

# Enantioselective, decarboxylative [3+2] cycloaddition of azomethine ylides and chromone-3-carboxylic acids

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## 1. General methods

NMR spectra were acquired on a Bruker Ultra Shield 700 instrument, running at 700 MHz for  $^1\text{H}$  and 176 MHz for  $^{13}\text{C}$ , respectively. Chemical shifts ( $\delta$ ) are reported in ppm relative to residual solvent signals ( $\text{CDCl}_3$ : 7.26 ppm for  $^1\text{H}$  NMR, 77.16 ppm for  $^{13}\text{C}$  NMR). Mass spectra were recorded on a Bruker Maxis Impact spectrometer using electrospray (ES+) ionization (referenced to the mass of the charged species). Analytical thin layer chromatography (TLC) was performed using pre-coated aluminum backed plates (Merck Kieselgel 60 F254) and visualized by ultraviolet irradiation. Unless otherwise noted, analytical grade solvents and commercially available reagents were used without further purification. For flash chromatography (FC) silica gel (Silica gel 60, 230-400 mesh, Fluka and Davisil Grade 643, 200-425 mesh). The enantiomeric ratio (er) of the products were determined by Ultra Performance Convergence Chromatography (UPC2) or HPLC using Daicel Chiralpak IA, IB, IC and IG columns as chiral stationary phases. Diethyl 2-hydroxyarylideneaminomalonates **3** and 2-hydroxyarylideneaminolactones **4** were synthesized according to the literature procedure<sup>1</sup>. Chromone-3- carboxylic acids<sup>2</sup> **2** were prepared from the corresponding substituted 2-hydroxybenzaldehydes following the literature procedure.

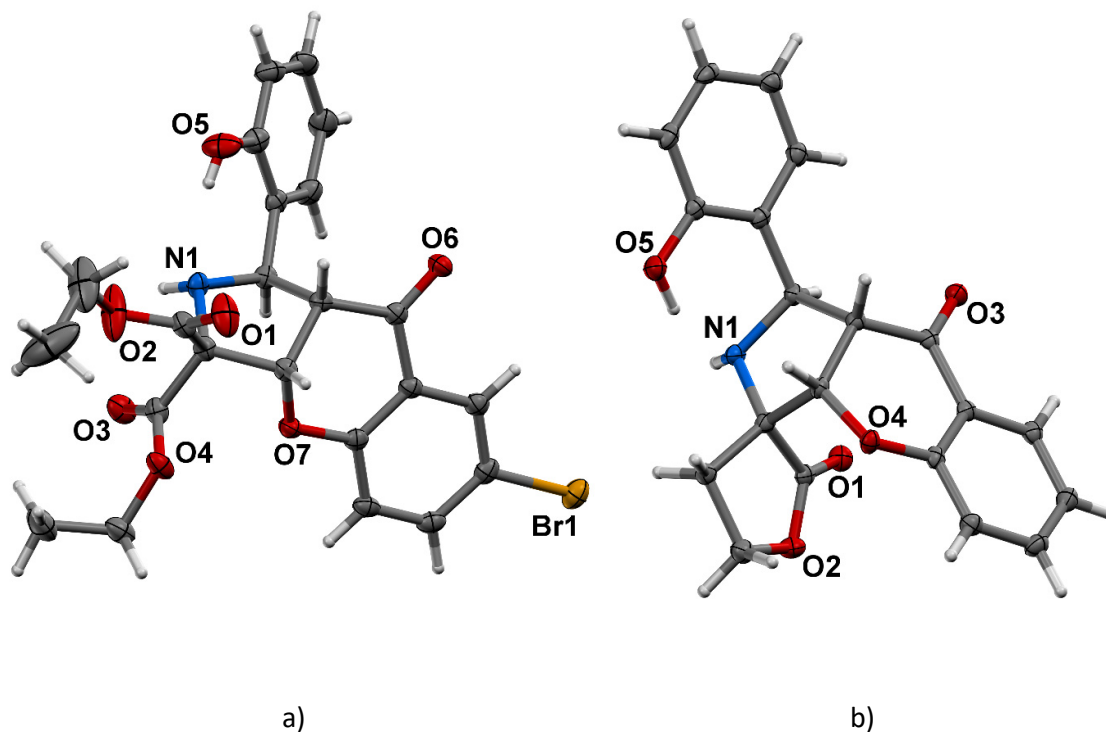
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<sup>1</sup> Tian, L., Xu, G. Q., Li, Y. H., Liang, Y. M., Xu, P. F., *Chemical Community*, **2014**, 50(19), 2428-2430

<sup>2</sup> Ishizuka, N., Matsumura, K. I., Sakai, K., Fujimoto, M., Mihara, S. I., Yamamori, T., *Journal of medicinal chemistry*, **2002**, 45(10), 2041-2055.

## 2. Crystal and X-ray data

Single-crystal X-ray diffraction data were collected at temperature of 100 K. The compound **1e** ( $C_{23}H_{22}BrNO_7$ ) crystallizes in centrosymmetric triclinic space group  $P\bar{1}$  as racemic mixture, and **1m** ( $C_{20}H_{17}NO_5$ ) crystallizes in the non-centrosymmetric orthorhombic space group  $P2_12_12_1$ . The asymmetric unit of both crystals contains one crystallographically independent molecule (Fig. 1).



**Figure S1.** Views of the molecules of **1e** (a) and **1m** (b) at 100 K, with the atom-labeling schemes and 50% probability ellipsoids. Hydrogen atoms are drawn with an arbitrary radius.

Single crystal X-ray diffraction data were collected at 100 K by the  $\omega$ -scan technique using a RIGAKU XtaLAB Synergy, Dualflex, Pilatus 300K diffractometer<sup>3</sup> with PhotonJet micro-focus X-ray Source Cu-K $\alpha$  ( $\lambda = 1.54184$  Å). Data collection, cell refinement, data reduction and absorption correction were performed using CrysAlis PRO software.<sup>3</sup> The crystal structure was solved by using direct methods with the SHELXT 2018/2 program.<sup>4</sup> Atomic scattering factors

<sup>3</sup> Rigaku OD. CrysAlis PRO. Rigaku Oxford Diffraction Ltd, Yarnton, Oxfordshire, England, **2019**.

<sup>4</sup> Sheldrick, G.M. "SHELXT - integrated space-group and crystal-structure determination", *Acta Cryst.* **2015**, A71, 3-8.

were taken from the International Tables for X-ray Crystallography. Positional parameters of non-H-atoms were refined by a full-matrix least-squares method on  $F^2$  with anisotropic thermal parameters by using the SHELXL 2018/3 program.<sup>5</sup> All hydrogen atoms were placed in calculated positions ( $C-H = 0.95-1.00 \text{ \AA}$ ) and included as riding contributions with isotropic displacement parameters set to 1.2-1.5 times the  $U_{eq}$  of the parent atom.

**1e:** Formula  $C_{23}H_{22}BrNO_7$ , triclinic, space group  $P-1$ ,  $Z = 4$ , unit cell constants  $a = 11.1608(1)$ ,  $b = 14.1880(2)$ ,  $c = 16.0038(2) \text{ \AA}$ ,  $\alpha = 69.852(1)$ ,  $\beta = 75.621(1)$ ,  $\gamma = 74.609(1)^\circ$ ,  $V = 2259.06(5) \text{ \AA}^3$ . The integration of the data yielded a total of 53433 reflections with  $\theta$  angles in the range of  $2.99$  to  $66.59^\circ$  of which 7974 were unique ( $R_{int} = 2.69\%$ ), and 7815 were greater than  $2\sigma(F^2)$ . The final anisotropic full-matrix least-squares refinement on  $F^2$  with 597 parameters converged to the final  $R_1 = 0.0320$  (for  $I > 2\sigma(I)$ ) and  $wR_2 = 0.0806$  (all data). The goodness-of-fit was 1.037. The largest peak in the final difference electron density synthesis was  $0.93 \text{ e \AA}^{-3}$  and the largest hole was  $-0.67 \text{ e \AA}^{-3}$ .

**1m:** Formula  $C_{20}H_{17}NO_5$ , orthorhombic, space group  $P2_12_12_1$ ,  $Z = 4$ , unit cell constants  $a = 6.6243(1)$ ,  $b = 10.9183(1)$ ,  $c = 22.0348(1) \text{ \AA}$ ,  $V = 1593.69(3) \text{ \AA}^3$ . The integration of the data yielded a total of 37878 reflections with  $\theta$  angles in the range of  $4.01$  to  $66.58^\circ$  of which 2806 reflections were unique ( $R_{int} = 2.46\%$ ), and 2786 were greater than  $2\sigma(F^2)$ . The final anisotropic full-matrix least-squares refinement on  $F^2$  with 244 parameters converged to the final  $R_1 = 0.0211$  (for  $I > 2\sigma(I)$ ) and  $wR_2 = 0.0530$  for all data. The goodness-of-fit was 1.056. The largest peak in the final difference electron density synthesis was  $0.20 \text{ e \AA}^{-3}$  and the largest hole was  $-0.12 \text{ e \AA}^{-3}$ . The absolute configuration was determined from anomalous scattering, by calculating the  $x$  Flack parameter<sup>6</sup> of  $0.00(2)$  using 1154 quotients.

CCDC 2167375 (**1e**) and CCDC 2167382 (**1m**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/structure](http://www.ccdc.cam.ac.uk/structure).

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<sup>5</sup> Sheldrick, G.M. "Crystal structure refinement with SHELXL", *Acta Cryst.* **2015**, *C71*, 3-8.

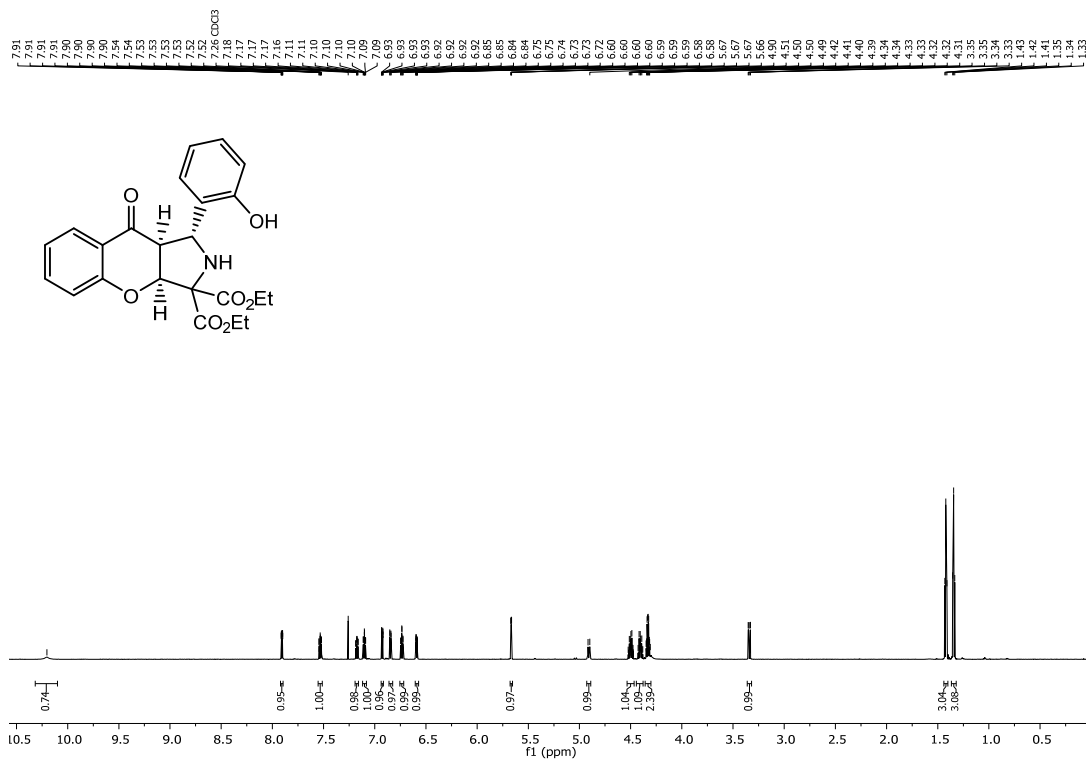
<sup>6</sup> Parsons, S.; Flack, H.D.; Wagner, T. "Use of intensity quotients and differences in absolute structure refinement" *Acta Cryst.* **2013**, *B69*, 249-259.



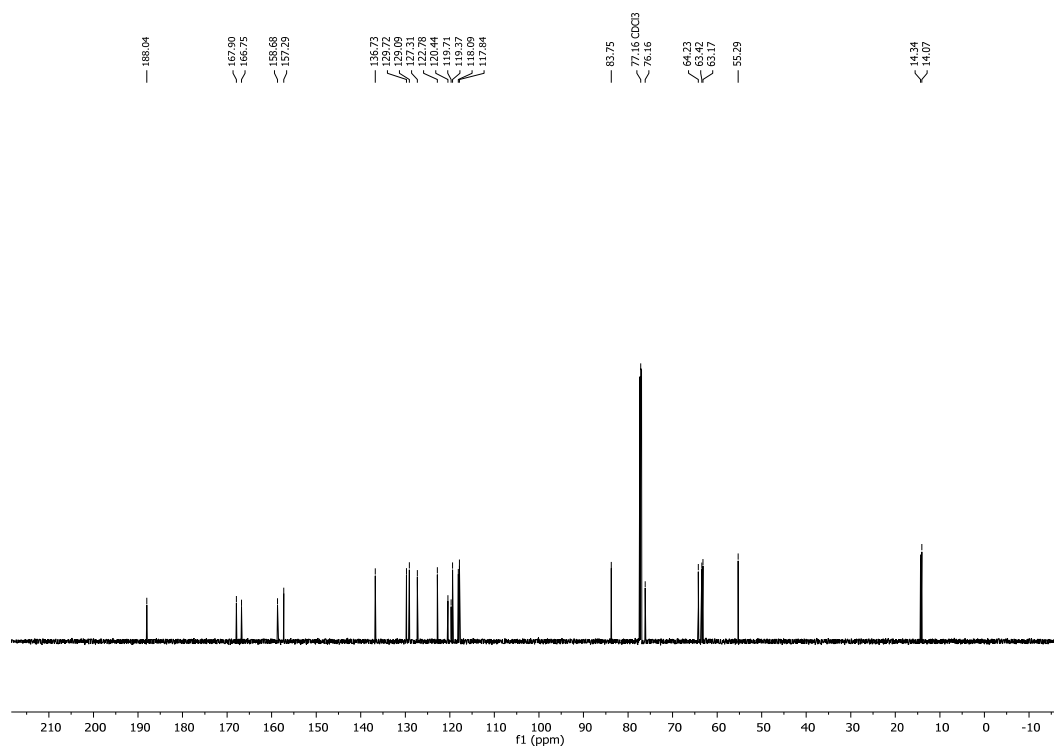
### 3. NMR Data

#### (1*R*,3*aS*,9*aS*)-Diethyl 1-(2-hydroxyphenyl)-9-oxo-1,2,9,9*a*-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1a

##### <sup>1</sup>H NMR

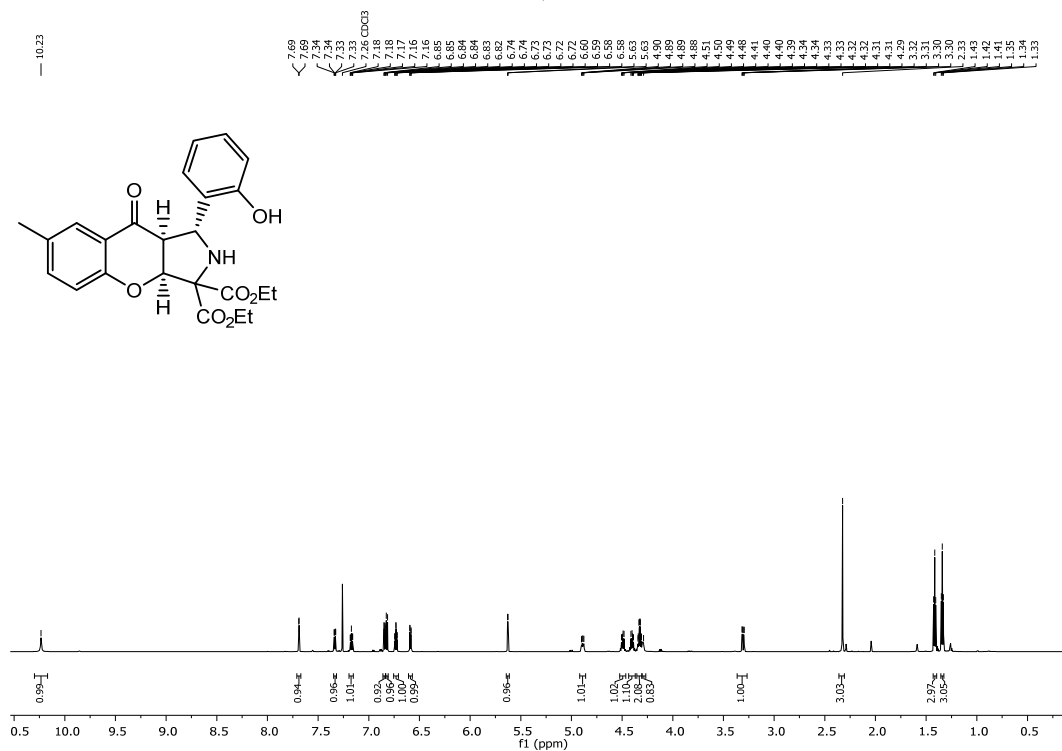


##### <sup>13</sup>C NMR

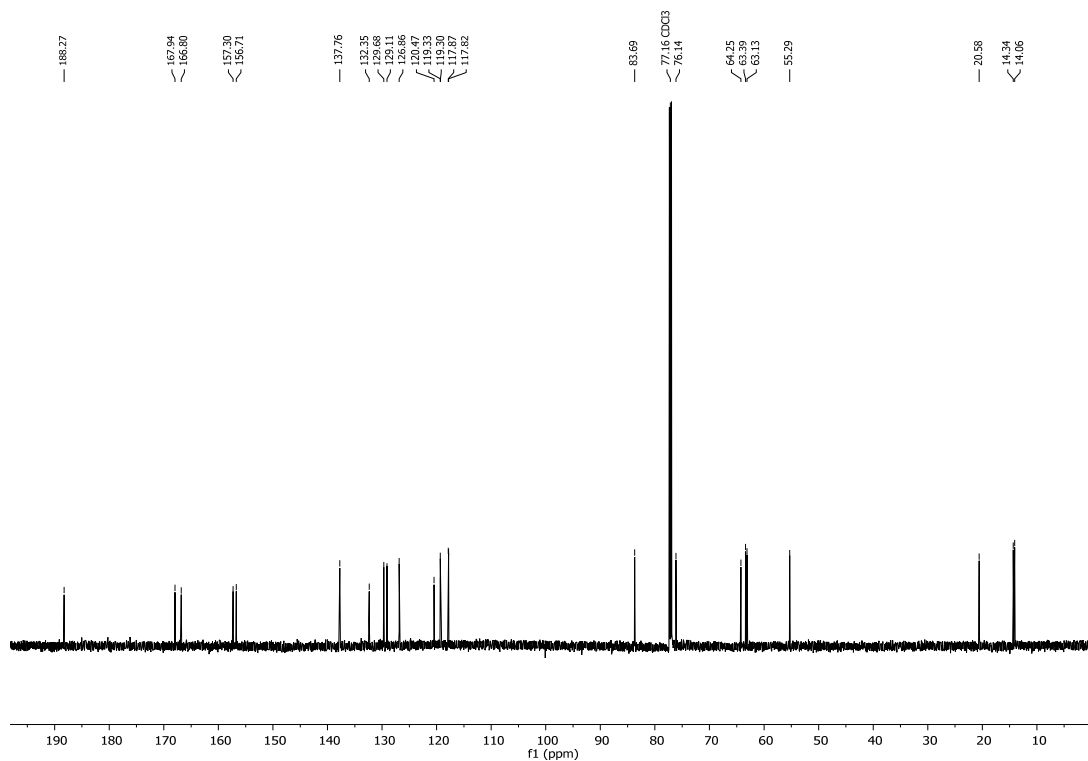


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<sup>1</sup>H NMR

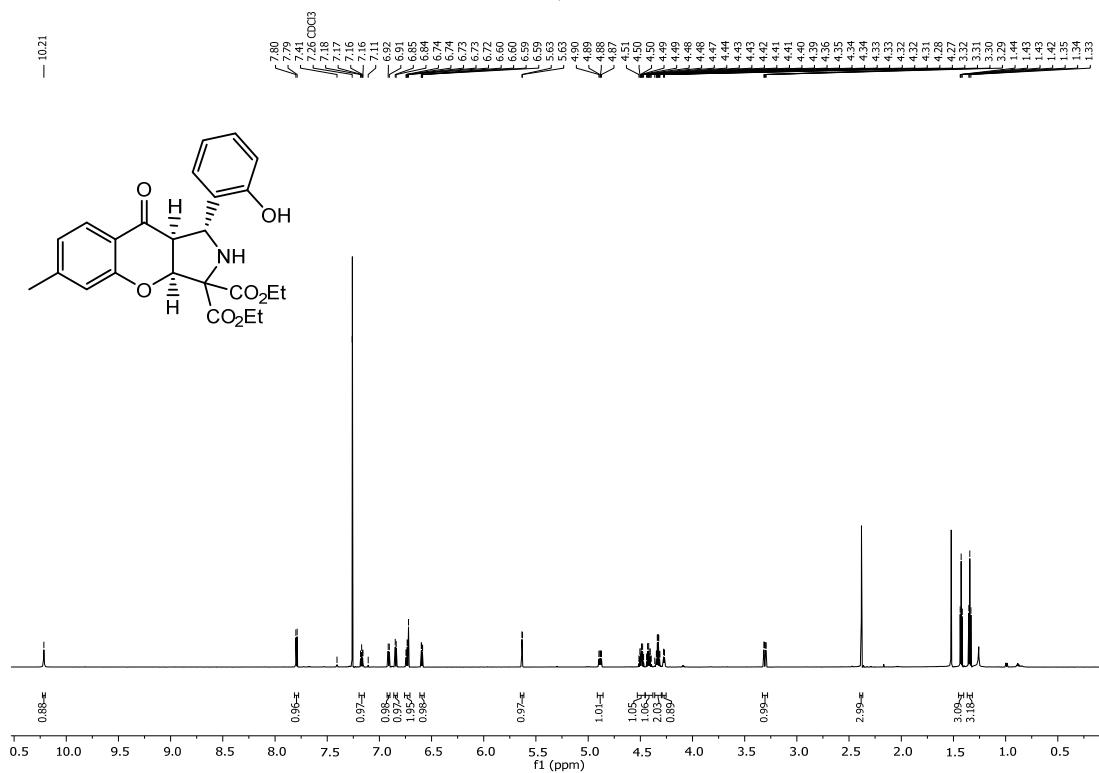


<sup>13</sup>C NMR

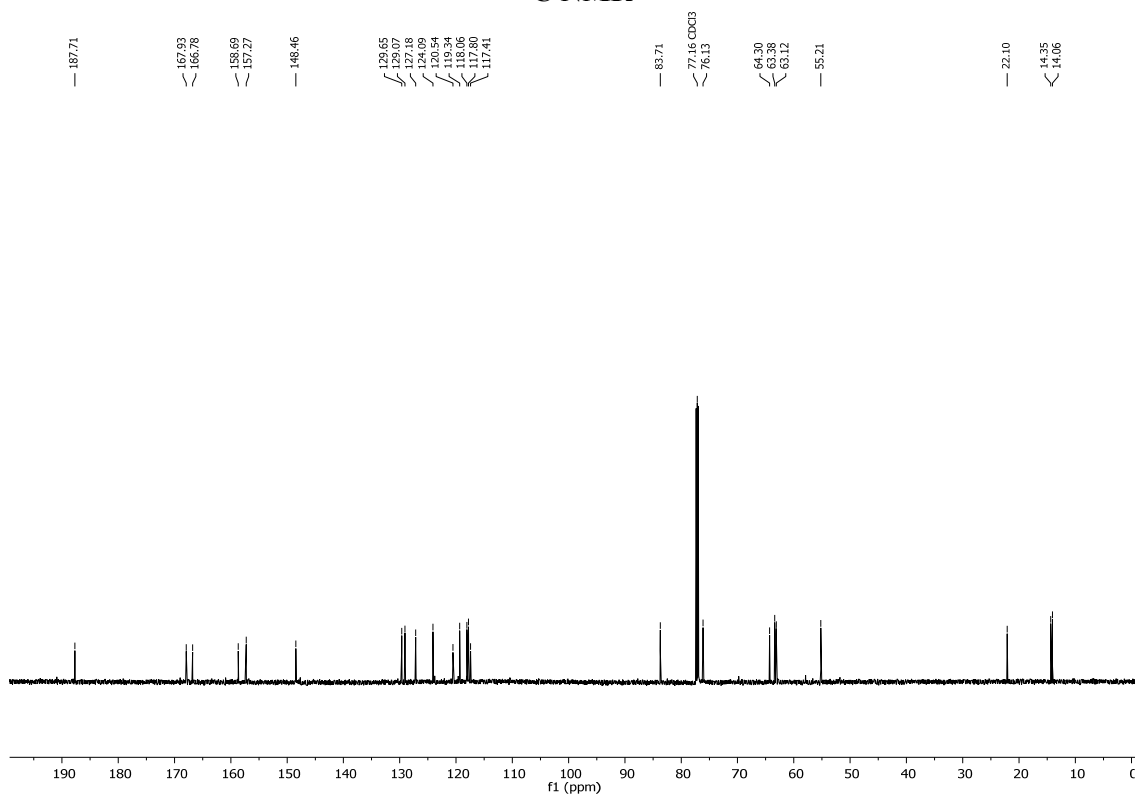


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<sup>1</sup>H NMR

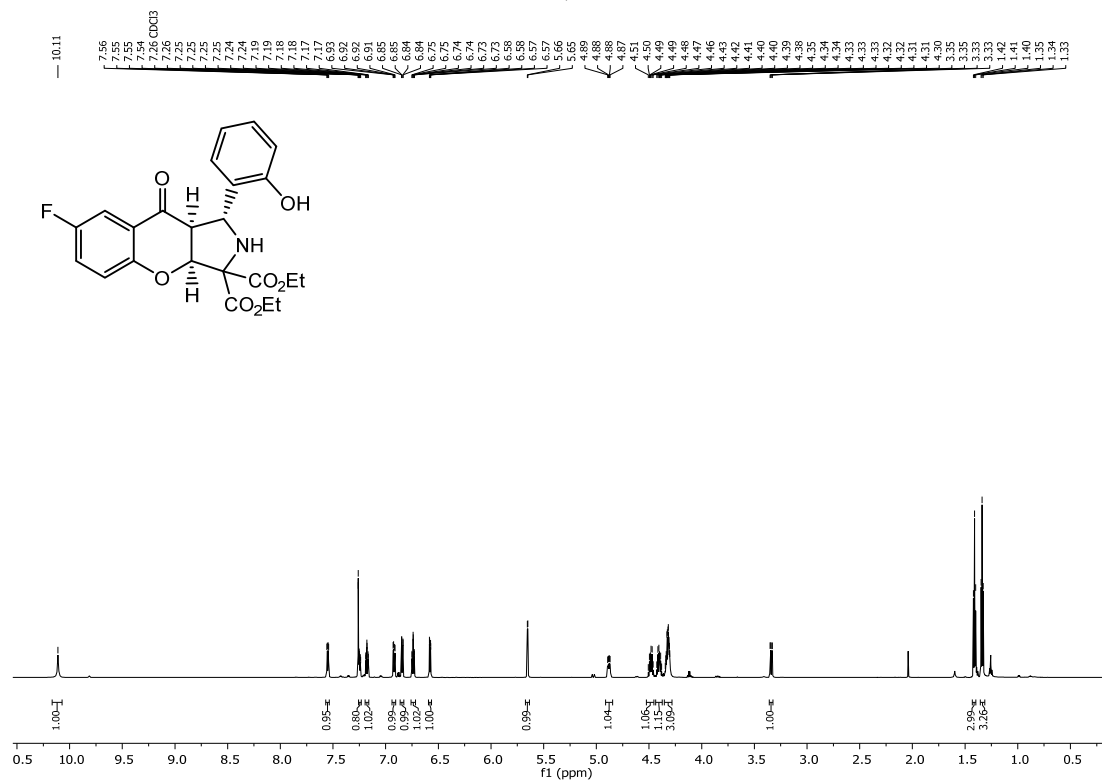


<sup>13</sup>C NMR

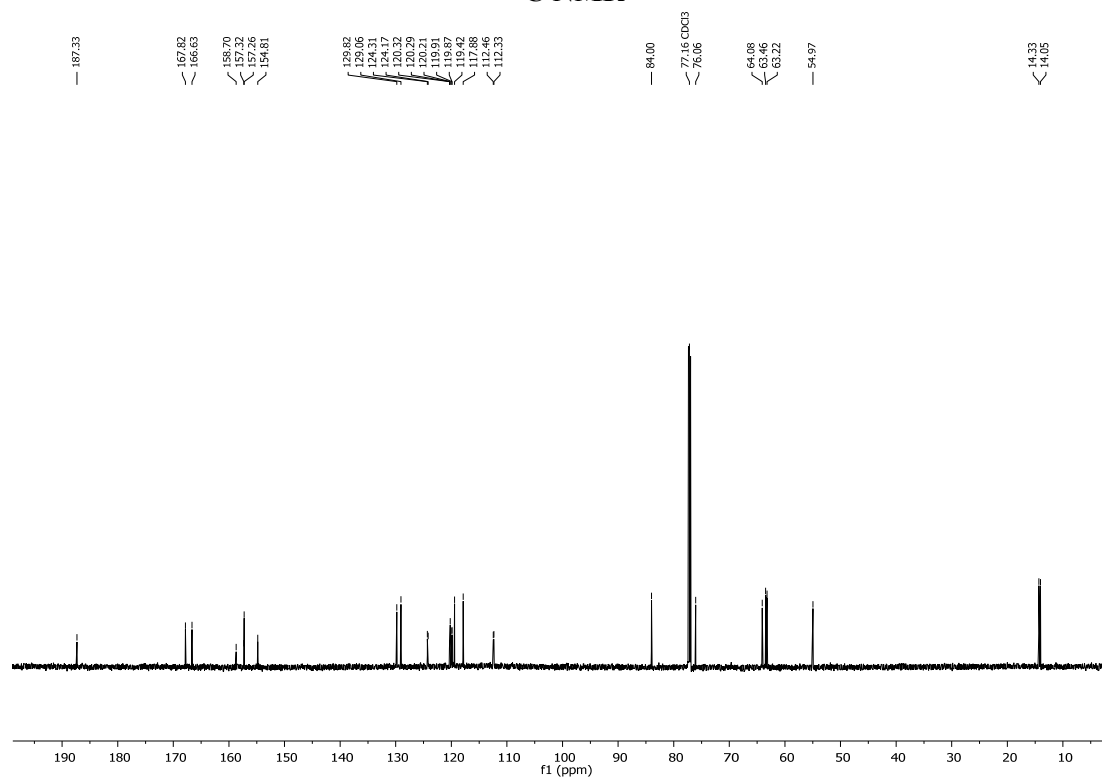


**(1*R*,3*aS*,9*aS*)-Diethyl 7-fluoro-1-(2-hydroxyphenyl)-9-oxo-1,2,9*a*-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1d**

**<sup>1</sup>H NMR**

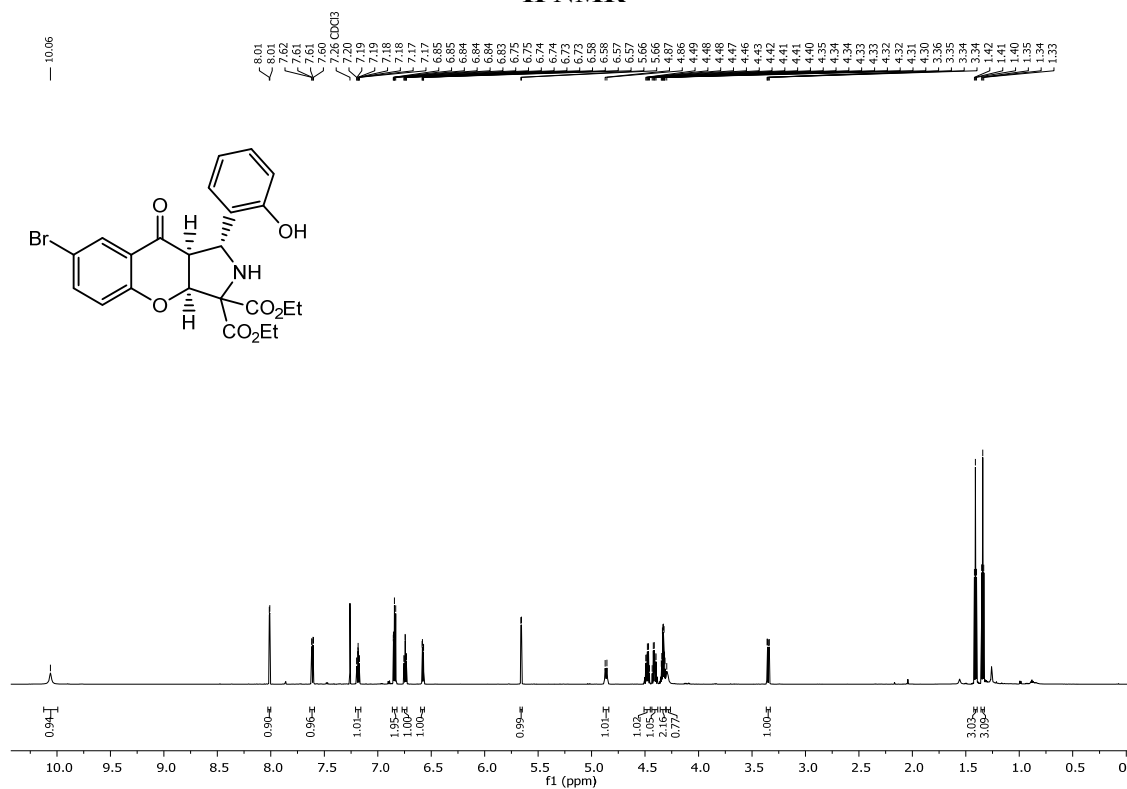


**<sup>13</sup>C NMR**

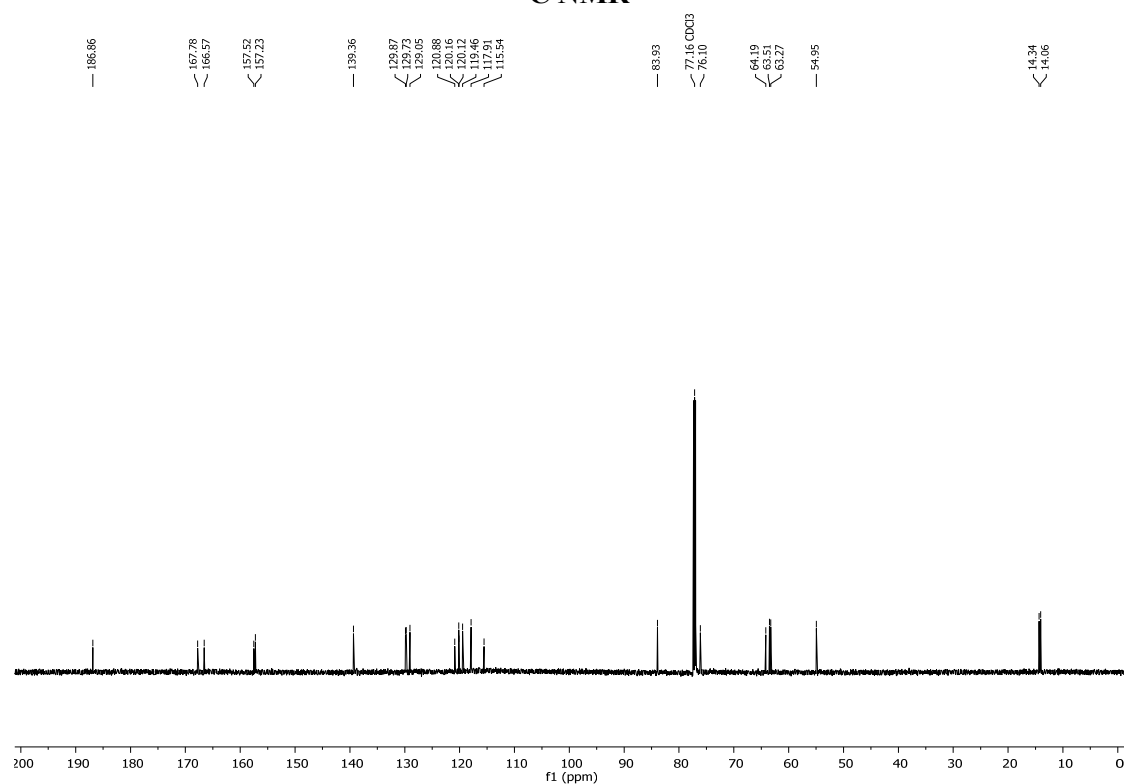


**(1*R*,3*aS*,9*aS*)-Diethyl 7-bromo-1-(2-hydroxyphenyl)-9-oxo-1,2,9,9*a*-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1e**

