

ELECTRONIC SUPPLEMENTARY INFORMATION

**Diastereoselective liquid assisted grinding: ‘Cracking’ functional resins to
advance chromatography-free synthesis**

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Contents:

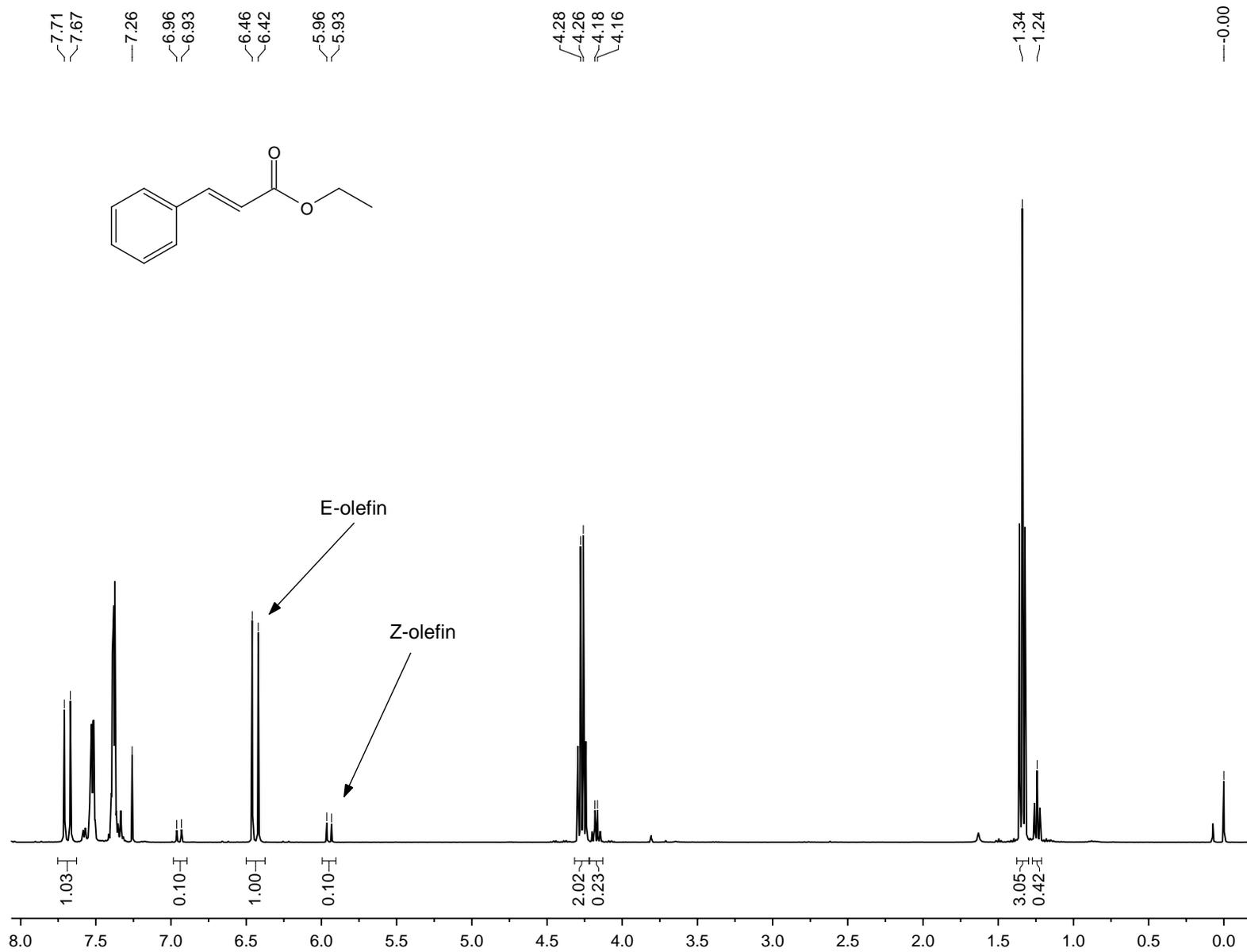
General Information.....	S2
Experimental Conditions.....	S2
Selected ¹H-NMR Spectra.....	S3-S12

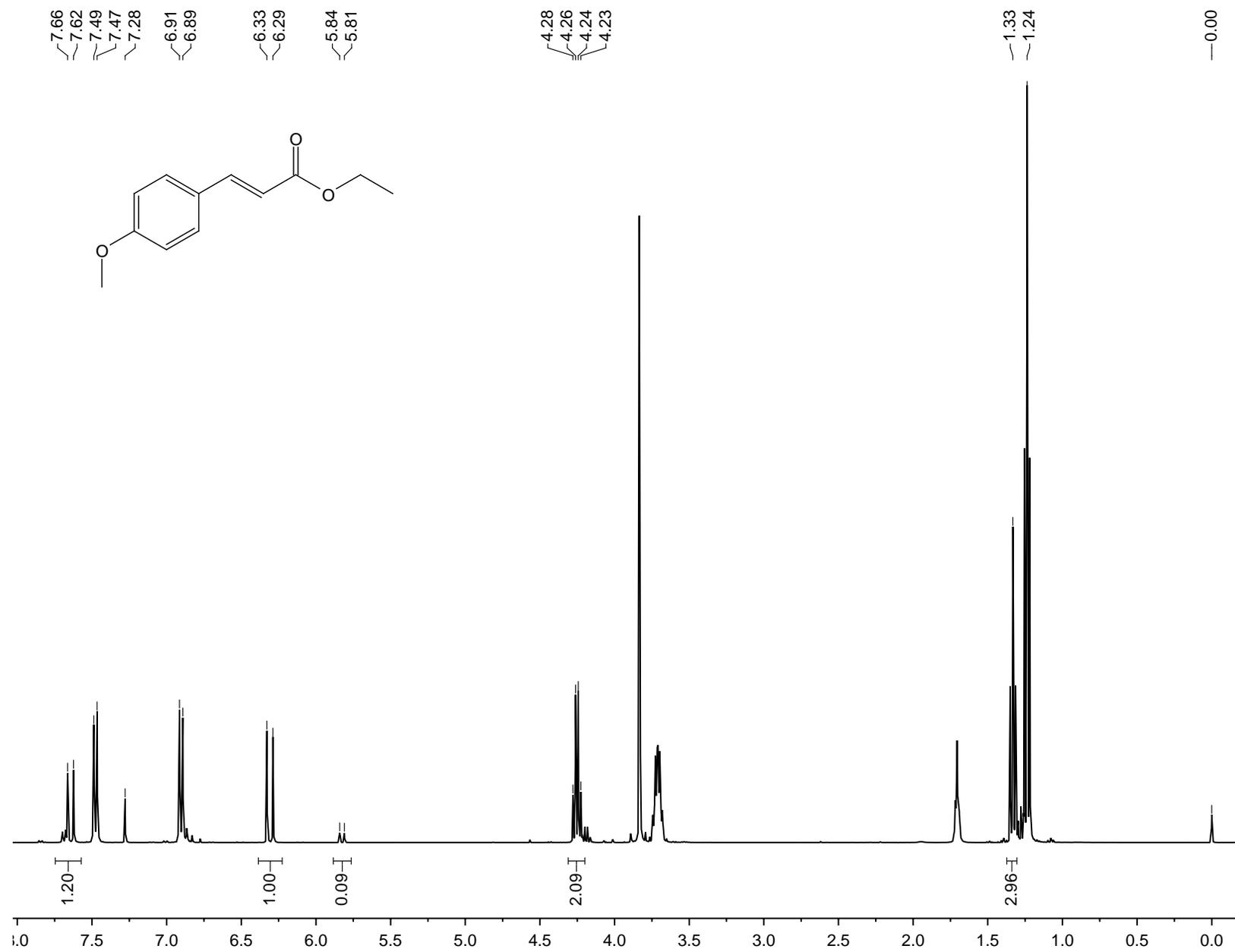
