Synthesis of manganese(III) acetylacetonate

Summary of the analysed protocols

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<td>4MnCl₂·4H₂O + KMnO₄ + 15(Hacac) → 5[Mn(acac)₃] + 20H₂O + 7HCl + KCl (1)</td>
<td>KMnO₄ + 4(Hacac) → [Mn(acac)₃] + 2H₂O + Kacac + O₂ (2)</td>
<td>KMnO₄ + 4MnSO₄ + 15(Hacac) → 5[Mn(acac)₃] + 4H₂O + KHSO₄ + 3H₂SO₄ (3)</td>
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Reaction (R₁): equation (1), 122% exc. acetylacetone, water and sodium acetate (auxiliary substances), T ~ 60-70 ºC
Isolation (I₁): cooling → filtration (suction) → washing (cold water) → drying (desiccator over anhydrous calcium chloride)
Purification (P₁): recrystallization – dissolution (cyclohexane, petroleum ether) → reflux (water bath) → cooling (ice bath) → filtration (suction) → washing (cold petroleum ether) → drying (air)

Reaction (R₂): ≡ Pr A (scale decreased 10 times)
Isolation (I₂): ≡ Pr A (but is done drying in the air)
Purification (P₂): recrystallization – dissolution (cyclohexane, petroleum ether) → reflux (steam bath) → cooling (ice bath) → filtration (suction) → washing (cold petroleum ether) → drying (air)

Reaction (R₃): ≡ Pr A (but 113% exc. acetylacetone is used)
Isolation (I₃): ≡ Pr A
Purification: not prescribed

Reaction (R₄): equation (1), 108% exc. acetylacetone, water and sodium acetate (auxiliary substances), T ~ 100 ºC
Isolation (I₄): ≡ Pr C
Purification (P₄): recrystallization – dissolution (toluene, petroleum ether) → reflux (steam bath) → filtration (glass funnel) → cooling (ice bath) → filtration (suction) → washing (petroleum ether) → drying (air)

Reaction (R₅): equation (1), 108% exc. acetylacetone, water and sodium acetate (auxiliary substances), T ~ 100 ºC
Isolation (I₅): ≡ Pr C
Purification (P₅): recrystallization – dissolution (hot toluene, petroleum ether) → filtration (glass funnel) → cooling (slowly)

Reaction (R₆): ≡ Pr F (but 117% exc. acetylacetone is used)
Isolation (I₆): cooling → filtration (suction) → washing (water) → drying (oven at 60-70 ºC)
Purification (P₆): recrystallization – dissolution (benzene, petroleum ether) → filtration (glass funnel) → cooling (ice bath) → filtration (suction) → drying (oven at 60 ºC)

Reaction (R₇): equation (2), 73,5% exc. acetylacetone, water (auxiliary substance), T ~ 100 ºC
Isolation (I₇): filtration (suction) → washing (acetylacetone-water 1:1) → drying (suction)
Purification (P₇): recrystallization – dissolution (hot benzene, hot petroleum ether) → cooling (ice bath) → filtration (suction) → drying (air)

Reaction (R₈): equation (3), slight exc. acetylacetone, sodium acetate (auxiliary substance), room temperature
Isolation (I₈): washing (acetylacetone, acetone and ethyl ether)
Purification (P₈): recrystallization (hot acetone)

* → Sequential
References

(9) Cartledge, G.H. Equilibrium Between the Complexes of Tervalent Manganese with 2,4-Pentanediol. J. Am. Chem. Soc., 1951, 73 (9), 4416-4419.