Electronic Supplementary Material (ESI) for Chemical Communications


**Supporting Information**

# Neutral tetrathia[22]annulene[2.1.2.1] based field-effect transistors. improved *on/off* ratio defies ring puckering†

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## General Information

$^{1}H$ (400 MHz), $^{13}C$ (100 MHz) NMR spectra were recorded on a BRUKER AVANCE II 400 NMR Spectrometer and JEOL-FT NMR-AL Spectrometer at 300MHz. Tetramethylsilane (TMS) served as the internal standard (0 ppm for $^{1}H$ and 77.0 ppm for $^{13}C$) and $CDCl_3$ was used as solvent. The following abbreviations were used to express the multiplicities: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; br = broad. Data are reported as follows: chemical shifts in ppm (δ), integration, coupling constant J (Hz) and assignment. Mass spectra were recorded on Bruker Daltonics esquire3000_00037 mass spectrometer. Elemental analyses were performed with a Thermoelectron FLASH EA1112 CHNS analyzer and were within ±0.4% of the theoretical values. IR spectrum was recorded on VARIAN 660-IR Fourier-Transform Spectrophotometer in range 400-4000 $cm^{-1}$ using KBr as medium. UV-Vis spectra were recorded on a SHIMADZU 1601 PC spectrophotometer, with a quartz cuvette (path length, 1 cm) and studies were performed in AR grade DCM. TGA were performed on a TA instrument with a DTG-60 detector with a temp. rise of 10 $^{\circ}$C/minute under nitrogen atmosphere. Electrochemical studies were carried out on CHI 660C Electrochemical Workstation with a conventional three-electrode configuration consisting of platinum working electrode (2 mm diameter), counter electrode and Ag/AgCl as reference electrode. The experiments were carried out on $10^{-4}$ M solutions of samples in DCM containing 0.1 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$) as supporting electrolyte at room temperature. Deoxygenation of the solutions was achieved by bubbling nitrogen for 30 min and the working electrode was cleaned after each run. The cyclic voltammograms were recorded with a scan rate of 100 $mVs^{-1}$. All reactions were monitored by thin-layer chromatography carried out on Merck pre-coated TLC plates (silica gel 60 $F_{254}$, 0.25 mm), visualization by using UV (254 nm). Melting points were determined in open capillaries and are uncorrected. Reactions that required anhydrous conditions were carried out under the blanket of deoxygenated (BASF catalyst) anhydrous nitrogen gas in oven/flame dried glassware. The products were purified by flash column chromatography on silica gel 60-120 mesh. All reagents and chemicals were purchased from Sigma-Aldrich. DMF, Diethyl ether, $TiCl_4$, Pyridine and DCM were purchased locally, dried and distilled prior to use. n-Buli was prepared and stored under a blanket of dry nitrogen gas. THF and toluene were distilled from sodium/benzophenone (benzophenone ketyl). Anhydrous DCM was stored over fused $CaCl_2$ and distilled before use. Zinc dust was activated prior to use using standard (2M

HCl and subsequent water washing) methods. DDQ and hydrazine hydrate were purchased from Sigma-Aldrich, and were used as received. All theoretical studies were performed with a GAUSSIAN 09 software package.

## Experimental Section

### General procedure for the synthesis of *meso*-substituted dithienyl methane dialdehydes (9a,b):

To a solution of *meso-p*-florophenyl dithienyl methane 1g (3.65 mmol) in dry diethyl ether (33ml) was added dropwise, n-Buli (8.2 mmol) at r.t.. to the deep red solution so obtained was added dropwise, anhydrous DMF (8 mmol) in diethyl ether. After 1 hour stiring, the mixture was washed sucessively with water, dil. HCl, water and sodium bicarbonate. The organic phase was then dried and vaccum evaporated. The residue was chromatographed on silica to give (1) as a deep red oil (750 mg) 62.3%.

The characteristic data for **8a**, **8b**, **9a** and **9b** is presented below.

### *m*-Chlorophenyl-di(thien-2yl)methane, 8a

(Yield = 16%), $^{1}$H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.27 (1H, m), 7.15-7.22 (5H, m), 6.90-6.93 (2H, m), 6.79-6.80 (2H, m), 5.81 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$): δ (ppm) 146.5, 145.5, 134.3, 129.7, 128.4, 127.3, 126.6, 126.4, 126.2, 124.9, 46.9; *m/z* 290.5, colourless oil.

### *p*-Florophenyl-di(thien-2yl)methane, 8b

(Yield = 15%), $^{1}$H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.27 (2H, m), 7.19-7.22 (4H, m), 6.93 (2H, t, J= 4.22 hz), 6.79 (2H, d, J= 4 hz), 5.82 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$): δ (ppm) 147, 142.2, 133, 129.7, 128.7, 126.7, 126.2, 124.9, 46.8; *m/z* 274.2, colourless oil.

### *m*-Chlorophenyl-di(5-formylthien-2yl)methane, 9a

(Yield = 64%), $^{1}$H NMR (400 MHz, $CDCl_3$) 9.86 (2H, s), 7.65 (2H, d, J=3.6 Hz), 7.30-7.32 (2H, m), 7.26 (1H, s), 7.16-7.18 (1H, m), 6.98 (2H, dd, J=2.8 Hz), 5.87 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$) 182.7, 155.5, 143.5, 142.8, 136.3, 135, 130.4, 128.5, 128.4, 127.9, 126.4, 48.1; *m/z* 347 (M, 100%).

***p*-Florophenyl-di(5-formylthien-2yl)methane**, **9b**

(Yield = 62.3%), $^{1}$H NMR (400 MHz, $CDCl_3$) 9.83 (2H, s), 7.63-7.65 (2H, m), 7.23-7.27 (2H, m), 7.01-7.06 (2H, m), 6.95-6.96 (2H, m), 5.89 (1H, s); $^{13}$C NMR (100 MHz, $CDCl_3$) 182.7, 156.2, 143.2, 136.2, 129.9, 129.8, 127.7, 116.1, 115.8, 47.7; *m/z* 331 (M+, 100%).

**General procedure for the synthesis of the *meso*-substituted dihydrotetrathia annulenes 10a and 10b:**

To a stirring suspension of zinc dust (38 mmol) in 200 ml of THF maintained under nitrogen atmosphere, a solution of 19.6 ml of 1.0 M $TiCl_4$ (in $CH_2Cl_2$) was added over 20 minutes. The reaction mixture was refluxed for 1 hour, and treated with a solution of appropriate dialdehyde (1.78 mmol) and pyridine (35.6 mmol) dissolved in 200 ml of THF. The addition was made using a hypodermic syringe over 40 minutes to the gently refluxing suspension. After refluxing under nitrogen for 18 hours, the reaction was carefully quenched with a solution of aqueous $K_2CO_3$ (10%, 100 ml). The reaction mixture was filtered, and the filtrate was concentrated under reduced pressure and the residue extracted with 300 ml methylene chloride. The extract was washed with water (2 x 50 ml) and dried over anhydrous sodium sulfate. The solvent was evaporated under reduced pressure and the residue was chromatographed (over silica) to isolate the **10a,b** as light green solids (25%),(**10a**,**b**, m.pt. >280 ˚C).

***Meso-m*-chlorophenyldihydrotetrathia[22]annulene[2,1,2,1] 10a**, (Yield = 13%), $^{1}$H NMR (400 MHz, $CDCl_3$) 7.31 (2H, s), 7.20-7.27 (6H, m), 6.82-6.83 (4H, m), 6.65 (1H, dd, J= 4 Hz), 6.61 (3H, dd, J= 2.72 Hz), 6.51 (4H, s), 5.71 (2 H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) 148.2, 144.7, 138.7, 134.3, 129.8, 128.6, 128.5, 127.3, 126.7, 125.8, 123.6, 47.8; IR (KBr): 687, 763, 807,889, 953, 1025, 1254, 1427, 1473, 1570, 1590, 2961, 3014, 3060, 3433 $cm^{-1}$; (M+K) 668.

***Meso-p*-florophenyldihydrotetrathia[22]annulene[2,1,2,1] 10b**, (Yield =15%), $^{1}$H NMR (400 MHz, $CDCl_3$) 7.26-7.30 (4H, m), 6.99-7.03 (4H, m), 6.80-6.82 (4H, m), 6.63 (2H, dd, J=4 Hz), 6.60 (2H, dd, J= 4 Hz), 6.5 (4H, m), 5.73-5.75 (2H, m); $^{13}$C NMR (100 MHz, $CDCl_3$) 148.9, 138.6, 130, 128.6, 125.6, 123.6, 123.4, 115.5, 115.2, 47.4; IR (KBr):724, 793, 810, 860, 1025, 1158, 1222, 1389, 1506, 1600, 1621, 1892, 2970, 3020, 3065, 3401 $cm^{-1}$; (M + K) 635.

**General procedure for the synthesis of the *meso*-substituted tetrathia[22]annulenes 11a and 11b:**

To a solution of **10a** (0.2 mmol) in 5 ml toluene, was added under nitrogen with stirring, a solution of DDQ (0.5 mmol) in 5 ml toluene. Shortly after mixing the two solutions, purple precipitates formed and the reaction mixture was stirred for additional 3 h. The purple precipitates were filtered and added to 3 ml of hydrazine hydrate (98%). After boiling for 10 minutes, the solid was filtered, washed with water, and dried. The resulting product was dissolved in methylene chloride and chromatographed on silica (DCM). Evaporation of the purple solution gave **11a** (50%) as shining metallic purple solid (**11a**,**b**, m.pt. >280 ˚C).

***Meso-m*-clorophenyltetrathia[22]annulene[2,1,2,1] 11a**, (Yield = 50%), $^1$H NMR (400 MHz, $CDCl_3$) 11.09 (4H, s), 10.41 (4H, d, J= 4 Hz), 9.98 (4H, d, J= 4.4 Hz ), 8.47 (2H, s), 8.33-8.35 (2H, m), 7.91-7.97 (4H, m); IR (KBr): 776, 810, 1069, 1168, 1359, 1464, 1588, 1632, 2923, 3398 cm$^{-1}$; Anal. Calcd. (%) for $C_{34}H_{20}S_4Cl_2$: C, 65.07; H, 3.19; S, 20.41; Found: C, 65.09; H, 3.19; S, 20.40; *m/z* 627.9 (100%).

***Meso-p*-florophenyltetrathia[22]annulene[2,1,2,1] 11b**, (Yield = 45%), $^1$H NMR (400 MHz, $CDCl_3$) 11.10 (4H, s), 10.41 (4H, d, J= 4.8 Hz), 9.98 (4H, d, J= 4.8 Hz), 8.41-8.44 (4H, m), 7.67-7.72 (4H, m); IR (KBr): 809, 1154, 1217, 1504, 3055 cm$^{-1}$; Anal. Calcd. (%) for $C_{34}H_{20}S_4F_2$ : C, 68.68; H, 3.36; S, 21.54 ; Found: C, 68.66; H, 3.35; S, 21.53; *m/z* 594 (100%).

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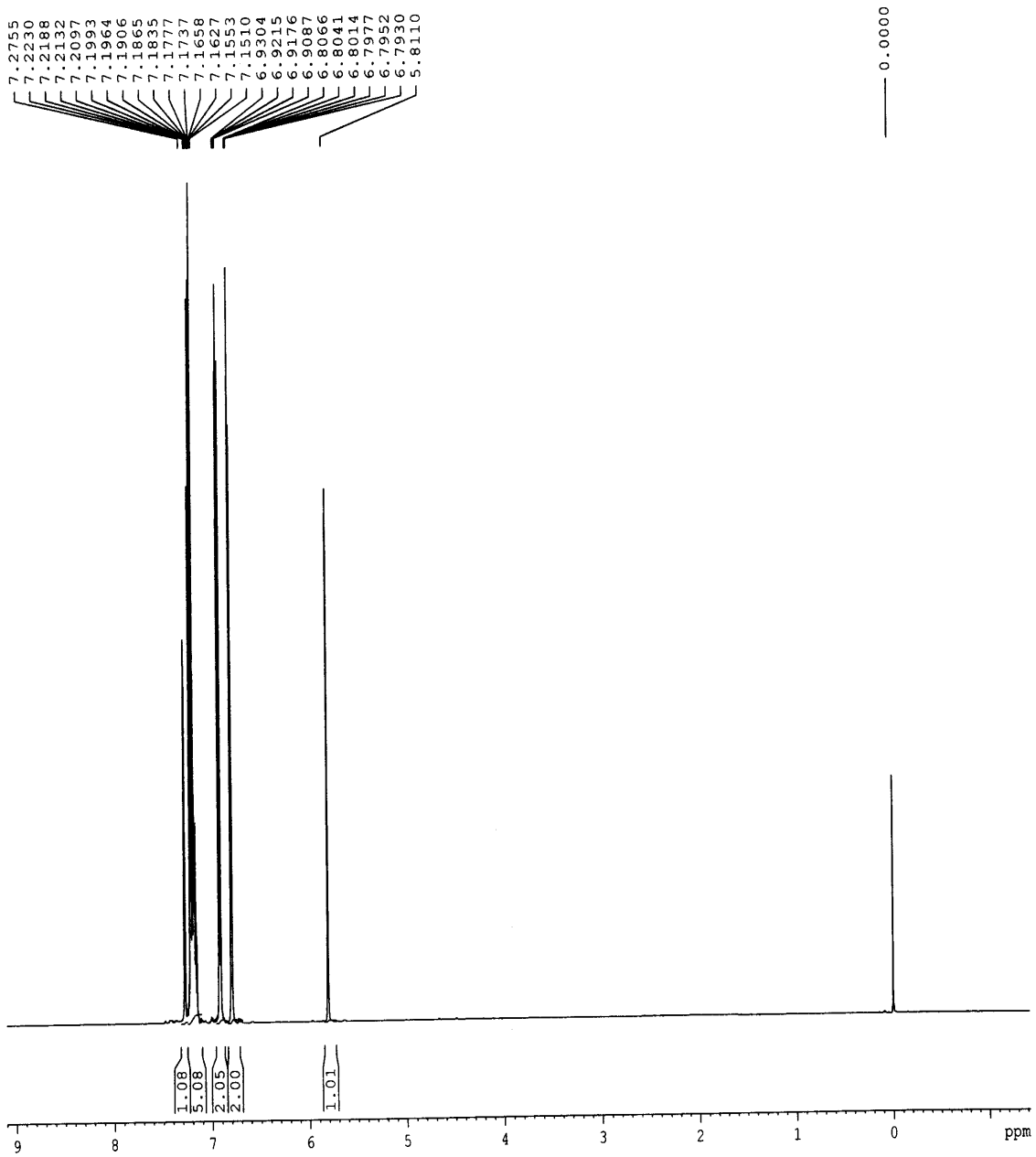



**Figure S1**: $^{1}$H NMR spectrum of **8a**.

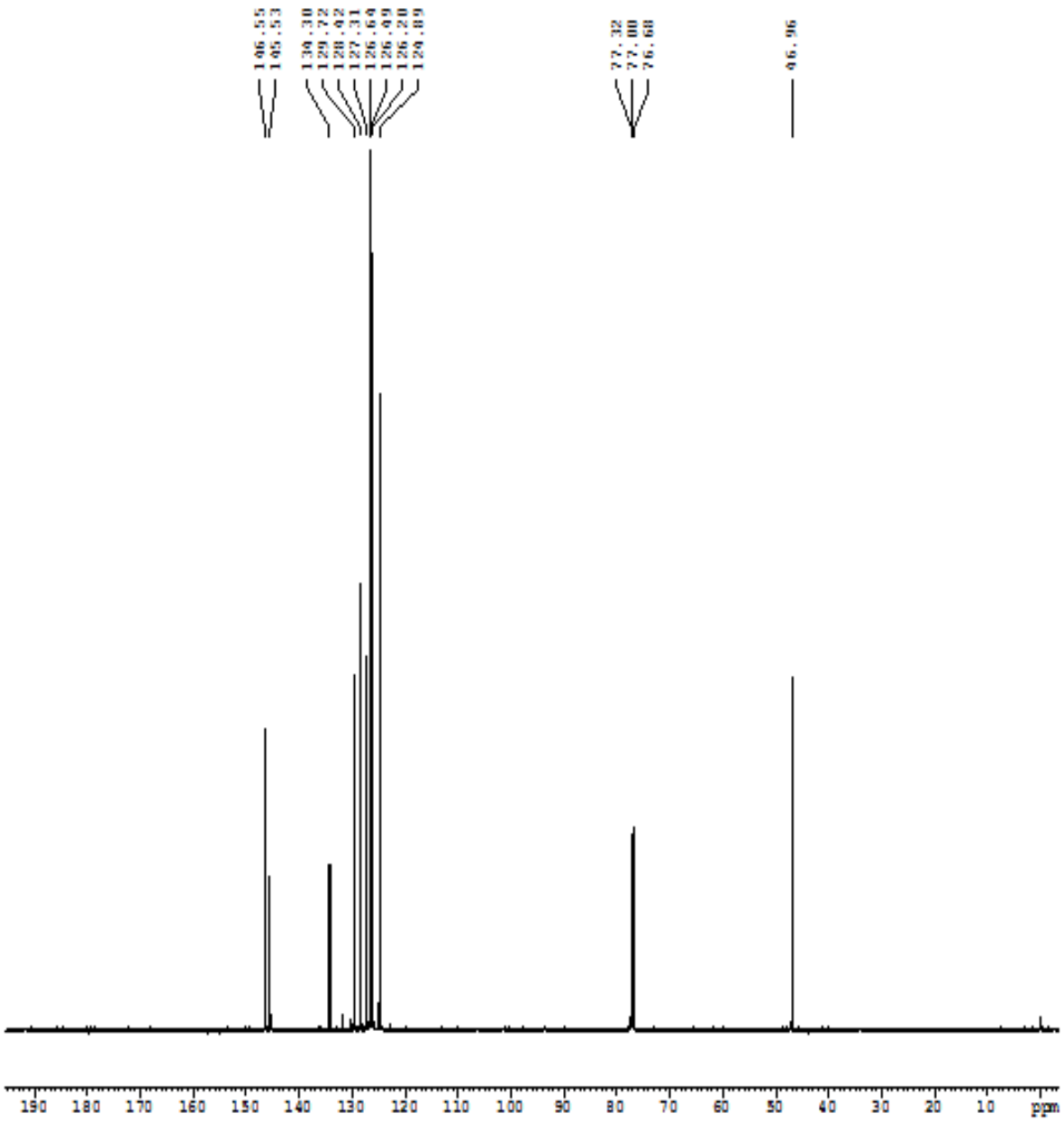



**Figure S2**: $^{13}$C NMR spectrum of **8a**.

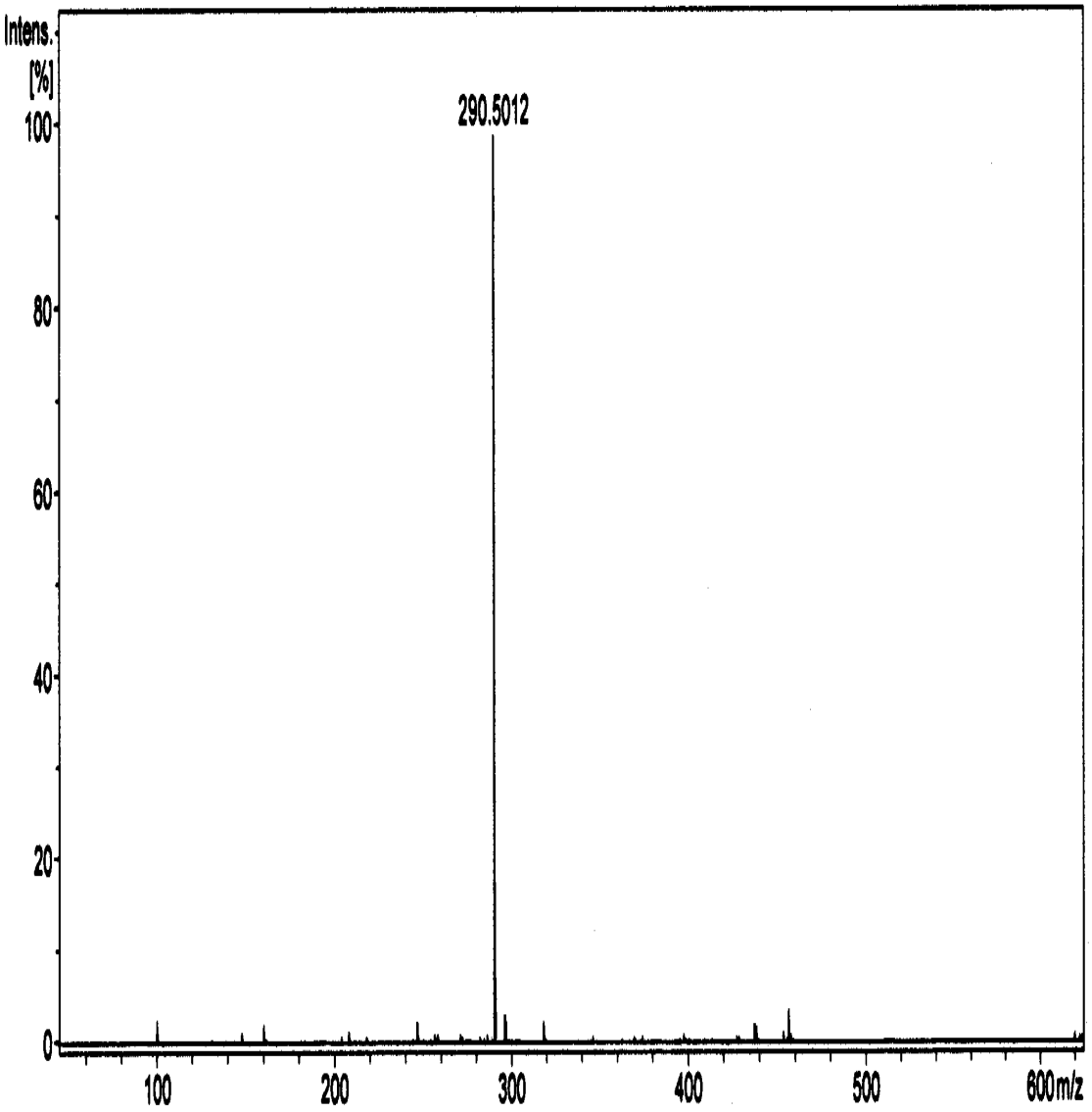



Figure **S3**: Mass spectrum of **8a**.

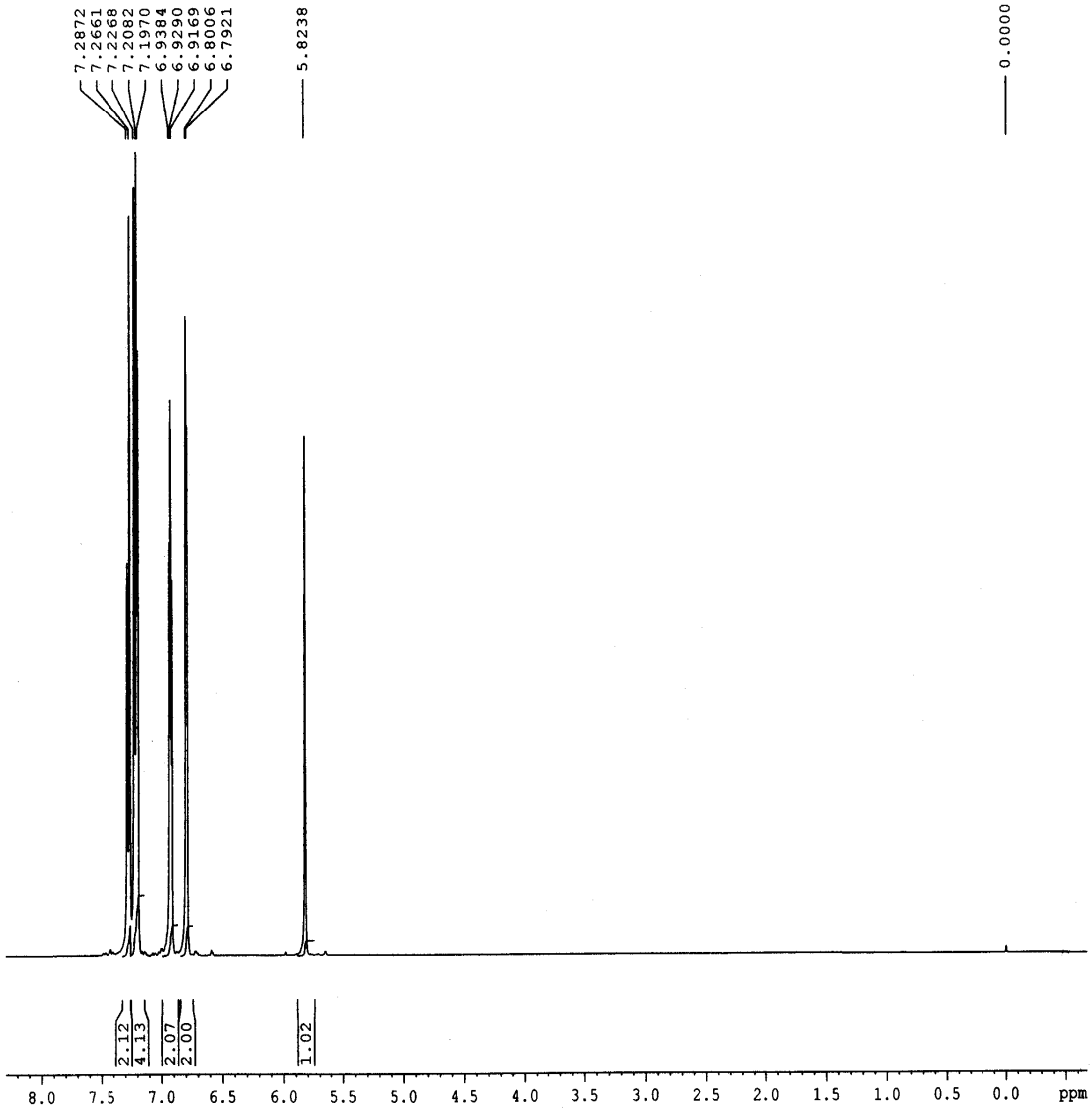



Figure **S4**: $^{1}$H NMR spectrum of **8b**.

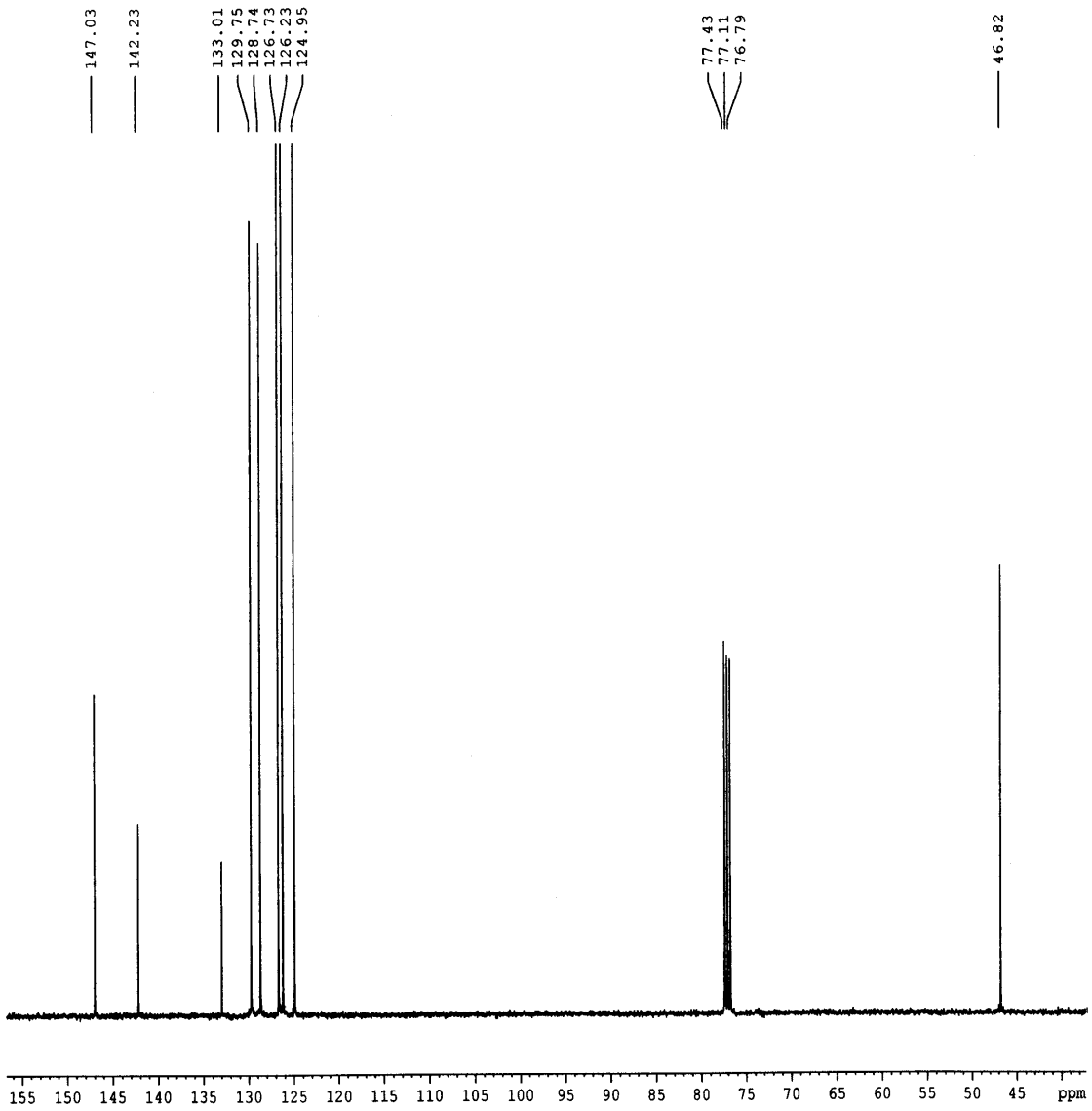



Figure **S5**: $^{13}$C NMR spectrum of **8b**.

