Synthesis of vanadyl acetylacetonate

Greenness optimization

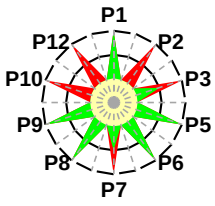
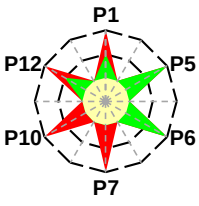
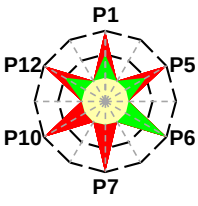
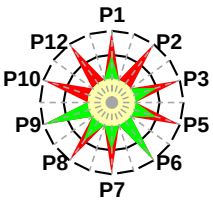
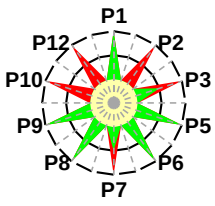
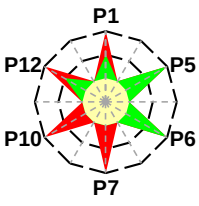
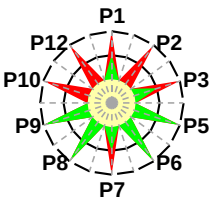
The greenest procedures for each step are: R_{14}[1] for the reaction, I_5[1-6] for the isolation, and Pu_8[5] for the purification. Combining these procedures it is possible to obtain greener procedures than those of the original protocols.

In Table 1 are presented two combinations (1 and 2). Combination 2 coincides with protocol O,[1] the greenest of the evaluated (GSAI = 55.00) and where purification is not prescribed.

Combination 1 has a more limited greenness than combination 2 (GSAI = 45.00), because a purification of the product is performed. However, it is obtained a global protocol greener than those analysed where the three steps are performed (reaction, isolation and purification).

Optimized protocols are described below.

Table 1. Green star obtained by combining the greenest procedures of each step

Combination	Reaction	Isolation	Purification	Global process
1	**R_{14}**  **GSAI = 60.00**	**I_5**  **GSAI = 50.00**	**Pu_8**  **GSAI = 41.67**	 **GSAI = 45.00**
2 = Pr O	**R_{14}**  **GSAI = 60.00**	**I_5**  **GSAI = 50.00**	Without purification	 **GSAI = 55.00**

Optimized protocol 1

Reaction. Dissolve 10.8 g (54 mmol) of vanadyl sulphate dihydrate (about 71% excess) in 80 mL of a 0.05 M sulphuric acid solution, in a nitrogen stream. Add 13 mL (63 mmol) of acetylacetone, followed by ethanol just enough for homogenization (about 4 mL). Pre-saturate a 14% sodium carbonate aqueous solution with nitrogen and add it in small increments with vigorous stirring, the pH being measured after each addition.

Isolation. Filter the precipitate and dry it in air.

Purification. Recrystallize the solid from acetylacetone.

Optimized protocol 2

Reaction. Dissolve 10.8 g (54 mmol) of vanadyl sulphate dihydrate (about 71% excess) in 80 mL of a 0.05 M sulphuric acid solution, in a nitrogen stream. Add 13 mL (63 mmol) of acetylacetone, followed by ethanol just enough for homogenization (about 4 mL). Pre-saturate a 14% sodium carbonate aqueous solution with nitrogen and add it in small increments with vigorous stirring, the pH being measured after each addition.

Isolation. Filter the precipitate and dry it in air.

Purification. Not prescribed.

References

(1) Osorio, V.K.L.; Ferreira, M. The Synthesis of Oxobis(2,4-pentanodiato)vanadium(IV) Revisited. *Química Nova.* **1991**, *14(3)*, 162-164.

(2) Adams, D.M.; Raynor, J.B. *Advanced Practical Inorganic Chemistry*. John Wiley & Sons, Ltd: London, 1965, pp. 49.

(3) Universiti Malaya, *http://www.kimia.um.edu.my/images/kimia/lab%20manual/level%203/Lab%20Manual%20Yr%203%20Inorganic.pdf* (accessed June 2011).

(4) Marr, G.; Rockett, B.W. *Practical Inorganic Chemistry*. Van Nostrand Reinhold Company: London, 1972, pp. 243.

(5) Moeller, T. *et al*. *Inorganic Synthesis – vol. V*. McGraw-Hill Book Company, Inc: New York, 1957, pp. 113-116.

(6) University of Lethbridge, *http://classes.uleth.ca/200203/chem38201/Expt7-3820.pdf* (accessed June 2011).