

Supporting information for

Chemistry of 9,10-phenanthrenequinone revisited: Iron(III) chloride catalyzed reactions of 9,10-phenanthrenequinone with acyclic and cyclic ketones provide furan annulated products

H. Surya Prakash Rao*, V. Satish

Department of Chemistry, Pondicherry University, Pondicherry – 605 014. INDIA

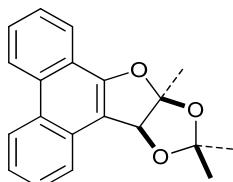
Email: hspr.che@pondiuni.edu.in.

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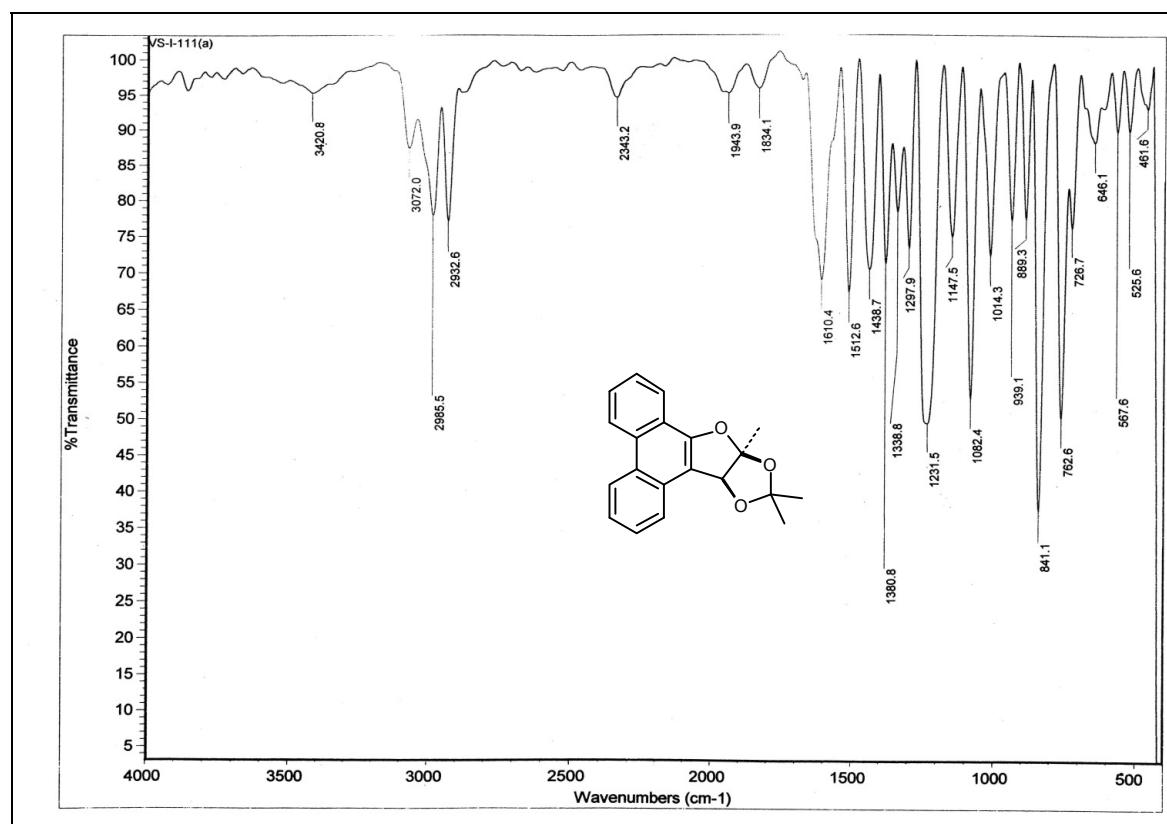
1) Copies of IR, ¹H NMR, ¹³C NMR, DEPT-135 and HRMS spectra for new compounds.

A) 9a,11,11-Trimethyl-9a,12a-dihydrophenanthro[9',10':4,5]furo[2,3-d][1,3]dioxole **3**.

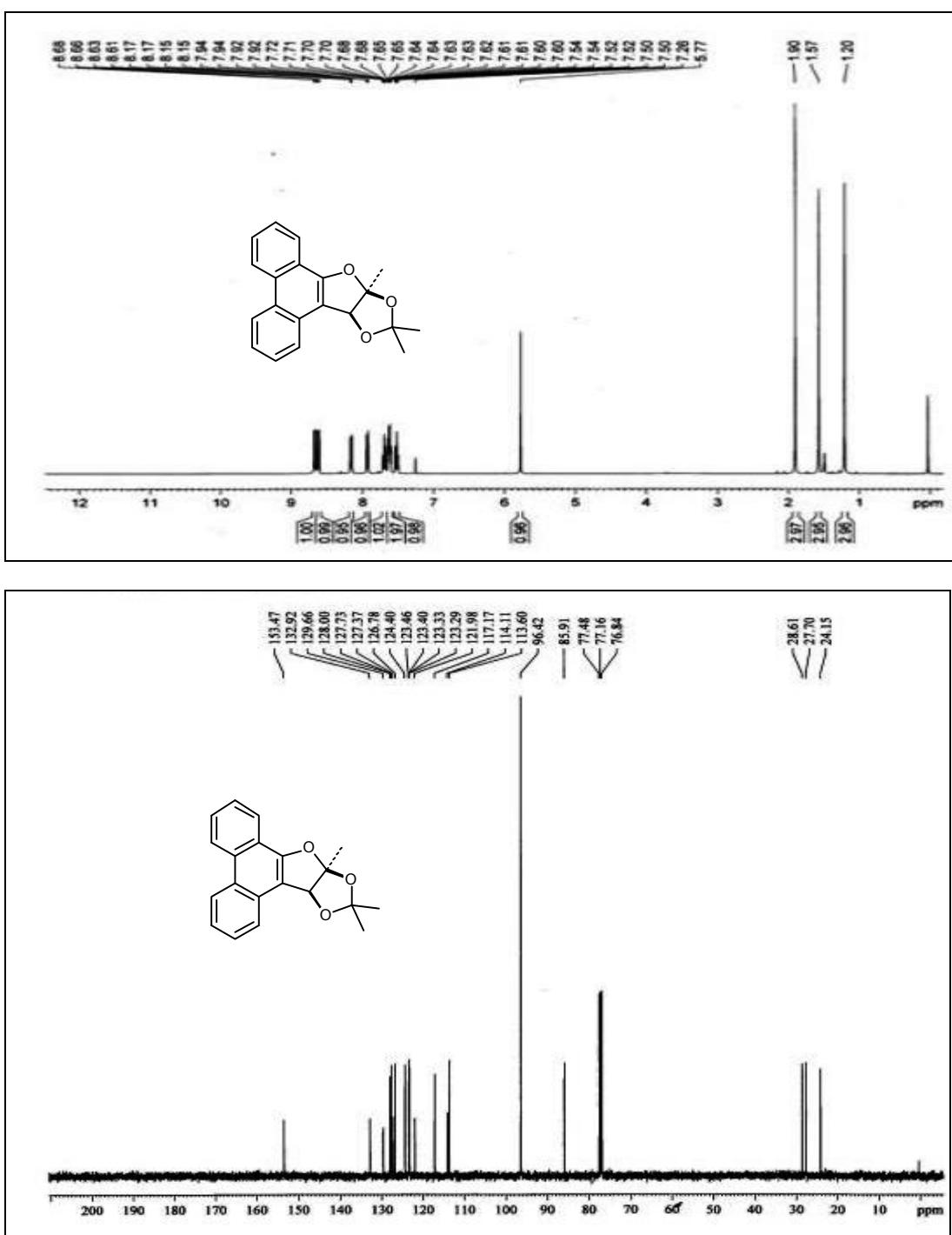


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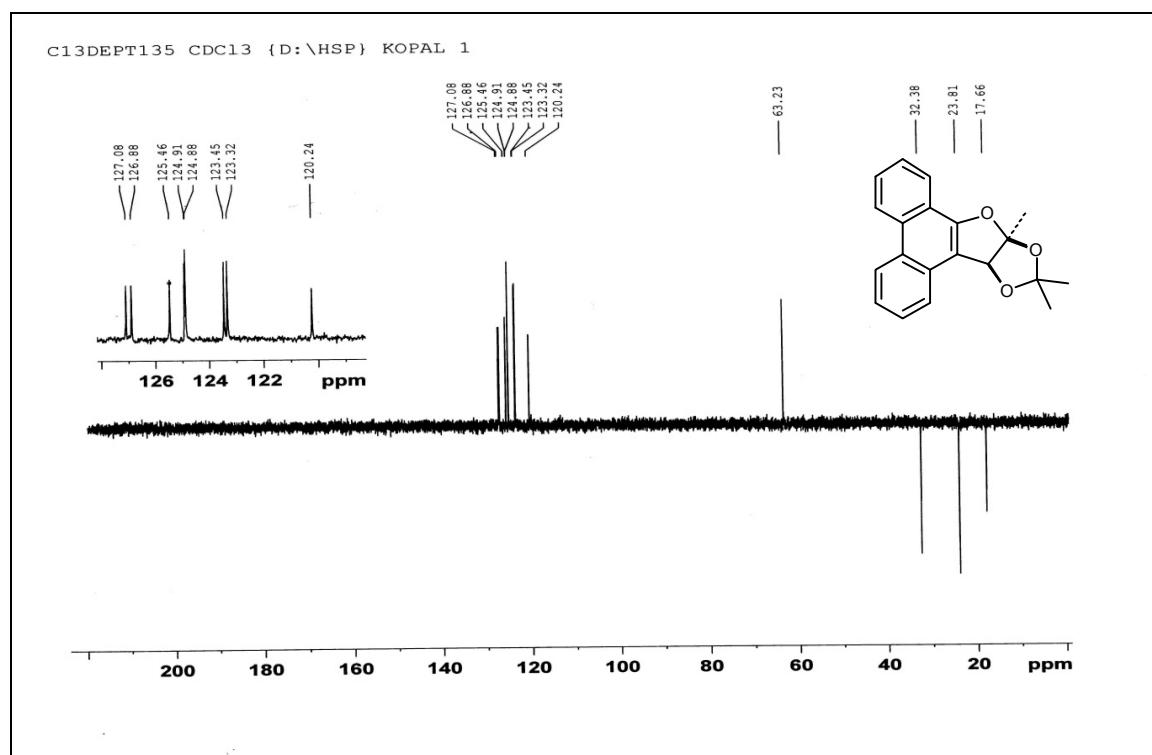
IR spectrum of **3**.



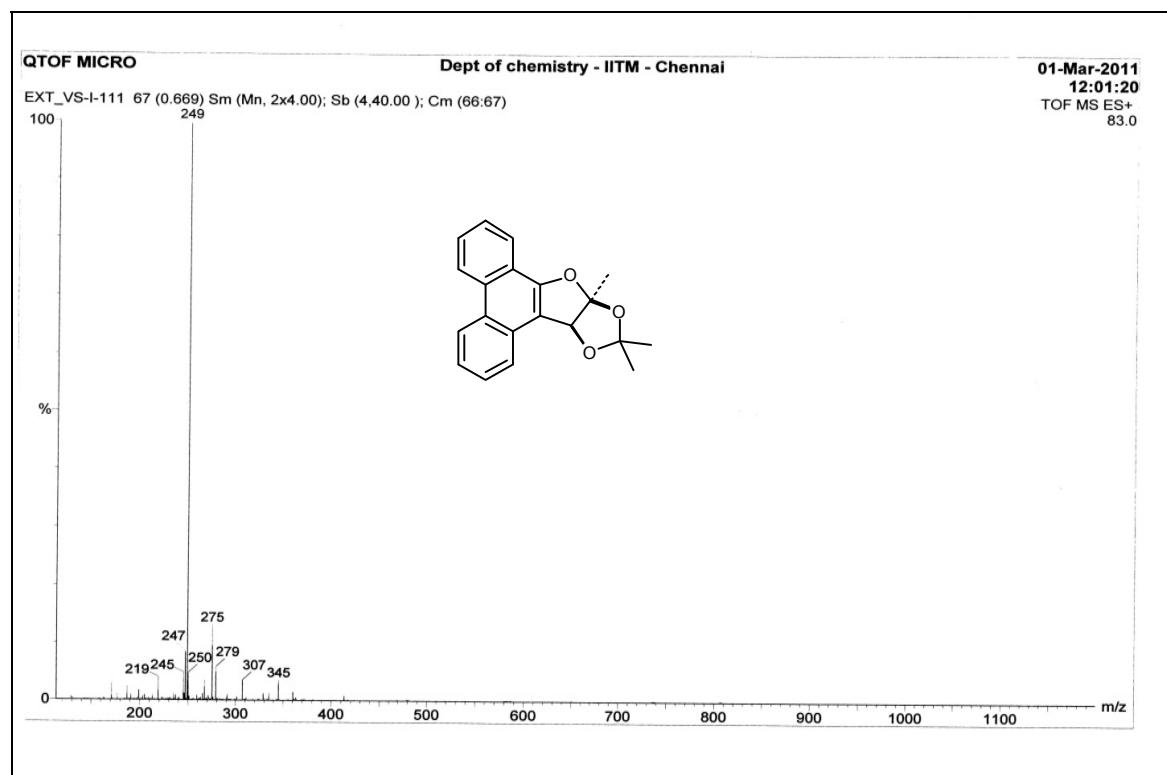
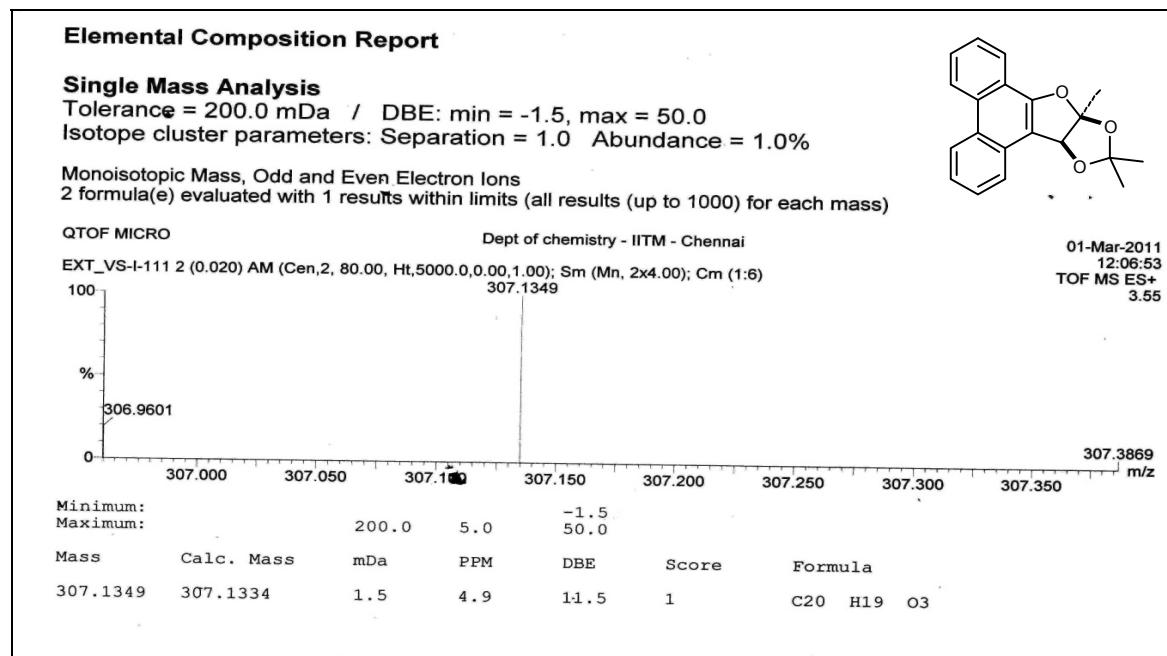
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) NMR spectra of **3**.



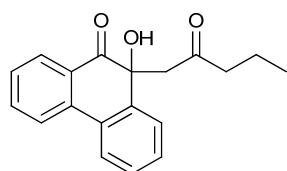
DEPT-135 NMR spectrum of **3**.



ESIMS HRMS of 3.

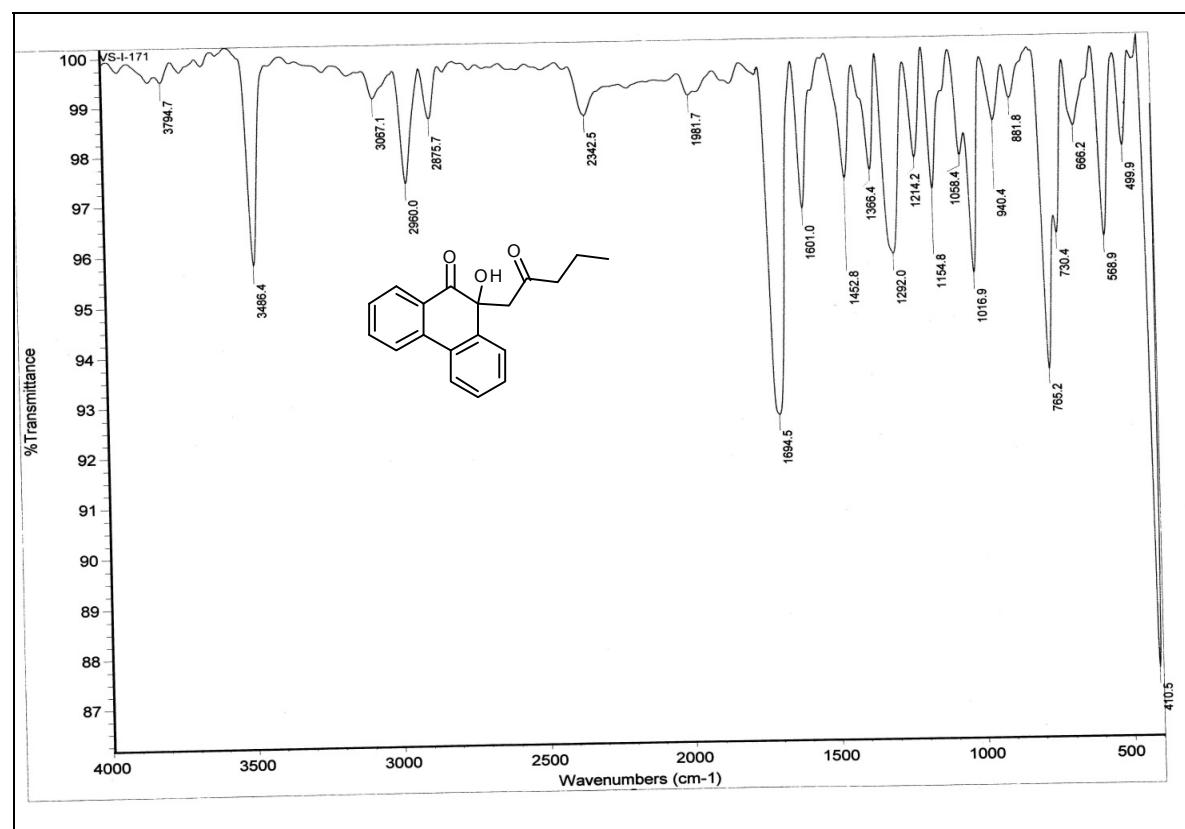


B) 10-Hydroxy-10-(2-oxopentyl)phenanthren-9(10H)-one **2b**.

