

Supporting Information

Fast Chemical Reaction Work-Up Using Silicone Elastomer-Coated Glass Powders

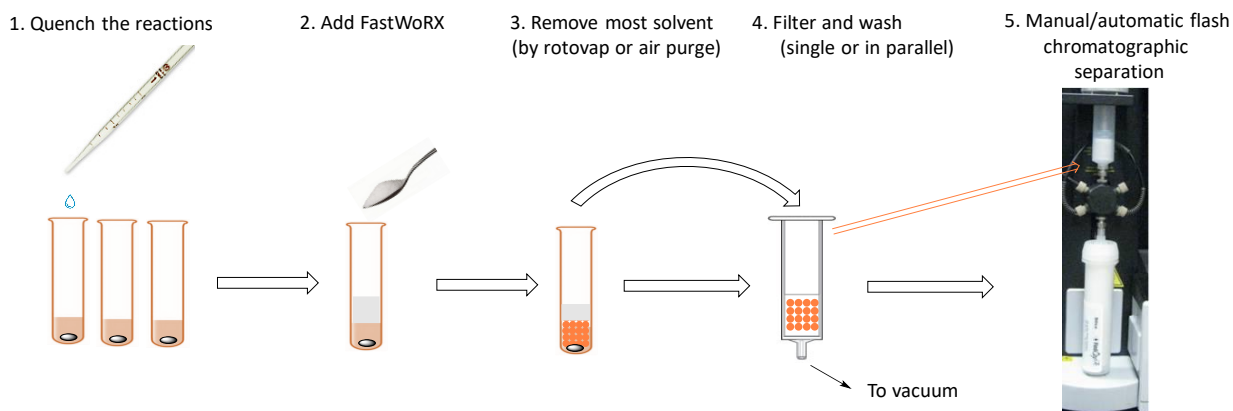
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1. General procedure for FastWoRX-based work-up.

The following is a general procedure for FastWoRX-based work-up.



Step 1: Quench the reactions – The reaction is conducted in the usual way in an organic solvent and is quenched at completion with an appropriate aqueous solution. If quenching of the reaction is not required, FastWoRX powder can be added directly to the sample (go to Step 2). If your products have appreciable water solubility, a saturated solution of a suitable salt such as NaCl or NH_4Cl is recommended for quenching. Also, unlike liquid-liquid extraction, the FastWoRX method works well with just a small amount of quench liquid – around 5 mL of water or brine per gram of organic product usually works. If the reaction was done in a water miscible solvent, an appropriate water immiscible solvent should be added after quenching to form an immiscible organic phase that the FastWoRX powder can better absorb.

Step 2: Add FastWoRX Powder to the Quenched Reaction Mixture – 6 to 10 grams of FastWoRX powder should be added for every gram of the reaction mixture's organic phase (unreacted starting compounds, products, by-products and solvent remaining after evaporation (see Step 3)) you would like to absorb. The absorption of more polar/water soluble compounds can be improved by quenching with a salt solution.

Step 3: Reduce the Solvent Volume – In our experience, the fastest way to do a work-up while using the least amount of FastWoRX powder is to evaporate excess solvent at this step of the process. This can be done by rotovaping or air or inert gas sparging the quenched reaction mixture containing the FastWoRX powder. We recommend evaporating essentially all of solvent (90 - 95%) in this step – there should be no organic liquid visible in the mixture.

Step 4: Filter and Wash the FastWoRX Powder – Filter the FastWoRX powder out with a convenient and appropriate filter. In our experience, if you will be doing flash chromatography later, it is usually

fastest and most efficient to use an appropriate dry loading cartridge or vessel as the filter. Dry the filtered powder by drawing air or inert gas through the powder for about 2 minutes to remove any remaining aqueous phase. Use water (or brine for more water-soluble compounds) or acid or base solutions as appropriate to wash away reactants, salts, catalysts and by-products from the filtered powder (the approximate volume of wash solution in mLs is numerically equal to the weight in grams of FastWoRX powder added in Step 2). If using acid or base washes, finish with water washes to remove any residual acid or base (the approximate volume of wash solution in mLs is numerically equal to the weight in grams of FastWoRX powder added in Step 2). If a target compound is highly water soluble, a minimal amount of a saturated inorganic salt solution (like saturated NaCl solution) should be used for the washing step – the use of salt will significantly reduce the solubility of the target during washing. Use vacuum or an air or inert gas stream to evaporate any solvent remaining in the powder, leaving only the target compound(s) in the FastWoRX powder.

Step 5: Chromatography separation. Elute the target(s) from the FastWoRX powder with a suitable organic solvent. The powder can be loaded into a commercial flash chromatography system or elute the target(s) manually with your desired solvent.

2. Copies of NMR spectra

Note: In the purification of compounds **3** and **14**, the hexane solvents used for chromatography were of relatively low quality (containing high boiling point residues). The peaks in the 1.0 - 1.5 ppm range are from these residues.

