

Domino Michael-Aldol Reactions on 1,4-Diarylbut-2-ene-1,4-diones with Methyl Acetoacetate furnish Methyl 2-Aroyl-4-hydroxy-6-oxo-4-arylcyclohexane-1-carboxylate Derivatives

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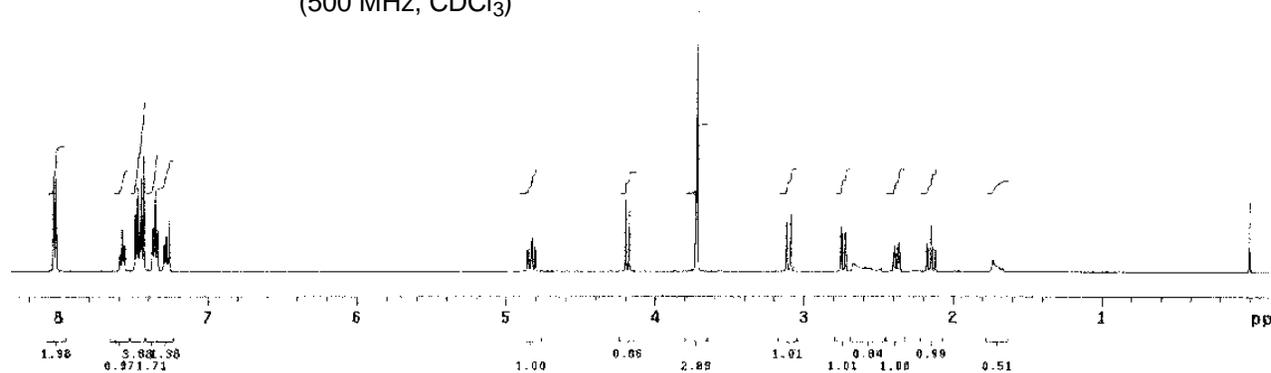
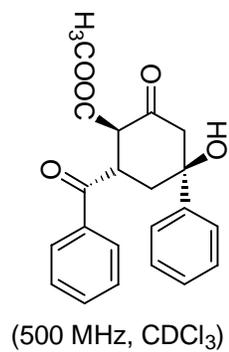
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Experimental Section, General

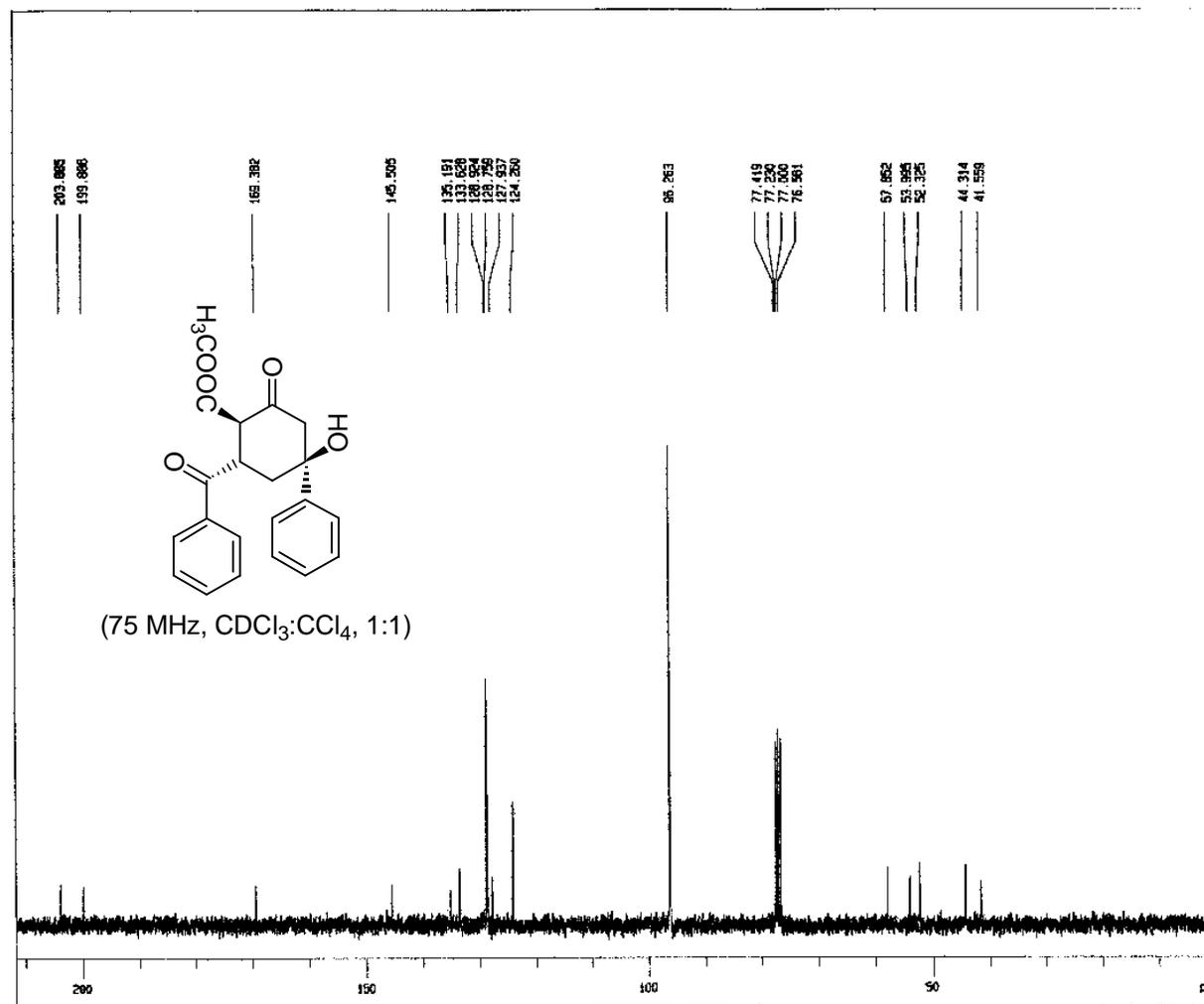
The progress of all the reactions was monitored by TLC using a hexanes / ethyl acetate mixture as eluent. Column chromatography was accomplished on silica gel (100-200 mesh) using a hexanes / ethyl acetate mixture as eluent. The IR spectra were recorded as a solution in KBr or neat. The ^1H NMR and ^{13}C NMR spectra were recorded in CDCl_3 or $\text{CDCl}_3:\text{CCl}_4$ (1:1). The microwave reactions were carried out in a mono-made, multi power; (power source: 230 V, 50 Hz, microwave frequency: 2450 MHz) microwave oven. The *trans*-DBE and derivatives (**1**, **8-11**) were prepared according to the literature procedure.¹ Activated $\text{Ba}(\text{OH})_2$ was prepared by heating commercial $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ at 200 °C for 3 h in a furnace and then stored in a desiccator.²

