

Electronic Supplementary Information

A Regioselective Synthesis of the Dephospho Ditholene Protected Molybdopterin

by

Igor V. Pimkov, Antoinette Peterson, David N. Vaccarello, Partha Basu*

In situ microscale synthesis and optimization of condensation reaction of 4a with 3a. Small amount of 4a and 3a in 1 mL of DMSO-d₆, with or without Na₂SO₃, in presence of 1,4-dimethoxybenzene as an internal standard for quantitative determination of concentration. The reaction mixture was heated with stirring in water bath (100 °C) or oil bath (130, 160 °C), with periodic probing by ¹H NMR.

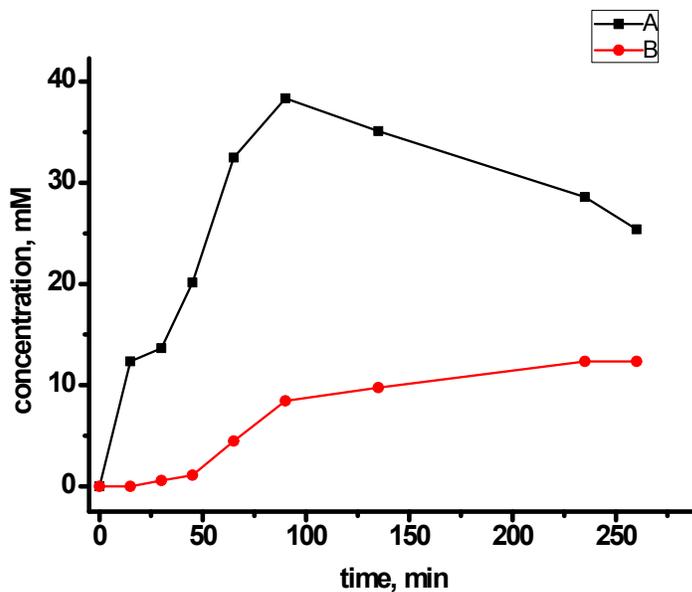


Figure S1. Change of concentrations of compound 8 (A) and compound 9 (B) during the condensation reaction (in situ microscale synthesis) of 4a with 3a in presence of Na₂SO₃ at 100 °C.

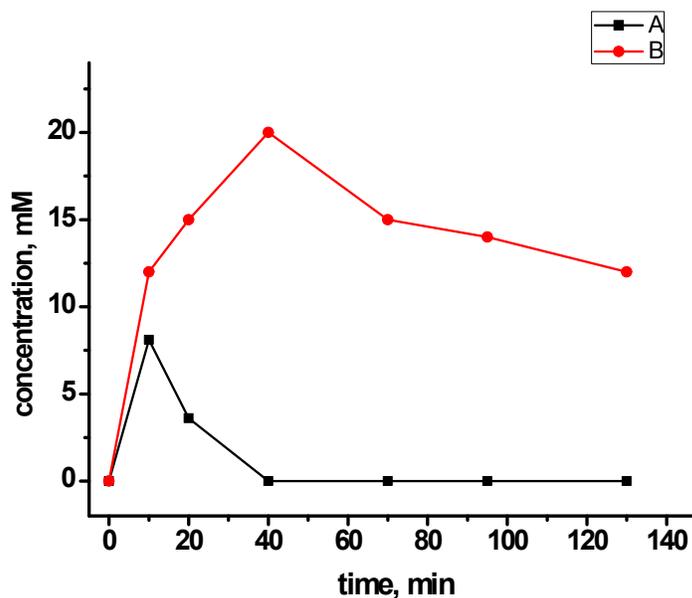


Figure S2. Change of concentrations of compound **8** (A) and compound **9** (B) during the condensation reaction (in situ microscale synthesis) of **4a** with **3a** at 130 °C.

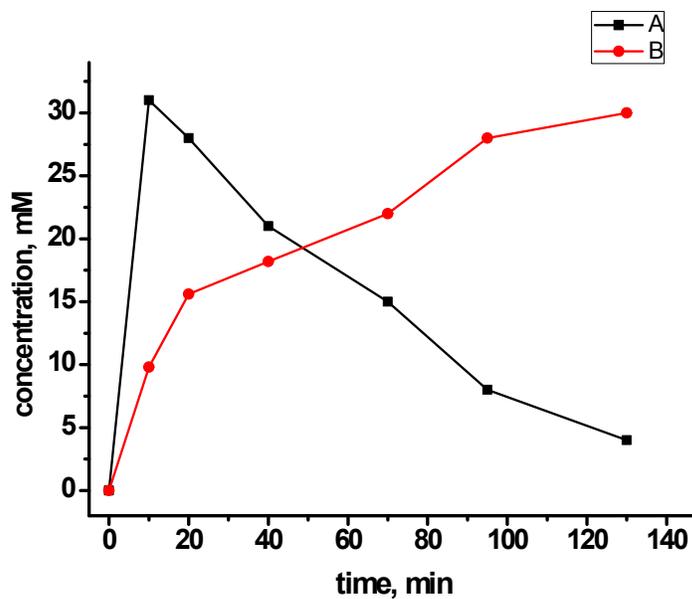


Figure S3. Change of concentrations of compound **8** (A) and compound **9** (B) during the condensation reaction (in situ microscale synthesis) of **4a** with **3a** in presence of Na_2SO_3 at 130 °C.

