

## Supporting Information

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### One-step synthesis of a singly bridged biscalicx[6]arene and evaluation of its alkali metal recognition properties

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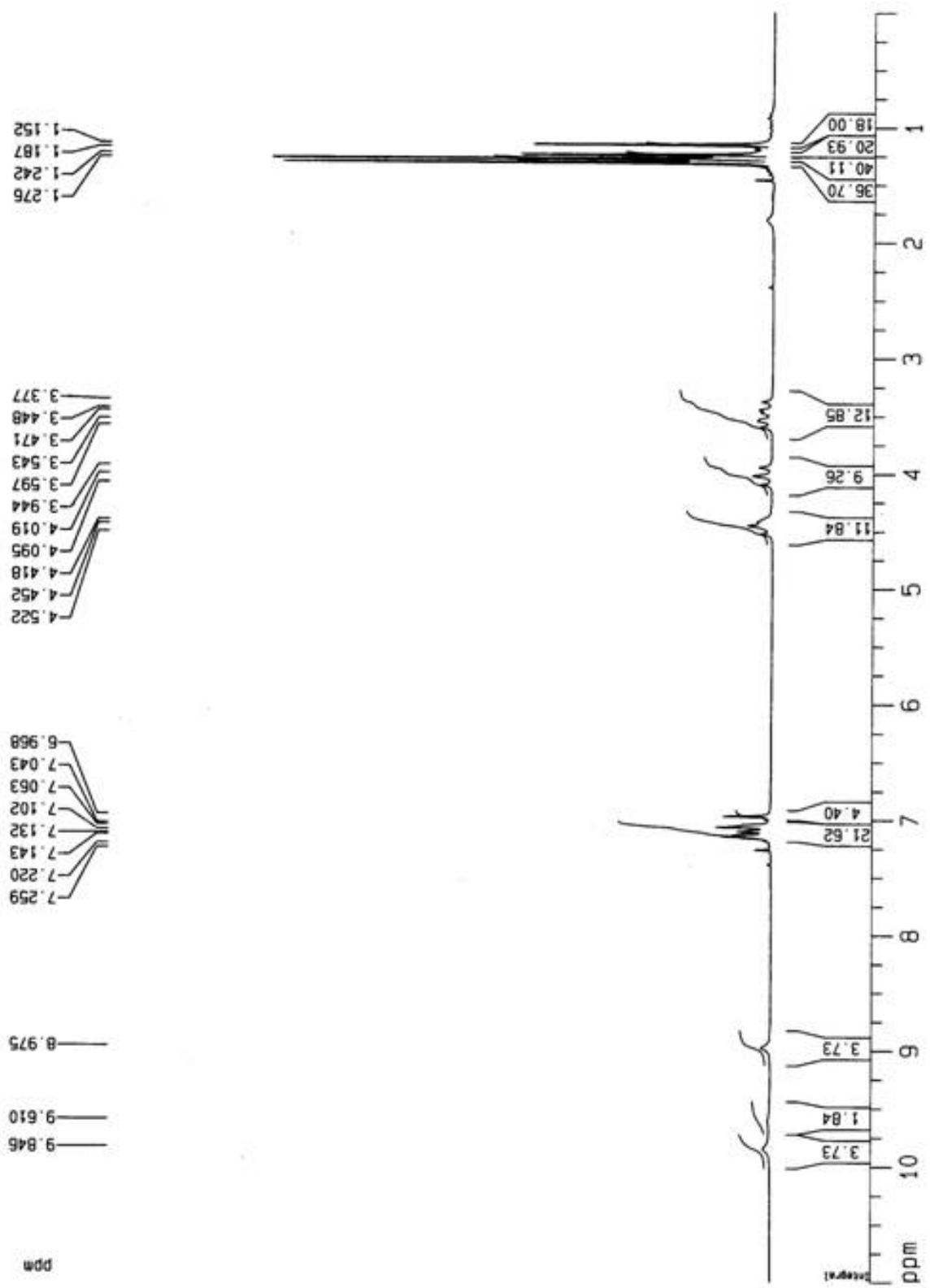
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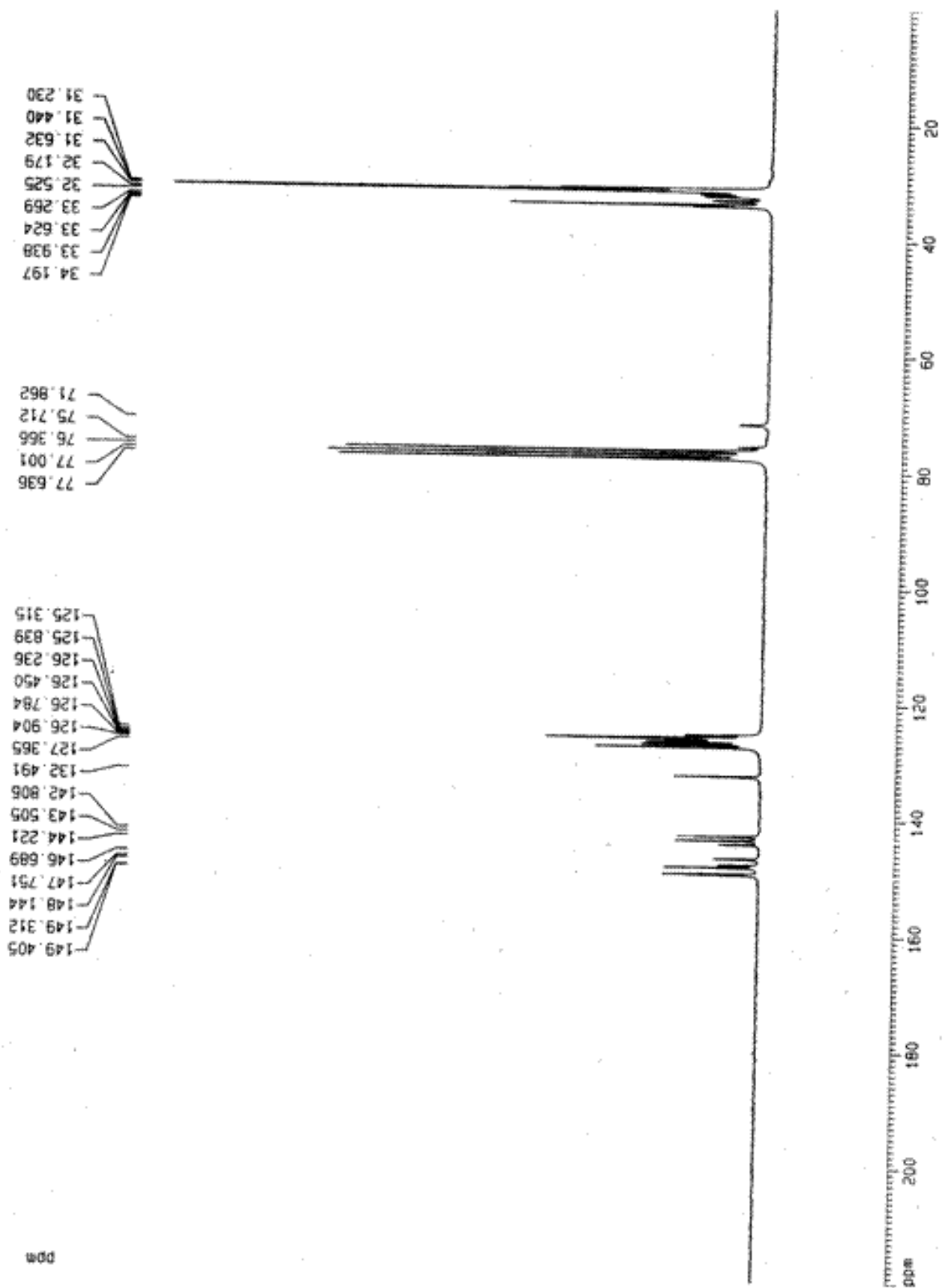
