Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Rh-catalyzed direct synthesis of 2,2'-dihydroxybenzophenones and xanthones

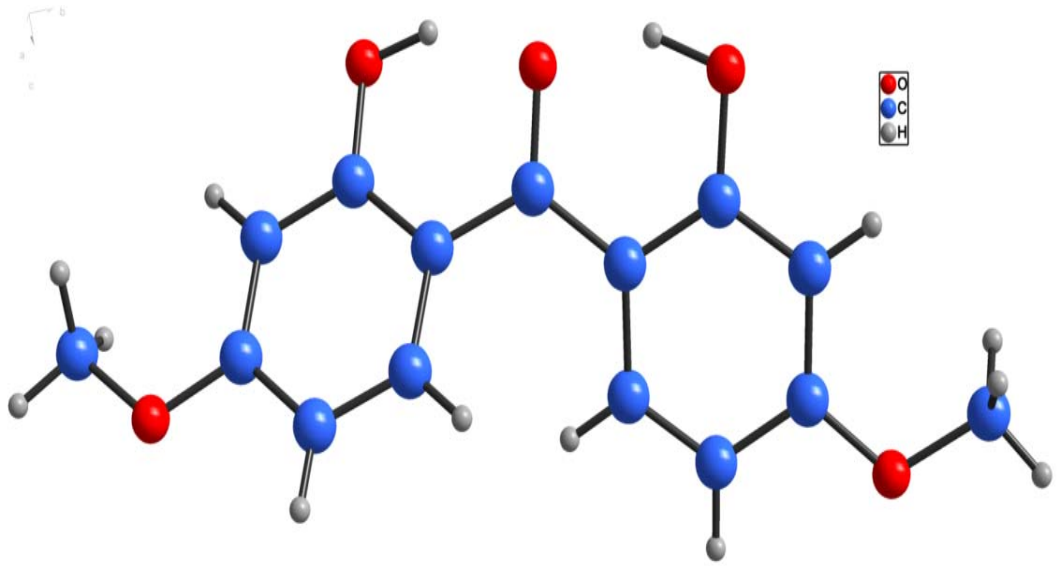
Maddali L. N. Rao* **and Boddu S. Ramakrishna**

Department of Chemistry, Indian Institute of Technology Kanpur, Kanpur-208016, India

maddali@iitk.ac.in

Contents

1. Crystallographic data for Compound 2.2 (CCDC-1480785):

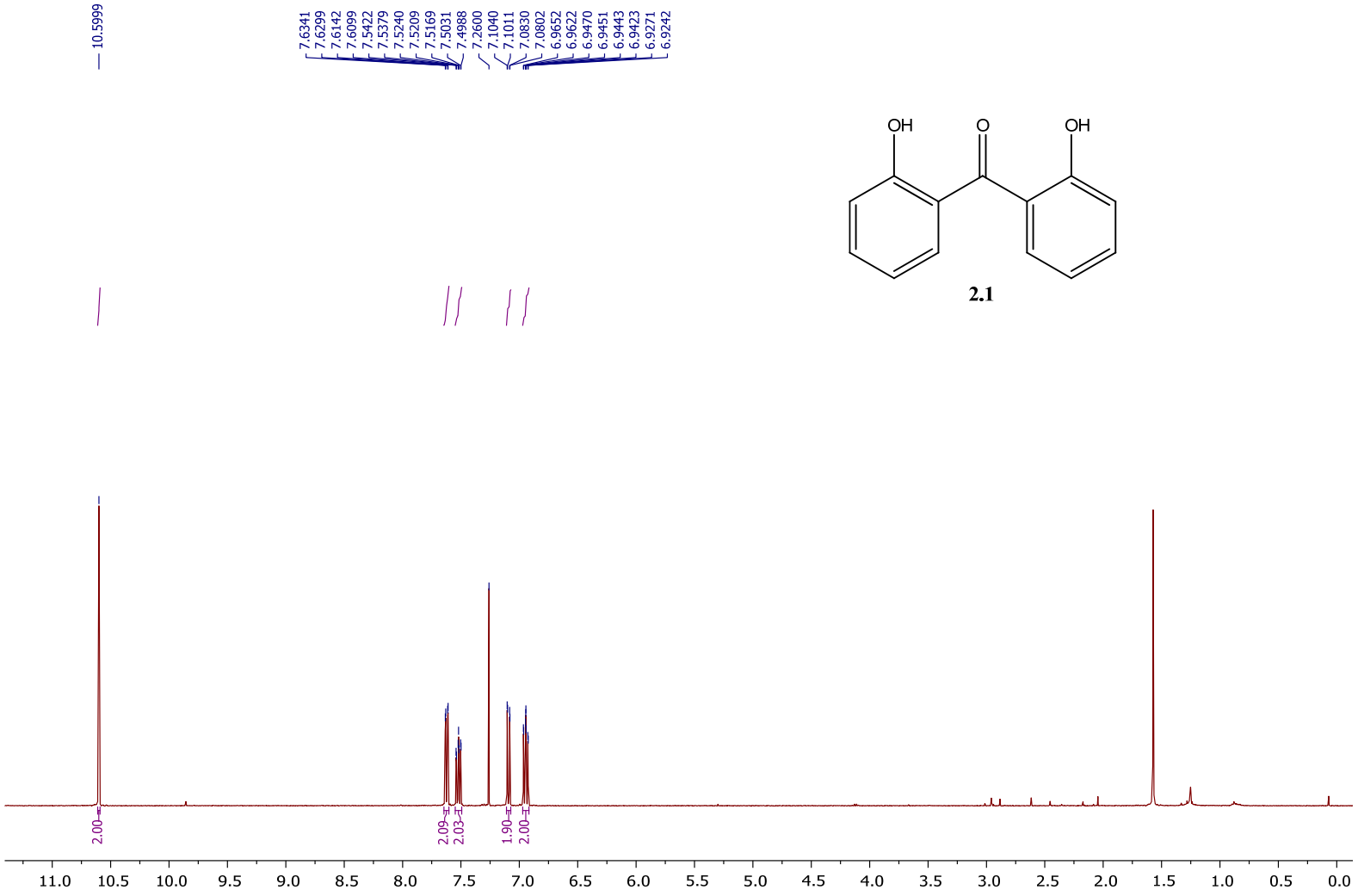


Crystal data and structure refinement for 2marb_0m.

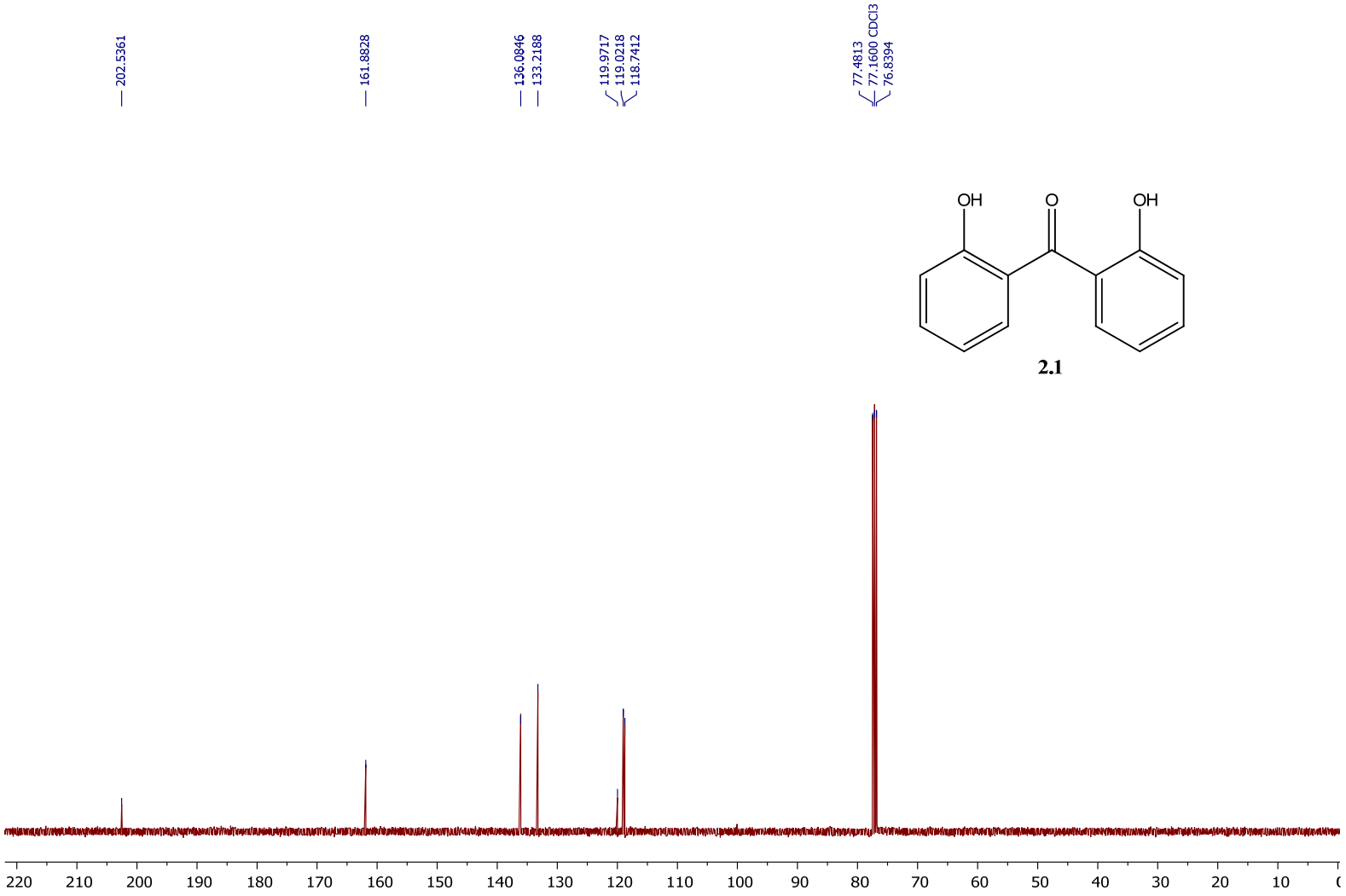
Identification code	2marb_0m	
Empirical formula	C15 H14 O5	
Formula weight	274.26	
Temperature	298(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/n	
Unit cell dimensions	a = 3.8416(3) Å	a= 90°.
	b = 25.1212(16) Å	b= 92.567(2)°.
	c = 12.9636(9) Å	g = 90°.

Volume	1249.80(15) $Å^3$
Z	4
Density (calculated)	1.458 Mg/m^3
Absorption coefficient	0.110 mm^{-1}
F(000)	576
Crystal size	0.16 x 0.13 x 0.12 mm^3
Theta range for data collection	2.26 to 28.35°
Index ranges	-5<=h<=5, -33<=k<=33, -13<=l<=17
Reflections collected	12538
Independent reflections	3107 [R(int) = 0.0674]
Completeness to theta = 28.35°	99.8 %
Absorption correction	Empirical
Max. and min. transmission	0.987 and 0.983
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3107 / 0 / 190
Goodness-of-fit on F^2	1.055
Final R indices [I>2sigma(I)]	R1 = 0.0581, wR2 = 0.1219
R indices (all data)	R1 = 0.1180, wR2 = 0.1535
Largest diff. peak and hole	0.292 and -0.362 $e.Å^{-3}$

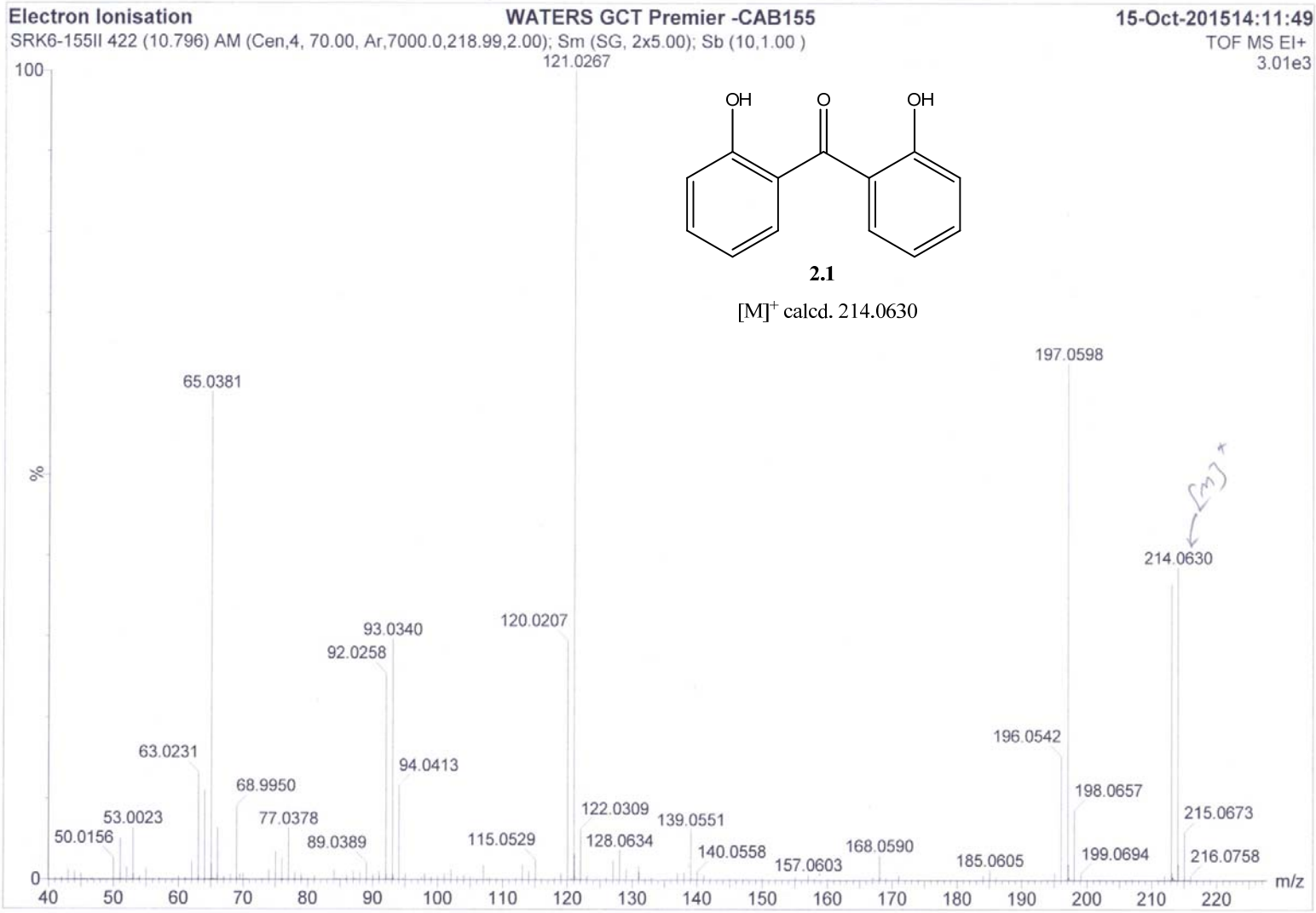
2. Copies of ^{1}H NMR, ^{13}C NMR and HRMS spectra of Compound 2.1 to 2.11 (Table 2):



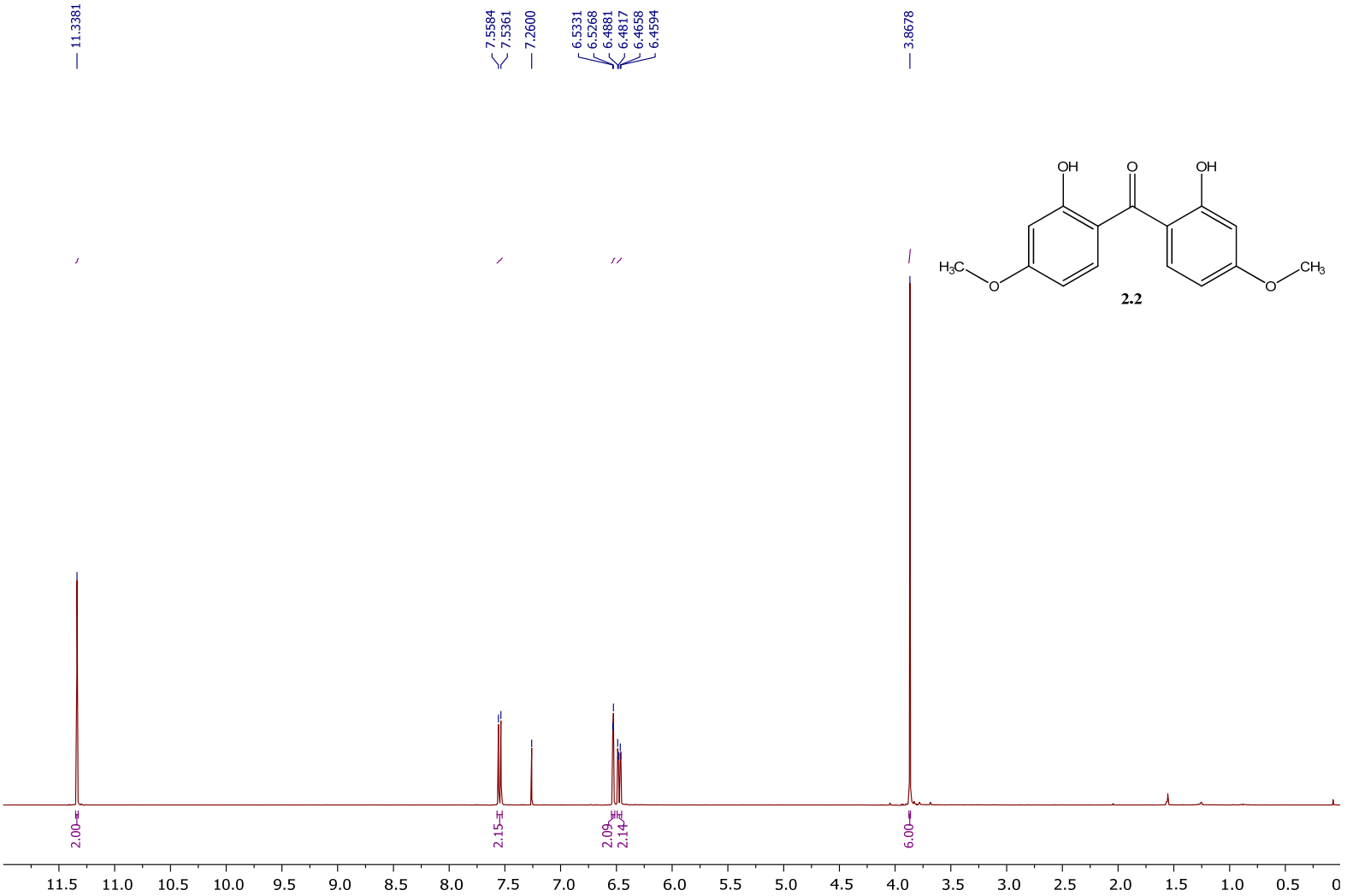
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(2-hydroxyphenyl)methanone (**2.1**)



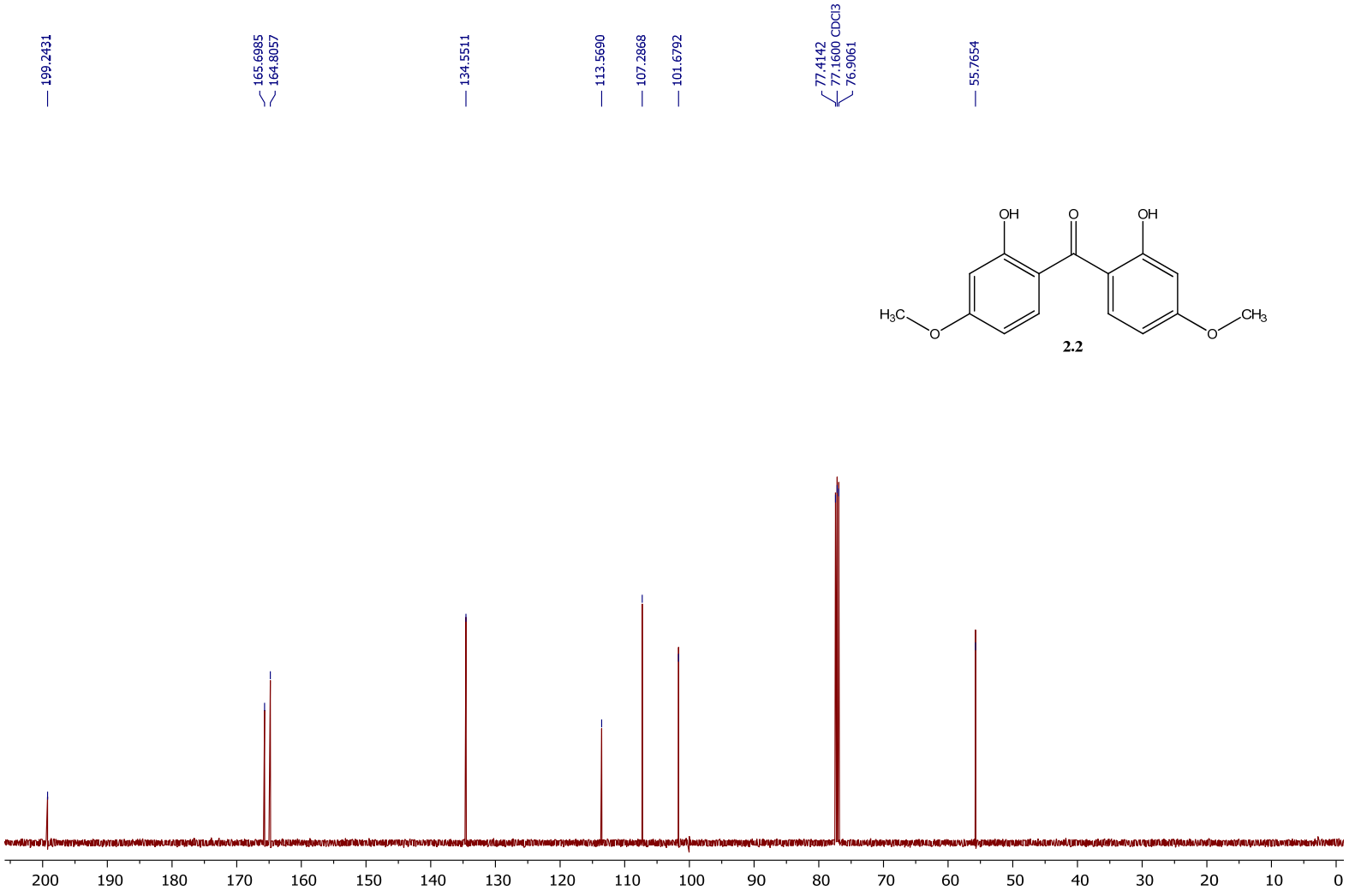
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of bis(2-hydroxyphenyl)methanone (**2.1**)



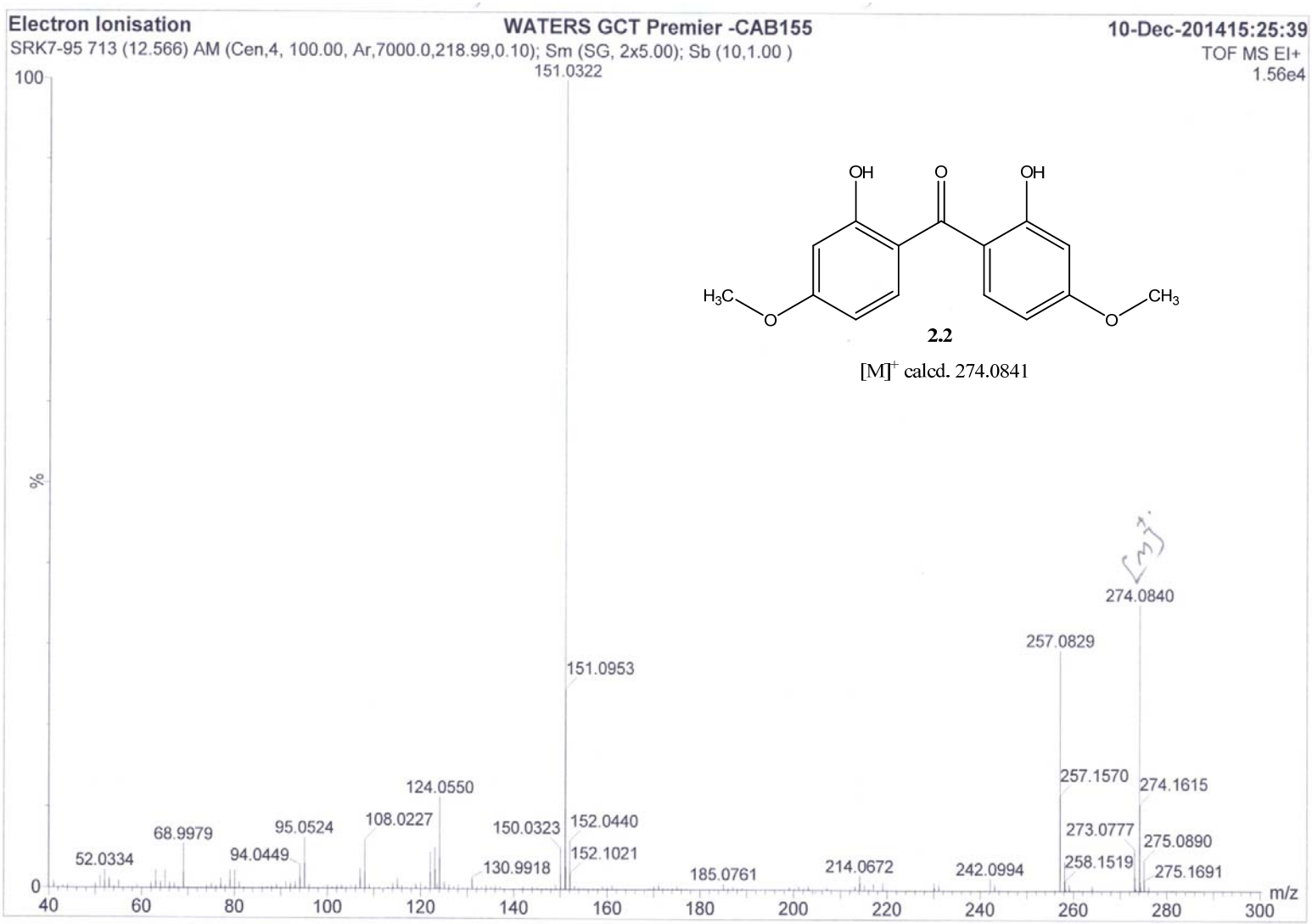
EI(HRMS) spectrum of bis(2-hydroxyphenyl)methanone (**2.1**)



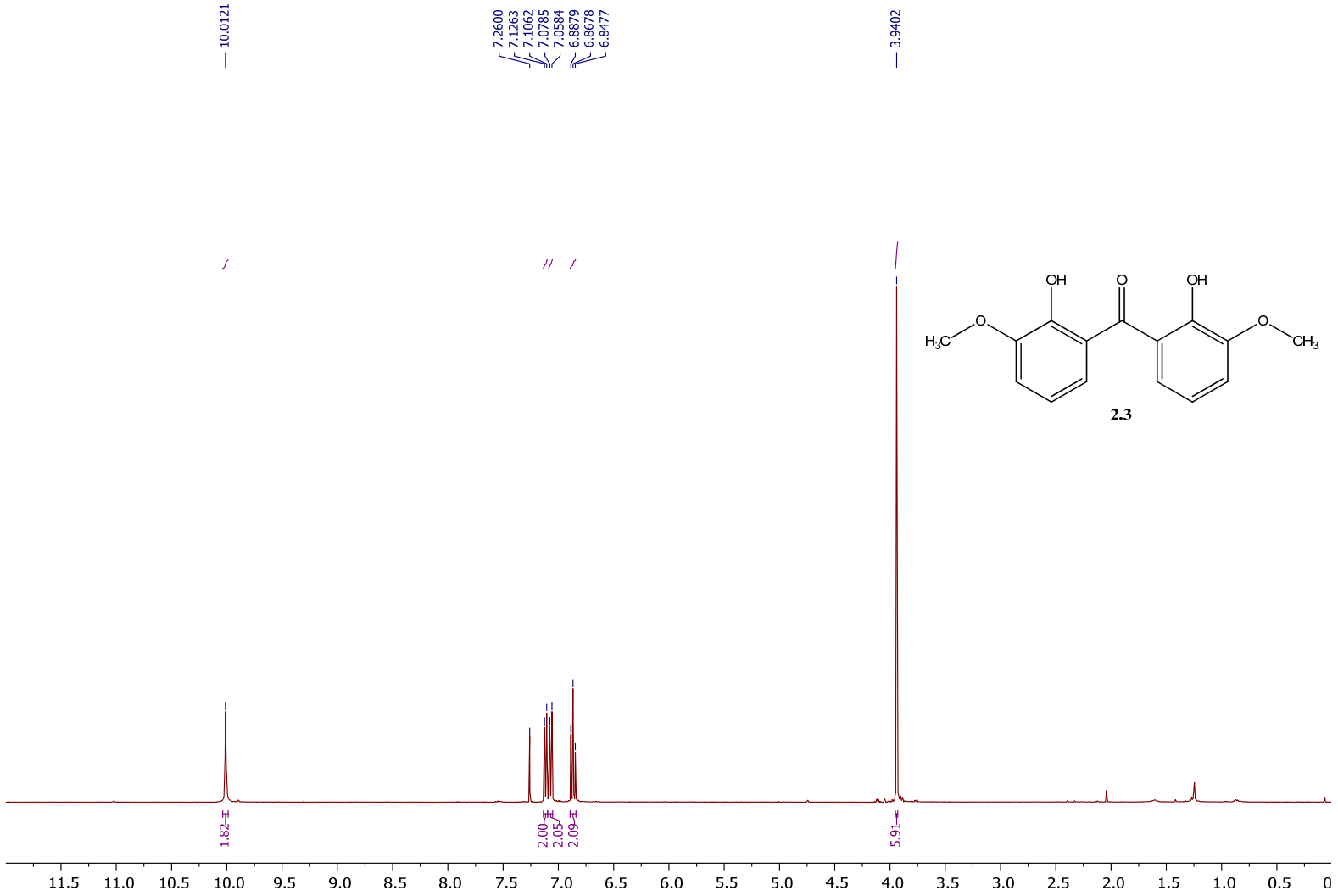
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-4-methoxyphenyl)methanone (**2.2**)



