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Coremodified Smaragdyrins; First examples of stable meso substituted Expanded Corrole

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Supporting Information :

1. The ^{1}H NMR spectra for **5a, 5b, 5c, 5d, 5e, 5f, 6a, 6b** and **6c**. (16 pages)
2. 2D COSY Spectra for **5a** and **6a** and 2D TCOSY spectra for **6a**. (3 pages)
3. Representative mass spectra for **5a, 5b, 5d, 5e, 6a, 6b** and **6c**. (7 pages)
4. The electronic (a) and fluorescence (b) spectra of **5a** (solid line) and **6a** (dashed line in CH_2Cl_2. Excitation wavelengths are 443nm (**5a**) and 411nm (**6a**). (1 page)
5. Table containing the selected physical data for the new compounds (2 pages)
6. General synthetic procedure (1 page)
7. Table containing the yields of various compounds (1 page)
8. The ORTEP plots showing the cystal structure of **6a** (top: plane view, bottom: side view - phenyl rings are omitted for clarity). (1 page)
9. The ORTEP plot showing two independent molecules of **5a** trapping three solvent methanol molecules with weak interactions in the unit cell. (1 page)
10. The ORTEP plots of Rh(I) Carbonyl complex of **5a.** (top: plane view, bottom: side view) (1 page)
11. Tables of crystal data, structure solution and refinement, atomic coordinates, bond lengths and angles and anisotropic themal parameters for compound **5a**. (15 pages)
12. Tables of crystal data, structure solution and refinement, atomic coordinates, bond lengths and angles and anisotropic themal parameters for compounds **6a**. (9 pages)

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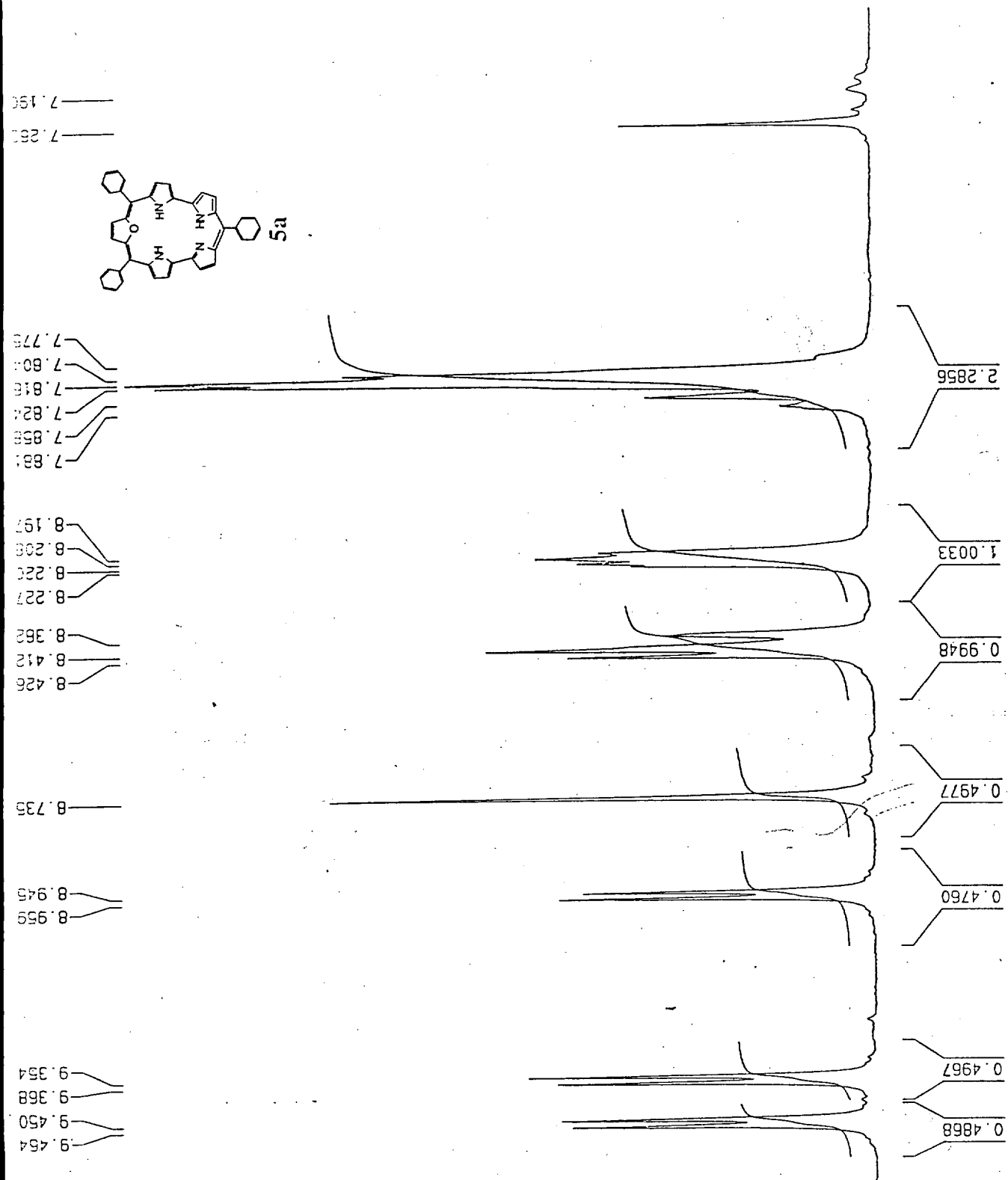

^{1}HNMR (300 MHz) spectrum of **5a** in $CDCl_3$.