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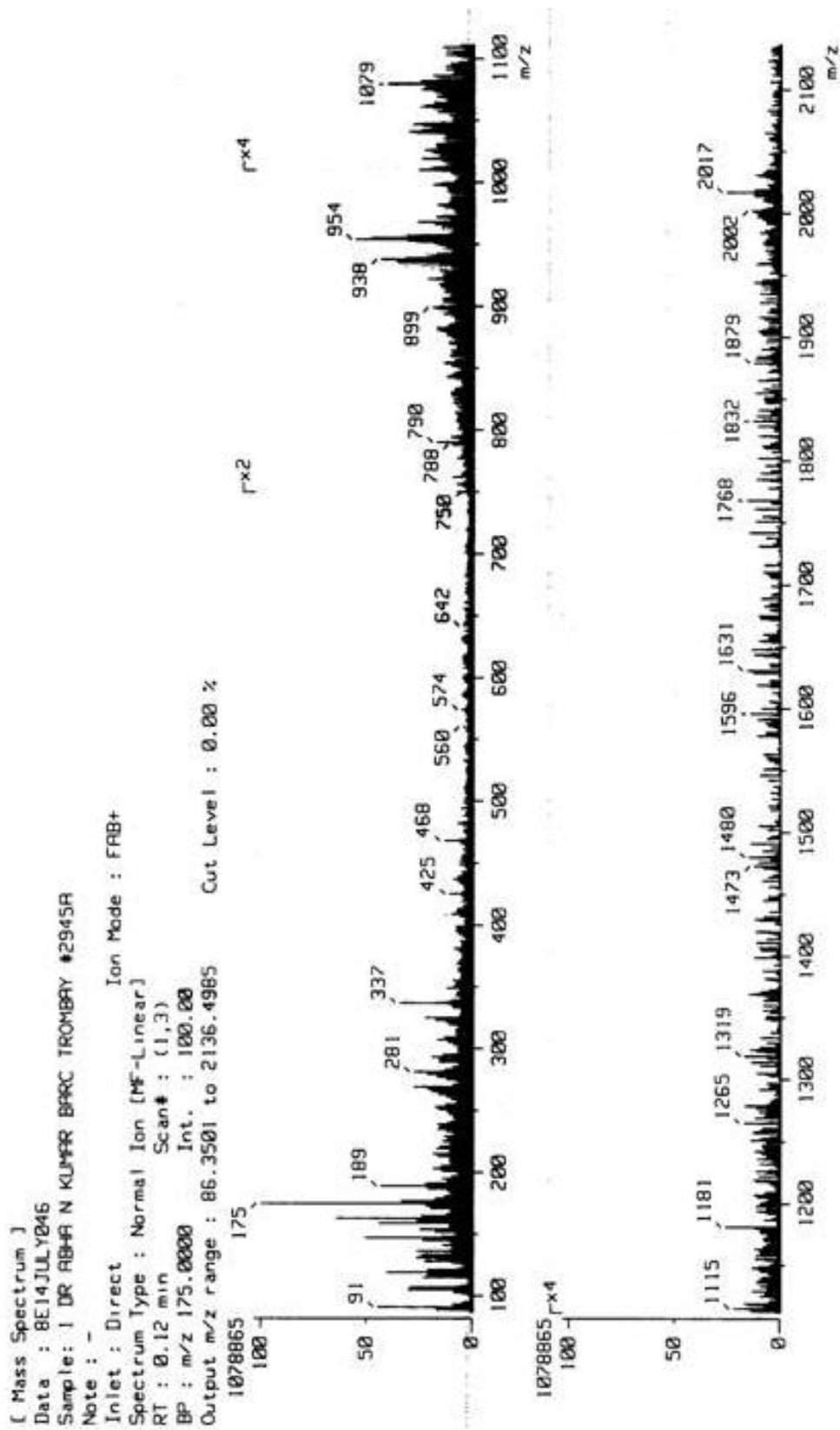
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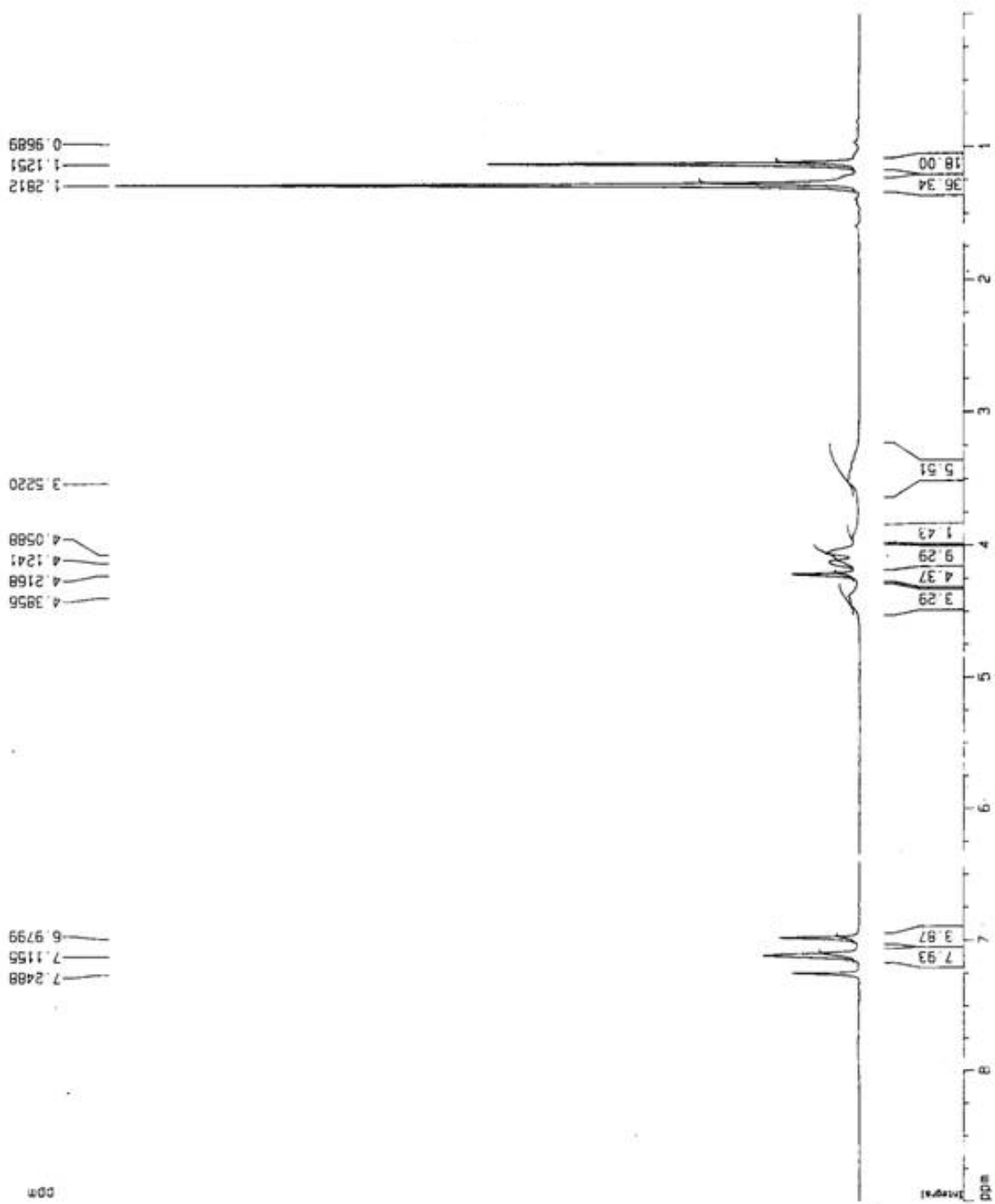
**Figure S1.** <sup>1</sup>H NMR spectrum of bis[2-(5,11,17,23,29,35-hexa-*tert*-butyl-37,38,39,40,41-pentahydroxy-42-calix[6]arenyloxy)ethyl] ether (**2**).



