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Supplementary Material

A safe and simple synthesis of 1,4-bis(trimethylsilyl)buta-1,3-diyne

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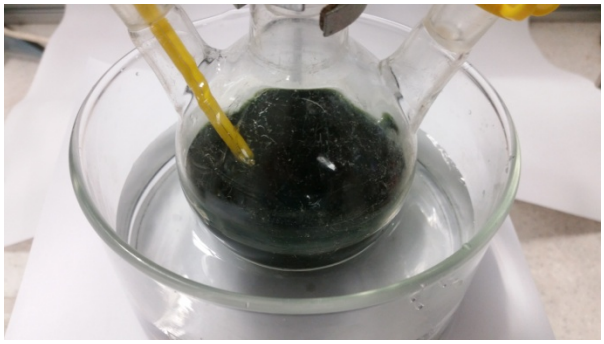


Figure S1: Green-brown colour of the catalyst solution prior to addition of trimethylsilylacetylene.

Figure S2: The reaction setup showing the three-neck flask, glass thermometer, dry-ice condenser and O_2 inlet.

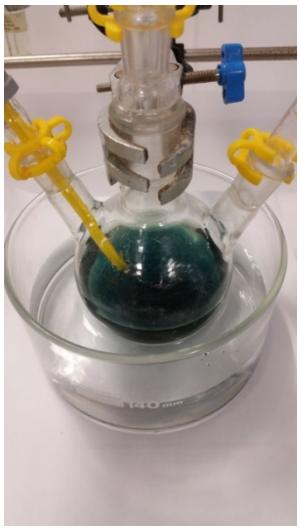


Figure S3: The blue-green colour of the reaction solution obtained 5 minutes after the addition of trimethylsilylacetylene.

Spectroscopic characterisation of 1,4-bis(trimethylsilyl)buta-1,3-diyne (**1**)

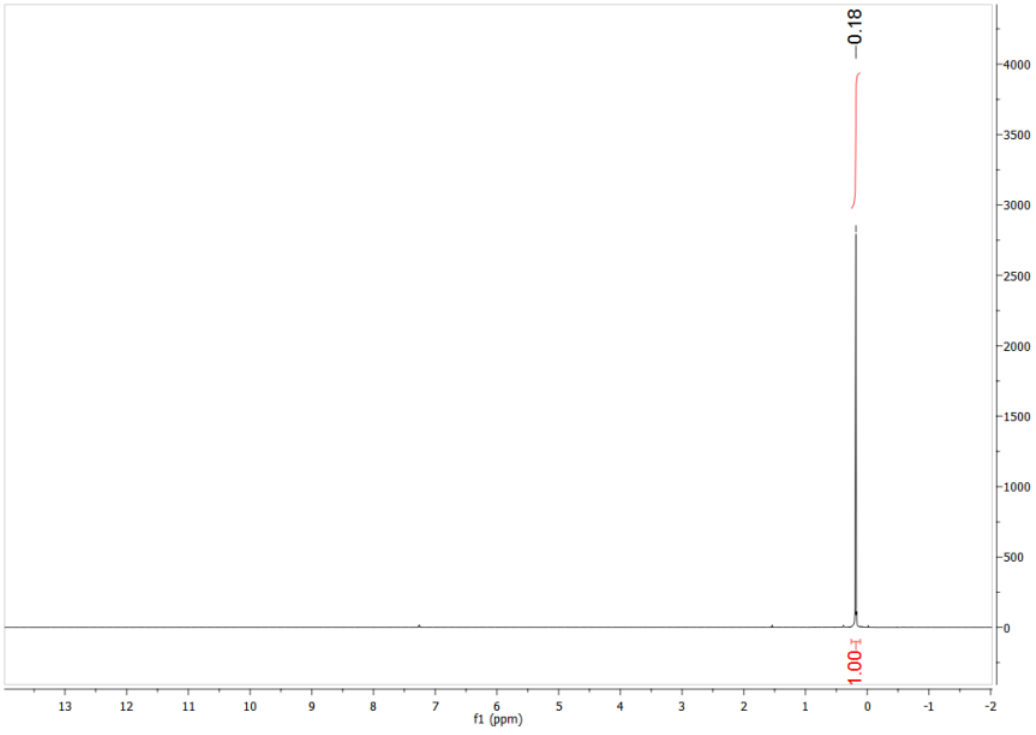


Figure S4: ^{1}H-NMR spectrum of **1** (300 MHz, $CDCl_3$)