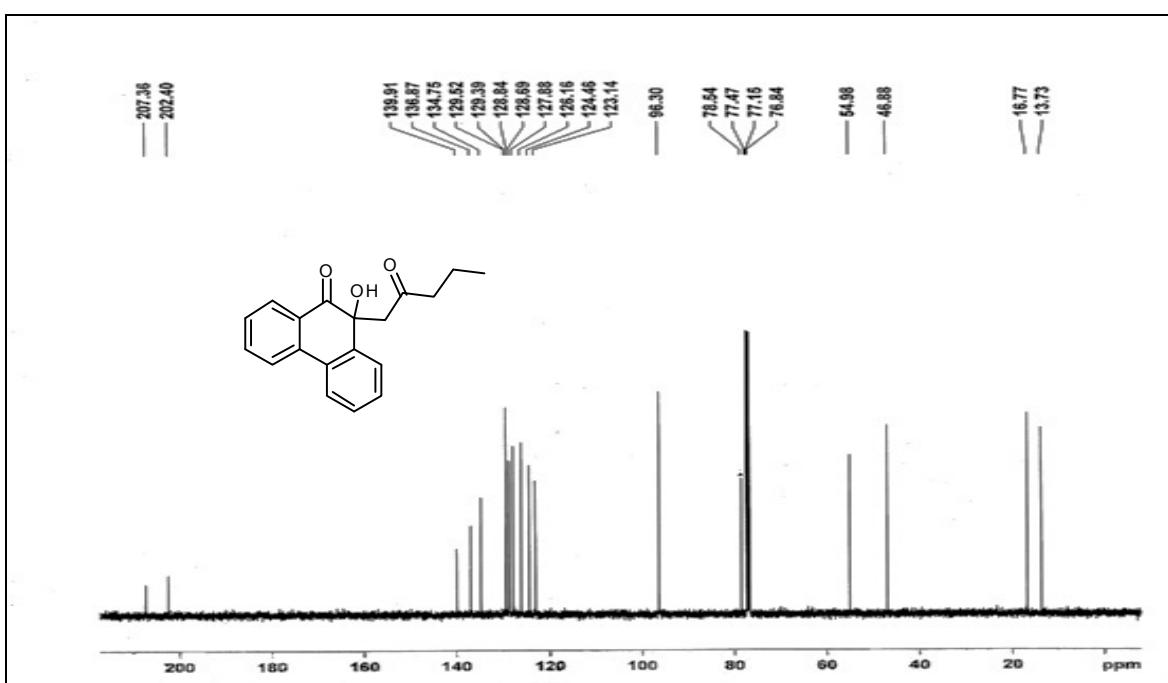
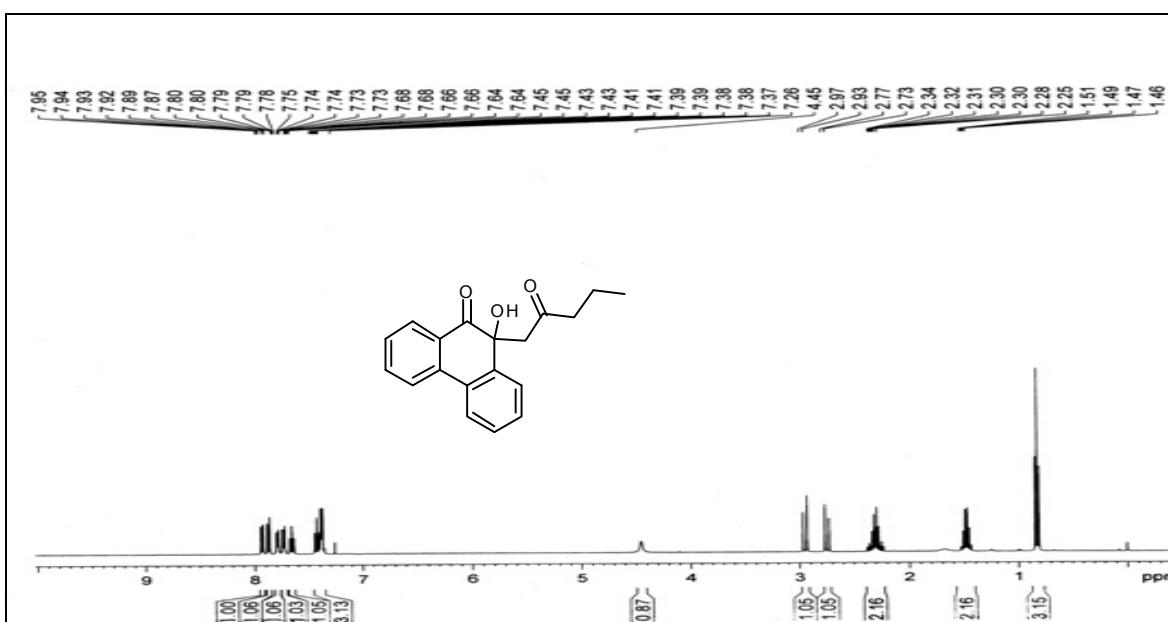


2b

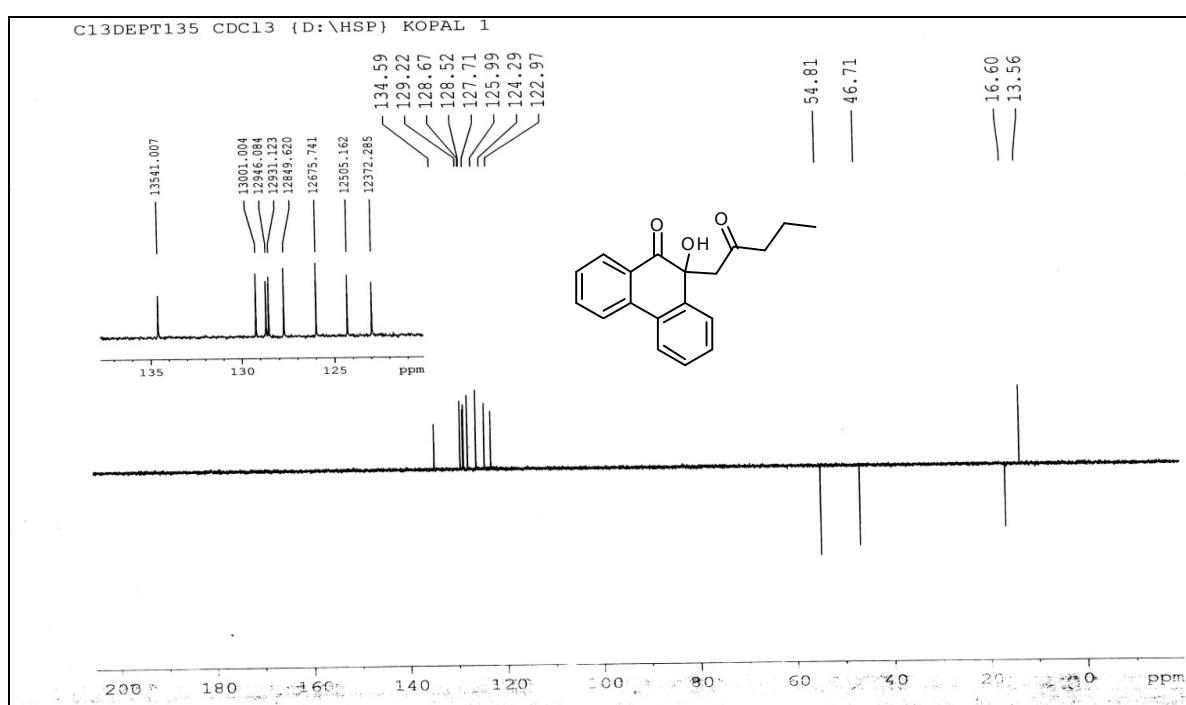
IR spectrum of **2b**.



¹H (400 MHz, CCl₄:CDCl₃, 1:1) and ¹³C (100 MHz, CCl₄:CDCl₃, 1:1) NMR spectra of **2b**.



DEPT-135 NMR Spectra of **2b**.



ESIMS HRMS of **2b**.

Elemental Composition Report

Single Mass Analysis

Tolerance = 200.0 mDa / DBE: min = -1.5, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

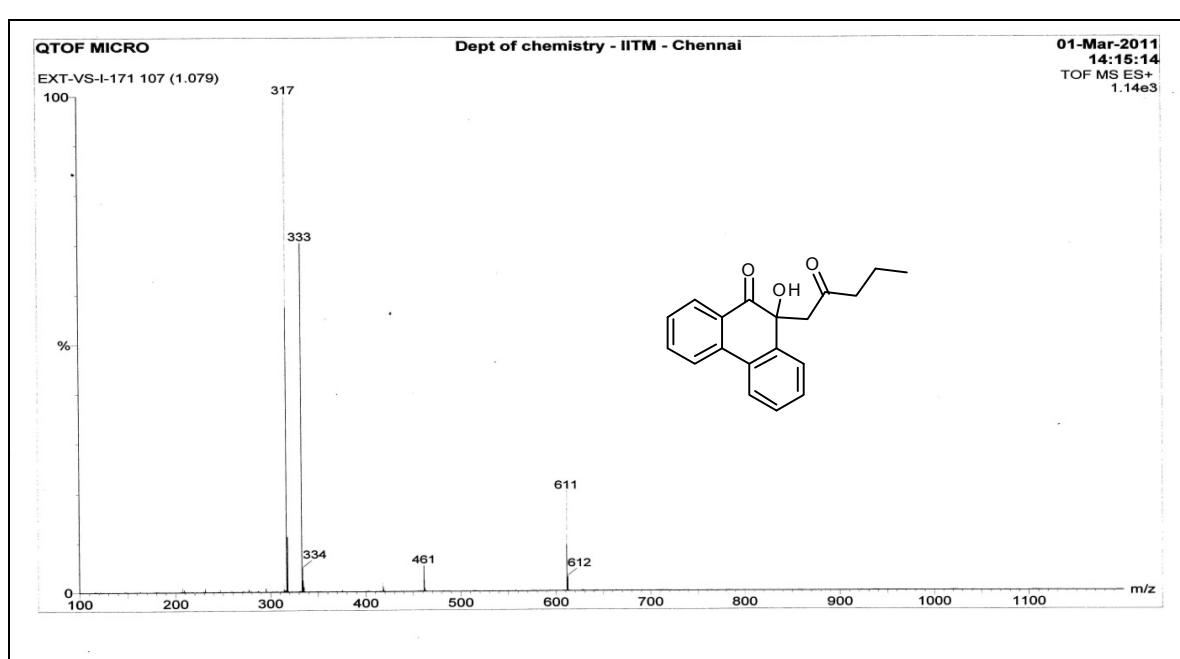
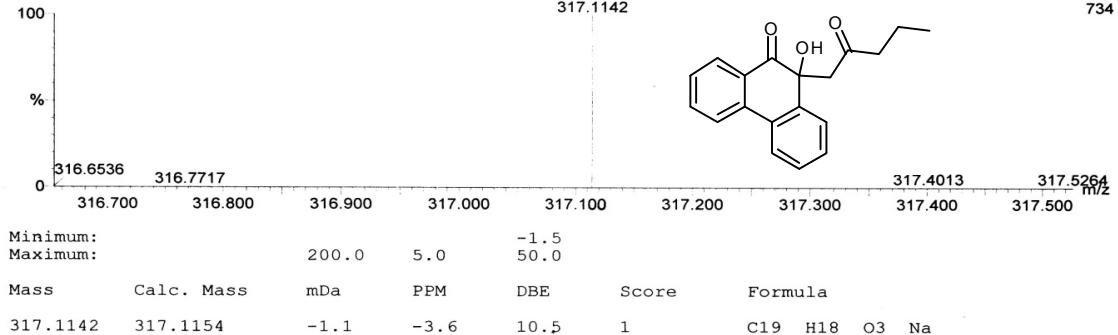
QTOF MICRO

Dept of chemistry - IITM - Chennai

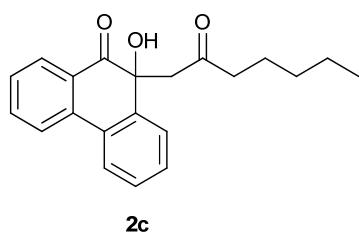
01-Mar-2011

01-Mar-2011
14:17:46

TOF MS ES+
724

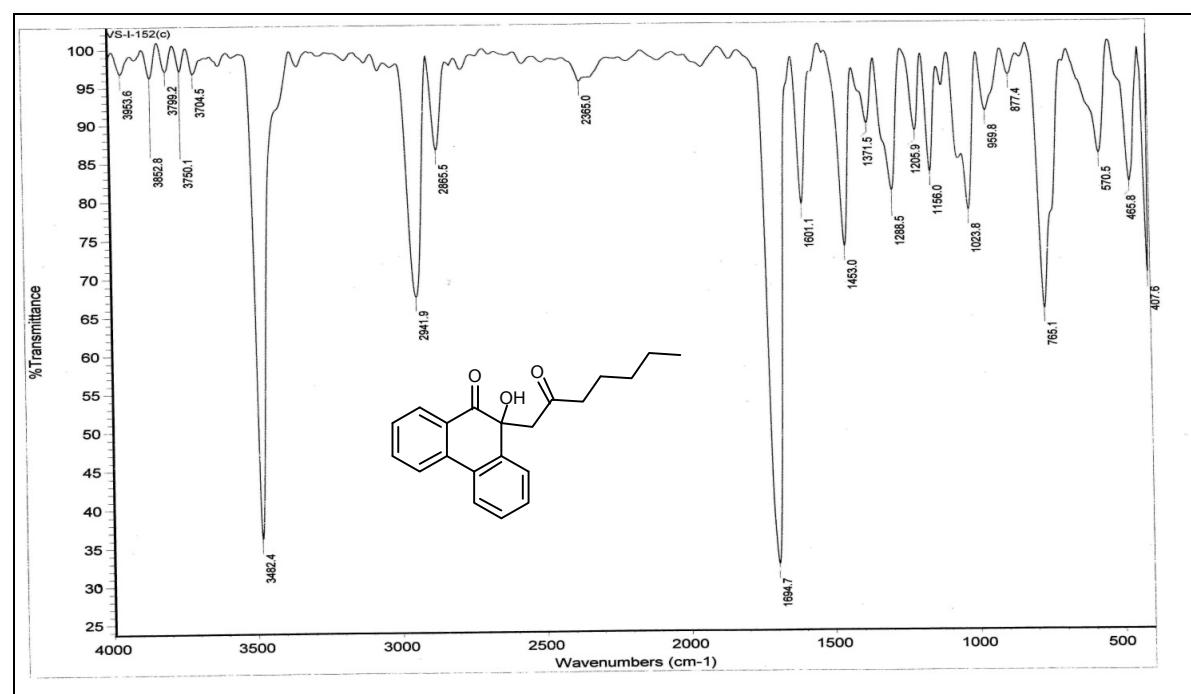


C) 10-Hydroxy-10-(2-oxoheptyl)phenanthren-9(10H)-one **2c**.

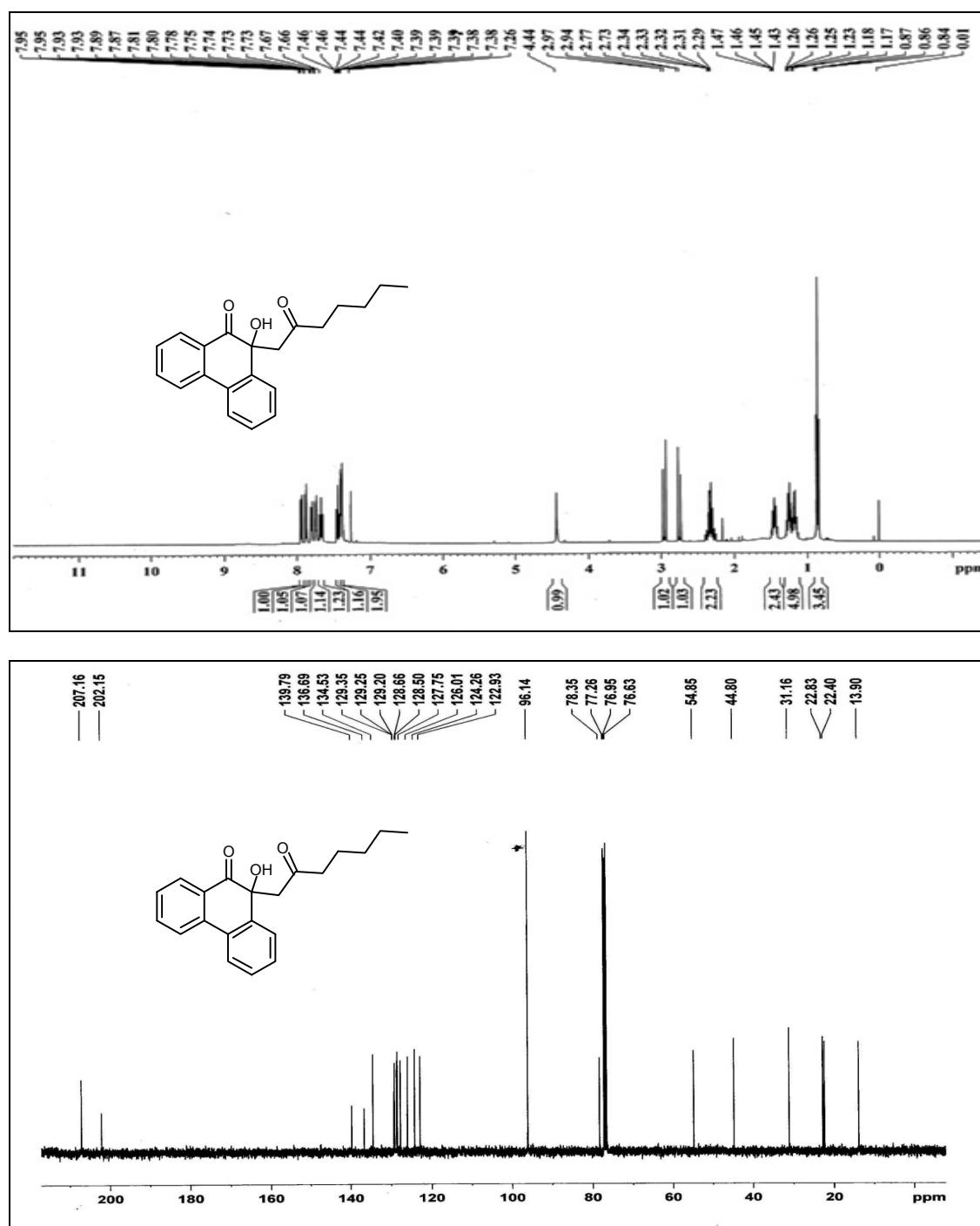


2c

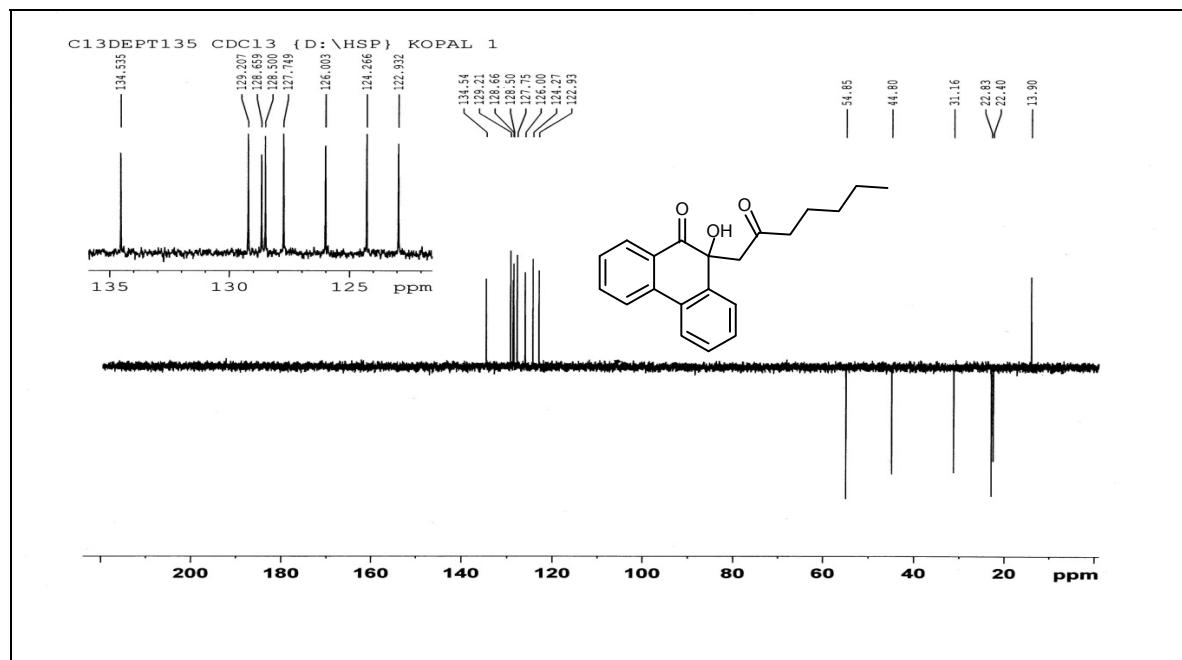
IR spectrum of **2c**.