References

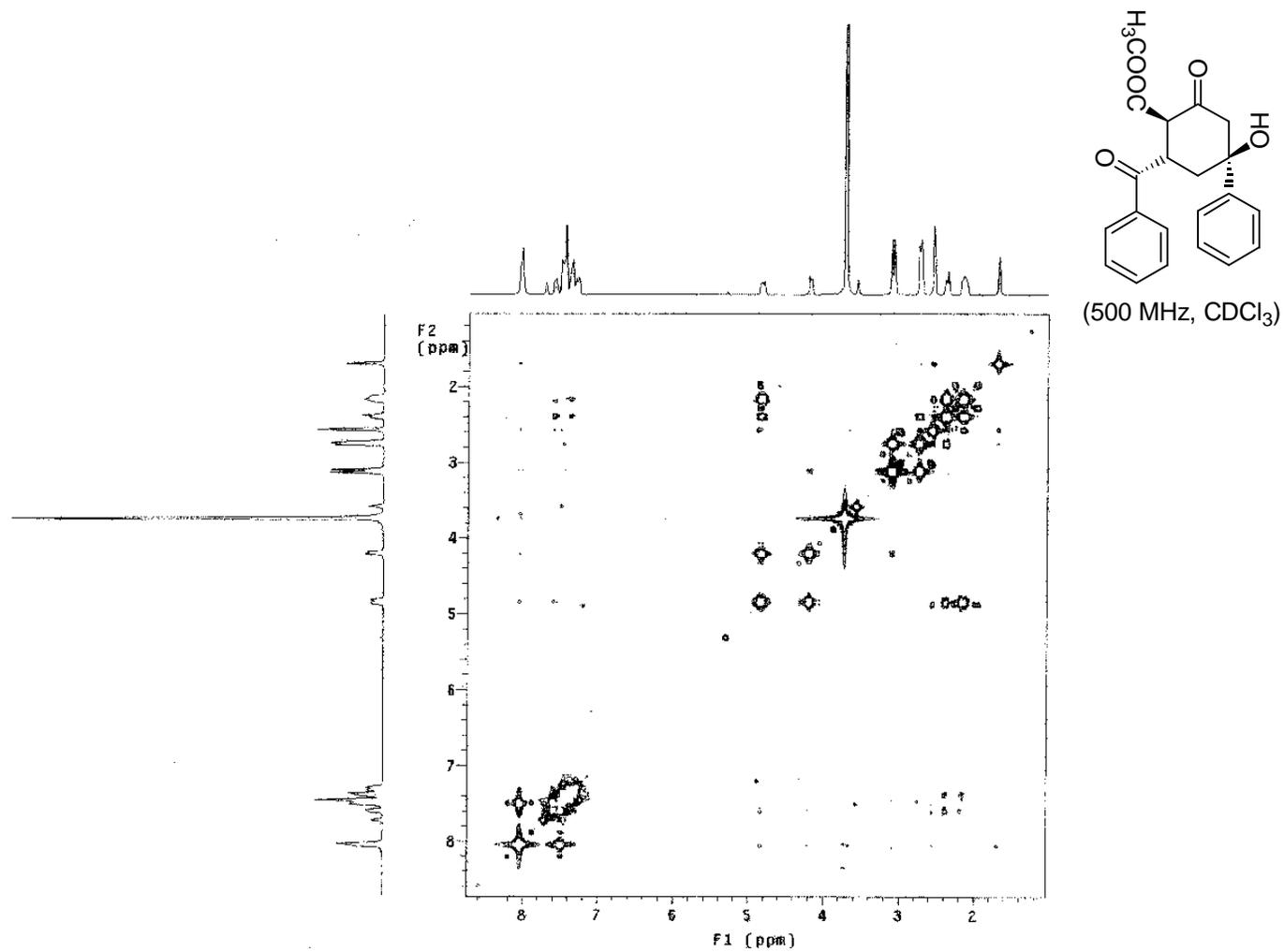
1. Conant, J. B.; Lutz, R. E. *J. Am. Chem. Soc.*, **1923**, *45*, 1303. (b) Campaigne, E.; Foye, W. O. *J. Org. Chem.*, **1952**, *17*, 1405.
2. Garcia-Raso, A.; Garcia-Raso, J.; Campaner, B.; Mestres, R.; Sinistera, J. V. *Synthesis*, **1982**, 1037.



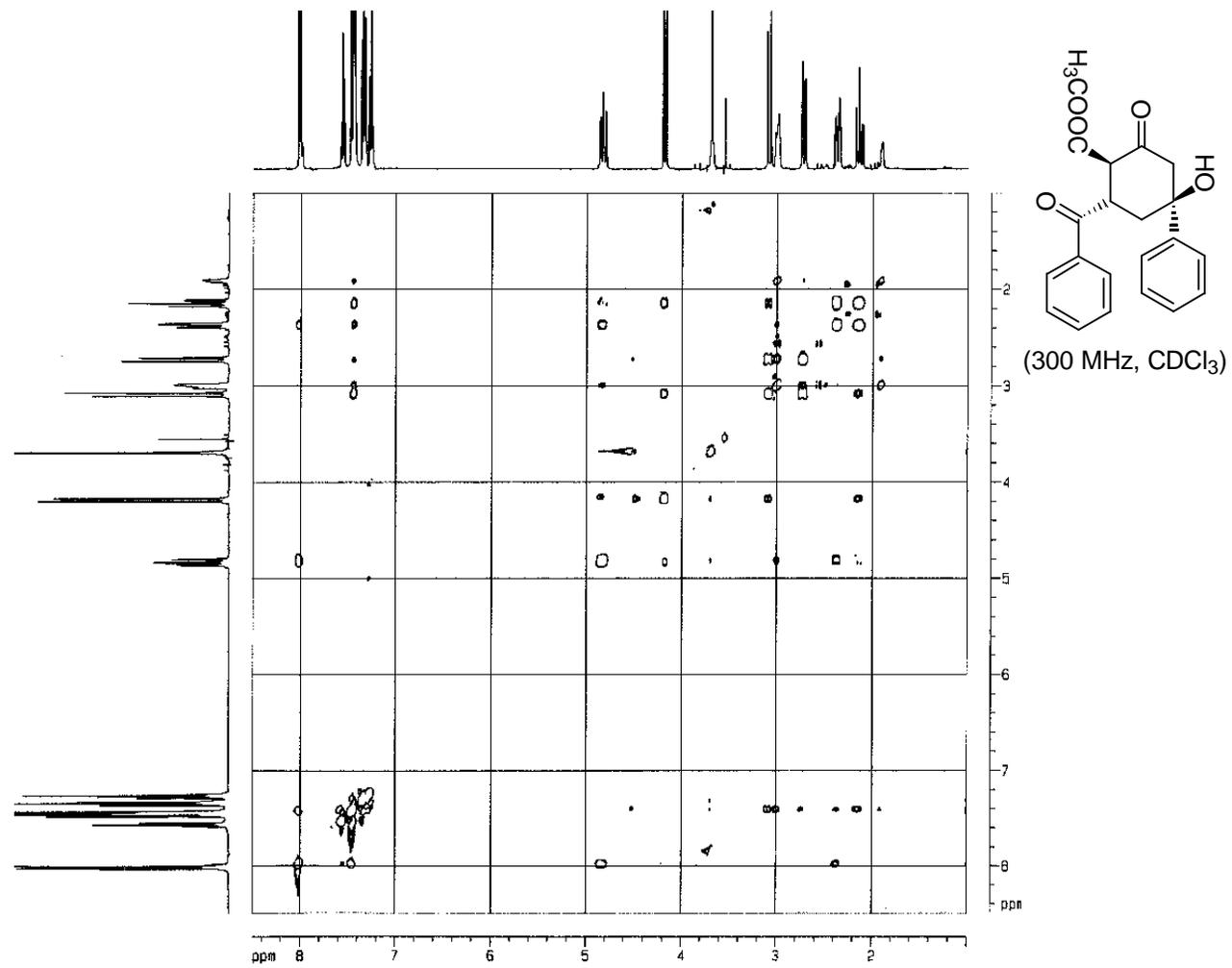
500 MHz (CDCl₃) ¹H NMR spectrum of methyl (1*R**,2*S**,4*S**)-2-benzoyl-4-hydroxy-6-oxo-4-phenylcyclohexane-1-carboxylate (**5**)