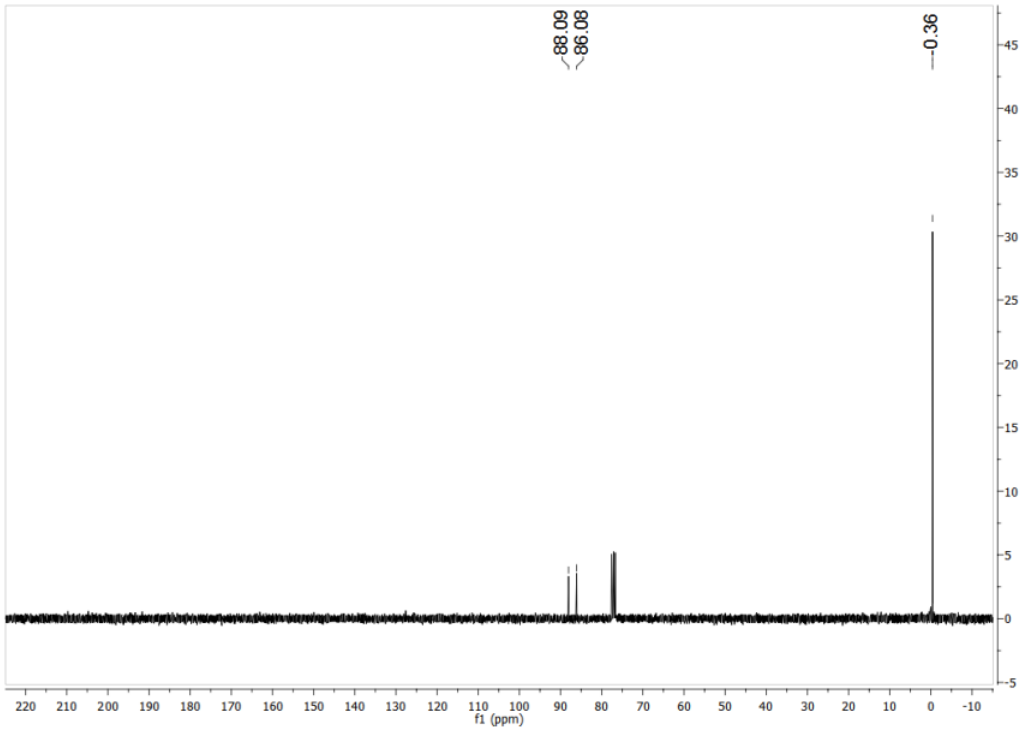


Figure S5: ^{13}C-NMR of **1** (75 MHz, $CDCl_3$)

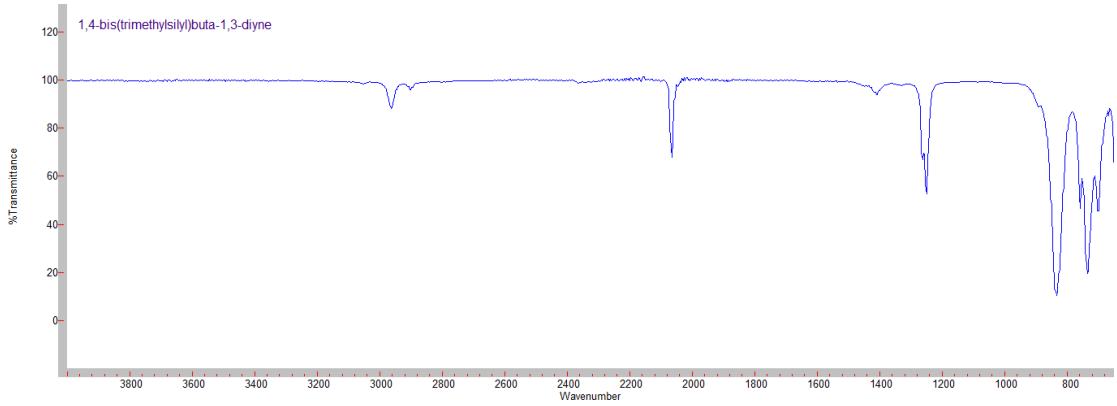


Figure S6: IR(ATR)-spectrum of **1**

Standard GC-MS analysis of **1** was performed using a GC-MS was recorded on an Agilent 6890 GC connected to an Agilent 5973 mass-selective detector (Agilent Technologies, USA) using a BPX-5 column (5% phenyl polysilphenylene–siloxane, 30 m x 0.25 mm i.d. x 0.25 µm film thickness, SGE, Australia). Helium was used as the carrier gas with a constant flow rate of 1.0 mL/min. A scan range of m/z 45–400

