

Supporting Information

Preparation of Glycosyl Carboxylic Acids via Stereoselective Synthesis and Oxidative Cleavage of C-Vinyl Glycosides: An Improved Cyanide-Free Strategy

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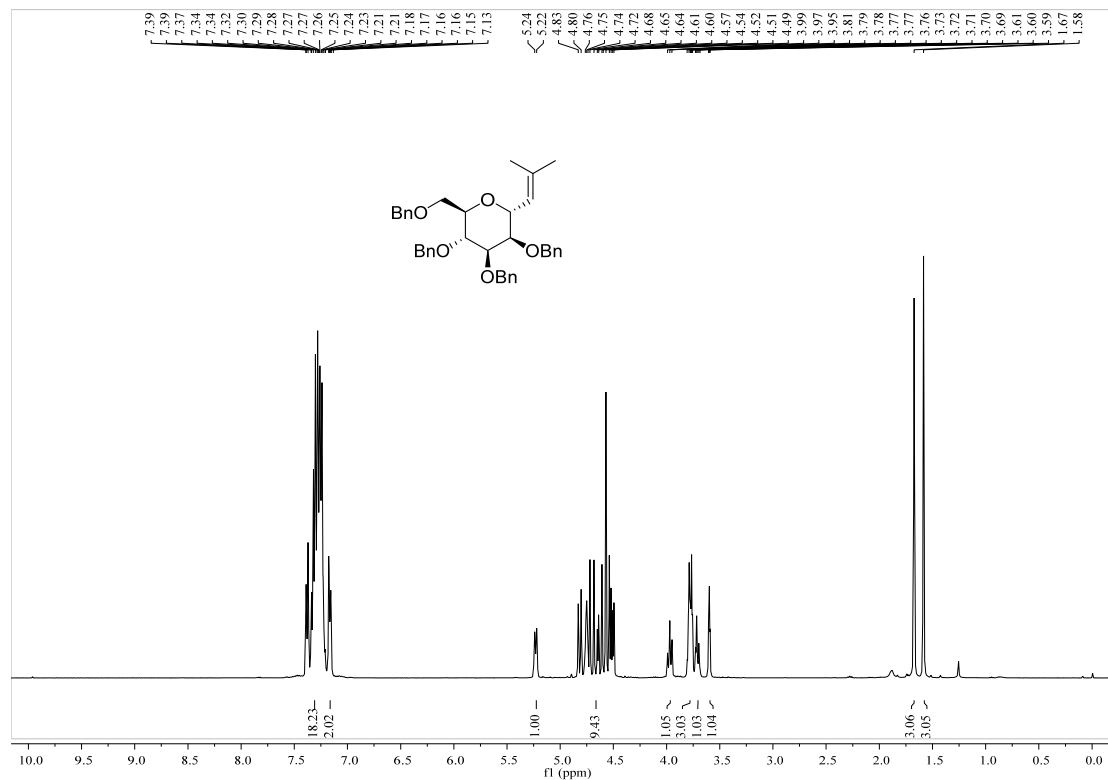
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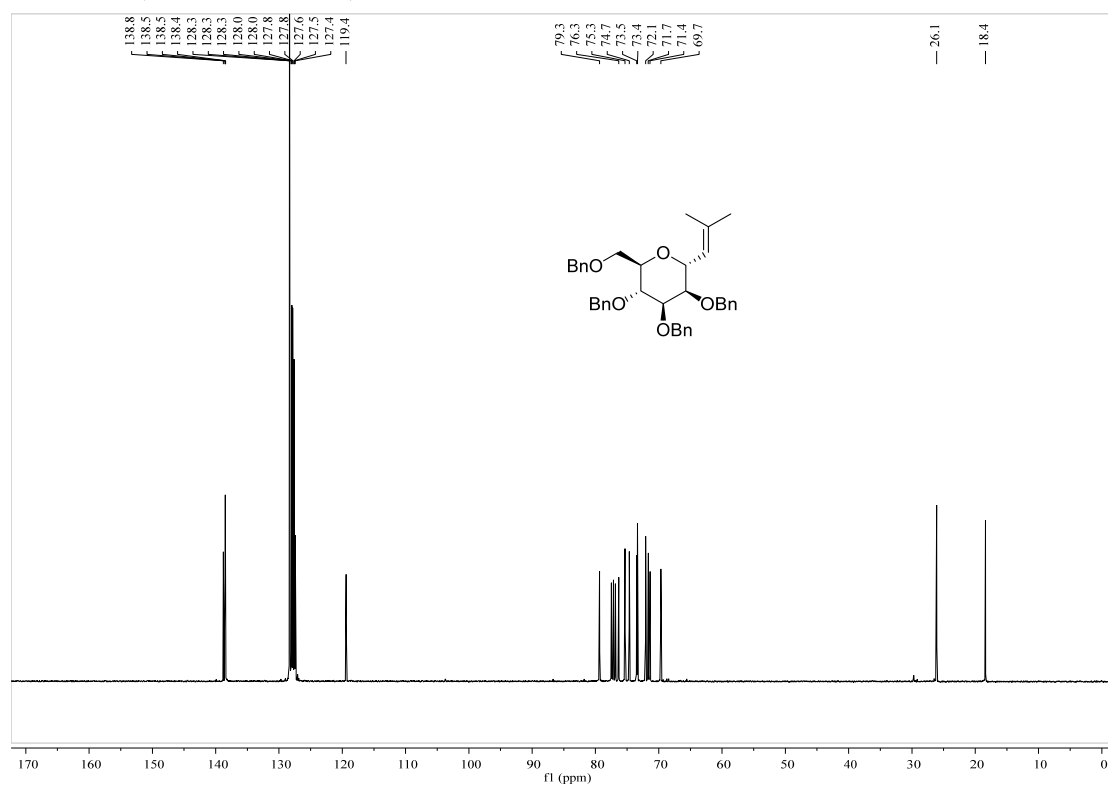
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Copies of NMR Spectra

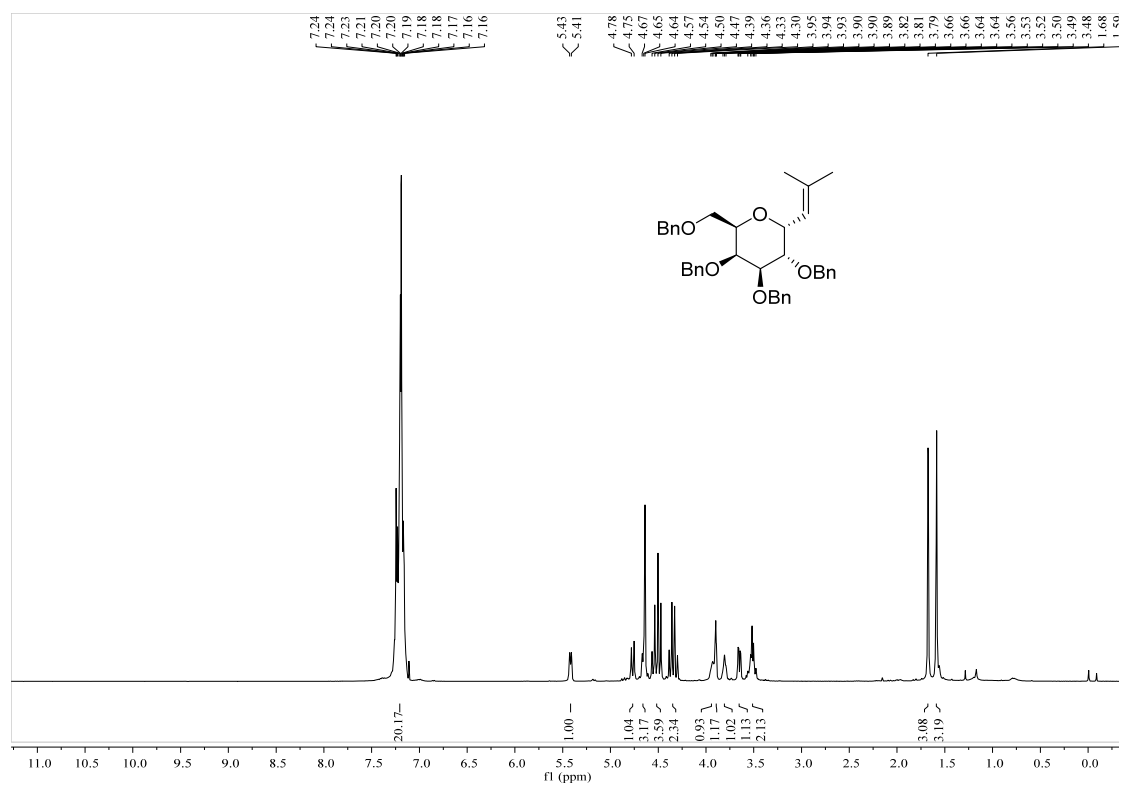
¹H NMR (400 MHz, CDCl₃) of 4d



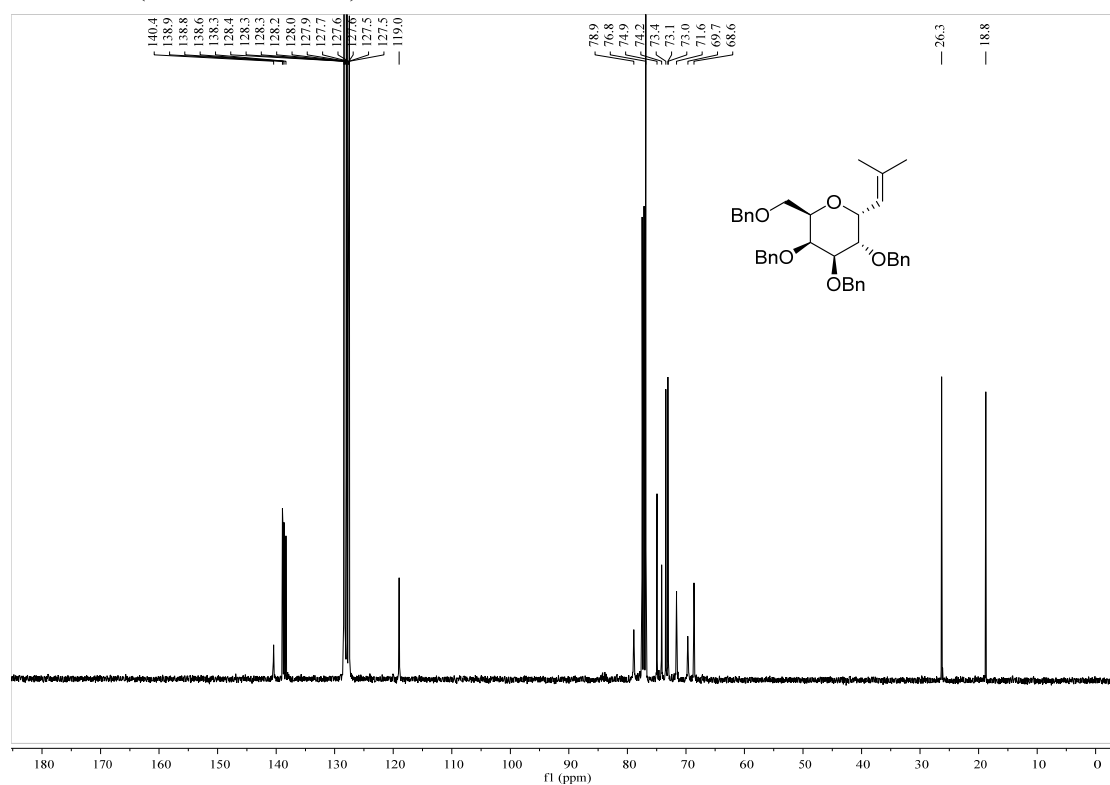
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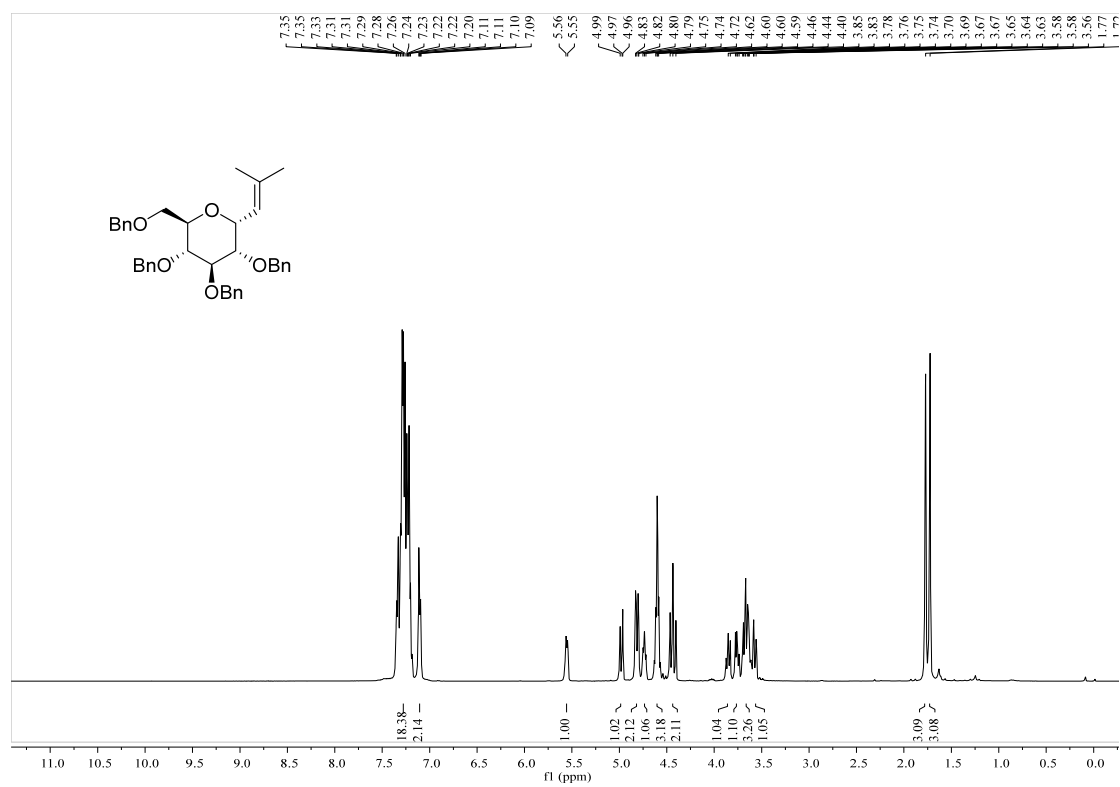
¹H NMR (400 MHz, CDCl₃) of 4e



