A Facile Catalyst-Free Synthesis of *gem*-Dihydroperoxide with Aqueous Hydrogen Peroxide

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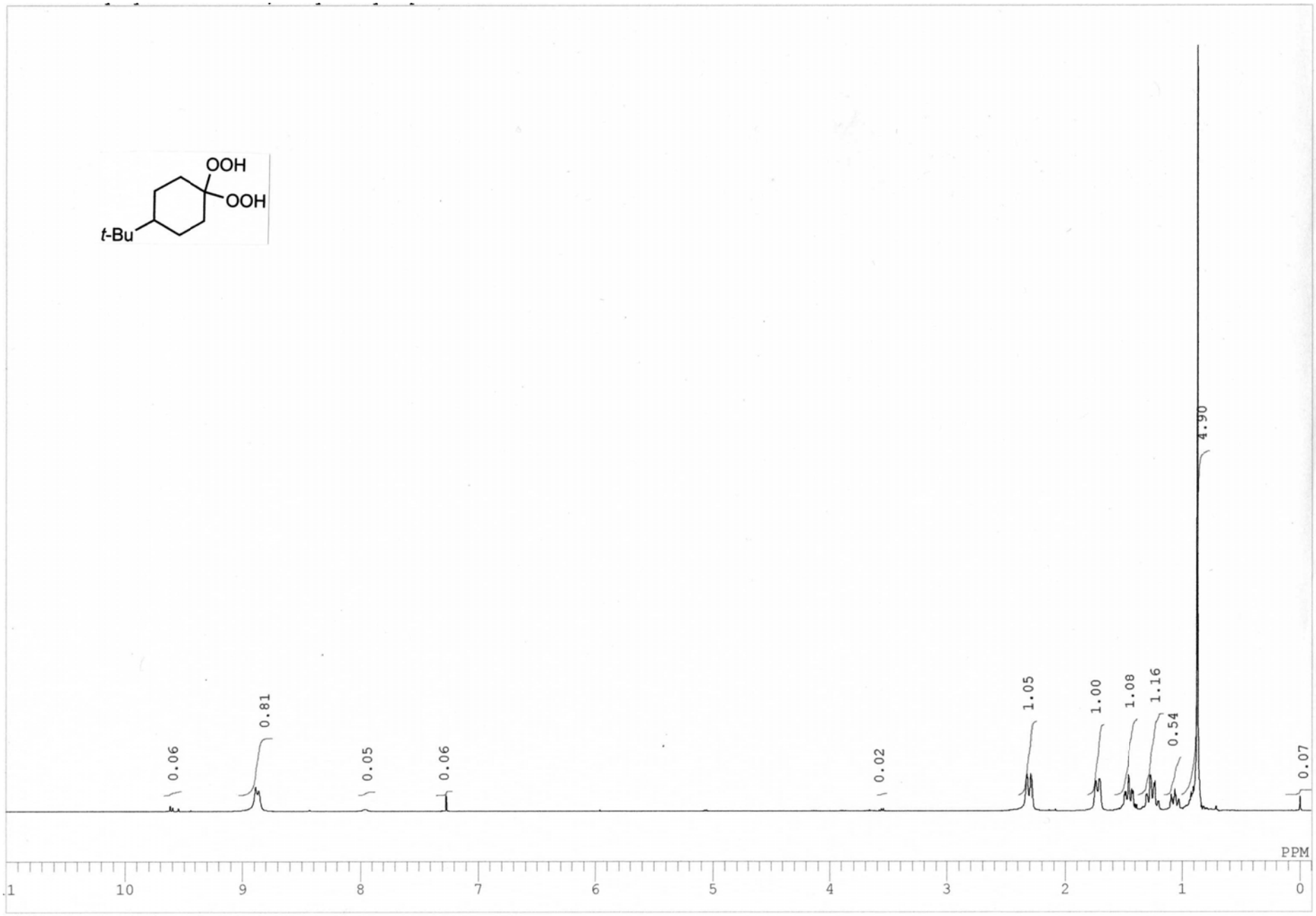
Experimental Section

General. 35% aqueous hydrogen peroxide was purchased from Tokyo Chemical Industry. 1,2-Dimethoxyethane (dehydrated) and other dry solvent were purchased from Kanto Kagaku. The ^{1}H NMR spectra were recorded using JEOL JNM-EX-400 and JEOL JNM-AL-400 (400 MHz) spectrometers. Part