and a solvent delay of 5 min were used with splitless injections of 1.0 µL for 1.0 min. The ion source was set to 230°C, and the transfer line temperature to 250°C. The oven temperature program was 40°C, held for 1 min, then ramped at 10°C /min to 250°C, and held for 10 min. The chromatogram only showed one peak in the chromatogram suggesting a 100% purity based on GC. In addition the mass spectrum of the peak present provided further confirmation of the identity of the compound with the molecular ion peal for M^+ at m/z 194 and the fragment peak for $[M\text{-}CH_3]^+$ at m/z 179 (Figure S7).

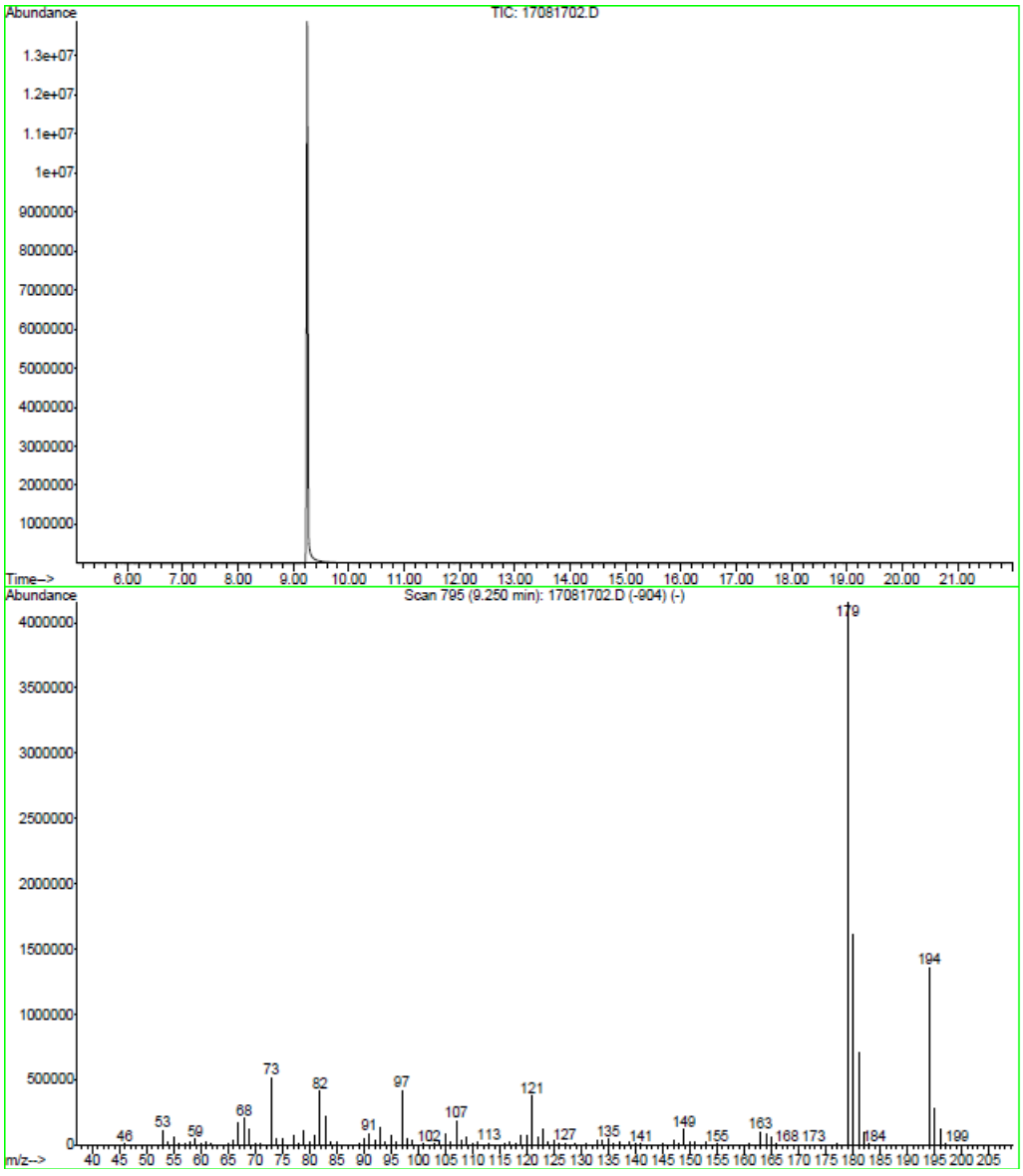


Figure S7: Gas chromatogram (top) and mass spectrum (bottom) of **1**

*Quantitative NMR measurements of **1***

1,3,5-trimethoxybenzene (99 %; Sigma-Aldrich) was used as internal standard. The purity of the sample was calculated from the following relationships.

$$molar\ ratio = \frac{\frac{I_{cpd}}{nH_{cpd}}}{\frac{I_{std}}{nH_{std}}}$$

$$wt\% = \frac{mg_{std} \times MW_{cpd} \times molar\ ratio \times P_{std}}{mg_{cpd} \times MW_{std}}$$

$$wt\% = \frac{mg_{std} \times MW_{cpd} \times I_{cpd} \times P_{std} \times nH_{std}}{mg_{cpd} \times MW_{std} \times I_{std} \times nH_{cpd}}$$

wt% … purity of the compound (**1**)

I_{cpd} … proton integral area of the compound (**1**)

I_{std} … proton integral area of the internal standard

nH_{cpd} … number of hydrogens associated with the compound (**1**) NMR resonance

