Synthesis of hexaamminecobalt(III) chloride – Protocol B

$$2CoCl_2 + 10NH_3 + 2NH_4Cl + H_2O_2 \rightarrow 2[Co(NH_3)_6]Cl_3 + 2H_2O$$

Reaction. Dissolve 2.5 g (10.5 mmol) of cobalt(II) chloride hexahydrate and 1.7 g (32 mmol) of ammonium chloride (about 195% excess) in 15 mL of distilled water. Add 0.5 g of activated charcoal and 23 mL (380 mmol) of concentrated aqueous ammonia (about 624% excess). Cool the reaction mixture to 0 ºC in an ice bath. Slowly add 2 mL (19.5 mmol) of 30% hydrogen peroxide (about 271% excess), with stirring – maintain the temperature below 10 ºC. After the hydrogen peroxide has been added, heat the reaction mixture to 60 ºC for about 30 minutes. Cool the reaction mixture to 0 ºC to cause precipitation of the product.

Isolation. Suction filter the mixture of charcoal and hexaamminecobalt(III) chloride.

Purification. Place the mixture of solids in a 250 mL Erlenmeyer flask, then add 20 mL of hot water and 0.5 mL of concentrated hydrochloric acid. Heat the mixture to 70 ºC, then gravity filter while it is still hot. Let the filtrate cool to room temperature, then place the filtrate in an ice bath and add 0.5 mL of cold concentrated hydrochloric acid. Leave the mixture to cool. Isolate the product by suction filtration, wash with 13 mL of ice-cold 95% ethanol and air dry.

Safety. Synthesis should be performed in a fume hood. 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.

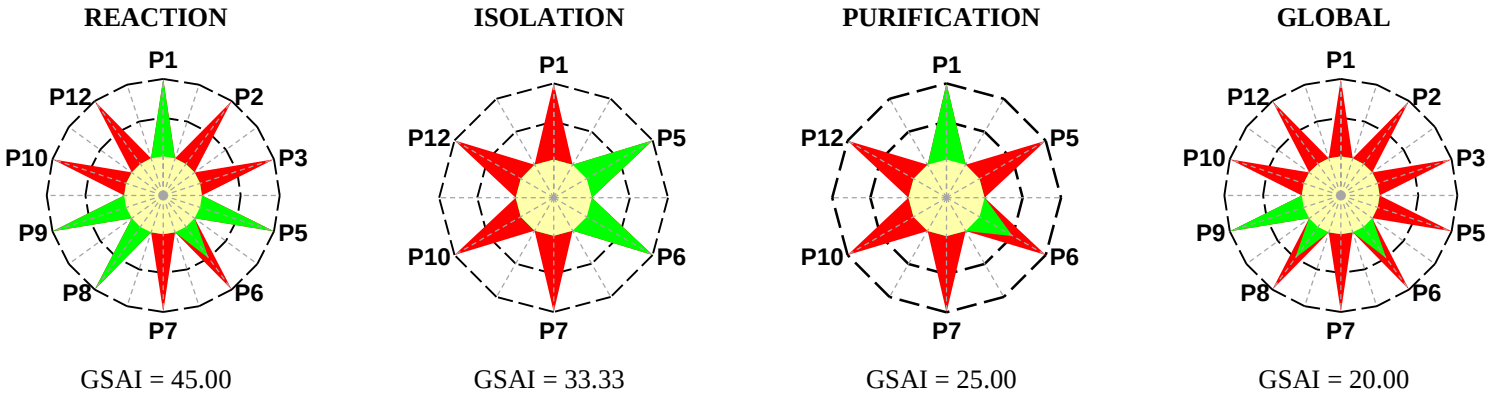

Figure 1. Greenness assessment (GS) for the synthesis of hexaamminecobalt(III) chloride

Construction of the GS

$$2CoCl_2 + 10NH_3 + 2NH_4Cl + H_2O_2 \rightarrow 2[Co(NH_3)_6]Cl_3 + 2H_2O$$

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 hexaamminecobalt(III) chloride, protocol B[α]

Substances involved	Step			Hazard code	Score: hazards to...		
	R	I	Pu		HH	E	P
Stoichiometric reagents							
30% Hydrogen peroxide (CAS 7722-84-1)	✓			H302, H318	3	1	1
Ammonia (concentrated solution)	✓			H290, H314, H335, H400	3	3	2
Ammonium chloride (CAS 12125-02-9)	✓			H302, H319	2	1	1
Cobalt(II) chloride hexahydrate (CAS 7791-13-1)	✓			H302, H317, H334, H341, H350i, H360F, H410	3	3	1
Auxiliary substances							
Solvents							
Ethanol[b] (CAS 64-17-5)			✓	H225	1	1	3
Hydrochloric acid (CAS 7647-01-0)			✓	H314, H335	3	1	1
Water[a,b]	✓		✓	-	1	1	1
Other auxiliary substances							
Activated charcoal (CAS 7440-44-0)	✓			-	1	1	1
Product							
Hexaamminecobalt(III) chloride (10534-89-1)	✓	✓	✓	H315, H319, H335	2	1	1
Waste							
Activated charcoal			✓	-	1	1	1
Ammonia (aqueous solution)		✓		H315, H318, H400	3	3	1
Ammonium chloride (excess, aqueous solution)		✓		H302, H319	2	1	1
Ethanol[b]			✓	H225	1	1	3
Hydrochloric acid (dilute solution)			✓	-	1	1	1
Hydrogen peroxide (dilute 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

Table 2. Scores used to construct the green star for the synthesis of hexaamminecobalt(III) chloride, protocol B[α]

Green Chemistry Principle	Reaction		Isolation		Purification		Global	
	s	**Explanation**	**s**	**Explanation**	**s**	**Explanation**	**s**	**Explanation**
P1 Prevention	3	Without waste	1	Excess of ammonia solution, H318 and H400	3	Waste is innocuous	1	Excess of ammonia solution, H318 and H400
P2 Atom Economy	1	Excess of reagents > 10%, formation of by-products		NA		NA	1	Excess of reagents > 10%, formation of by-products
P3 Less hazardous chemical synthesis	1	Cobalt(II) chloride hexahydrate, H334, H350, H360 and H410, ammonia solution, H314 and H400, and hydrogen peroxide, H318		NA		NA	1	Cobalt(II) chloride hexahydrate, H334, H350, H360 and H410, ammonia solution, H314 and H400, hydrogen peroxide, H318, hydrochloric acid, H314
P5 Safer solvents and auxiliary substances	3	Solvents and auxiliary substances are innocuous	3	Solvents and auxiliary substances are not used	1	Hydrochloric acid, H314	1	Hydrochloric acid, H314
P6 Increase energy efficiency	2	0 ºC ≤ T ≤ 100 ºC	3	Room temperature	2	0 ºC ≤ T ≤ 100 ºC	2	0 ºC ≤ 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	Activated charcoal is innocuous		NA		NA	3	Activated charcoal is innocuous
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	Cobalt(II) chloride hexahydrate, H334, H350, and H360, ammonia solution, H314, and hydrogen peroxide, H318	1	Excess of ammonia solution, H318	1	Hydrochloric acid, H314, and ethanol, H225	1	Cobalt(II) chloride hexahydrate, H334, H350, and H360, ammonia solution, H314, hydrogen peroxide, H318, hydrochloric acid, H314, and ethanol, H225

[α]s – Score; NA – Not applicable

References

Gustavus Adolphus College, *http://homepages.gac.edu/~bobrien/Inorganic_Lab/cold/CO-AMMIN.S01.pdf* (accessed June 2011).