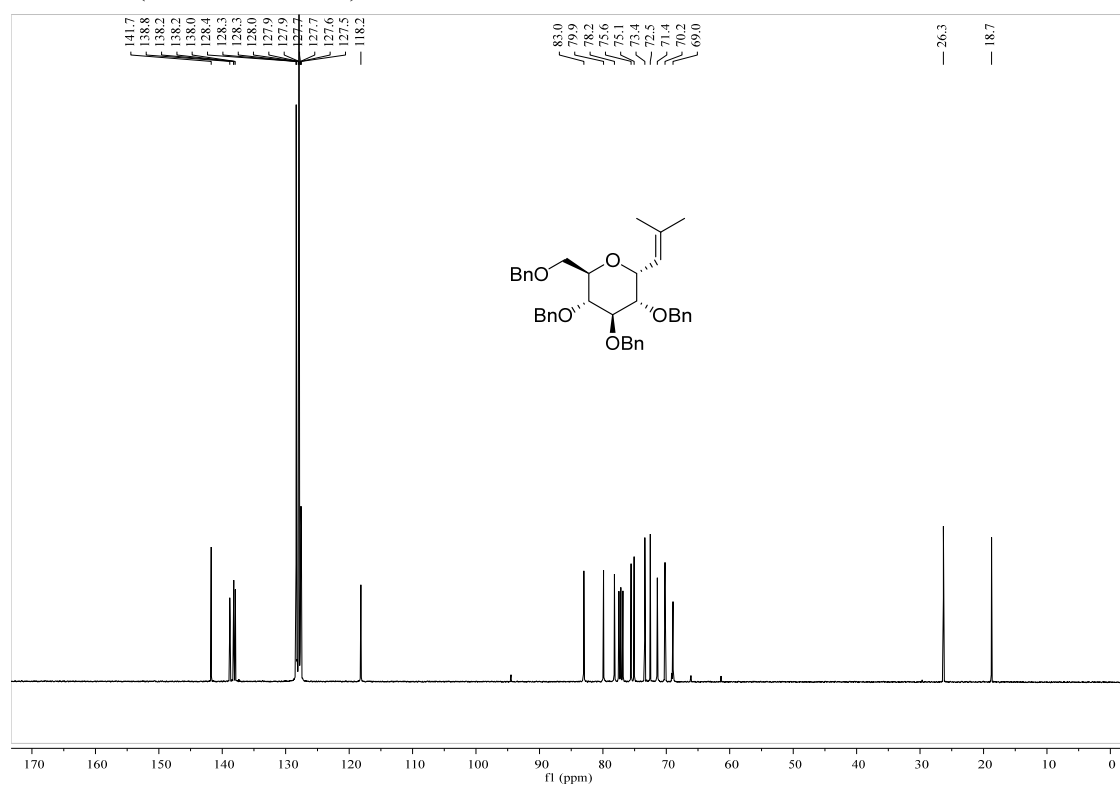
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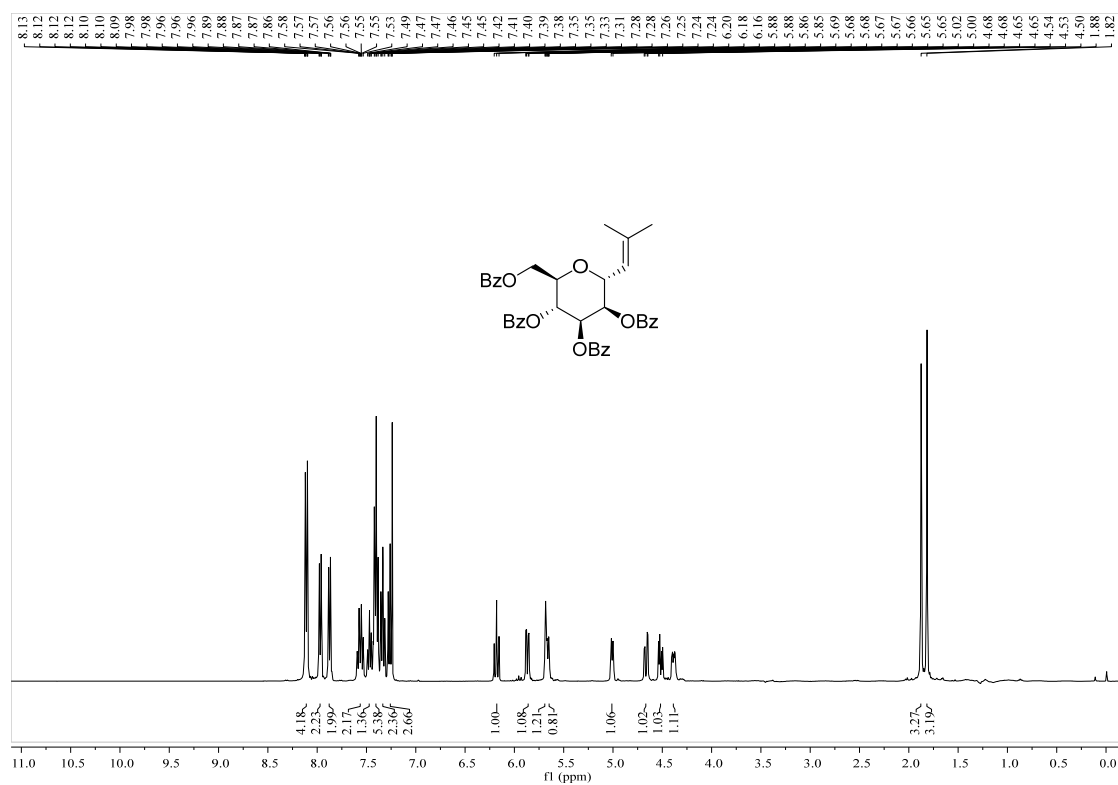
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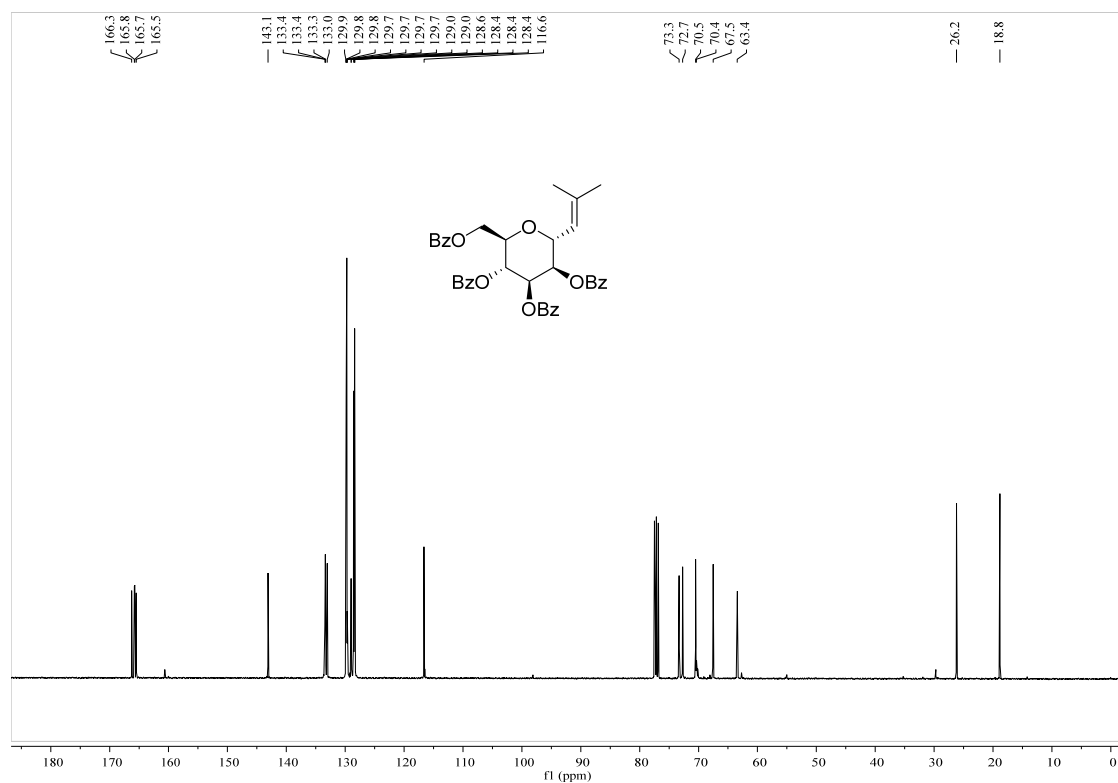
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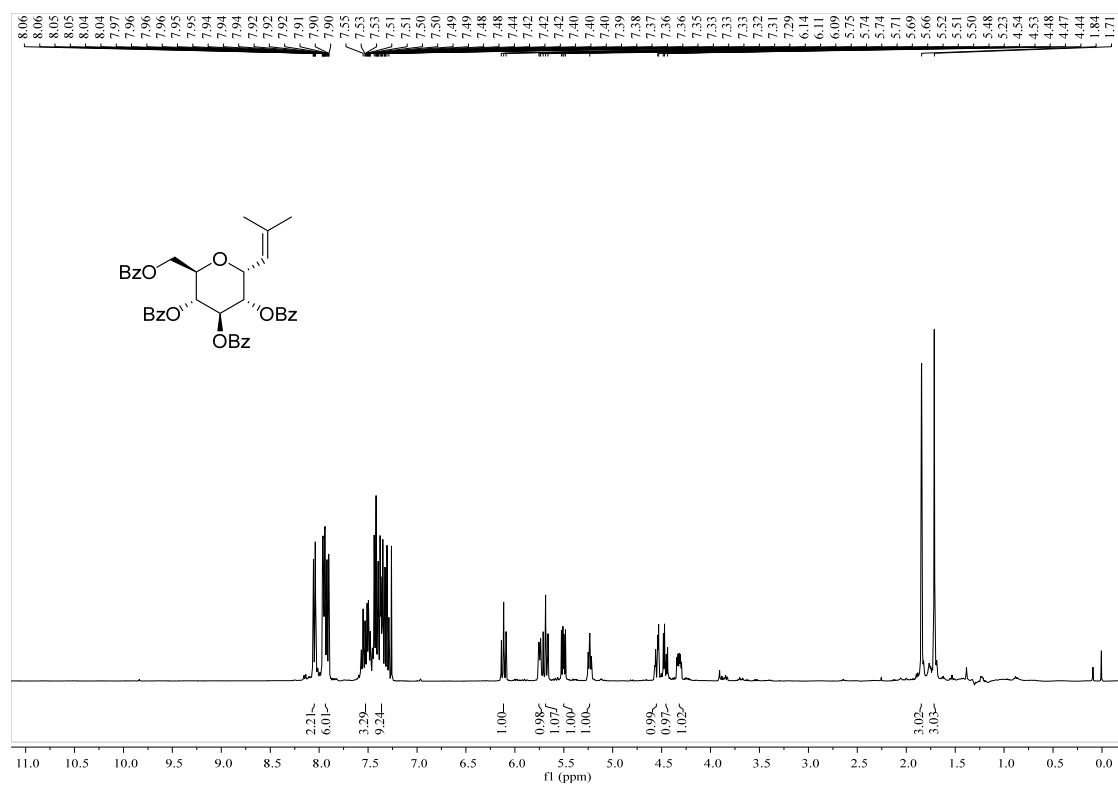
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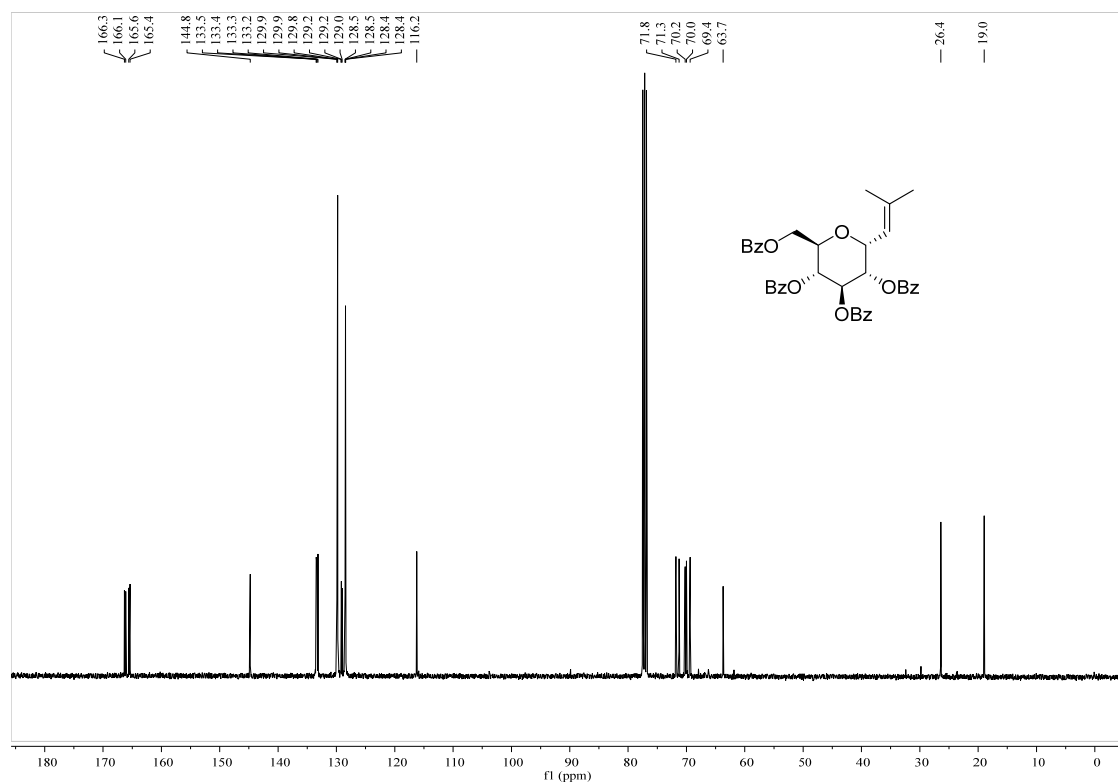
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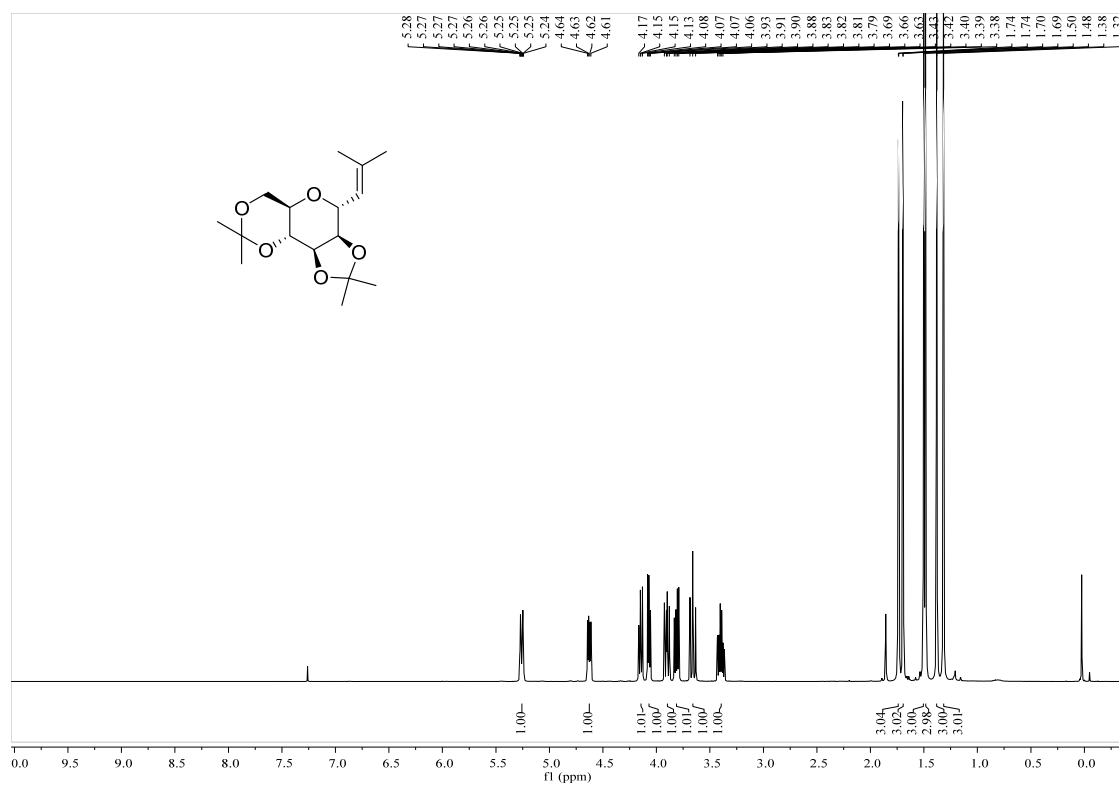
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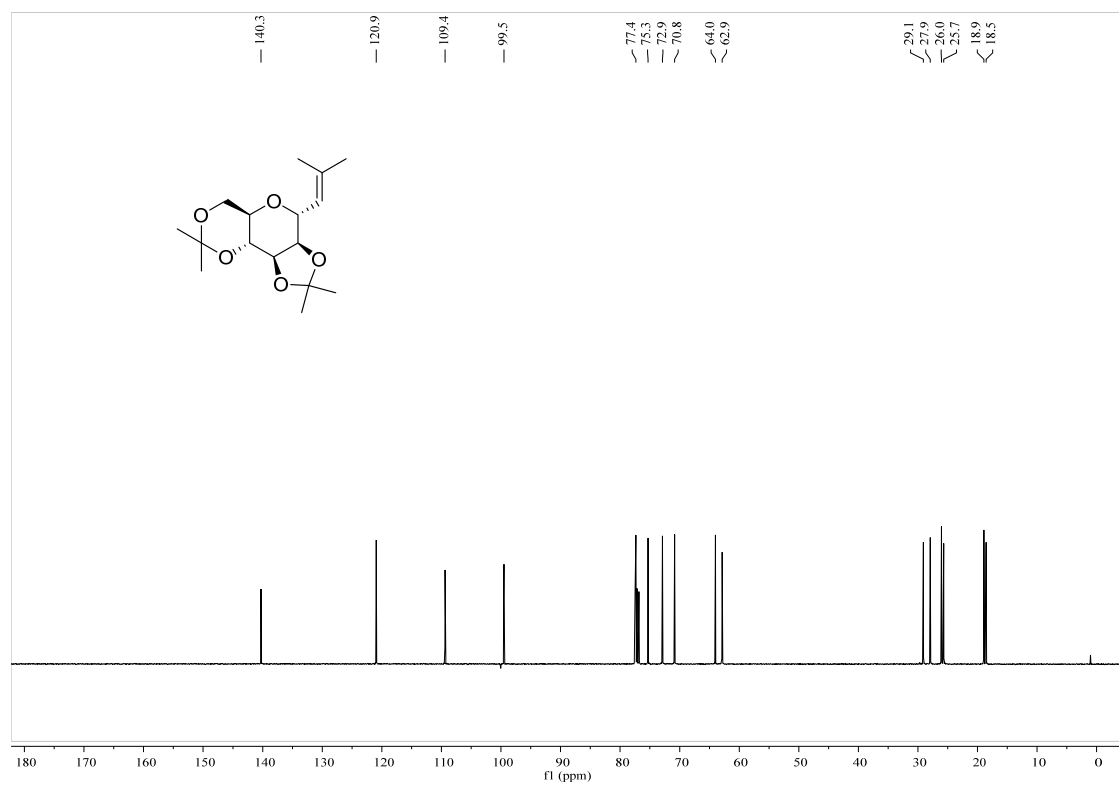
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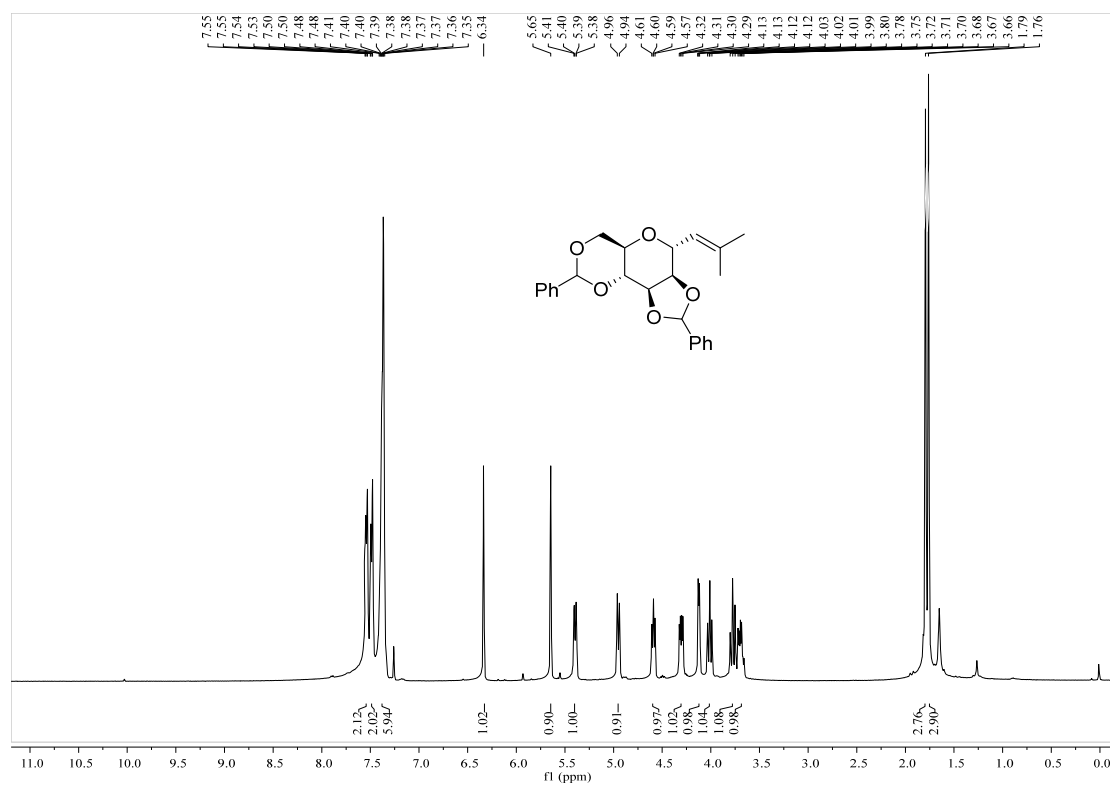
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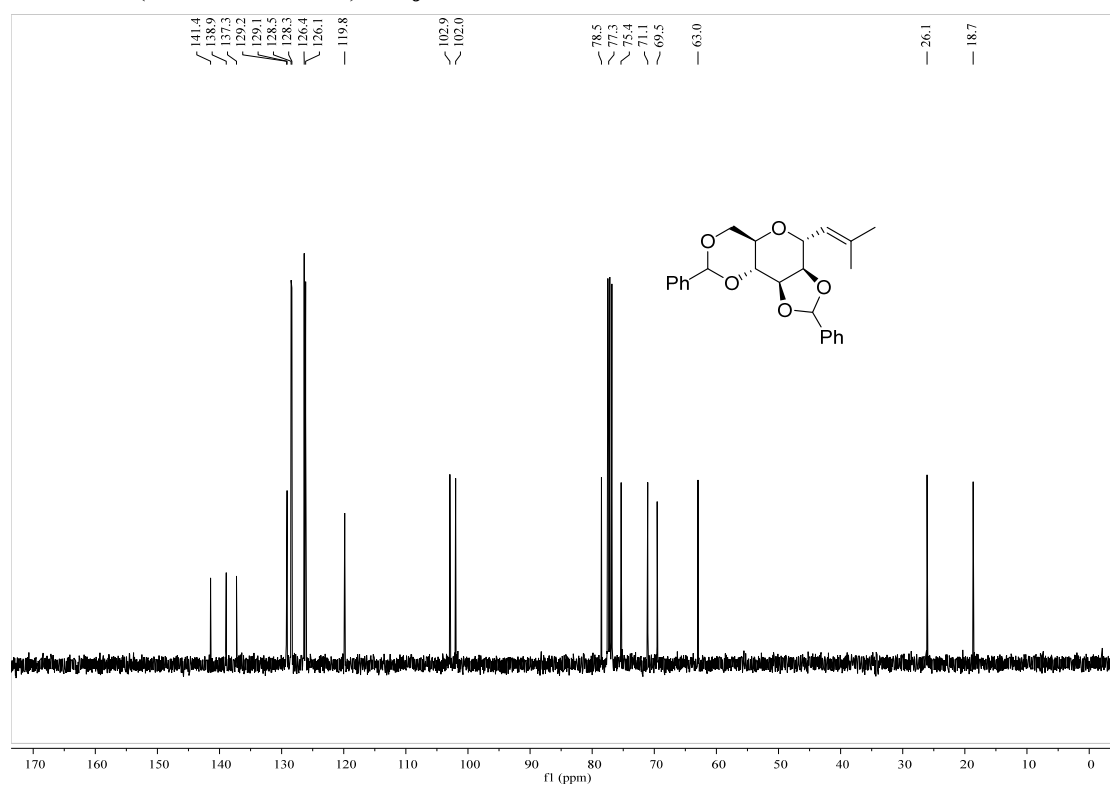
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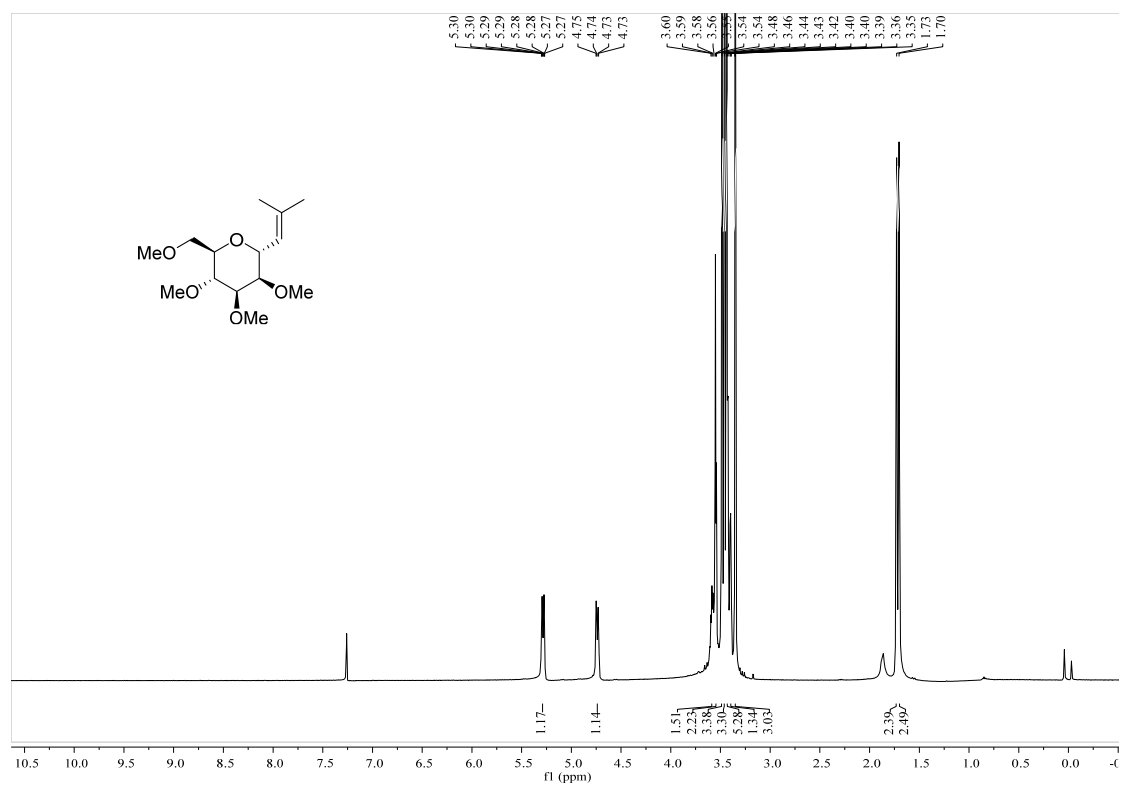
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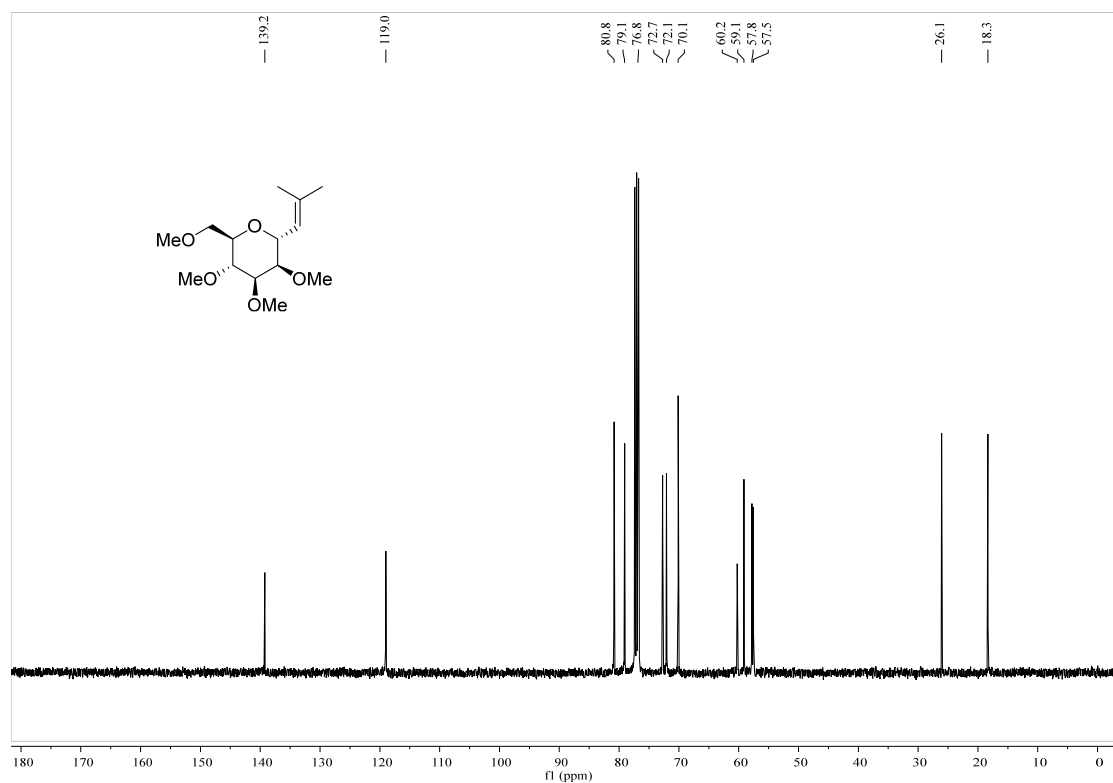
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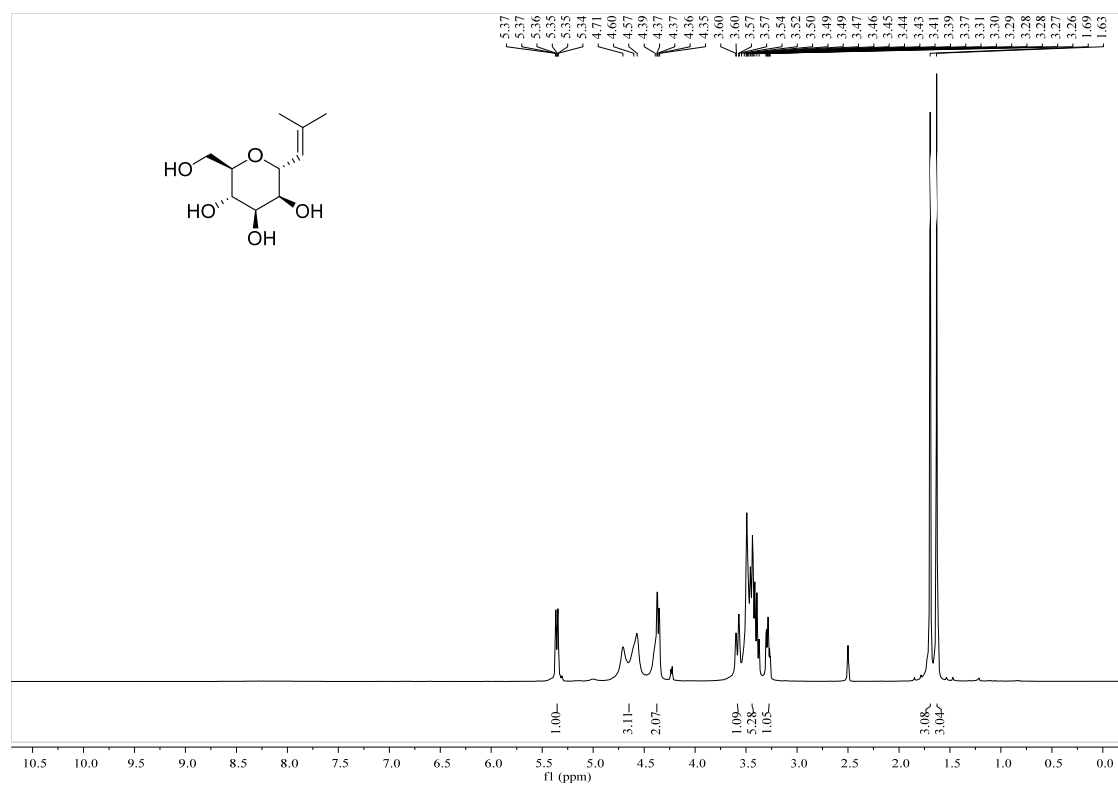
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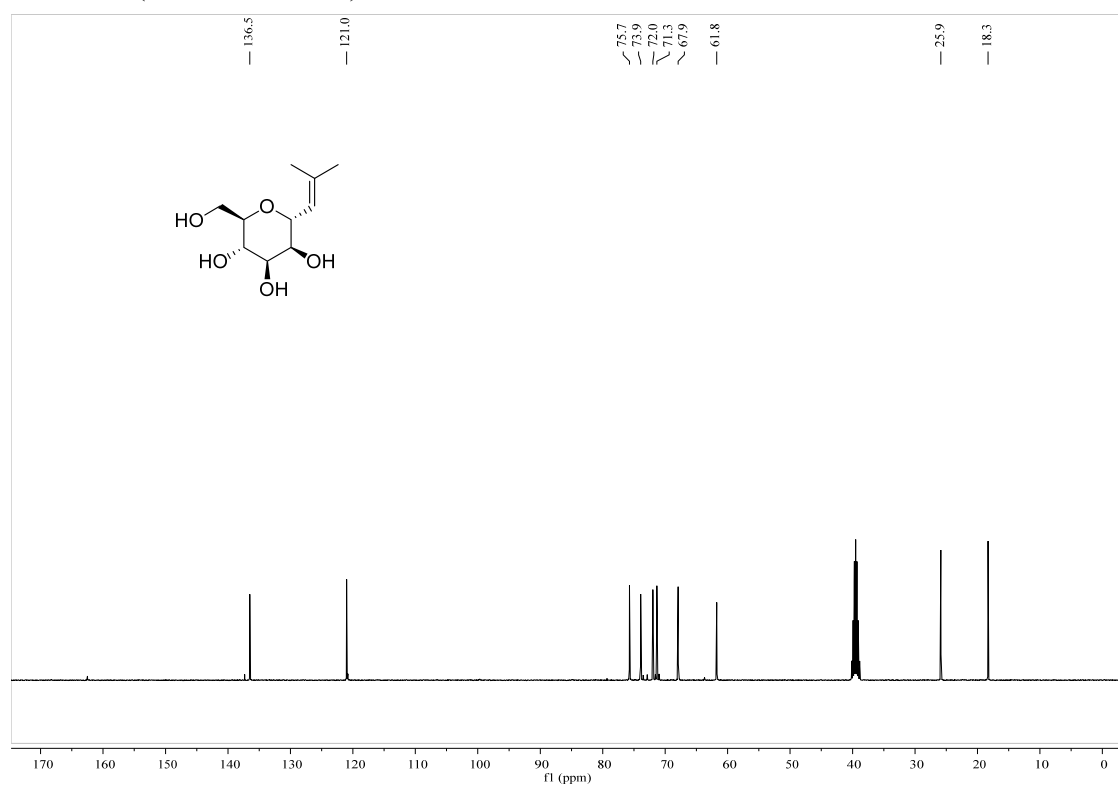
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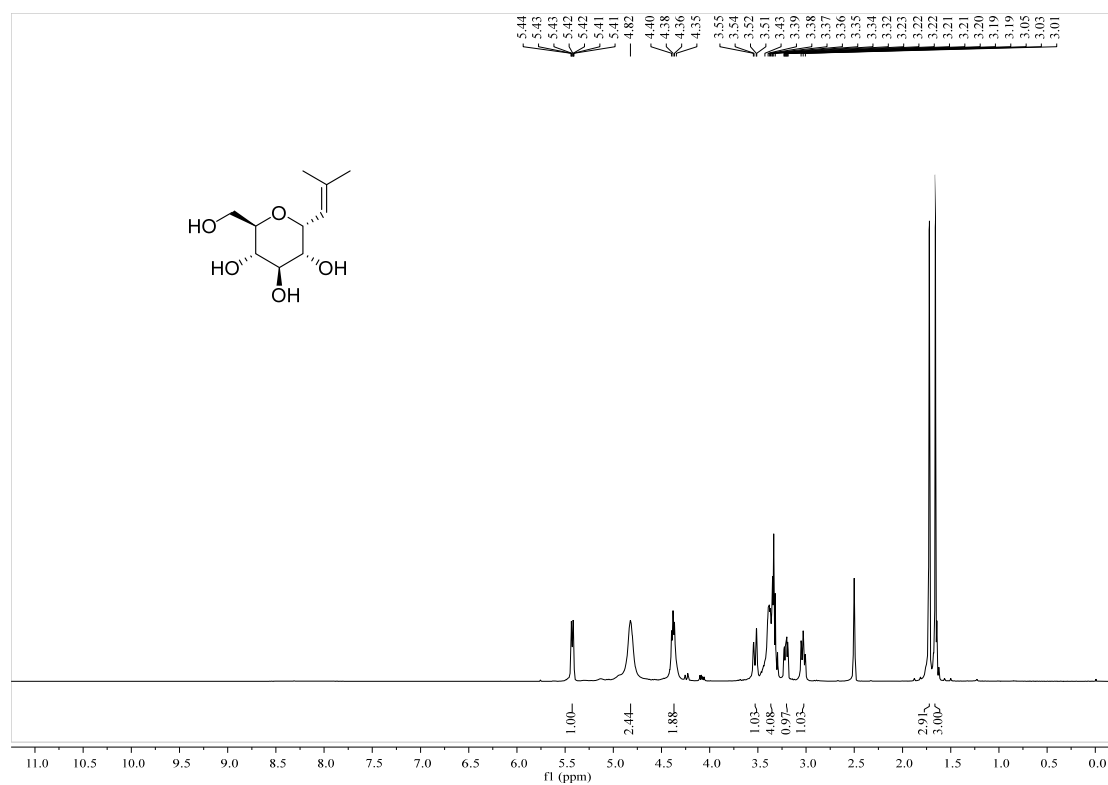
