

# Synthesis of 3-nitrobenzoic acid

## Summary of the analysed protocols

$m\text{-NO}_2\text{C}_6\text{H}_4\text{COOCH}_3 + \text{NaOH} + \text{HCl} \rightarrow m\text{-NO}_2\text{C}_6\text{H}_4\text{COOH} + \text{CH}_3\text{OH} + \text{NaCl}$	(1)
$\text{C}_6\text{H}_5\text{COOCH}_3 + \text{HNO}_3 \rightarrow m\text{-NO}_2\text{C}_6\text{H}_4\text{COOCH}_3 + \text{H}_2\text{O}$	(2)
$\text{C}_6\text{H}_5\text{COOH} + \text{HNO}_3 \rightarrow m\text{-NO}_2\text{C}_6\text{H}_4\text{COOH} + \text{H}_2\text{O}$	(3)
$m\text{-NO}_2\text{C}_6\text{H}_4\text{COCH}_3 + \text{NaOH} + \text{NaOCl} + \text{HCl} \rightarrow m\text{-NO}_2\text{C}_6\text{H}_4\text{COOH} + \text{CH}_3\text{OH} + \text{NaCl}$	(4)
<b>Protocol A<sup>1</sup></b>	
<b>Reaction (R<sub>1</sub>):</b> equation (1), 695% exc. 10% sodium hydroxide solution, reflux, T > 100 °C	
<b>Isolation (I<sub>1</sub>):</b> filtration (suction) → washing (water)	
<b>Purification (Pu<sub>1</sub>):</b> recrystallization – dissolution (water) → drying (air)	
<b>Protocol B<sup>2,3</sup></b>	
<b>Reaction (R<sub>2</sub>):</b> equation (1), 100% exc. sodium hydroxide, 200% exc. hydrochloric acid, water (solvent), reflux, T > 100 °C	
<b>Isolation (I<sub>2</sub>):</b> cooling → filtration (suction)	
<b>Purification (Pu<sub>2</sub>):</b> recrystallization (1% hydrochloric acid aqueous solution)	
<b>Protocol C<sup>4</sup></b>	
<b>Reaction (R<sub>2</sub>):</b> ≡ Pr B (scale decreased to half)	
<b>Isolation (I<sub>3</sub>):</b> cooling → filtration (suction) → washing (water) → drying (at 100 °C)	
<b>Purification (Pu<sub>2</sub>):</b> ≡ Pr B	
<b>Protocol D<sup>5</sup></b>	
<b>Preparation of reagents (Prep<sub>1</sub>):</b> equation (2), 58% exc. nitric acid, sulphuric acid and methanol (auxiliary substances), T ~ 0-5 °C	
<b>Reaction (R<sub>3</sub>):</b> equation (1), 64-100% exc. sodium hydroxide, 195-260% exc. hydrochloric acid, water and activated charcoal (auxiliary substances), reflux, T > 100 °C	
<b>Isolation (I<sub>4</sub>):</b> cooling (ice bath) → filtration (suction) → washing (water) → drying (air)	
<b>Purification (Pu<sub>2</sub>):</b> ≡ Pr B	
<b>Protocol E<sup>6</sup></b>	
<b>Reaction (R<sub>4</sub>):</b> equation (3), 28% exc. nitric acid, water and sulphuric acid (auxiliary substances), T ~ 0-5 °C	
<b>Isolation (I<sub>5</sub>):</b> filtration (suction) → washing (cold water) → drying (air)	
<b>Purification (Pu<sub>3</sub>):</b> recrystallization (boiling water)	
<b>Protocol F<sup>6</sup></b>	
<b>Reaction (R<sub>5</sub>):</b> equation (4), 384% exc. 5% sodium hypochlorite solution, 35% exc. 10% sodium hydroxide solution, acetone (auxiliary substance), T ~ 100 °C	
<b>Isolation (I<sub>6</sub>):</b> cooling (ice bath) → filtration (suction) → drying (air)	
<b>Purification (Pu<sub>4</sub>):</b> recrystallization (hot water)	

<sup>a</sup> → – Sequential

## References

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- (6) Stradling, S.S.; Gage, C.L. And the winner is... A multistep synthesis for the Introductory Organic Course. *J. Chem. Educ.* **1985**, 62 (12), 1116-1117.