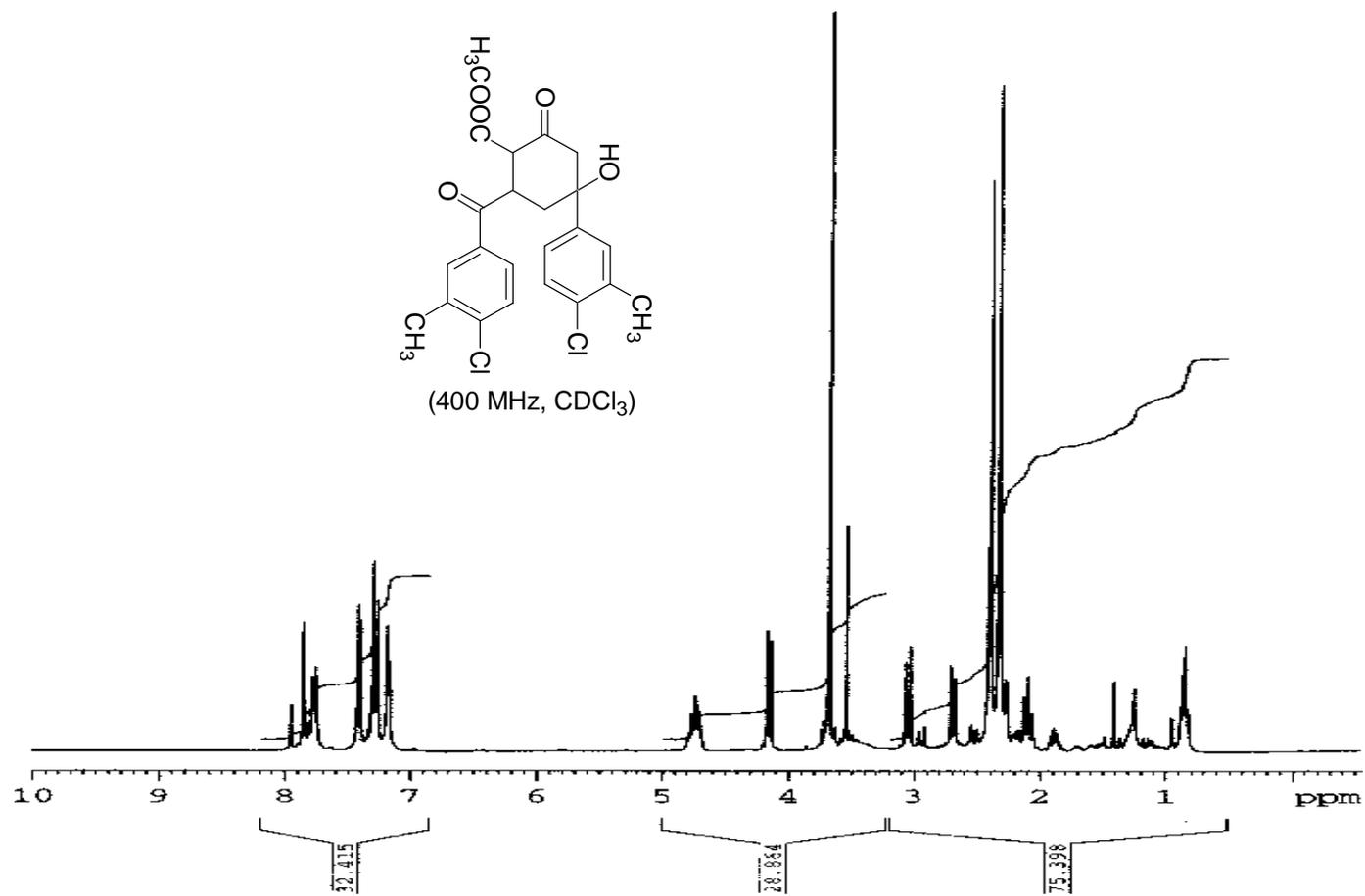
75 MHz (CDCl₃/CCl₄, 1:1) ¹³C NMR spectrum of methyl (1*R**,2*S**,4*S**)-2-benzoyl-4-hydroxy-6-oxo-4-phenylcyclohexane-1-carboxylate (**5**)