**<sup>1</sup>H NMR**

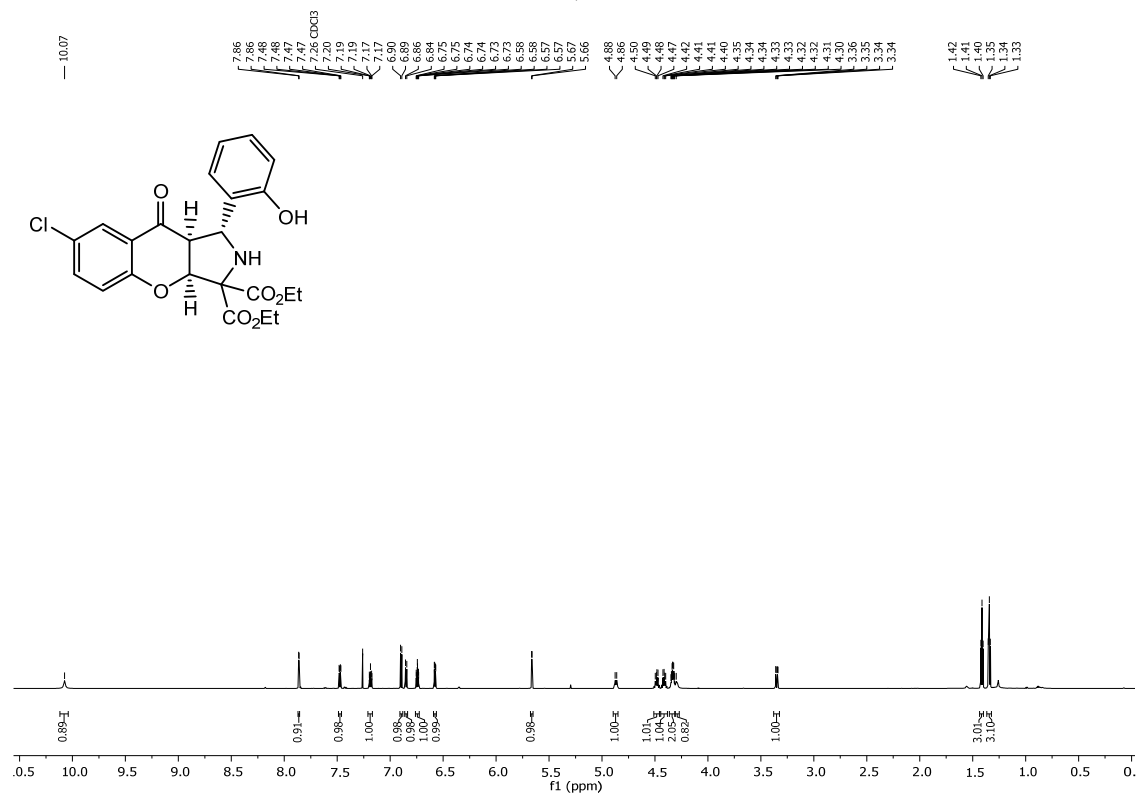


**<sup>13</sup>C NMR**

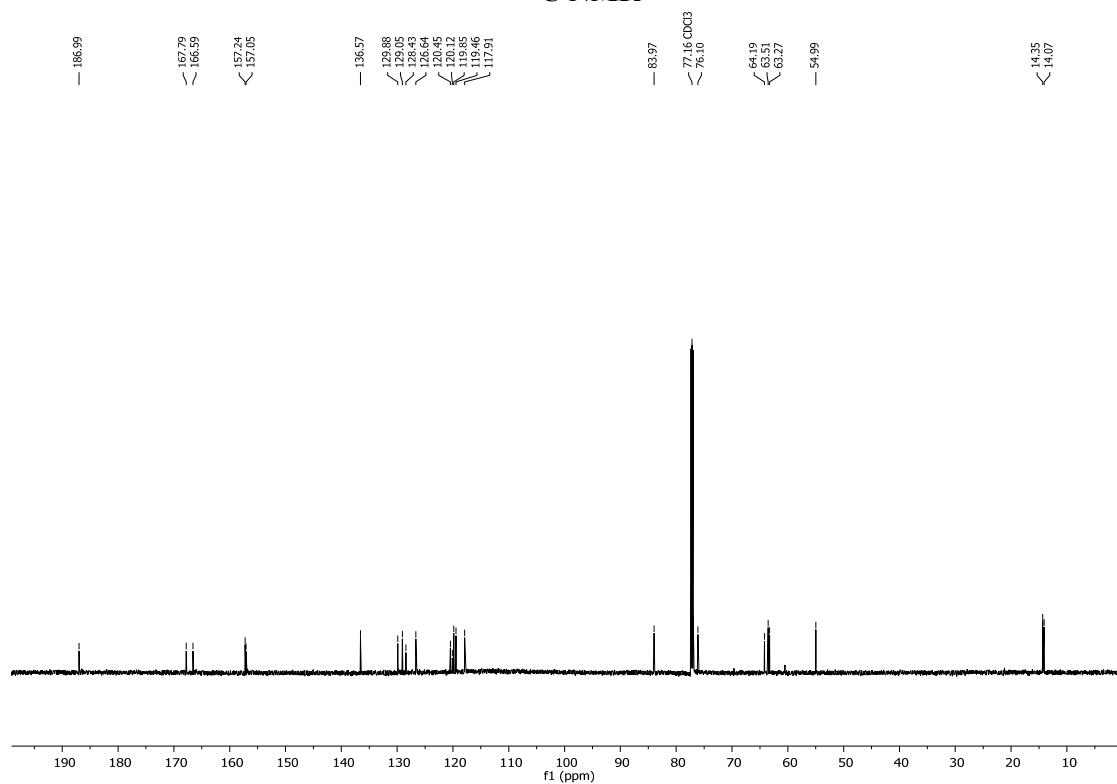


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<sup>1</sup>H NMR

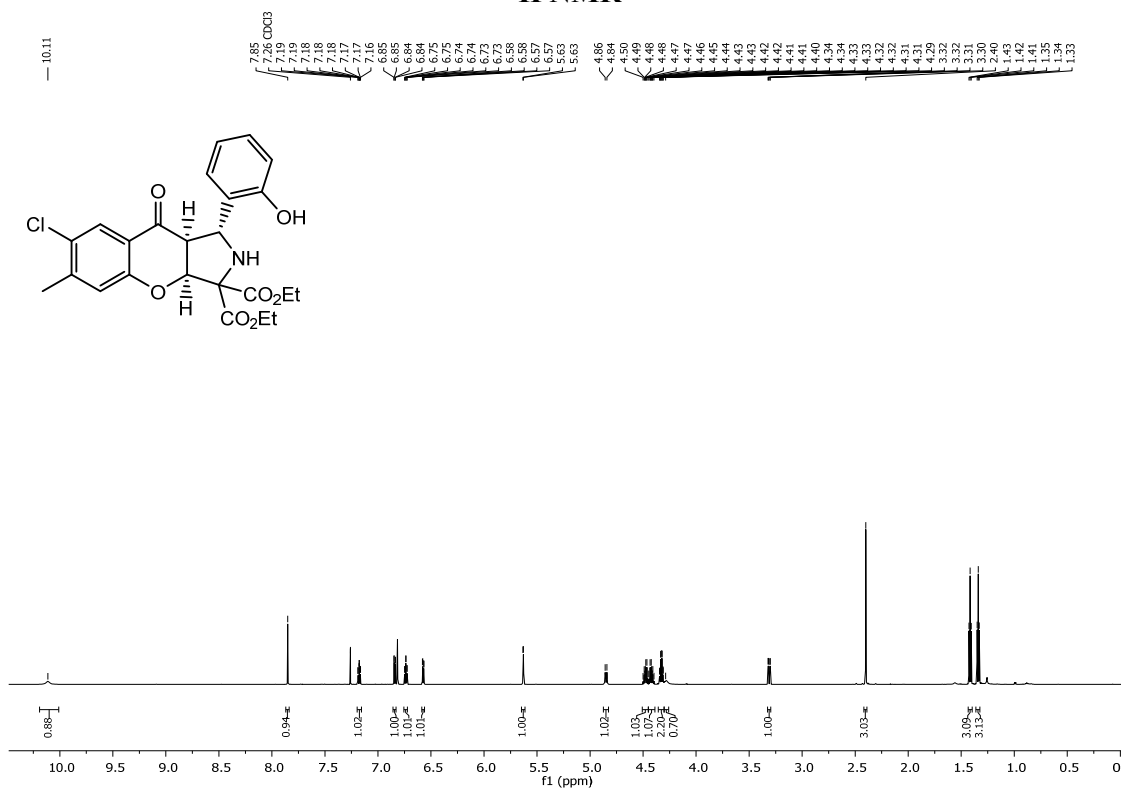


<sup>13</sup>C NMR

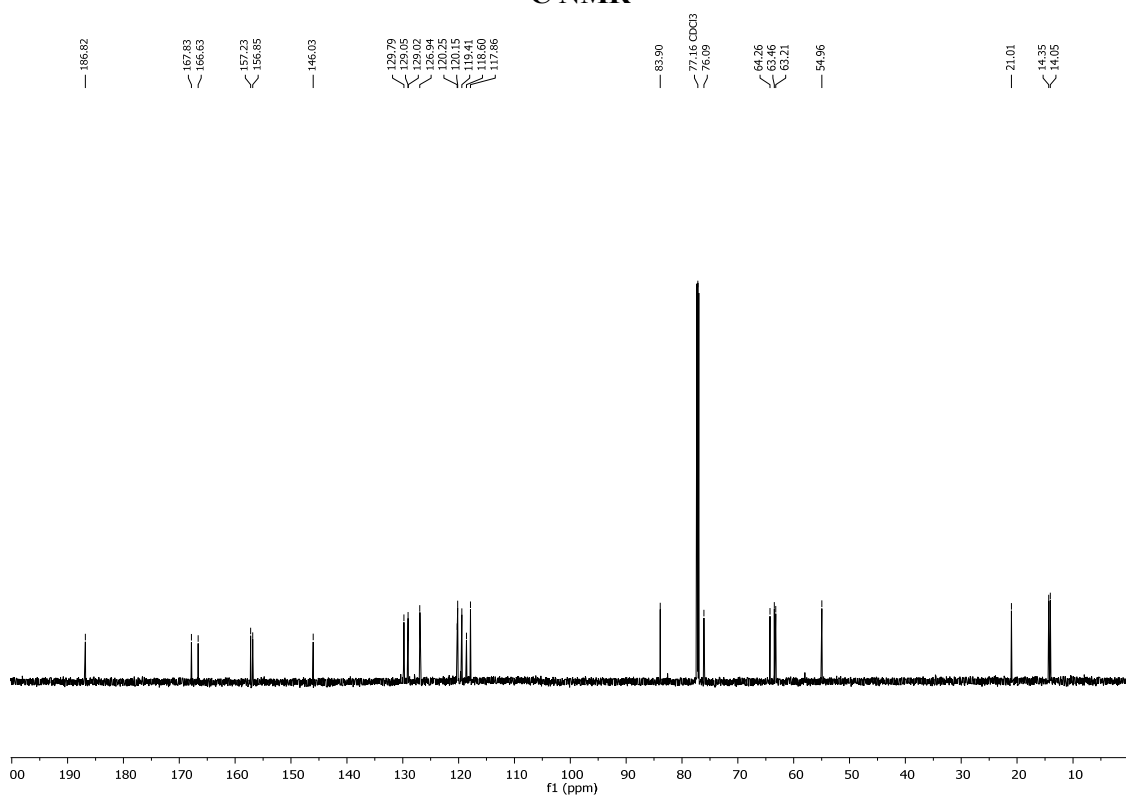


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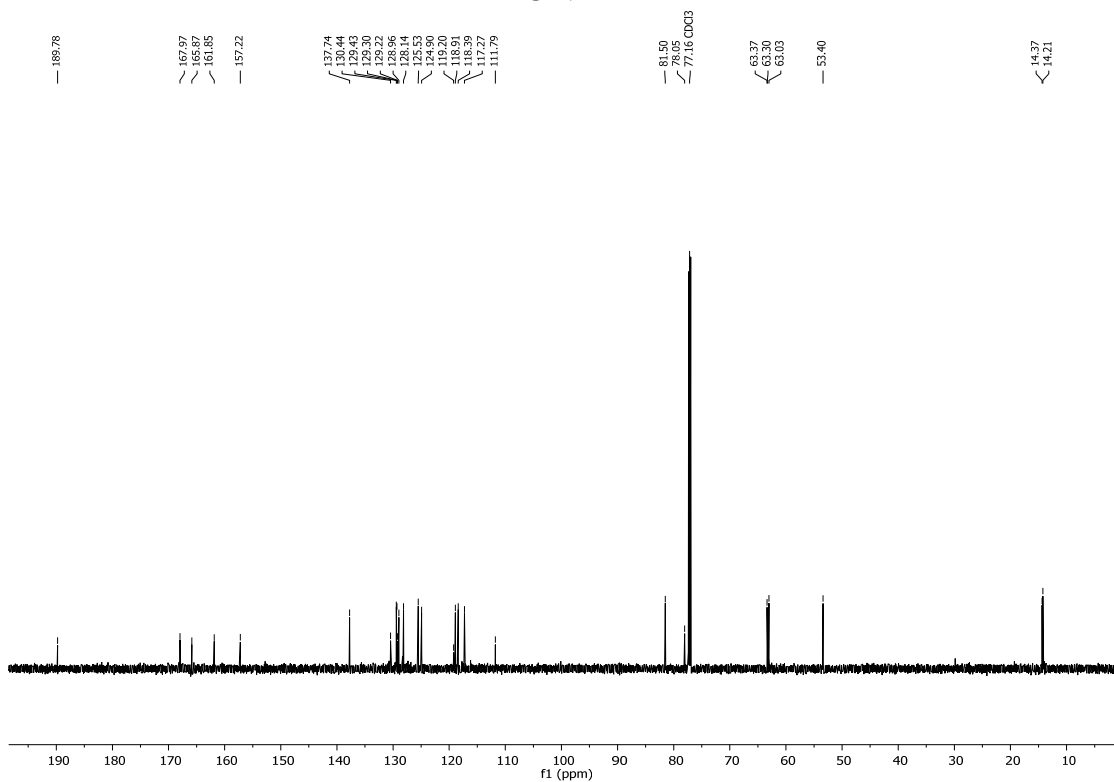
**<sup>1</sup>H NMR**



**<sup>13</sup>C NMR**



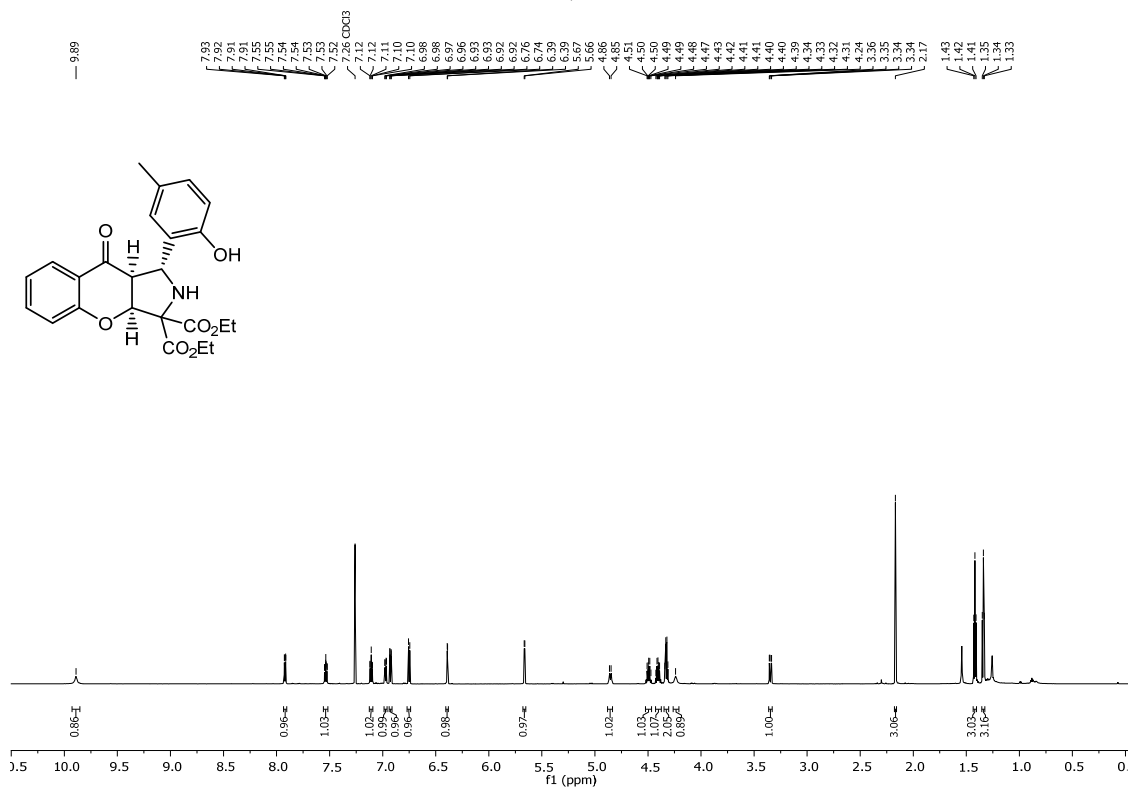
### <sup>1</sup>H NMR



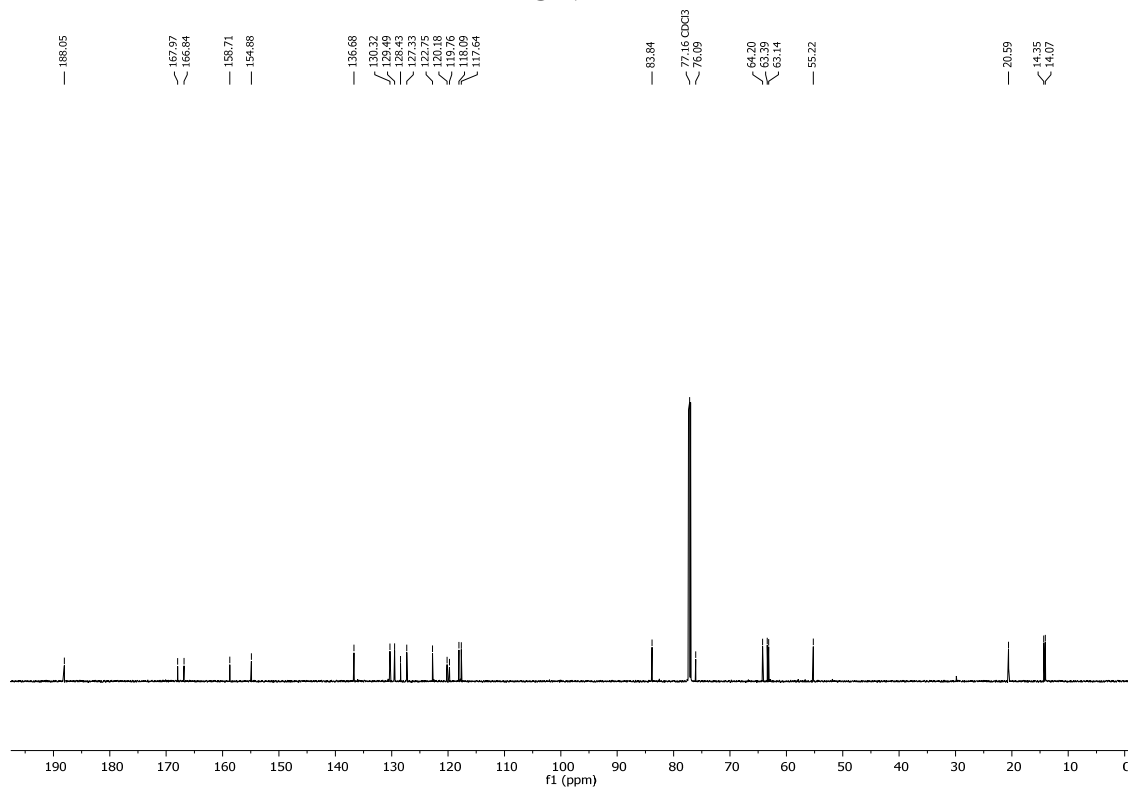


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**<sup>1</sup>H NMR**

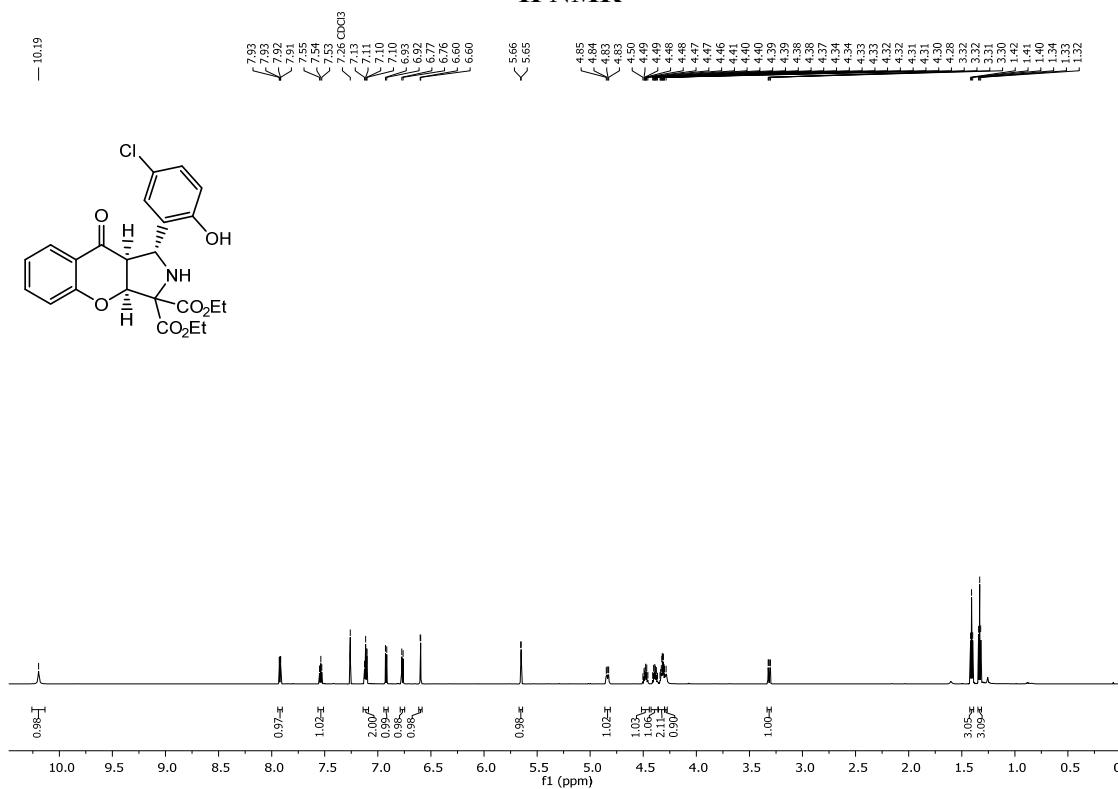


**<sup>13</sup>C NMR**

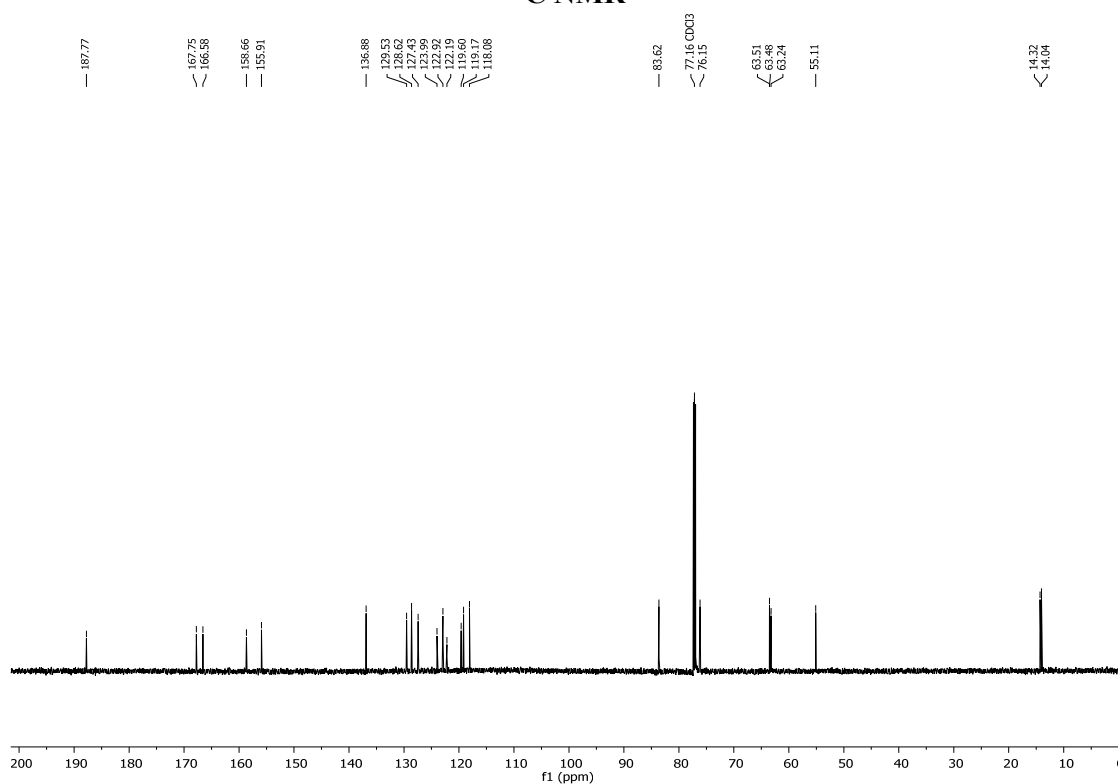


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**<sup>1</sup>H NMR**

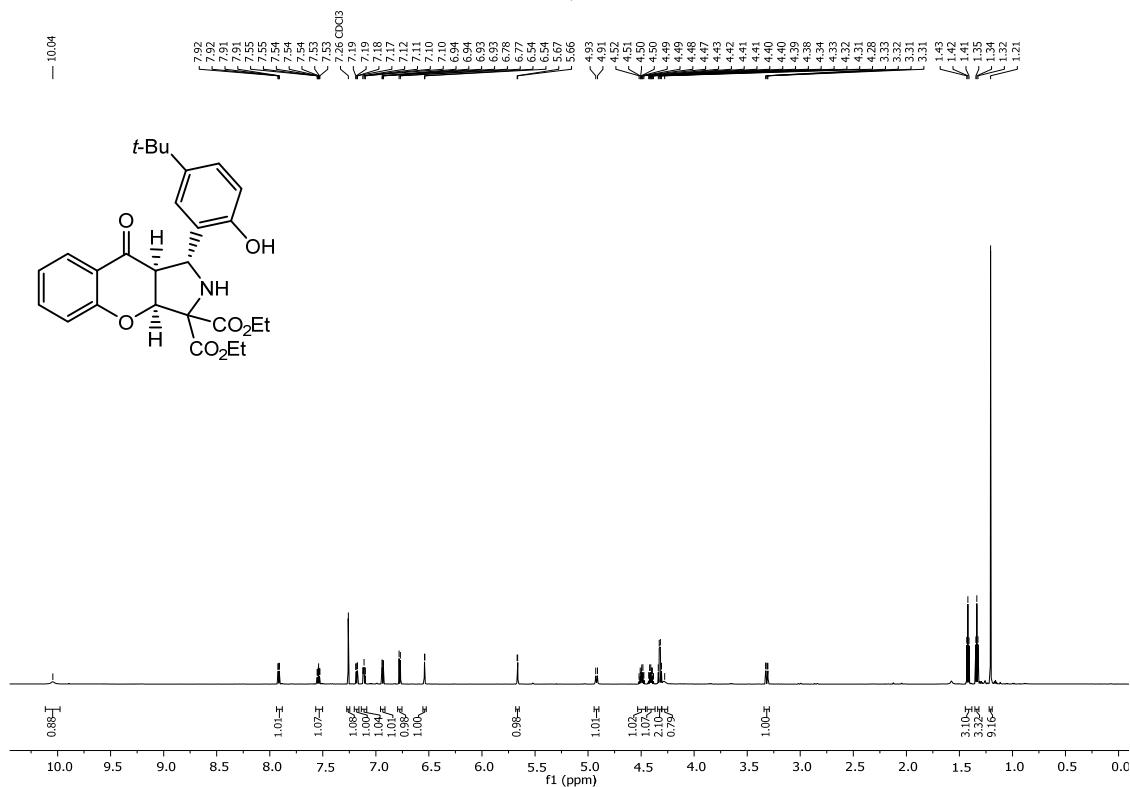


**<sup>13</sup>C NMR**

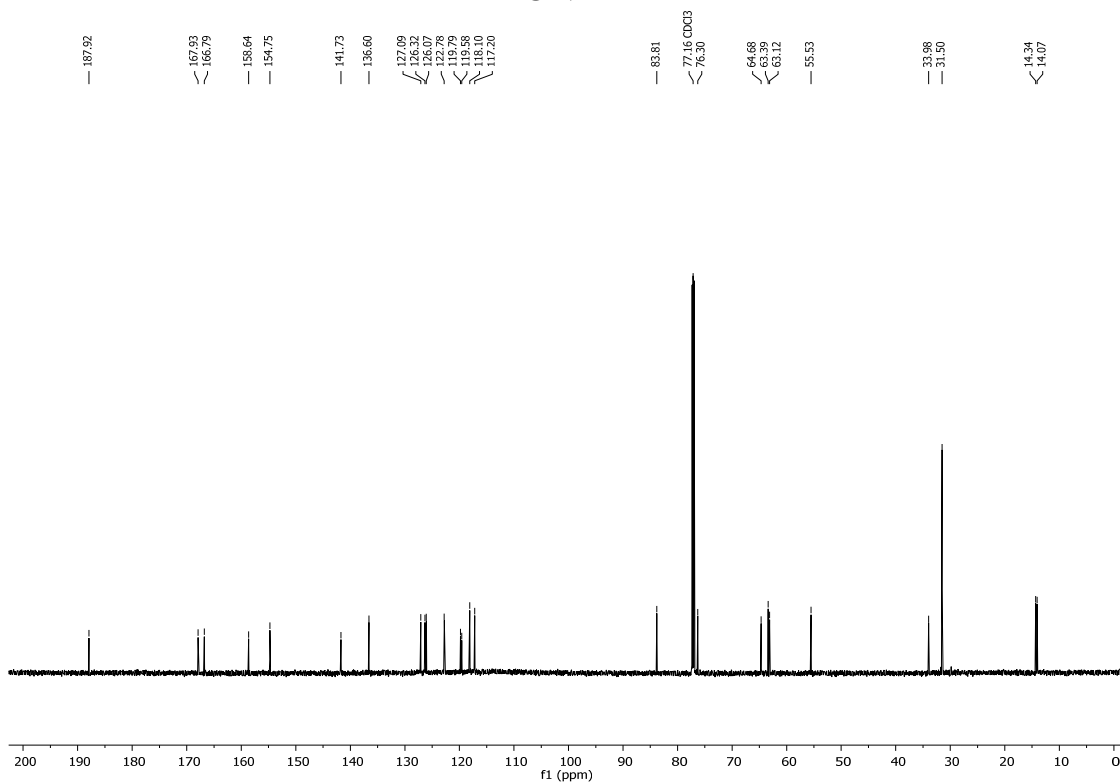


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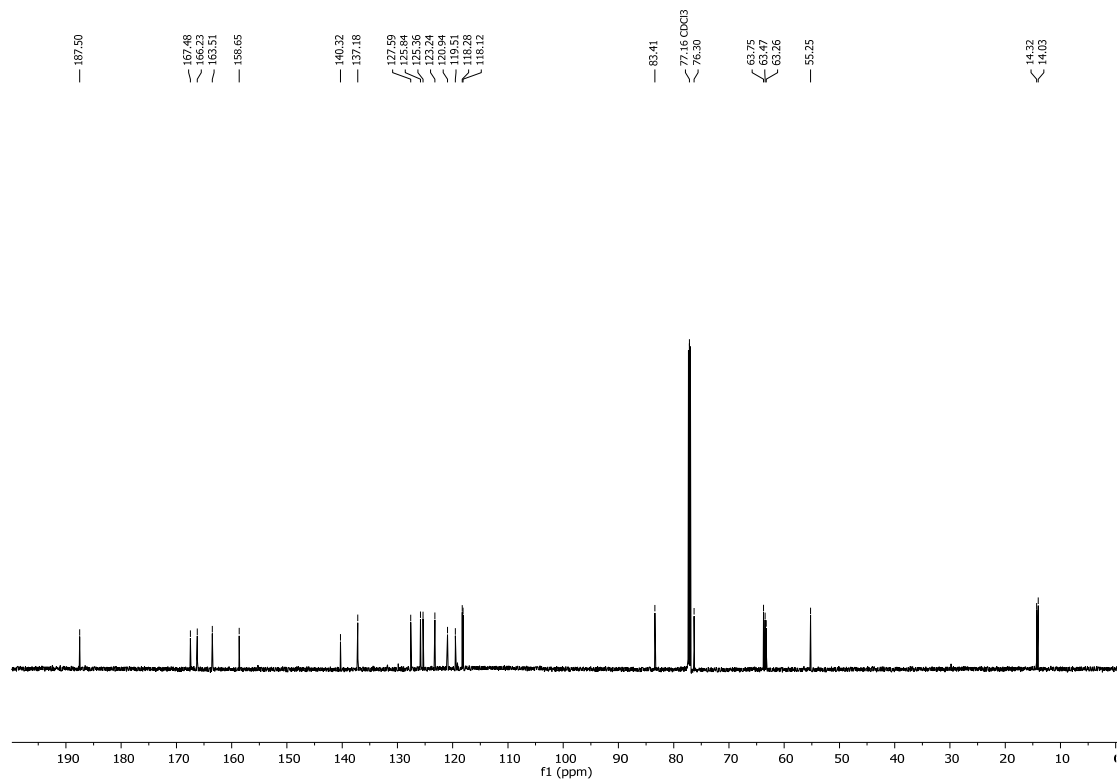
**<sup>1</sup>H NMR**



**<sup>13</sup>C NMR**

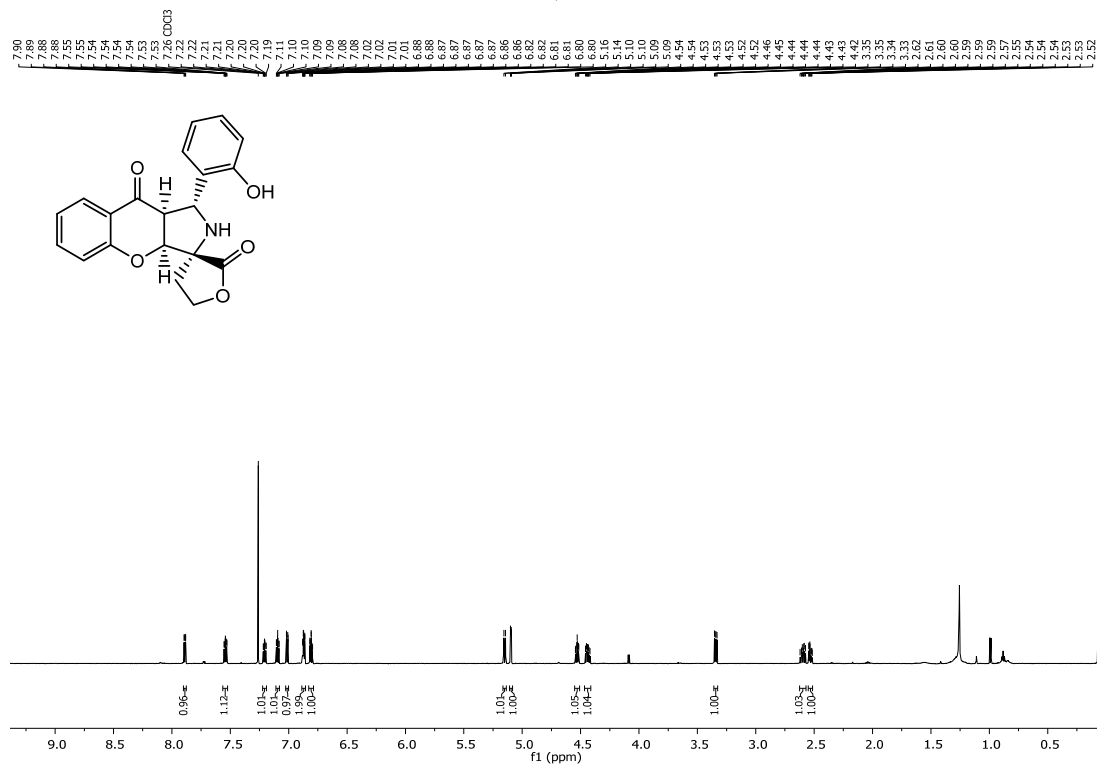


## <sup>1</sup>H NMR

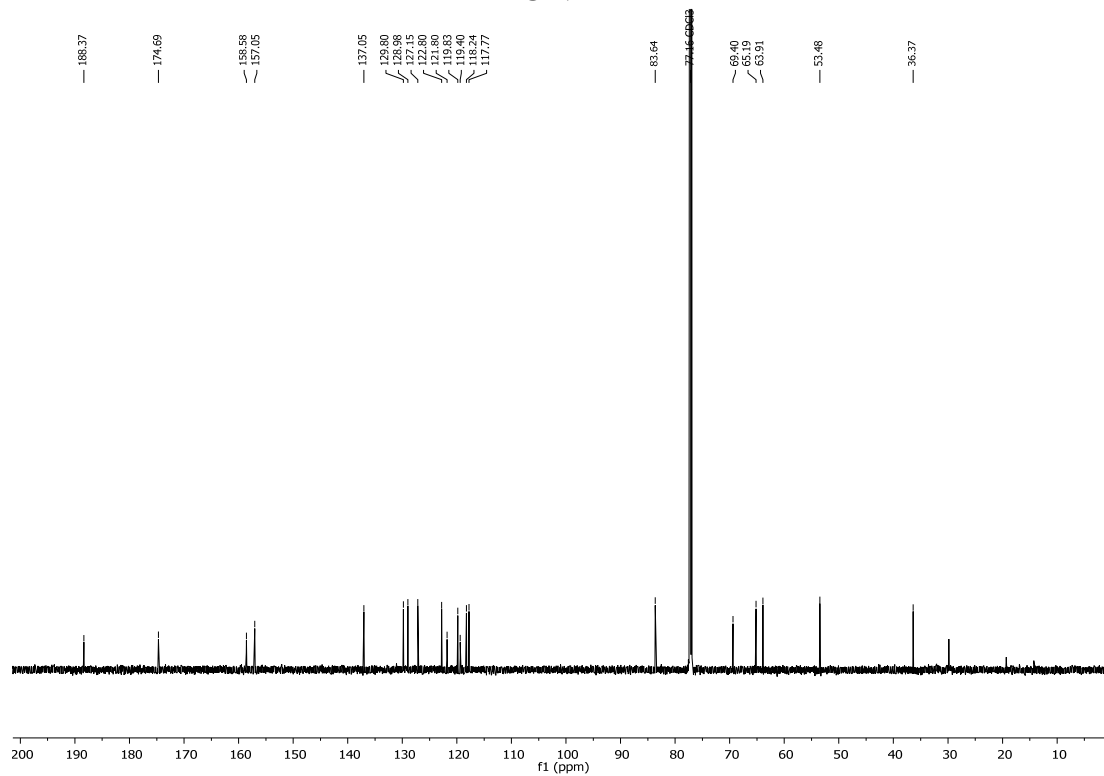


(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1m

<sup>1</sup>H NMR

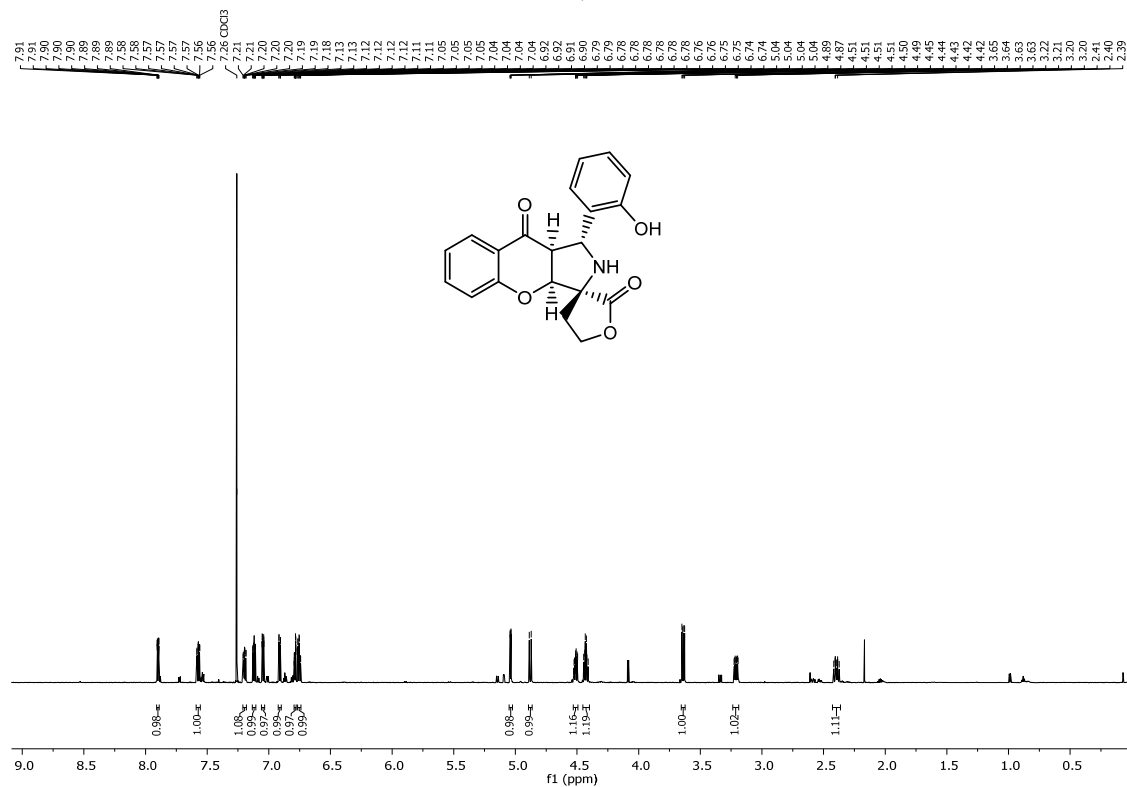


<sup>13</sup>C NMR

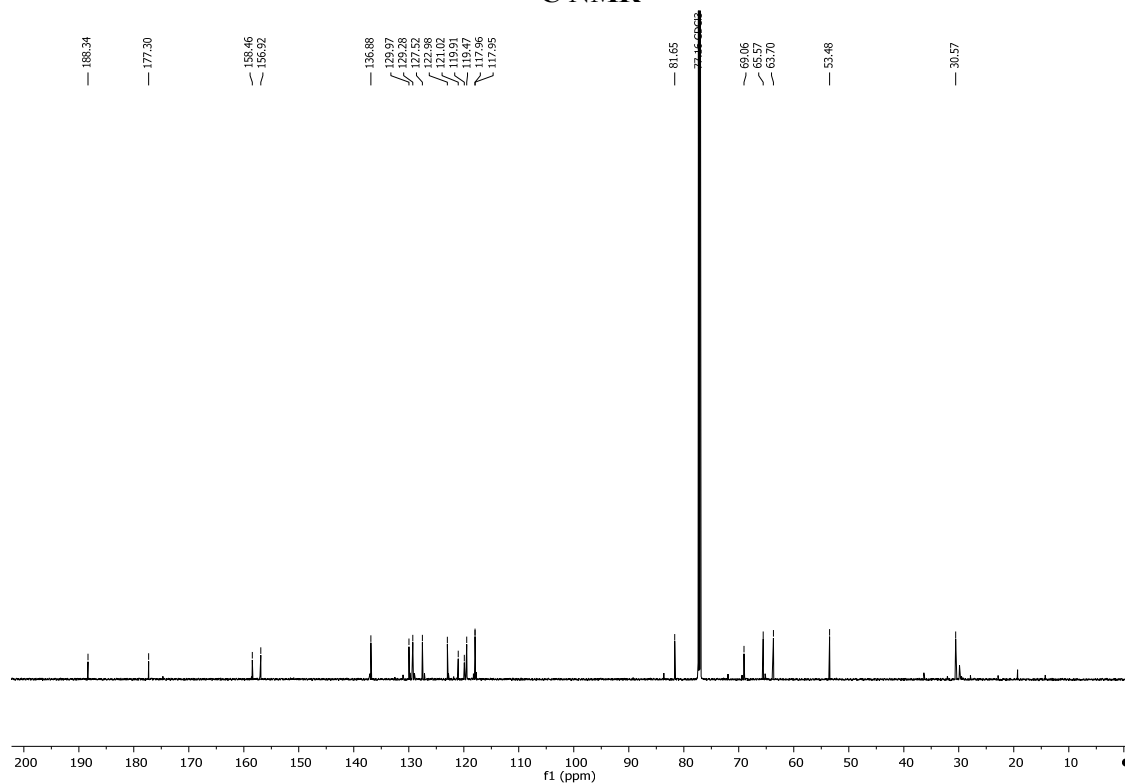


(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
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<sup>1</sup>H NMR

