

Synthesis of acetanilide

Summary of the analysed protocols

$\text{C}_6\text{H}_5\text{NH}_2 + (\text{CH}_3\text{CO})_2\text{O} \rightarrow \text{C}_6\text{H}_5\text{NHCOCH}_3 + \text{CH}_3\text{COOH}$	(1)
$\text{C}_6\text{H}_5\text{NH}_2 + \text{CH}_3\text{COOH} \rightarrow \text{C}_6\text{H}_5\text{NHCOCH}_3 + \text{H}_2\text{O}$	(2)
Protocol A¹	
Reaction (R₁): equation (1), 20% exc. acetic anhydride, water, hydrochloric acid and sodium acetate (auxiliary substances), T ~ 0 °C	
Isolation (I₁): filtration (suction) → washing (cold water)	
Purification (Pu₁): recrystallization – dissolution (hot water) → heating → filtration (suction) → cooling → filtration (suction) → washing (cold water) → drying (oven at 80 °C), activated charcoal (auxiliary substance)	
Protocol B²	
Reaction (R₂): equation (1), 22% exc. acetic anhydride, water, hydrochloric acid and sodium acetate (auxiliary substances), T ~ 0 °C	
Isolation (I₂): filtration (suction)	
Purification (Pu₂): recrystallization (hot water)	
Protocol C²	
Reaction (R₂): ≡ Pr B	
Isolation (I₂): ≡ Pr B	
Purification (Pu₃): recrystallization (water and ethanol)	
Protocol D³	
Reaction (R₃): equation (1), 45% exc. acetic anhydride, water, hydrochloric acid, activated charcoal and sodium acetate trihydrate (auxiliary substances), T ~ 0 °C	
Isolation (I₃): filtration (suction) → washing (water) → drying (desiccator)	
Purification (Pu₄): recrystallization – dissolution (hot water) → drying (air)	
Protocol E³	
Reaction (R₃): ≡ Pr D	
Isolation (I₃): ≡ Pr D	
Purification (Pu₅): recrystallization – dissolution (water-ethanol) → drying (air)	
Protocol F⁴	
Reaction (R₄): equation (1), 15% exc. acetic anhydride, water, hydrochloric acid, activated charcoal and sodium acetate trihydrate (auxiliary substances), T ~ 0 °C	
Isolation (I₄): filtration (suction) → washing (cold water) → drying (air)	
Purification (Pu₄): ≡ Pr D	
Protocol G⁴	
Reaction (R₄): ≡ Pr F	
Isolation (I₄): ≡ Pr F	
Purification (Pu₅): ≡ Pr E	
Protocol H⁵	
Reaction (R₅): equation (1), 24% exc. acetic anhydride, water, hydrochloric acid, activated charcoal and sodium acetate (auxiliary substances), 0 °C < T < 100 °C	
Isolation (I₅): filtration (suction) → washing (water) → drying (air)	
Purification (Pu₆): recrystallization (boiling water, ethanol:methanol solution)	
Protocol I⁶	
Reaction (R₅): ≡ Pr H (scale enlarged 4 times)	
Isolation (I₅): ≡ Pr H	
Purification (Pu₆): ≡ Pr H (scale enlarged 4 times)	

Protocol J⁷
Reaction (R₆): equation (1), 39% exc. acetic anhydride, water, glacial acetic acid and sodium acetate (auxiliary substances), T ~ 0 °C Isolation (I₄): ≡ Pr F Purification (Pu₇): recrystallization – dissolution (hot water) → heating → filtration (gravity) → cooling → filtration (suction) → drying (air), activated charcoal (auxiliary substance)
Protocol K⁶
Reaction (R₇): equation (1), stoichiometric proportions of aniline and acetic anhydride, water, glacial acetic acid and powdered zinc (auxiliary substances), reflux, T > 100 °C Isolation (I₄): ≡ Pr F Purification (Pu₆): ≡ Pr H (scale enlarged 4 times)
Protocol L⁸
Reaction (R₈): equation (1), stoichiometric proportions of aniline and acetic anhydride, water and glacial acetic acid (auxiliary substances), reflux, T > 100 °C Isolation (I₆): filtration (suction) → washing (water) Purification (Pu₈): recrystallization – dissolution (acetic acid-water 1:2) → filtration (suction) → washing (water) → drying (air)
Protocol M⁸
Reaction (R₉): equation (1), stoichiometric proportions of aniline and acetic anhydride, water and glacial acetic acid (auxiliary substances), reflux, T > 100 °C Isolation (I₄): ≡ Pr F Purification (Pu₉): recrystallization – dissolution (boiling water) → filtration (gravity) → cooling → filtration (suction)
Protocol N⁵
Reaction (R₁₀): equation (2), 100% exc. glacial acetic acid, water and powdered zinc (auxiliary substances), reflux, T > 100 °C Isolation (I₁): ≡ Pr A Purification (Pu₁₀): recrystallization – dissolution (boiling water, ethanol:methanol solution) → cooling → filtration (suction) → drying (air), activated charcoal (auxiliary substance)
Protocol O⁶
Reaction (R₁₁): equation (2), 99% exc. glacial acetic acid, water and powdered zinc (auxiliary substances), reflux, T > 100 °C Isolation (I₁): ≡ Pr A Purification (Pu₁₁): recrystallization – dissolution (boiling water, ethanol) → filtration (suction) → cooling → filtration (suction) → drying (air), activated charcoal (auxiliary substance)
Protocol P²
Reaction (R₁₂): equation (2), 546% exc. glacial acetic acid, water and powdered zinc (auxiliary substances), reflux, T > 100 °C Isolation (I₆): ≡ Pr L Purification: not prescribed

^a → – Sequential

References

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- (5) Vogel, A.I. *Elementary Practical Organic Chemistry*. Longmans, Green and Co: London, 1958, pp. 237-239.
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(8) Mann, F.G.; Saunders, B.C. *Practical Organic Chemistry - 4th edition*. Longmans, Green and Co: London, 1960, pp. 107.