Supporting Information

Manuscript Title:

3-D Brickwall Polymeric Structure of TMEDA-Supported 1,4-Dilithio-1,3-Butadiene

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Contents:

1) X-ray crystallographic studies

The single crystals of **5**-THF and **5**-TMEDA suitable for X-ray analysis were grown as shown in the experimental section. The crystals were manipulated in the glovebox and were sealed in thin-walled glass capillaries. Data collections for **5**-THF and **5**-TMEDA were performed at −100 °C on a RIGAKU CCD SATURN 724 diffractometer, using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The determination of crystal class and unit cell parameters was carried out by the CrystalClear (Rigaku Inc., 2007) program package. The raw frame data were processed using Crystal Structure (Rigaku/MSC 2000) to yield the reflection data file. These structures were solved by use of SHELXTL program. Refinement was performed on F^2 anisotropically for all the non-hydrogen atoms by the full-matrix least-squares method. The hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters. Crystal data, data collection and processing parameters for compounds **5**-THF and **5**-TMEDA are summarized in **Table S1**. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-939424 (**5**-THF), CCDC-939423 (**5**-TMEDA). Copies of these data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. [1]

Table S1. Crystallographic data and structure refinement details for **5**-THF and **5**-TMEDA.

	5-THF	**5**-TMEDA
Formula	$C_{40}H_{72}Li_4O_4$	$(C_{36}H_{72}Li_4N_4)_n$
Mw	644.74	$(558.72)_n$
crystal system	Monoclinic	Tetragonal
space group	C2/c	I4(1)/a
a [Å]	25.138(9)	17.806(3)
b [Å]	8.419(3)	17.806(3)
c [Å]	20.378(7)	12.312(3)
α	90	90
β [°]	97.560(6)	90
γ	90	90
V [Å^3]	4275(2)	3903.7(11)
Z	4	16
ρ_{calcd} [gcm^{-3}]	1.002	1.002
μ [mm^{-1}]	0.060	0.056
F(000)	1424	1312
θ range [°]	2.71-25.36	3.05-26.36
no of reflns collected	9884	5593
no of indep reflns	3889	1449
no of variables	219	100

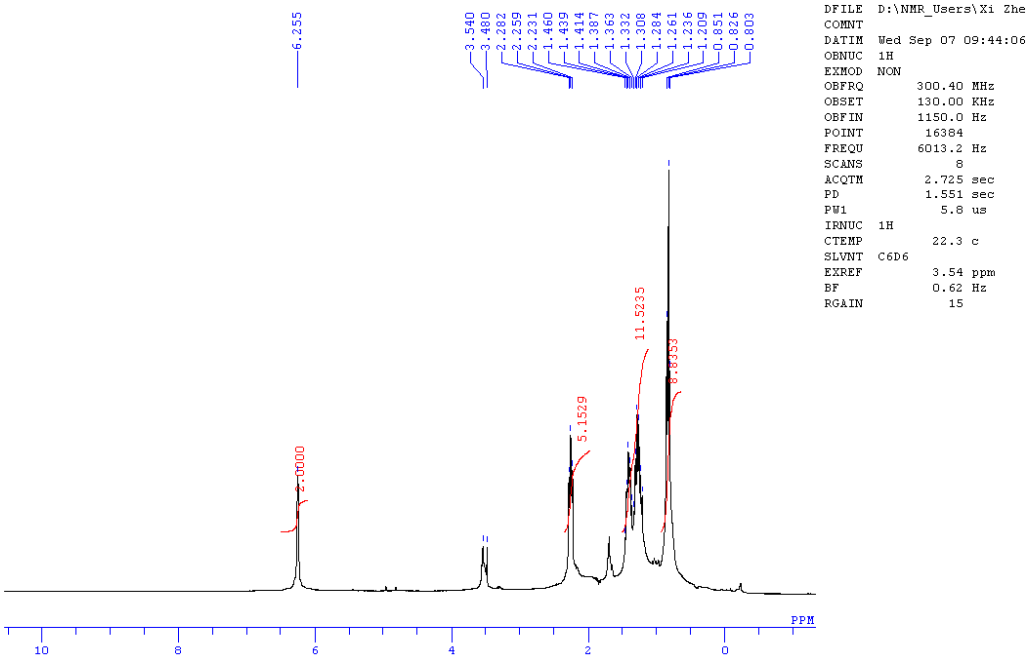
GOF	1.18	1.074
R [*I* > 2σ (*I*)]	0.09350.1168	0.05795
Rw	0.22930.2453	0.2048

References:

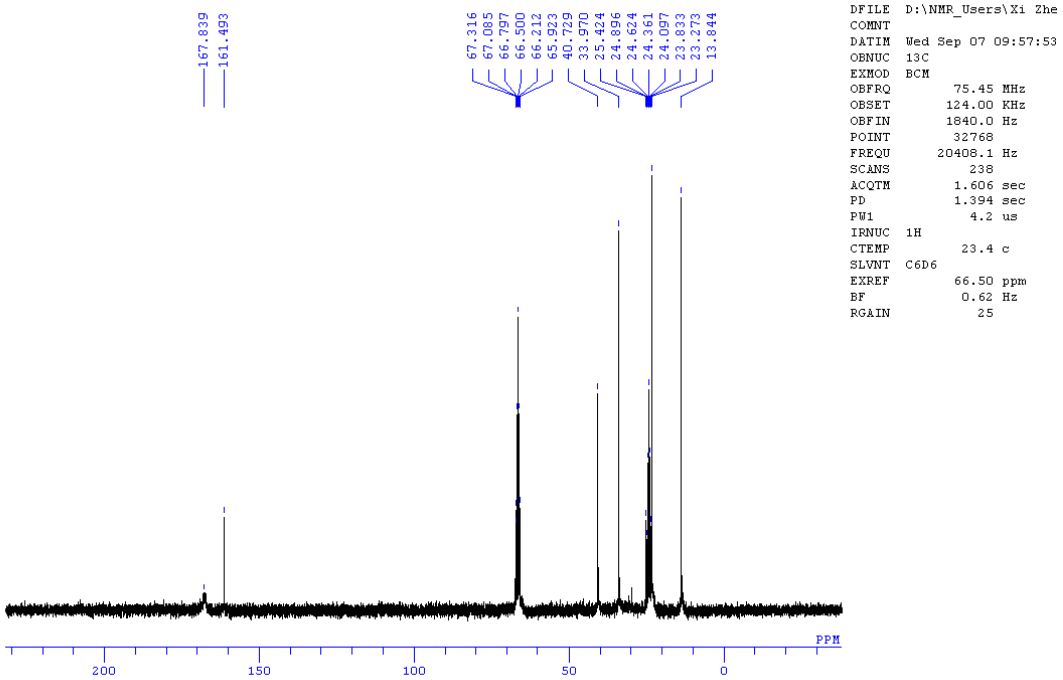
(1) G. M. Sheldrick, SHELXTL 5.10 for Windows NT: *Structure Determination Software Programs* Bruker Analytical X-ray Systems, Inc.: (Madison, WI, 1997).

2) Scanned ^{1}H NMR and ^{13}C NMR spectra of all new compounds

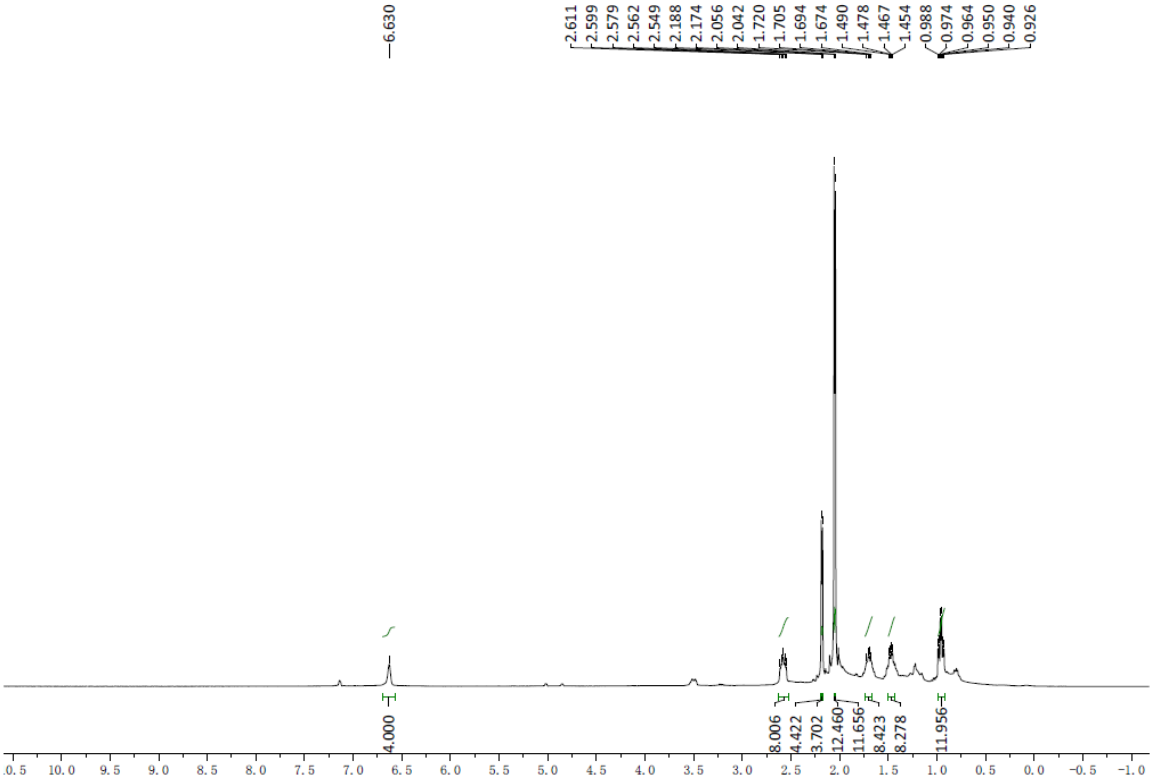
^{1}H NMR-**5**



^{13}C NMR-**5**



^{1}H NMR-**5**-TMEDA



^{13}C NMR-**5**-TMEDA