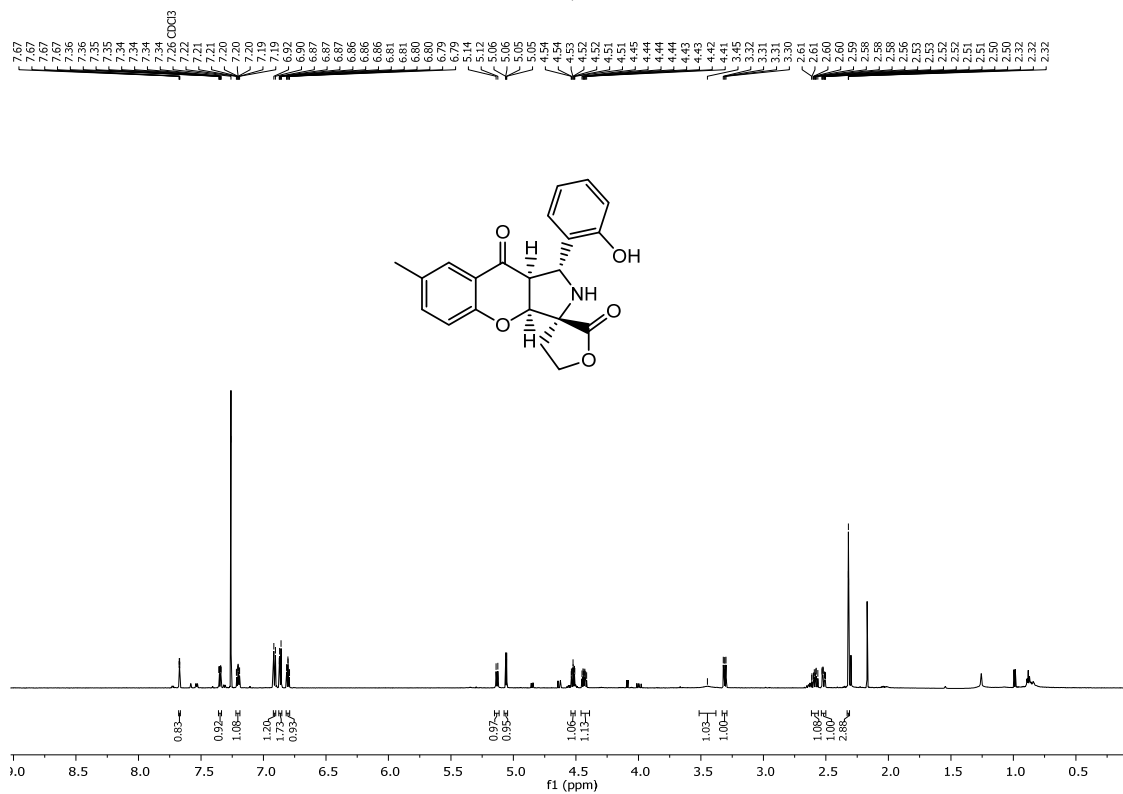


<sup>13</sup>C NMR

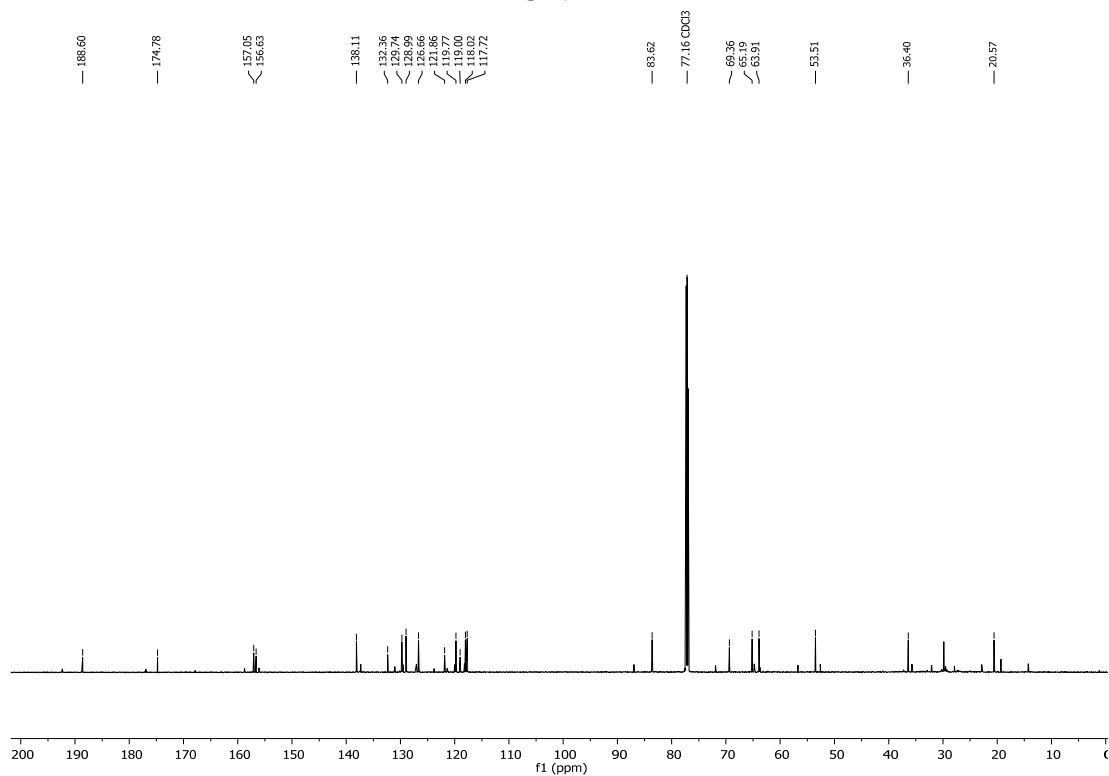


**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-7-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1n**

**<sup>1</sup>H NMR**

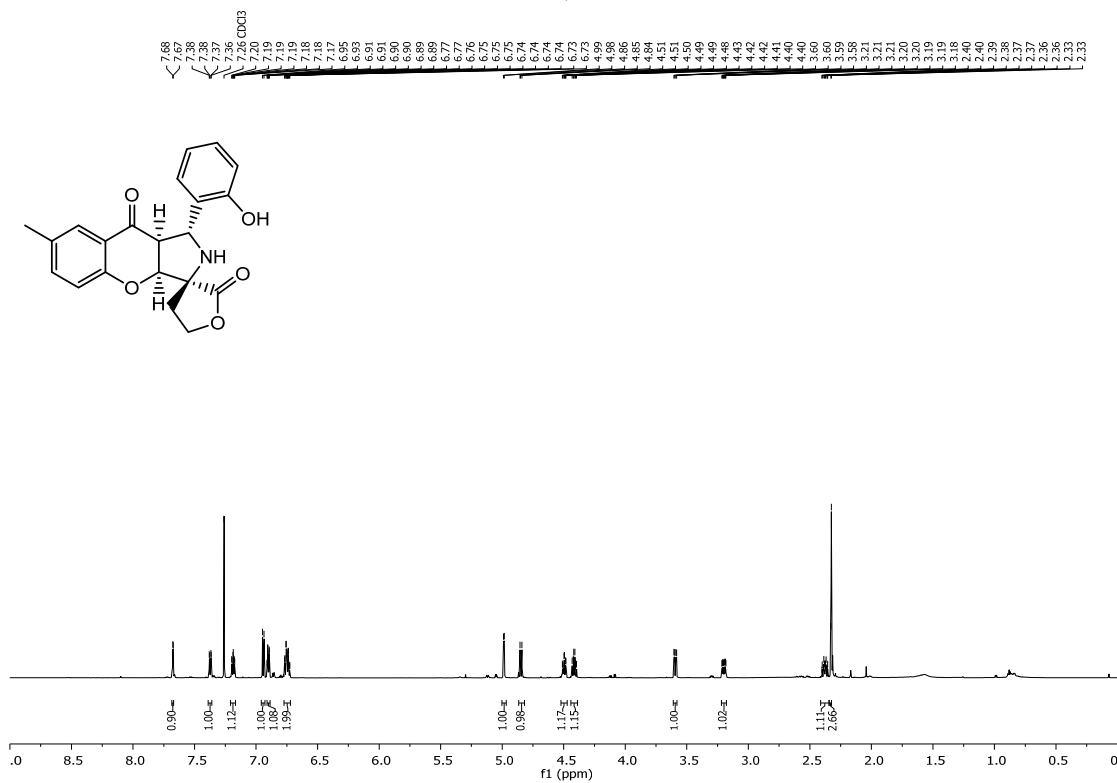


**<sup>13</sup>C NMR**



**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-7-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1n'**

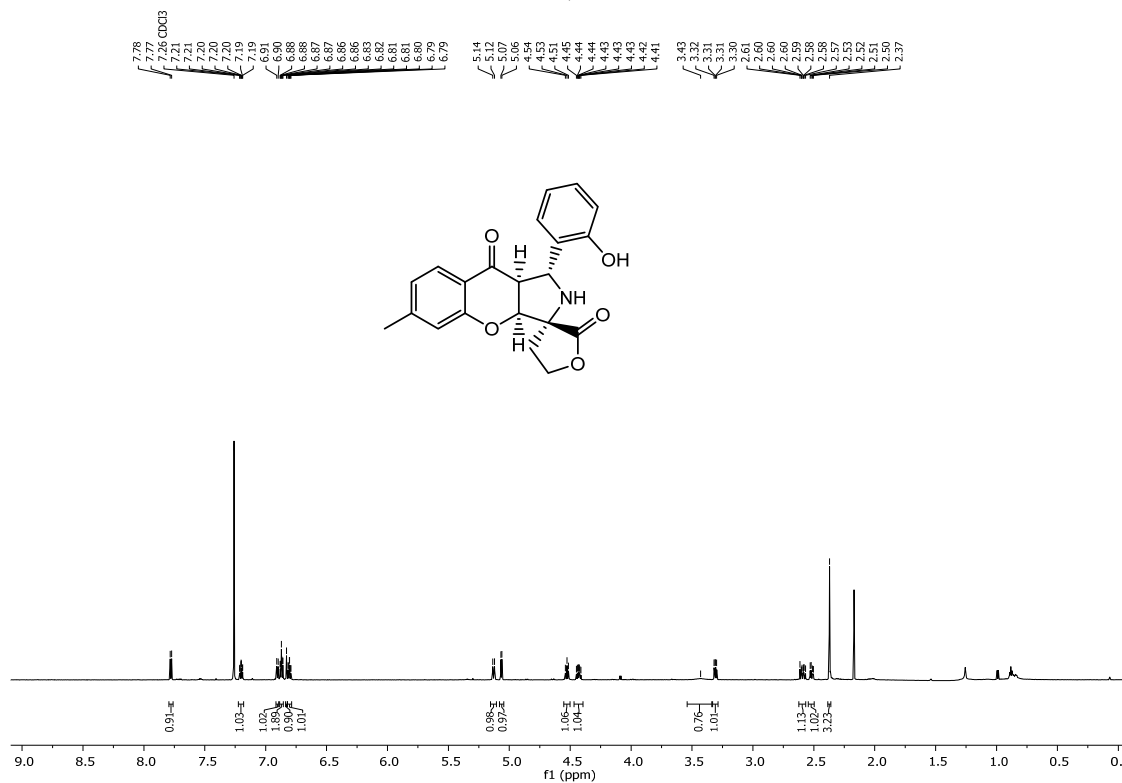
**<sup>1</sup>H NMR**



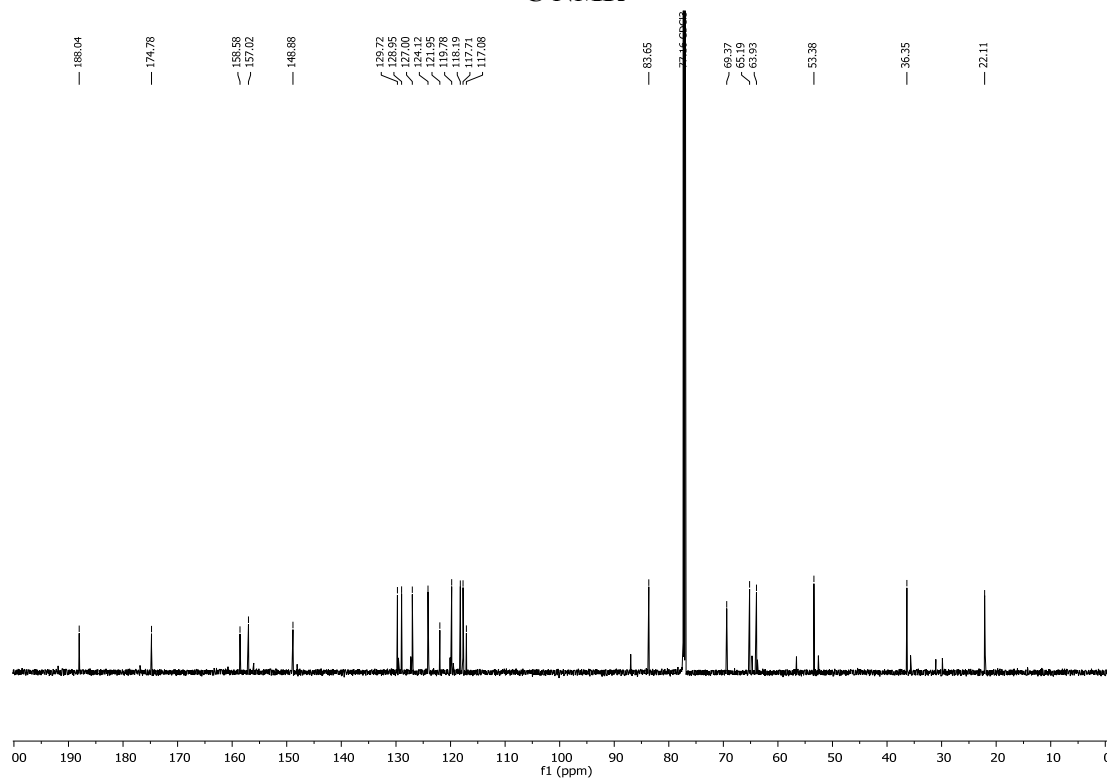


**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1o**

**<sup>1</sup>H NMR**

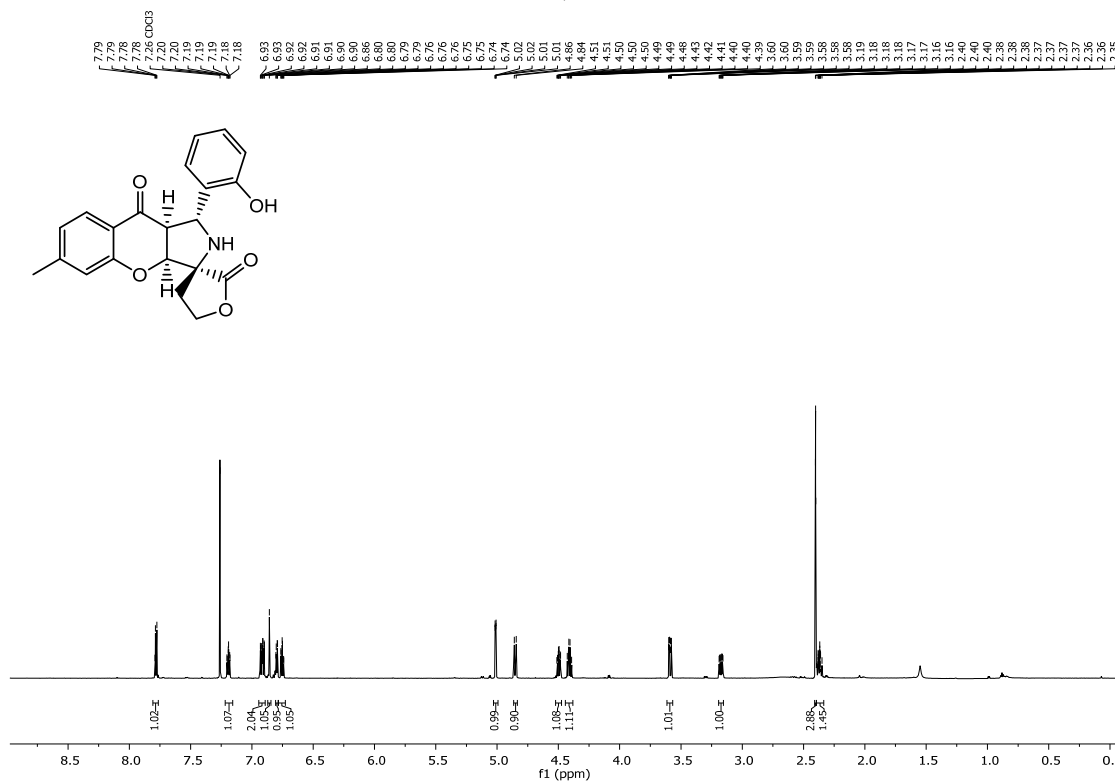


**<sup>13</sup>C NMR**

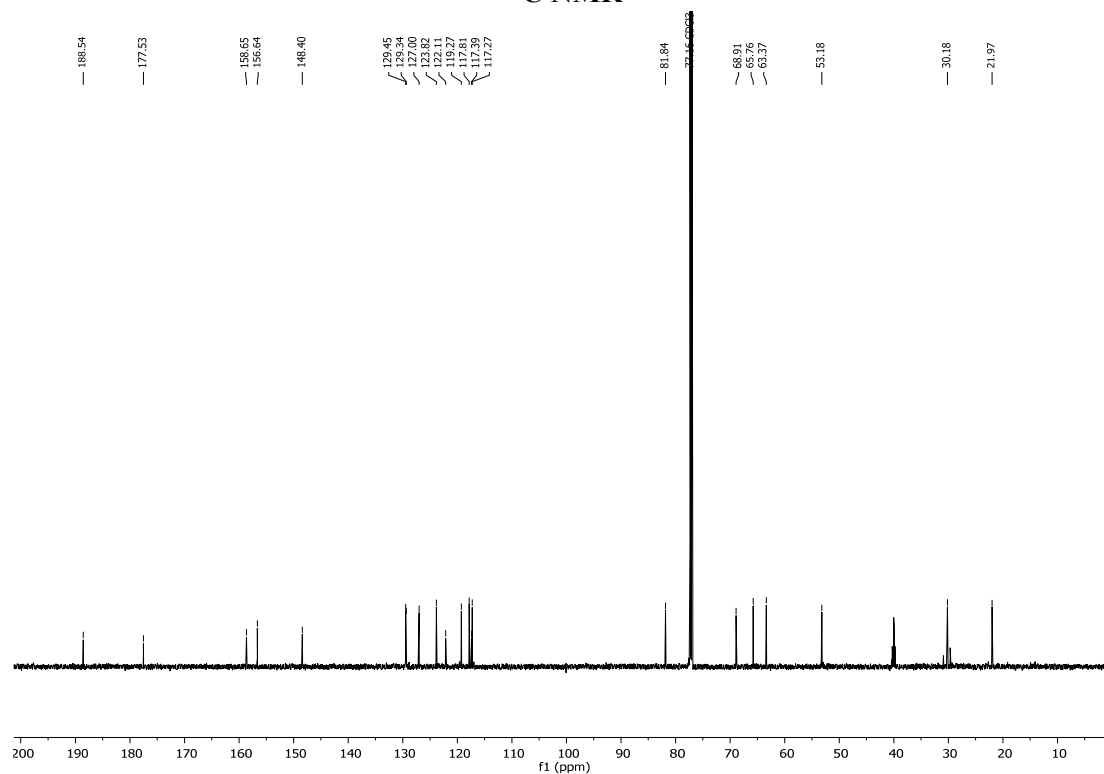


**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1o'**

**<sup>1</sup>H NMR**

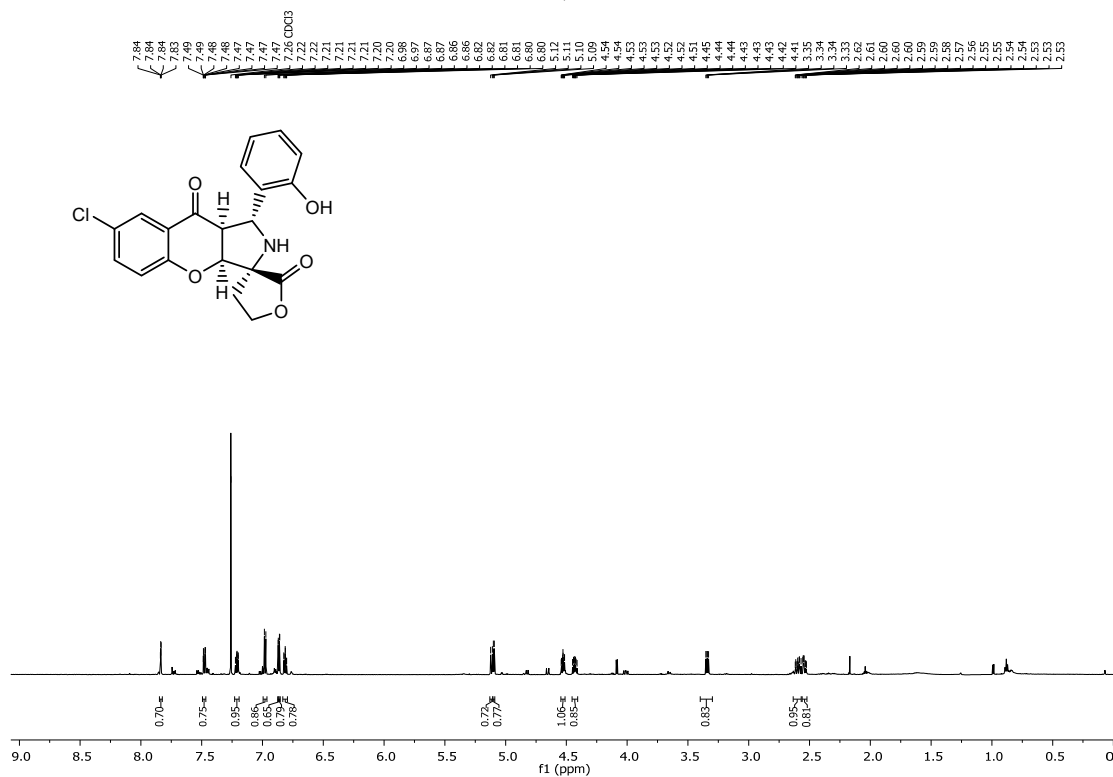


**<sup>13</sup>C NMR**

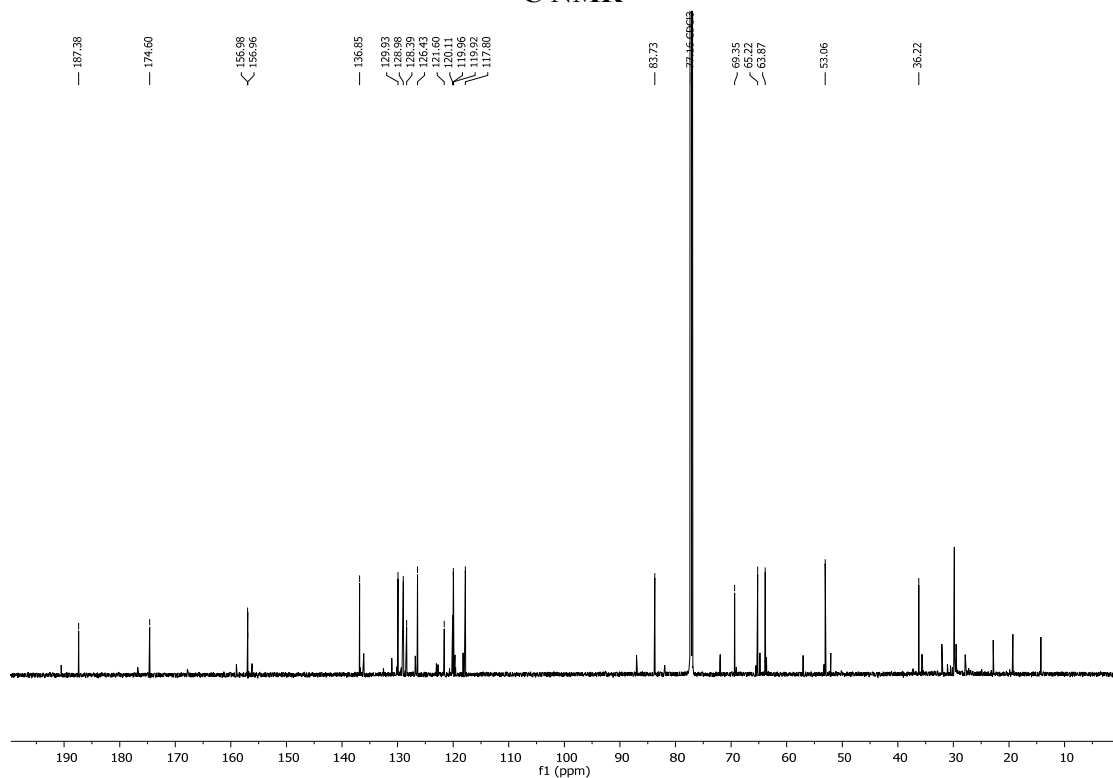


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spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1p**

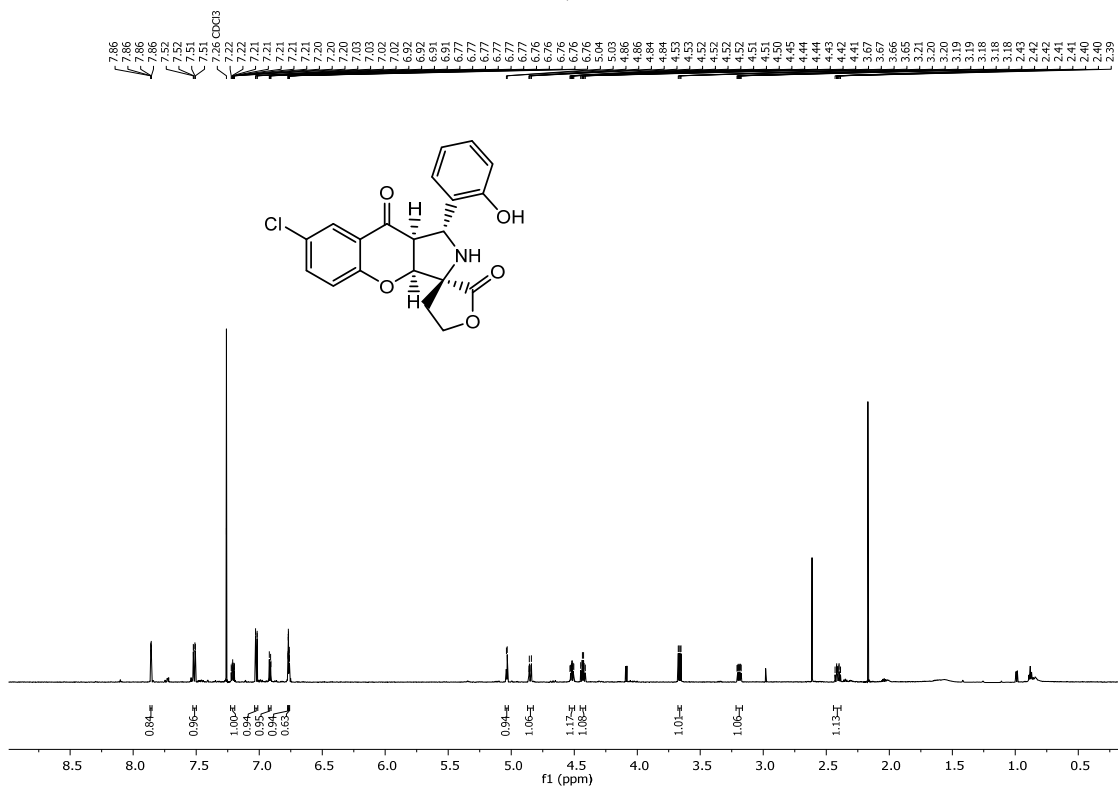
**<sup>1</sup>H NMR**



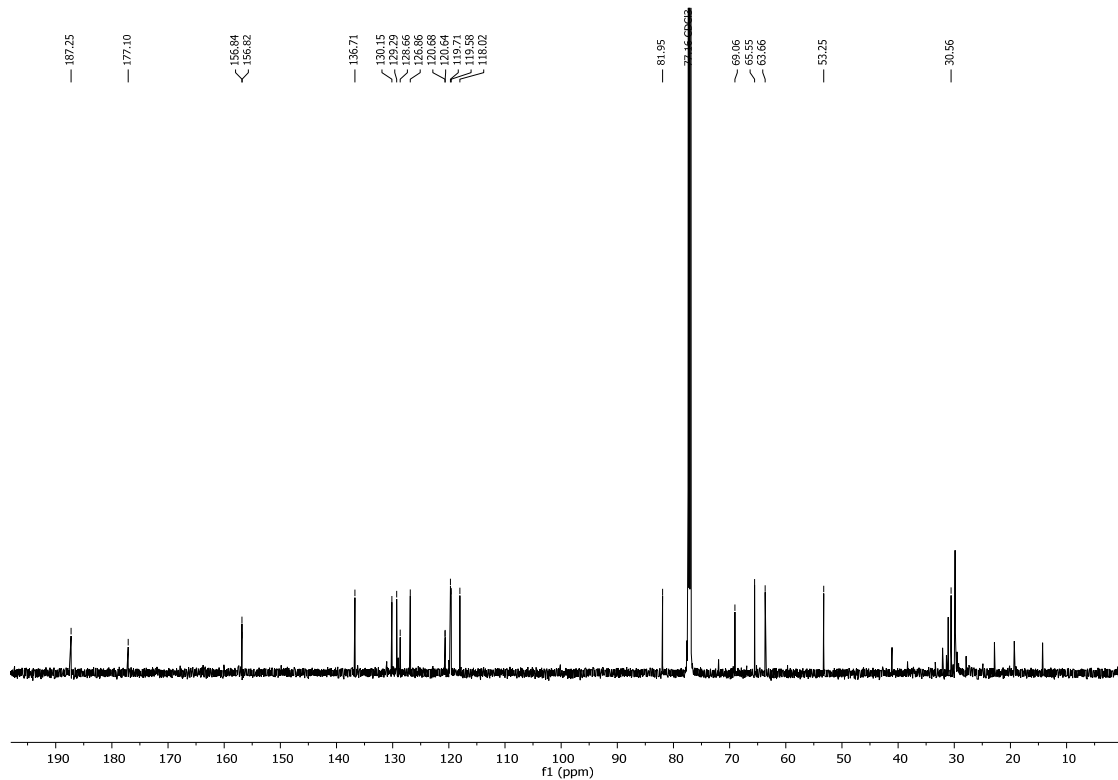
**<sup>13</sup>C NMR**



### <sup>1</sup>H NMR

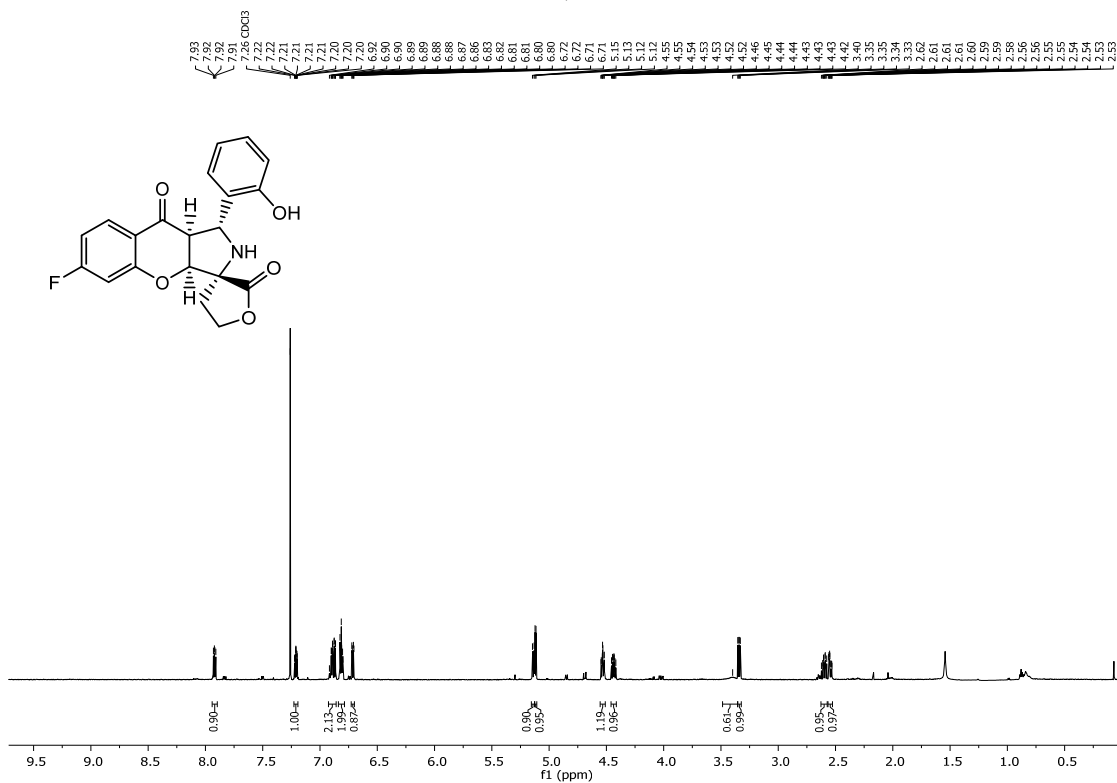


### <sup>13</sup>C NMR

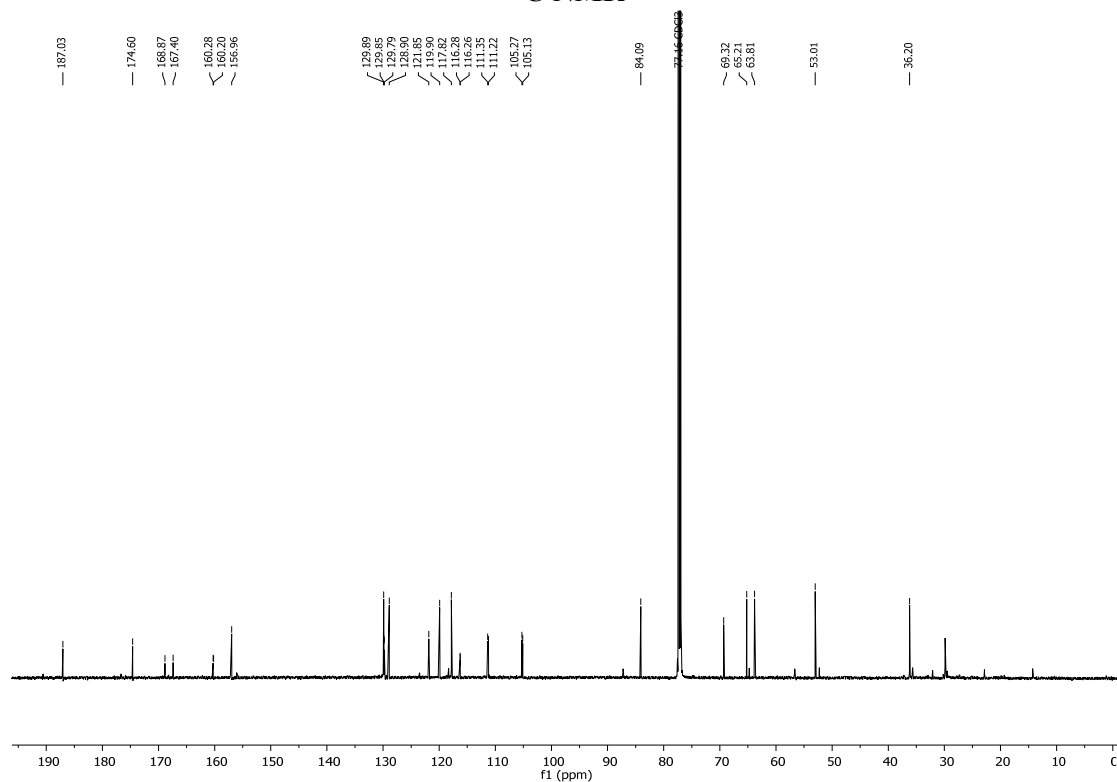


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spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1r**