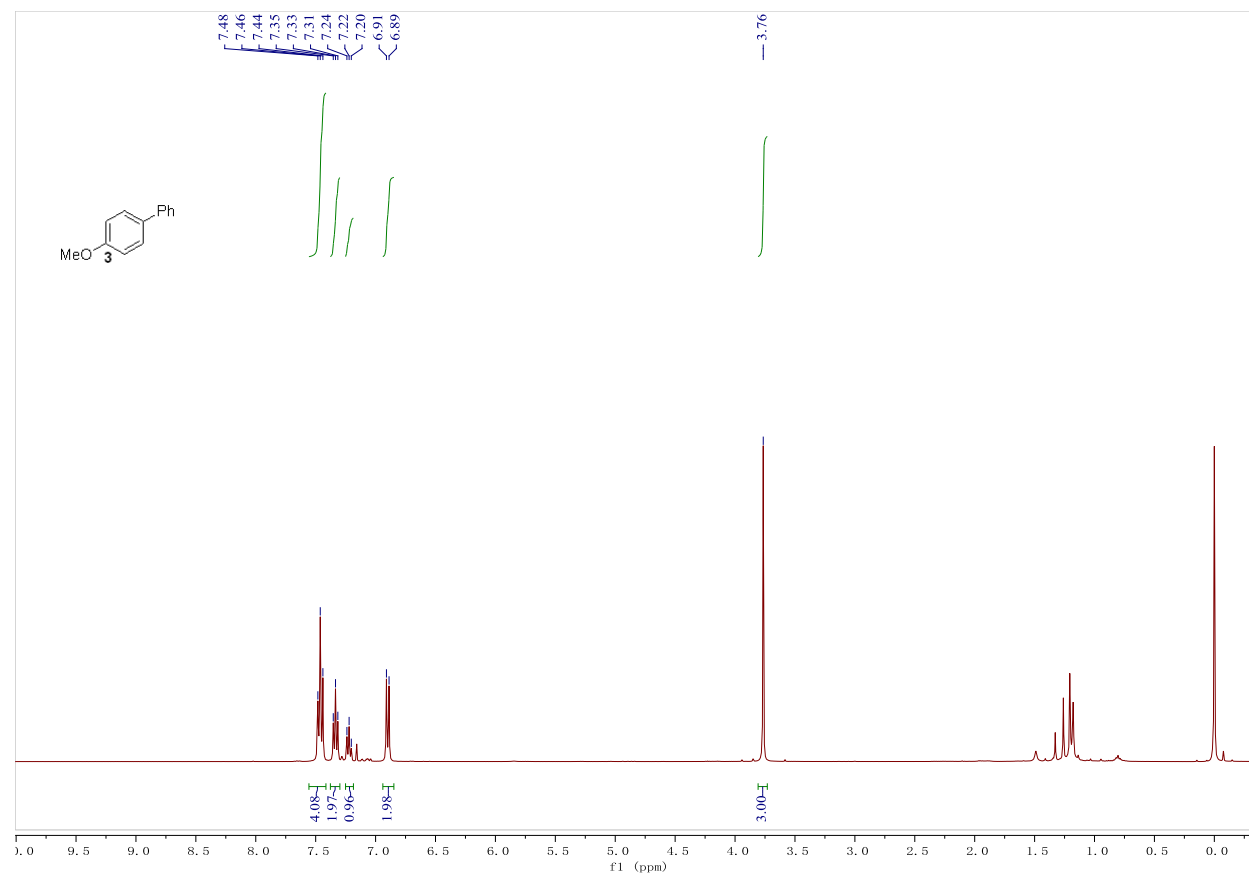


Figure S 1

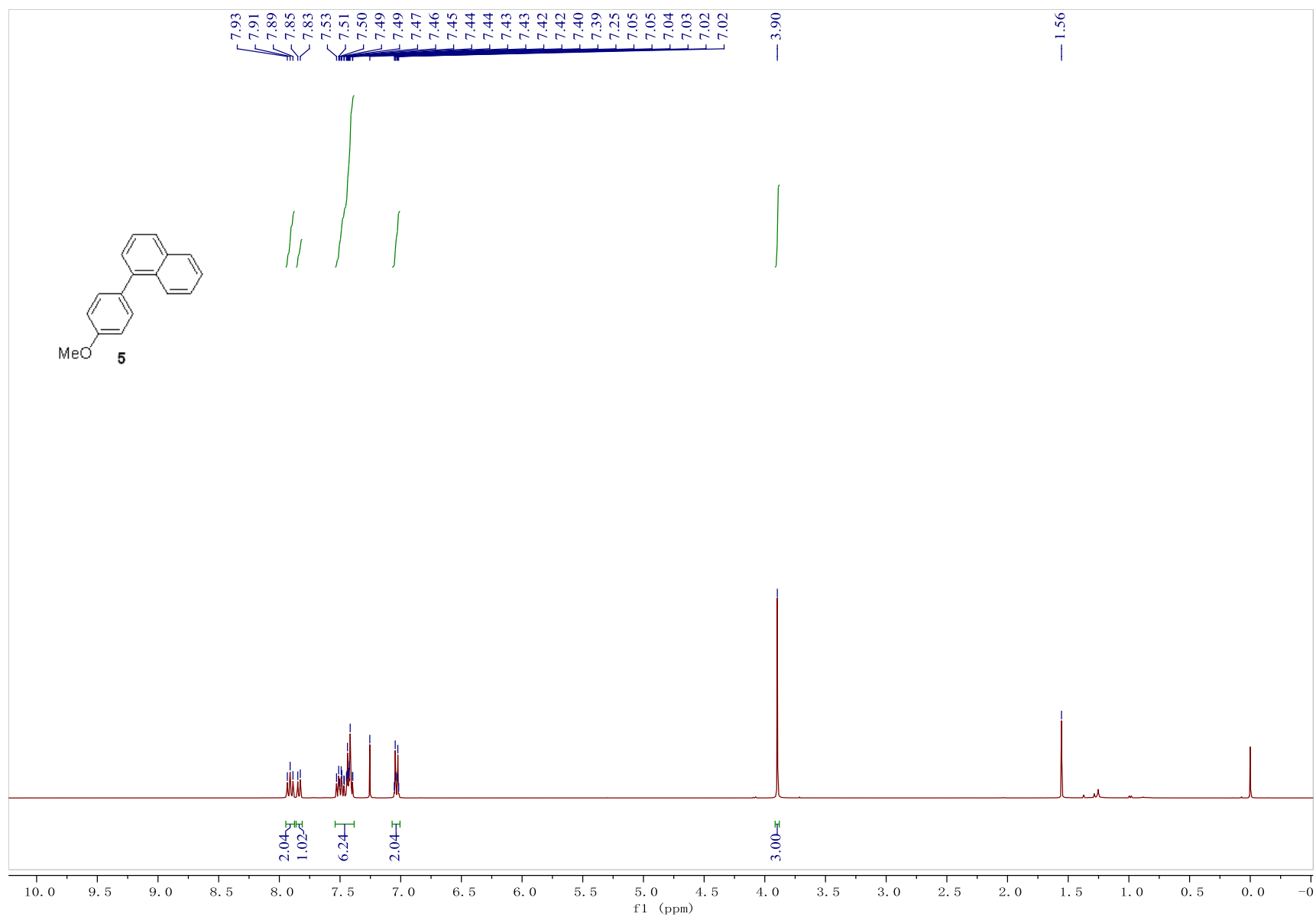


Figure S2

