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A Cautionary Note on the use of Diazomethane to Methylate RAFT-Synthesized Polymers†

Ming C **A Potential New RAFT-“Click” Reaction.**

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Supporting information

The thermal decomposition of the 1,3-dithiolanes. The decomposition of the 1,3-dithiolane **10** was monitored by the changes in the ^{1}H NMR spectra of **10** in d_5-chlorobenzene (Figure 2). Similar decomposition behaviour was also observed for compound **11** when it was heated in d_5-chlorobenzene.

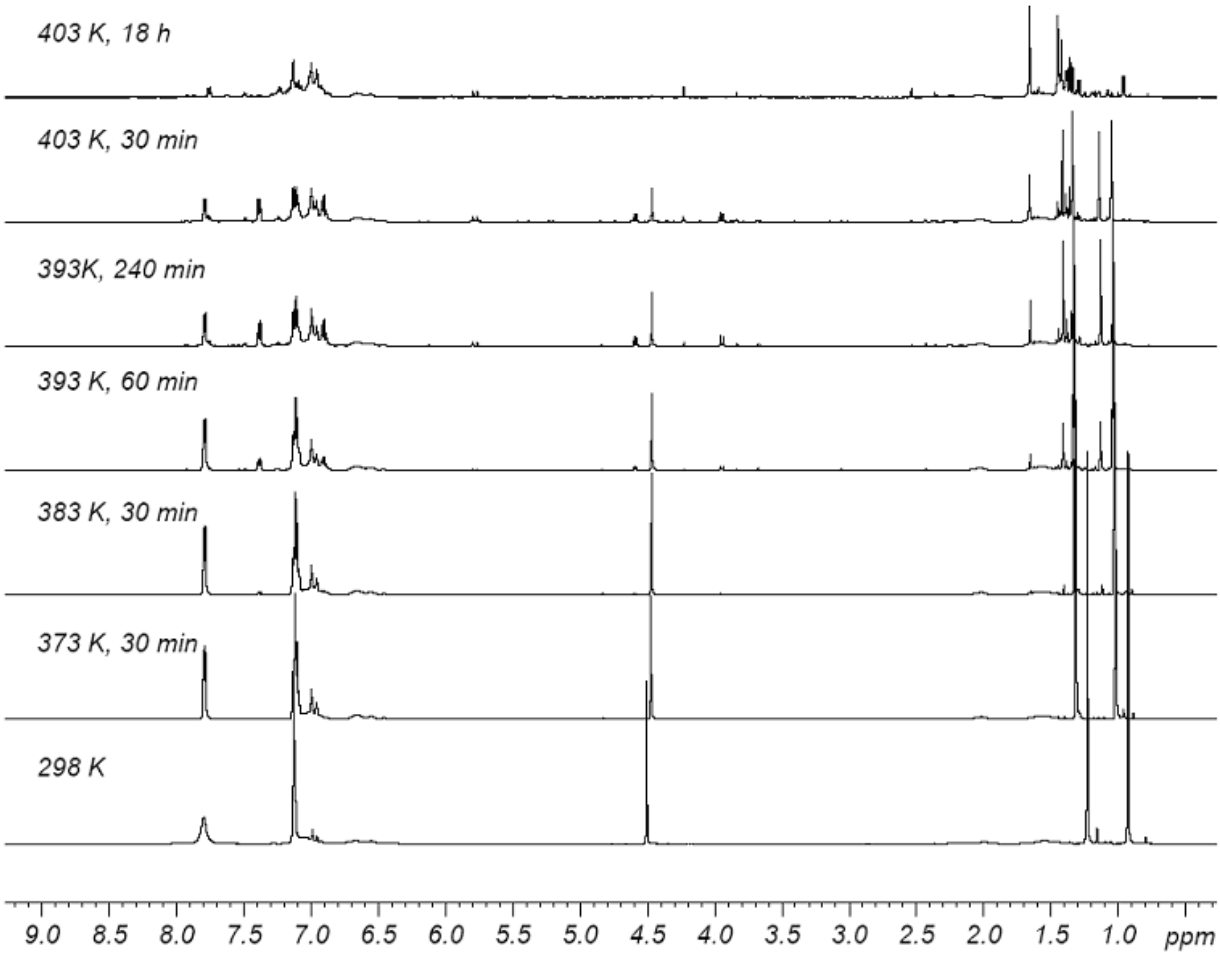

Figure 2. ^{1}H NMR spectra of compound **10** in d_5-chlorobenzene (~5 mg ml^{-1}), which was heated in the probe of the NMR spectrometer for the time and temperatures indicated.

It was observed that **10** first underwent stereo-equilibration during heating. The formation of **11** is evidenced by the ^{1}H NMR signals at δ 3.91, 4.62 and at 7.36. When the temperature reached to 393 K, the formation of dithiobenzoate **9** is indicated by the appearance of a signal at 1.65. The formation of Compound **9** was confirmed by isolation using column

chromatography on silica gel after the experiment. On keeping the solution at 403 K for 18h, the 1,3-dithiolanes were completely decomposed to **9** and other by-products.