

Green Strategies for the Preparation of Enantiomeric 5–8-Membered Carbocyclic β -Amino Acid Derivatives through CALB-Catalyzed Hydrolysis

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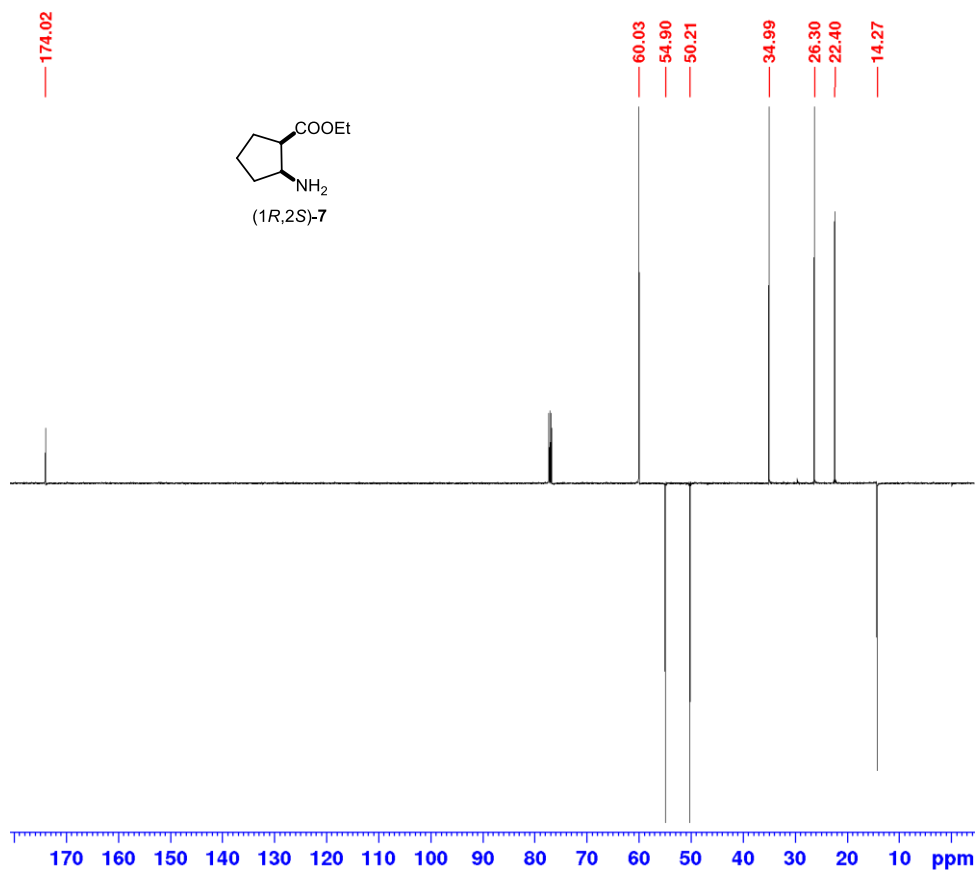
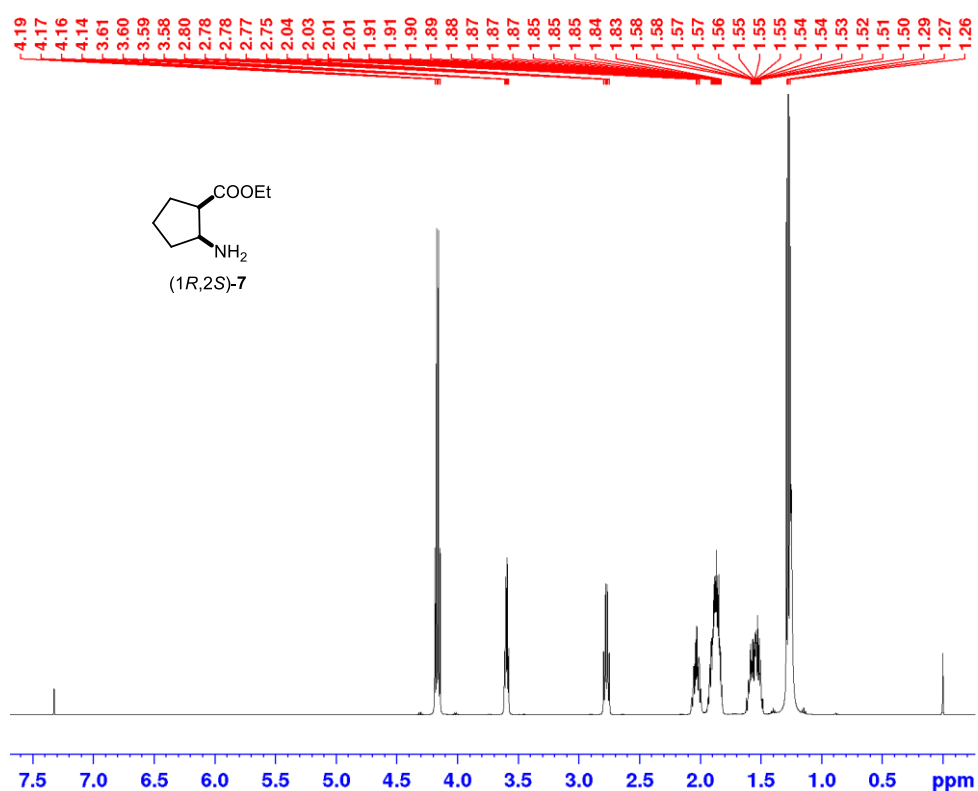
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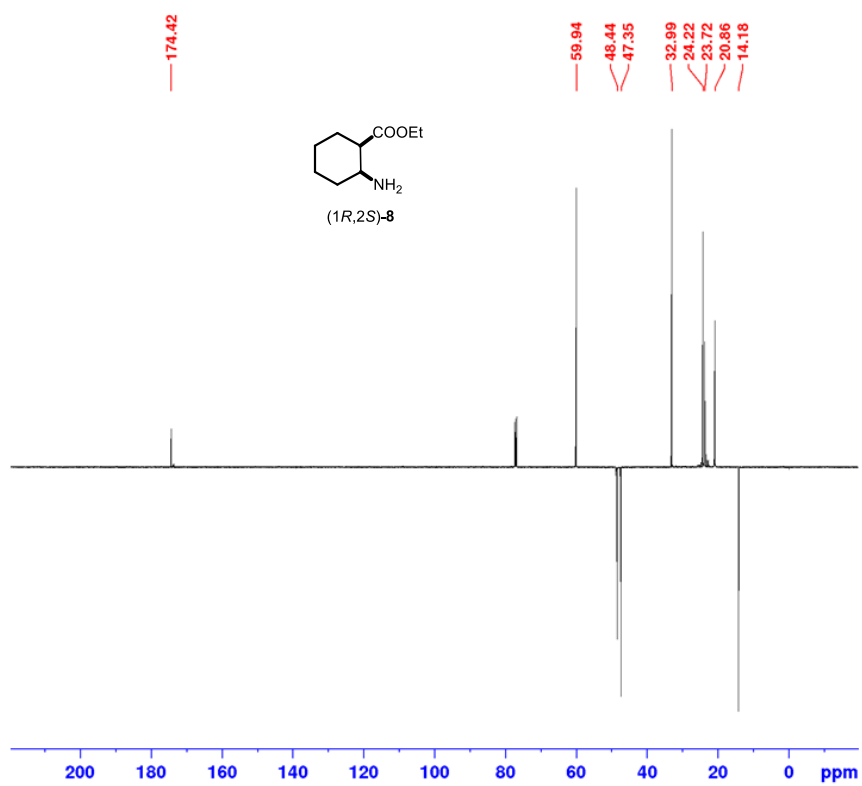
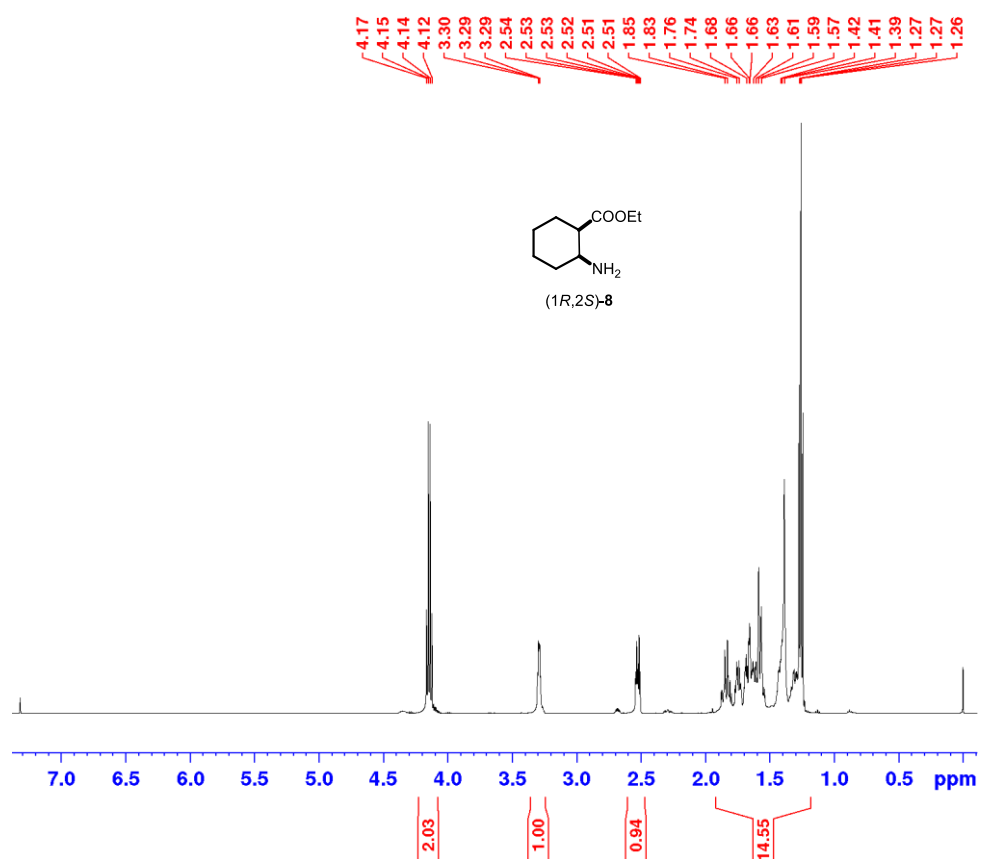
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1. ^1H - and ^{13}C -NMR spectra of ester enantiomers (1*R*,2*S*)-7-9 and (1*R*,2*S*)-13

