

Synthesis of vanadyl acetylacetonate

Summary of the analysed protocols

$V_2O_5 + 2H_2SO_4 + C_2H_6O + 4(Hacac) + 2Na_2CO_3 \rightarrow 2[VO(acac)_2] + 2Na_2SO_4 + C_2H_4O + 5H_2O + 2CO_2$	(1)
$V_2O_5 + 2H_2SO_4 + C_2H_6O + 4(Hacac) + 2K_2CO_3 \rightarrow 2[VO(acac)_2] + 2K_2SO_4 + C_2H_4O + 5H_2O + 2CO_2$	(2)
$2NH_4VO_3 + 3H_2SO_4 + C_2H_6O + 4(Hacac) + 2Na_2CO_3 \rightarrow 2[VO(acac)_2] + C_2H_4O + (NH_4)_2SO_4 + 2Na_2SO_4 + 6H_2O + 2CO_2$	(3)
$VO(SO_4) + 2(Hacac) + Na_2CO_3 \rightarrow [VO(acac)_2] + Na_2SO_4 + H_2O + CO_2$	(4)
$2V_2O_5 + 9(Hacac) \rightarrow 4[VO(acac)_2] + (CH_3CO)_2CO + 5H_2O$	(5)
$CO(NH_2)_2 + H_2O \rightarrow 2NH_3 + CO_2$	(6)
$VO(SO_4) + 2(Hacac) + 2NH_3 \rightarrow [VO(acac)_2] + (NH_4)_2SO_4$	(7)
Protocol A¹	
Reaction (R₁): equation (1), 207% exc. sulphuric acid, 1768% exc. ethanol, 14% exc. acetylacetonate, 243% exc. sodium carbonate, water (solvent), reflux, 0 °C < T < 100 °C	
Isolation (I₁): filtration → washing (cold water → ethanol → ethyl ether)	
Purification (Pu₁): recrystallization – dissolution (dichloromethane and petroleum ether) → cooling (ice bath) → filtration → washing (cold petroleum ether) → drying (air)	
Protocol B²	
Reaction (R₂): equation (1), 1740% exc. sulphuric acid, 8385% exc. ethanol, 468% exc. vanadium pentoxide, 543% exc. sodium carbonate, water (solvent), reflux, 0 °C < T < 100 °C	
Isolation (I₂): filtration (suction) → washing (cold water) → drying (suction, air)	
Purification (Pu₂): recrystallization – dissolution (dichloromethane and petroleum ether) → filtration (gravity) → filtration (suction) → washing (cold petroleum ether) → drying (suction)	
Protocol C³	
Reaction (R₃): equation (1), 229% exc. sulphuric acid, 1395% exc. ethanol, 6% exc. acetylacetonate, 586% exc. sodium carbonate, water (solvent), reflux, 0 °C < T < 100 °C	
Isolation (I₃): filtration (suction) → washing (cold water) → drying (desiccator over anhydrous calcium chloride)	
Purification: not prescribed	
Protocol D⁴	
Reaction (R₄): equation (1), 207% exc. sulphuric acid, 1458% exc. ethanol, 14% exc. acetylacetonate, water (solvent), T < 100 °C	
Isolation (I₄): filtration (suction) → washing (cold water) → drying (desiccator over phosphorus pentoxide)	
Purification: not prescribed	
Protocol E⁵	
Reaction (R₅): equation (1), 207% exc. sulphuric acid, 1458% exc. ethanol, 14% exc. acetylacetonate, 243% exc. sodium carbonate, water (solvent), reflux, T < 100 °C	
Isolation (I₅): filtration → drying (air)	
Purification (Pu₃): recrystallization (chloroform)	
Protocol F⁶	
Reaction (R₅): ≡ Pr E (scale decreased to half)	
Isolation (I₅): ≡ Pr E	
Purification (Pu₄): recrystallization (hot chloroform and ethyl ether)	
Protocol G⁷	
Reaction (R₆): equation (1), 241% exc. sulphuric acid, 1458% exc. ethanol, 15% exc. acetylacetonate, water (solvent), reflux, T < 100 °C	
Isolation (I₅): ≡ Pr E	
Purification (Pu₃): ≡ Pr E	
Protocol H⁸	
Reaction (R₇): equation (1), 191% exc. sulphuric acid, 1457% exc. ethanol, 11% exc. acetylacetonate, 243% exc. sodium carbonate, water (solvent), T < 100 °C	
Isolation (I₅): ≡ Pr E	
Purification (Pu₃): ≡ Pr E	