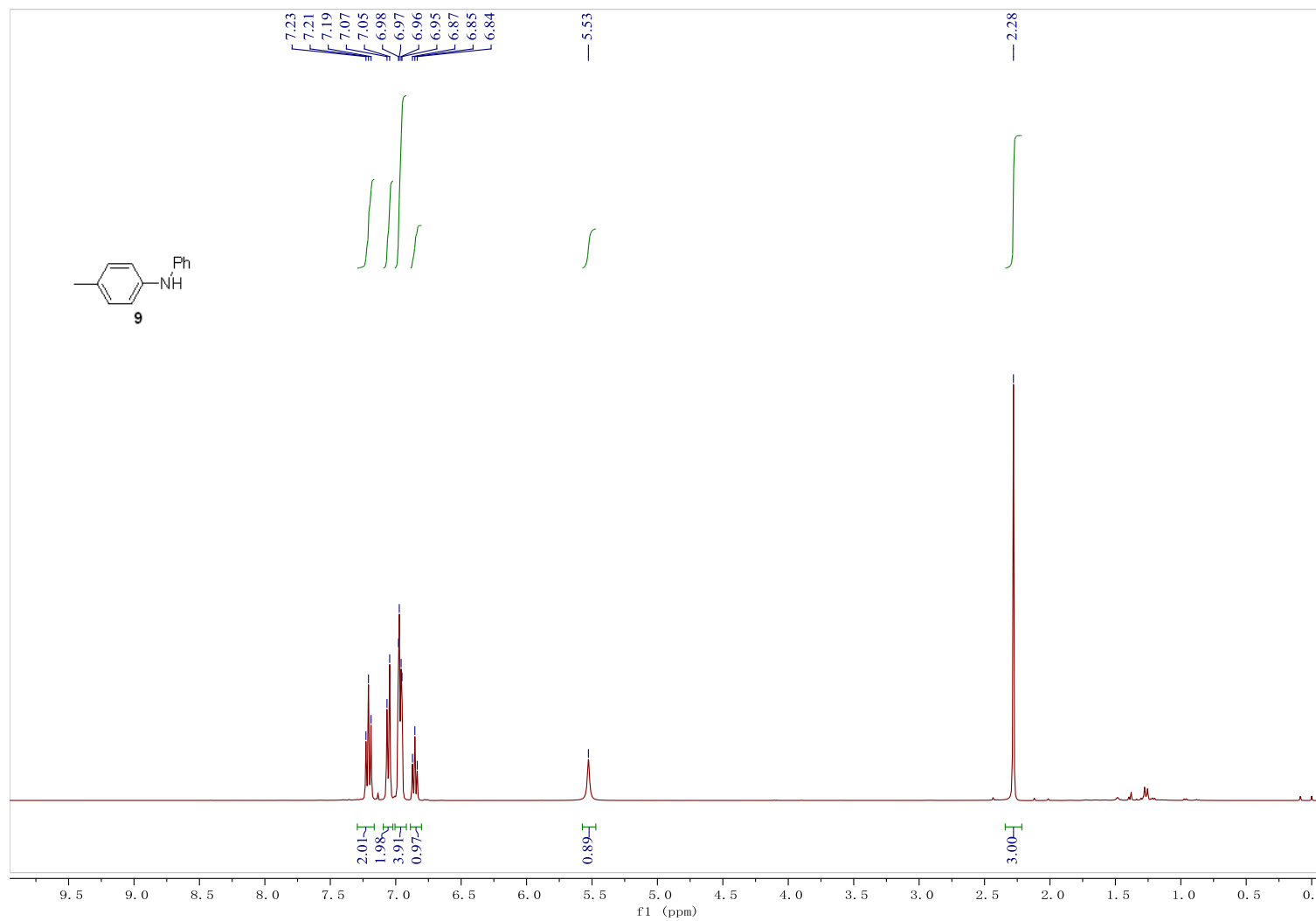


Figure S3

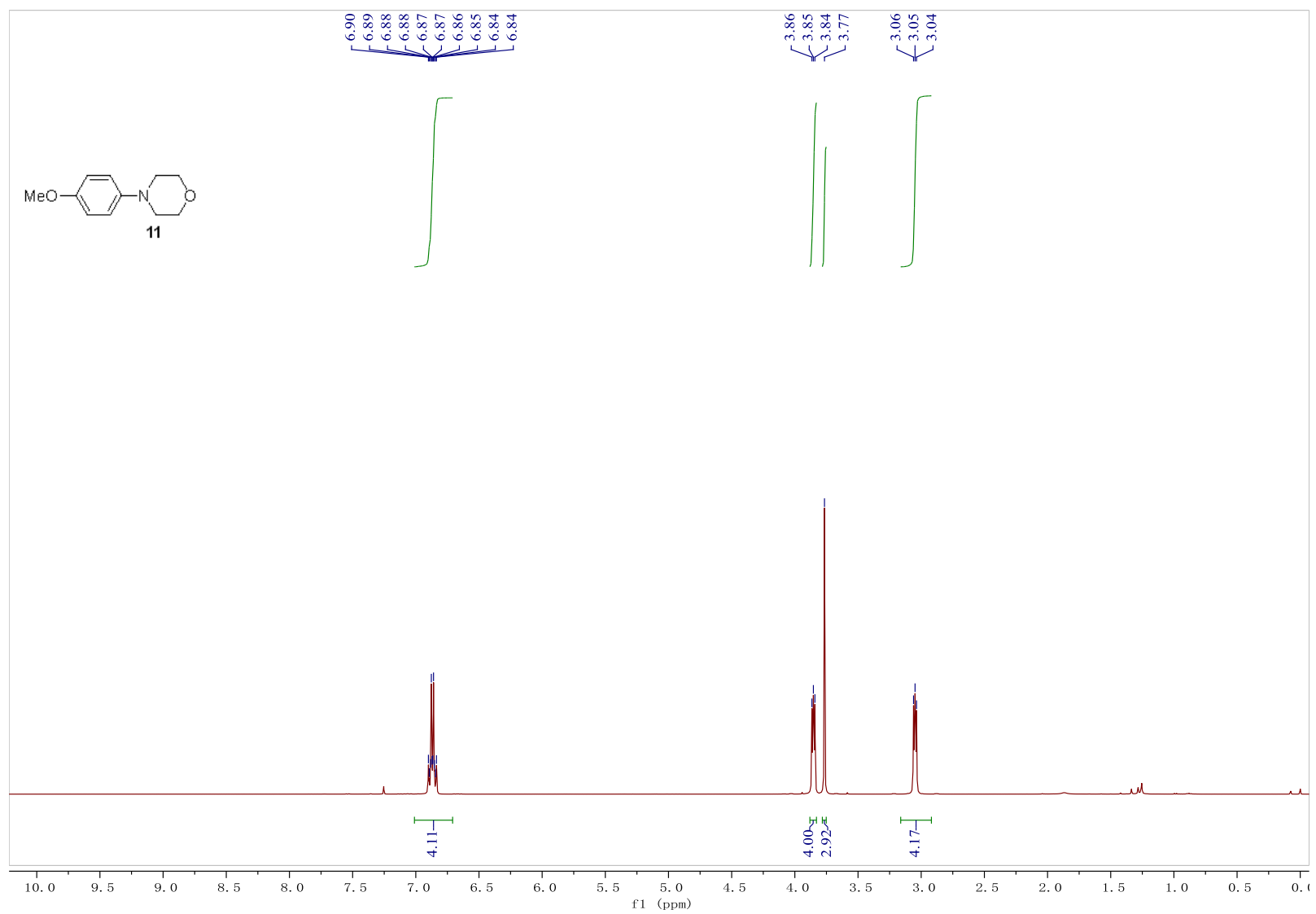


Figure S4