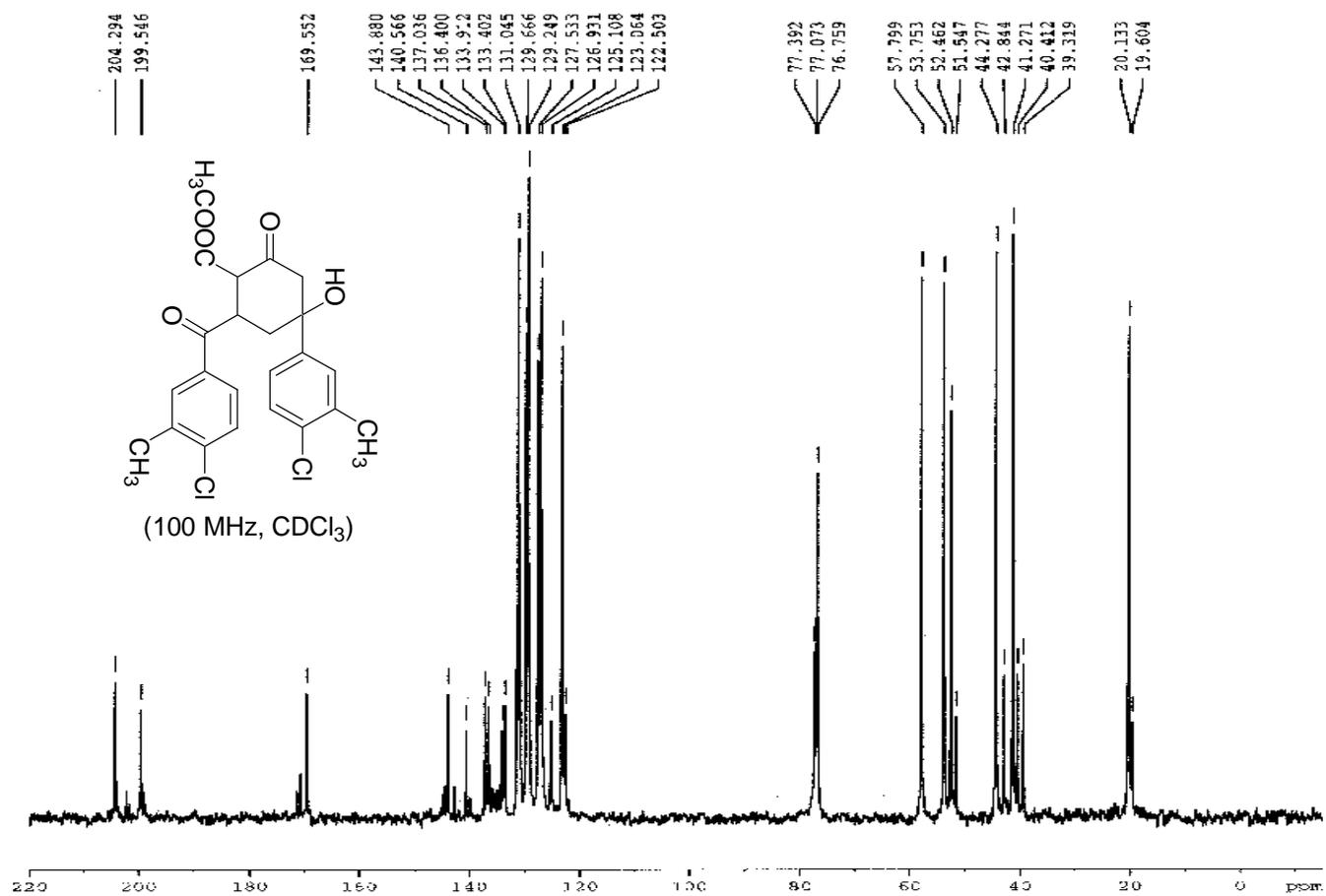
500 MHz (CDCl₃) ¹H-¹H COSY spectrum of methyl (1*R**,2*S**,4*S**)-2-benzoyl-4-hydroxy-6-oxo-4-phenylcyclohexane-1-carboxylate (**5**)



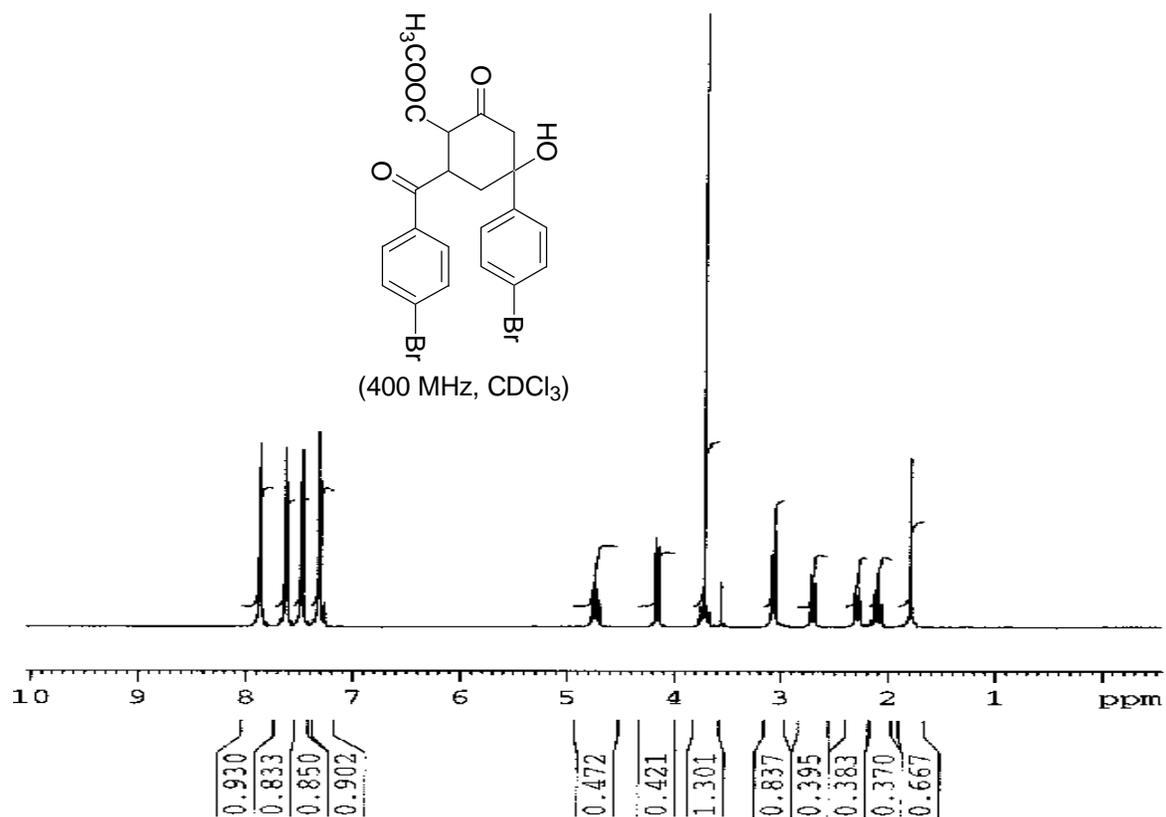
300 MHz (CDCl₃) NOESY spectrum of methyl (1*R**,2*S**,4*S**)-2-benzoyl-4-hydroxy-6-oxo-4-phenylcyclohexane-1-carboxylate (**5**)