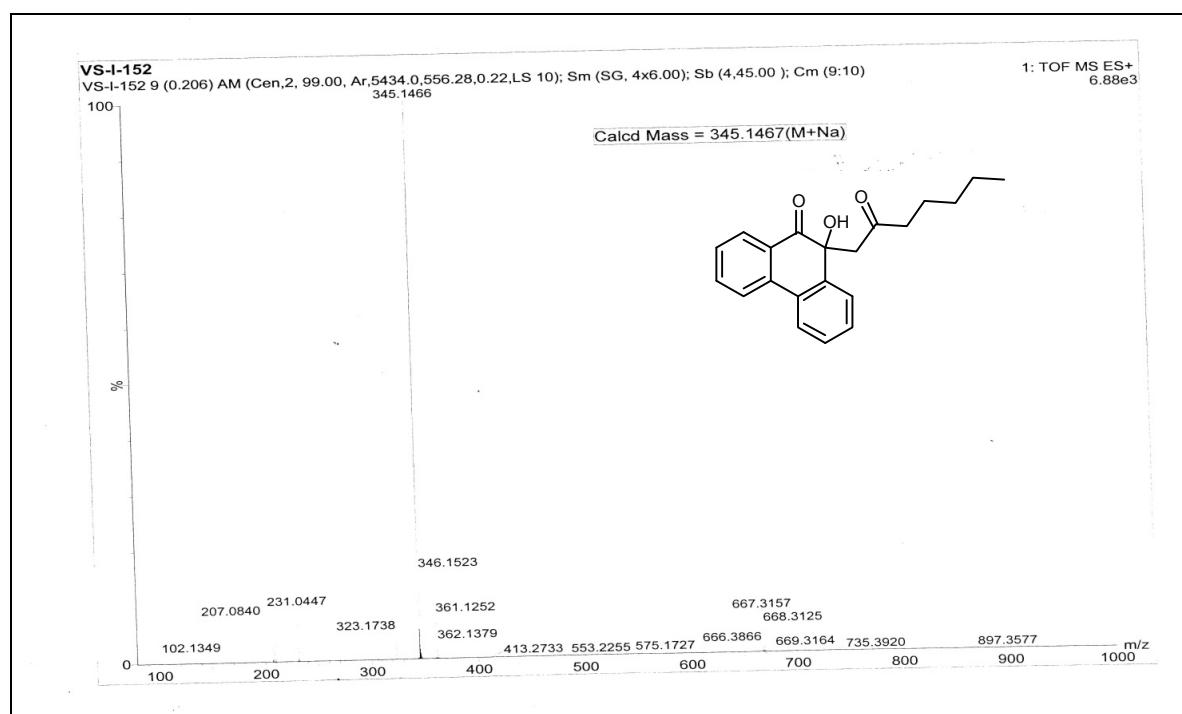
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) NMR spectra of **2c**.



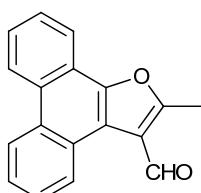
DEPT-135 NMR spectrum of **2c**.



ESIMS HRMS of **2c**.

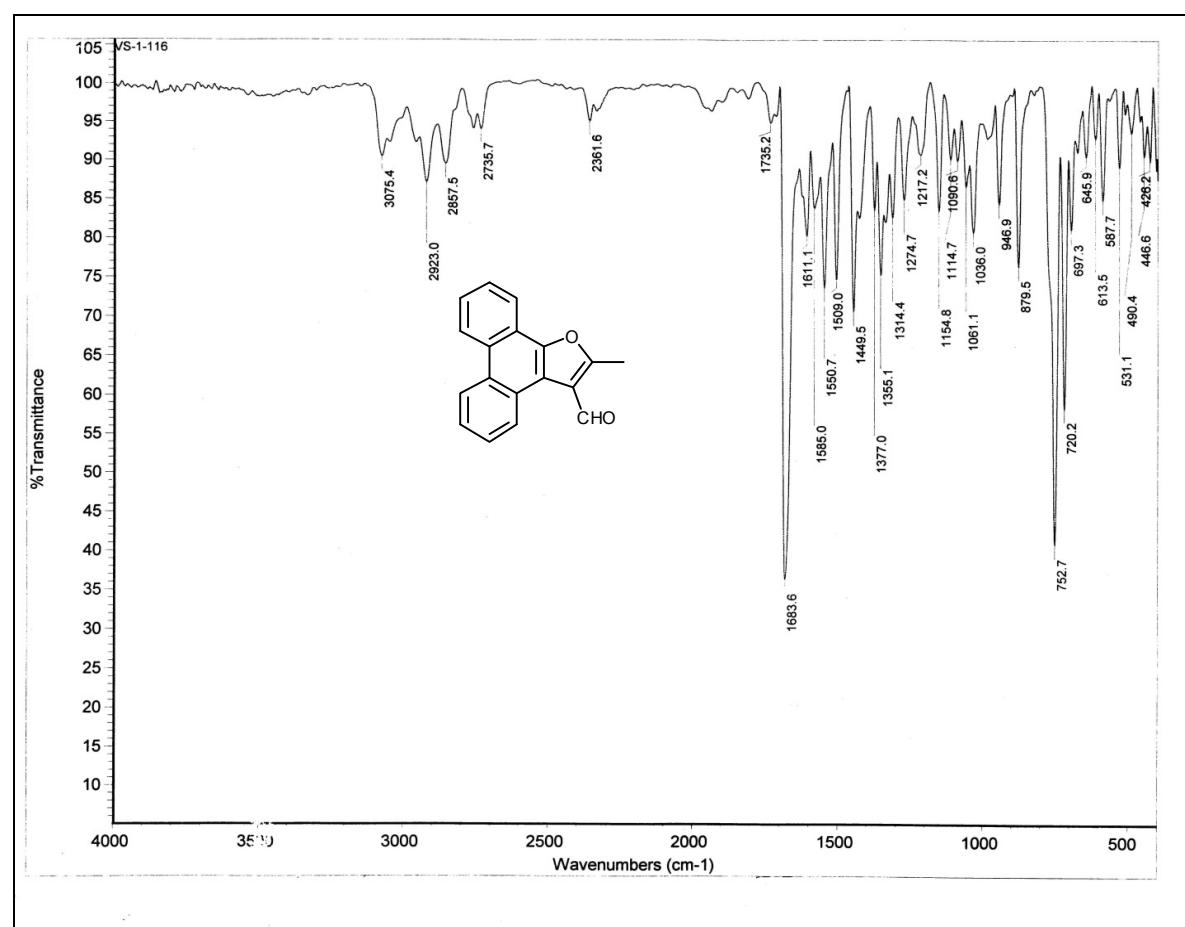


D) 2-Methylphenanthro[9,10-*b*]furan-3-carbaldehyde **9a**.

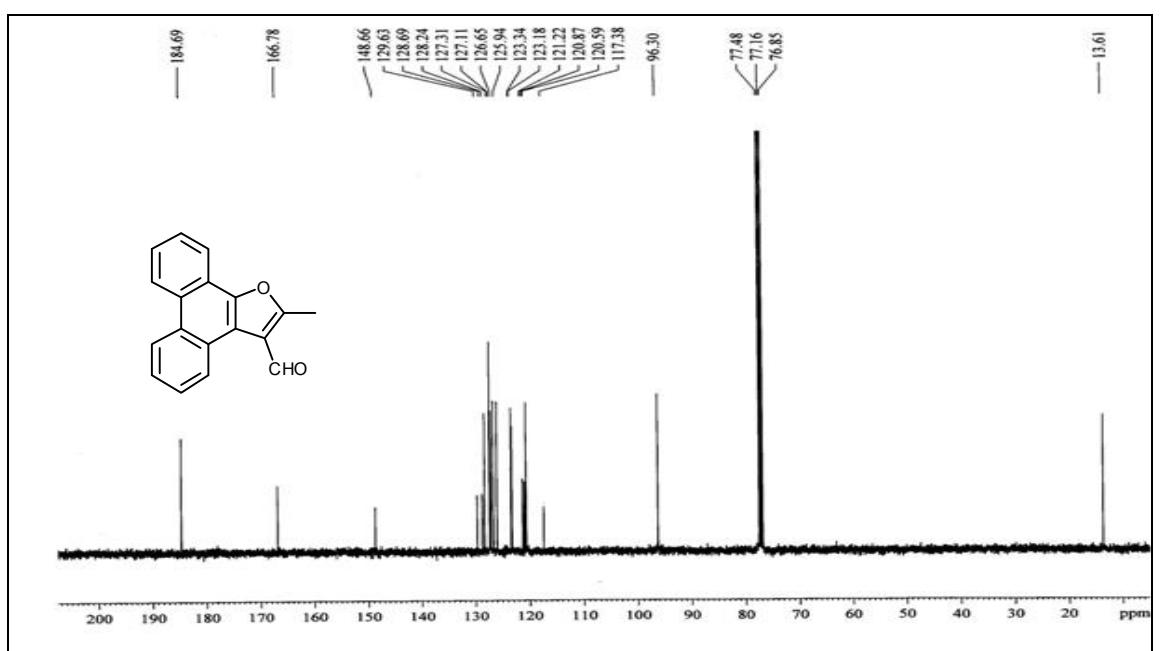
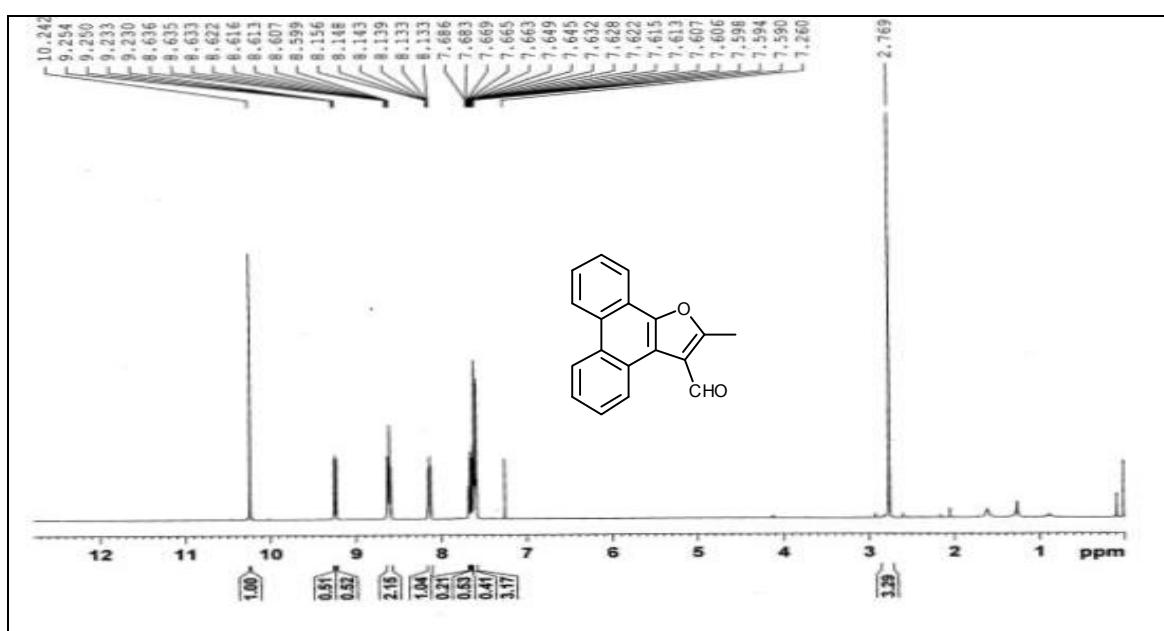


9a

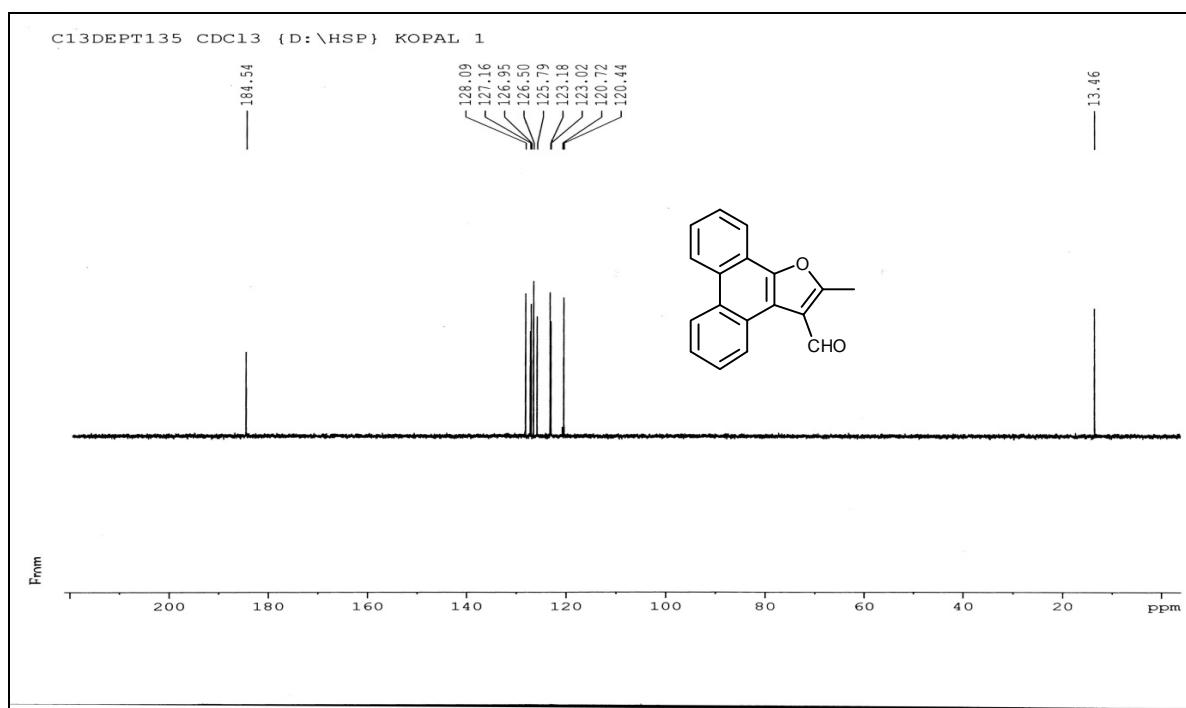
IR spectrum of **9a**.



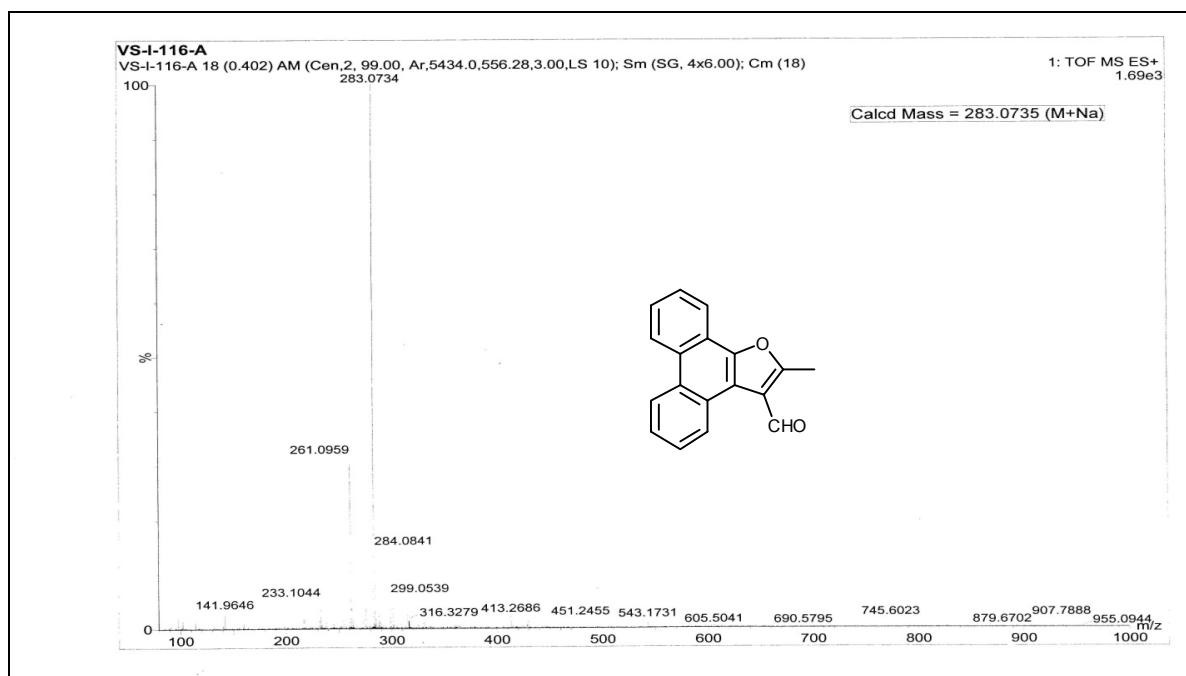
¹H (400 MHz, CCl₄:CDCl₃, 1:1) and ¹³C (100 MHz, CCl₄:CDCl₃, 1:1) NMR spectra of **9a**.



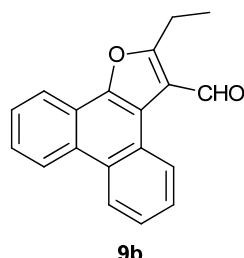
DEPT-135 NMR spectra of **9a**.



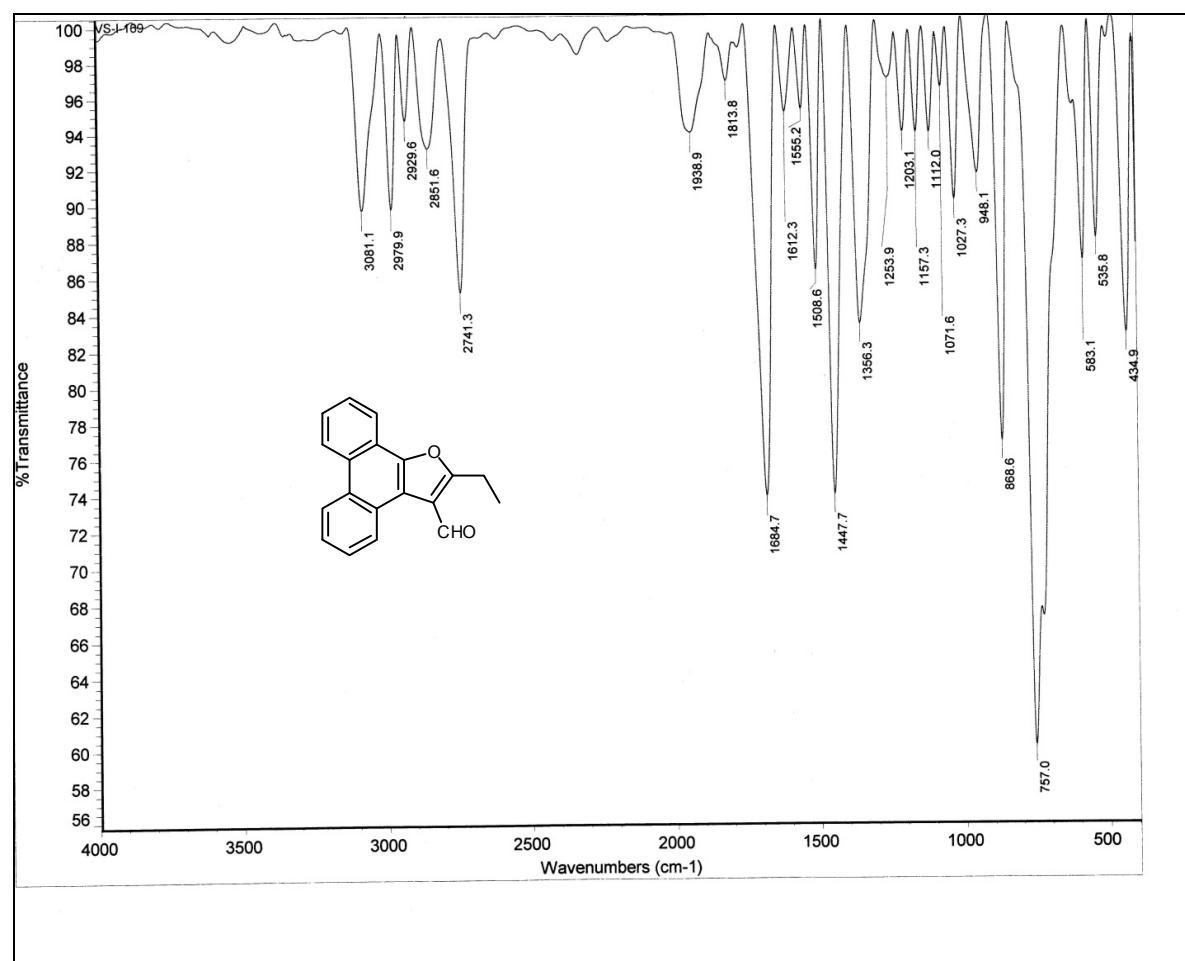
ESIMS HRMS of **9a**.