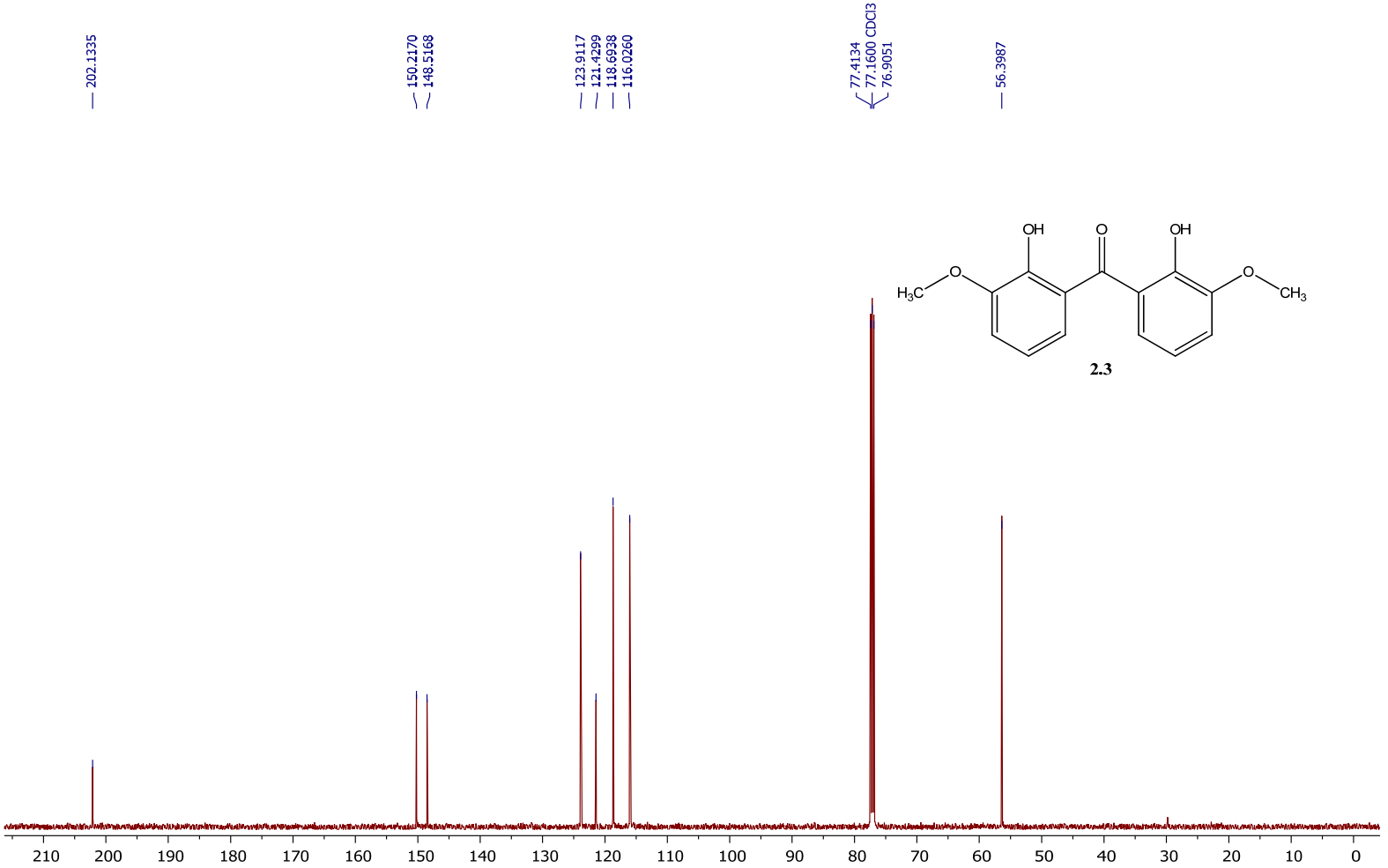
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-4-methoxyphenyl)methanone (**2.2**)



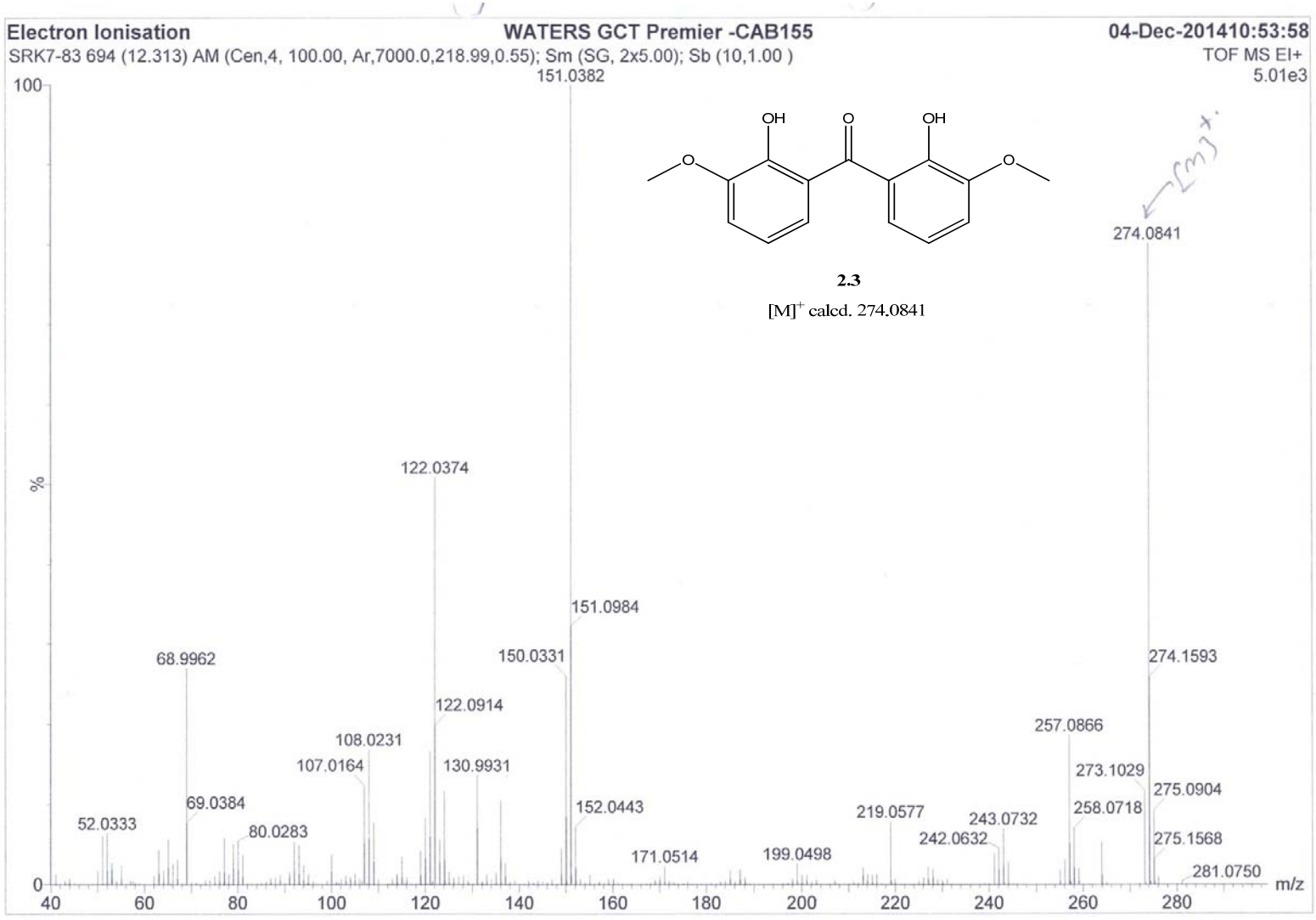
EI(HRMS) spectrum of bis(2-hydroxy-4-methoxyphenyl)methanone (**2.2**)



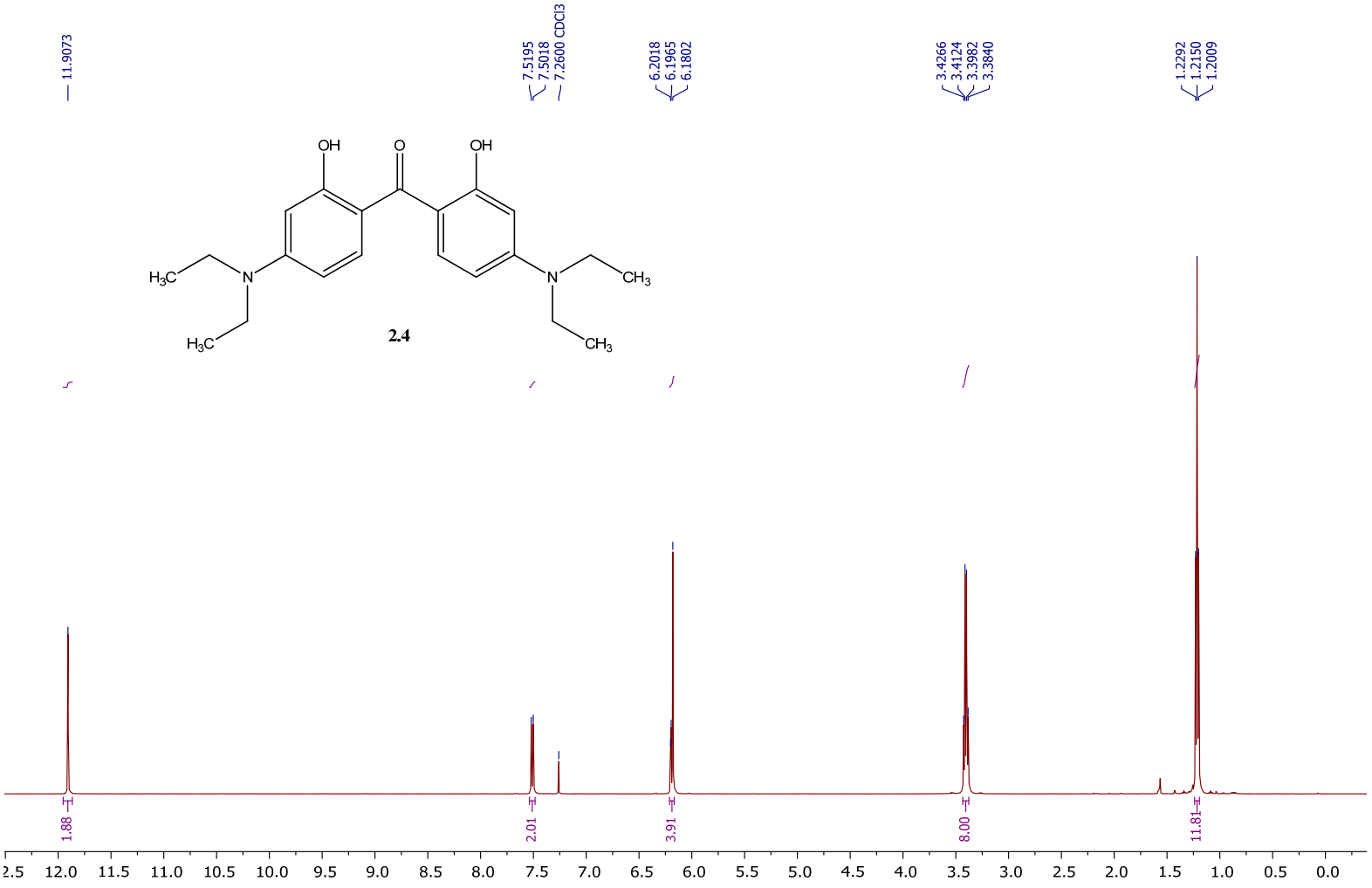
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-3-methoxyphenyl)methanone (**2.3**)



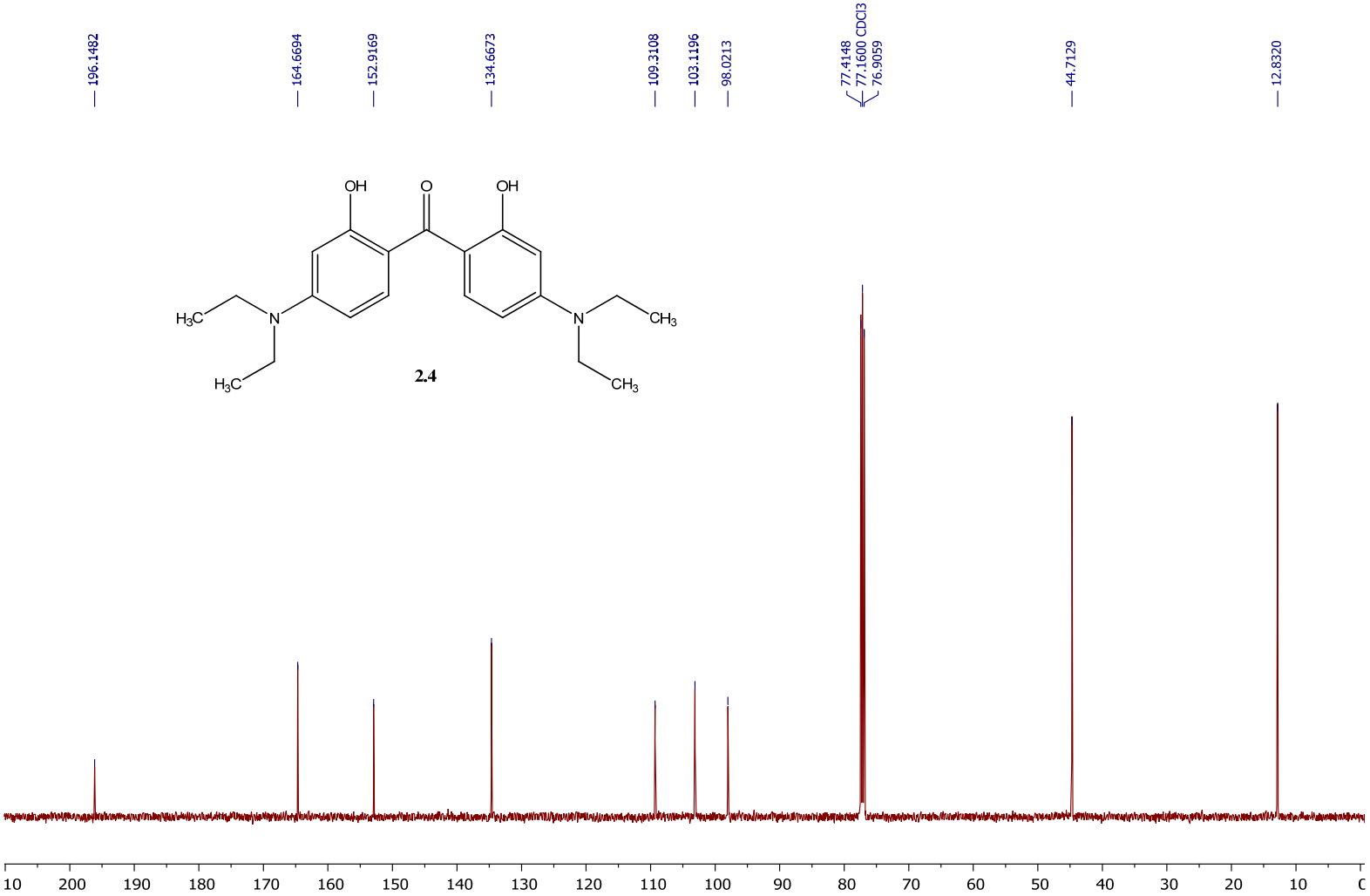
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-3-methoxyphenyl)methanone (**2.3**)



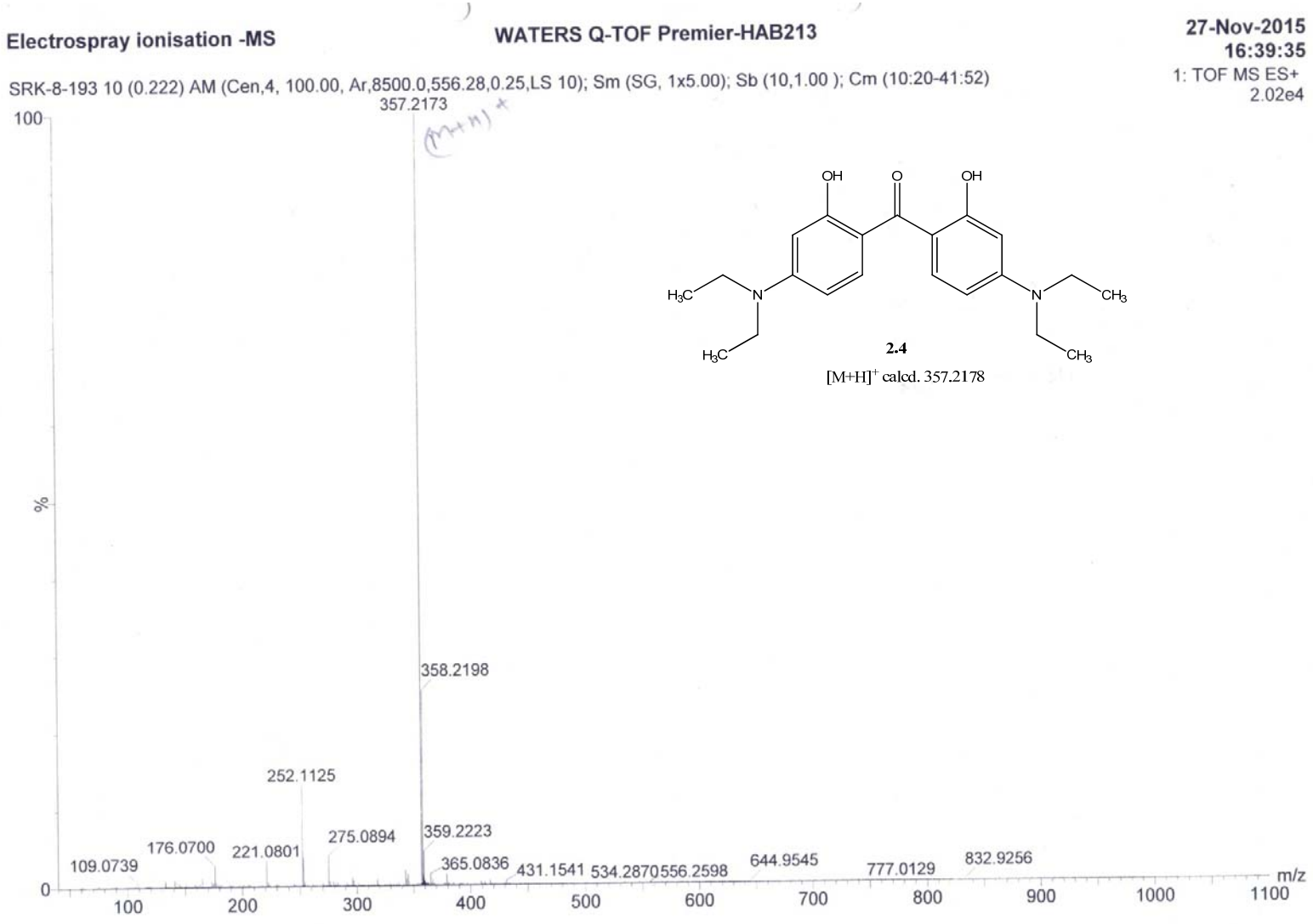
EI(HRMS) spectrum of bis(2-hydroxy-3-methoxyphenyl)methanone (**2.3**)



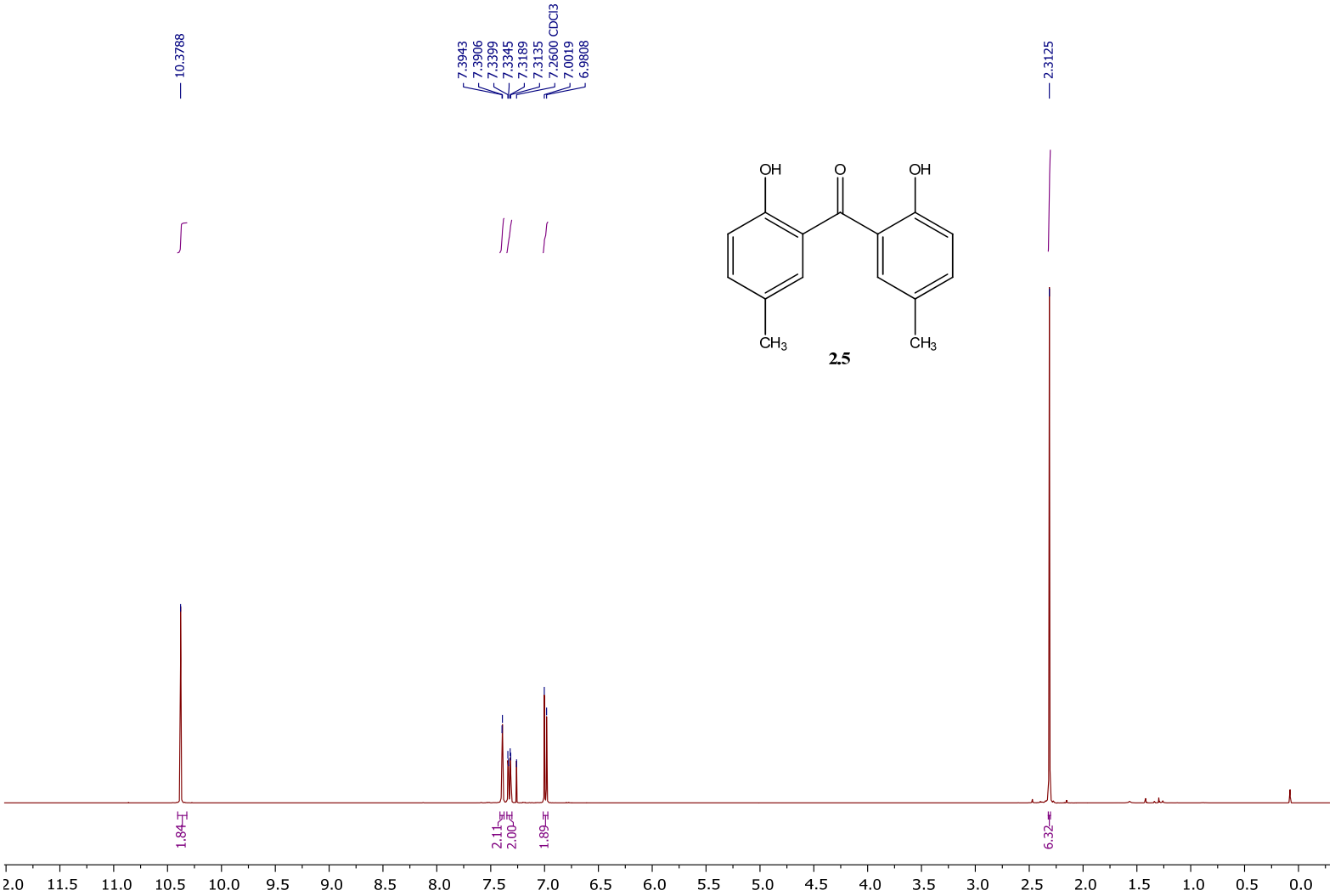
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of bis(4-(diethylamino)-2-hydroxyphenyl)methanone (**2.4**)



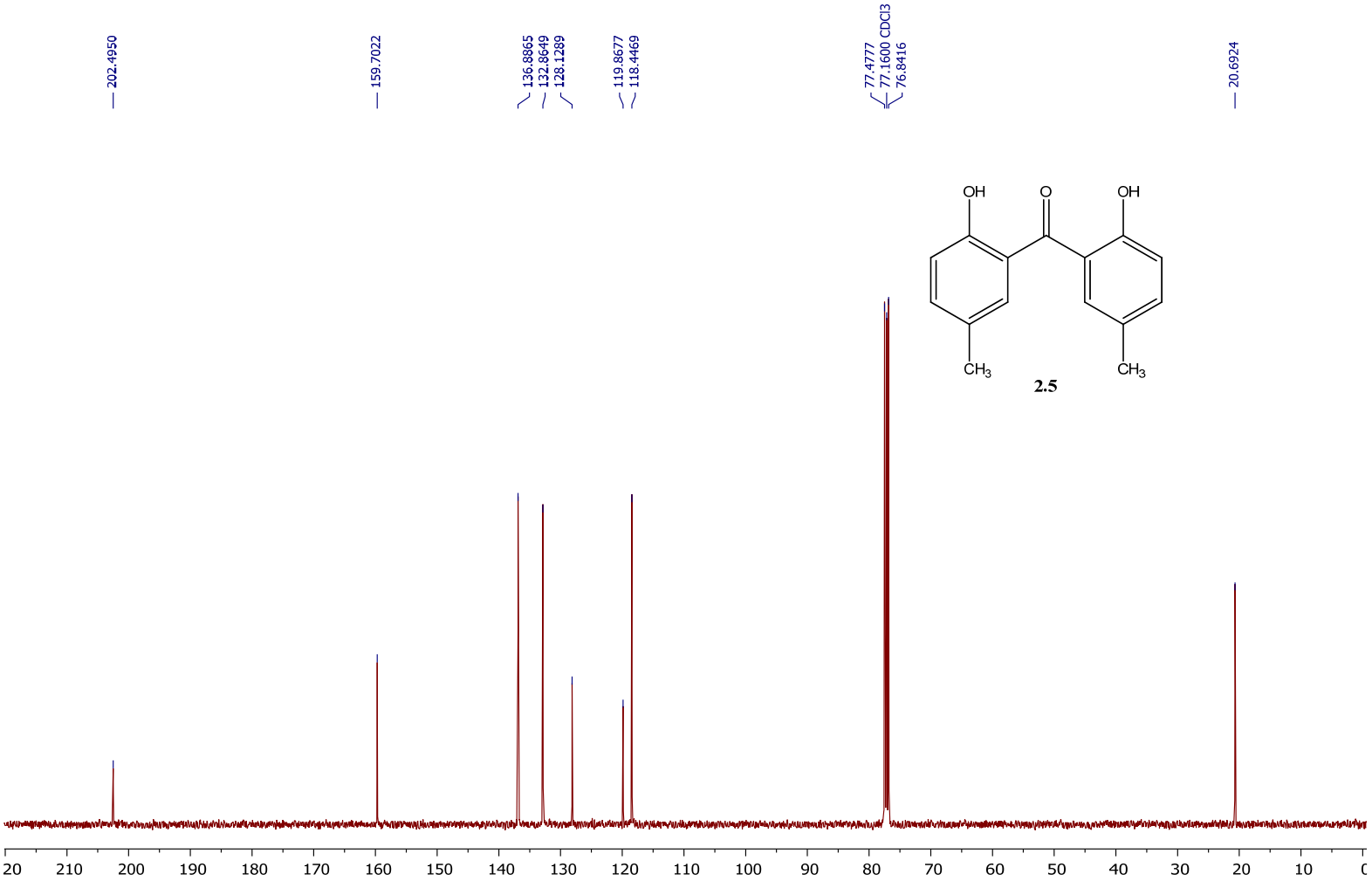
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of bis(4-(diethylamino)-2-hydroxyphenyl)methanone (**2.4**)



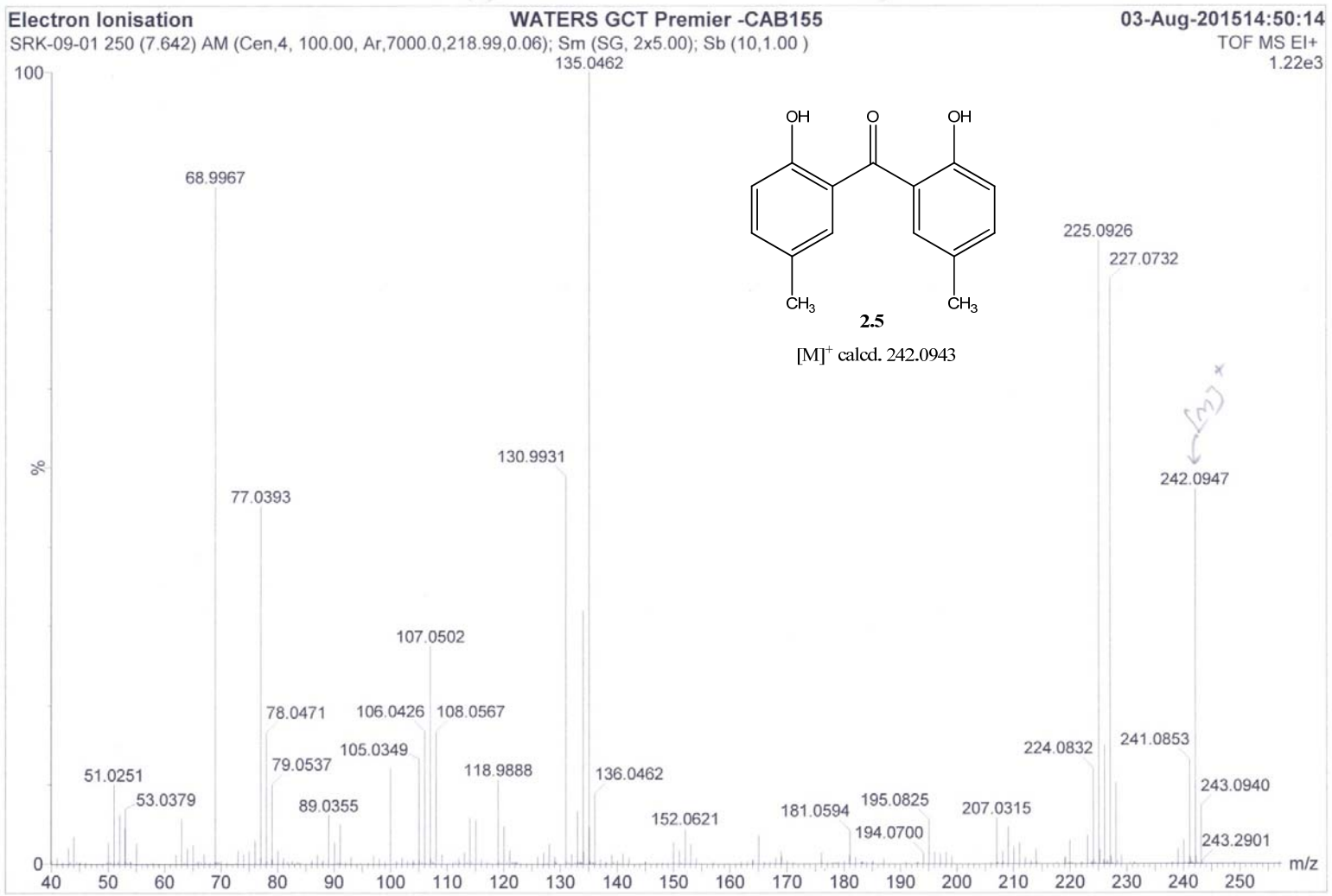
ESI(HRMS) spectrum of bis(4-(diethylamino)-2-hydroxyphenyl)methanone (**2.4**)



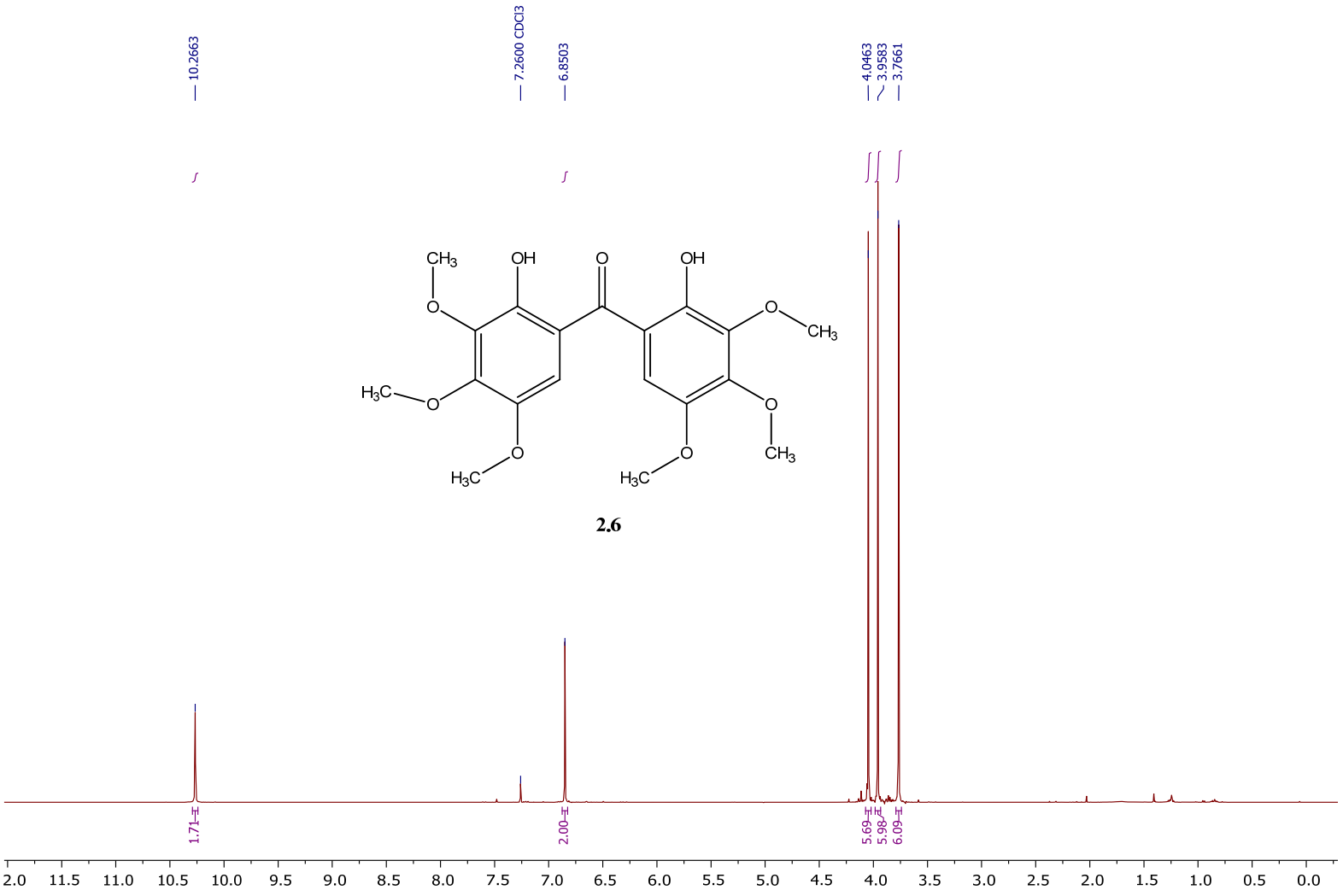
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-5-methylphenyl)methanone (**2.5**)