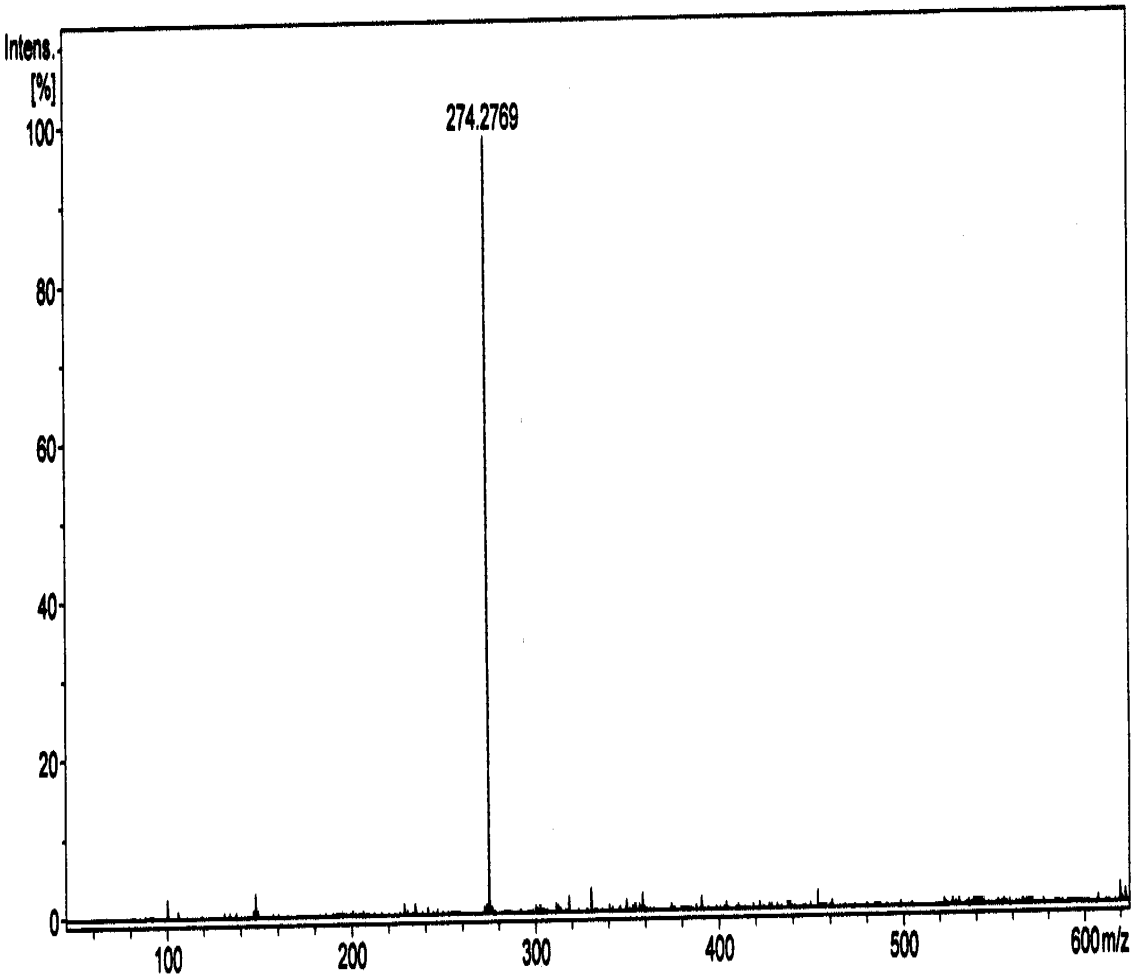



Figure **S6**: Mass spectrum of **8b**.

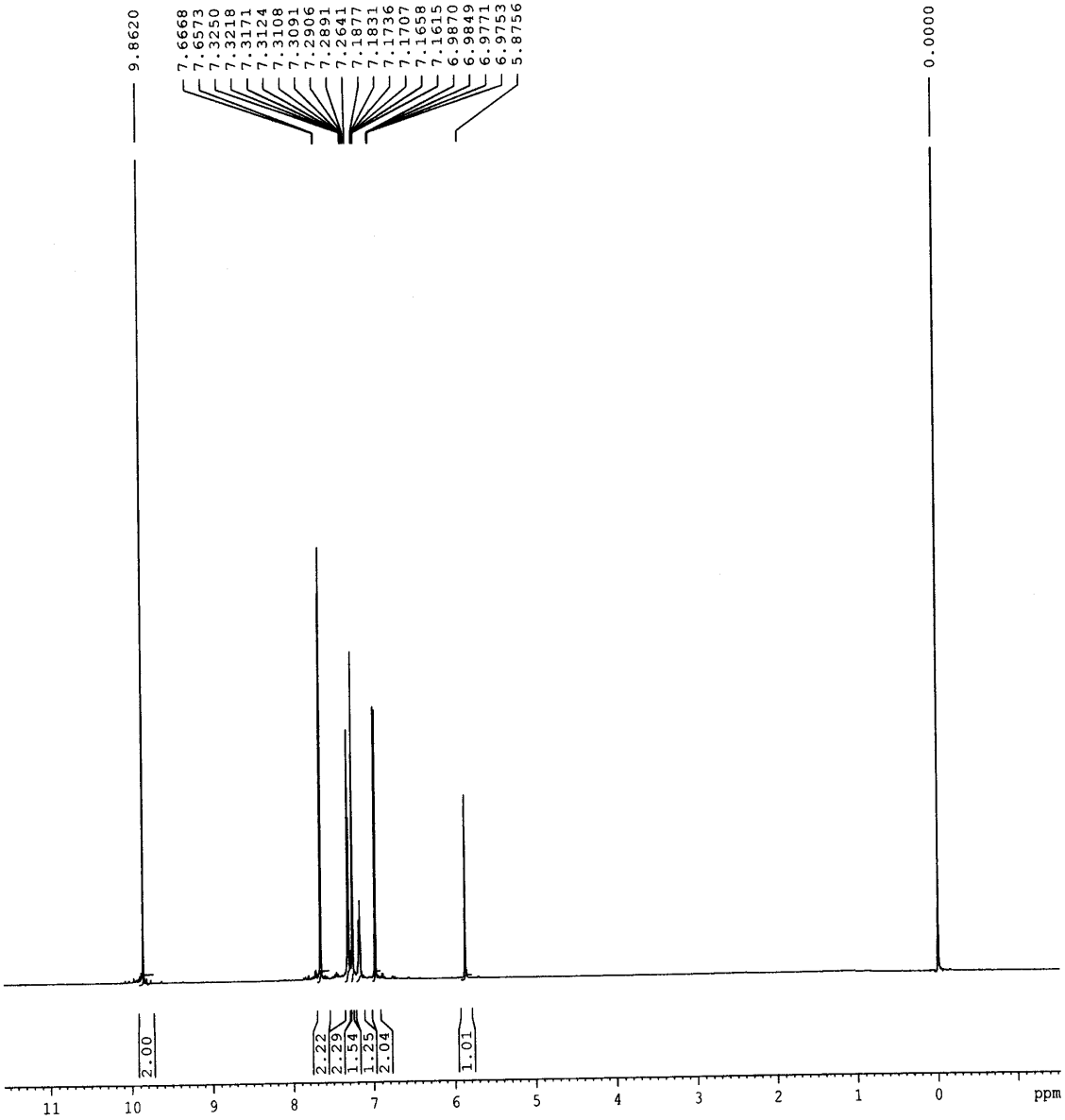



Figure **S7**: $^{1}$H NMR spectrum of **9a**.

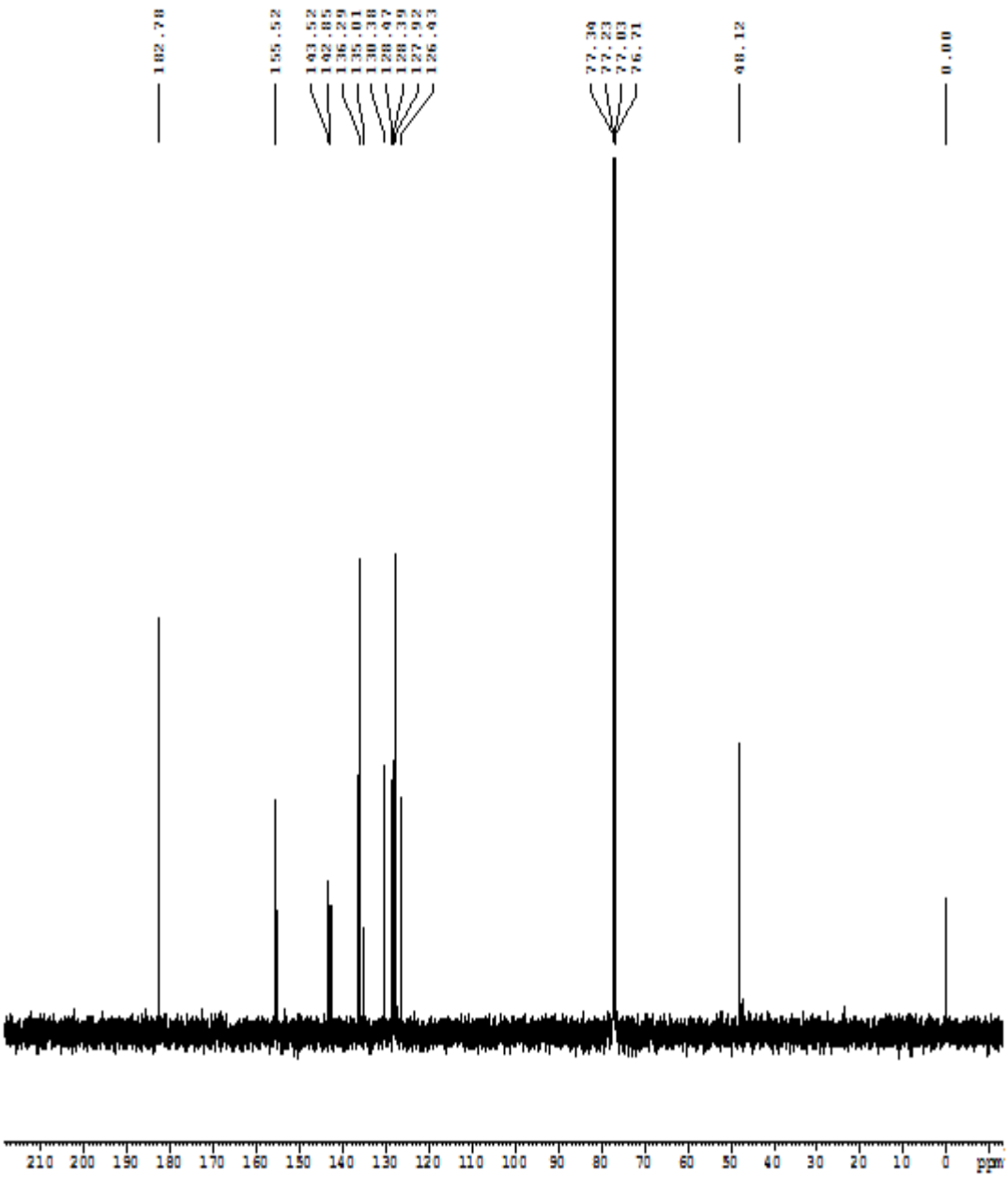



Figure **S8**: $^{13}$C NMR spectrum of **9a.**

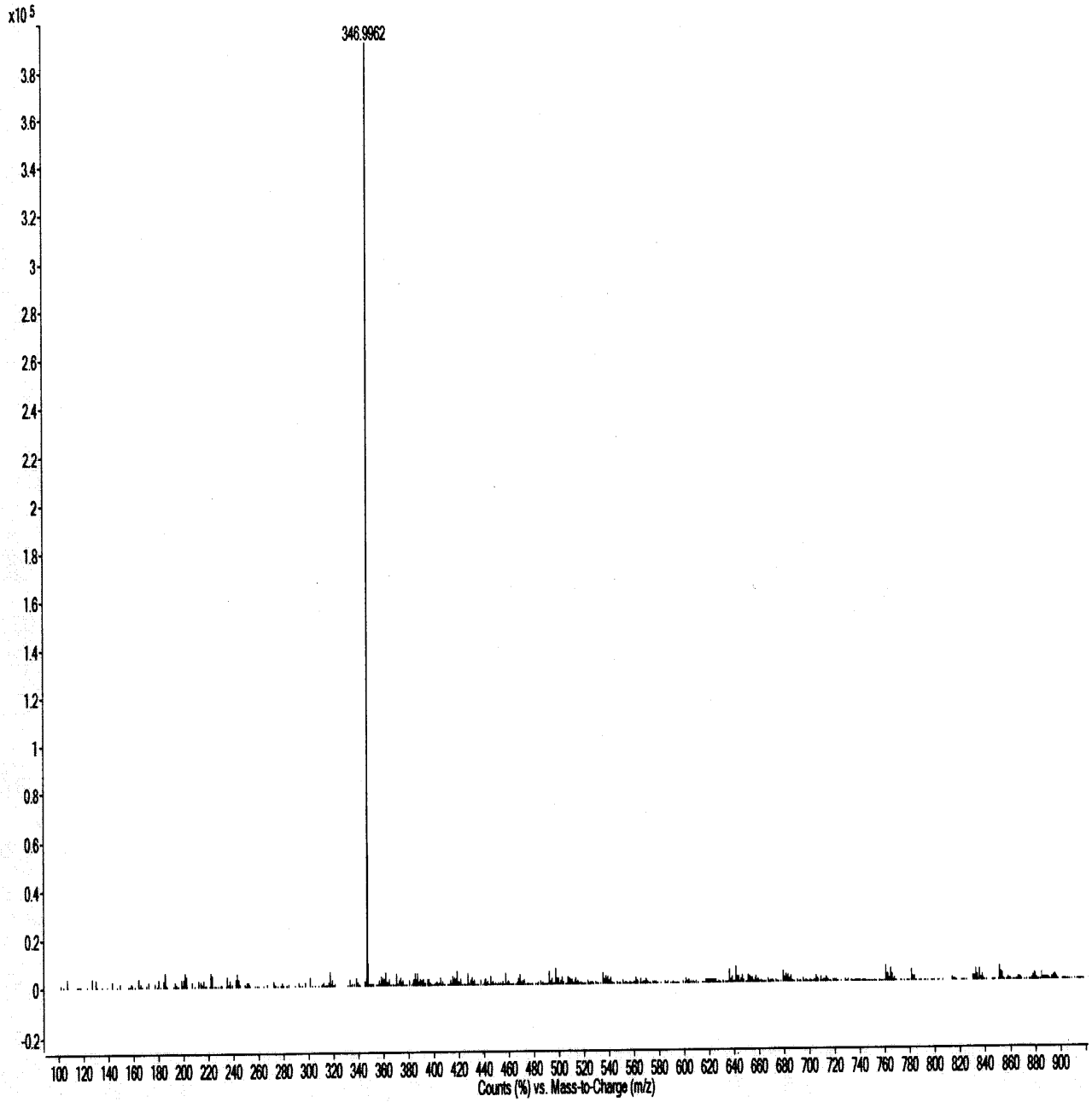



Figure **S9**: Mass spectrum spectrum of **9a.**

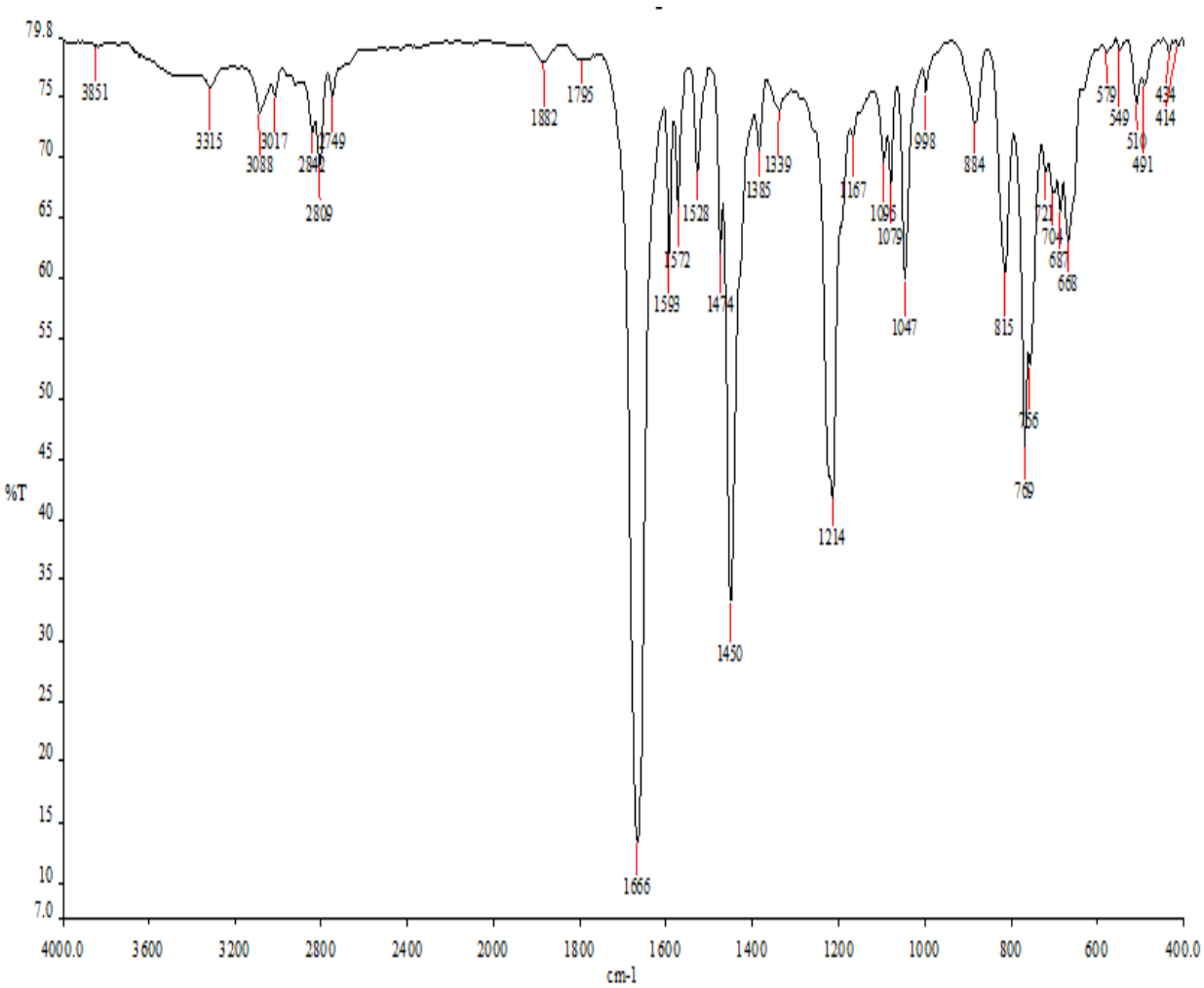



Figure **S10**: IR Spectrum of **9a**.

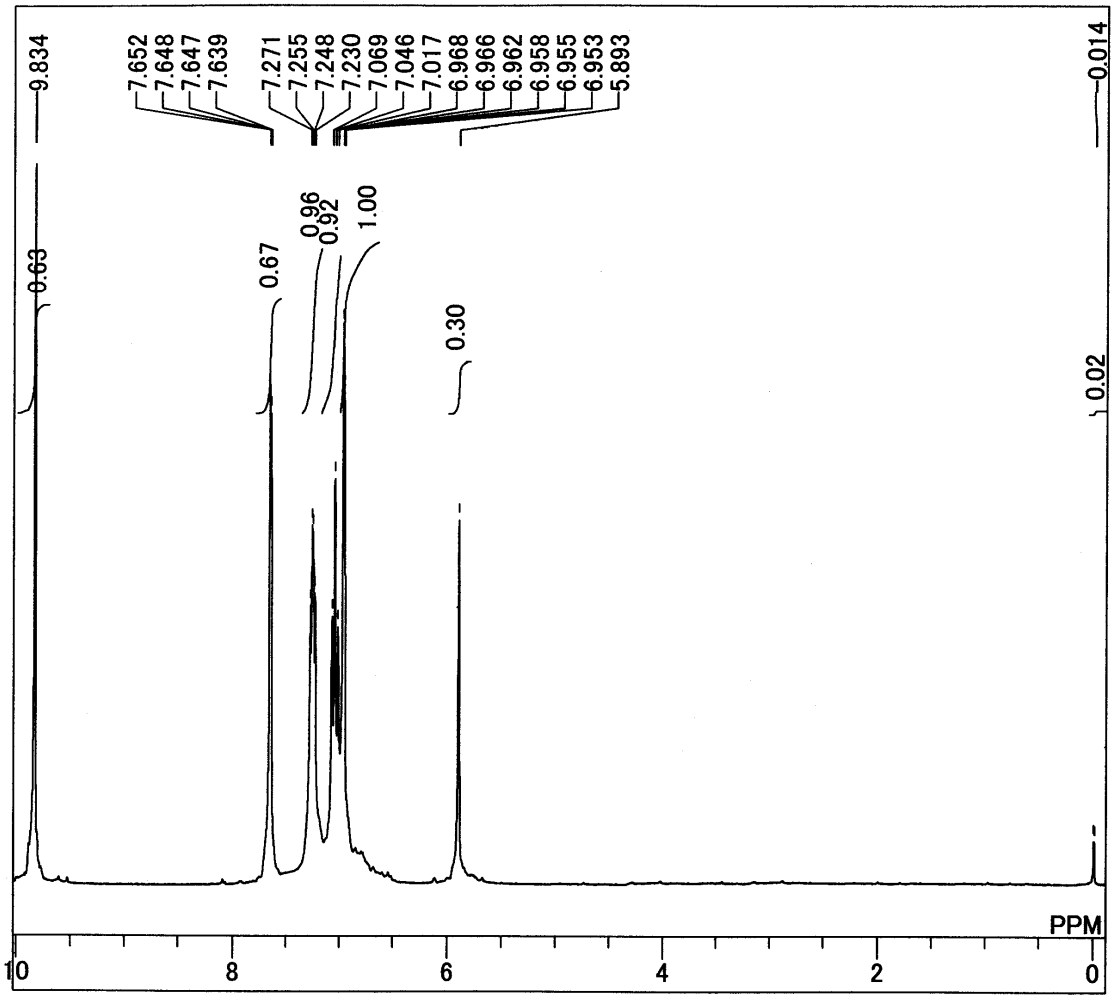



Figure **S11**: $^{1}$H NMR spectrum of **9b**.

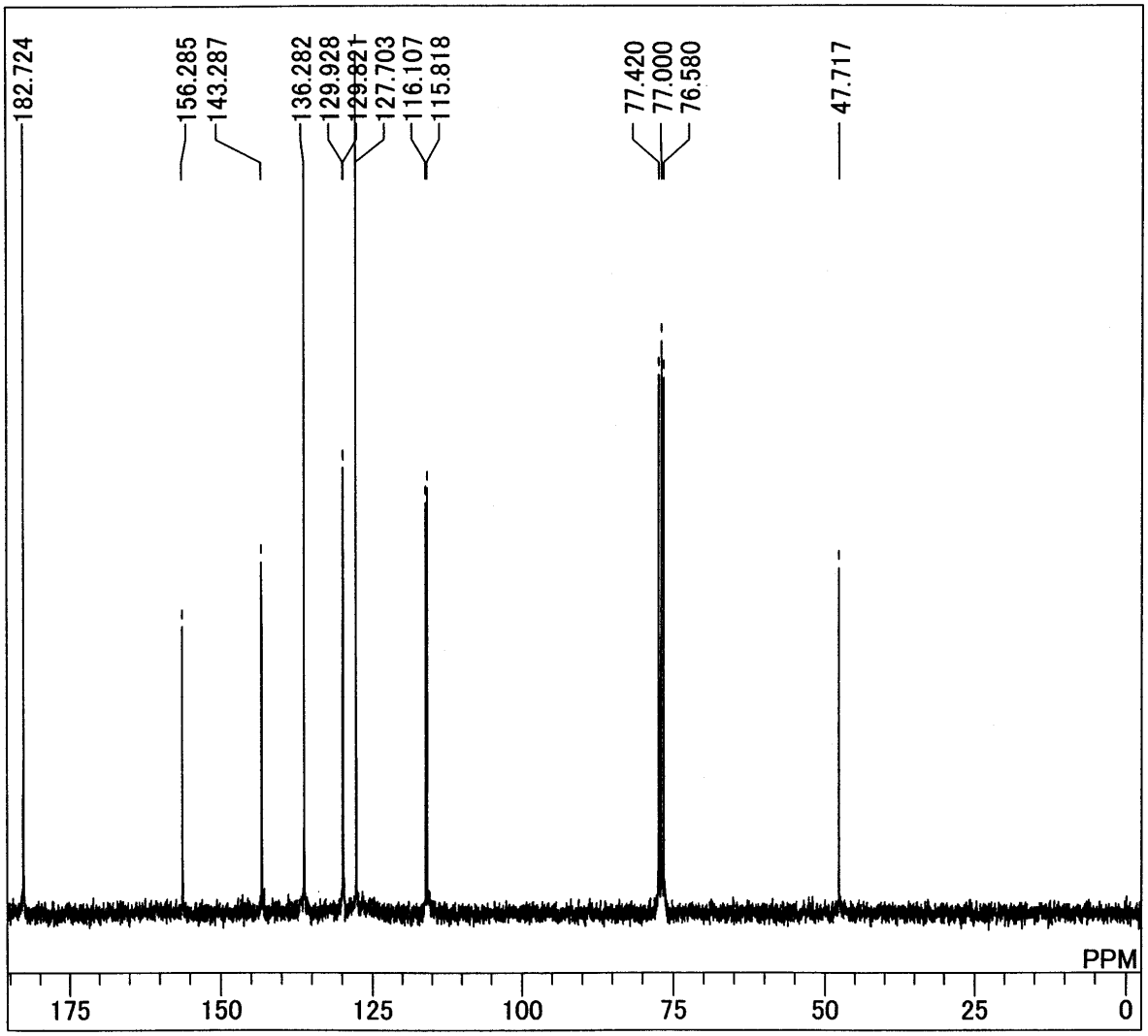



Figure **S12**: $^{13}$C NMR spectrum of **9b**.