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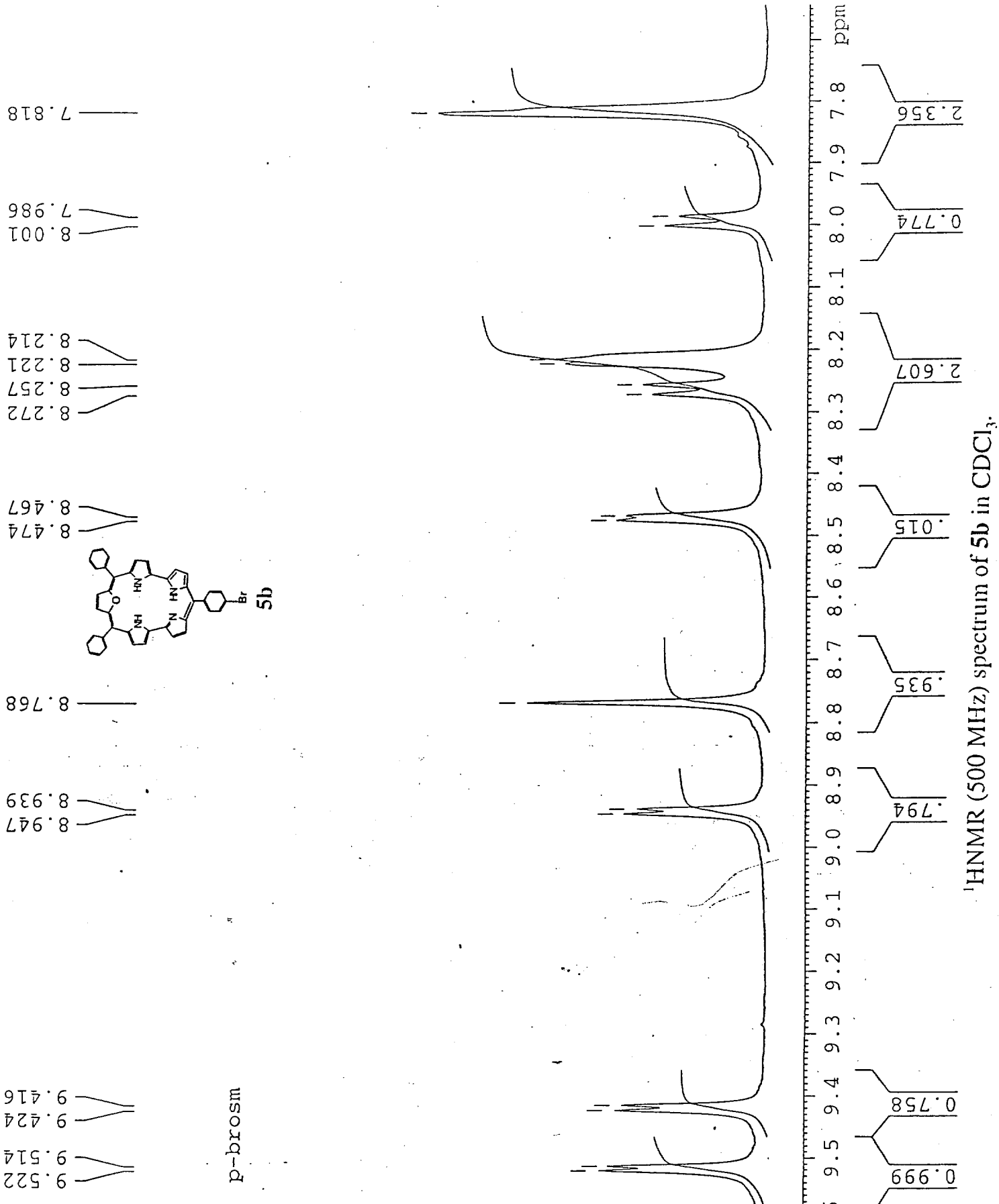
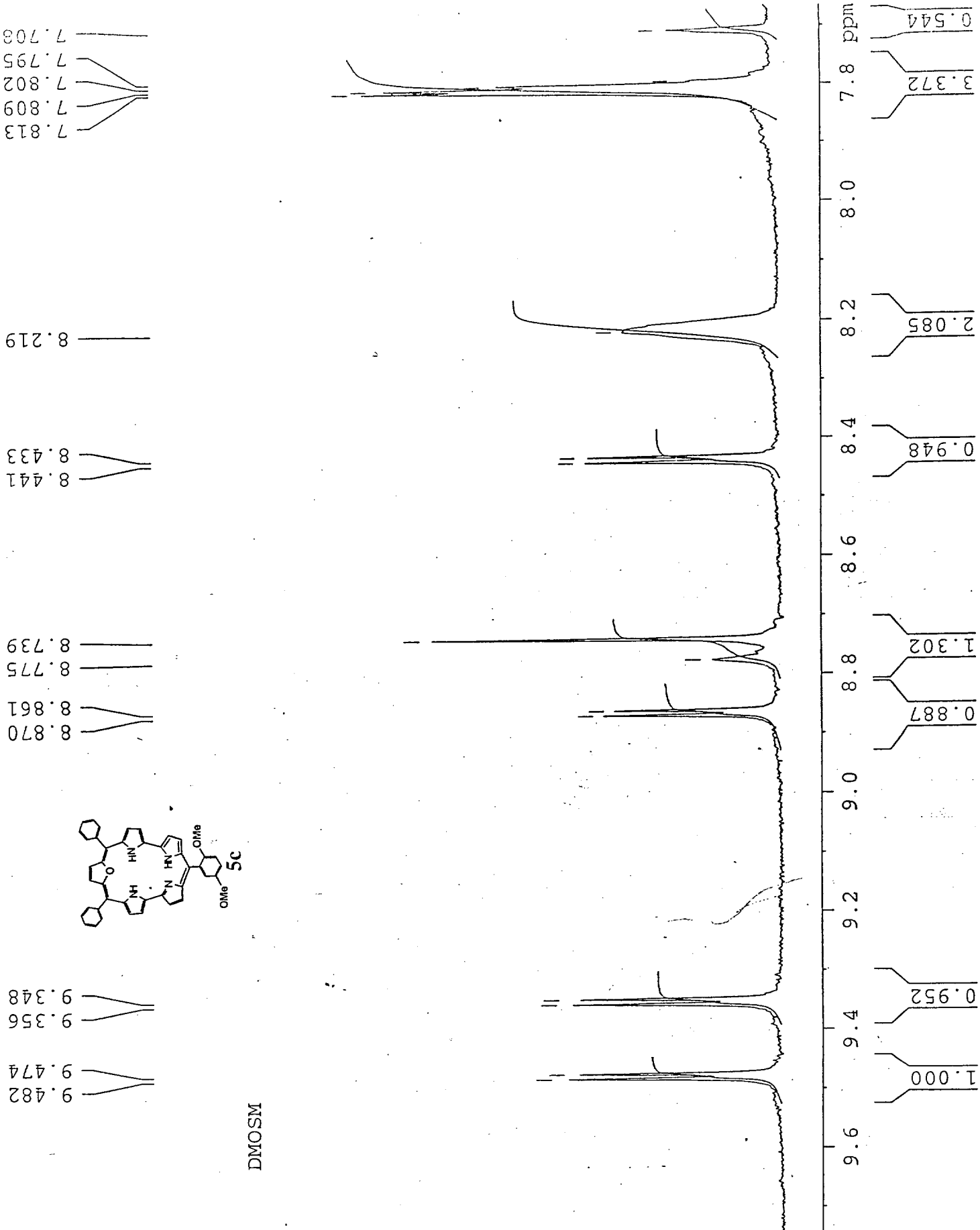