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-5-methylphenyl)methanone (**2.5**)

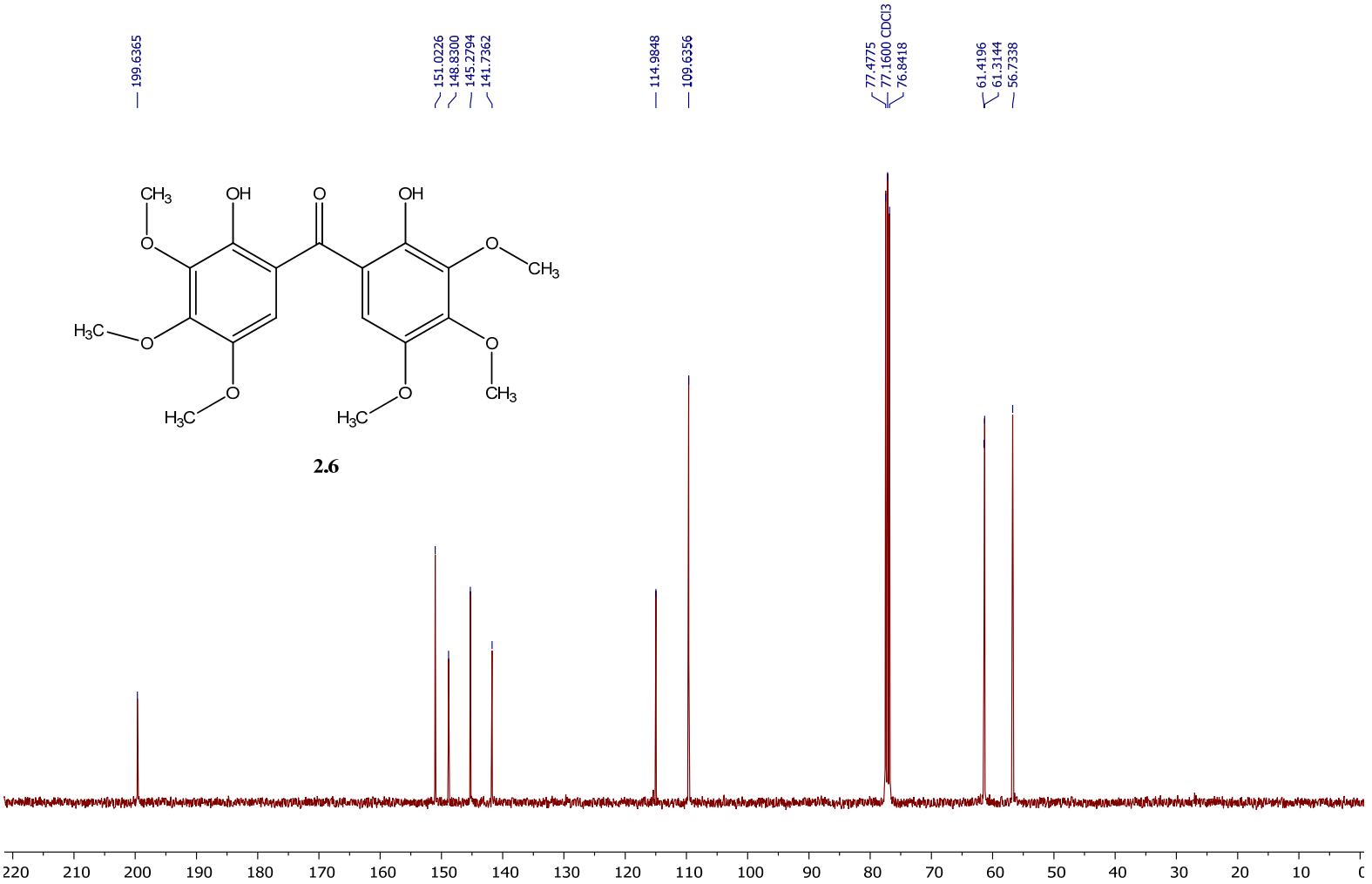


EI(HRMS) spectrum of bis(2-hydroxy-5-methylphenyl)methanone (**2.5**)



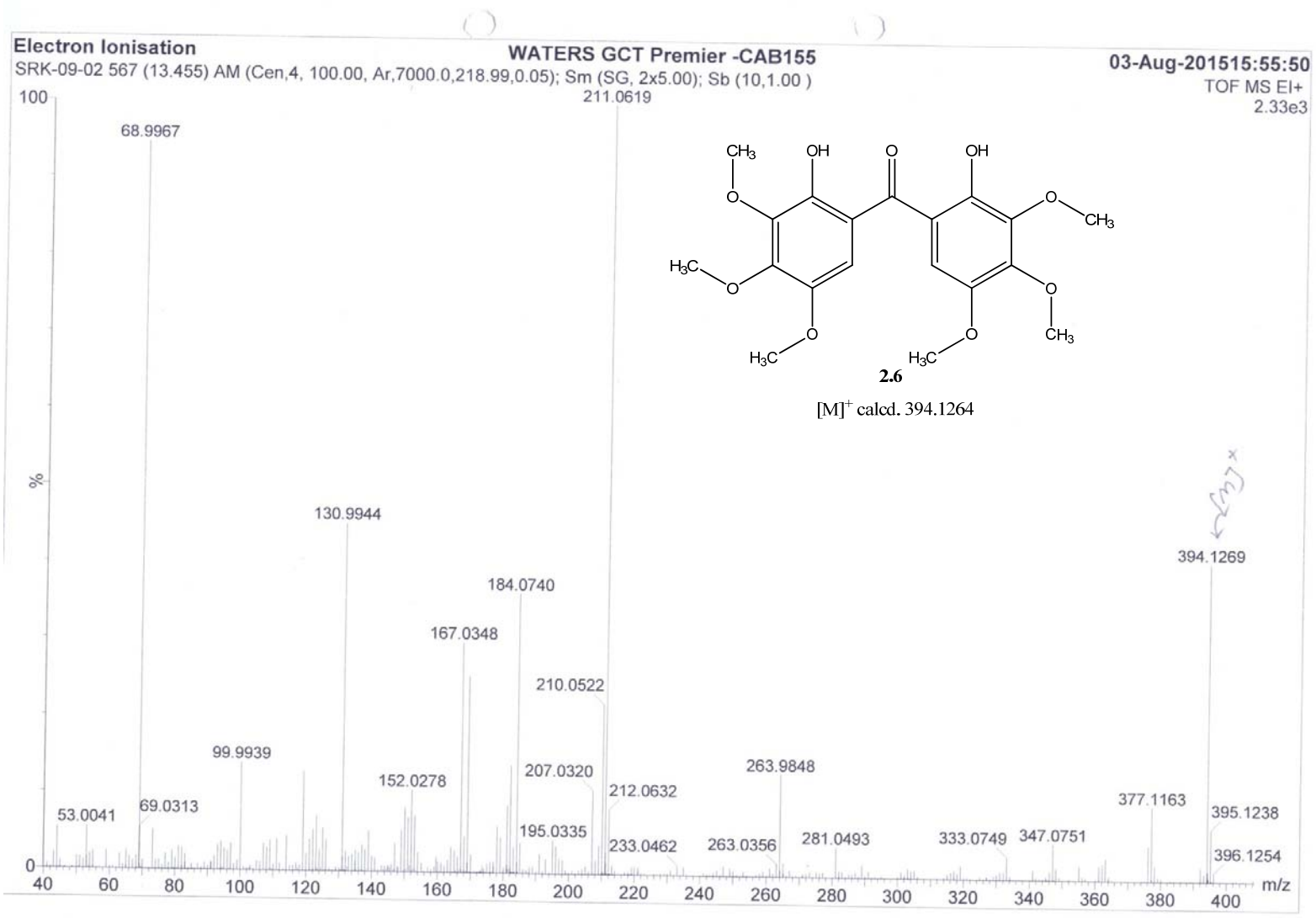
2.6

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-3,4,5-trimethoxyphenyl)methanone (**2.6**)

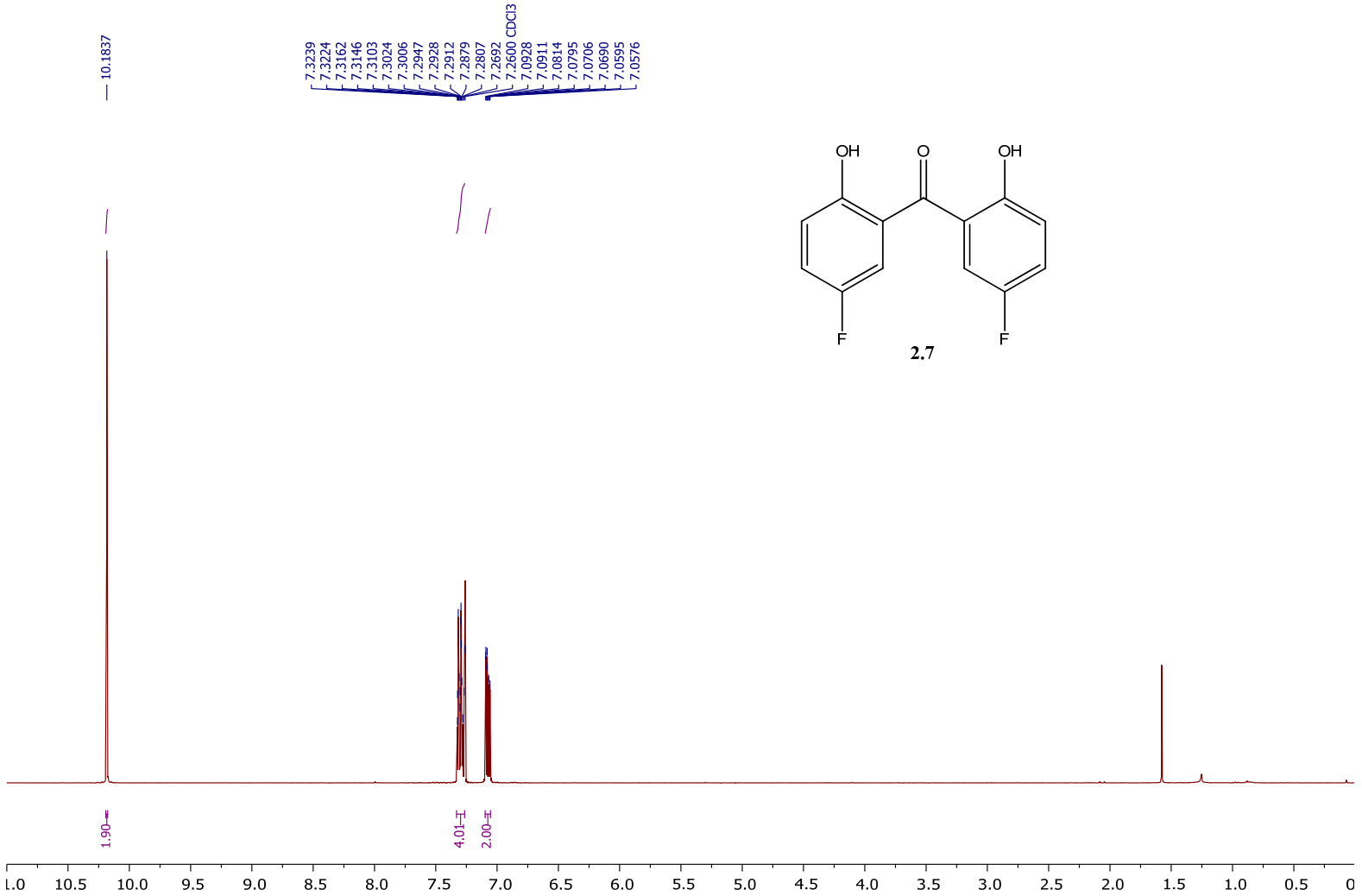


2.6

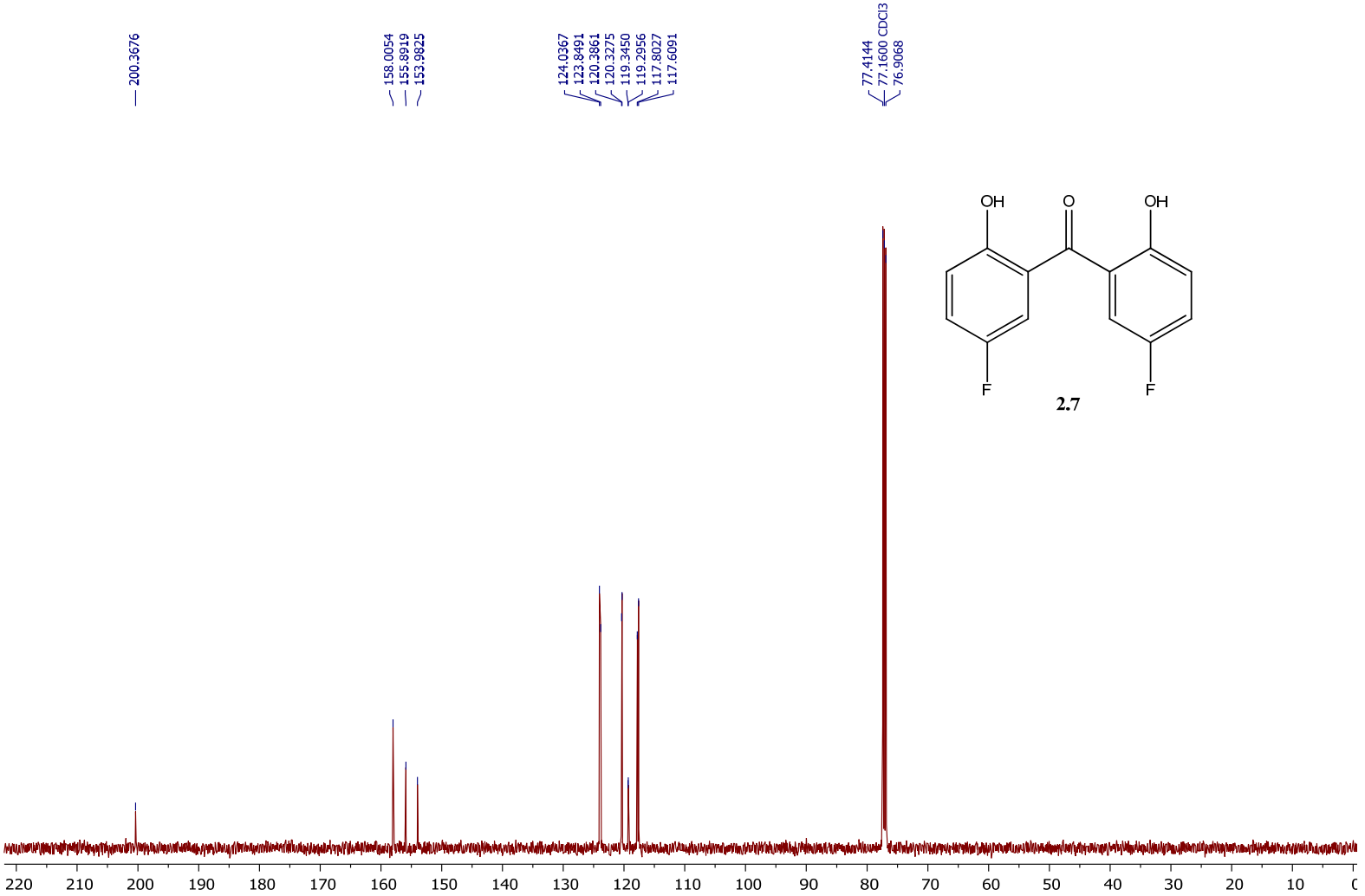
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of bis(2-hydroxy-3,4,5-trimethoxyphenyl)methanone (**2.6**)



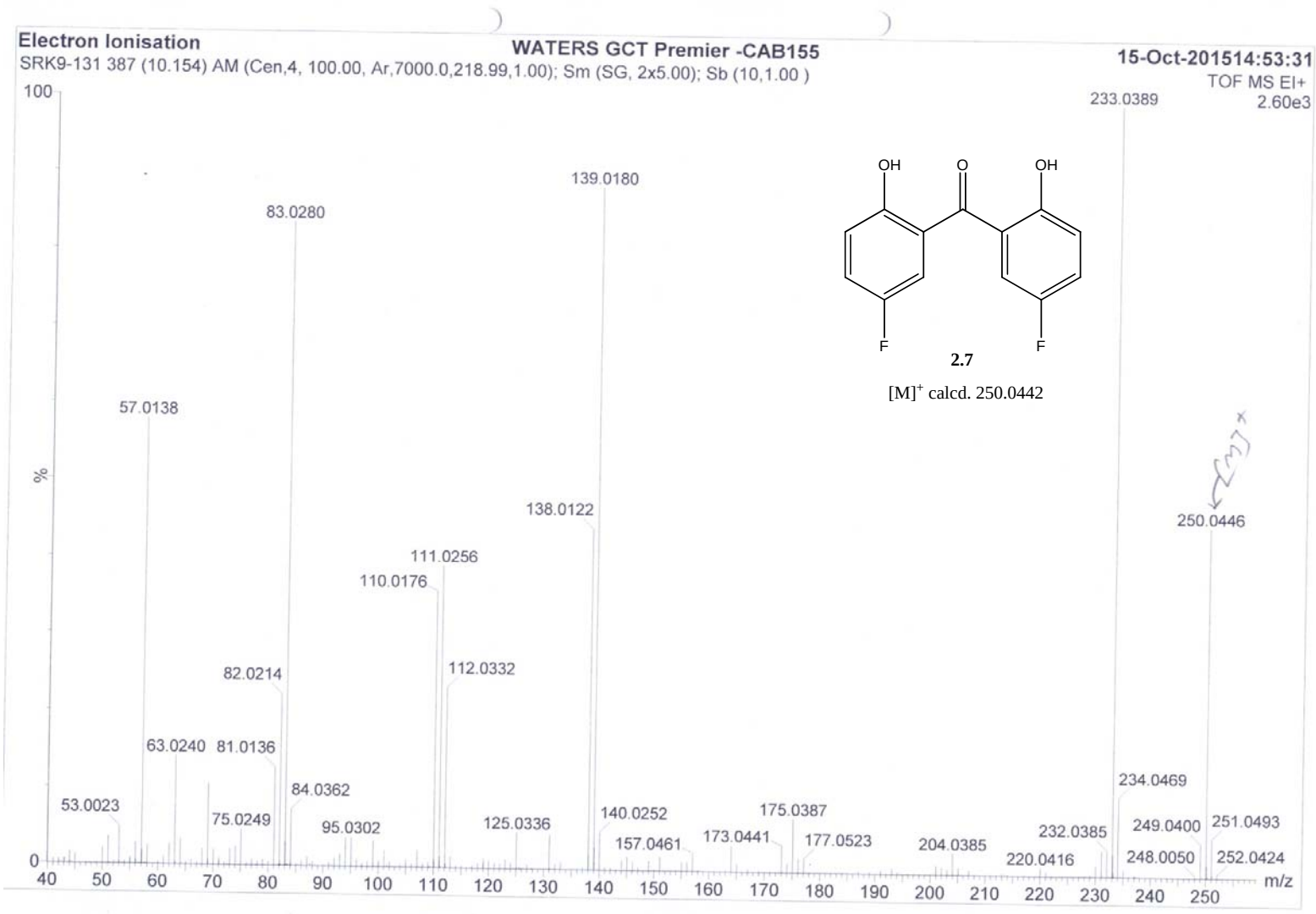
EI(HRMS) spectrum of bis(2-hydroxy-3,4,5-trimethoxyphenyl)methanone (**2.6**)



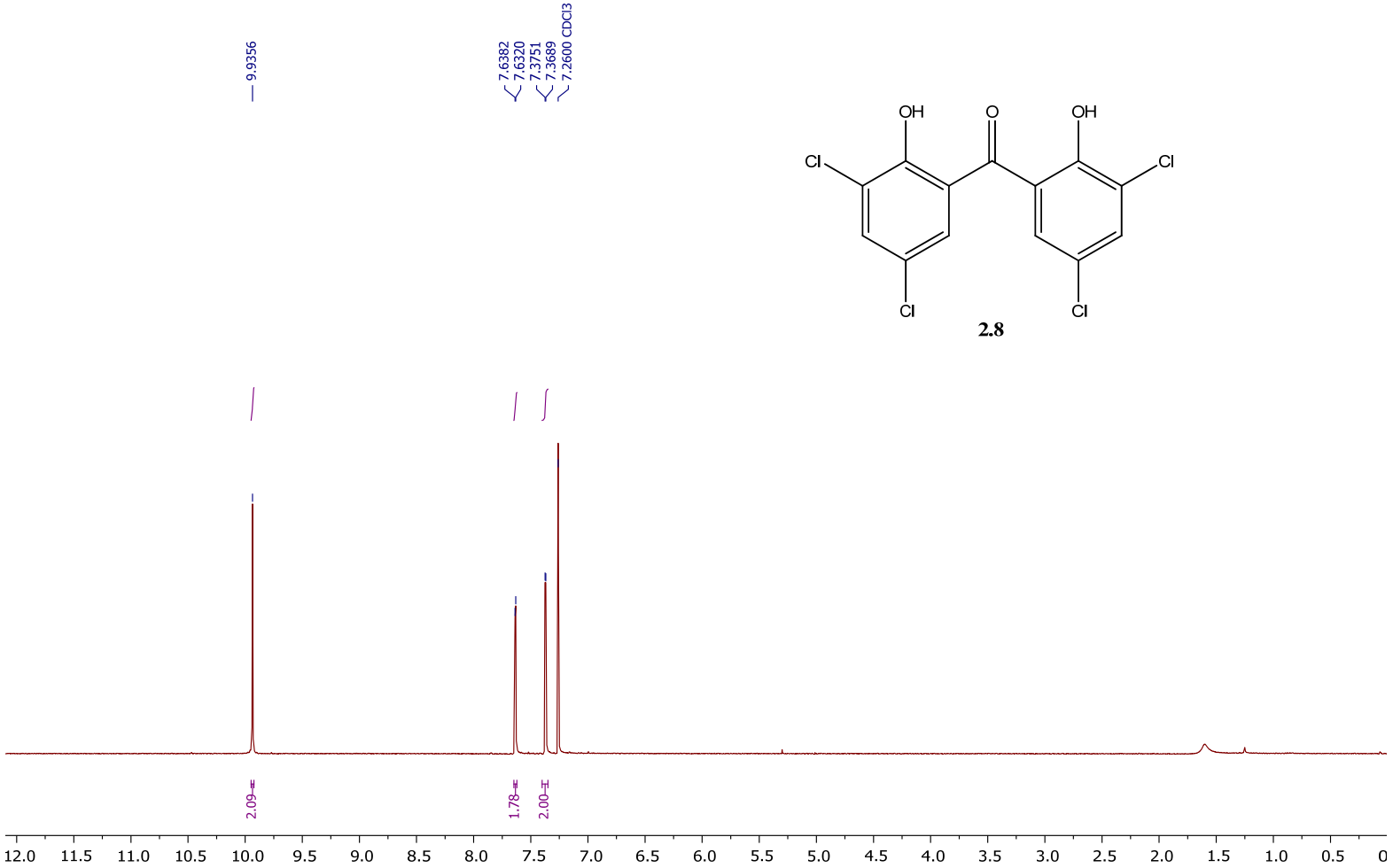
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(5-fluoro-2-hydroxyphenyl)methanone (**2.7**)



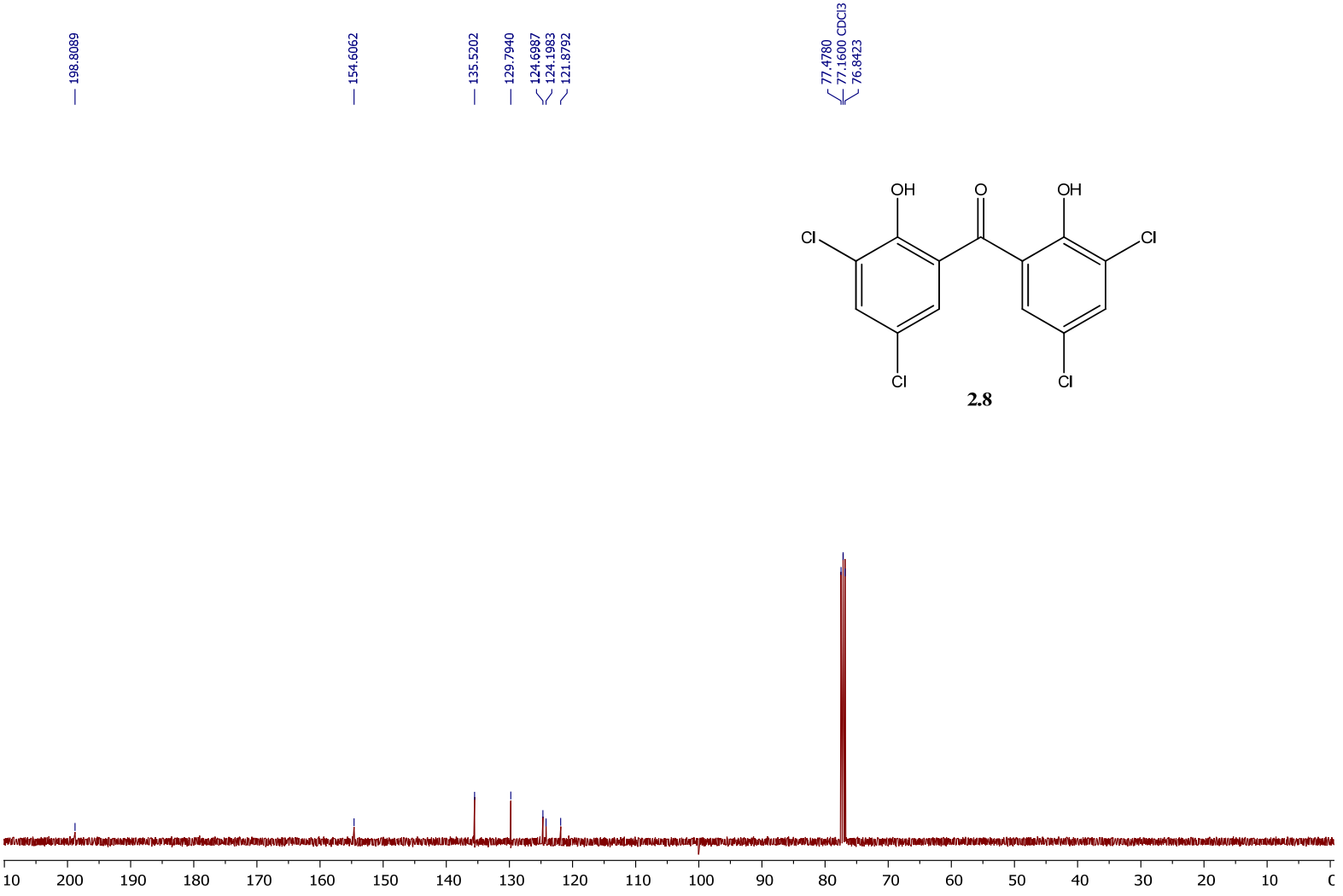
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of bis(5-fluoro-2-hydroxyphenyl)methanone (**2.7**)



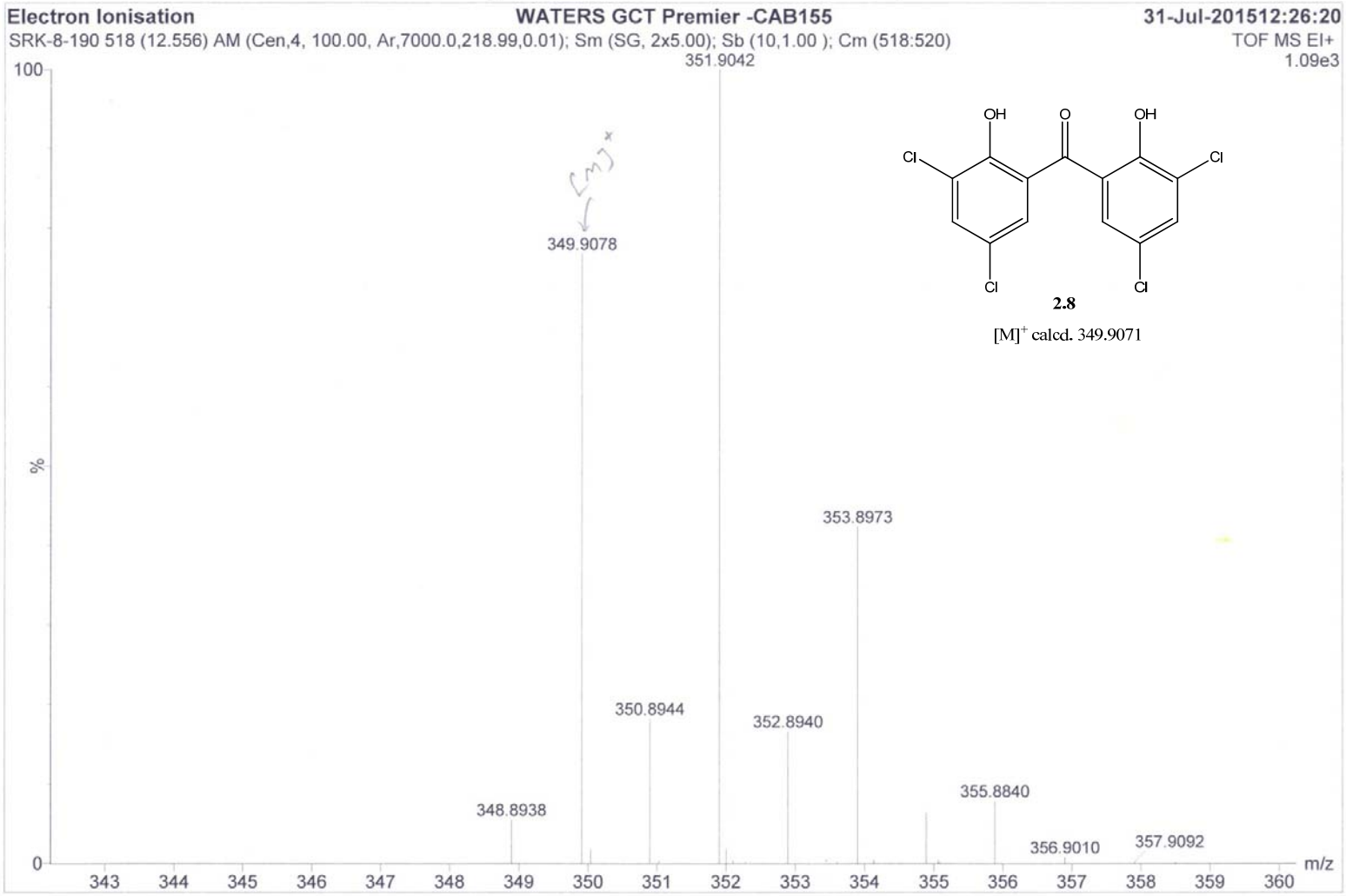
EI(HRMS) spectrum of bis(5-fluoro-2-hydroxyphenyl)methanone (**2.7**)