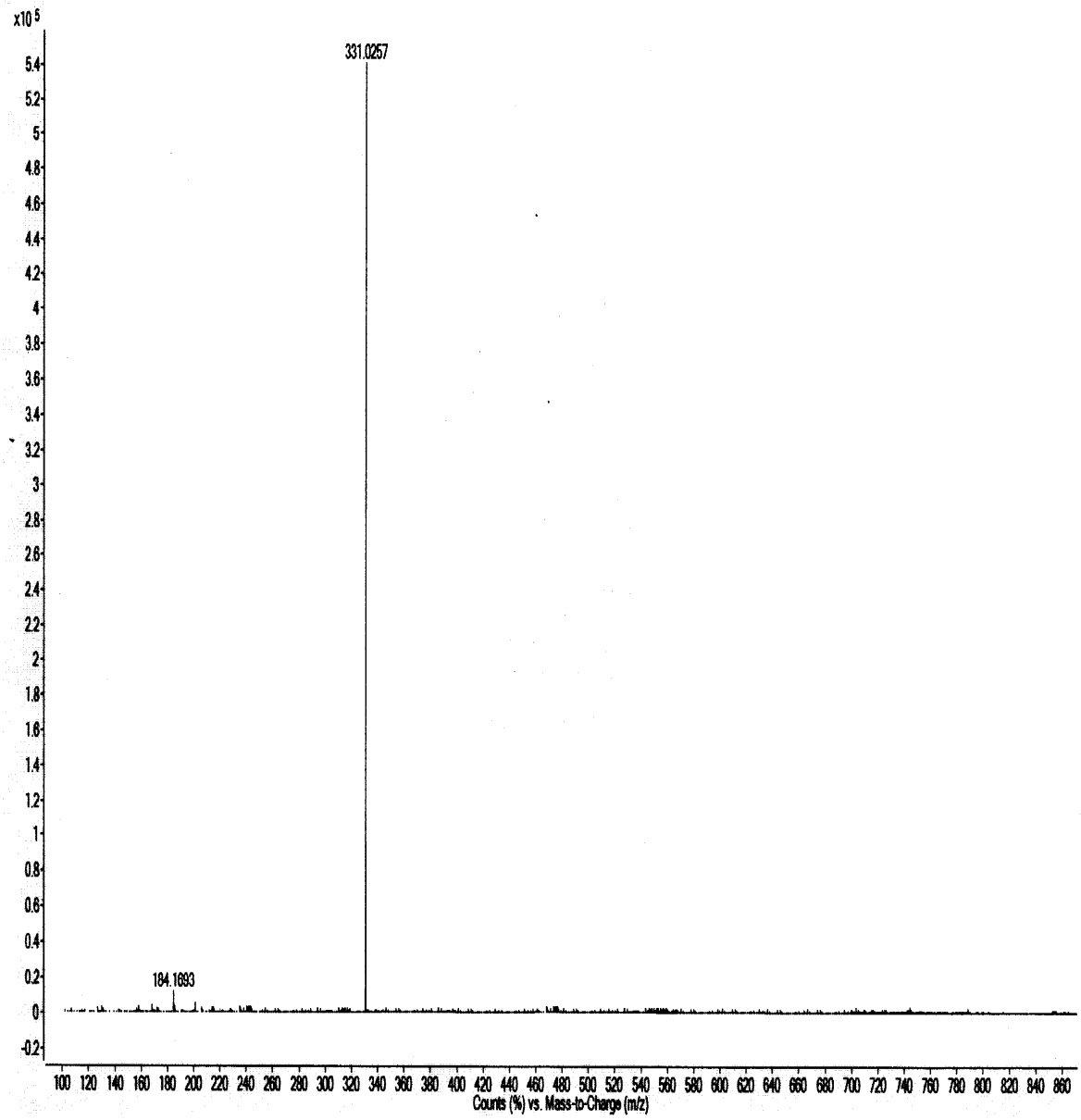



Figure **S13**: Mass spectrum of **9b.**

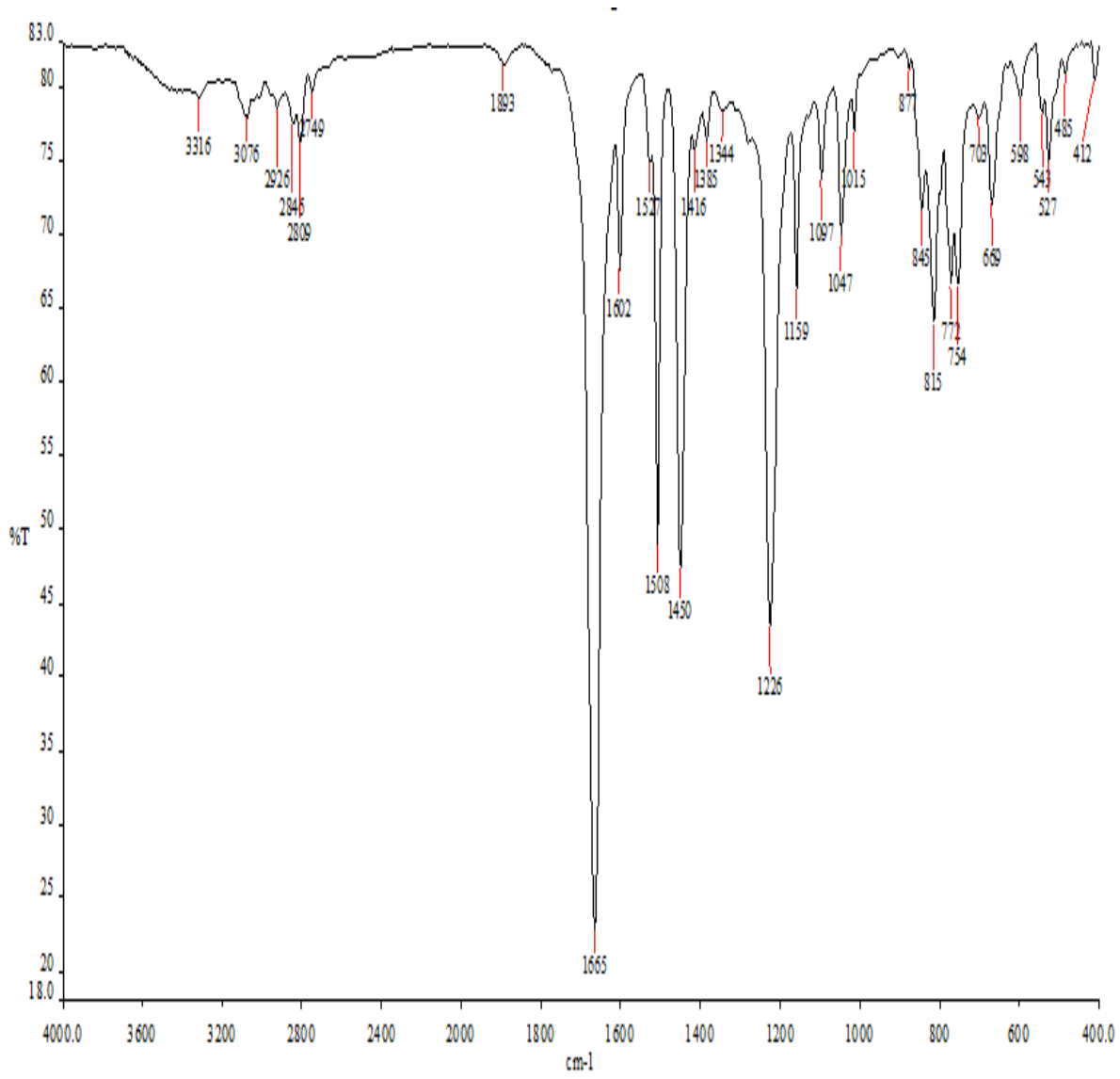



Figure **S14**: IR Spectrum of **9b**.

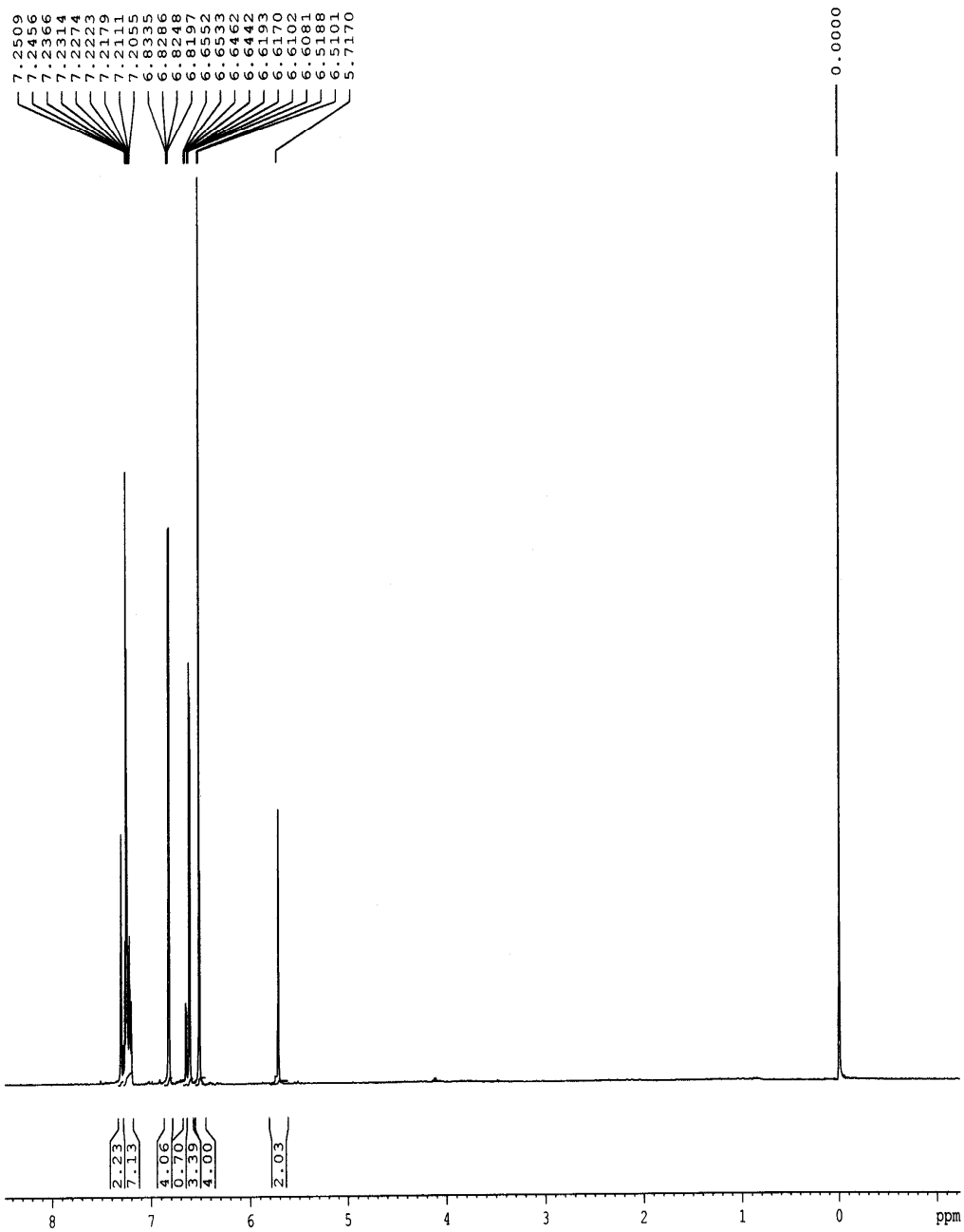



Figure **S15**: $^{1}$H NMR Spectrum of **10a**.

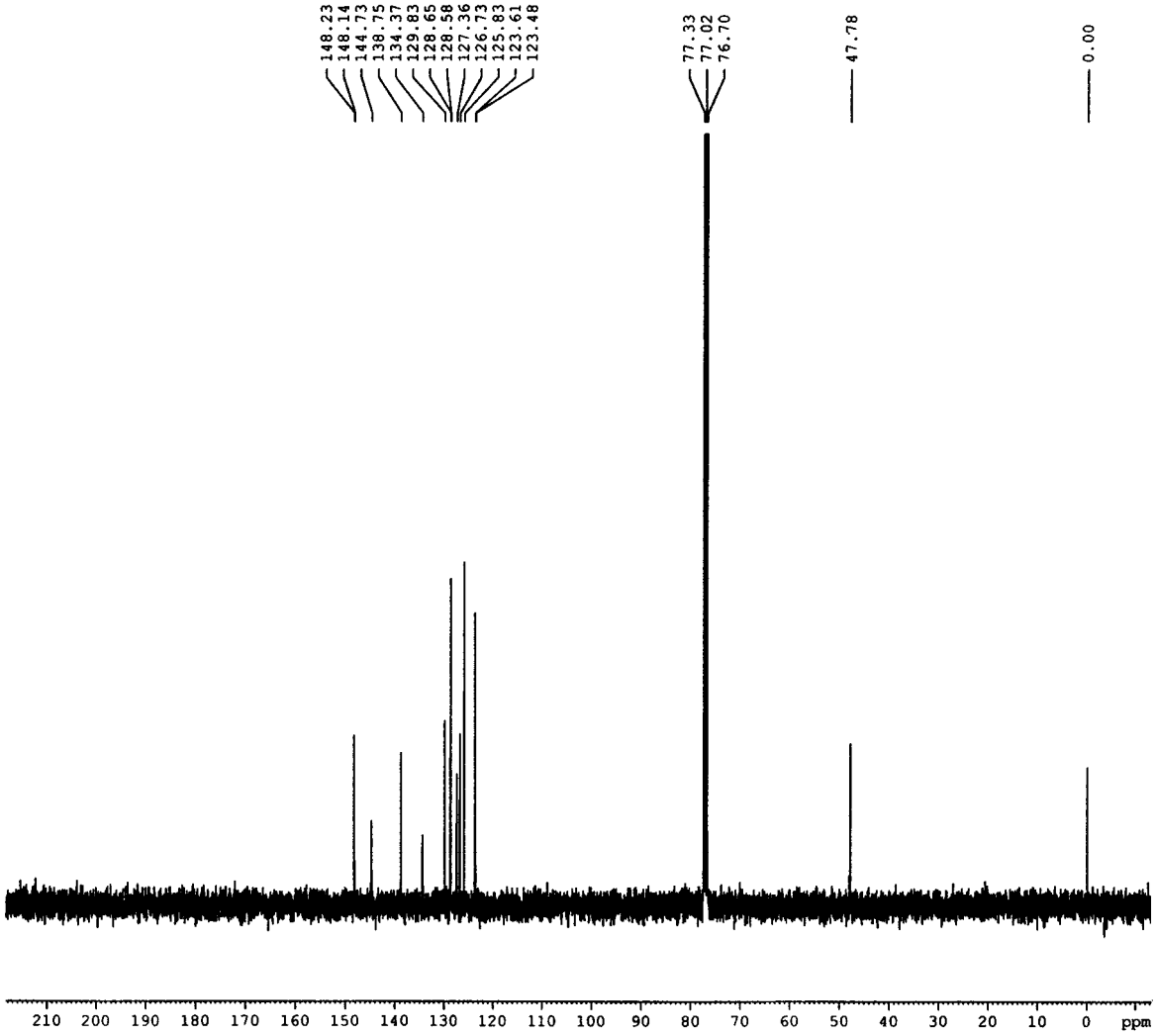



Figure **S16**: $^{13}$C NMR spectrum of **10a**.

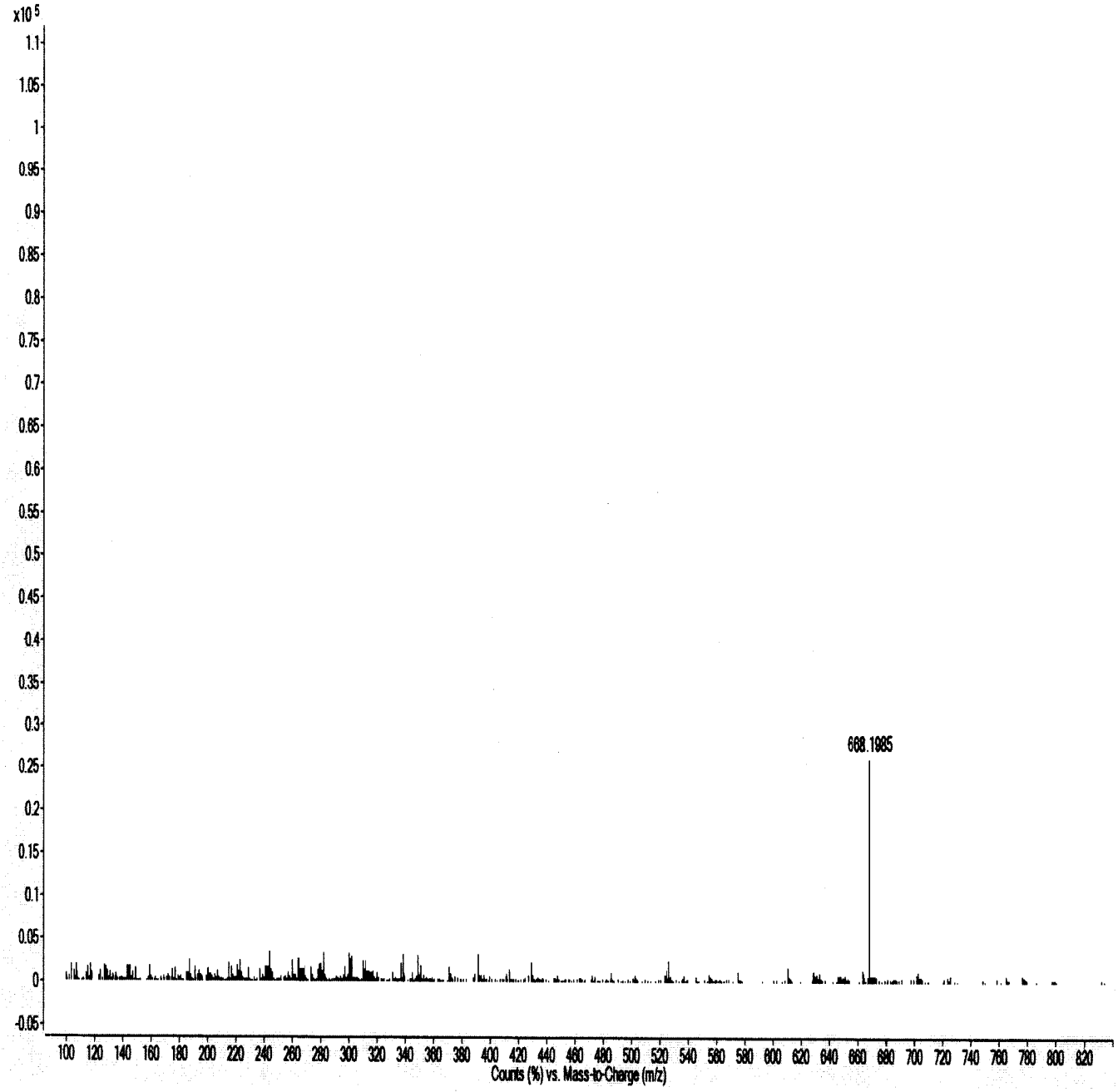



Figure **S17**: Mass spectrum of **10a**.

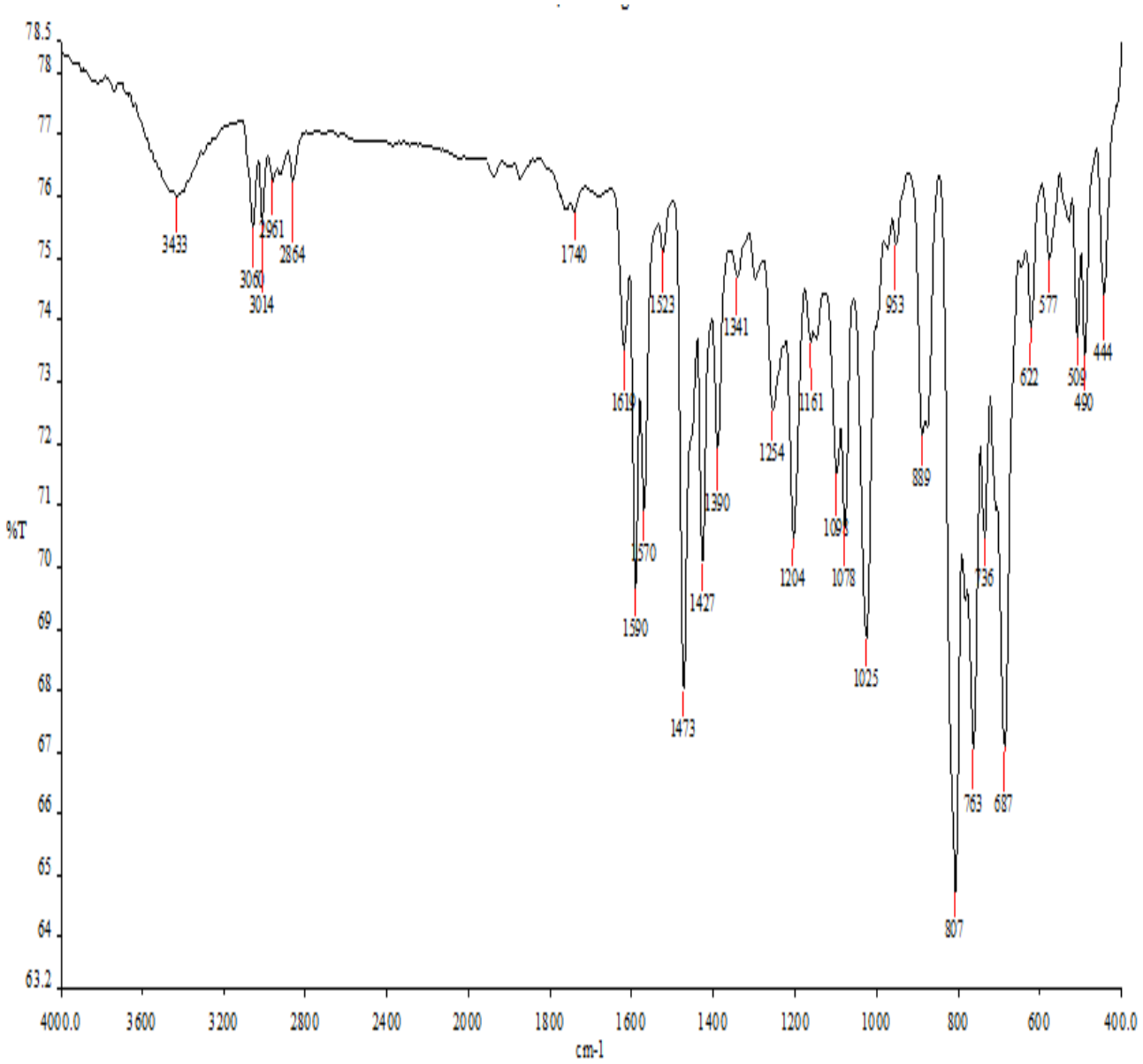



Figure **S18**: IR Spectrum of **10a**.

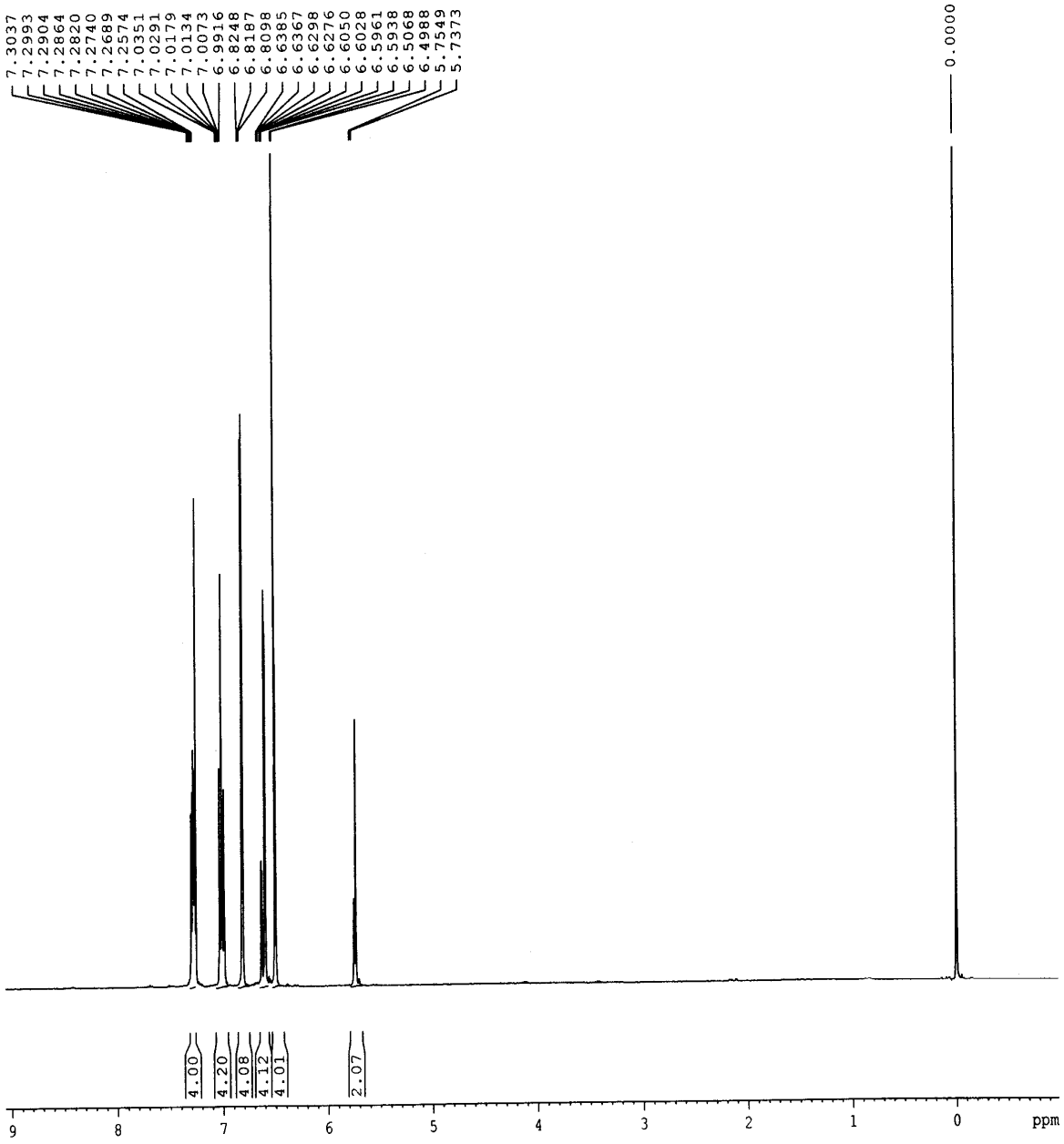



Figure **S19**: $^{1}$H NMR Spectrum of **10b**.

