

## Experimental.

**General.** Unless otherwise indicated, NMR spectra were recorded at 20 °C on a Bruker AC 250 FT-NMR spectrometer operating at 250 MHz ( $^1\text{H}$ ) and 62.9 MHz ( $^{13}\text{C}$ ), respectively. Chemical shifts were referenced against the solvent signal. The NMR samples of acid salts were prepared in a glovebox (Vacuum Atmospheres CS-40). Diacid **10** was prepared according to earlier work.<sup>1</sup> All other chemicals including tetrabutylammonium hydroxide (1.0 M in methanol) and dicarboxylic acids were obtained commercially (Aldrich) and used without further purification. NMR solvents were obtained from Aldrich (THF- $d_8$ ,  $\text{CD}_2\text{Cl}_2$ ) and Cambridge Isotope Labs ( $\text{CD}_2\text{Cl}_2$ ). Dichloromethane- $d_2$  was dried and distilled from phosphorus pentoxide prior to use.

**Table S1.**  $^1\text{H}$  NMR resonances<sup>a</sup> of the corresponding acid salts of compounds **1–11** (0.15 M in  $\text{CD}_2\text{Cl}_2$ ).

Carboxylic Acid	Hemisalt	$\delta_{\text{OH}}$	$\delta_{\text{CHCO}}$	$\delta_{\text{CHCCO}}$	$\delta_{\text{CHCCCCO}}$
Benzoic acid	<b>1a</b>	18.27	8.05	7.37	---
<i>p</i> -Toluic acid <sup>b</sup>	<b>2a</b>	18.02	3.00	5.69	---
<i>p</i> -Nitrobenzoic acid	<b>3a</b>	18.05	8.19	---	---
<i>trans</i> -1,2-Cyclohexanedicarboxylic acid	<b>4a</b>	16.96	2.39	1.98	1.21
<i>trans</i> -1,2-Cyclopropanedicarboxylic acid	<b>5a</b>	16.90	---	---	---
<i>trans</i> -3-Hexenedioic acid	<b>6a</b>	16.60	2.91	5.59	---
2-Norbornene-2,3-dicarboxylic acid	<b>7a</b>	19.99	3.32	---	---
1-Cyclopentene-1,2-dicarboxylic acid	<b>8a</b>	20.35	---	2.72	1.43
4-Cyclohexene- <i>cis</i> -1,2-dicarboxylic acid	<b>9a</b>	19.75	2.81	2.45	5.61
<i>cis</i> -1,2-Dimethylcyclopropane-1,2-dicarboxylic acid	<b>10a</b>	18.31	1.81	0.84	---
1,1-Cyclobutanedicarboxylic acid	<b>11a</b>	18.91	2.40	2.05	---

<sup>a</sup> $\delta$  in ppm. <sup>b</sup> Signals obscured by the ammonium signals.

**Table S2.**  $^{13}\text{C}\{^1\text{H}\}$  NMR resonances<sup>a</sup> of the corresponding acid salts of compounds **1–11** (0.15 M in  $\text{CD}_2\text{Cl}_2$ ).

Carboxylic Acid	Hemisalt	$\delta_{\text{CO}}$	$\delta_{\text{CCO}}$	$\delta_{\text{CCCO}}$	$\delta_{\text{CCCCO}}$	$\delta_{\text{CCCCCO}}$
Benzoic acid	<b>1a</b>	170.37	137.11	129.93	128.10	130.82
<i>p</i> -Toluic acid <sup>b</sup>	<b>2a</b>	170.30	134.30	130.10	128.80	141.10
<i>p</i> -Nitrobenzoic acid	<b>3a</b>	168.39	142.39	130.86	123.33 <sup>c</sup>	149.68 <sup>d</sup>
<i>trans</i> -1,2-Cyclohexanedicarboxylic acid	<b>4a</b>	177.36	30.04	45.79	---	---
<i>trans</i> -1,2-Cyclopropanedicarboxylic acid	<b>5a</b>	174.77	14.94	23.35	---	---
<i>trans</i> -3-Hexenedioic acid	<b>6a</b>	175.80	127.50	41.30	---	---
2-Norbornene-2,3-dicarboxylic acid	<b>7a</b>	167.69	148.40	46.24	25.55	46.12
1-Cyclopentene-1,2-dicarboxylic acid	<b>8a</b>	168.32	144.50	37.39	20.50	---
4-Cyclohexene- <i>cis</i> -1,2-dicarboxylic acid	<b>9a</b>	178.17	43.91	28.99	126.94	---
<i>cis</i> -1,2-Dimethylcyclopropane-1,2-dicarboxylic acid	<b>10a</b>	178.37	33.70	32.30	19.80	---
1,1-Cyclobutanedicarboxylic acid	<b>11a</b>	179.34	50.16	30.28	17.51	---

<sup>a</sup> $\delta$  in ppm. <sup>b</sup> $\delta$  ( $\text{CCH}_3$ ) = 35.17 ppm and  $\delta$  ( $\text{CCH}_3$ ) = 31.56 ppm. <sup>c</sup> $\delta$  ( $\text{CCNO}_2$ ). <sup>d</sup> $\delta$  ( $\text{CNO}_2$ ) = 31.56 ppm.

<sup>1</sup> McCoy, L. L. *J. Am. Chem. Soc.* **1958**, *80*, 6568-6572.