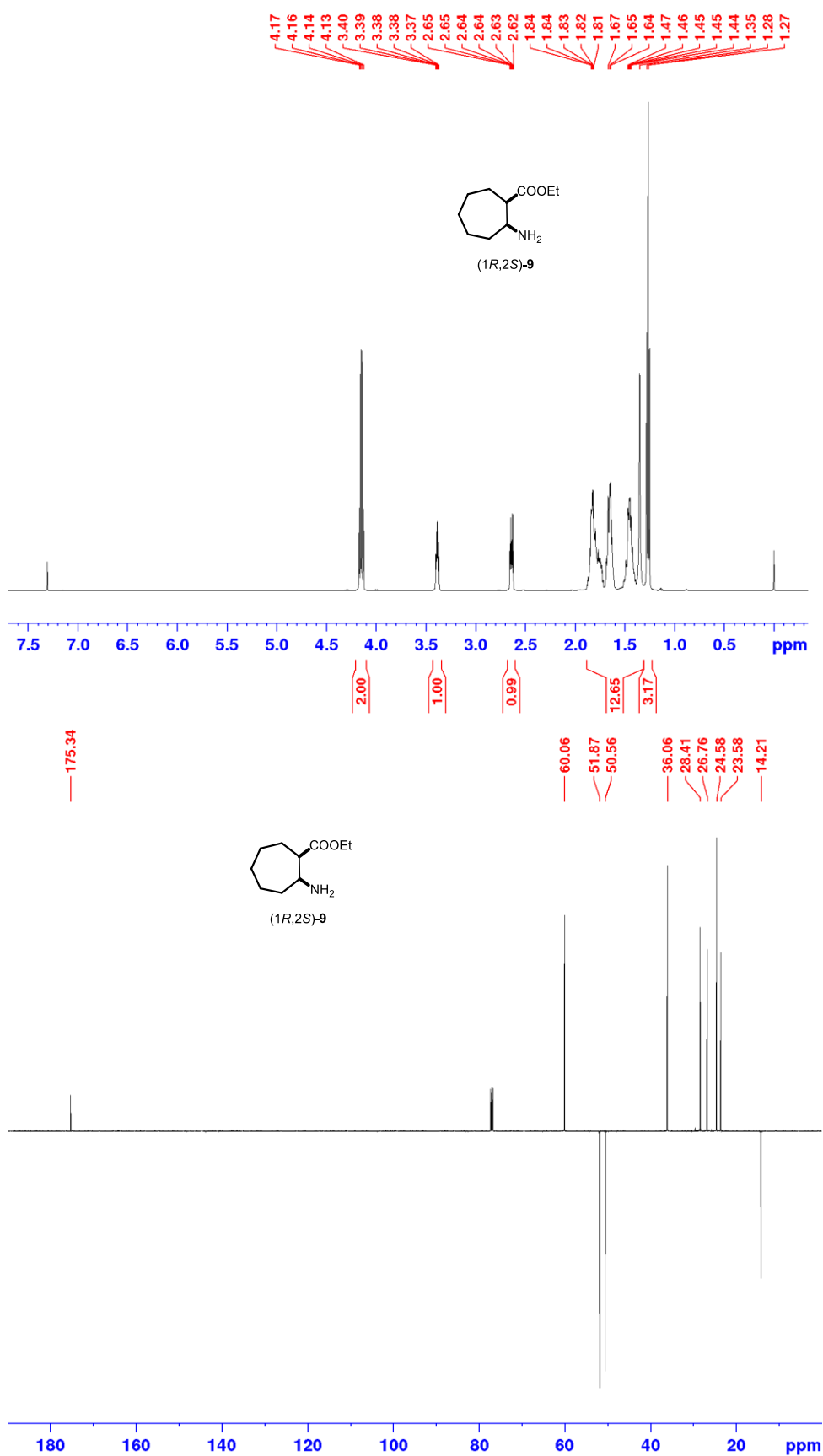
Ethyl (1*R*,2*S*)-2-aminocyclopentanecarboxylate (**7**)



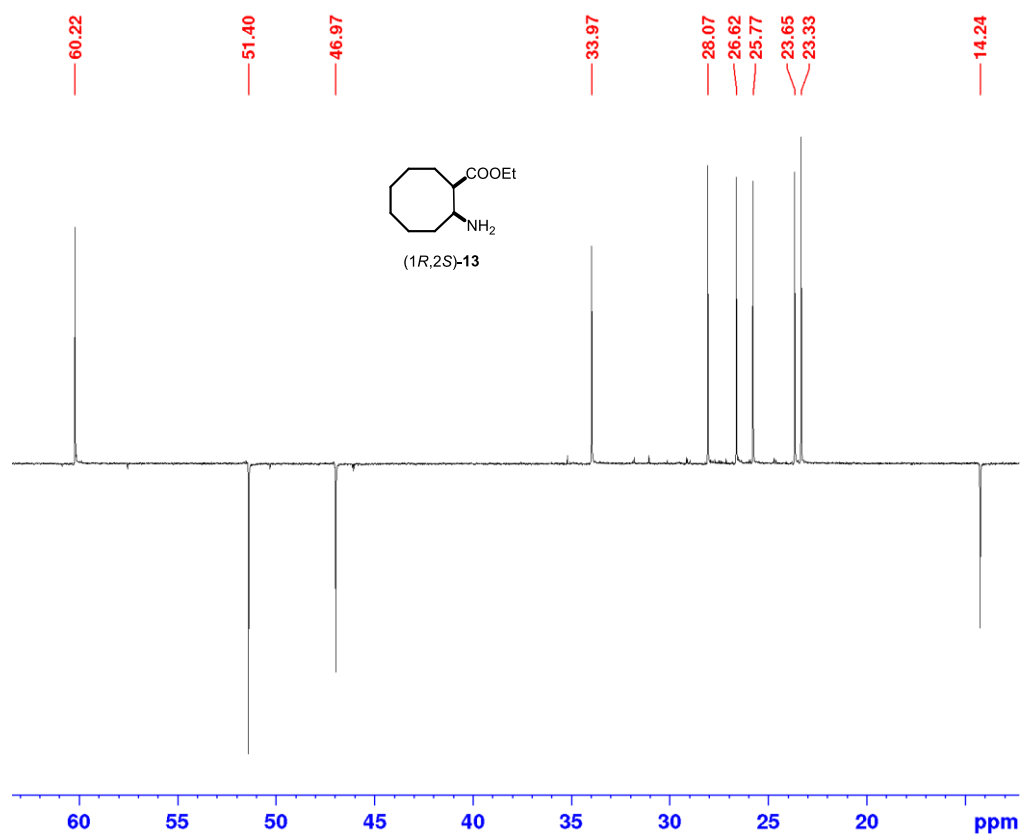
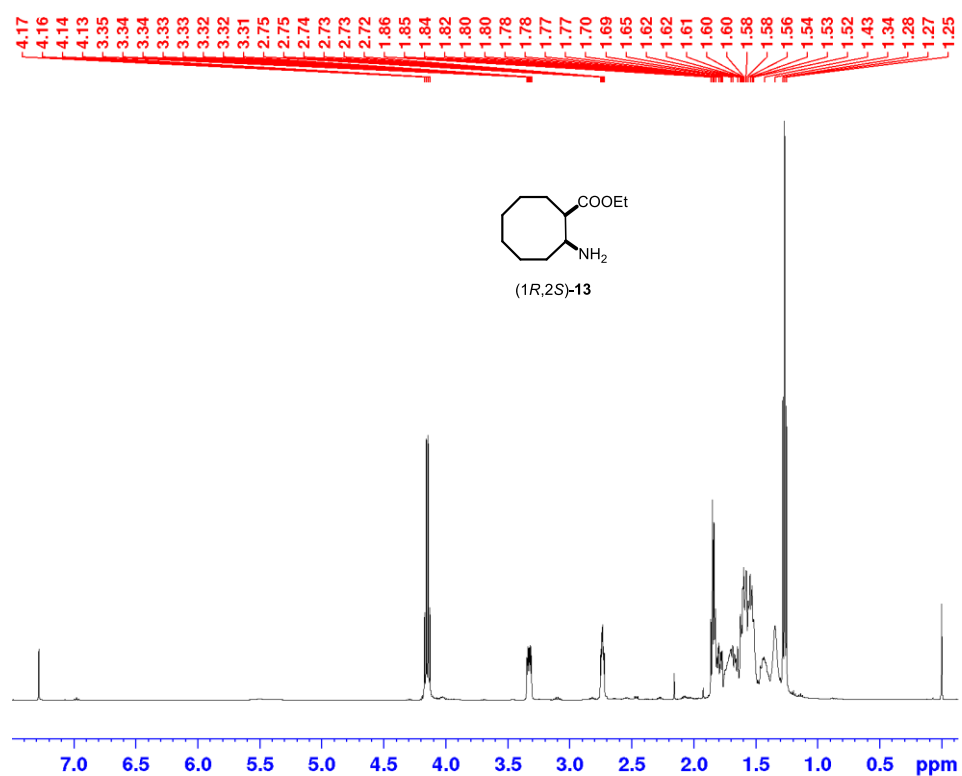
Ethyl (1*R*,2*S*)-2-aminocyclohexanecarboxylate (**8**)



Ethyl (1*R*,2*S*)-2-aminocycloheptanecarboxylate (**9**)