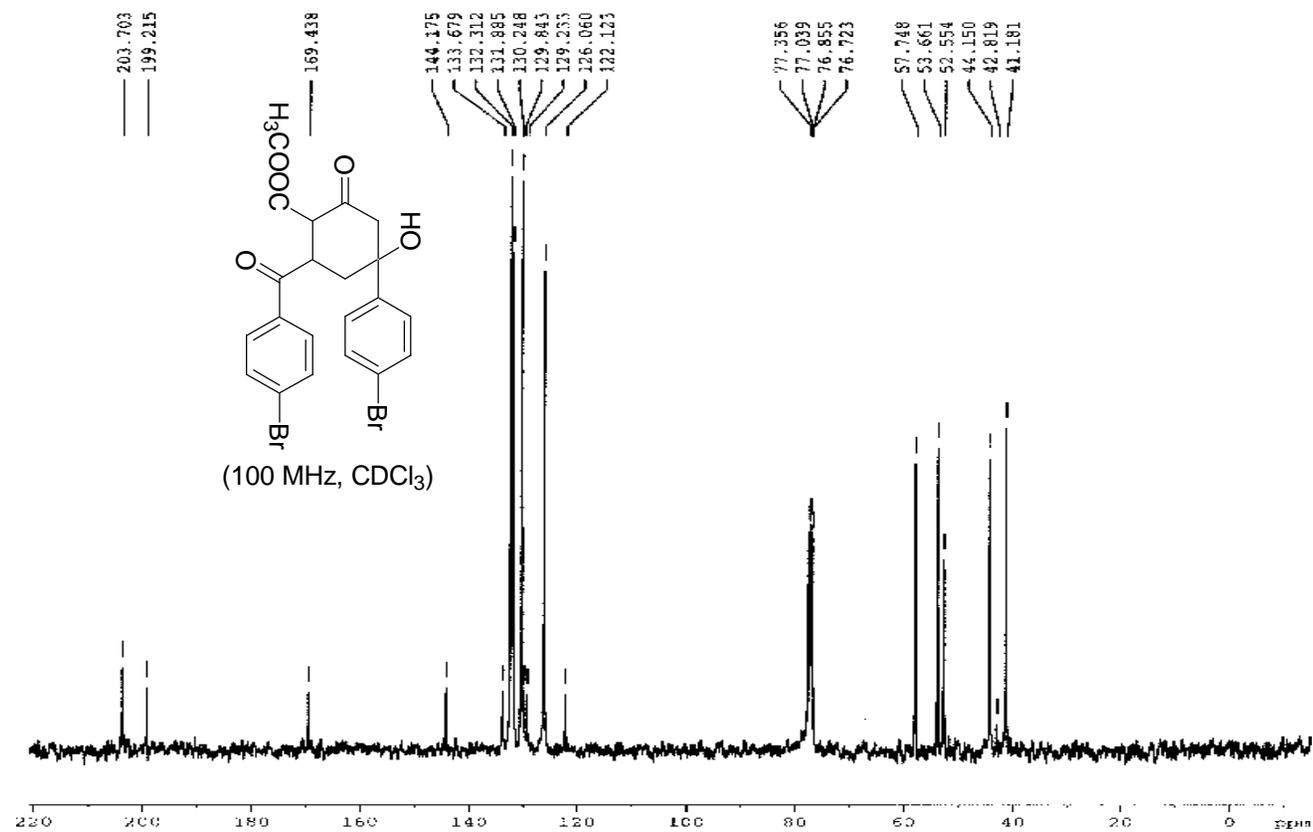
400 MHz (CDCl₃) ¹H NMR spectrum of diastereomeric mixture of methyl 2-(4-chloro-3-methylbenzoyl)-4-(4-chloro-3-methylphenyl)-4-hydroxy-6-oxocyclohexane-1-carboxylate (**12**)



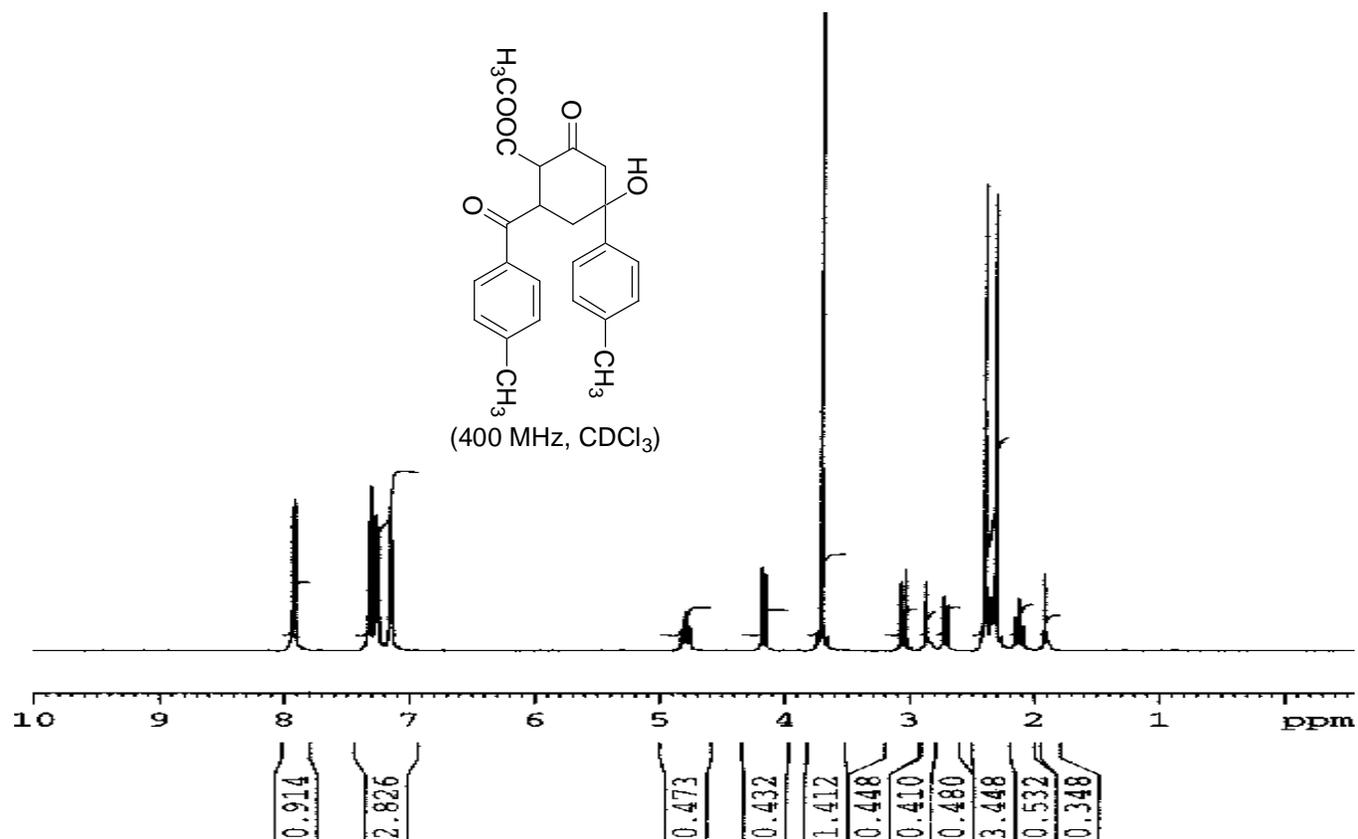
100 MHz (CDCl₃) ¹³C NMR spectrum of diastereomeric mixture of methyl 2-(4-chloro-3-methylbenzoyl)-4-(4-chloro-3-methylphenyl)-4-hydroxy-6-oxocyclohexane-1-carboxylate (**12**)



