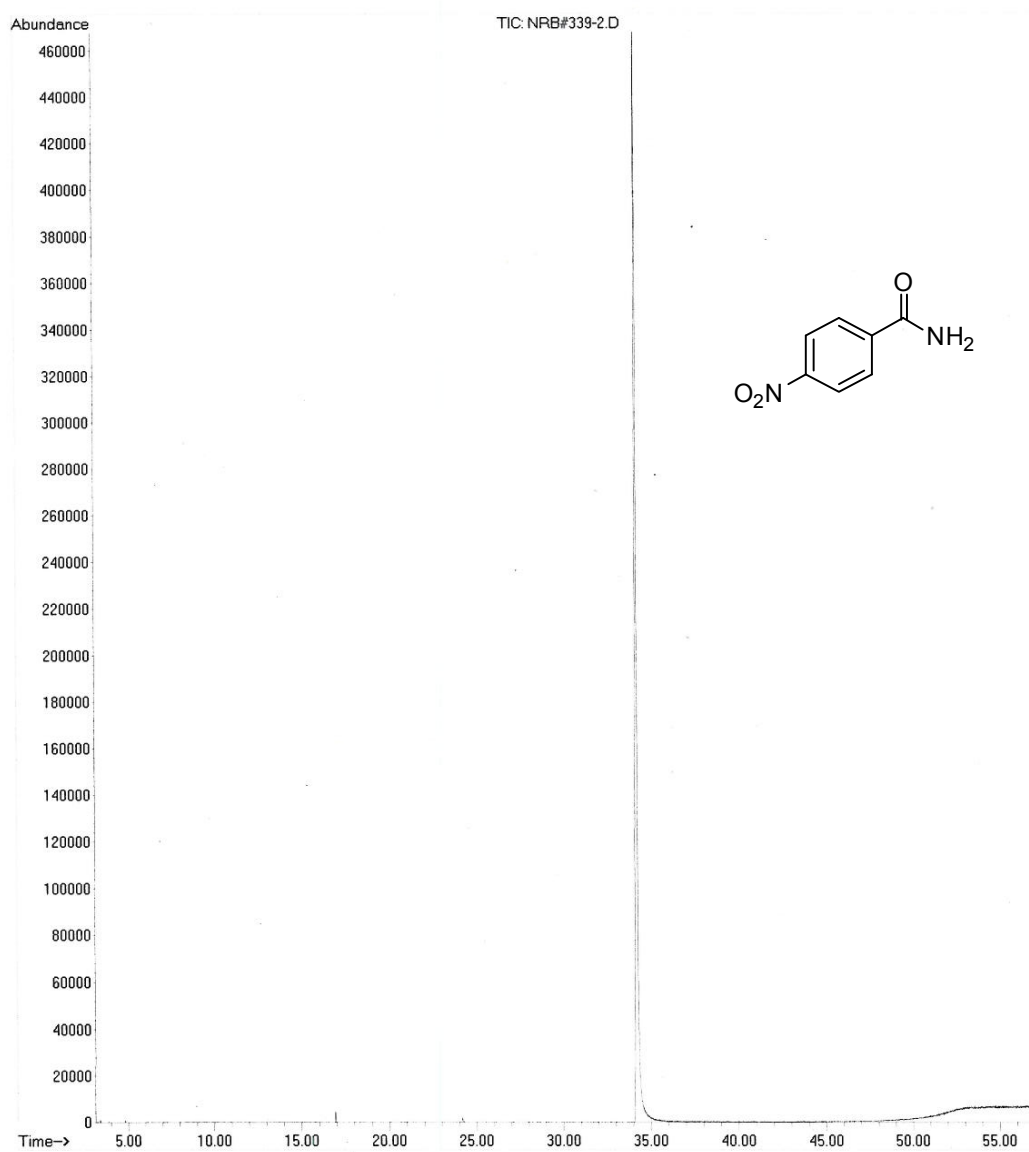
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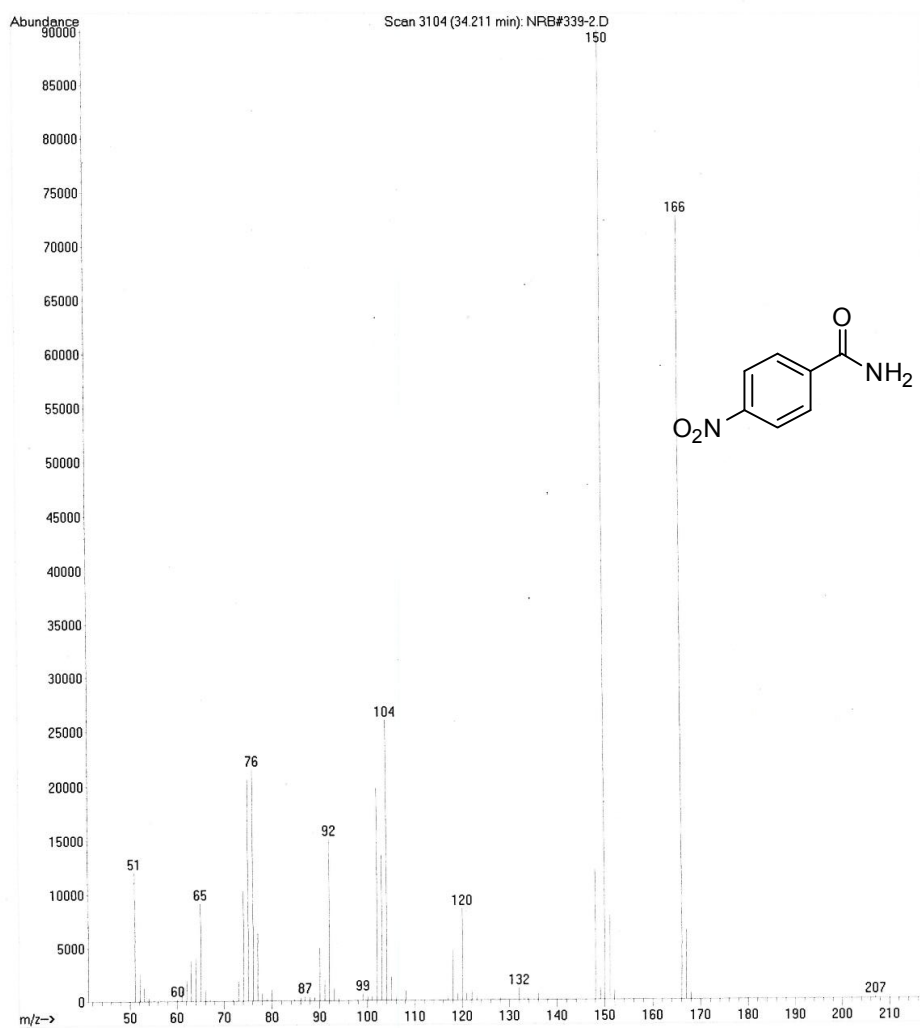
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