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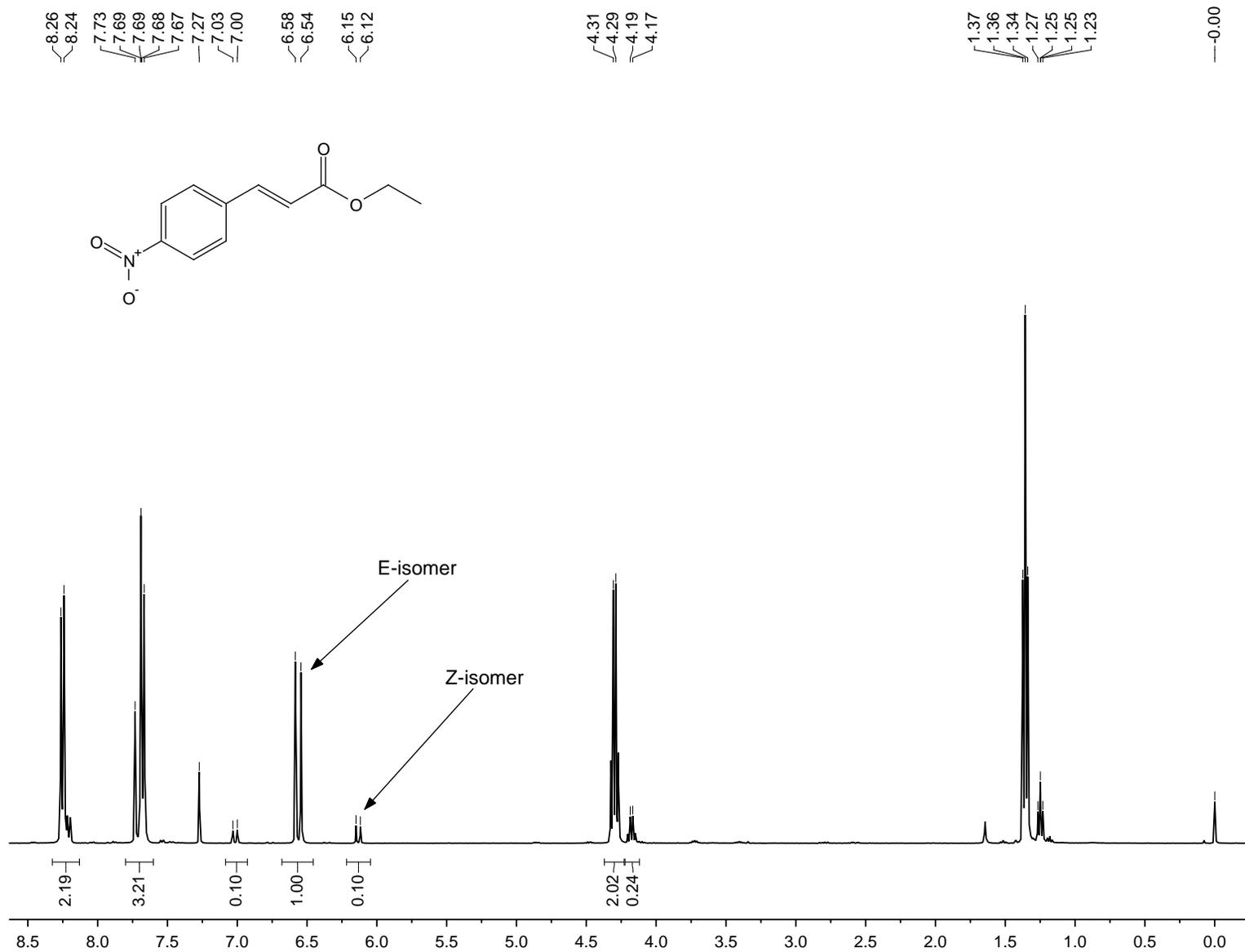
¹H-NMR spectra were recorded on a Bruker-Avance 400 MHz spectrometer. Deuterated NMR solvents were obtained from Cambridge Isotope Laboratories, Inc., Andover MA, and used without further purification. Starting aldehydes and alkyl halides were purchased from Acros® Organics and used without further purification. Triphenyl phosphine functionalized polystyrene (2% cross-linked with DVB, 100 μm diameter, ~2.24 mmol/g loading) was donated by Biotage Ltd.. Milling balls (5 mm) were purchased from McMaster-Carr Industrial Supply Co.. Ball milling was carried out in a Spex8000M Mixer/Mill purchased from SpexCertiprep. Digital microscope images were captured using a Keyence VHX-S50 digital microscope with a VH-220R lens.

