

Experimental.

General. Unless otherwise indicated, NMR spectra were recorded at 20 °C on a Bruker AC 250 FT-NMR spectrometer operating at 250 MHz (¹H) and 62.9 MHz (¹³C), 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.¹ 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, CD₂Cl₂) and Cambridge Isotope Labs (CD₂Cl₂). Dichloromethane-*d*₂ was dried and distilled from phosphorus pentoxide prior to use.

Table S1. ¹H NMR resonances^a of the corresponding acid salts of compounds **1–11** (0.15 M in CD₂Cl₂).

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

^aδ in ppm. ^b Signals obscured by the ammonium signals.

Table S2. ¹³C{¹H}NMR resonances^a of the corresponding acid salts of compounds **1–11** (0.15 M in CD₂Cl₂).

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

^aδ in ppm. ^bδ (CCH₃) = 35.17 ppm and δ (CCH₃) = 31.56 ppm. ^cδ (CCNO₂). ^dδ (CNO₂) = 31.56 ppm.

¹ McCoy, L. L. *J. Am. Chem. Soc.* **1958**, 80, 6568-6572.