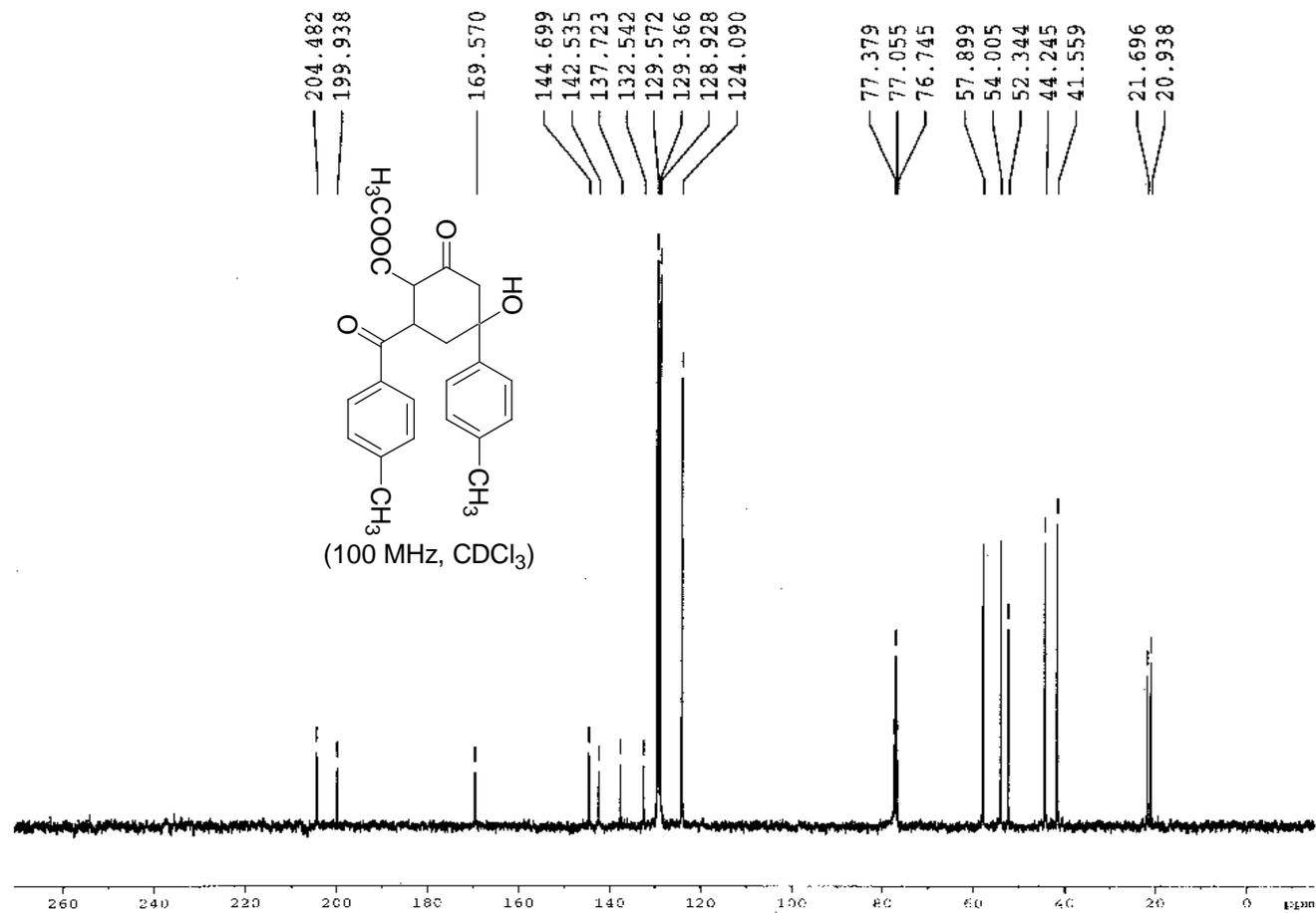
400 MHz (CDCl₃) ¹H NMR spectrum of methyl (1*R**2*S**,4*S**)-2-(4-bromobenzoyl)-4-(4-bromophenyl)-4-hydroxy-6-oxocyclohexane-1-carboxylate (**13**)



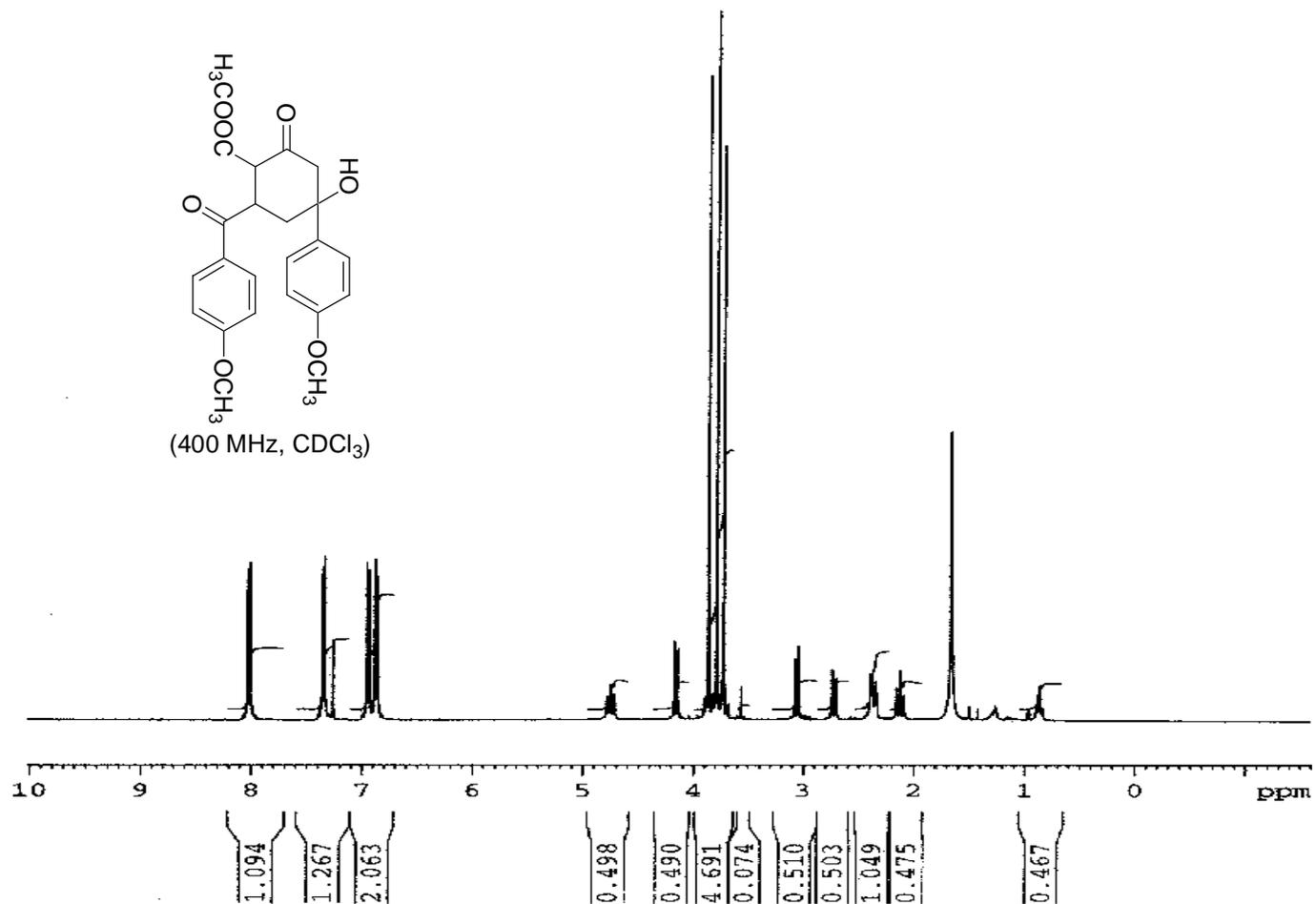
100 MHz (CDCl₃) ¹³C NMR spectrum of methyl (1*R**,2*S**,4*S**)-2-(4-bromobenzoyl)-4-(4-bromophenyl)-4-hydroxy-6-oxocyclohexane-1-carboxylate (**13**)