nH_{std} … number of hydrogens associated with the internal standard NMR resonance

mg_{cpd} … mass of the compound (**1**) weighed out (in mg)

mg_{std} … mass of the internal standard weighed out (in mg)

MW_{cpd} … molecular weight of the compound (**1**) (194.42 g/mol)

MW_{std} … molecular weight of the internal standard (168.19 g/mol)

P_{std} … wt% purity of the internal standard (0.99)

All ^{1}H-NMR spectra were recorded on a Bruker Avance IIIHD 500MHz NMR spectrometer using a 30 degree pulse with an acquisition time of 5 seconds, 32 scans per spectrum and a relaxation delay of 30 seconds. The spectra were referenced against the protio solvent residue signal of $CDCl_3$ at 7.26 ppm. The methoxy resonance (3.60 – 3.94 ppm) of 1,3,5-trimethoxybenzene was used as the internal standard resonance representing 9 hydrogen atoms. The trimethylsilyl resonance of 1,4-bis(trimethylsilyl)buta-1,3-diyne was used as compound resonance (0.30 – 0.35 ppm) representing 18 hydrogen atoms. The purity was determined in triplicate (Table S1, Figure S8-S10). The average purity as determined from the 3 quantitative NMR measurements was 99.5%.

Run	I_{cpd}	I_{std}	nH_{cpd}	nH_{std}	MW_{cpd} / g/mol	MW_{std} / g/mol	mg_{cpd} / mg	mg_{std} / mg	P_{std}	wt%
1	18.2116	8.9942	18	9	194.42	168.19	17.54	14.96	0.99	98.82
2	18.8258	8.9913	18	9	194.42	168.19	18.47	15.34	0.99	99.50
3	11.9868	8.9859	18	9	194.42	168.19	19.64	25.8	0.99	100.27
Average										**99.53**