of the products was isolated by preparative TLC (Merck, TLC plates, silica gel 60 F_{254}, Art 5744) or column chromatography on silica gel (Kanto Kagaku, silica gel 60N, spherical, neutral, 40-50μm).

A typical procedure of the dihydroperoxidation is as follows: To a solution of 4-*t*-butylcyclohexanone (46.3 mg, 0.30 mmol) in dry DME solution (3 mL) was added 35 % H_2O_2 (130 μL, 1.50 mmol) at room temperature. After stirring at room temperature for 20 h, the reaction mixture was concentrated under reduced pressure. The residue was purified by preparative TLC (hexanen:AcOEt = 2 : 1) to afford the pure 4-*t*-butylcyclohexylidenebishydroperoxide (60.7 mg, 99 %).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PPM
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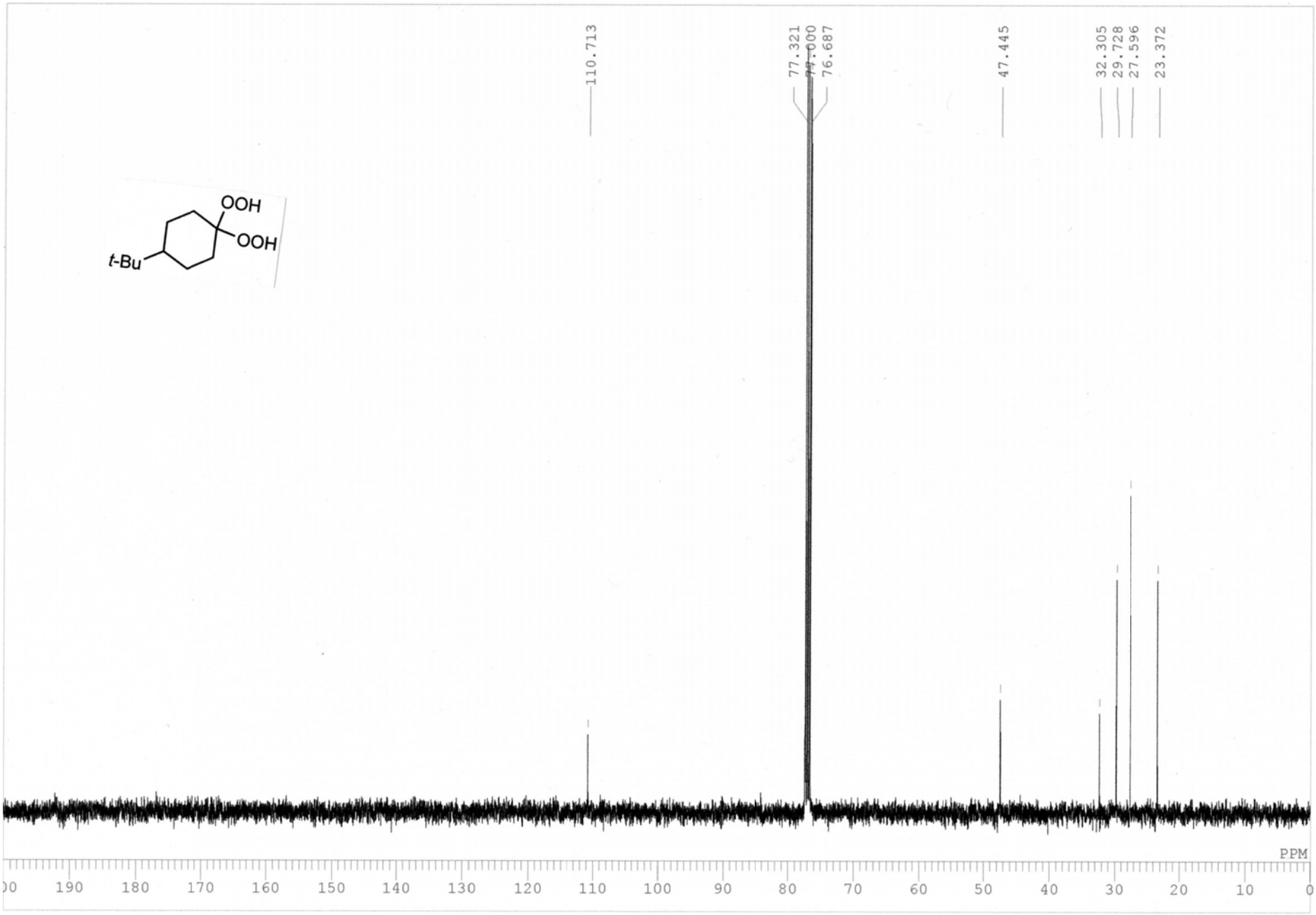
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PPM
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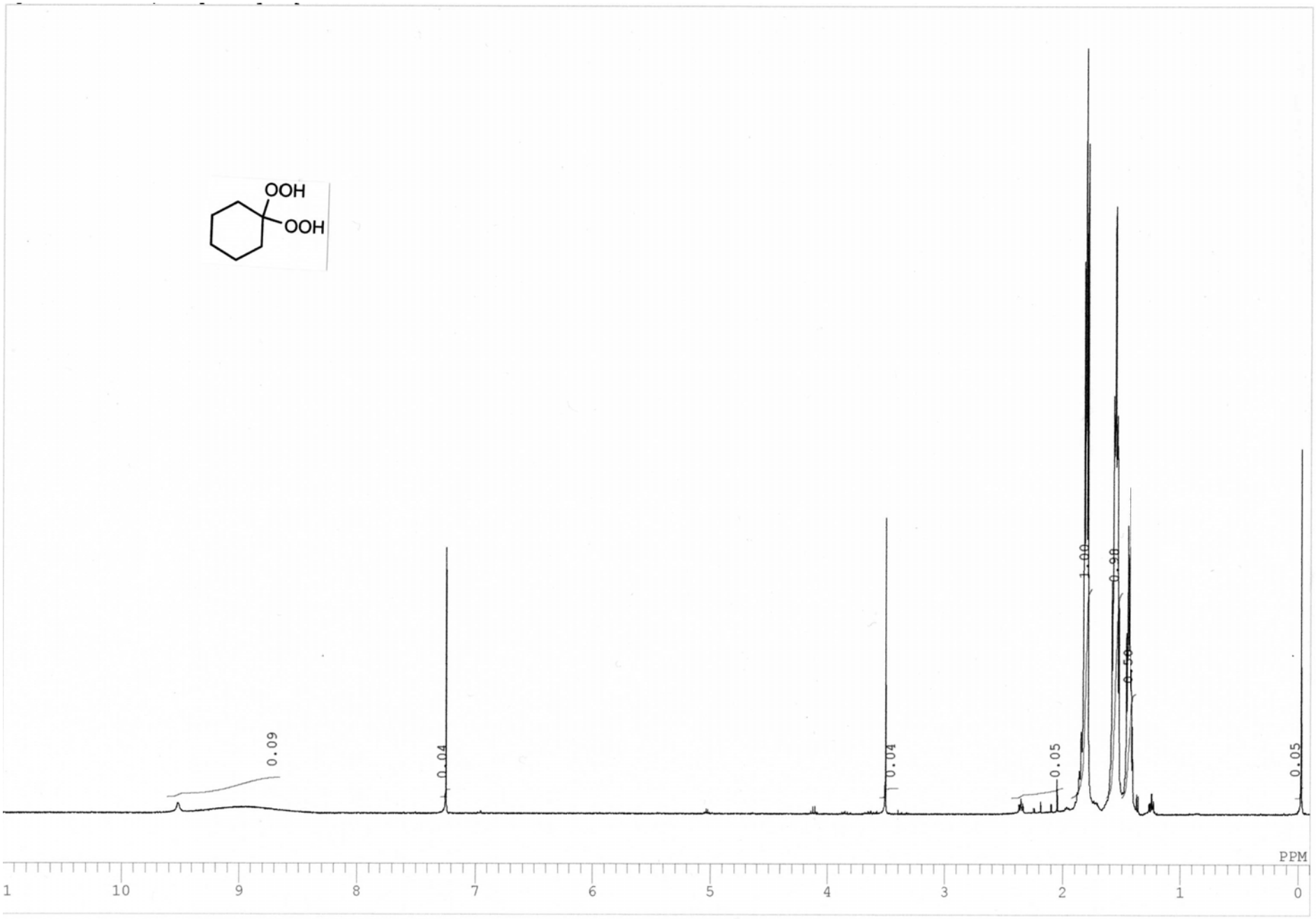