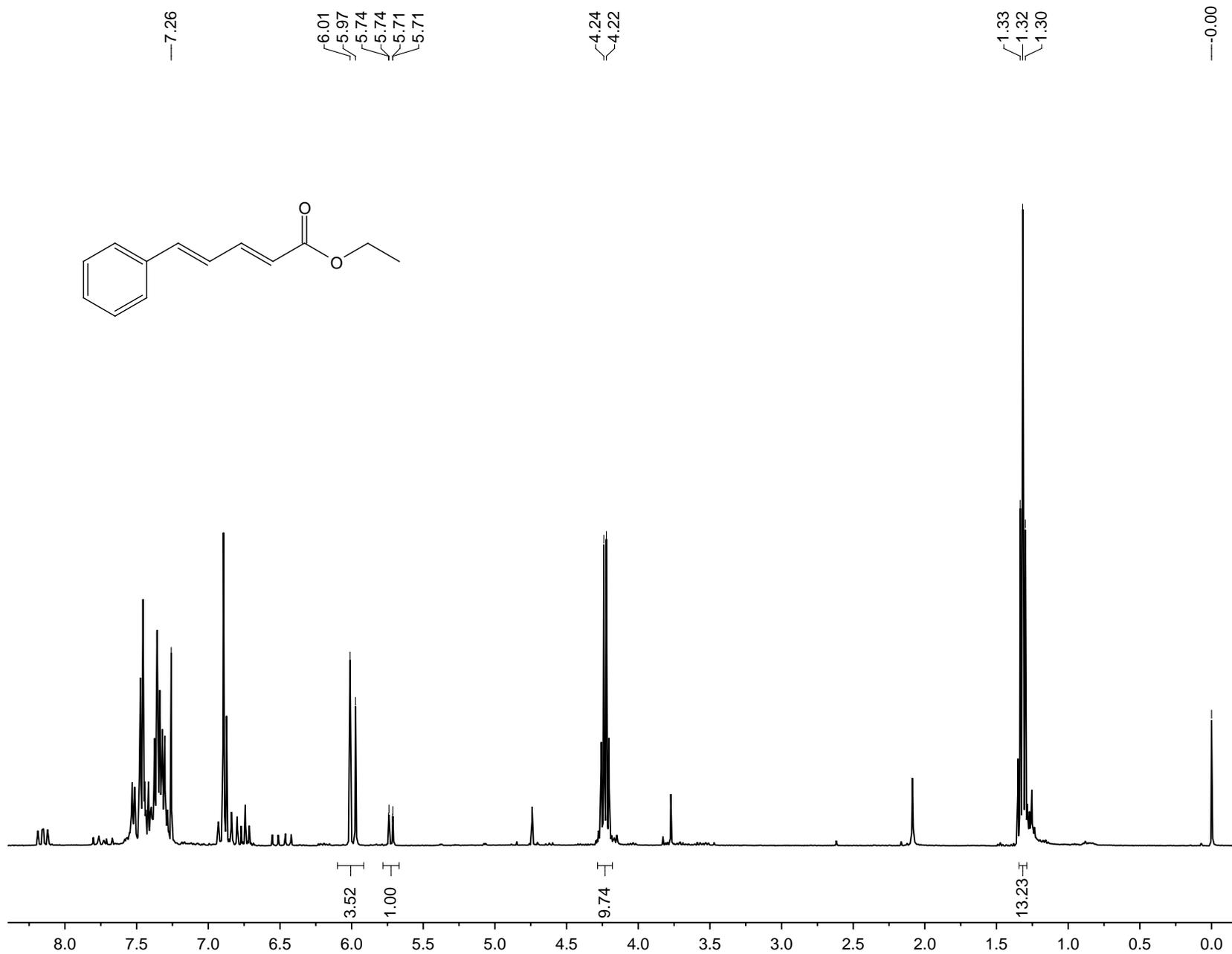
Experimental Conditions:

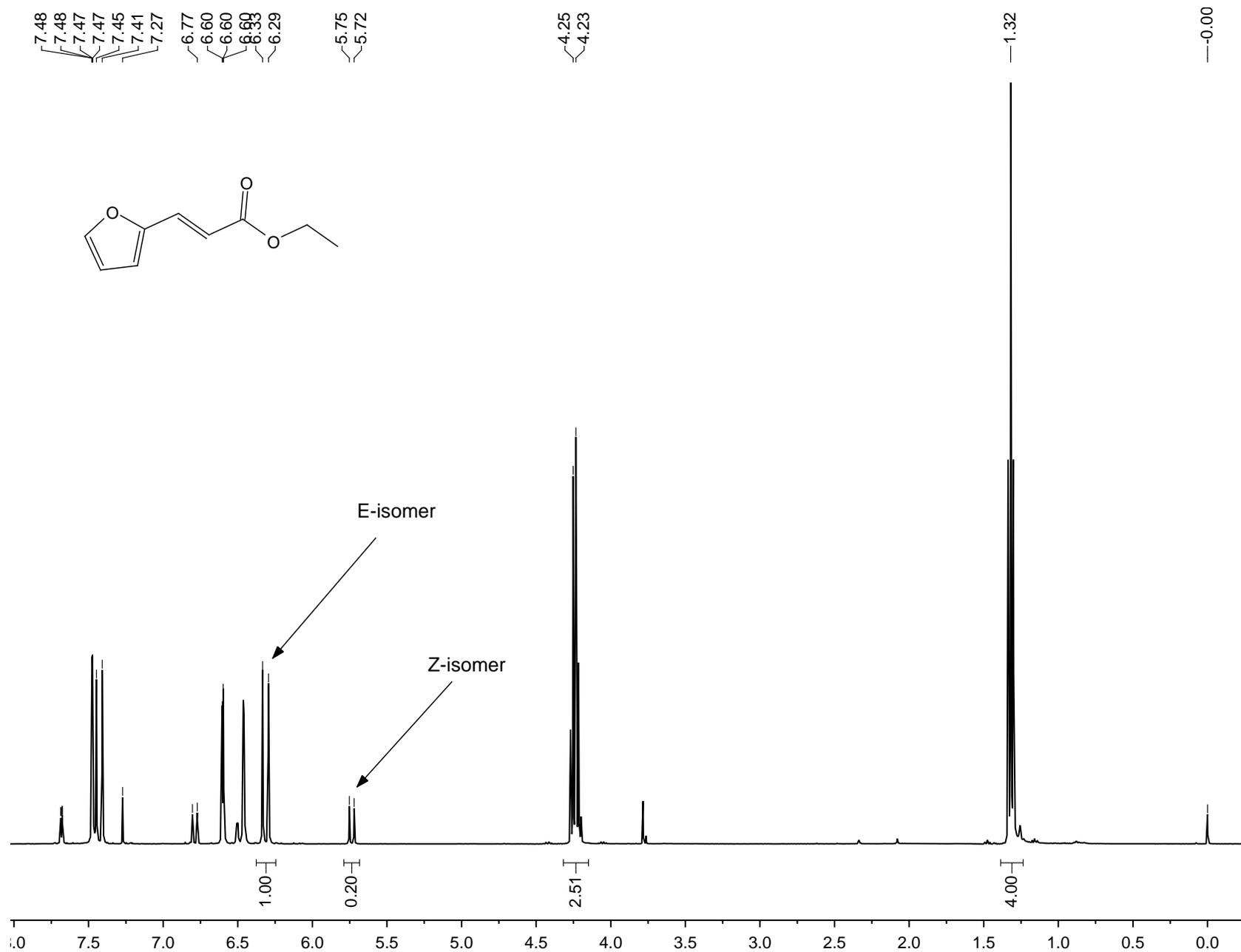
A customized 3.0 mL stainless steel vial was charged with 500 mg (2.24 mmol/g) of triphenylphosphine-functionalized polystyrene (2% cross-linked with DVB, 100 μm diameter), 0.680 mmol of aldehyde, 0.975 mmol of alkyl halide, and 1.30 mmol of potassium carbonate. A 5 mm milling ball was added to the vial with 1 mL of 95% ethanol. The vial was shaken at 18 Hz for 1-2 hours in a Spex8000M mixer mill. The resulting mixture was suspended in approximately 20 mL of ethyl acetate, filtered, and the solvent was removed under reduced pressure affording the pure olefin product. ¹H-NMR and GC-MS were performed to assess the extent and purity of the reaction products.