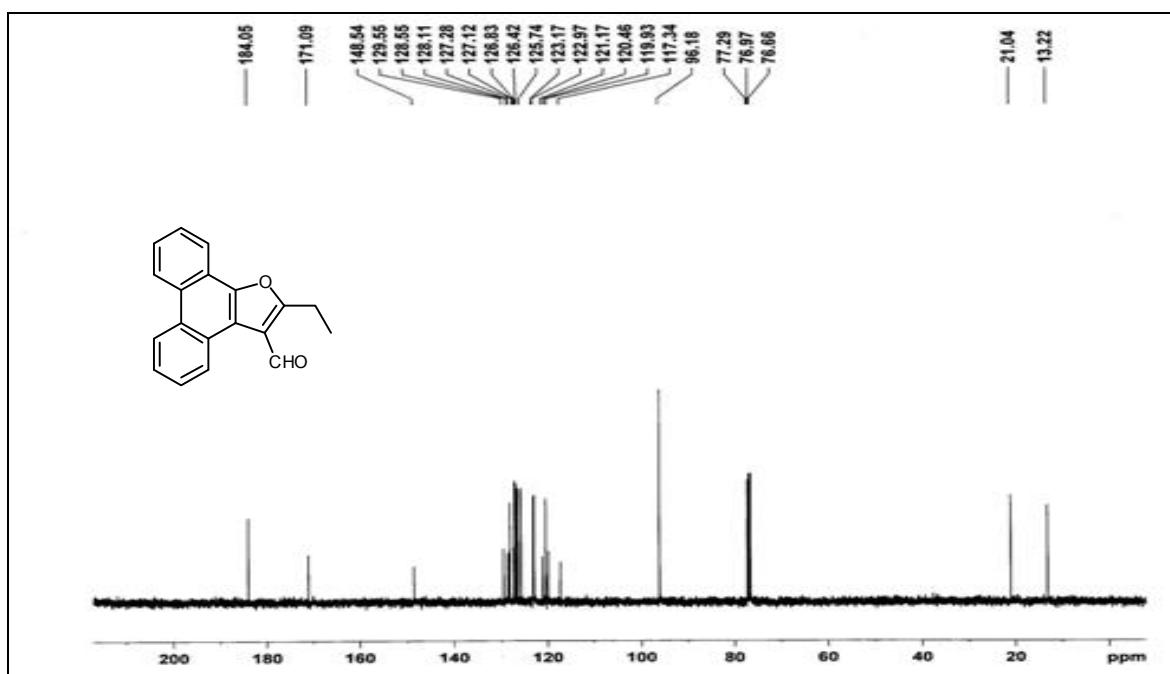
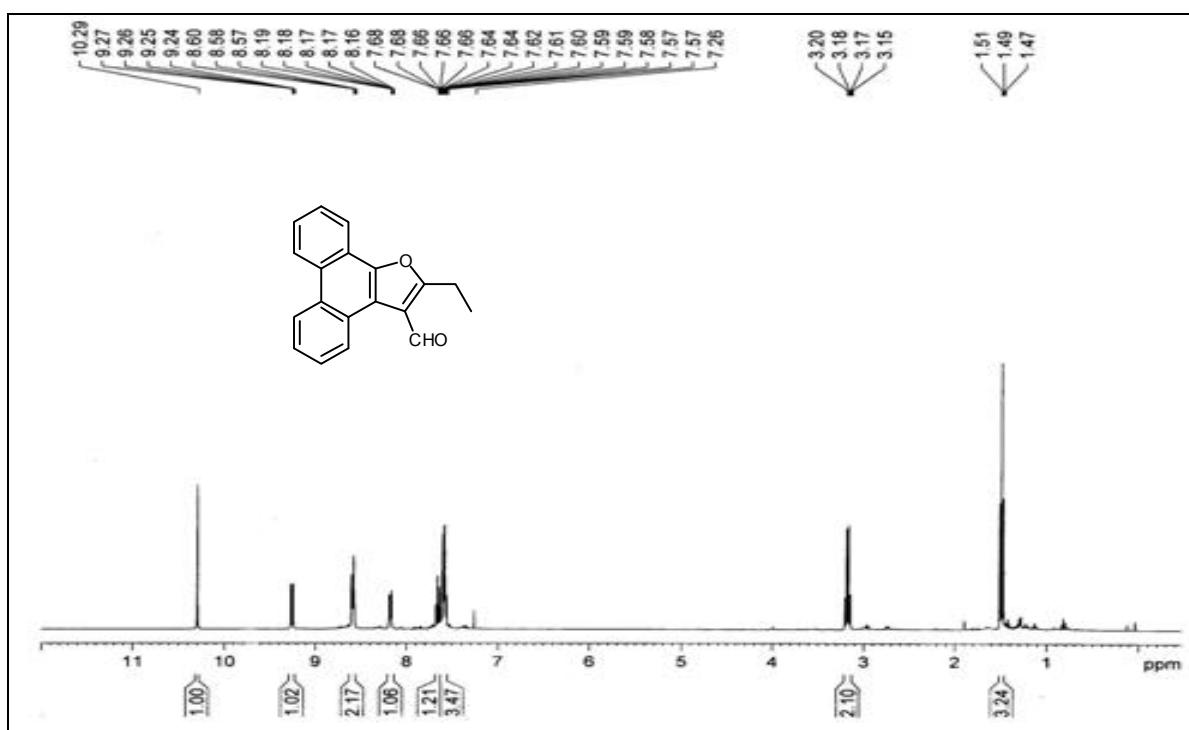
E) 2-Ethylphenanthro[9,10-*b*]furan-3-carbaldehyde **9b**.



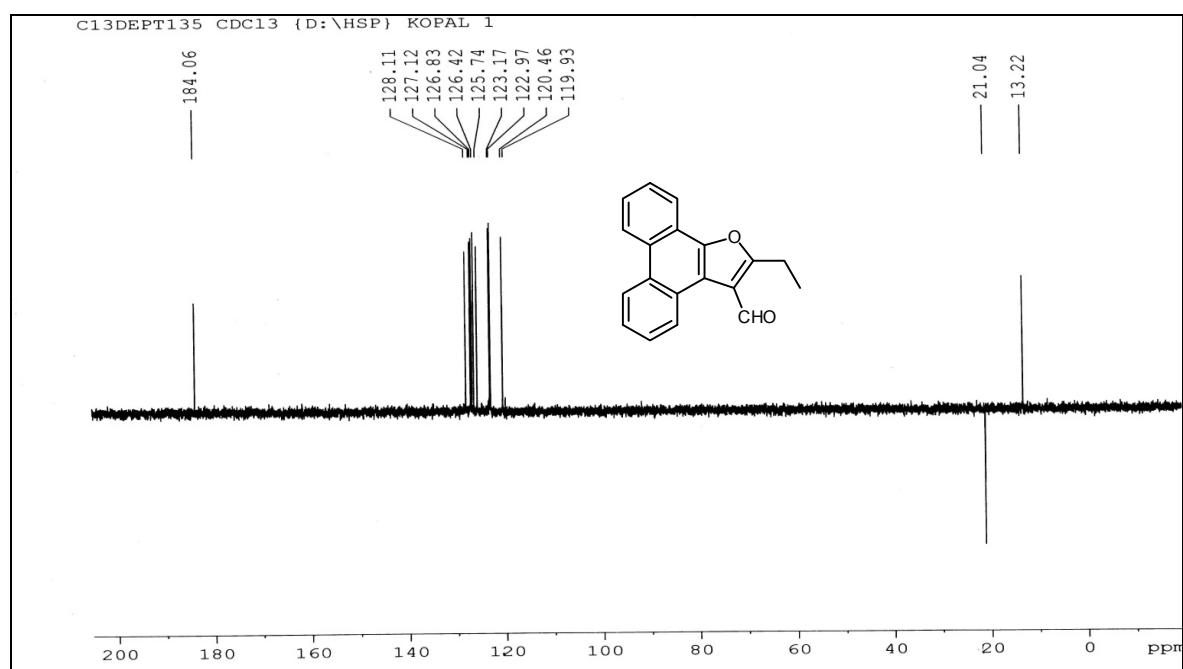
IR spectrum of **9b**.



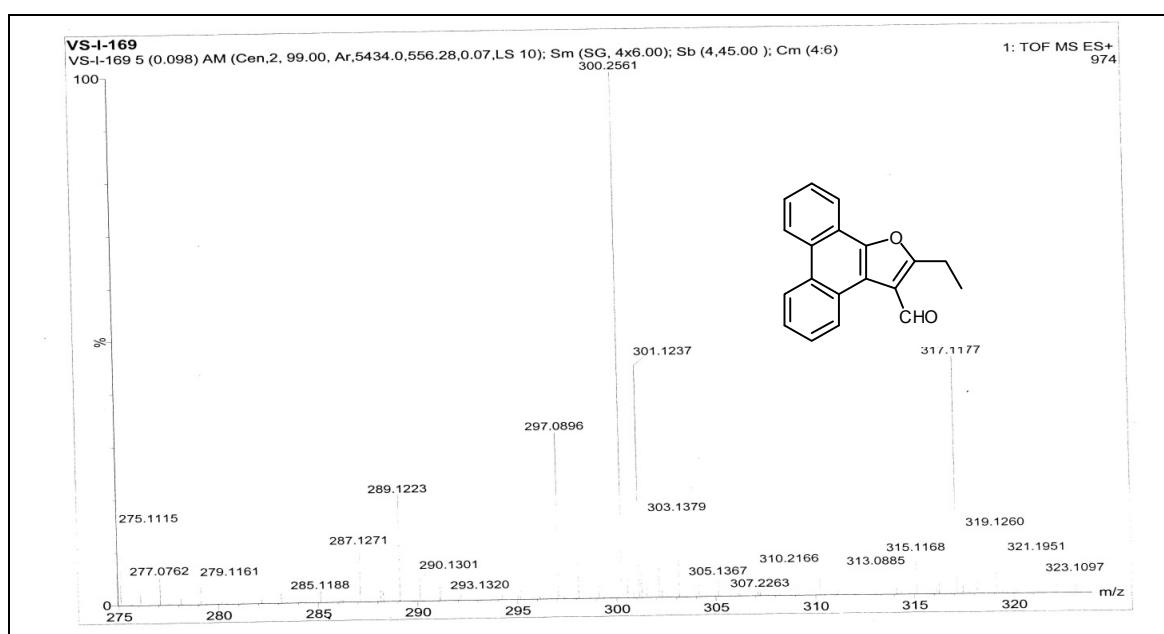
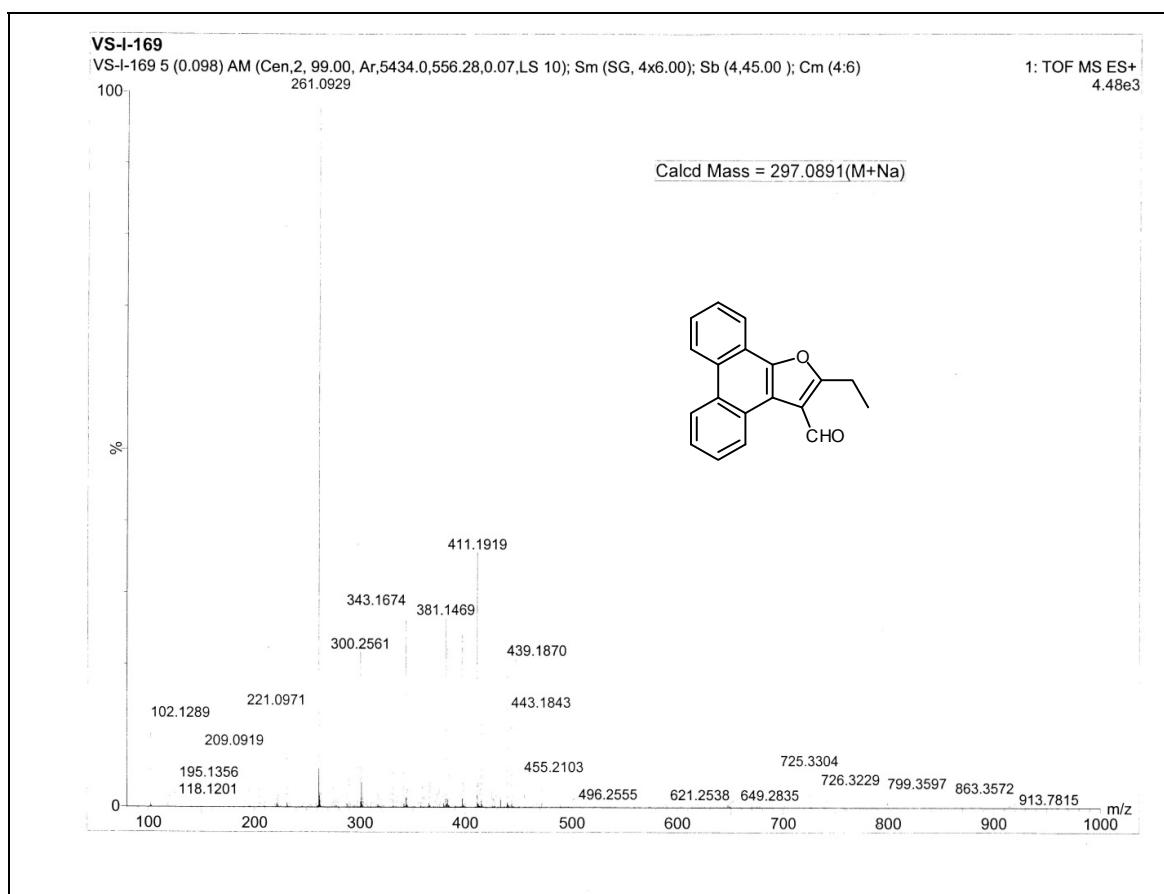
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) NMR spectra of **9b**.



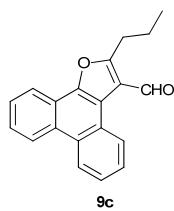
DEPT-135 spectrum of **9b**.



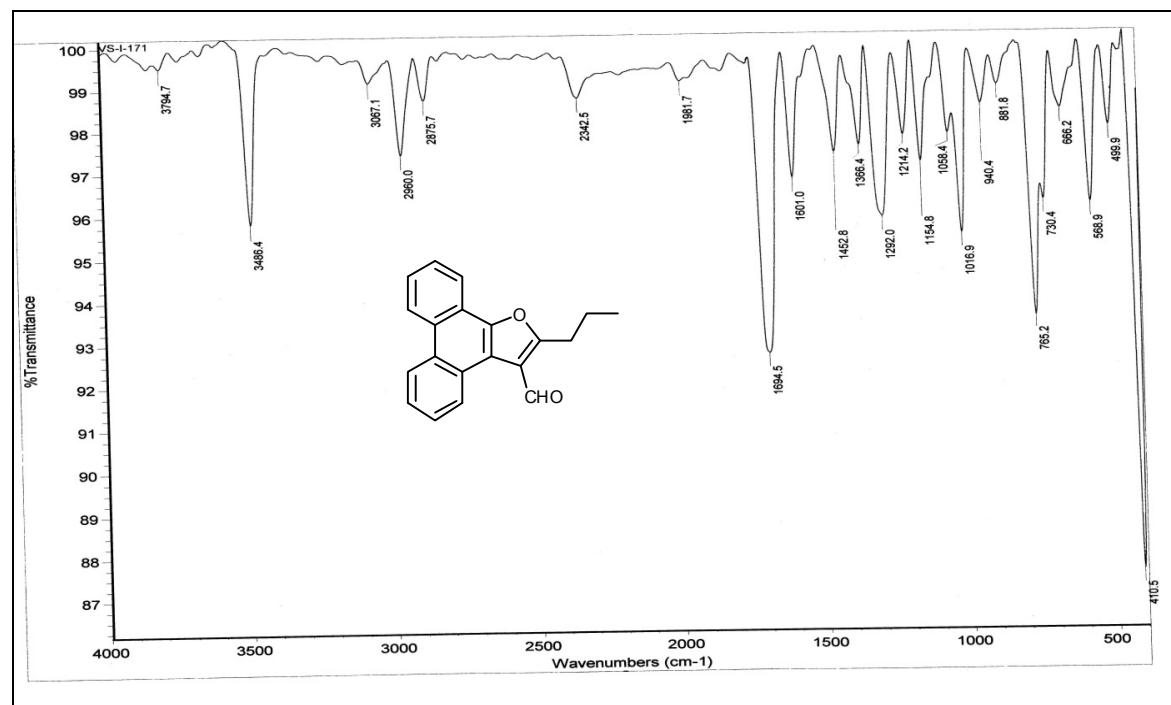
ESIMS HRMS of **9b**.



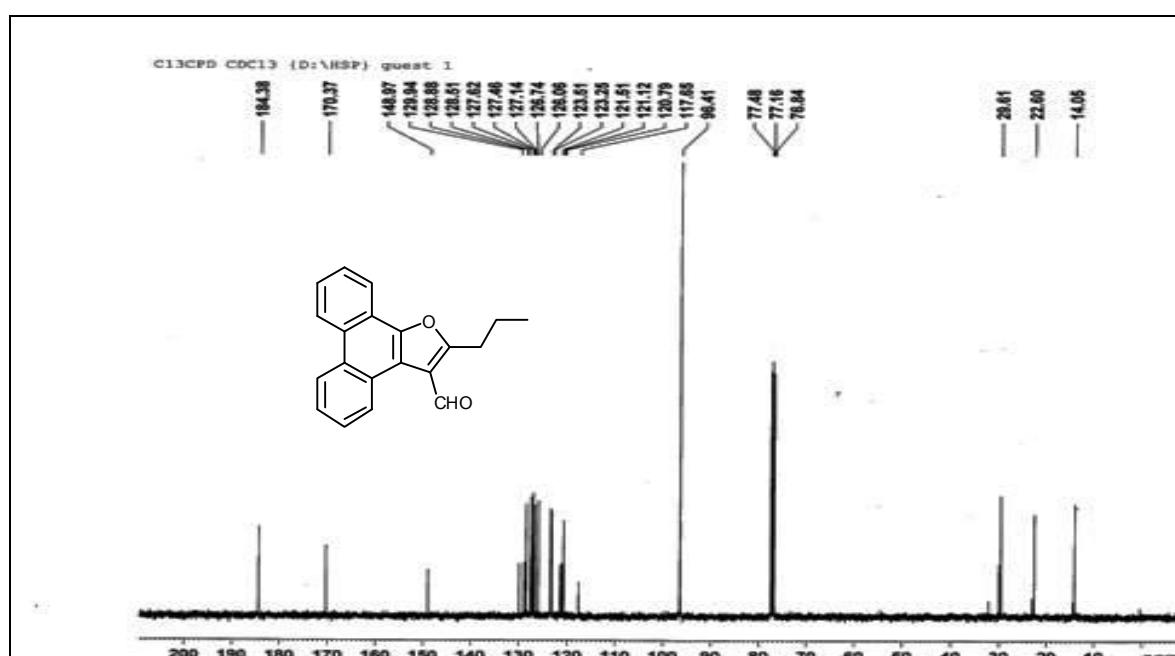
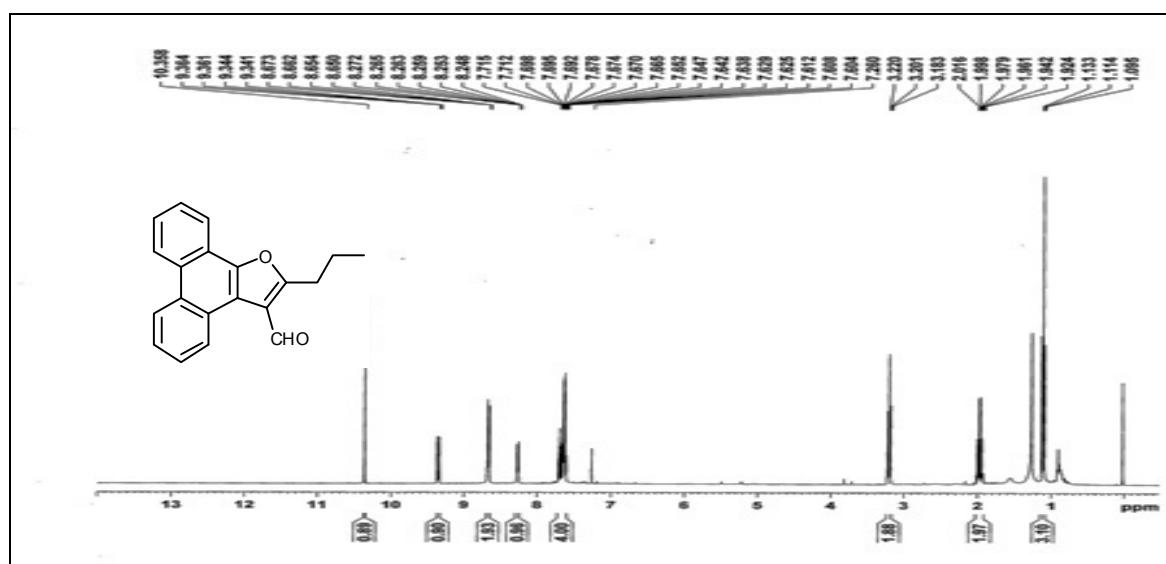
F) 2-Propylphenanthro[9,10-*b*]furan-3-carbaldehyde **9c**.



