

Synthesis of ethyl acetate

Summary of the analysed protocols

$\text{CH}_3\text{COOH} + \text{C}_2\text{H}_5\text{OH} \rightarrow \text{CH}_3\text{COOC}_2\text{H}_5 + \text{H}_2\text{O}$	(1)
Protocol A¹	
<p>Reaction (R₁): equation (1), stoichiometric proportions of ethanol and glacial acetic acid, sulphuric acid (catalyst), reflux, $0\text{ }^\circ\text{C} \leq T < 100\text{ }^\circ\text{C}$</p> <p>Isolation (I₁): simple distillation → washing (10% sodium carbonate solution → calcium chloride solution) → drying (anhydrous calcium chloride) → filtration (gravity)</p> <p>Purification (Pu₁): simple distillation</p>	
Protocol B²	
<p>Reaction (R₁): ≡ Pr A (scale enlarged to double)</p> <p>Isolation (I₂): simple distillation → washing (30% sodium carbonate solution → calcium chloride solution) → drying (anhydrous calcium chloride) → filtration (gravity)</p> <p>Purification (Pu₁): ≡ Pr A</p>	
Protocol C²	
<p>Reaction (R₂): equation (1), stoichiometric proportions of ethanol and glacial acetic acid, sulphuric acid (catalyst), reflux, $T < 100\text{ }^\circ\text{C}$</p> <p>Isolation (I₃): simple distillation → washing (30% sodium carbonate solution → calcium chloride solution) → drying (anhydrous calcium chloride) → decantation</p> <p>Purification (Pu₁): ≡ Pr A</p>	
Protocol D³	
<p>Reaction (R₃): equation (1), 96% exc. ethanol, sulphuric acid (catalyst), simple distillation, $T \sim 150\text{ }^\circ\text{C}$</p> <p>Isolation (I₄): washing (saturated sodium carbonate solution → calcium chloride solution) → drying (anhydrous calcium chloride) → filtration (gravity)</p> <p>Purification (Pu₁): ≡ Pr A</p>	
Protocol E⁴	
<p>Reaction (R₄): equation (1), 10% exc. ethanol, sulphuric acid (catalyst), simple distillation, $T \sim 140\text{ }^\circ\text{C}$</p> <p>Isolation (I₄): ≡ Pr D</p> <p>Purification (Pu₁): ≡ Pr A</p>	
Protocol F⁵	
<p>Reaction (R₅): equation (1), 23% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, $T \sim 100\text{ }^\circ\text{C}$</p> <p>Isolation (I₅): simple distillation → washing (saturated sodium carbonate solution)</p> <p>Purification: not prescribed</p>	
Protocol G^{6,7}	
<p>Reaction (R₆): equation (1), 23% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, $T < 100\text{ }^\circ\text{C}$</p> <p>Isolation (I₆): washing (saturated sodium carbonate solution) → decantation</p> <p>Purification (Pu₁): ≡ Pr A</p>	
Protocol H⁸	
<p>Reaction (R₇): equation (1), stoichiometric proportions of ethanol and glacial acetic acid, sulphuric acid (catalyst), simple distillation, $T \sim 140\text{-}160\text{ }^\circ\text{C}$</p> <p>Isolation (I₇): washing (dilute sodium carbonate solution)</p> <p>Purification: not prescribed</p>	
Protocol I⁹	
<p>Reaction (R₈): equation (1), stoichiometric proportions of ethanol and glacial acetic acid, sulphuric acid (catalyst), reflux, $T < 100\text{ }^\circ\text{C}$</p> <p>Isolation (I₈): simple distillation</p> <p>Purification: not prescribed</p>	

Protocol J¹⁰

Reaction (R₉): equation (1), 22% exc. ethanol, hydrochloric acid (catalyst), anhydrous calcium chloride (auxiliary substance), reflux, T < 100 °C

Isolation (I₉): simple distillation → washing (saturated sodium chloride solution → saturated sodium carbonate solution → 45% calcium chloride solution) → drying (anhydrous calcium chloride)

Purification (Pu₁): ≡ Pr A

Protocol K⁴

Reaction (R₁₀): equation (1), 30% exc. ethanol, hydrogen chloride (catalyst); sulphuric acid (auxiliary substance), cooling (ice bath)

Isolation (I₁₀): washing (cold water → sodium carbonate solution) → drying (anhydrous sodium sulphate) → decantation

Purification (Pu₁): ≡ Pr A

Protocol L¹¹

Reaction (R₁₁): equation (1), 100% exc. glacial acetic acid, DTPA^b/ K10^c (catalyst), reflux, T < 100 °C

Isolation (I₁₁): filtration

Purification: not prescribed

^a → – Sequential; ^a DTPA – Dodecatungstophosphoric acid; ^b Montmorillonite K10

References

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