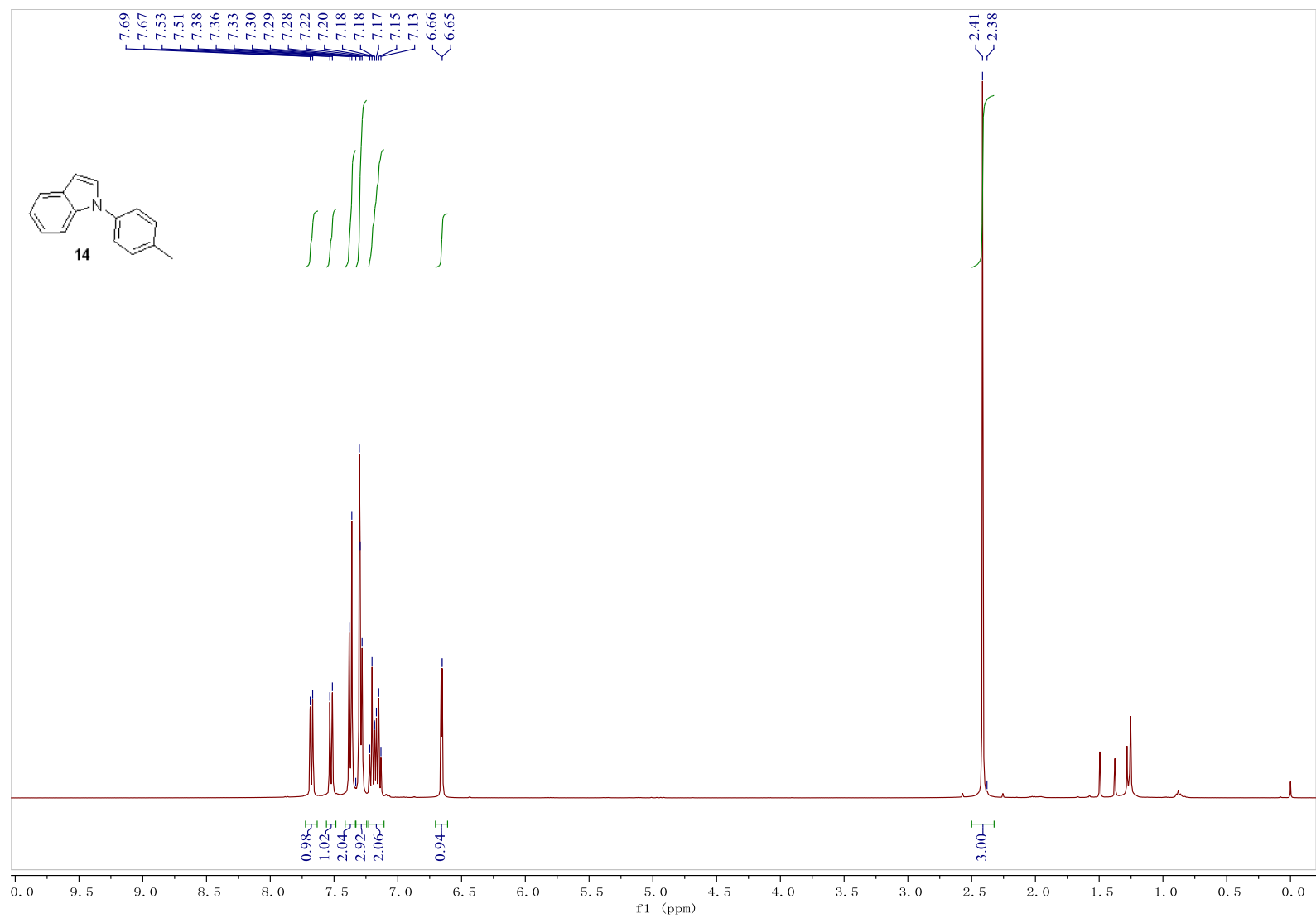


Figure S 5

Figure S6

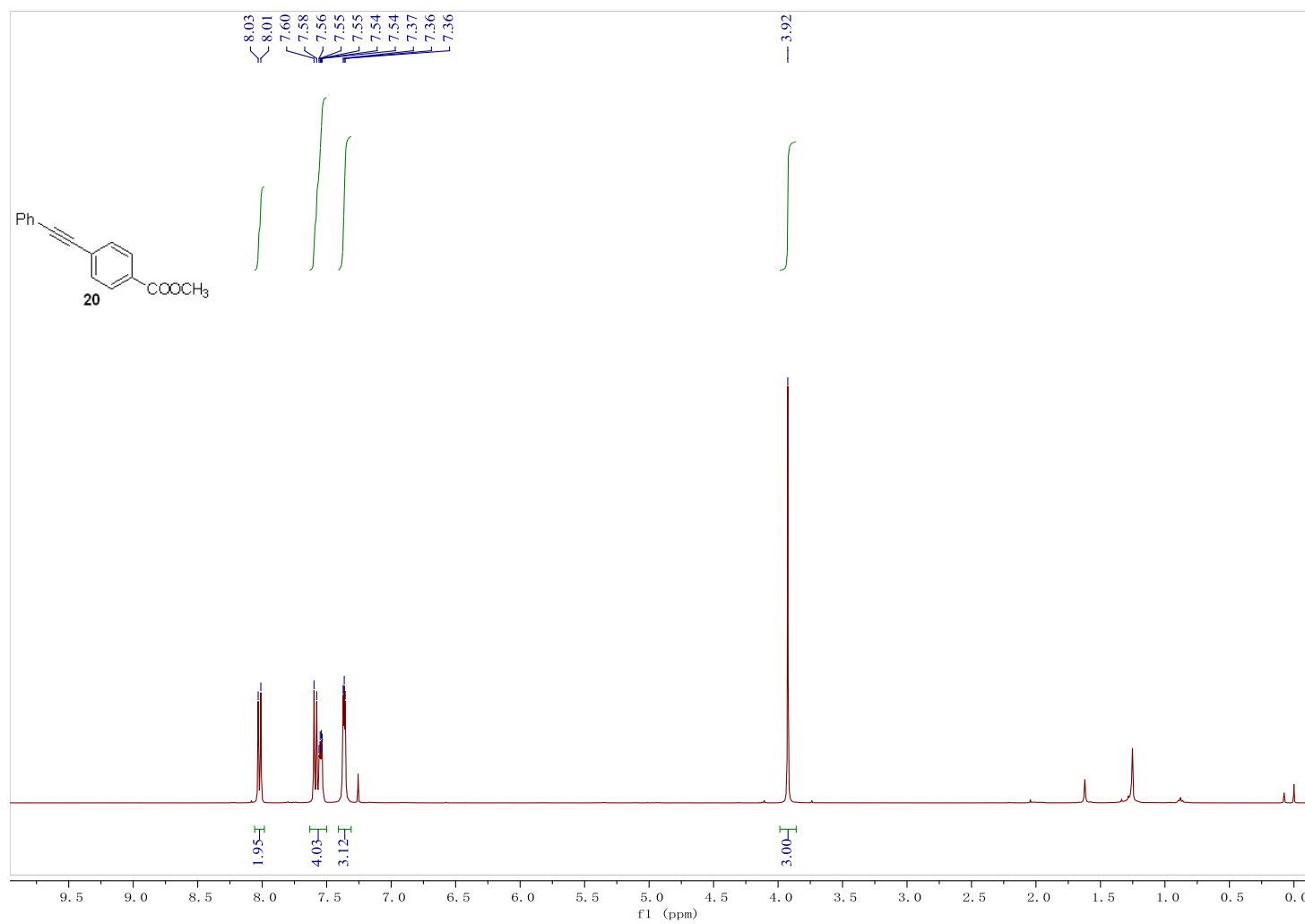


Figure S7