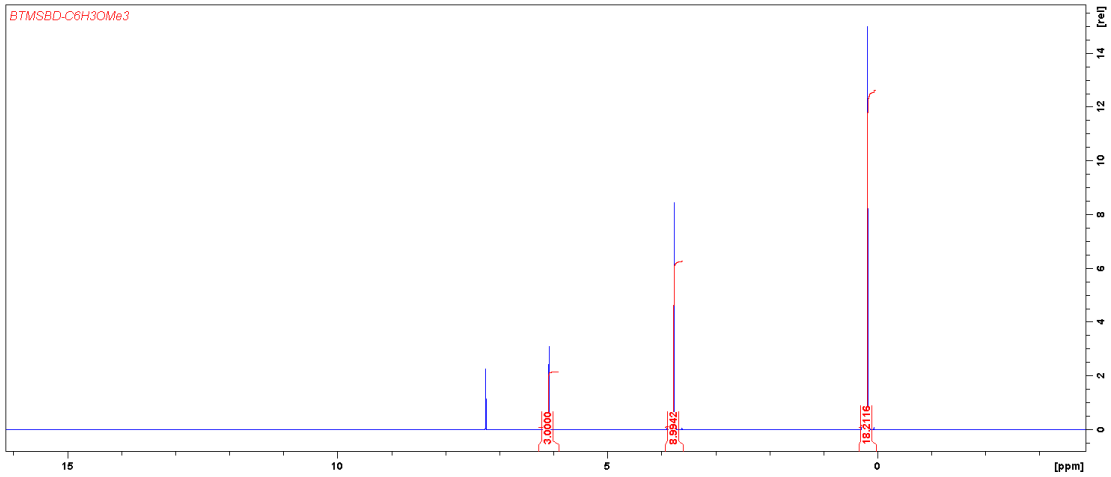


Figure S8: ^{1}H-NMR spectrum of run 1 of the quantitative NMR determination

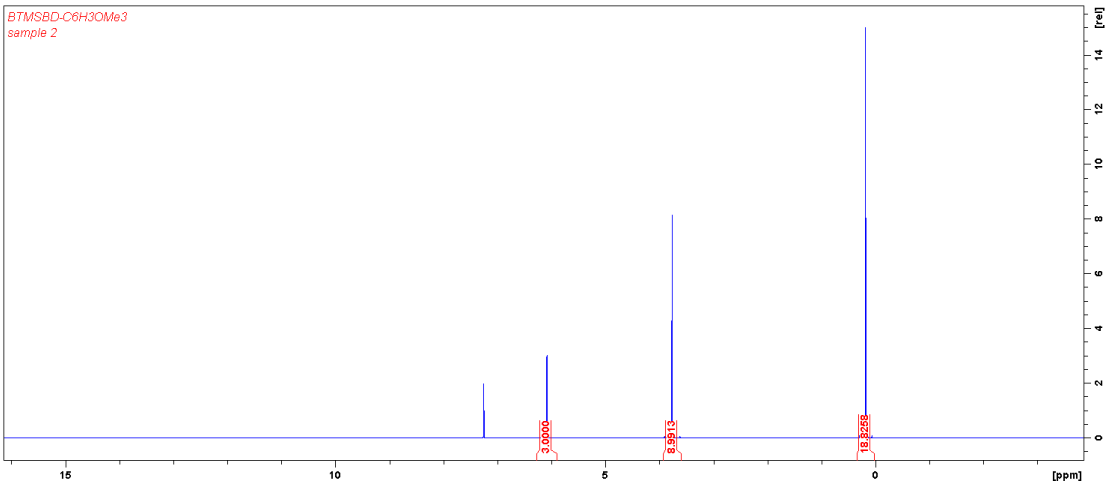


Figure S9: ^{1}H-NMR spectrum of run 2 of the quantitative NMR determination

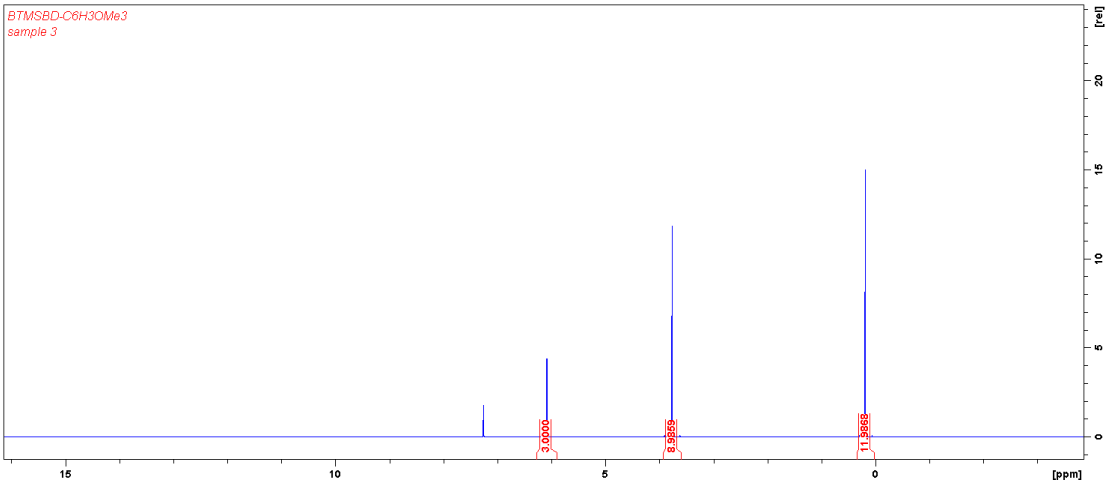
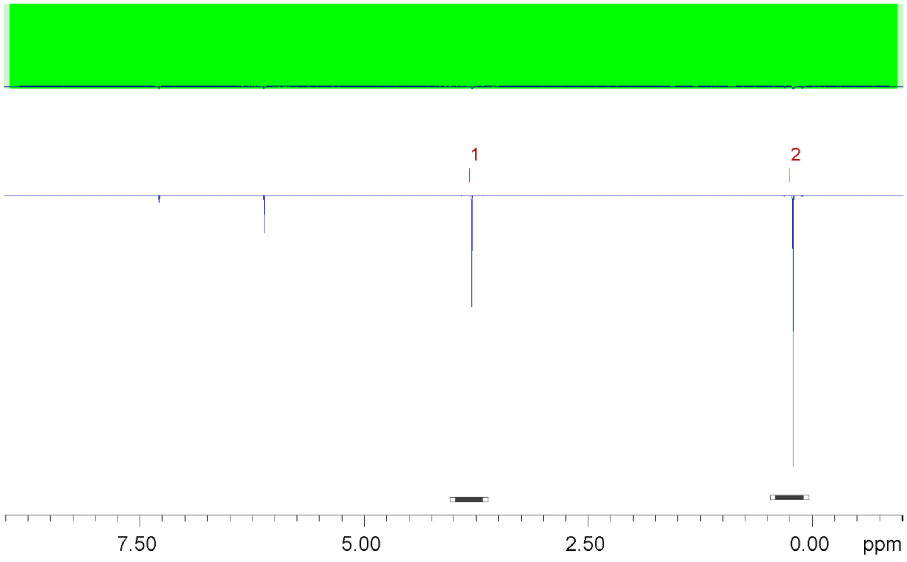


Figure S10: ^{1}H-NMR spectrum of run 3 of the quantitative NMR determination

Determination of the T1 values for the internal standard and **1** were determined via an inversion recovery sequence on a Bruker Avance IIIHD 600MHz NMR spectrometer (Figure S11). After calibration of the 90° pulse using the automated routines in Topspin 3.5, the T1 values were determined to be 2.0 s for the methoxy resonance of 1,3,5-trimethoxybenzene (as internal standard) and 3.0 s for the trimethylsilyl resonance of **1**. The relaxation time for quantitative NMR with a 90° pulse is recommended to be five times the longest T1, thus requiring a 15s delay in the current system.[1] In this case, using a 30° pulse and a relaxation delay of 30 seconds is extremely conservative and therefore fully ensures reliable quantitative results.



Fitted function:	f(t) = Io * [1 - a*exp (-t/T1)]
Random error estimation of data:	RMS per spectrum (or trace/plane)
Systematic error estimation of data:	worst case per peak scenario
Fit parameter Error estimation method:	from fit using arbitray y uncertainties
Confidence level:	95%
Used peaks:	peaks from C:/Data/Low/SB6-57-T1/31/pdata/1/peaklist1D.xml
Used integrals:	area integral

Peak name	F2 [ppm]	T1 [s]	error
1	3.824	2.03	0.002588
2	0.249	2.99	0.06334

Figure S11: T1 analysis of 1,3,5-trimethoxybenzene and **1**

[1] S. K. Bharti, R. Roy, *Trends in Analytical Chemistry* **2012**, *35*, 5-26.