

Synthesis of isoamyl acetate

Summary of the analysed protocols

$\text{CH}_3\text{COOH} + \text{HOCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2 \rightarrow \text{CH}_3\text{COOCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2 + \text{H}_2\text{O}$	(1)
$\text{HOCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2 + \text{CH}_3\text{OCOCH}_2\text{CH}(\text{OCOCH}_3)\text{CH}_2\text{OCOCH}_3 \rightarrow$ $\rightarrow \text{CH}_3\text{COOCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2 + \text{HOCH}_2\text{CH}(\text{OCOCH}_3)\text{CH}_2\text{OCOCH}_3$	(2)
$\text{HOCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2 + (\text{CH}_3\text{CO})_2\text{O} \rightarrow \text{CH}_3\text{COOCH}_2\text{CH}_2\text{CH}(\text{CH}_3)_2 + \text{CH}_3\text{COOH}$	(3)
Protocol A¹	
Reaction (R₁): equation (1), 340% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C	
Isolation (I₁): cooling → washing (saturated sodium chloride solution → ethyl ether → water → 10% sodium carbonate solution) → drying (magnesium sulphate) → evaporation of the solvent	
Purification (Pu): not prescribed	
Protocol B²	
Reaction (R₂): equation (1), 166% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C	
Isolation (I₂): cooling → washing (water → saturated sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous sodium sulphate) → filtration (gravity)	
Purification (Pu): not prescribed	
Protocol C^{3,4}	
Reaction (R₃): ≡ Pr B (but a different amount of catalyst is used)	
Isolation (I₃): cooling → washing (water → 5% sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous sodium sulphate) → decantation	
Purification (Pu₁): distillation (collecting the product in an ice bath)	
Protocol D⁵	
Reaction (R₄): equation (1), 198% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C	
Isolation (I₄): cooling → washing (cold water → 5% sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous sodium sulphate) → decantation	
Purification (Pu₂): simple distillation	
Protocol E⁶	
Reaction (R₅): equation (1), 138% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C	
Isolation (I₅): cooling → washing (water → 5% sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous sodium sulphate or anhydrous magnesium sulphate) → decantation, pH test with pH indicator paper	
Purification (Pu₁): ≡ Pr C	
Protocol F⁷	
Reaction (R₆): equation (1), 153% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C	
Isolation (I₆): cooling → washing (water → 5% sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous magnesium sulphate), pH test with litmus paper	
Purification (Pu₂): ≡ Pr D	
Protocol G⁸	
Reaction (R₆): ≡ Pr F	
Isolation (I₇): cooling → washing (cold water → 5% sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous magnesium sulphate) → decantation, pH test with litmus paper	
Purification (Pu₁): ≡ Pr C	
Protocol H⁹	
Reaction (R₇): equation (1), 217% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C	
Isolation (I₈): cooling → washing (cold water → saturated sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous magnesium sulphate or anhydrous sodium sulphate) → decantation	
Purification (Pu₂): ≡ Pr D	

Protocol I¹⁰
<p>Reaction (R₈): equation (1), 183% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₉): cooling → washing (water → sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) Purification (Pu₂): ≡ Pr D</p>
Protocol J¹¹
<p>Reaction (R₉): equation (1), 100% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₀): cooling → washing (water → 5% sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) Purification (Pu₂): ≡ Pr D</p>
Protocol K¹²
<p>Reaction (R₁₀): equation (1), 129% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₁): cooling → washing (water → saturated sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) → filtration (gravity) Purification (Pu₂): ≡ Pr D</p>
Protocol L¹³
<p>Reaction (R₁₁): equation (1), 140% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₂): cooling → washing (water → 5% sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) → filtration Purification (Pu₂): ≡ Pr D</p>
Protocol M¹⁴
<p>Reaction (R₈): ≡ Pr I Isolation (I₁₂): ≡ Pr L (scale reduced to one third) Purification (Pu₂): ≡ Pr D</p>
Protocol N¹⁵
<p>Reaction (R₁₂): equation (1), 314% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₃): cooling → washing (ice → ethyl ether → iron(II) sulphate solution → saturated sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) → filtration → evaporation of the solvent Purification (Pu₂): ≡ Pr D</p>
Protocol O¹⁶
<p>Reaction (R₁₂): ≡ Pr N Isolation (I₁₄): cooling (cold water) → washing (ice → ethyl ether → iron(II) sulphate solution → sodium carbonate solution) → drying (anhydrous magnesium sulphate) → filtration (gravity) → evaporation of the solvent Purification (Pu₂): ≡ Pr D</p>
Protocol P^{17,18}
<p>Reaction (R₁₃): equation (1), 103% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₄): ≡ Pr O Purification (Pu₂): ≡ Pr D</p>
Protocol Q¹⁹
<p>Reaction (R₁₄): equation (1), 200% exc. glacial acetic acid, sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₅): washing (water → ethyl acetate → saturated sodium carbonate solution) → drying (anhydrous magnesium sulphate) → evaporation of the solvent Purification: not prescribed</p>
Protocol R²⁰
<p>Reaction (R₁₅): equation (1), 183% exc. glacial acetic acid, Amberlyst resin (catalyst), reflux, T > 100 °C Isolation (I₁₆): cooling → washing (5% sodium hydrogen carbonate solution) → drying (anhydrous sodium sulphate) → decantation Purification (Pu₂): ≡ Pr D</p>
Protocol S²⁰
<p>Reaction (R₁₆): equation (1), 183% exc. glacial acetic acid, concentrated sulphuric acid (catalyst), reflux, T > 100 °C Isolation (I₁₆): ≡ Pr R Purification (Pu₂): ≡ Pr D</p>
Protocol T²¹
<p>Reaction (R₁₇): equation (2), 1991% exc. triacetin, Amberlyst 36 resin (catalyst), T < 100 °C Isolation (I₁₇): cooling → washing (petroleum ether) → concentration of the organic phase Purification: not prescribed</p>

Protocol U²¹

Reaction (R₁₈): equation (2), 1991% exc. triacetin, Amberlyst 36 resin (catalyst), T > 100 °C

Isolation (I₁₈): cooling (ice bath) → washing (petroleum ether)

Purification: not prescribed

Protocol V²²

Reaction (R₁₉): equation (3), 27% exc. acetic anhydride, sulphuric acid and water (auxiliary substances), 0 °C < T < 100 °C

Isolation (I₁₉): cooling → washing (demi-saturated sodium chloride solution → saturated sodium hydrogen carbonate solution → saturated sodium chloride solution) → drying (anhydrous magnesium sulphate)

Purification: not prescribed

^α → – Sequential

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