400 MHz (CDCl₃) ¹H NMR spectrum of methyl (1R*,2S*,4S*)-4-hydroxy-2-(4-methylbenzoyl)-4-(4-methylphenyl)-6-oxocyclohexane-1-carboxylate (**14**)

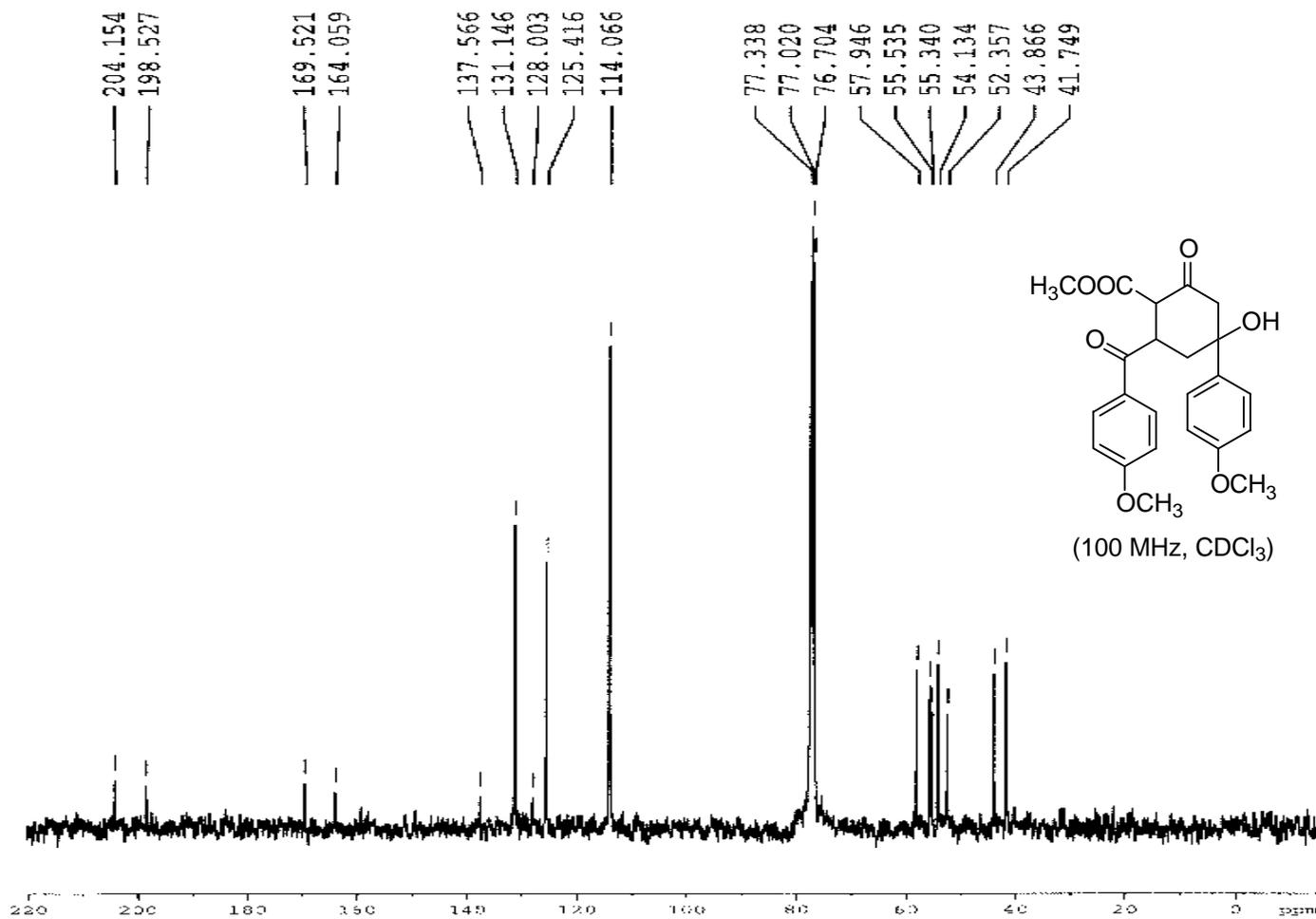


100 MHz (CDCl_3) ^{13}C NMR spectrum of methyl (1*R**,2*S**,4*S**)-4-hydroxy-2-(4-methylbenzoyl)-4-(4-methylphenyl)-6-oxocyclohexane-1-carboxylate (**14**)

