

2014

Experiment #3 - Dess-Martin Oxidation of Betulin Monoacetate to Betulone Acetate (2014)

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- 4) What is the role of the sodium thiosulfate solution?
- 5) Would you expect **2** to be more or less polar than **1**? Based on your experience with betulin and derivatives, what would be a good TLC solvent system?

NOTES:

- 1) This reaction has worked well with both freshly prepared Dess-Martin reagent,³ and commercially available DMP.
- 2) Attempts to adopt the calcium hypochlorite oxidation⁴ for this reaction were only partially successful, with roughly a 50% conversion after four hours.
- 3) CAUTION: Dess Martin reagent has been reported to explode upon improper handling.⁵

¹ (a) Dess, D. B.; Martin, J. C. *J. Org. Chem.* **1983**, *48*, 4155. (b) Dess, D. B.; Martin, J. C. *J. Am. Chem. Soc.* **1991**, *113*, 7277.

² Reed, N. A.; Rapp, R. D.; Hamann, C. S.; Artz, P. G. *J. Chem. Ed.* **2005**, *82*, 1053.

³ (a) Ireland, R. E.; Liu, L. *J. Org. Chem.* **1993**, *58*, 2899. Also, see: (b) Boeckman, Jr., R. K.; Shao, P.; Mullins, J. J. *Org. Syn. Coll. Vol.* **2004**, *10*, 696.

⁴ Geiger, H. C.; Donohoe, J. S. *J. Chem. Ed.* **2012**, *89*, 1572

⁵ Plumb, J. B.; Harper, D. J. *Chem. Eng. News* **1990**, *68*, 3.