^{1}HNMR (500 MHz) spectrum of **5b** in $CDCl_3$.



^{1}HNMR (500 MHz) spectrum of **5c** in $CDCl_3$.

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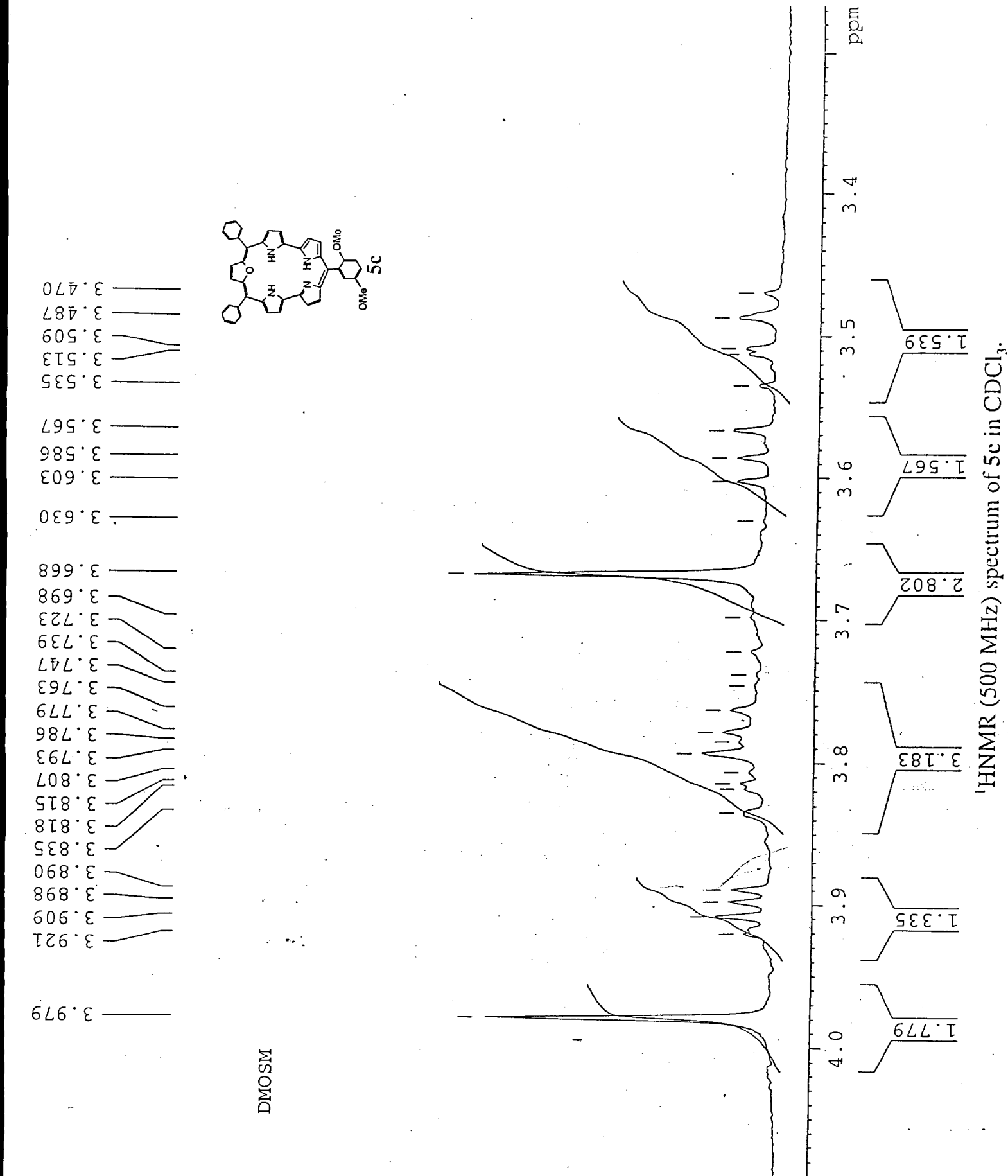

^{1}HNMR (500 MHz) spectrum of **5c** in $CDCl_3$.