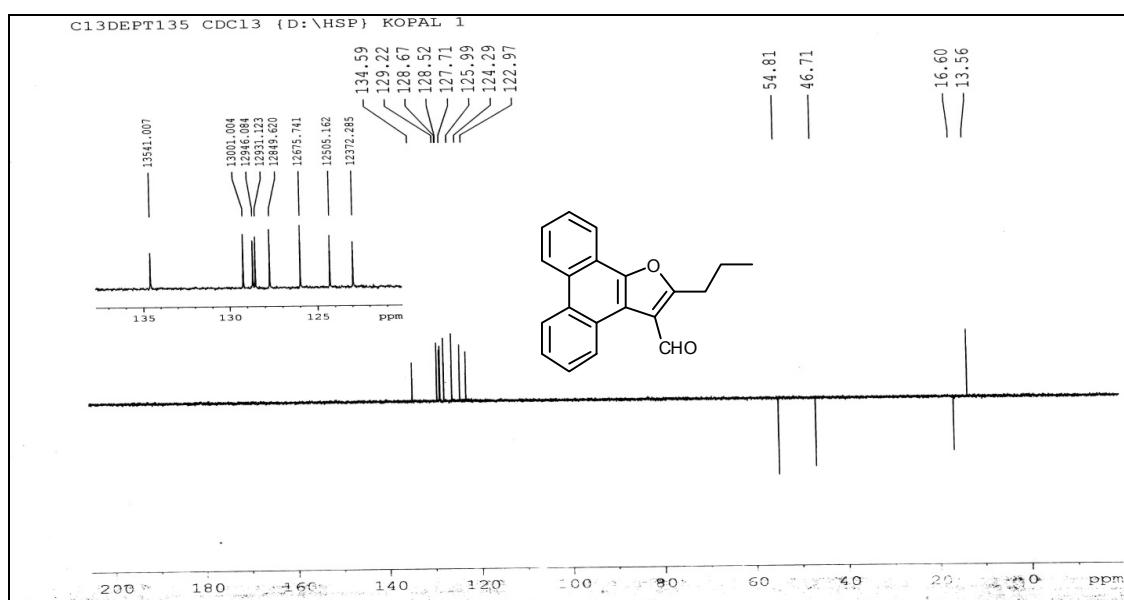
IR spectra of **9c**.



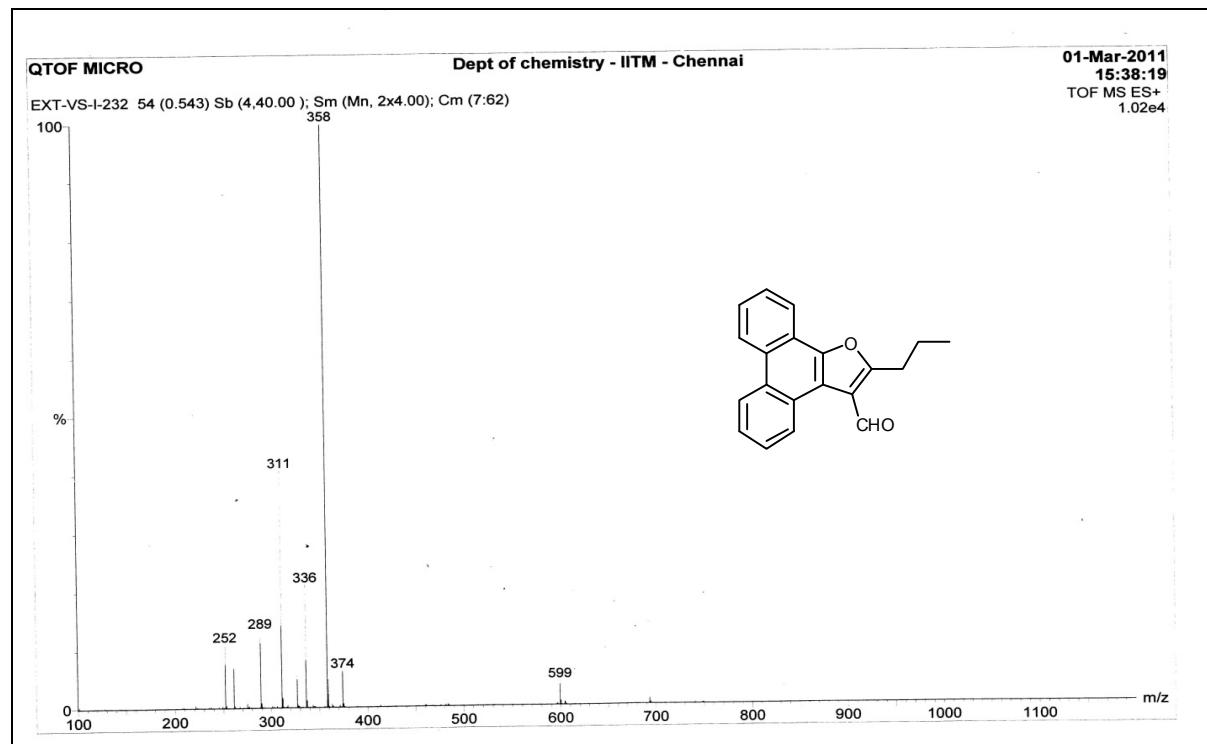
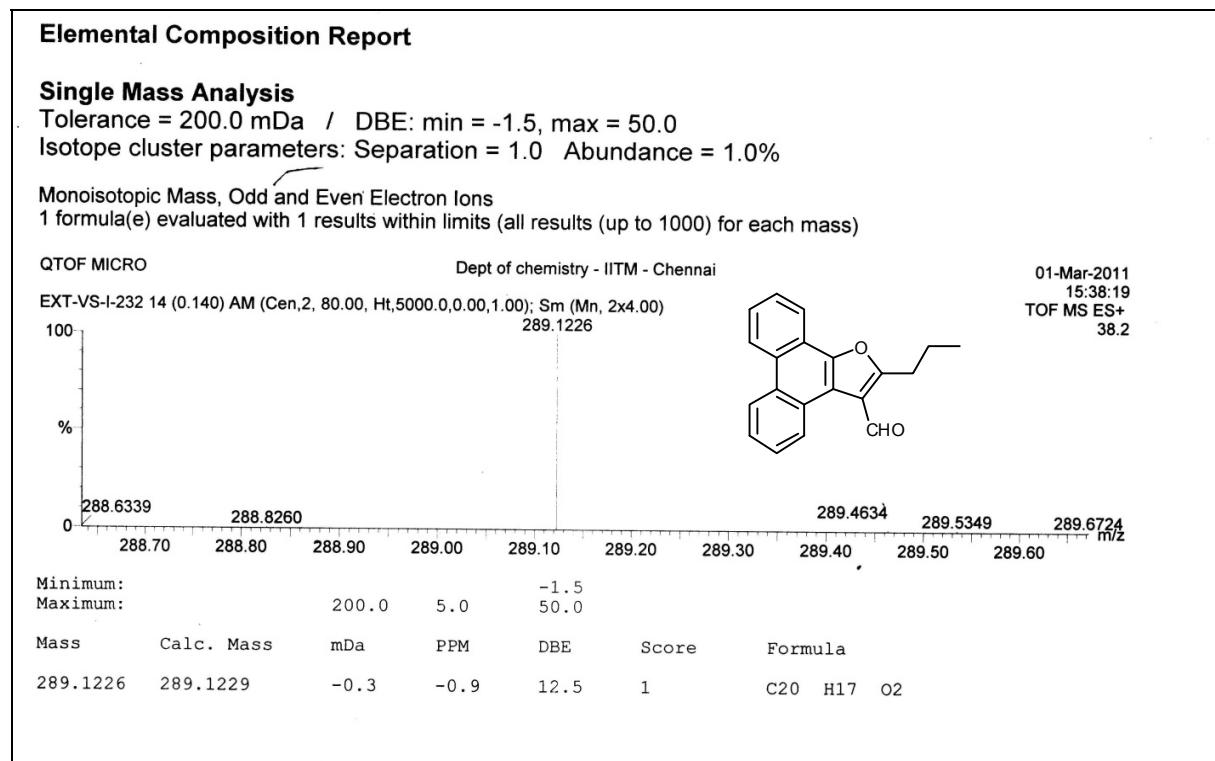
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) NMR spectra of **9c**.



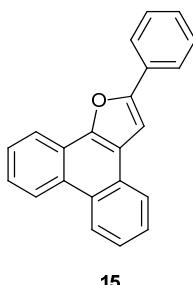
DEPT-135 NMR spectrum of **9c**.



ESIMS HRMS of 9c.

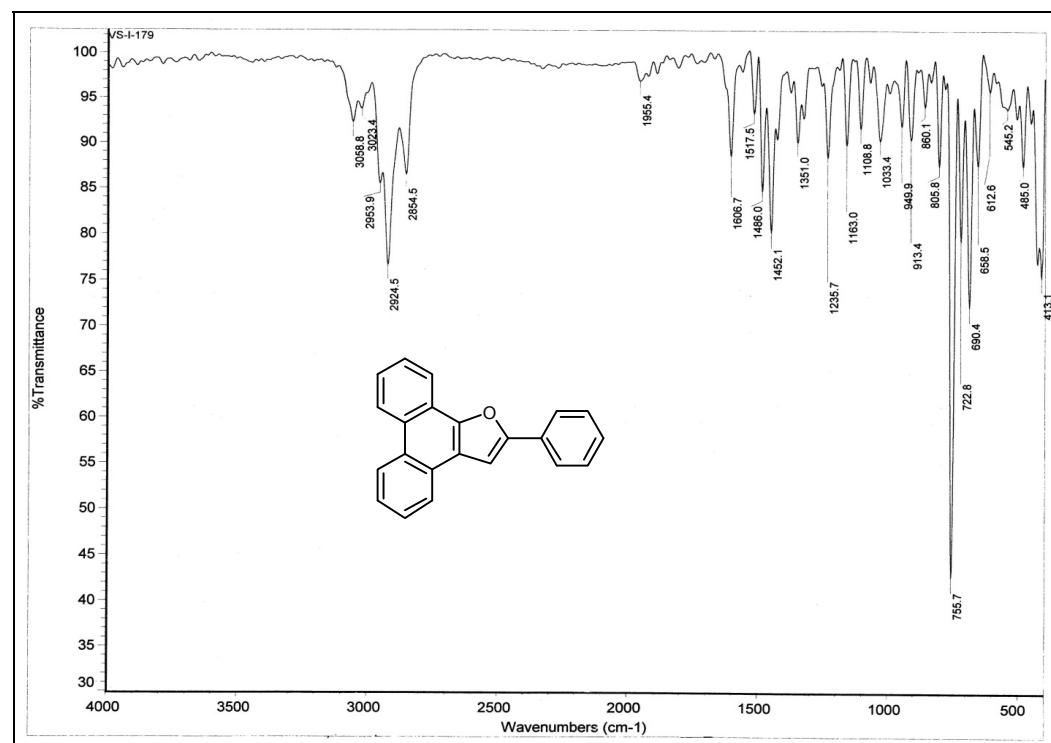


G) 2-Phenylphenanthro[9,10-*b*]furan **15**.

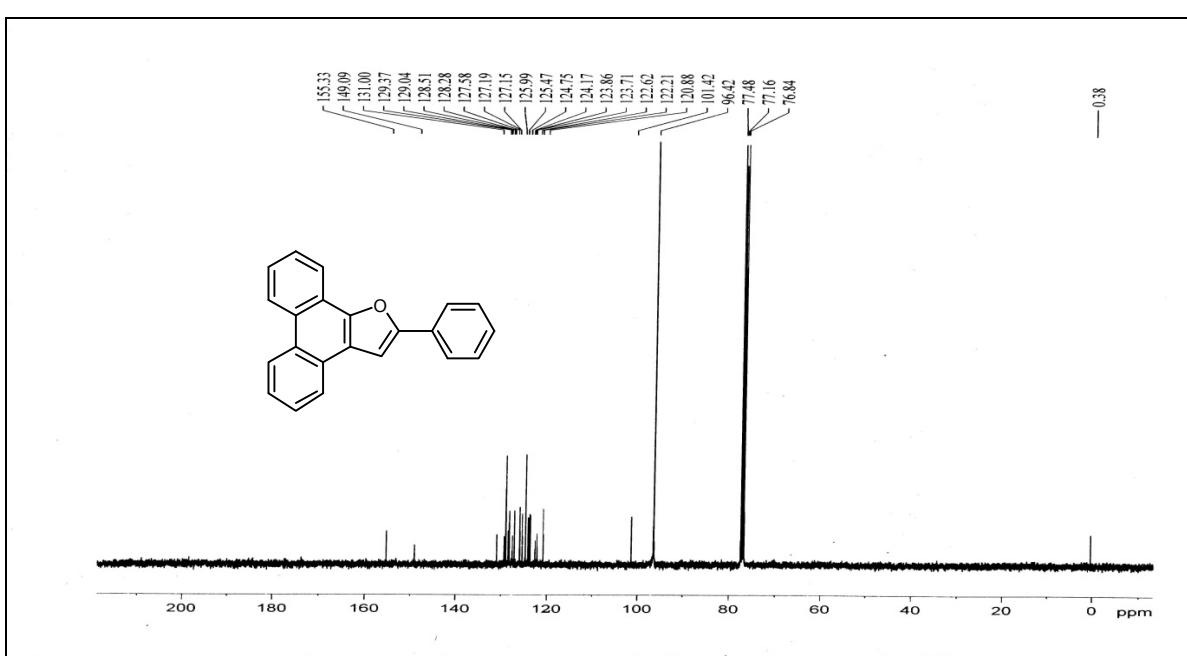
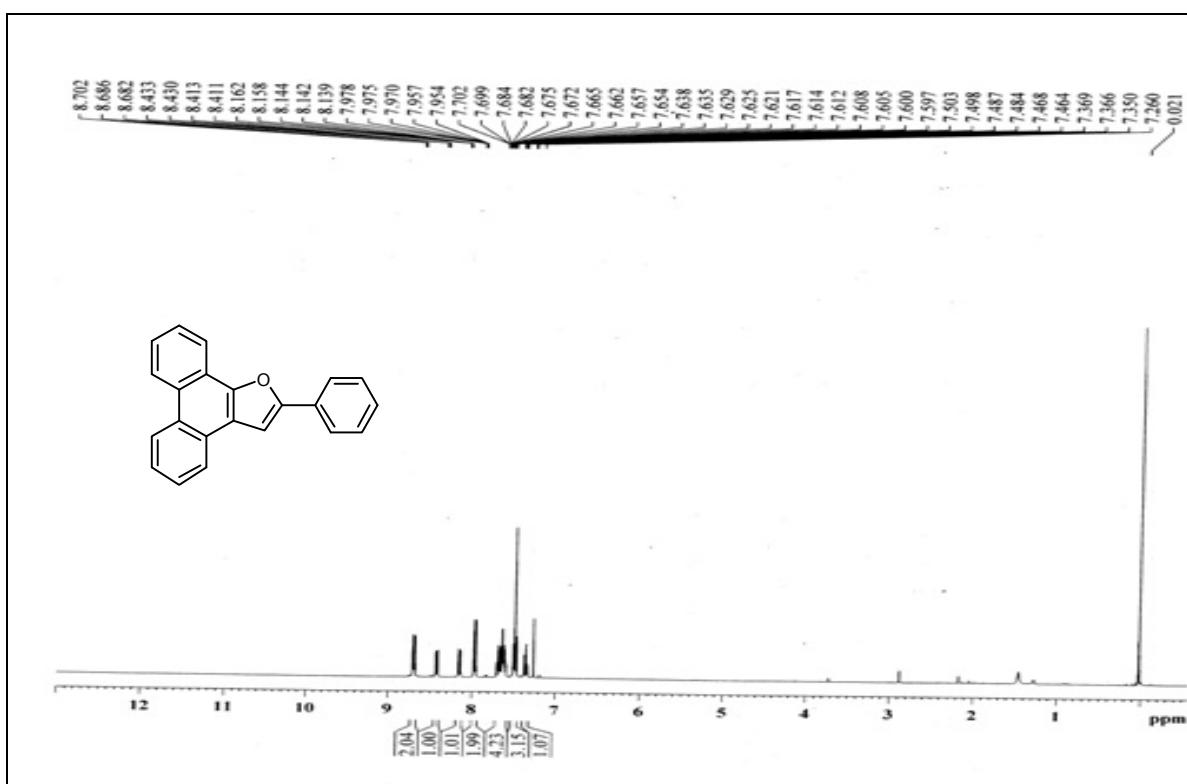


15

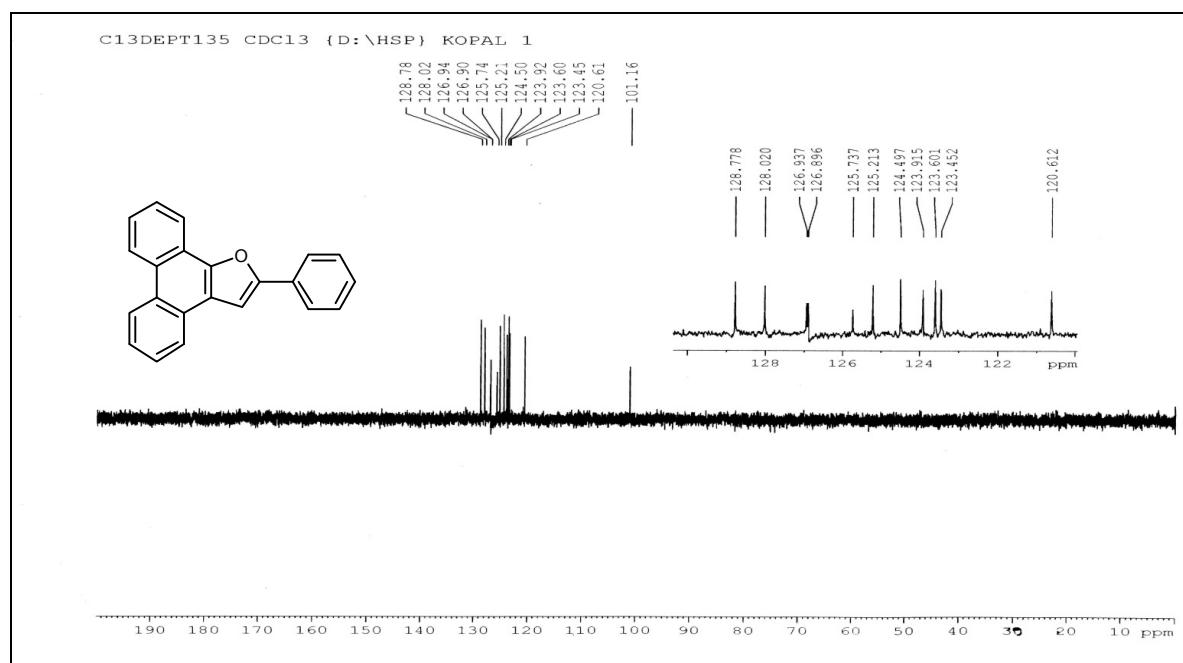
IR spectra of **15**.