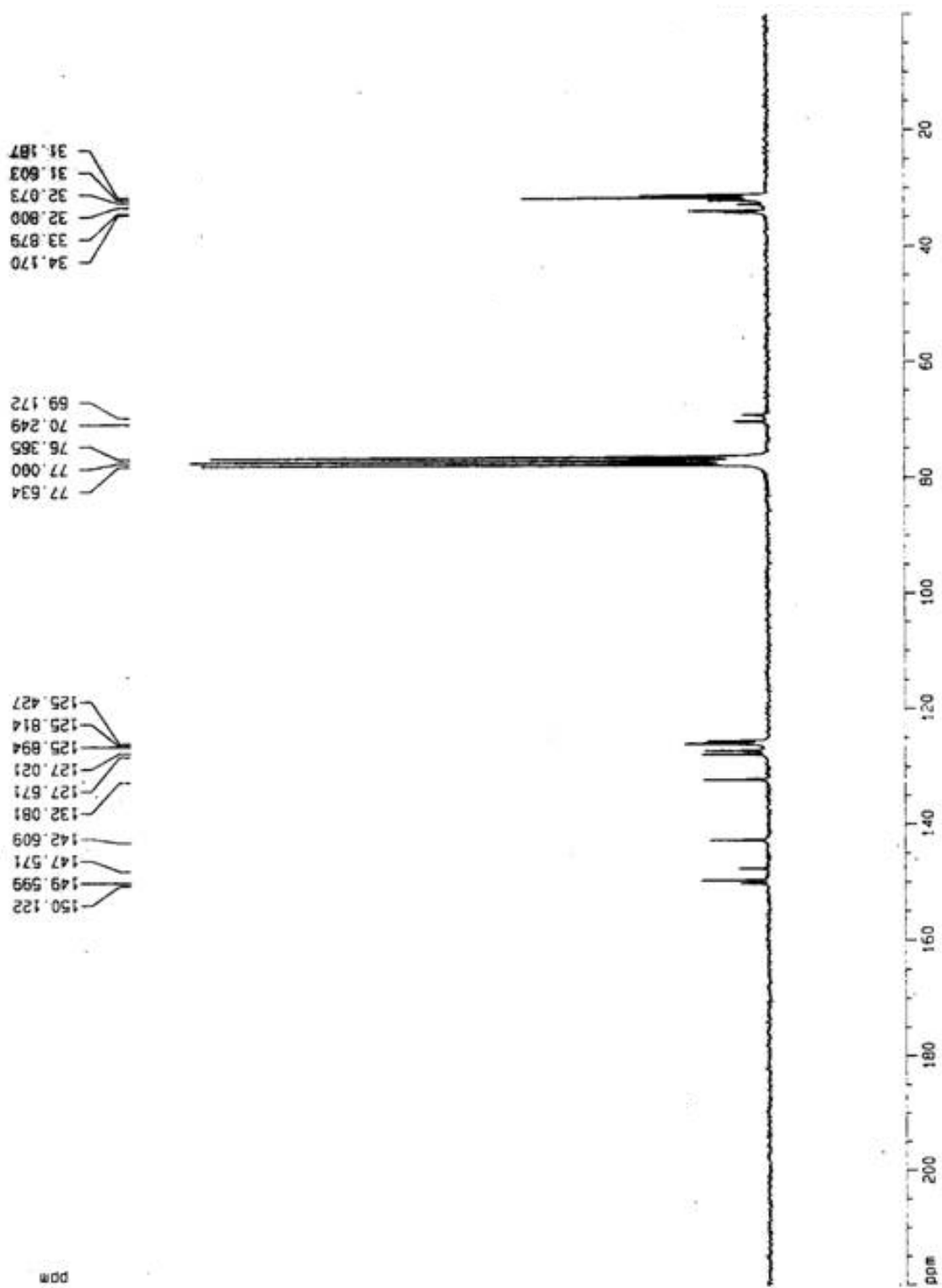
**Figure S2.**  $^{13}\text{C}$  NMR spectrum of bis[2-(5,11,17,23,29,35-hexa-*tert*-butyl-37,38,39,40,41-pentahydroxy-42-calix[6]arenyloxy)ethyl] ether (**2**).



