## **Supporting Information**

### for

# Synthesis of functionalized macrocyclic derivatives of trioxabicyclo[3.3.0]nonadiene

Sabine Leber<sup>1,2</sup>, Gert Kollenz\*<sup>1</sup> and Curt Wentrup\*<sup>2</sup>

Address: <sup>1</sup>Institute of Chemistry, Karl-Franzens University of Graz, Heinrichstrasse 28, A-8010 Graz, Austria and <sup>2</sup>School of Chemistry and Molecular Biosciences, The University of Queensland, Brisbane, QLD 4072, Australia

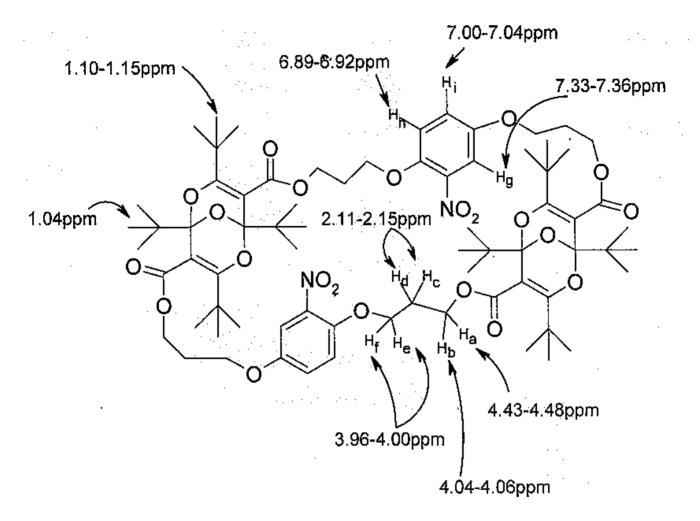
 $Email: Gert\ Kollenz\ -\ gert.kollenz\ @uni-graz.at; Curt\ Wentrup\ -\ wentrup\ @uq.edu.au$ 

## Assignment of <sup>1</sup>H NMR spectra and copies of <sup>13</sup>C NMR spectra of 8, 11, and 13.

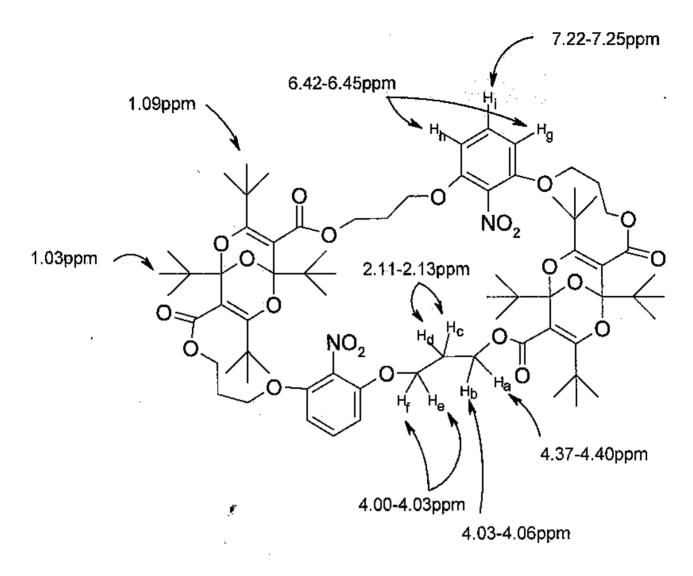
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<sup>\*</sup> Corresponding author