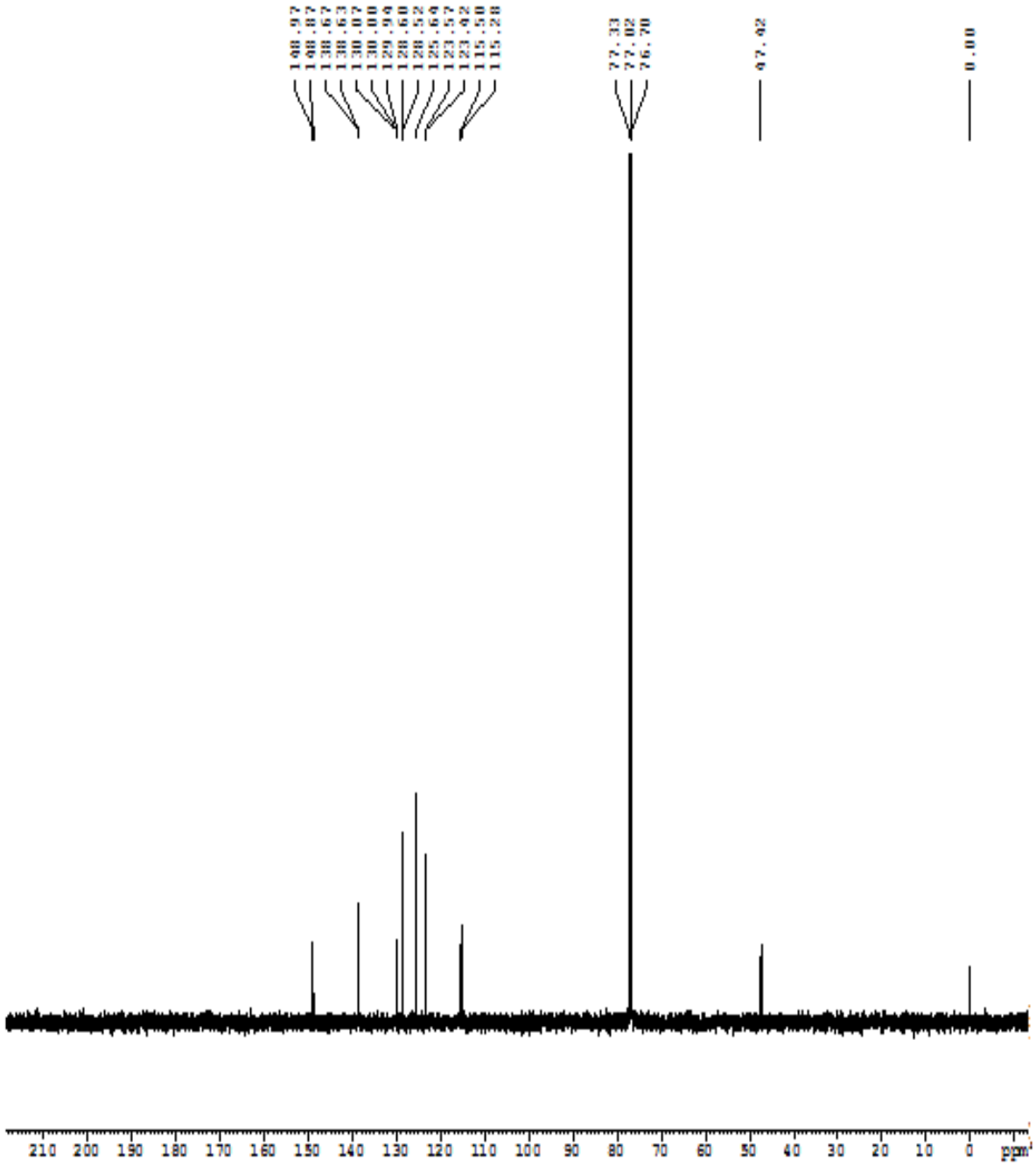



Figure **S20**: $^{13}$C NMR Spectrum of **10b**.

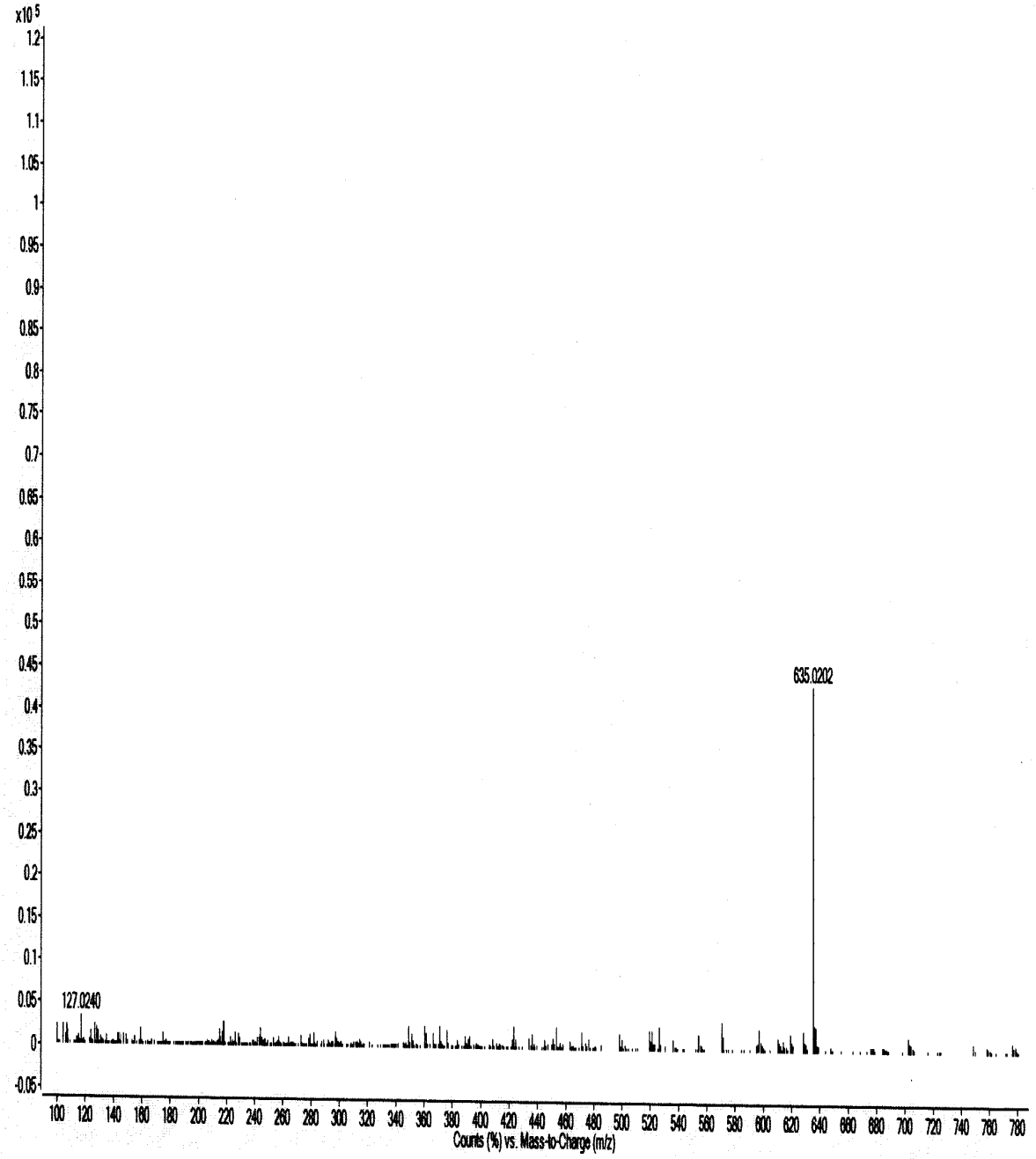



Figure **S21**: Mass spectrum of **10b**.

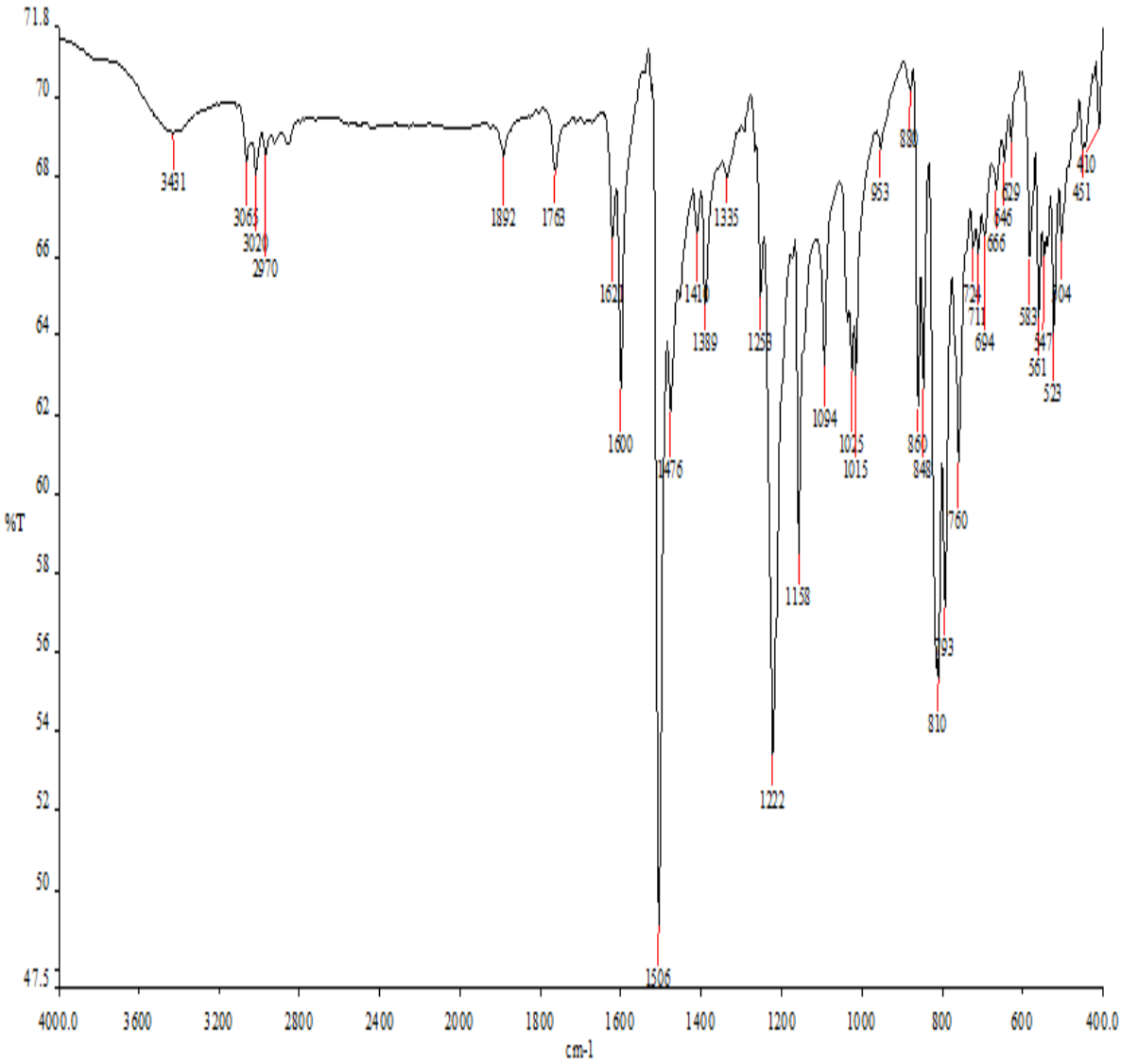



Figure **S22**: IR Spectrum of **10b**.

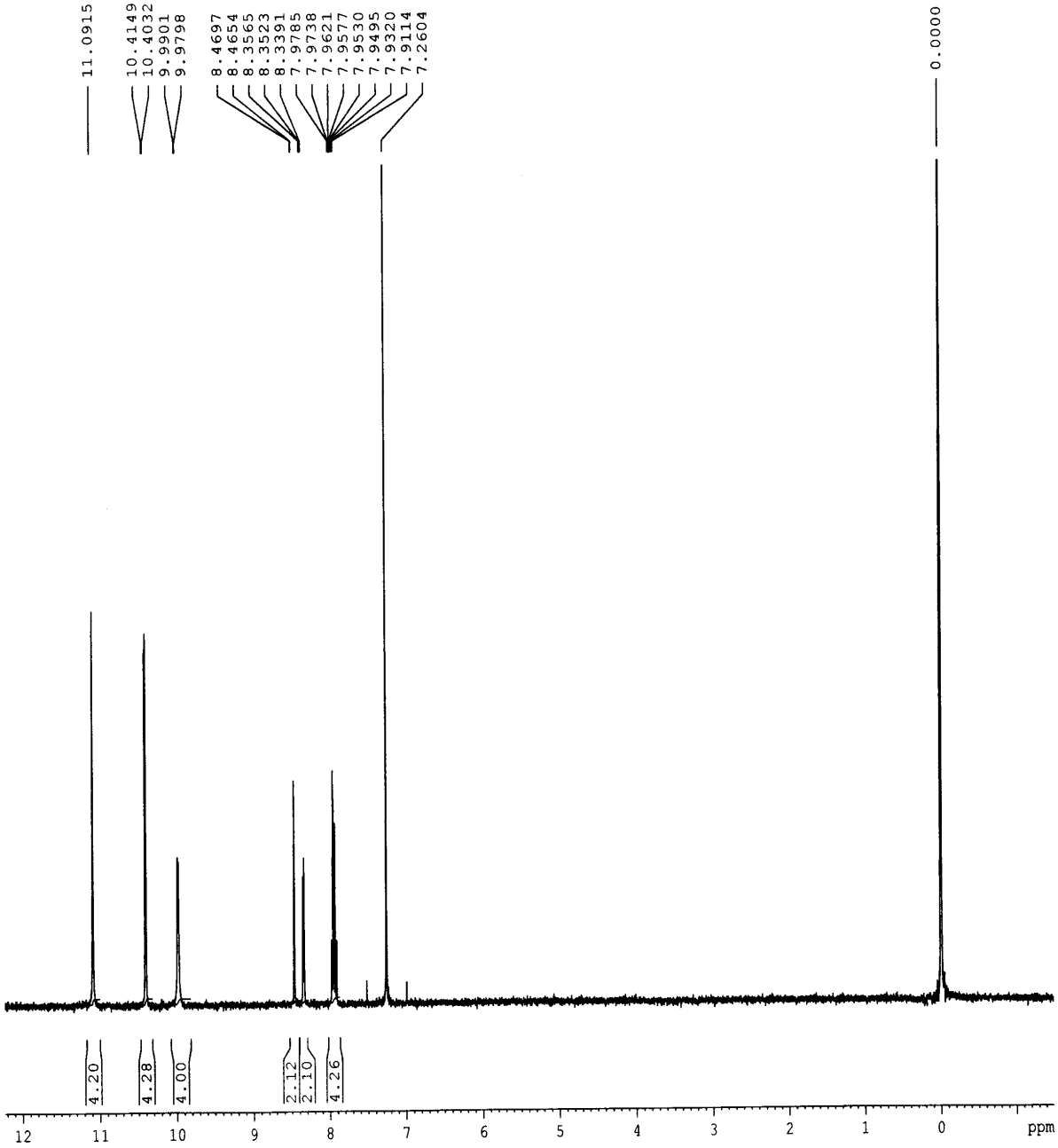



Figure **S23**: $^{1}$H NMR Spectrum of **11a**.

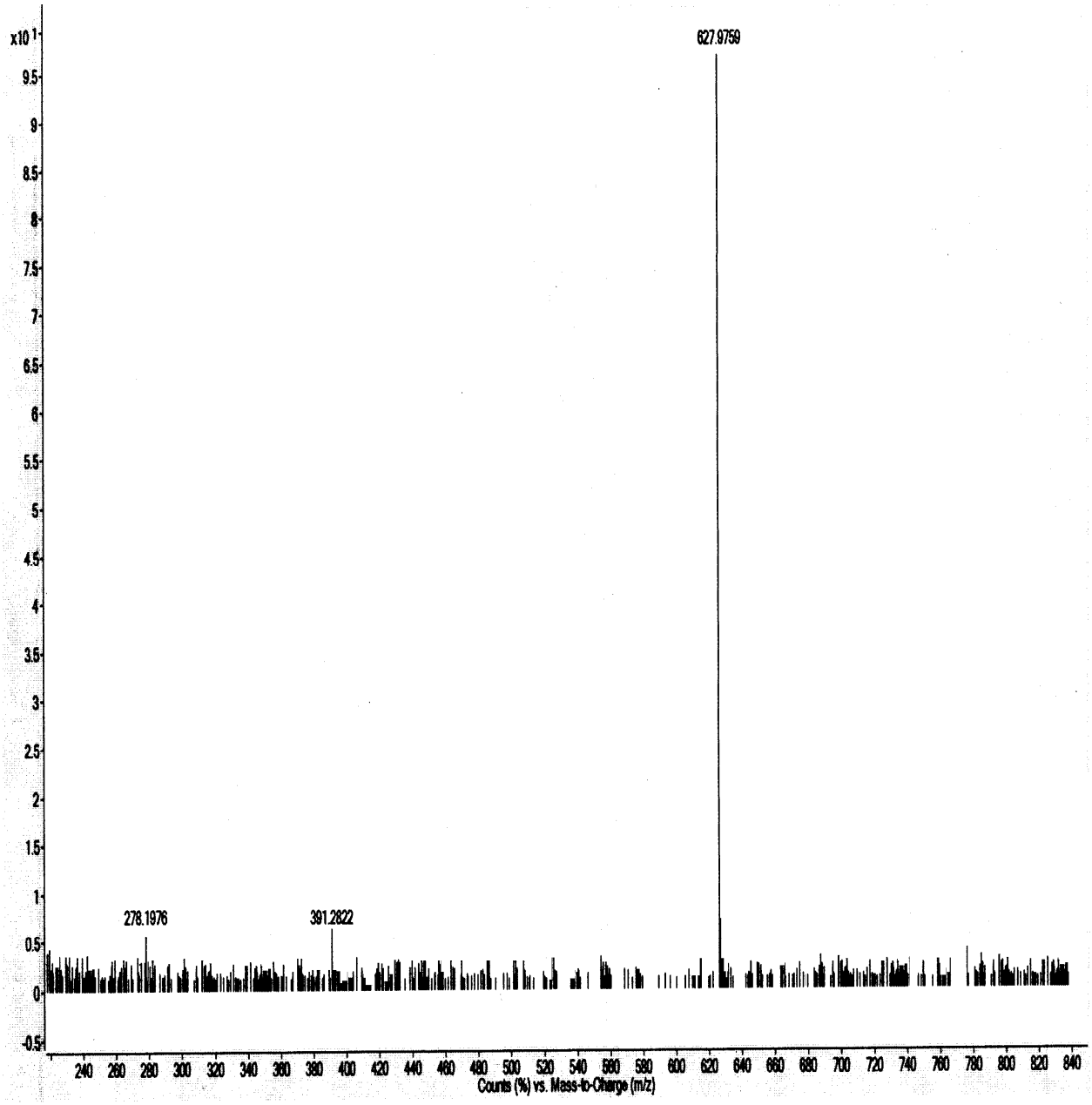



Figure **S24**: Mass spectrum of **11a**.

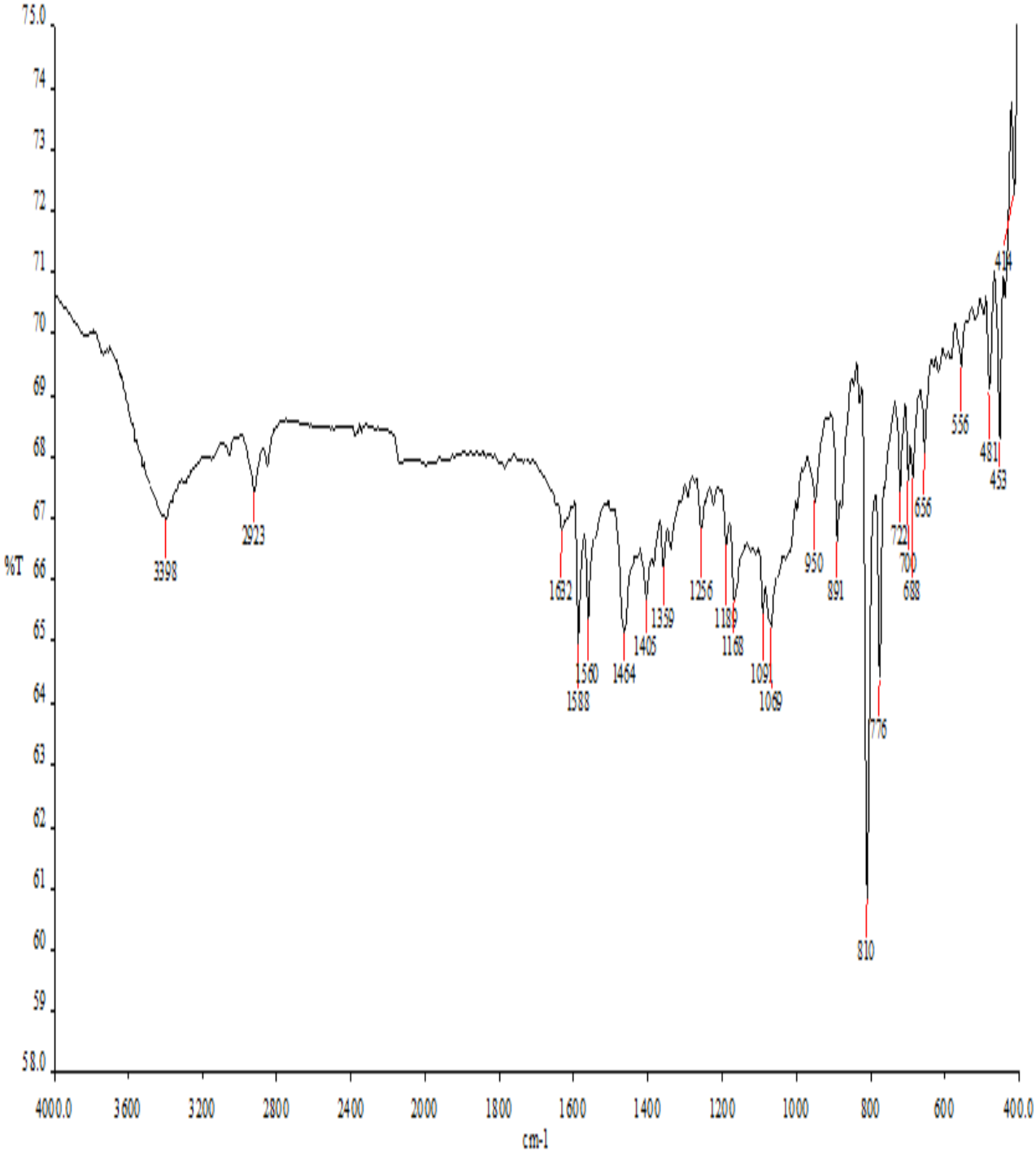



Figure **S25**: IR Spectrum of **11a**.

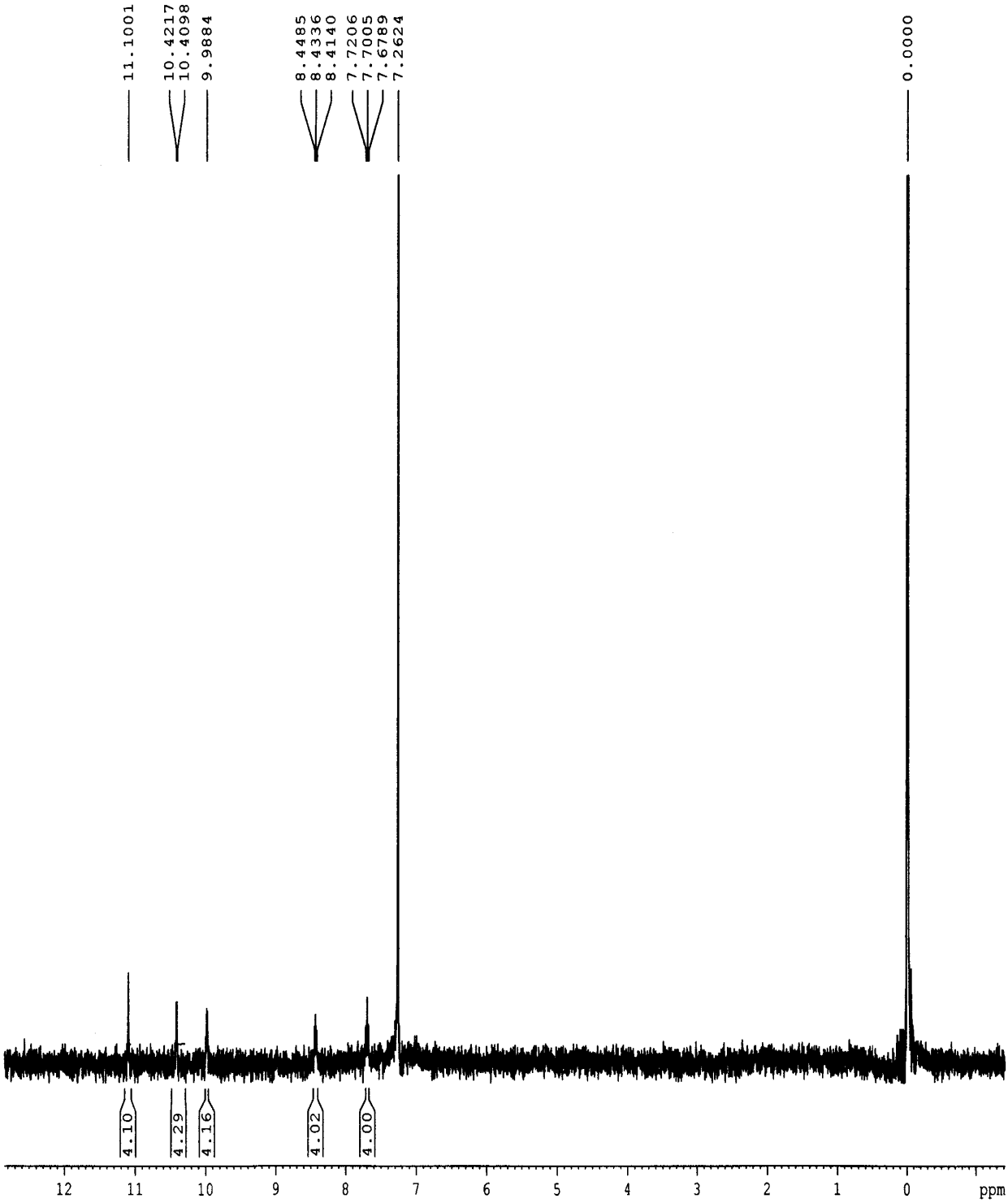



Figure **S26**: 1H NMR Spectrum of **11b**.

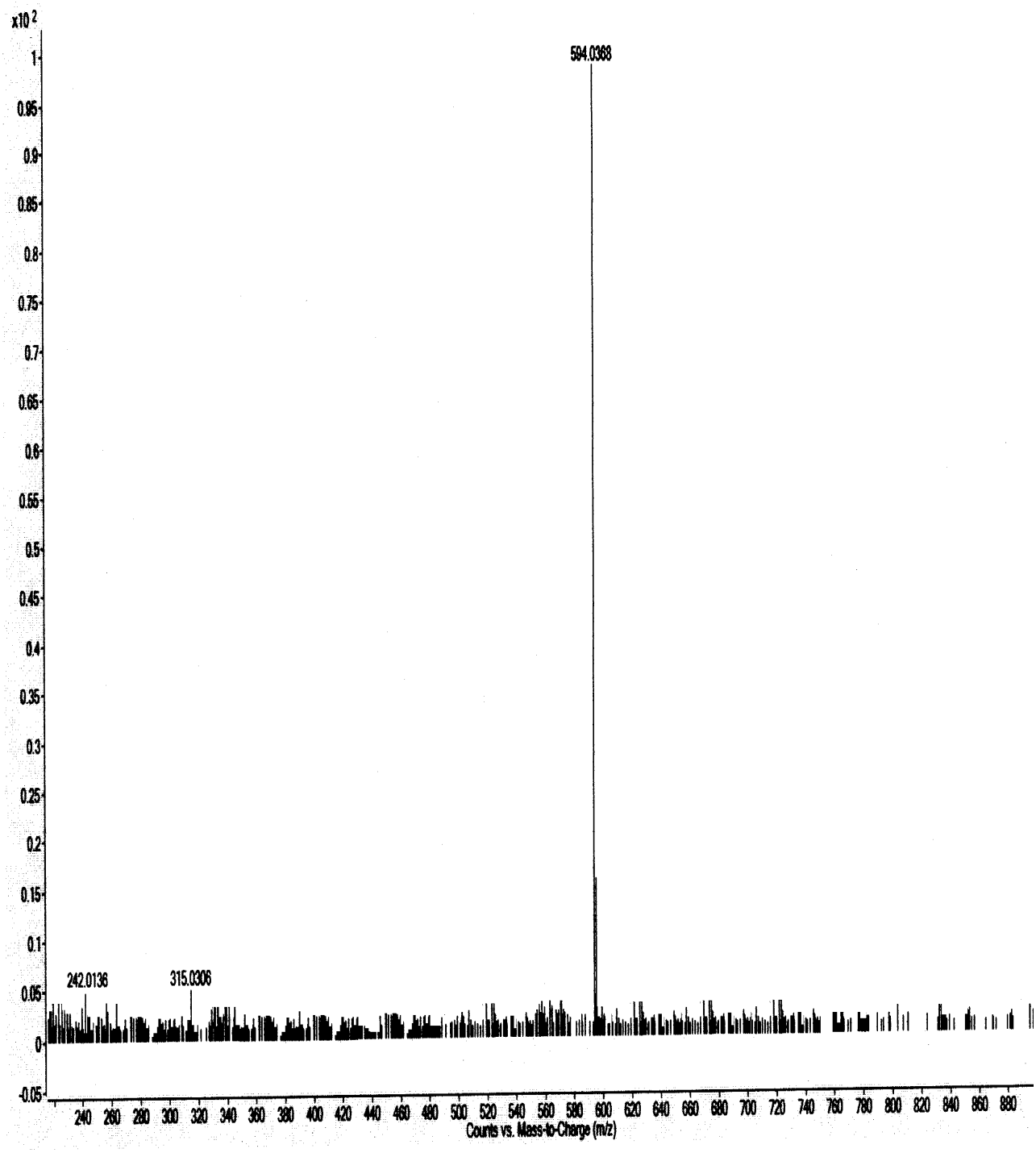



Figure **S27**: Mass spectrum of **11b**.