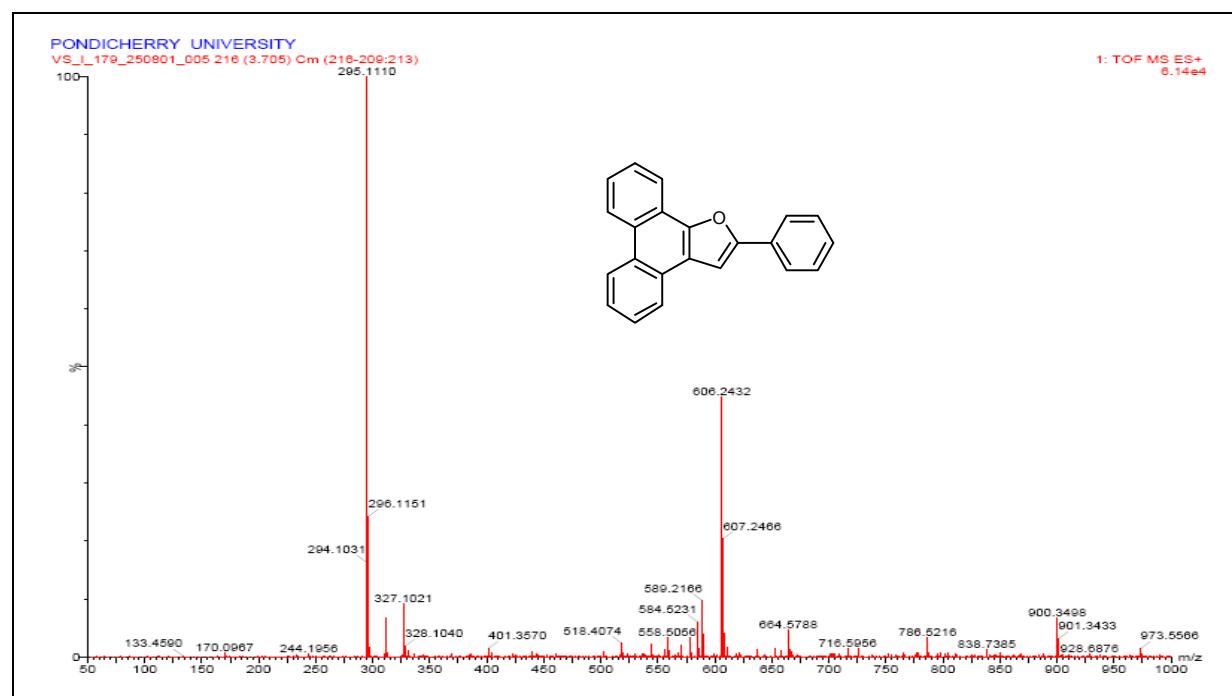
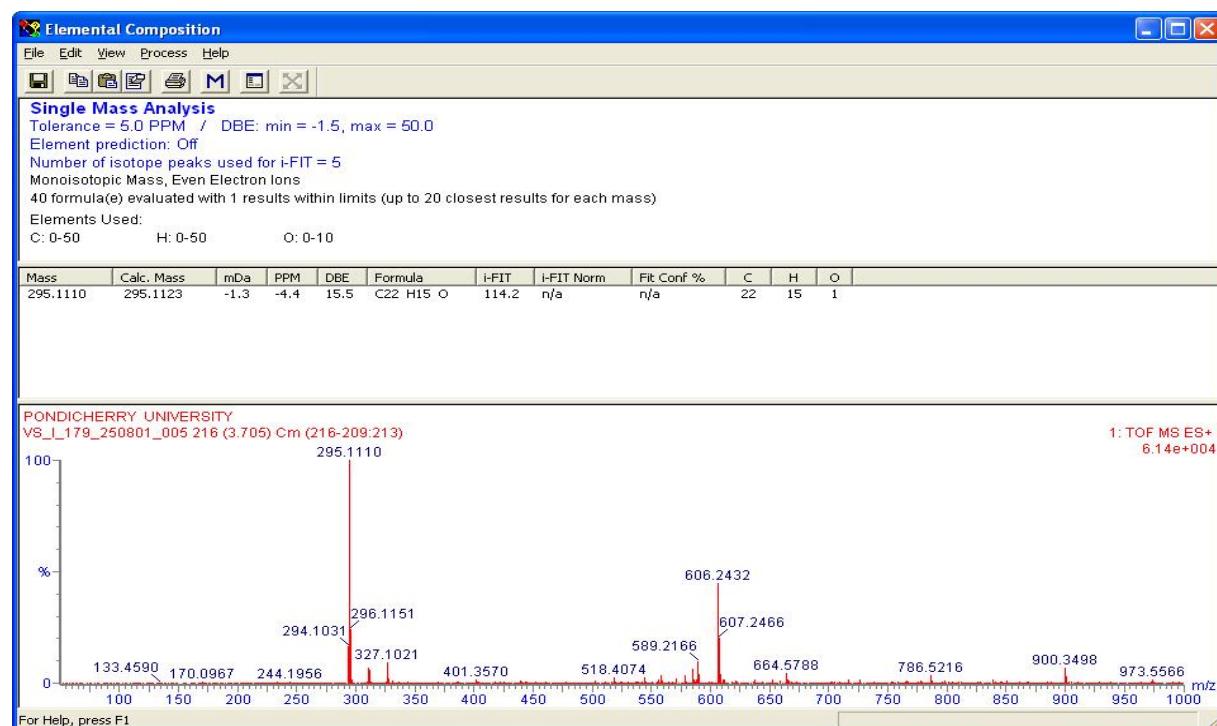
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) NMR spectra of **15**.



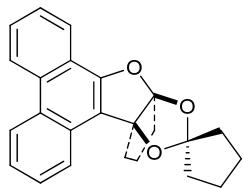
DEPT-135 NMR spectrum of **15**.



ESIMS HRMS of 15.

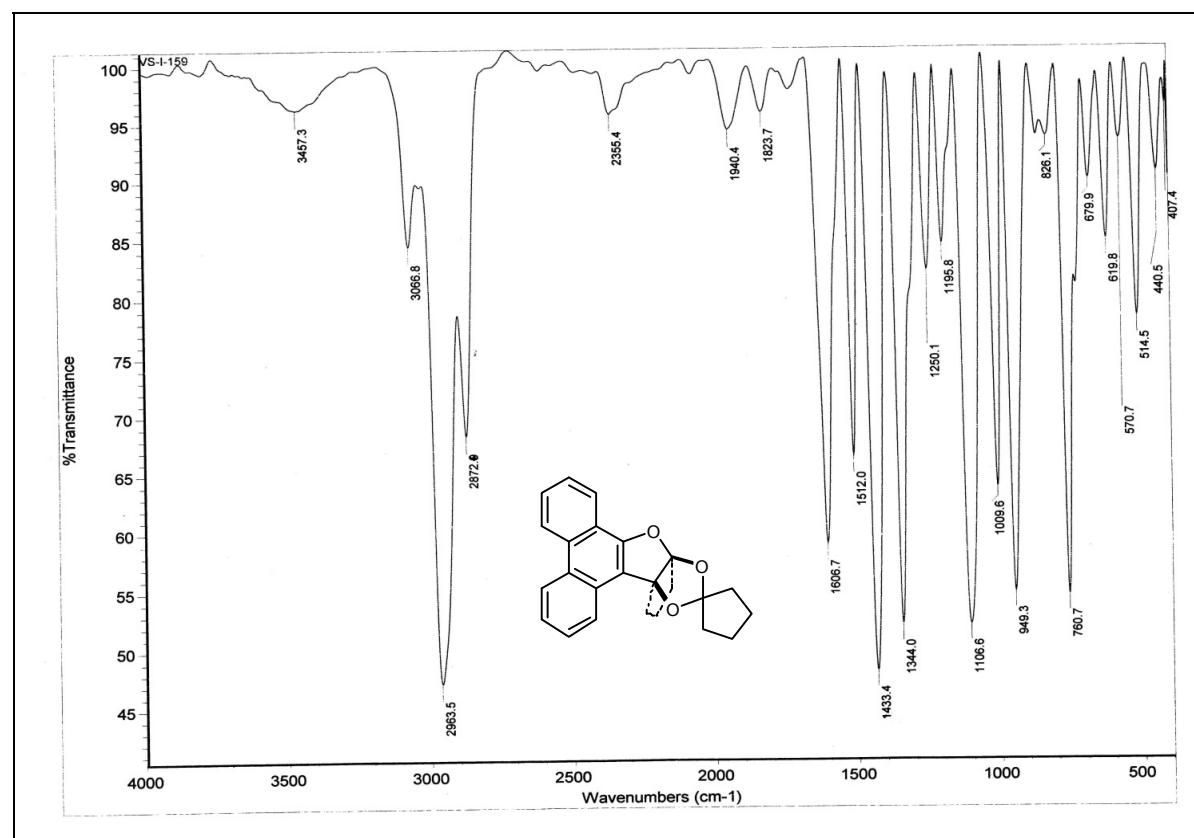


H).(9a*S*,12a*S*)-11,12-Dihydro-10*H*-spiro[9a,12a
(epoxymethanooxy)cyclopenta[*b*]phenanthro[9,10-*d*]furan-14,1'-cyclopentane] **17**.

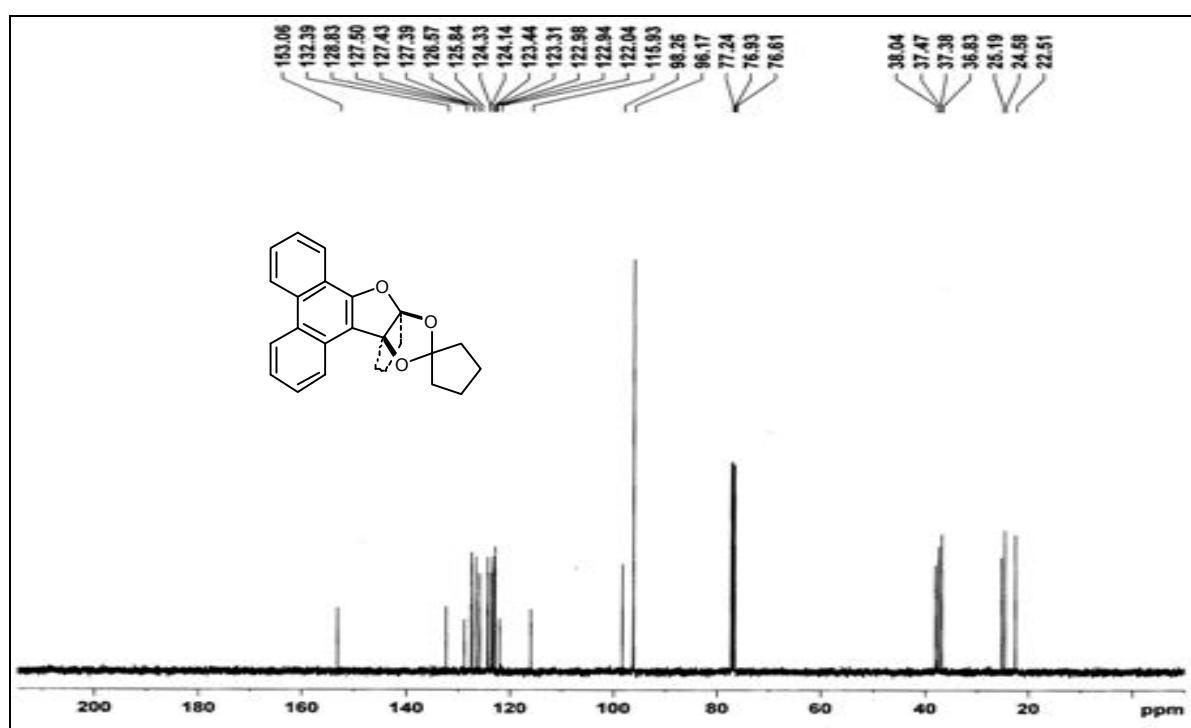
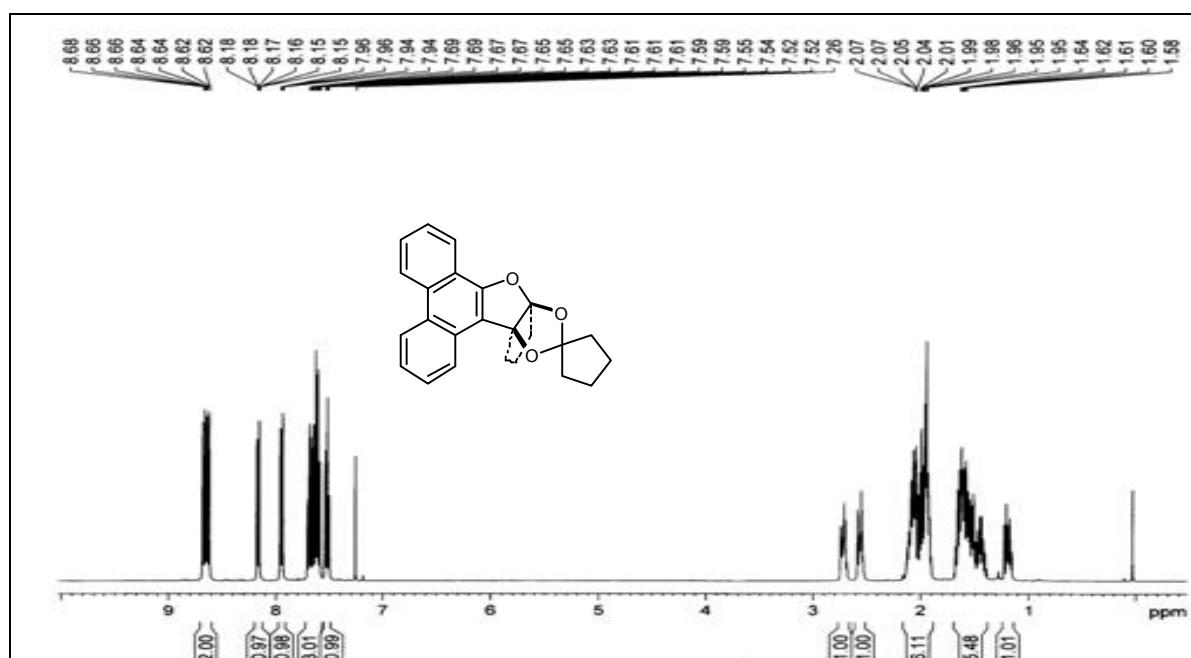


17

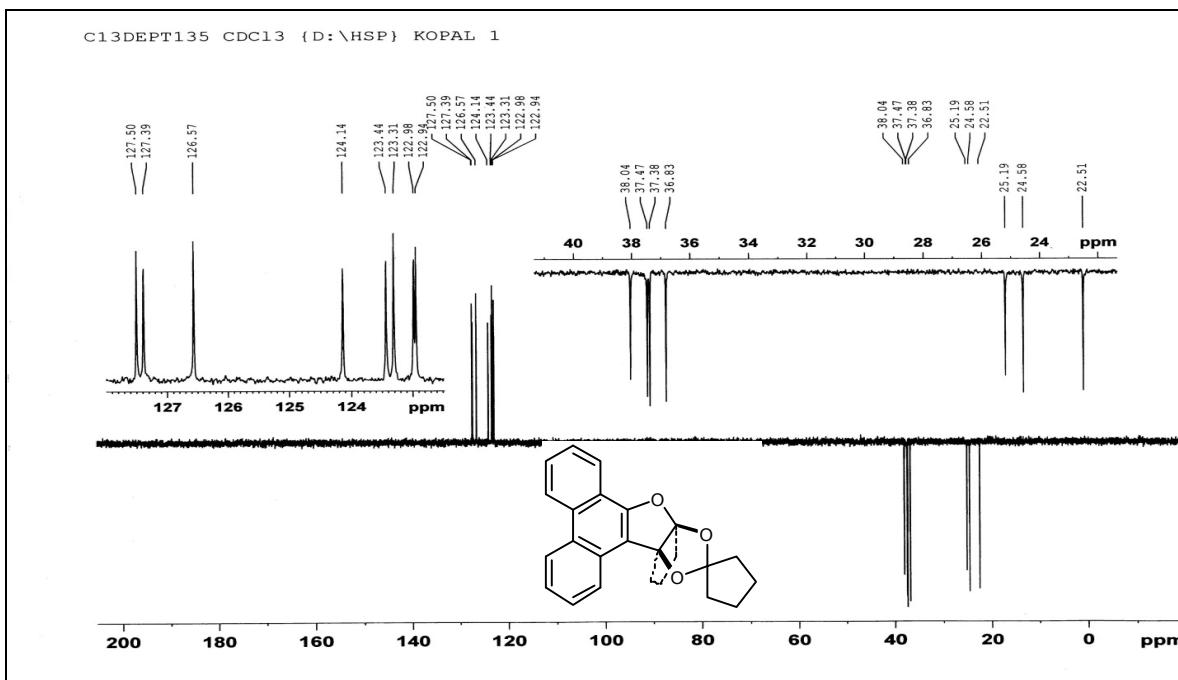
IR spectrum of **17**.



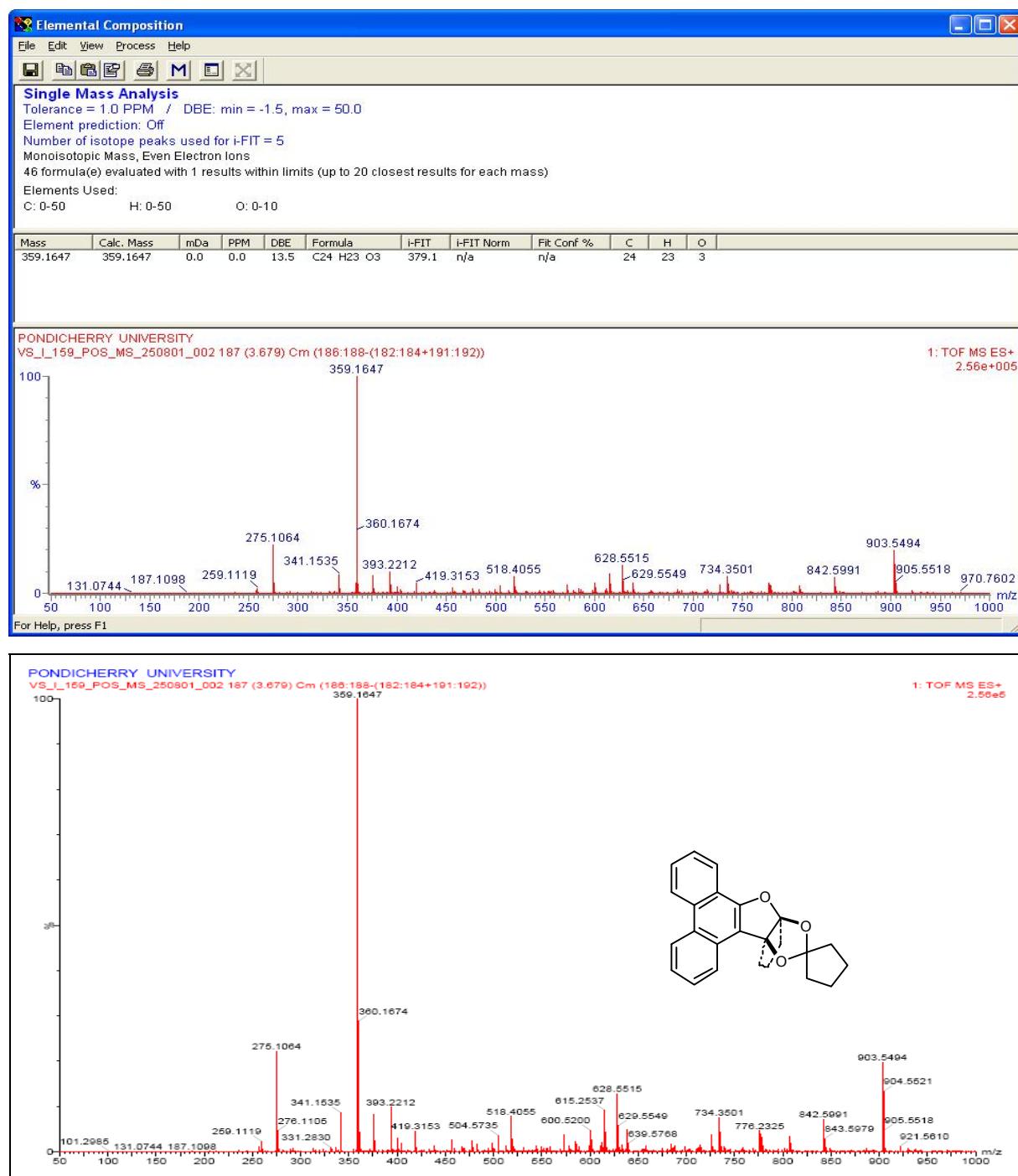
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1)NMR spectra of **17**.



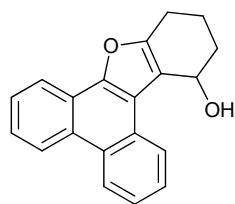
DEPT-135 NMR spectrum of **17**.



ESIMS HRMS of 17.