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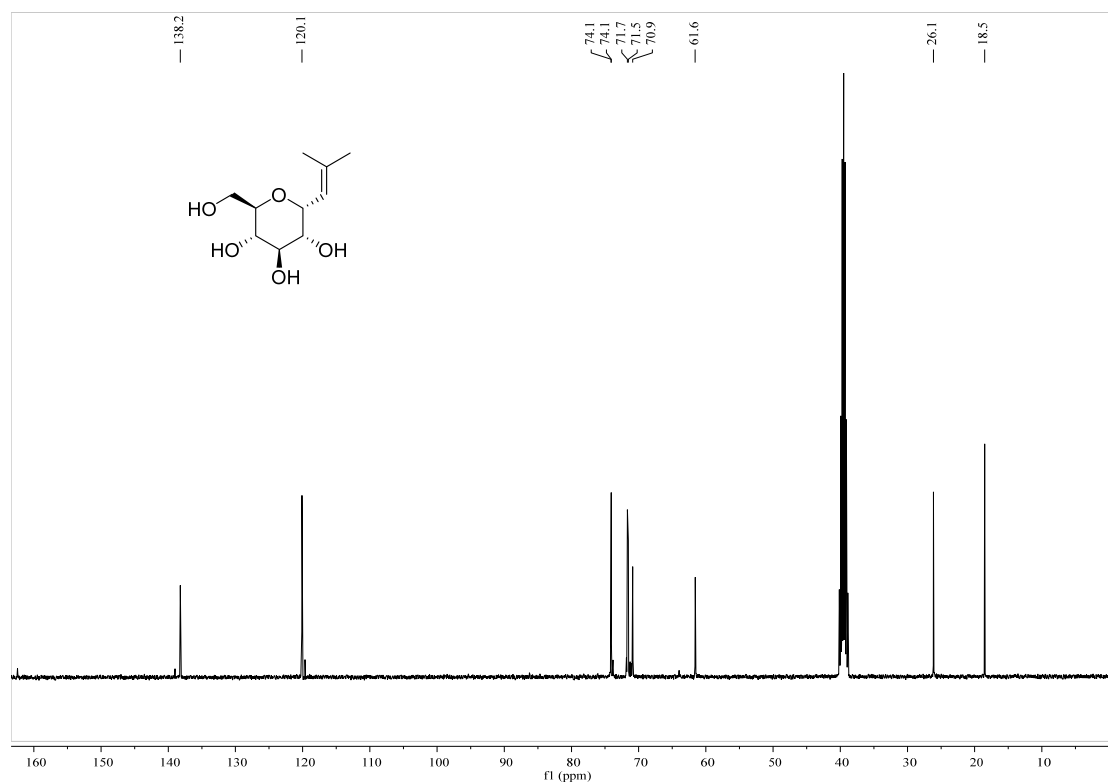
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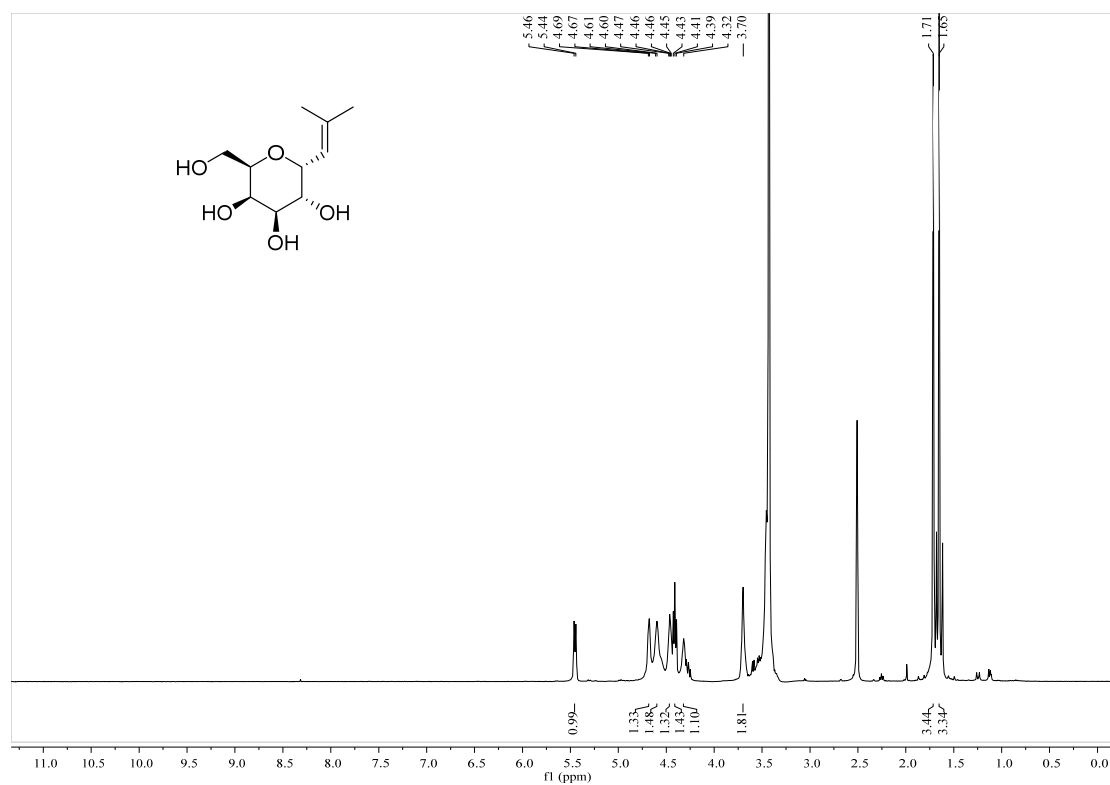
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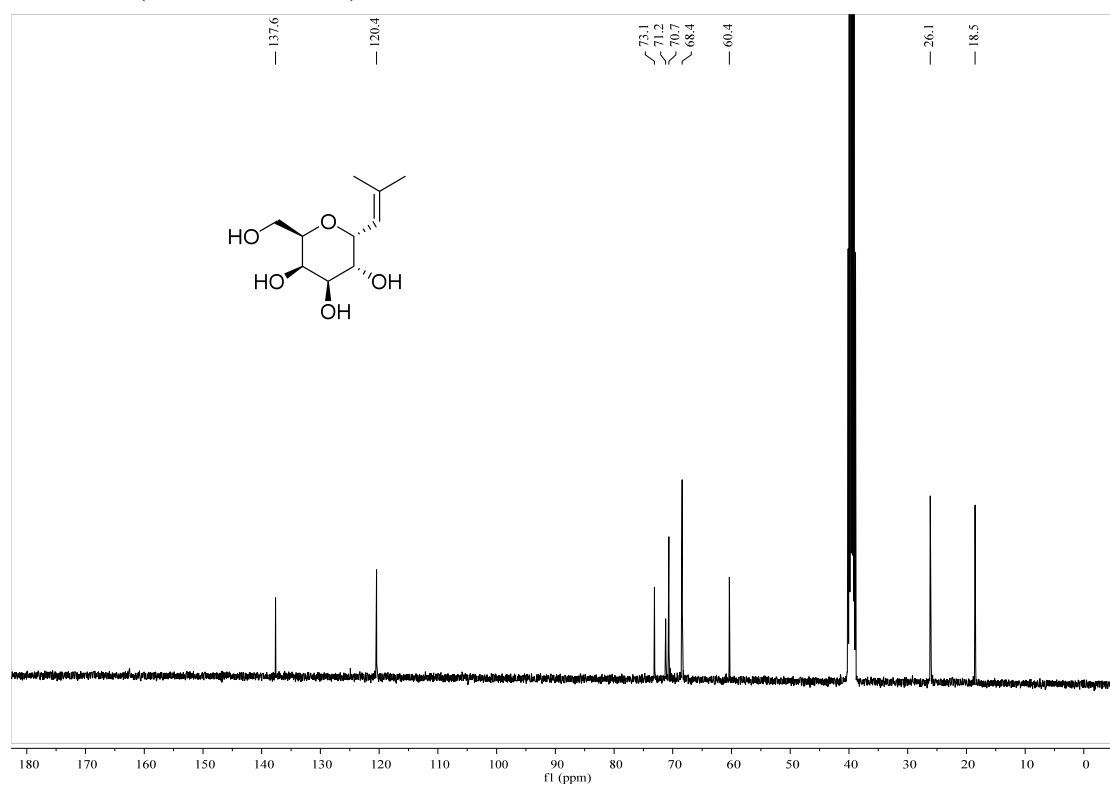
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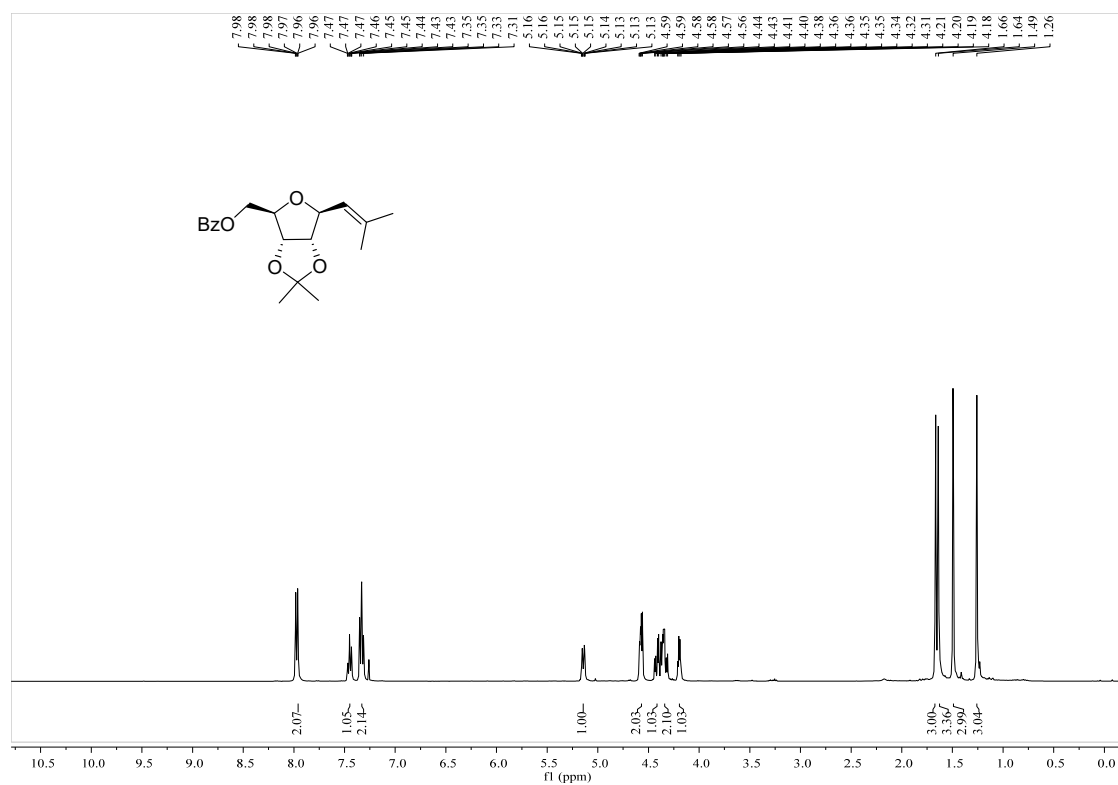
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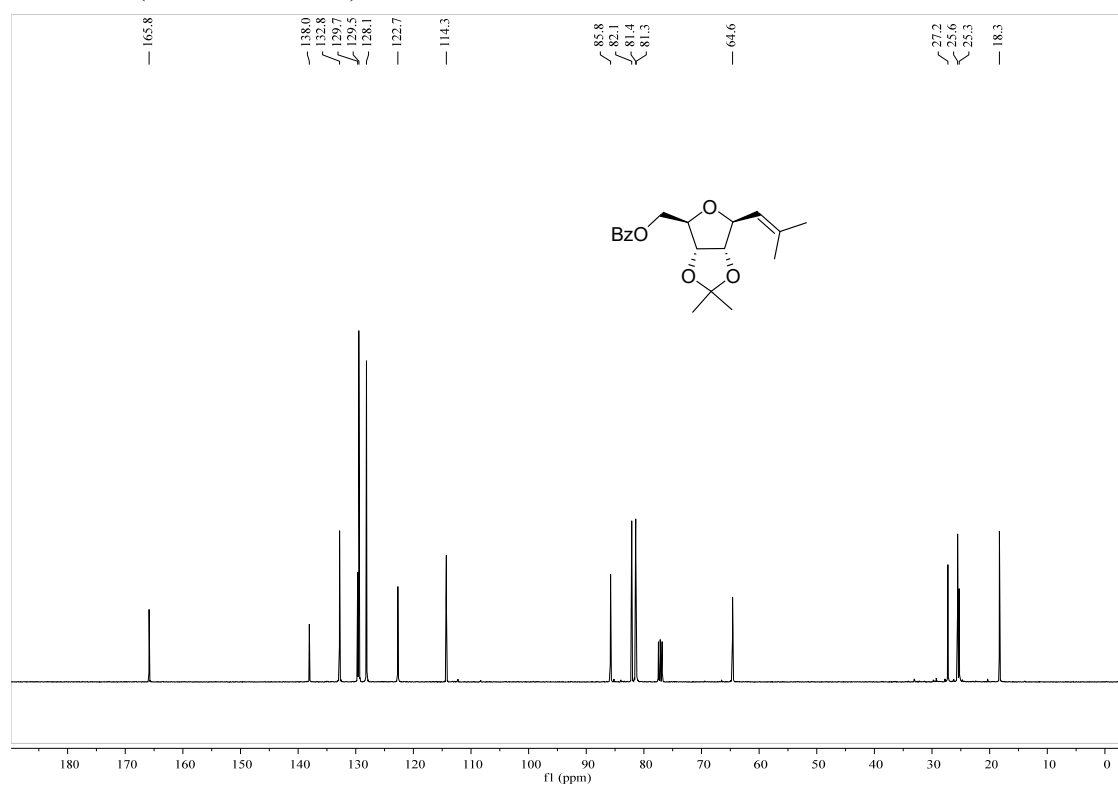
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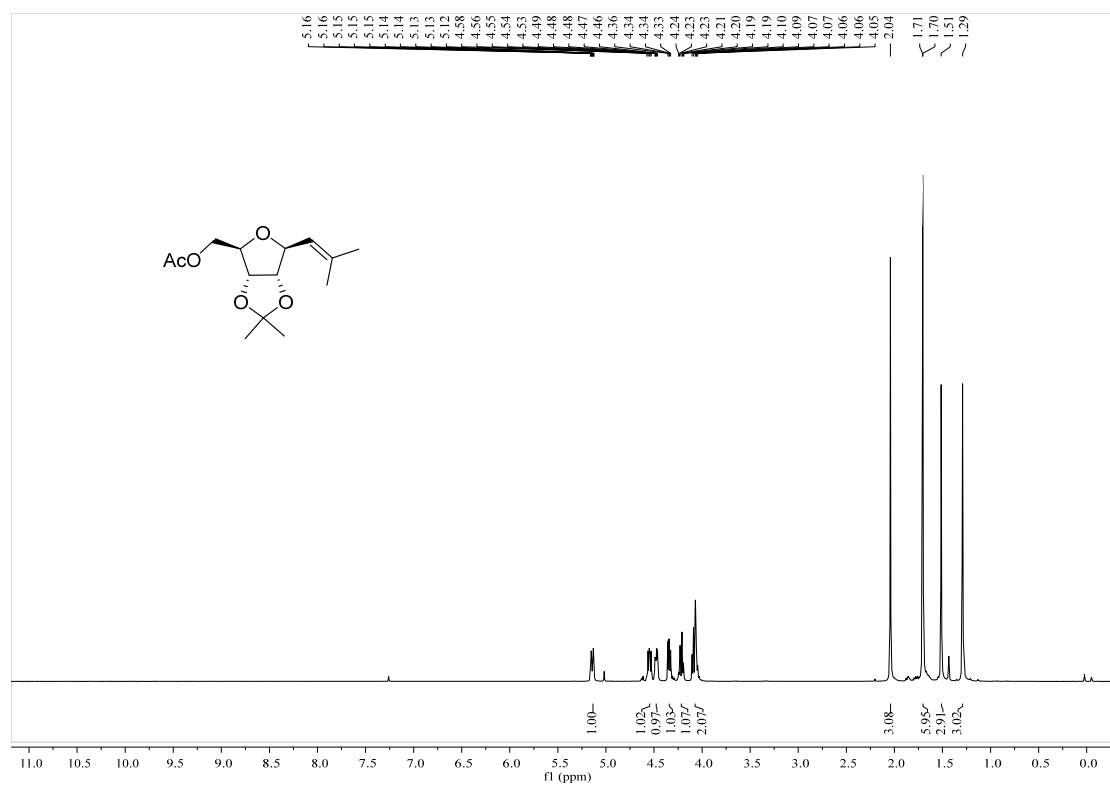
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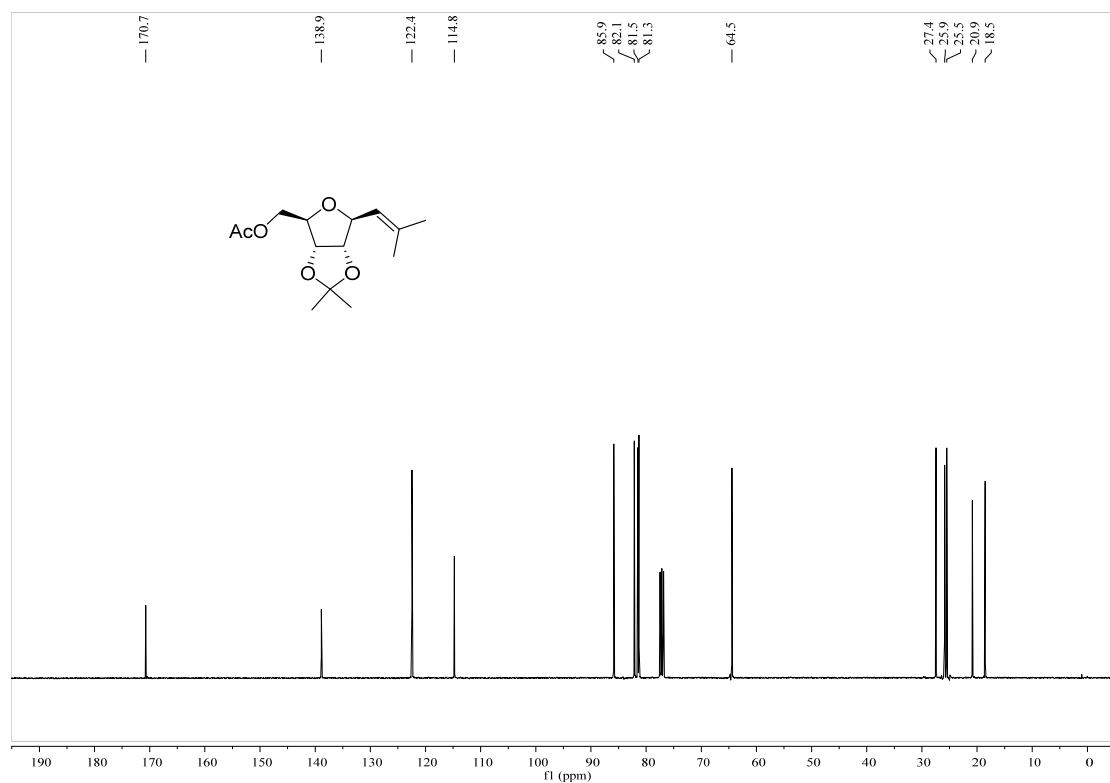
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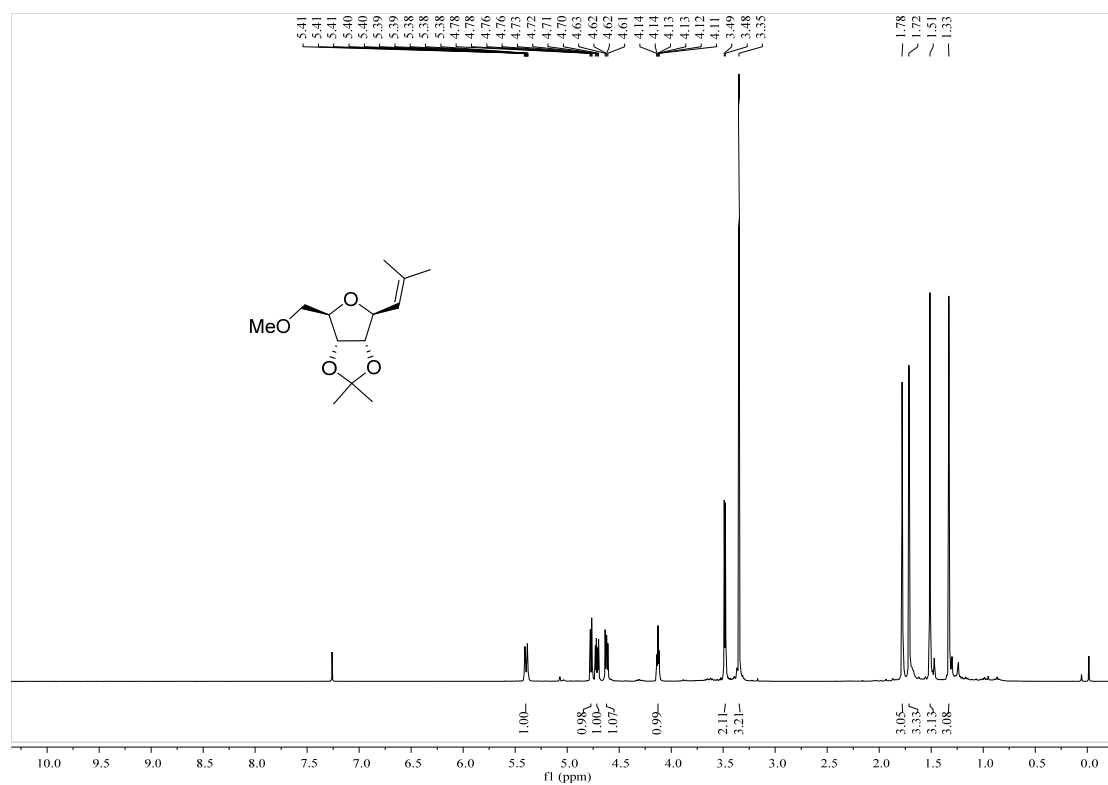
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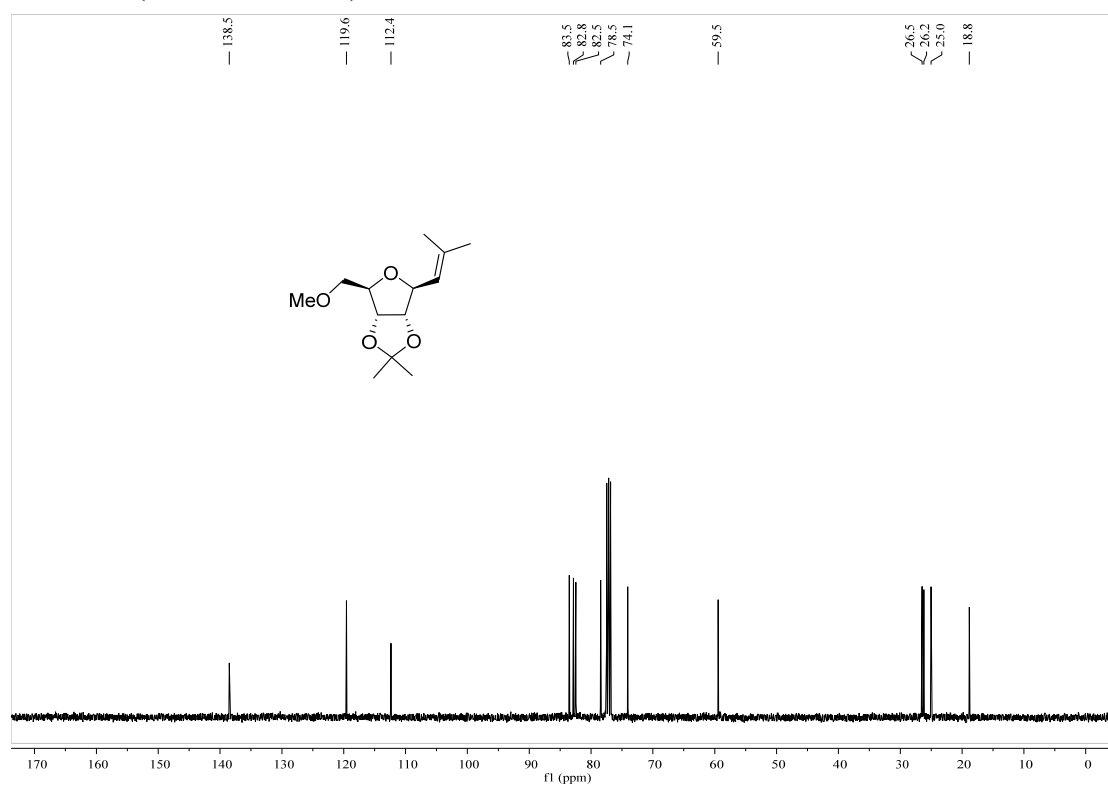
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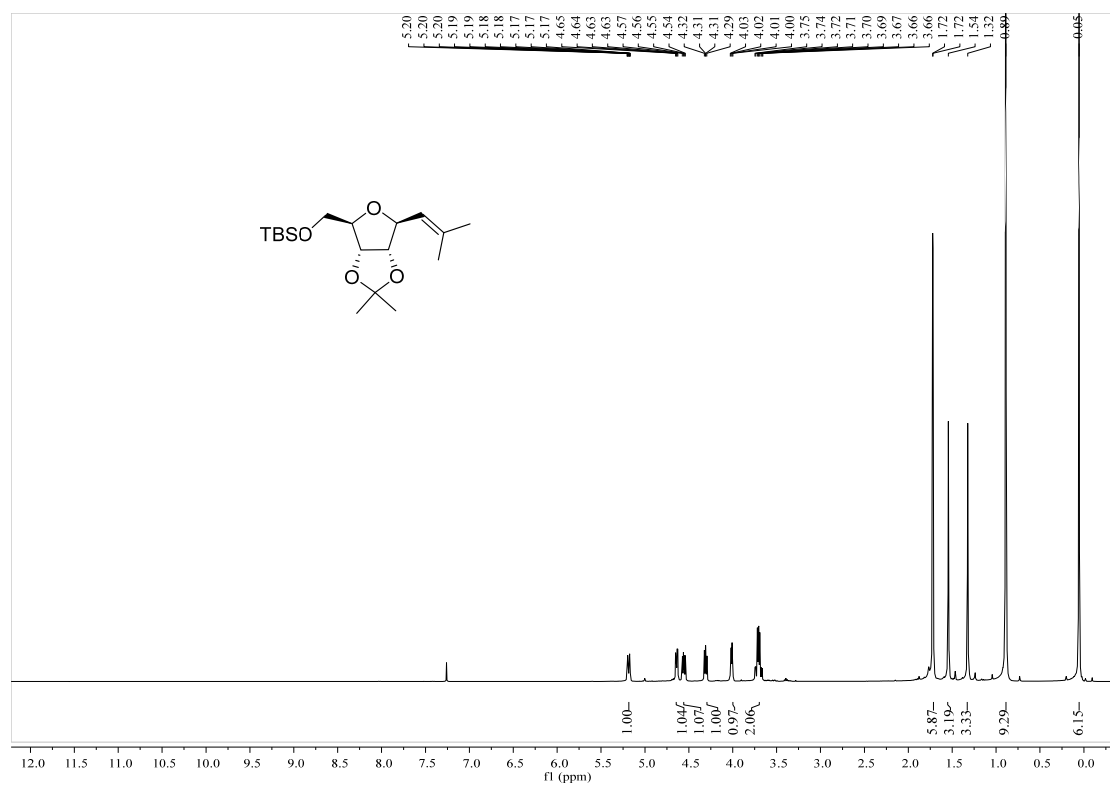
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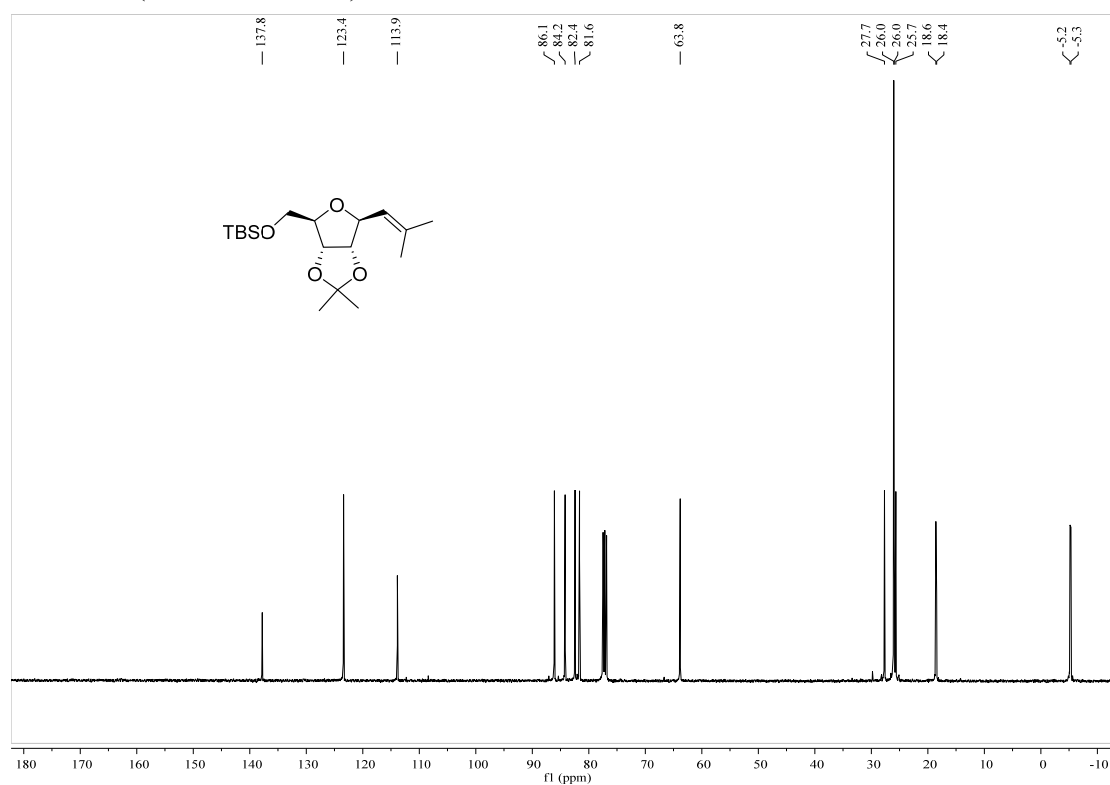
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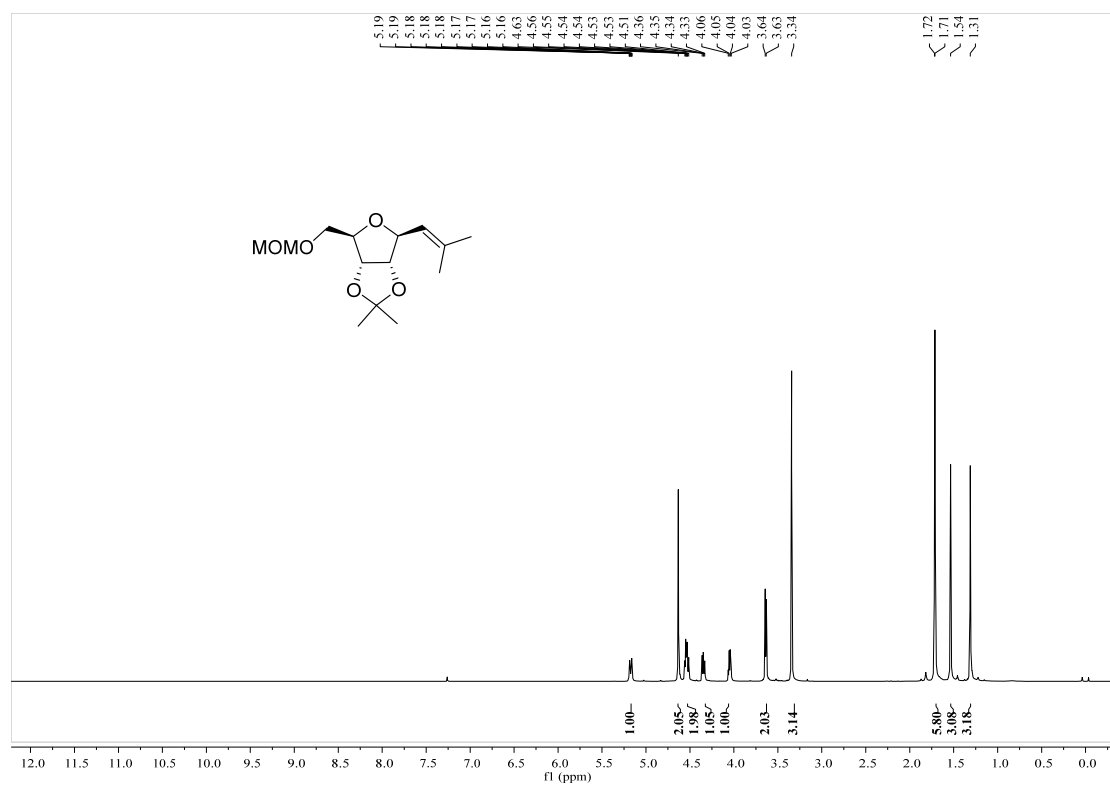
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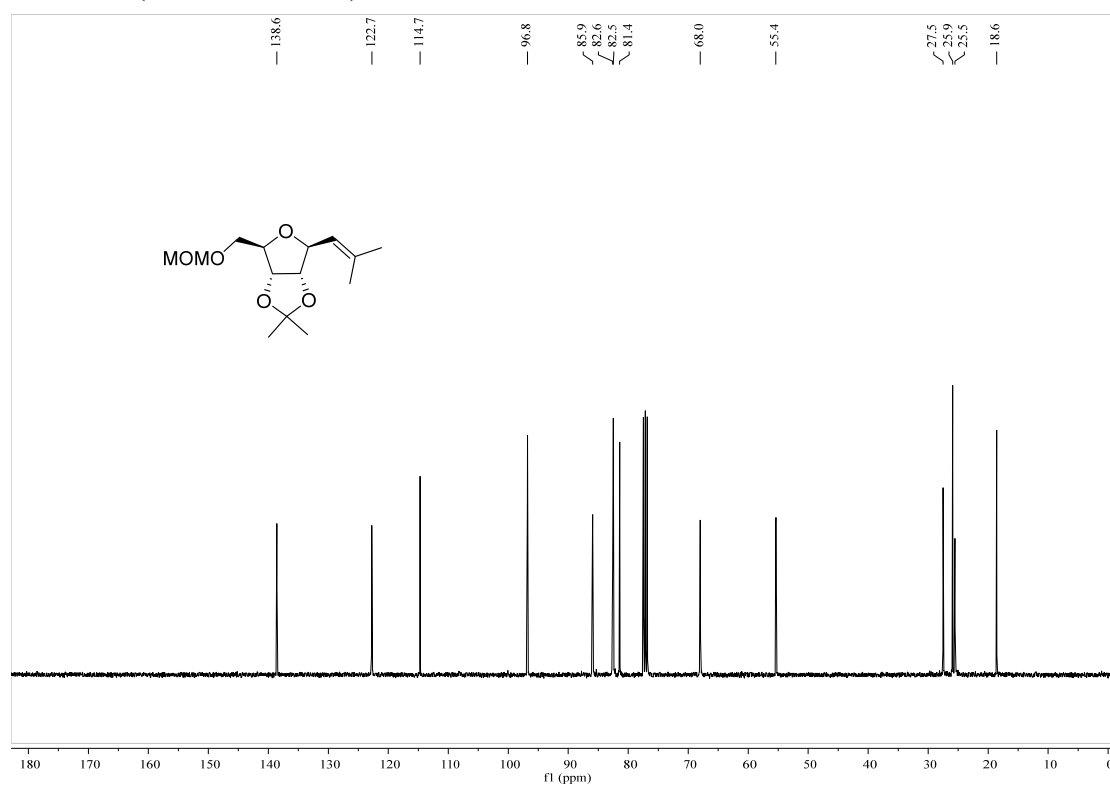
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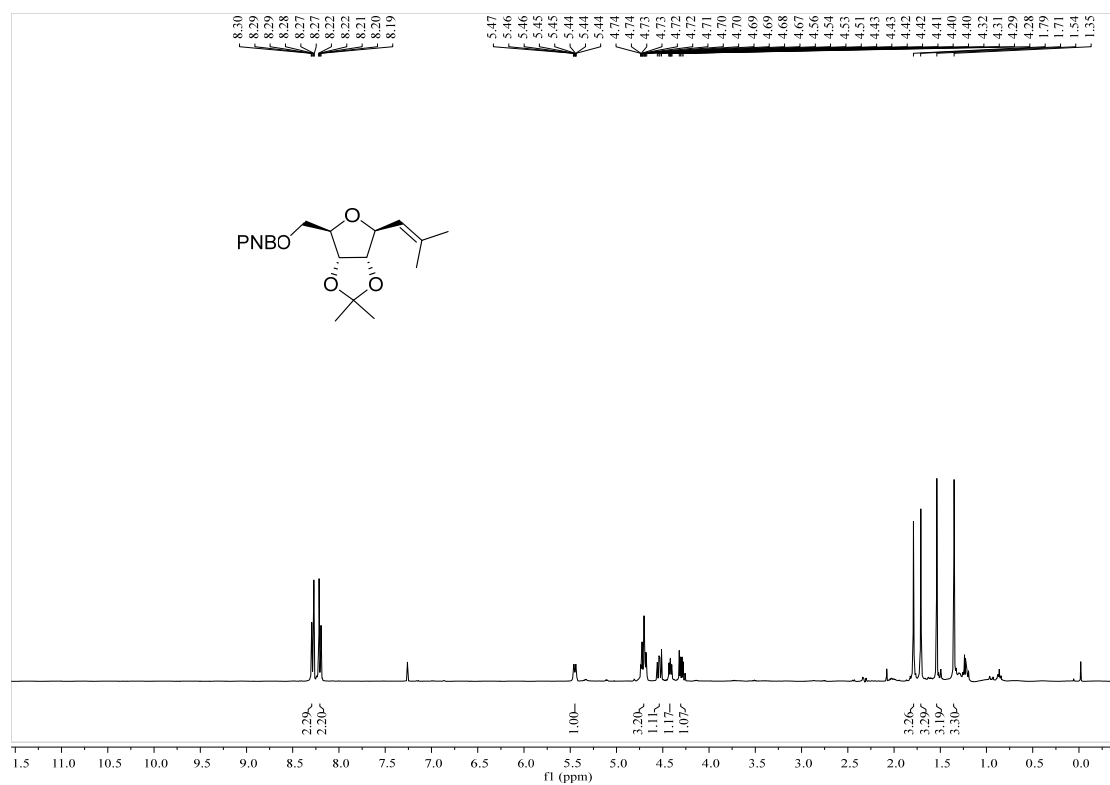
