Synthesis of vanadyl acetylacetonate – Protocol N $VO(SO_4) + 2(Hacac) + Na_2CO_3 \rightarrow [VO(acac)_2] + Na_2SO_4 + H_2O + CO_2$

Reaction. Prepare a solution of 1 g (9.4 mmol) of anhydrous sodium carbonate (about 95% excess) and 1 mL (9.7 mmol) of acetylacetone in about 15 mL of distilled water and put it to one side. Then prepare a second solution, containing 1 g (6.1 mmol) of vanadyl sulphate hydrate (about 26% excess) in 15 mL distilled water; warm the solution to persuade the solid to dissolve. Do not heat it to more than 60 °C. Quickly add this warm, blue solution to the sodium carbonate/ acetylacetone solution. If no precipitate appears after two minutes, warm the mixture to a maximum of 70 °C.

Isolation. Cool the solution in ice, filter with a Hirsch funnel, then wash the solid with a little icecold water followed by two 5 mL portions of ice-cold ethanol. Continue the suction until the product is as dry as possible. Dry the solid by heating it on a steam bath in a watch glass for a few minutes.

Purification. Not prescribed.

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 vanadyl acetylacetonate

Construction of the GS

$VO(SO_4) + 2(Hacac) + Na_2CO_3 \rightarrow [VO(acac)_2] + Na_2SO_4 + H_2O + CO_2$

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

Substances involved	Step			Hazard code	Score: hazards to		
Substances involved	R	Ι	Pu	inizar a couc	нн	Е	Р
Stoichiometric reagents							
Acetylacetone ^c (CAS 123-54-6)	✓			H226, H302	2	1	2
Sodium carbonate (CAS 497-19-8)	\checkmark			H319	2	1	1
Vanadyl sulphate hydrate (CAS 123334-20-3)	\checkmark			H302	2	1	1
Auxiliary substances							
Solvents							
Ethanol (CAS 64-17-5)		✓		H225	1	1	3
Water ^{a,b}	\checkmark	✓		-	1	1	1
Product							
Vanadyl acetylacetonate (3153-26-2)	\checkmark	✓		H302, H315, H319, H335	2	1	1
Waste							
Carbon dioxide	\checkmark			H280	1	1	2
Ethanol ^b		✓		H225	1	1	3
Sodium carbonate (aqueous solution)		✓		-	1	1	1
Sodium sulphate (aqueous solution)		✓		-	1	1	1
Vanadyl sulphate hydrate (excess)		✓		H302	2	1	1
Water ^{a,b}		\checkmark		-	1	1	1

Table 1. Hazards for the synthesis of vanadyl acetylacetonate, protocol N^{α}

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

^a Renewable; ^b Degradable to innocuous products; ^c Degradable

Green Chemistry Principle		Reaction	Isolation			Global		
		Explanation		Explanation	s	Explanation		
P1 Prevention	3	Carbon dioxide	2	Excess of vanadyl sulphate, H302		Excess of vanadyl sulphate, H302		
P2 Atom Economy	1	Excess of reagents > 10%, formation of by- products		NA	1	Excess of reagents > 10%, formation of by- products		
P3 Less hazardous chemical synthesis	2	Acetylacetone and vanadyl sulphate, H302, sodium carbonate, H319, vanadyl acetylacetonate, H302, H315, H319, H335		NA 2		Acetylacetone and vanadyl sulphate, H302, sodium carbonate, H319, vanadyl acetylacetonate, H302, H315, H319, H335		
P5 Safer solvents and auxiliary substances	3	Water	3	Water and ethanol	3	Water and ethanol		
P6 Increase energy efficiency	2	$0 \ ^{\circ}\text{C} \le T \le 100 \ ^{\circ}\text{C}$	2	$0 \ ^{\circ}C \le T \le 100 \ ^{\circ}C$	2	$0 \text{ °C} \le T \le 100 \text{ °C}$		
P7 Use renewable feedstocks	1	Substances not renewable		Substances not renewable	1	Substances not renewable		
P8 Reduce derivatives	3	One stage		NA	3	One stage		
P9 Catalysts	3	Without catalysts		NA	3	Without catalysts		
P10 Design for degradation	1	Substances not degradable	1	Substances not degradable	1	Substances not degradable		
P12 Safer chemistry for accident prevention	2	Vanadyl sulphate, H302, acetylacetone, H226 and H302, sodium carbonate, H319, vanadyl acetylacetonate, H302, H315, H319, H335	1	Ethanol, H225	1	Ethanol, H225		

Table 2. Scores used to construct the green star for the synthesis of vanadyl acetylacetonate, protocol N^{α}

 $^{\alpha}s$ – Score; NA – Not applicable

References

University of Bristol, *http://www.chm.bris.ac.uk/teaching-labs/1Einorganic2005-6/experiment3.pdf* (accessed June 2011).