Electronic Supplementary Material (ESI) for Dalton Transactions

Supporting Information

3-Methyl-1,2-BN-Cyclopentane: A Promising H_2 Storage Material?

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Viscosity

The rheological properties of compound **1**[1] were studied using a dynamic mechanical rheometer (Rheometric Scientific: SR 2000). Liquid **1** was placed between two parallel plates having a diameter of 25 mm and a gap of 0.5 mm. The data were collected at a constant stress of 4.0 dyne cm^{-2}, a frequency of 1.0 rad s^{-1}, and a heating rate of 0.2 °C min^{-1} between the temperatures of 15-50 °C. The viscosity of compound **1** was measured at room temperature at a constant value of 25 ±5 cP. No shear behavior was observed in the time range. Temperature dependant viscosity studies in the range of 15-50 °C didn't show significant change in the viscosity which stayed within the 25-35 cP range.

Thermogravimetric Analysis / Mass Spectrometry (TGA/MS)

TGA/MS analyses were performed on a TG/DSC STA 449 Jupiter Netzsch instrument equipped with an Aelos QMS 403C MS by heating the samples under flowing argon. The MS uses a standard electron impact ionization detector. Typically, the samples were loaded in aluminium crucibles in the drybox. The samples were transported under inert atmosphere and loaded in the instrument. The data were obtained by heating the samples under argon gas from room temperature to 30 ºC, 40 ºC, 50 ºC and 60 ºC at a rate of 10 °K/min, and maintaining each of these temperatures for 3 hours.

Residual Gas Analysis (RGA)

A 300 cm^3 high pressure reactor (Parr, model 4760) was modified with an internal heater assembly. The internal electrical heater used was a 1.0 x 1.5" pyrolytic boron nitride (PBN) – coated graphite stage heater (Momentum Performance Materials, Inc. model HTR 1001) capable of near instantaneous heating to temperatures above 200 °C. The heater was suspended from the reactor body using two lengths of threaded stainless steel rod. A nickel-chromium crucible cover (Fischer # 13-812-121) was held in place against the heater stage and was used to hold and transmit heat to the sample. Heater control was

(1) Compound **1** was prepared according to literature procedures: W. Luo, P. G. Campbell, L. N. Zakharov, S. Y. Liu *J. Am. Chem. Soc.* **2011,** *133,* 19326-19329.

performed manually using a variac to supply power to the heater stage. Temperature feedback was provided to the controller using a 0.020" diameter stainless steel sheathed thermocouple inserted into a thermocouple well drilled into one end of the heater body. The thermocouple and a pair of Teflon-coated heater lead wires were brought through the reactor lid using a high-pressure feed-through fitting (Conax model MTG 24T (Cu)-4-T). For RGA analysis, a turbomolecular-pumped vacuum system containing a 1-200 amu range RGA head (Stanford Research Systems SRS 200) was connected to the interior of the reactor with a ~36" length of 100 um ID fused silica capillary tubing.

^{11}B NMR

The NMR experiments were performed on a Varian spectrometer (500 MHz for ^{1}H). The ^{11}B chemical shifts were referenced to BF_3•OEt_2 (δ=0 ppm) at 23 °C.

NMR was used to follow the thermal stability of the neat material at 40 °C and 50 °C.

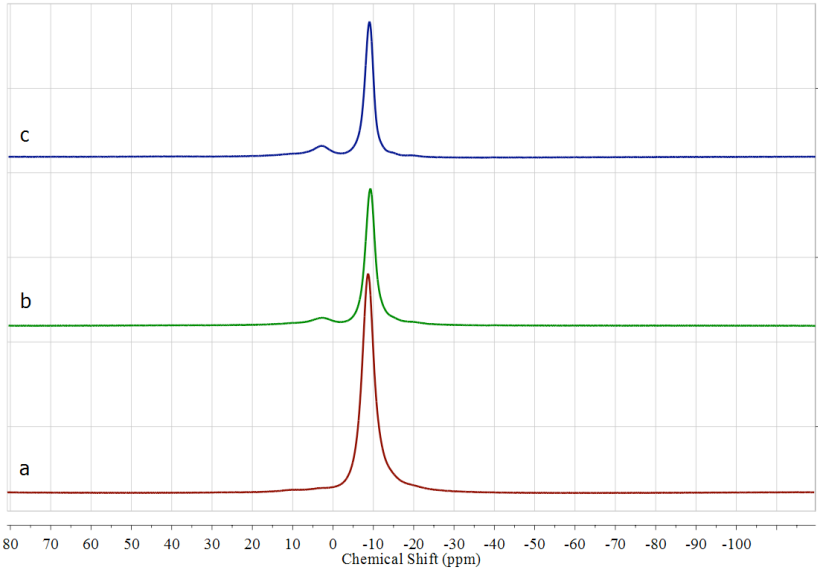

Figure S1. ^{11}B NMR data for 3-methyl-1,2-BN-cyclopentane **1**, a) 23 °C, b) 40 °C for 15 hours, c) 50 °C for 3 hours in addition to 40 °C for 15 hours.

Theoretical calculations

Molecule **1** was optimized at the density functional theory (DFT) level with the B3LYP hybrid exchange-correlation functional[2] and the DZVP2 basis set;[3] the electrostatic potential surface (ESP) was calculated at this level. Dipole moments were calculated at the MP2 level[4] with the aug-cc-cpVTZ correlation consistent basis set.[5] The calculations were done with the Gaussian09 program system.[6]

G3MP2 optimized Coordinates in angstroms for 1

B	-0.038767	1.337250	-0.032992
N	-1.563194	0.651390	0.162032
C	0.823483	0.005918	-0.393487
C	-0.001432	-1.139869	0.200052
H	0.108606	-1.153915	1.293420
C	-1.438964	-0.803822	-0.157958
H	-2.202498	-1.387250	0.366225
H	0.271665	-2.137051	-0.167377
H	-1.595804	-0.911345	-1.234448
H	-0.141952	2.173674	-0.903322
H	-2.273280	1.108136	-0.411424
H	-1.850778	0.771583	1.133684
H	0.228980	1.808413	1.054215
C	2.258580	0.023750	0.119721
H	2.799952	-0.901342	-0.114790
H	2.818248	0.855853	-0.318313
H	2.272578	0.158768	1.206297
H	0.850477	-0.147363	-1.483401

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Solvatochromism

UV-Vis spectra were recorded on an Agilent 8453 spectrometer with ChemStation software.

Experimental Procedure

A 0.01 M stock solution of Reichardt's betaine dye in acetone was prepared. 20 µL from this stock solution were transferred to a 2 mL volumetric flask, and the acetone was allowed to evaporate. 2 mL of the corresponding solvent was added to obtain a dye concentration equal to 1 x 10^{-4} M for each solvent investigated.