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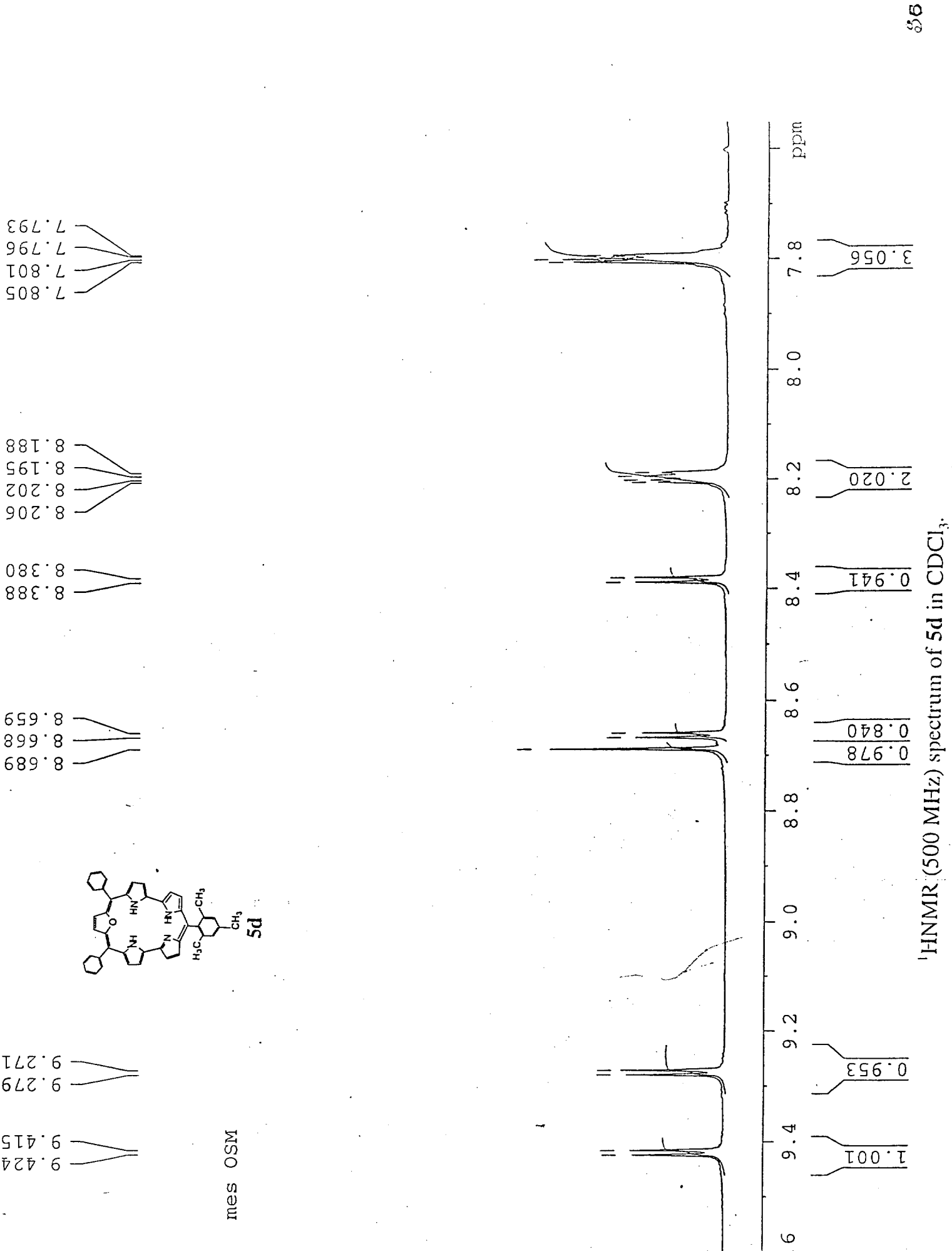

^{1}HNMR (500 MHz) spectrum of **5d** in $CDCl_3$.

