

Synthesis of *tert*-butyl chloride

Summary of the analysed protocols

$(\text{CH}_3)_3\text{COH} + \text{HCl} \rightarrow (\text{CH}_3)_3\text{CCl} + \text{H}_2\text{O}$	(1)
Protocol A¹	
Reaction (R₁): equation (1), 362% exc. hydrochloric acid, T ~ 0 °C	
Isolation (I₁): washing (water → 5% potassium carbonate solution) → drying (anhydrous calcium chloride) → filtration (gravity)	
Purification (Pu₁): simple distillation	
Protocol B²	
Reaction (R₂): equation (1), 362% exc. hydrochloric acid, room temperature	
Isolation (I₂): washing (5% potassium carbonate solution) → drying (anhydrous calcium chloride) → decantation	
Purification (Pu₁): ≡ Pr A	
Protocol C³	
Reaction (R₃): equation (1), 286% exc. hydrochloric acid, T ~ 0 °C	
Isolation (I₃): washing (water → 5% sodium hydrogen carbonate solution → water) → drying (anhydrous magnesium sulphate) → filtration (gravity)	
Purification: not prescribed	
Protocol D⁴	
Reaction (R₄): equation (1), 236% exc. hydrochloric acid, T ~ 0 °C	
Isolation (I₄): washing (water → 5% sodium hydrogen carbonate solution → water) → drying (anhydrous calcium chloride) → decantation	
Purification (Pu₁): ≡ Pr A	
Protocol E⁵	
Reaction (R₅): equation (1), 174% exc. hydrochloric acid, room temperature	
Isolation (I₅): washing (water → 5% sodium hydrogen carbonate solution → water) → drying (anhydrous calcium chloride) → decantation	
Purification (Pu₂): simple distillation → cooling in ice	
Protocol F⁵	
Reaction (R₅): ≡ Pr E	
Isolation (I₆): washing (water → 5% sodium hydrogen carbonate solution → water) → drying (anhydrous sodium sulphate) → decantation	
Purification (Pu₂): ≡ Pr E	
Protocol G⁶	
Reaction (R₆): equation (1), 200% exc. hydrochloric acid, T ~ 5-8 °C	
Isolation (I₇): washing (cold water → cold 10% sodium hydrogen carbonate solution → cold water) → drying (anhydrous calcium chloride) → decantation, pH test: litmus	
Purification: not prescribed	
Protocol H⁷	
Reaction (R₇): equation (1), 200% exc. hydrochloric acid, room temperature	
Isolation (I₈): washing (5% sodium hydrogen carbonate solution → water) → drying (anhydrous magnesium sulphate) → decantation	
Purification (Pu₁): ≡ Pr A	
Protocol I⁸	
Reaction (R₇): ≡ Pr H (scale enlarged 4 times)	
Isolation (I₉): washing (5% sodium hydrogen carbonate solution → water) → drying (anhydrous calcium chloride) → decantation	
Purification (Pu₁): ≡ Pr A	

Protocol J⁸
<p>Reaction (R₇): ≡ Pr H (scale enlarged 4 times) Isolation (I₁₀): washing (5% sodium hydrogen carbonate solution → water) → drying (anhydrous calcium sulphate) → decantation Purification (Pu₁): ≡ Pr A</p>
Protocol K⁹
<p>Reaction (R₇): ≡ Pr H (scale decreased to half) Isolation (I₁₁): washing (saturated sodium hydrogen carbonate solution → water) → drying (anhydrous sodium sulphate) → filtration (suction) Purification (Pu₁): ≡ Pr A</p>
Protocol L¹⁰
<p>Reaction (R₇): ≡ Pr K Isolation (I₁₂): washing (5% hydrogen carbonate solution → water) → drying (anhydrous calcium chloride) → filtration (gravity) Purification: not prescribed</p>
Protocol M¹⁰
<p>Reaction (R₇): ≡ Pr K Isolation (I₁₃): washing (5% hydrogen carbonate solution → water) → drying (anhydrous sodium sulphate) → filtration (gravity) Purification: not prescribed</p>
Protocol N^{11,12}
<p>Reaction (R₇): ≡ Pr H (scale enlarged 12 times) Isolation (I₁₄): washing (5% hydrogen carbonate solution → water) → drying (anhydrous calcium chloride), pH test: litmus Purification (Pu₁): ≡ Pr A</p>
Protocol O¹³
<p>Reaction (R₈): equation (1), 220% exc. hydrochloric acid, room temperature Isolation (I₁₄): ≡ Pr N Purification (Pu₂): ≡ Pr E</p>
Protocol P¹⁴
<p>Reaction (R₉): equation (1), 253% exc. hydrochloric acid, room temperature Isolation (I₁₅): washing (water → 5% hydrogen carbonate solution) → drying (anhydrous sodium sulphate) Purification (Pu₁): ≡ Pr A</p>
Protocol Q¹⁵
<p>Reaction (R₁₀): equation (1), 246% exc. hydrochloric acid, room temperature Isolation (I₁₆): washing (saturated sodium hydrogen carbonate solution → water) → drying (anhydrous calcium chloride) → decant or pipet Purification: not prescribed</p>
Protocol R¹⁶
<p>Reaction (R₁₁): equation (1), 300% exc. hydrochloric acid, room temperature Isolation (I₁₇): washing (dilute sodium hydrogen carbonate solution) → drying (anhydrous calcium chloride) Purification (Pu₁): ≡ Pr A</p>
Protocol S¹⁷
<p>Reaction (R₁₂): equation (1), 278% exc. hydrochloric acid, T ~ 0 °C Isolation (I₁₈): washing (water → 5% sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous sodium sulphate) → decantation Purification (Pu₁): ≡ Pr A</p>
Protocol T¹⁸
<p>Reaction (R₁₃): equation (1), 210% exc. hydrochloric acid, T ~ 0 °C Isolation (I₁₉): saturation (sodium chloride) → liquid-liquid extraction → drying (anhydrous sodium sulphate) → filtration (gravity) Purification (Pu₁): ≡ Pr A</p>

Protocol U¹⁹

Reaction (R₁₄): equation (1), 500% exc. hydrochloric acid, room temperature

Isolation (I₂₀): liquid-liquid extraction → drying (anhydrous calcium chloride)

Purification (Pu₁): ≡ Pr A

^α → – Sequential

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