**Figure S2:** Assignment of <sup>1</sup>H NMR signals for compound **8**.



**Figure S3:** Assignment of <sup>1</sup>H NMR signals for compound **11**.

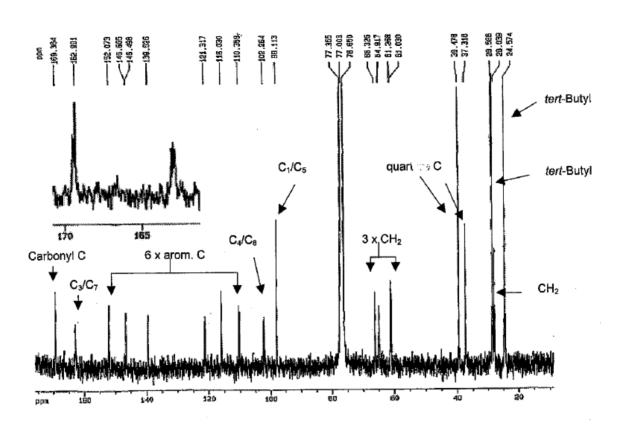


Figure S4: <sup>13</sup>C NMR spectrum of compound 8. C1/C5, C3/C7 and C4/C8 refer to the bisdioxine unit.

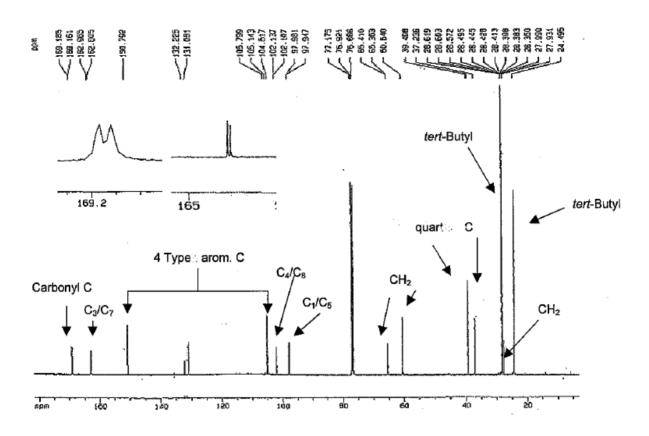
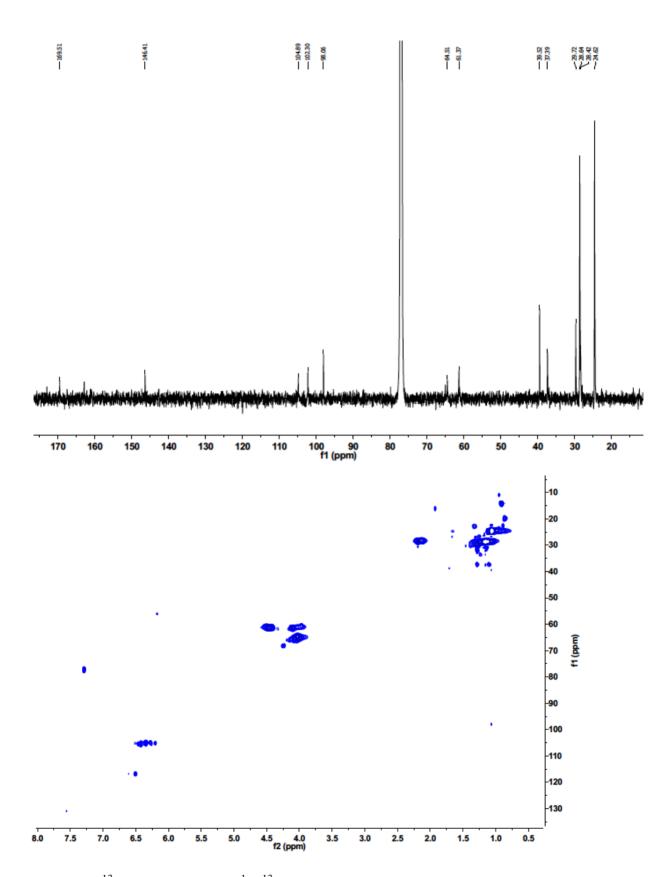


Figure S5: <sup>13</sup>C NMR spectrum of compound 11. C1/C5, C3/C7 and C4/C8 refer to the bisdioxine unit.



**Figure S6:** <sup>13</sup>C NMR and HSQC <sup>1</sup>H/<sup>13</sup>C NMR correlation for compound **13**.