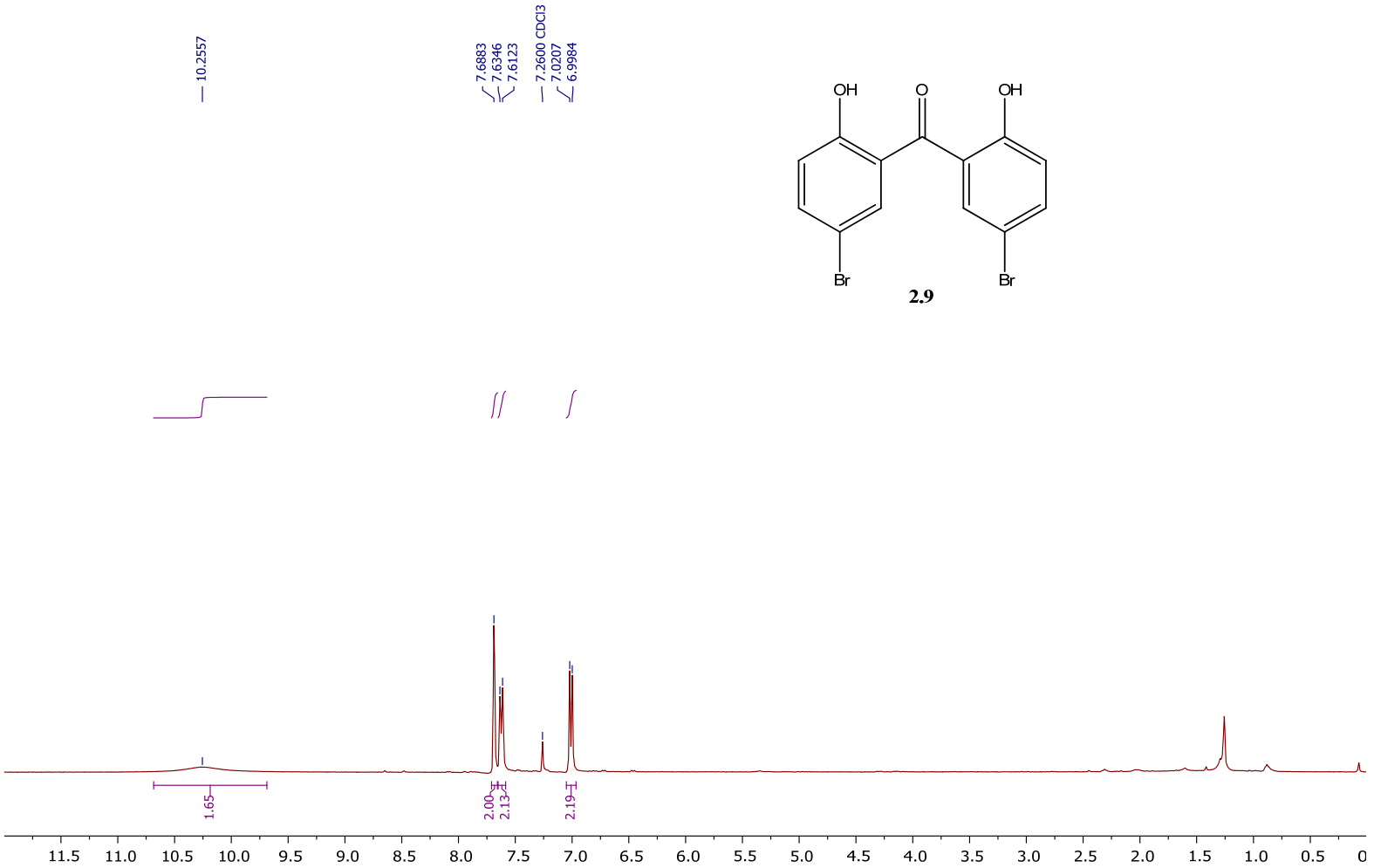
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(3,5-dichloro-2-hydroxyphenyl)methanone (**2.8**)



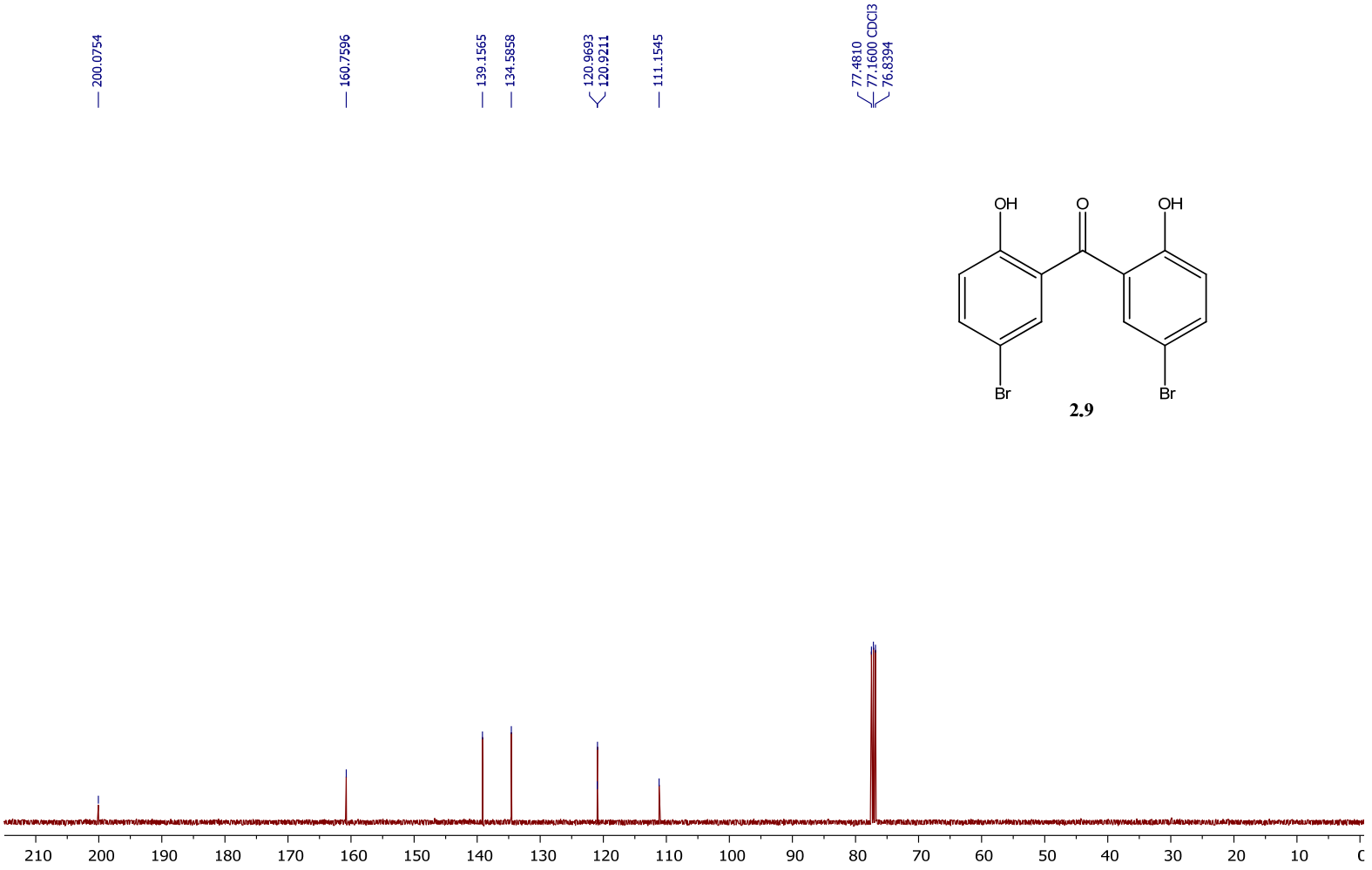
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of bis(3,5-dichloro-2-hydroxyphenyl)methanone (**2.8**)



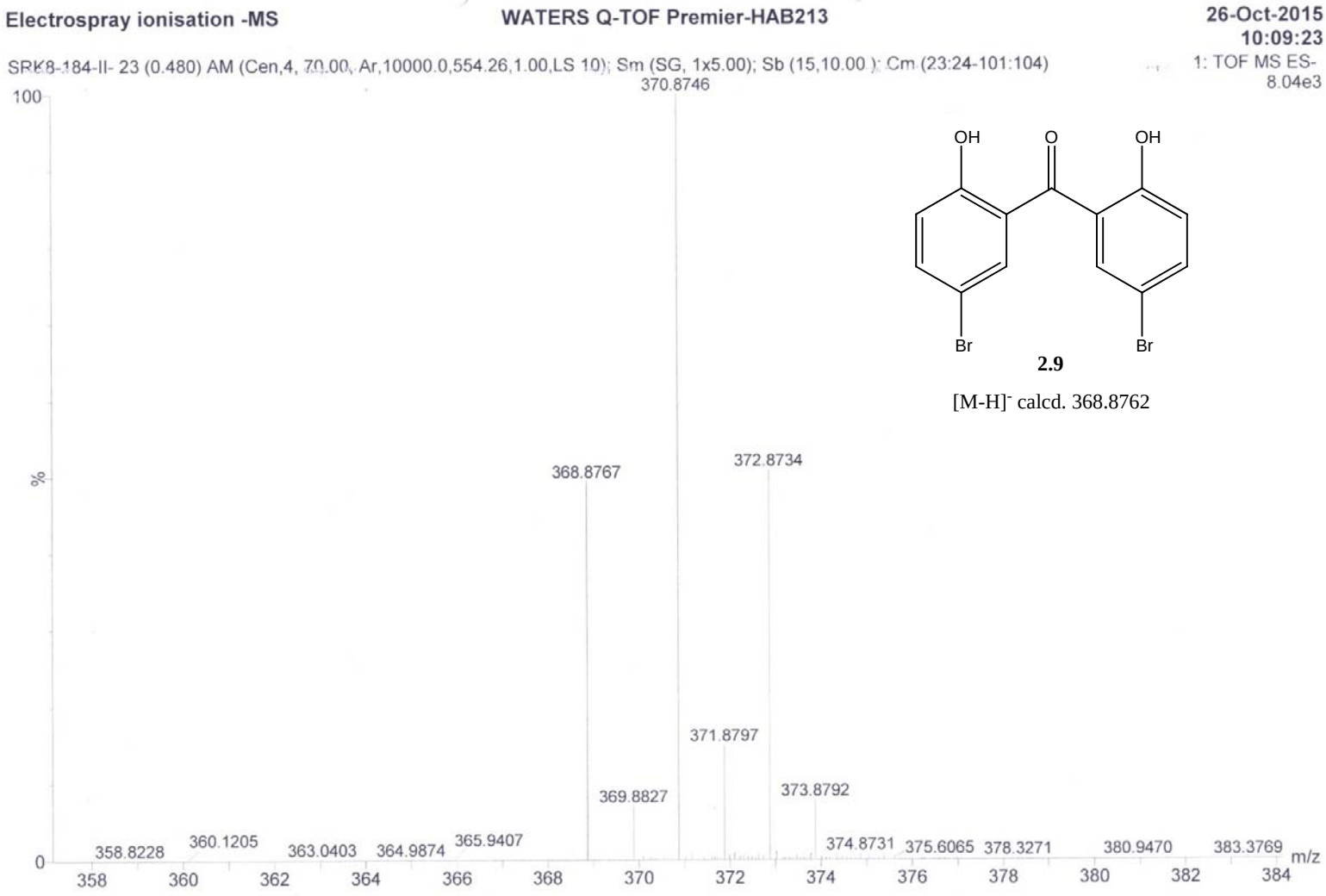
EI(HRMS) spectrum of bis(3,5-dichloro-2-hydroxyphenyl)methanone (**2.8**)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(5-bromo-2-hydroxyphenyl)methanone (**2.9**)



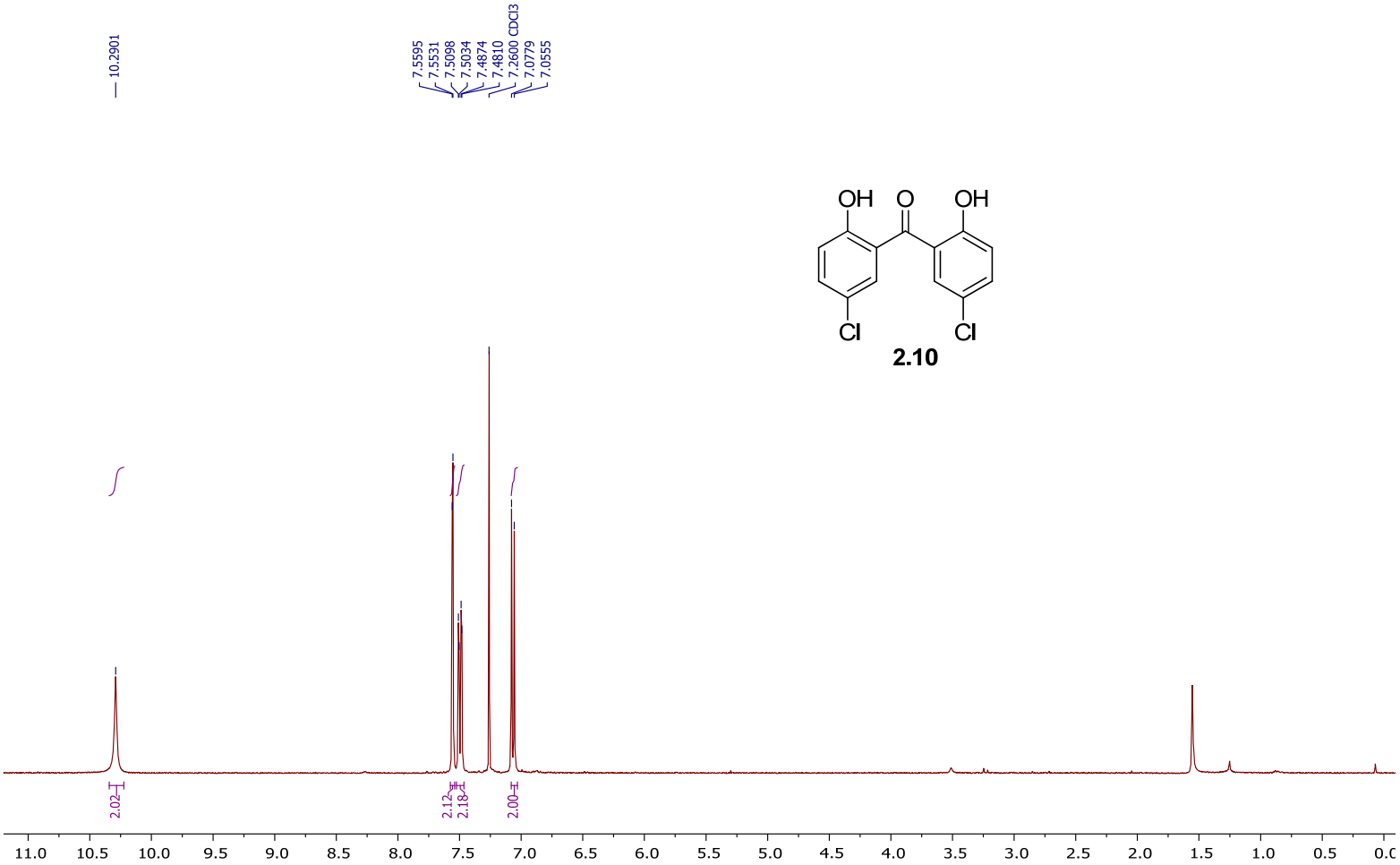
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of bis(5-bromo-2-hydroxyphenyl)methanone (**2.9**)



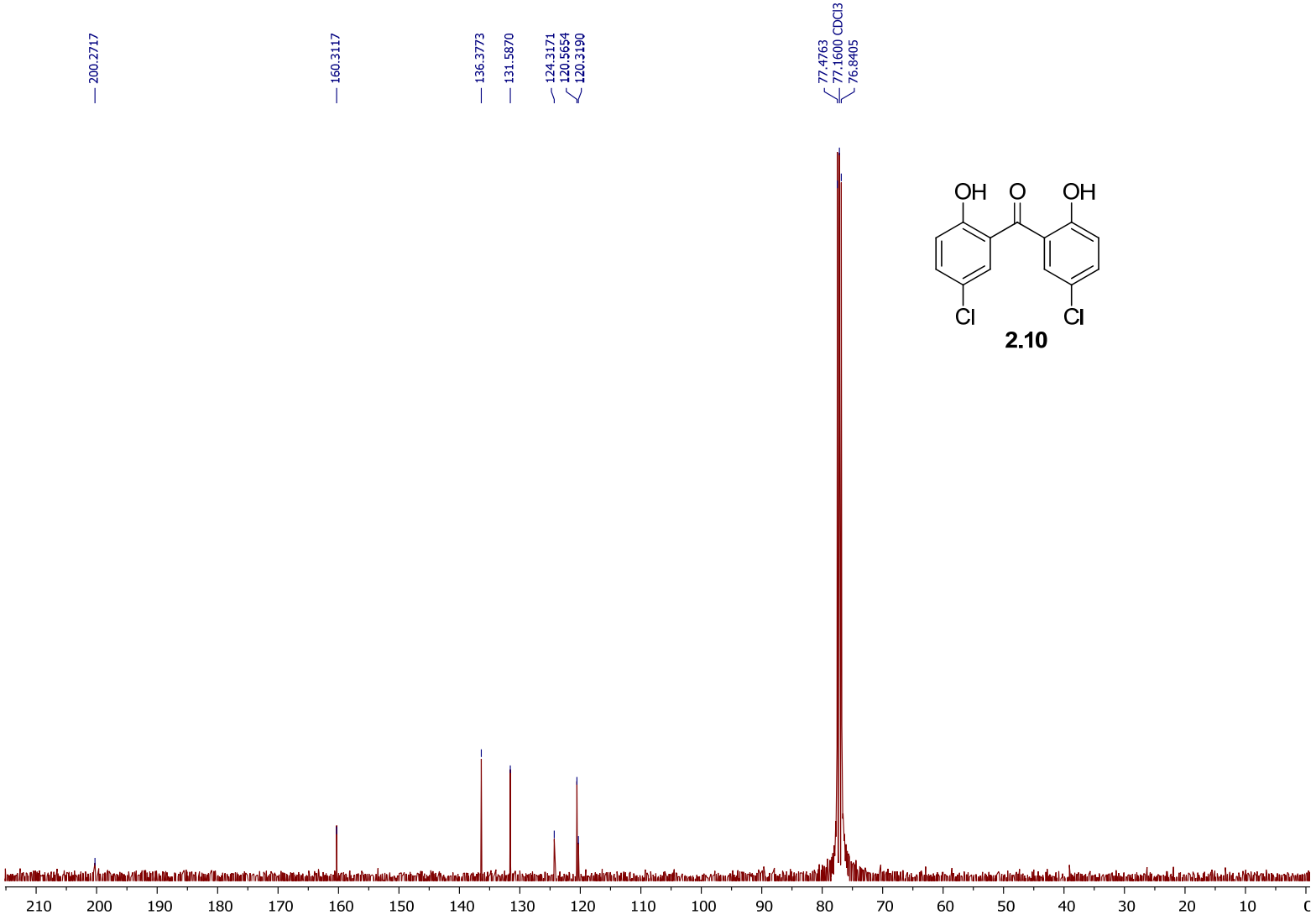
2.9

$[M-H]^-$ calcd. 368.8762

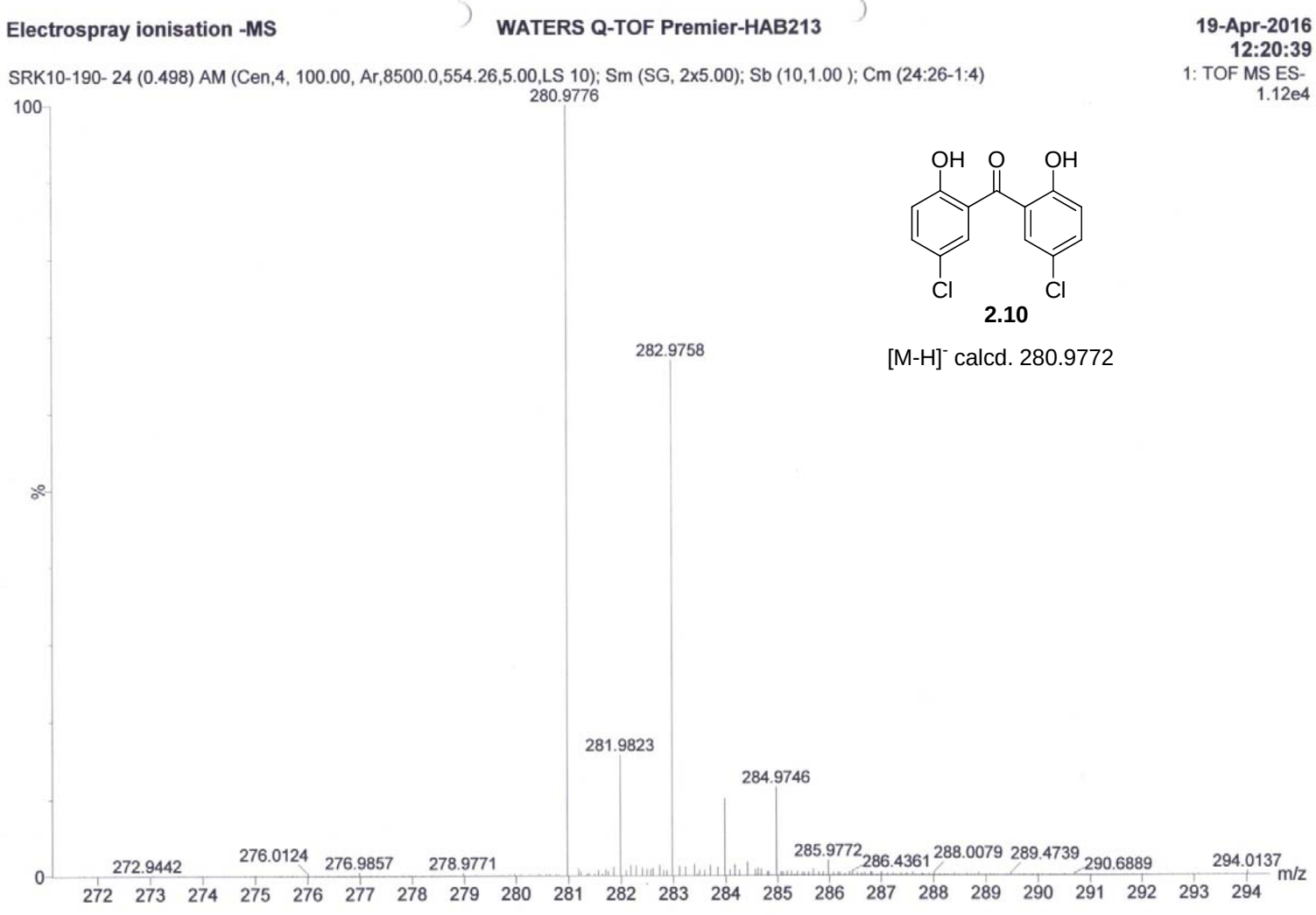
ESI(HRMS) spectrum of bis(5-bromo-2-hydroxyphenyl)methanone (**2.9**)



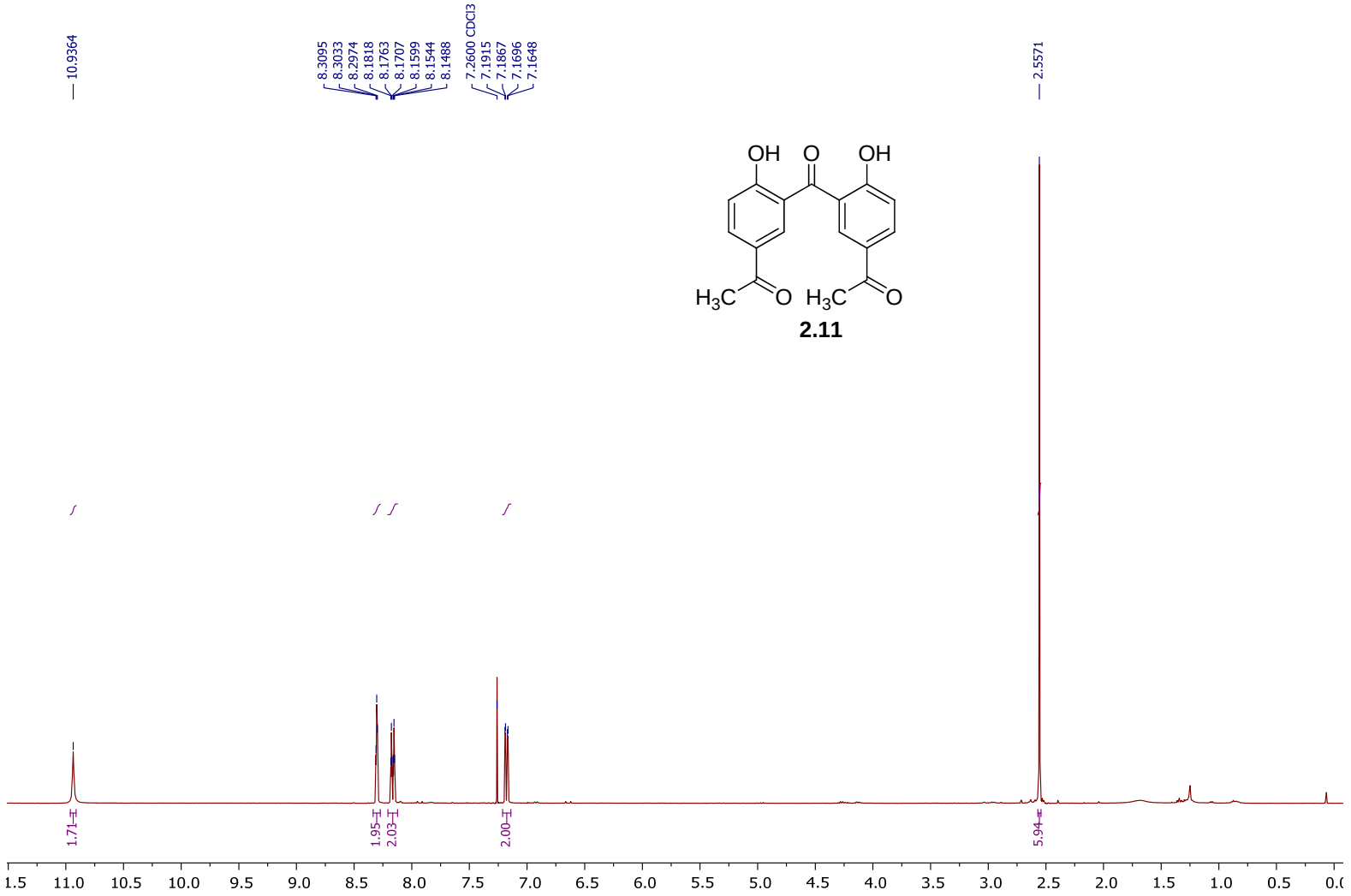
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of bis(5-chloro-2-hydroxyphenyl)methanone (**2.10**)



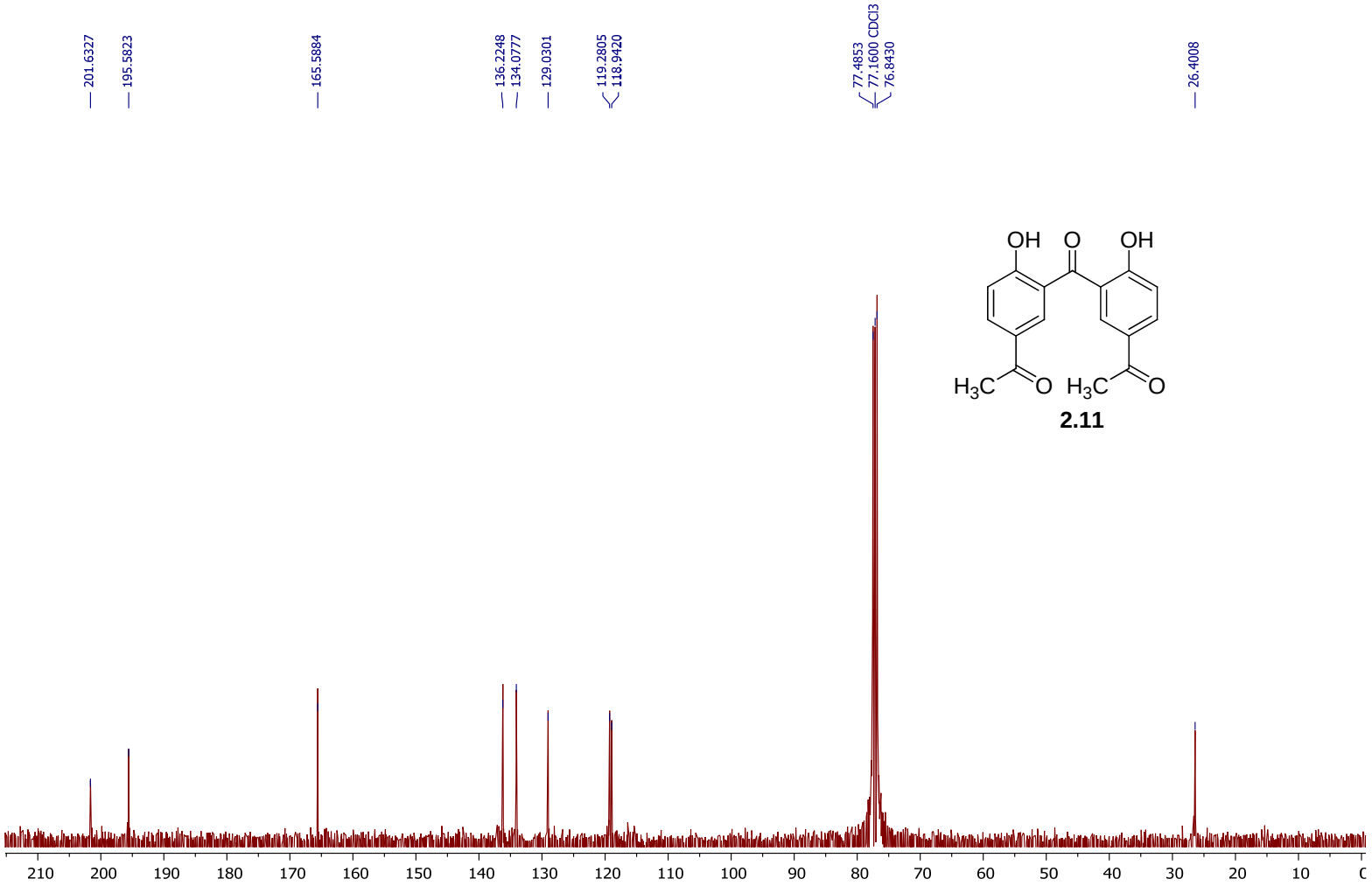
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of bis(5-chloro-2-hydroxyphenyl)methanone (**2.10**)