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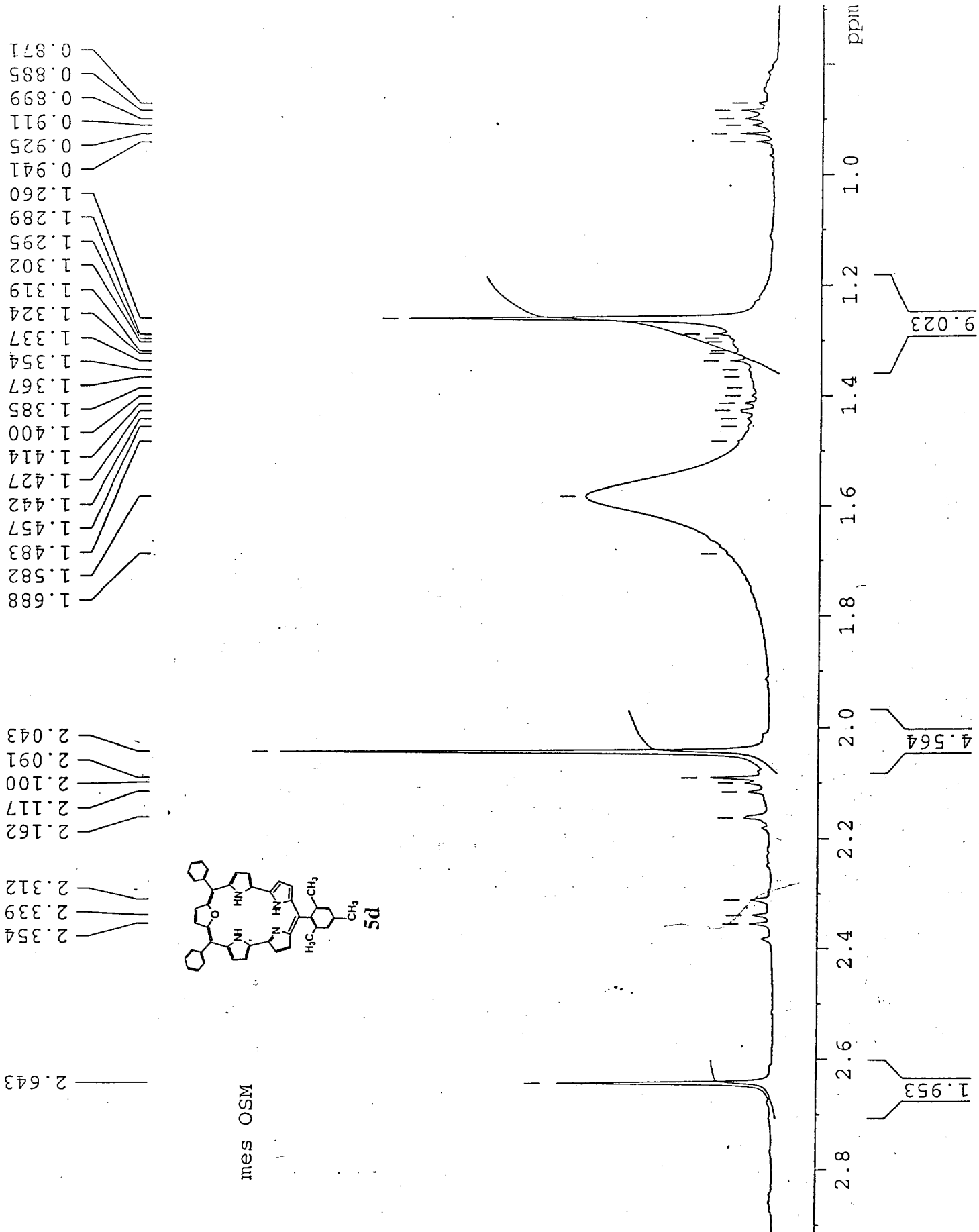
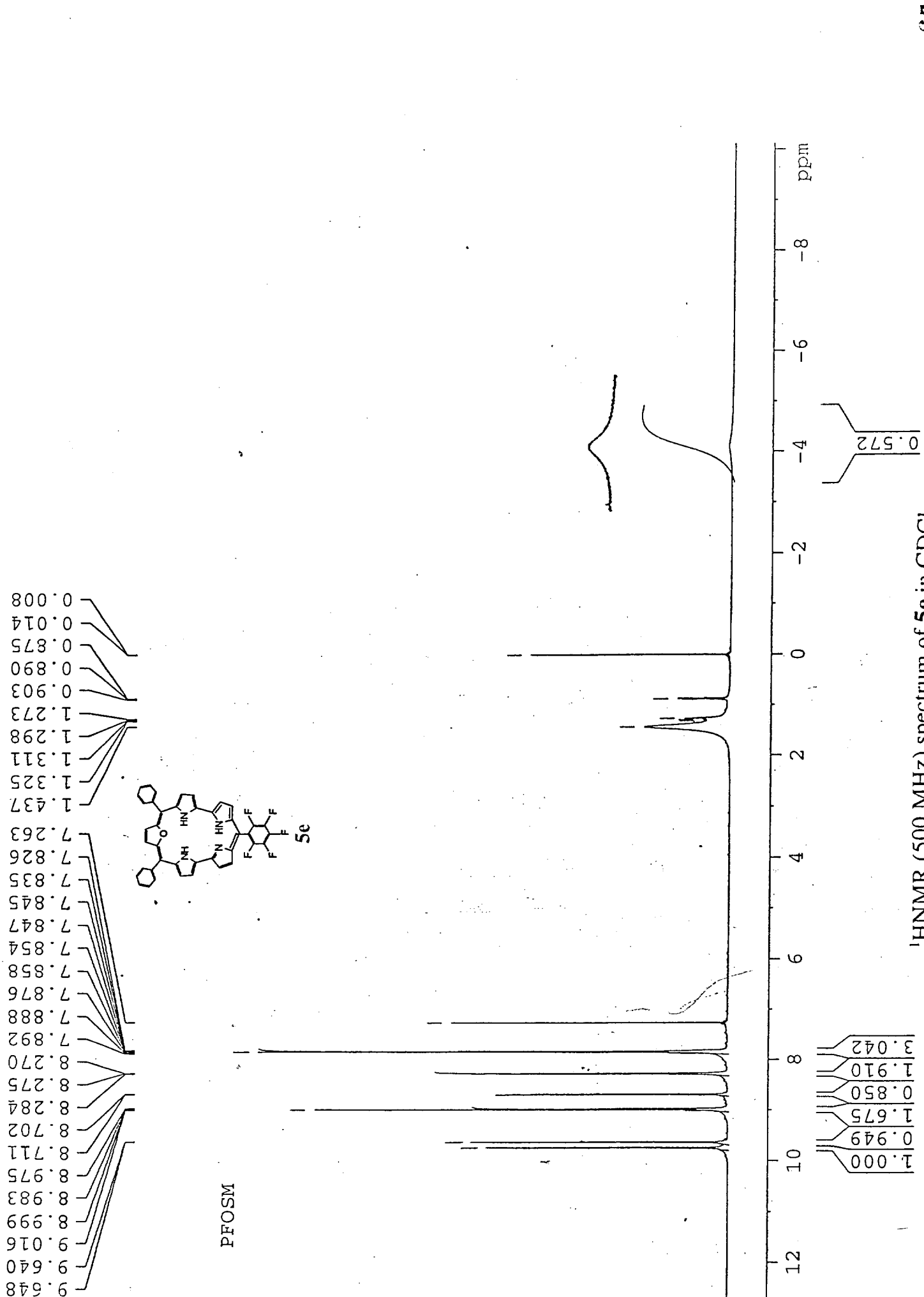


^{1}HNMR (500 MHz) spectrum of **5d** in $CDCl_3$.



^{1}HNMR (500 MHz) spectrum of **5e** in $CDCl_3$.