**<sup>1</sup>H NMR**

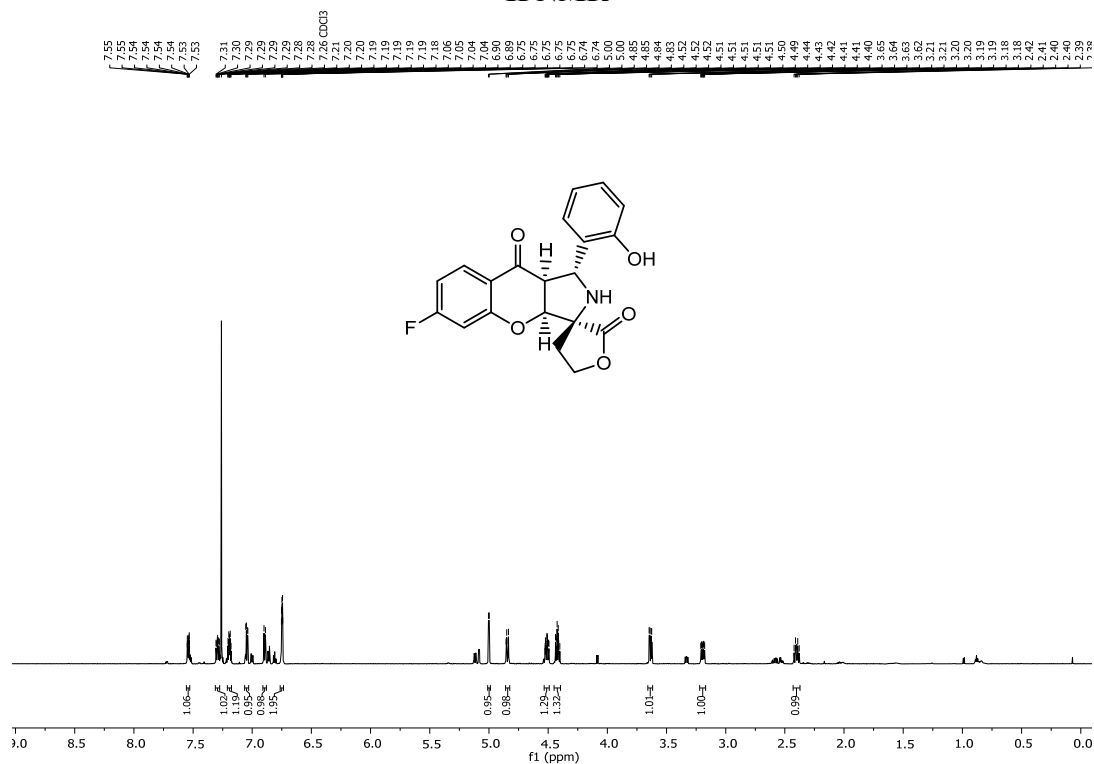


**<sup>13</sup>C NMR**

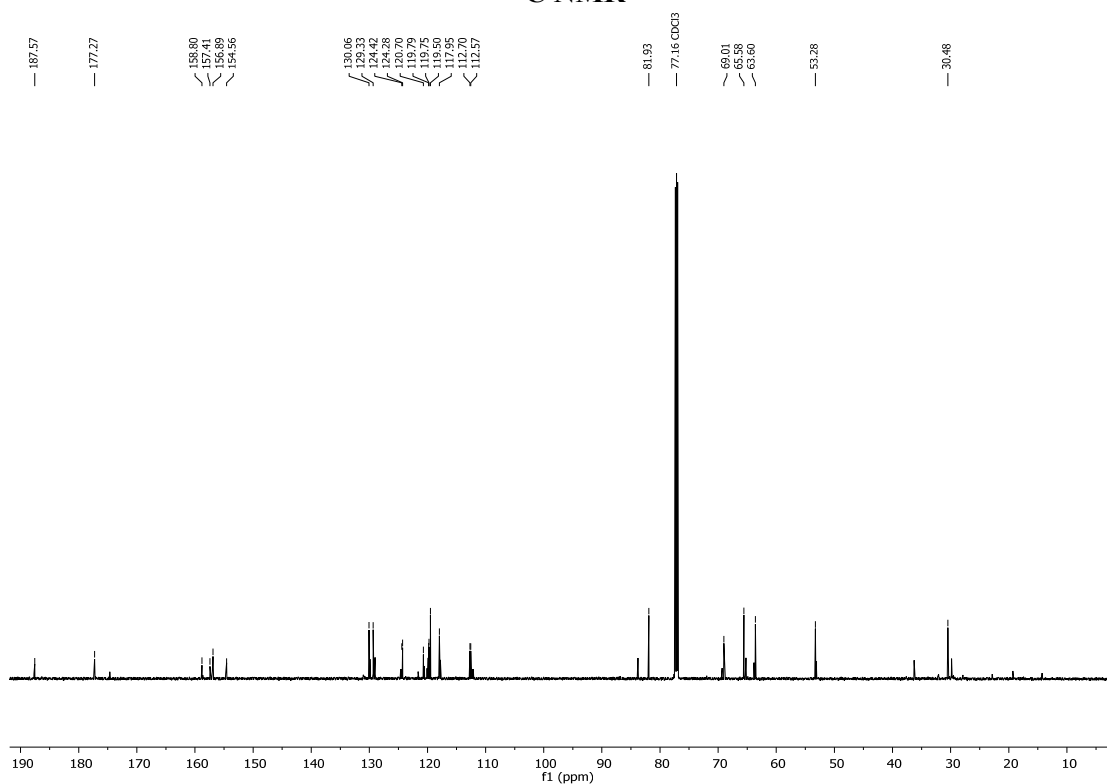


**(1*R*,3*S*,3*aS*,9*aS*)-6-Fluoro-1-(2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1*r*'**

**<sup>1</sup>H NMR**

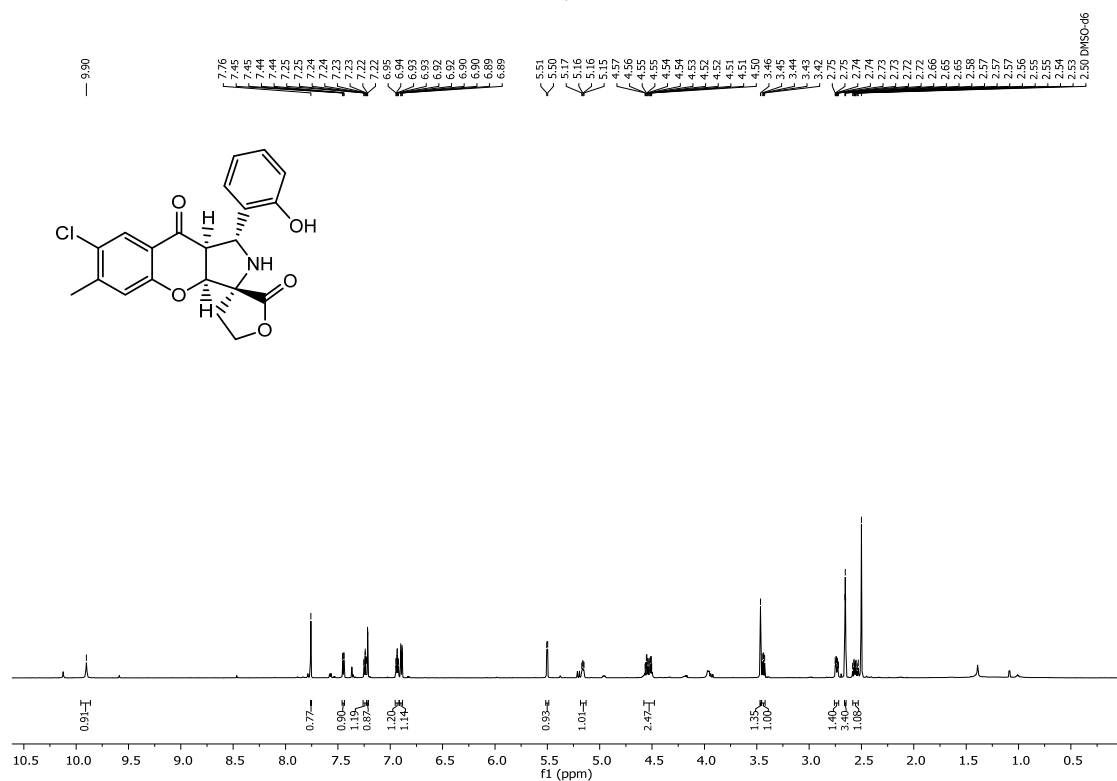


**<sup>13</sup>C NMR**

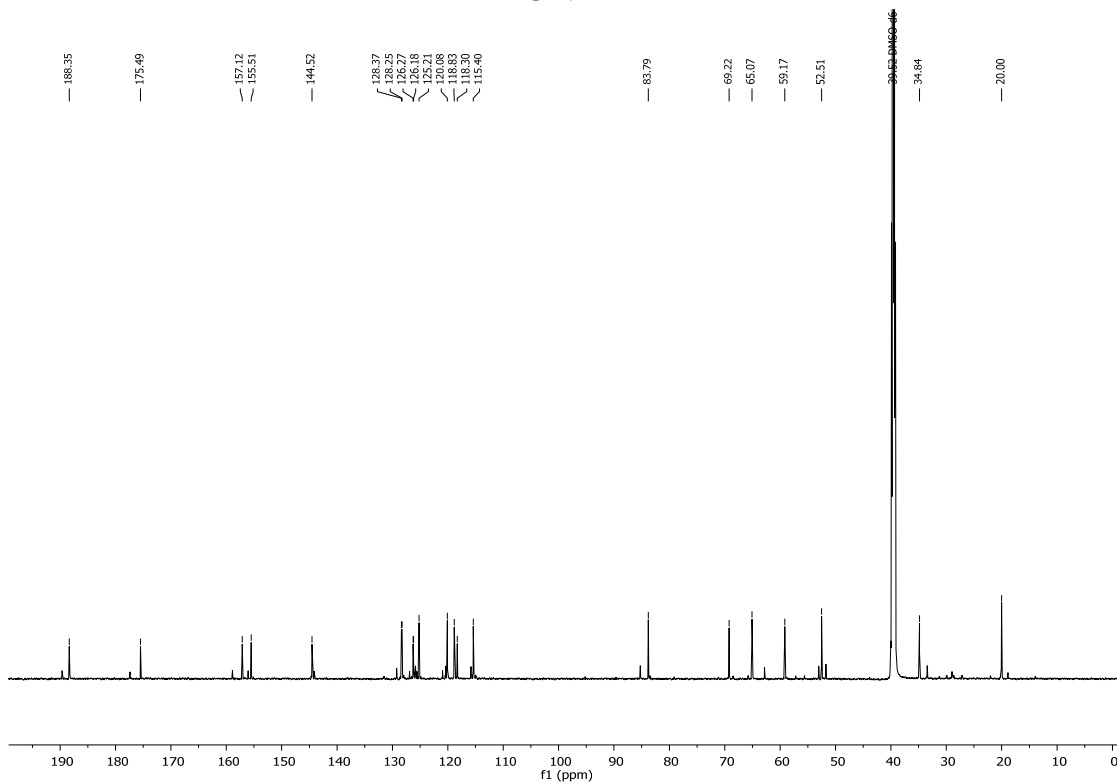


**(1*R*,3*R*,3*aS*,9*aS*)-7-Chloro-1-(2-hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1s**

**<sup>1</sup>H NMR**

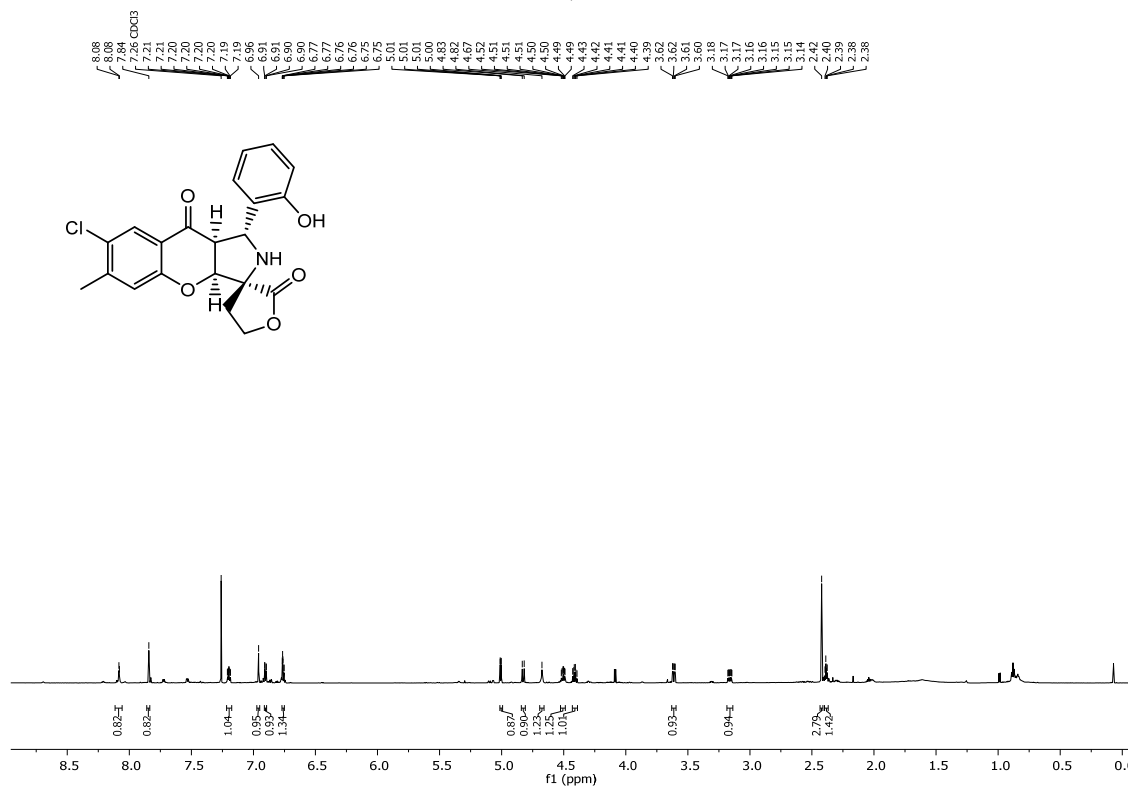


**<sup>13</sup>C NMR**

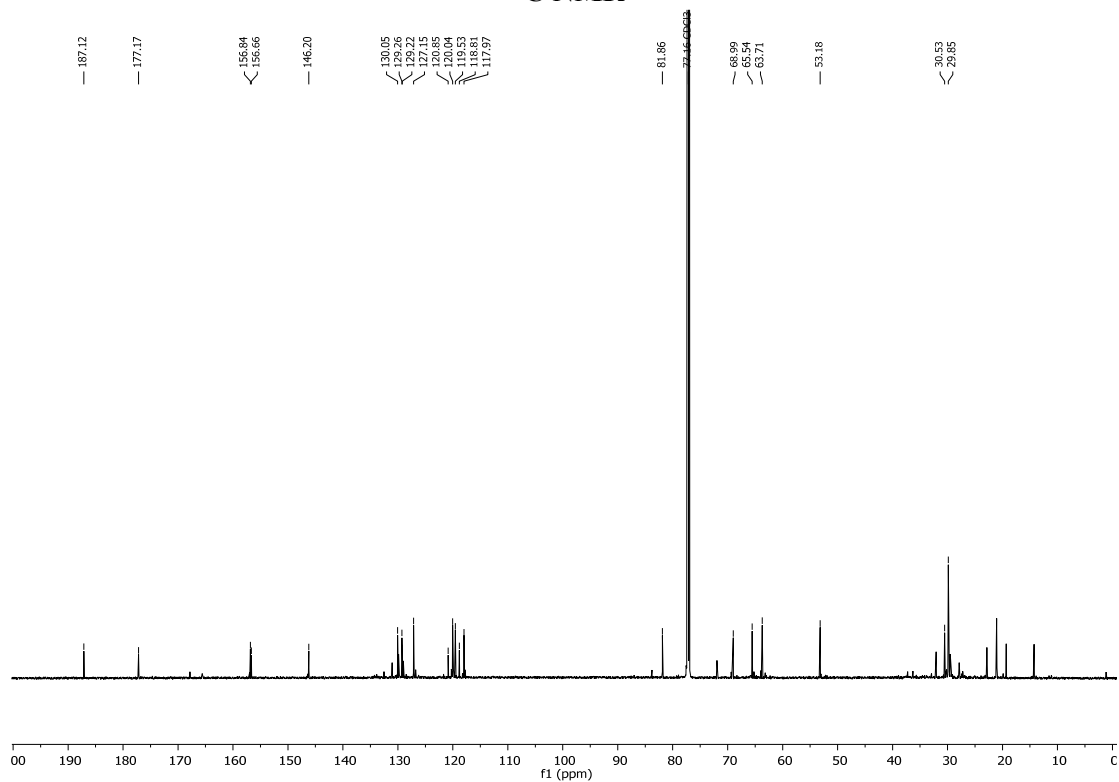


**(1*R*,3*S*,3*aS*,9*aS*)-7-Chloro-1-(2-hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1*s*'**

**<sup>1</sup>H NMR**



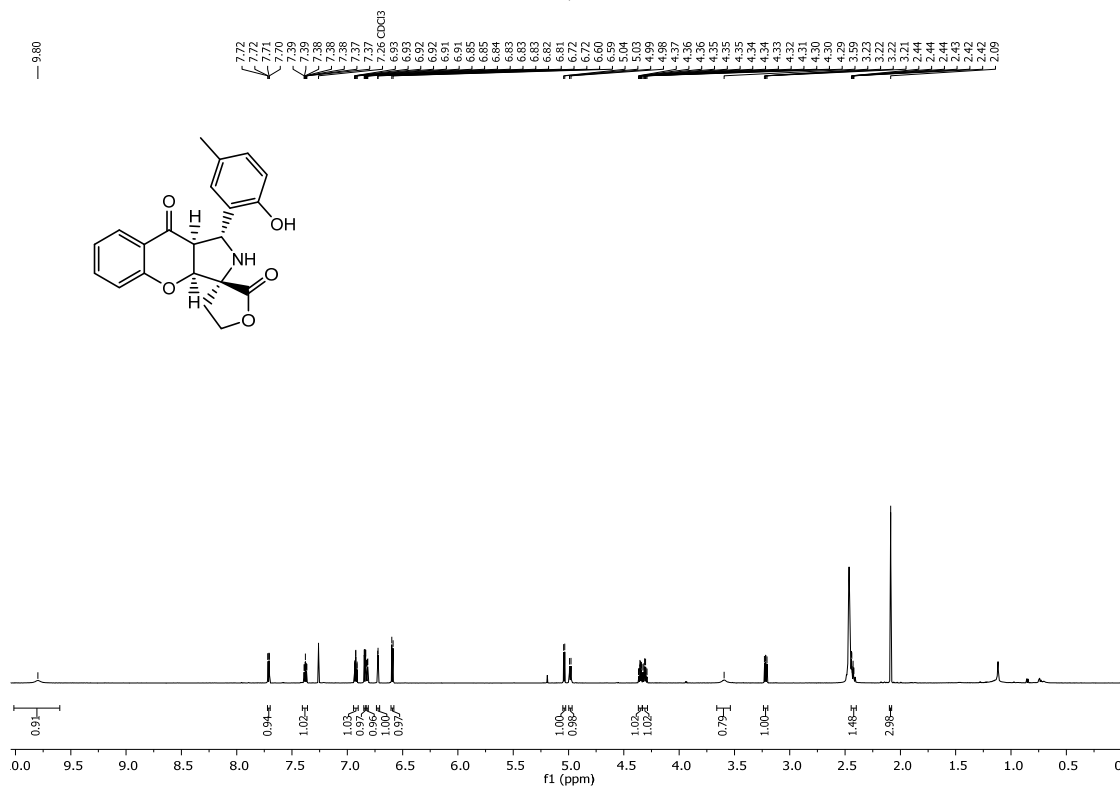
**<sup>13</sup>C NMR**



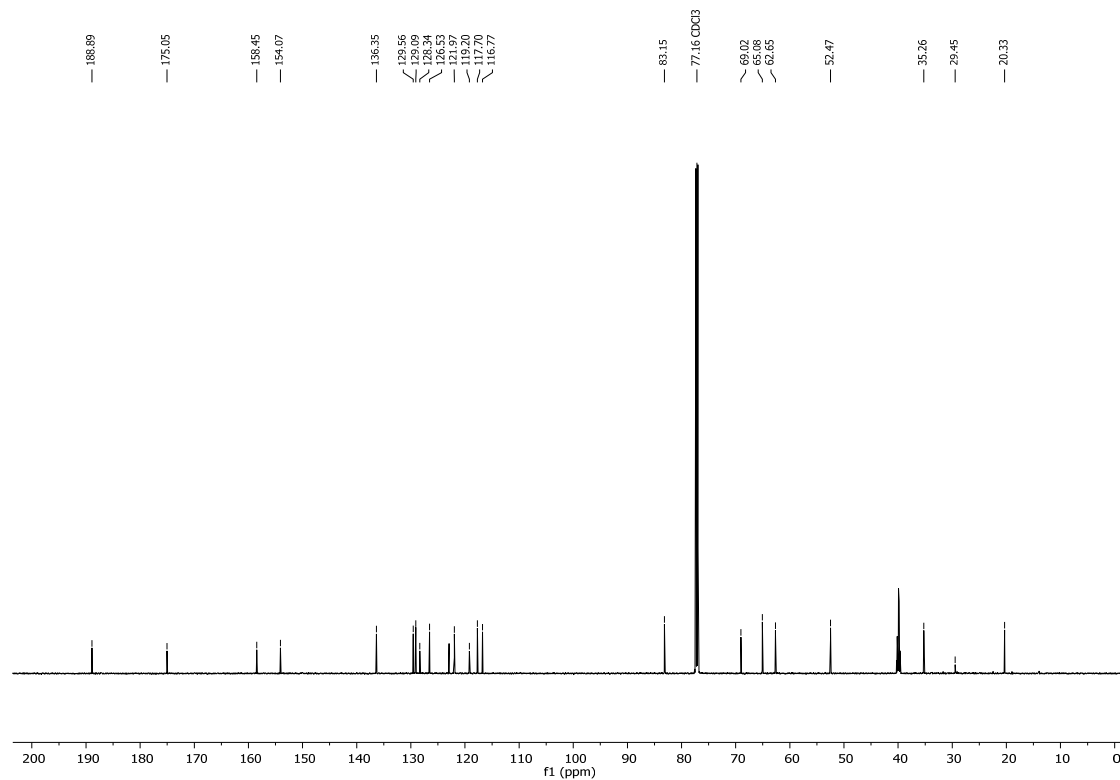


**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxy-5-methylphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1t**

**<sup>1</sup>H NMR**

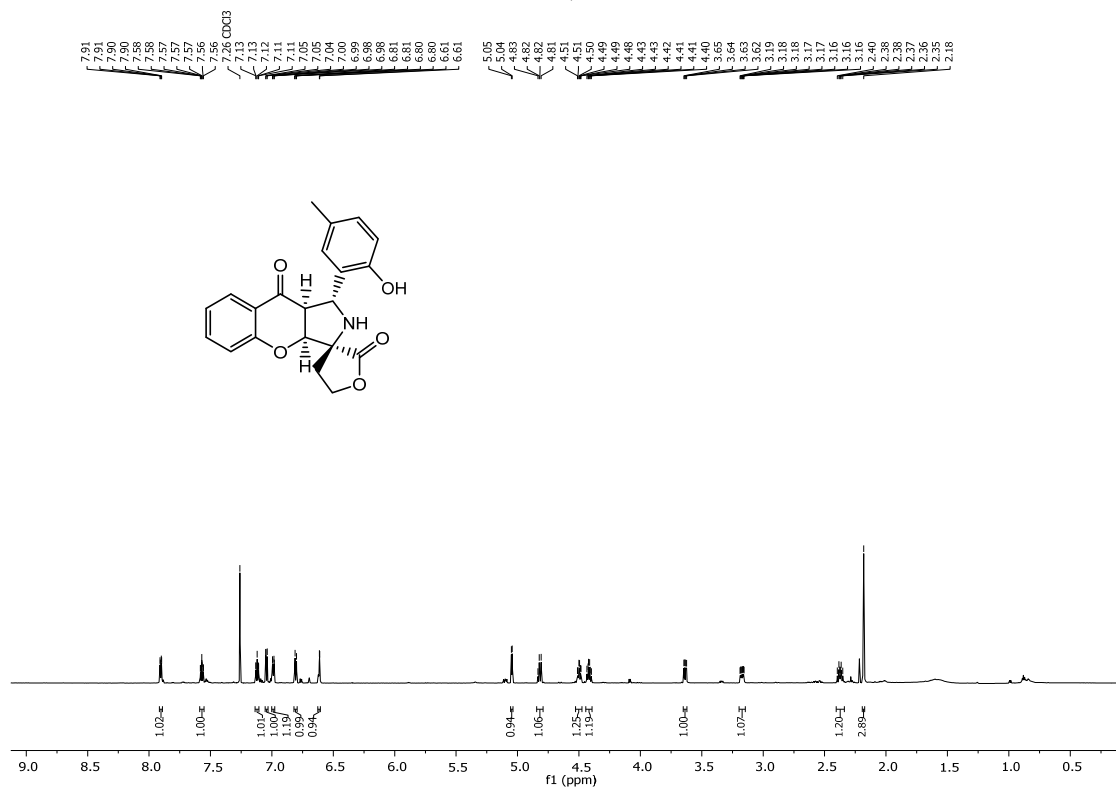


**<sup>13</sup>C NMR**

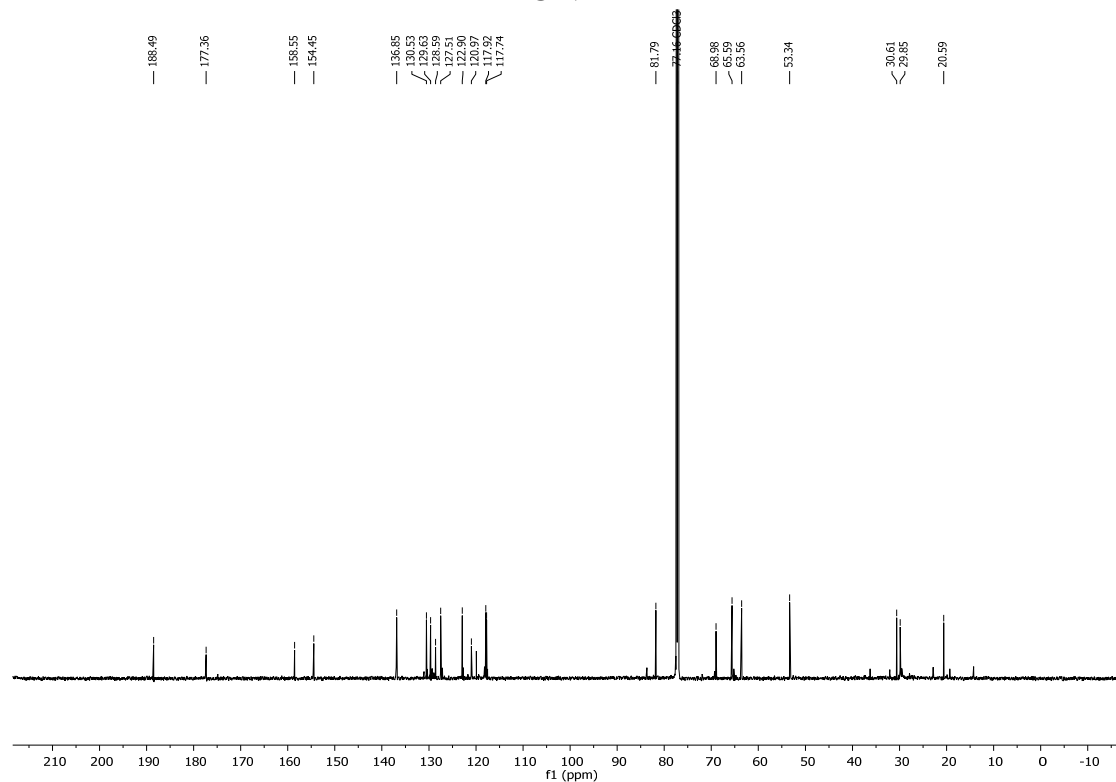


**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxy-5-methylphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1t'**