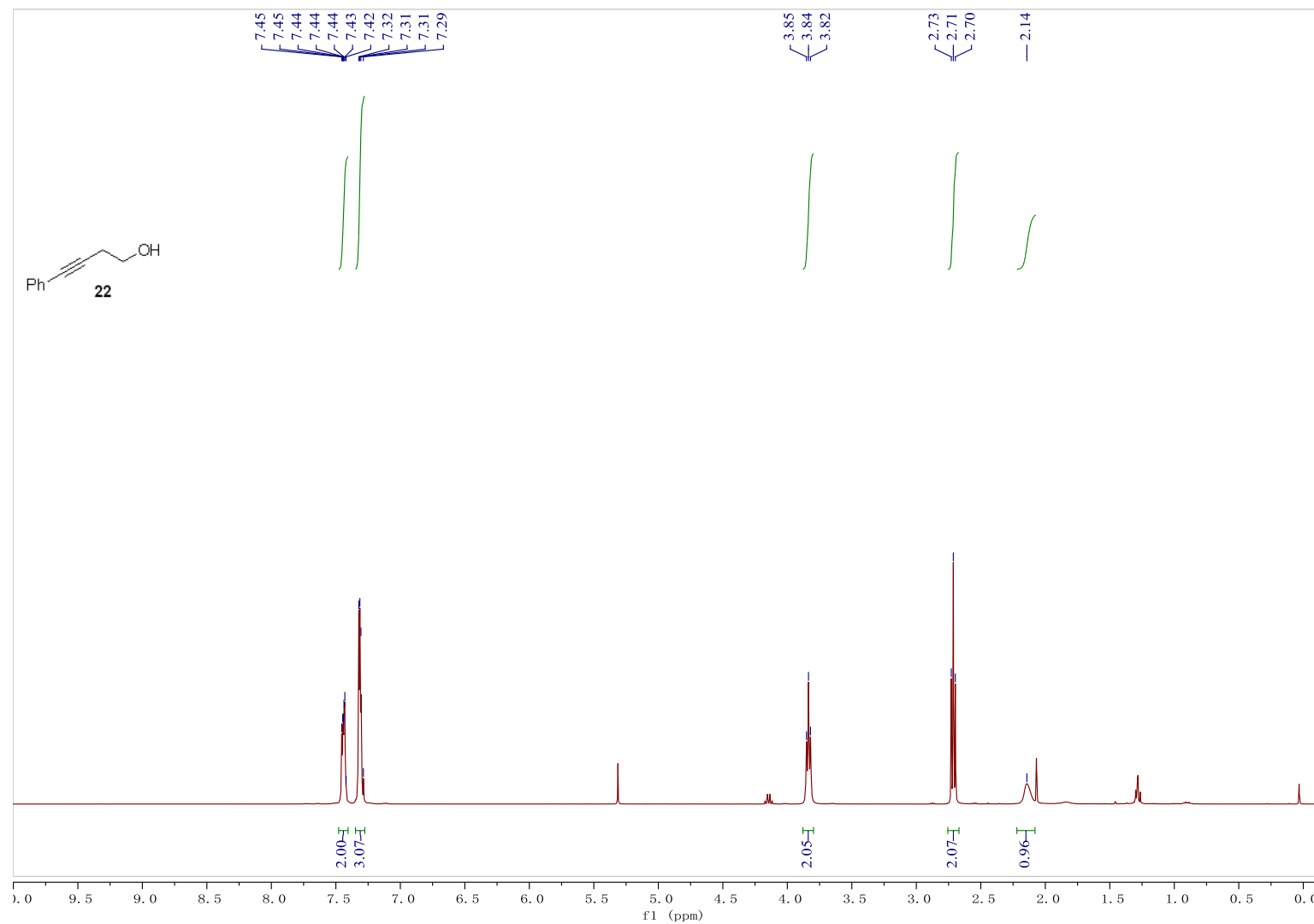


Figure S8

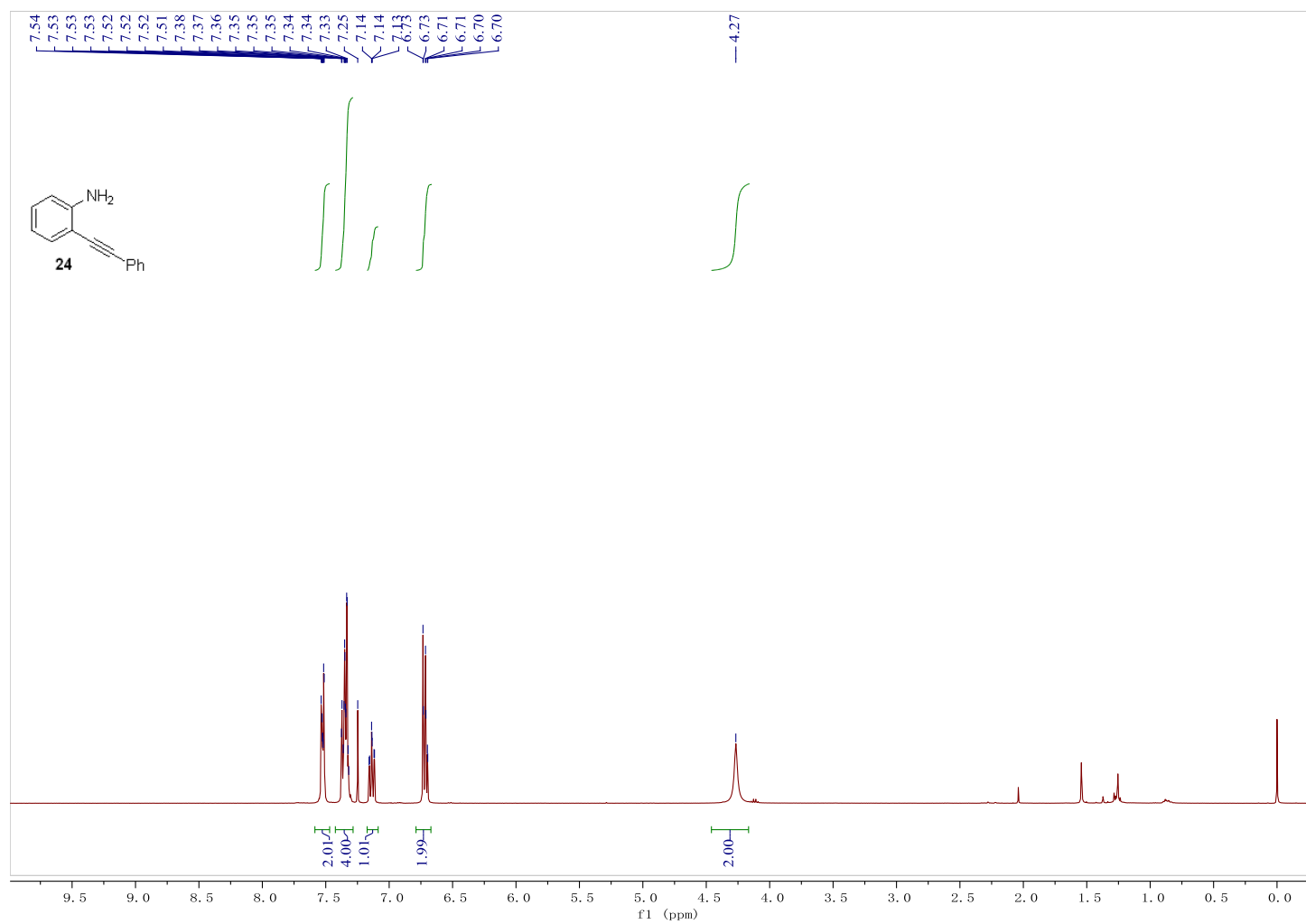


Figure S9