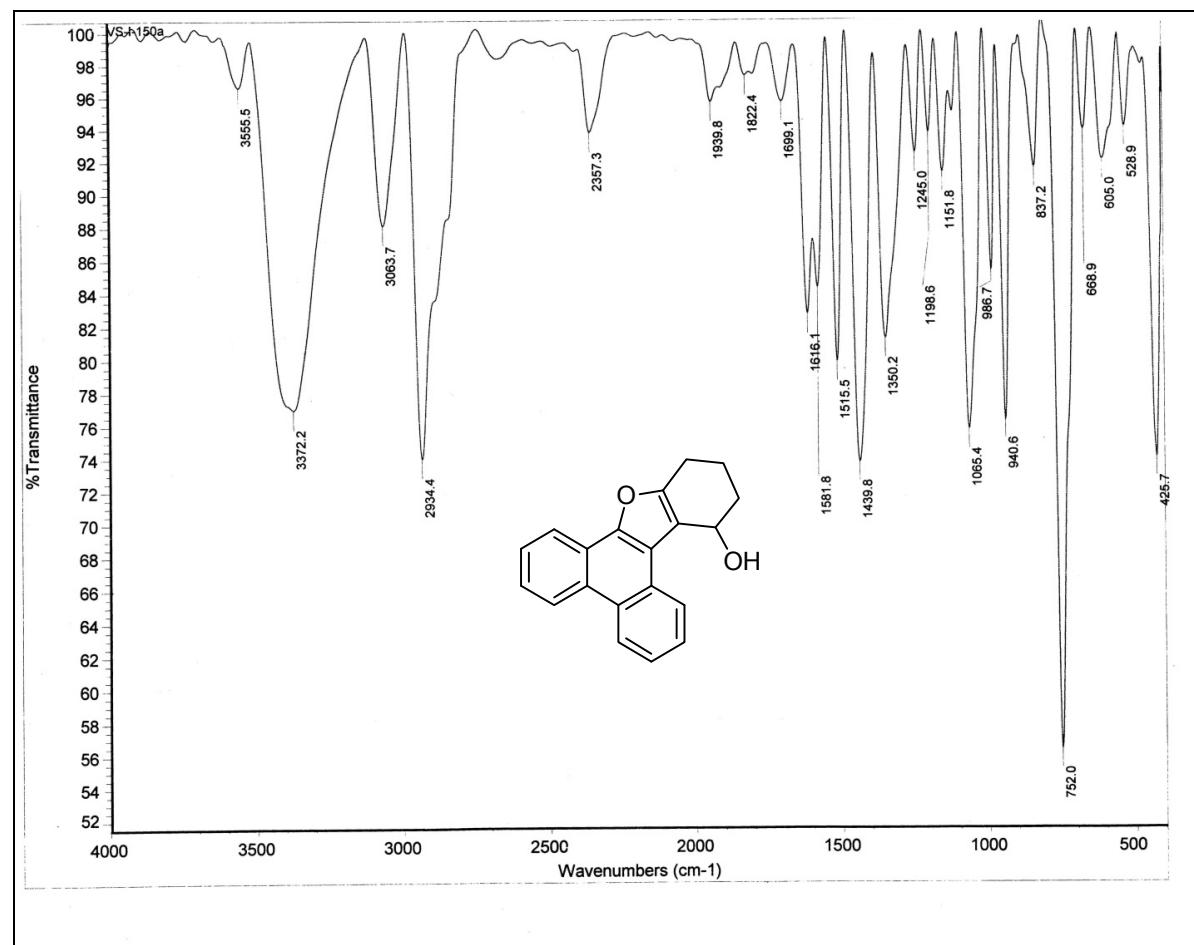


I) 10,11,12,13-Tetrahydrophenanthro[9,10-*b*]benzofuran-13-ol **19**.

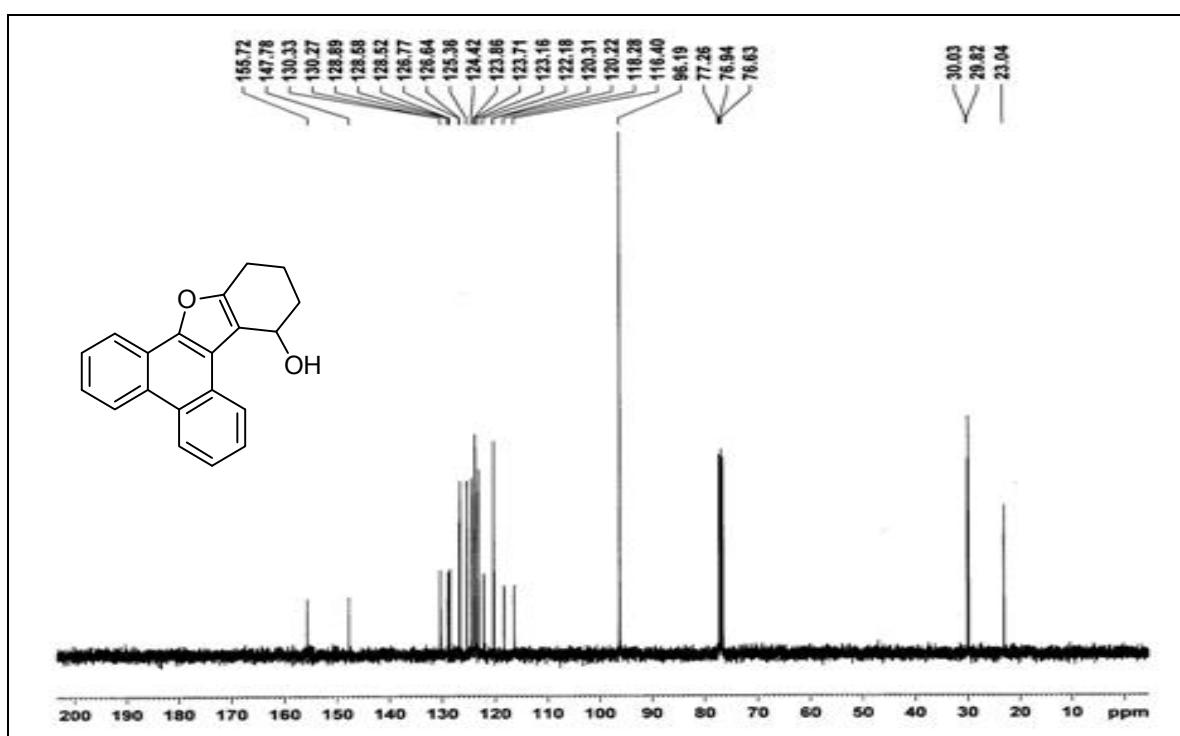
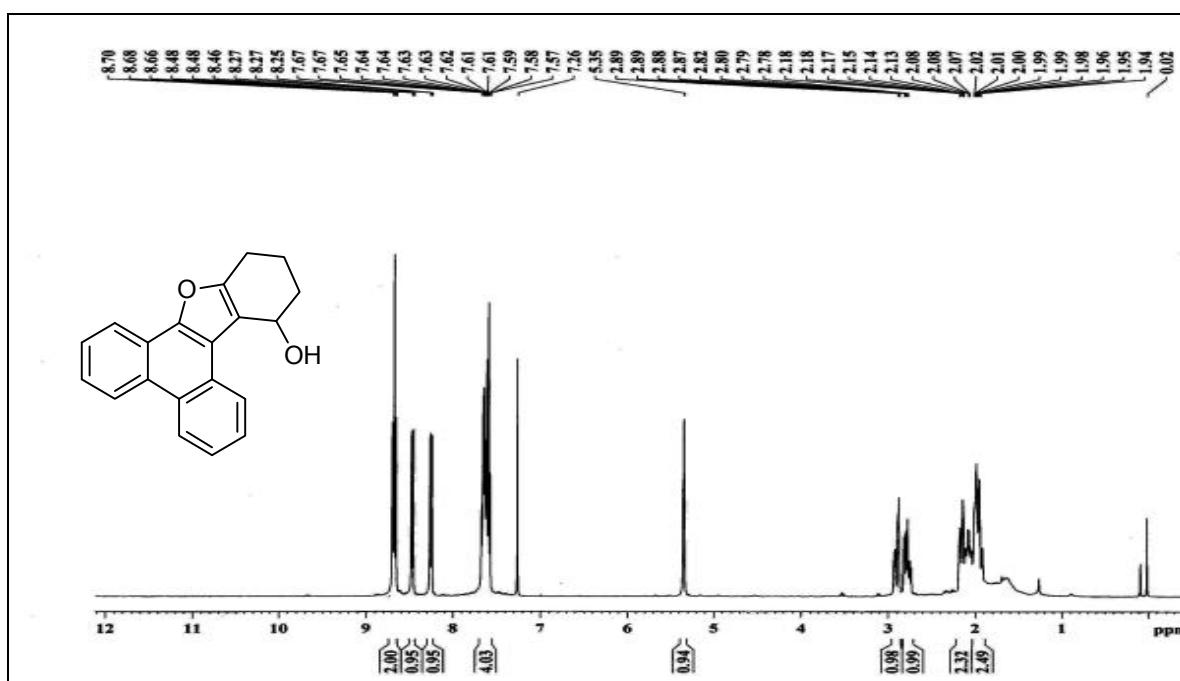


19

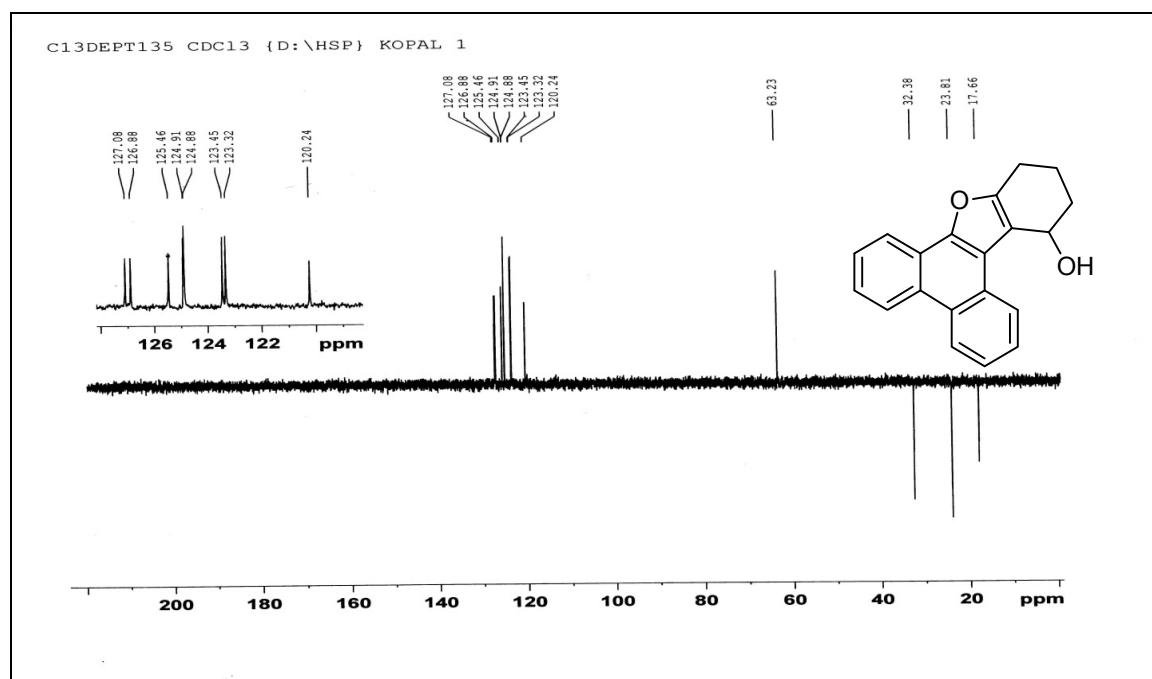
IR spectrum of **19**.



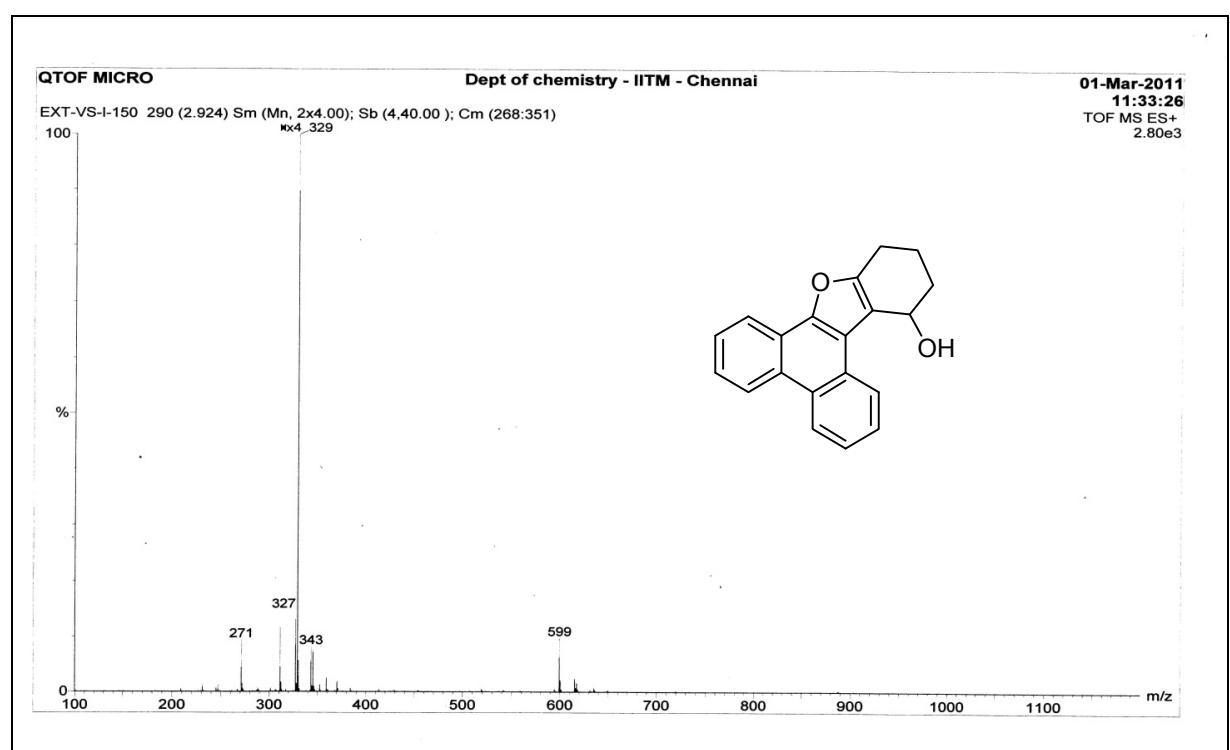
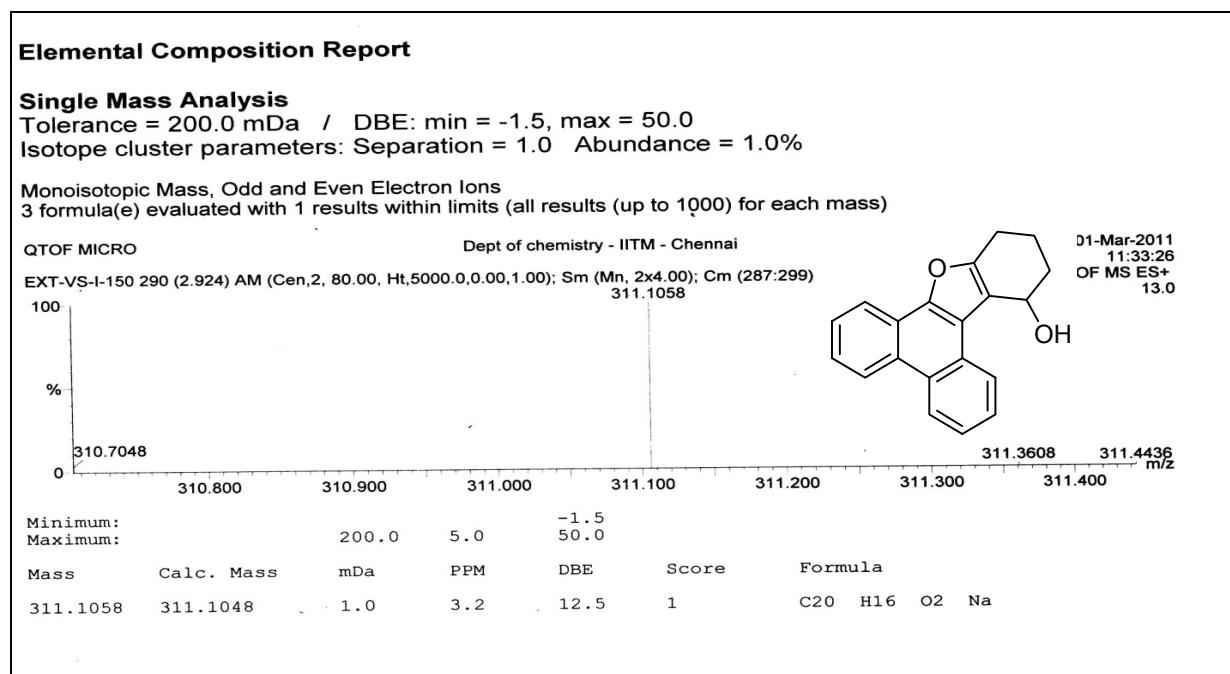
¹H (400 MHz, CCl₄:CDCl₃, 1:1) and ¹³C (100 MHz, CCl₄:CDCl₃, 1:1) NMR spectra of **19**.



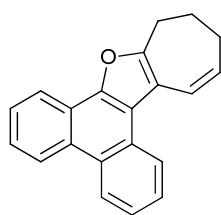
DEPT-135 NMR spectrum of **19**



ESIMS HRMS of 19.

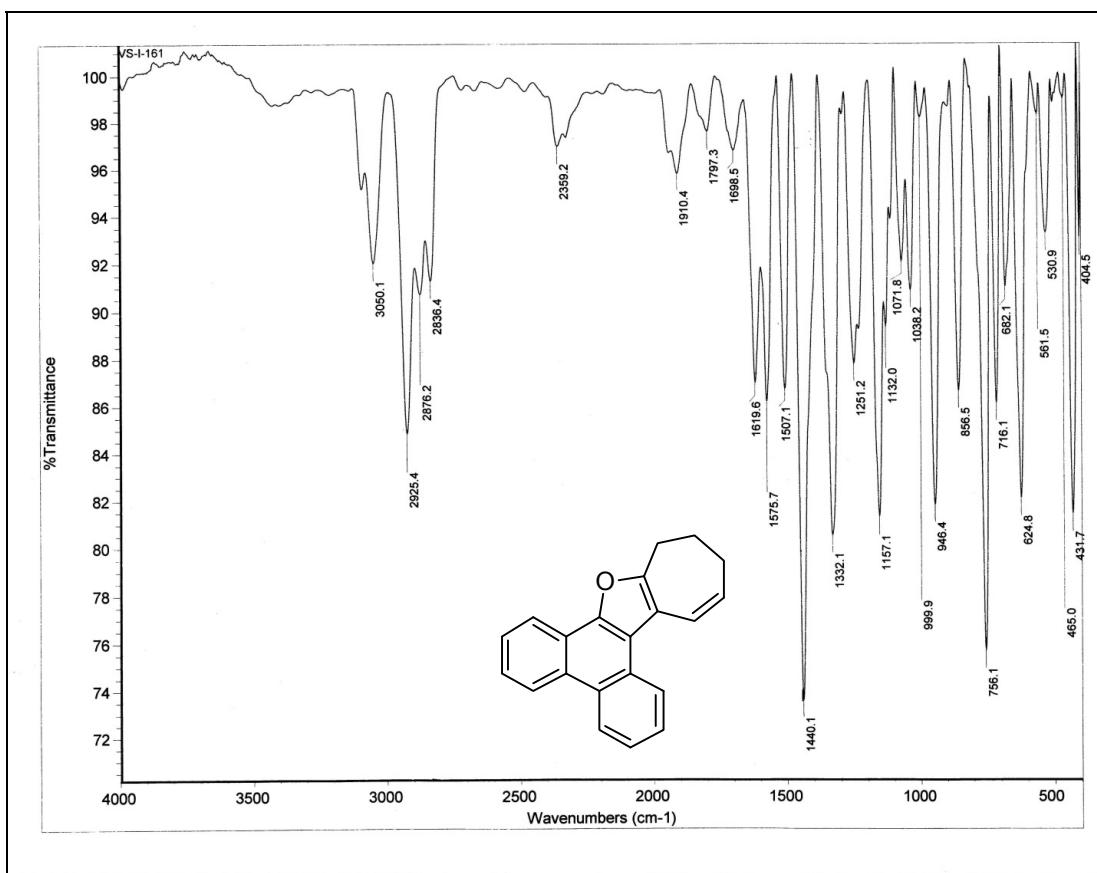


J) 11,12-Dihydro-10*H*-cyclohepta[*d*]phenanthro[9,10-*b*]furan **25a**.