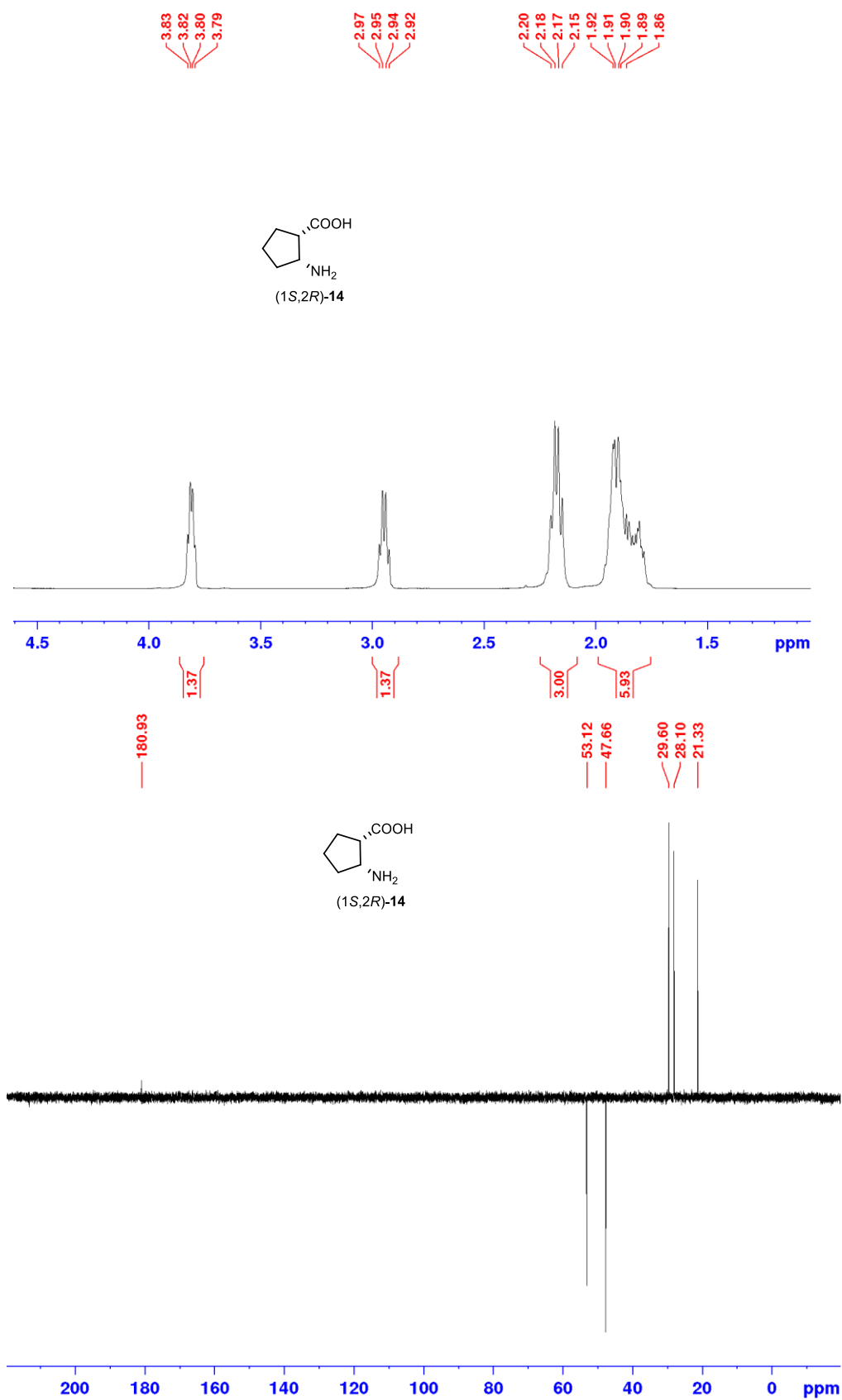


Ethyl (1*R*,2*S*)-2-aminocyclooctanecarboxylate (**13**)

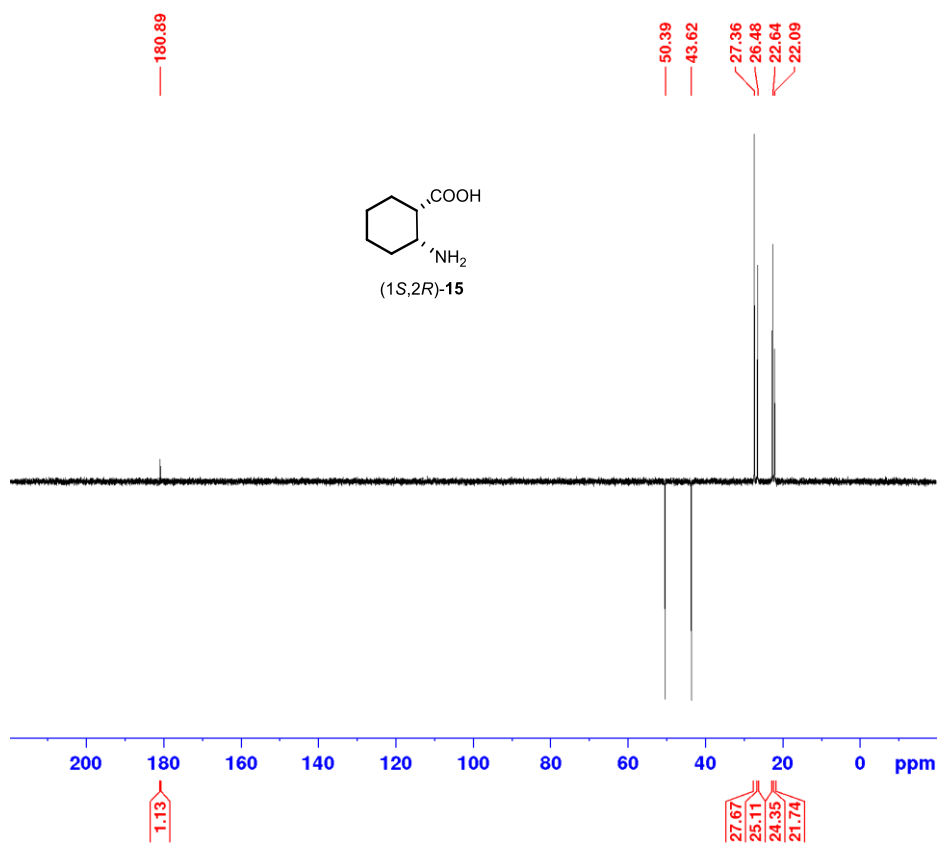
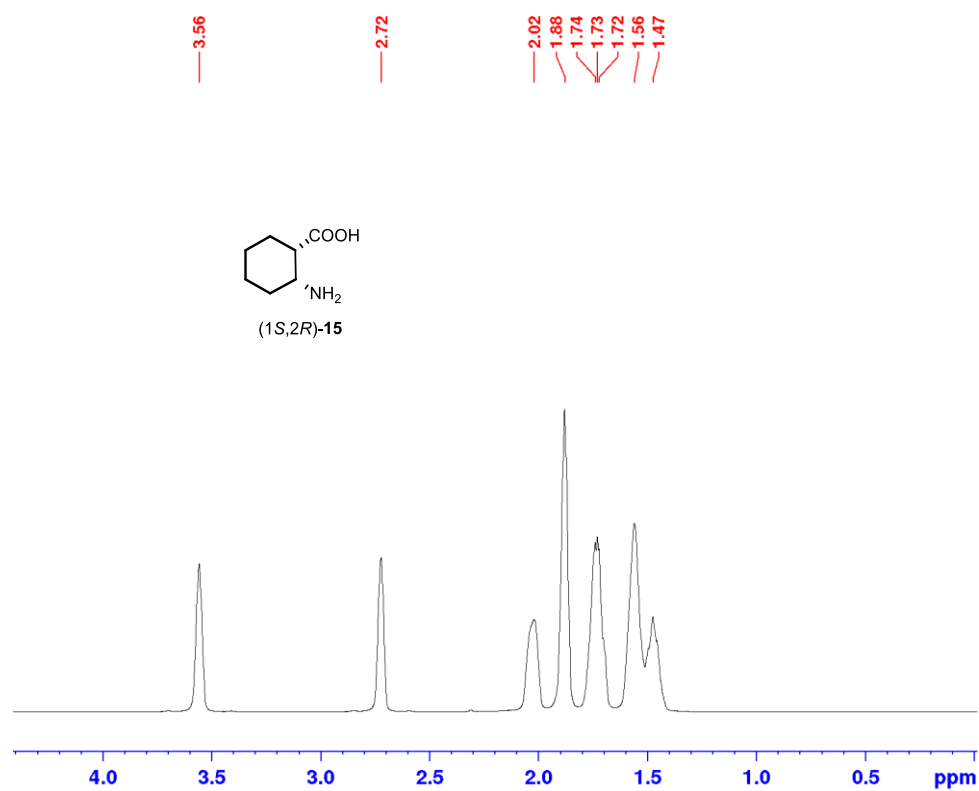


2. ^1H - and ^{13}C -NMR spectra of amino acid enantiomers (1*S*,2*R*)-14-17

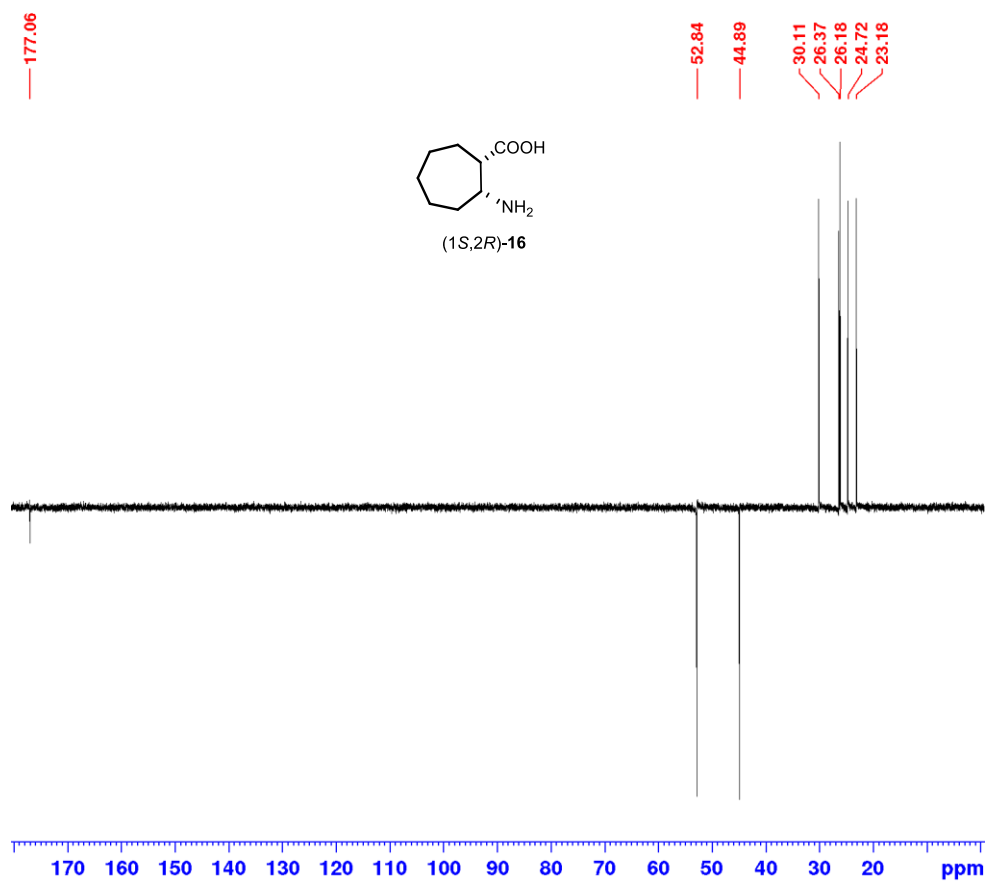
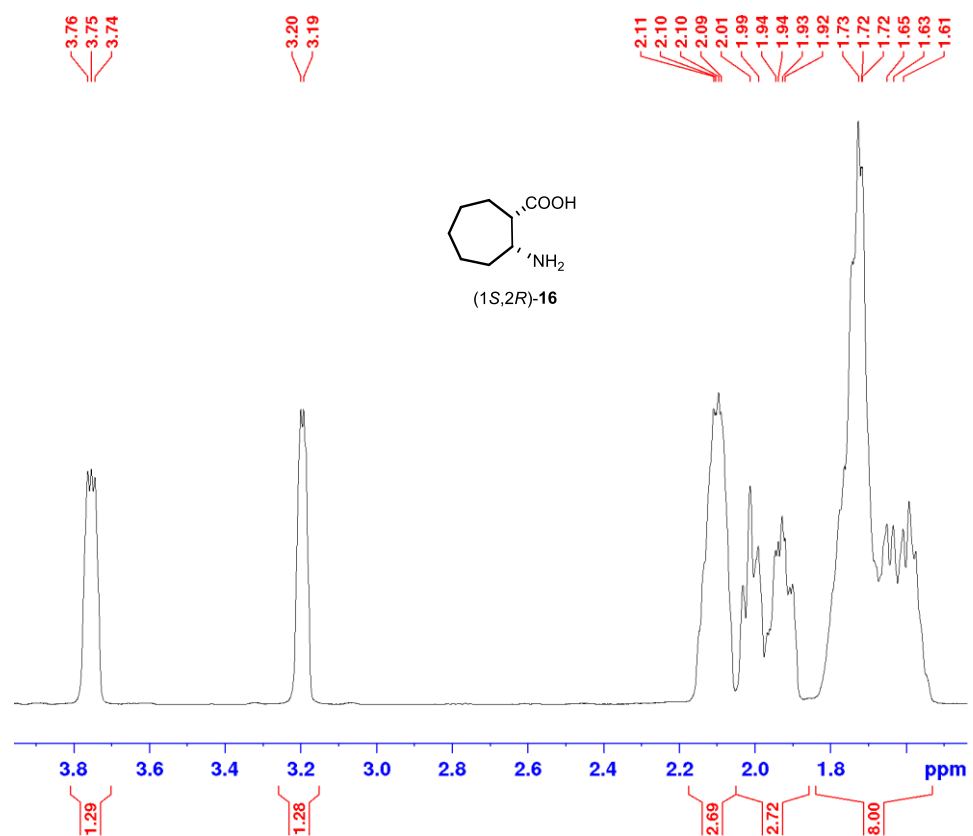
(1*S*,2*R*)-2-aminocyclopentanecarboxylic acid (**14**)