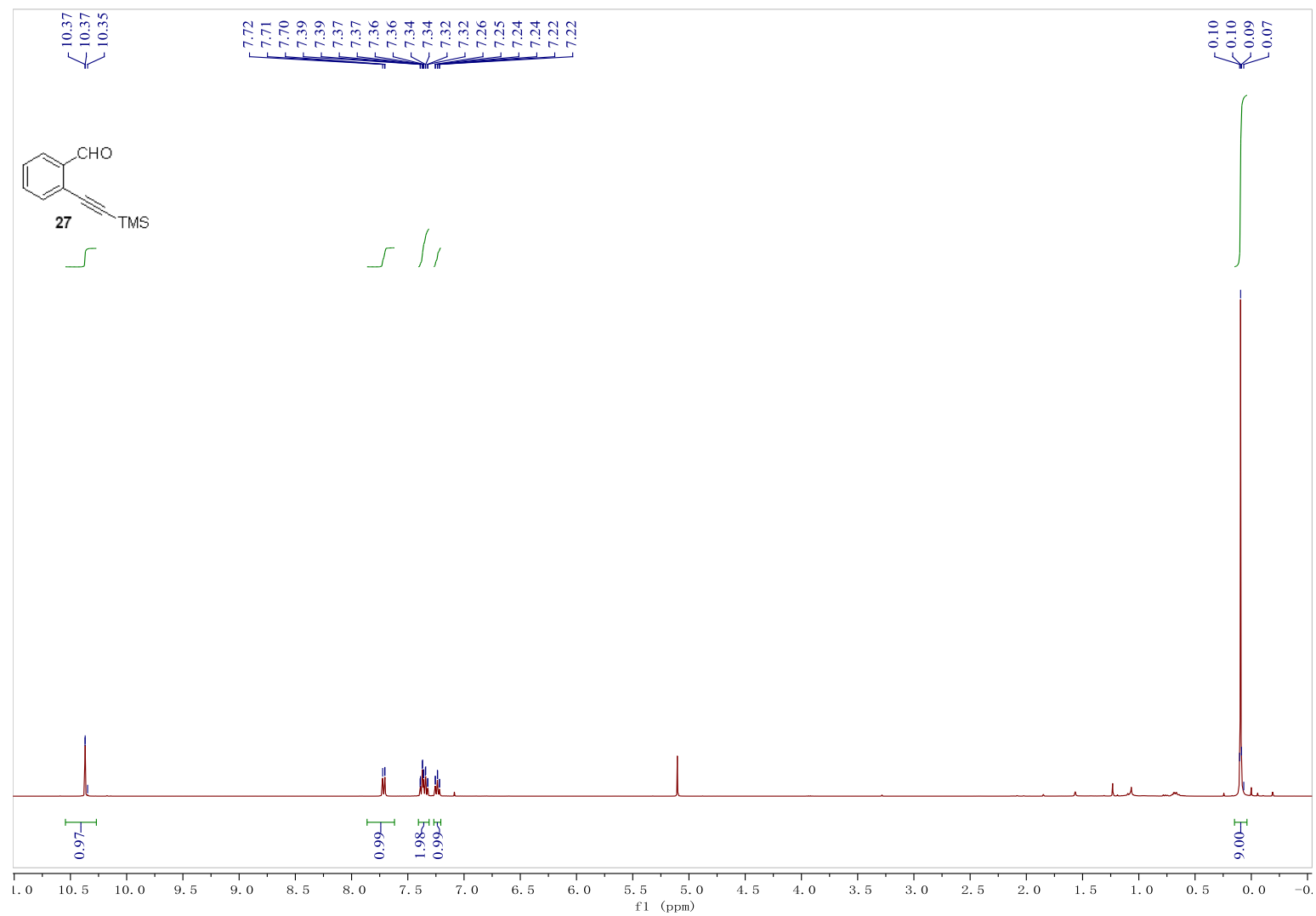


Figure S10

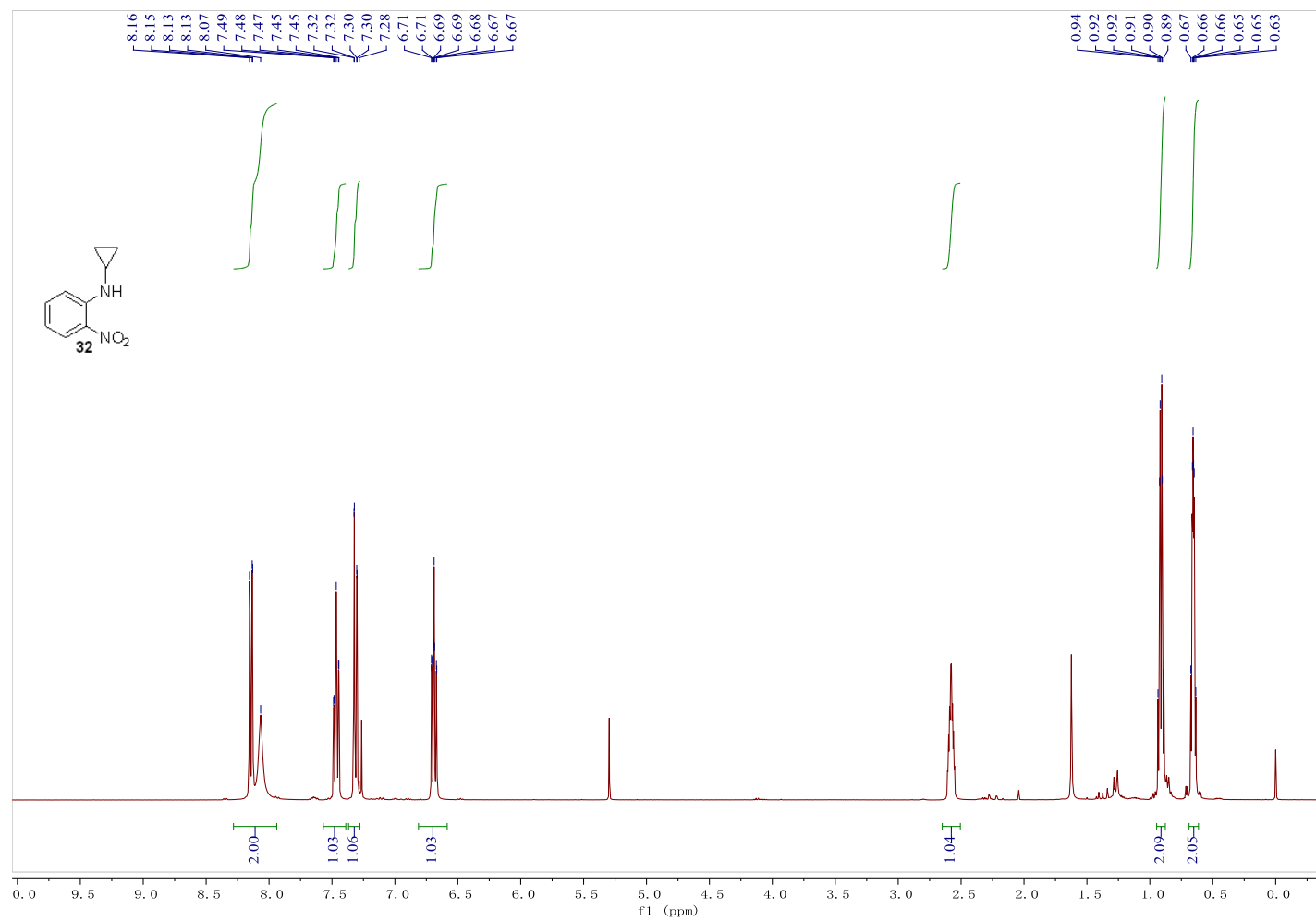


Figure S11