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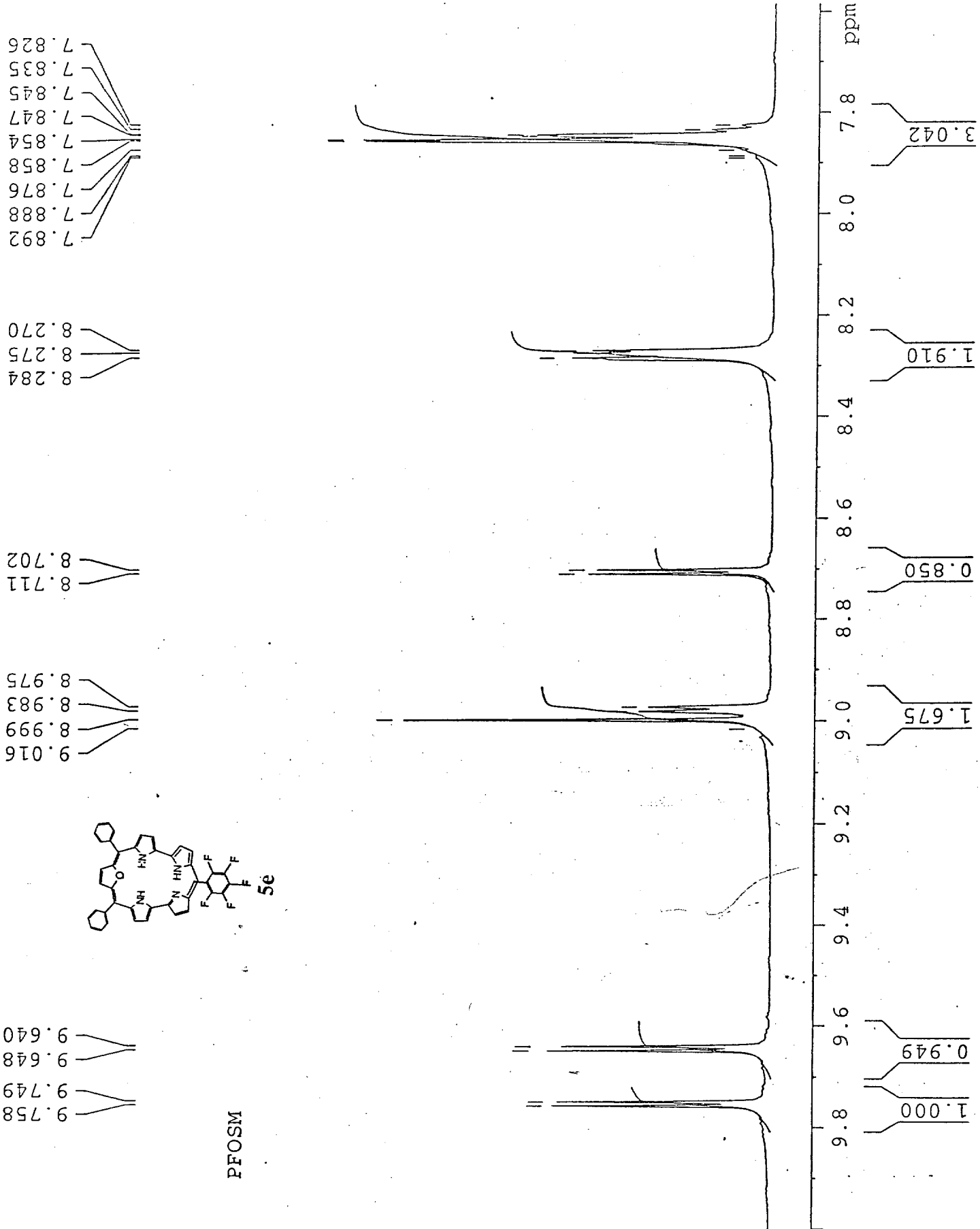

^{1}HNMR (500 MHz) spectrum of **5e** in $CDCl_3$.

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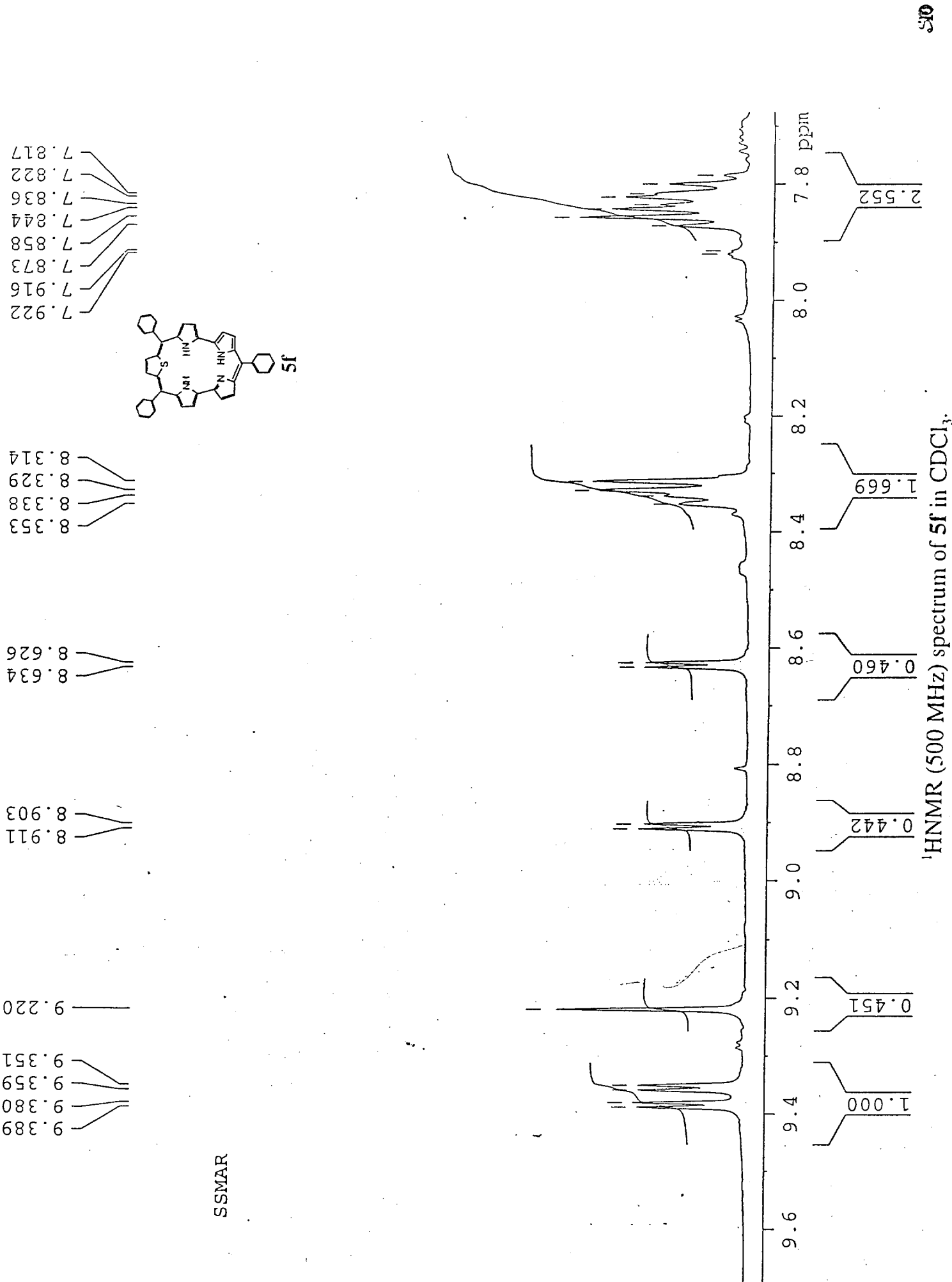

^{1}HNMR (500 MHz) spectrum of **5f** in $CDCl_3$.