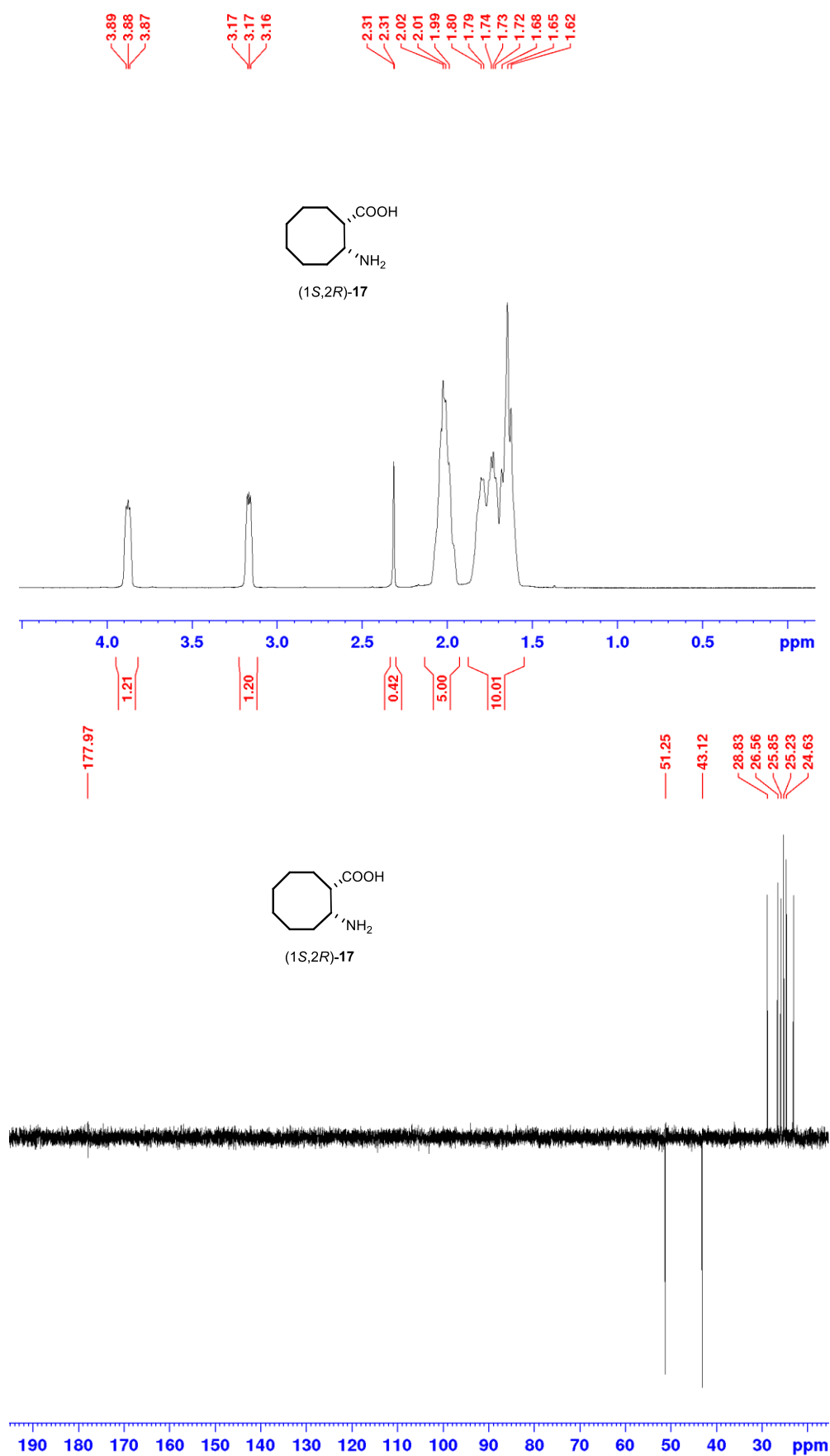
(1*S*,2*R*)-2-aminocyclohexanecarboxylic acid (**15**)



(1*S*,2*R*)-2-aminocycloheptanecarboxylic acid (**16**)

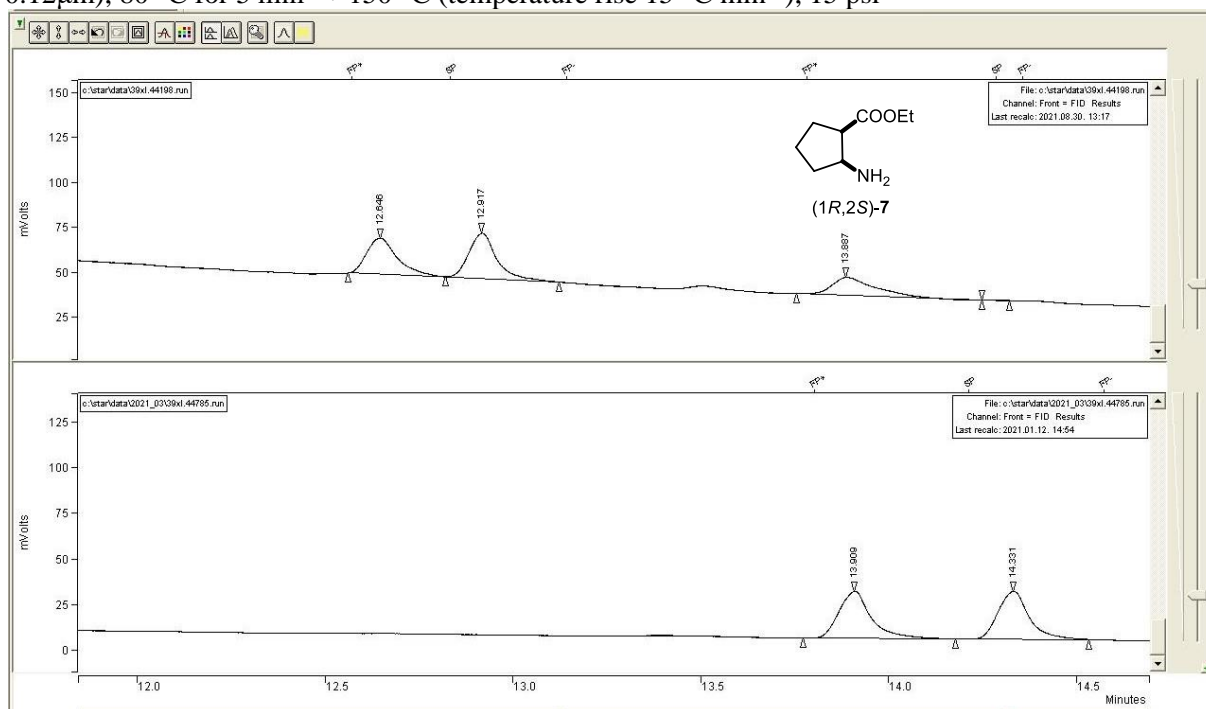


(1*S*,2*R*)-2-aminocyclooctanecarboxylic acid (**17**)