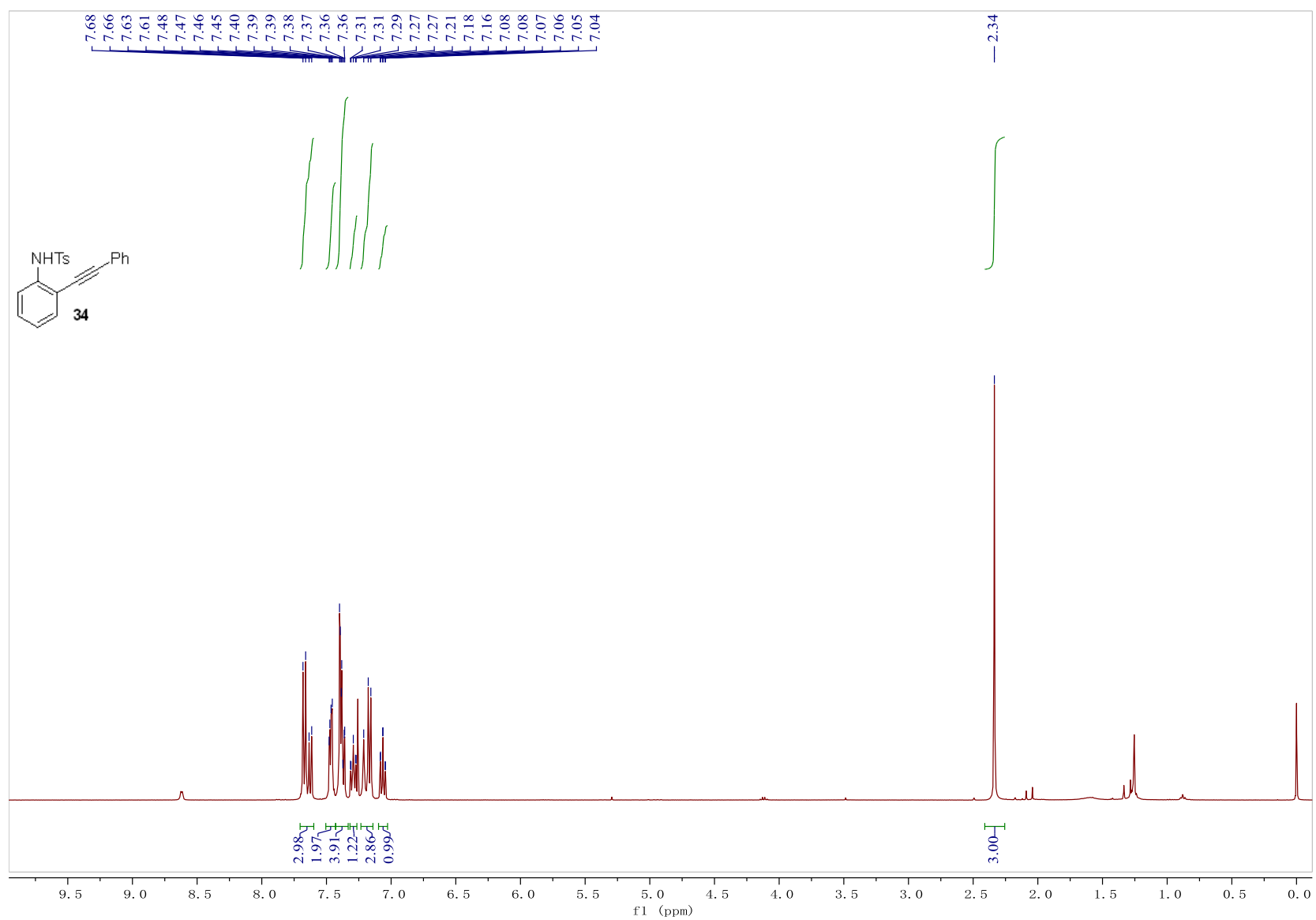


Figure S 12

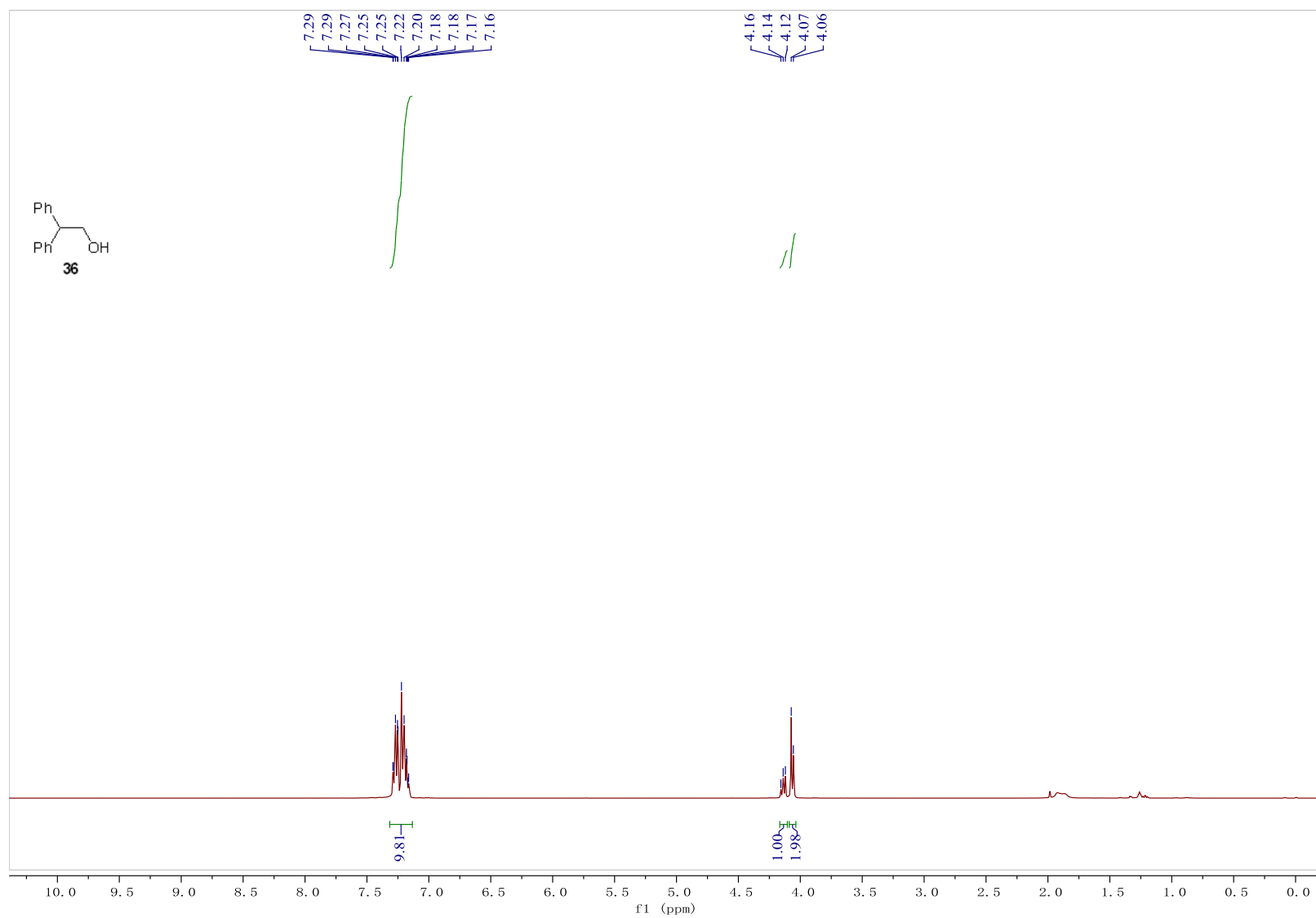


Figure S13