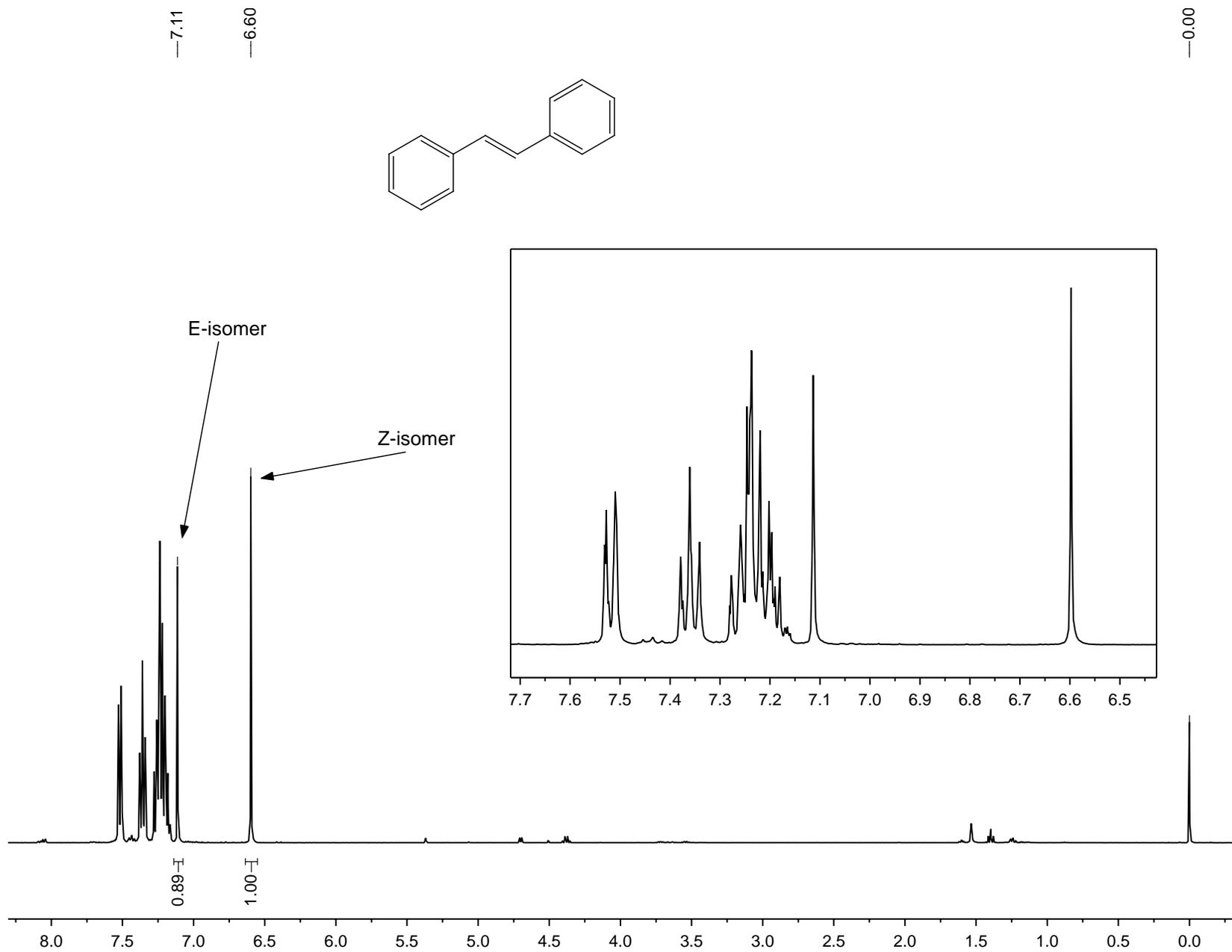


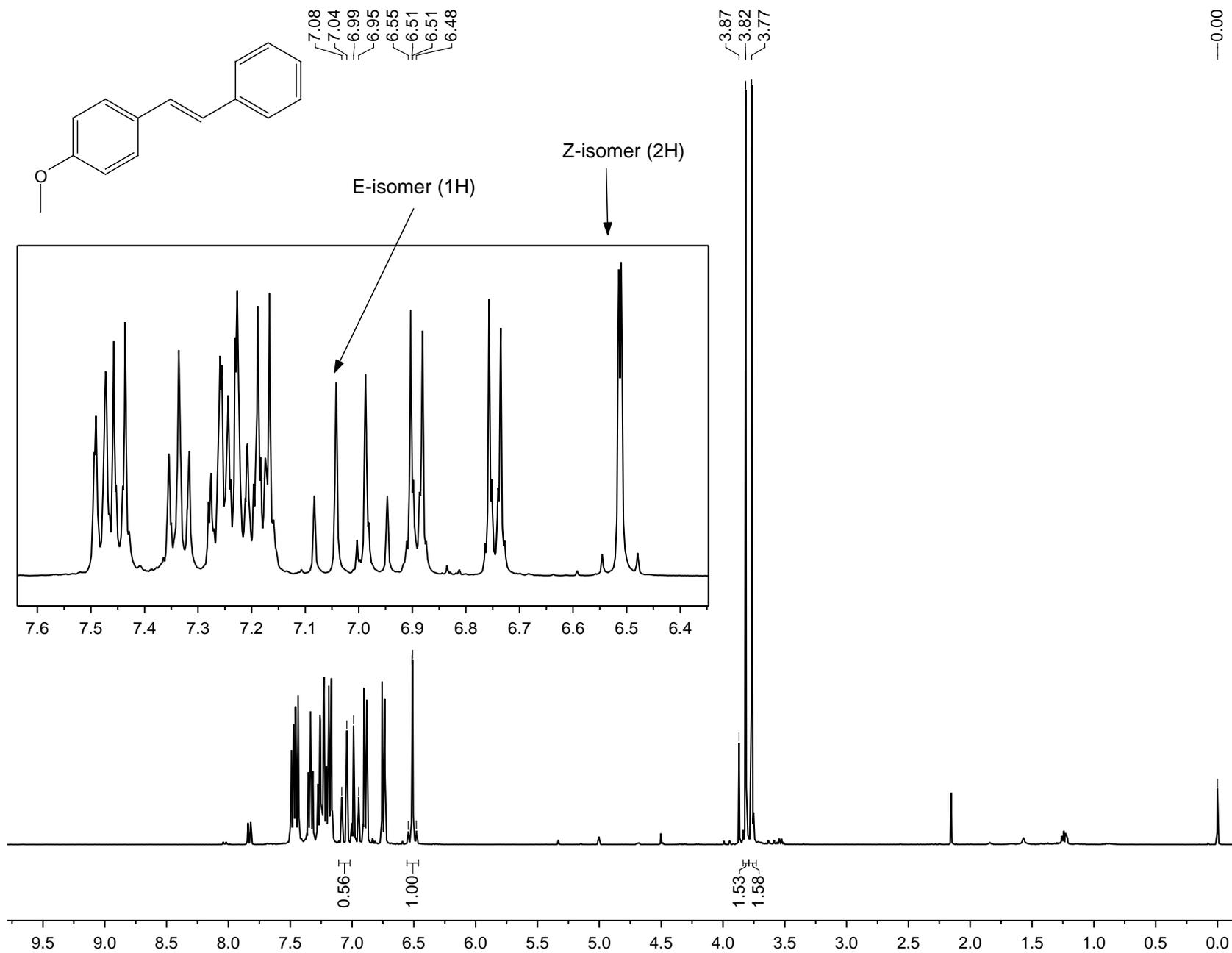