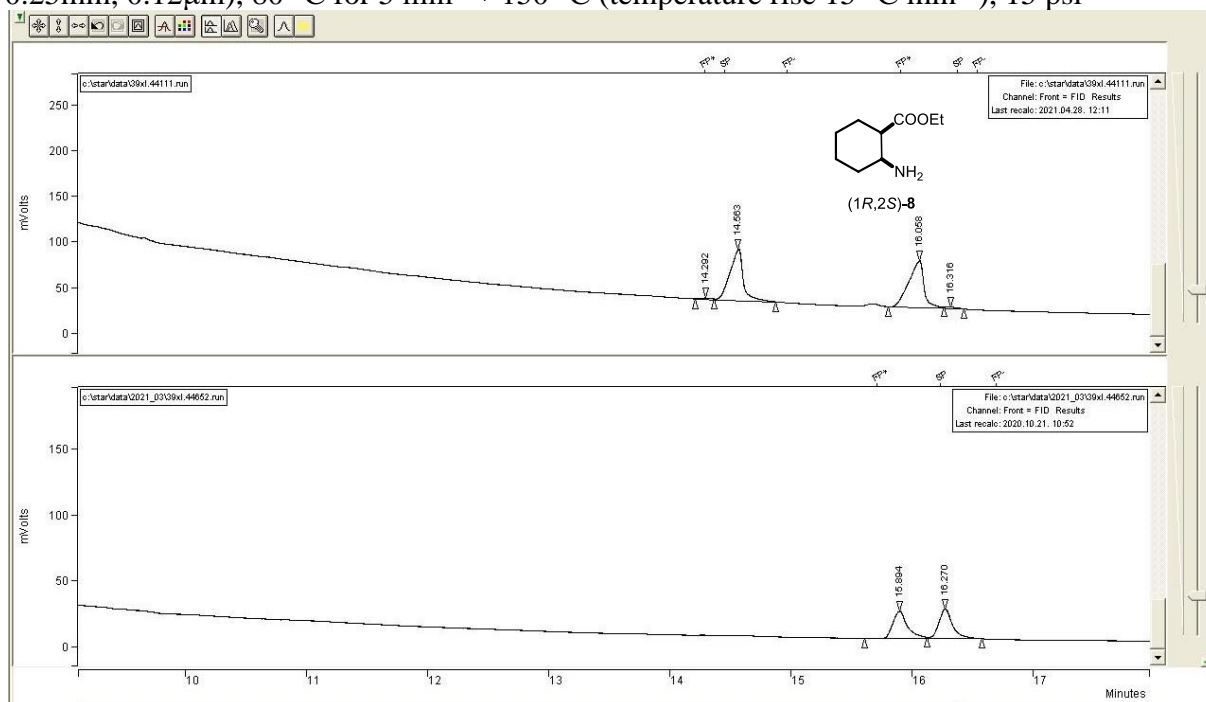


3. GC Chromatograms of ester enantiomers 7-9 and 13:

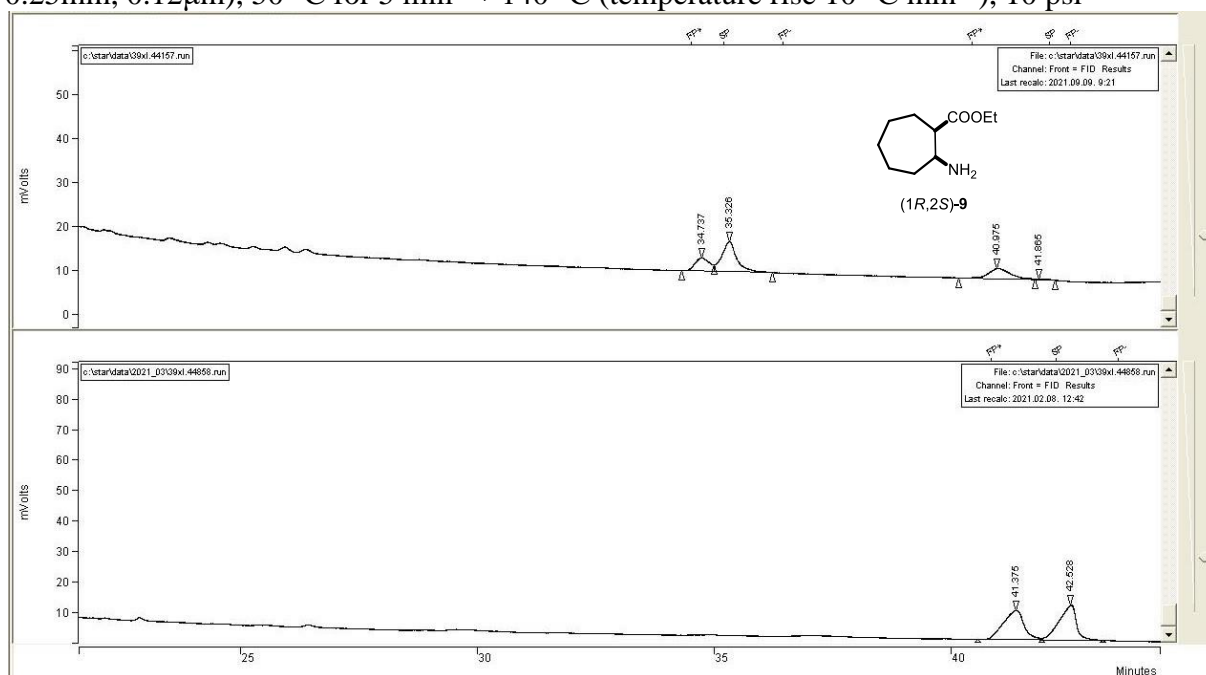
GC Chromatogram of ethyl-2-aminocyclopentanecarboxylate (**7**): retention times (min) for (1*R*,2*S*)-**7**:13.887 (antipode: 14.331): GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12µm), 80 °C for 5 min → 150 °C (temperature rise 15 °C min⁻¹), 15 psi



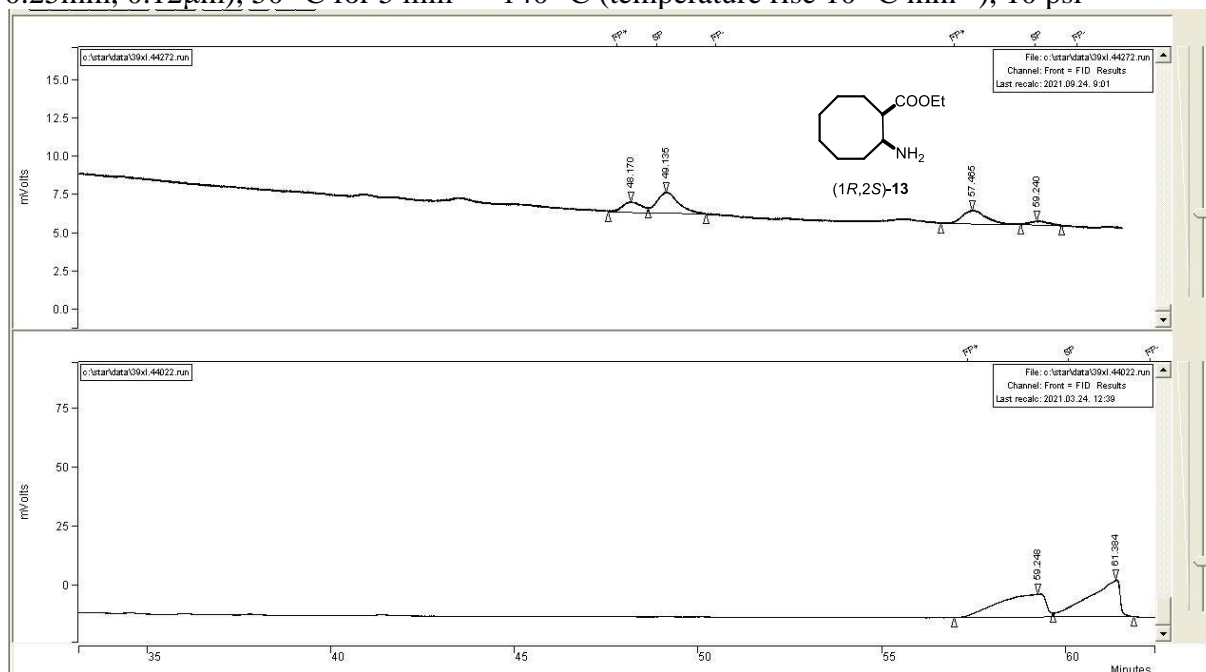
GC Chromatogram of ethyl-2-aminocyclohexanecarboxylate (**8**): retention times (min) for (1*R*,2*S*)-**8**: 16.058 (antipode: 16.316): GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12µm), 80 °C for 5 min → 150 °C (temperature rise 15 °C min⁻¹), 15 psi



GC Chromatogram of ethyl-2-aminocycloheptanecarboxylate (**9**): retention times (min) for (1*R*,2*S*)-**9**: 40.975 (antipode: 41.865), GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12μm), 50 °C for 5 min → 140 °C (temperature rise 10 °C min⁻¹), 10 psi

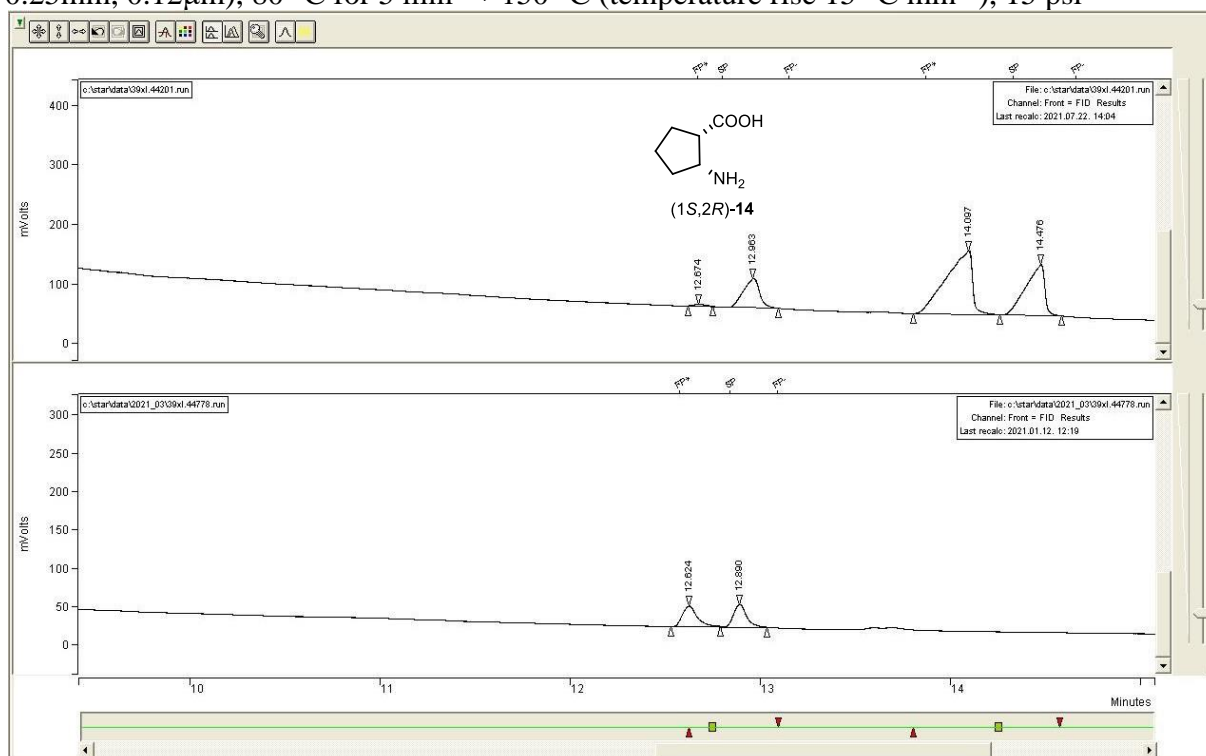


GC Chromatogram of ethyl-2-aminocyclooctanecarboxylate (**13**): retention times (min) for (1*R*,2*S*)-**13**: 57.405 (antipode: 59.240), GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12μm), 50 °C for 5 min → 140 °C (temperature rise 10 °C min⁻¹), 10 psi