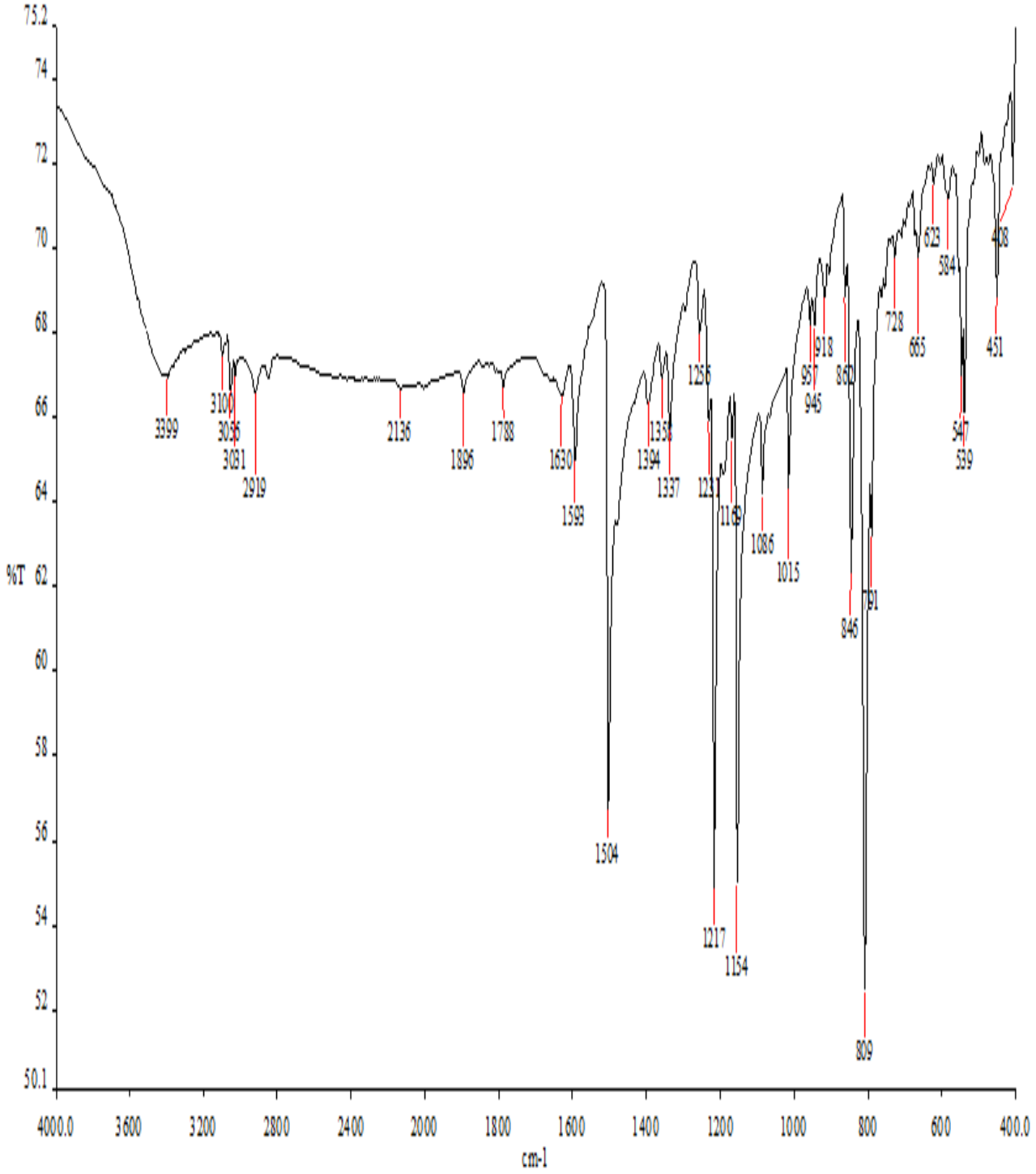



Figure **S28**: IR Spectrum of **11b**.

## Theoretical Calculations

### Computational methods

All the calculations were performed at the density functional theory (DFT) level with the B3LYP functional, the gradient correction of the exchange functional by Becke and the correlation functional by Lee, Yang and Parr. The 6-311G(d) split valence plus polarization basis set was used in Gaussian 09 program.[1],[2] The results were analyzed and visualized on Gauss View 5.0.9.

[1] Gaussian 09, Revision B.01, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, W. H. G. E.; Robb, M. A.;Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.;Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M; Toyota, K.; Fukuda,R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, J. A.; Peralta,Jr., J. E.; Ogliaro, F.;Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Keith, T.; Kobayashi,R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.;Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski,V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, O.; Foresman, J. B.; Ortiz, J.V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, 2010.

[2] Details including the references for the DFT method and basis set can be found online at the homepage of Gaussian, Inc.; http://www.gaussian.com/

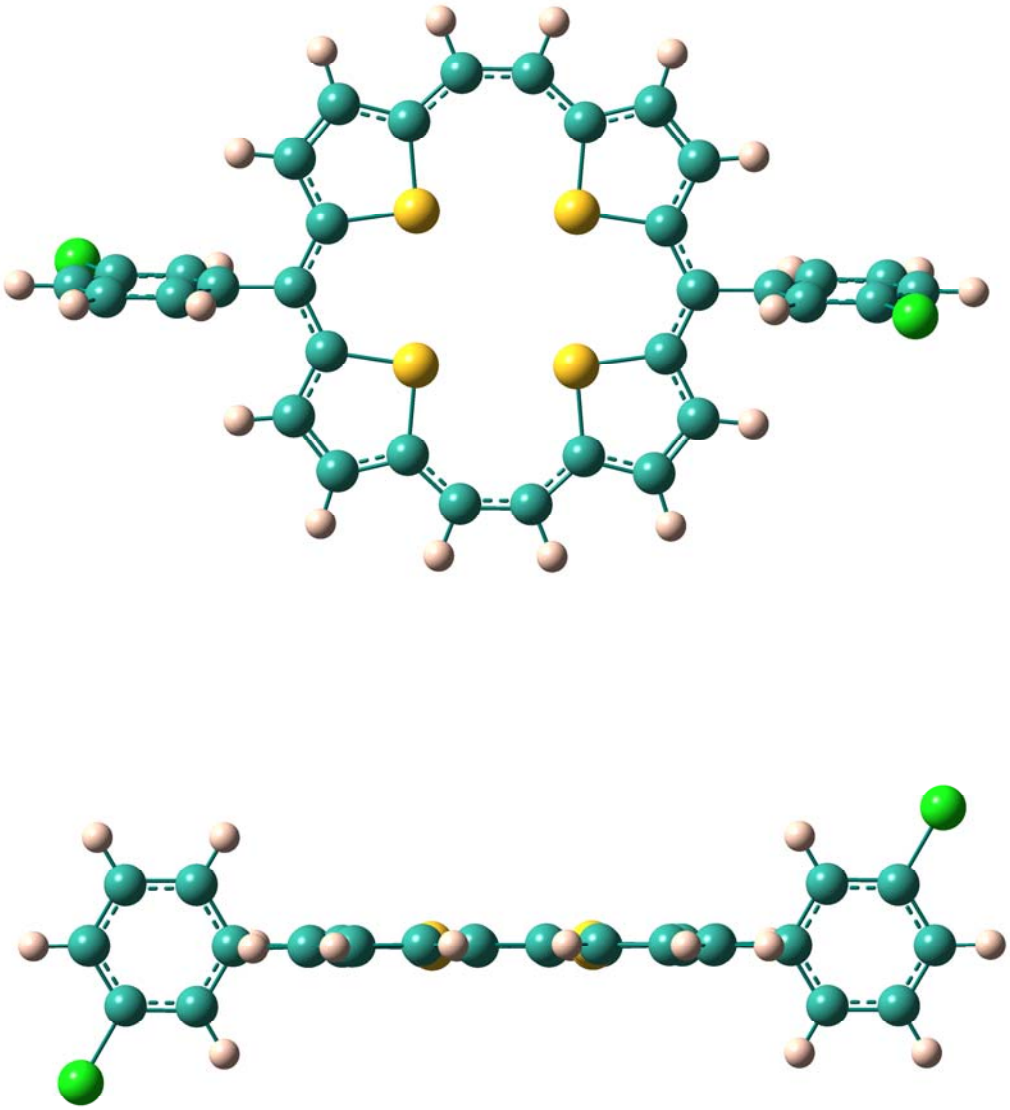
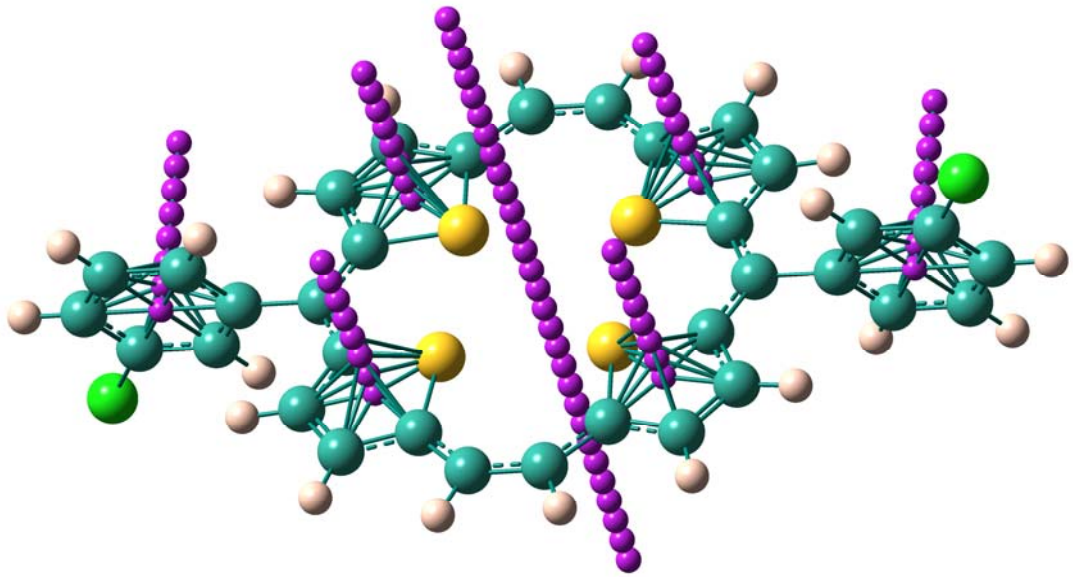


**Figure S29**: Energy minimized structure of **11a** by DFT method at B3LYP/6-311G(d) level using the Gaussion09 program (Top and side views shown above).



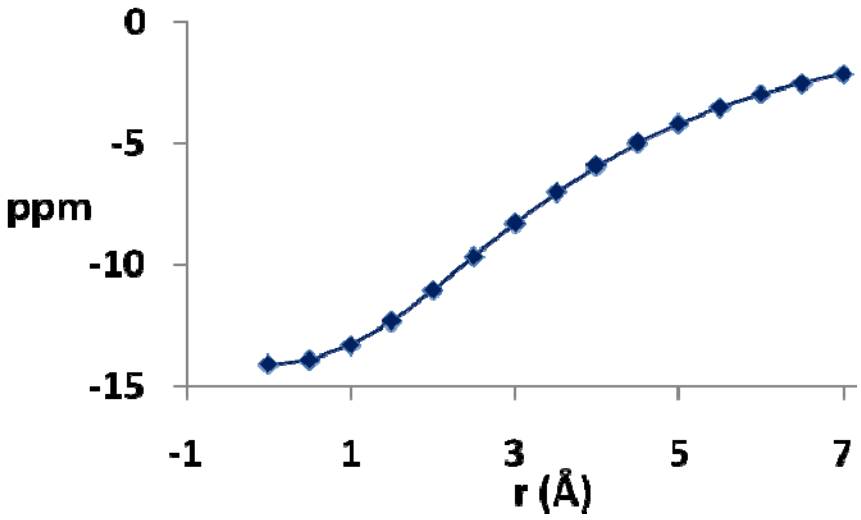
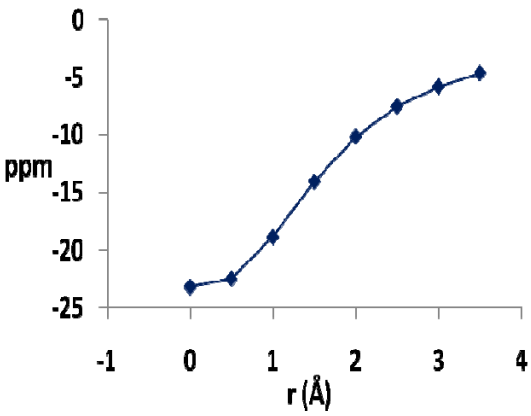
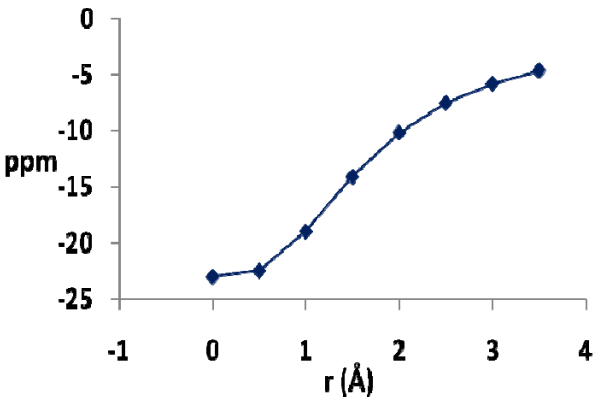


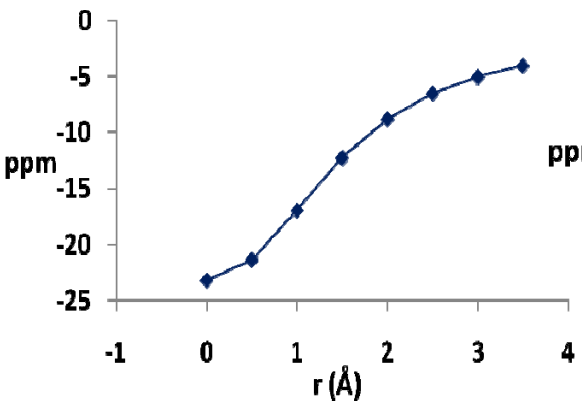

**Figure S30**: The ghost (Bq) atoms (in purple) were placed (0.5 Å interval) in the centre of the molecule. The -ve NICS values clearly indicate the aromaticity of **11a**. NICS is maximum at 0 Å and decreases as the distance of the ghost atom is increased in 0.5 Å intervals.



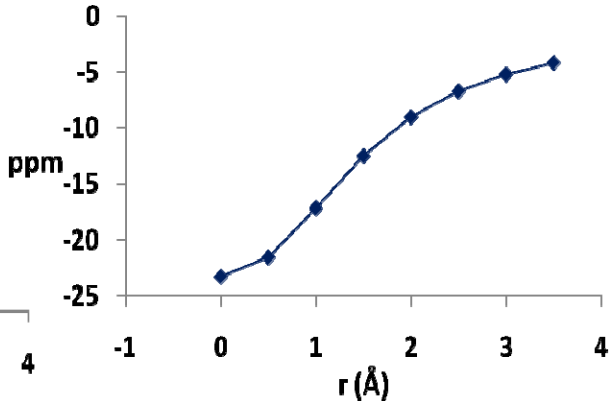

Thiophene ring 1



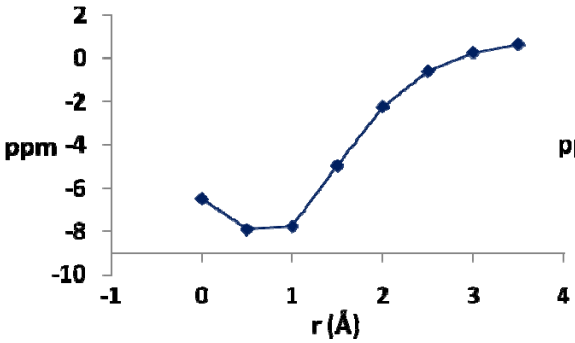

Thiophene ring 2



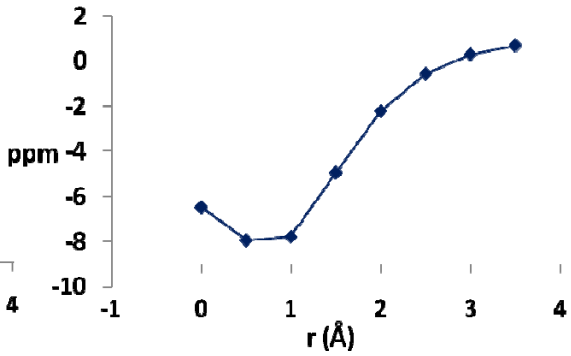

Thiophene ring 3




Thiophene ring 4




Phenyl ring 1




Phenyl ring 2

**Figure S31**: As shown above is the behaviour of the four thiophene rings of **11a**. The NICS are negative and maximum at their centres and decreases as we move farther. Further the NICS at the centre is far higher than the normal thiophene showing their increased aromatic character when they are part of the annulene ring. Phenyl rings are showing the normal behavior like benzene but with decreased NICS values.

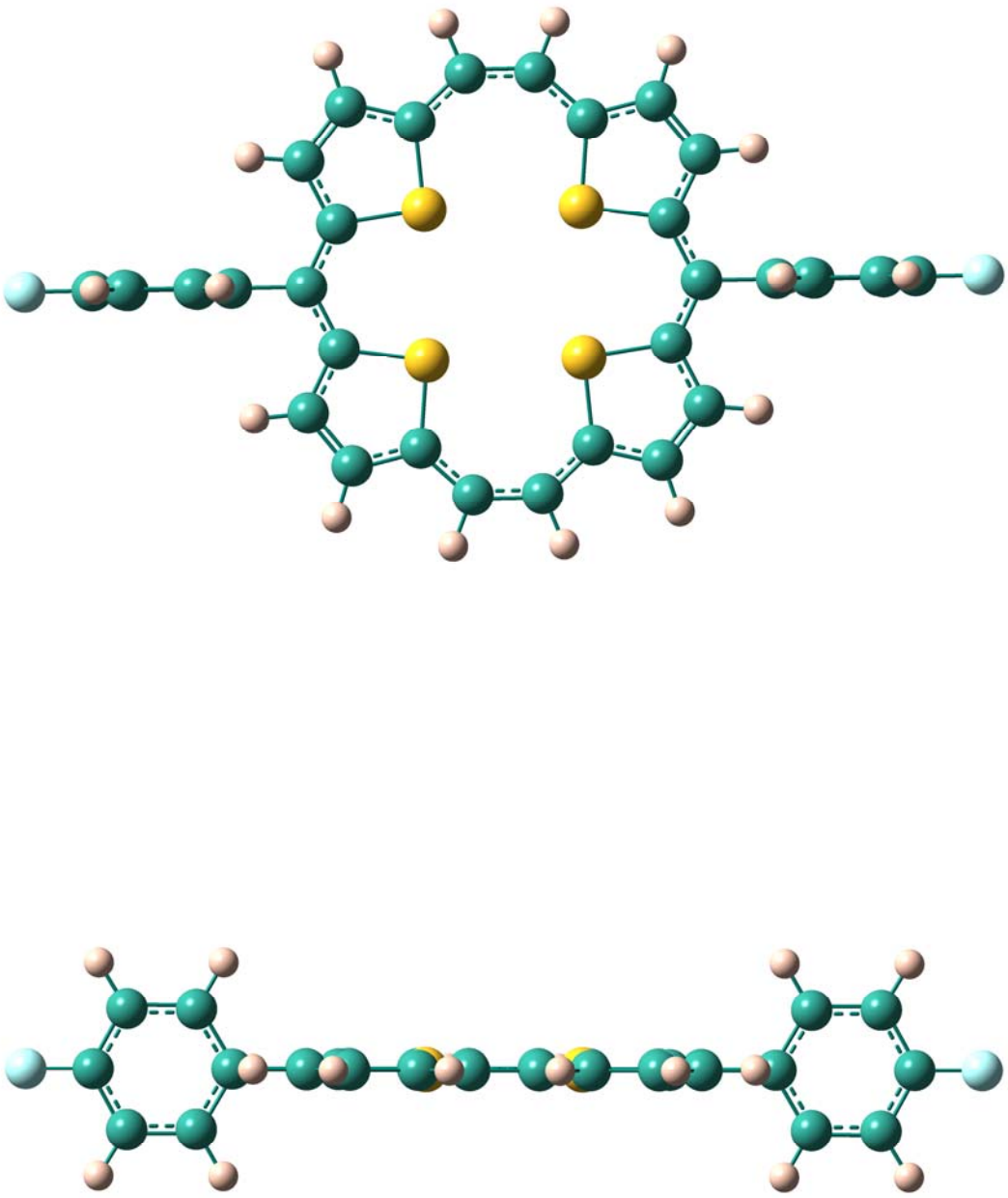
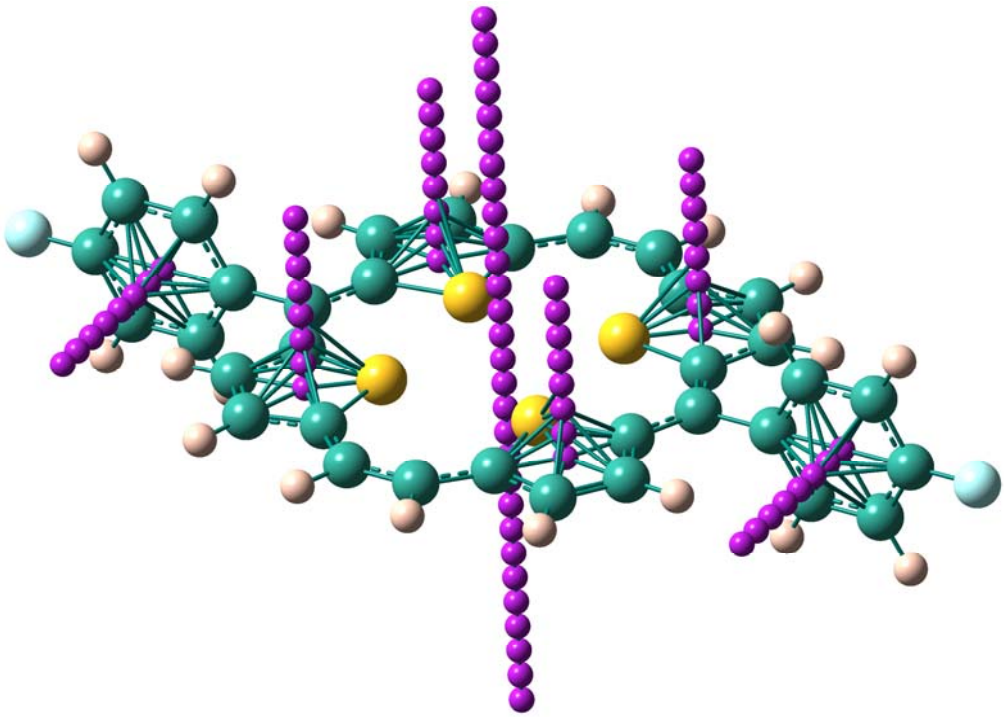


**Figure S32**: Energy minimized structure of **11b** by DFT method at B3LYP/6-311G(d) level using the Gaussion09 program (Top and side views shown above).



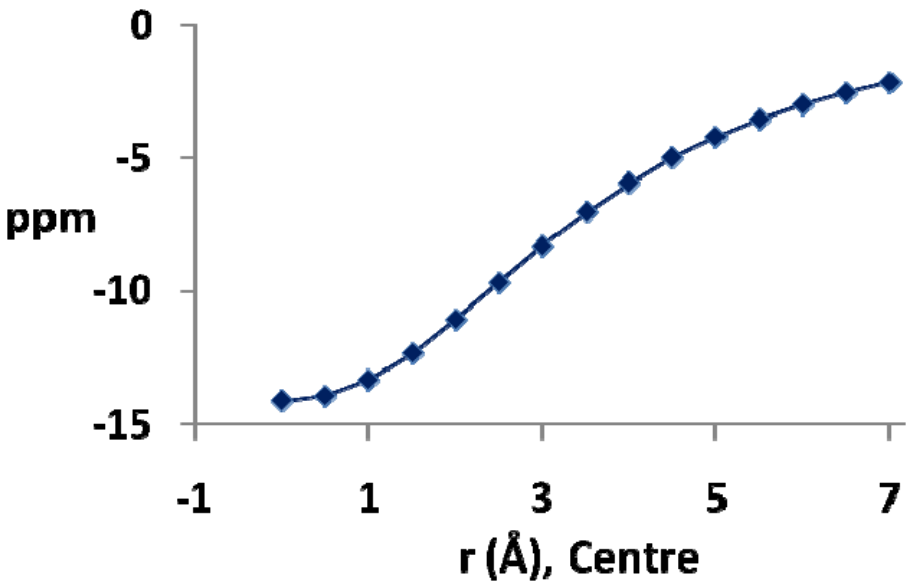
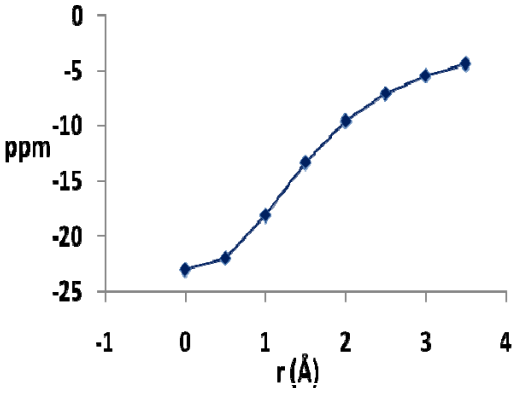
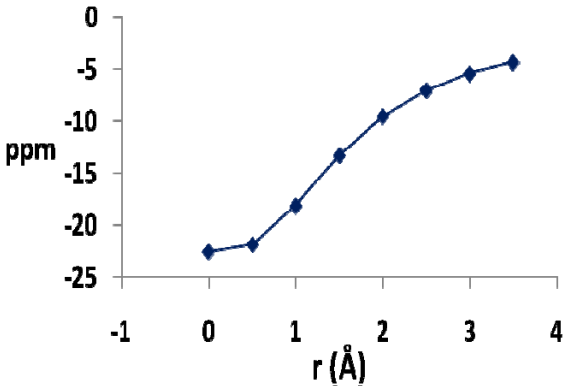


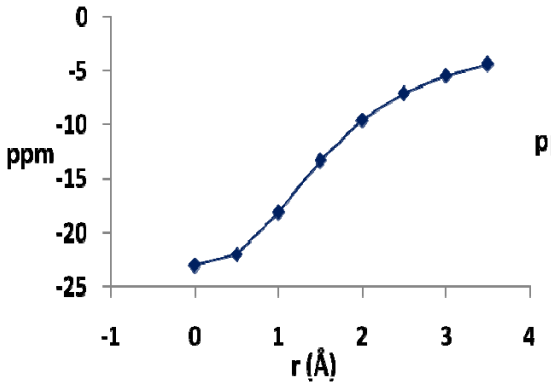

**Figure S33**: The ghost (Bq) atoms were placed (0.5 Å interval) in the centre of the molecule (in purple). The -ve NICS values clearly indicate the aromaticity of **11b**. NICS is maximum at 0 Å and decreases as the distance of the ghost atom is increased in 0.5 Å intervals.