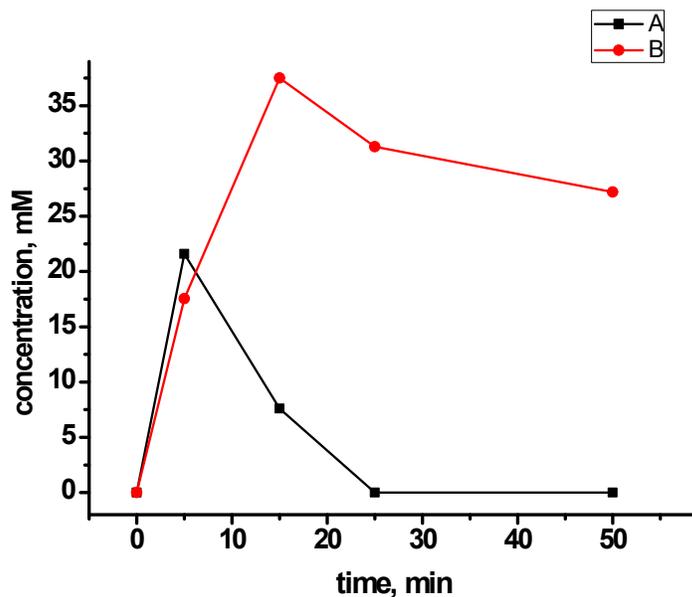


Figure S4. Change of concentrations of compound **8** (A) and compound **9** (B) during the condensation reaction (in situ microscale synthesis) of **4a** with **3a** in presence of Na_2SO_3 at 130 °C.

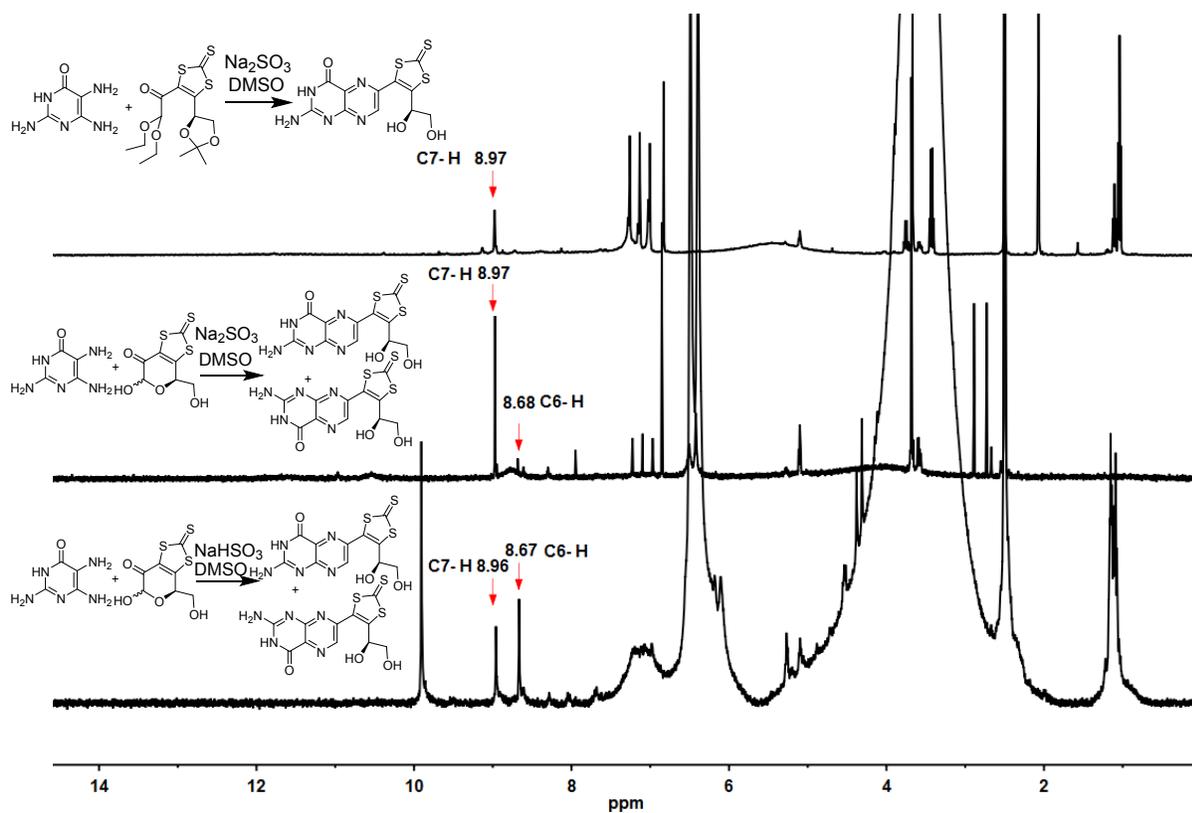


Figure S5. ¹H NMR spectra of three different reaction mixtures indicating the the formation of only 6-isomer (top), and mixture of 6- and 7-isomers in the other two. The large broad peak in the bottom spectrum is due water present in NaHSO₃.