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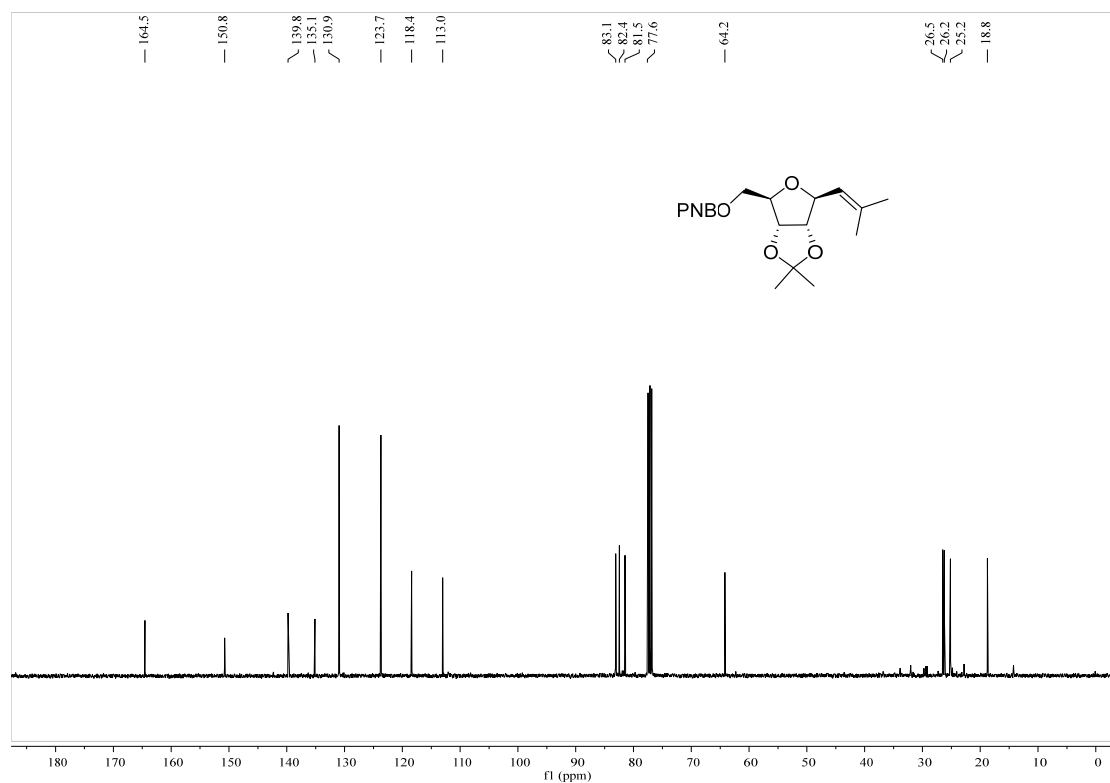
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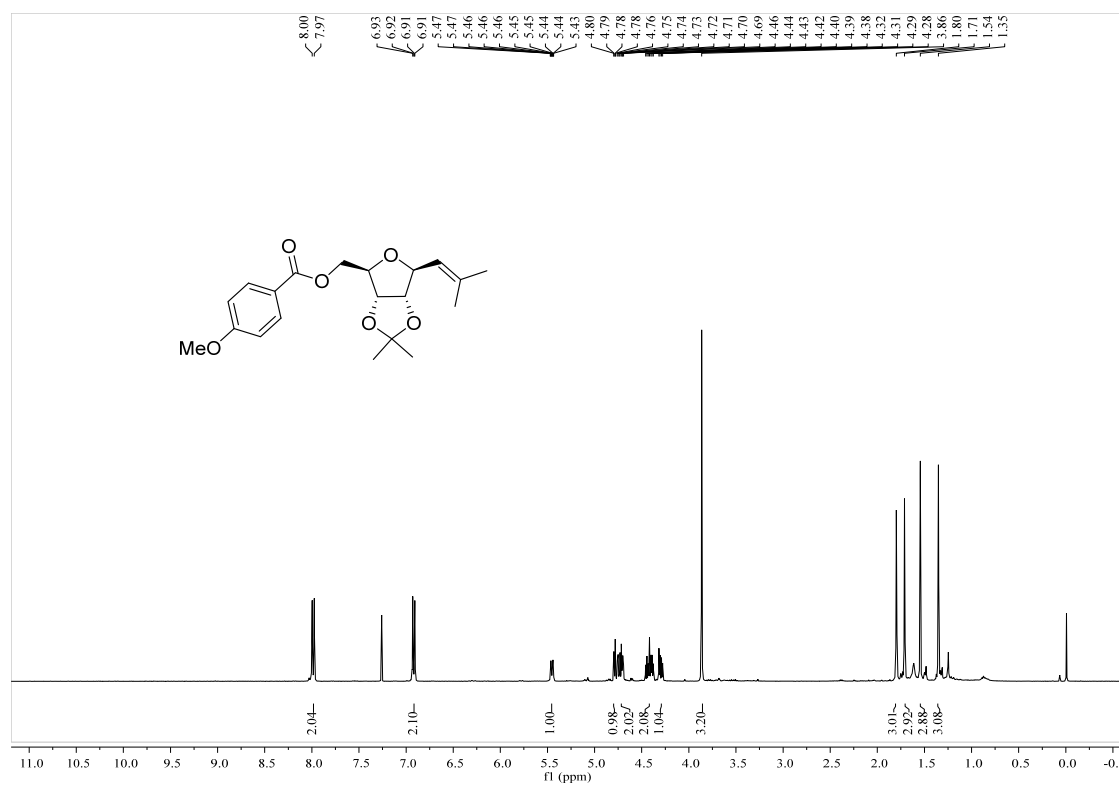
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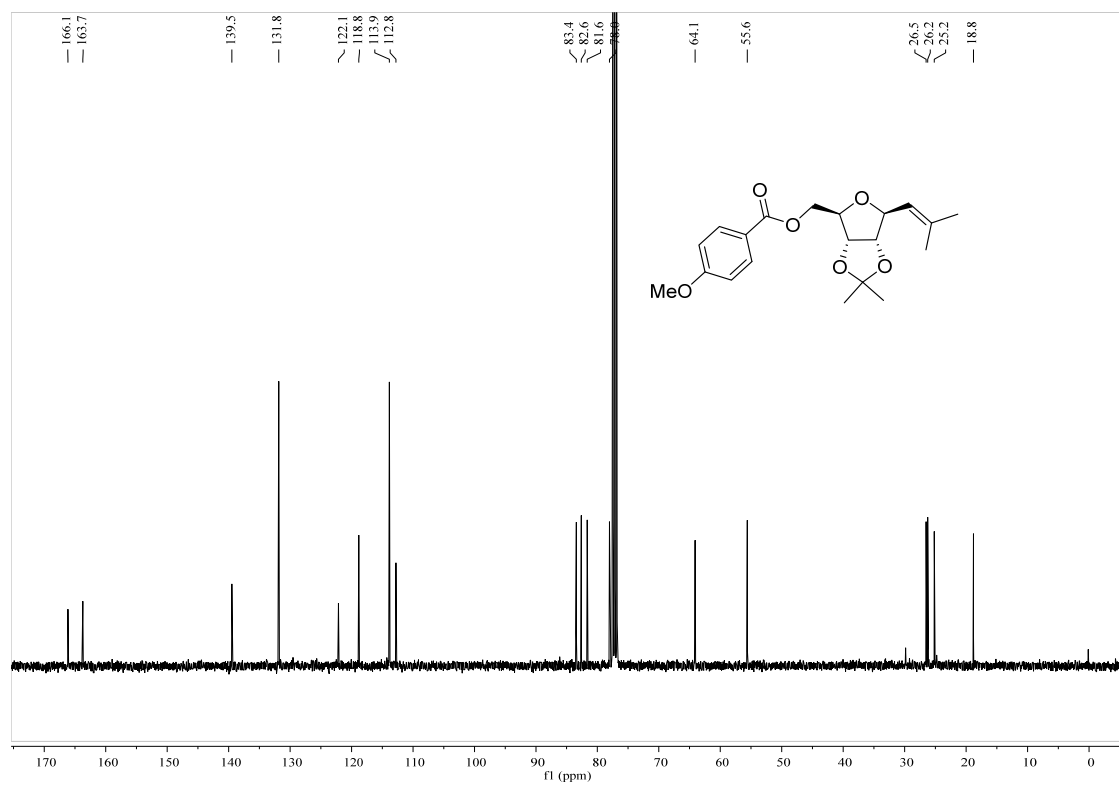
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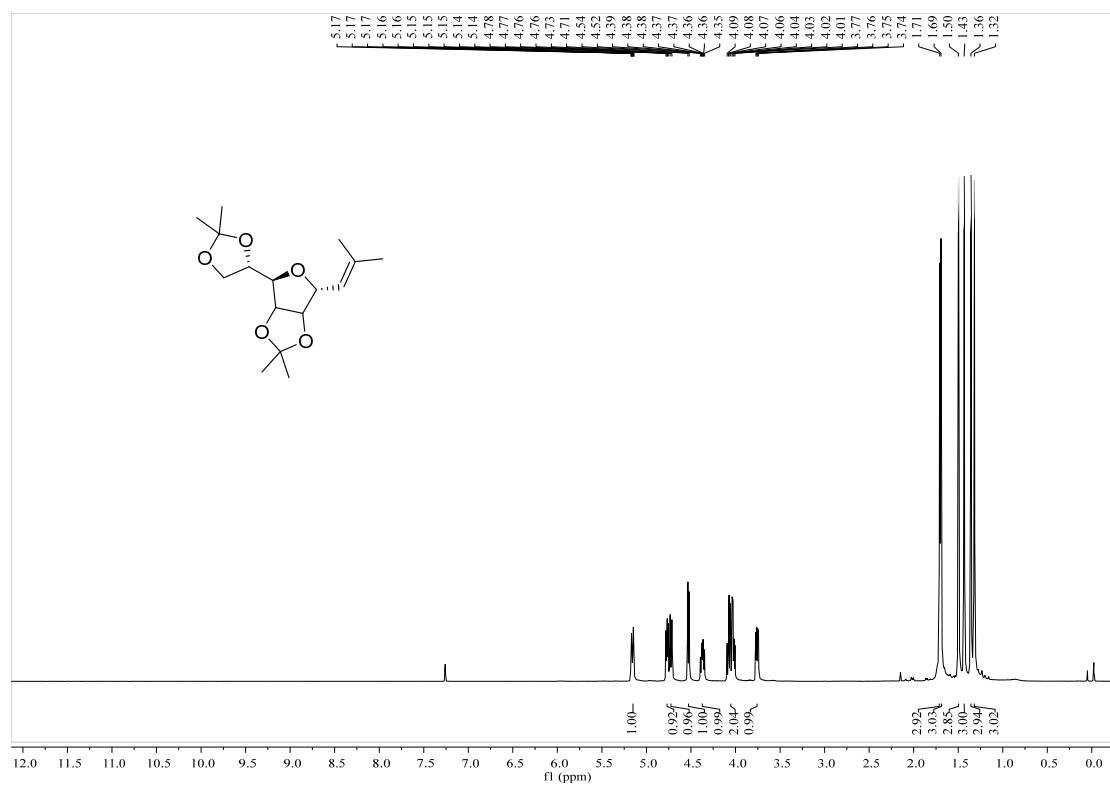
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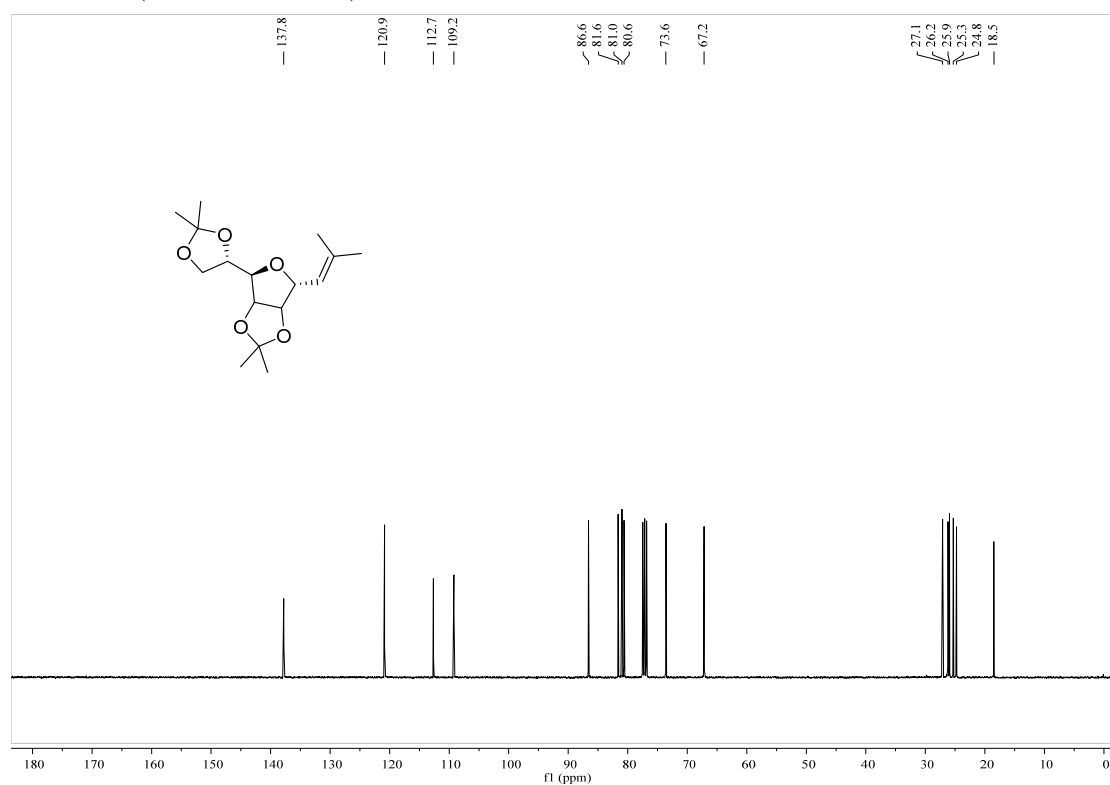
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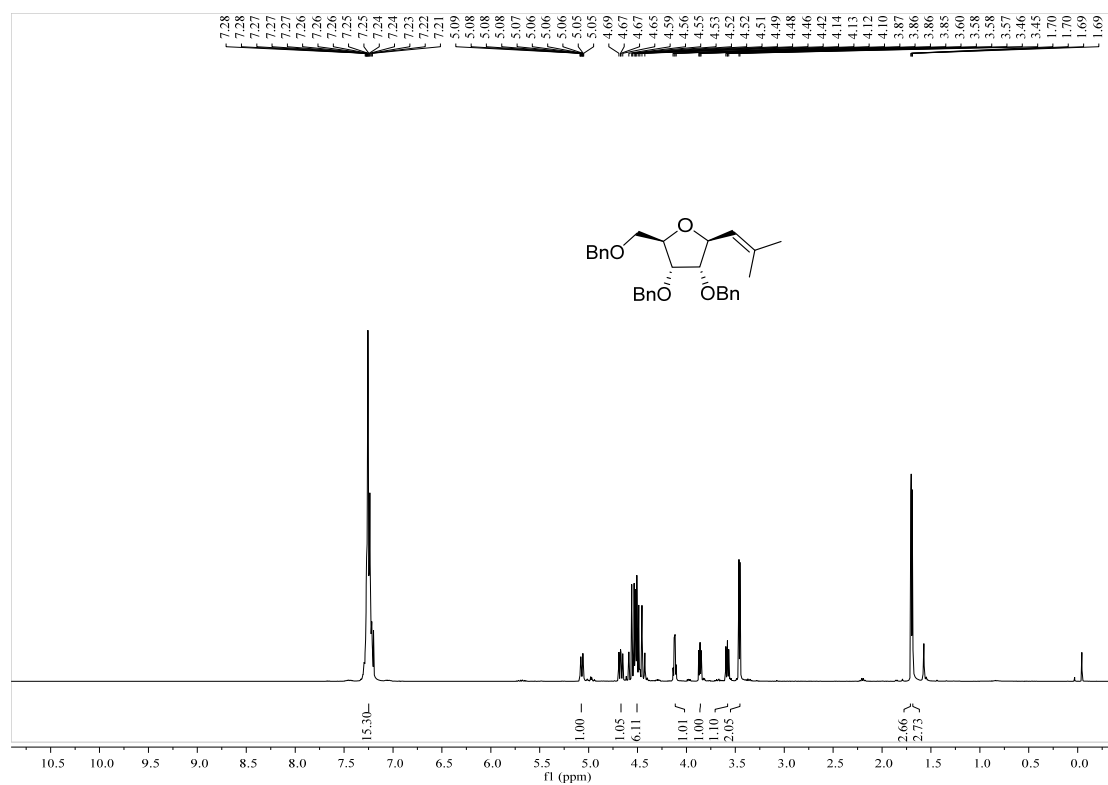
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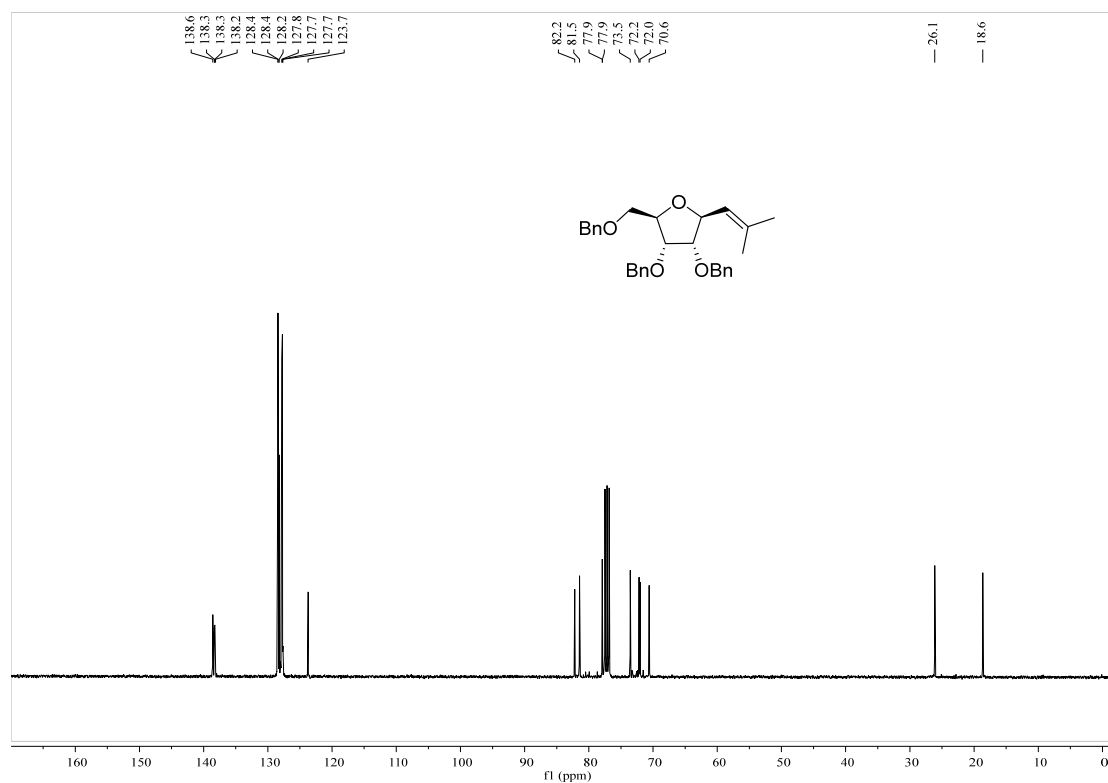
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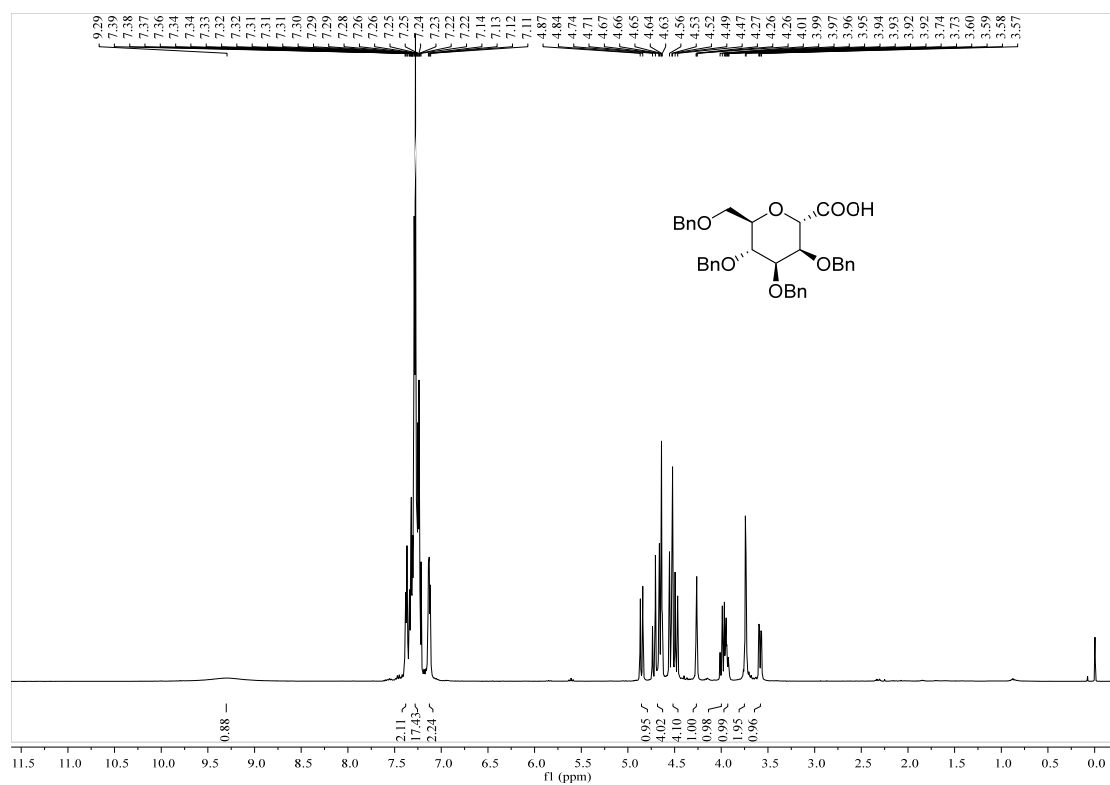
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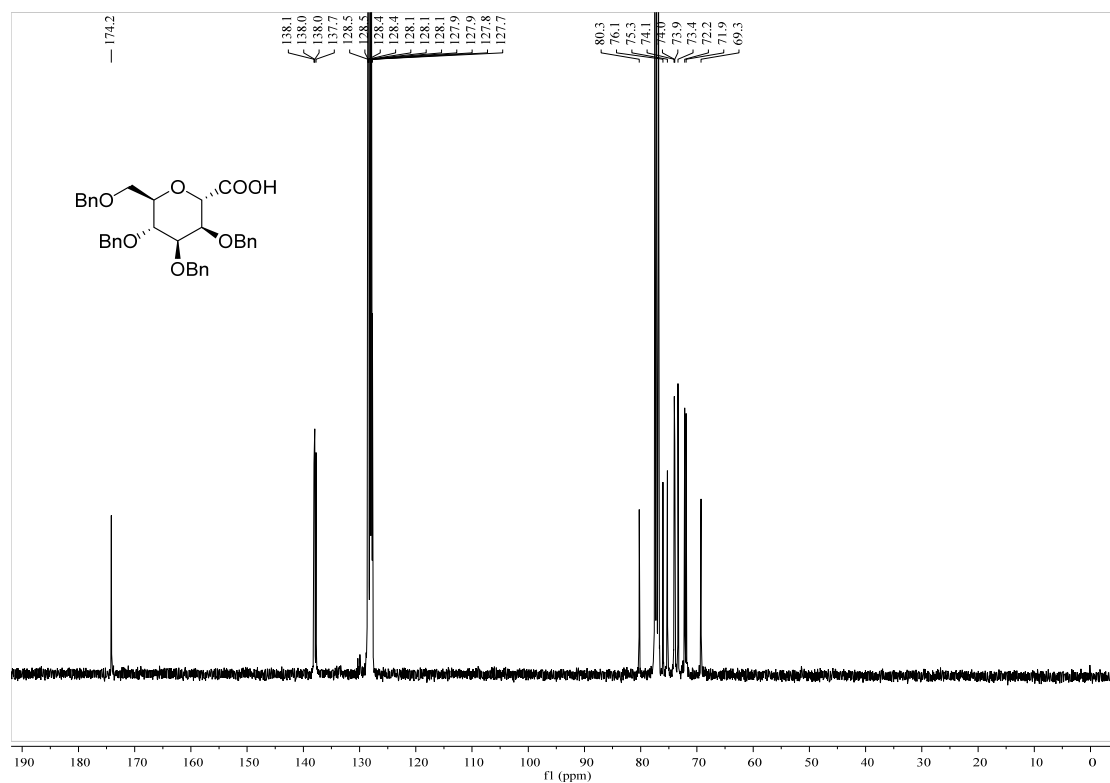
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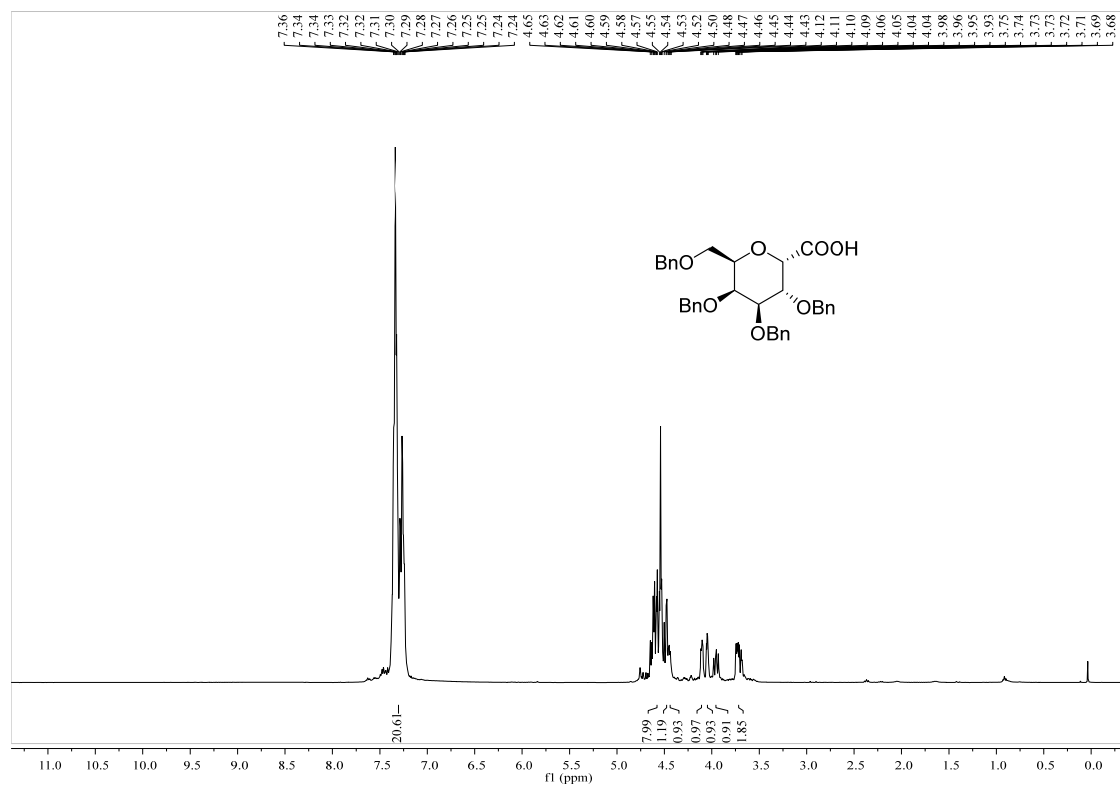
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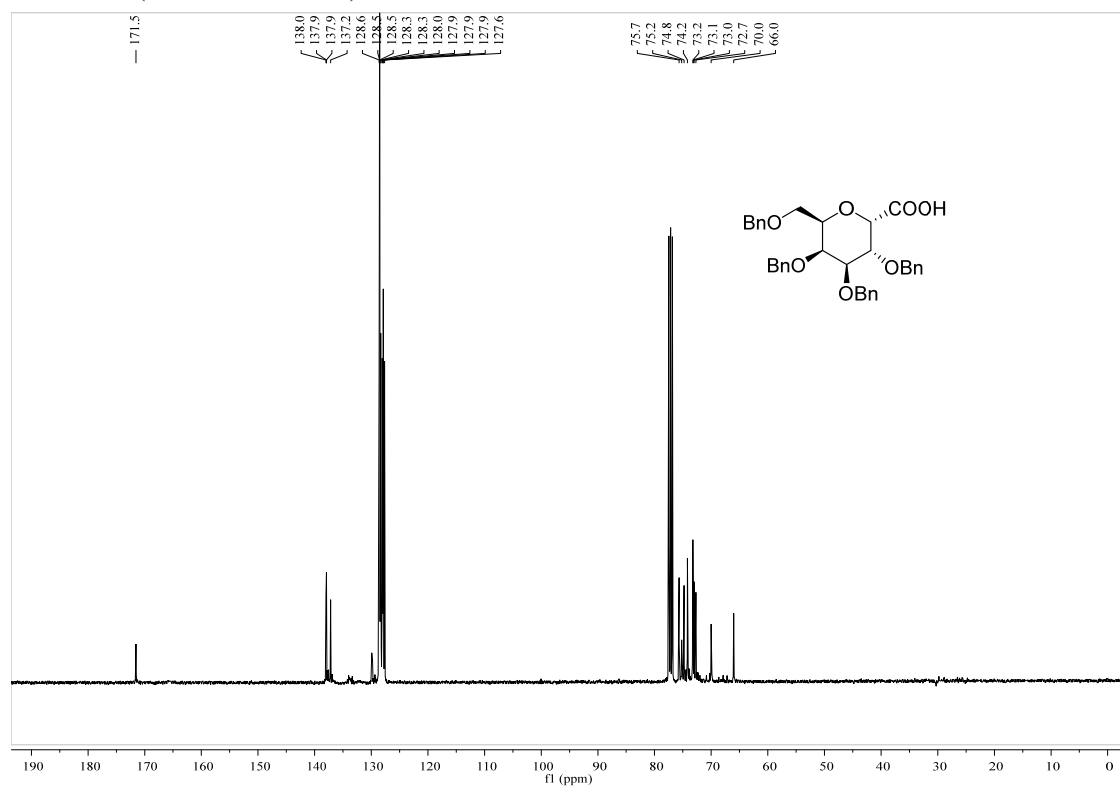
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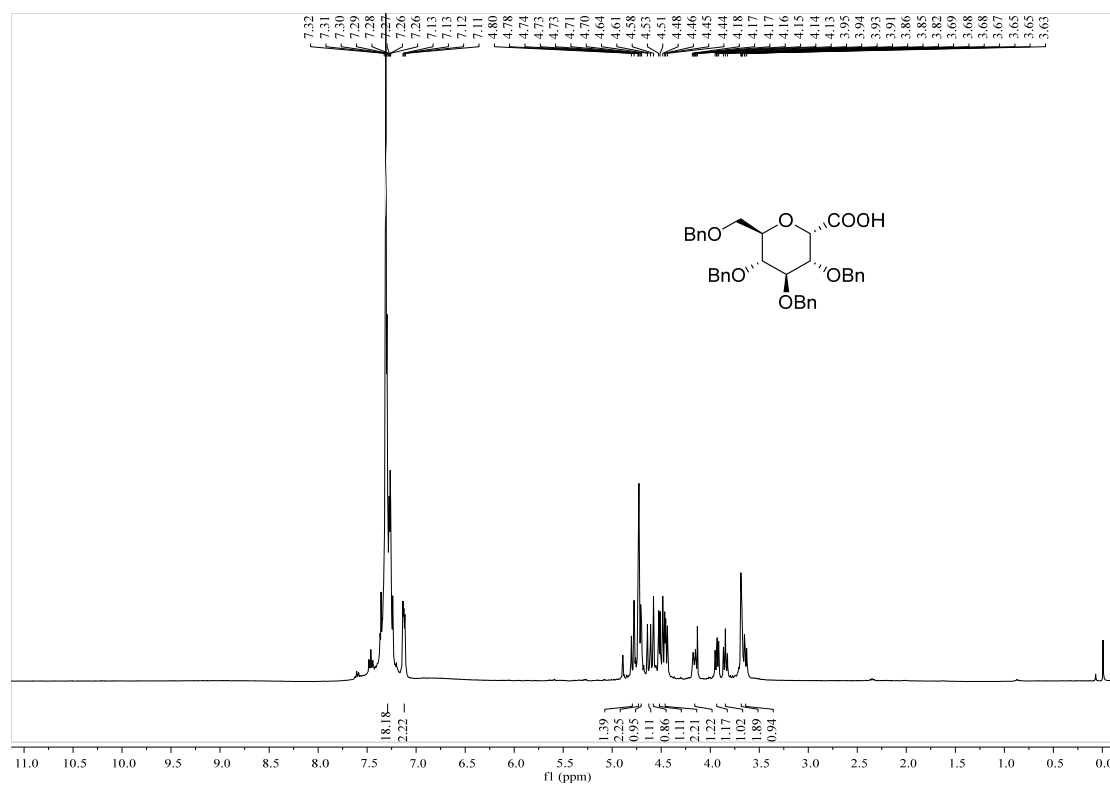
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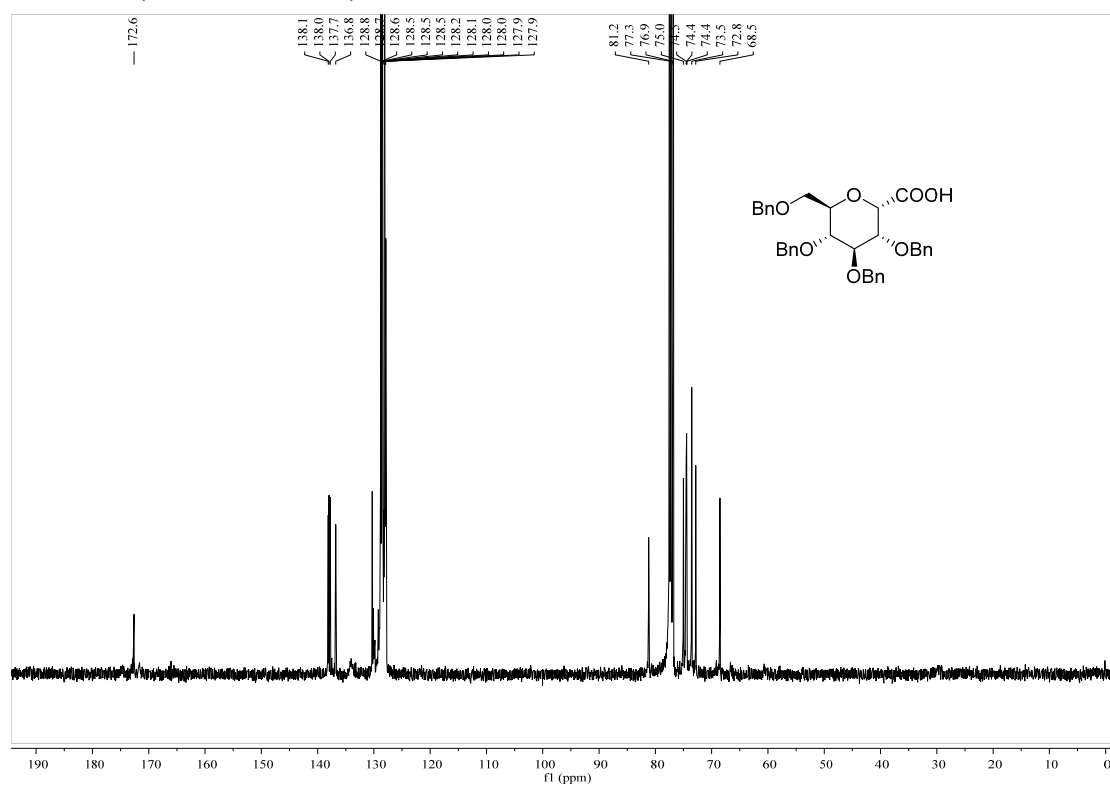