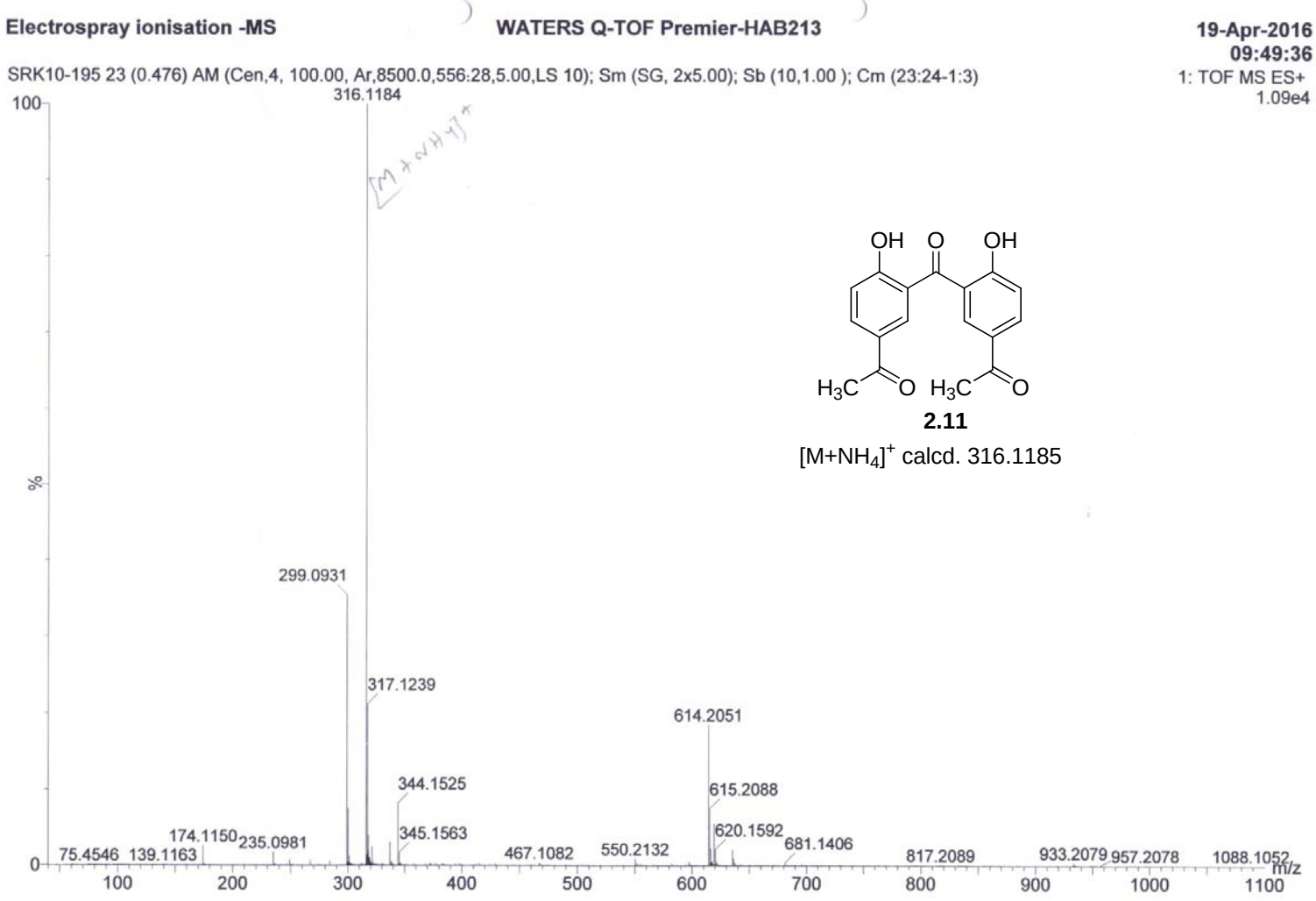
ESI(HRMS) spectrum of bis(5-chloro-2-hydroxyphenyl)methanone (**2.10**)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1'-(3,3'-carbonylbis(4-hydroxy-3,1-phenylene))diethanone (**2.11**)

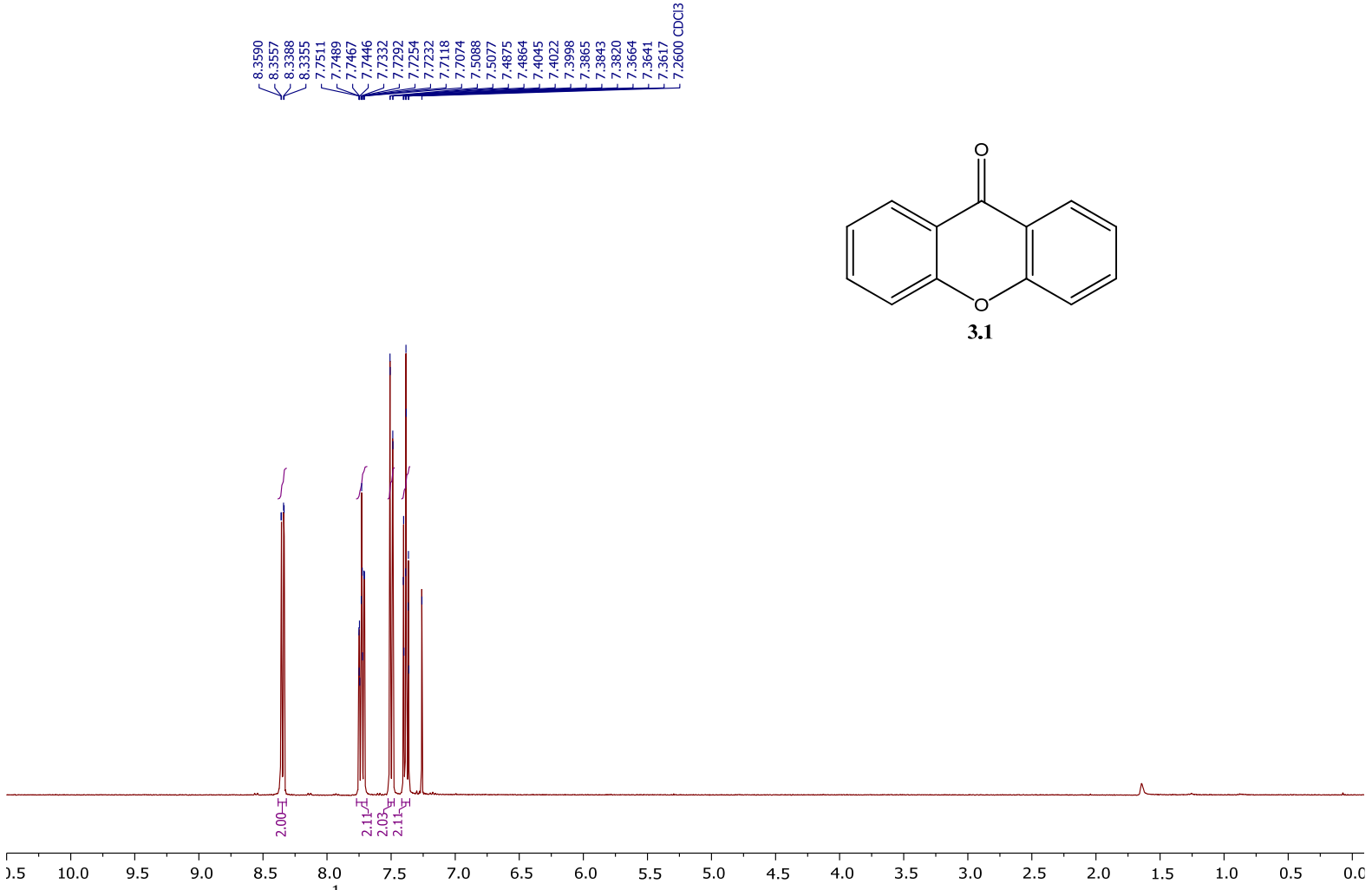


^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 1,1'-(3,3'-carbonylbis(4-hydroxy-3,1-phenylene))diethanone (**2.11**)

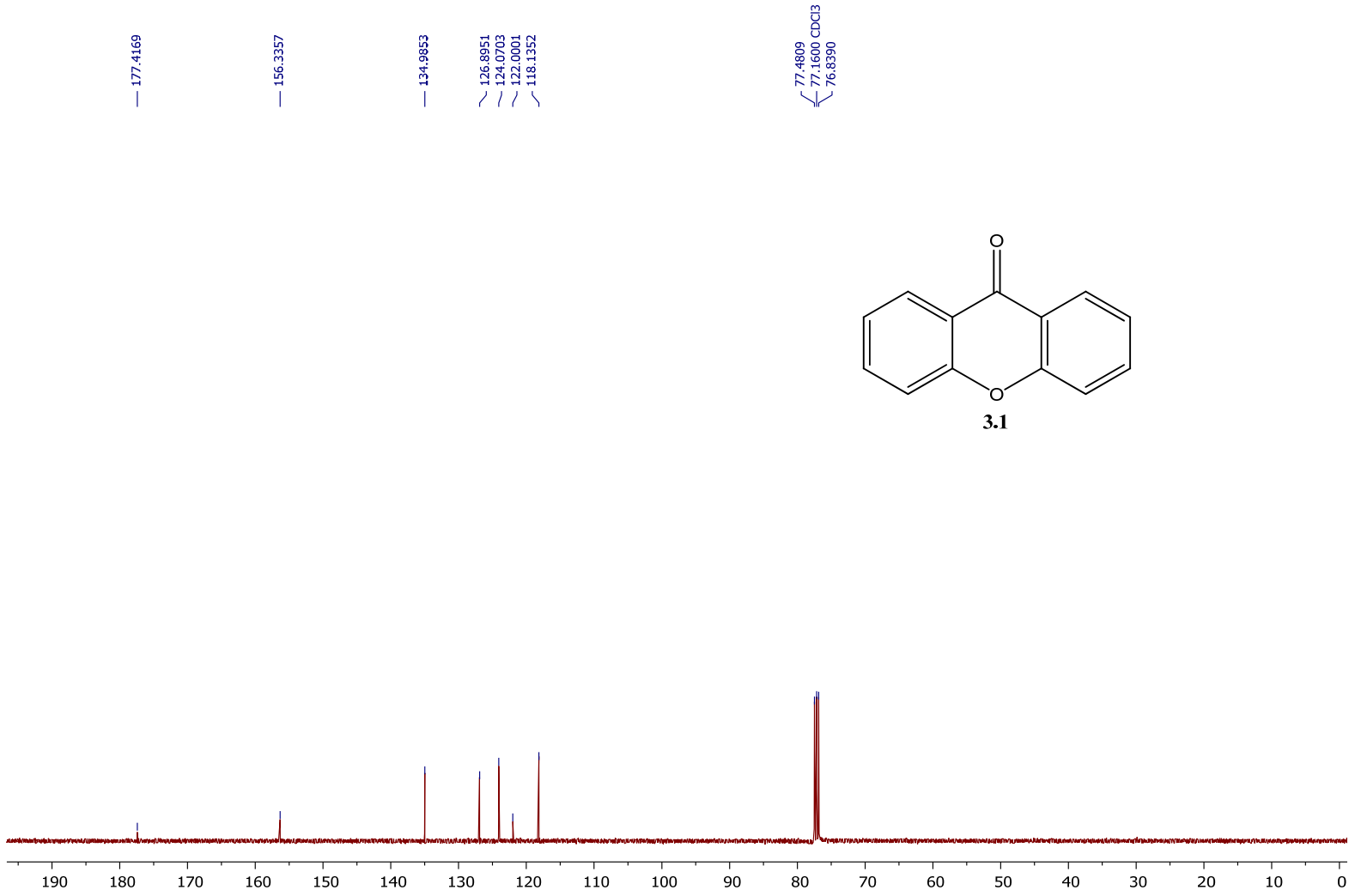


ESI(HRMS) spectrum of 1,1'-(3,3'-carbonylbis(4-hydroxy-3,1-phenylene))diethanone (**2.11**)

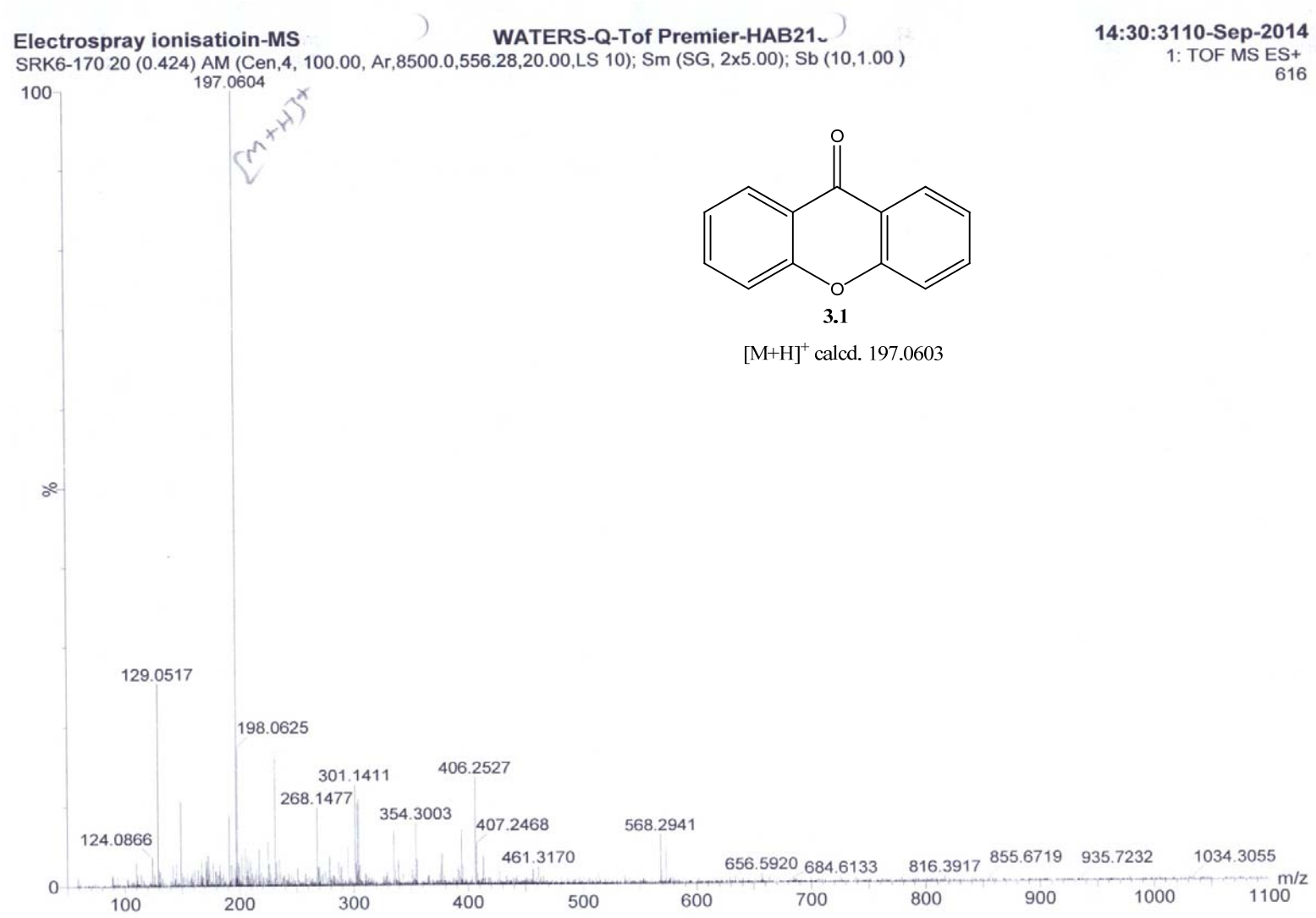
3. Copies of ^{1}H NMR, ^{13}C NMR and HRMS spectra of Compound 3.1 to 3.10 (Table 3):



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 9*H*-xanthen-9-one (**3.1**)



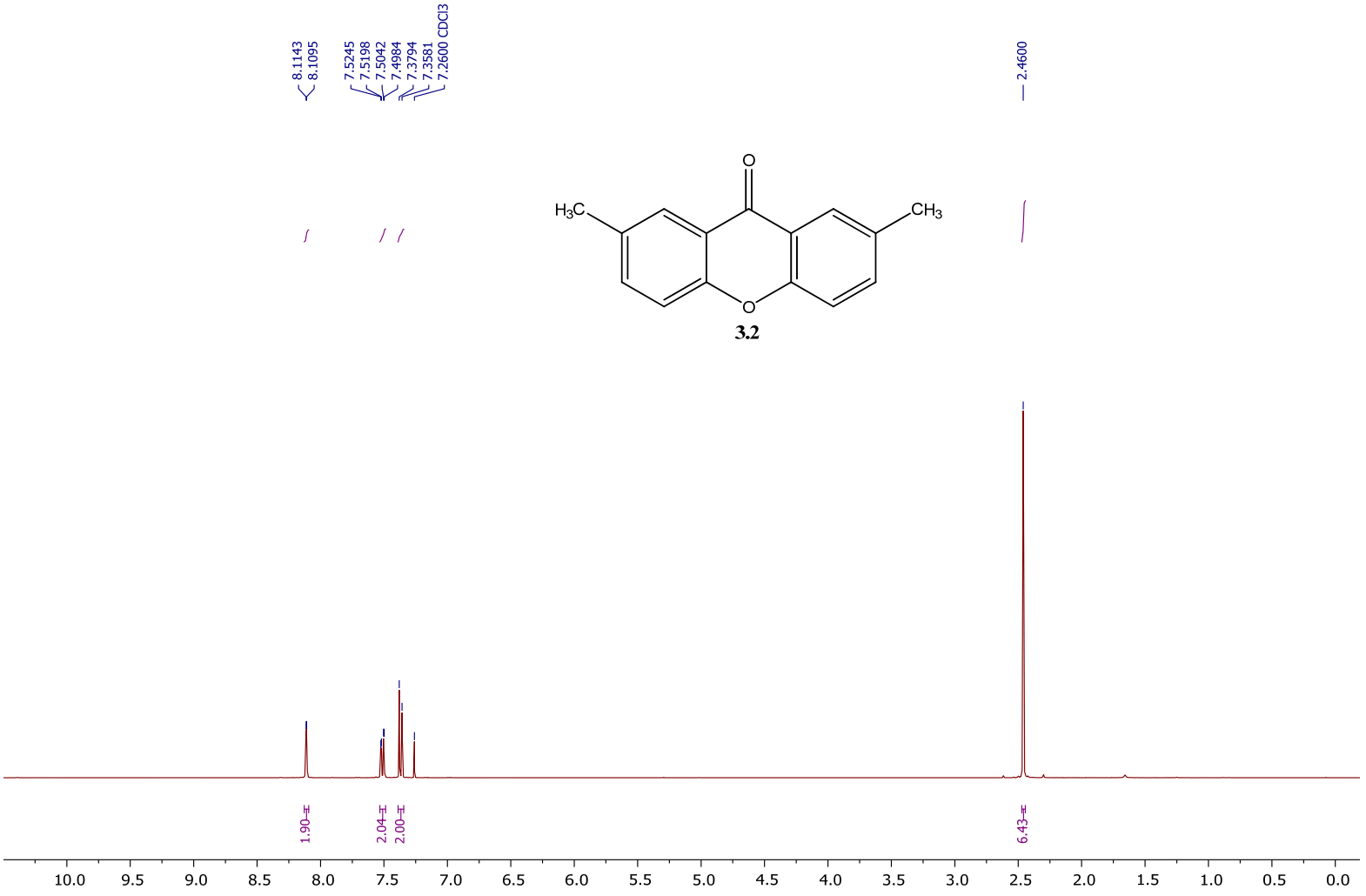
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 9*H*-xanthen-9-one (**3.1**)



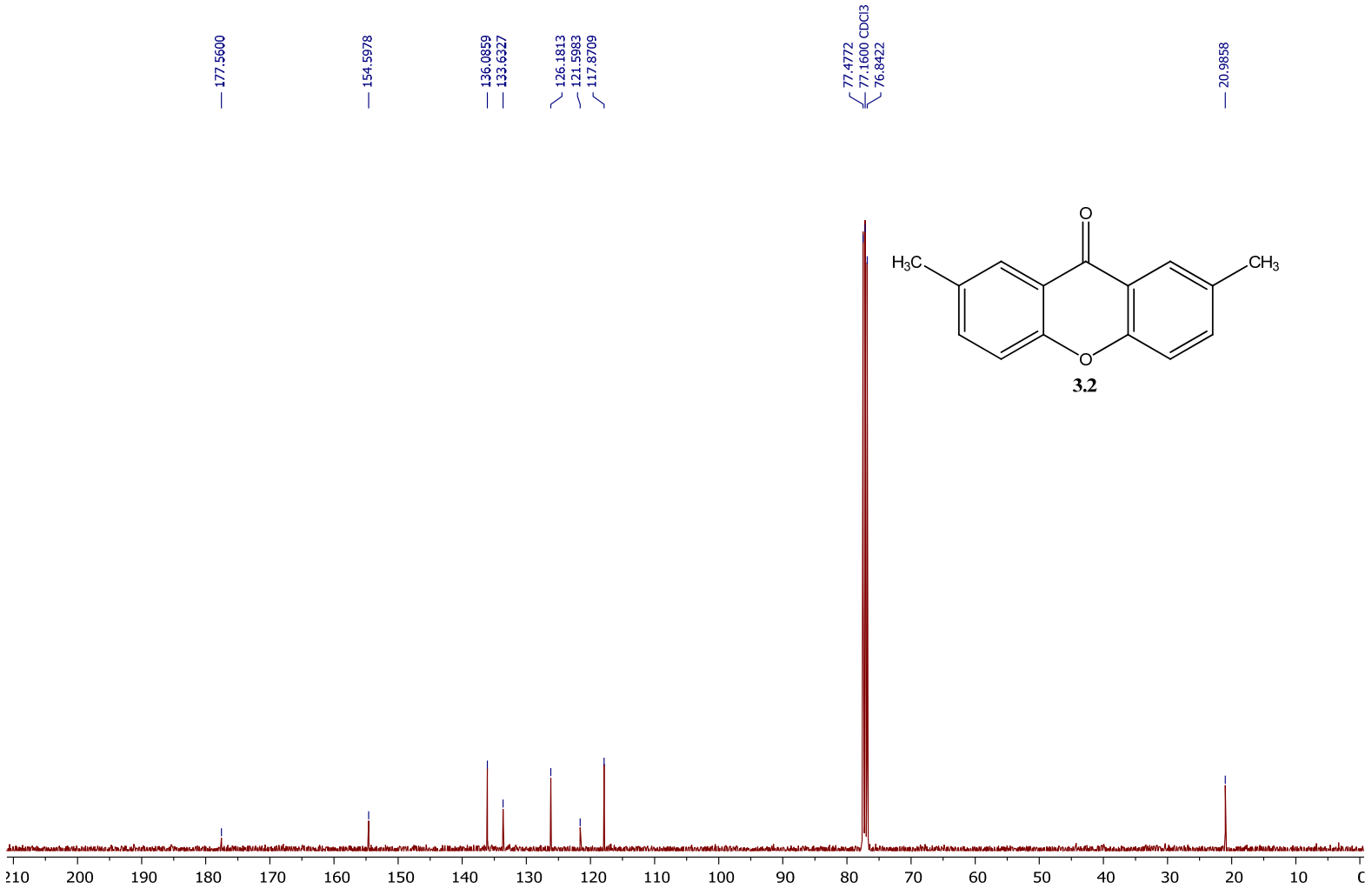
3.1

$[M+H]^+$ calcd. 197.0603

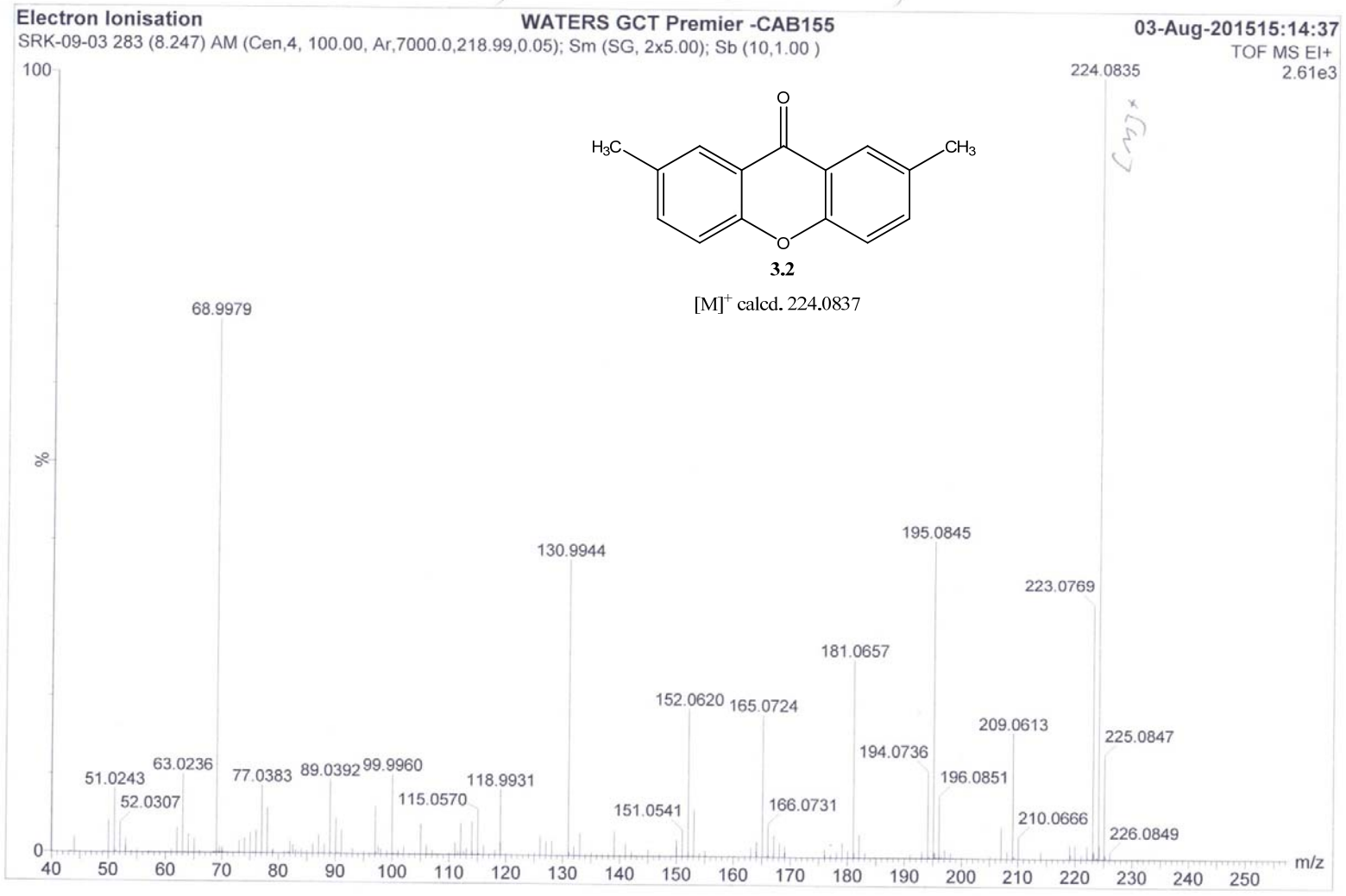
ESI(HRMS) spectrum of 9*H*-xanthen-9-one (**3.1**)



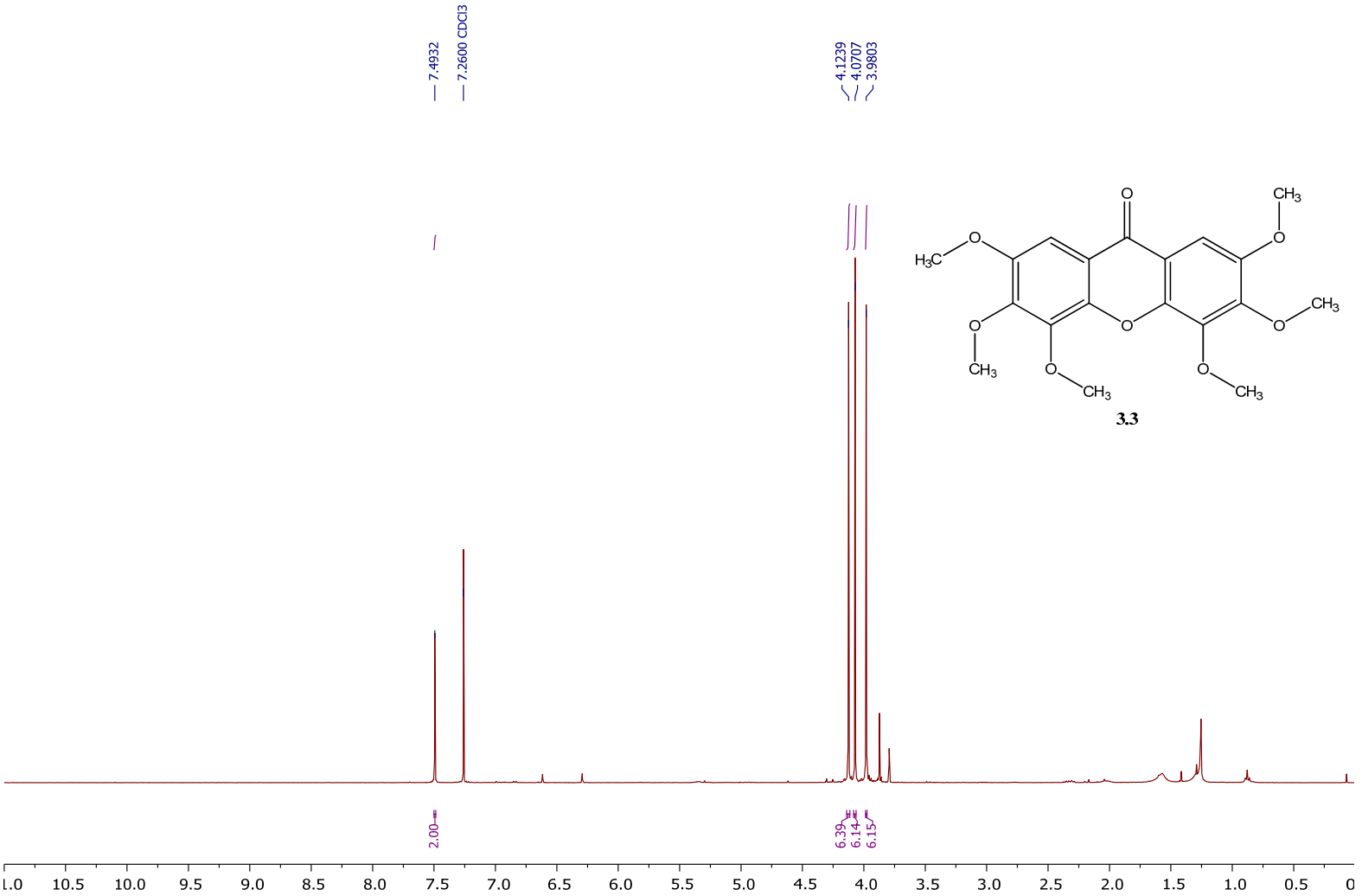
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2,7-dimethyl-9*H*-xanthen-9-one (**3.2**)



