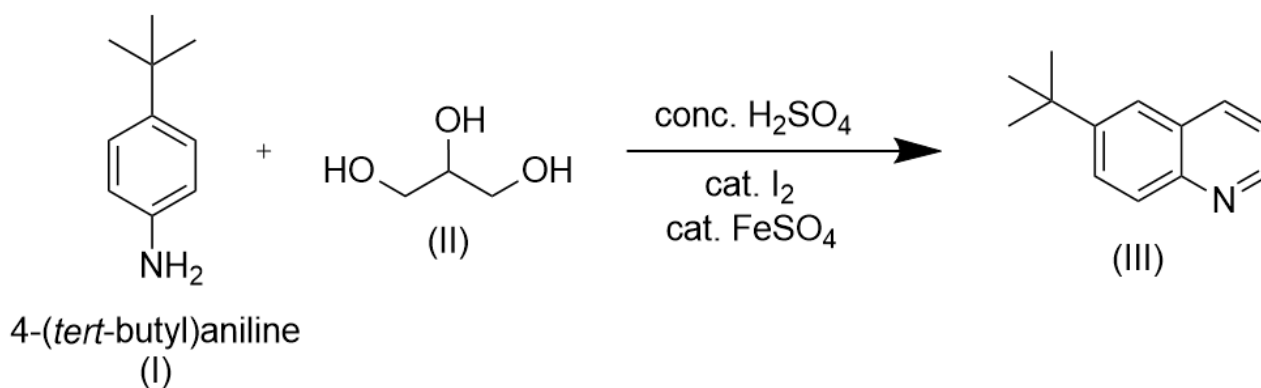


Synthesis of a substituted quinolineAbstract

In this experiment, a mono-substituted quinoline was synthesised via the Skraup synthesis by refluxing a substituted aniline with glycerol under strongly acidic and oxidizing conditions (conc.  $\text{H}_2\text{SO}_4$ ,  $\text{I}_2$ ,  $\text{FeSO}_4$ ). Glycerol dehydrated to form acrolein, followed by coupling of the aniline with the acrolein to produce a quinoline ring system. After 90 minutes of reflux at  $150^\circ\text{C}$  the reaction mixture was then neutralized and worked up by extraction, drying and removing solvent. The product was then analysed using  $^1\text{H}$  NMR, including COSY and NOESY data which allowed for precise assignment of proton signal and confirmation of the unknown starting aniline.

Experimental

The experiment was performed as detailed in the CHEM202 laboratory manual (p29), without modification. The accurate masses used of the reagents are detailed below.

Substance	Formula	$M_r$	mass (g)	amount (mmol)
4-( <i>tert</i> -butyl)aniline	$\text{C}_{10}\text{H}_{15}\text{N}$	149.23	0.863	5.78
Glycerol	$\text{C}_3\text{H}_8\text{O}_3$	92.09	3.09	33.55

During the reaction acid vapours were minimised by maintaining a proper reflux under the fume hood. After the reaction was completed the acid solution containing the product was neutralised using 5M NaOH until it tested basic on litmus paper. The celite used to gravity filter the solution was disposed of in the celite waste container. After washing of the solution, the aqueous layer was disposed of down the fume hood sink with excess water, the organic extract was dried using anhydrous  $\text{MgSO}_4$  which was filtered of and disposed of down the fume hood sink with excess water. Diethyl ether was distilled of from the product using the rotary evaporator.

## **Results and calculations**

### **Percentage yield calculation**

The limiting reagent in this reaction was (**4-(tert-butyl)aniline**).

$$\therefore n \text{ (product) expected} = 0.00578 \text{ mol}$$

$$M \text{ (product)} = 185.270 \text{ g mol}^{-1}$$

$$\text{Theoretical yield} = N \times M$$

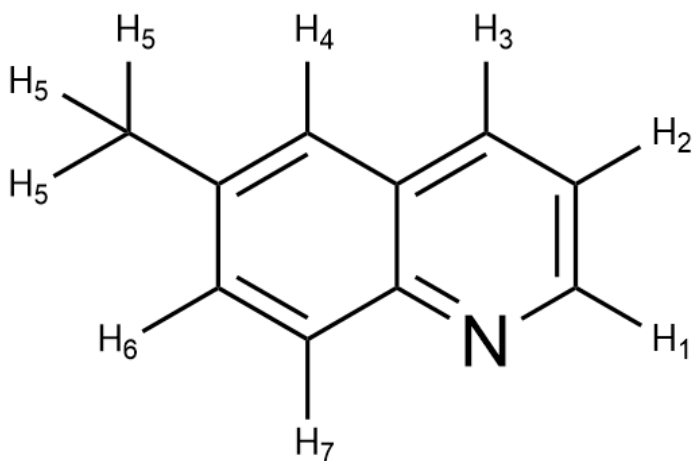
$$= 0.00578 \text{ mol} \times 185.270 \text{ g mol}^{-1}$$

$$= 1.071 \text{ g}$$

$$\text{Actual yield} = 0.192 \text{ g}$$

$$\therefore \text{Percentage yield} = \frac{0.192}{1.071} \times 100$$

$$= 17.92 \%$$



*<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)*

$\delta/\text{pm}$	Integration	multiplicity	J / Hz	COSY shows coupling to	Assignment
8.85	1H	dd	$J_{1,2} 7.7, J_{1,3} 1.7$	H <sub>2</sub>	H <sub>1</sub>
8.12	1H	d	$J_{3,2} 8.2$	H <sub>2</sub>	H <sub>3</sub>
8.05	1H	d	$J_{7,6} 8.9$	H <sub>6</sub>	H <sub>7</sub>
7.81	1H	dd	$J_{6,7} 8.9, J_{6,4} 2.2$	H <sub>4</sub> , H <sub>7</sub>	H <sub>6</sub>
7.72	1H	d	$J_{4,6} 2.2$	H <sub>6</sub>	H <sub>4</sub>
7.36	1H	dd	$J_{2,3} 8.2, J_{2,1} 4.2$	H <sub>1</sub> , H <sub>3</sub>	H <sub>2</sub>
1.43	12H	s	~	~	H <sub>5</sub>

## ESI-MS

<i>m/z</i>	[Assignment] <sup>charge</sup>
171.09	[C <sub>13</sub> H <sub>16</sub> N – CH <sub>3</sub> ] <sup>+</sup>
186.12	[C <sub>13</sub> H <sub>16</sub> N] <sup>•</sup>
187.13	[C <sub>13</sub> H <sub>16</sub> N + H] <sup>+</sup>

## Discussion

### **ADD YOUR DISCUSSION SECTION HERE**

- *Remember to append to your report your risk assessment form, and **all** spectral data.*
- *Make sure each appended item is clearly labelled with a title and explanatory legend.*