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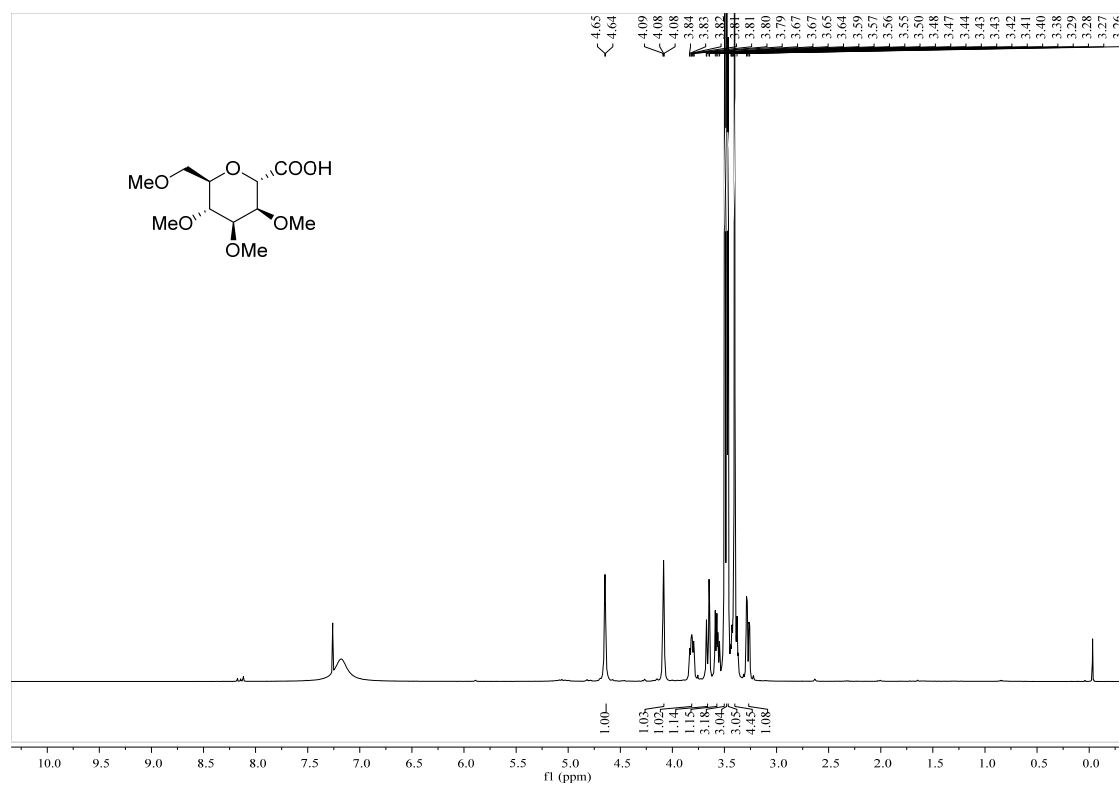
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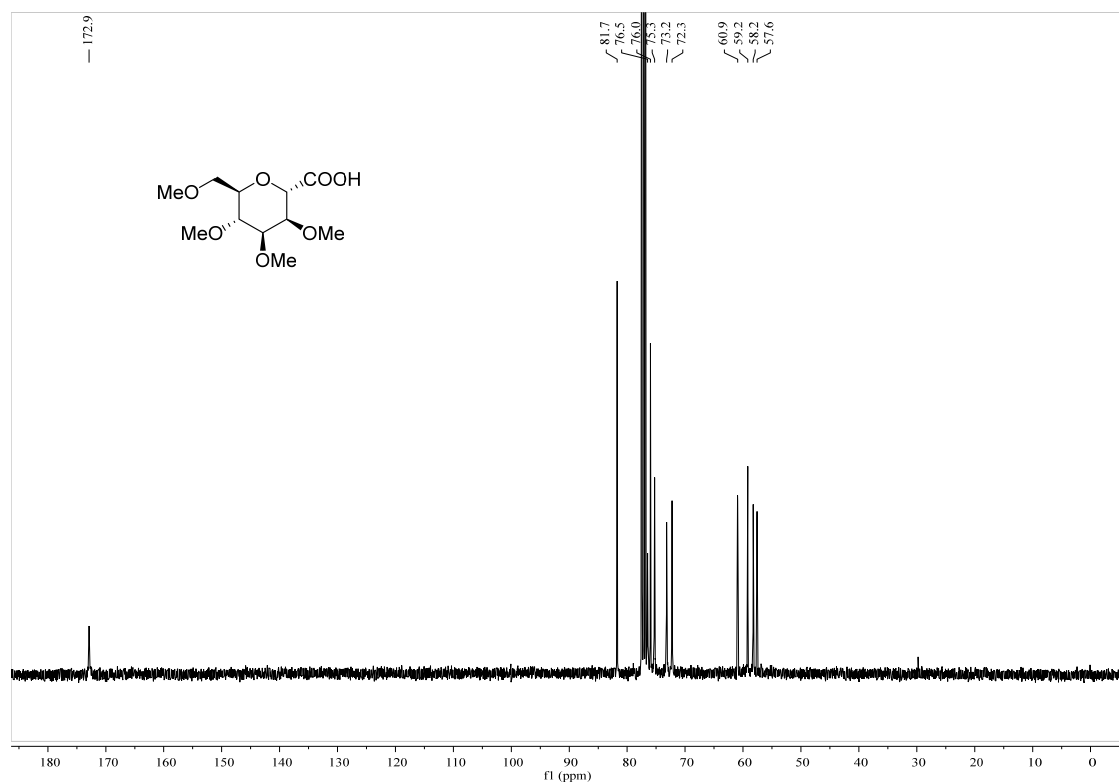
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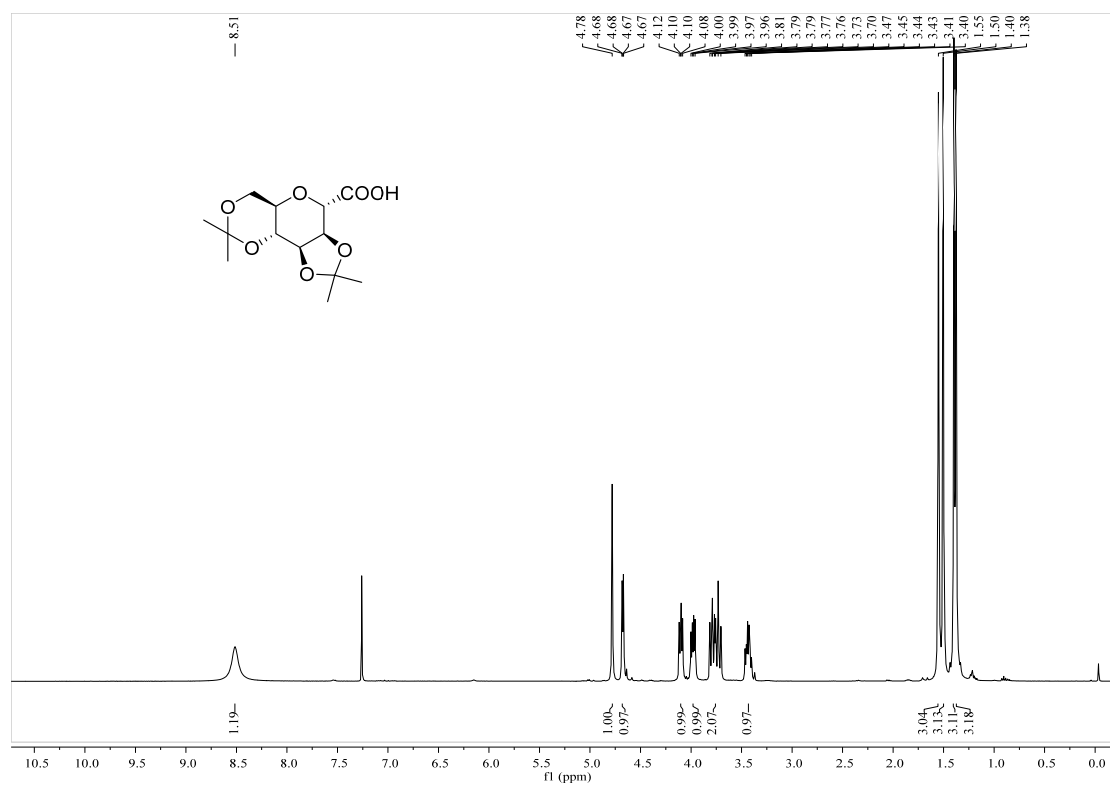
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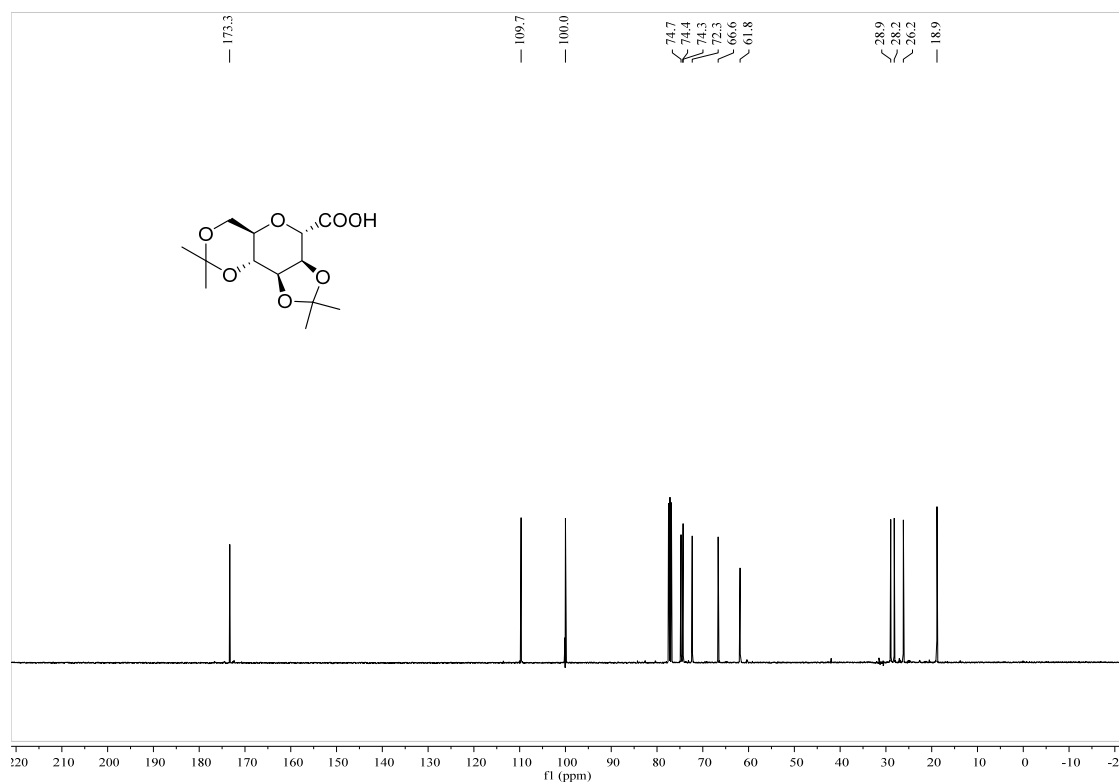
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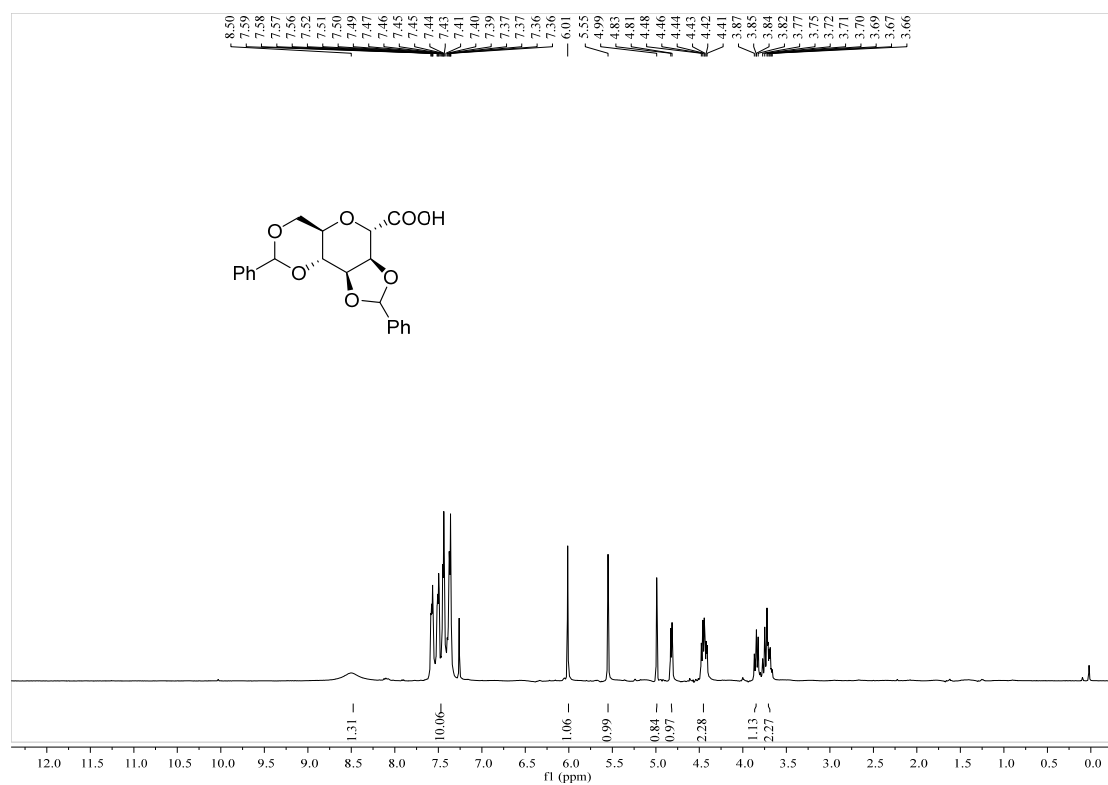
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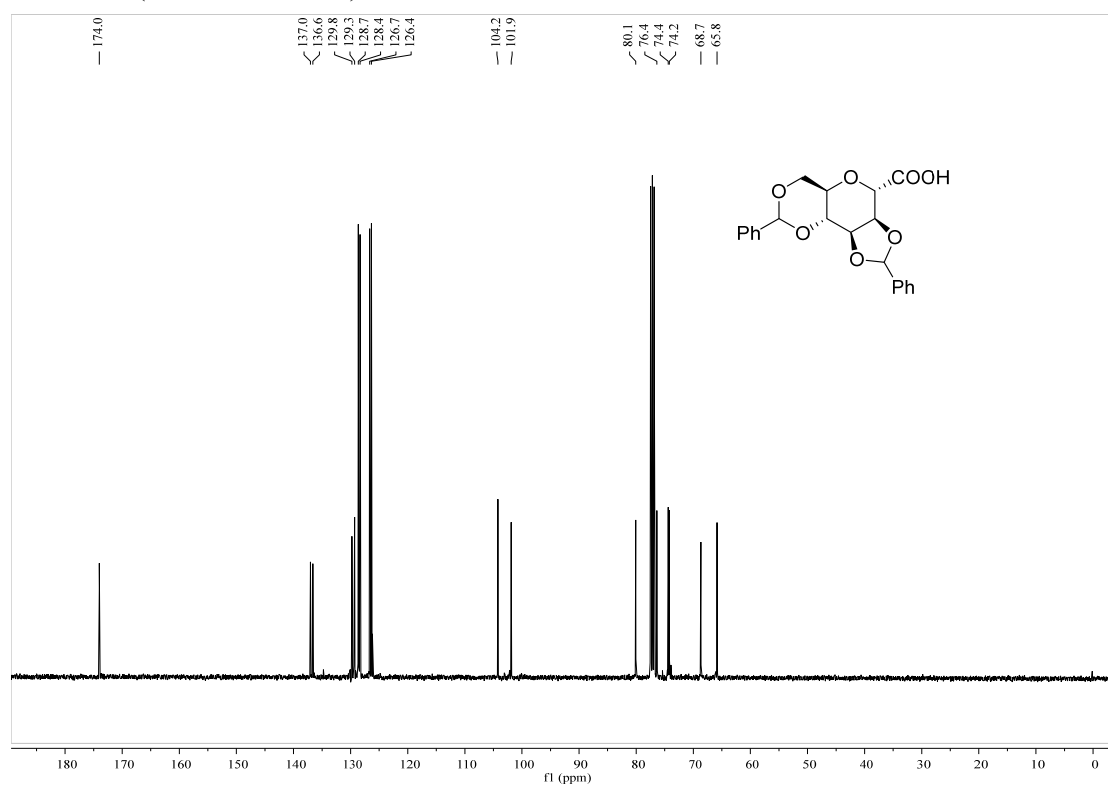
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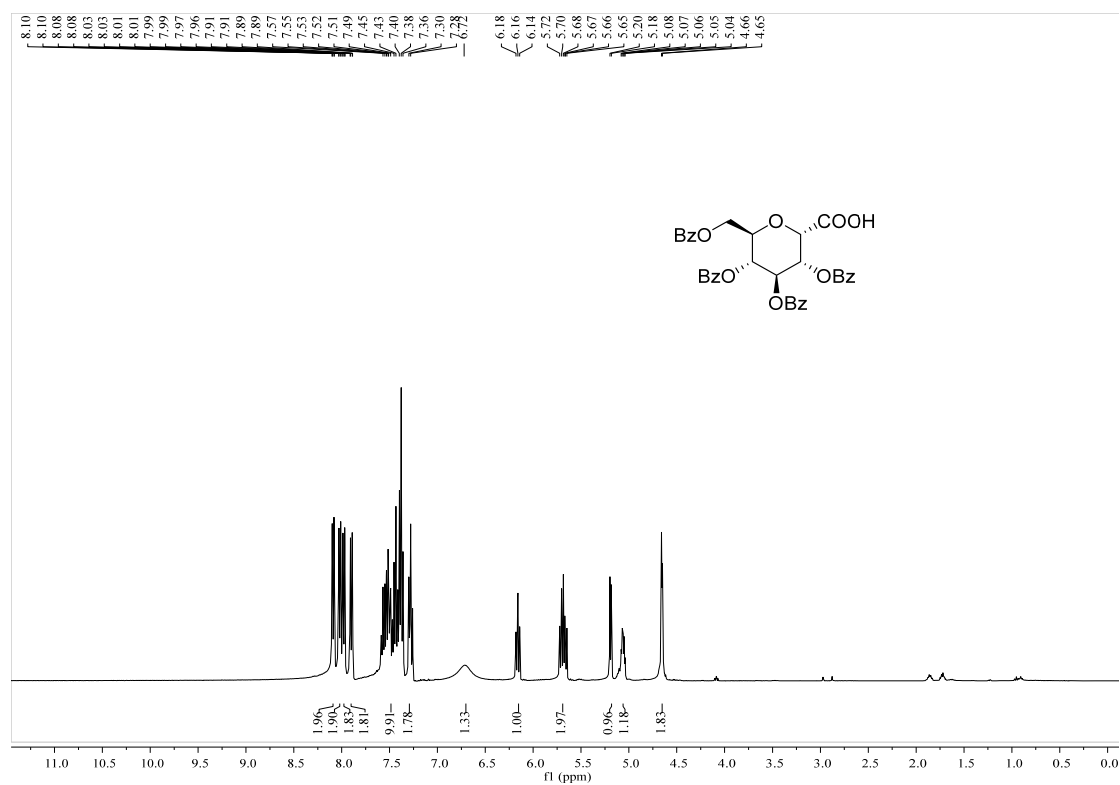
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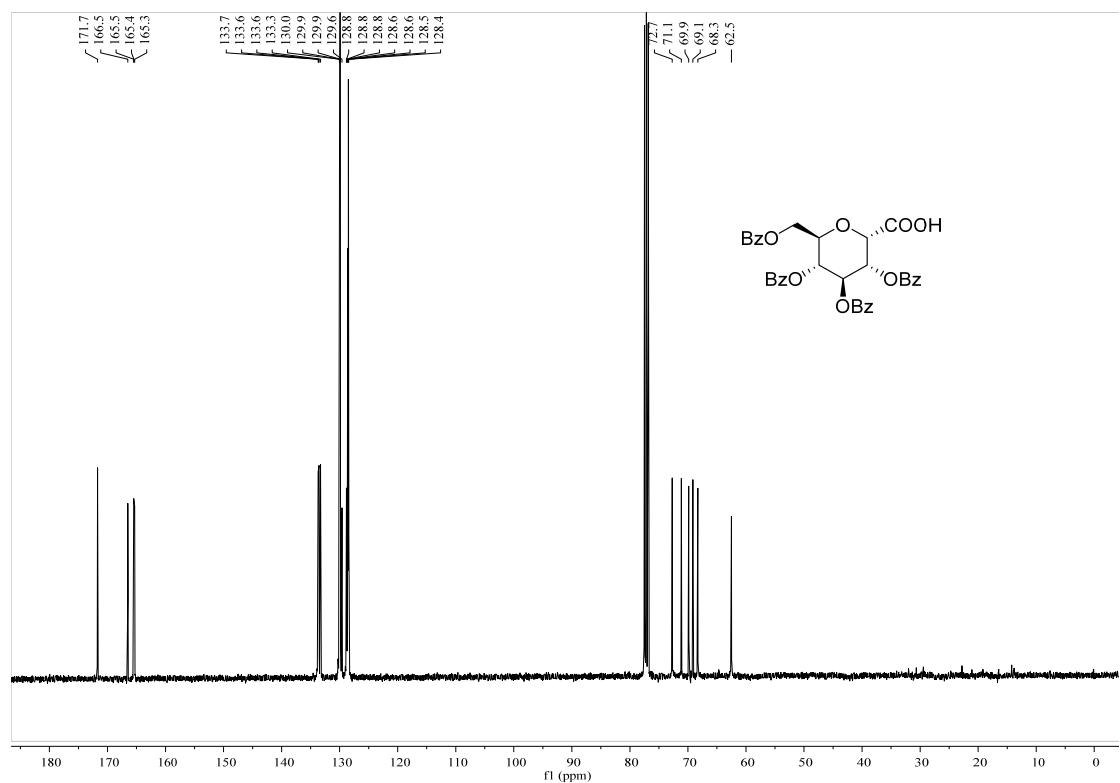
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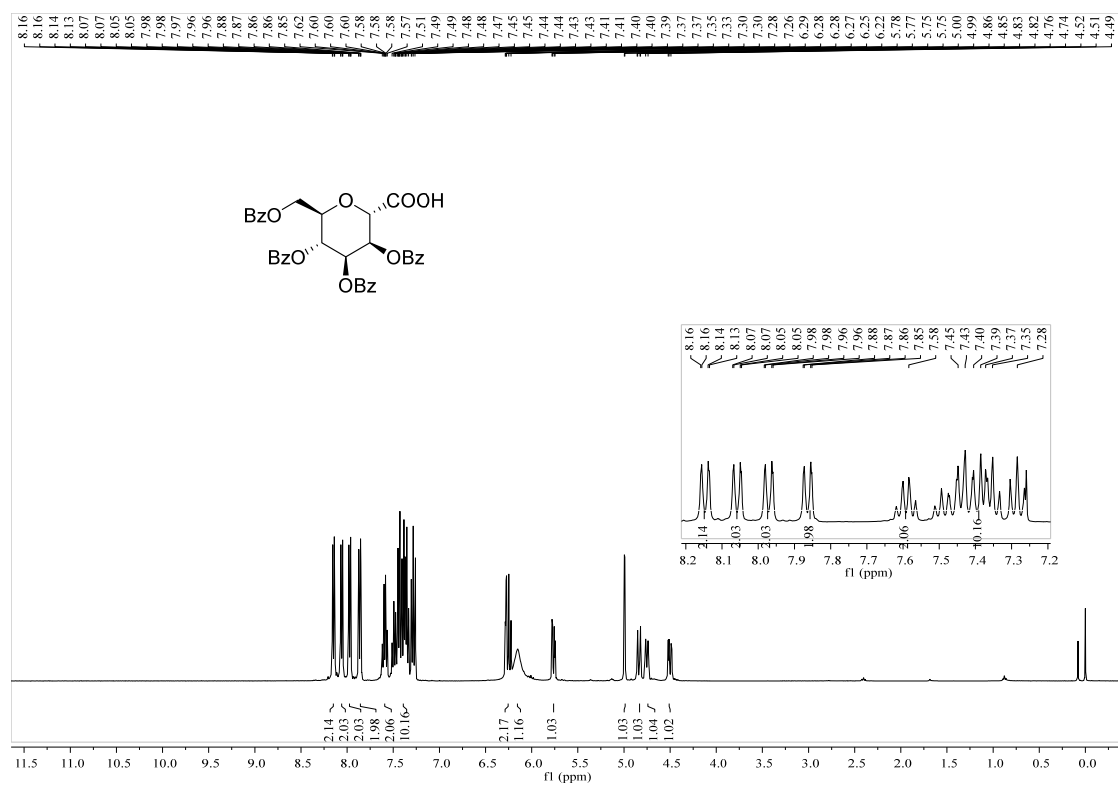
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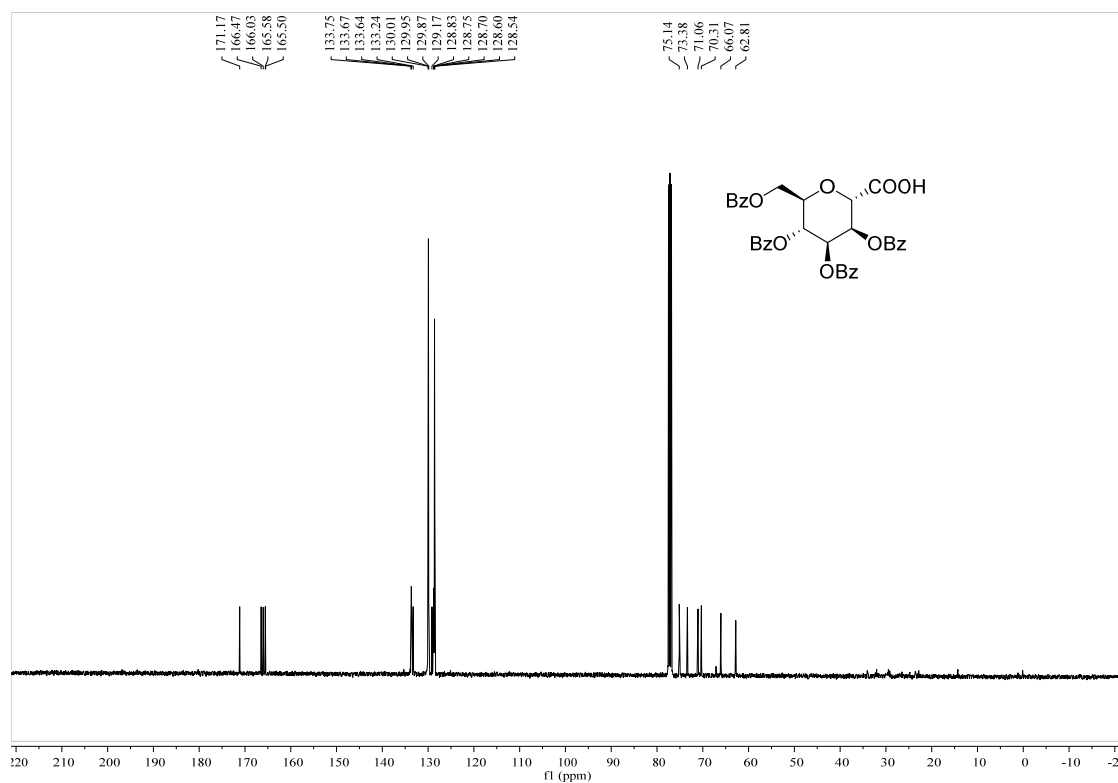
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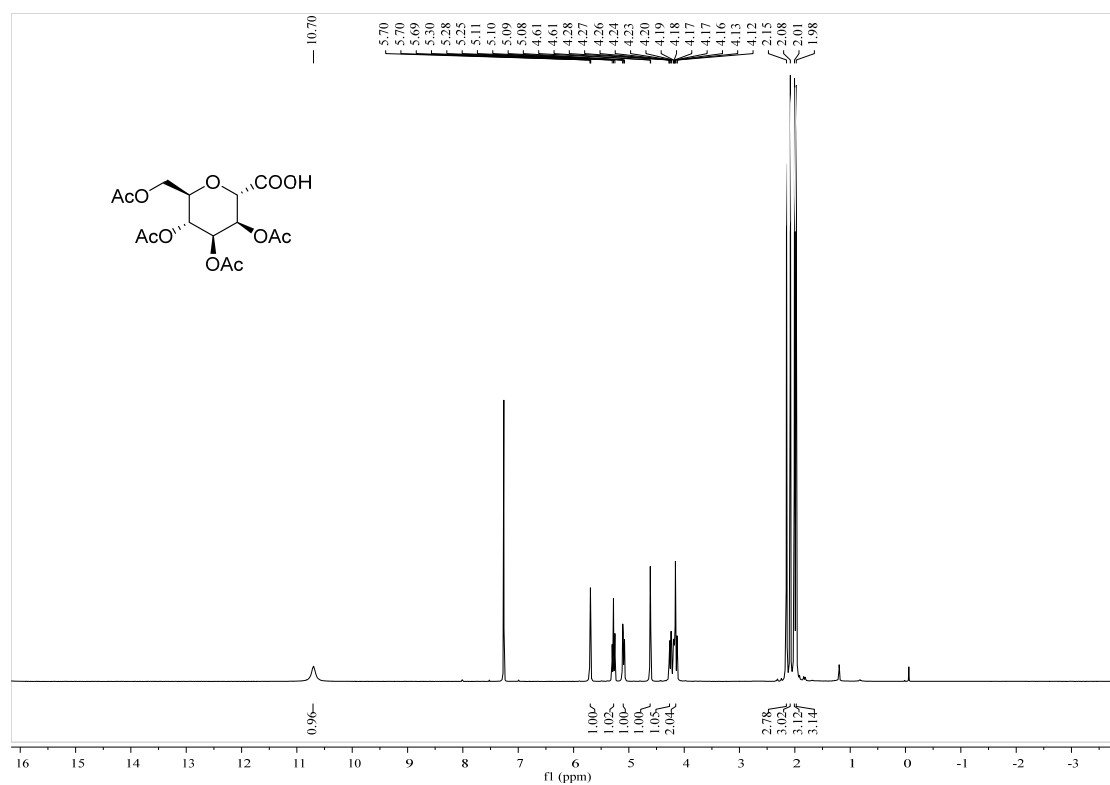
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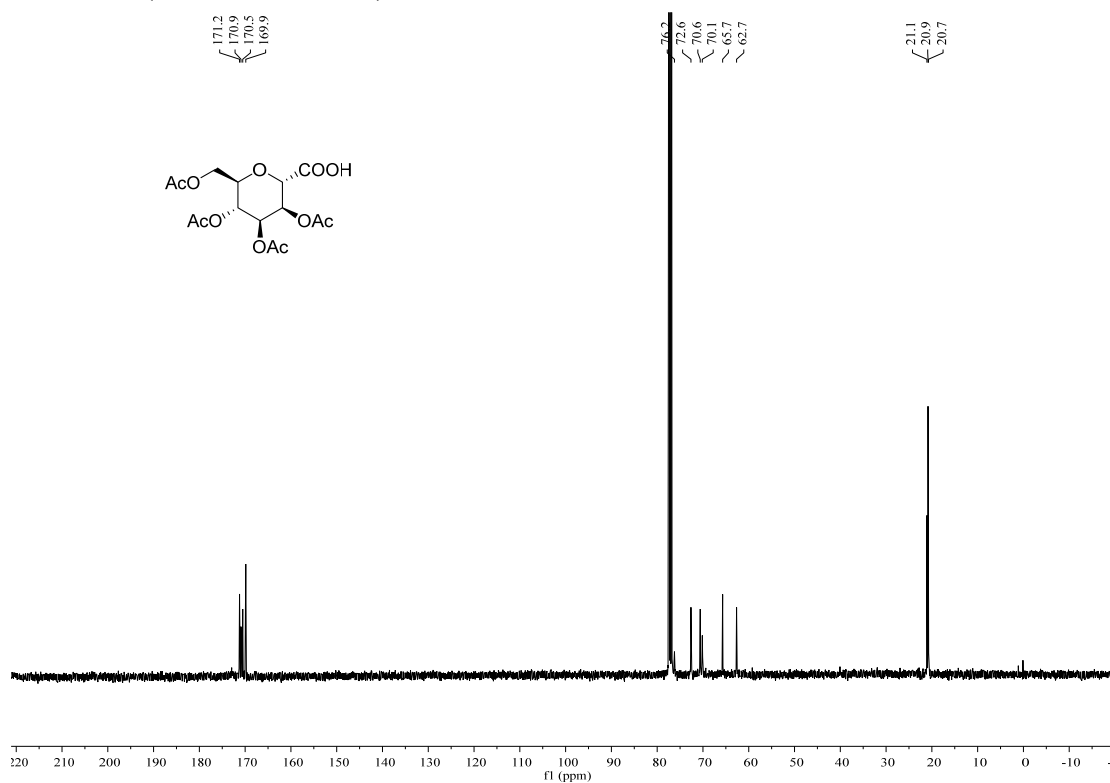
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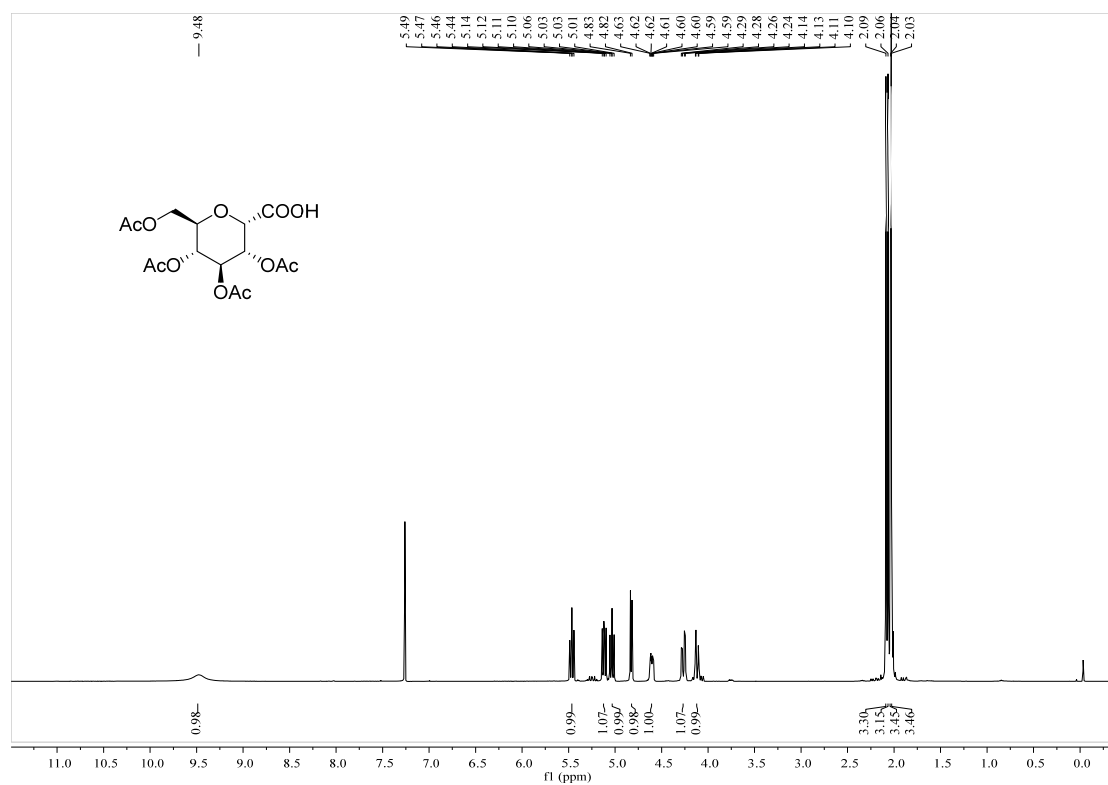
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¹³C NMR (100 MHz, CDCl₃) of **16**