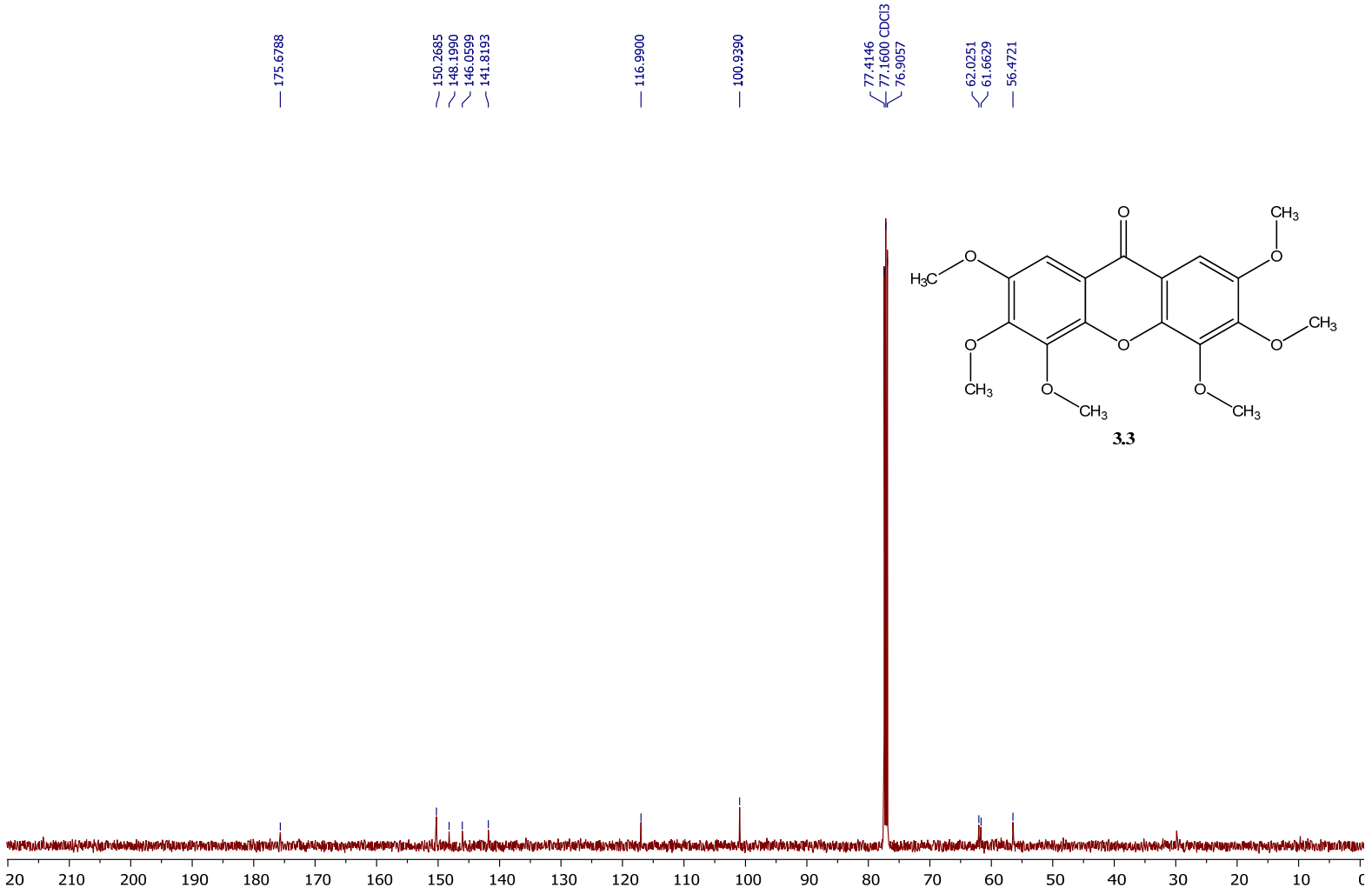
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2,7-dimethyl-9*H*-xanthen-9-one (**3.2**)



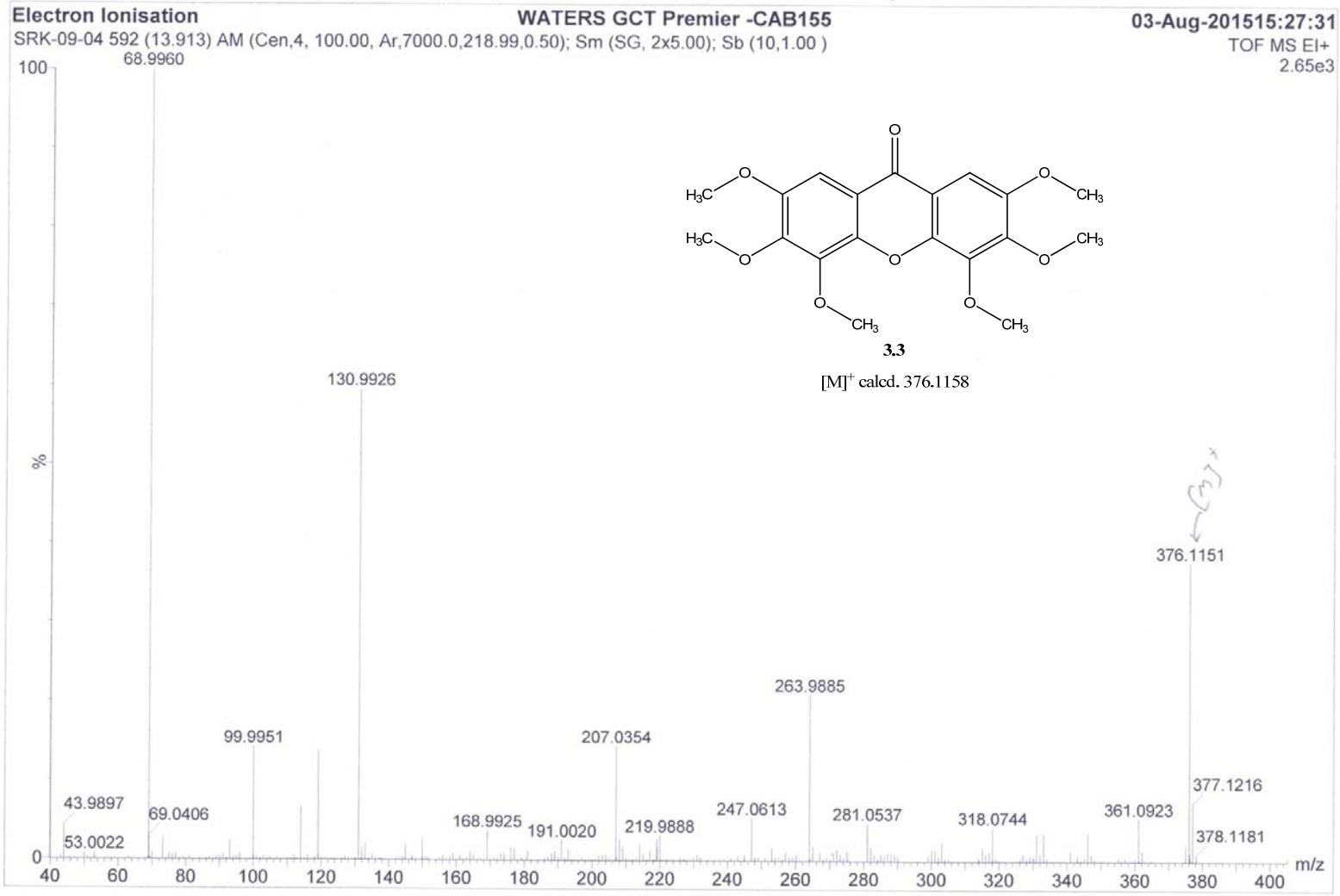
EI(HRMS) spectrum of 2,7-dimethyl-9*H*-xanthen-9-one (**3.2**)



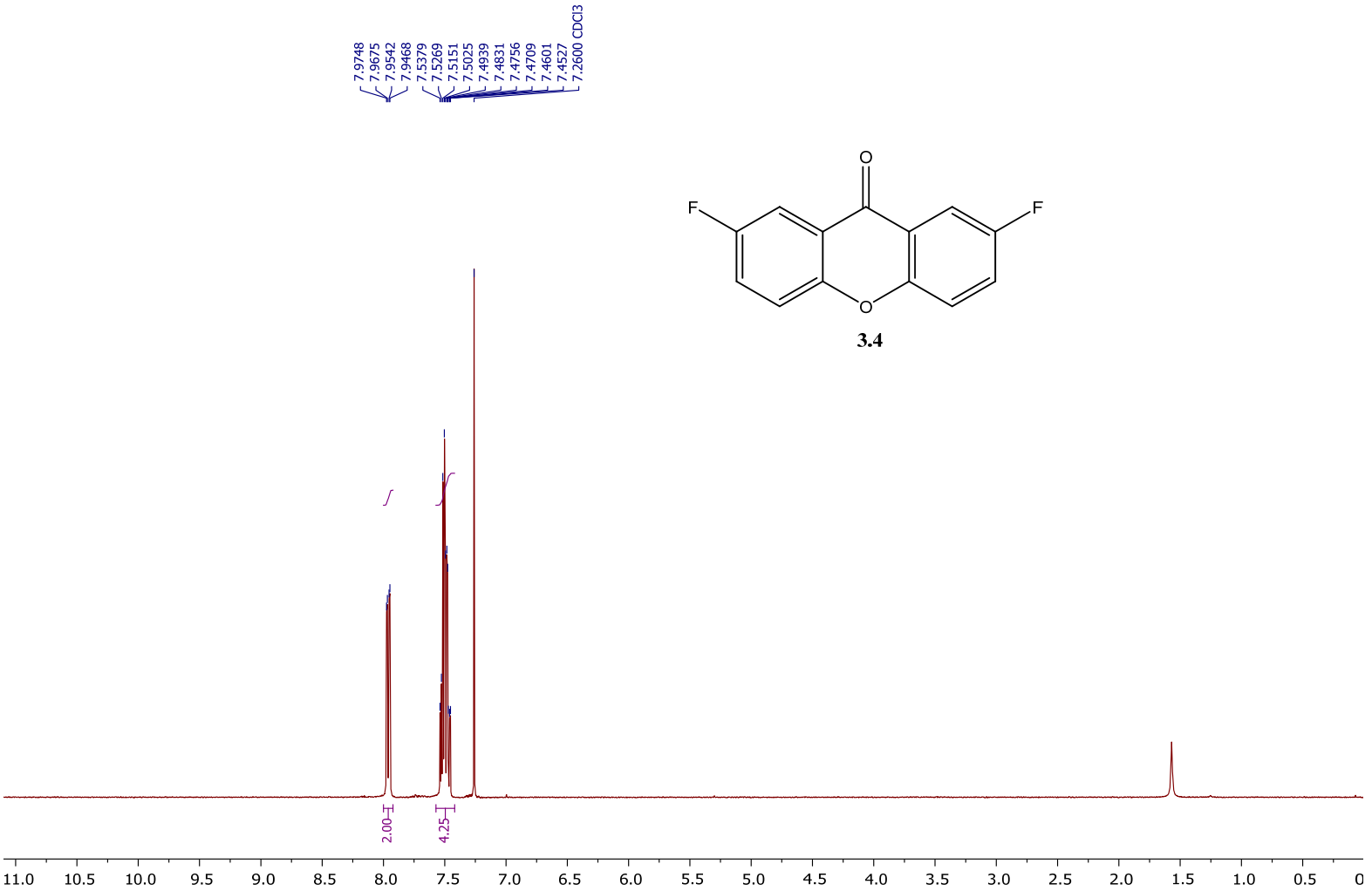
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2,3,4,5,6,7-hexamethoxy-9*H*-xanthen-9-one (**3.3**)



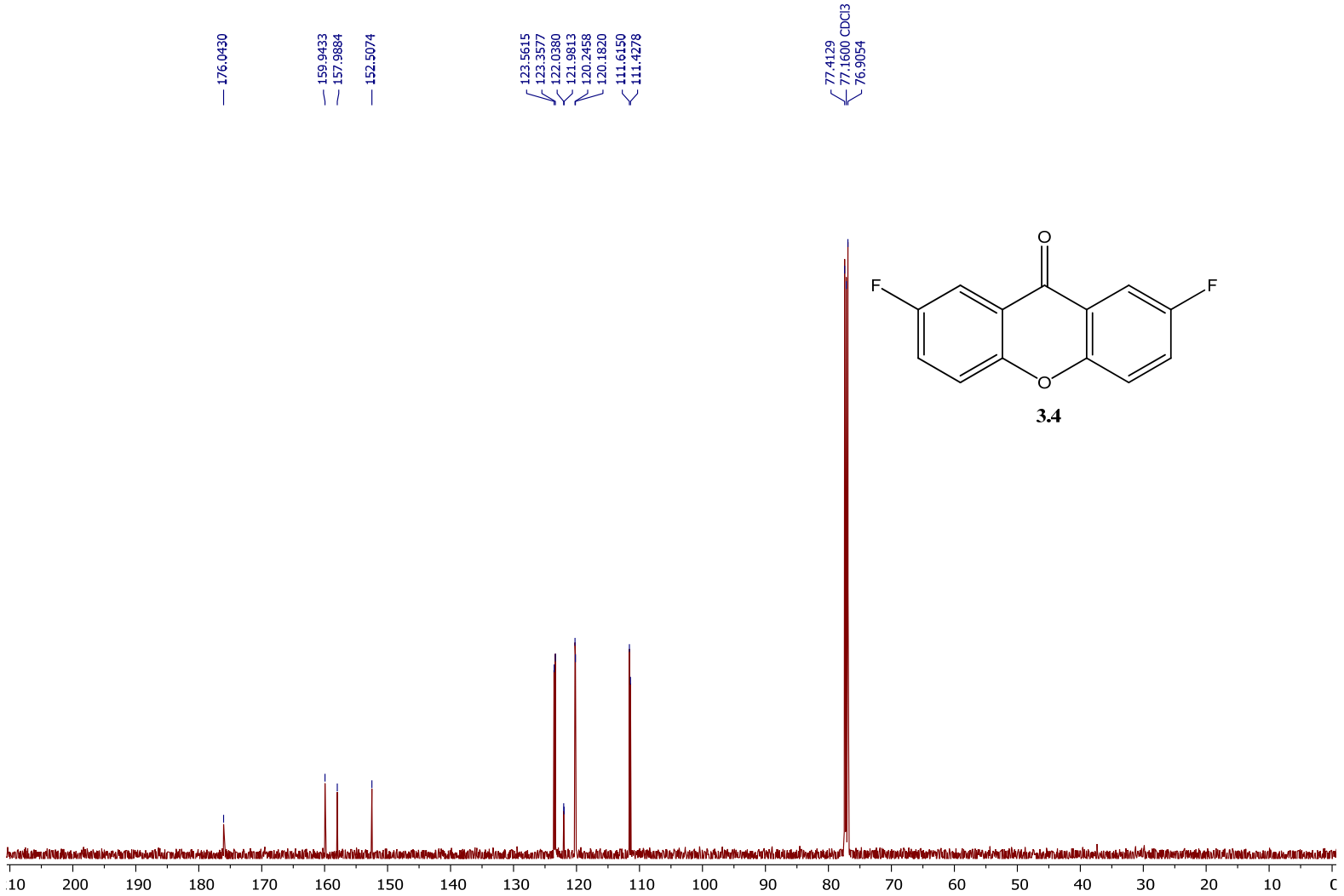
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of 2,3,4,5,6,7-hexamethoxy-9*H*-xanthen-9-one (**3.3**)



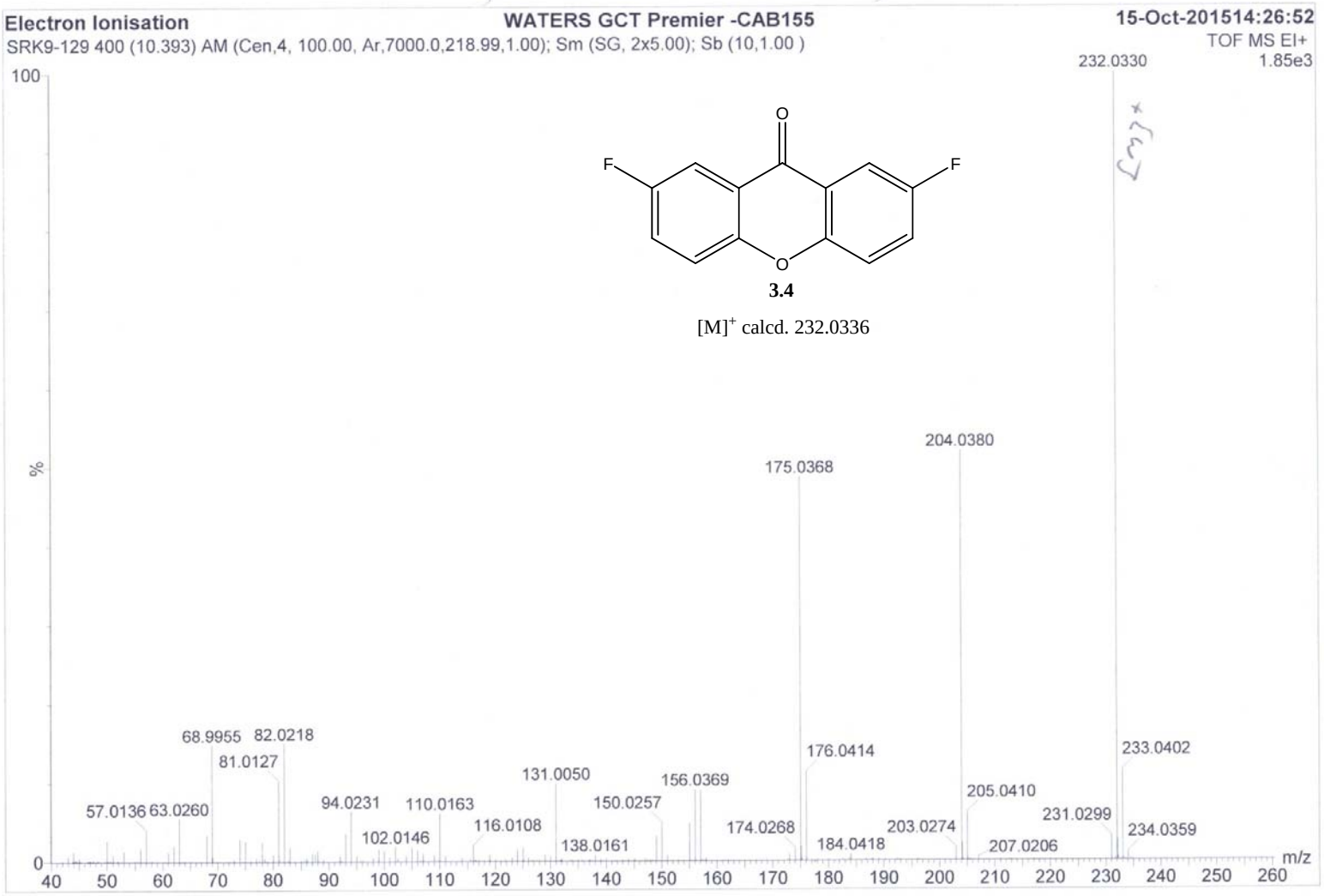
EI(HRMS) spectrum of 2,3,4,5,6,7-hexamethoxy-9*H*-xanthen-9-one (**3.3**)



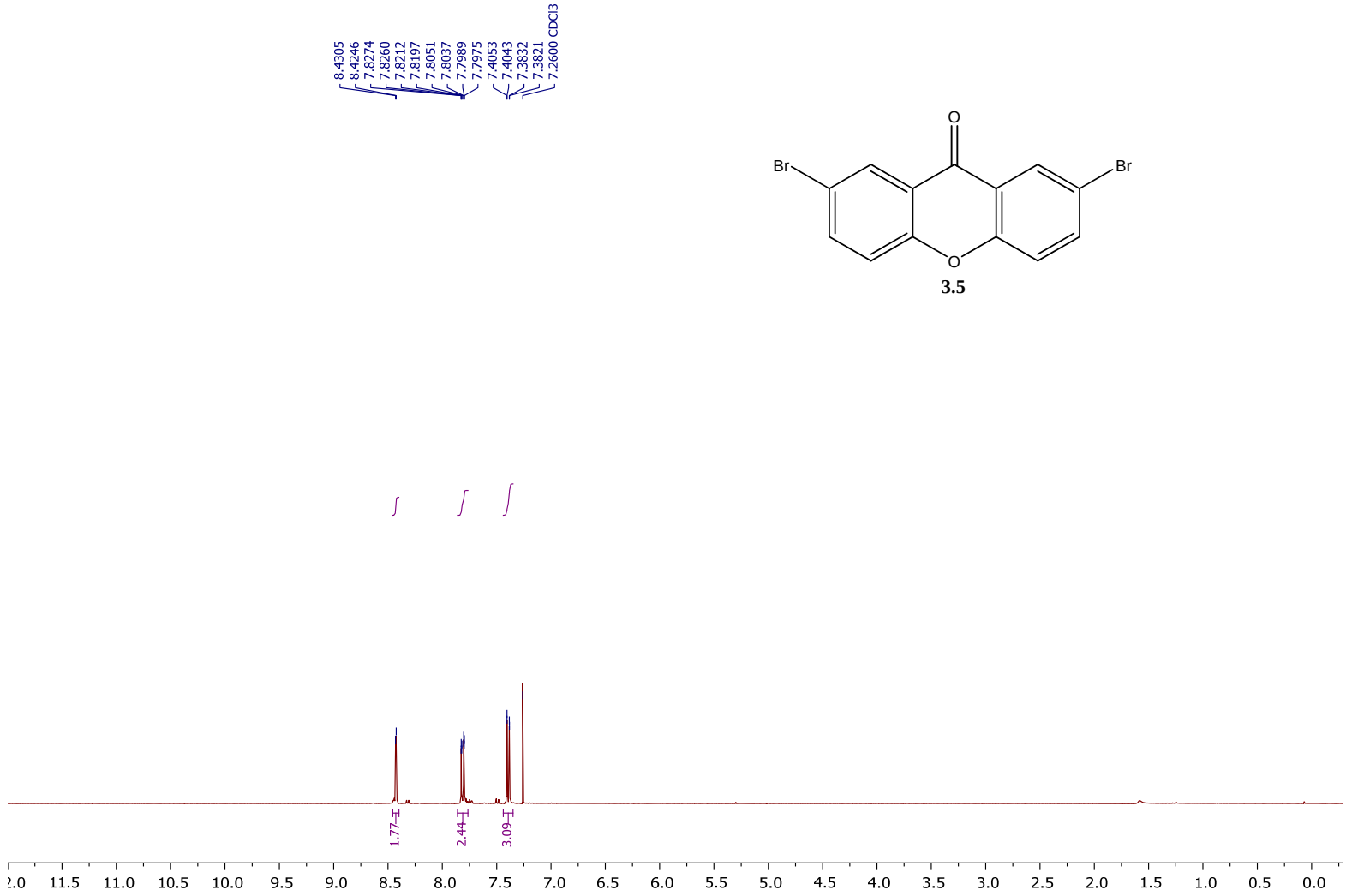
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2,7-difluoro-9*H*-xanthen-9-one (**3.4**)



^{13}C NMR (125 MHz, $CDCl_3$) spectrum of 2,7-difluoro-9*H*-xanthen-9-one (**3.4**)

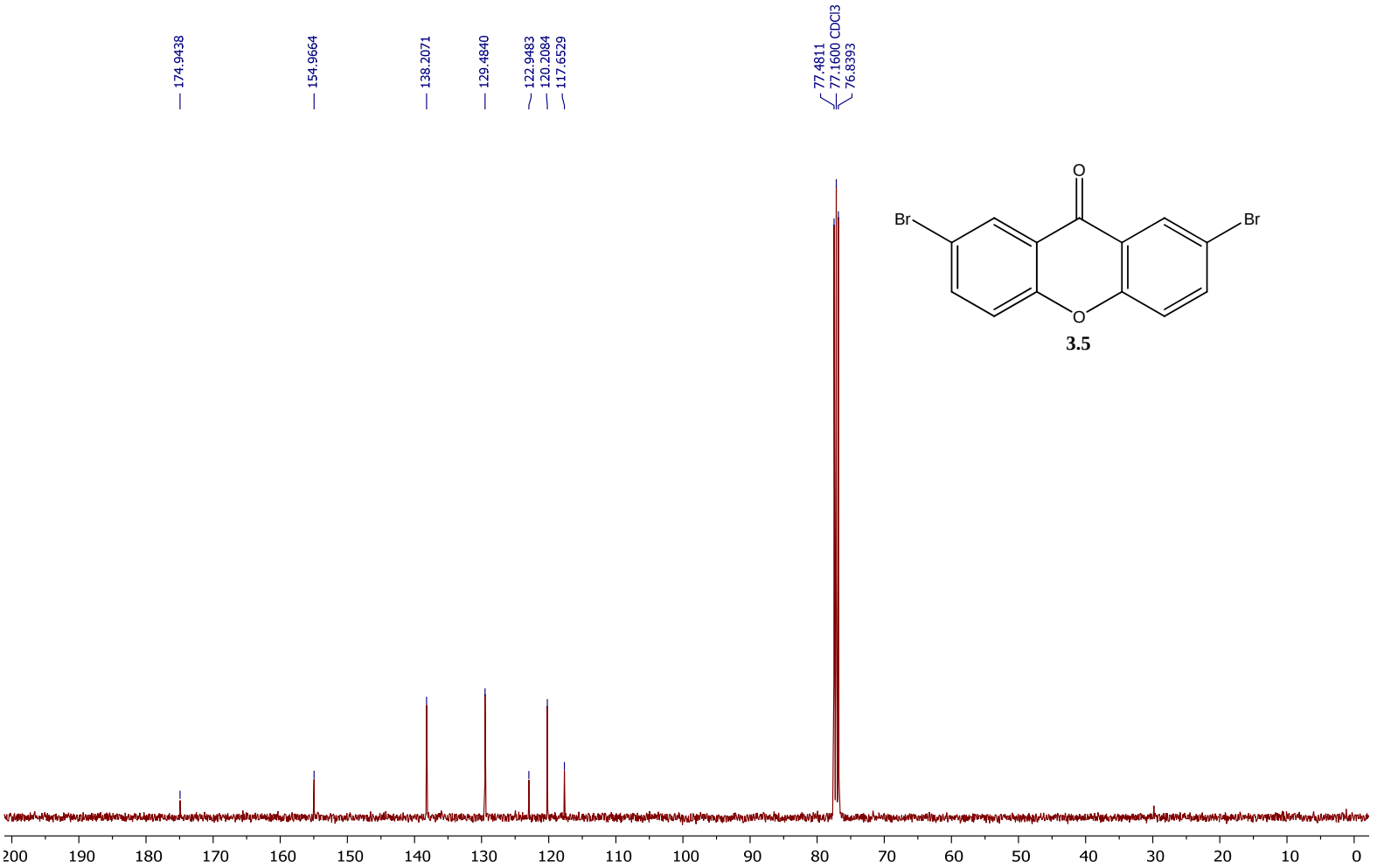


EI(HRMS) spectrum of 2,7-difluoro-9*H*-xanthen-9-one (**3.4**)

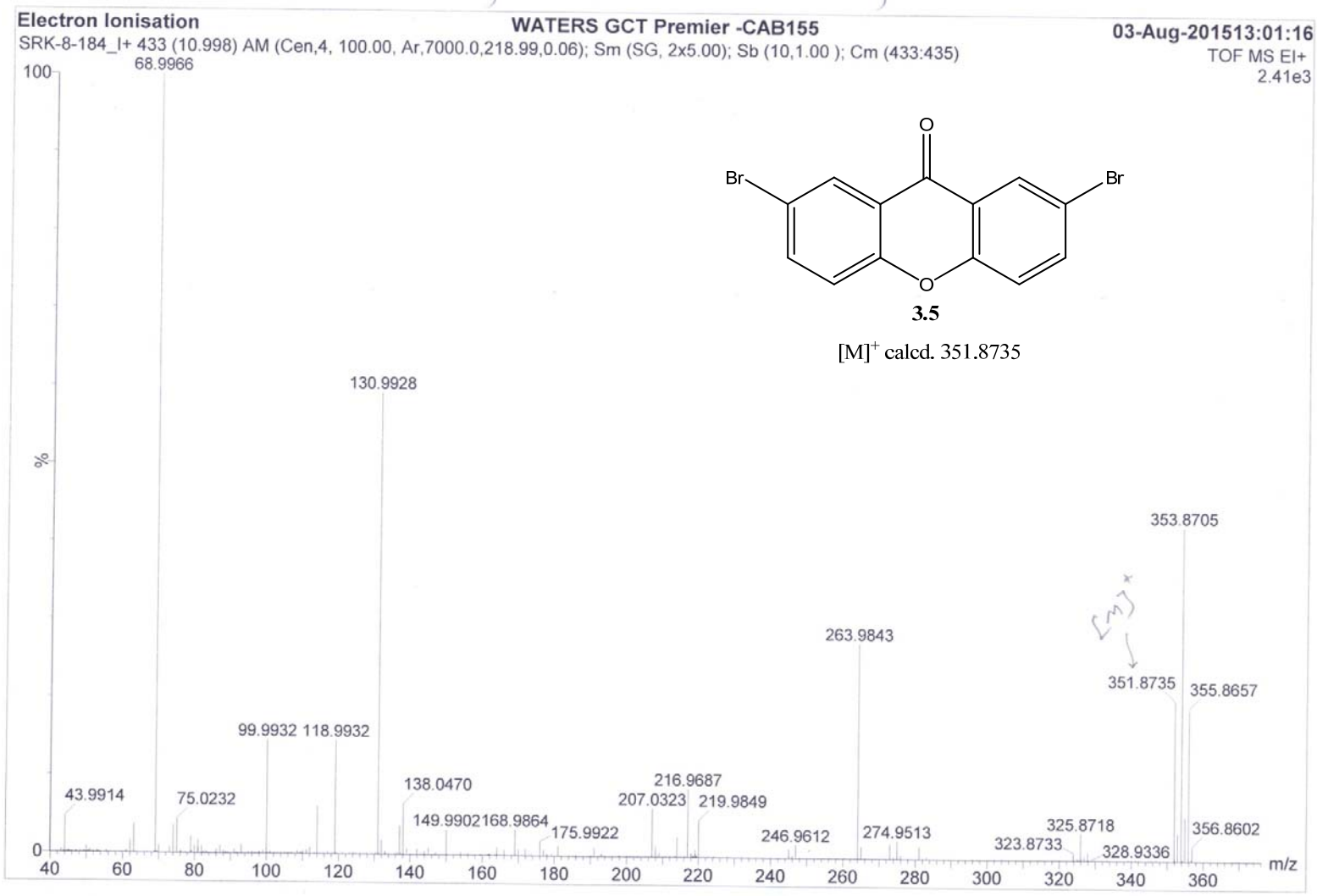


3.5

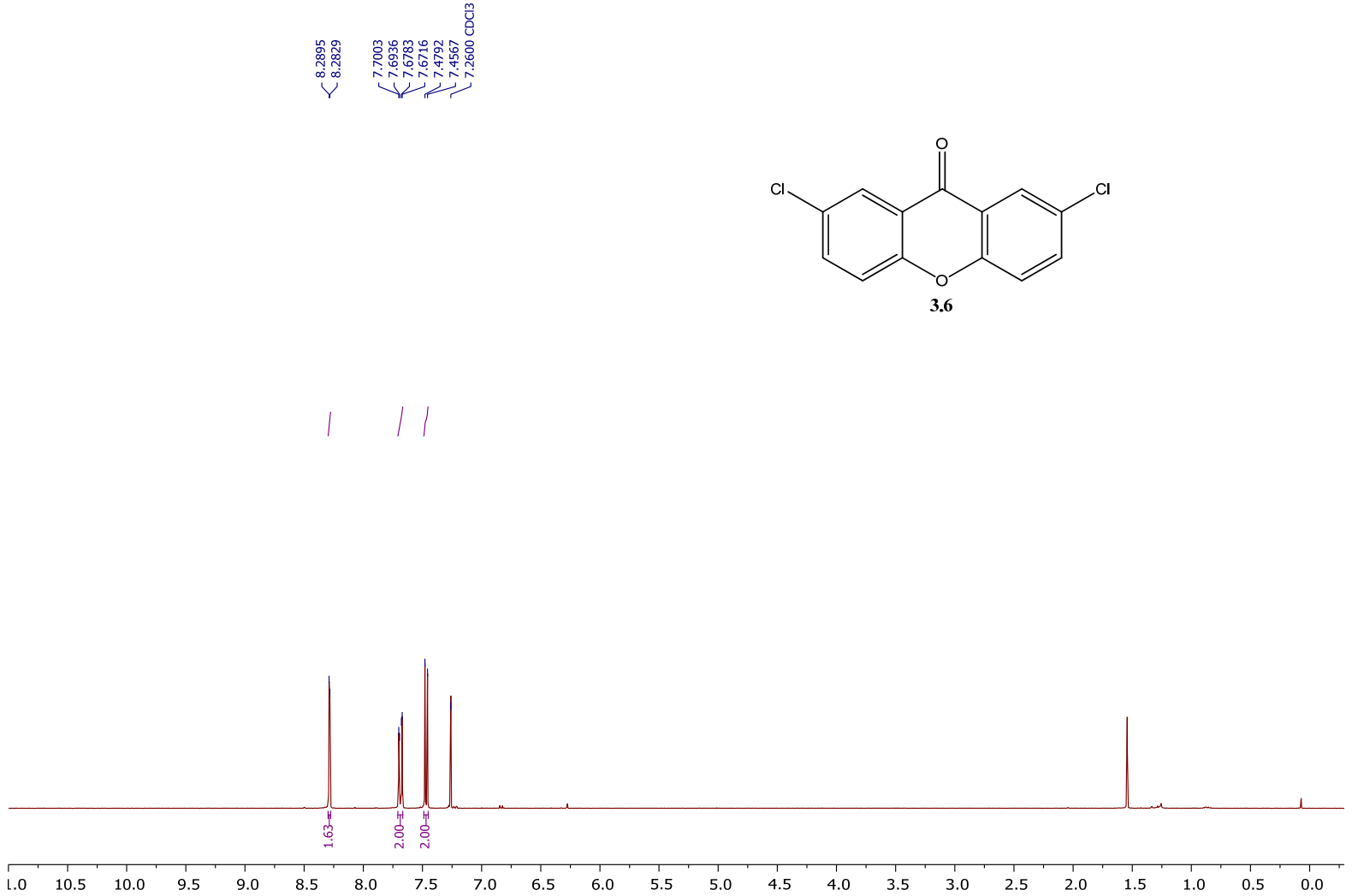
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2,7-dibromo-9*H*-xanthen-9-one (**3.5**)