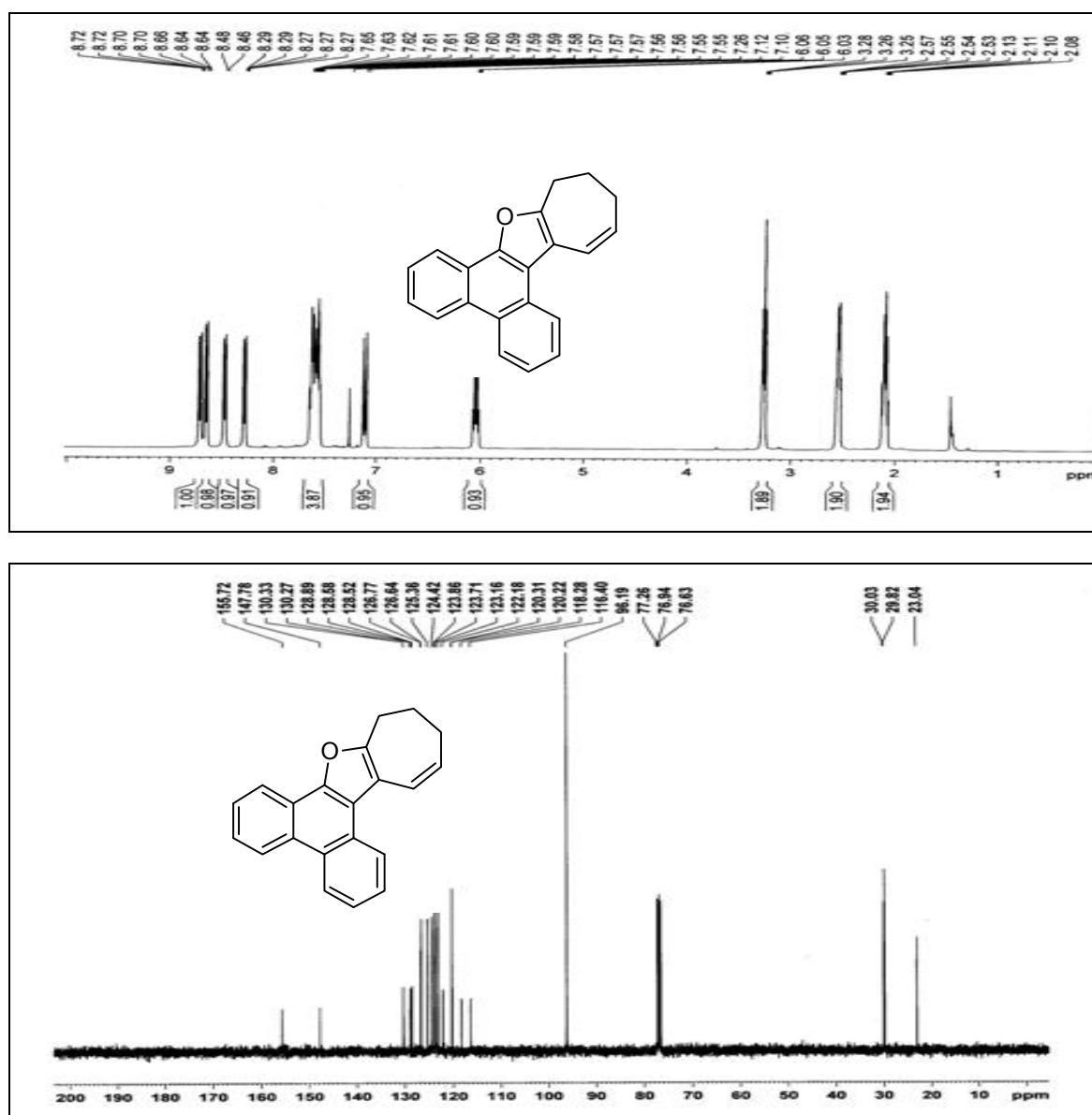


25a

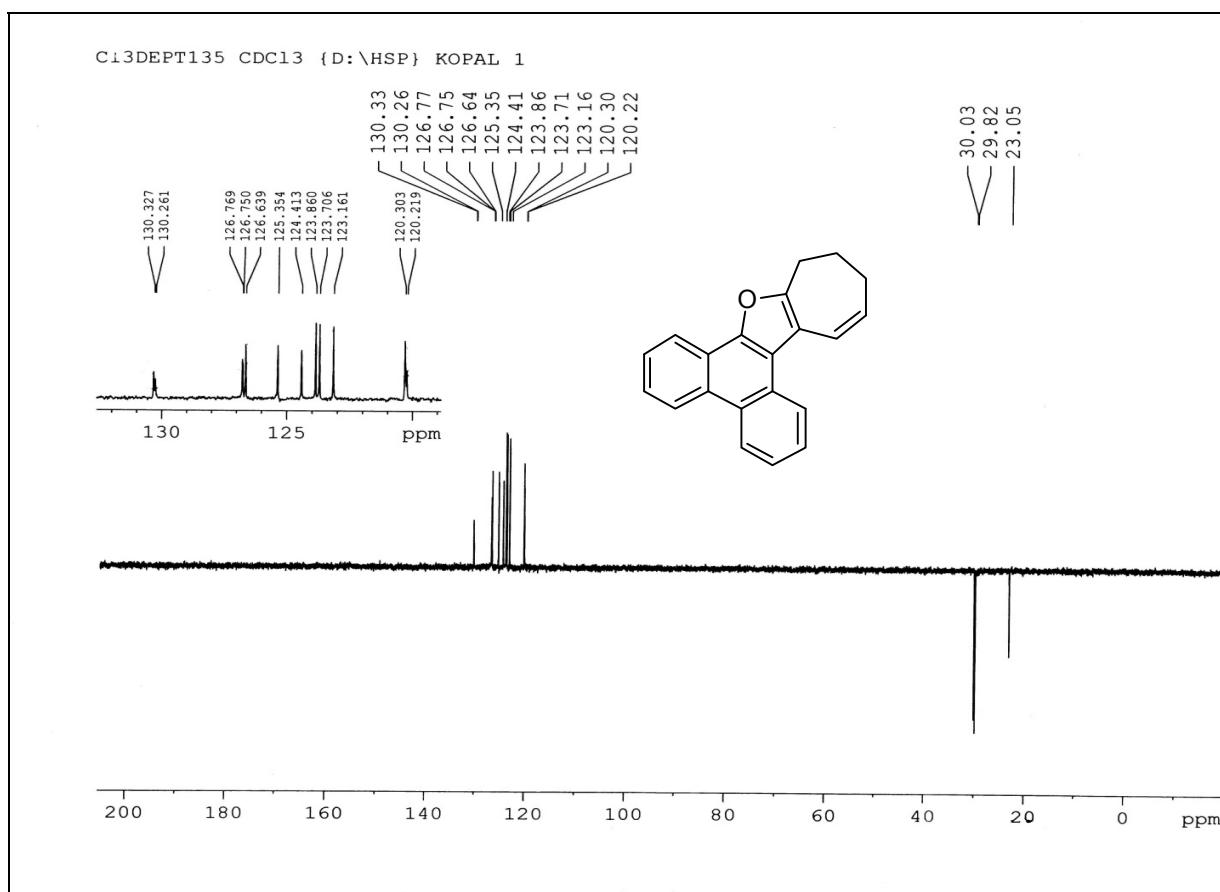
IR spectrum of **25a**.



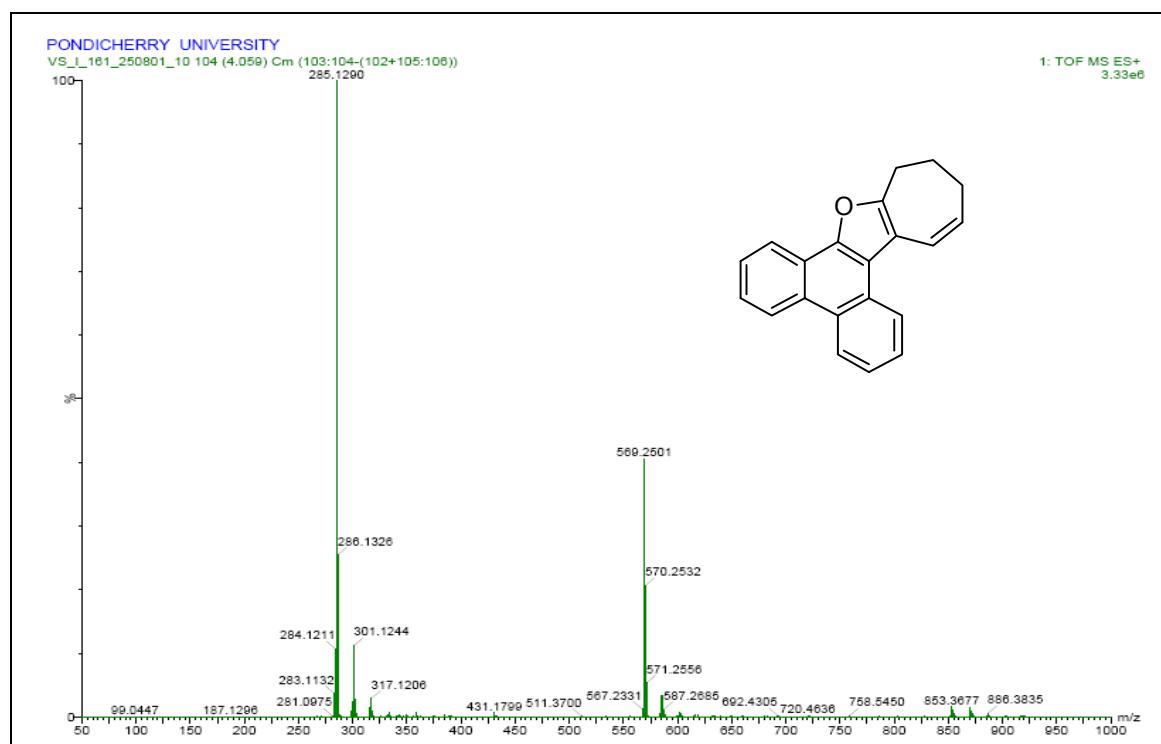
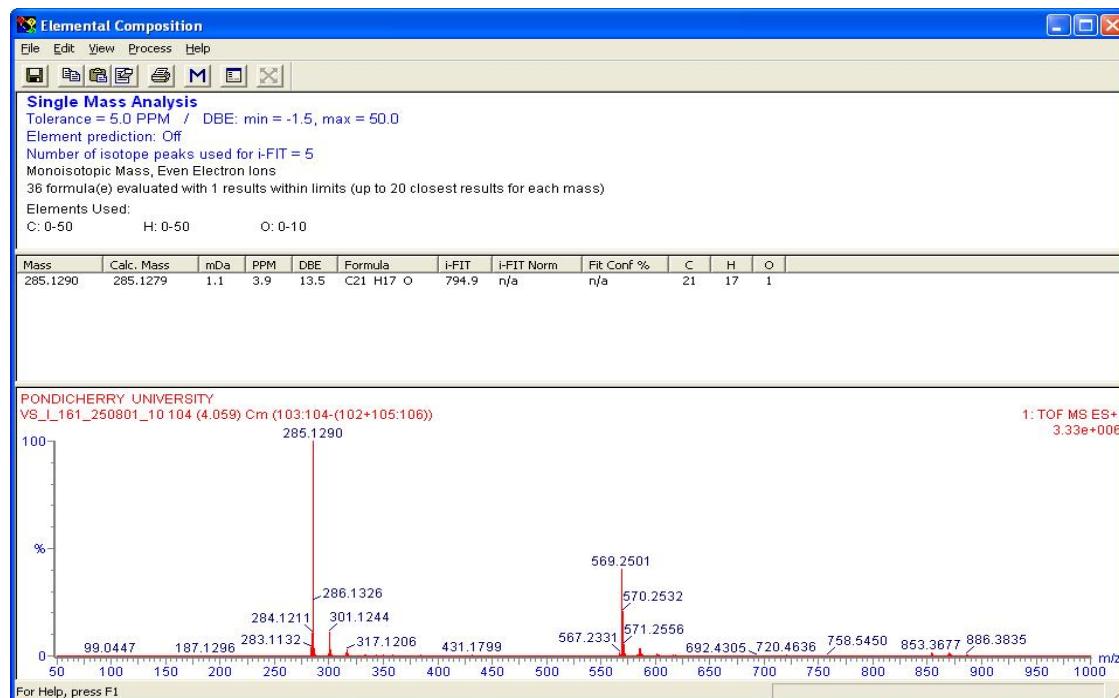
^1H (400 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1) and ^{13}C (100 MHz, $\text{CCl}_4:\text{CDCl}_3$, 1:1)NMR spectra of **25a**.



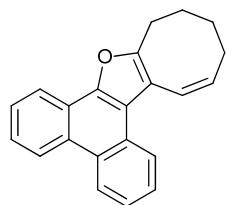
DEPT-135 NMR spectrum of **25a**.



ESIMS HRMS of **25a**.

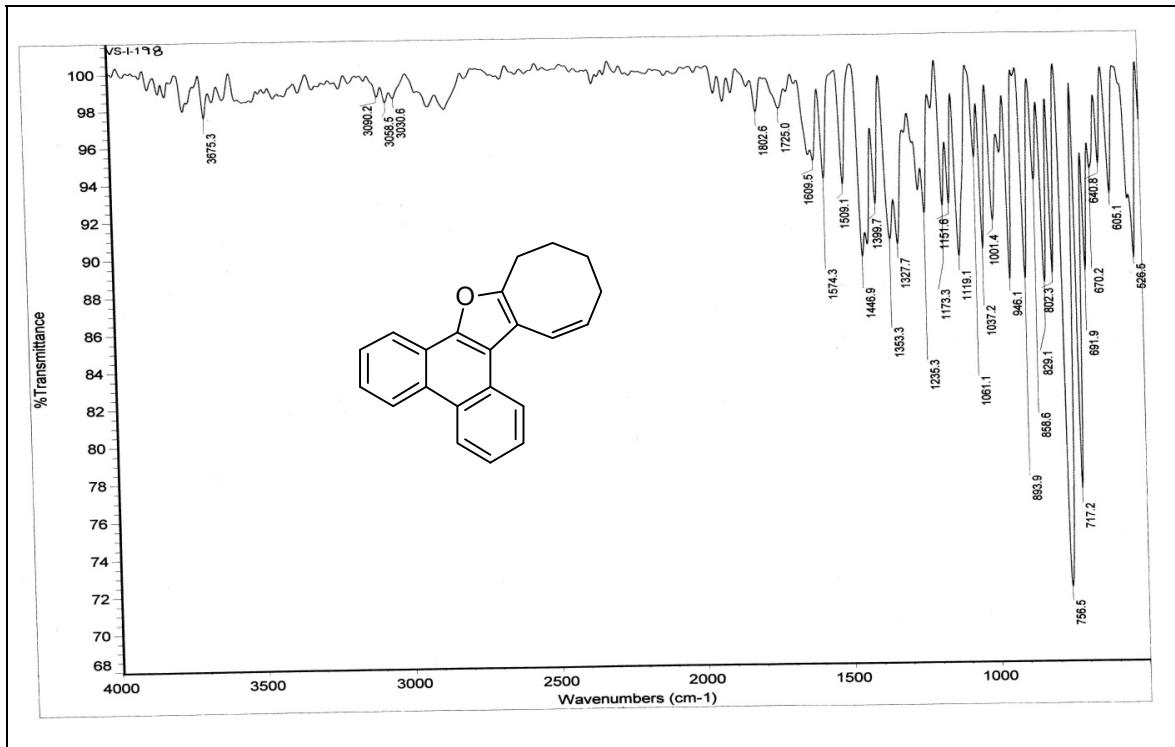


K) (*Z*)-10,11,12,13-tetrahydrocycloocta[*d*]phenanthro[9,10-*b*]furan **25b**.

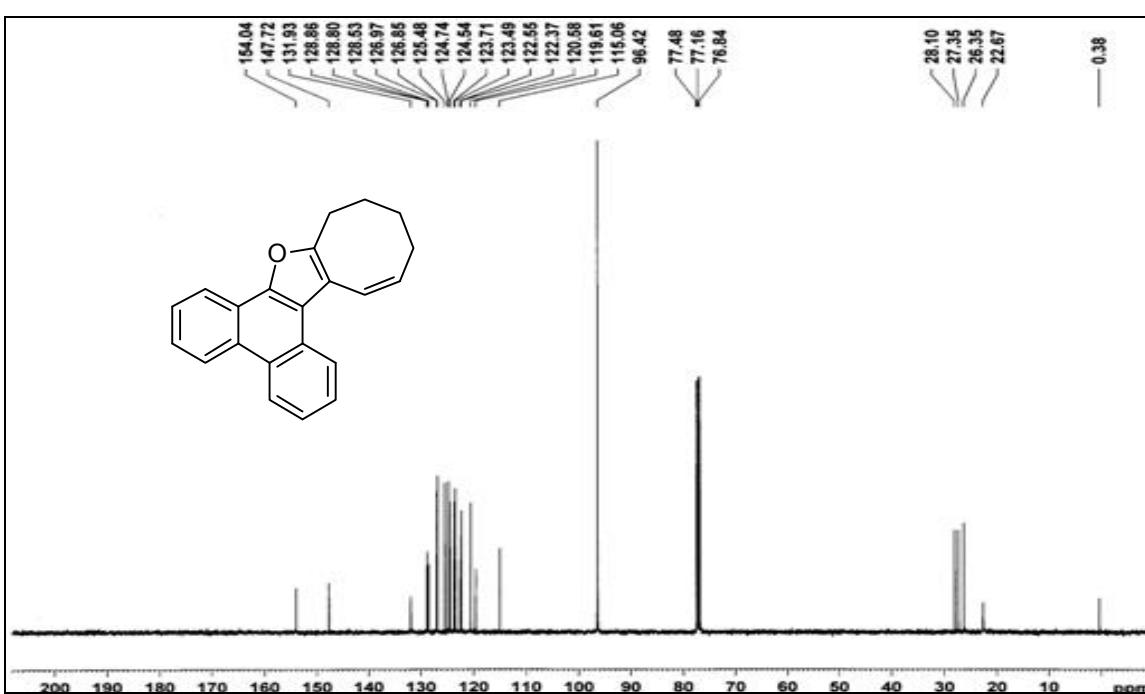


25b

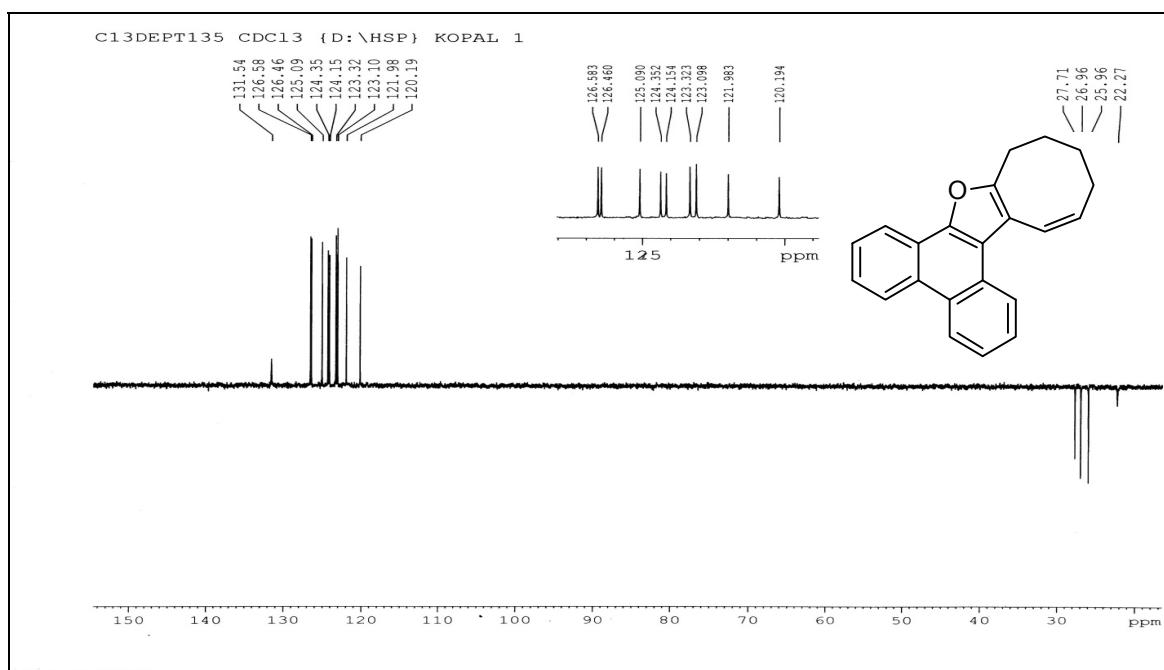
IR spectrum of **25b**.



¹H (400 MHz, CCl₄:CDCl₃, 1:1) and ¹³C (100 MHz, CCl₄:CDCl₃, 1:1)NMR spectra of **25b**.



DEPT-135 NMR spectrum of **25b**.



ESIMS HRMS of **25b**.