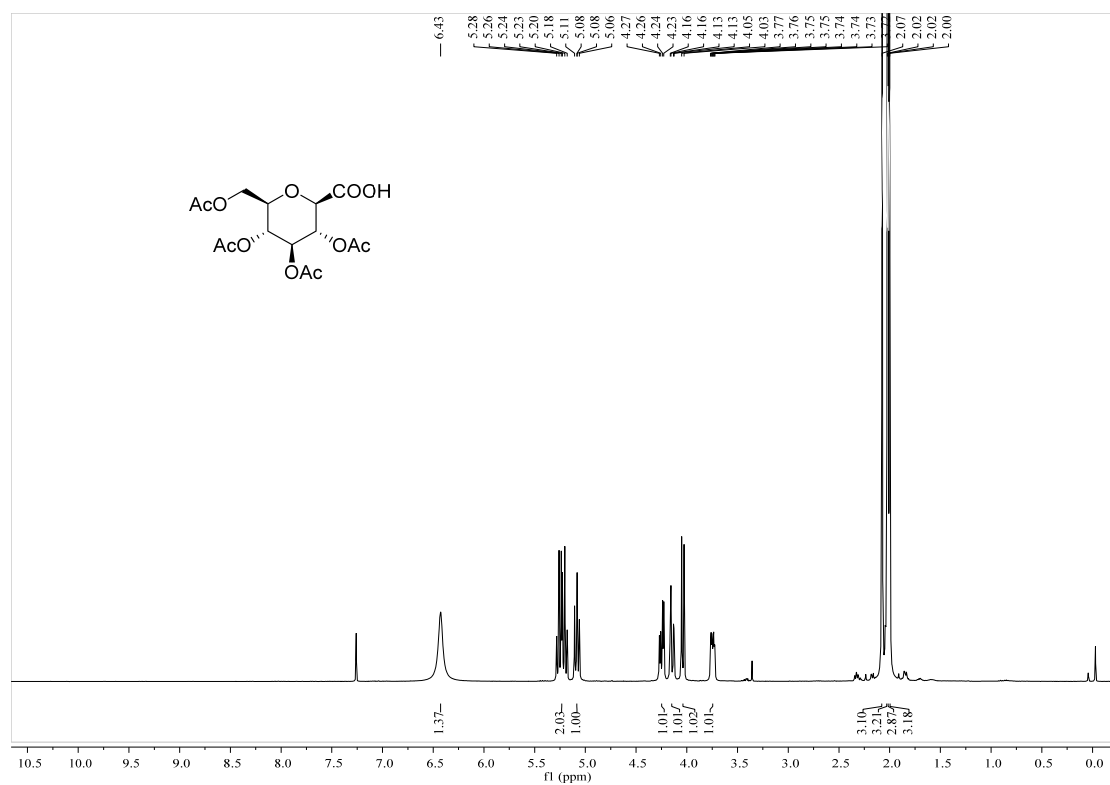
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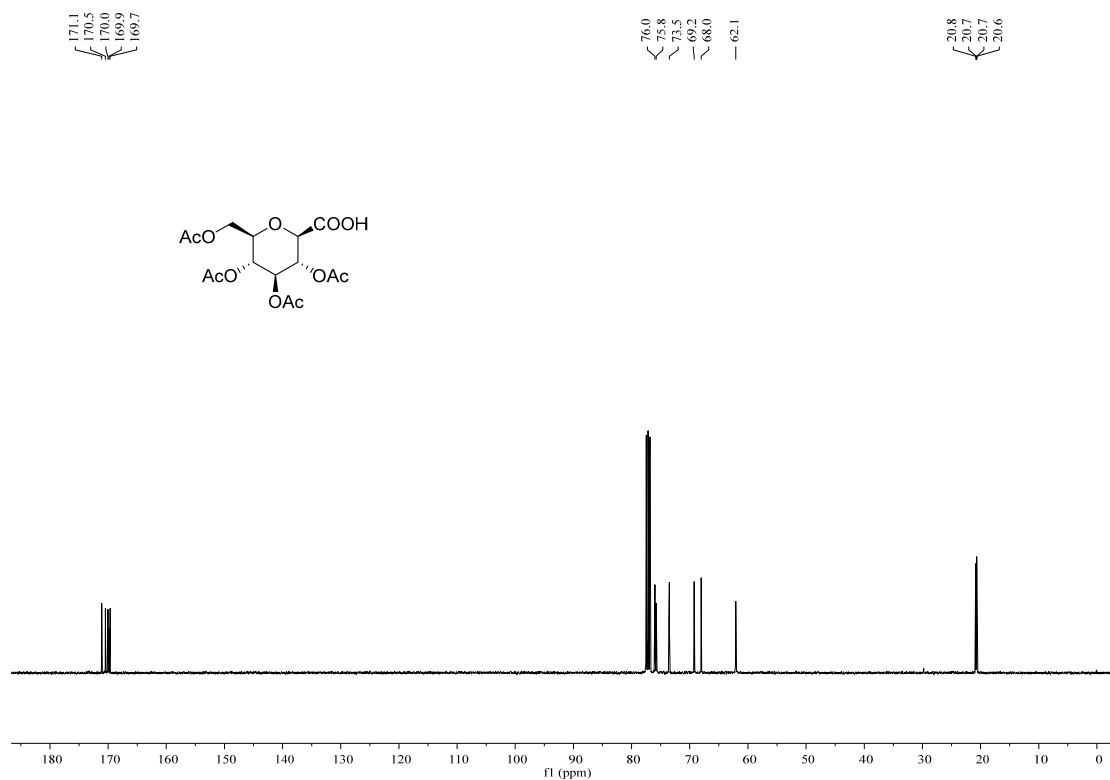
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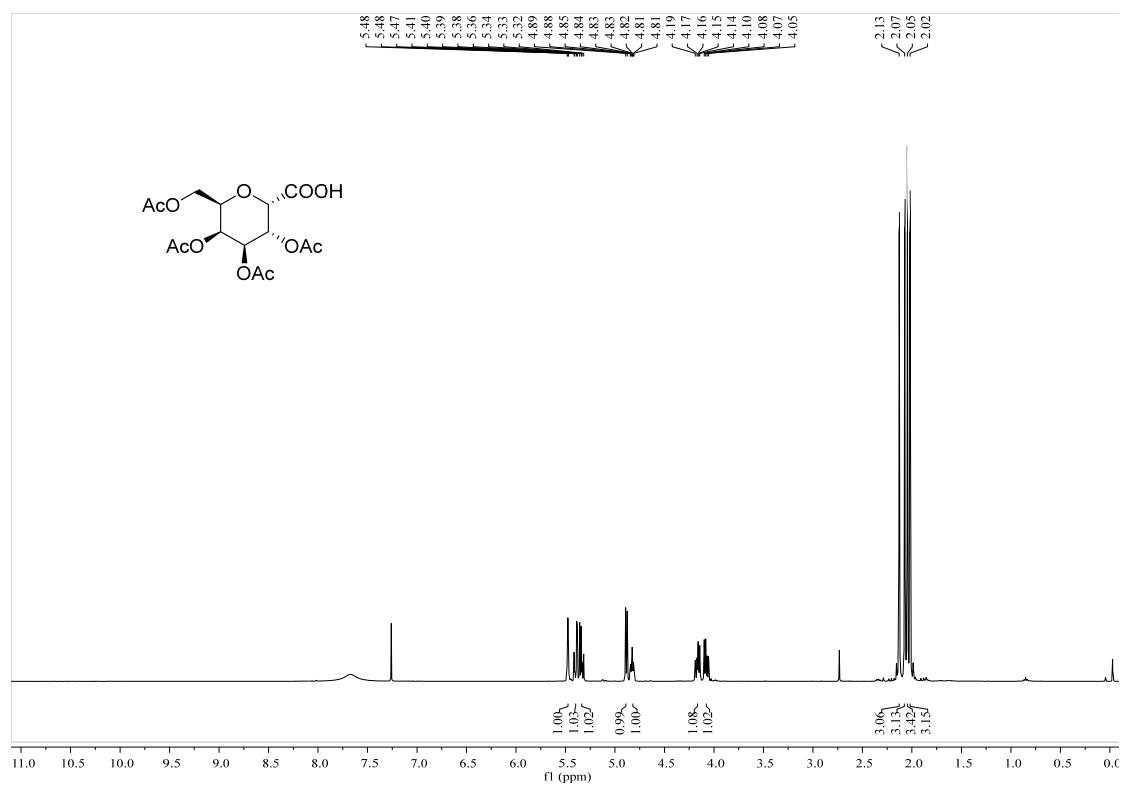
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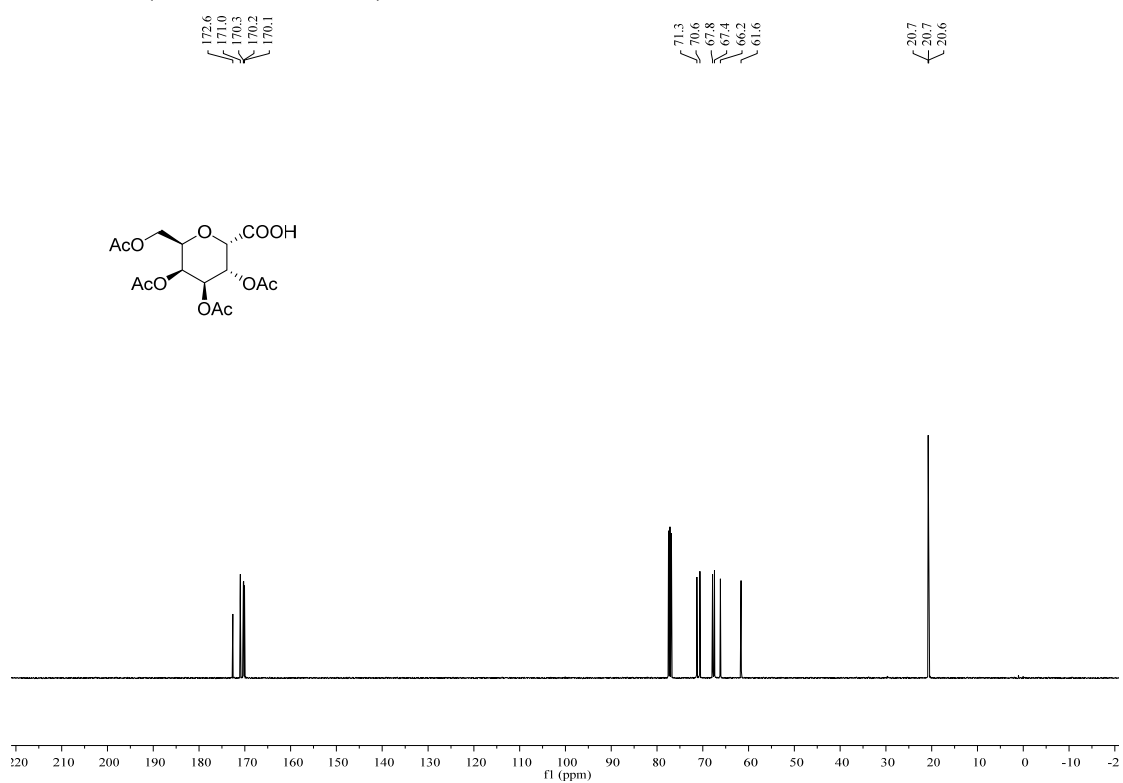
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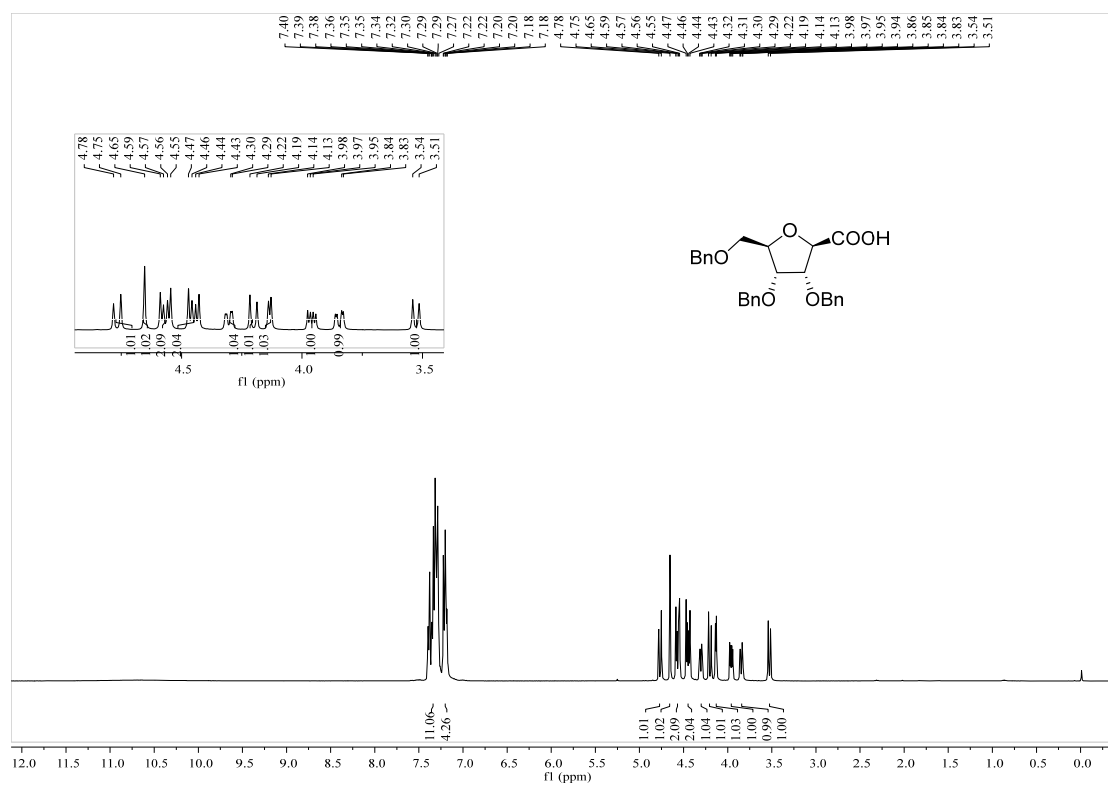
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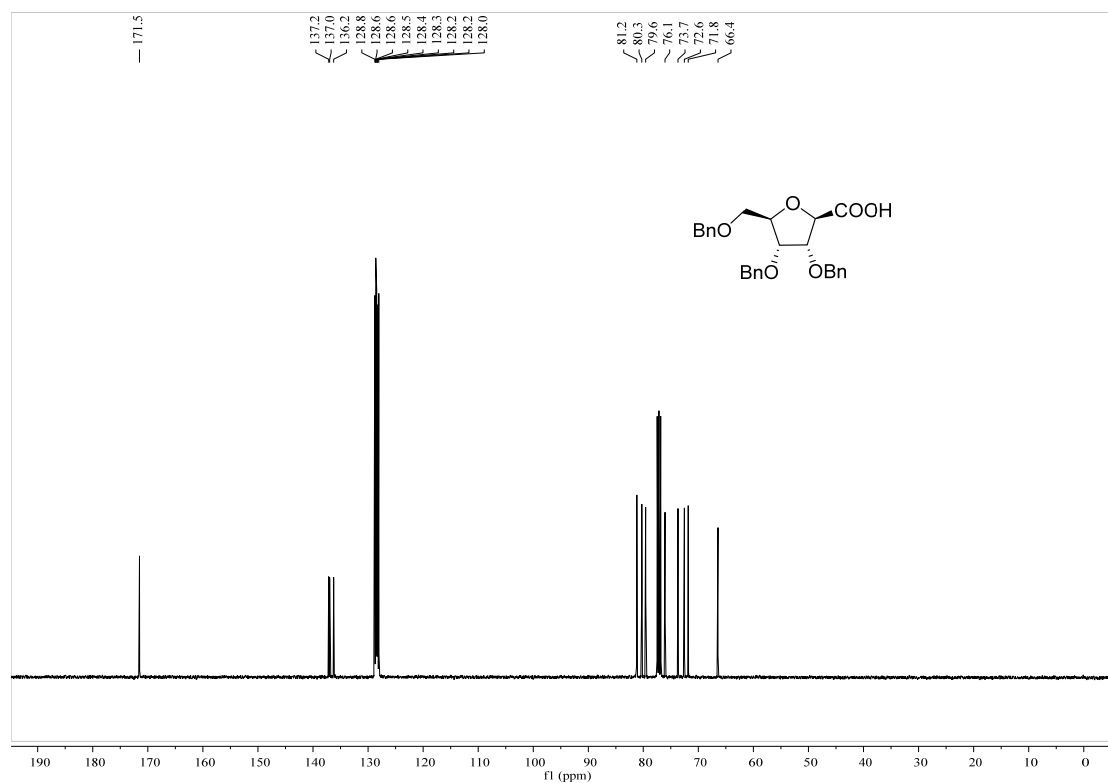
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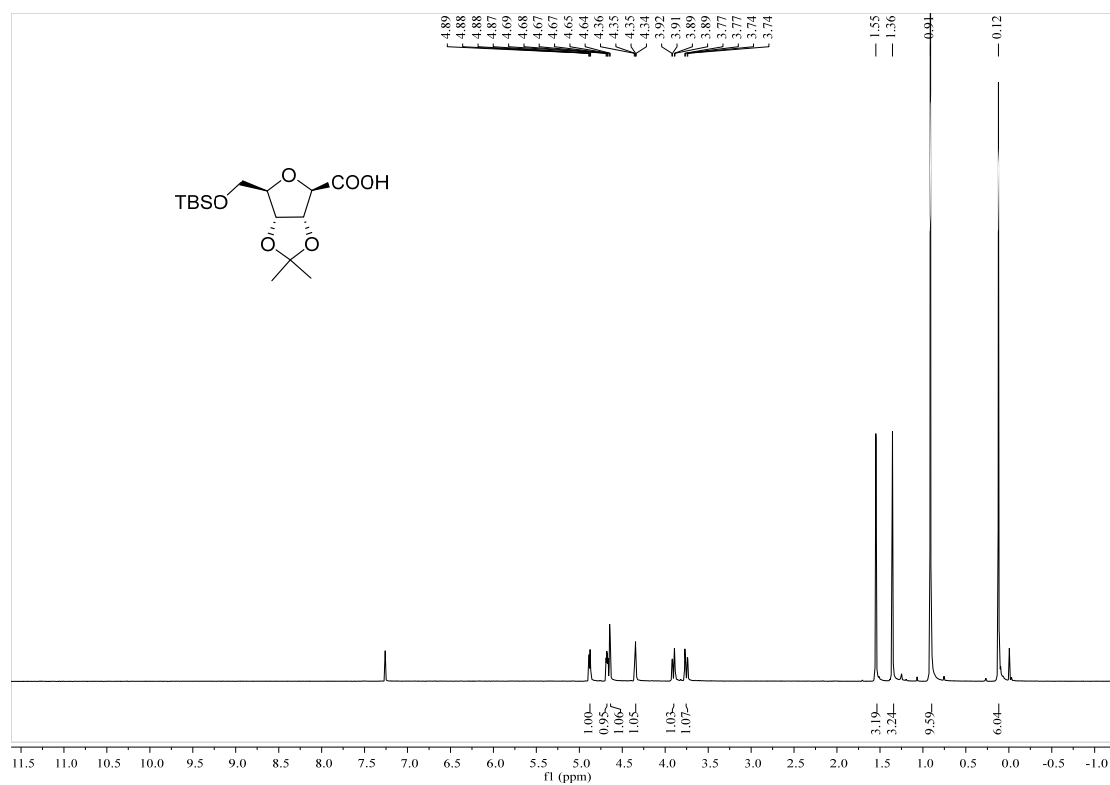
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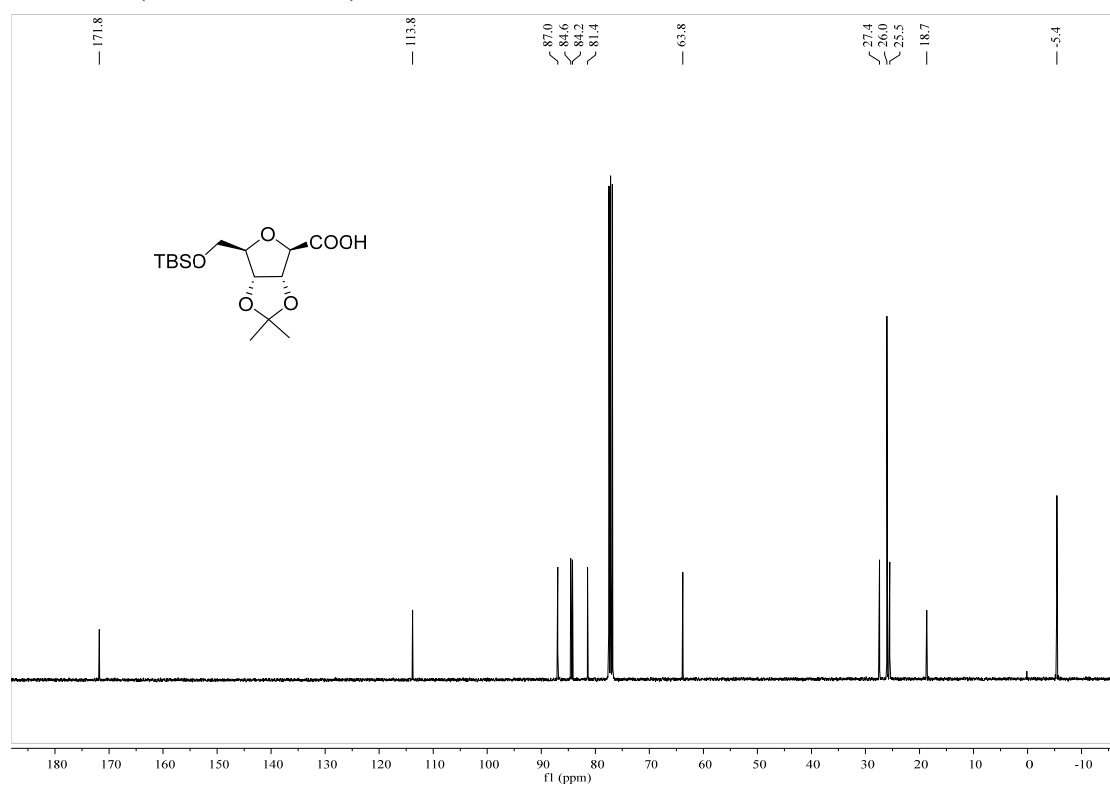
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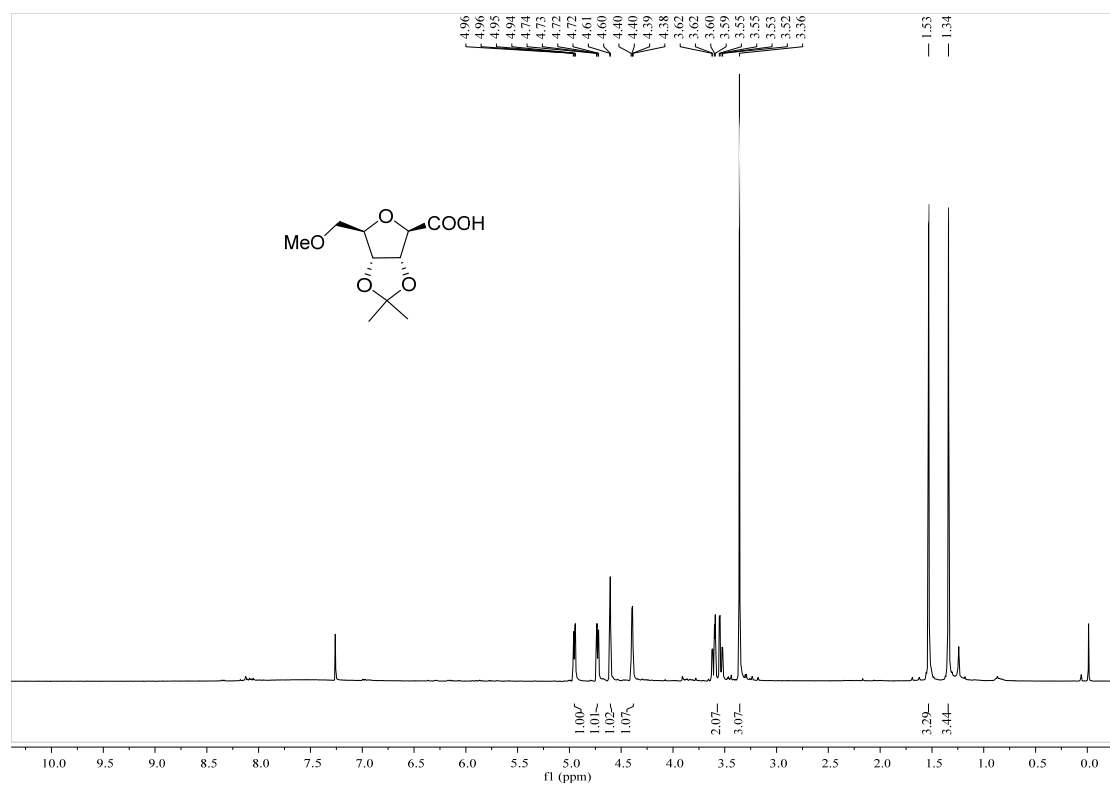
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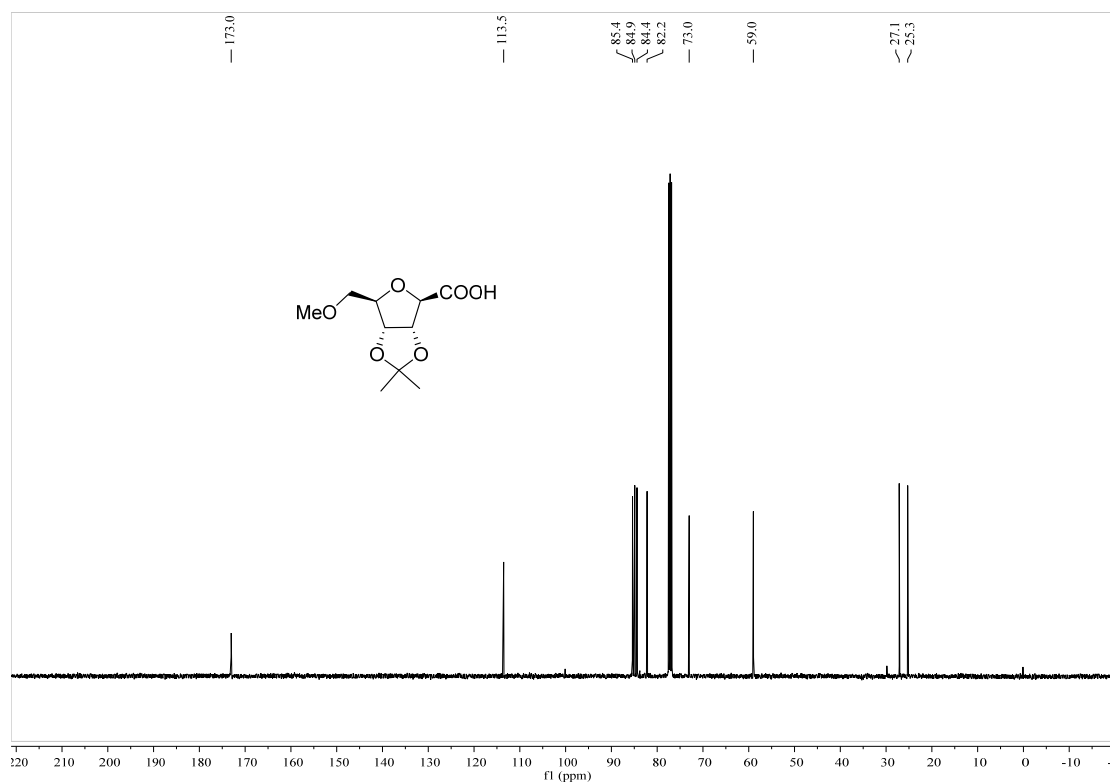
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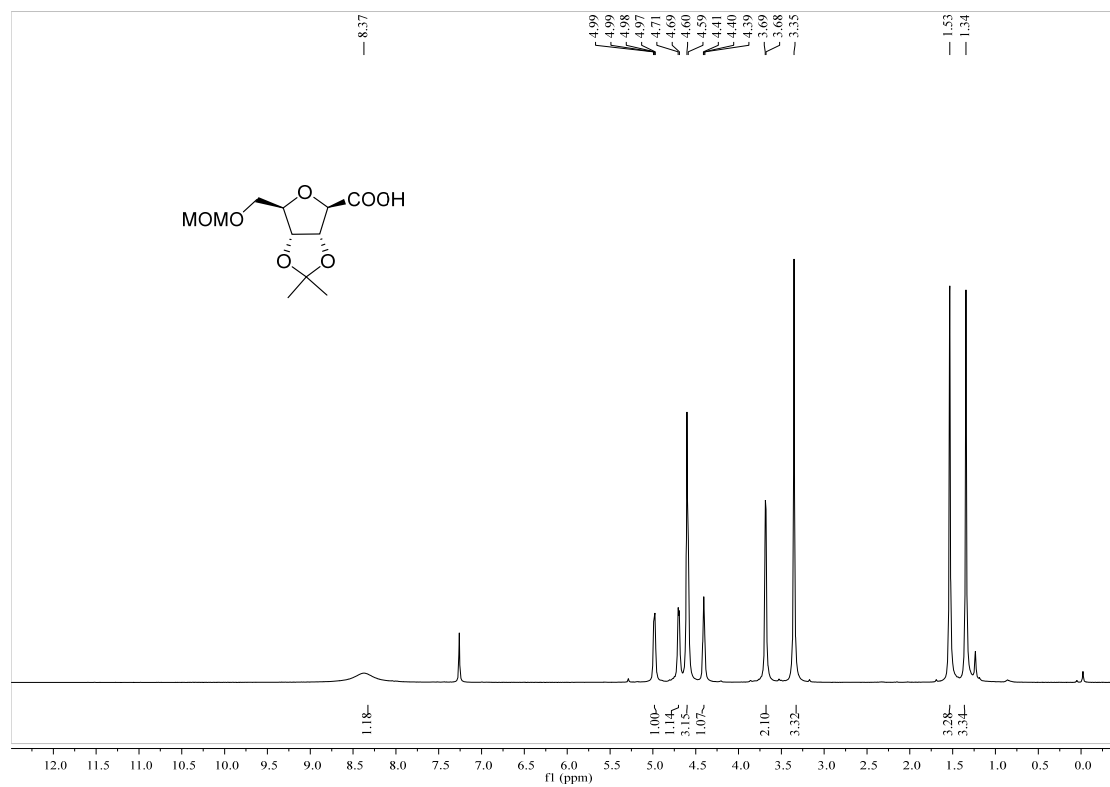
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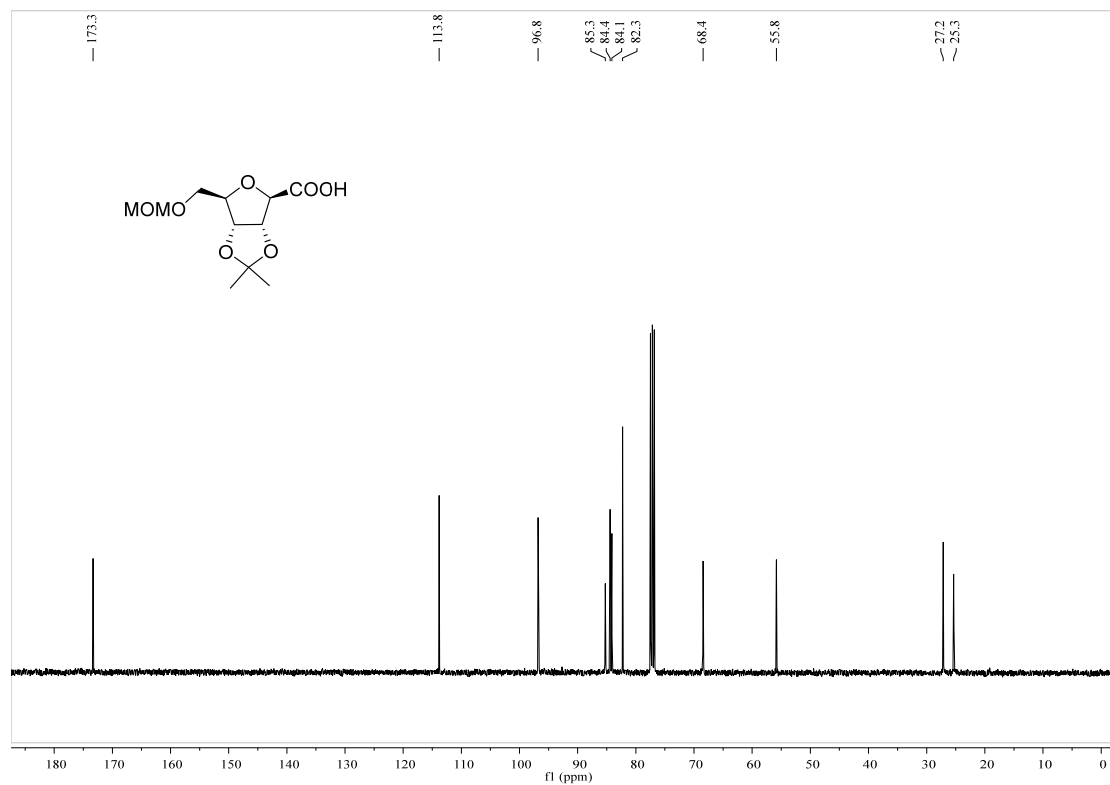
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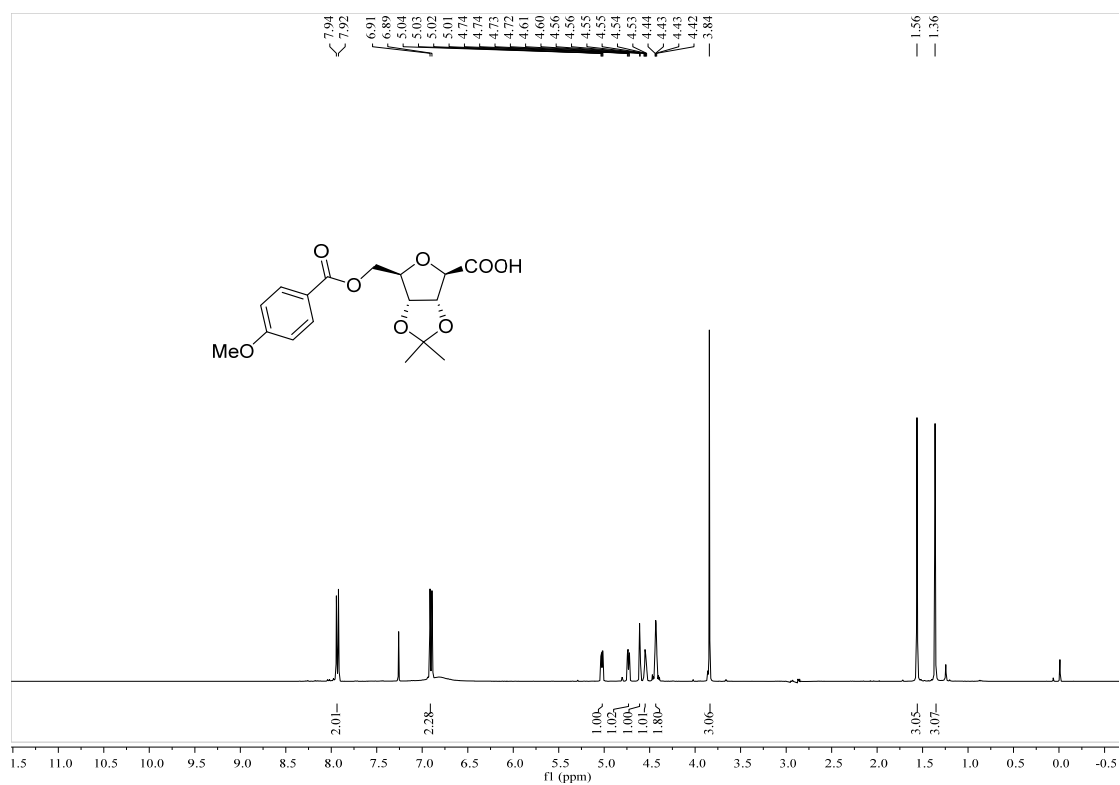
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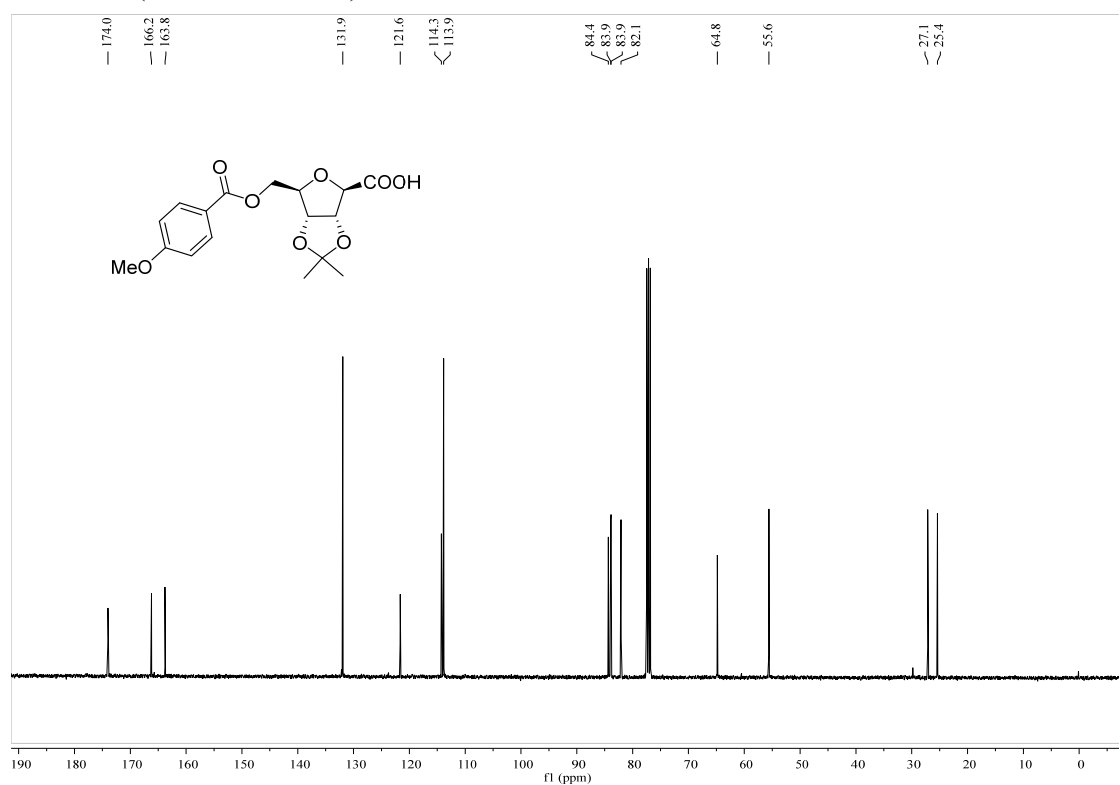
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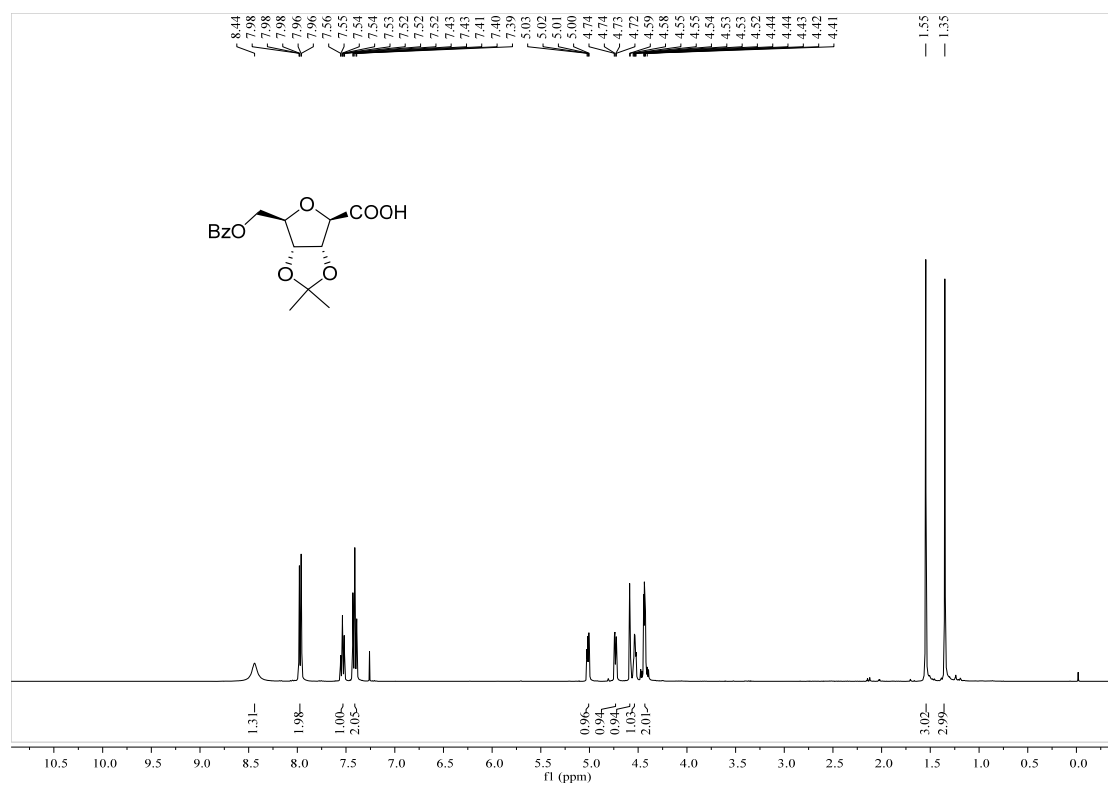
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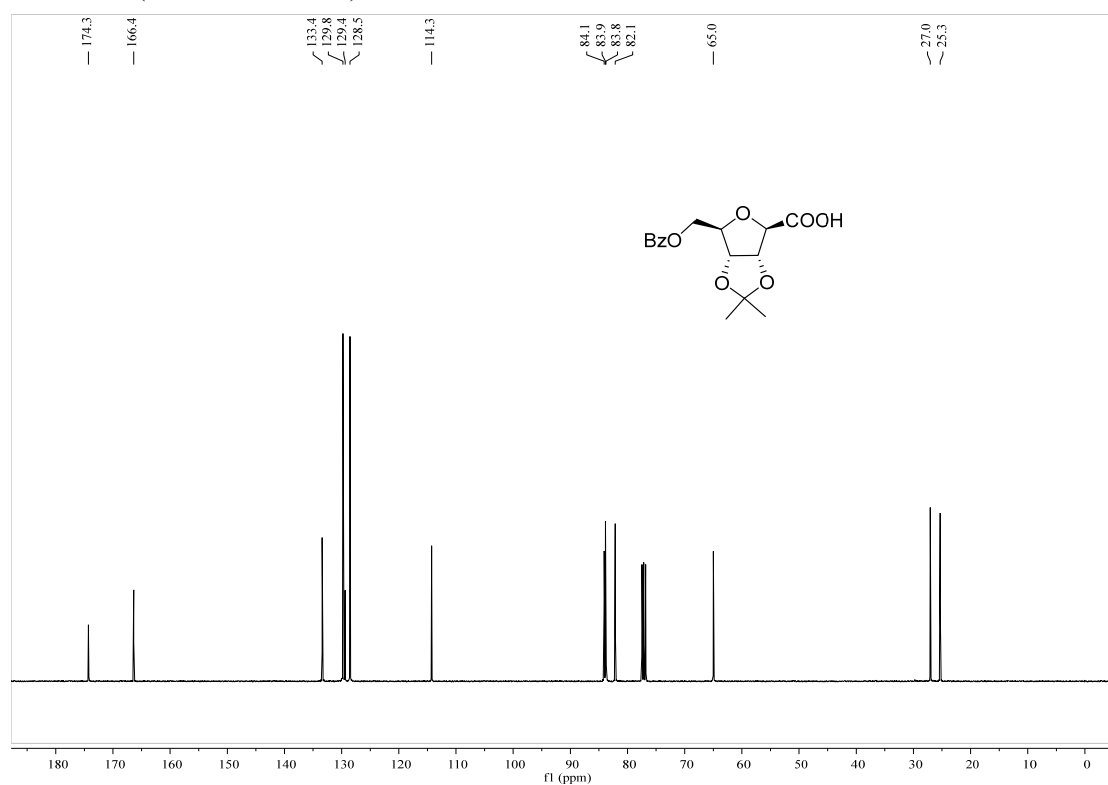
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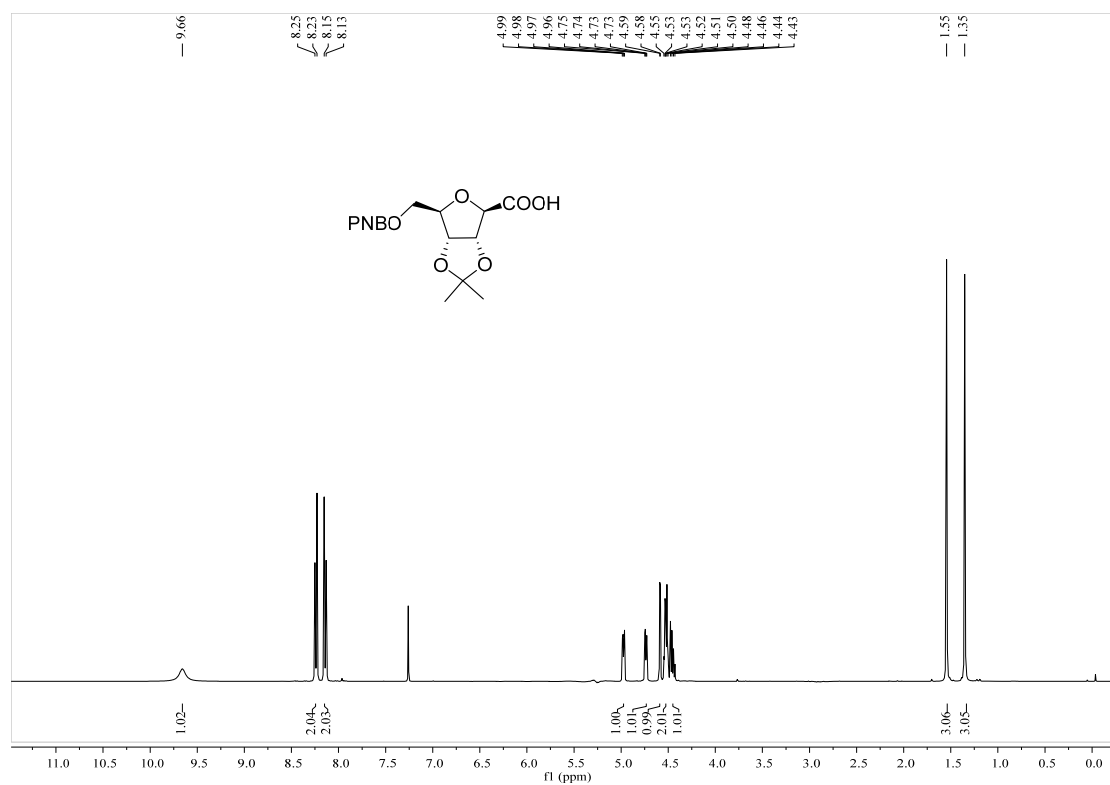
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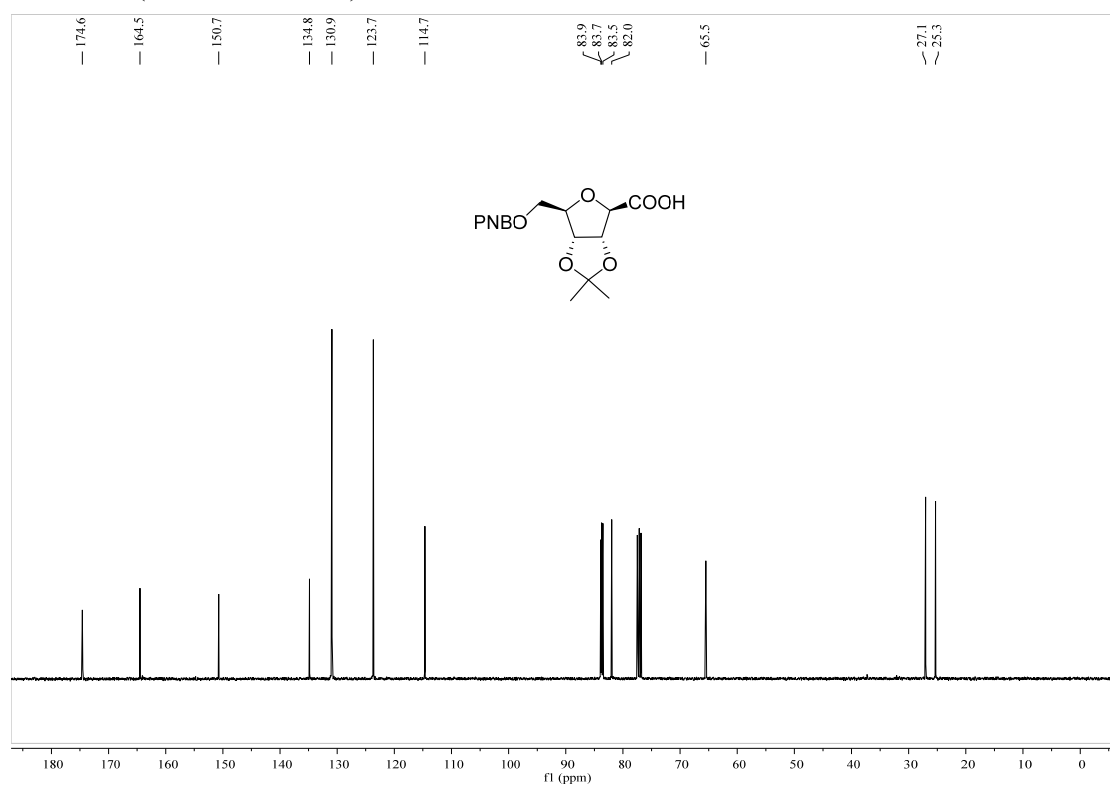
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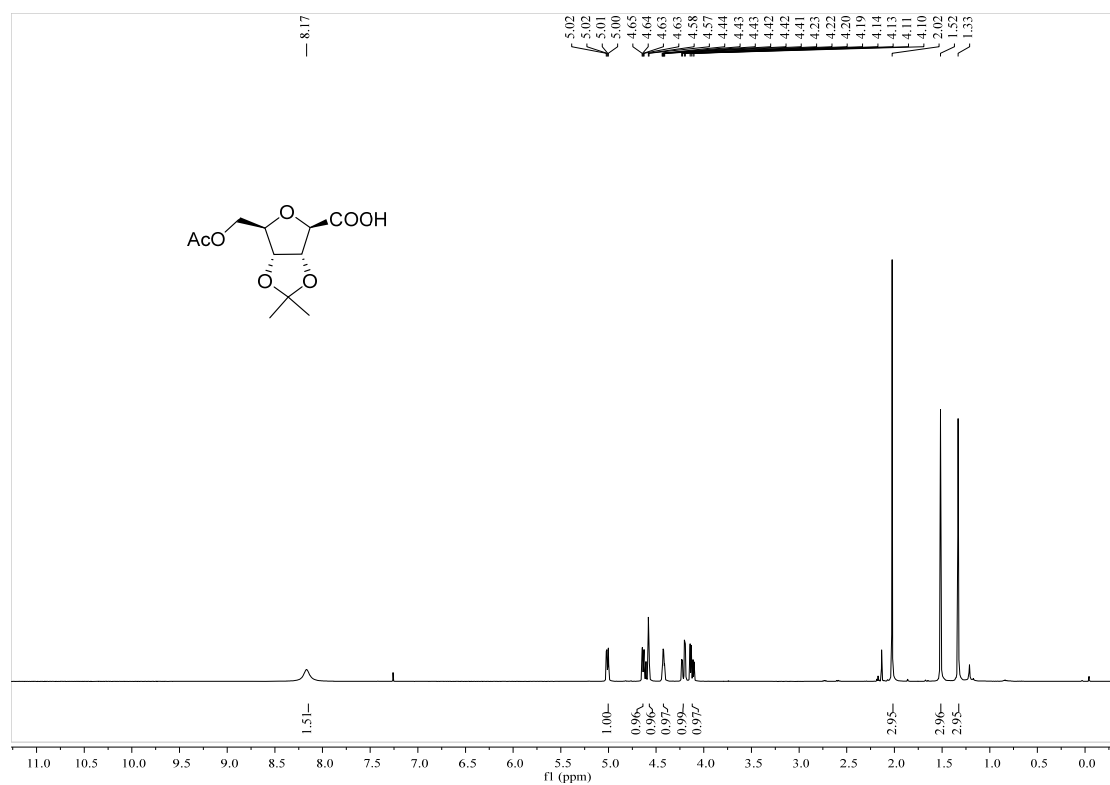
¹H NMR (400 MHz, CDCl₃) of 26