**<sup>1</sup>H NMR**

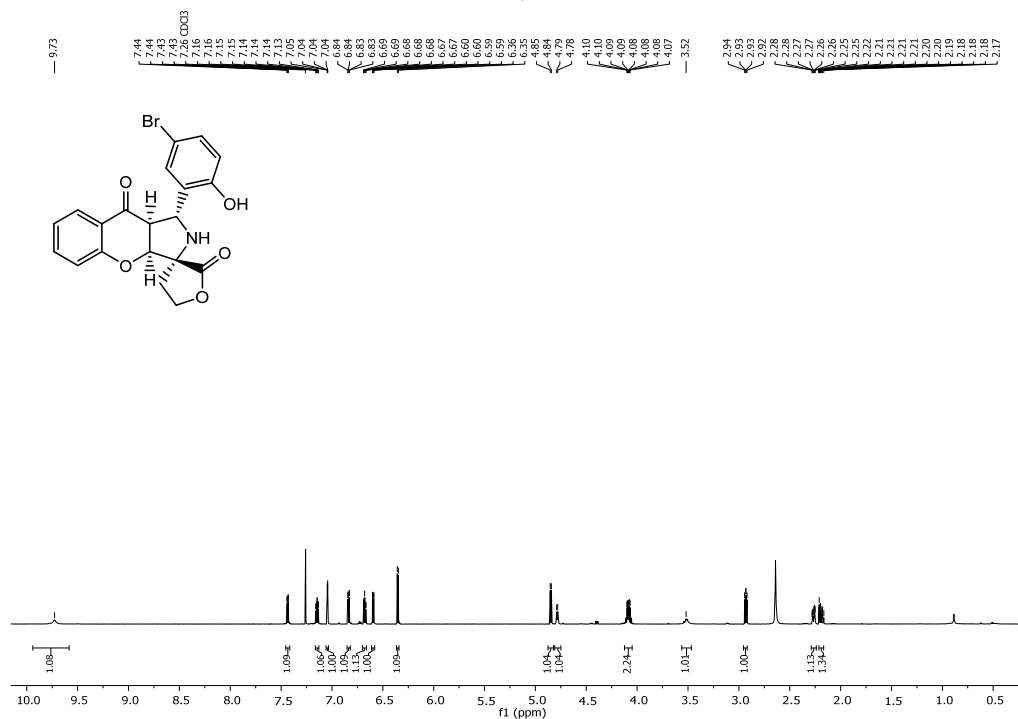


**<sup>13</sup>C NMR**

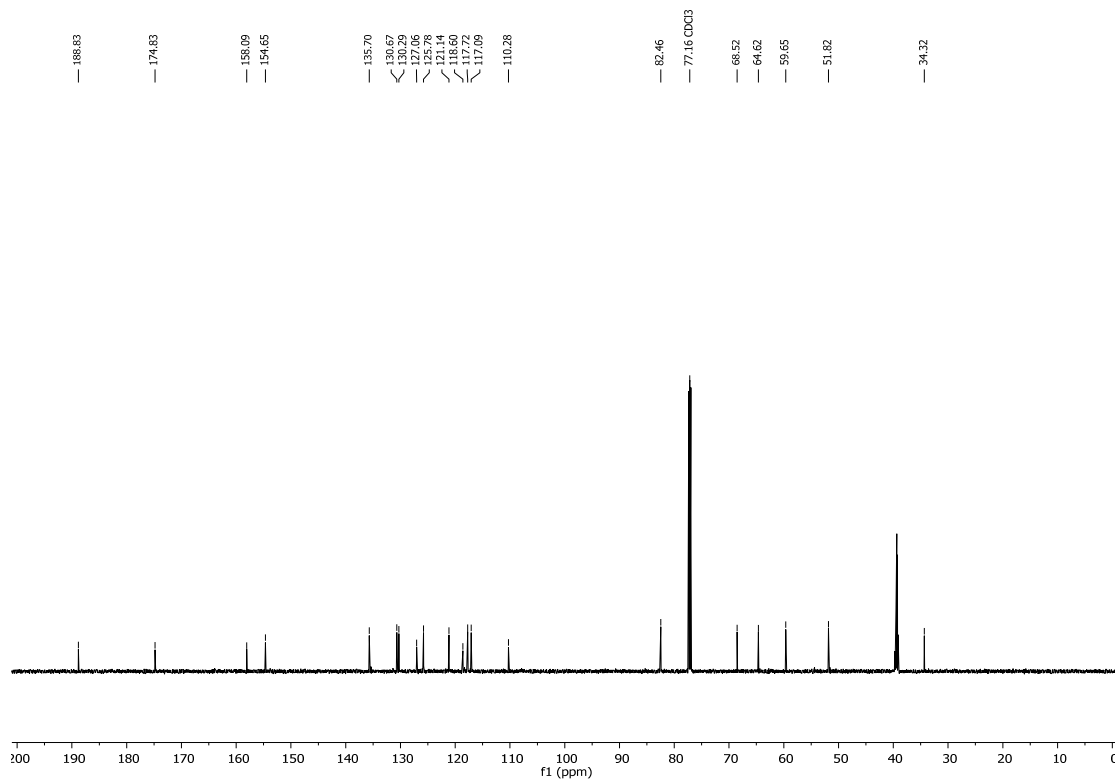


**(1*R*,3*R*,3*aS*,9*aS*)-1-(5-Bromo-2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1u**

**<sup>1</sup>H NMR**

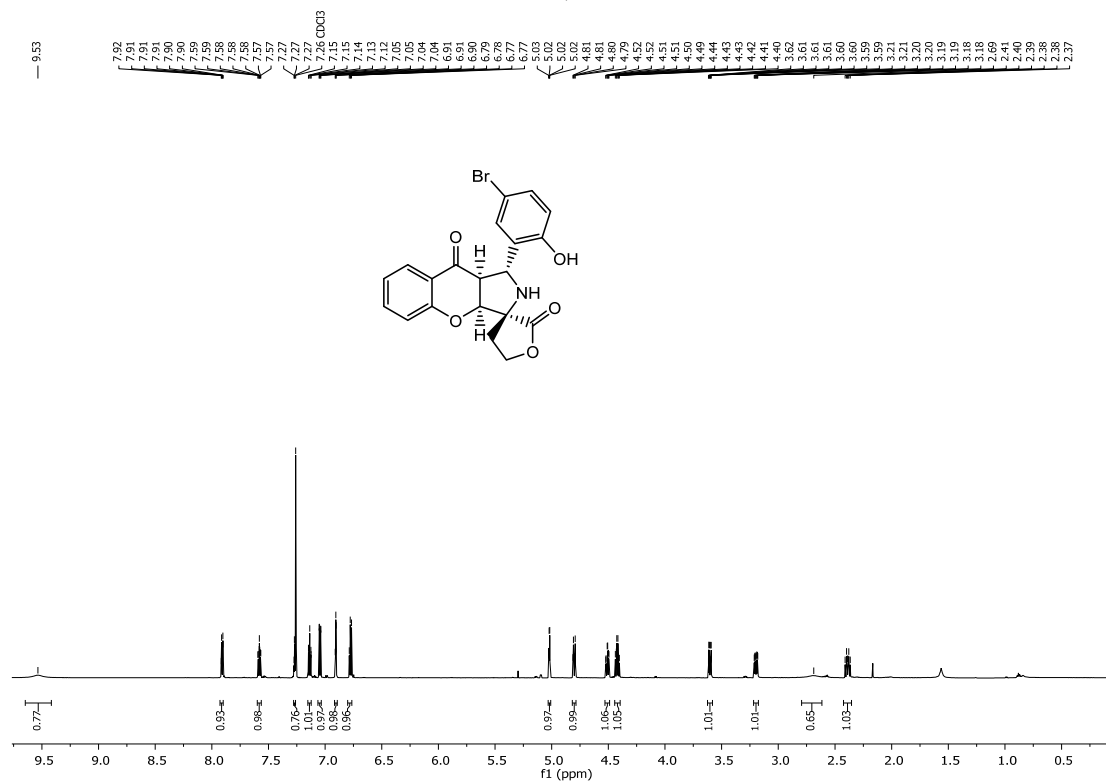


**<sup>13</sup>C NMR**

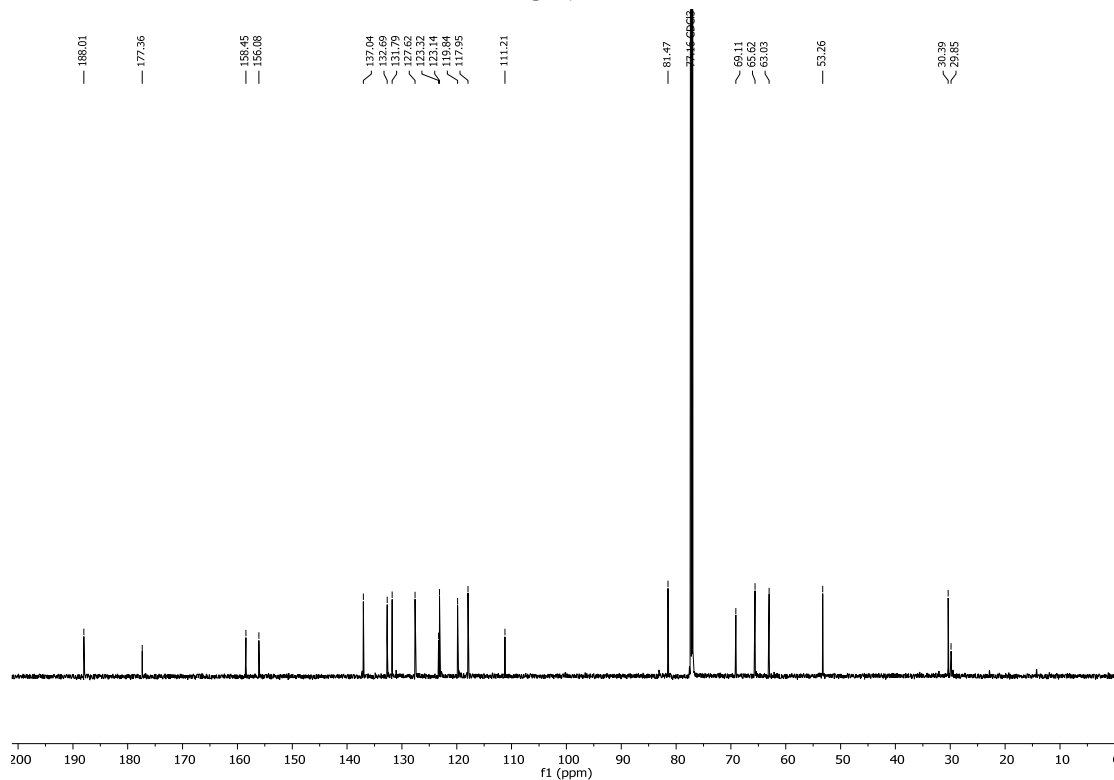


**(1*R*,3*S*,3*aS*,9*aS*)-1-(5-Bromo-2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1u'**

**<sup>1</sup>H NMR**



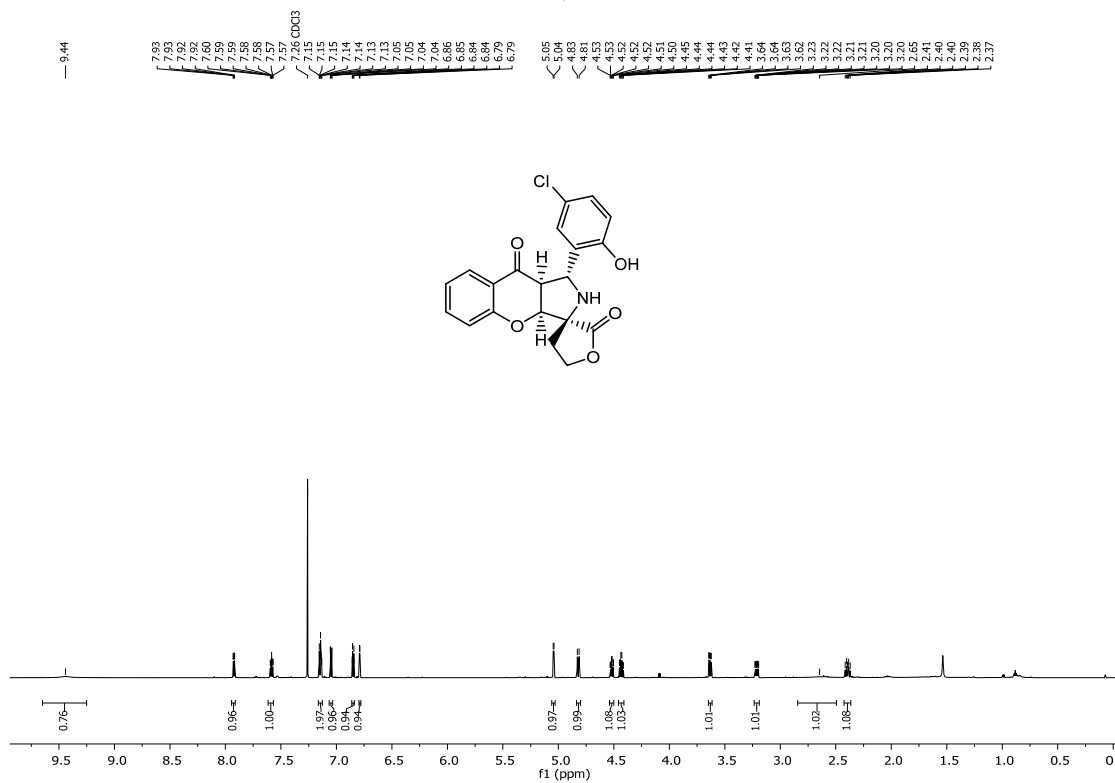
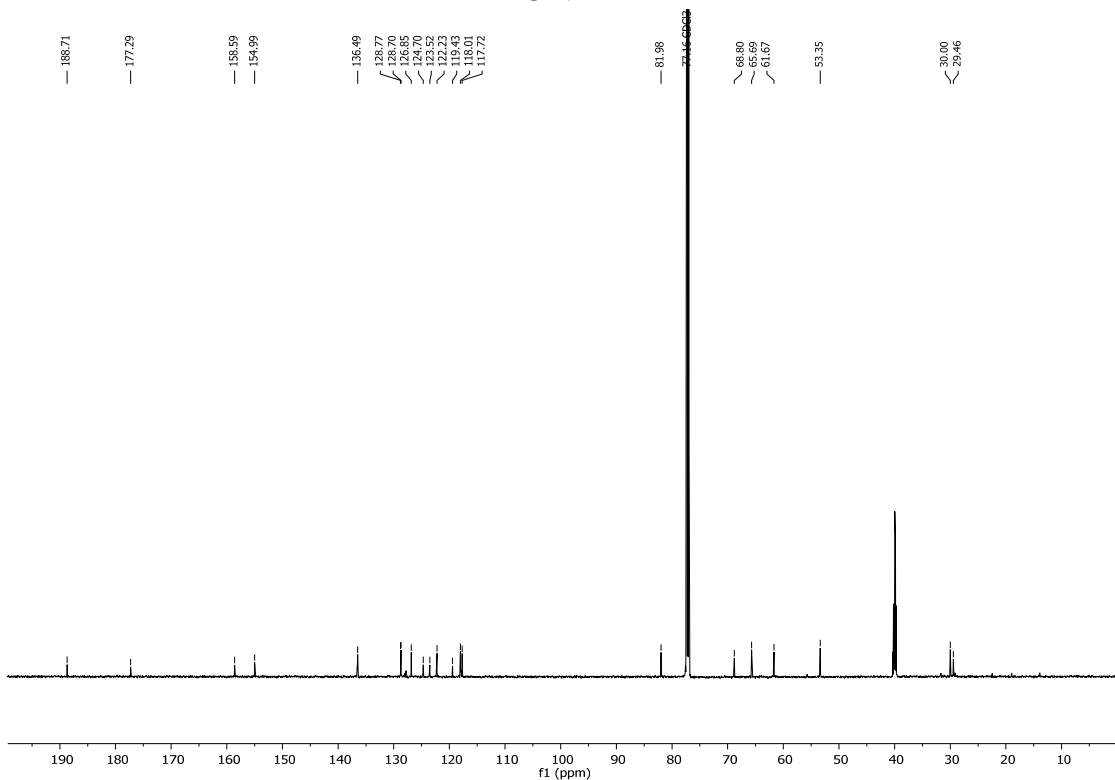
**<sup>13</sup>C NMR**



[illegible]

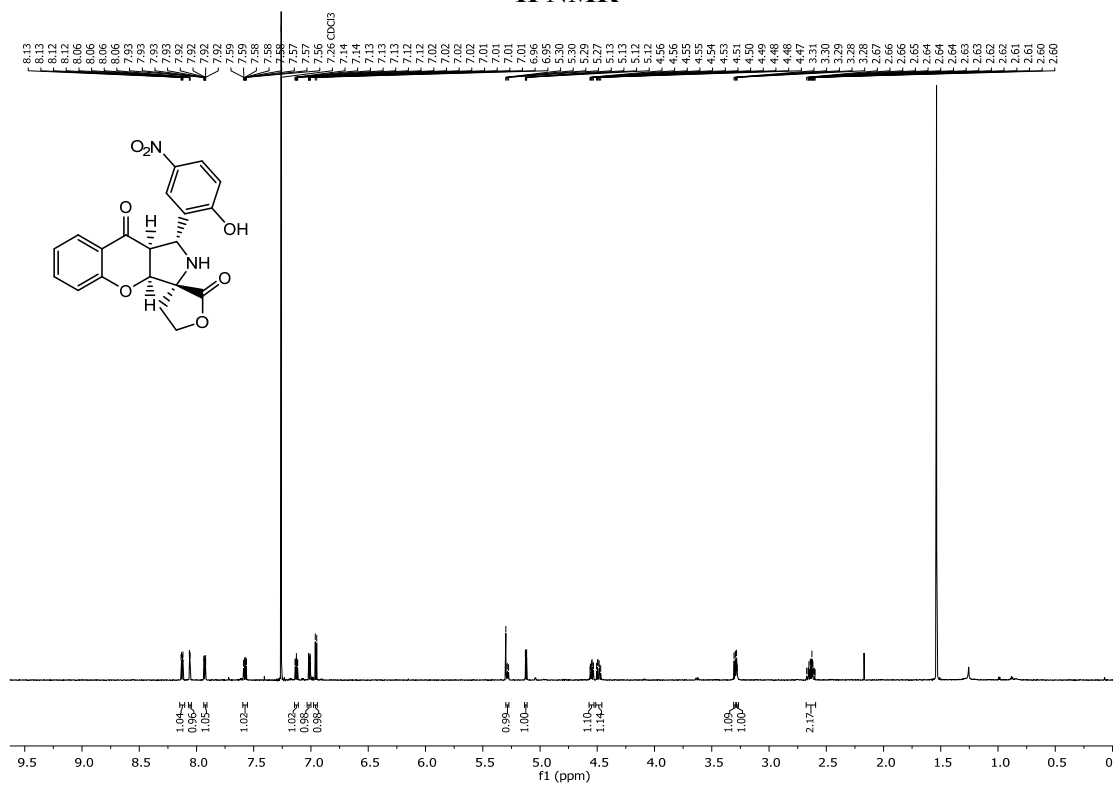
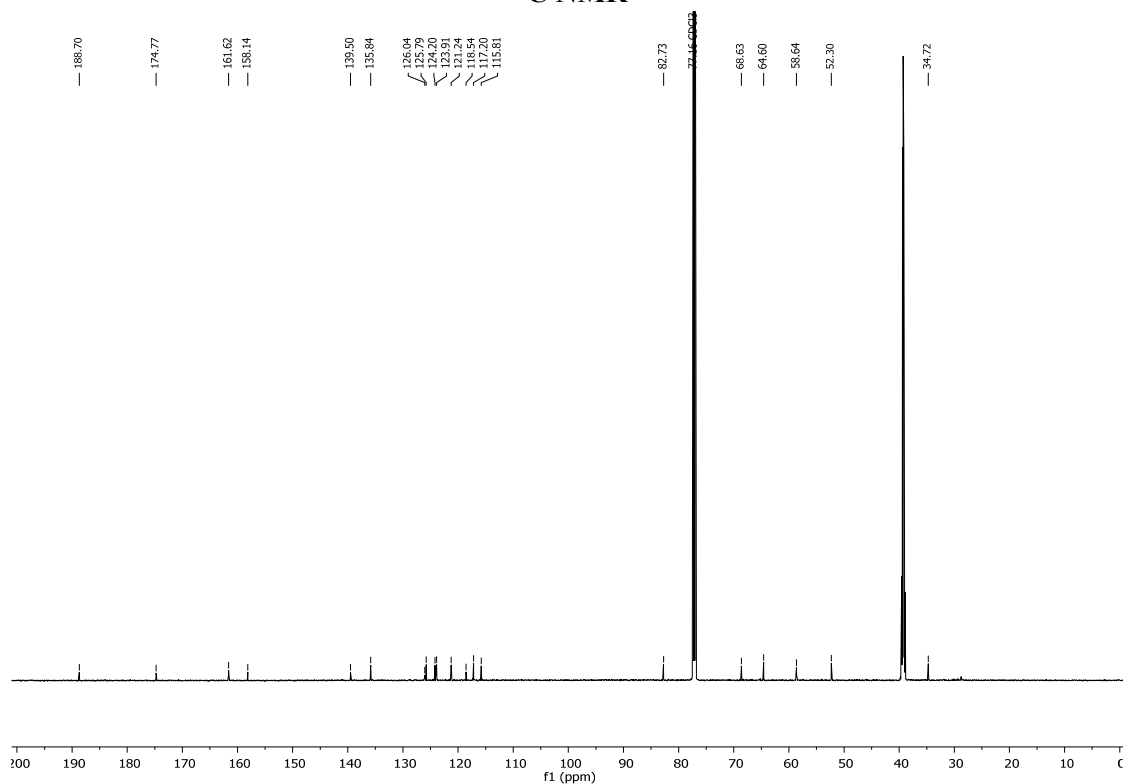
13C NMR spectrum of compound 10 in CDCl<sub>3</sub>. The x-axis is labeled 'f1 (ppm)' and ranges from 0 to 190. The spectrum shows several sharp peaks. A triplet for the CDCl<sub>3</sub> solvent is visible at approximately 77 ppm. Numerous other peaks are present, with chemical shifts labeled above the spectrum: 188.13, 174.17, 157.49, 153.34, 134.85, 126.73, 126.69, 126.65, 126.55, 121.84, 120.26, 117.97, 116.38, 116.12, 82.04, 77.16 CDCl<sub>3</sub>, 67.92, 63.87, 58.14, 51.43, 38.63 DMSO, and 33.79.

## <sup>1</sup>H NMR

<sup>13</sup>C NMR

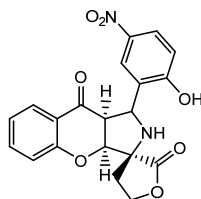
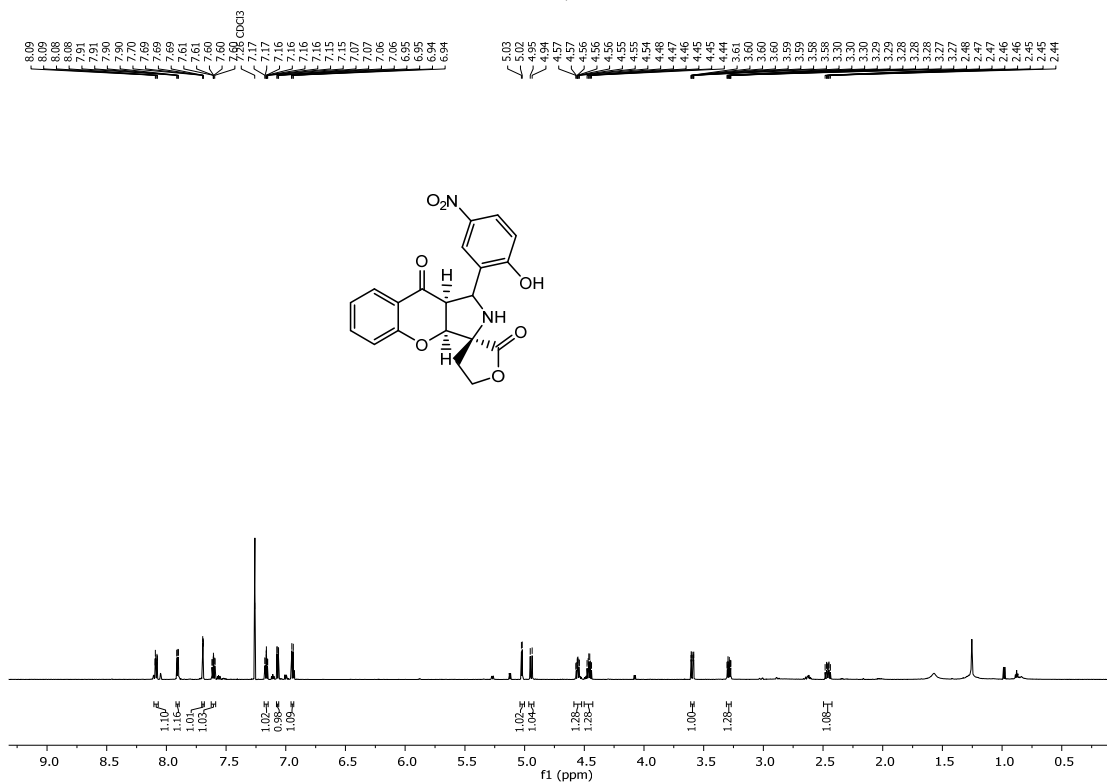
**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxy-5-nitrophenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1x**

## <sup>1</sup>H NMR

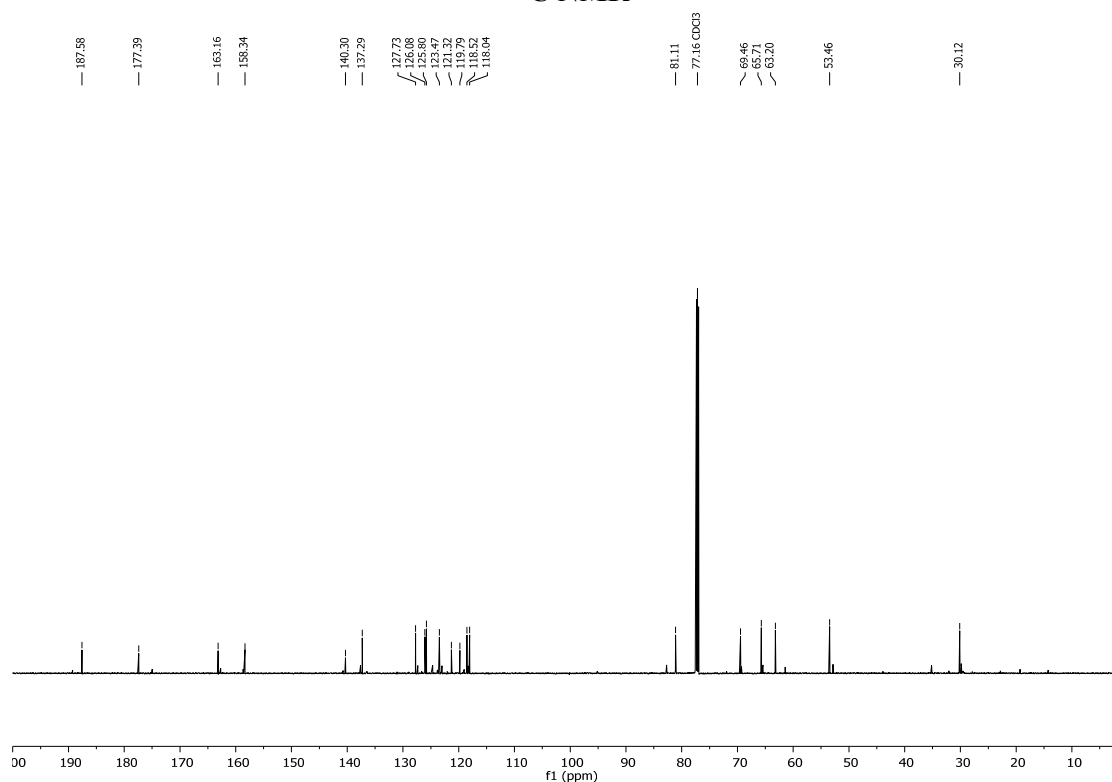
<sup>13</sup>C NMR

**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxy-5-nitrophenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1x'**

**<sup>1</sup>H NMR**



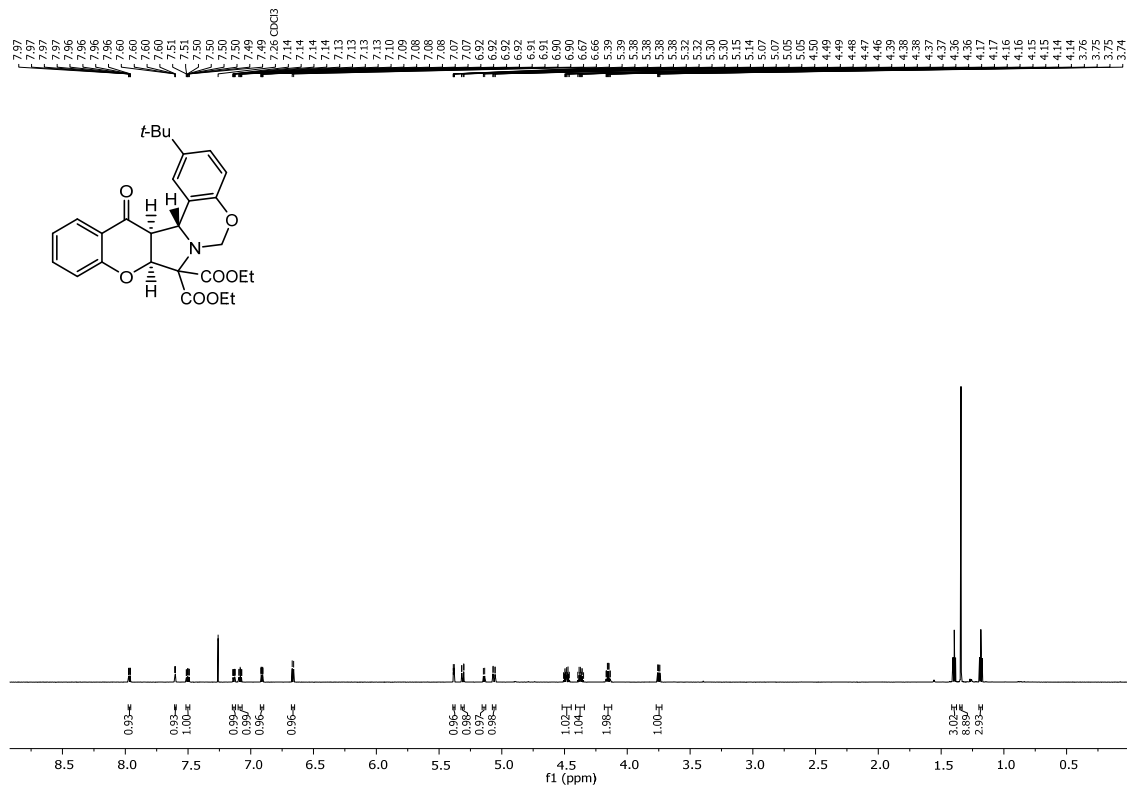
**<sup>13</sup>C NMR**





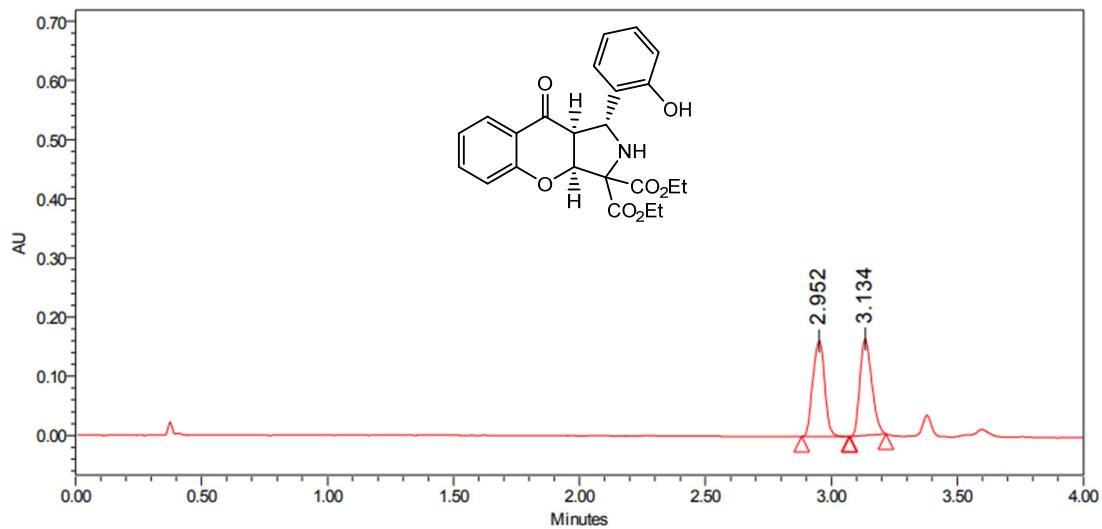
**(8a*S*,14a*S*,14b*R*)-Diethyl 2-(*tert*-butyl)-14-oxo-8a,14,14a,14b-tetrahydrobenzo-  
[*e*]chromeno[3',2':3,4]pyrrolo[1,2-*c*][1,3]oxazine-8,8(6*H*)-dicarboxylate 9a**

**<sup>1</sup>H NMR**



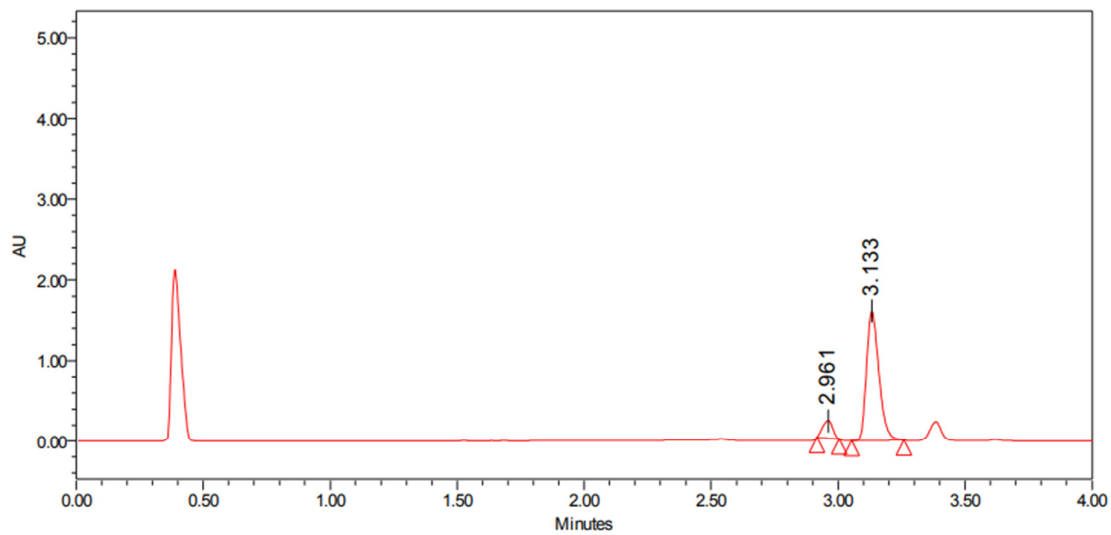
#### 4. HPLC data

**(1*R*,3*aS*,9*aS*)-Diethyl 1-(2-hydroxyphenyl)-9-oxo-1,2,9,9*a*-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1a**



##### Peak Results

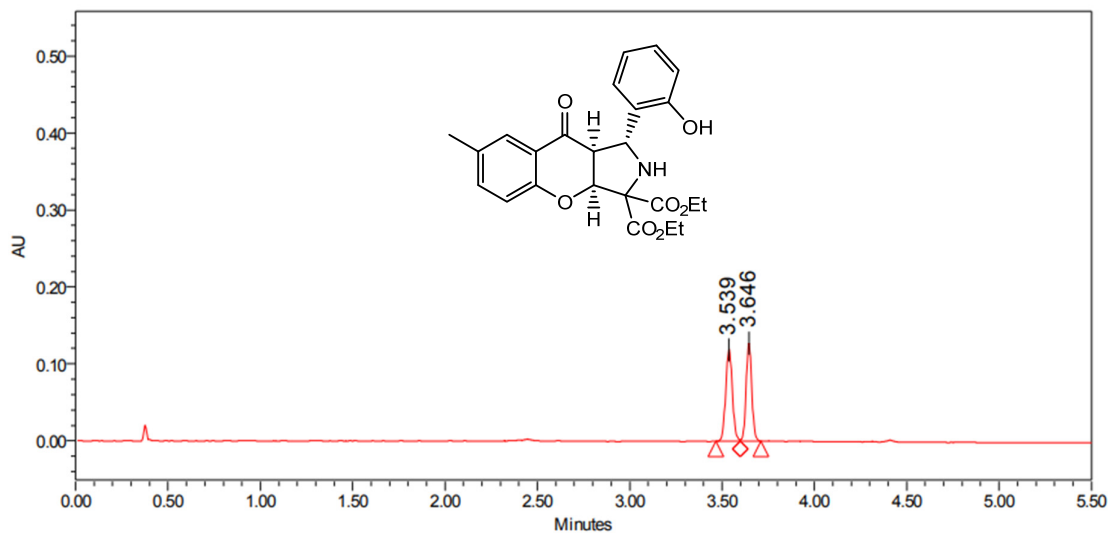
	RT	% Area
1	2.952	49.80
2	3.134	50.20



##### Peak Results

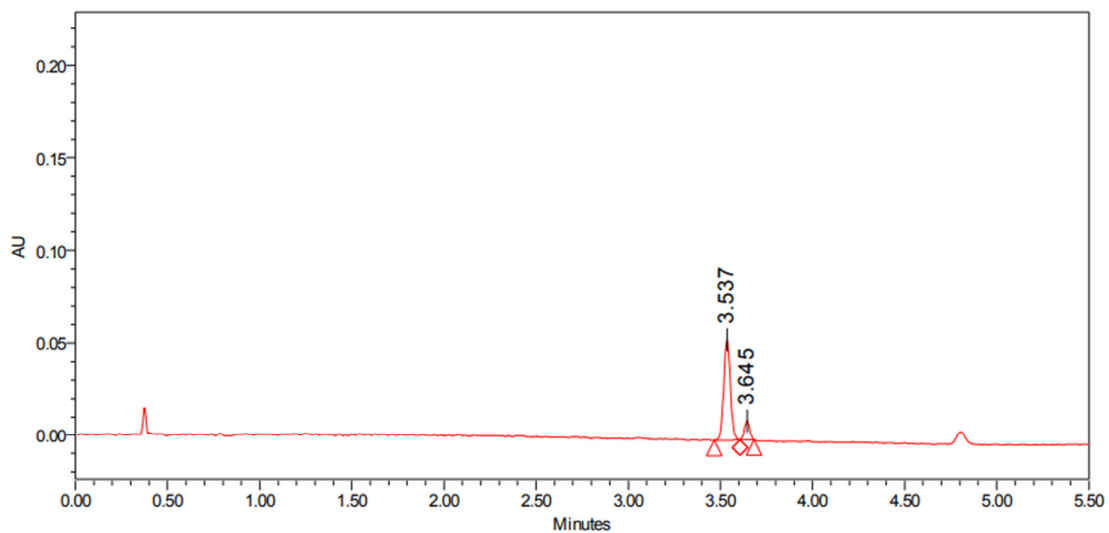
	RT	% Area
1	2.961	10.22
2	3.133	89.78

**(1*R*,3*aS*,9*aS*)-Diethyl 1-(2-hydroxyphenyl)-7-methyl-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1b**



**Peak Results**

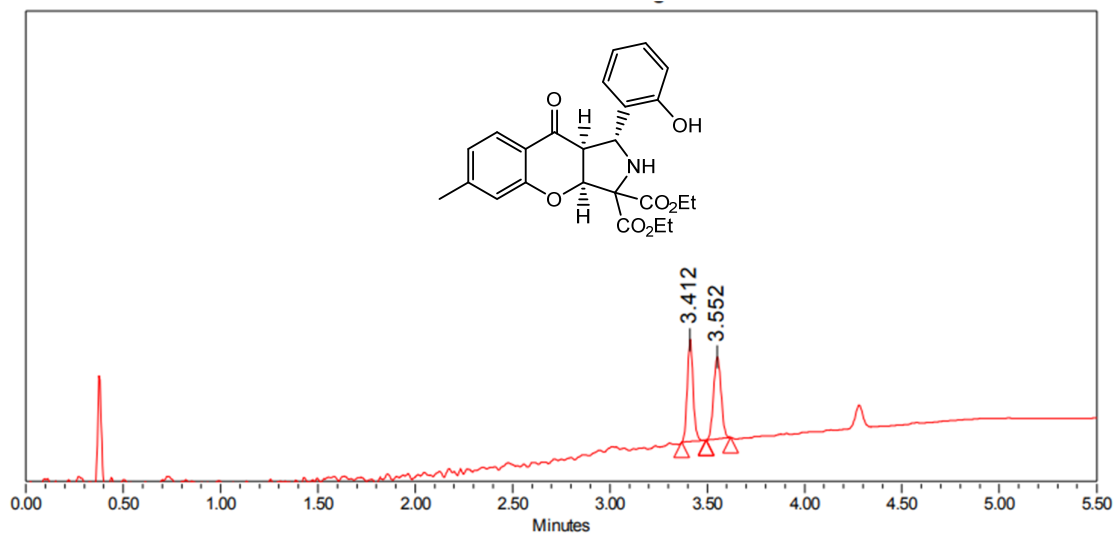
	RT	% Area
1	3.539	52.63
2	3.646	47.37



**Peak Results**

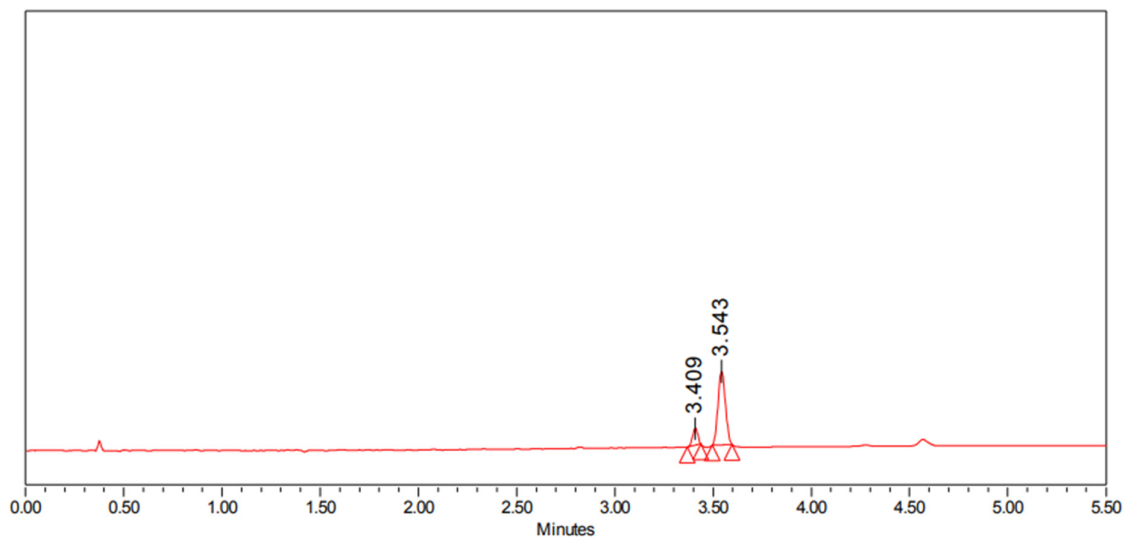
	RT	% Area
1	3.537	86.66
2	3.645	13.34

(1*R*,3*aS*,9*aS*)-Diethyl 1-(2-hydroxyphenyl)-6-methyl-9-oxo-1,2,9,9*a*-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1c