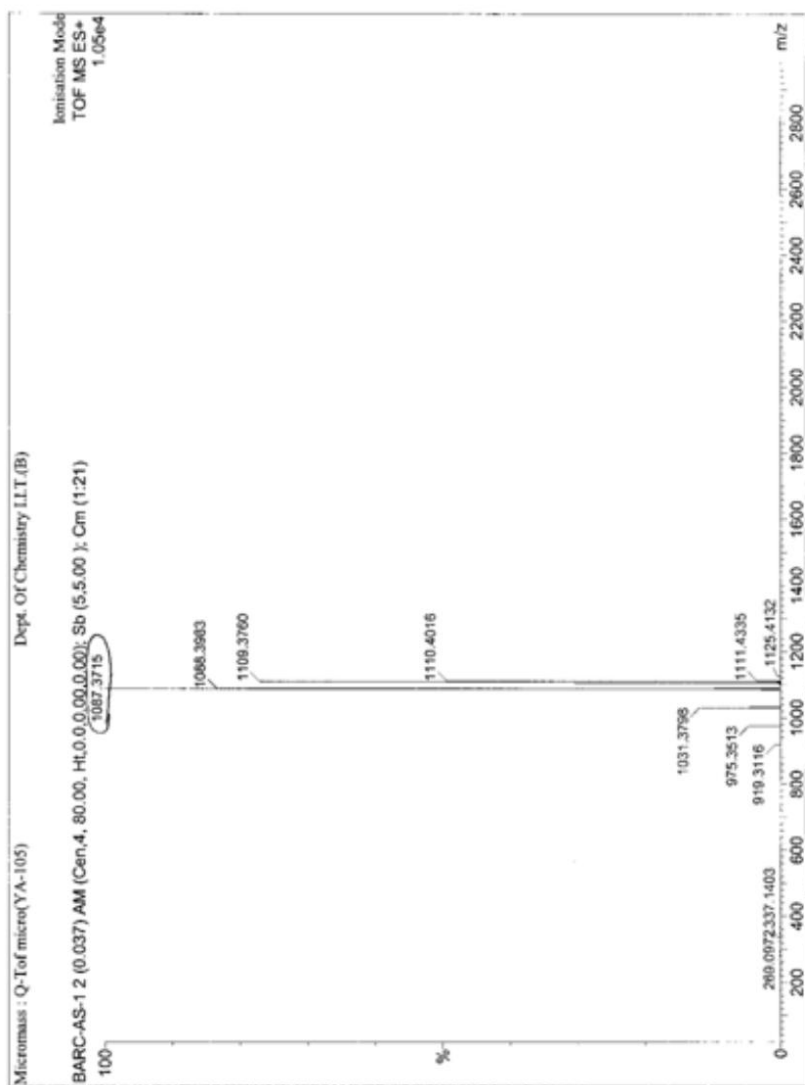
**Figure S3.** Mass spectrum of bis[2-(5,11,17,23,29,35-hexa-*tert*-butyl-37,38,39,40,41-pentahydroxy-42-calix[6]arenyloxy)ethyl] ether (2).



**Figure S4.** <sup>1</sup>H NMR spectrum of 5,11,17,23,29,35-hexa-*tert*-butyl-37,38,40,41-tetrahydroxycalix[6]arene-39,42-(crown-4) (**3**).



**Figure S5.**  $^{13}\text{C}$  NMR spectrum of 5,11,17,23,29,35-hexa-*tert*-butyl-37,38,40,41-tetrahydroxycalix[6]arene-39,42-(crown-4) (**3**).



**Figure S6.** Mass spectrum of 5,11,17,23,29,35-hexa-*tert*-butyl-37,38,40,41-tetrahydroxycalix[6]arene-39,42-(crown-4) (**3**).