

### Preparation of materials.

Approximately 2.7g of Bromine (Acros 99.6%) was weight into a 25mL round bottomed flask. This was cooled in an ice bath and 1.6 mL of 1,4-dioxaneD8 (Cambridge isotopes Limited 98% enrichment) was slowly added. An orange precipitate was formed. This was dissolved in the minimum amount of  $\text{CH}_2\text{Cl}_2$  and left overnight. This was then cooled in an acetone / $\text{CO}_2$  slush bath. The resultant precipitate was scraped out with cable tie and the flak was washed with pet ether, and left to dry. 2.68g. Melting point 60.8 - 62.2 °C (lit 61-62 °C )<sup>1</sup>

2.65g of iodine (Alfa- Aessar 99.9%) was dissolved in 2o of diethyl ether. To this 1ml of 1,4-dioxaneD8 (Cambridge isotopes Limited 98% enrichment) was added slowly. The sample was left overnight. It was then cooled in an acetone/ $\text{CO}_2$  slush bath and nitrogen gas was passed over the sample to cause evaporation of some of the solvent. The sample was filtered and dried 2.81g Melting point 77 -79°C (lit 84-85 °C )<sup>2</sup>

### Reference

1 S. K. Chaudhuri, S. Roy, and S. Bhar *Beilstein J. Org. Chem.* **2012**, 8, 323–329.

2 H Rheinboldt and R Boy *J. Prakt. Chemie* 1931, **129**, 268-271.