**Peak Results**

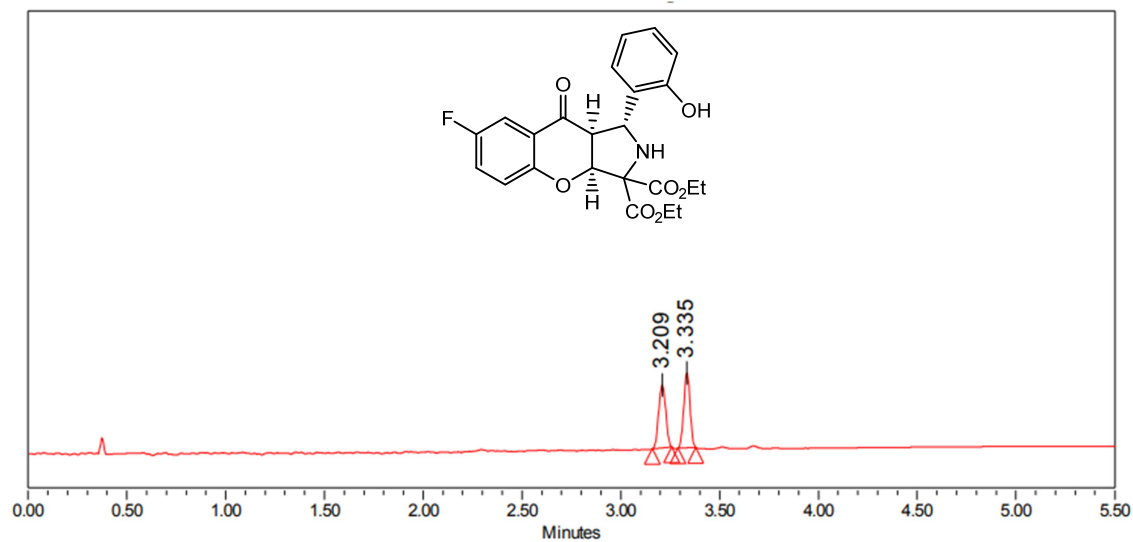
	RT	% Area
1	3.412	49.51
2	3.552	50.49



**Peak Results**

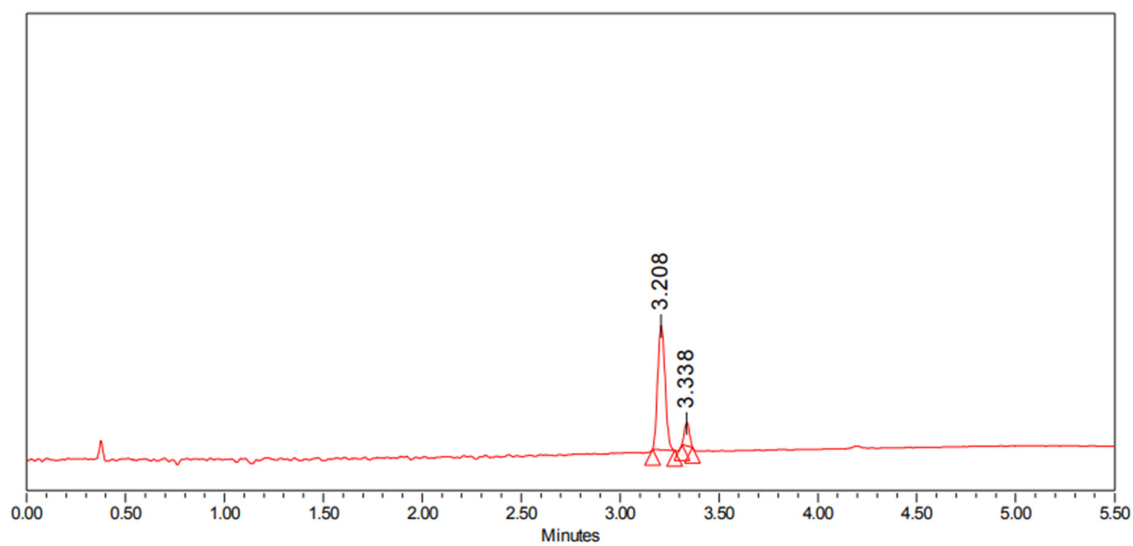
	RT	% Area
1	3.409	13.86
2	3.543	86.14

**(1*R*,3*aS*,9*aS*)-Diethyl 7-fluoro-1-(2-hydroxyphenyl)-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1d**



**Peak Results**

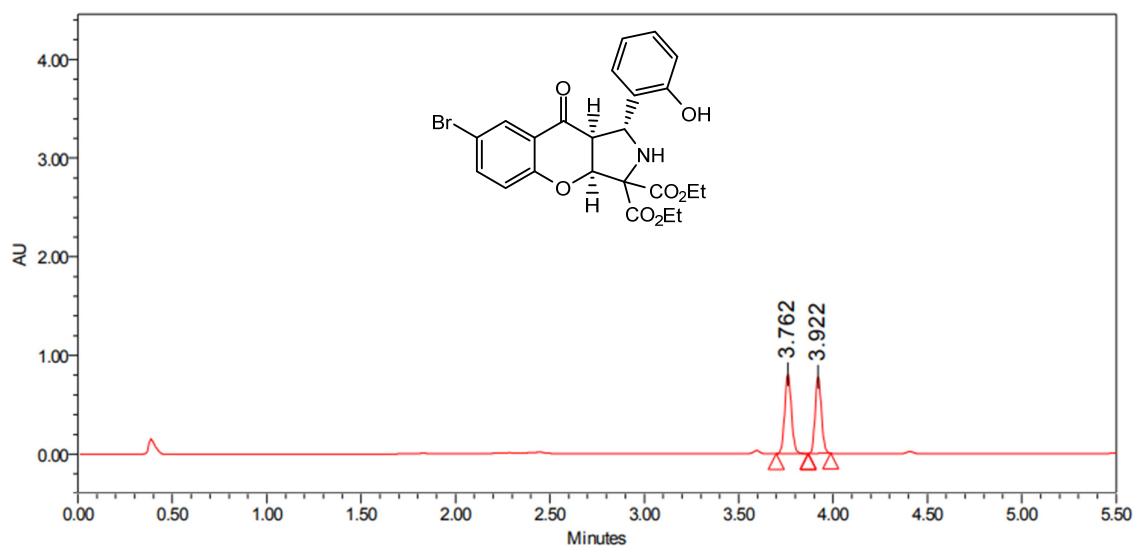
	RT	% Area
1	3.209	49.08
2	3.335	50.92



**Peak Results**

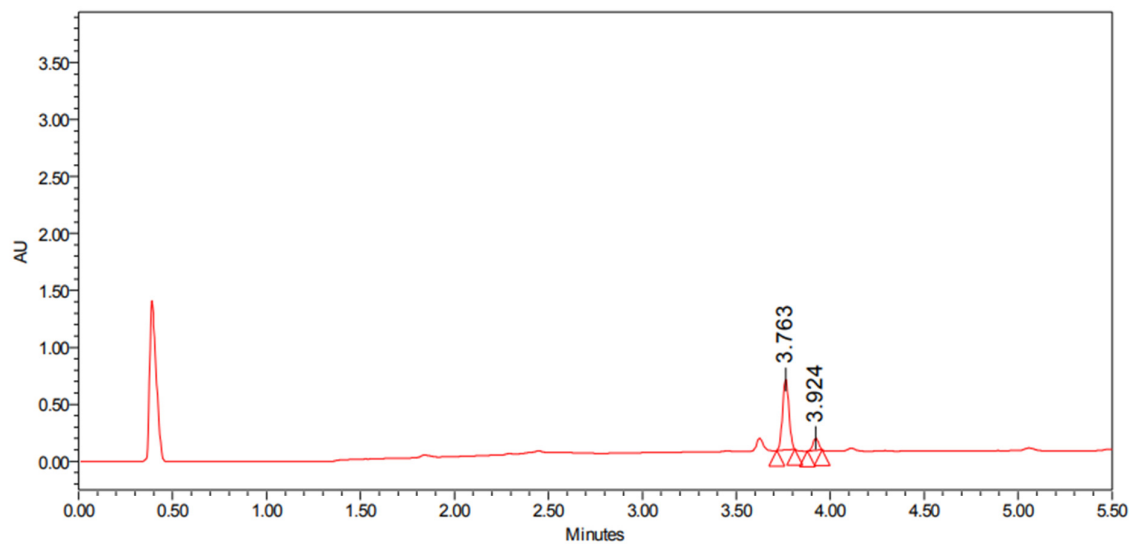
	RT	% Area
1	3.208	88.87
2	3.338	11.13

**(1*R*,3*aS*,9*aS*)-Diethyl 7-bromo-1-(2-hydroxyphenyl)-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1e**



**Peak Results**

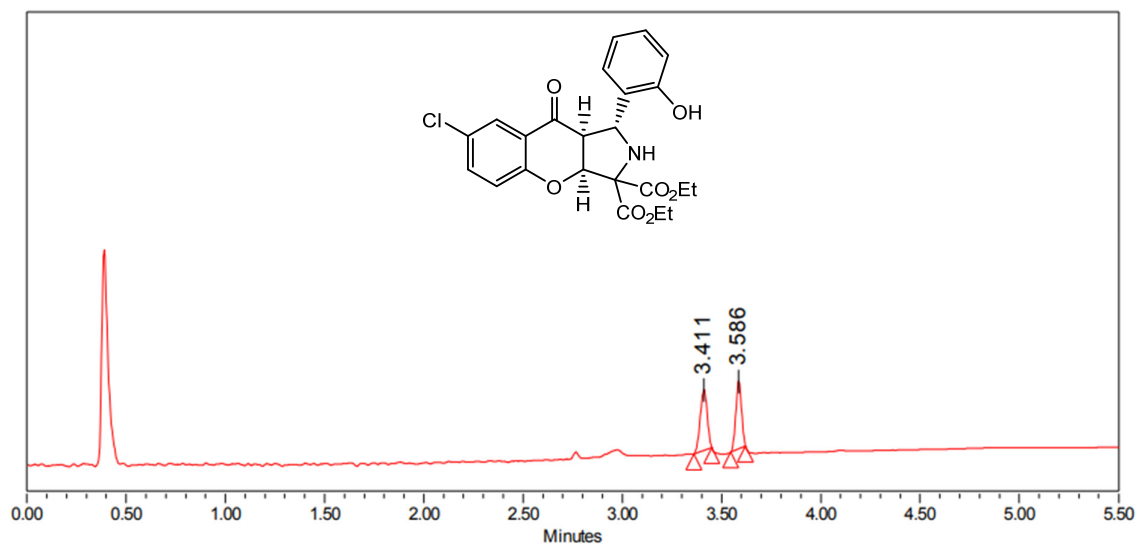
	RT	% Area
1	3.762	52.82
2	3.922	47.18



**Peak Results**

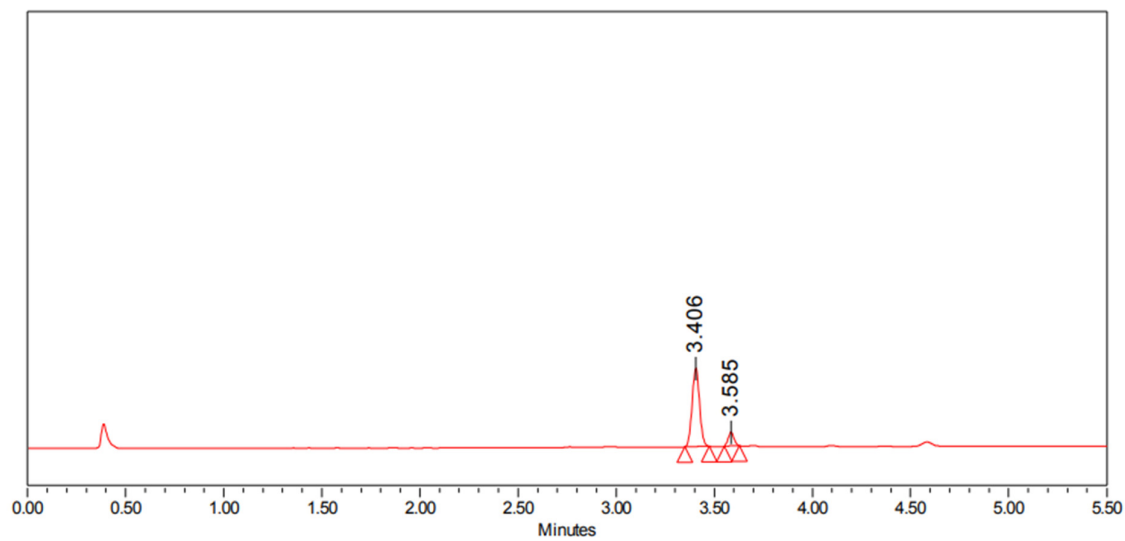
	RT	% Area
1	3.763	86.83
2	3.924	13.17

**(1*R*,3*aS*,9*aS*)-Diethyl 7-chloro-1-(2-hydroxyphenyl)-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1f**



**Peak Results**

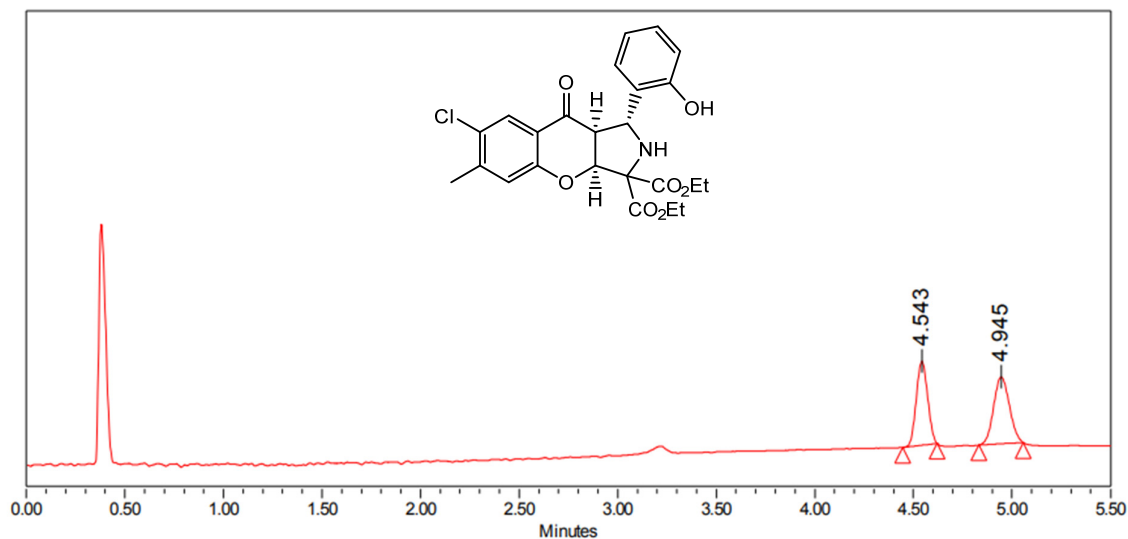
	RT	% Area
1	3.411	52.32
2	3.586	47.68



**Peak Results**

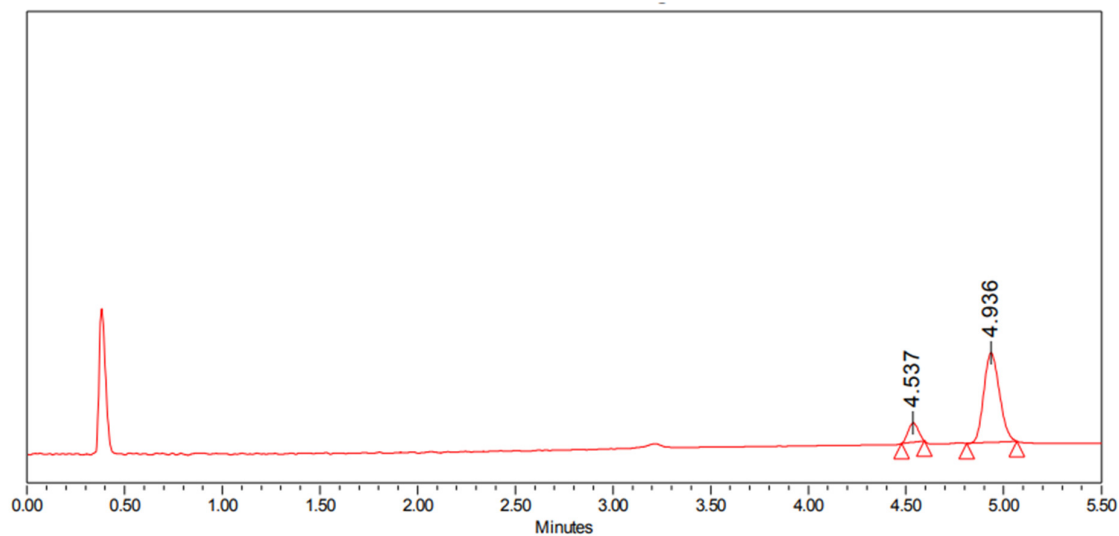
	RT	% Area
1	3.406	87.86
2	3.585	12.14

**(1*R*,3*aS*,9*aS*)-Diethyl 7-chloro-1-(2-hydroxyphenyl)-6-methyl-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1g**



**Peak Results**

	RT	% Area
1	4.543	47.51
2	4.945	52.49

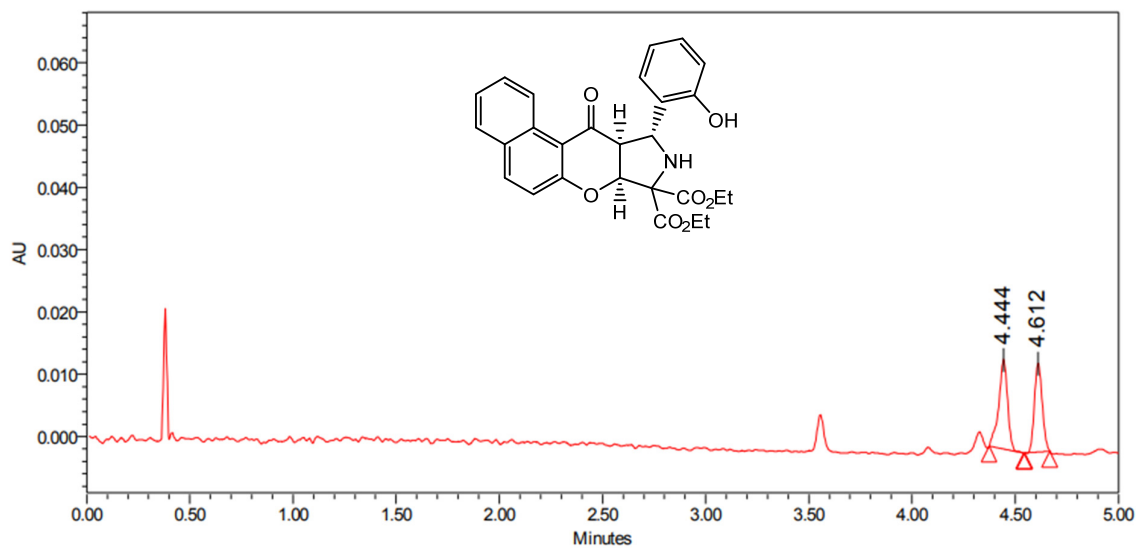


**Peak Results**

	RT	% Area
1	4.537	12.27
2	4.936	87.73

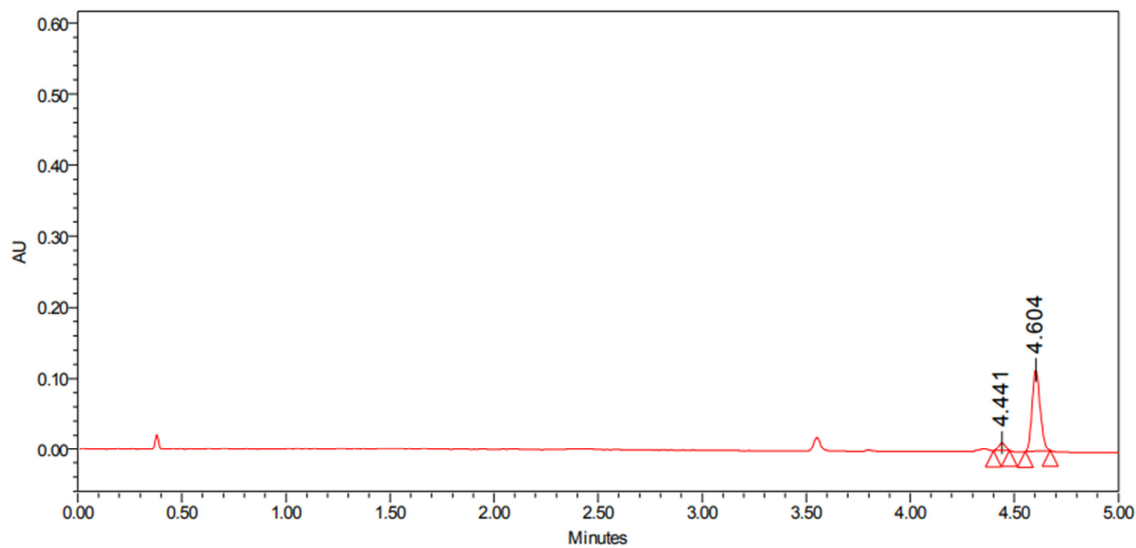


**(7a*S*,10*R*,10a*S*)-Diethyl 10-(2-hydroxyphenyl)-11-oxo-9,10,10a,11-tetrahydrobenzo[5,6]chromeno[2,3-*c*]pyrrole-8,8(7a*H*)-dicarboxylate 1h**



**Peak Results**

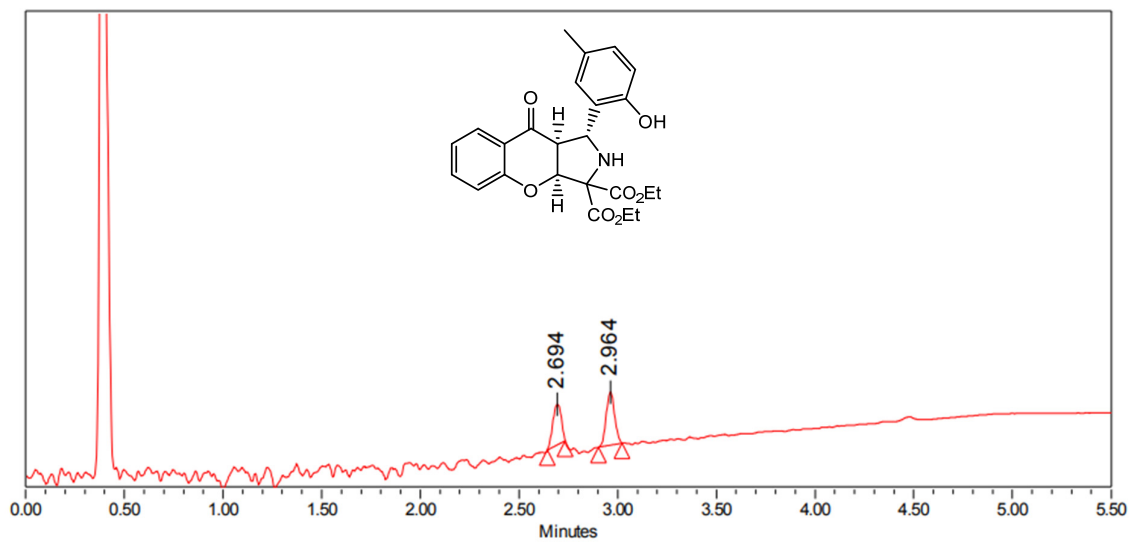
	RT	% Area
1	4.444	53.31
2	4.612	46.69



**Peak Results**

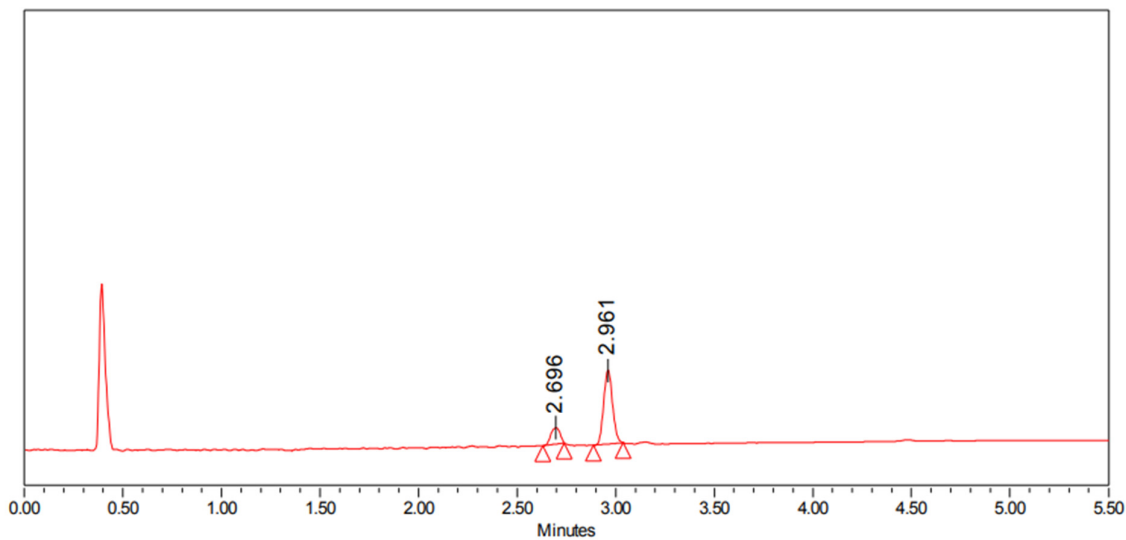
	RT	% Area
1	4.441	7.78
2	4.604	92.22

**(1*R*,3*aS*,9*aS*)-Diethyl 1-(2-hydroxy-5-methylphenyl)-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1i**



**Peak Results**

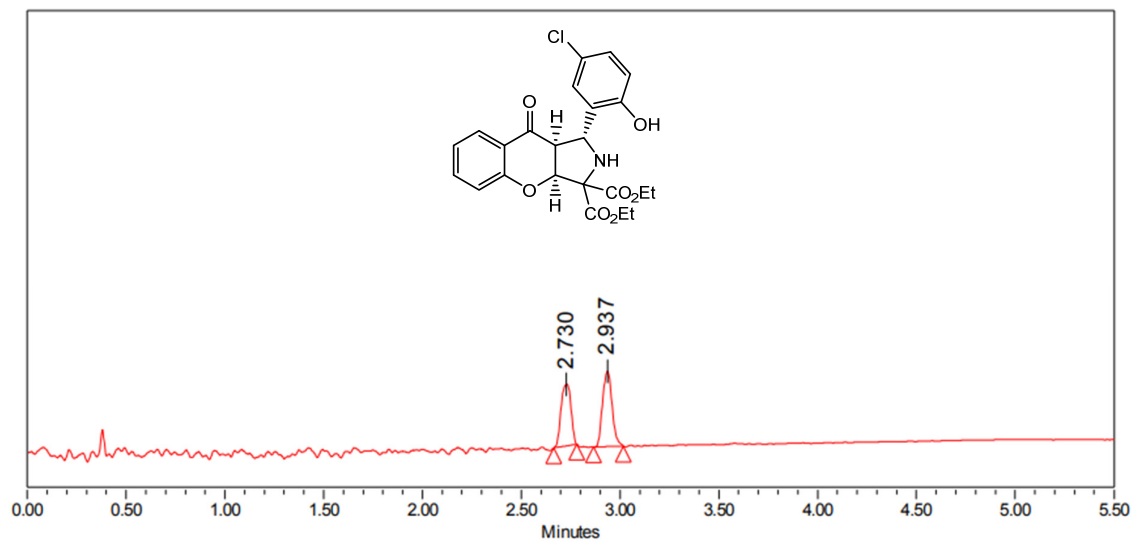
	RT	% Area
1	2.694	43.33
2	2.964	56.67



**Peak Results**

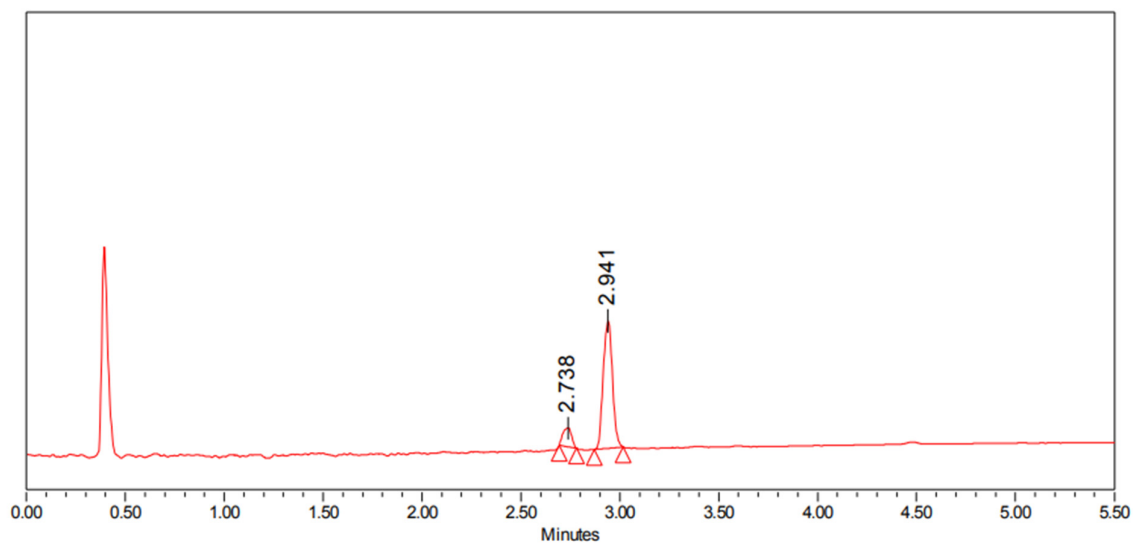
	RT	% Area
1	2.696	18.28
2	2.961	81.72

**(1*R*,3*aS*,9*aS*)-Diethyl 1-(5-chloro-2-hydroxyphenyl)-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1j**



**Peak Results**

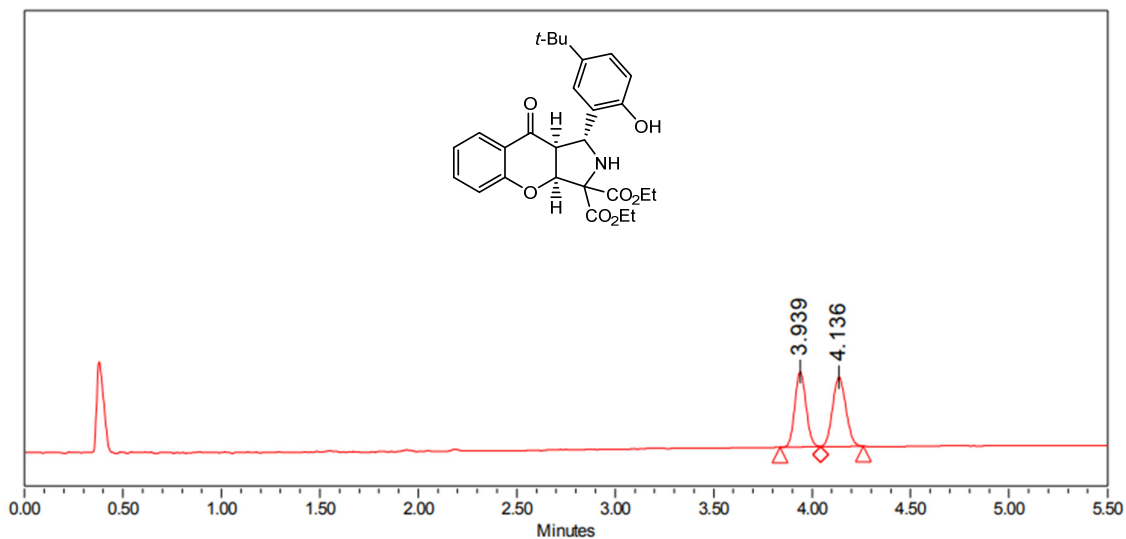
	RT	% Area
1	2.730	46.10
2	2.937	53.90



**Peak Results**

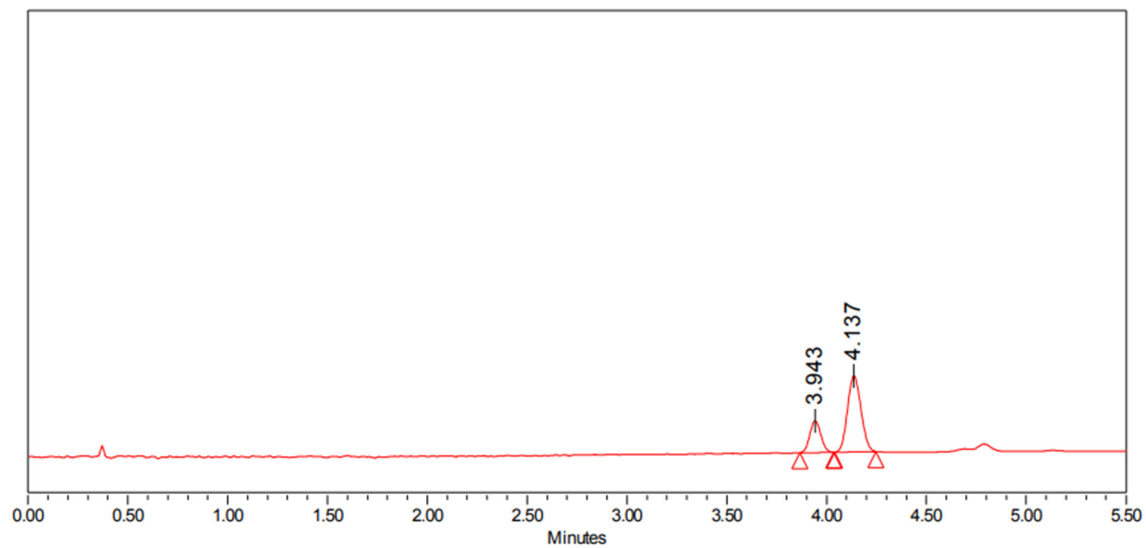
	RT	% Area
1	2.738	12.28
2	2.941	87.72

(1*R*,3*aS*,9*aS*)-Diethyl 1-(5-(*tert*-butyl)-2-hydroxyphenyl)-9-oxo-1,2,9,9a-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 1k



**Peak Results**

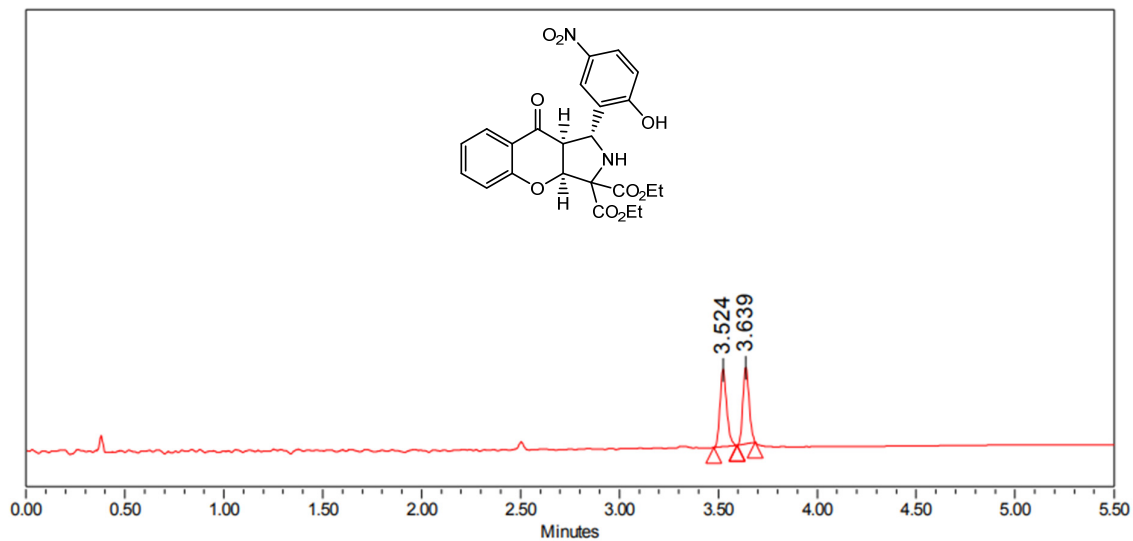
	RT	% Area
1	3.939	47.55
2	4.136	52.45



**Peak Results**

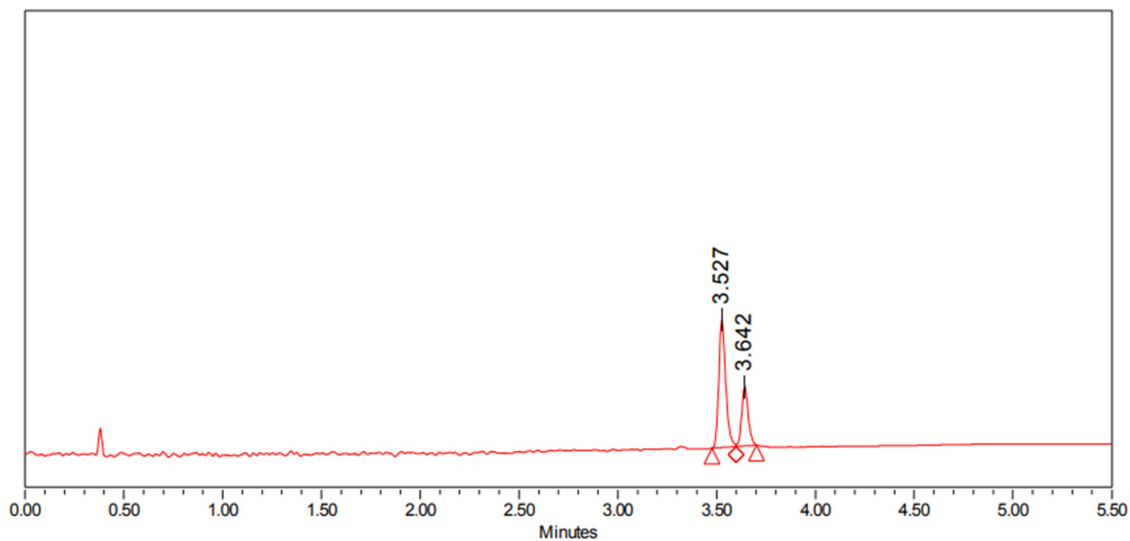
	RT	% Area
1	3.943	25.49
2	4.137	74.51

**(1*R*,3*aS*,9*aS*)-Diethyl 1-(2-hydroxy-5-nitrophenyl)-9-oxo-1,2,9*a*-tetrahydrochromeno[2,3-*c*]pyrrole-3,3(3*aH*)-dicarboxylate 11**