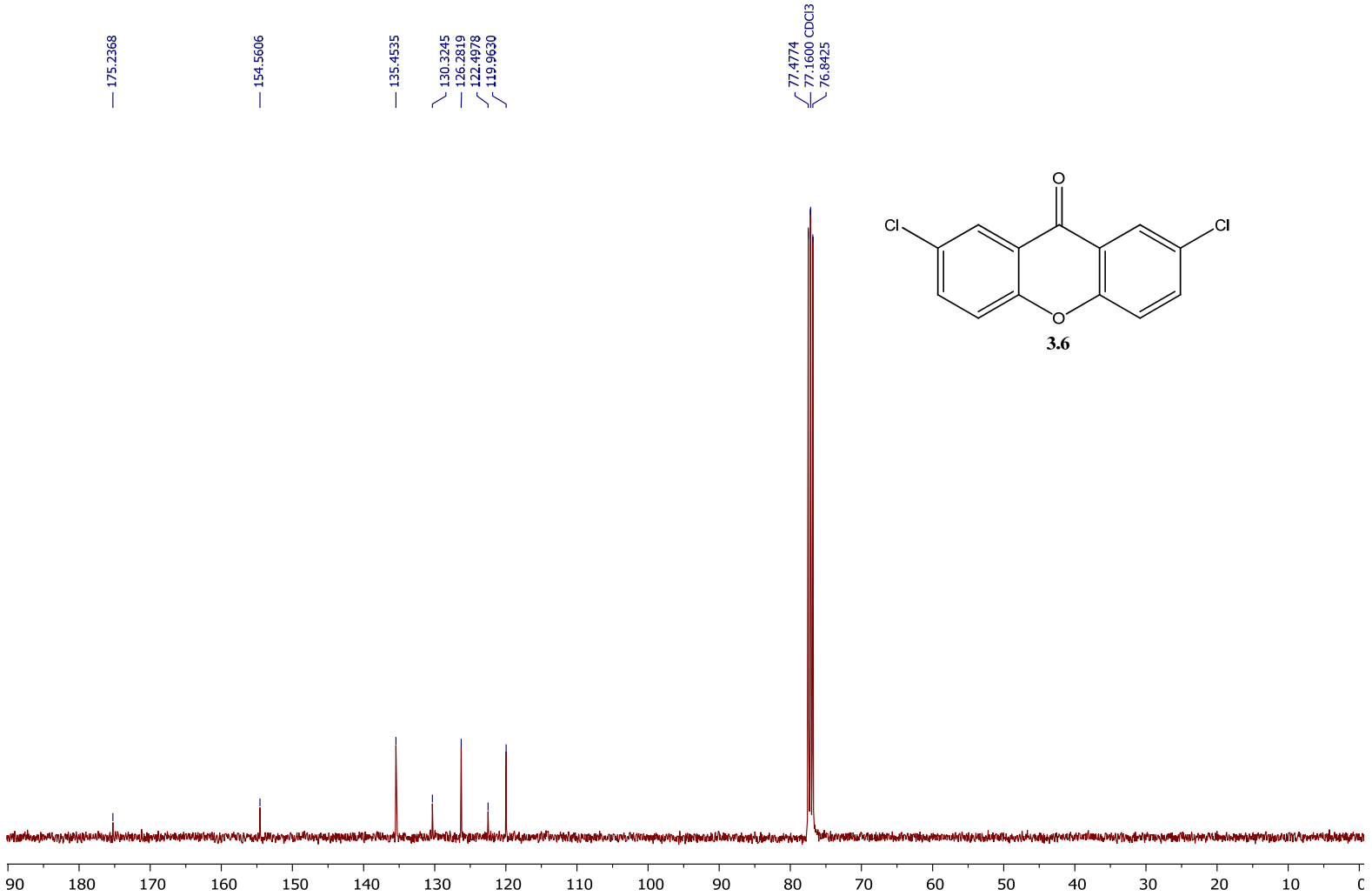
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2,7-dibromo-9*H*-xanthen-9-one (**3.5**)



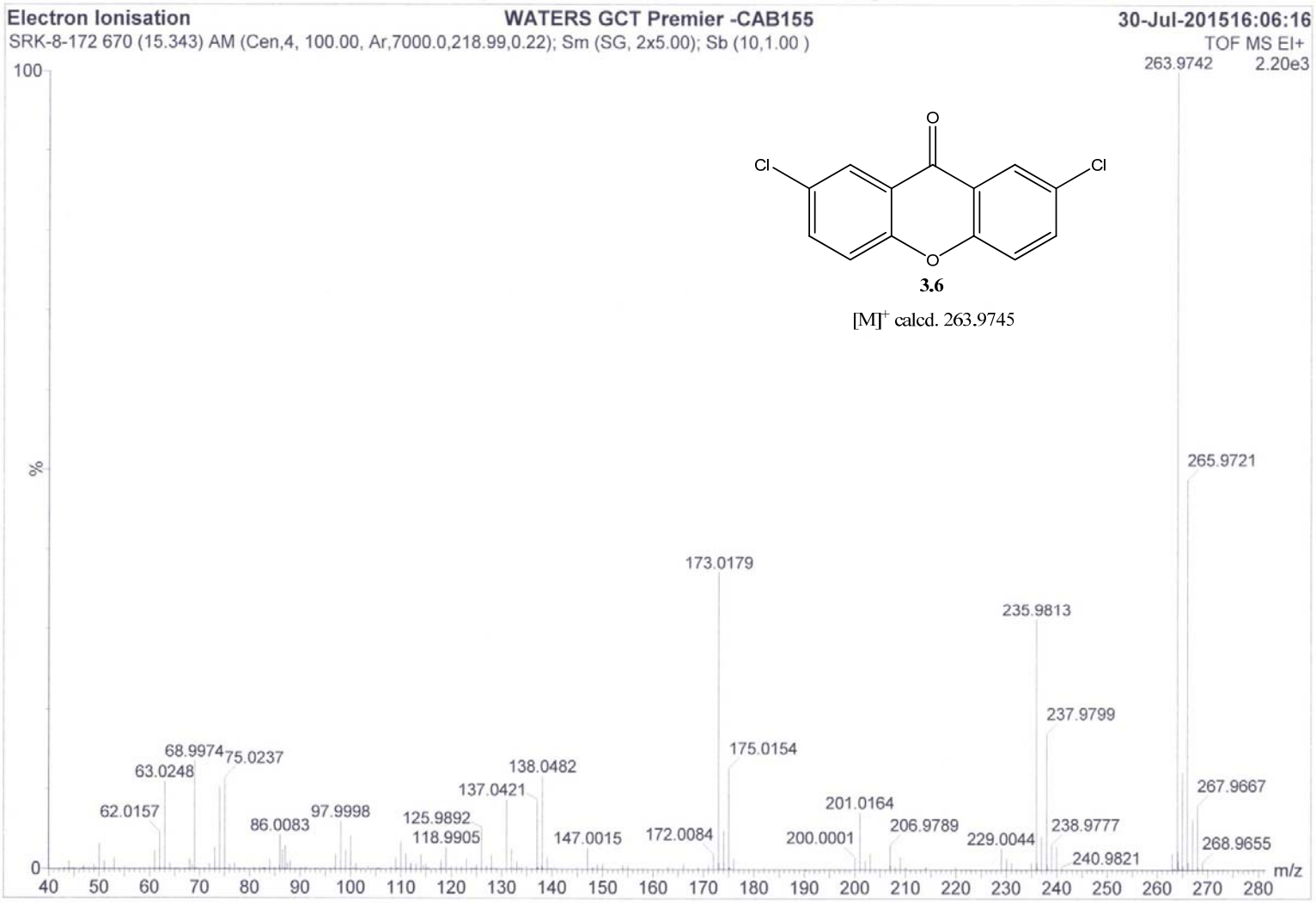
EI(HRMS) spectrum of 2,7-dibromo-9*H*-xanthen-9-one (**3.5**)



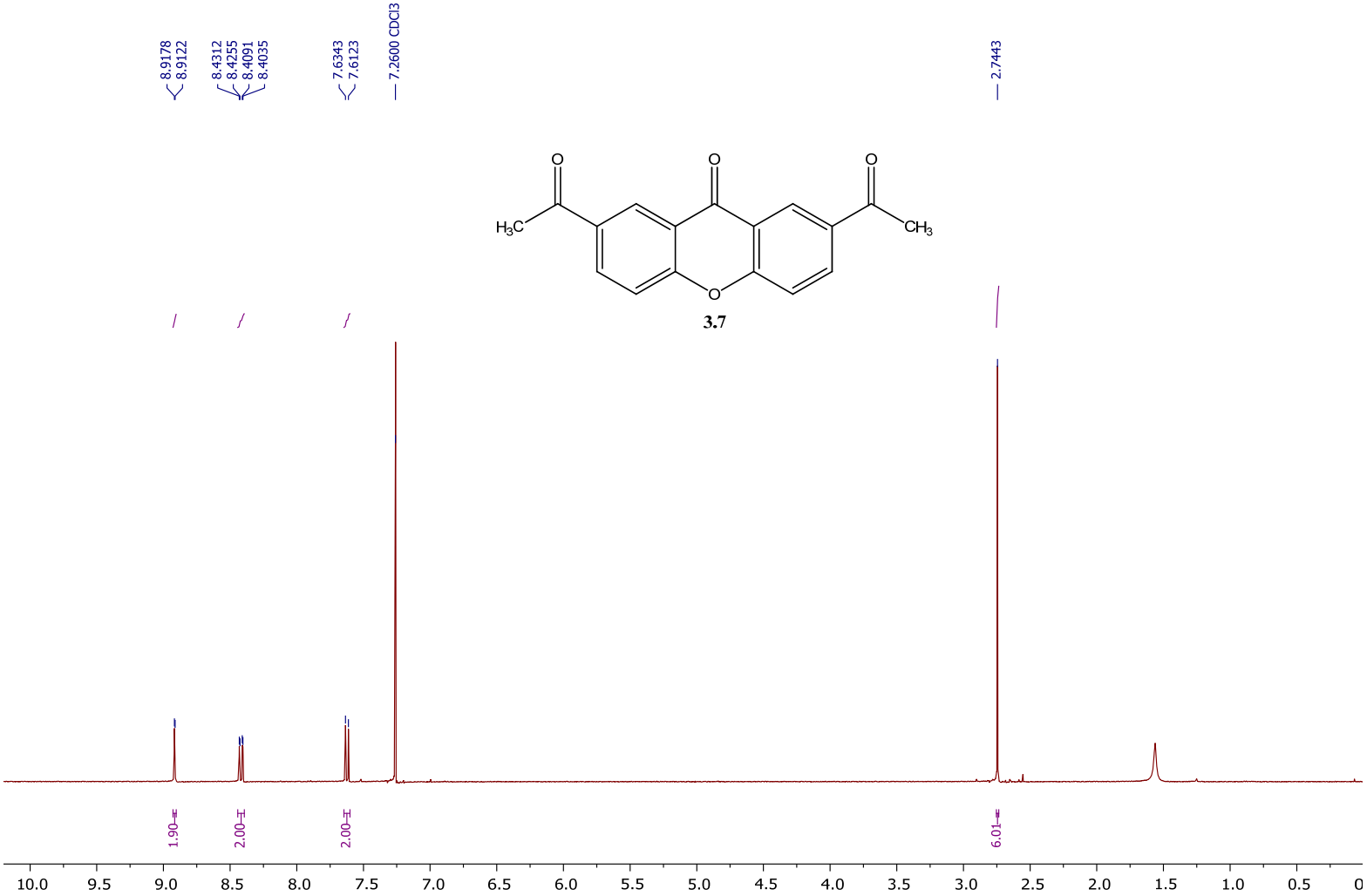
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2,7-dichloro-9*H*-xanthen-9-one (**3.6**)



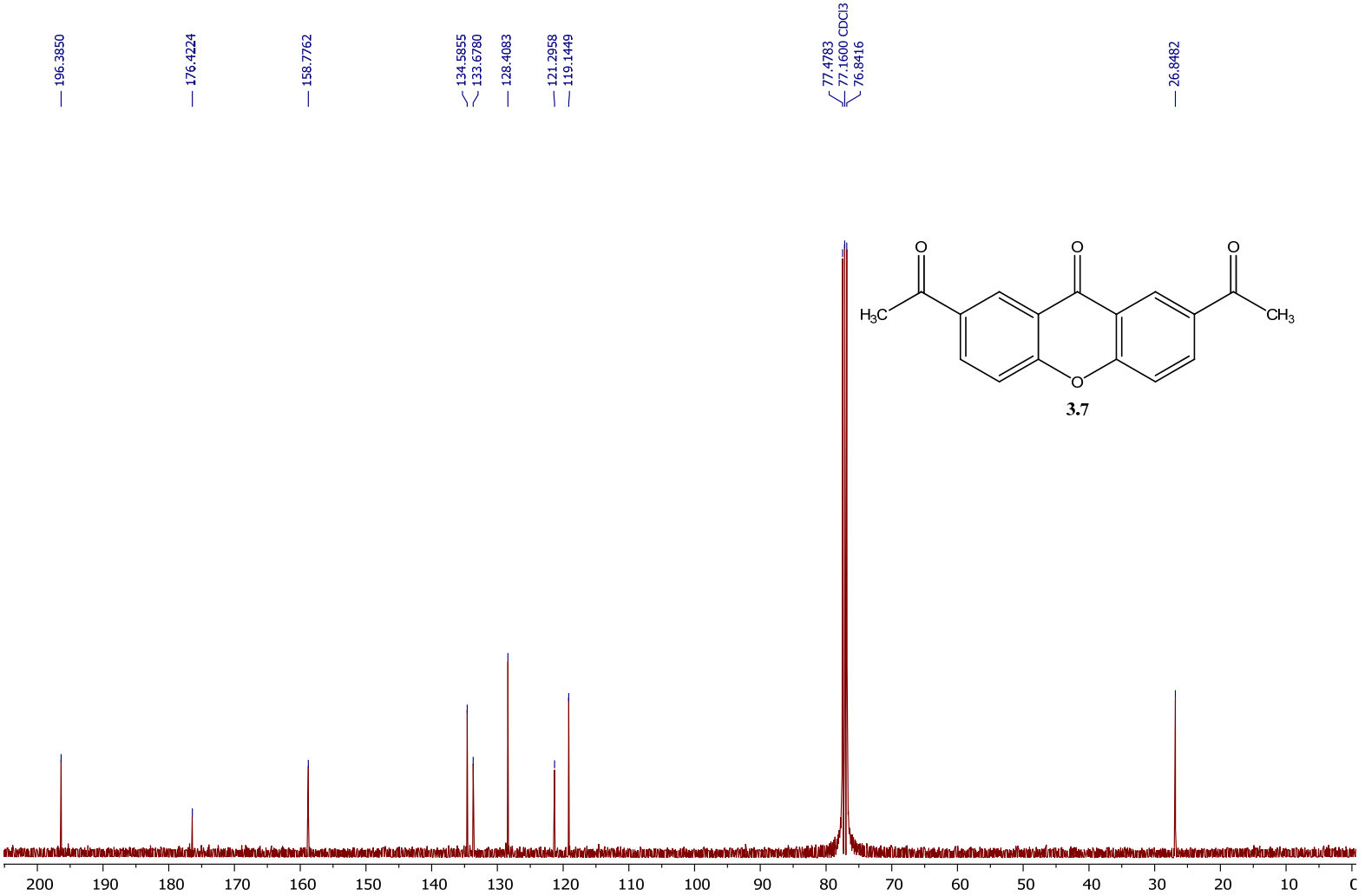
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2,7-dichloro-9*H*-xanthen-9-one (**3.6**)



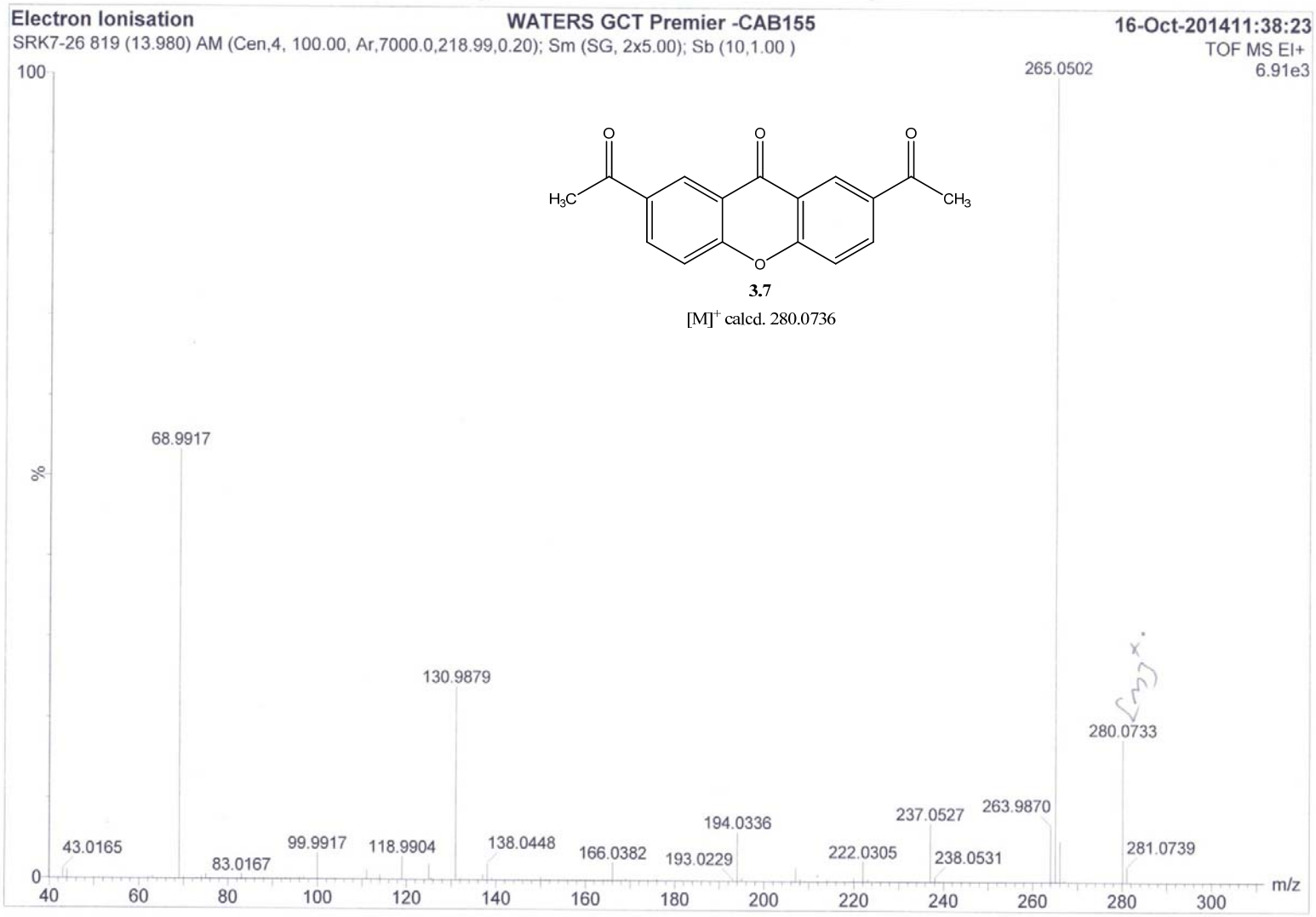
EI(HRMS) spectrum of 2,7-dichloro-9*H*-xanthen-9-one (**3.6**)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1'-(9-oxo-9*H*-xanthene-2,7-diyl)diethanone (**3.7**)



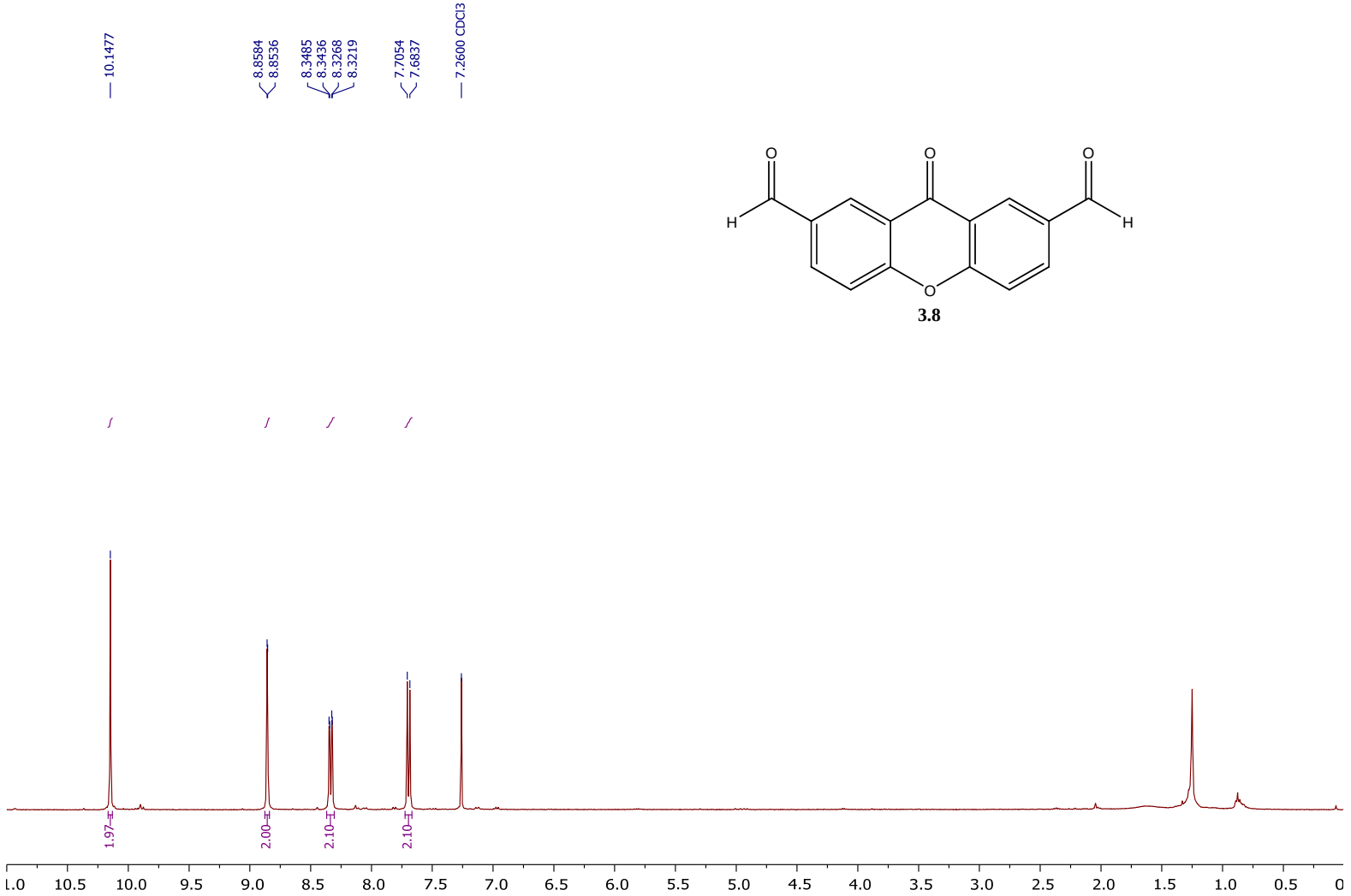
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 1,1'-(9-oxo-9*H*-xanthene-2,7-diyl)diethanone (**3.7**)



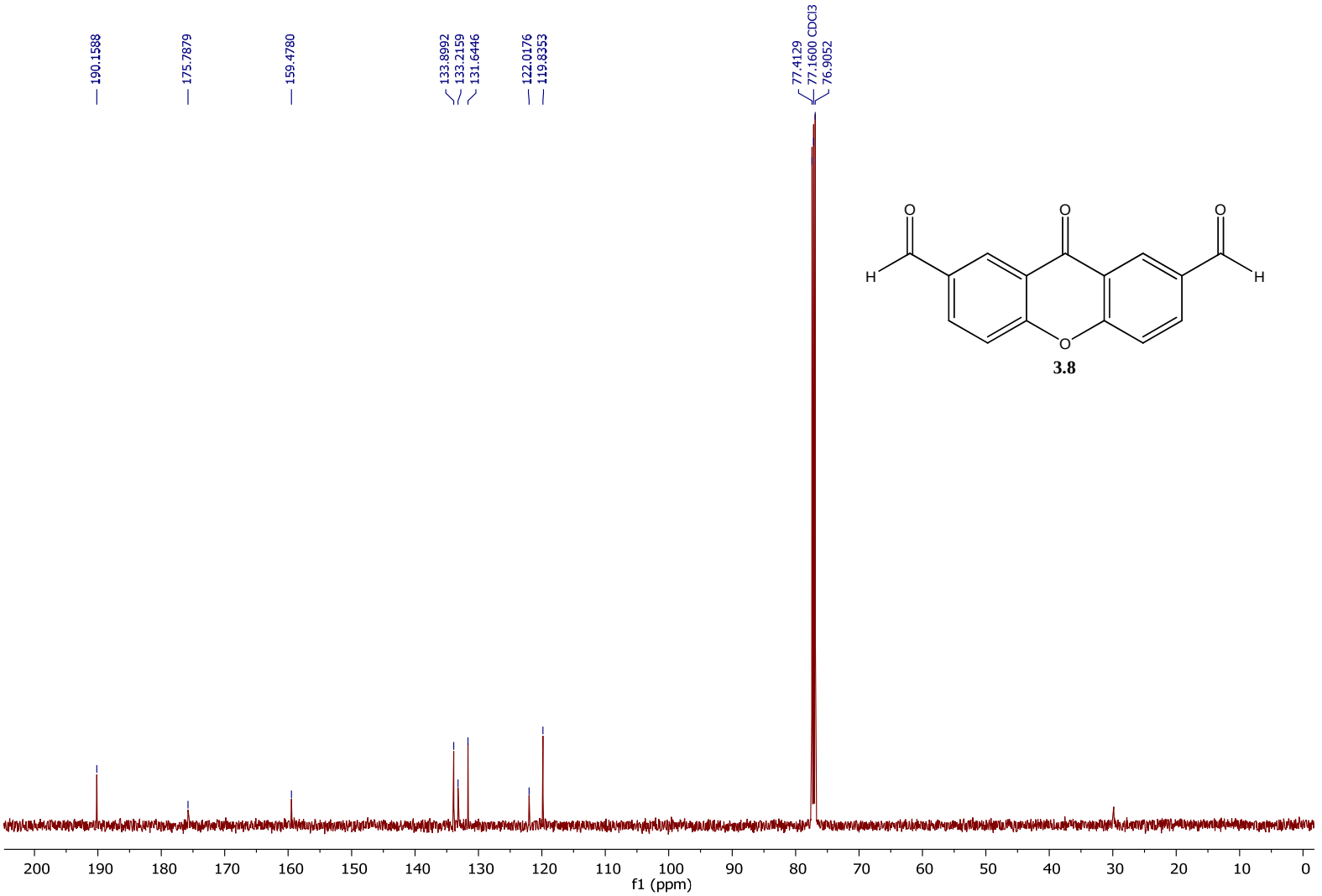
3.7

$[M]^+$ calcd. 280.0736

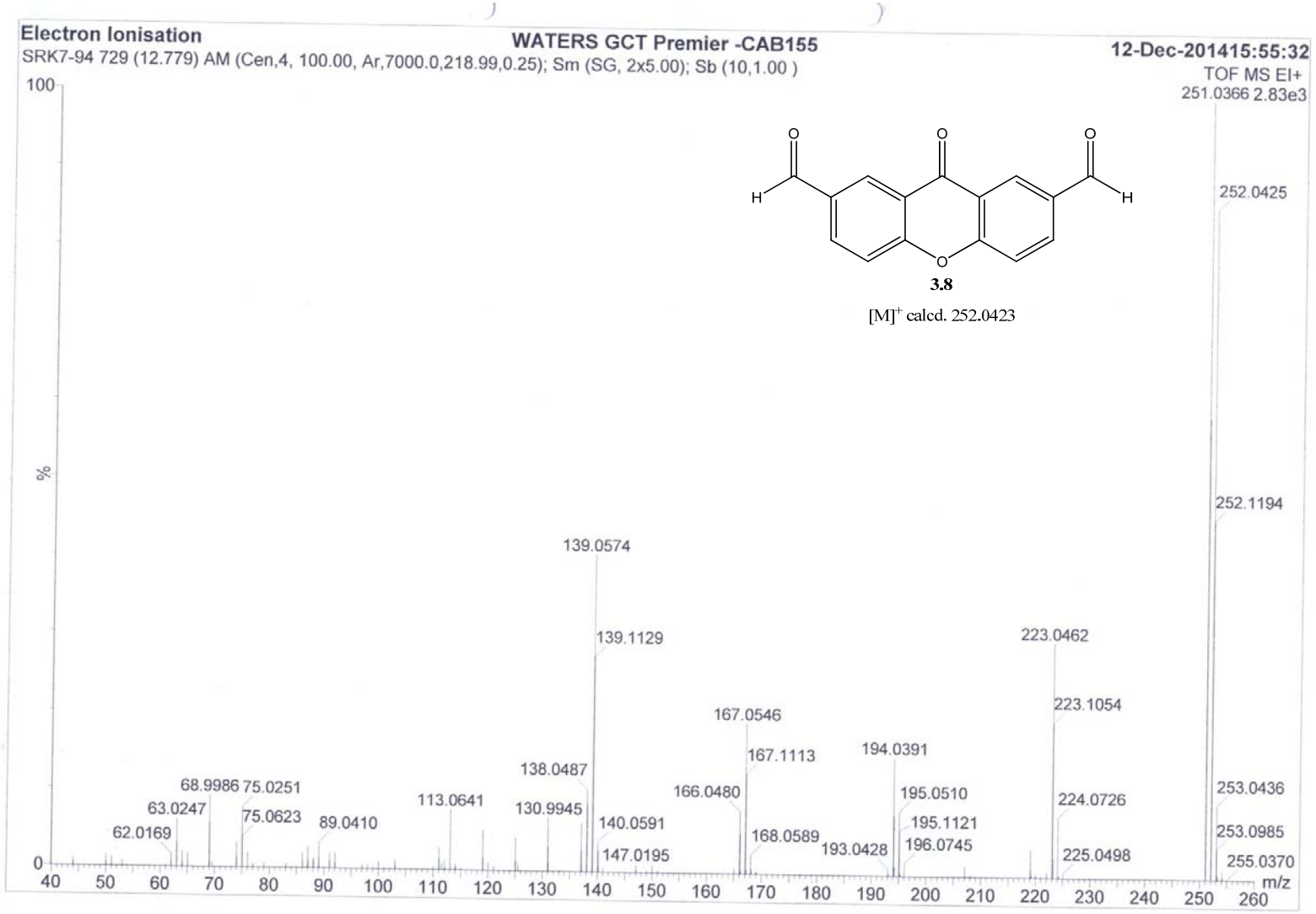
EI(HRMS) spectrum of 1,1'-(9-oxo-9*H*-xanthene-2,7-diyl)diethanone (**3.7**)