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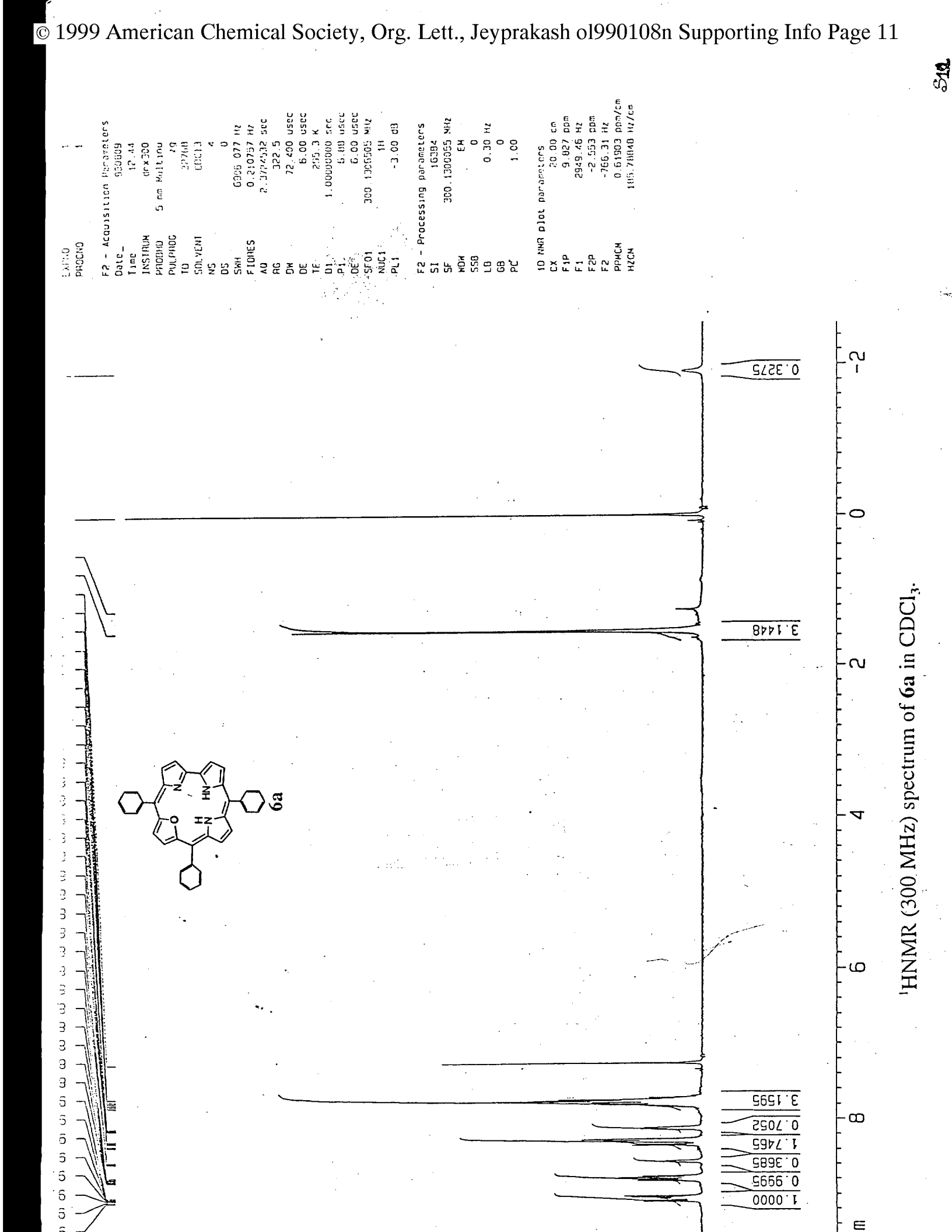
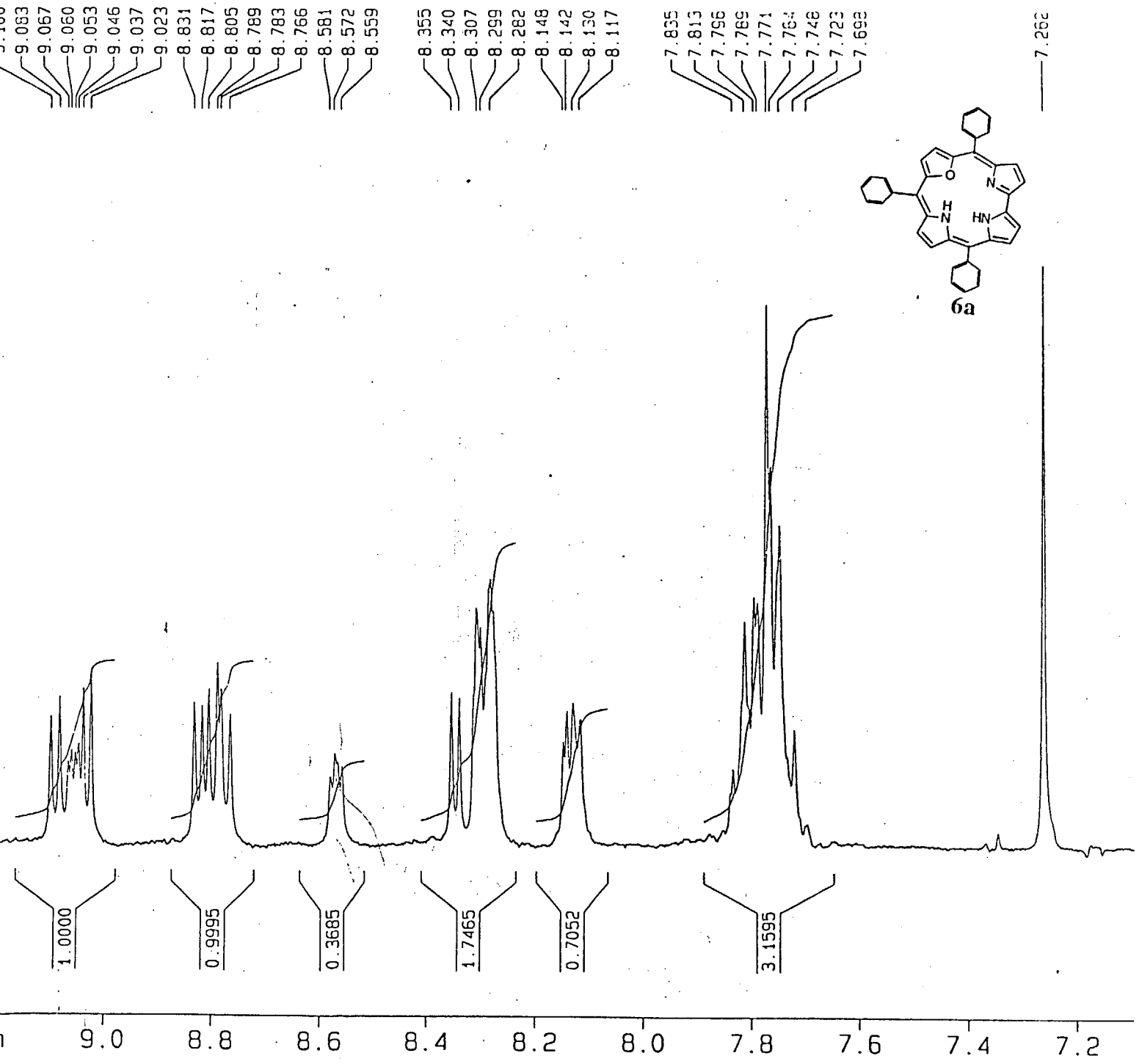