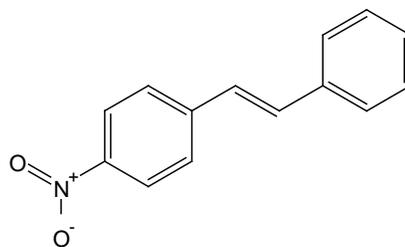




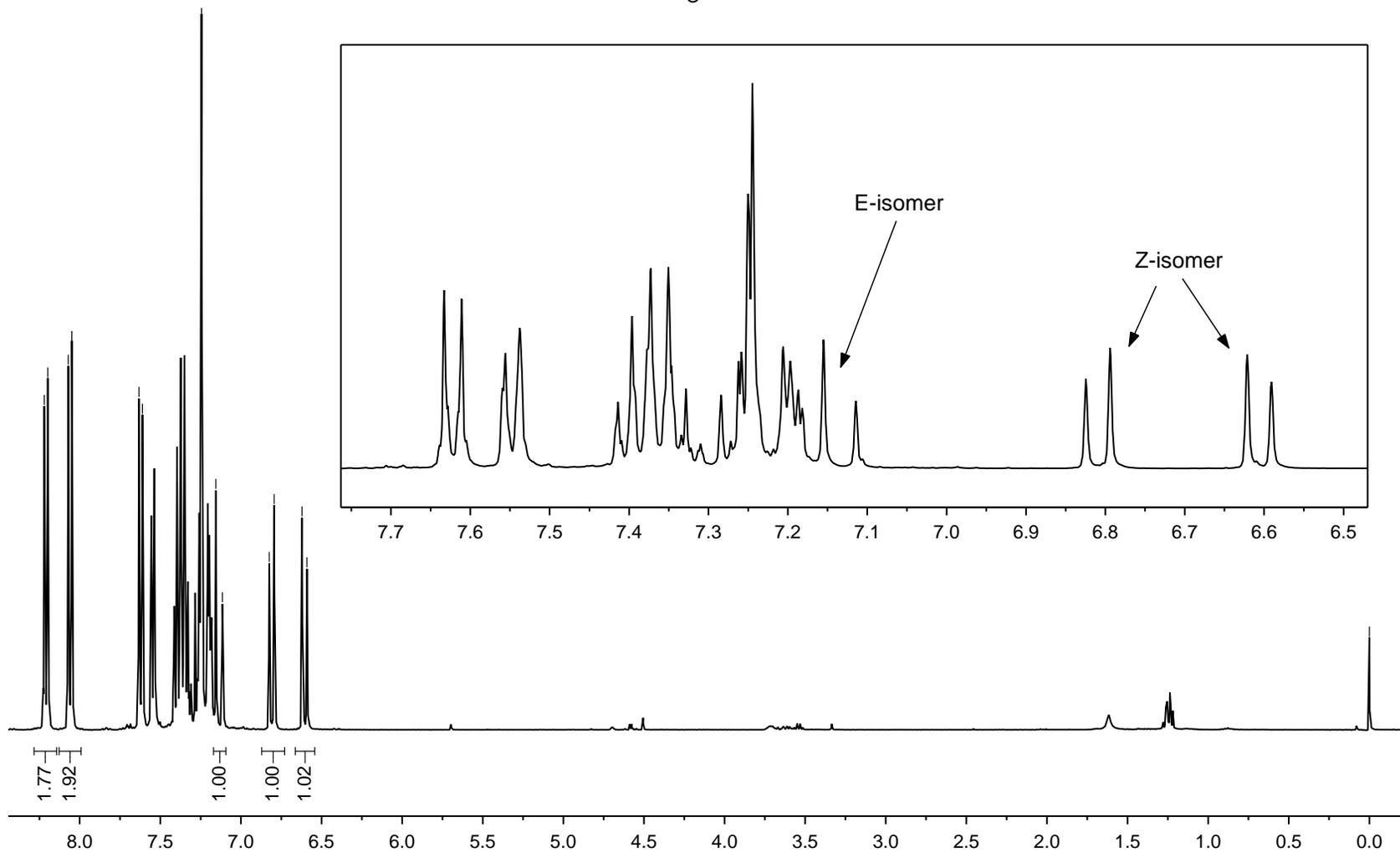




8.22
8.20
8.07
8.05
7.63
7.61
7.24
7.16
7.11
6.82
6.79
6.62
6.59



0.00



S10