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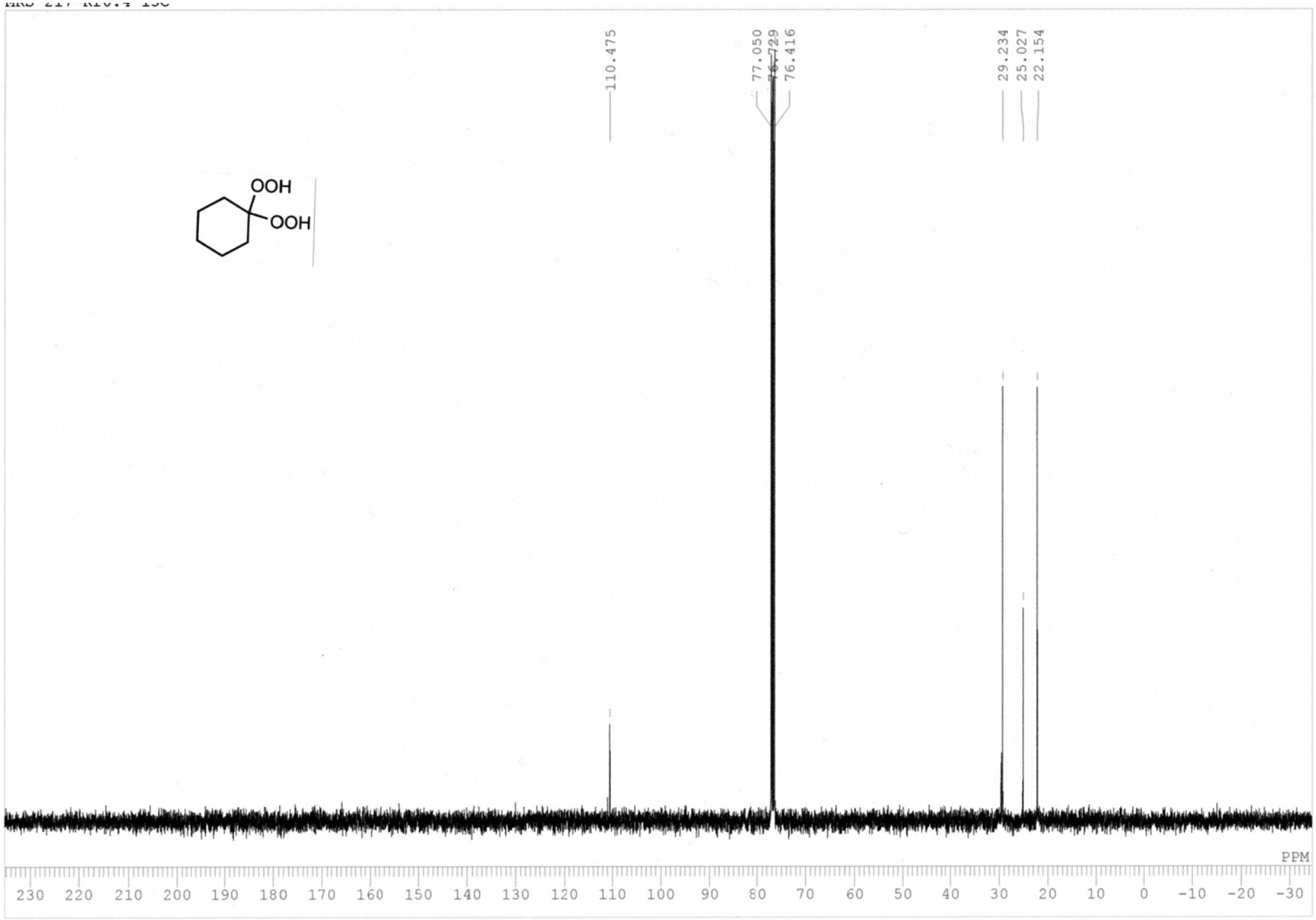
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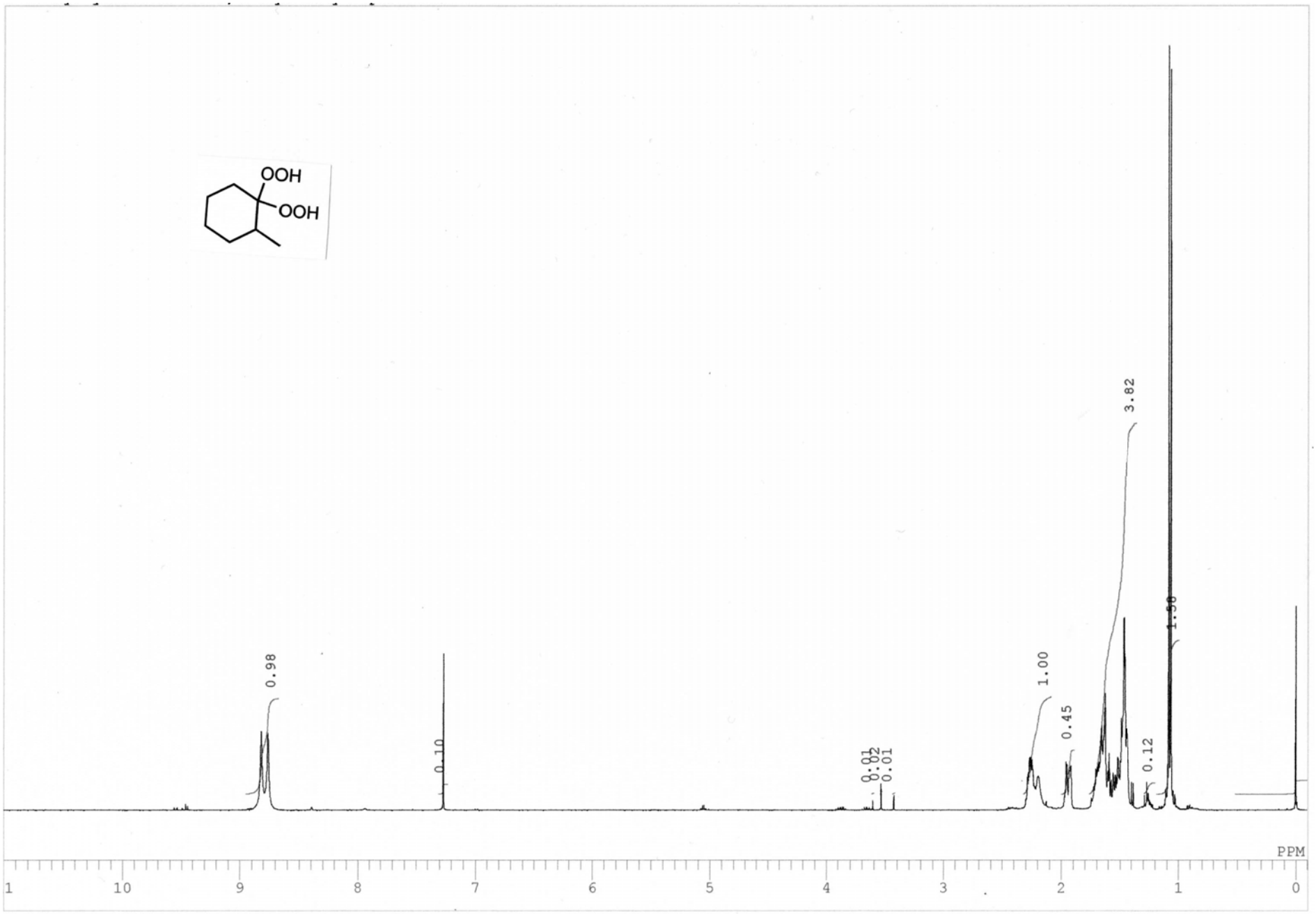
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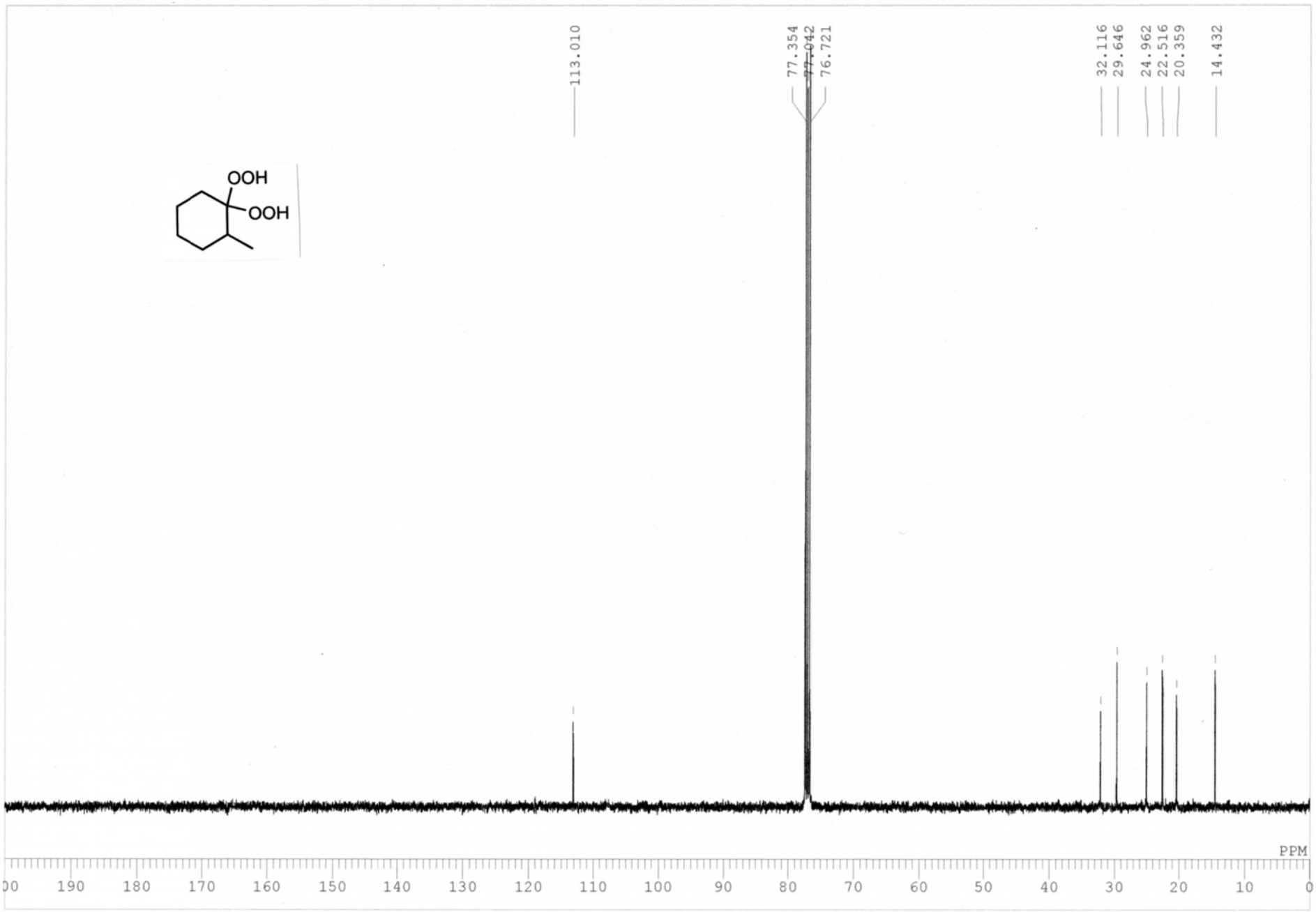
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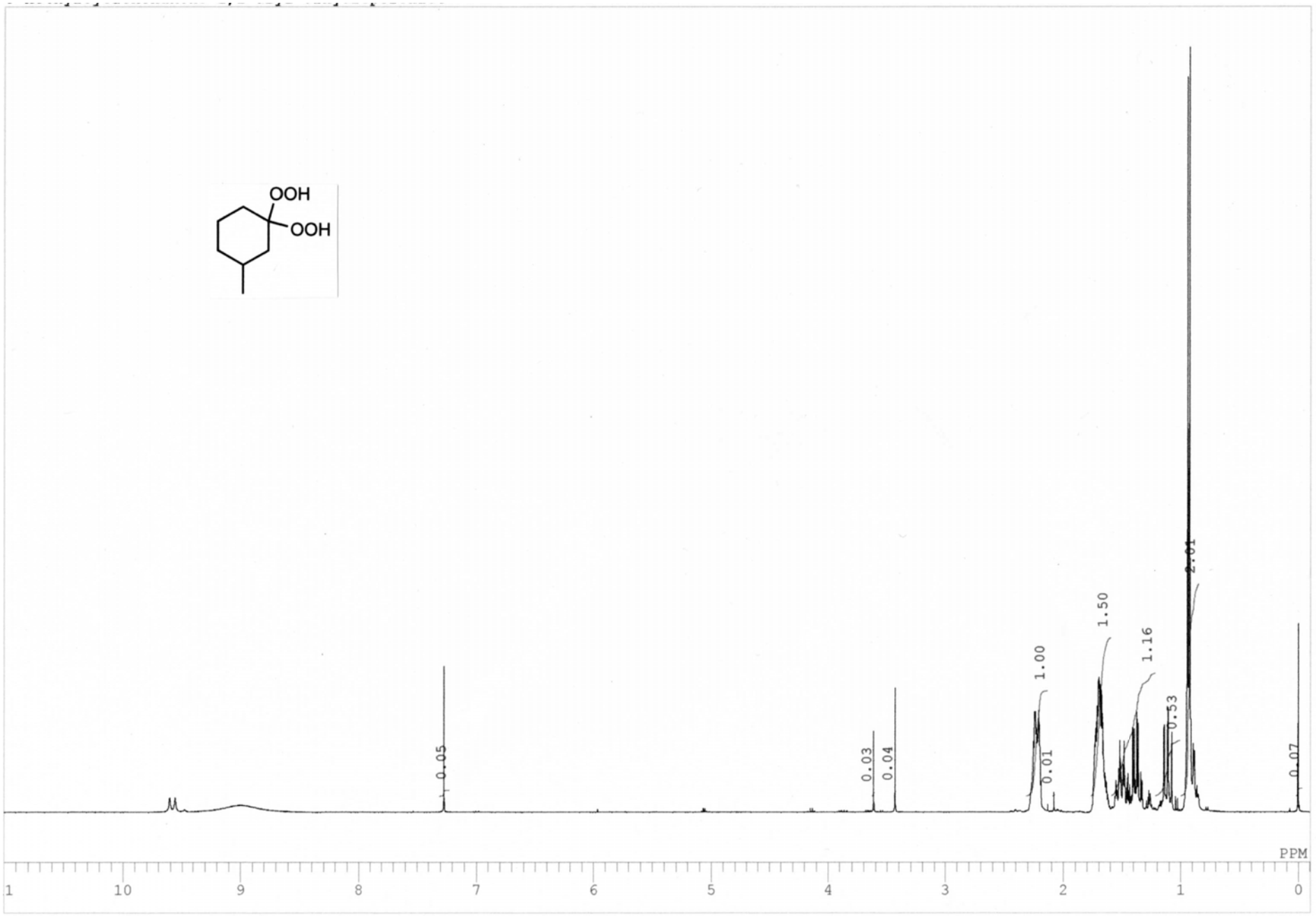
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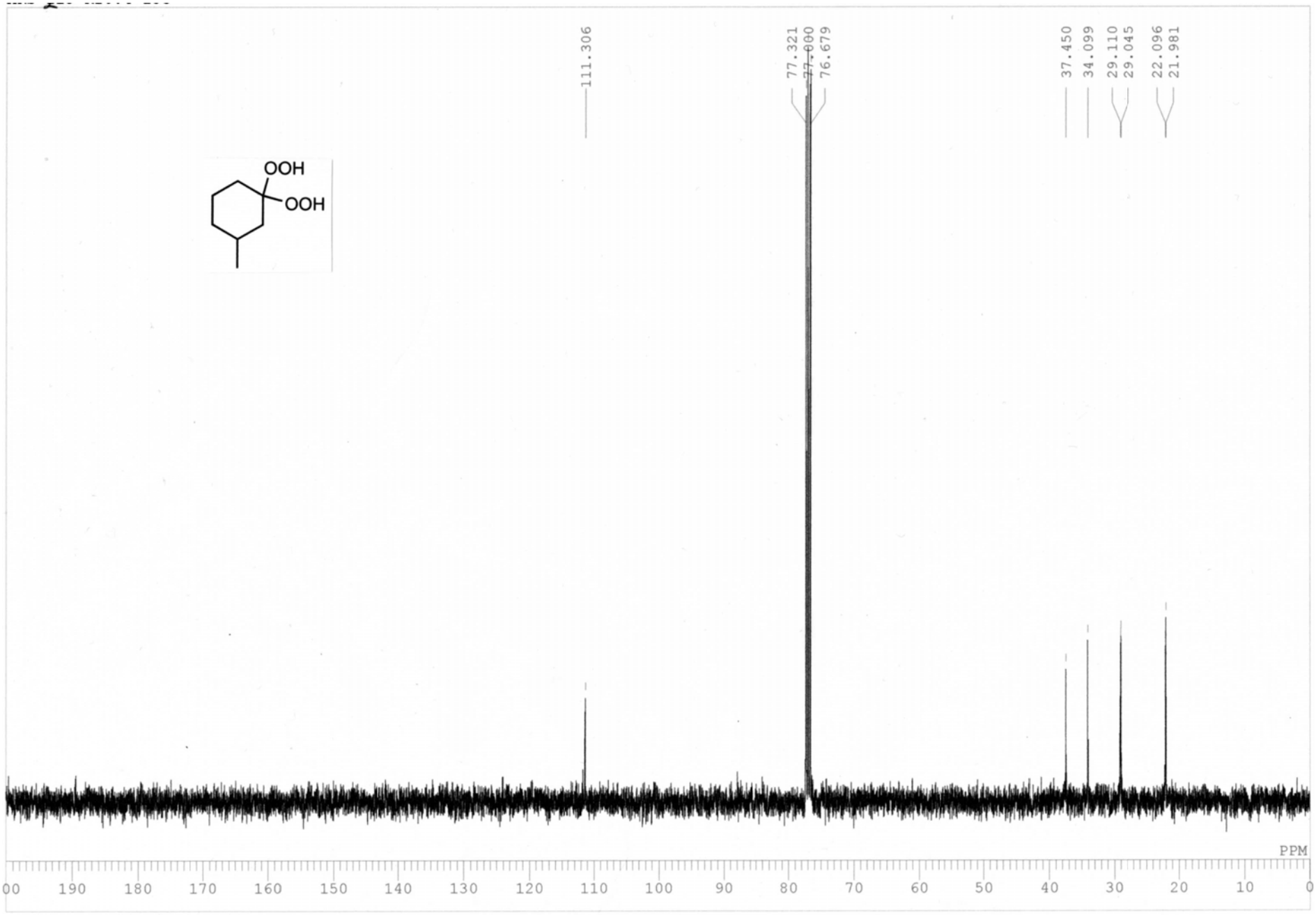