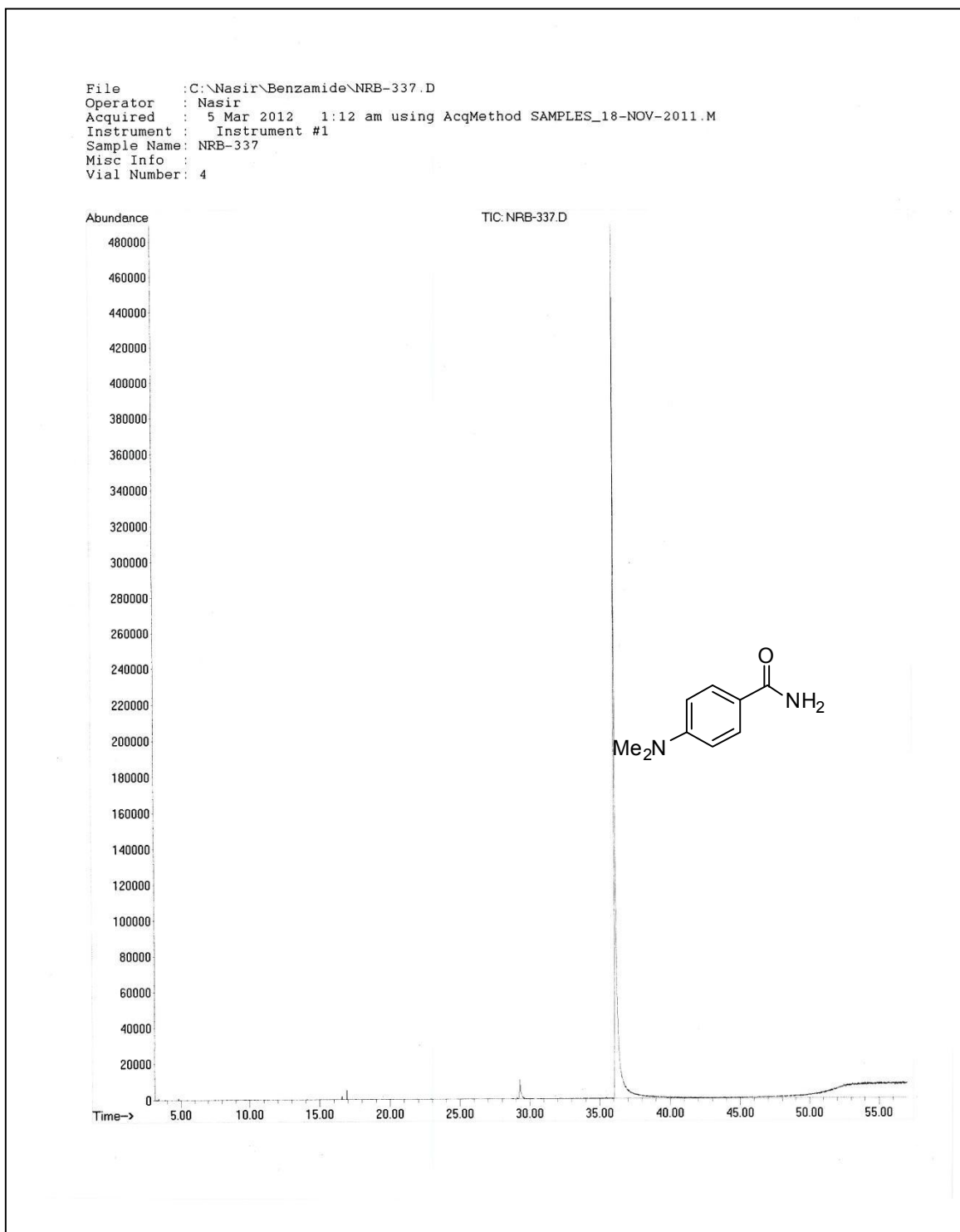


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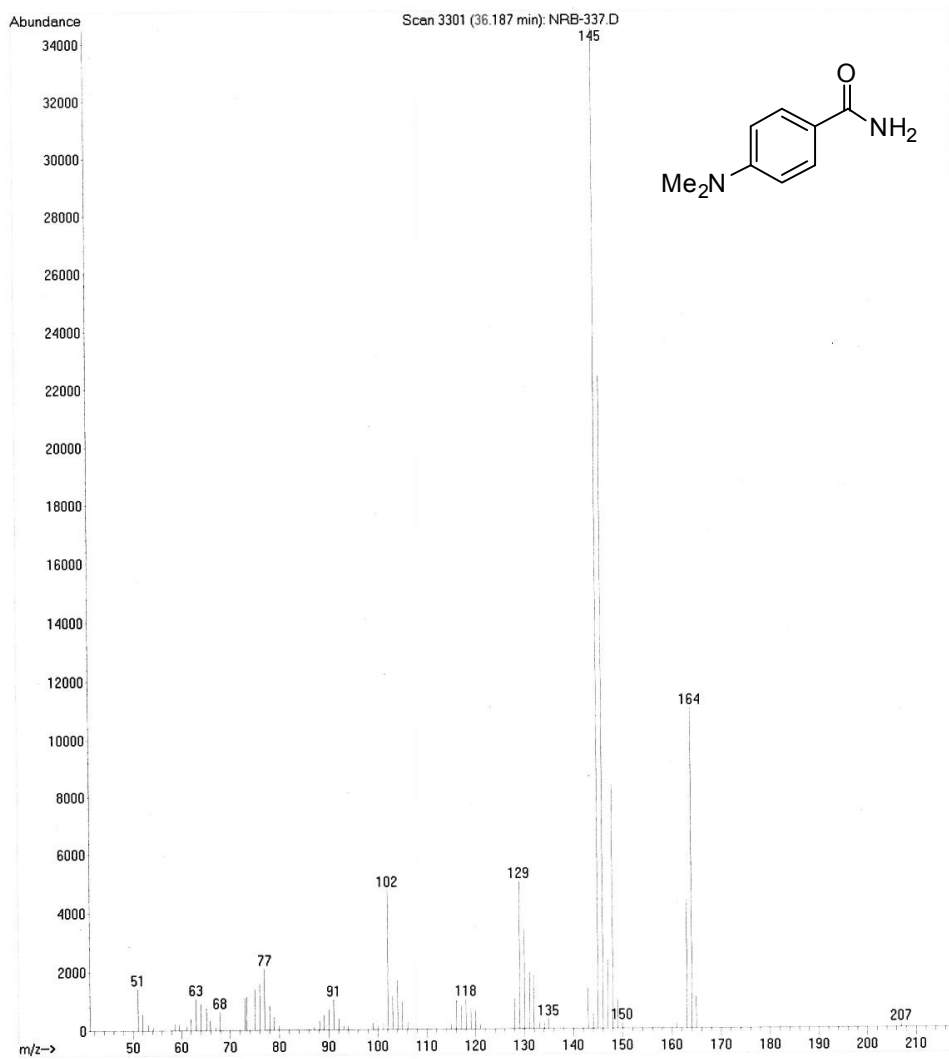


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