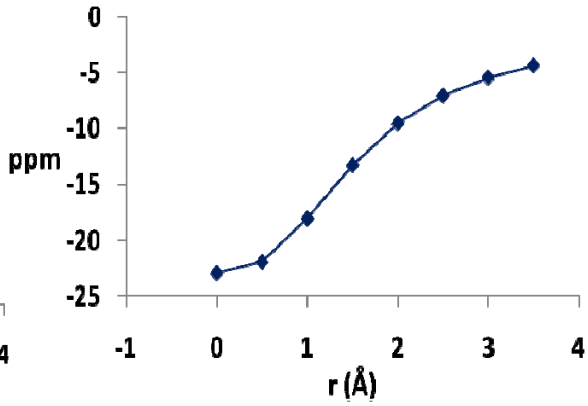

Thiophene ring 1



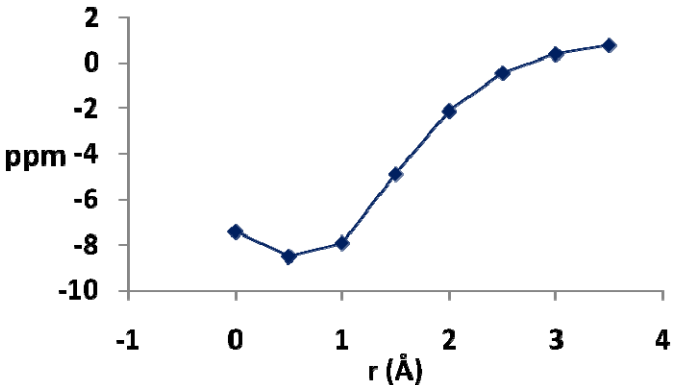

Thiophene ring 2




Thiophene ring 3




Thiophene ring 4




Phenyl ring 1, 2

**Figure S34**: As shown above is the behaviour of the four thiophene rings of **11b**. The NICS are negative and maximum at their centres and decreases as we move farther. Further the NICS at the centre is far higher than the normal thiophene showing their increased aromatic character when they are part of the annulene ring. Phenyl rings are showing the normal behavior like benzene but with decreased NICS values.

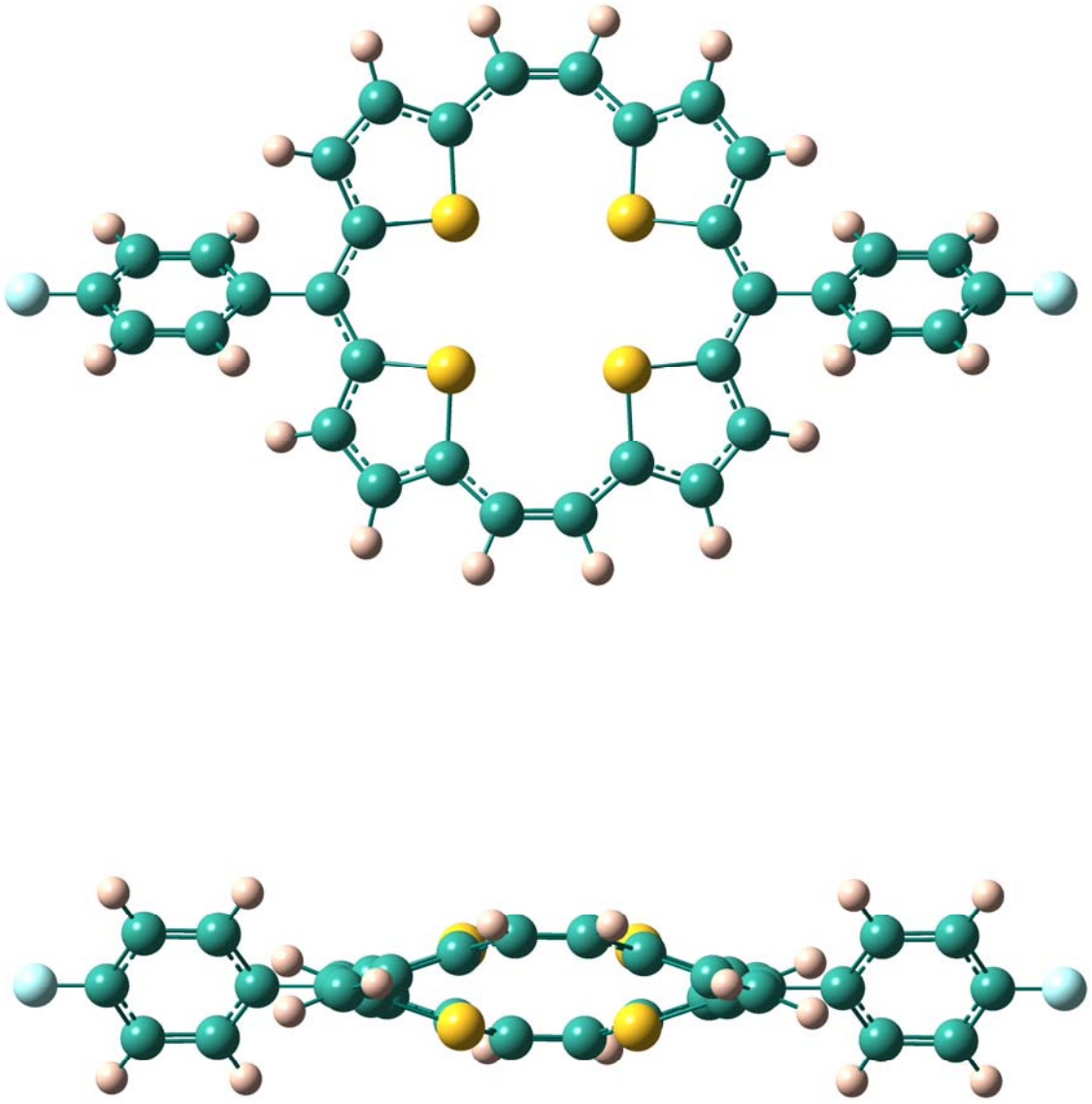
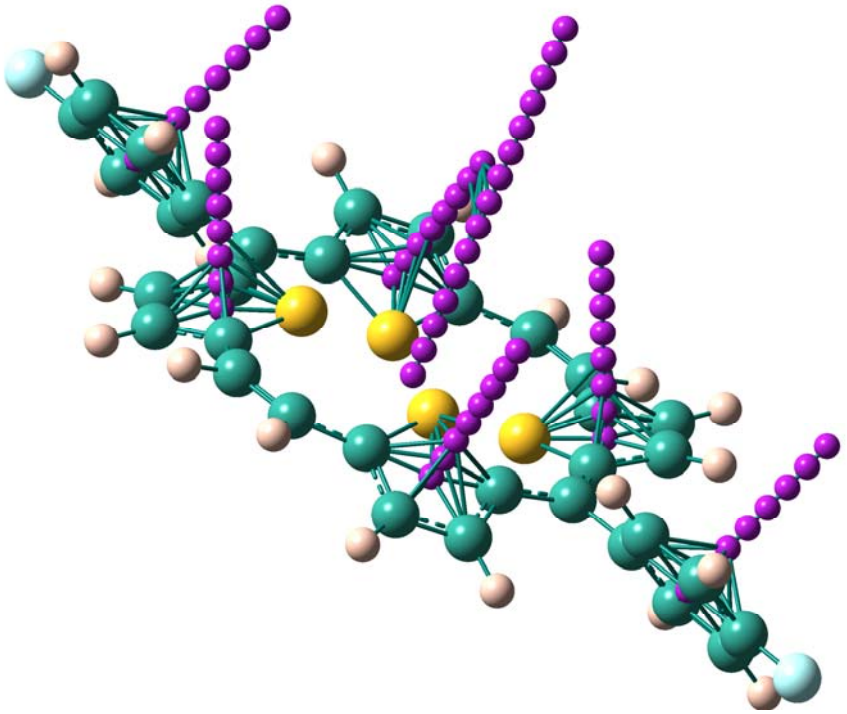


**Figure S35**: Energy minimized structure of antiaromatic dication **12b**. Upper (top view), lower (side view).



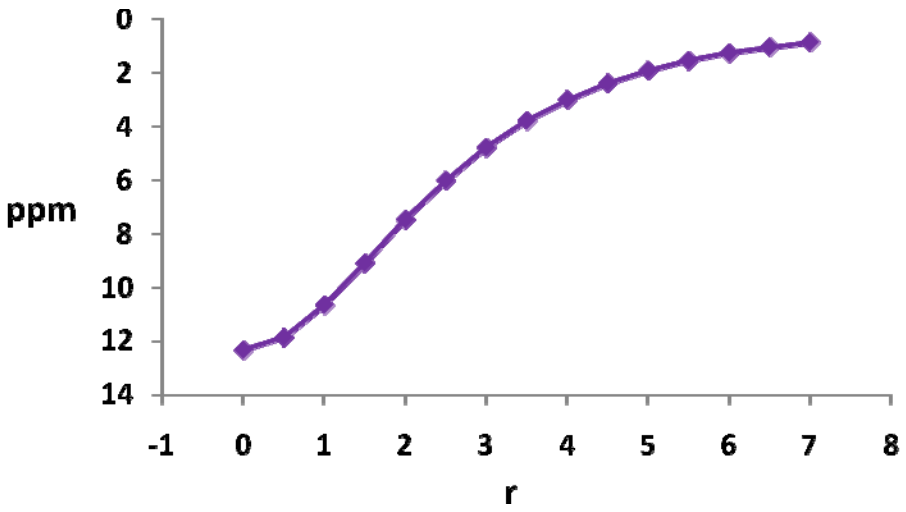
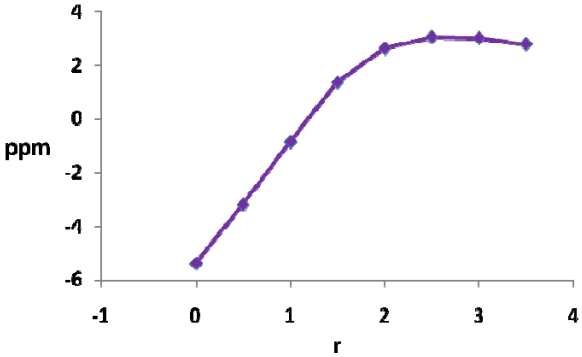
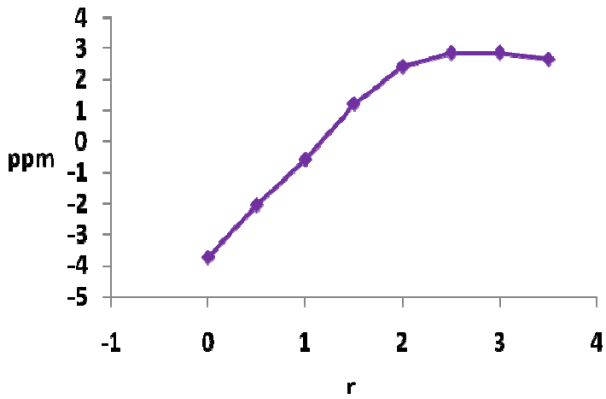


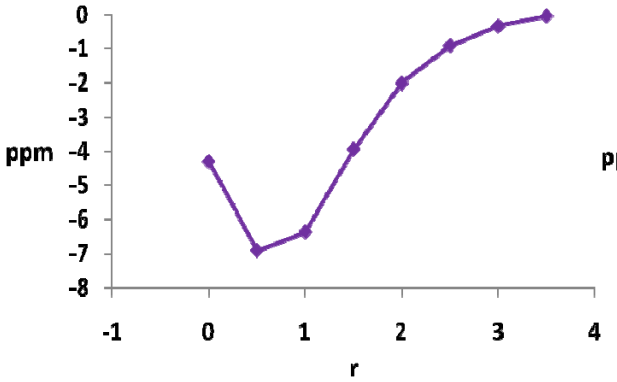

**Figure S36**: Placement of the ghost atoms (at 0.5 Å intervals) at the centre of all the individual rings as well as the main ring of **12b** (anti aromatic). The +ve NICS values (NICS 1 = 10.65) indicates the antiaromatic behaviour of the dication **12b**. The resulting NICS vs. r (distance from centre in Å) graph is also shown for the main centre of the **12b**. The graphs for the four thiophene rings are also shown (Figure S37).



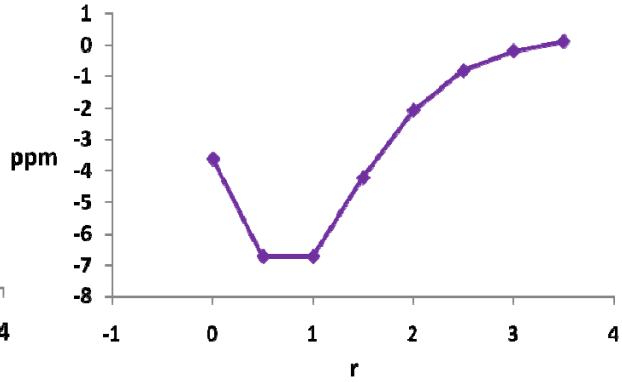

Thiophene ring 1



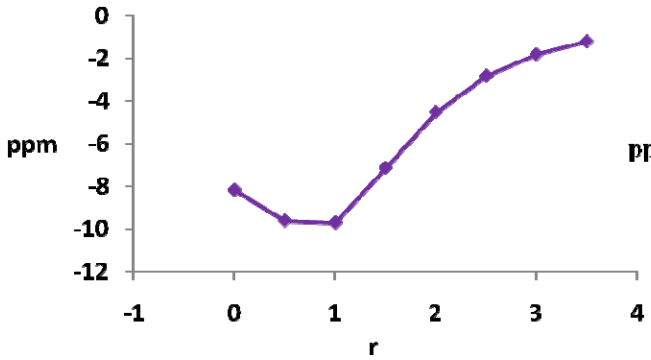

Thiophene ring 2



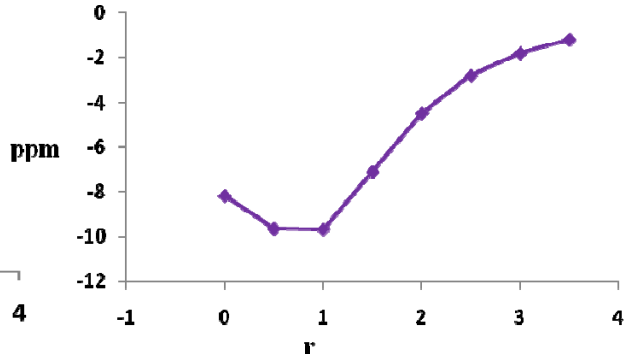

Thiophene ring 3




Thiophene ring 4



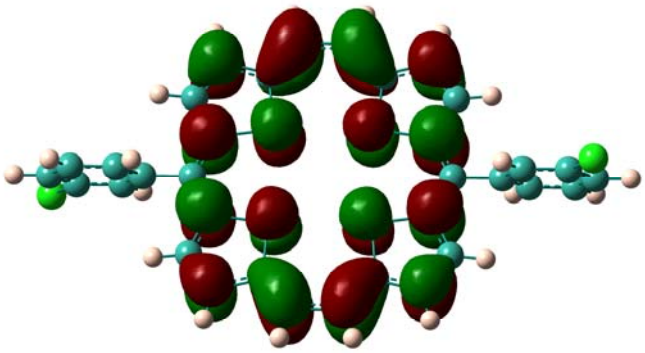

Phenyl ring 1



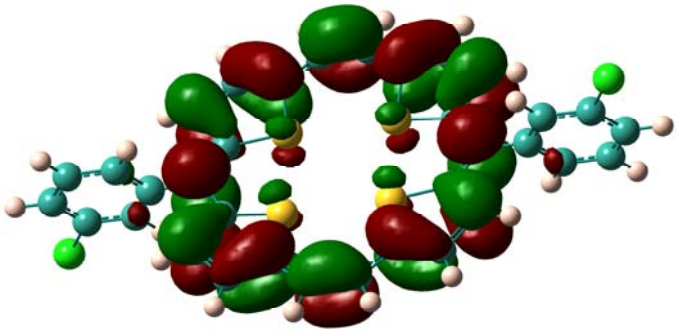

Phenyl ring 2

**Figure S37**: For **12b** As shown at the top, at the centre of a set of two thiophene rings aromaticity (-ve NICS) is lost at about 1 Å distance and antiaromatic ring current is then observed due to the impact of the main annulene ring. The other set of thiophenes maintain their aromaticity as they points outwards to the annulene core as shown in Fig. S36. They also show a dip at 1 Å which is not shown by a free thiophene ring. The *p*-florophenyl rings shows the normal aromaticity.

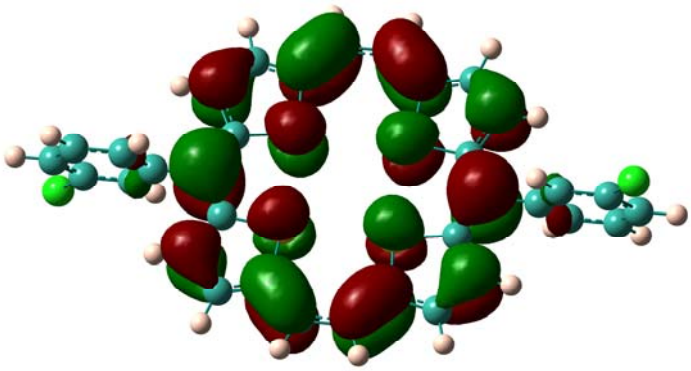
**163 (LUMO+1), -0.08132 a.u.**



**162 (LUMO), -0.10188 a.u.**



**161 (HOMO), -0.18024 a.u.**



**160 (HOMO-1), -0.19319 a.u.**

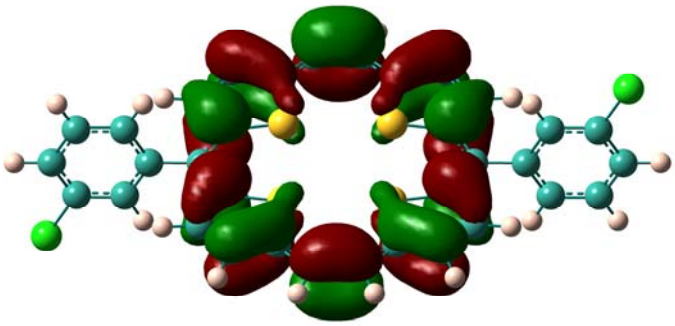
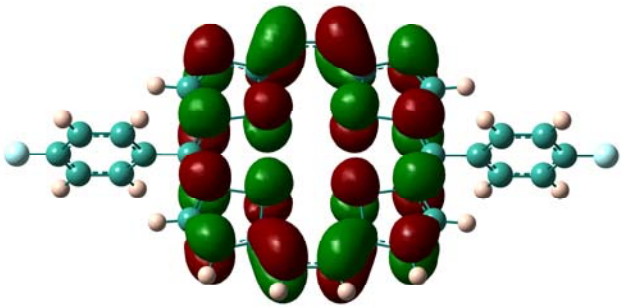
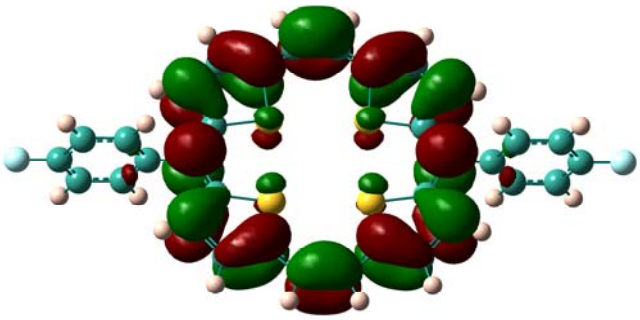


**Figure S38**: HOMO-LUMO and their neighbouring energy levels for **11a** are shown along with their energies (left) in a.u. HOMO - LUMO levels are showing high degree of delocalisation on the 22Π annulene ring periphery. The energy of HOMO = -0.18024 a.u. and that of LUMO = -0.10188 a.u.

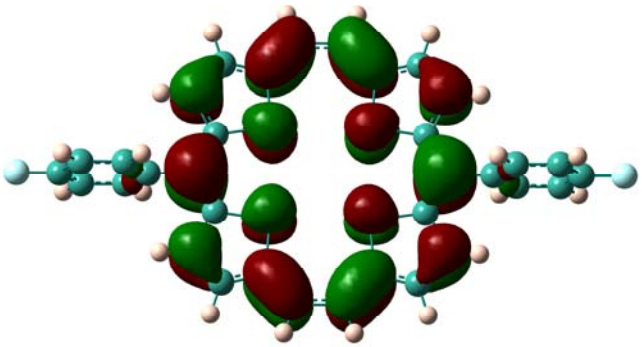
**155 (LUMO+1), -0.07982 a.u.**



**154 (LUMO), -0.10007 a.u.**



**153 (HOMO), -0.17860 a.u.**



**152 (HOMO-1), -0.19168 a.u.**

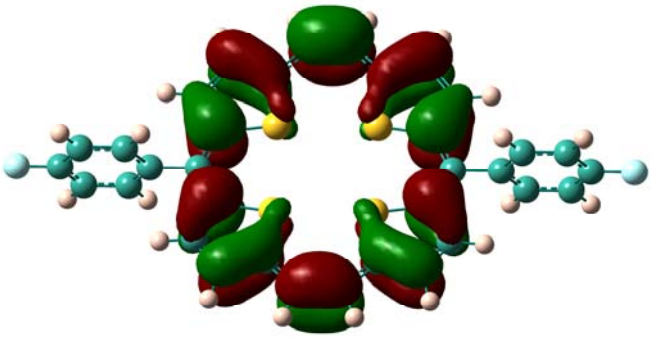


**Figure S39**: HOMO-LUMO and their neighbouring energy levels for **11b** are shown along with their energies (left) in a.u. HOMO - LUMO levels are showing high degree of delocalisation on the 22Π annulene ring periphery. The energy of HOMO = -0.17860 a.u. and that of LUMO = -0.10007 a.u..

**Cartesian coordinates**

**Table S1**: Cartesian coordinates of **11a**

SCF Done: E (RB3LYP) = -3821.16360453 a.u..

| Center Number | Atomic Number | Atomic Type | Coordinates (Angstroms) X | Y | Z |
|---|---|---|---|---|---|
| 1 | 16 | 0 | 1.531384 | 1.550566 | -0.136219 |
| 2 | 6 | 0 | 0.616009 | 4.241159 | 0.058379 |
| 3 | 6 | 0 | 1.655553 | 3.304537 | -0.070987 |
| 4 | 6 | 0 | 3.019430 | 3.663395 | -0.166820 |
| 5 | 1 | 0 | 3.331399 | 4.701394 | -0.145682 |
| 6 | 6 | 0 | 3.894801 | 2.605820 | -0.278562 |
| 7 | 1 | 0 | 4.967675 | 2.721984 | -0.352155 |
| 8 | 6 | 0 | 3.278349 | 1.330467 | -0.282769 |
| 9 | 6 | 0 | 3.922479 | 0.082407 | -0.377136 |
| 10 | 6 | 0 | 5.417280 | 0.114888 | -0.530463 |
| 11 | 6 | 0 | 6.001194 | 0.338373 | -1.781666 |
| 12 | 1 | 0 | 5.364074 | 0.488042 | -2.646428 |
| 13 | 6 | 0 | 7.387040 | 0.365558 | -1.917886 |
| 14 | 1 | 0 | 7.832552 | 0.537041 | -2.892251 |
| 15 | 6 | 0 | 8.211885 | 0.171465 | -0.812425 |
| 16 | 1 | 0 | 1.009752 | 5.255799 | 0.060949 |
| 17 | 16 | 0 | 1.593335 | -1.485276 | -0.161228 |
| 18 | 6 | 0 | 0.770346 | -4.212211 | -0.189886 |
| 19 | 6 | 0 | 1.775879 | -3.232787 | -0.254923 |
| 20 | 6 | 0 | 3.146930 | -3.534363 | -0.419886 |
| 21 | 1 | 0 | 3.492342 | -4.558717 | -0.500783 |
| 22 | 6 | 0 | 3.982748 | -2.440916 | -0.474073 |
| 23 | 1 | 0 | 5.054065 | -2.512847 | -0.604389 |
| 24 | 6 | 0 | 3.325642 | -1.192153 | -0.349837 |
| 25 | 6 | 0 | 6.241901 | -0.079660 | 0.582697 |
| 26 | 1 | 0 | 5.806569 | -0.250874 | 1.559851 |
| 27 | 6 | 0 | 7.623798 | -0.049276 | 0.428309 |
| 28 | 1 | 0 | 1.198289 | -5.209666 | -0.270588 |
| 29 | 16 | 0 | -1.531384 | -1.550563 | 0.136208 |
| 30 | 6 | 0 | -0.616009 | -4.241156 | -0.058386 |
| 31 | 6 | 0 | -1.655553 | -3.304534 | 0.070984 |
| 32 | 6 | 0 | -3.019429 | -3.663392 | 0.166826 |
| 33 | 1 | 0 | -3.331398 | -4.701391 | 0.145693 |
| 34 | 6 | 0 | -3.894799 | -2.605816 | 0.278569 |
| 35 | 1 | 0 | -4.967673 | -2.721981 | 0.352170 |
| 36 | 6 | 0 | -3.278348 | -1.330464 | 0.282768 |
| 37 | 6 | 0 | -3.922478 | -0.082405 | 0.377133 |
| 38 | 6 | 0 | -5.417278 | -0.114886 | 0.530466 |
| 39 | 6 | 0 | -6.001185 | -0.338356 | 1.781675 |
| 40 | 1 | 0 | -5.364062 | -0.488017 | 2.646436 |
| 41 | 6 | 0 | -7.387030 | -0.365540 | 1.917902 |

| | | | | | |
|---|---|---|---|---|---|
| 42 | 1 | 0 | -7.832538 | -0.537012 | 2.892271 |
| 43 | 6 | 0 | -8.211881 | -0.171459 | 0.812443 |
| 44 | 1 | 0 | -1.009752 | -5.255797 | -0.060953 |
| 45 | 16 | 0 | -1.593334 | 1.485278 | 0.161222 |
| 46 | 6 | 0 | -0.770347 | 4.212213 | 0.189875 |
| 47 | 6 | 0 | -1.775879 | 3.232789 | 0.254911 |
| 48 | 6 | 0 | -3.146931 | 3.534365 | 0.419869 |
| 49 | 1 | 0 | -3.492344 | 4.558719 | 0.500760 |
| 50 | 6 | 0 | -3.982748 | 2.440918 | 0.474058 |
| 51 | 1 | 0 | -5.054066 | 2.512849 | 0.604371 |
| 52 | 6 | 0 | -3.325642 | 1.192155 | 0.349828 |
| 53 | 6 | 0 | -6.241904 | 0.079649 | -0.582691 |
| 54 | 1 | 0 | -5.806577 | 0.250852 | -1.559850 |
| 55 | 6 | 0 | -7.623801 | 0.049267 | -0.428296 |
| 56 | 1 | 0 | -1.198290 | 5.209668 | 0.270573 |
| 57 | 1 | 0 | 9.290710 | 0.190275 | -0.908948 |
| 58 | 1 | 0 | -9.290705 | -0.190267 | 0.908971 |
| 59 | 17 | 0 | 8.650456 | -0.294641 | 1.837998 |
| 60 | 17 | 0 | -8.650465 | 0.294616 | -1.837983 |

**Table S2**: Cartesian coordinates of **11b**

SCF Done: E (RB3LYP) = -3100.44898875 a.u.