**Peak Results**

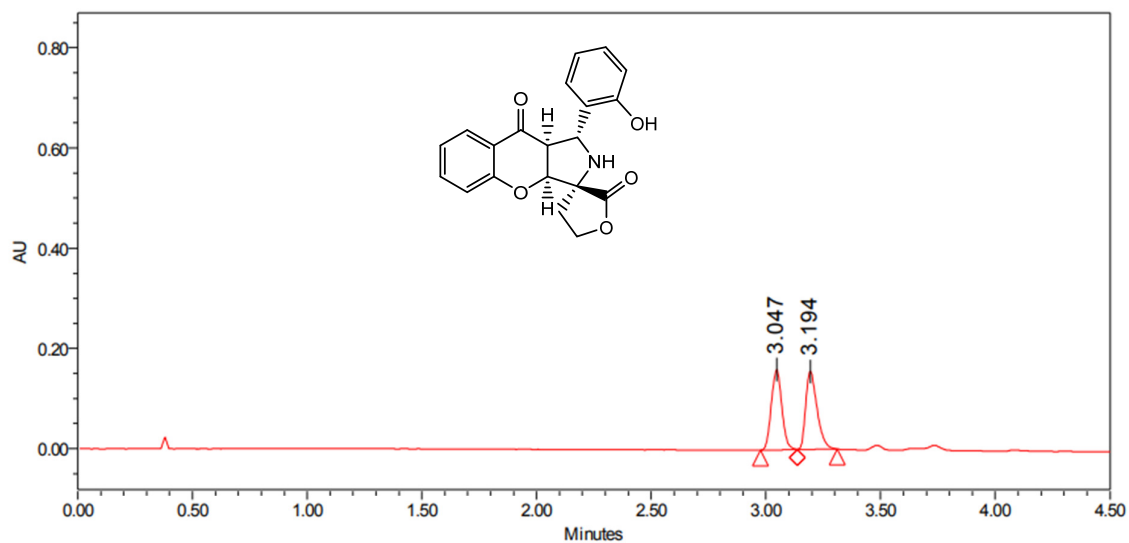
	RT	% Area
1	3.524	52.06
2	3.639	47.94



**Peak Results**

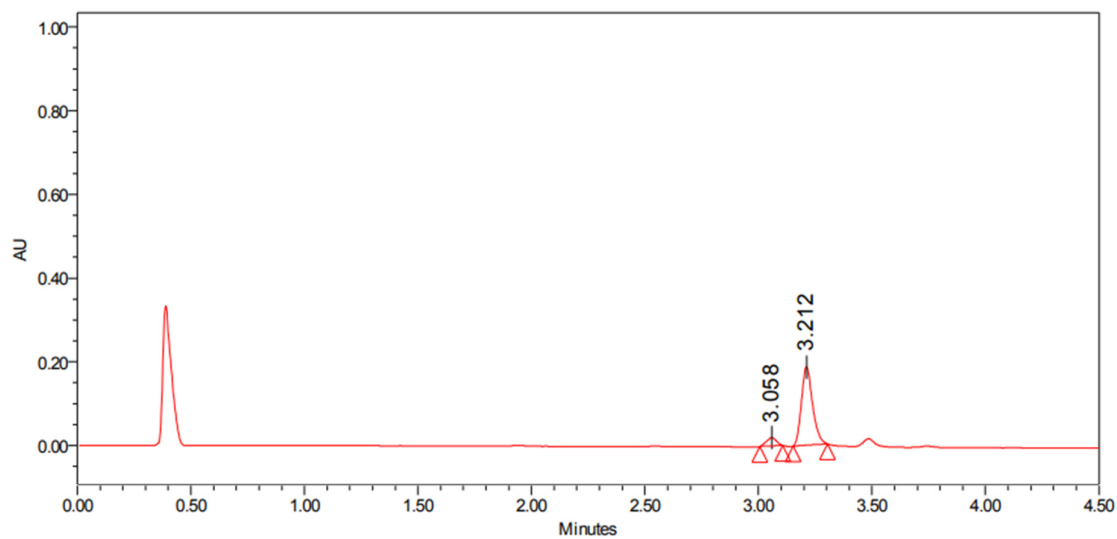
	RT	% Area
1	3.527	69.58
2	3.642	30.42

**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1m**



**Peak Results**

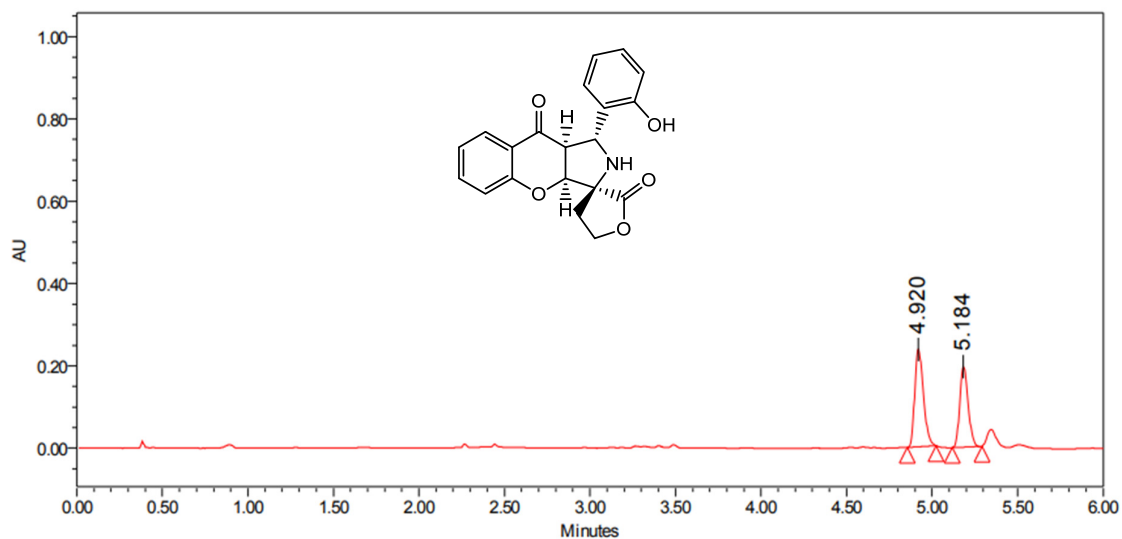
	RT	% Area
1	3.047	48.93
2	3.194	51.07



**Peak Results**

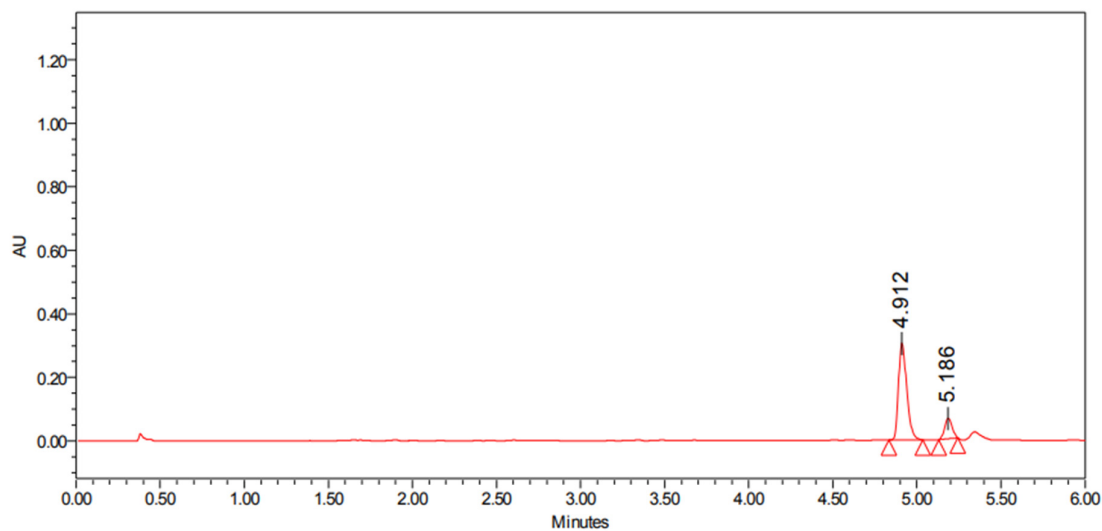
	RT	% Area
1	3.058	8.47
2	3.212	91.53

**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1m'**



**Peak Results**

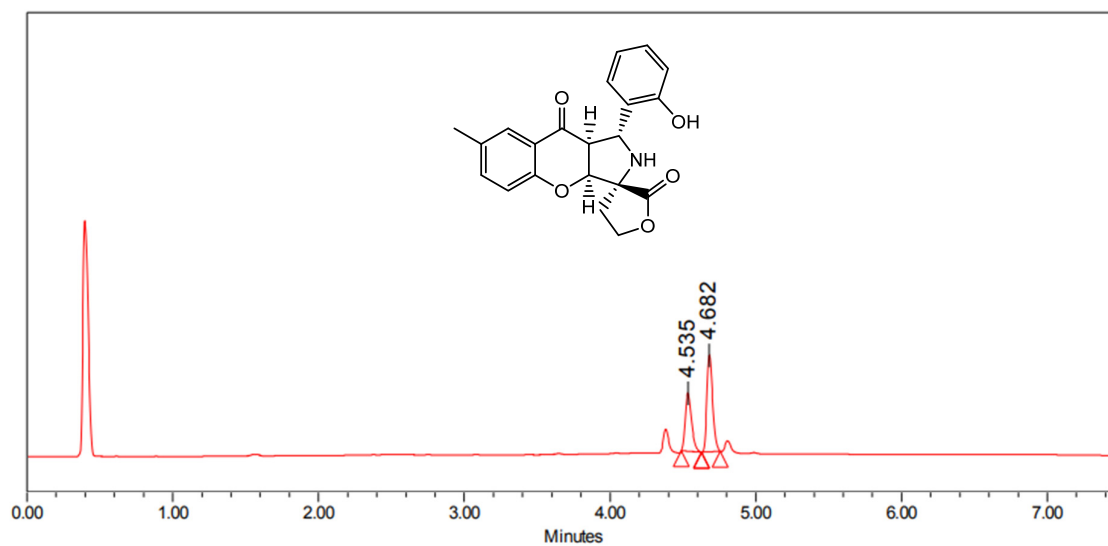
	RT	% Area
1	4.920	55.72
2	5.184	44.28



**Peak Results**

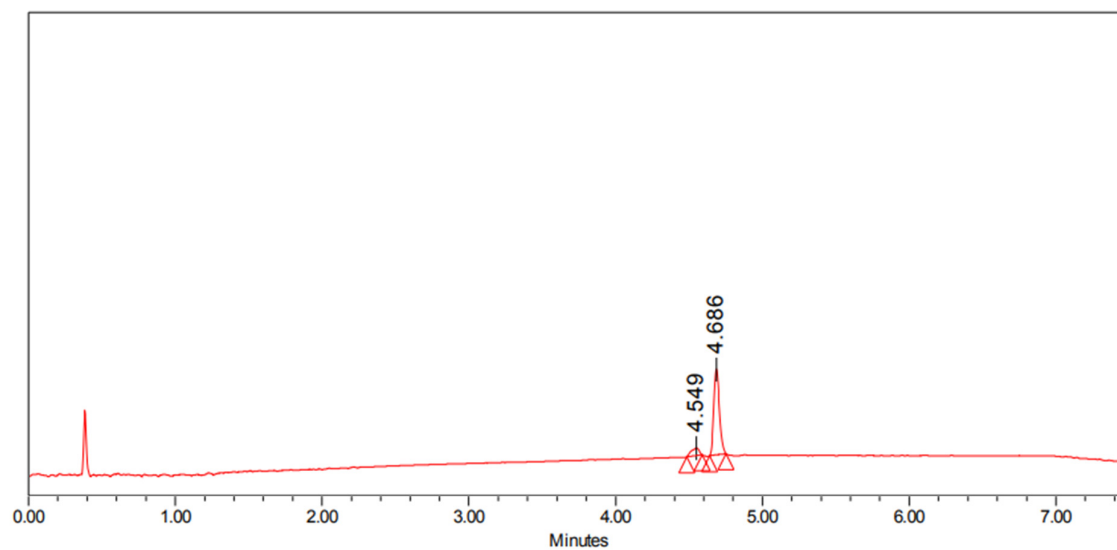
	RT	% Area
1	4.912	83.96
2	5.186	16.04

**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-7-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1n**



**Peak Results**

	RT	% Area
1	4.535	39.71
2	4.682	60.29

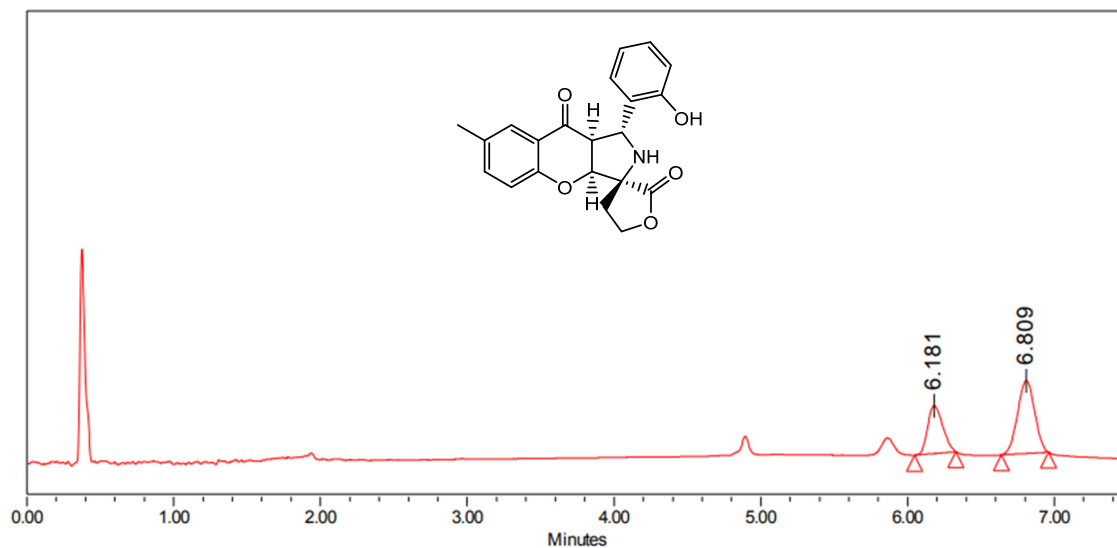


**Peak Results**

	RT	% Area
1	4.549	11.05
2	4.686	88.95

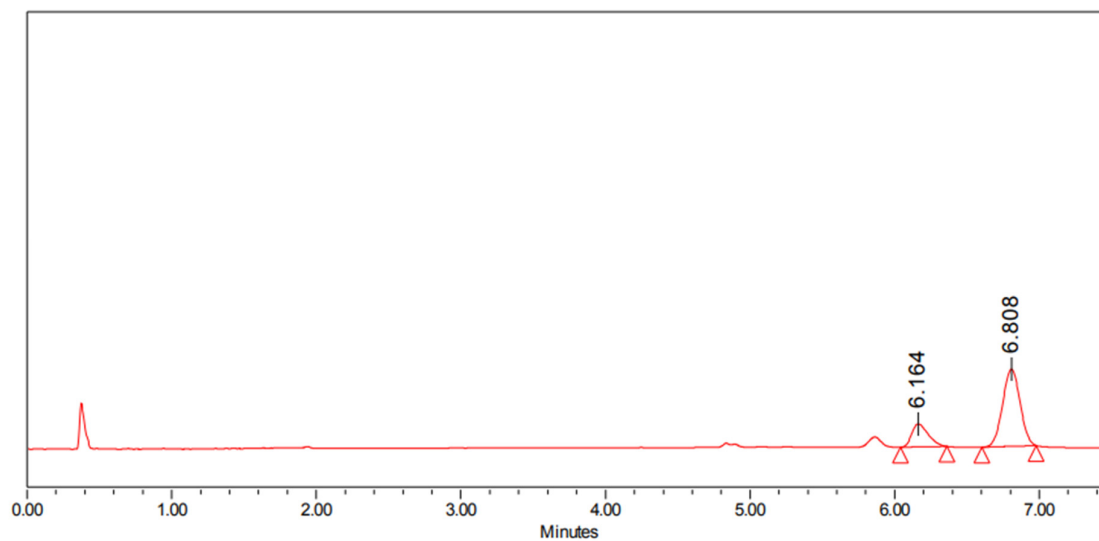


**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-7-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1n**



**Peak Results**

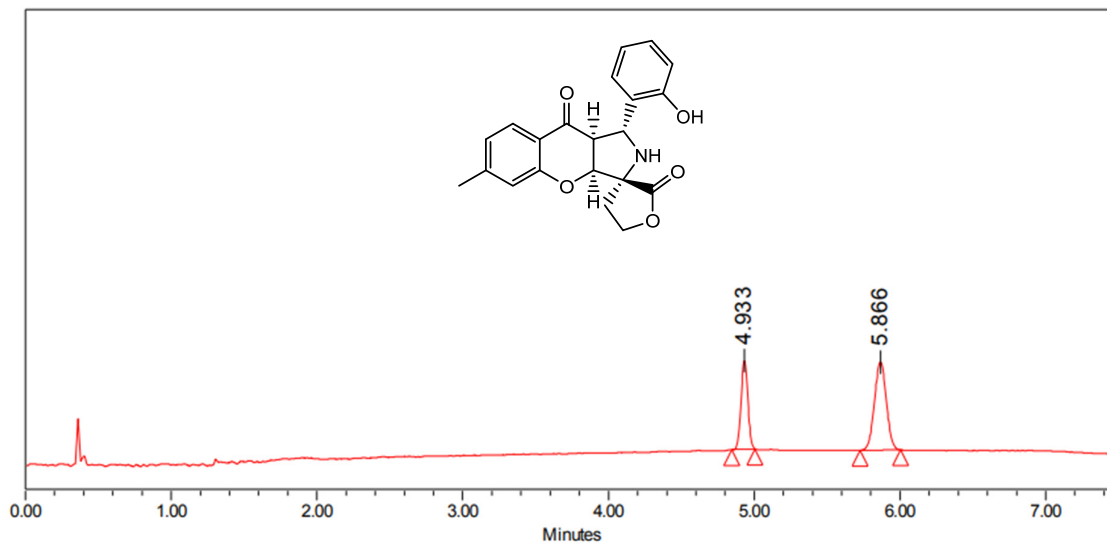
	RT	% Area
1	6.181	36.64
2	6.809	63.36



**Peak Results**

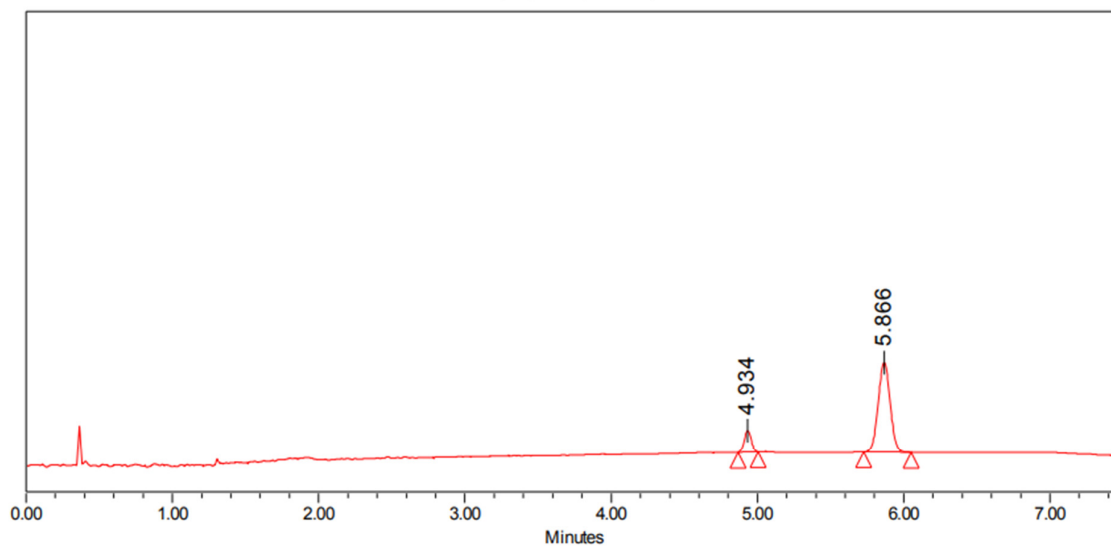
	RT	% Area
1	6.164	21.85
2	6.808	78.15

**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1o**



**Peak Results**

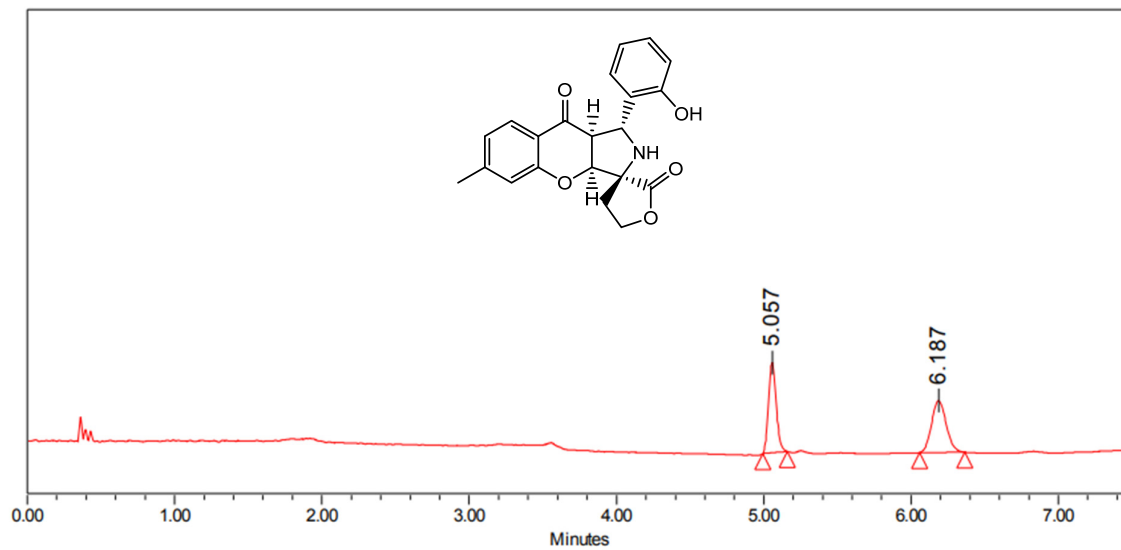
	RT	% Area
1	4.933	36.27
2	5.866	63.73



**Peak Results**

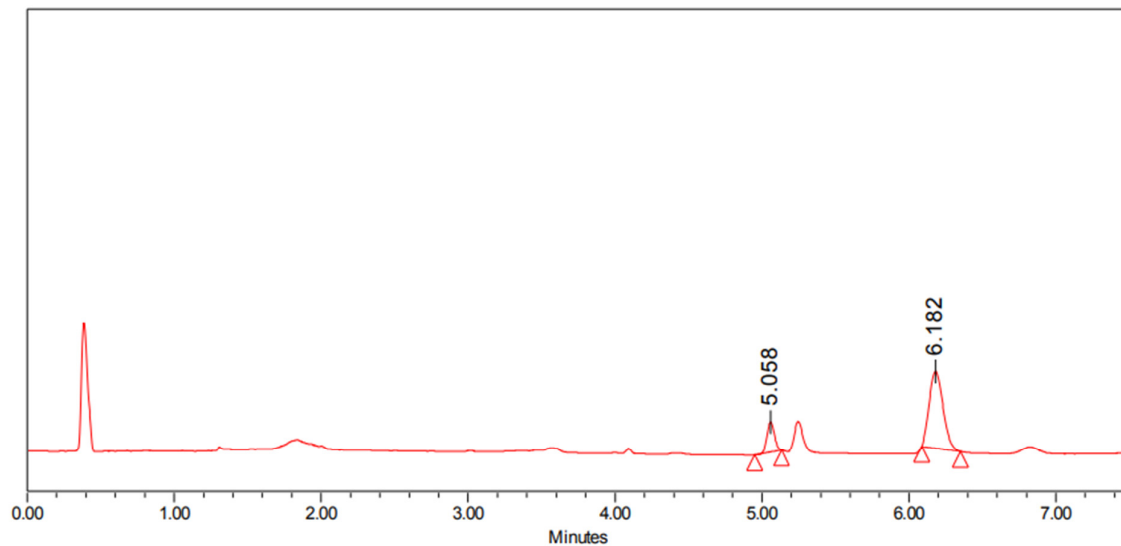
	RT	% Area
1	4.934	12.43
2	5.866	87.57

**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1o'**



**Peak Results**

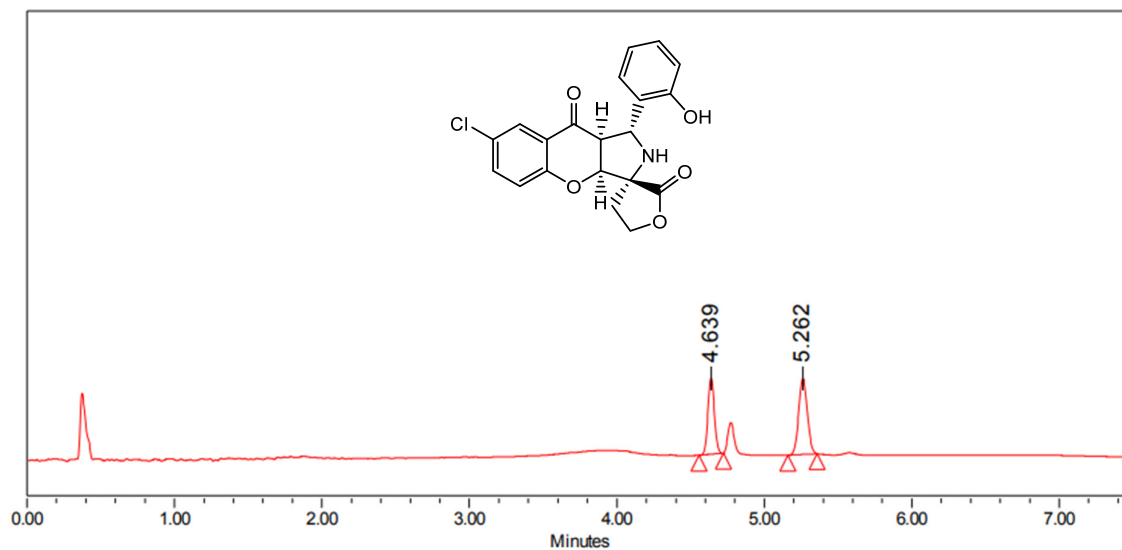
	RT	% Area
1	5.057	47.80
2	6.187	52.20



**Peak Results**

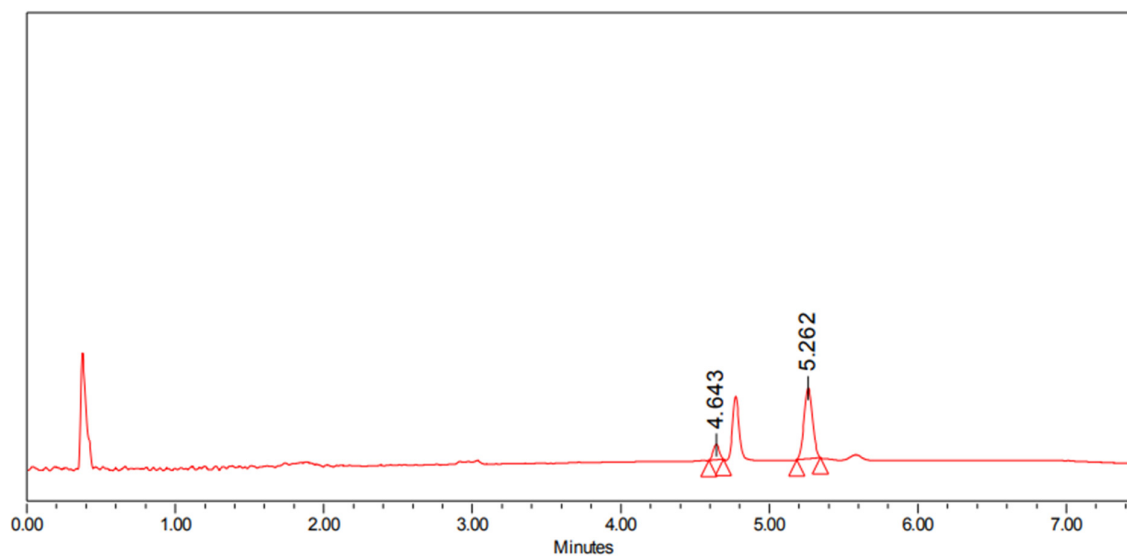
	RT	% Area
1	5.058	17.48
2	6.182	82.52

**(1*R*,3*R*,3*aS*,9*aS*)-7-Chloro-1-(2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1p**



**Peak Results**

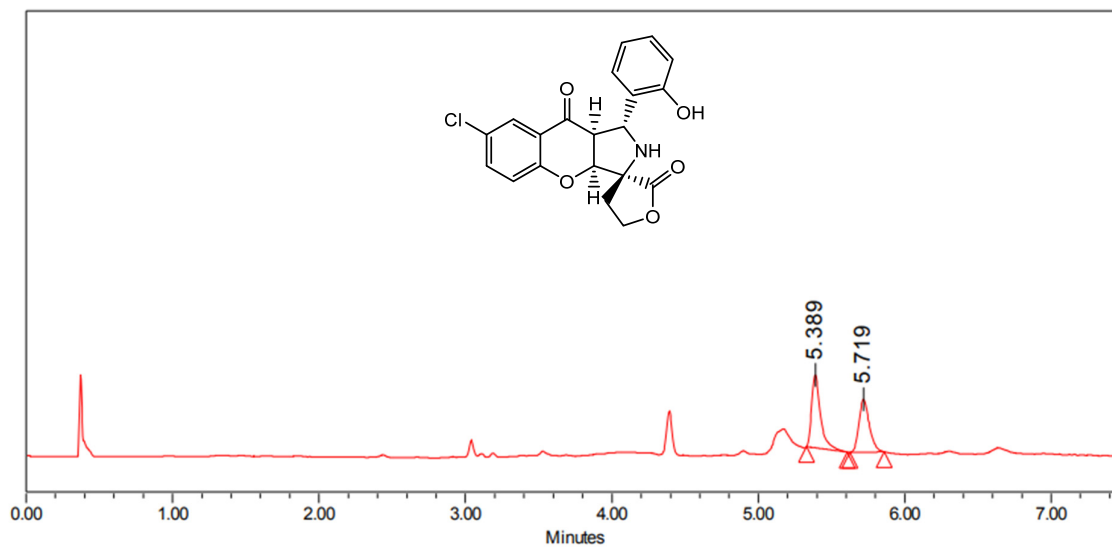
	RT	% Area
1	4.639	42.70
2	5.262	57.30



**Peak Results**

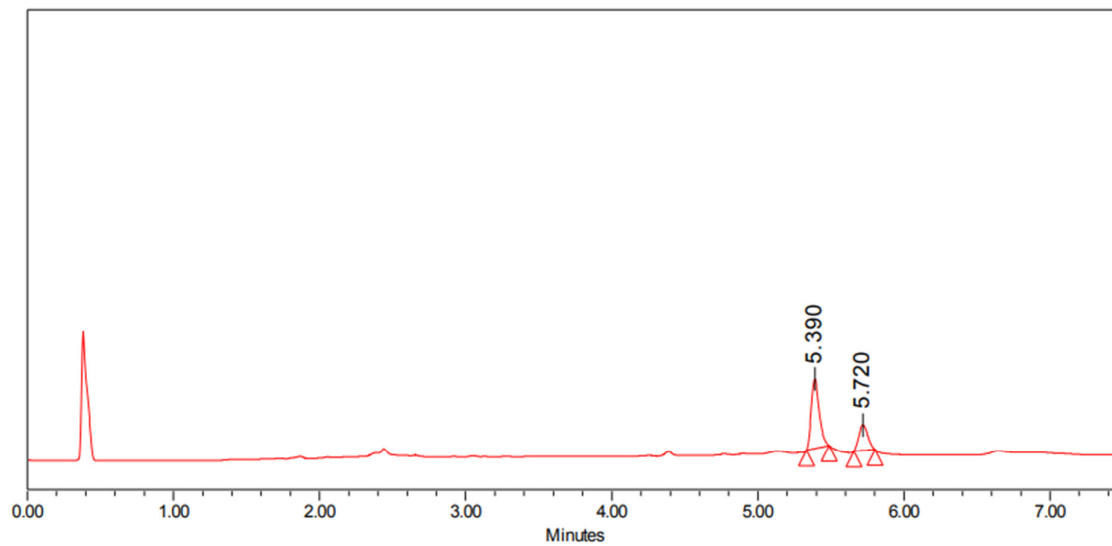
	RT	% Area
1	4.643	12.91
2	5.262	87.09

**(1*R*,3*S*,3*aS*,9*aS*)-7-Chloro-1-(2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1p'**



**Peak Results**

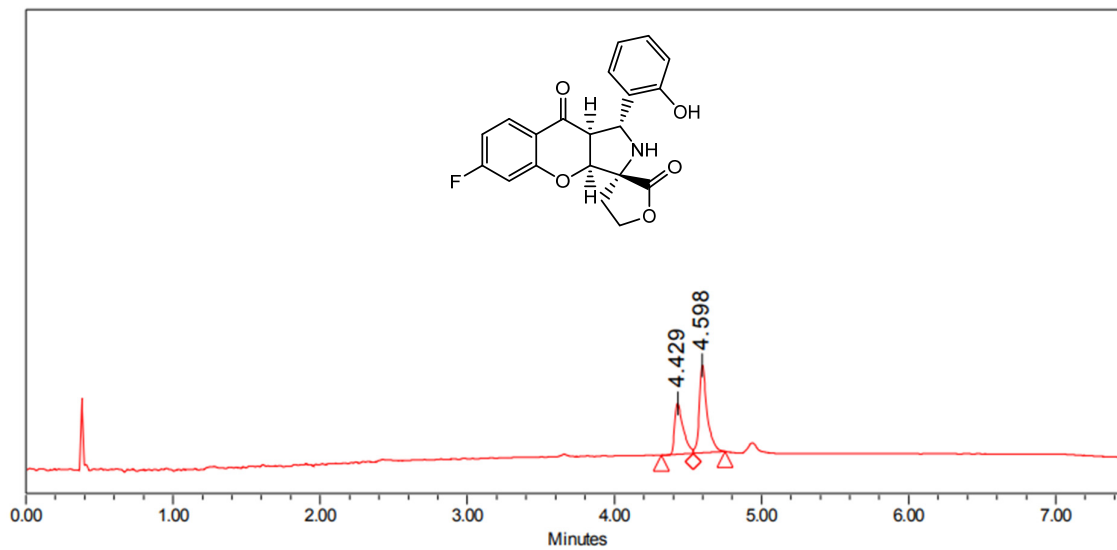
	RT	% Area
1	5.389	54.43
2	5.719	45.57



**Peak Results**

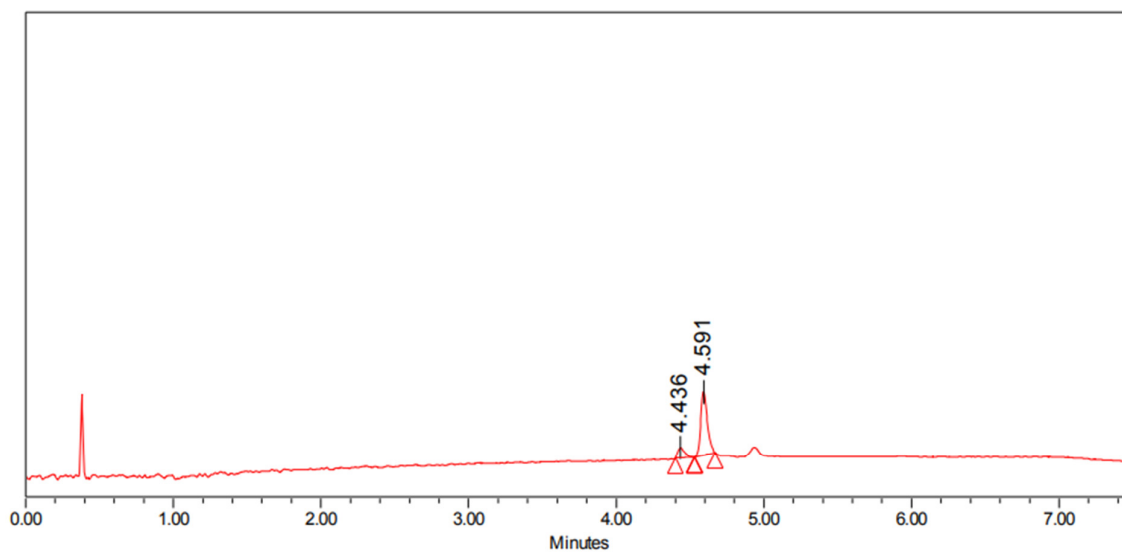
	RT	% Area
1	5.390	70.77
2	5.720	29.23

**(1*R*,3*R*,3*aS*,9*aS*)-6-Fluoro-1-(2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1r**



**Peak Results**

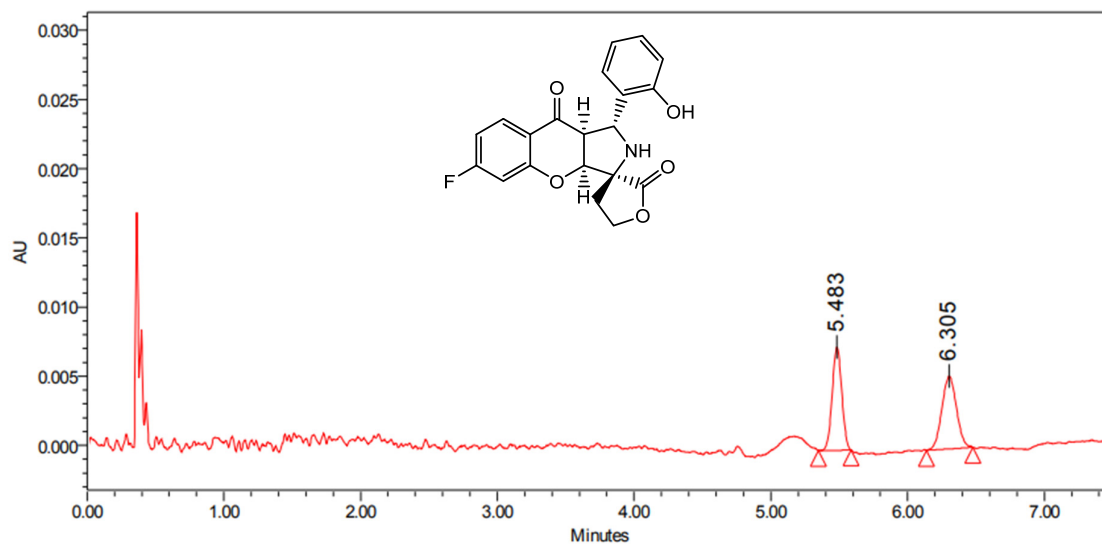
	RT	% Area
1	4.429	38.75
2	4.598	61.25



**Peak Results**

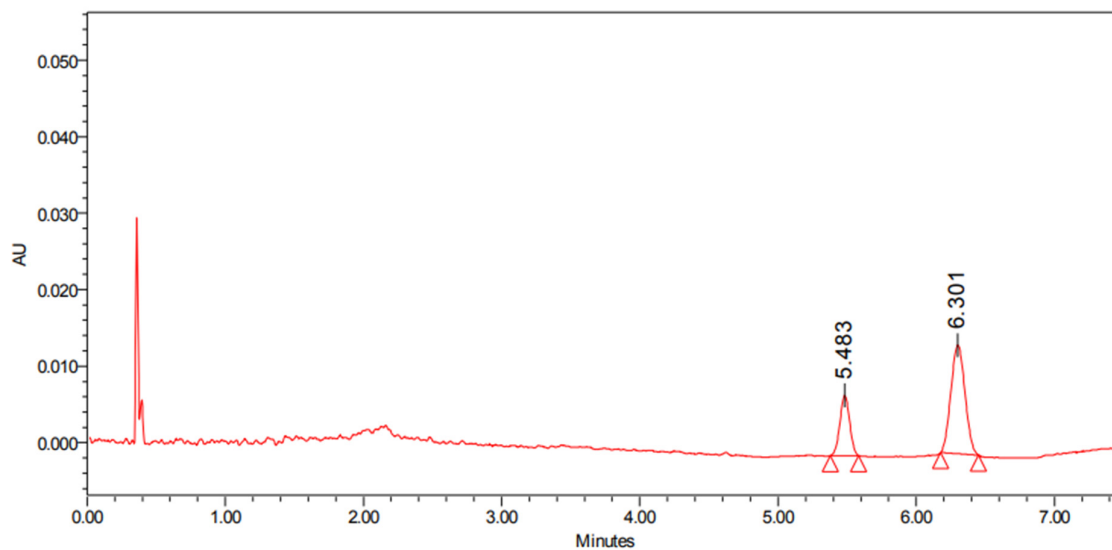
	RT	% Area
1	4.436	14.17
2	4.591	85.83

**(1*R*,3*S*,3*aS*,9*aS*)-6-Fluoro-1-(2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1*r*'**



**Peak Results**

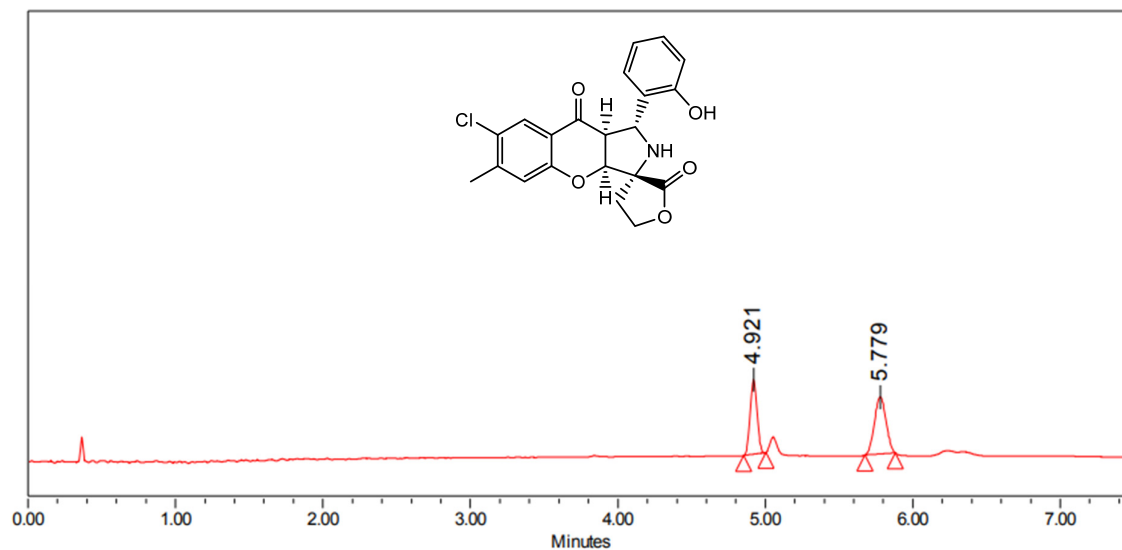
	RT	% Area