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OOH
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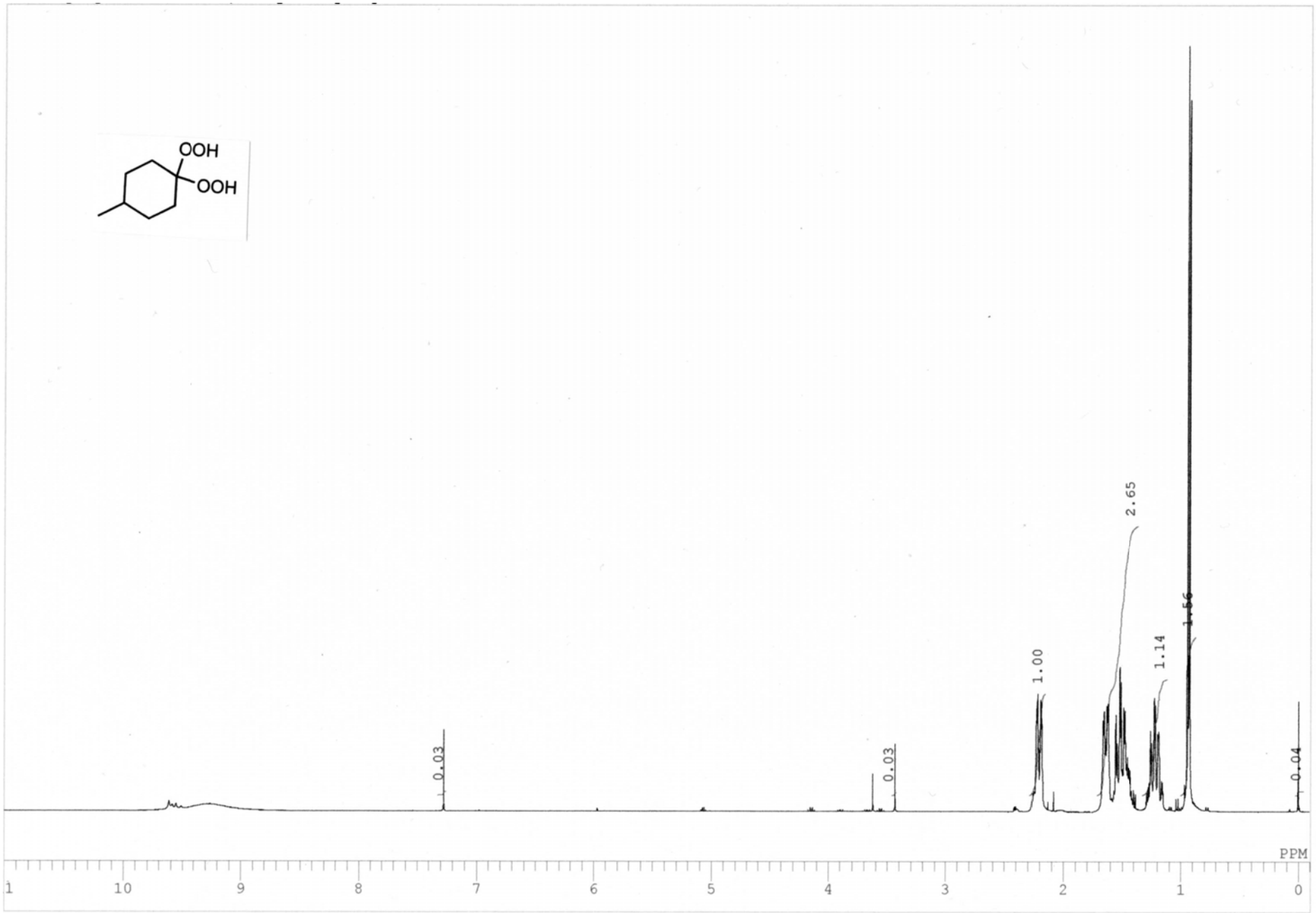
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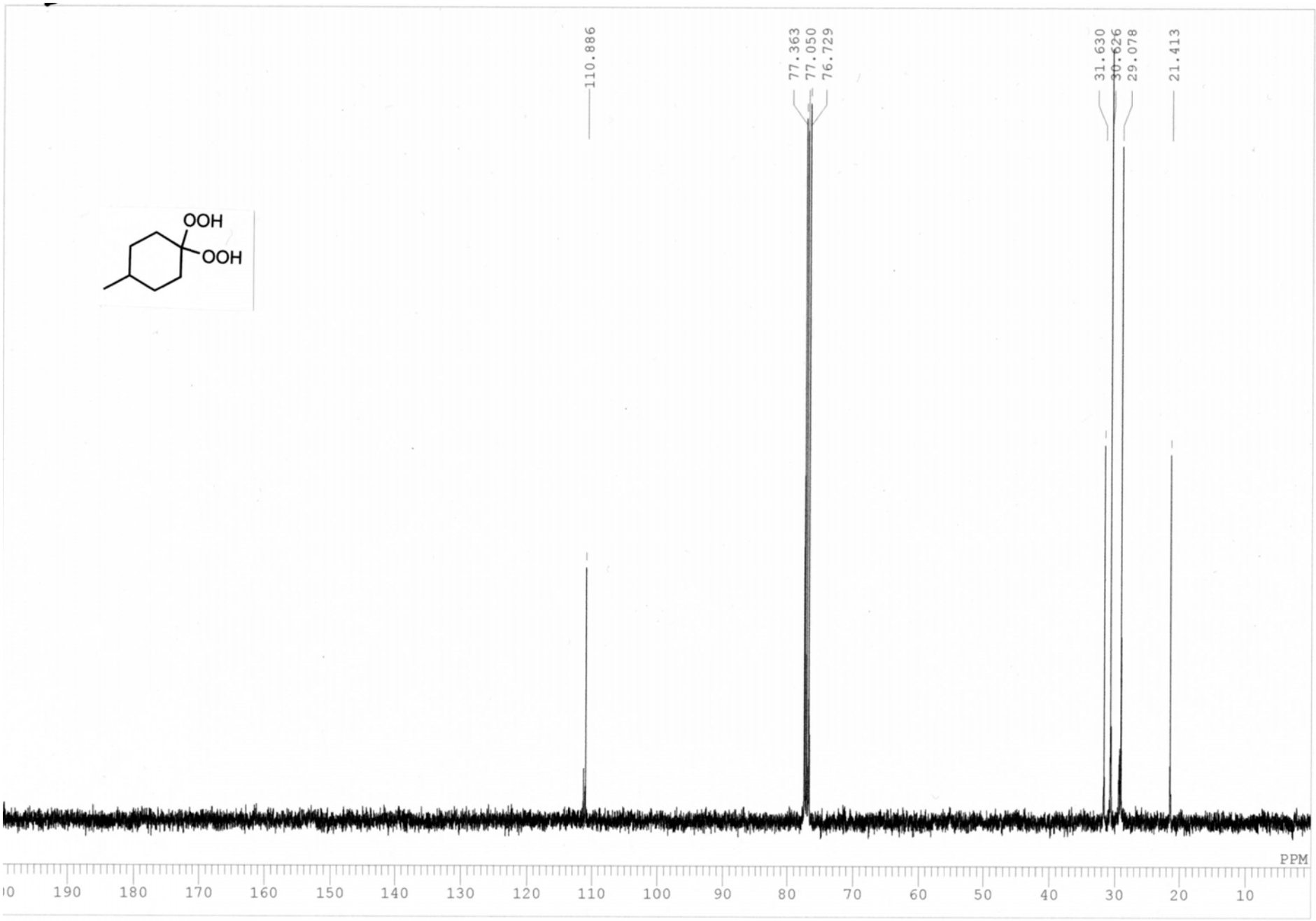
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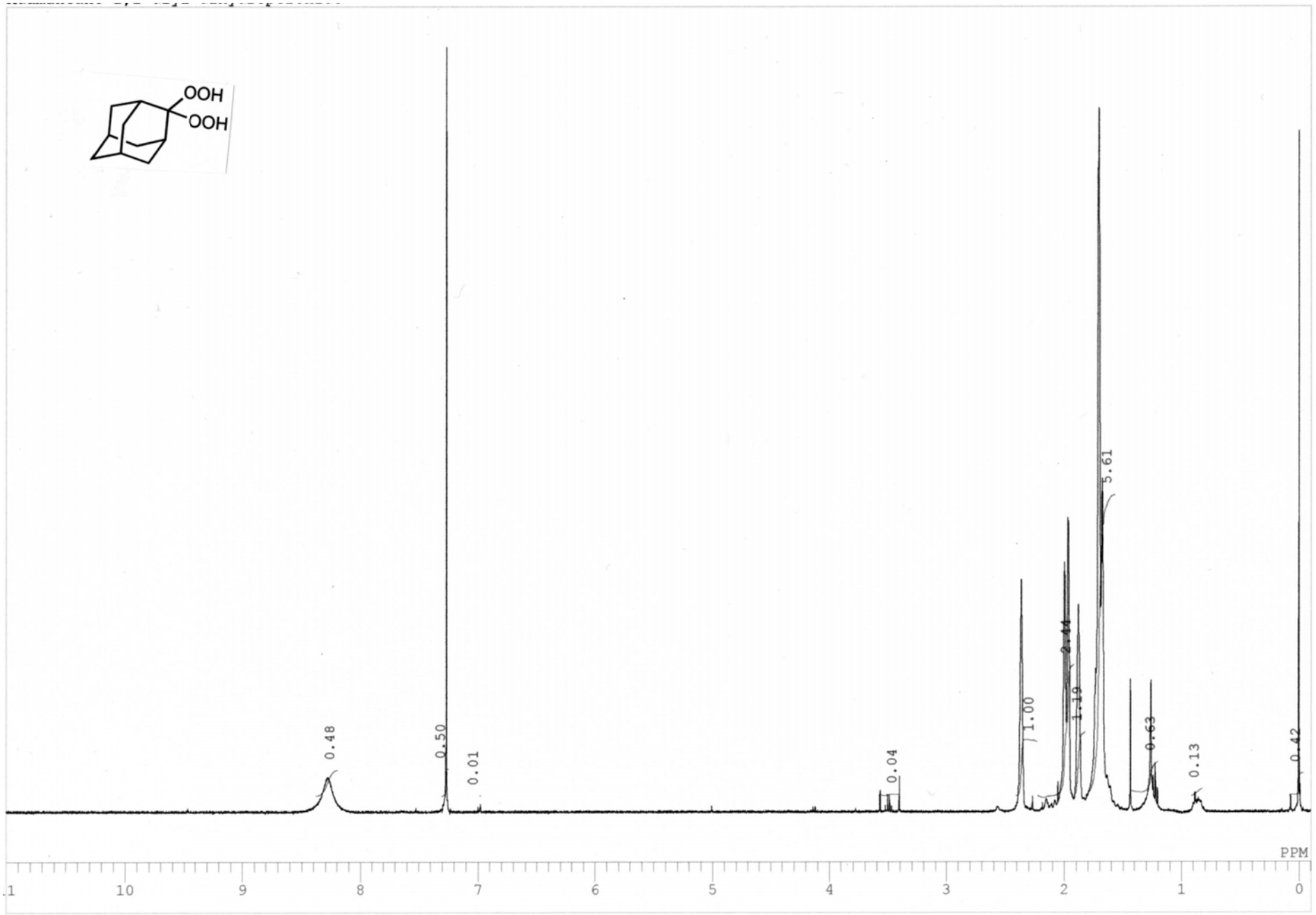
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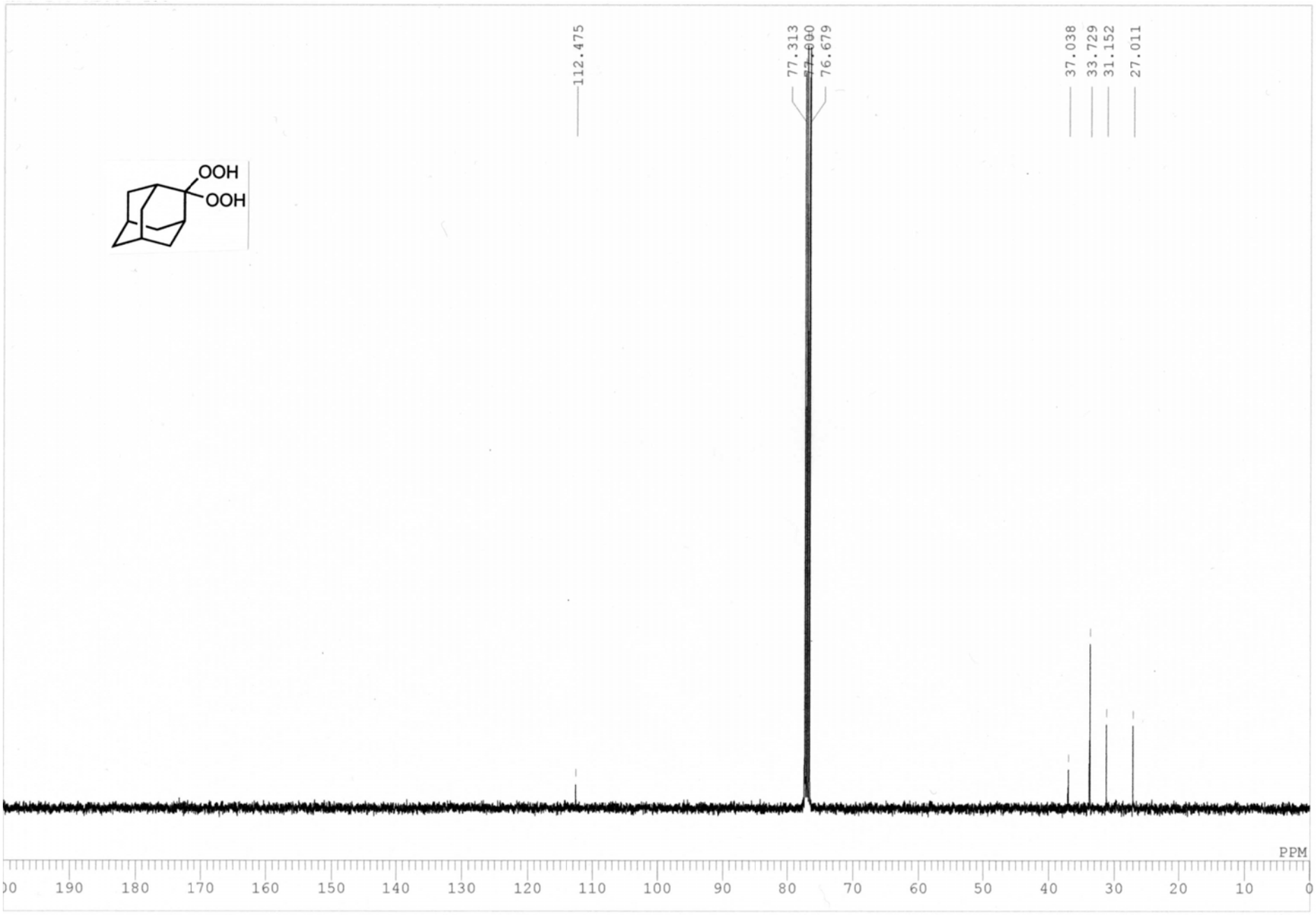