Protocol I⁹
<p>Reaction (R₈): equation (1), 207% exc. sulphuric acid, 1457% exc. ethanol, 14% exc. acetylacetone, 243% exc. sodium carbonate, water (solvent), T ~ 100 °C</p> <p>Isolation (I₅): ≡ Pr E</p> <p>Purification (Pu₅): recrystallization – dissolution (hot chloroform and ethyl ether) → filtration → cooling → filtration → drying (air)</p>
Protocol J¹⁰
<p>Reaction (R₉): equation (1), 475% exc. sulphuric acid, 2575% exc. ethanol, 97% exc. acetylacetone, 491% exc. sodium carbonate, water (solvent), T < 100 °C</p> <p>Isolation (I₆): filtration (suction) → washing (water → ethanol) → drying (air)</p> <p>Purification: not prescribed</p>
Protocol K¹¹
<p>Reaction (R₁₀): equation (2), 337% exc. sulphuric acid, 2126% exc. ethanol, 26% exc. acetylacetone, 276% exc. potassium carbonate, water (solvent), reflux, T < 100 °C</p> <p>Isolation (I₇): filtration → washing (water) → drying (air)</p> <p>Purification (Pu₃): ≡ Pr E</p>
Protocol L¹²
<p>Reaction (R₁₁): equation (3), 157% exc. sulphuric acid, 1892% exc. ethanol, 47% exc. acetylacetone, 339% exc. sodium carbonate, water (solvent), T < 100 °C</p> <p>Isolation (I₇): ≡ Pr K</p> <p>Purification (Pu₆): recrystallization – dissolution (dichloromethane) → drying (anhydrous sodium carbonate) → filtration → rotary evaporation</p>
Protocol M¹⁰
<p>Reaction (R₁₂): equation (3), 185% exc. sulphuric acid, 1892% exc. ethanol, 47% exc. acetylacetone, 339% exc. sodium carbonate, water (solvent), T < 100 °C</p> <p>Isolation (I₆): ≡ Pr J</p> <p>Purification: not prescribed</p>
Protocol N¹³
<p>Reaction (R₁₃): equation (4), 26% exc. vanadyl sulphate hydrate, 95% exc. sodium carbonate, water (solvent), T ~ 60-70 °C</p> <p>Isolation (I₈): cooling (ice bath) → filtration (suction) → washing (cold water → cold ethanol) → drying (suction, heating on a steam bath)</p> <p>Purification: not prescribed</p>
Protocol O⁴
<p>Reaction (R₁₄): equation (4), 71% exc. vanadyl sulphate dihydrate, 0.05 M sulphuric acid solution, ethanol, nitrogen (auxiliary substances), room temperature</p> <p>Isolation (I₅): ≡ Pr E</p> <p>Purification: not prescribed</p>
Protocol P⁸
<p>Reaction (R₁₅): equation (5), 698% exc. acetylacetone, reflux, T > 100 °C</p> <p>Isolation (I₉): filtration → cooling → evaporation in a stream of air → washing (acetone → ethyl ether) → drying (oven at 110 °C)</p> <p>Purification (Pu₇): recrystallization (acetone)</p>
Protocol Q⁸
<p>Reaction (R₁₅): ≡ Pr P</p> <p>Isolation (I₉): ≡ Pr P</p> <p>Purification (Pu₈): recrystallization (acetylacetone)</p>
Protocol R¹⁴
<p>Reaction (R₁₆): equation (6) and (7), 190% exc. acetylacetone, 3230% exc. urea, water (solvent), T ~ 100 °C</p> <p>Isolation (I₁₀): washing (water) → drying (air)</p> <p>Purification (Pu₃): ≡ Pr E</p>

^α → – Sequential

References

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