| Center Numbe | Atomic Number | Atomic Type | Coordinates (Angstroms) X | Y | Z |
|---|---|---|---|---|---|
| 1 | 16 | 0 | 1.517613 | 0.000059 | 1.569273 |
| 2 | 6 | 0 | 4.229223 | 0.000182 | 0.696513 |
| 3 | 6 | 0 | 3.270420 | 0.000137 | 1.723818 |
| 4 | 6 | 0 | 3.601244 | 0.000139 | 3.098206 |
| 5 | 1 | 0 | 4.633765 | 0.000177 | 3.428694 |
| 6 | 6 | 0 | 2.524907 | 0.000086 | 3.957796 |
| 7 | 1 | 0 | 2.618636 | 0.000076 | 5.035369 |
| 8 | 6 | 0 | 1.261141 | 0.000042 | 3.317893 |
| 9 | 6 | 0 | 0.000000 | 0.000000 | 3.942829 |
| 10 | 6 | 0 | 0.000000 | 0.000000 | 5.445866 |
| 11 | 6 | 0 | 0.000000 | -1.203883 | 6.159156 |
| 12 | 1 | 0 | -0.000012 | -2.143791 | 5.617619 |
| 13 | 6 | 0 | 0.000002 | -1.212480 | 7.552237 |
| 14 | 1 | 0 | -0.000004 | -2.138853 | 8.115171 |

| | | | | | |
|---|---|---|---|---|---|
| 15 | 6 | 0 | 0.000000 | 0.000000 | 8.224963 |
| 16 | 1 | 0 | 5.236320 | 0.000224 | 1.109286 |
| 17 | 16 | 0 | -1.517613 | -0.000060 | 1.569273 |
| 18 | 6 | 0 | -4.229223 | -0.000182 | 0.696513 |
| 19 | 6 | 0 | -3.270420 | -0.000137 | 1.723818 |
| 20 | 6 | 0 | -3.601244 | -0.000139 | 3.098206 |
| 21 | 1 | 0 | -4.633765 | -0.000177 | 3.428694 |
| 22 | 6 | 0 | -2.524907 | -0.000086 | 3.957796 |
| 23 | 1 | 0 | -2.618636 | -0.000076 | 5.035369 |
| 24 | 6 | 0 | -1.261141 | -0.000042 | 3.317893 |
| 25 | 6 | 0 | 0.000000 | 1.203883 | 6.159156 |
| 26 | 1 | 0 | 0.000012 | 2.143791 | 5.617619 |
| 27 | 6 | 0 | -0.000002 | 1.212481 | 7.552237 |
| 28 | 1 | 0 | 0.000004 | 2.138854 | 8.115170 |
| 29 | 1 | 0 | -5.236320 | -0.000225 | 1.109286 |
| 30 | 16 | 0 | -1.517613 | -0.000060 | -1.569273 |
| 31 | 6 | 0 | -4.229223 | -0.000182 | -0.696513 |
| 32 | 6 | 0 | -3.270420 | -0.000137 | -1.723818 |
| 33 | 6 | 0 | -3.601244 | -0.000139 | -3.098206 |
| 34 | 1 | 0 | -4.633765 | -0.000177 | -3.428694 |
| 35 | 6 | 0 | -2.524907 | -0.000086 | -3.957796 |
| 36 | 1 | 0 | -2.618636 | -0.000076 | -5.035369 |
| 37 | 6 | 0 | -1.261141 | -0.000042 | -3.317893 |
| 38 | 6 | 0 | 0.000000 | 0.000000 | -3.942829 |
| 39 | 6 | 0 | 0.000000 | 0.000000 | -5.445866 |
| 40 | 6 | 0 | 0.000000 | 1.203883 | -6.159156 |
| 41 | 1 | 0 | 0.000012 | 2.143791 | -5.617619 |
| 42 | 6 | 0 | -0.000002 | 1.212481 | -7.552237 |
| 43 | 1 | 0 | 0.000004 | 2.138854 | -8.115170 |
| 44 | 6 | 0 | 0.000000 | 0.000000 | -8.224963 |
| 45 | 1 | 0 | -5.236320 | -0.000225 | -1.109286 |
| 46 | 16 | 0 | 1.517613 | 0.000059 | -1.569273 |
| 47 | 6 | 0 | 4.229223 | 0.000182 | -0.696513 |
| 48 | 6 | 0 | 3.270420 | 0.000137 | -1.723818 |
| 49 | 6 | 0 | 3.601244 | 0.000139 | -3.098206 |
| 50 | 1 | 0 | 4.633765 | 0.000177 | -3.428694 |
| 51 | 6 | 0 | 2.524907 | 0.000086 | -3.957796 |
| 52 | 1 | 0 | 2.618636 | 0.000076 | -5.035369 |
| 53 | 6 | 0 | 1.261141 | 0.000042 | -3.317893 |
| 54 | 6 | 0 | 0.000000 | -1.203883 | -6.159156 |
| 55 | 1 | 0 | -0.000012 | -2.143791 | -5.617619 |
| 56 | 6 | 0 | 0.000002 | -1.212480 | -7.552237 |
| 57 | 1 | 0 | -0.000004 | -2.138853 | -8.115171 |
| 58 | 1 | 0 | 5.236320 | 0.000224 | -1.109286 |
| 59 | 9 | 0 | 0.000000 | 0.000000 | 9.576524 |
| 60 | 9 | 0 | 0.000000 | 0.000000 | -9.576524 |

---

**Table S3**: Cartesian coordinates of **12b**

SCF Done: E (RB3LYP) = -3099.91380272 a.u.

| Center Number | Atomic Number | Atomic Type | Coordinates (Angstroms) X | Y | Z |
|---|---|---|---|---|---|
| 1 | 16 | 0 | -1.633314 | -1.552067 | 0.436315 |
| 2 | 6 | 0 | -0.679356 | -4.224876 | 0.095413 |
| 3 | 6 | 0 | -1.715469 | -3.224541 | -0.023758 |
| 4 | 6 | 0 | -2.981453 | -3.533913 | -0.527206 |
| 5 | 1 | 0 | -3.238239 | -4.526589 | -0.874831 |
| 6 | 6 | 0 | -3.847072 | -2.448196 | -0.576672 |
| 7 | 1 | 0 | -4.841726 | -2.495129 | -0.997932 |
| 8 | 6 | 0 | -3.294427 | -1.257893 | -0.083724 |
| 9 | 6 | 0 | -3.959290 | 0.000000 | -0.000004 |
| 10 | 6 | 0 | -5.421555 | 0.000000 | 0.000000 |
| 11 | 6 | 0 | -6.144183 | 0.925401 | -0.790573 |
| 12 | 1 | 0 | -5.611147 | 1.612479 | -1.436648 |
| 13 | 6 | 0 | -7.526729 | 0.915908 | -0.806892 |
| 14 | 1 | 0 | -8.093450 | 1.592083 | -1.435653 |
| 15 | 6 | 0 | -8.200984 | 0.000000 | 0.000009 |
| 16 | 1 | 0 | -1.108067 | -5.223936 | 0.098060 |
| 17 | 16 | 0 | -1.633316 | 1.552066 | -0.436338 |
| 18 | 6 | 0 | -0.679357 | 4.224876 | -0.095447 |
| 19 | 6 | 0 | -1.715469 | 3.224540 | 0.023734 |
| 20 | 6 | 0 | -2.981449 | 3.533913 | 0.527189 |
| 21 | 1 | 0 | -3.238234 | 4.526589 | 0.874815 |
| 22 | 6 | 0 | -3.847068 | 2.448196 | 0.576663 |
| 23 | 1 | 0 | -4.841719 | 2.495129 | 0.997930 |
| 24 | 6 | 0 | -3.294426 | 1.257892 | 0.083712 |
| 25 | 6 | 0 | -6.144178 | -0.925401 | 0.790580 |
| 26 | 1 | 0 | -5.611138 | -1.612479 | 1.436650 |
| 27 | 6 | 0 | -7.526724 | -0.915908 | 0.806906 |
| 28 | 1 | 0 | -8.093441 | -1.592084 | 1.435670 |

| | | | | | |
|---|---|---|---|---|---|
| 29 | 1 | 0 | -1.108068 | 5.223936 | -0.098106 |
| 30 | 16 | 0 | 1.633317 | 1.552066 | -0.436334 |
| 31 | 6 | 0 | 0.679356 | 4.224876 | -0.095446 |
| 32 | 6 | 0 | 1.715468 | 3.224540 | 0.023738 |
| 33 | 6 | 0 | 2.981447 | 3.533914 | 0.527196 |
| 34 | 1 | 0 | 3.238230 | 4.526590 | 0.874822 |
| 35 | 6 | 0 | 3.847066 | 2.448197 | 0.576672 |
| 36 | 1 | 0 | 4.841716 | 2.495131 | 0.997941 |
| 37 | 6 | 0 | 3.294425 | 1.257893 | 0.083720 |
| 38 | 6 | 0 | 3.959290 | 0.000001 | 0.000006 |
| 39 | 6 | 0 | 5.421555 | 0.000001 | 0.000014 |
| 40 | 6 | 0 | 6.144176 | -0.925401 | 0.790594 |
| 41 | 1 | 0 | 5.611134 | -1.612478 | 1.436664 |
| 42 | 6 | 0 | 7.526722 | -0.915908 | 0.806924 |
| 43 | 1 | 0 | 8.093437 | -1.592084 | 1.435690 |
| 44 | 6 | 0 | 8.200984 | 0.000000 | 0.000029 |
| 45 | 1 | 0 | 1.108067 | 5.223936 | -0.098103 |
| 46 | 16 | 0 | 1.633314 | -1.552067 | 0.436320 |
| 47 | 6 | 0 | 0.679357 | -4.224876 | 0.095414 |
| 48 | 6 | 0 | 1.715470 | -3.224540 | -0.023754 |
| 49 | 6 | 0 | 2.981455 | -3.533912 | -0.527200 |
| 50 | 1 | 0 | 3.238242 | -4.526588 | -0.874825 |
| 51 | 6 | 0 | 3.847074 | -2.448195 | -0.576664 |
| 52 | 1 | 0 | 4.841728 | -2.495128 | -0.997922 |
| 53 | 6 | 0 | 3.294427 | -1.257892 | -0.083716 |
| 54 | 6 | 0 | 6.144185 | 0.925402 | -0.790559 |
| 55 | 1 | 0 | 5.611151 | 1.612480 | -1.436634 |
| 56 | 6 | 0 | 7.526731 | 0.915908 | -0.806874 |
| 57 | 1 | 0 | 8.093454 | 1.592083 | -1.435633 |
| 58 | 1 | 0 | 1.108068 | -5.223936 | 0.098062 |
| 59 | 9 | 0 | -9.527029 | 0.000000 | 0.000013 |
| 60 | 9 | 0 | 9.527029 | 0.000000 | 0.000036 |

---

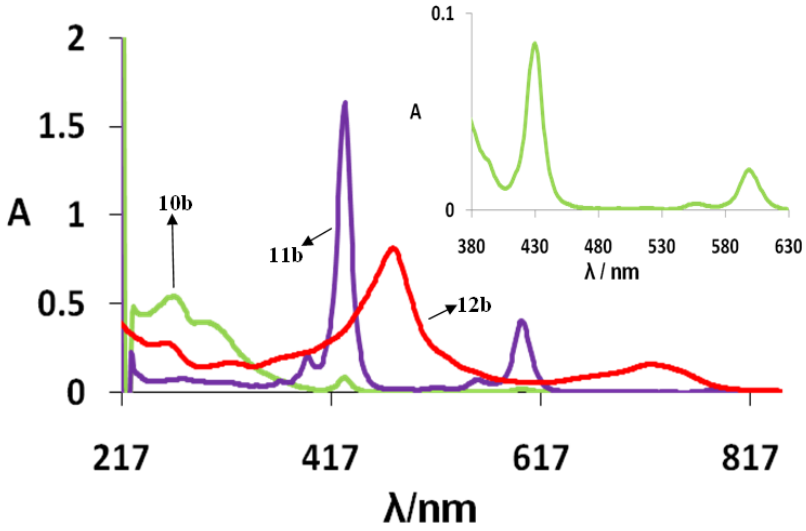



**Figure S40**: UV-Vis. spectra of **10b** (DCM), **11b** (DCM) and **12b** ($H_2SO_4$). (Inset shows the partial auto-oxidation of the **10b** into **11b**)

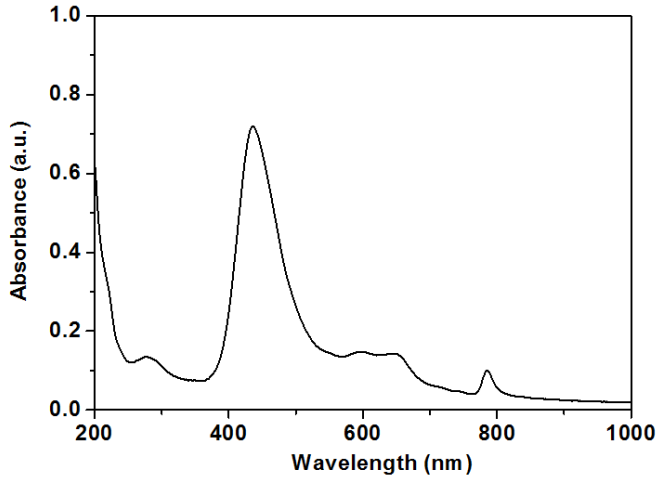



**Figure S40a**: UV-Vis. spectra of thin film of **11a.**

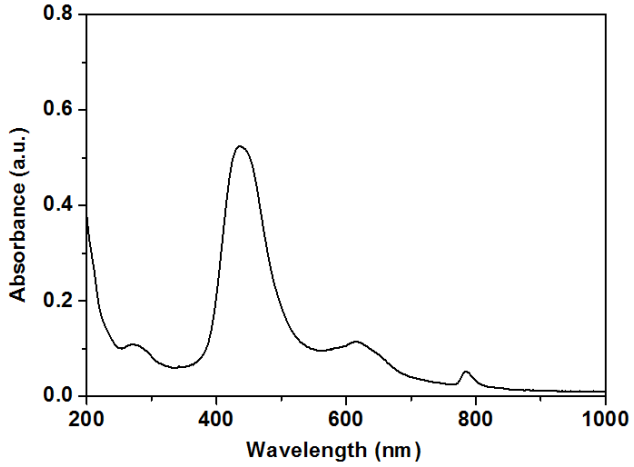



**Figure S40b**: UV-Vis. spectra of thin film of **11b.**

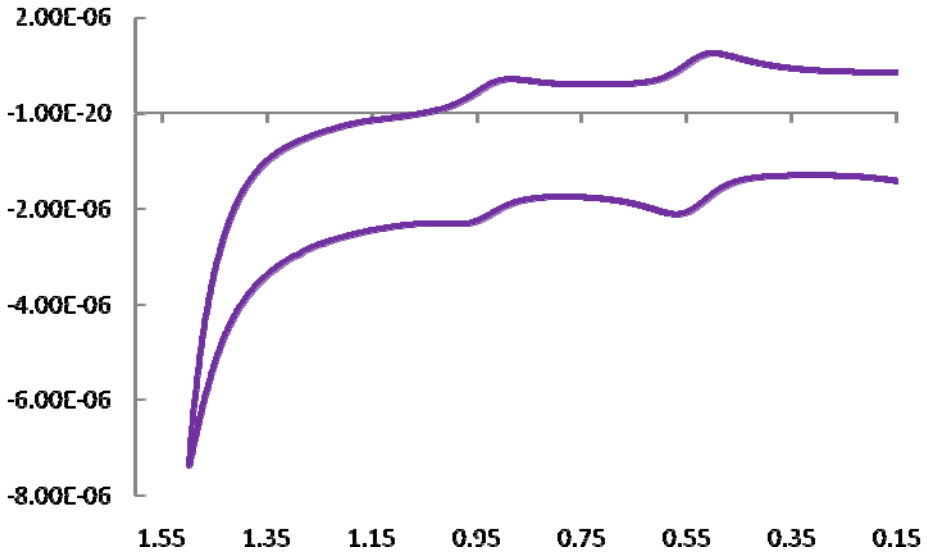



**Figure S41**: Cyclic voltammogram (CV) for **11b** (DCM, electrolyte $TBAPF_6$; working electrode: Pt; ref. electrode: Ag/AgCl; Scan rate 100 mV $s^{-1}$.

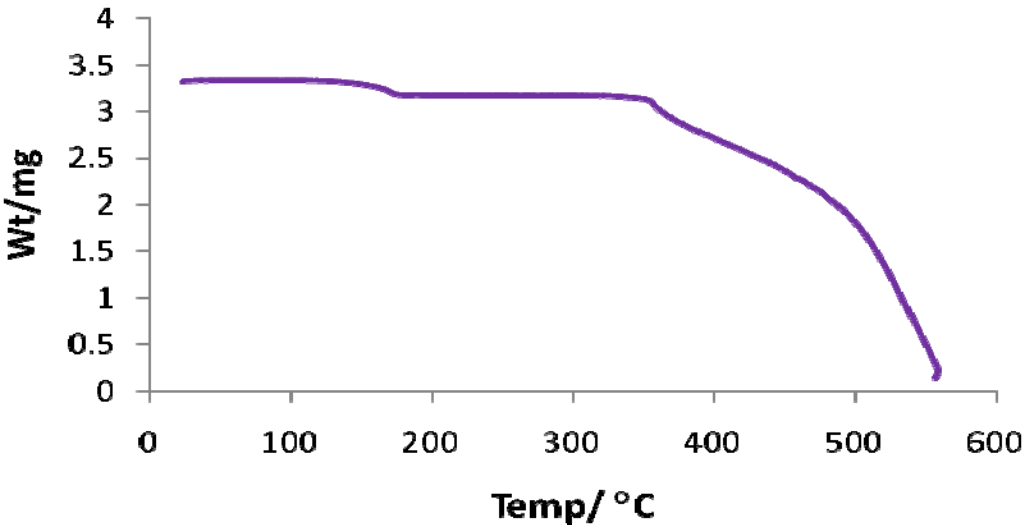



**Figure S42**: TGA Analysis of **11a** under $N_2$ with temperature rise of 10°C per minute. Thus**11a** is highly stable as it is having a high thermal decomposition temp. of about >355°C.

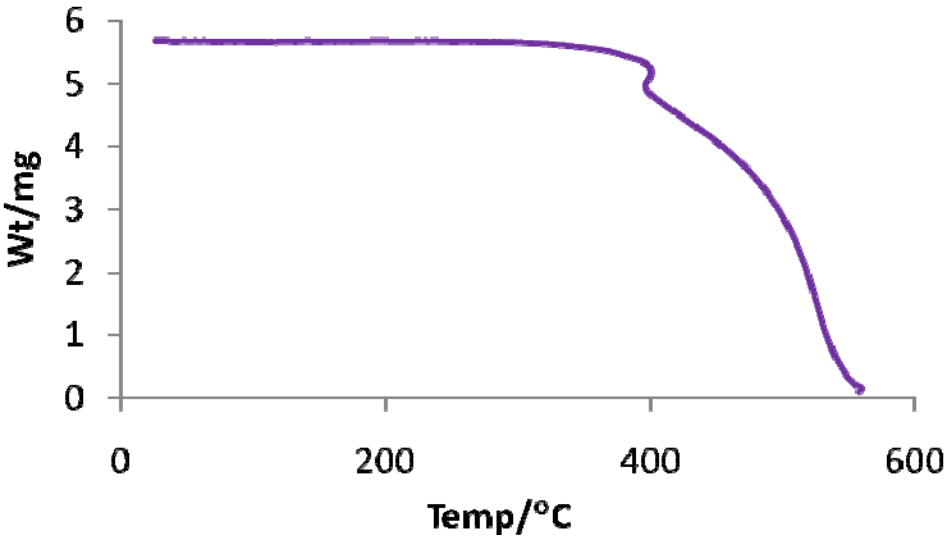



**Figure S43**: TGA Analysis of **11b** under $N_2$ with temperature rise of 10°C per minute. Thus**11b** is highly stable as it is having a high thermal decomposition temp. of about >355°C.

**X-Ray Diffraction Analysis of 11a:**
Single crystals of **11a** suitable for an X-Ray crystal structure determination were grown in a dark, quiet and undisturbed place from dry DCM with a toluene layer upon it (2 weeks).
**Table S4**: The crystallographic data for **11a**.

| | |
|---|---|
| Empirical formula | $C_{136}$ $H_{80}$ $Cl_8$ $S_{16}$ |
| Formula weight | 2510.56 |
| Temperature | 150(2) K |
| Wavelength | 1.54180 A |
| Crystal system, space group | Orthorhombic, F d d 2 |
| Unit cell dimensions | a = 60.299(6) A alpha = 90 deg.<br>b = 19.5214(15) A beta = 90 deg.<br>c = 9.5973(8) A gamma = 90 deg. |
| Volume | 11297.2(17) A^3 |
| Z, Calculated density | 4, 1.476 Mg/m^3 |
| Absorption coefficient | 5.018 mm^-1 |
| F(000) | 5152 |
| Crystal size | 0.23 x 0.18 x 0.14 mm |
| Theta range for data collection | 2.93 to 72.40 deg. |
| Limiting indices | -72<=h<=73, -22<=k<=23, -9<=l<=11 |
| Reflections collected / unique | 19859 / 4940 [R(int) = 0.0462] |
| Completeness to theta = 72.40 | 99.3 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.5401 and 0.3915 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 4940 / 1 / 361 |
| Goodness-of-fit on F^2 | 1.072 |
| Final R indices [I>2sigma(I)] | R1 = 0.0503, wR2 = 0.1381 |

| | |
|---|---|
| R indices (all data) | R1 = 0.0529, wR2 = 0.1411 |
| Absolute structure parameter | 0.00(2) |
| Largest diff. peak and hole | 0.397 and -0.321 e.A^-3 |

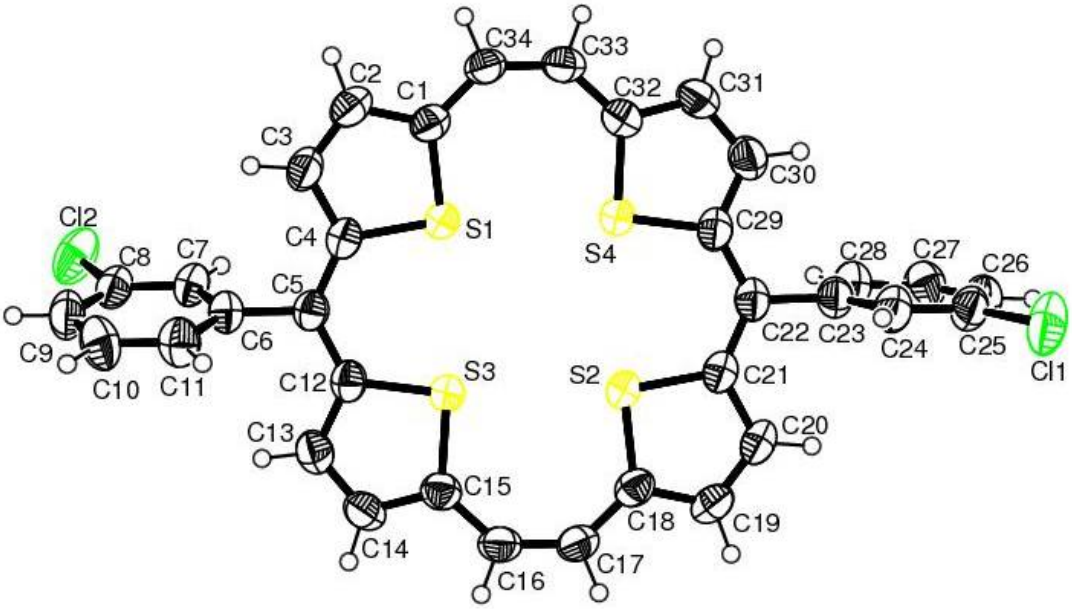



**Table S5**: Torsion angles [deg] for **11a**.

| | |
|---|---|
| C(4)-S(1)-C(1)-C(34) | -176.7(4) |
| C(4)-S(1)-C(1)-C(2) | -0.2(3) |
| C(34)-C(1)-C(2)-C(3) | 176.9(4) |
| S(1)-C(1)-C(2)-C(3) | 0.1(5) |
| C(1)-C(2)-C(3)-C(4) | 0.0(6) |
| C(2)-C(3)-C(4)-C(5) | 179.3(4) |
| C(2)-C(3)-C(4)-S(1) | -0.1(5) |
| C(1)-S(1)-C(4)-C(5) | -179.3(3) |
| C(1)-S(1)-C(4)-C(3) | 0.1(3) |
| C(3)-C(4)-C(5)-C(12) | -173.0(4) |
| S(1)-C(4)-C(5)-C(12) | 6.3(6) |
| C(3)-C(4)-C(5)-C(6) | 7.0(6) |
| S(1)-C(4)-C(5)-C(6) | -173.8(3) |
| C(4)-C(5)-C(6)-C(7) | 69.9(5) |
| C(12)-C(5)-C(6)-C(7) | -110.1(5) |
| C(4)-C(5)-C(6)-C(11) | -110.1(5) |
| C(12)-C(5)-C(6)-C(11) | 69.9(5) |
| C(11)-C(6)-C(7)-C(8) | 0.0(7) |
| C(5)-C(6)-C(7)-C(8) | 180.0(4) |
| C(6)-C(7)-C(8)-C(9) | 0.6(7) |
| C(6)-C(7)-C(8)-Cl(2) | -178.5(4) |
| C(7)-C(8)-C(9)-C(10) | -0.5(8) |
| Cl(2)-C(8)-C(9)-C(10) | 178.6(4) |
| C(8)-C(9)-C(10)-C(11) | -0.1(9) |

| | |
|---|---|
| C(9)-C(10)-C(11)-C(6) | 0.7(9) |
| C(7)-C(6)-C(11)-C(10) | -0.6(8) |
| C(5)-C(6)-C(11)-C(10) | 179.4(5) |
| C(4)-C(5)-C(12)-C(13) | -178.1(4) |
| C(6)-C(5)-C(12)-C(13) | 1.9(6) |
| C(4)-C(5)-C(12)-S(3) | 2.7(6) |
| C(6)-C(5)-C(12)-S(3) | -177.2(3) |
| C(15)-S(3)-C(12)-C(13) | -1.3(3) |
| C(15)-S(3)-C(12)-C(5) | 178.1(3) |
| C(5)-C(12)-C(13)-C(14) | -178.0(4) |
| S(3)-C(12)-C(13)-C(14) | 1.3(5) |
| C(12)-C(13)-C(14)-C(15) | -0.7(6) |
| C(13)-C(14)-C(15)-C(16) | -179.5(4) |
| C(13)-C(14)-C(15)-S(3) | -0.3(5) |
| C(12)-S(3)-C(15)-C(16) | -179.9(4) |
| C(12)-S(3)-C(15)-C(14) | 0.9(3) |
| C(14)-C(15)-C(16)-C(17) | 179.3(5) |
| S(3)-C(15)-C(16)-C(17) | 0.3(8) |
| C(15)-C(16)-C(17)-C(18) | -0.1(10) |
| C(16)-C(17)-C(18)-C(19) | -173.0(5) |
| C(16)-C(17)-C(18)-S(2) | 3.0(8) |
| C(21)-S(2)-C(18)-C(19) | 4.1(3) |
| C(21)-S(2)-C(18)-C(17) | -172.3(4) |
| C(17)-C(18)-C(19)-C(20) | 173.2(4) |
| S(2)-C(18)-C(19)-C(20) | -3.4(5) |
| C(18)-C(19)-C(20)-C(21) | 0.6(6) |
| C(19)-C(20)-C(21)-C(22) | -173.1(4) |
| C(19)-C(20)-C(21)-S(2) | 2.5(5) |
| C(18)-S(2)-C(21)-C(22) | 171.9(4) |
| C(18)-S(2)-C(21)-C(20) | -3.8(3) |
| C(20)-C(21)-C(22)-C(29) | 175.4(4) |
| S(2)-C(21)-C(22)-C(29) | 0.5(6) |
| C(20)-C(21)-C(22)-C(23) | 0.5(6) |
| S(2)-C(21)-C(22)-C(23) | -174.4(3) |
| C(21)-C(22)-C(23)-C(24) | -78.5(6) |
| C(29)-C(22)-C(23)-C(24) | 105.9(5) |
| C(21)-C(22)-C(23)-C(28) | 100.8(5) |
| C(29)-C(22)-C(23)-C(28) | -74.7(6) |
| C(28)-C(23)-C(24)-C(25) | -1.2(7) |
| C(22)-C(23)-C(24)-C(25) | 178.2(4) |
| C(23)-C(24)-C(25)-C(26) | -0.9(8) |
| C(23)-C(24)-C(25)-Cl(1) | -179.9(4) |
| C(24)-C(25)-C(26)-C(27) | 1.8(8) |
| Cl(1)-C(25)-C(26)-C(27) | -179.2(4) |
| C(25)-C(26)-C(27)-C(28) | -0.6(8) |
| C(26)-C(27)-C(28)-C(23) | -1.4(8) |
| C(24)-C(23)-C(28)-C(27) | 2.3(8) |
| C(22)-C(23)-C(28)-C(27) | -177.0(5) |
| C(21)-C(22)-C(29)-C(30) | -177.7(4) |
| C(23)-C(22)-C(29)-C(30) | -2.8(7) |
| C(21)-C(22)-C(29)-S(4) | 0.7(6) |
| C(23)-C(22)-C(29)-S(4) | 175.6(3) |
| C(32)-S(4)-C(29)-C(22) | -178.0(4) |
| C(32)-S(4)-C(29)-C(30) | 0.7(4) |
| C(22)-C(29)-C(30)-C(31) | 178.4(4) |
| S(4)-C(29)-C(30)-C(31) | -0.2(5) |
| C(29)-C(30)-C(31)-C(32) | -0.4(7) |
| C(30)-C(31)-C(32)-C(33) | 177.0(4) |

| | |
|---|---|
| C(30)-C(31)-C(32)-S(4) | 0.9(6) |
| C(29)-S(4)-C(32)-C(33) | -176.7(4) |
| C(29)-S(4)-C(32)-C(31) | -0.9(4) |
| C(31)-C(32)-C(33)-C(34) | -179.4(5) |
| S(4)-C(32)-C(33)-C(34) | -4.2(8) |
| C(32)-C(33)-C(34)-C(1) | -7.9(10) |
| C(2)-C(1)-C(34)-C(33) | -177.8(5) |
| S(1)-C(1)-C(34)-C(33) | -1.7(8) |

The crystal structure has been deposited at the Cambridge Crystallographic Data Centre (CCDC 900288). The data can be obtained free of charge via the Internet at www.ccdc.cam.ac.uk/data_request/cif.