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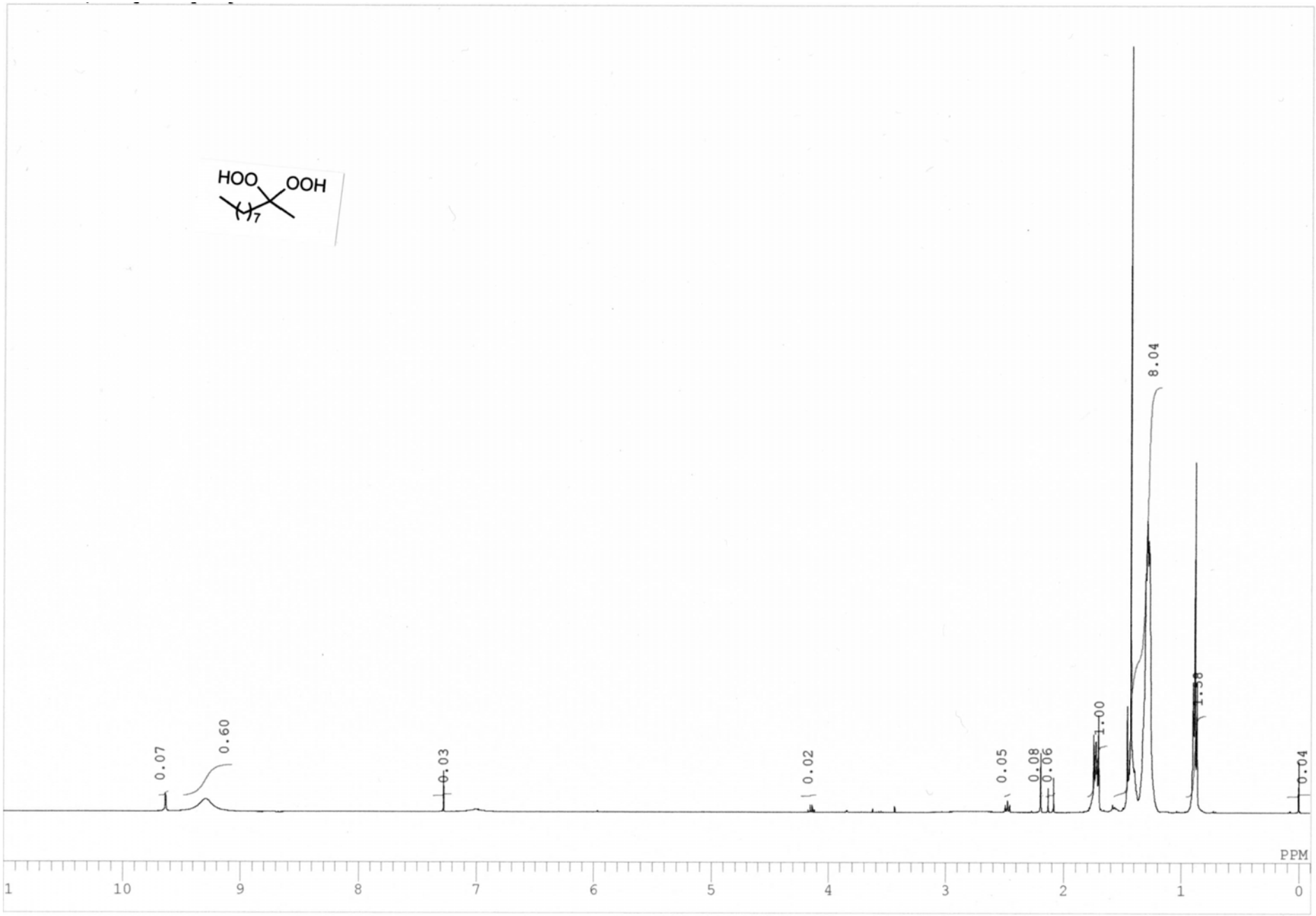
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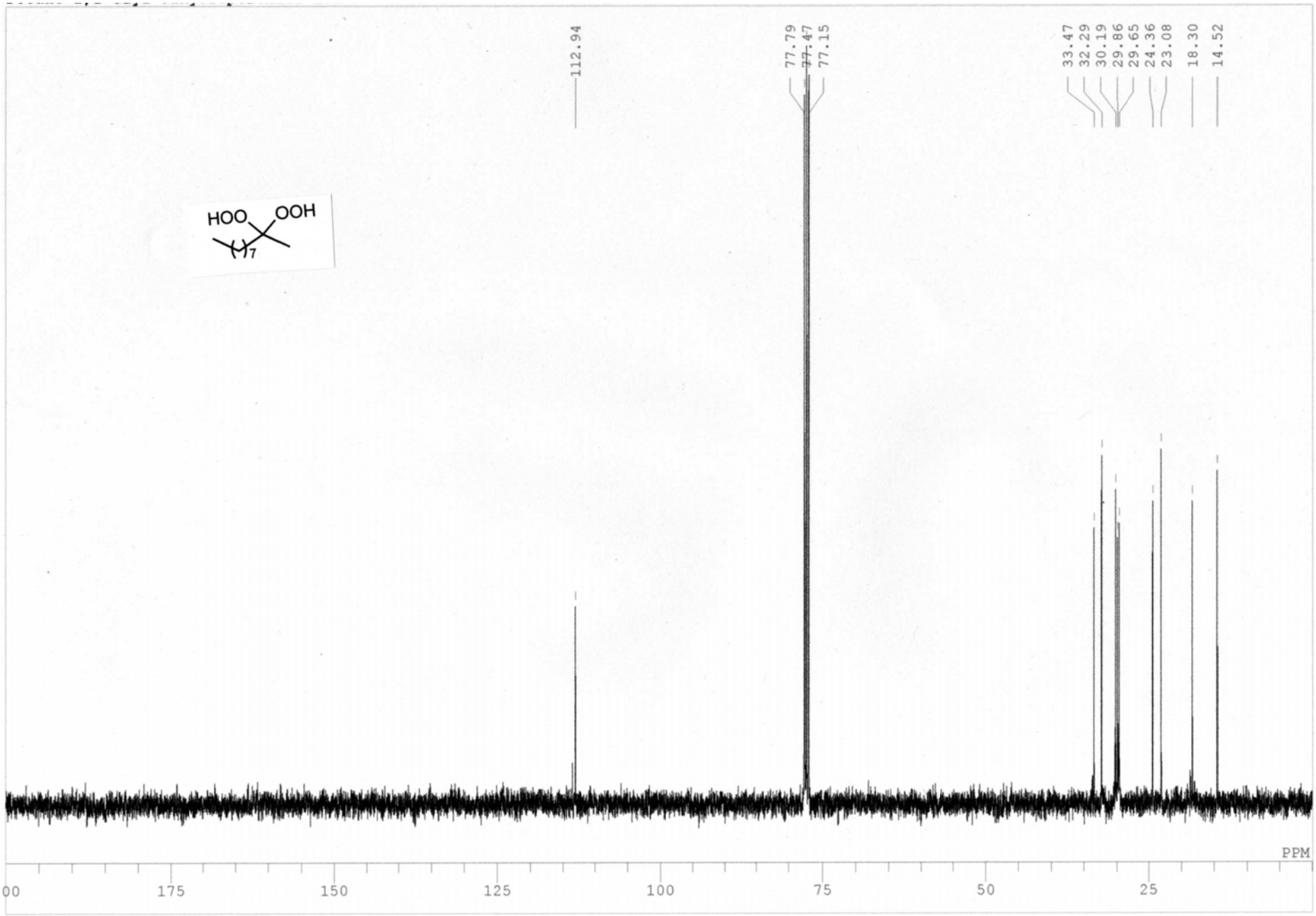
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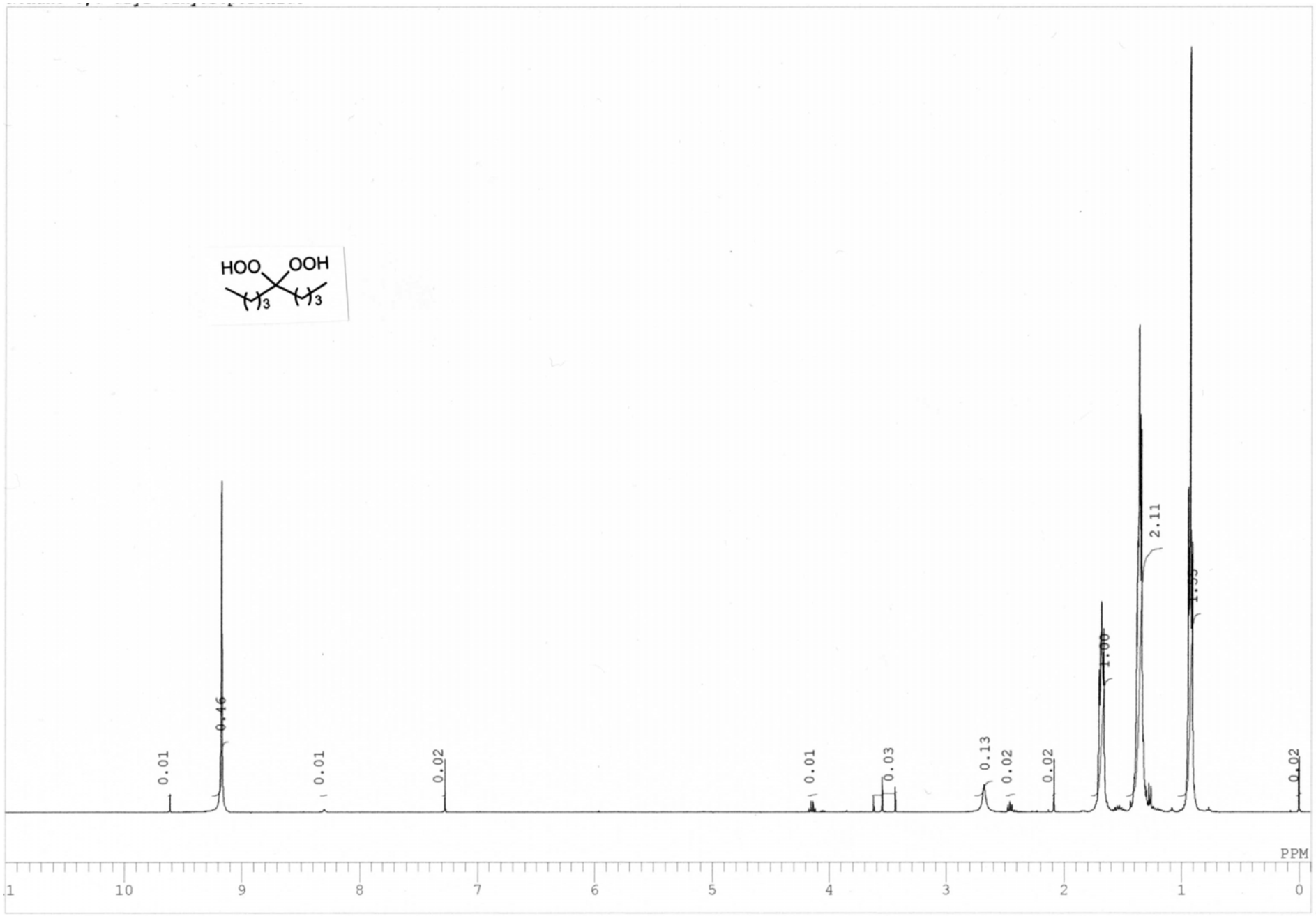
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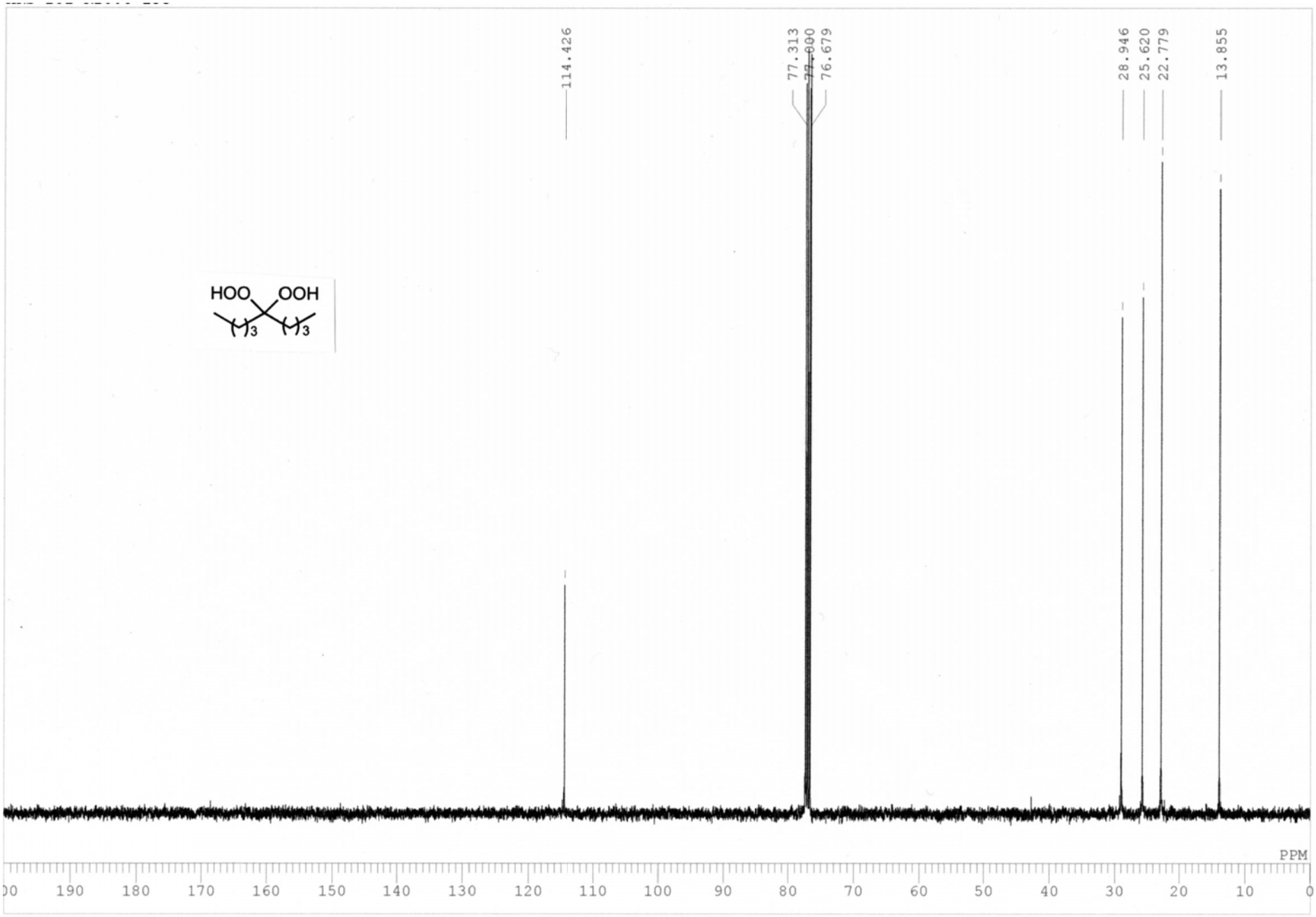
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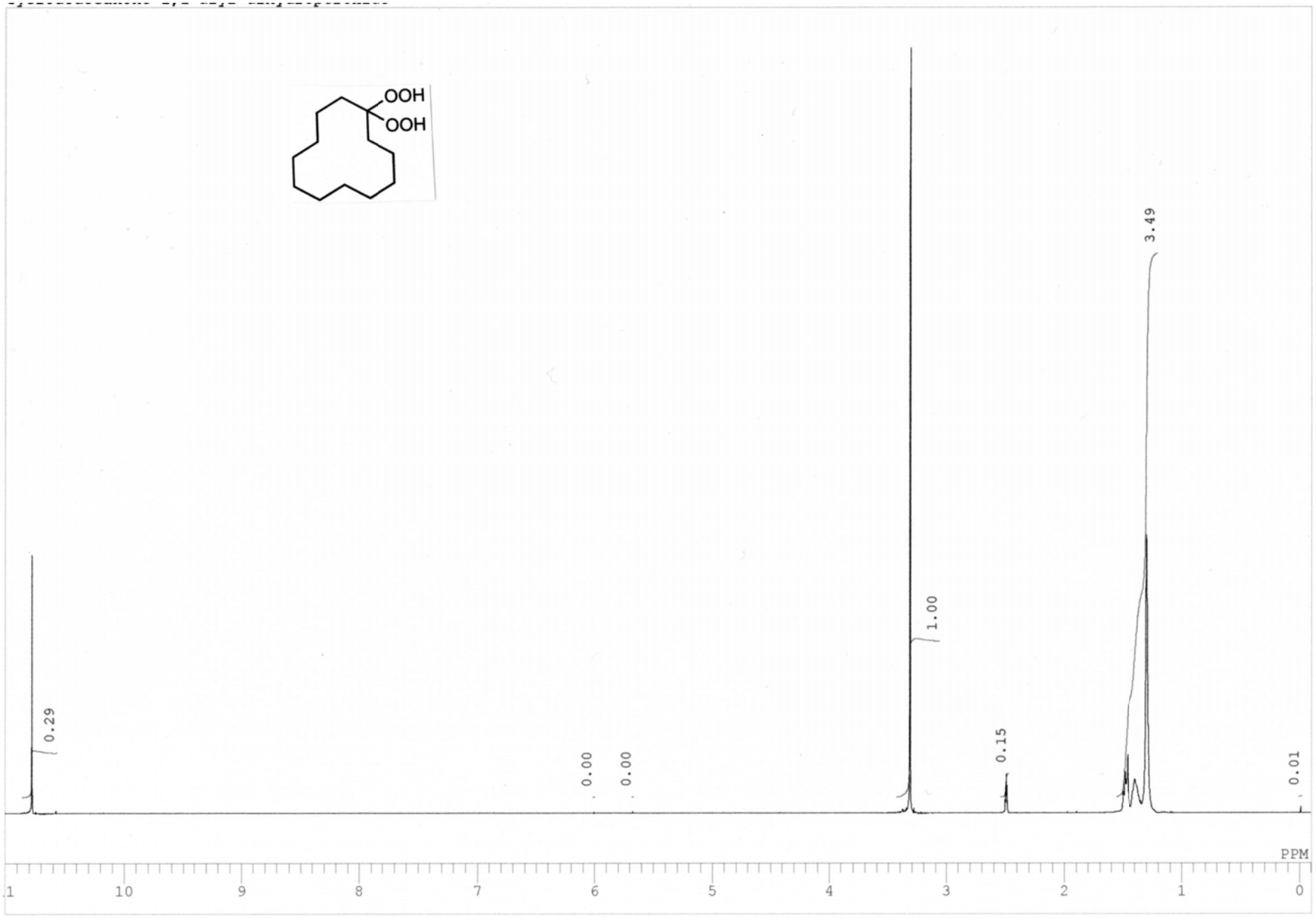
