

Synthesis of 1-bromobutane

Summary of the analysed protocols

$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{NaBr} + \text{H}_2\text{SO}_4 \rightarrow \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{NaHSO}_4 + \text{H}_2\text{O}$ (1)
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{HBr} \rightarrow \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{H}_2\text{O}$ (2)
$\text{Br}_2 + \text{SO}_2 + \text{H}_2\text{O} \rightarrow 2\text{HBr} + \text{SO}_3$ (3)
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{KBr} + \text{H}_2\text{SO}_4 \rightarrow \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{KHSO}_4 + \text{H}_2\text{O}$ (4)
Protocol A¹
Reaction (R₁): equation (1), 60% exc. sodium bromide, 147% exc. sulphuric acid, water (solvent), reflux, T > 100 °C Isolation (I₁): washing (ethyl ether → water → 5% sodium hydrogen carbonate solution) → drying (magnesium sulphate) → filtration (gravity) → evaporation of the solvent Purification (Pu₁): not prescribed
Protocol B²
Reaction (R₂): equation (1), 51% exc. sodium bromide, 137% exc. sulphuric acid, water (solvent), reflux, T > 100 °C Isolation (I₂): washing (9M sulphuric acid solution → water → saturated sodium hydrogen carbonate solution) → drying (anhydrous calcium chloride) Purification (Pu₁): simple distillation
Protocol C³
Reaction (R₂): ≡ Pr B Isolation (I₂): ≡ Pr B Purification (Pu₁): not prescribed
Protocol D⁴
Reaction (R₂): ≡ Pr B (scale 7 times reduced) Isolation (I₃): washing (9M sulphuric acid solution → water → saturated sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) Purification (Pu₁): ≡ Pr B
Protocol E⁵
Reaction (R₃): equation (1), 16% exc. sodium bromide, 83% exc. sulphuric acid, water (solvent), reflux, T > 100 °C Isolation (I₄): simple distillation → washing (water → cold sulphuric acid → 10% sodium hydroxide solution) → drying (magnesium sulphate) Purification (Pu₁): not prescribed
Protocol F⁶
Reaction (R₃): ≡ Pr E Isolation (I₅): simple distillation → washing (water → cold sulphuric acid → 10% sodium hydroxide solution) → drying (anhydrous calcium chloride) → decantation Purification (Pu₁): ≡ Pr B
Protocol G⁷
Reaction (R₄): equation (1), 20% exc. sodium bromide, 88% exc. sulphuric acid, water (solvent), reflux, T > 100 °C Isolation (I₆): simple distillation → washing (water → sulphuric acid → 3M sodium hydroxide solution) → drying (magnesium sulphate) → decantation Purification (Pu₁): not prescribed
Protocol H⁸
Reaction (R₅): equation (1), 19% exc. sodium bromide, 87% exc. sulphuric acid, water (solvent), reflux, T > 100 °C Isolation (I₇): simple distillation → washing (water → sulphuric acid → 3M sodium hydroxide solution) → drying (anhydrous calcium chloride) → decantation → washing (<i>p</i> -xylene) Purification (Pu₁): ≡ Pr B
Protocol I⁸
Reaction (R₆): equation (1), 18% exc. sodium bromide, 94% exc. sulphuric acid, water (solvent), reflux, T > 100 °C Isolation (I₈): simple distillation → washing (water → cold sulphuric acid → 3M sodium hydroxide solution) → drying (anhydrous calcium chloride) → decantation Purification (Pu₁): ≡ Pr B

Protocol J⁹
<p>Reaction (R₆): ≡ Pr I (scale enlarged to double)</p> <p>Isolation (I₉): simple distillation → washing (water → cold sulphuric acid → 10% sodium hydroxide solution) → drying (anhydrous calcium chloride) → heating (steam bath) → decantation</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol K¹⁰
<p>Reaction (R₇): ≡ Pr J (but different time of the reflux)</p> <p>Isolation (I₉): ≡ Pr J</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol L¹¹
<p>Reaction (R₈): equation (1), 21% exc. sodium bromide, 113% exc. sulphuric acid, water (solvent), reflux, T > 100 °C</p> <p>Isolation (I₁₀): simple distillation → washing (sulphuric acid) → decantation</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol M¹²
<p>Reaction (R₉): equation (1), stoichiometric proportions of butan-1-ol and sodium bromide, 84% exc. sulphuric acid, water (solvent), reflux, T > 100 °C</p> <p>Isolation (I₁₁): simple distillation → washing (water → 2M sodium hydroxide solution → saturated sodium chloride solution) → drying (anhydrous sodium sulphate)</p> <p>Purification: not prescribed</p>
Protocol N¹³
<p>Reaction (R₁₀): equation (1), 14% exc. sodium bromide, 84% exc. sulphuric acid, water (solvent), reflux, T > 100 °C</p> <p>Isolation (I₁₂): simple distillation → washing (water → hydrochloric acid → 5% sodium hydrogen carbonate solution) → drying (anhydrous magnesium sulphate) → filtration (glass funnel)</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol O^{14,15}
<p>Reaction (R₁₁): equation (1), 25% exc. sodium bromide, 67% exc. sulphuric acid, water (solvent), reflux, T > 100 °C</p> <p>Isolation (I₁₃): simple distillation → washing (water → cold sulphuric acid → sodium carbonate solution) → drying (anhydrous calcium chloride)</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol P¹⁶
<p>Reaction (R₁₂): equation (1), 11% exc. sodium bromide, 132% exc. sulphuric acid, reflux, T > 100 °C</p> <p>Isolation (I₁₄): simple distillation → washing (water → cold 80% sulphuric acid solution → saturated sodium hydrogen carbonate solution) → drying (anhydrous calcium chloride)</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol Q¹⁷
<p>Reaction (R₁₃): equation (1), 13% exc. sodium bromide, 64% exc. sulphuric acid, water (solvent), reflux, T > 100 °C</p> <p>Isolation (I₁₅): simple distillation → washing (cold sulphuric acid → water → diluted sodium carbonate solution) → drying (anhydrous calcium chloride or anhydrous magnesium sulphate) → decantation</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol R¹³
<p>Reaction (R₁₄): equation (2), 24% exc. hydrobromic acid, sulphuric acid (catalyst), reflux, T > 100 °C</p> <p>Isolation (I₁₂): ≡ Pr N</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol S¹⁸
<p>Preparation of reagents (Prep₁): equation (3), excess of sulphur dioxide, T ~ 0 °C</p> <p>Reaction (R₁₄): ≡ Pr R (scale enlarged 5 times)</p> <p>Isolation (I₁₆): simple distillation → washing (water → hydrochloric acid → 5% sodium hydrogen carbonate or sodium carbonate solution) → drying (anhydrous calcium chloride or anhydrous magnesium sulphate) → filtration (glass funnel)</p> <p>Purification (Pu₁): ≡ Pr B</p>
Protocol T^{14,15}
<p>Preparation of reagents (Prep₁): ≡ Pr S (scale enlarged 10 times)</p> <p>Reaction (R₁₄): ≡ Pr R (scale enlarged 50 times)</p> <p>Isolation (I₁₃): ≡ Pr O</p> <p>Purification (Pu₁): ≡ Pr B</p>

Protocol U¹⁸

Reaction (R₁₅): equation (4), 65% exc. potassium bromide, 381% exc. sulphuric acid, water (solvent), reflux, T > 100 °C
Isolation (I₁₇): simple distillation → washing (hydrochloric acid → water → 10% sodium carbonate solution) → drying (anhydrous calcium chloride) → filtration (glass funnel)

Purification (Pu₁): ≡ Pr B

^a → – Sequential

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