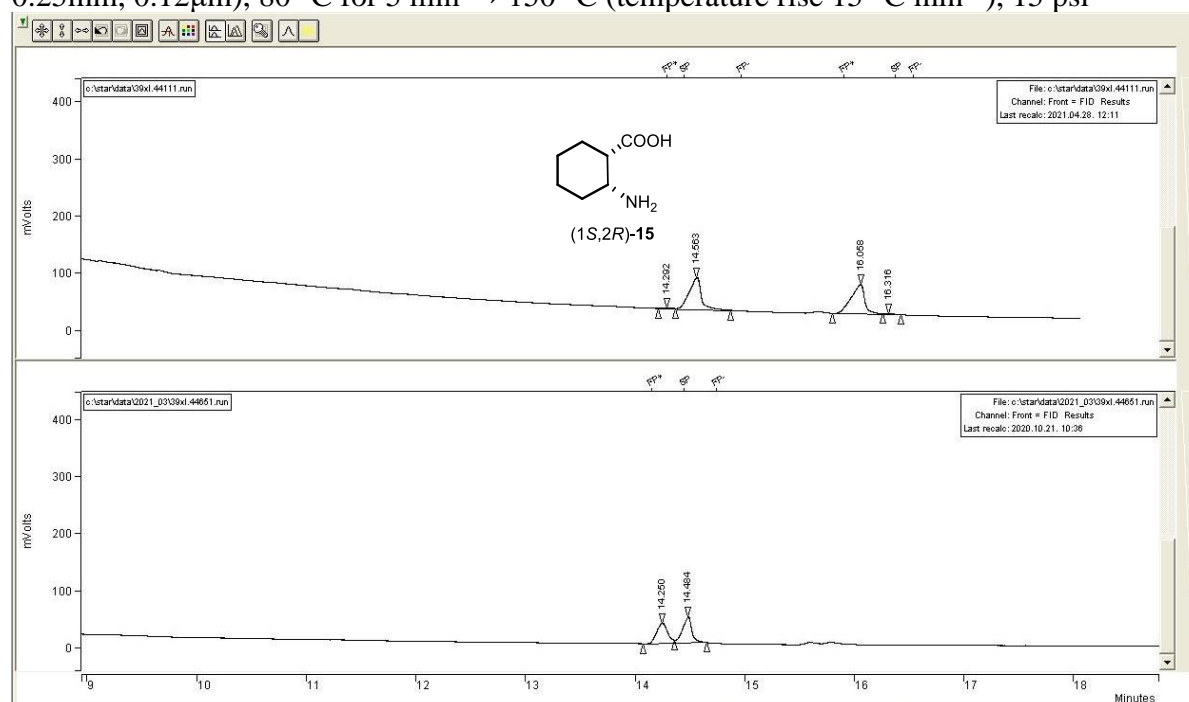


4. GC Chromatograms of amino acid enantiomers 14-17:

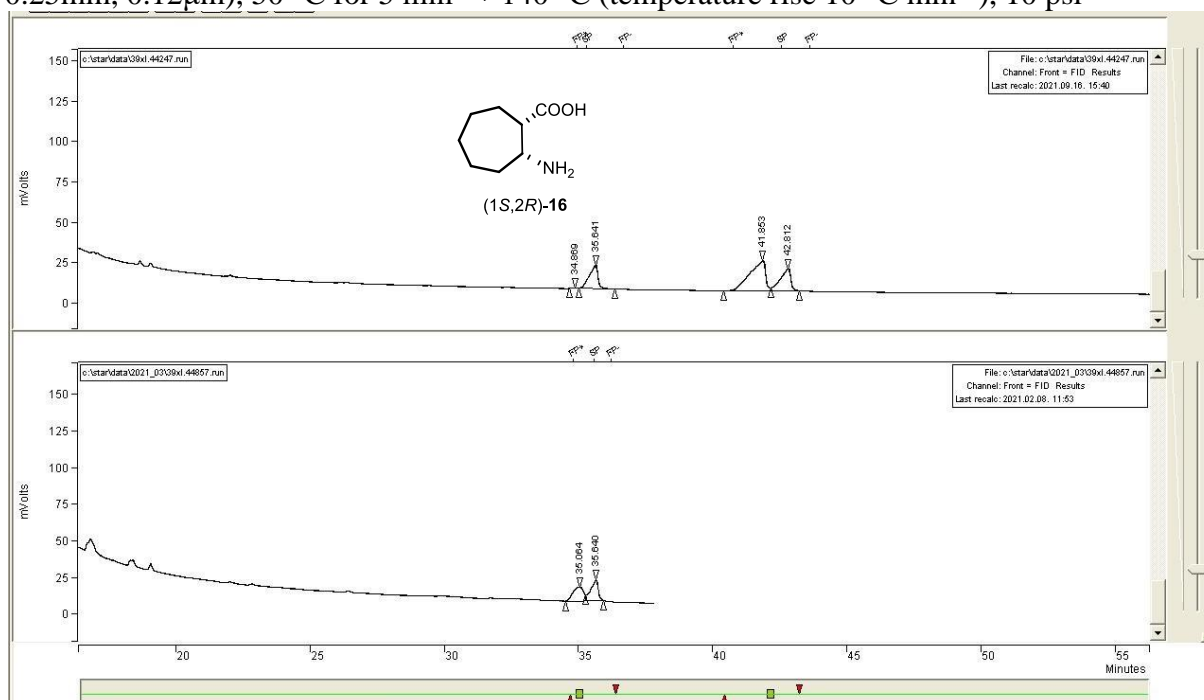
GC Chromatograms of 2-aminocyclopentanecarboxylic acid (**14**): retention times (min) for (1*S*,2*R*)-**14**: 12.963 (antipode: 12.674), GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12μm), 80 °C for 5 min → 150 °C (temperature rise 15 °C min⁻¹), 15 psi



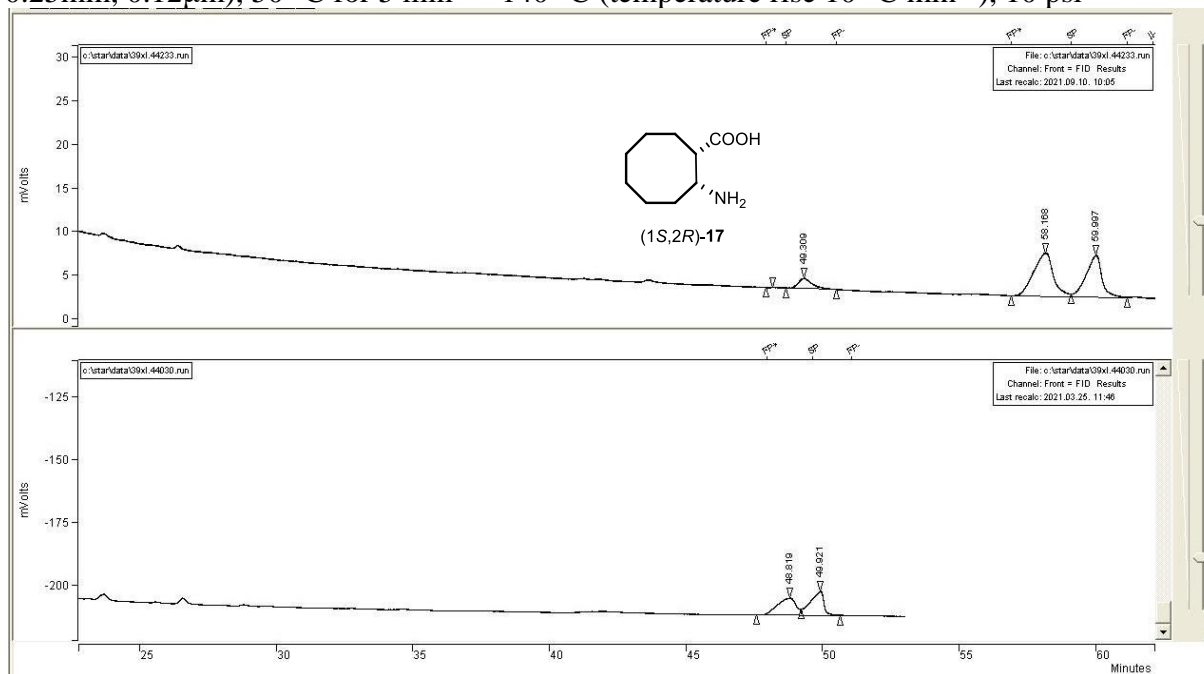
GC Chromatograms of 2-aminocyclohexanecarboxylic acid (**15**): retention times (min) for (1*S*,2*R*)-**15**: 14.563 (antipode: 14.292), GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12μm), 80 °C for 5 min → 150 °C (temperature rise 15 °C min⁻¹), 15 psi



GC Chromatograms of 2-aminocycloheptanecarboxylic acid (**16**): retention times (min) for (1*S*,2*R*)-**16**: 35.641 (antipode: 34.869) GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12 μ m), 50 °C for 5 min \rightarrow 140 °C (temperature rise 10 °C min⁻¹), 10 psi

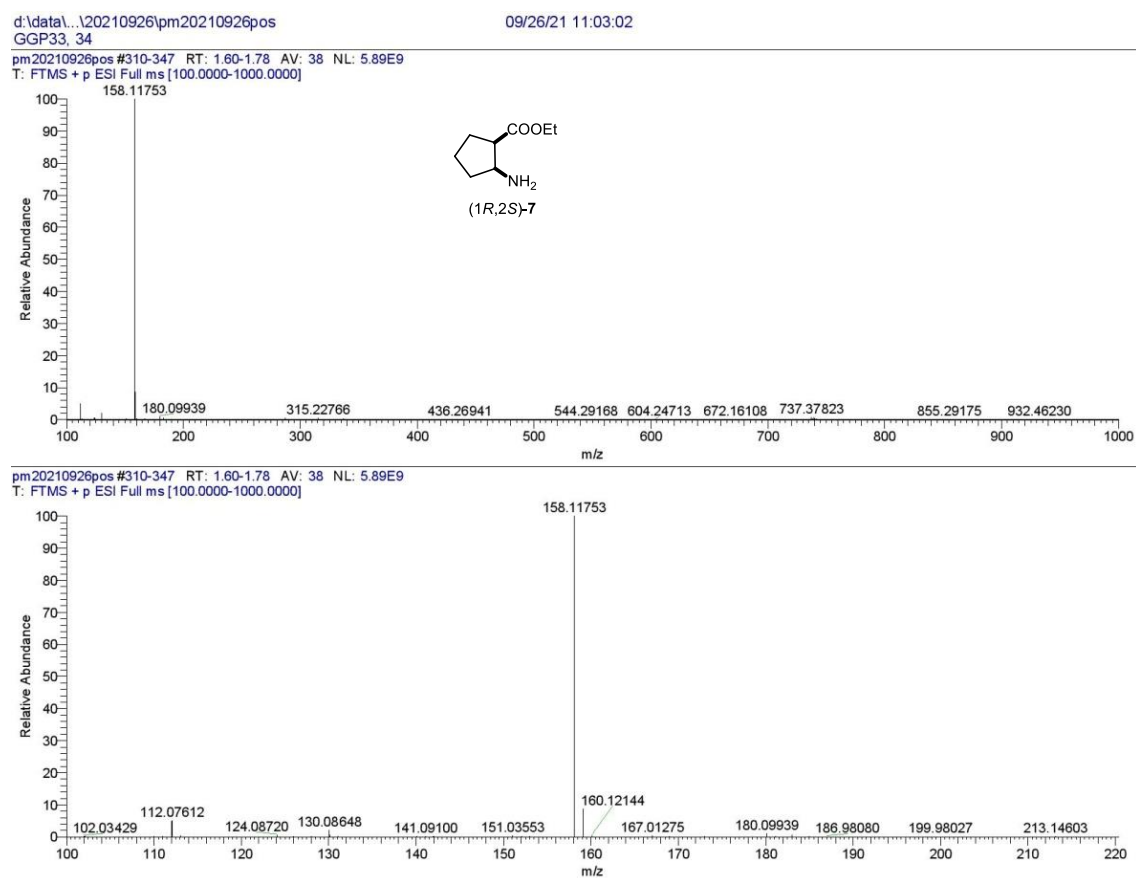


GC Chromatograms of 2-aminocyclooctanecarboxylic acid (**17**): retention times (min) for (1*S*,2*R*)-**17**: 49.309 (antipode: 48.819), GC equipped with a Chirasil-L-Val column (25m, 0.25mm, 0.12 μ m), 50 °C for 5 min \rightarrow 140 °C (temperature rise 10 °C min⁻¹), 10 psi