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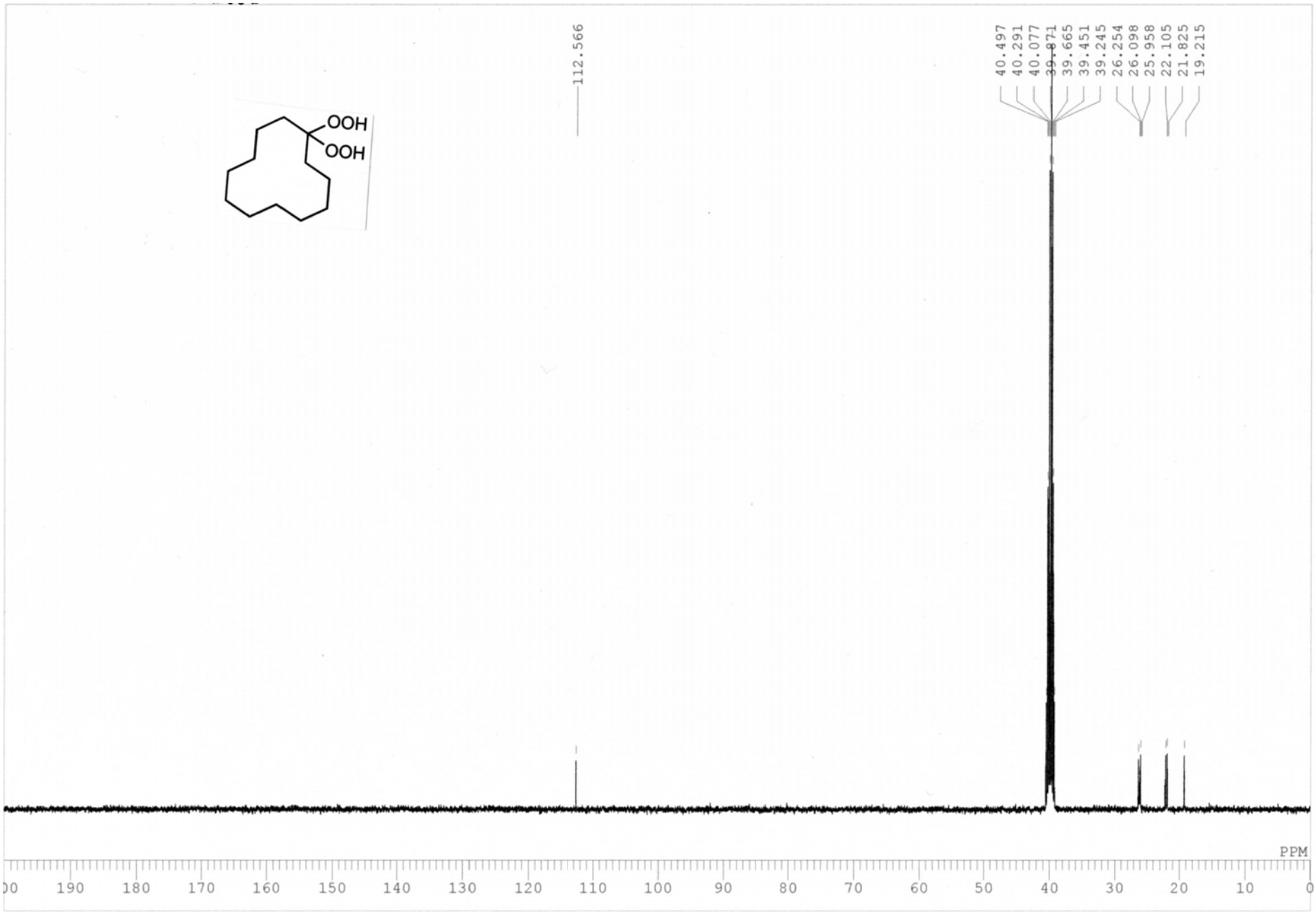
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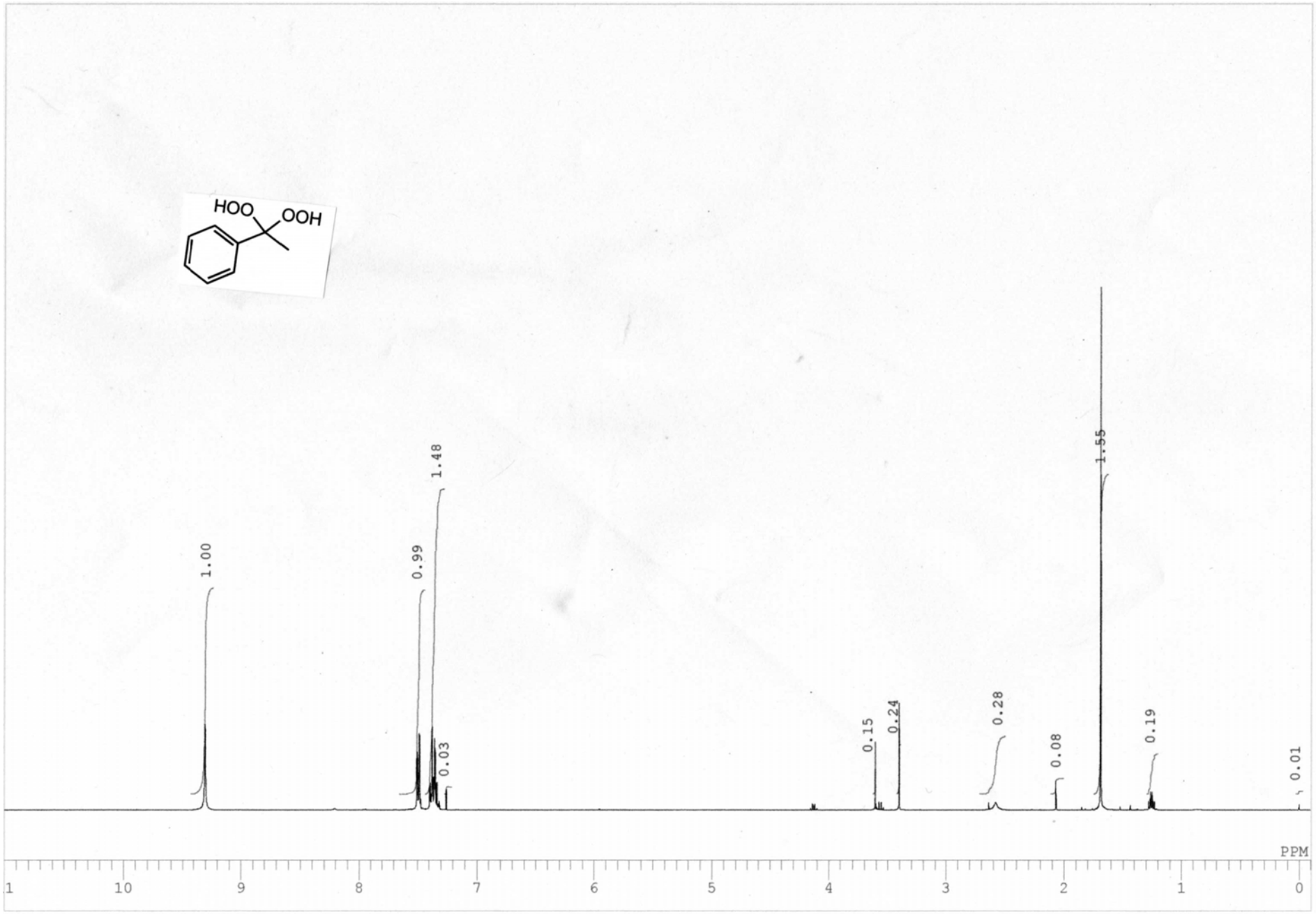
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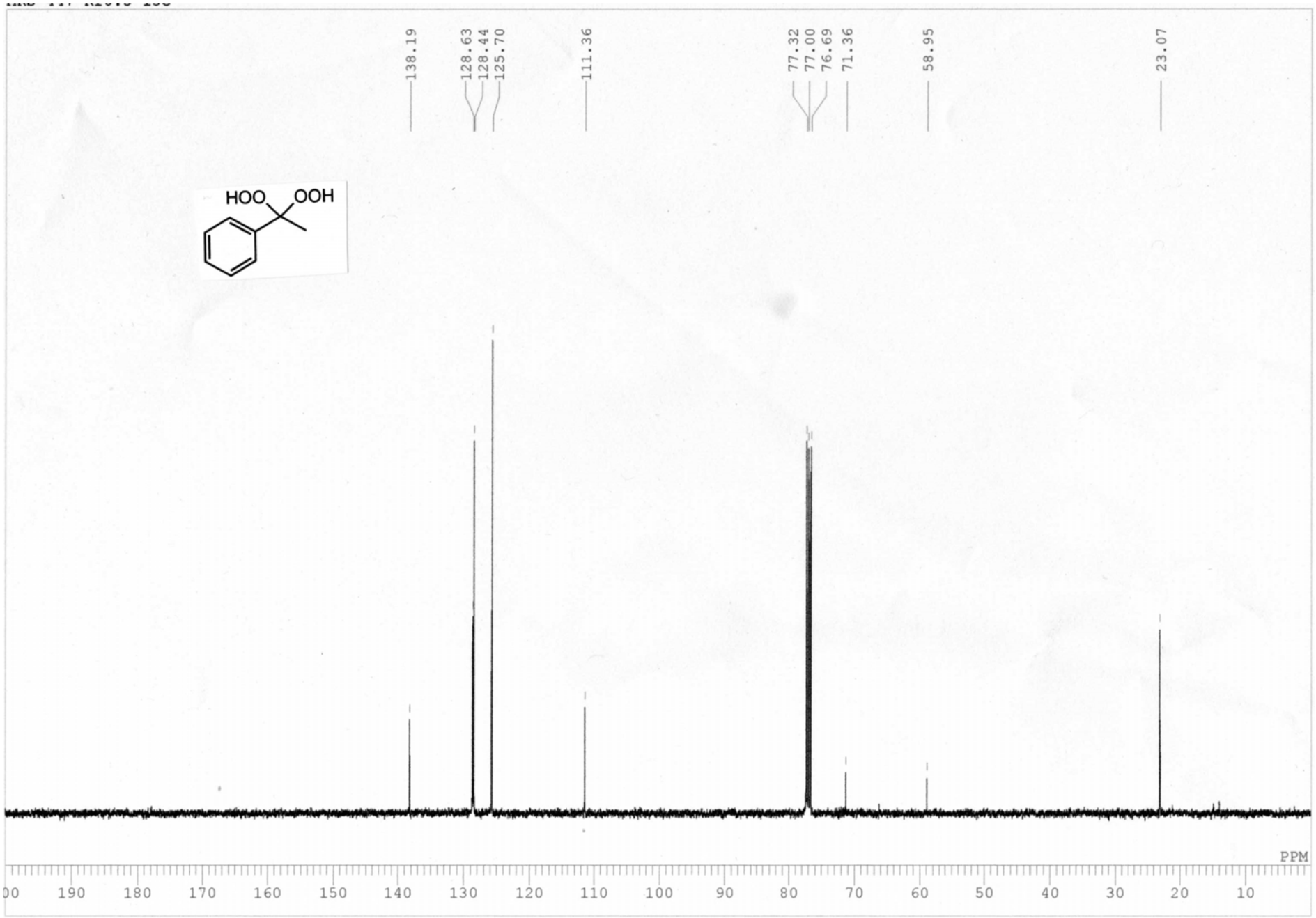
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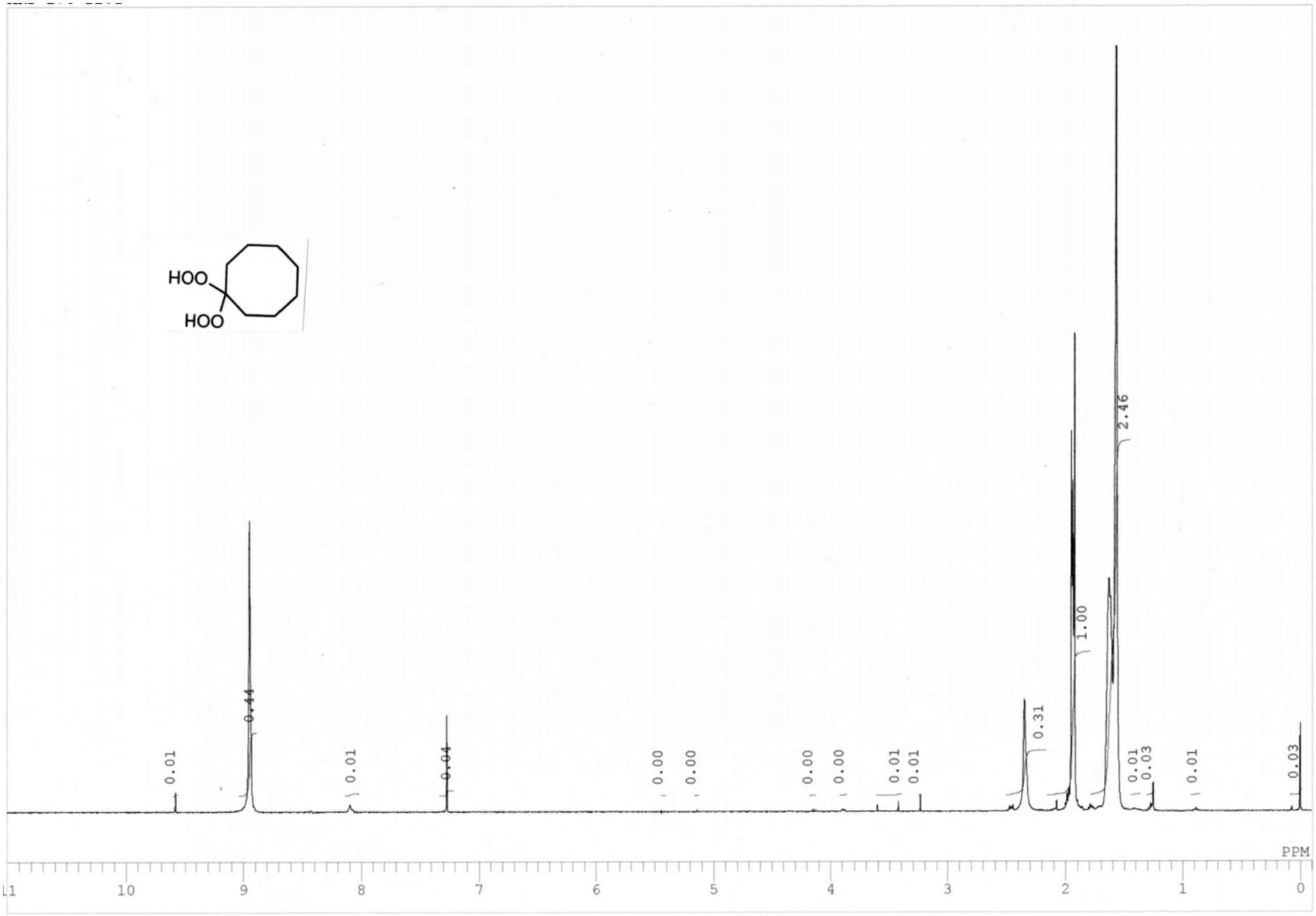
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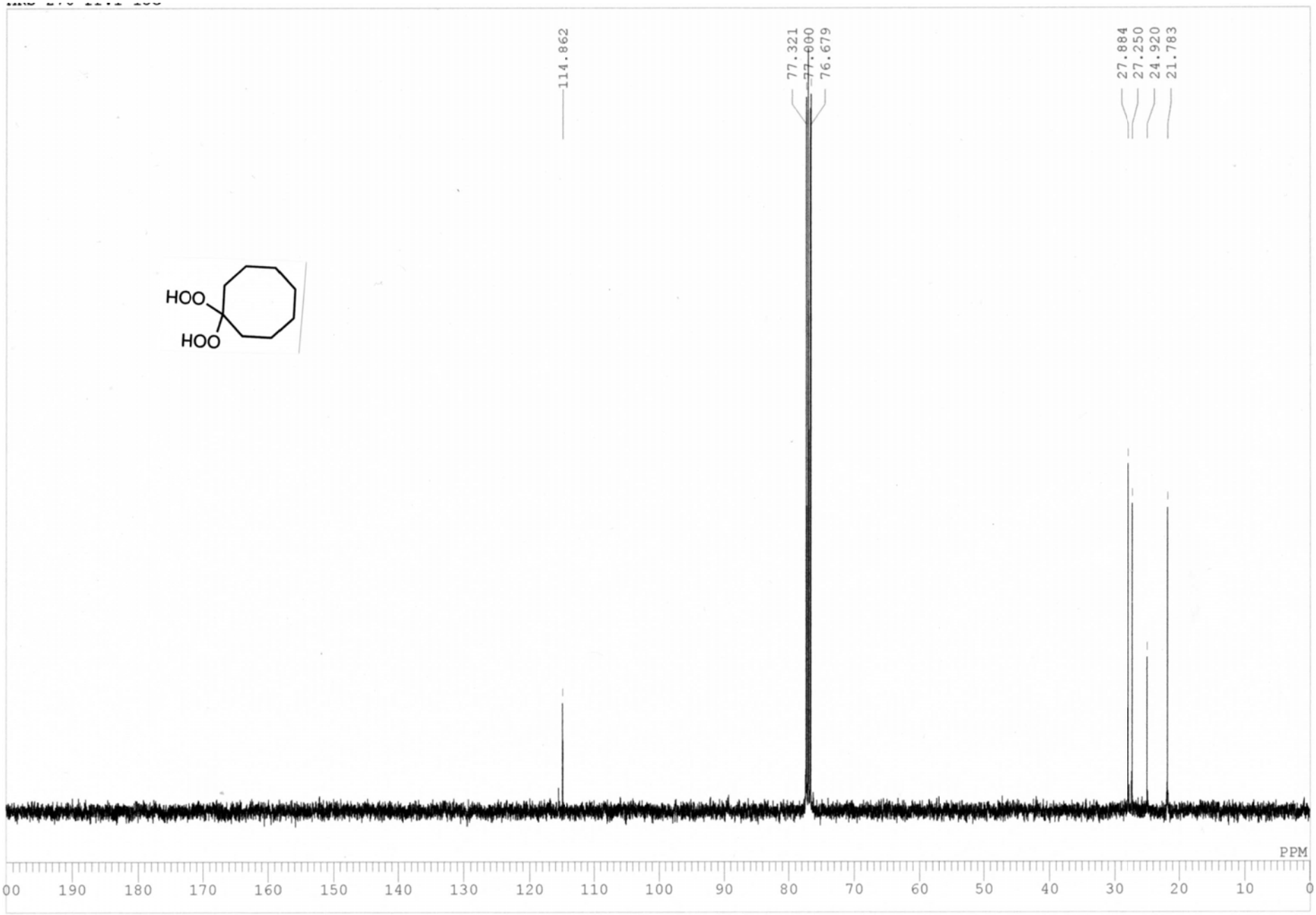
