Synthesis of copper(II) acetylacetonate – Protocol A

$$CuCl_2 + 2(Hacac) \rightarrow [Cu(acac)_2] + 2H^+ + 2Cl^-$$

Reaction. In a 250 mL beaker, dissolve 4 g (25 mmol) of copper(II) chloride dihydrate in 25 mL of water and add dropwise, with stirring, a solution of 5 mL (50 mmol) of acetylacetone (stoichiometric proportions) in 10 mL of methanol. To the resulting mixture, add a solution of 6.8 g (83 mmol) of sodium acetate dissolved in 15 mL of water over a period of 5 minutes. Heat to about 80 ºC for a period of 15 minutes, maintaining rapid stirring and monitor the temperature.

Isolation. Cool the solution to room temperature and then in an ice-water bath. Filter off the gray solid, wash with cold distilled water and dry in an oven at 110 ºC.

Purification. Place a small amount of solid, 300 mg, in 25 mL of methanol and reflux for 5 minutes. Decant the blue solution. Cool the solution to room temperature and then in an ice bath until crystals appear. Filter off the product, wash with cold methanol and dry.

Safety. See hazards associated with the reagents in Table 1.

Greenness Assessment. The evaluation was performed using the Green Star (GS) and the results are shown in Figure 1.

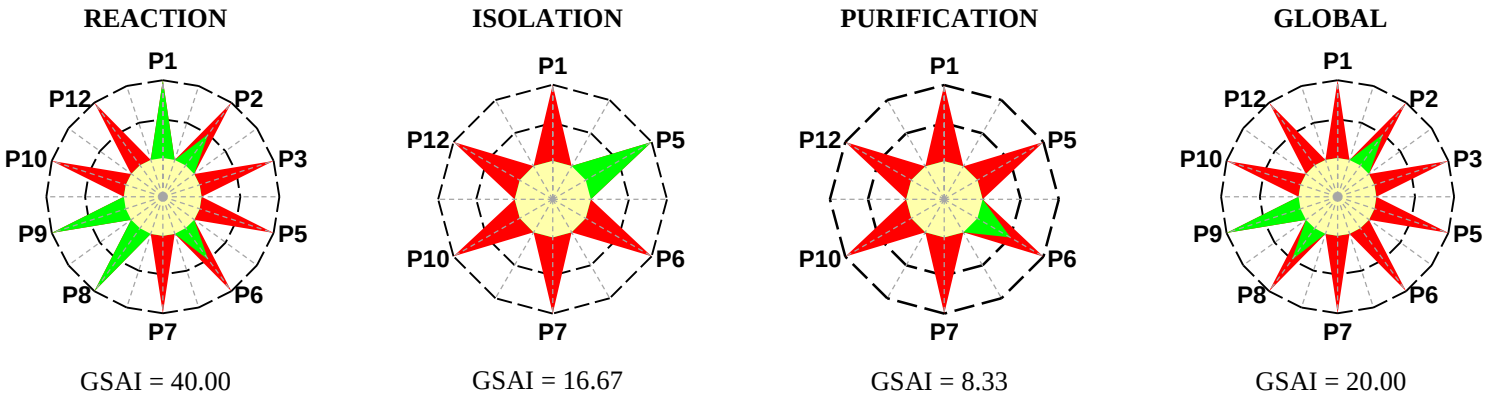

Figure 1. Greenness assessment (GS) for the synthesis of copper(II) acetylacetonate

Construction of the GS

$$CuCl_2 + 2(Hacac) \rightarrow [Cu(acac)_2] + 2H^+ + 2Cl^-$$

Table 1 presents the hazards and scores associated with the substances involved and Table 2 presents the scores used to construct the green stars.

Table 1. Hazards for the synthesis of copper(II) acetylacetonate, protocol A[α]

Substances involved	Step			Hazard code	Score: hazards to...		
	R	I	Pu		HH	E	P
Stoichiometric reagents							
Acetylacetone[c] (CAS 123-54-6)	✓			H226, H302	2	1	2
Copper(II) chloride dihydrate (CAS 10125-13-0)	✓			H302, H315, H319, H335, H400	2	3	1
Auxiliary substances							
Solvents							
Methanol (CAS 67-56-1)	✓		✓	H225, H301, H311, H331, H370	3	1	3
Water[a,b]	✓	✓		-	1	1	1
Other auxiliary substances							
Sodium acetate (CAS 127-09-3)	✓			-	1	1	1
Product							
Copper(II) acetylacetonate (13395-16-9)	✓	✓	✓	H315, H319, H335	2	1	1
Waste							
Acetic acid (dilute solution)		✓		-	1	1	1
Hydrochloric acid (dilute solution)		✓		-	1	1	1
Methanol		✓	✓	H225, H301, H311, H331, H370	3	1	3
Sodium acetate (aqueous solution)		✓		-	1	1	1
Water[a,b]		✓		-	1	1	1

[α] R – Reaction; I – Isolation; Pu – Purification; HH – Human Health; E – Environment; P – Physical

[a] Renewable; [b] Degradable to innocuous products; [c] Degradable

Table 2. Scores used to construct the green star for the synthesis of copper(II) acetylacetonate, protocol A[α]

Green Chemistry Principle	Reaction		Isolation		Purification		Global	
	s	**Explanation**	**s**	**Explanation**	**s**	**Explanation**	**s**	**Explanation**
P1 Prevention	3	Without waste	1	Methanol, H301, H311, H331, H370	1	Methanol, H301, H311, H331, H370	1	Methanol, H301, H311, H331, H370
P2 Atom Economy	2	Stoichiometric proportions and formation of by-products		NA		NA	2	Stoichiometric proportions and formation of by-products
P3 Less hazardous chemical synthesis	1	Copper(II) chloride dihydrate, H400 and methanol, H301, H311, H331, H370		NA		NA	1	Copper(II) chloride dihydrate, H400 and methanol, H301, H311, H331, H370
P5 Safer solvents and auxiliary substances	1	Methanol, H301, H311, H331, H370	3	Water	1	Methanol, H301, H311, H331, H370	1	Methanol, H301, H311, H331, H370
P6 Increase energy efficiency	2	0 ºC ≤ T ≤ 100 ºC	1	T > 100 ºC	2	0 ºC ≤ T ≤ 100 ºC	1	T > 100 ºC
P7 Use renewable feedstocks	1	Substances not renewable	1	Substances not renewable	1	Substances not renewable	1	Substances not renewable
P8 Reduce derivatives	3	One stage		NA		NA	2	Two stages
P9 Catalysts	3	Without catalysts		NA		NA	3	Without catalysts
P10 Design for degradation	1	Substances not degradable	1	Substances not degradable	1	Substances not degradable	1	Substances not degradable
P12 Safer chemistry for accident prevention	1	Methanol, H225	1	Methanol, H225	1	Methanol, H225	1	Methanol, H225

[α]s – Score; NA – Not applicable

References

Faculdade de Ciências e Tecnologia da Universidade de Coimbra, *https://woc.**uc**.pt/**quimica**/getFile.do?tipo=2&id=1438* (accessed February 2011).