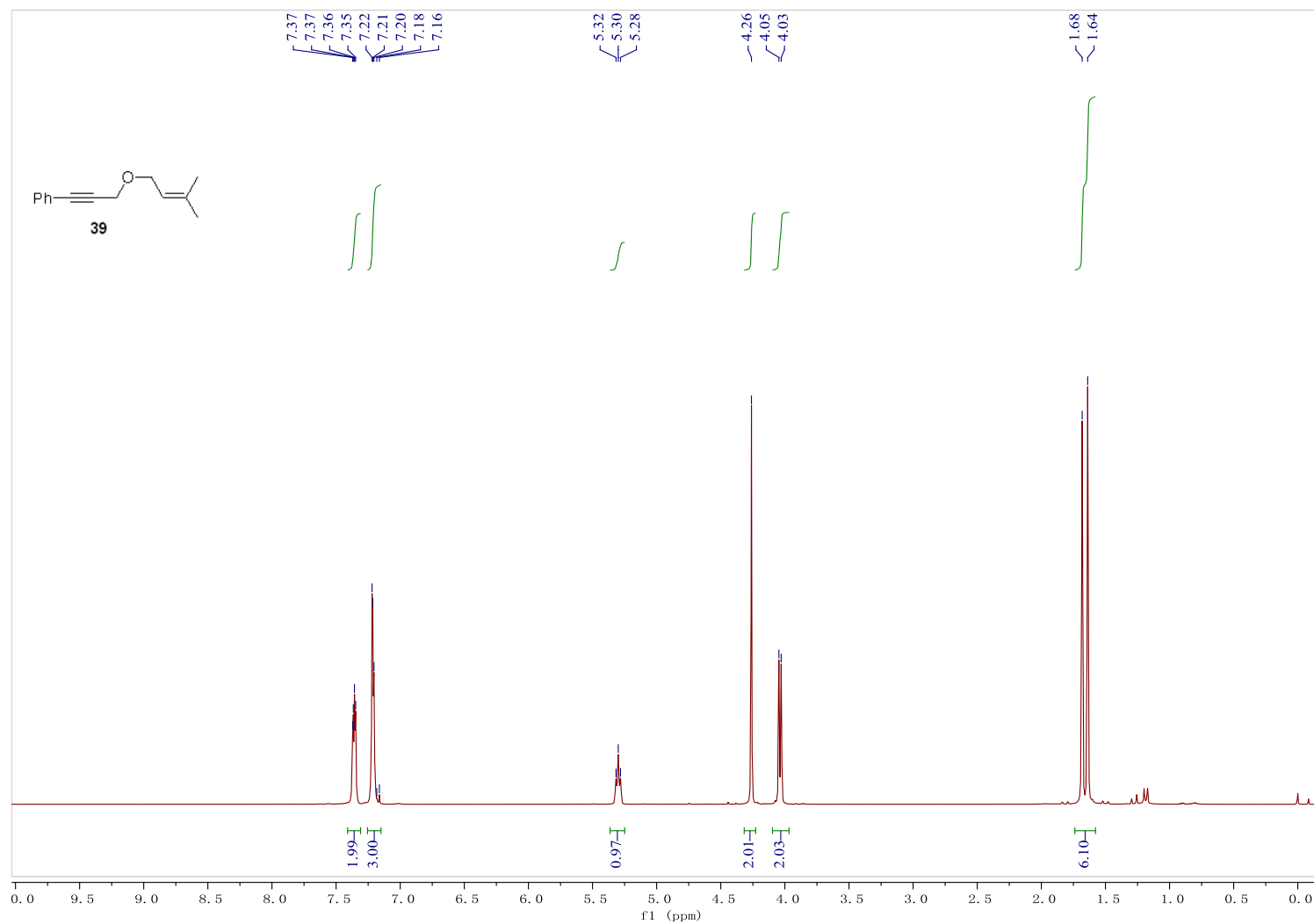


Figure S 14

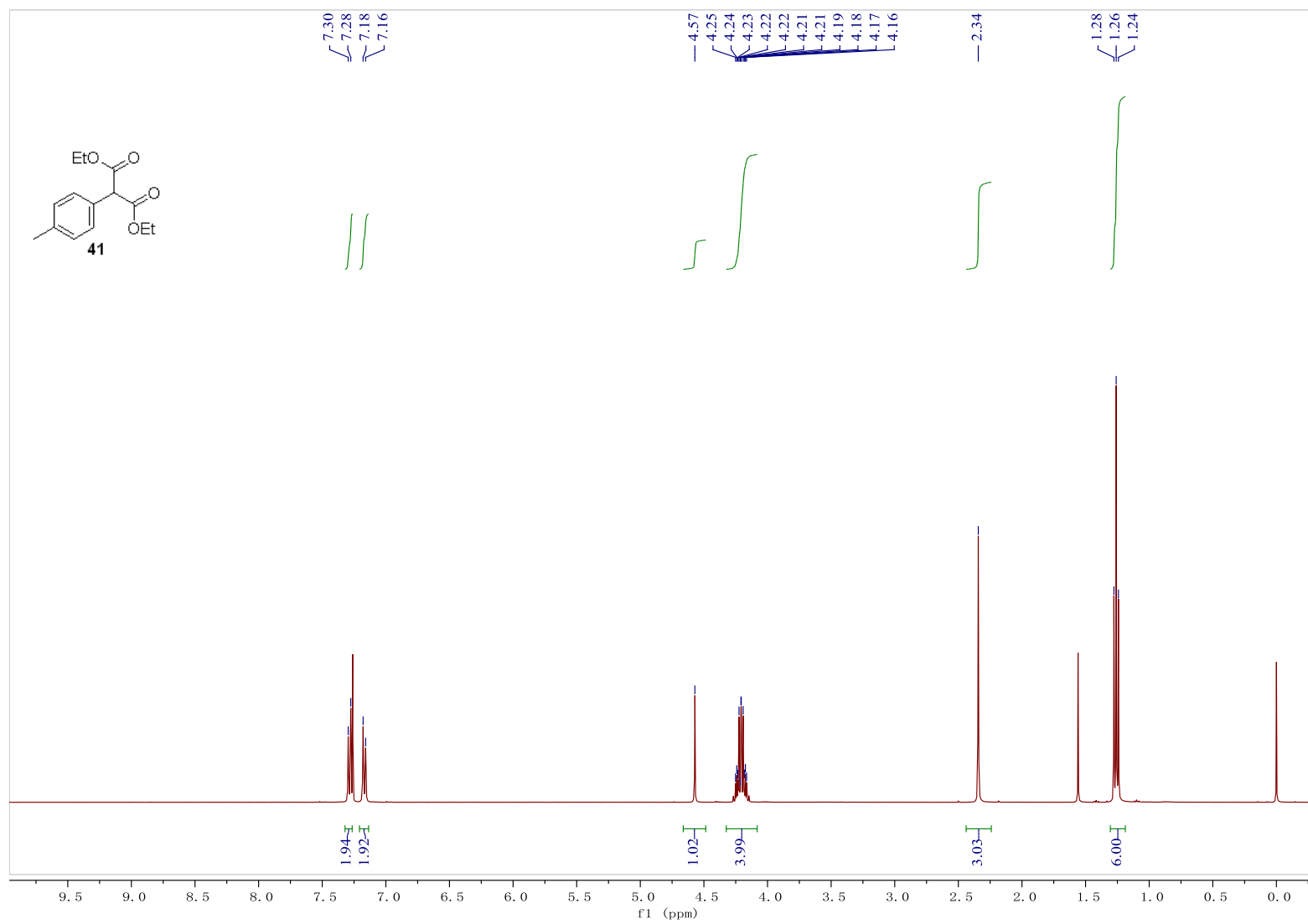


Figure S 15