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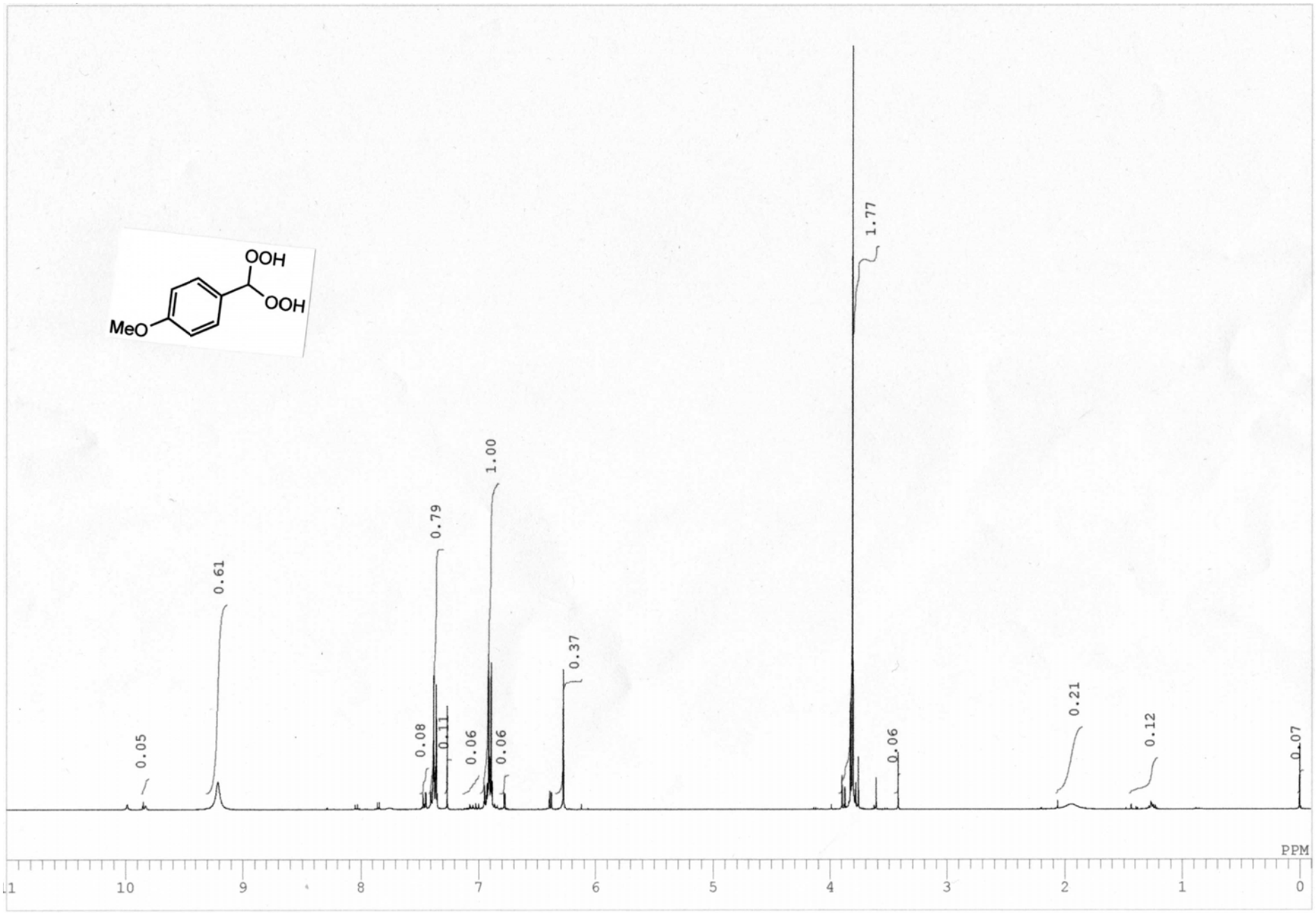
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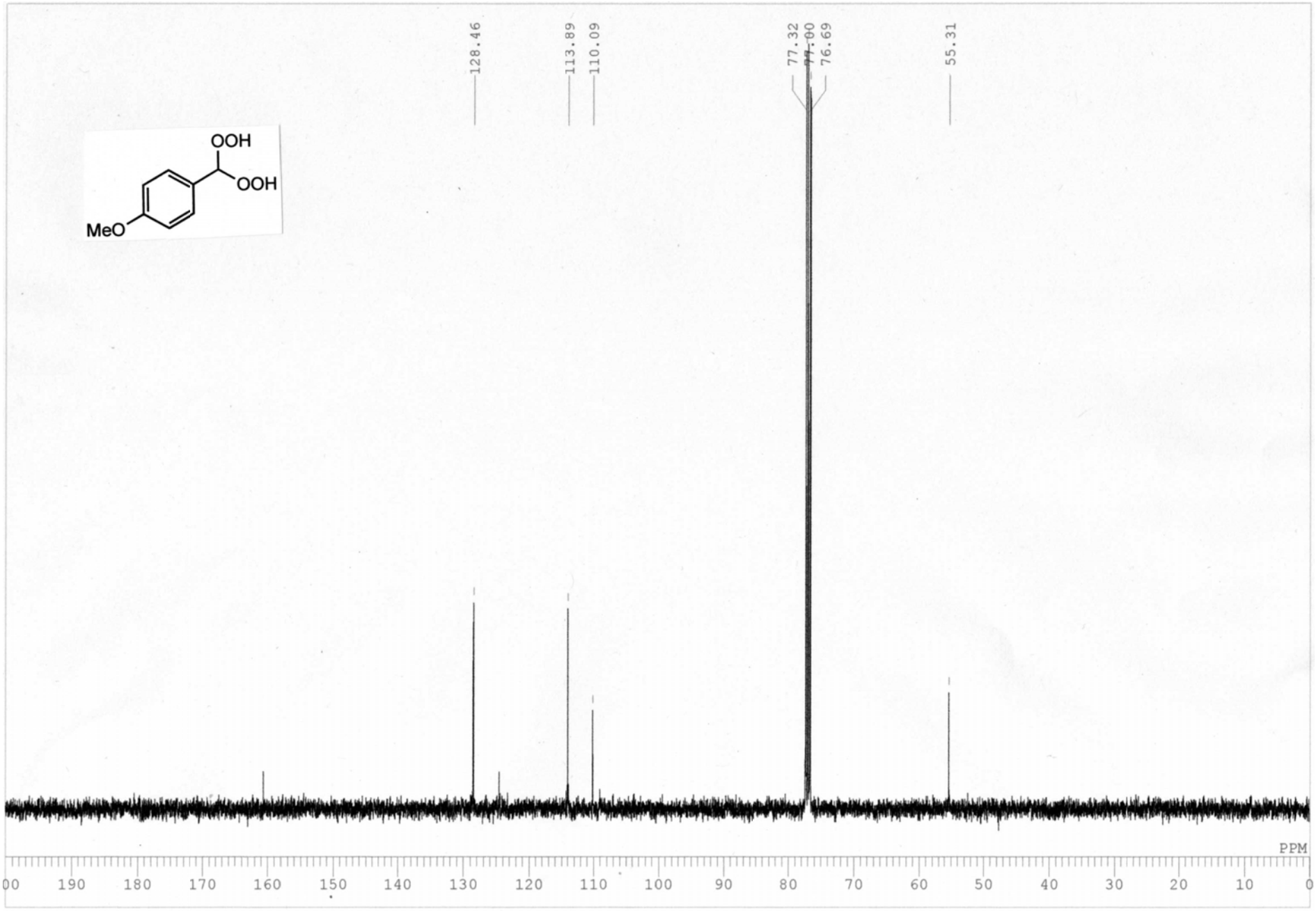
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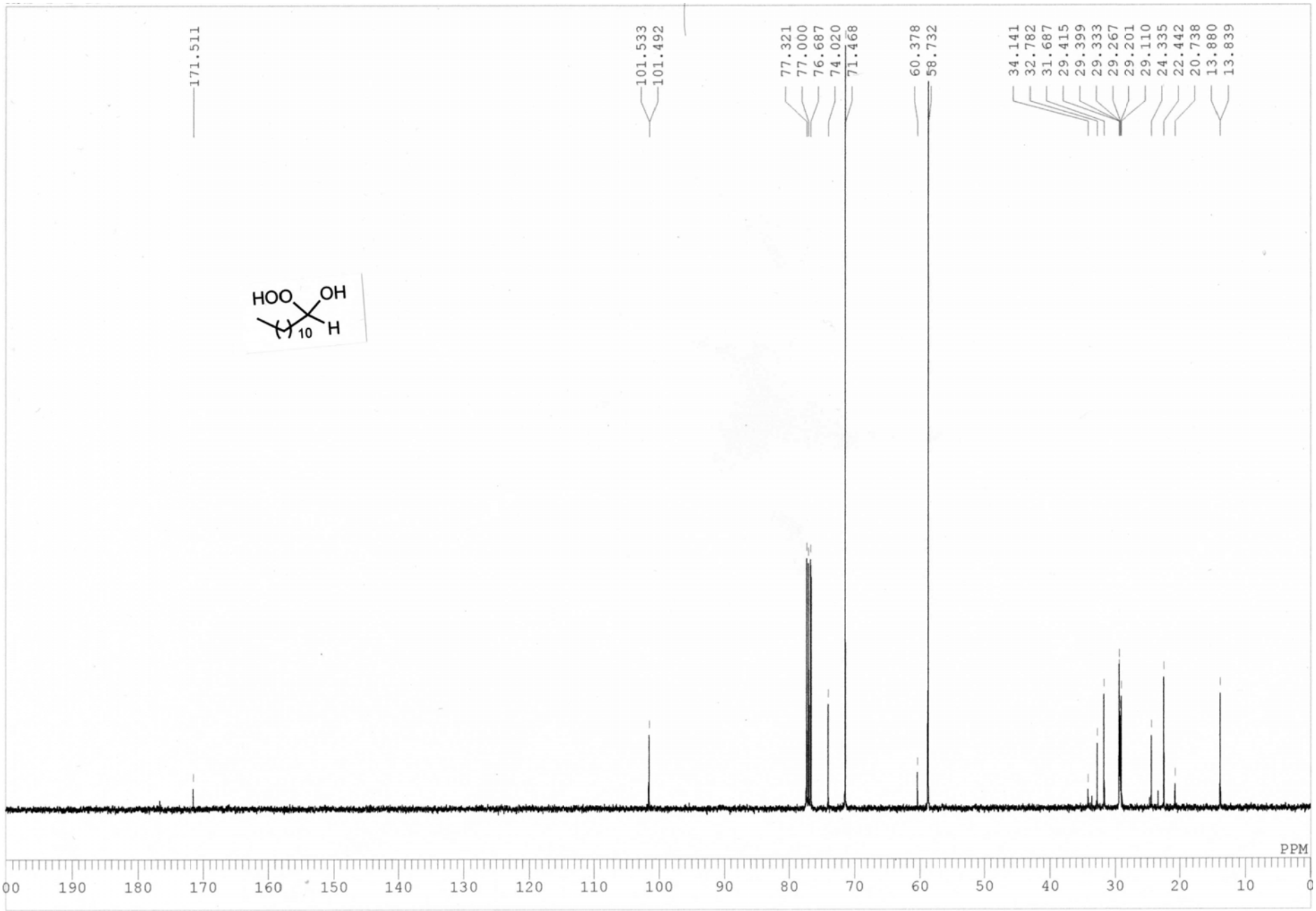
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OH
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H
PPM
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150
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