## Device Fabrication:

### Experimental:

OFET devices were fabricated in the top-contact device configuration. The substrate was heavily doped, n-type Si gate electrode with a 500 nm thick $SiO_2$ layer as the gate dielectric. The gate dielectric was treated with octadecyltrichlorosilane (OTS) by vapor deposition method. Subsequently, organic semiconductors were deposited on the substrate by thermal evaporation under a pressure of $8 \times 10^{-4}$ Pa at a deposition rate gradually increased from 0.1 Å $s^{-1}$ to 0.4 Å $s^{-1}$ at the first 20 nm and then maintained 0.5 Å $s^{-1}$ until the thickness of the film was 50 nm. The deposition rate and film thickness were monitored by a quartz crystal microbalance (ULVAC CRTM-6000). Finally, 20 nm thick gold source and drain electrode were deposited through a shadow mask. The channel length ($L$) and width ($W$) were 0.11 mm and 5.30 mm, respectively. The FET characteristics were measured at room temperature in air using Keithley 4200 SCS. Atomic force microscopy (AFM) measurements were carried out with a Nanoscope IIIa instrument (Digital Instruments) operating in tapping mode. UV-Vis spectra were recorded on a JASCO V-570 spectrometer.

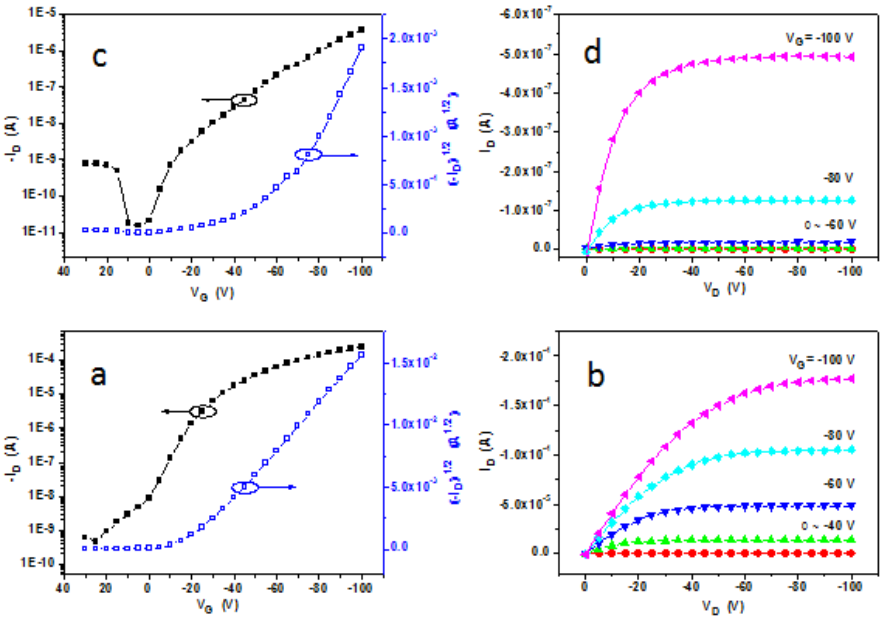



**Figure S44:** Typical transfer (a) and output (b) characteristics of FET devices based on **11a,** with OTS-treated $SiO_2$/Si substrate ($T_s$ = 100 $^oC$). Typical transfer (c) and output (d) characteristics of FET devices based on **11b,** with OTS-treated $SiO_2$/Si substrate ($T_s$ = 100 $^oC$).

CIF Files for **11a**:

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;
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 'x, y+1/2, z+1/2'
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 'x+1/2, y+1/2, z'
 '-x+1/2, -y+1/2, z'
 'x+3/4, -y+3/4, z+1/4'
 '-x+3/4, y+3/4, z+1/4'

_cell_length_a                    60.299(6)
_cell_length_b                    19.5214(15)
_cell_length_c                    9.5973(8)
_cell_angle_alpha                 90.00
_cell_angle_beta                  90.00
```

```
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_cell_formula_units_Z              4
_cell_measurement_temperature      150(2)
_cell_measurement_reflns_used    8176
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_exptl_crystal_colour              BLACK
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_exptl_crystal_size_min            0.14
_exptl_crystal_density_meas        ?
_exptl_crystal_density_diffrn      1.476
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_exptl_crystal_F_000               5152
_exptl_absorpt_coefficient_mu      5.018
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_exptl_absorpt_correction_T_max    0.5401
_exptl_absorpt_correction_type             multi-scan
_exptl_absorpt_process_details
;
(CrysAlis RED; Oxford Diffraction, 2009)
;

_diffrn_ambient_temperature        150(2)
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_diffrn_radiation_type             CuK\a
_diffrn_radiation_source           'Micro-Focus (Cu) X-ray Source'
_diffrn_radiation_monochromator    graphite
_diffrn_measurement_device_type    'OXFORD DIFFRACTION SUPER NOVA'
_diffrn_measurement_method         '\w/q-scan'
_diffrn_detector_area_resol_mean   15.9948
_diffrn_standards_number           ?
_diffrn_standards_interval_count   ?
_diffrn_standards_interval_time    ?
_diffrn_standards_decay_%          ?
_diffrn_reflns_number              19859
_diffrn_reflns_av_R_equivalents    0.0462
_diffrn_reflns_av_sigmaI/netI      0.0312
_diffrn_reflns_limit_h_min         -72
_diffrn_reflns_limit_h_max         73
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_diffrn_reflns_limit_k_max         23
_diffrn_reflns_limit_l_min         -9
_diffrn_reflns_limit_l_max         11
_diffrn_reflns_theta_min           2.93
_diffrn_reflns_theta_max           72.40
_reflns_number_total               4940
_reflns_number_gt                  4655
_reflns_threshold_expression       >2sigma(I)

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Ltd.,'
_computing_cell_refinement         'CrysAlis RED, Oxford Diffraction
Ltd.,'
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Ltd.,'
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR
and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold expression
of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
 not relevant to the choice of reflections for refinement.  R-factors
based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
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_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0970P)^2^+9.1277P] where
P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     riding
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details
 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack    0.00(2)
_refine_ls_number_reflns          4940
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_refine_ls_wR_factor_ref          0.1411
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_refine_ls_goodness_of_fit_ref    1.072
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_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000

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S2 S 0.184951(14) 0.33268(5) 0.90660(11) 0.0474(2) Uani 1 1 d . . .
S3 S 0.145561(15) 0.31985(5) 0.69777(11) 0.0487(2) Uani 1 1 d . . .
S4 S 0.152939(15) 0.39138(6) 1.12089(10) 0.0506(2) Uani 1 1 d . . .
Cl1 Cl 0.26458(2) 0.34249(9) 1.49820(19) 0.0886(5) Uani 1 1 d . . .
Cl2 Cl 0.03607(2) 0.42986(8) 0.35524(18) 0.0848(4) Uani 1 1 d . . .
C1 C 0.09336(6) 0.3904(2) 1.0407(5) 0.0477(9) Uani 1 1 d . . .
C2 C 0.07237(7) 0.3775(2) 0.9809(5) 0.0557(10) Uani 1 1 d . . .
H2 H 0.0589 0.3838 1.0307 0.067 Uiso 1 1 calc R . .
C3 C 0.07289(6) 0.3555(2) 0.8470(5) 0.0550(10) Uani 1 1 d . . .
H3 H 0.0599 0.3451 0.7954 0.066 Uiso 1 1 calc R . .
C4 C 0.09426(6) 0.34941(19) 0.7910(5) 0.0455(8) Uani 1 1 d . . .
C5 C 0.09968(6) 0.32730(18) 0.6566(5) 0.0448(8) Uani 1 1 d . . .
C6 C 0.08050(6) 0.3165(2) 0.5599(5) 0.0472(8) Uani 1 1 d . . .
C7 C 0.06835(6) 0.3715(2) 0.5097(5) 0.0505(9) Uani 1 1 d . . .
H7 H 0.0721 0.4169 0.5364 0.061 Uiso 1 1 calc R . .
C8 C 0.05068(6) 0.3599(2) 0.4202(5) 0.0537(9) Uani 1 1 d . . .
C9 C 0.04460(7) 0.2954(3) 0.3802(6) 0.0658(12) Uani 1 1 d . . .
H9 H 0.0324 0.2883 0.3195 0.079 Uiso 1 1 calc R . .
C10 C 0.05682(9) 0.2404(3) 0.4309(6) 0.0706(13) Uani 1 1 d . . .
H10 H 0.0529 0.1951 0.4045 0.085 Uiso 1 1 calc R . .
C11 C 0.07447(8) 0.2503(2) 0.5183(6) 0.0598(11) Uani 1 1 d . . .
H11 H 0.0827 0.2120 0.5509 0.072 Uiso 1 1 calc R . .
C12 C 0.12097(6) 0.31410(18) 0.6021(5) 0.0470(8) Uani 1 1 d . . .
C13 C 0.12608(7) 0.2938(2) 0.4658(5) 0.0548(9) Uani 1 1 d . . .
H13 H 0.1152 0.2883 0.3954 0.066 Uiso 1 1 calc R . .
C14 C 0.14810(7) 0.2825(2) 0.4432(5) 0.0560(9) Uani 1 1 d . . .
H14 H 0.1537 0.2680 0.3554 0.067 Uiso 1 1 calc R . .
C15 C 0.16194(7) 0.2936(2) 0.5582(5) 0.0501(9) Uani 1 1 d . . .
C16 C 0.18499(7) 0.2850(2) 0.5536(5) 0.0557(10) Uani 1 1 d . . .
H16 H 0.1898 0.2704 0.4642 0.067 Uiso 1 1 calc R . .
C17 C 0.20254(6) 0.2921(2) 0.6447(5) 0.0552(10) Uani 1 1 d . . .
H17 H 0.2163 0.2811 0.6019 0.066 Uiso 1 1 calc R . .
C18 C 0.20531(6) 0.31158(19) 0.7848(5) 0.0492(9) Uani 1 1 d . . .
C19 C 0.22626(6) 0.3210(2) 0.8462(6) 0.0570(10) Uani 1 1 d . . .
H19 H 0.2397 0.3100 0.7993 0.068 Uiso 1 1 calc R . .
C20 C 0.22593(6) 0.3468(2) 0.9773(5) 0.0566(11) Uani 1 1 d . . .
H20 H 0.2390 0.3558 1.0291 0.068 Uiso 1 1 calc R . .
C21 C 0.20435(6) 0.3593(2) 1.0305(5) 0.0486(9) Uani 1 1 d . . .
C22 C 0.19909(6) 0.3919(2) 1.1558(5) 0.0508(9) Uani 1 1 d . . .
C23 C 0.21852(6) 0.4165(2) 1.2416(5) 0.0523(9) Uani 1 1 d . . .
C24 C 0.23068(7) 0.3722(3) 1.3219(6) 0.0589(10) Uani 1 1 d . . .
H24 H 0.2268 0.3250 1.3275 0.071 Uiso 1 1 calc R . .
C25 C 0.24914(7) 0.3985(3) 1.3968(5) 0.0609(11) Uani 1 1 d . . .
C26 C 0.25502(7) 0.4655(3) 1.3886(6) 0.0672(12) Uani 1 1 d . . .
H26 H 0.2677 0.4819 1.4373 0.081 Uiso 1 1 calc R . .
C27 C 0.24256(8) 0.5091(3) 1.3097(6) 0.0700(13) Uani 1 1 d . . .
H27 H 0.2465 0.5561 1.3044 0.084 Uiso 1 1 calc R . .
C28 C 0.22424(7) 0.4857(2) 1.2373(5) 0.0611(11) Uani 1 1 d . . .
H28 H 0.2155 0.5168 1.1844 0.073 Uiso 1 1 calc R . .
C29 C 0.17796(6) 0.4090(2) 1.2067(5) 0.0511(9) Uani 1 1 d . . .
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C30 C 0.17314(7) 0.4439(3) 1.3311(5) 0.0608(11) Uani 1 1 d . . .
H30 H 0.1843 0.4583 1.3944 0.073 Uiso 1 1 calc R . .
C31 C 0.15105(8) 0.4553(3) 1.3535(5) 0.0621(11) Uani 1 1 d . . .
H31 H 0.1457 0.4781 1.4343 0.075 Uiso 1 1 calc R . .
C32 C 0.13680(7) 0.4310(2) 1.2489(5) 0.0504(9) Uani 1 1 d . . .
C33 C 0.11373(7) 0.4355(2) 1.2577(5) 0.0535(9) Uani 1 1 d . . .
H33 H 0.1090 0.4571 1.3413 0.064 Uiso 1 1 calc R . .
C34 C 0.09596(6) 0.4163(2) 1.1746(5) 0.0521(9) Uani 1 1 d . . .
H34 H 0.0821 0.4223 1.2206 0.062 Uiso 1 1 calc R . .

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S2 0.0359(4) 0.0573(5) 0.0489(5) -0.0034(4) -0.0029(3) 0.0018(3)
S3 0.0405(4) 0.0598(5) 0.0458(5) -0.0069(4) -0.0033(4) 0.0031(4)
S4 0.0396(4) 0.0676(6) 0.0446(5) -0.0063(4) -0.0029(4) 0.0027(4)
Cl1 0.0624(7) 0.1088(10) 0.0947(11) 0.0262(9) -0.0300(7) -0.0010(7)
Cl2 0.0546(6) 0.0986(9) 0.1013(11) 0.0351(8) -0.0167(6) 0.0079(6)
C1 0.0414(18) 0.052(2) 0.049(2) 0.0067(17) 0.0050(16) 0.0029(15)
C2 0.0388(19) 0.066(2) 0.062(3) 0.010(2) 0.0058(17) 0.0032(17)
C3 0.0379(18) 0.069(2) 0.058(3) 0.007(2) -0.0051(17) -0.0058(17)
C4 0.0399(17) 0.0445(18) 0.052(2) 0.0041(16) -0.0057(15) -0.0010(14)
C5 0.0425(17) 0.0427(16) 0.049(2) 0.0029(15) -0.0053(16) 0.0002(13)
C6 0.0456(18) 0.0467(19) 0.049(2) 0.0016(16) -0.0070(17) -0.0059(14)
C7 0.0417(18) 0.052(2) 0.057(2) 0.0077(18) -0.0081(17) -0.0015(15)
C8 0.0412(18) 0.067(2) 0.053(2) 0.012(2) -0.0047(17) -0.0032(16)
C9 0.051(2) 0.087(3) 0.059(3) 0.003(2) -0.014(2) -0.016(2)
C10 0.078(3) 0.063(3) 0.071(3) -0.006(2) -0.016(3) -0.021(2)
C11 0.064(2) 0.045(2) 0.071(3) 0.002(2) -0.009(2) -0.0065(17)
C12 0.0457(18) 0.0442(18) 0.051(2) 0.0027(16) -0.0085(16) 0.0003(14)
C13 0.059(2) 0.055(2) 0.050(2) -0.0021(18) -0.0114(19) 0.0009(17)
C14 0.066(2) 0.058(2) 0.043(2) -0.0075(19) 0.0000(19) 0.0043(18)
C15 0.054(2) 0.0478(19) 0.049(2) -0.0034(17) 0.0015(17) 0.0048(15)
C16 0.055(2) 0.056(2) 0.056(3) -0.0109(19) 0.0095(18) 0.0044(17)
C17 0.0440(18) 0.060(2) 0.062(3) -0.008(2) 0.0066(18) 0.0063(16)
C18 0.0429(18) 0.0437(18) 0.061(3) -0.0024(17) 0.0017(17) 0.0049(14)
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C20 0.0365(18) 0.066(2) 0.067(3) 0.005(2) -0.0071(18) 0.0007(16)
C21 0.0395(17) 0.053(2) 0.053(2) 0.0034(17) -0.0057(16) -0.0021(15)
C22 0.0457(19) 0.056(2) 0.051(2) 0.0020(18) -0.0093(17) -0.0067(15)
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C24 0.048(2) 0.068(3) 0.061(3) 0.006(2) -0.0088(19) -0.0056(18)
C25 0.0426(18) 0.083(3) 0.057(3) 0.005(2) -0.0049(19) -0.0047(19)
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C28 0.057(2) 0.062(2) 0.065(3) 0.003(2) -0.009(2) -0.0064(19)
C29 0.0440(19) 0.059(2) 0.051(2) 0.0051(19) -0.0082(17) -0.0049(16)
C30 0.057(2) 0.073(3) 0.052(3) -0.007(2) -0.0046(19) -0.0119(19)
C31 0.064(2) 0.073(3) 0.050(3) -0.017(2) 0.0034(19) -0.004(2)
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C33 0.056(2) 0.059(2) 0.045(2) -0.0010(18) 0.0091(18) 0.0060(17)
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planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters are
only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
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S1 C4 1.747(4) . ?
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S3 C12 1.747(4) . ?
S4 C32 1.748(4) . ?
S4 C29 1.753(4) . ?
Cl1 C25 1.734(5) . ?
Cl2 C8 1.741(4) . ?
C1 C34 1.389(7) . ?
C1 C2 1.412(6) . ?
C2 C3 1.356(7) . ?
C2 H2 0.9500 . ?
C3 C4 1.401(5) . ?
C3 H3 0.9500 . ?
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C5 C12 1.410(5) . ?
C5 C6 1.498(5) . ?
C6 C7 1.387(6) . ?
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C7 C8 1.387(6) . ?
C7 H7 0.9500 . ?
C8 C9 1.368(7) . ?
C9 C10 1.390(7) . ?
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C10 C11 1.369(7) . ?
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C16 C17 1.380(6) . ?
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C18 C19 1.406(6) . ?
C19 C20 1.355(7) . ?
C19 H19 0.9500 . ?
C20 C21 1.419(6) . ?
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C21 C22 1.397(7) . ?
C22 C29 1.405(6) . ?
C22 C23 1.510(5) . ?
C23 C24 1.371(6) . ?
C23 C28 1.395(6) . ?
C24 C25 1.421(6) . ?
C24 H24 0.9500 . ?
C25 C26 1.358(7) . ?
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C29 C30 1.405(7) . ?
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C30 H30 0.9500 . ?
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C34 C1 C2 122.8(4) . . ?
C34 C1 S1 128.5(3) . . ?
C2 C1 S1 108.6(3) . . ?
C3 C2 C1 114.9(4) . . ?
C3 C2 H2 122.5 . . ?
C1 C2 H2 122.5 . . ?
C2 C3 C4 114.3(4) . . ?
C2 C3 H3 122.8 . . ?
C4 C3 H3 122.8 . . ?
C5 C4 C3 126.5(4) . . ?
C5 C4 S1 124.4(3) . . ?
C3 C4 S1 109.1(3) . . ?
C4 C5 C12 127.7(4) . . ?
C4 C5 C6 115.7(3) . . ?
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C7 C6 C11 118.6(4) . . ?
C7 C6 C5 120.9(3) . . ?
C11 C6 C5 120.5(4) . . ?
C8 C7 C6 119.7(4) . . ?
C8 C7 H7 120.2 . . ?
C6 C7 H7 120.2 . . ?
C9 C8 C7 122.0(4) . . ?
C9 C8 Cl2 119.1(3) . . ?
C7 C8 Cl2 118.9(3) . . ?
C8 C9 C10 118.1(4) . . ?
C8 C9 H9 120.9 . . ?
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C10 C11 C6 120.5(4) . . ?
C10 C11 H11 119.8 . . ?
C6 C11 H11 119.8 . . ?
C13 C12 C5 126.7(4) . . ?
C13 C12 S3 108.8(3) . . ?
C5 C12 S3 124.5(3) . . ?
C14 C13 C12 114.1(4) . . ?
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C12 C13 H13 122.9 . . ?
C13 C14 C15 115.4(4) . . ?
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C15 C14 H14 122.3 . . ?
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C16 C15 S3 128.5(4) . . ?
C14 C15 S3 108.3(3) . . ?
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C18 C17 H17 111.8 . . ?
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C24 C23 C22 121.4(4) . . ?
C28 C23 C22 118.9(4) . . ?
C23 C24 C25 118.4(4) . . ?
C23 C24 H24 120.8 . . ?
C25 C24 H24 120.8 . . ?
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C26 C25 C24 121.5(4) . . ?
C26 C25 Cl1 120.0(4) . . ?
C24 C25 Cl1 118.5(4) . . ?
C25 C26 C27 119.3(4) . . ?
C25 C26 H26 120.4 . . ?
C27 C26 H26 120.4 . . ?
C26 C27 C28 120.9(5) . . ?
C26 C27 H27 119.6 . . ?
C28 C27 H27 119.6 . . ?
C27 C28 C23 120.2(5) . . ?
C27 C28 H28 119.9 . . ?
C23 C28 H28 119.9 . . ?
C22 C29 C30 126.7(4) . . ?
C22 C29 S4 124.8(4) . . ?
C30 C29 S4 108.4(3) . . ?
C31 C30 C29 114.5(4) . . ?
C31 C30 H30 122.8 . . ?
C29 C30 H30 122.8 . . ?
C30 C31 C32 115.4(4) . . ?
C30 C31 H31 122.3 . . ?
C32 C31 H31 122.3 . . ?
C33 C32 C31 123.0(4) . . ?
C33 C32 S4 128.7(3) . . ?
C31 C32 S4 108.1(3) . . ?
C34 C33 C32 135.8(4) . . ?
C34 C33 H33 112.1 . . ?
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C33 C34 C1 135.8(4) . . ?
C33 C34 H34 112.1 . . ?
C1 C34 H34 112.1 . . ?

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C4 S1 C1 C34 -176.7(4) . . . . ?
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C34 C1 C2 C3 176.9(4) . . . . ?
S1 C1 C2 C3 0.1(5) . . . . ?
C1 C2 C3 C4 0.0(6) . . . . ?
C2 C3 C4 C5 179.3(4) . . . . ?
C2 C3 C4 S1 -0.1(5) . . . . ?
C1 S1 C4 C5 -179.3(3) . . . . ?
C1 S1 C4 C3 0.1(3) . . . . ?
C3 C4 C5 C12 -173.0(4) . . . . ?
S1 C4 C5 C12 6.3(6) . . . . ?
C3 C4 C5 C6 7.0(6) . . . . ?
S1 C4 C5 C6 -173.8(3) . . . . ?
C4 C5 C6 C7 69.9(5) . . . . ?
C12 C5 C6 C7 -110.1(5) . . . . ?
C4 C5 C6 C11 -110.1(5) . . . . ?
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C11 C6 C7 C8 0.0(7) . . . . ?
C5 C6 C7 C8 180.0(4) . . . . ?
C6 C7 C8 C9 0.6(7) . . . . ?
C6 C7 C8 Cl2 -178.5(4) . . . . ?
C7 C8 C9 C10 -0.5(8) . . . . ?
Cl2 C8 C9 C10 178.6(4) . . . . ?
C8 C9 C10 C11 -0.1(9) . . . . ?
C9 C10 C11 C6 0.7(9) . . . . ?
C7 C6 C11 C10 -0.6(8) . . . . ?
C5 C6 C11 C10 179.4(5) . . . . ?
C4 C5 C12 C13 -178.1(4) . . . . ?
C6 C5 C12 C13 1.9(6) . . . . ?
C4 C5 C12 S3 2.7(6) . . . . ?
C6 C5 C12 S3 -177.2(3) . . . . ?
C15 S3 C12 C13 -1.3(3) . . . . ?
C15 S3 C12 C5 178.1(3) . . . . ?
C5 C12 C13 C14 -178.0(4) . . . . ?
S3 C12 C13 C14 1.3(5) . . . . ?
C12 C13 C14 C15 -0.7(6) . . . . ?
C13 C14 C15 C16 -179.5(4) . . . . ?
C13 C14 C15 S3 -0.3(5) . . . . ?
C12 S3 C15 C16 -179.9(4) . . . . ?
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S3 C15 C16 C17 0.3(8) . . . . ?
C15 C16 C17 C18 -0.1(10) . . . . ?
C16 C17 C18 C19 -173.0(5) . . . . ?
C16 C17 C18 S2 3.0(8) . . . . ?
C21 S2 C18 C19 4.1(3) . . . . ?
C21 S2 C18 C17 -172.3(4) . . . . ?
C17 C18 C19 C20 173.2(4) . . . . ?
S2 C18 C19 C20 -3.4(5) . . . . ?
C18 C19 C20 C21 0.6(6) . . . . ?
C19 C20 C21 C22 -173.1(4) . . . . ?
C19 C20 C21 S2 2.5(5) . . . . ?
C18 S2 C21 C22 171.9(4) . . . . ?
C18 S2 C21 C20 -3.8(3) . . . . ?
C20 C21 C22 C29 175.4(4) . . . . ?
S2 C21 C22 C29 0.5(6) . . . . ?
C20 C21 C22 C23 0.5(6) . . . . ?
S2 C21 C22 C23 -174.4(3) . . . . ?
C21 C22 C23 C24 -78.5(6) . . . . ?
C29 C22 C23 C24 105.9(5) . . . . ?
C21 C22 C23 C28 100.8(5) . . . . ?
C29 C22 C23 C28 -74.7(6) . . . . ?
C28 C23 C24 C25 -1.2(7) . . . . ?
C22 C23 C24 C25 178.2(4) . . . . ?
C23 C24 C25 C26 -0.9(8) . . . . ?
C23 C24 C25 Cl1 -179.9(4) . . . . ?
C24 C25 C26 C27 1.8(8) . . . . ?
Cl1 C25 C26 C27 -179.2(4) . . . . ?
C25 C26 C27 C28 -0.6(8) . . . . ?
C26 C27 C28 C23 -1.4(8) . . . . ?
C24 C23 C28 C27 2.3(8) . . . . ?
C22 C23 C28 C27 -177.0(5) . . . . ?
C21 C22 C29 C30 -177.7(4) . . . . ?
C23 C22 C29 C30 -2.8(7) . . . . ?
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C21 C22 C29 S4 0.7(6) . . . . ?
C23 C22 C29 S4 175.6(3) . . . . ?
C32 S4 C29 C22 -178.0(4) . . . . ?
C32 S4 C29 C30 0.7(4) . . . . ?
C22 C29 C30 C31 178.4(4) . . . . ?
S4 C29 C30 C31 -0.2(5) . . . . ?
C29 C30 C31 C32 -0.4(7) . . . . ?
C30 C31 C32 C33 177.0(4) . . . . ?
C30 C31 C32 S4 0.9(6) . . . . ?
C29 S4 C32 C33 -176.7(4) . . . . ?
C29 S4 C32 C31 -0.9(4) . . . . ?
C31 C32 C33 C34 -179.4(5) . . . . ?
S4 C32 C33 C34 -4.2(8) . . . . ?
C32 C33 C34 C1 -7.9(10) . . . . ?
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S1 C1 C34 C33 -1.7(8) . . . . ?

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