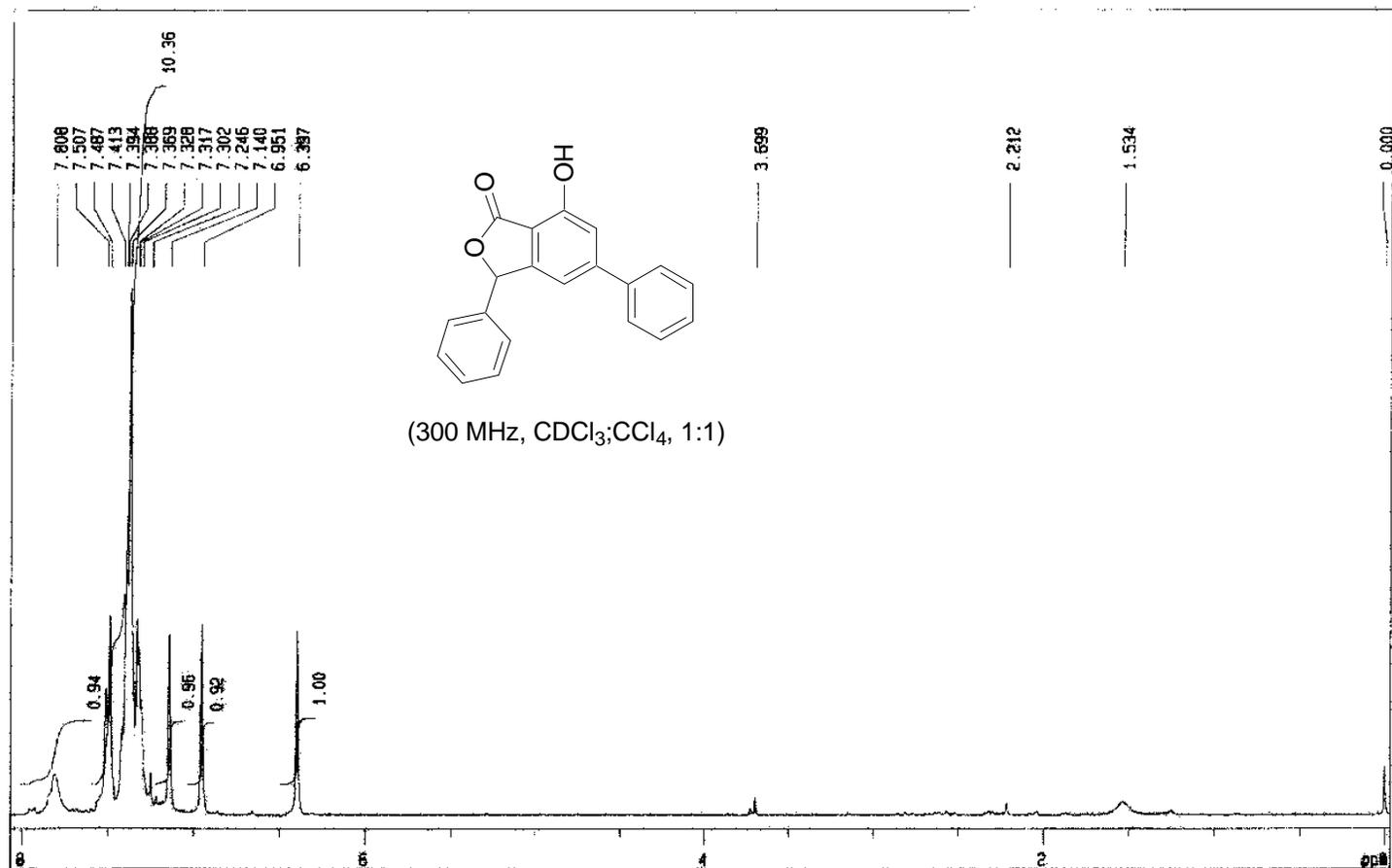


400 MHz (CDCl₃) ¹H NMR spectrum of methyl (1*R**,2*S**,4*S**)-4-hydroxy-2-(4-methoxybenzoyl)-4-(4-methoxyphenyl)-6-oxocyclohexane-1-carboxylate (**15**)

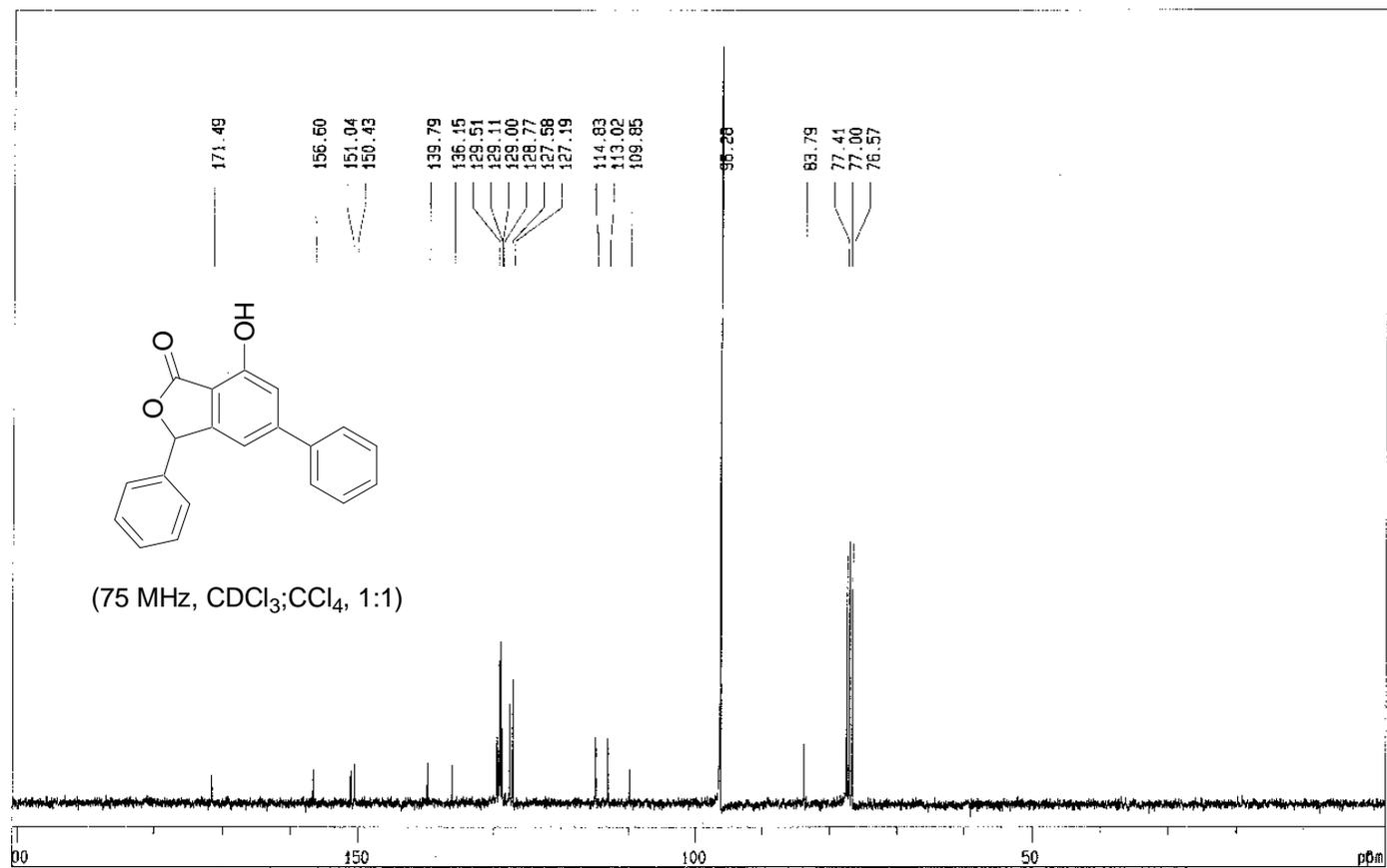
SIS



100 MHz (CDCl₃) ¹³C NMR spectrum of methyl (1*R**,2*S**,4*S**)-4-hydroxy-2-(4-methoxybenzoyl)-4-(4-methoxyphenyl)-6-oxocyclohexane-1-carboxylate (**15**)



300 MHz (CDCl₃:CCl₄, 1:1) ¹H NMR spectrum of 7-hydroxy-3,5-diphenyl-1,3-dihydro-1-isobenzofuranone (18)



75 MHz (CDCl₃:CCl₄, 1:1) ¹³C NMR spectrum of 7-hydroxy-3,5-diphenyl-1,3-dihydro-1-isobenzofuranone (**18**)