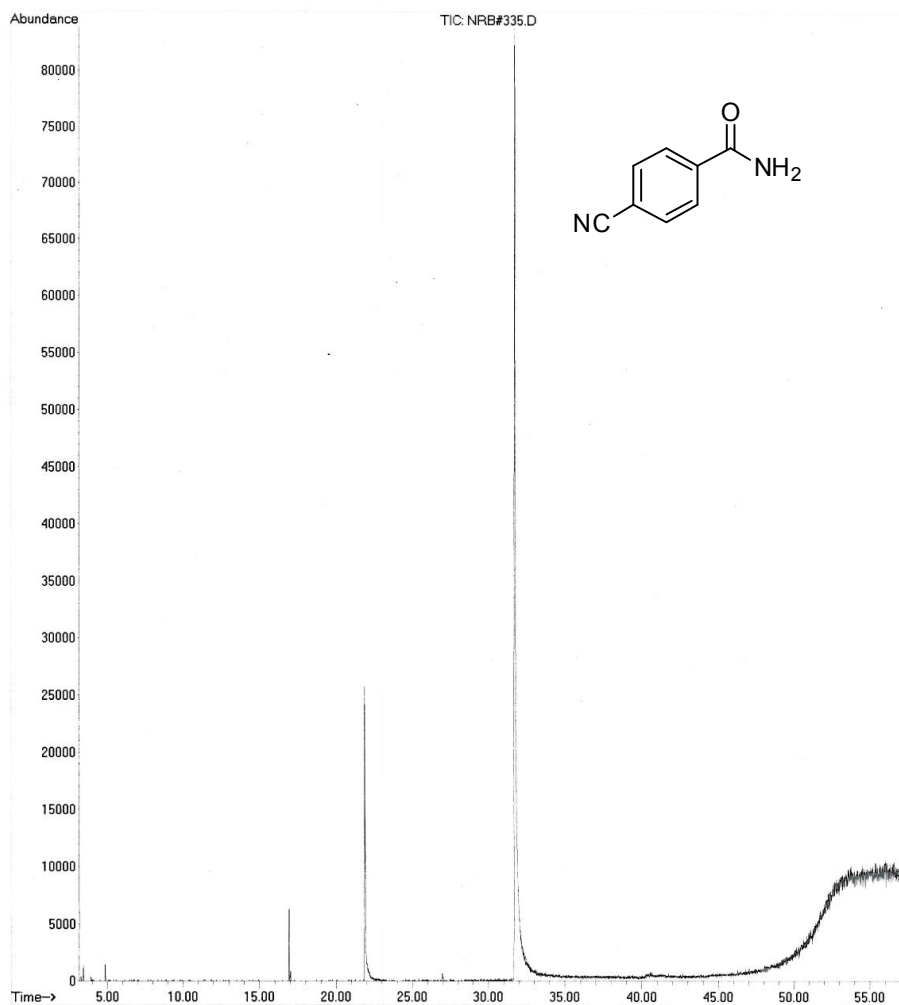




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Misc Info :
Vial Number: 4



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Instrument : Instrument #1
Sample Name: NRB#335
Misc Info :
Vial Number: 5



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Instrument : Instrument #1
Sample Name : NRB#335
Misc Info :
Vial Number : 5