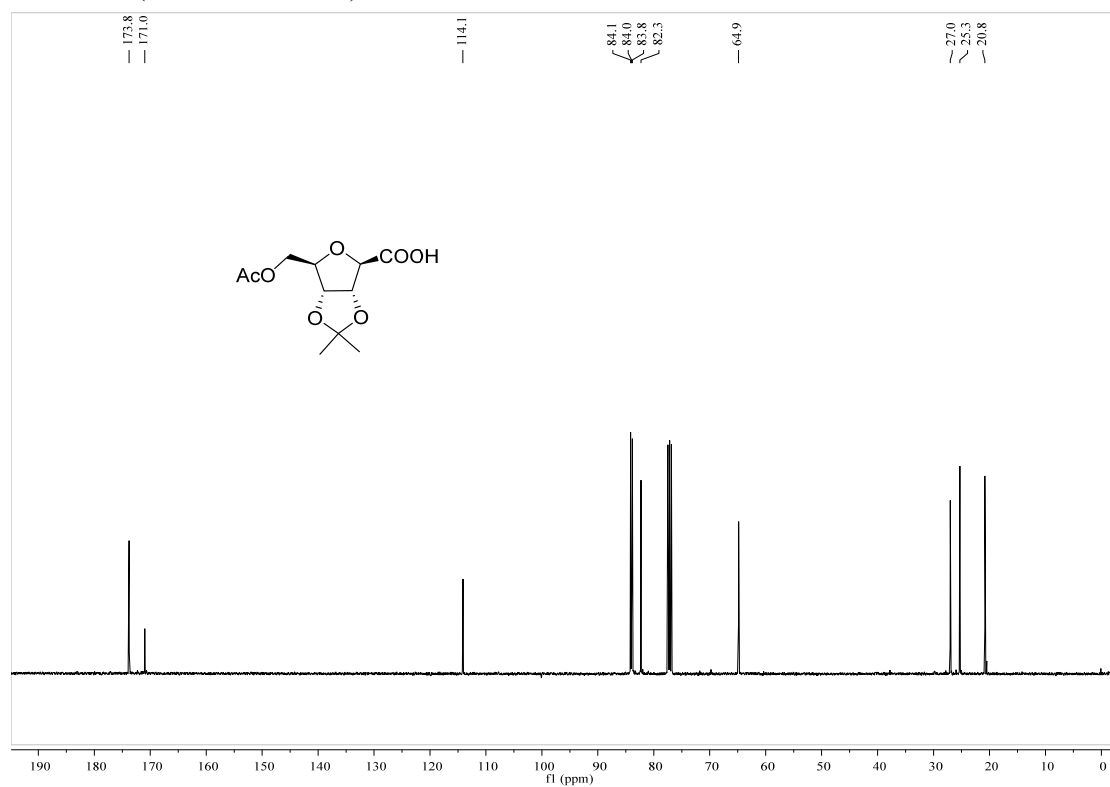
¹³C NMR (100 MHz, CDCl₃) of 26



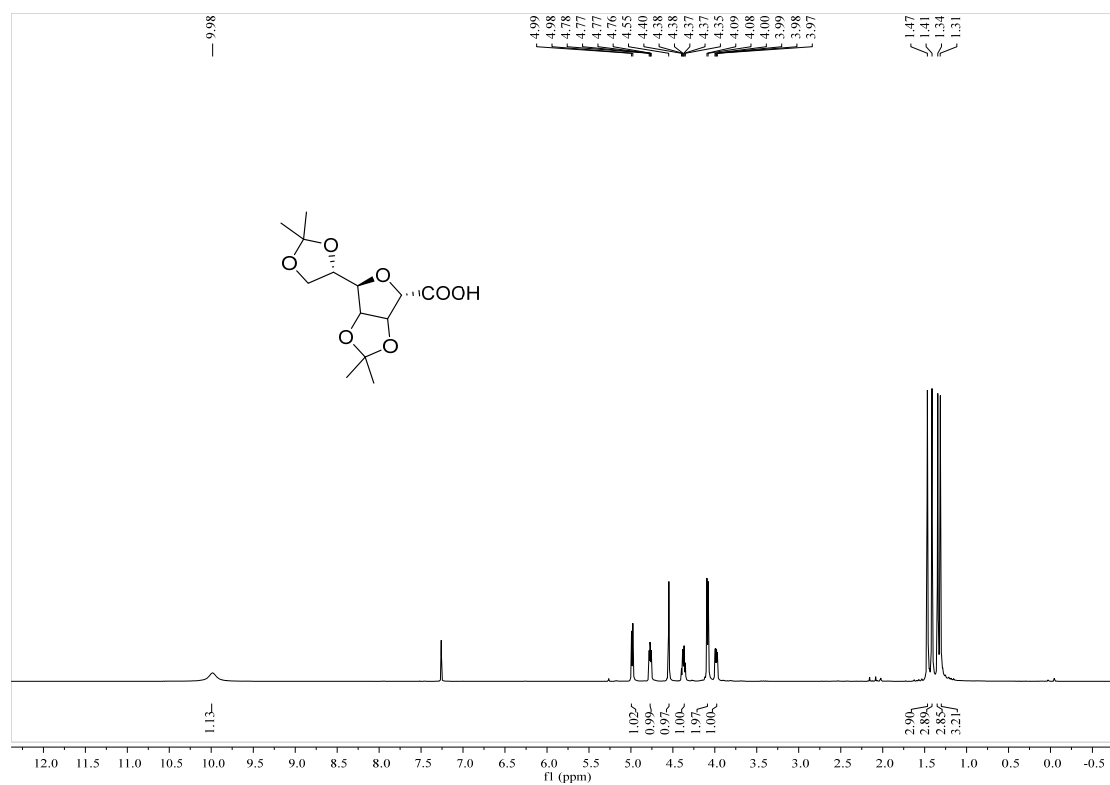
¹H NMR (400 MHz, CDCl₃) of 27



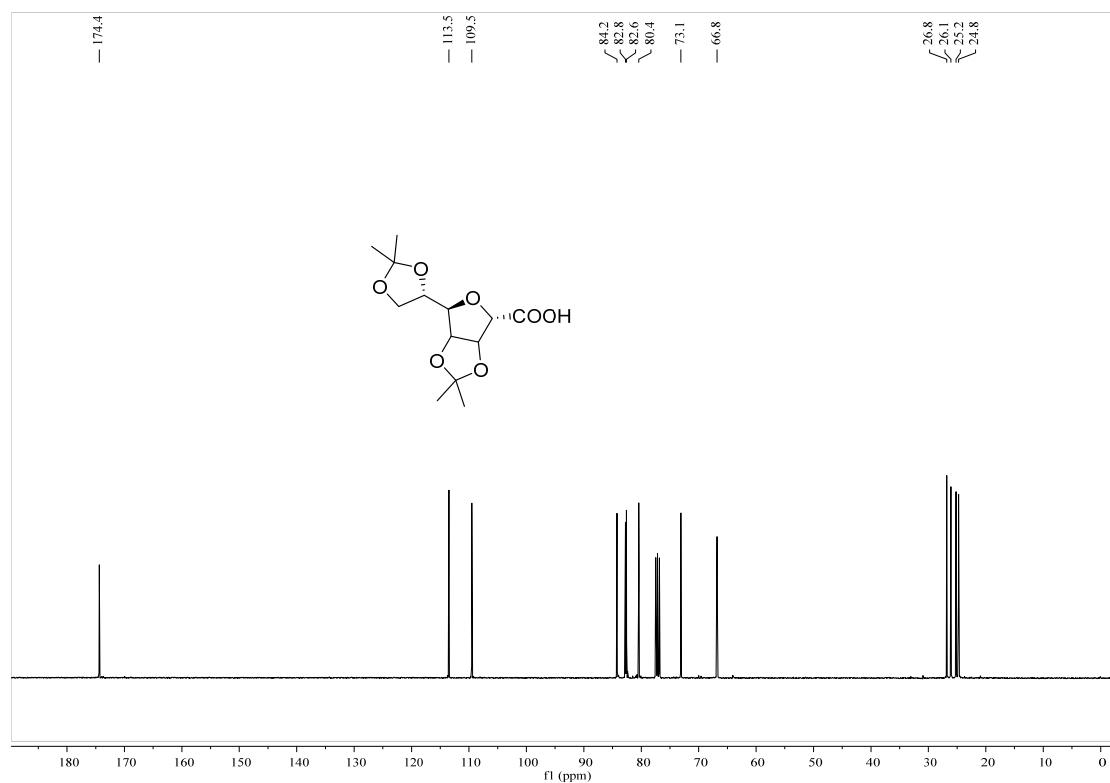
¹³C NMR (100 MHz, CDCl₃) of 27



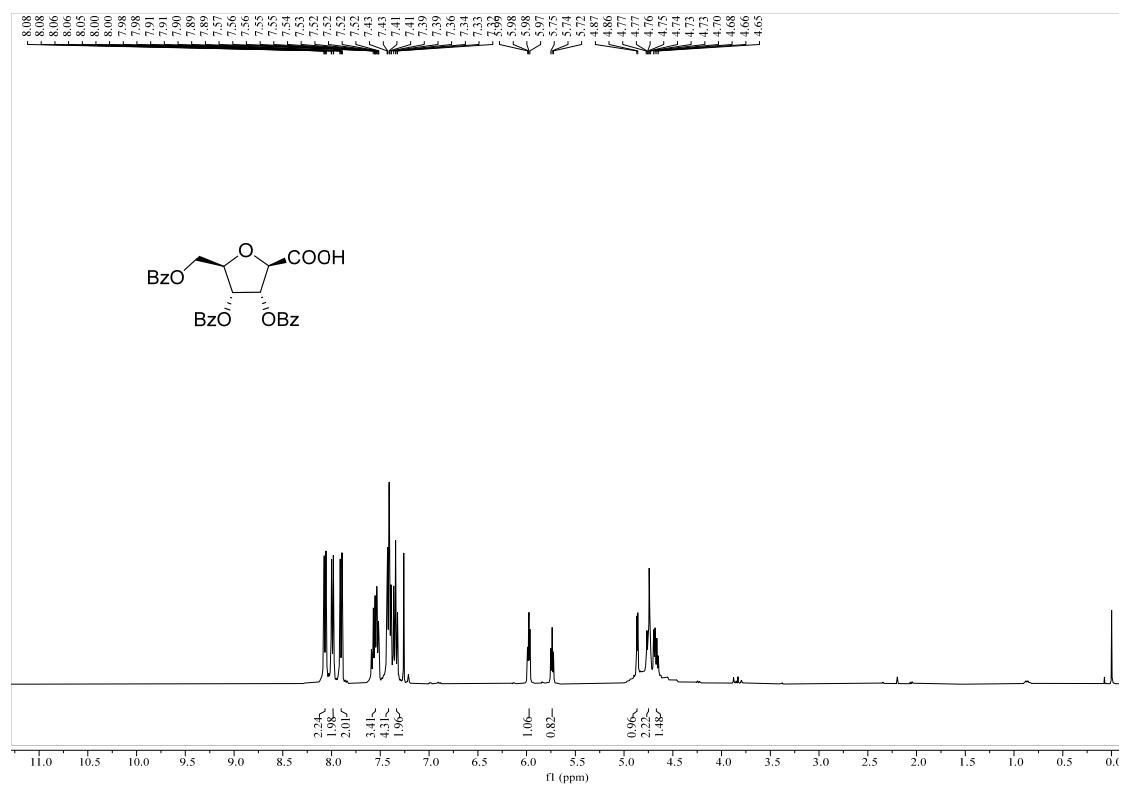
¹H NMR (400 MHz, CDCl₃) of 28



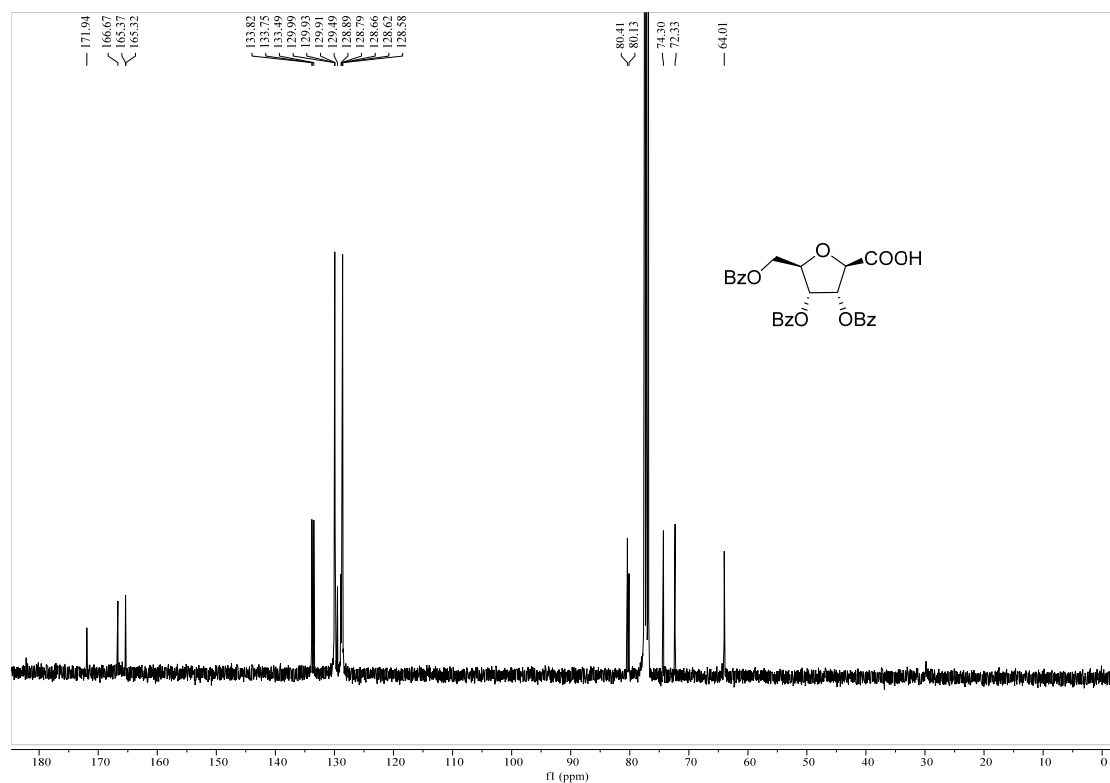
¹³C NMR (100 MHz, CDCl₃) of 28



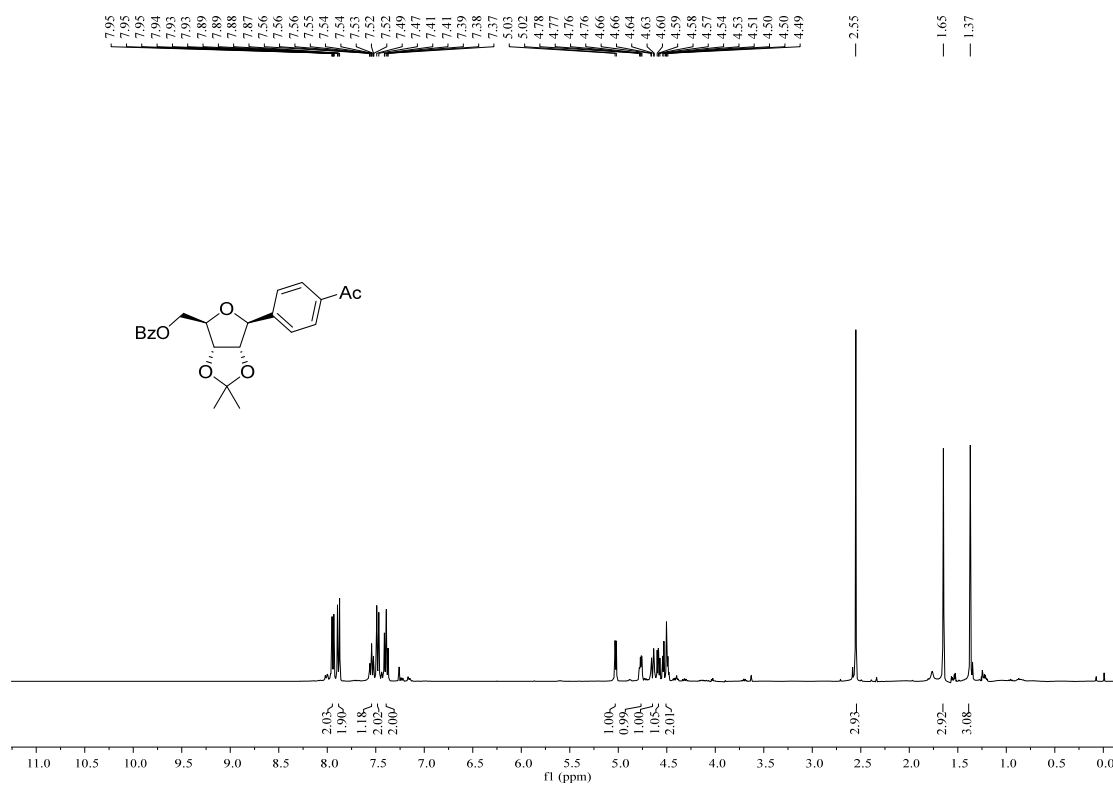
^1H NMR (400 MHz, CDCl_3) of **29**



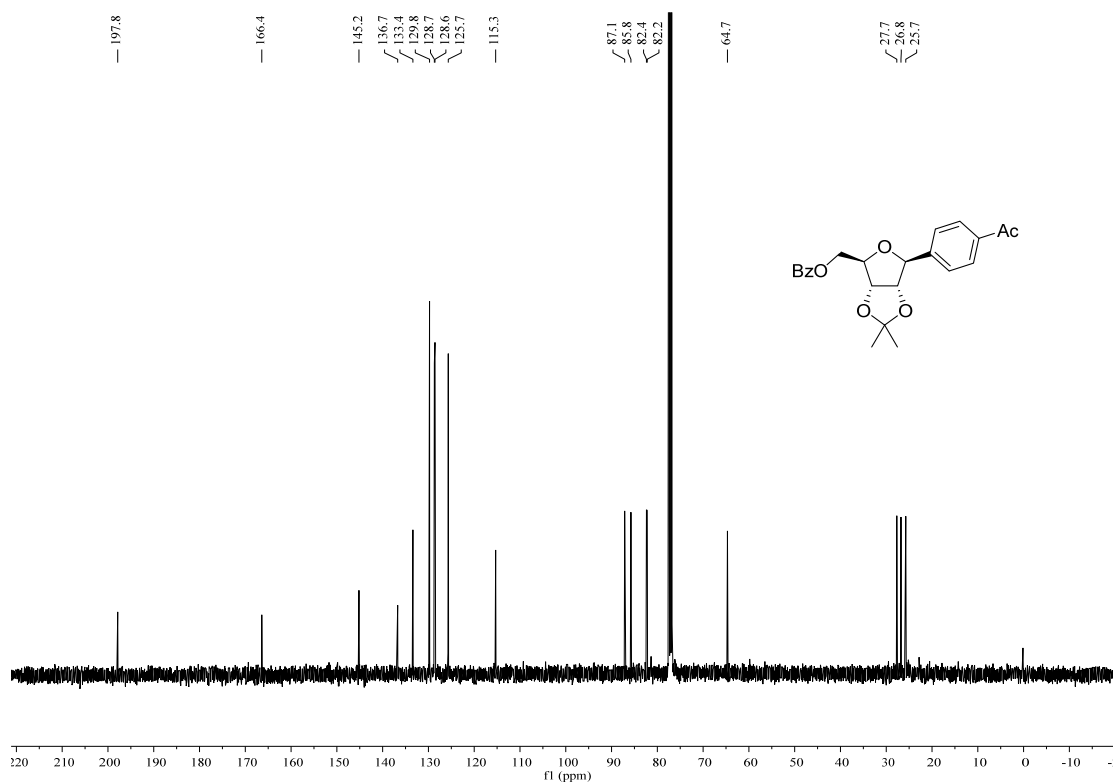
^{13}C NMR (100 MHz, CDCl_3) of **29**



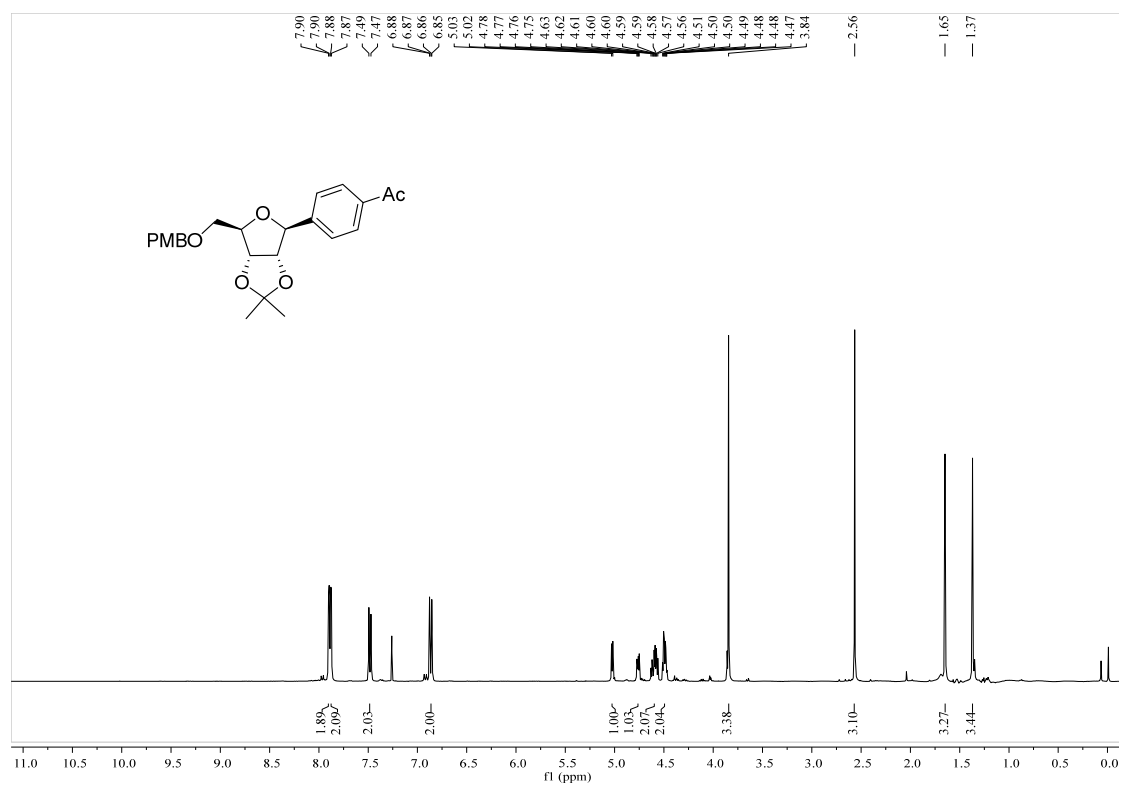
¹H NMR (400 MHz, CDCl₃) of 31



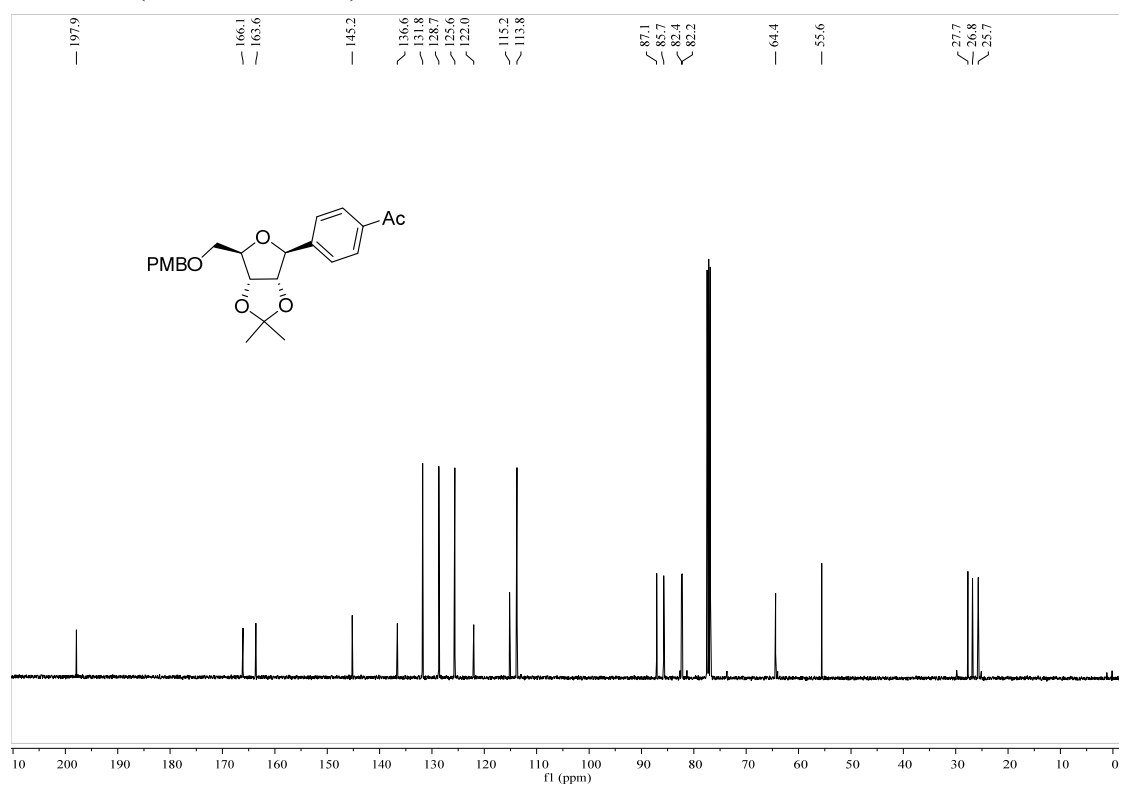
¹³C NMR (100 MHz, CDCl₃) of 31



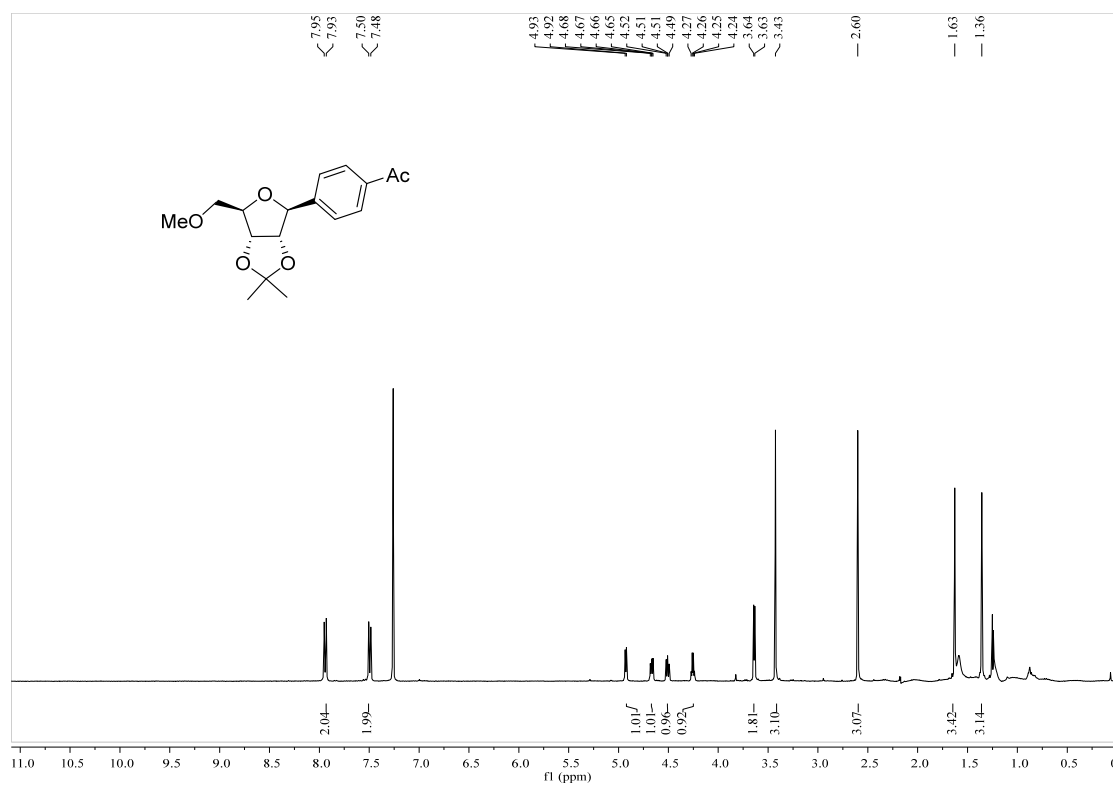
¹H NMR (400 MHz, CDCl₃) of **32**



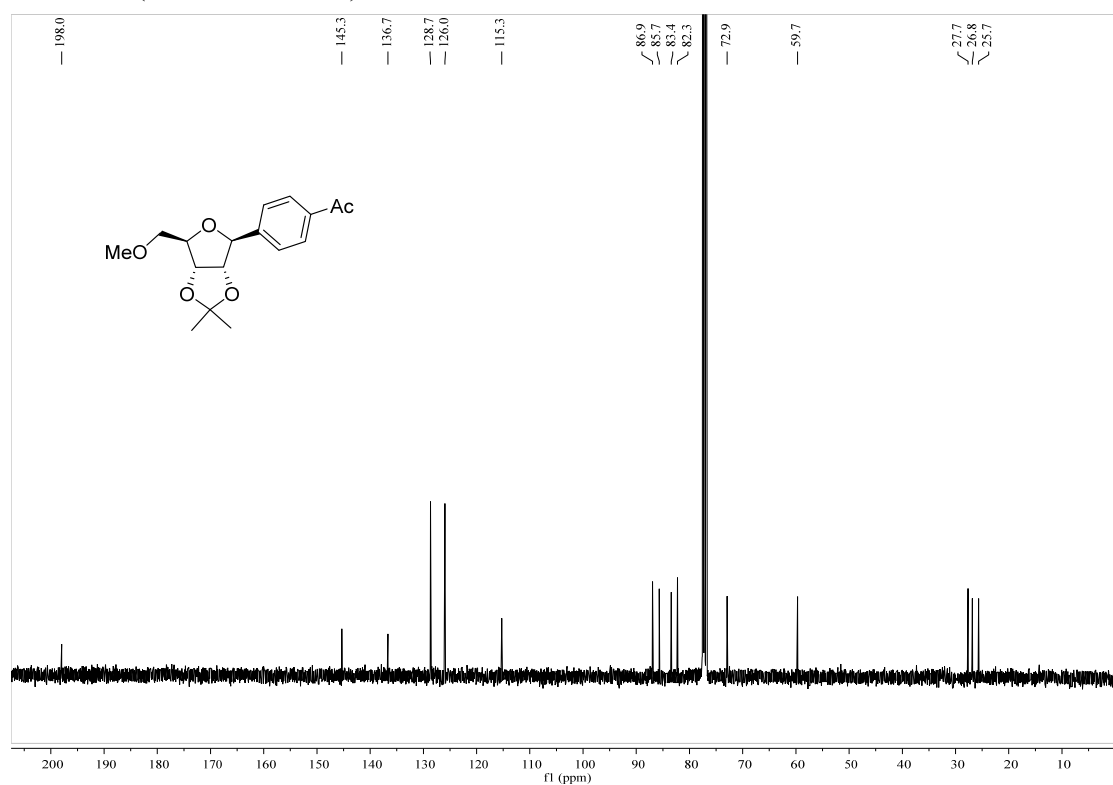
¹³C NMR (100 MHz, CDCl₃) of **32**



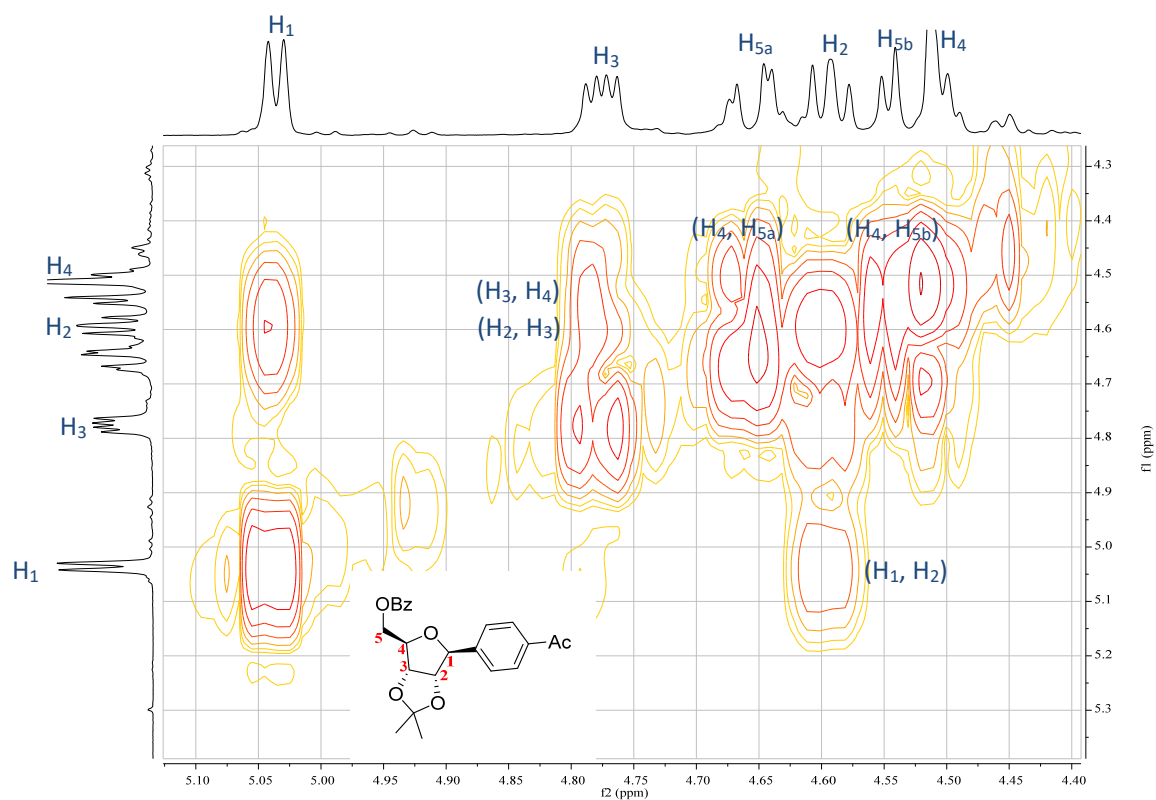
¹H NMR (400 MHz, CDCl₃) of 33



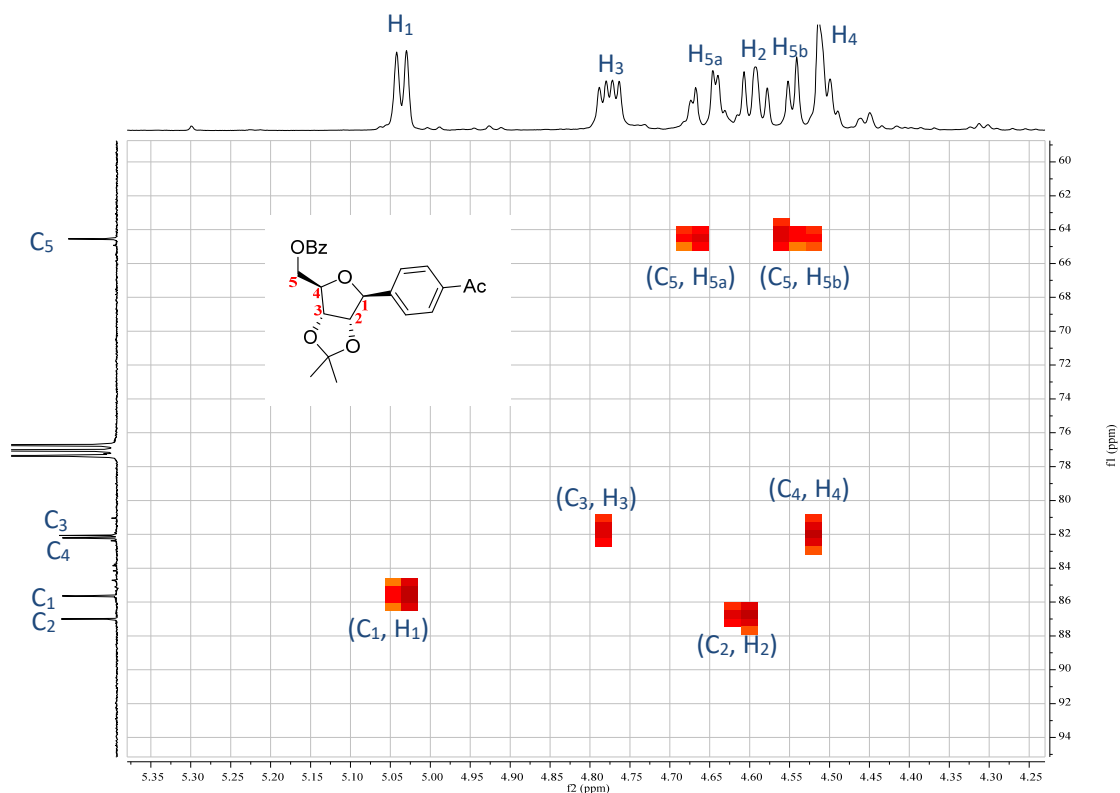
¹³C NMR (100 MHz, CDCl₃) of 33



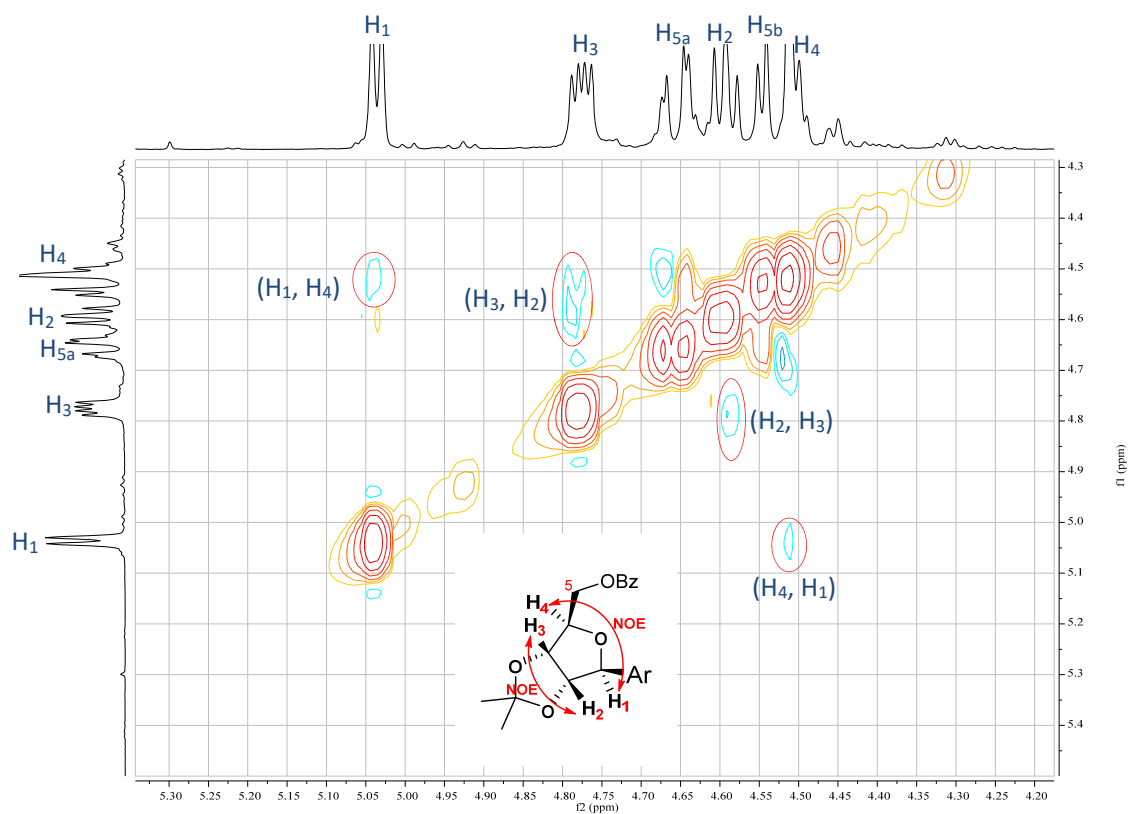
^1H - ^1H COSY (400 MHz, CDCl_3) of 31



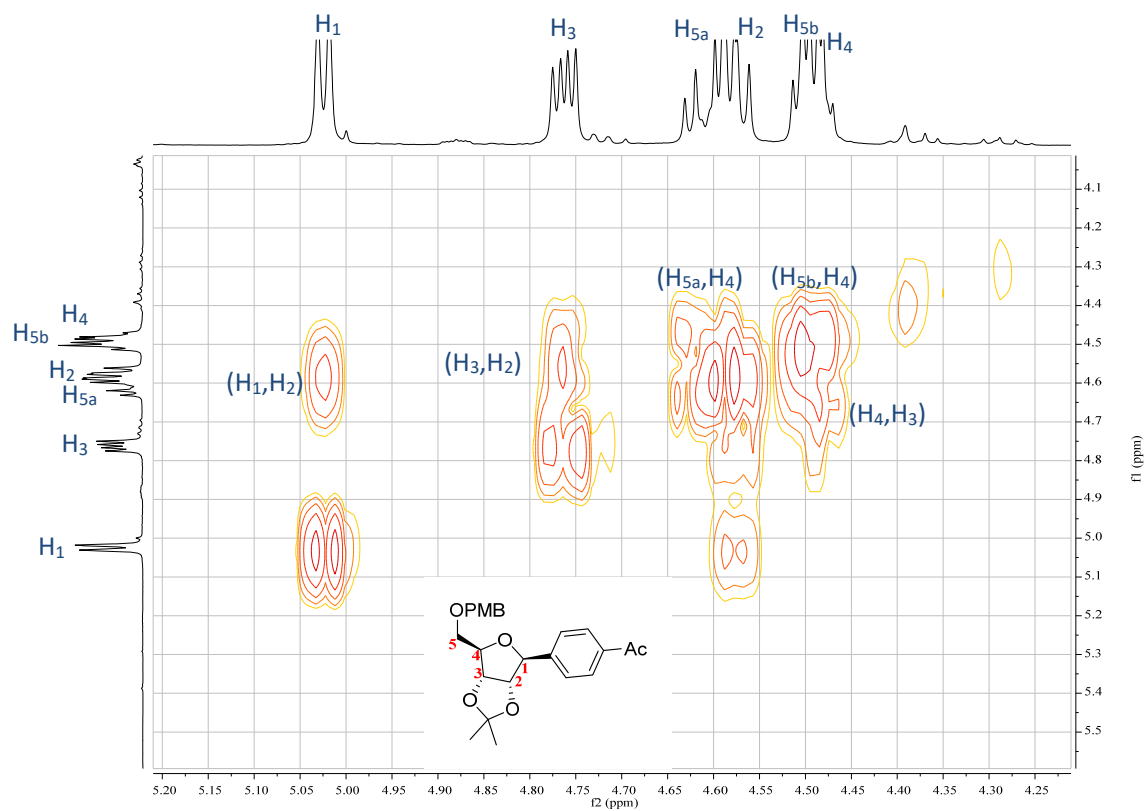
^{13}C - ^1H HSQC (400 MHz, CDCl_3) of 31



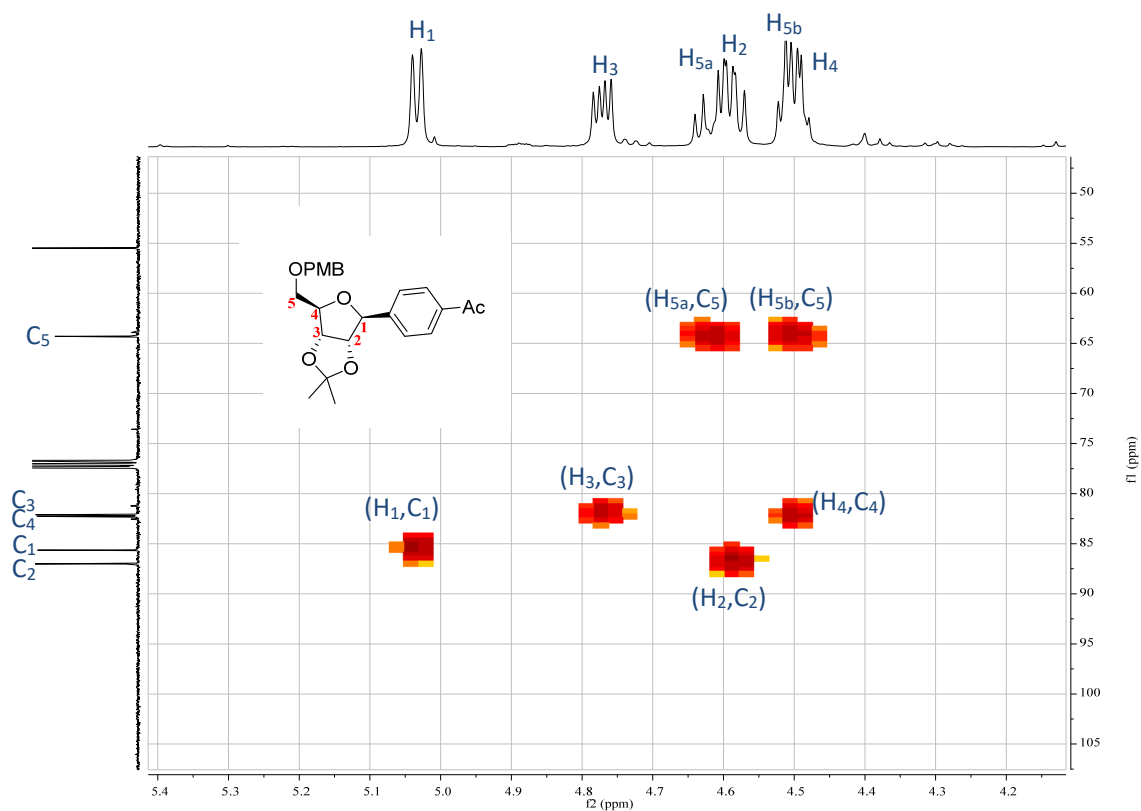