5. HRMS (ESI) spectra of ester enantiomers (1*R*,2*S*)-7-9 and (1*R*,2*S*)-13

HRMS spectrum of ethyl (1*R*,2*S*)-2-aminocyclopentanecarboxylate (**7**)

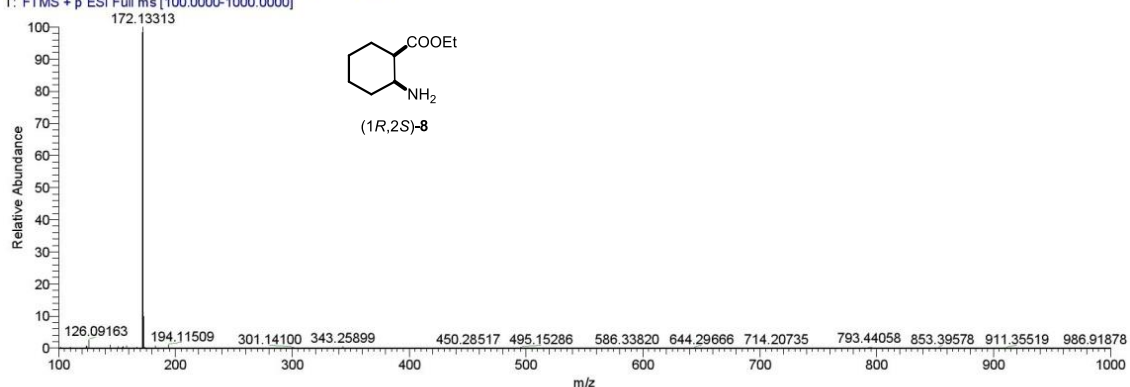


HRMS spectrum of ethyl (1*R*,2*S*)-2-aminocyclohexanecarboxylate (**8**)

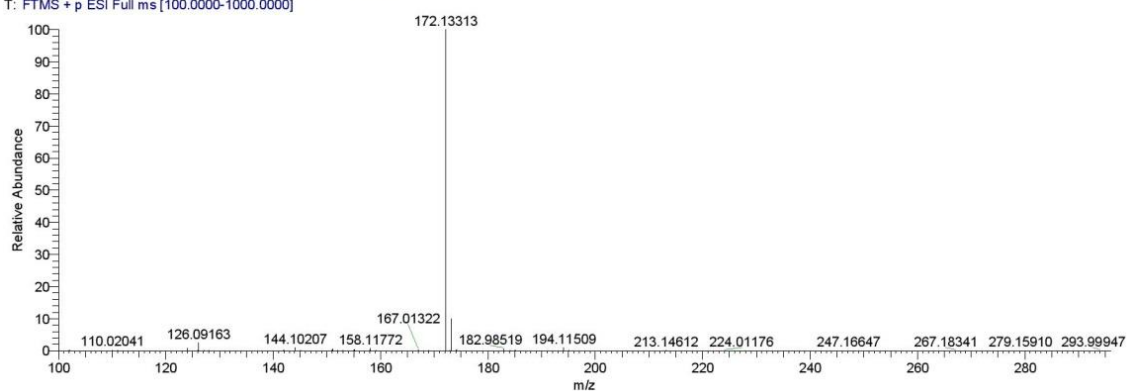
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09/26/21 11:03:02

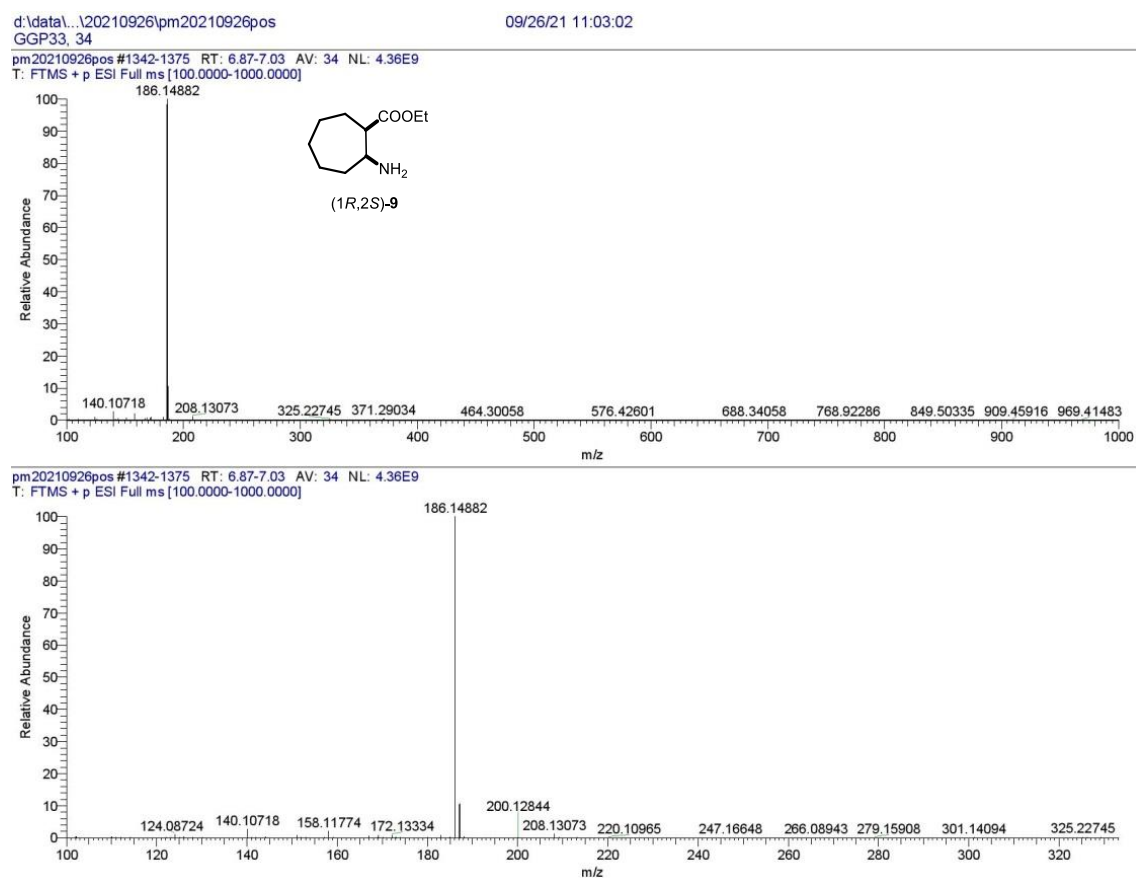
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pm20210926pos #925-962 RT: 4.74-4.92 AV: 38 NL: 5.52E9
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HRMS spectrum of ethyl (1*R*,2*S*)-2-aminocycloheptanecarboxylate (**9**)

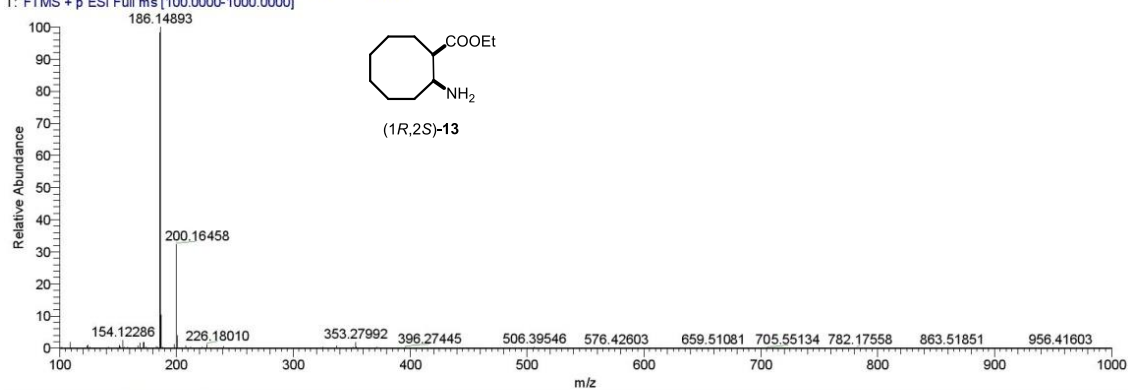


HRMS spectrum of ethyl (1*R*,2*S*)-2-aminocyclooctanecarboxylate (**13**)

