Synthesis of manganese(III) acetylacetonate

Summary of the analysed protocols

$4MnCl_2.4H_2O + KMnO_4 + 15(Hacac) \rightarrow 5[Mn(acac)_3] + 20H_2O + 7HCl + KCl$	(1)
$KMnO_4 + 4(Hacac) \rightarrow [Mn(acac)_3] + 2H_2O + Kacac + O_2$	(2)
$KMnO_4 + 4MnSO_4 + 15(Hacac) \rightarrow 5[Mn(acac)_3] + 4H_2O + KHSO_4 + 3H_2SO_4$	(3)

Protocol A[1]

Reaction (R_1): equation (1), 122% exc. acetylacetone, water and sodium acetate (auxiliary substances), T ~ 60-70 ºC
Isolation (I_1): cooling → filtration (suction) → washing (cold water) → drying (desiccator over anhydrous calcium chloride)
Purification (Pu_1): recrystallization – dissolution (cyclohexane, petroleum ether) → reflux (water bath) → cooling (ice bath) → filtration (suction) → washing (cold petroleum ether) → drying (air)

Protocol B[2]

Reaction (R_1): ≡ Pr A (scale decreased 10 times)
Isolation (I_2): ≡ Pr A (but is done drying in the air)
Purification (Pu_2): recrystallization – dissolution (cyclohexane, petroleum ether) → reflux (steam bath) → cooling (ice bath) → filtration (suction) → washing (cold petroleum ether) → drying (air)

Protocol C[3]

Reaction (R_2): ≡ Pr A (but 113% exc. acetylacetone is used)
Isolation (I_3): cooling → filtration (suction) → washing (water) → drying (air)
Purification (Pu_3): recrystallization – dissolution (cyclohexane, petroleum ether) → reflux (steam bath) → cooling (slowly) → filtration (suction) → drying (air)

Protocol D[4]

Reaction (R_3): ≡ Pr A (but 104% exc. acetylacetone is used)
Isolation (I_1): ≡ Pr A
Purification: not prescribed

Protocol E[5]

Reaction (R_4): equation (1), 108% exc. acetylacetone, water and sodium acetate (auxiliary substances), T ~ 100 ºC
Isolation (I_3): ≡ Pr C
Purification (Pu_4): recrystallization – dissolution (toluene, petroleum ether) → reflux (steam bath) → filtration (glass funnel) → cooling (ice bath) → filtration (suction) → washing (petroleum ether) → drying (air)

Protocol F[6]

Reaction (R_5): equation (1), 108% exc. acetylacetone, water and sodium acetate (auxiliary substances), T ~ 100 ºC
Isolation (I_3): ≡ Pr C
Purification (Pu_5): recrystallization – dissolution (hot toluene, petroleum ether) → filtration (glass funnel) → cooling (slowly)

Protocol G[7]

Reaction (R_6): ≡ Pr F (but 117% exc. acetylacetone is used)
Isolation (I_4): cooling → filtration (suction) → washing (water) → drying (oven at 60-70 ºC)
Purification (Pu_6): recrystallization – dissolution (benzene, petroleum ether) → filtration (glass funnel) → cooling (ice bath) → filtration (suction) → drying (oven at 60 ºC)

Protocol H[8]

Reaction (R_7): equation (2), 73,5% exc. acetylacetone, water (auxiliary substance), T ~ 100 ºC
Isolation (I_5): filtration (suction) → washing (acetylacetone-water 1:1) → drying (suction)
Purification (Pu_7): recrystallization – dissolution (hot benzene, hot petroleum ether) → cooling (ice bath) → filtration (suction) → drying (air)

Protocol I[9]

Reaction (R_8): equation (3), slight exc. acetylacetone, sodium acetate (auxiliary substance), room temperature
Isolation (I_6): washing (acetylacetone, acetone and ethyl ether)
Purification (Pu_8): recrystallization (hot acetone)

α → – Sequential

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