^1H - ^1H NOESY (400 MHz, CDCl_3) of **31**



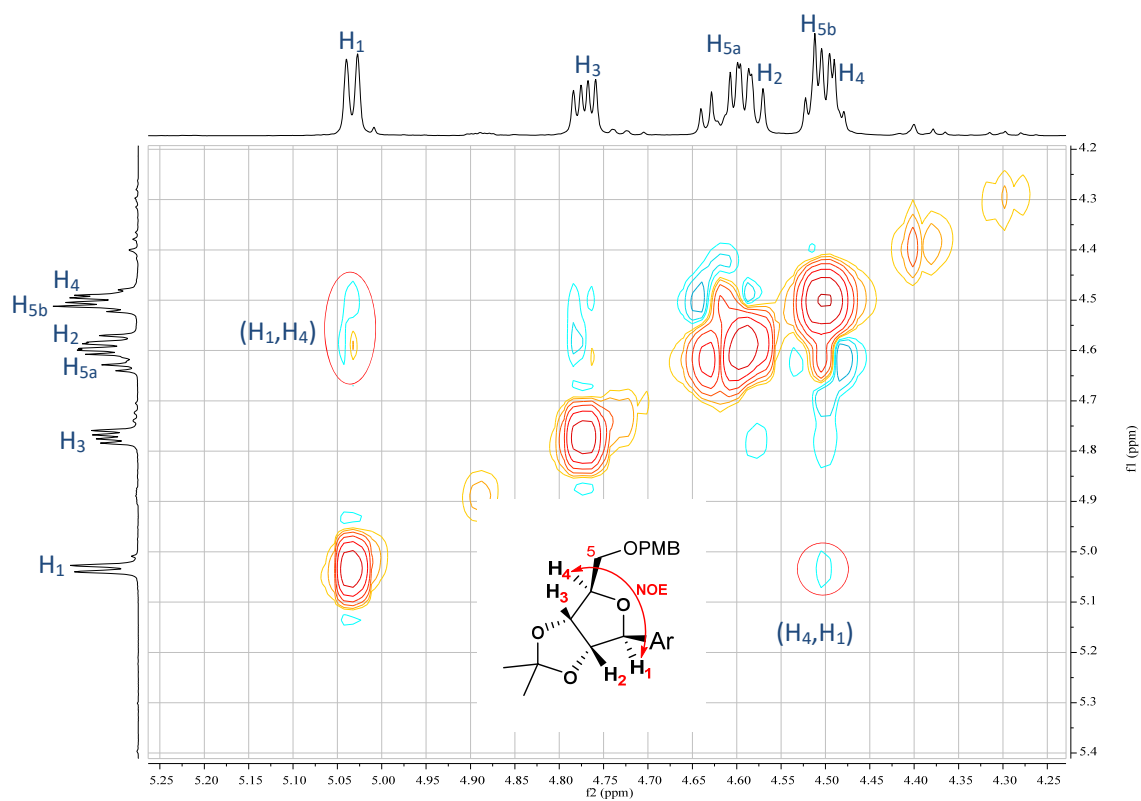
^1H - ^1H COSY (400 MHz, CDCl_3) of **32**



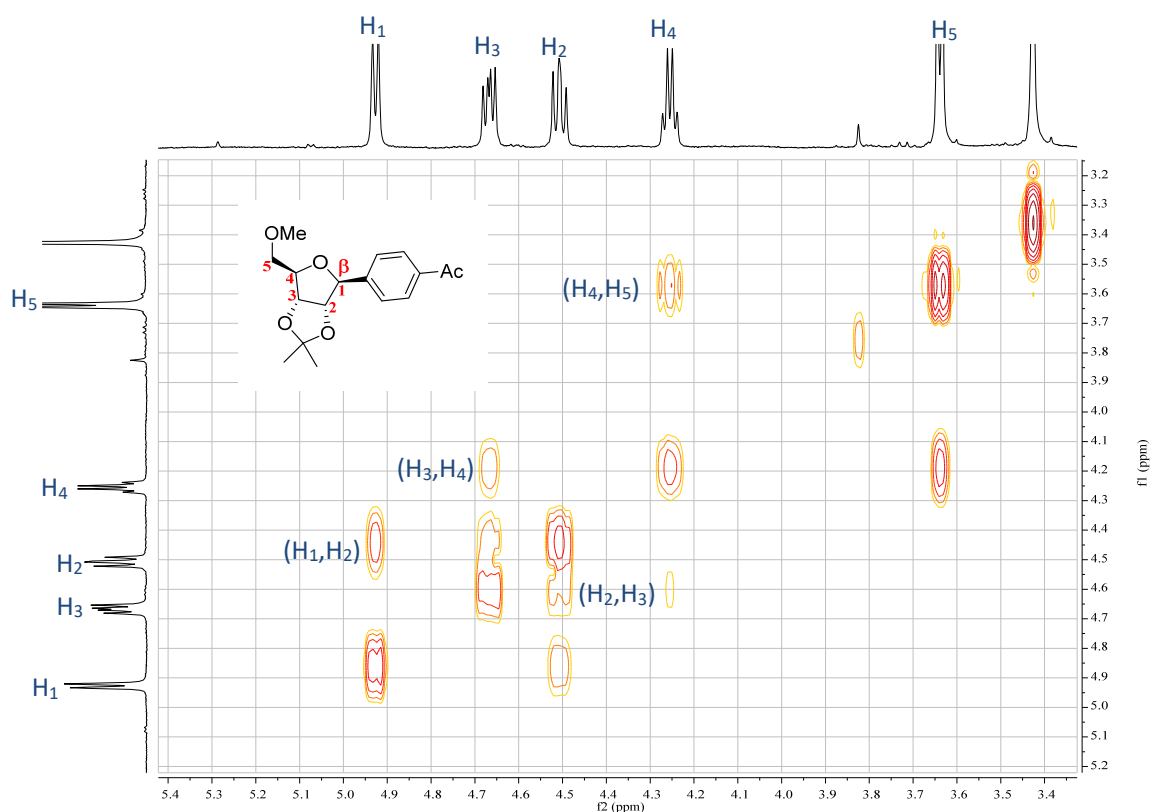
^{13}C - ^1H HSQC (400 MHz, CDCl_3) of **32**



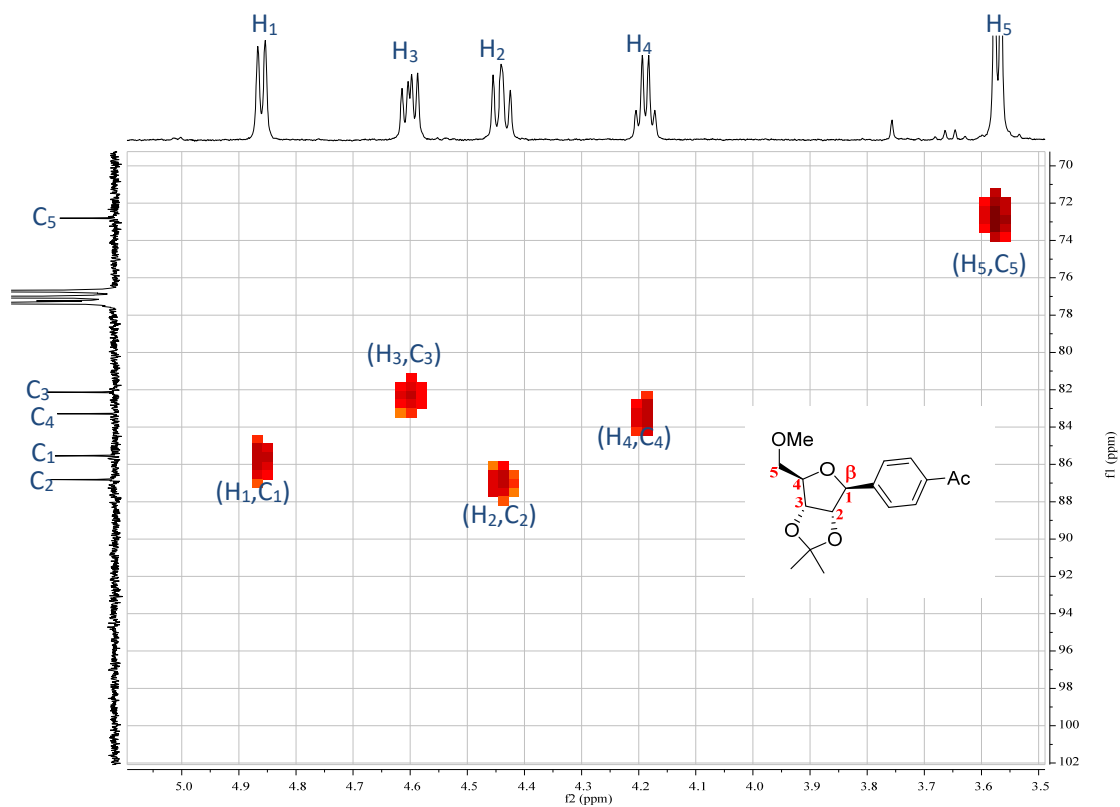
^1H - ^1H NOESY (400 MHz, CDCl_3) of **32**



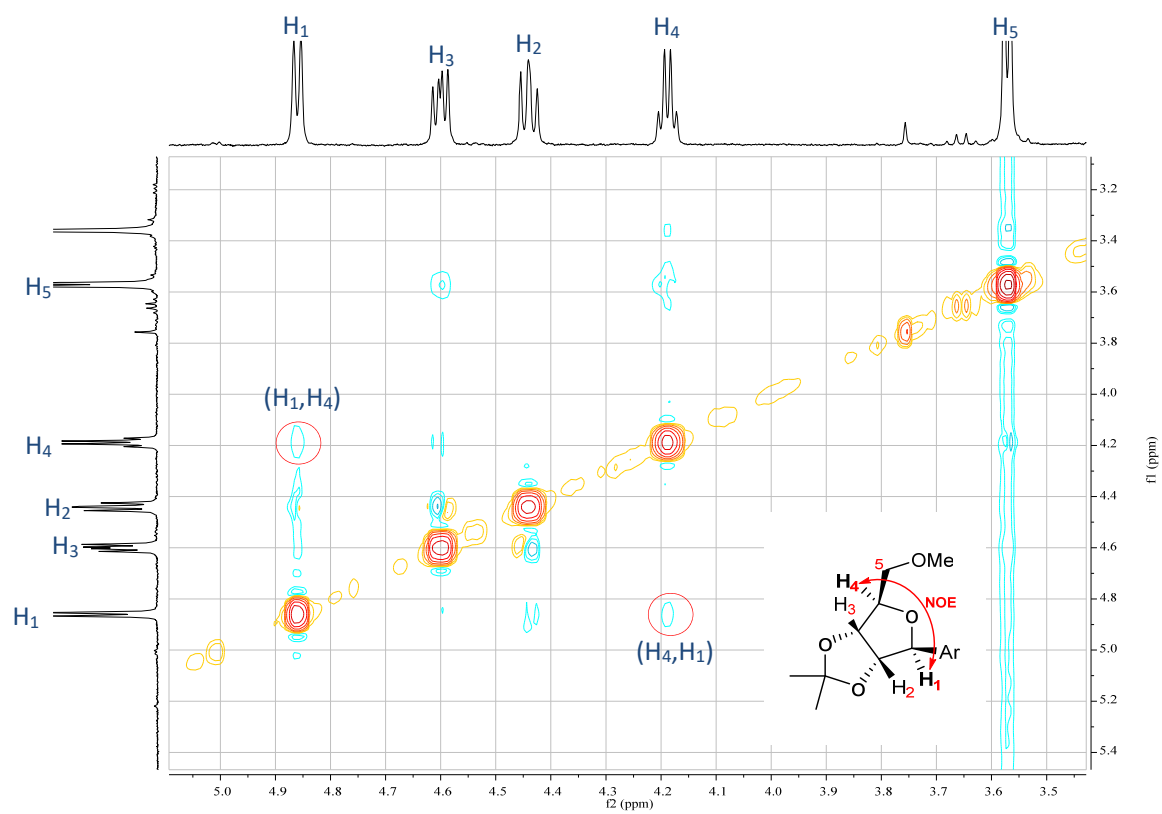
^1H - ^1H COSY (400 MHz, CDCl_3) of **33**



^{13}C - ^1H HSQC (400 MHz, CDCl_3) of **33**



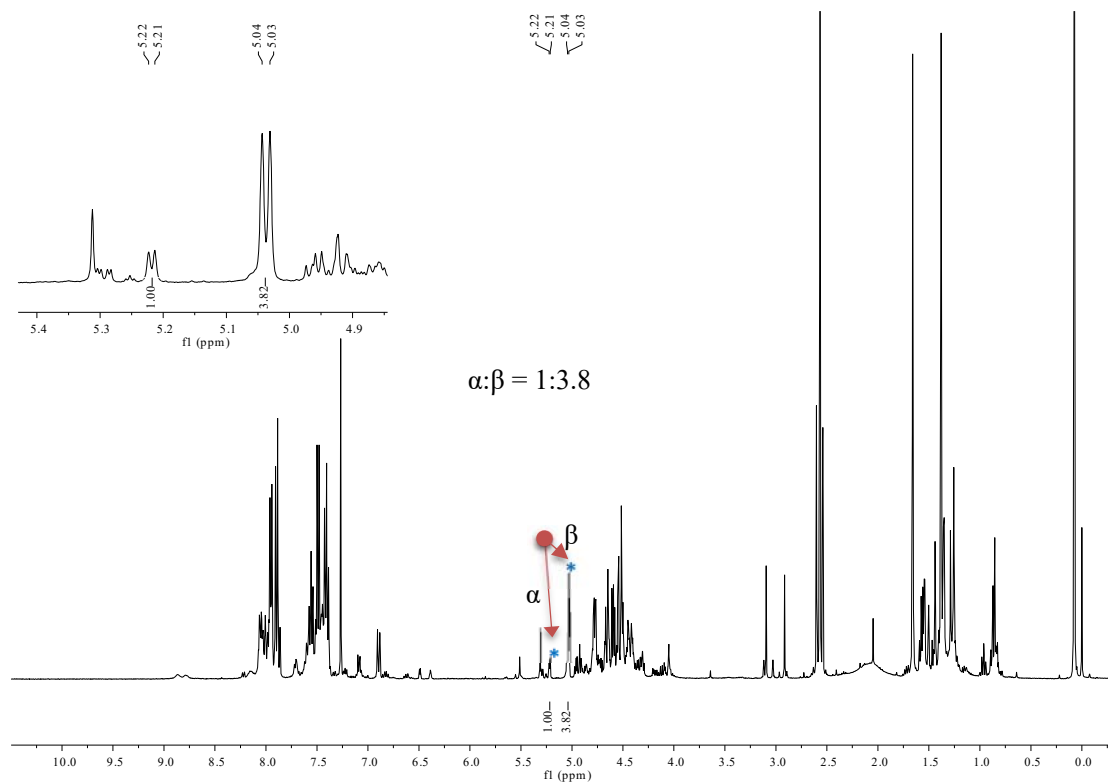
^1H - ^1H NOESY (400 MHz, CDCl_3) of **33**



The determination of the diastereomeric ratio

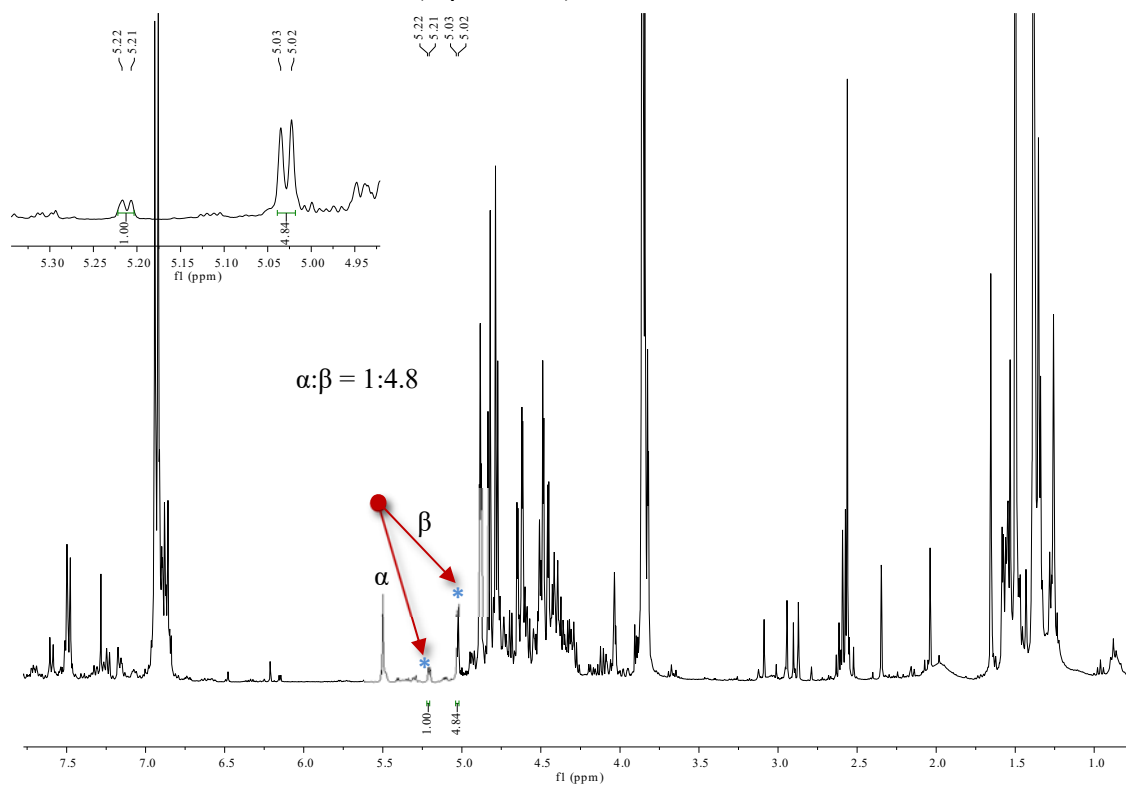
For **31** and **32**, we used the anomeric protons to determine the diastereomeric ratio. For compound **33**, we used the MeO to determine the diastereomeric ratio. The diastereomeric ratios determined are in the range of 3.5-4.8, consistent with the results reported by Messaoudi and co-workers in radical C-alkynyl glycosylation.¹

The ratio of isomer mixture **31** ($\alpha:\beta = 1:3.8$)



¹ M. Zhu and S. Messaoudi, *ACS Catalysis*, 2021, **11**, 6334–6342.

The ratio of isomer mixture **32** ($\alpha:\beta = 1:4.8$)



The ratio of isomer mixture **33** ($\alpha:\beta = 1:3.5$)