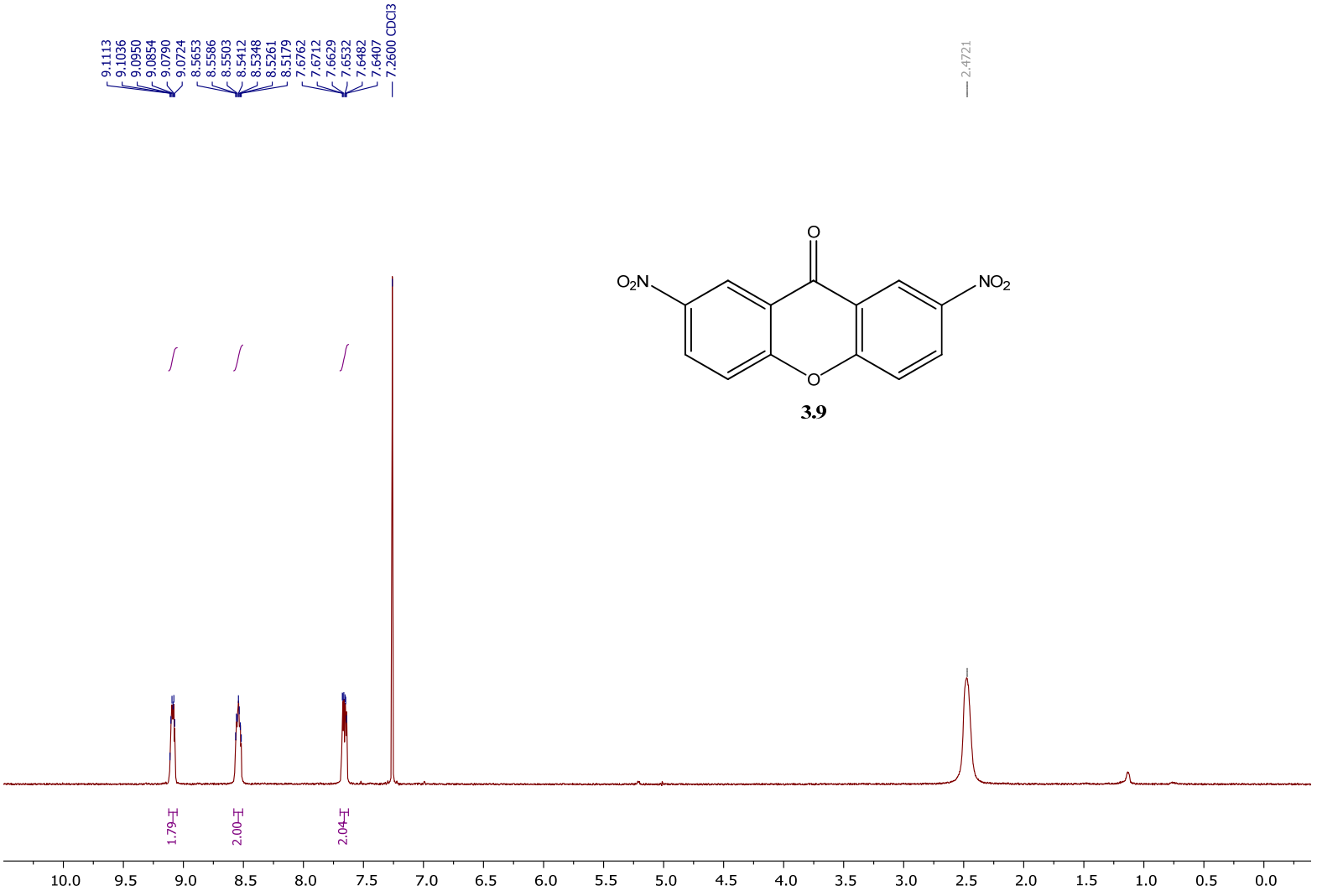
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 9-oxo-9*H*-xanthene-2,7-dicarbaldehyde (**3.8**)



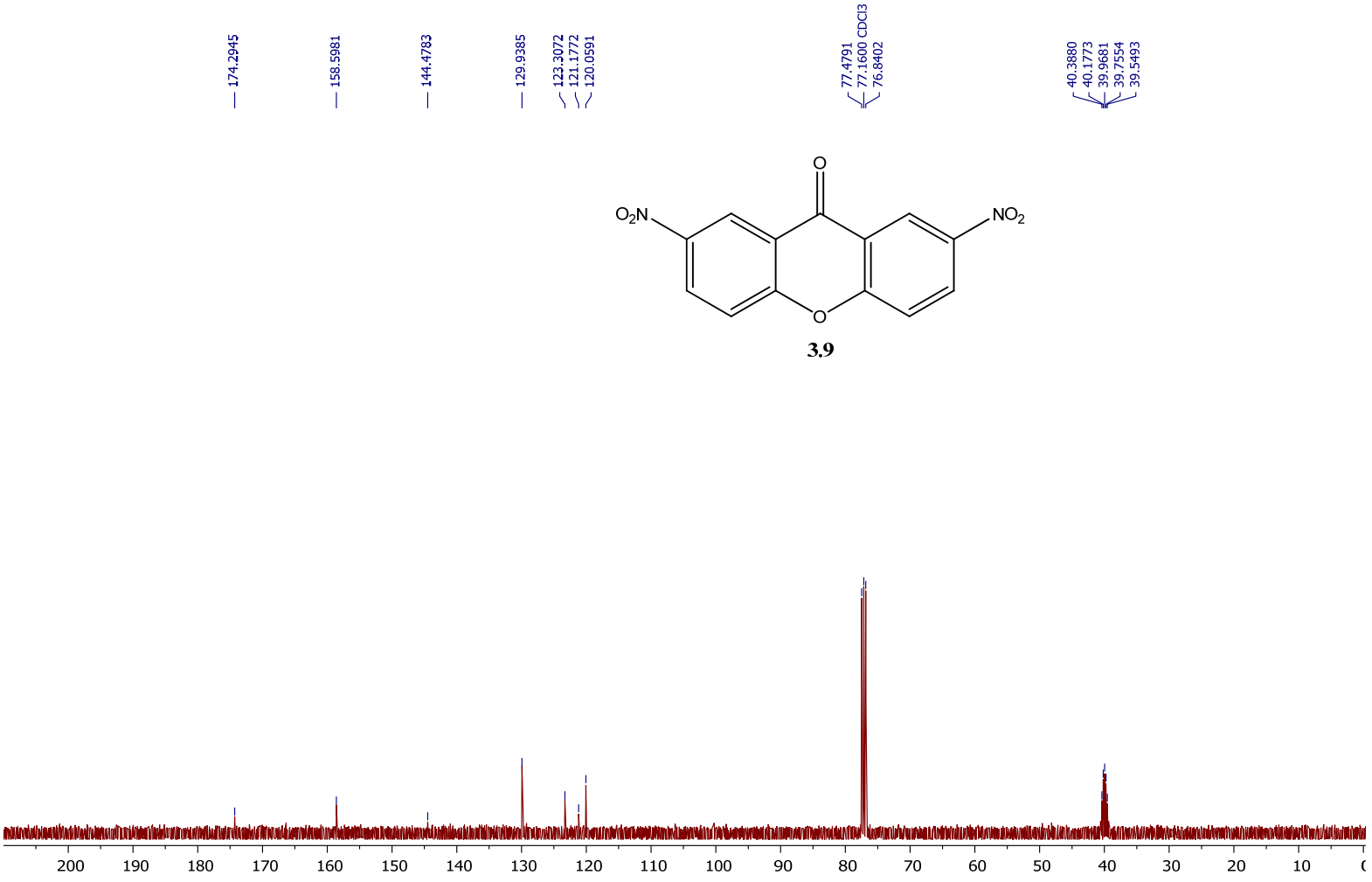
^{13}C NMR (125 MHz, $CDCl_3$) spectrum of 9-oxo-9*H*-xanthene-2,7-dicarbaldehyde (**3.8**)



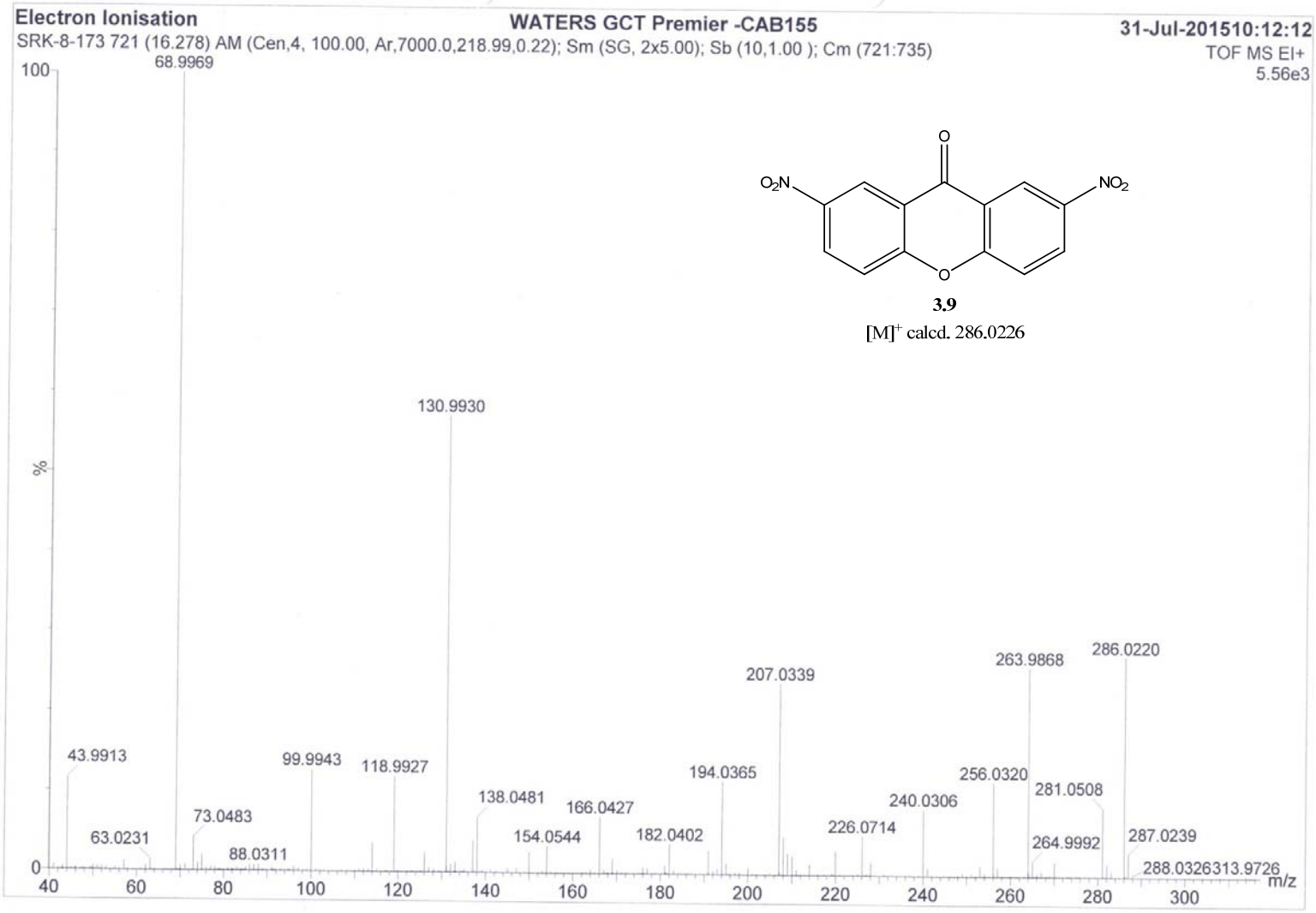
EI(HRMS) spectrum of 9-oxo-9*H*-xanthene-2,7-dicarbaldehyde (**3.8**)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 2,7-dinitro-9*H*-xanthen-9-one (**3.9**) (9:1)



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 2,7-dinitro-9*H*-xanthen-9-one (**3.9**) (9:1)

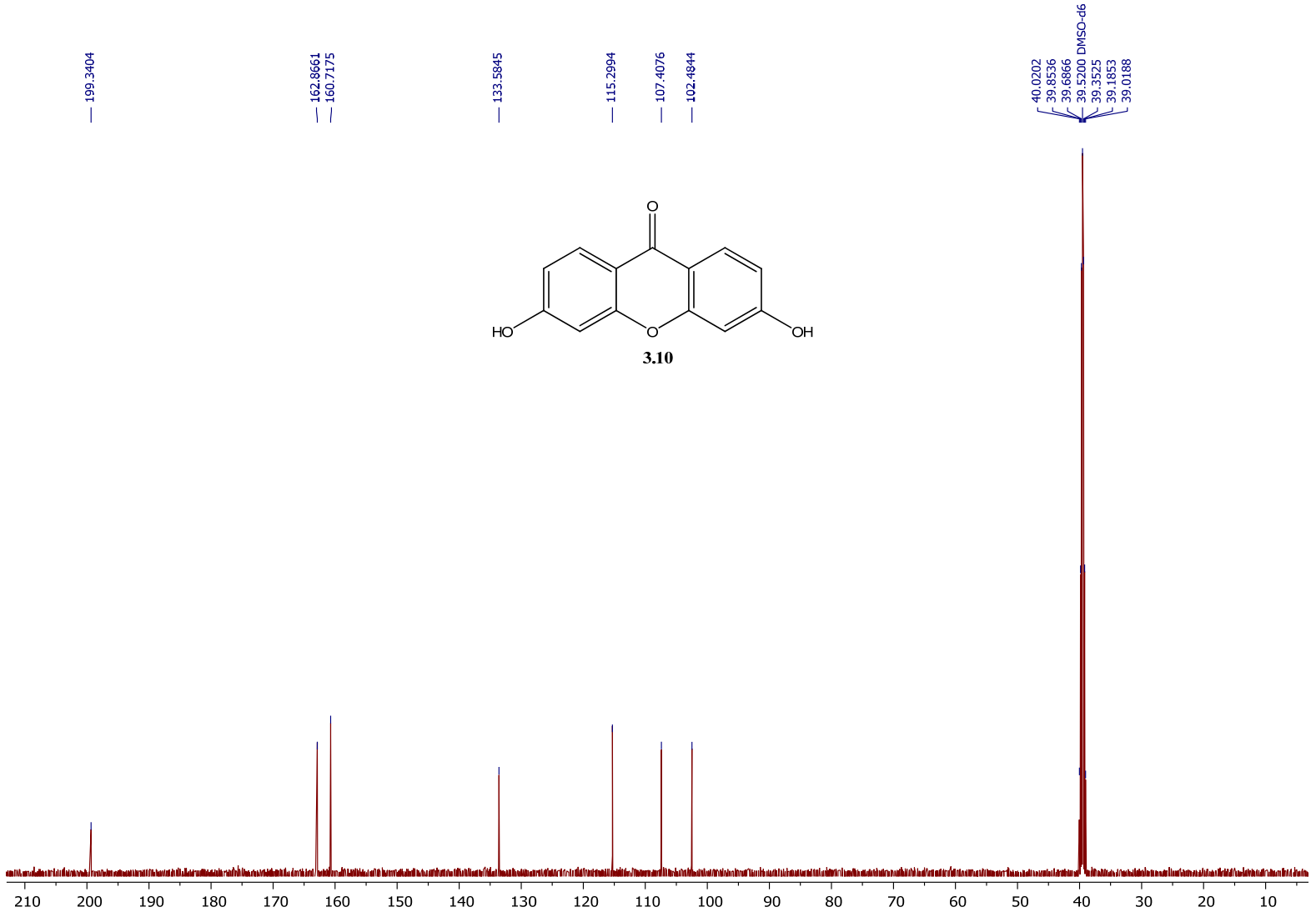


3.9

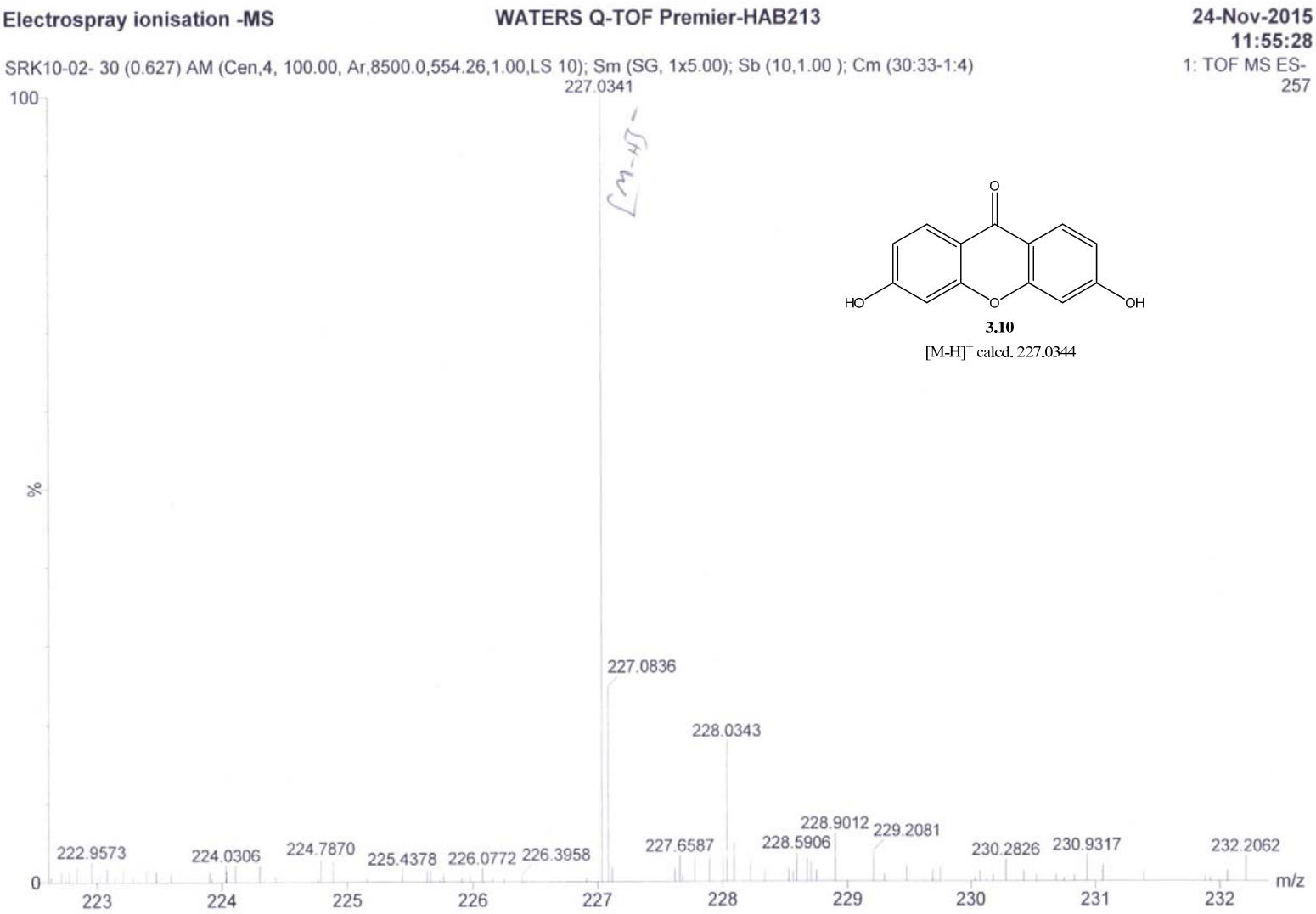
$[M]^+$ calcd. 286.0226

EI(HRMS) spectrum of 2,7-dinitro-9*H*-xanthen-9-one (**3.9**)

^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3,6-dihydroxy-9*H*-xanthen-9-one (**3.10**)

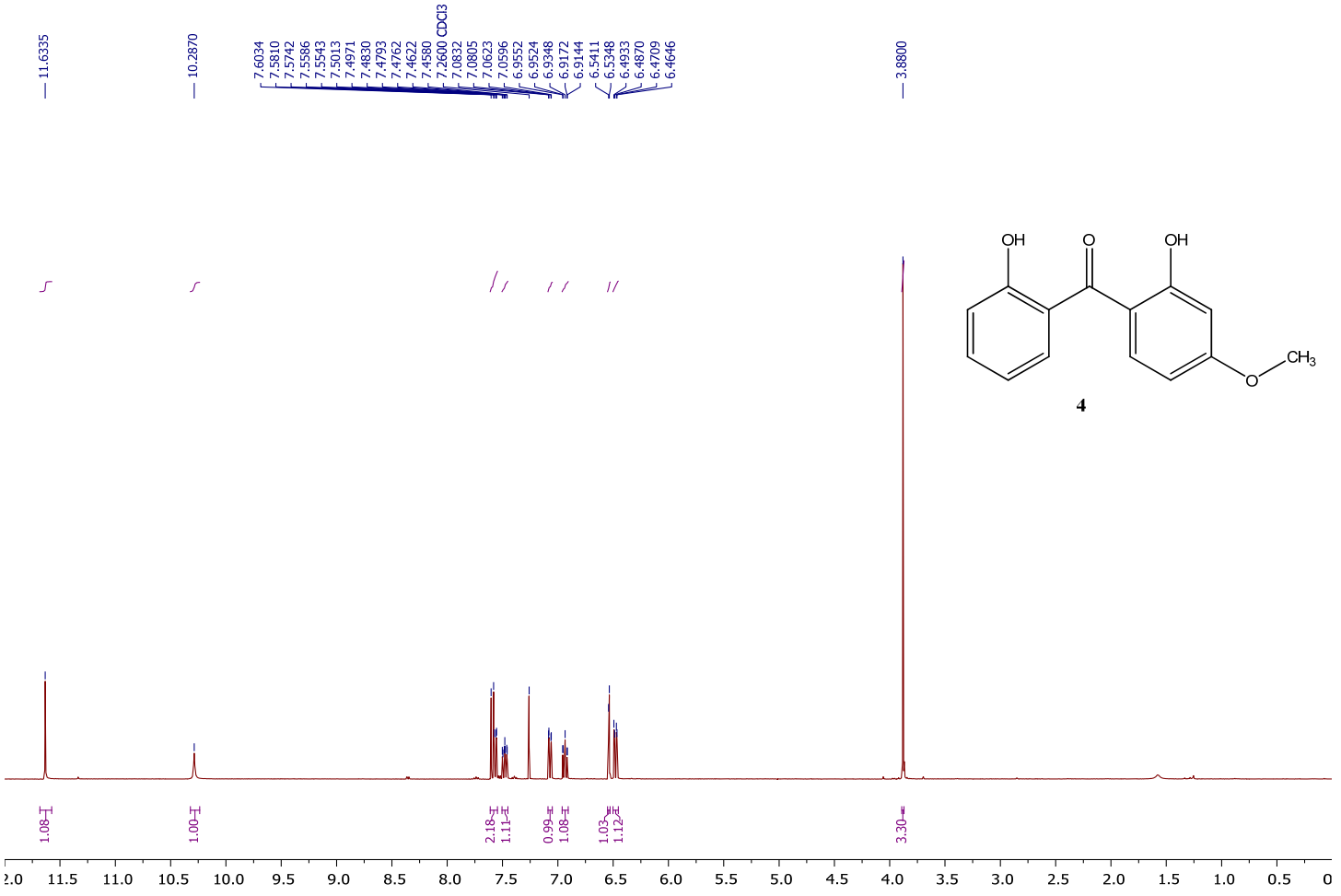


^{13}C NMR (125 MHz, DMSO-d_6) spectrum of 3,6-dihydroxy-9*H*-xanthen-9-one (**3.10**)

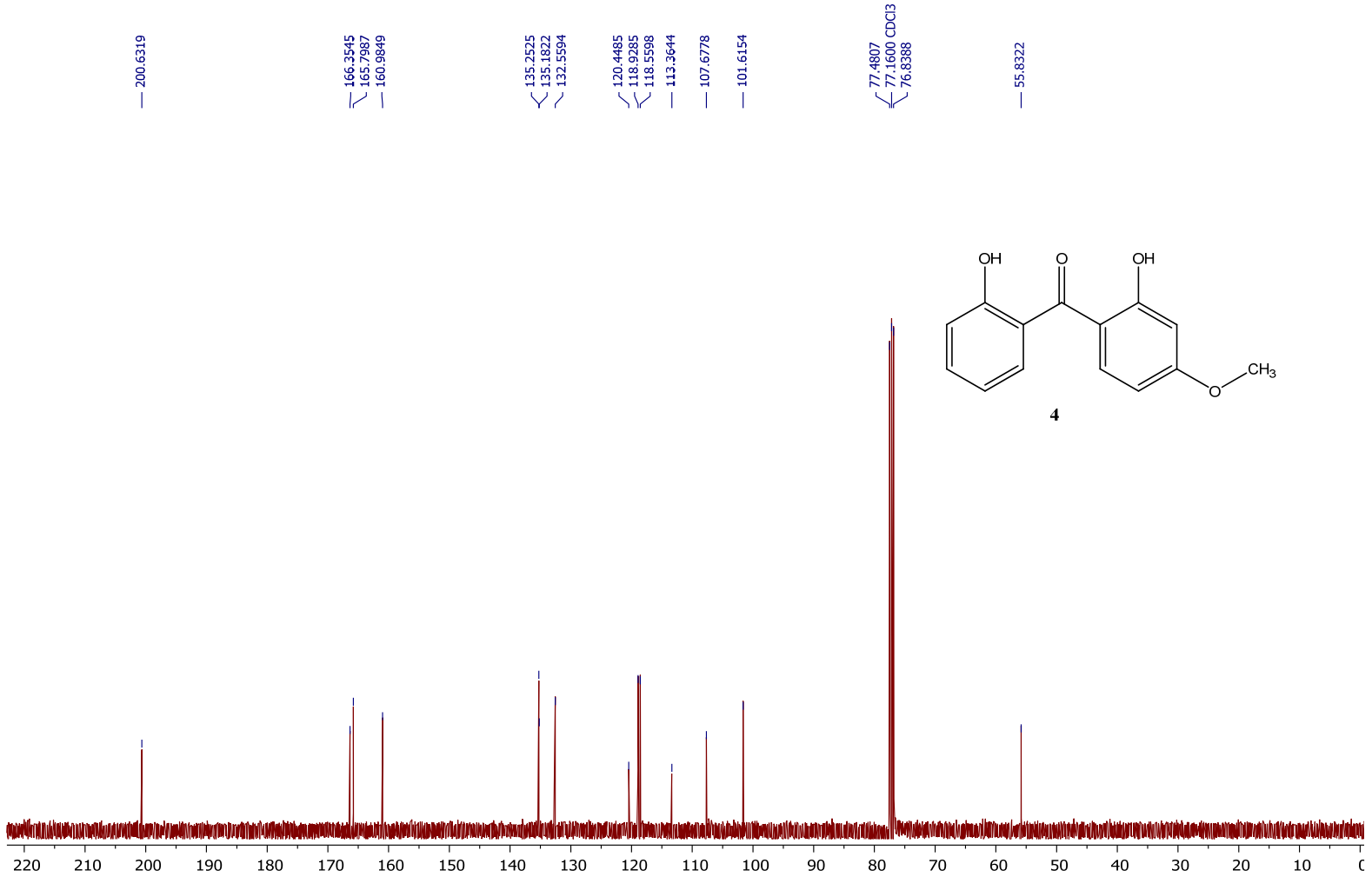


ESI(HRMS) spectrum of 3,6-dihydroxy-9*H*-xanthen-9-one (**3.10**)

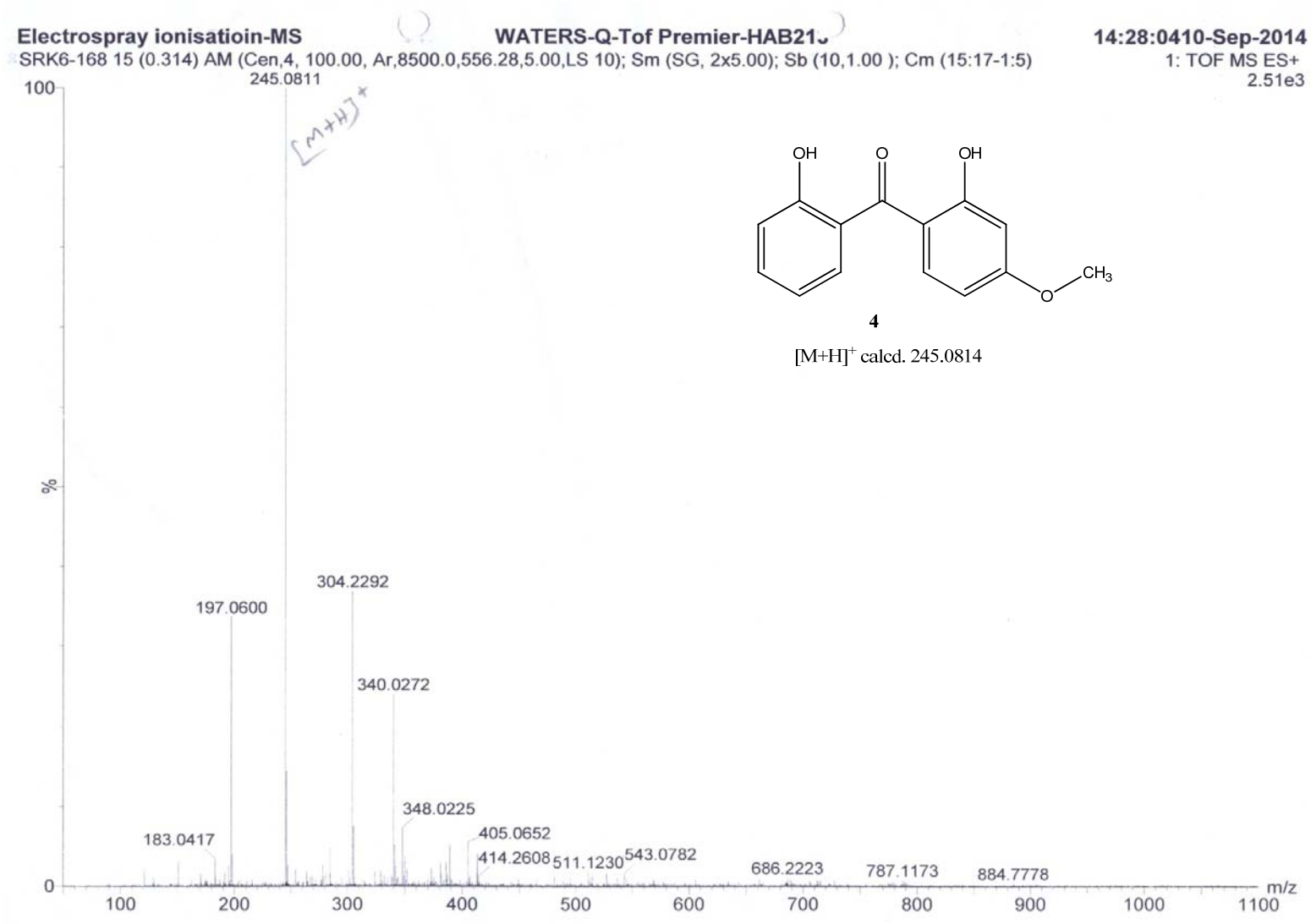
4. Copies of ^{1}H NMR, ^{13}C NMR and HRMS spectra of Compound 4 (Scheme 2):



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of (2-hydroxy-4-methoxyphenyl)(2-hydroxyphenyl)methanone (**4**)



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of (2-hydroxy-4-methoxyphenyl)(2-hydroxyphenyl)methanone (**4**)



ESI(HRMS) spectrum of (2-hydroxy-4-methoxyphenyl)(2-hydroxyphenyl)methanone (**4**)