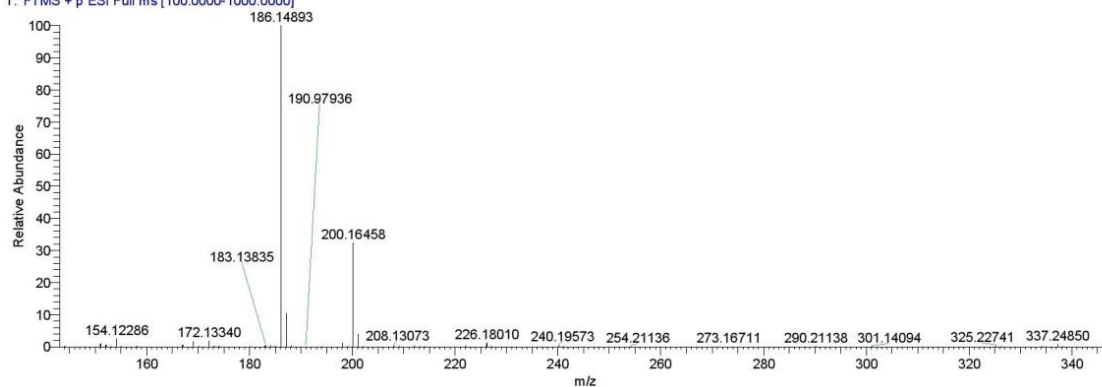
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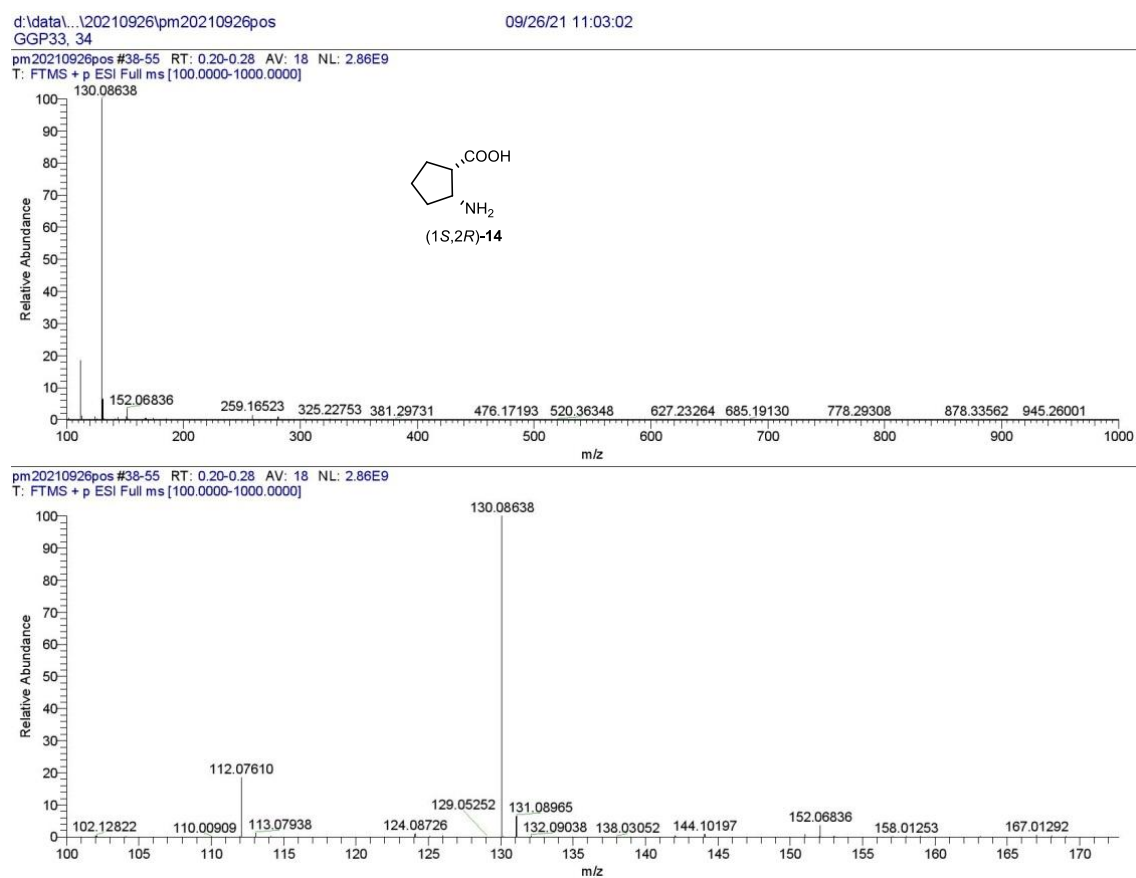


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T: FTMS + p ESI Full ms [100.0000-1000.0000]



6. HRMS (ESI) spectra of amino acid enantiomers (1*S*,2*R*)-14-17

HRMS spectrum of (1*S*,2*R*)-2-aminocyclopentanecarboxylic acid (**14**)

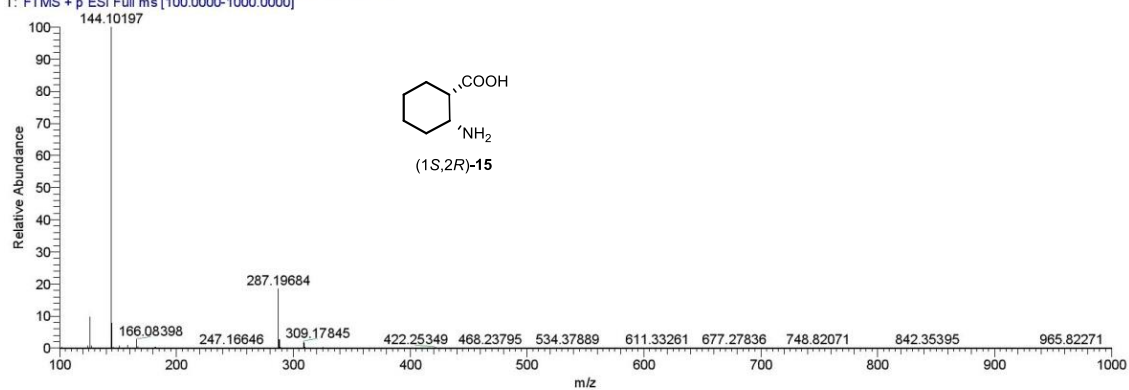


HRMS spectrum of (1*S*,2*R*)-2-aminocyclohexanecarboxylic acid (**15**)

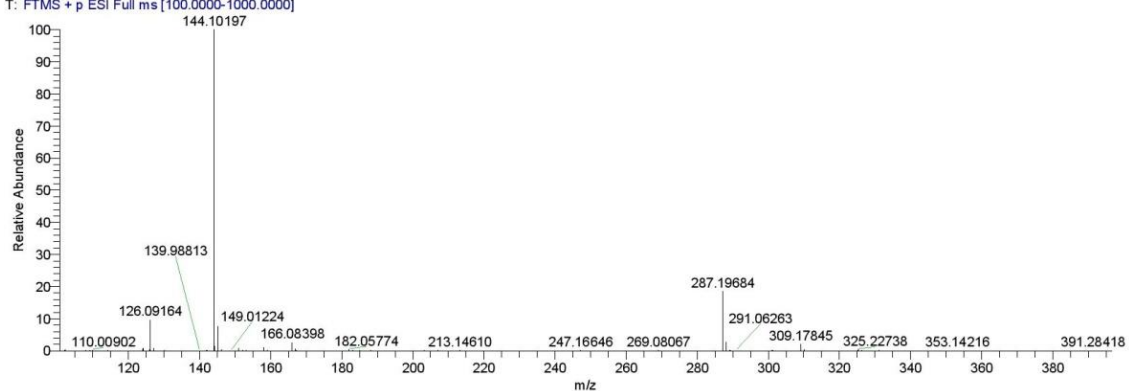
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09/26/21 11:03:02

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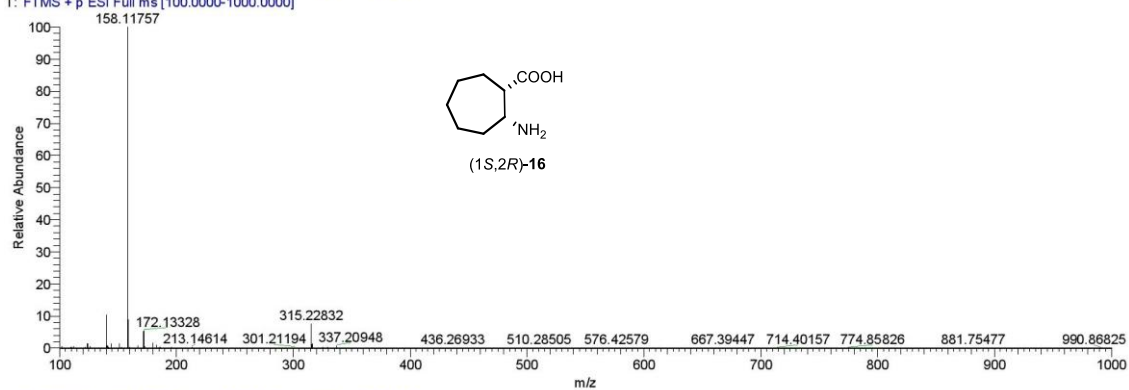


HRMS spectrum of (1*S*,2*R*)-2-aminocycloheptanecarboxylic acid (**16**)

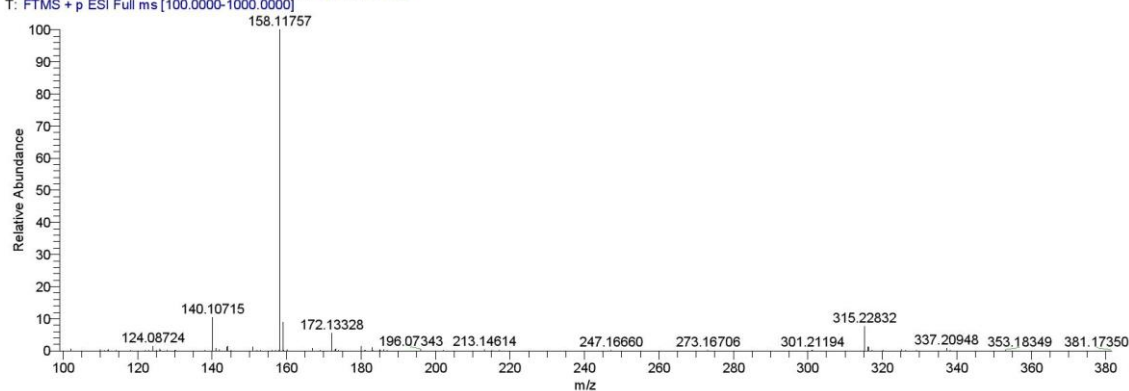
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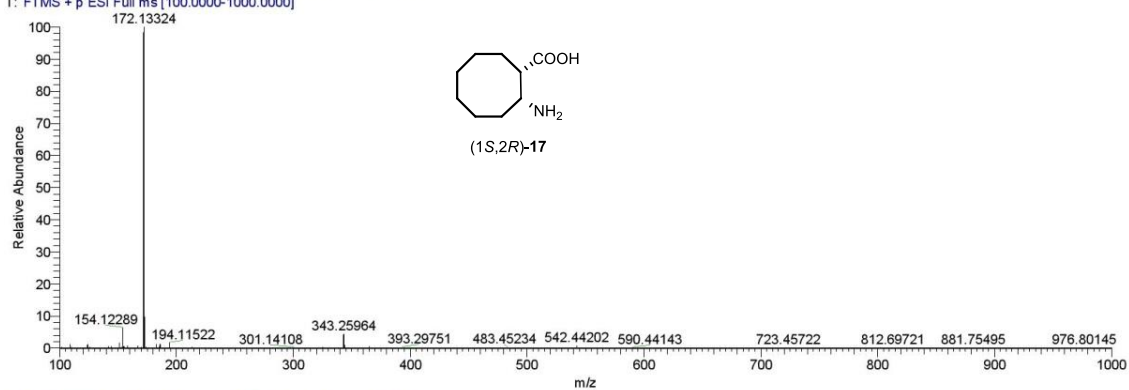


HRMS spectrum of (1*S*,2*R*)-2-aminocyclooctanecarboxylic acid (**17**)

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