1	5.483	48.40
2	6.305	51.60



**Peak Results**

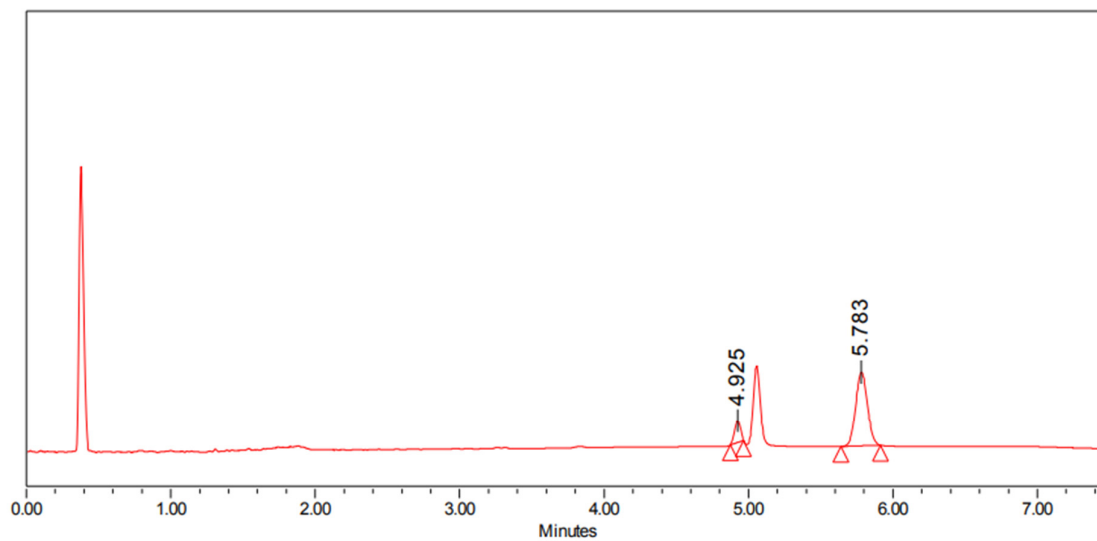
	RT	% Area
1	5.483	27.88
2	6.301	72.12

**(1*R*,3*R*,3*aS*,9*aS*)-7-Chloro-1-(2-hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1s**



**Peak Results**

	RT	% Area
1	4.921	44.00
2	5.779	56.00

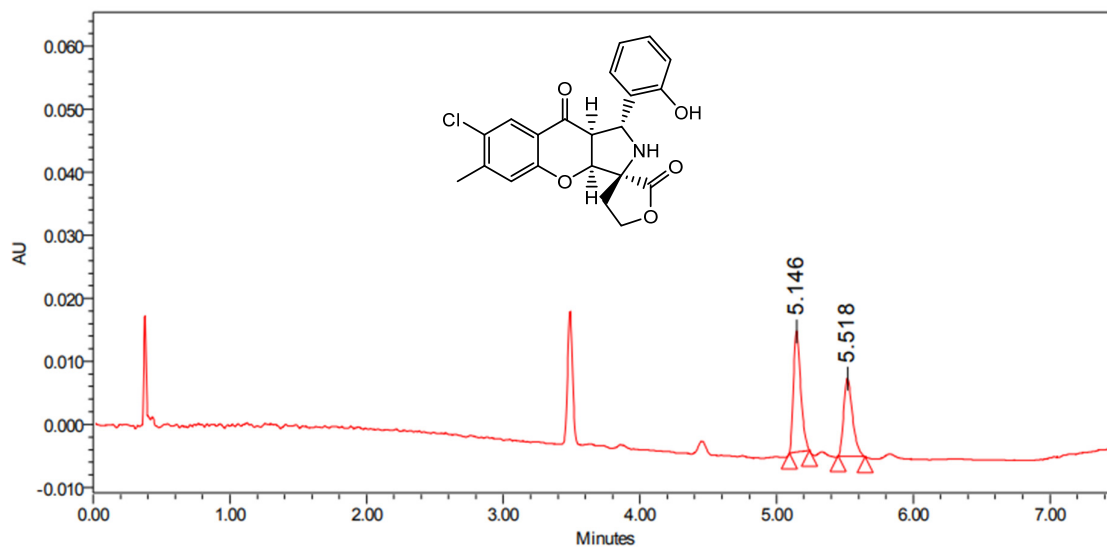


**Peak Results**

	RT	% Area
1	4.925	13.09
2	5.783	86.91

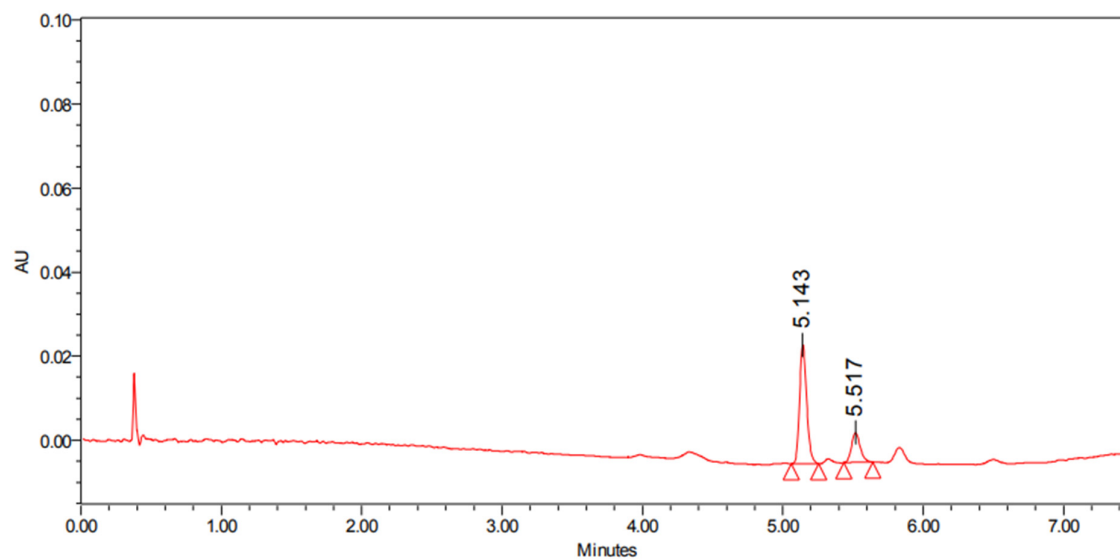


**(1*R*,3*S*,3*aS*,9*aS*)-7-Chloro-1-(2-hydroxyphenyl)-6-methyl-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1*s*'**



**Peak Results**

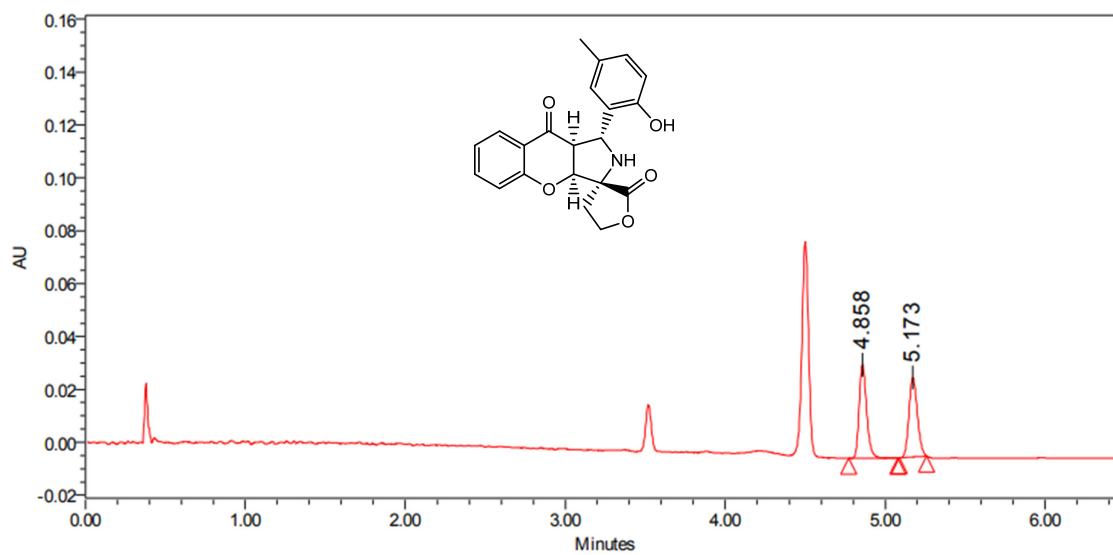
	RT	% Area
1	5.146	55.94
2	5.518	44.06



**Peak Results**

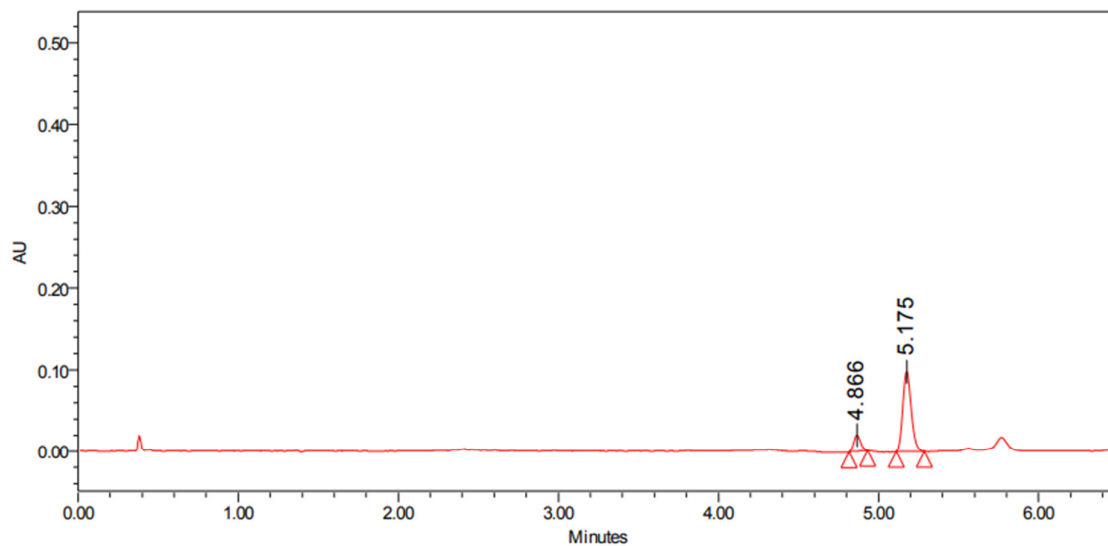
	RT	% Area
1	5.143	76.65
2	5.517	23.35

**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxy-5-methylphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1t**



**Peak Results**

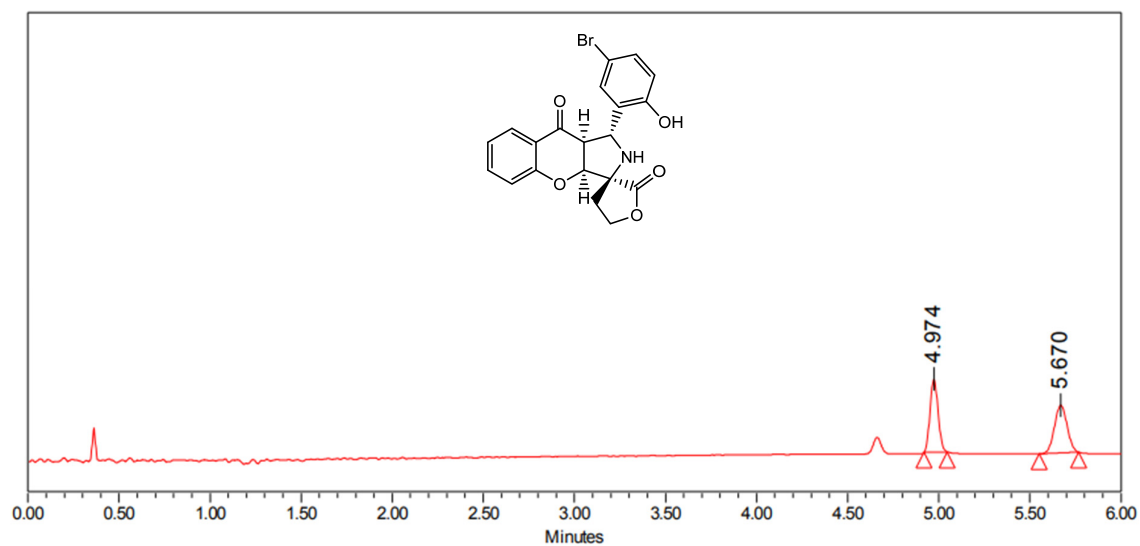
	RT	% Area
1	4.858	51.16
2	5.173	48.84



**Peak Results**

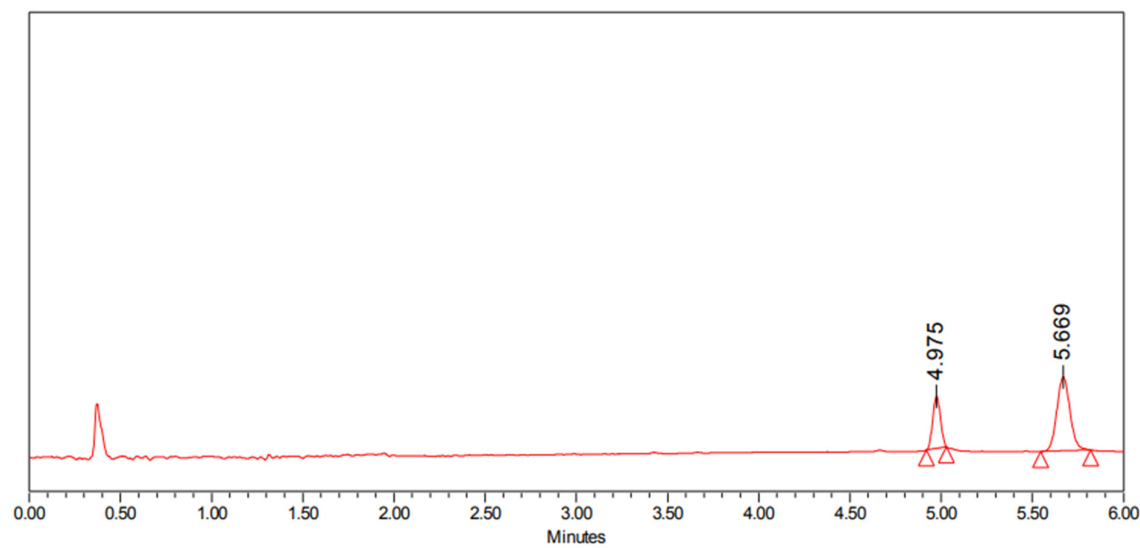
	RT	% Area
1	4.866	14.19
2	5.175	85.81

**(1*R*,3*S*,3*aS*,9*aS*)-1-(5-Bromo-2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1u'**



**Peak Results**

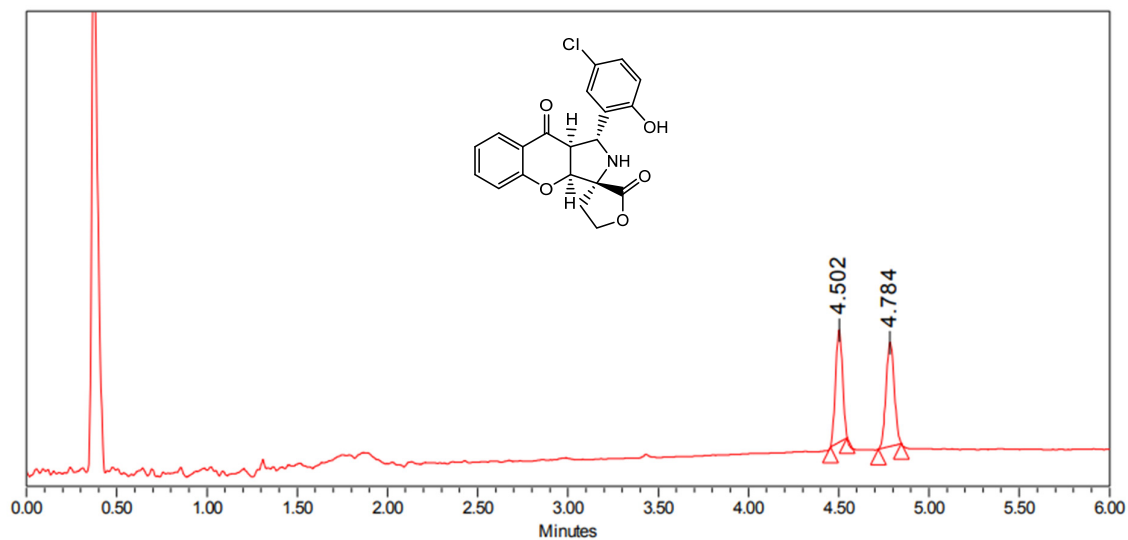
	RT	% Area
1	4.974	49.27
2	5.670	50.73



**Peak Results**

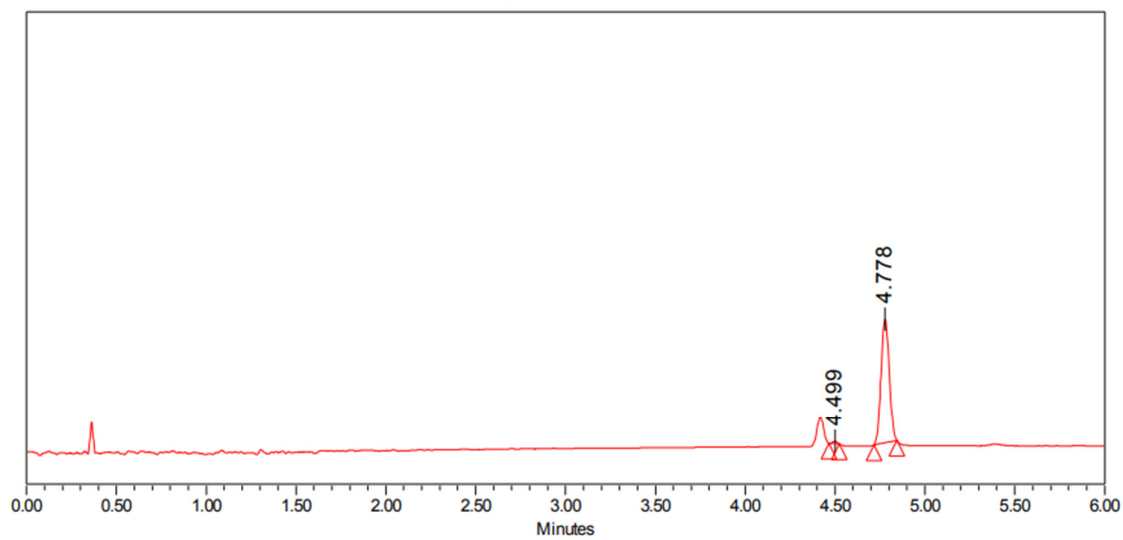
	RT	% Area
1	4.975	29.42
2	5.669	70.58

**(1*R*,3*R*,3*aS*,9*aS*)-1-(5-Chloro-2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1w**



**Peak Results**

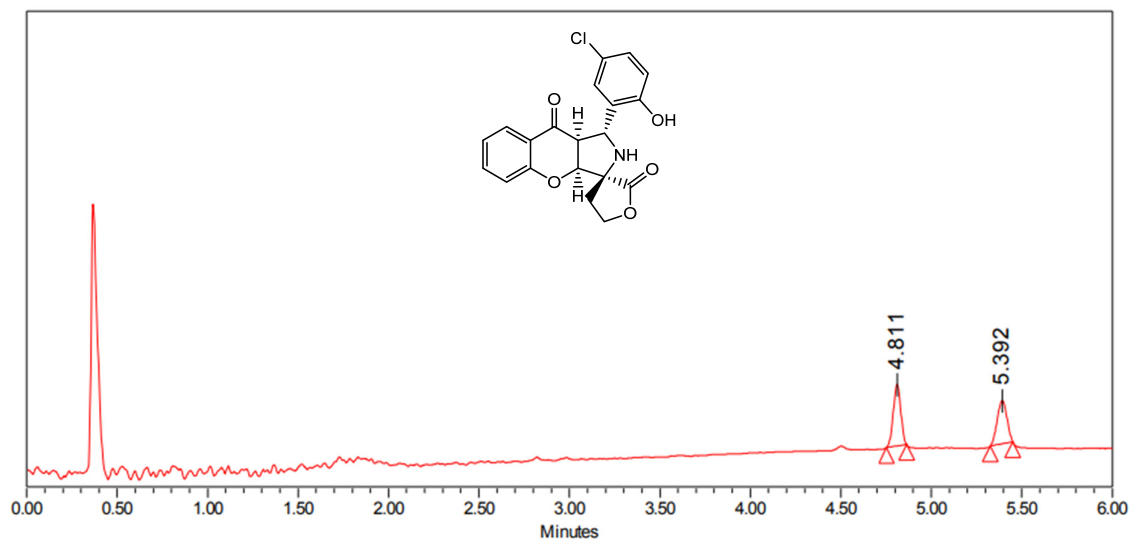
	RT	% Area
1	4.502	47.86
2	4.784	52.14



**Peak Results**

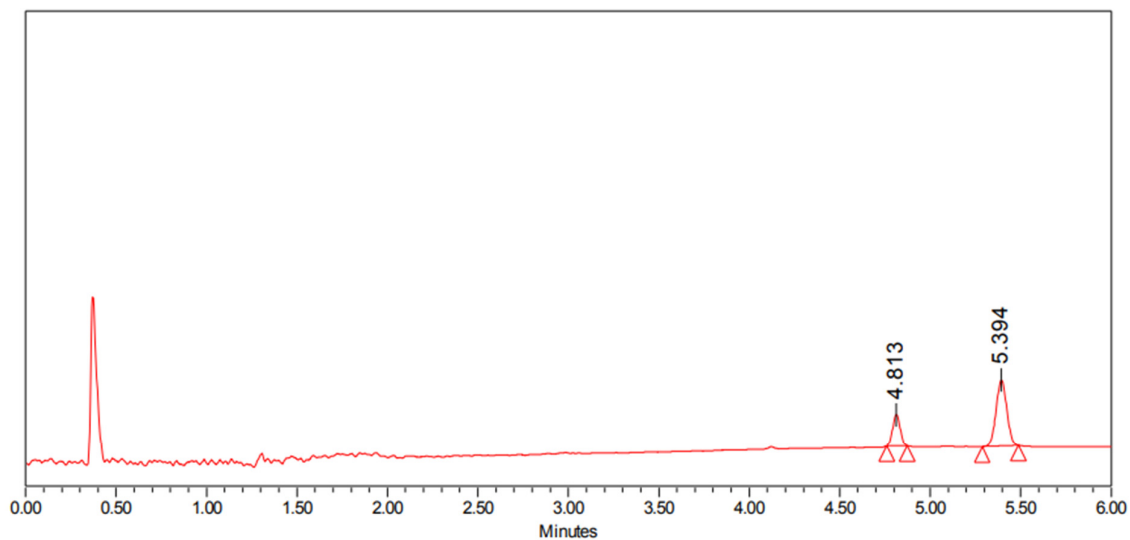
	RT	% Area
1	4.499	1.09
2	4.778	98.91

**(1*R*,3*S*,3*aS*,9*aS*)-1-(5-Chloro-2-hydroxyphenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1w'**



**Peak Results**

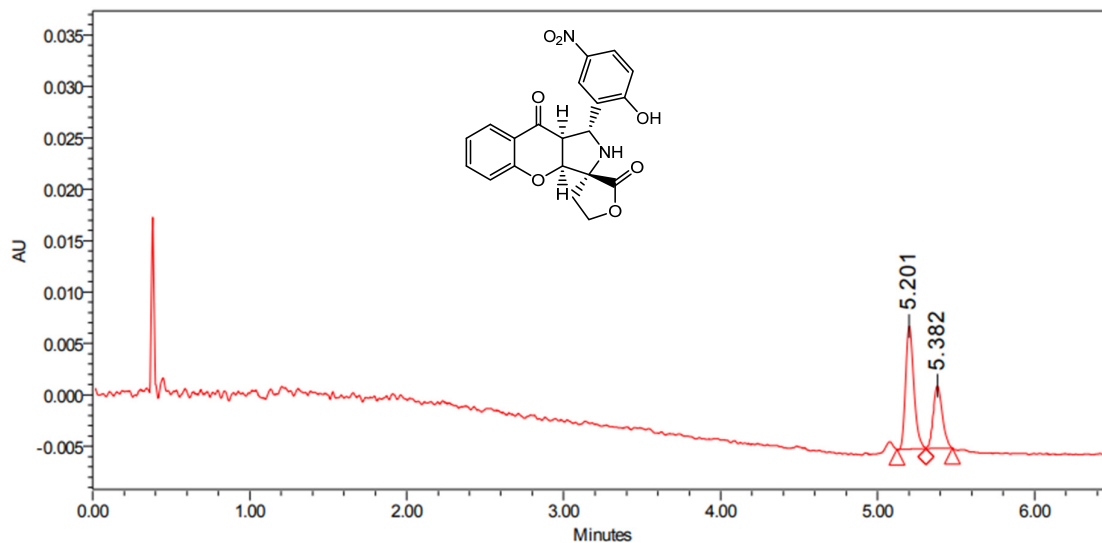
	RT	% Area
1	4.811	52.55
2	5.392	47.45



**Peak Results**

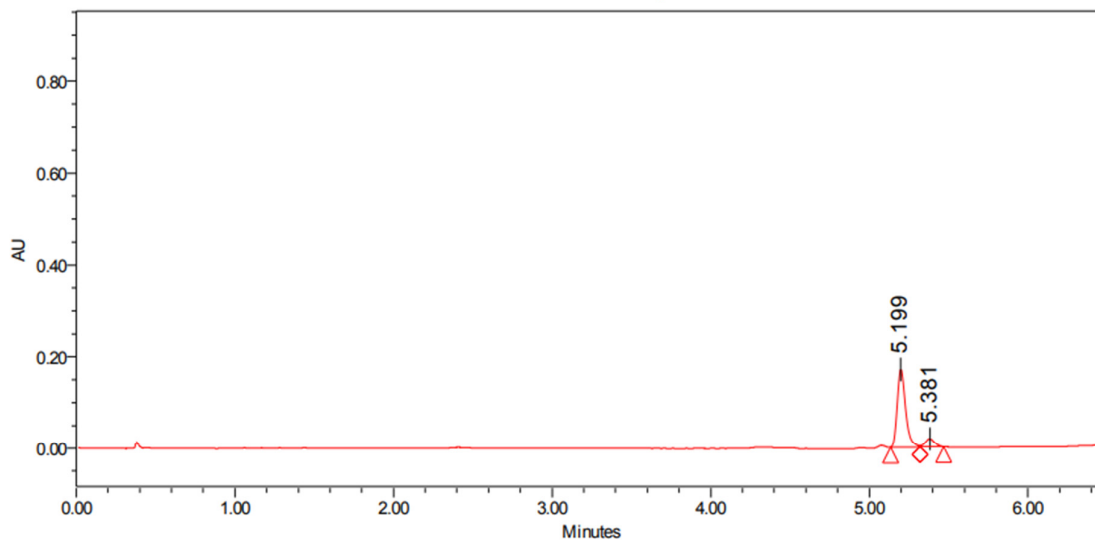
	RT	% Area
1	4.813	25.17
2	5.394	74.83

**(1*R*,3*R*,3*aS*,9*aS*)-1-(2-Hydroxy-5-nitrophenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1x**



**Peak Results**

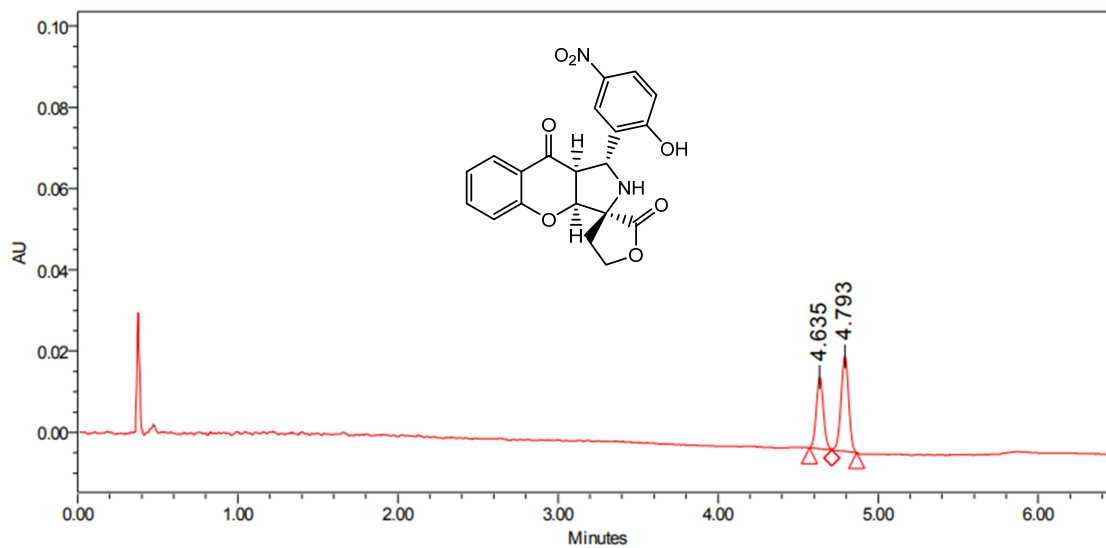
	RT	% Area
1	5.201	63.65
2	5.382	36.35



**Peak Results**

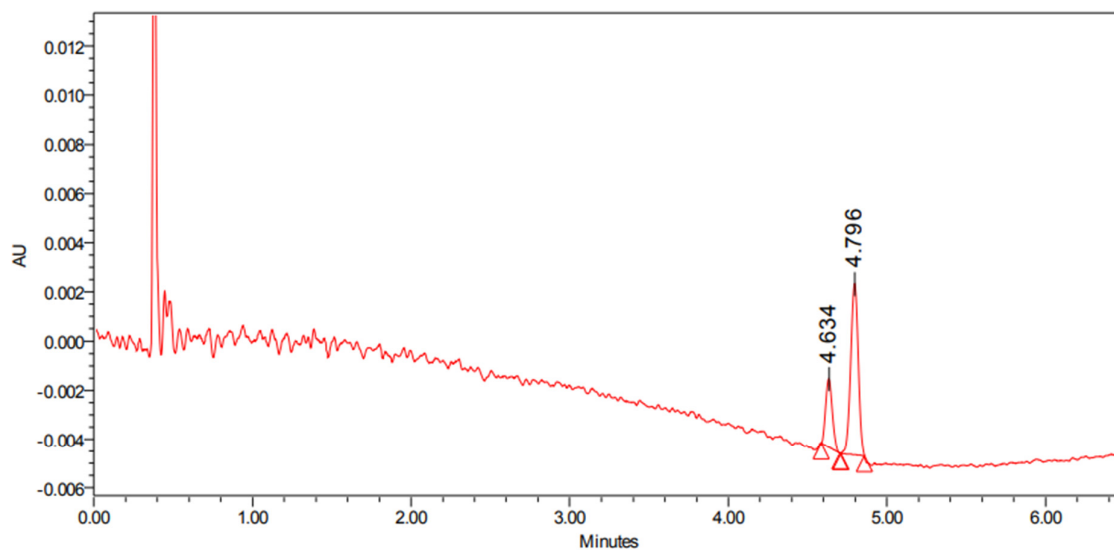
	RT	% Area
1	5.199	89.19
2	5.381	10.81

**(1*R*,3*S*,3*aS*,9*aS*)-1-(2-Hydroxy-5-nitrophenyl)-3*a*,4',5',9*a*-tetrahydro-1*H*,2'*H*-  
spiro[chromeno[2,3-*c*]pyrrole-3,3'-furan]-2',9(2*H*)-dione 1x'**



**Peak Results**

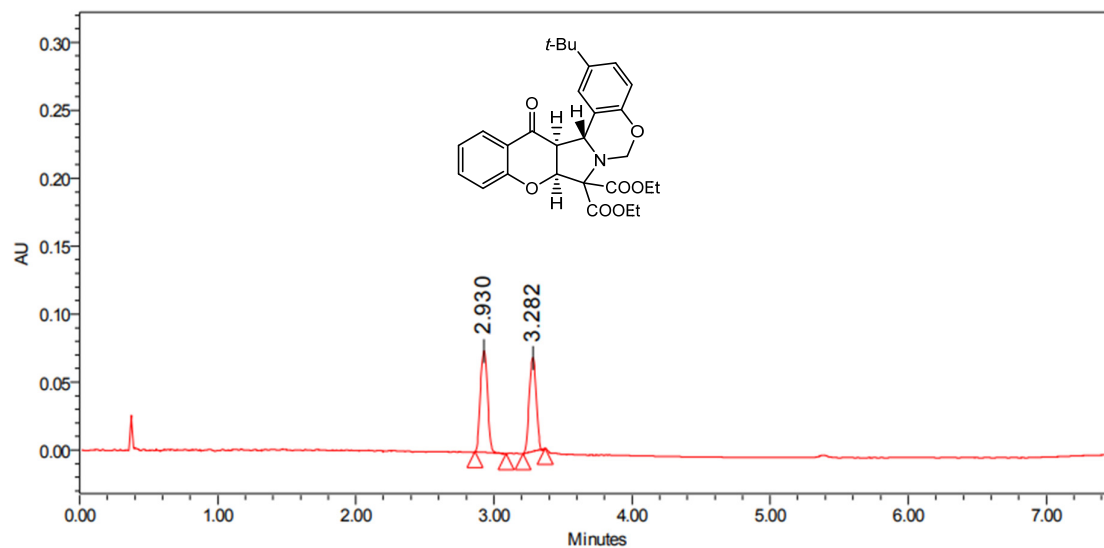
	RT	% Area
1	4.635	41.10
2	4.793	58.90



**Peak Results**

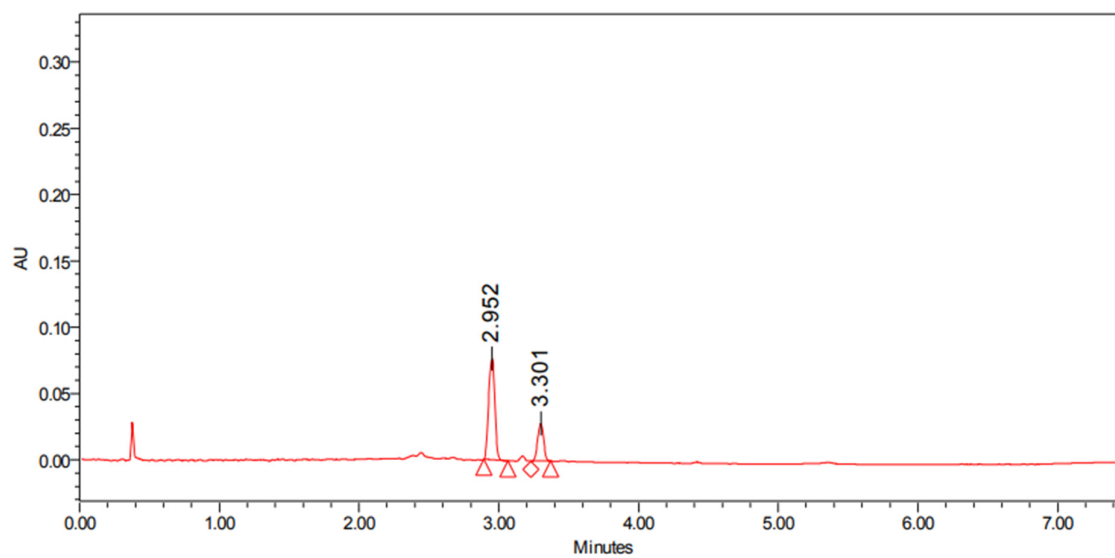
	RT	% Area
1	4.634	26.06
2	4.796	73.94

**(8a*S*,14a*S*,14b*R*)-Diethyl 2-(*tert*-butyl)-14-oxo-8a,14,14a,14b-tetrahydrobenzo-  
[*e*]chromeno[3',2':3,4]pyrrolo[1,2-*c*][1,3]oxazine-8,8(6*H*)-dicarboxylate 9a**



**Peak Results**

	RT	% Area
1	2.930	52.49
2	3.282	47.51



**Peak Results**

	RT	% Area
1	2.952	73.74
2	3.301	26.26