^{1}HNMR (300 MHz) spectrum of **6a** in $CDCl_3$.



6a

^{1}HNMR (300 MHz) spectrum of **6a** in $CDCl_3$.

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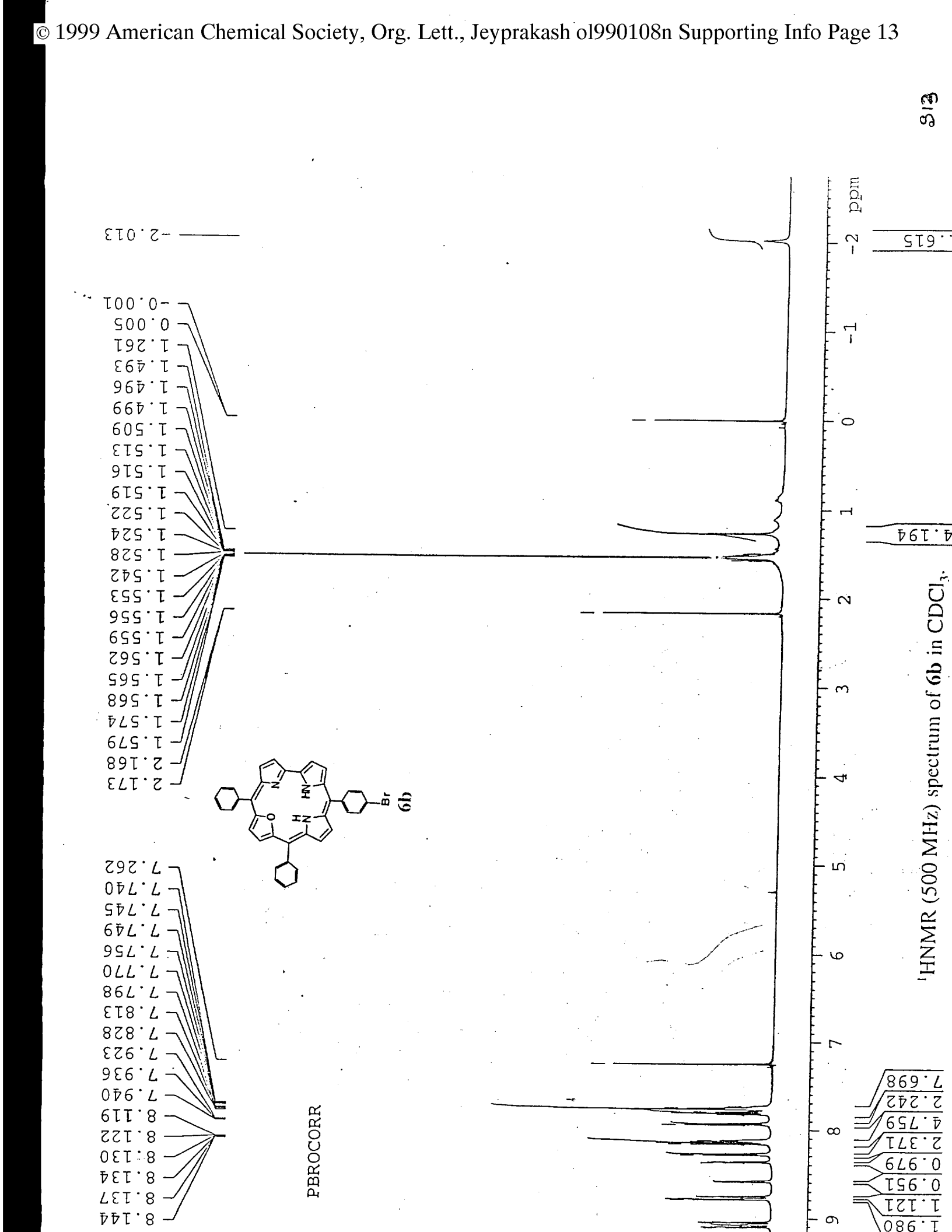
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^{1}HNMR (500 MHz) spectrum of **6b** in $CDCl_3$.

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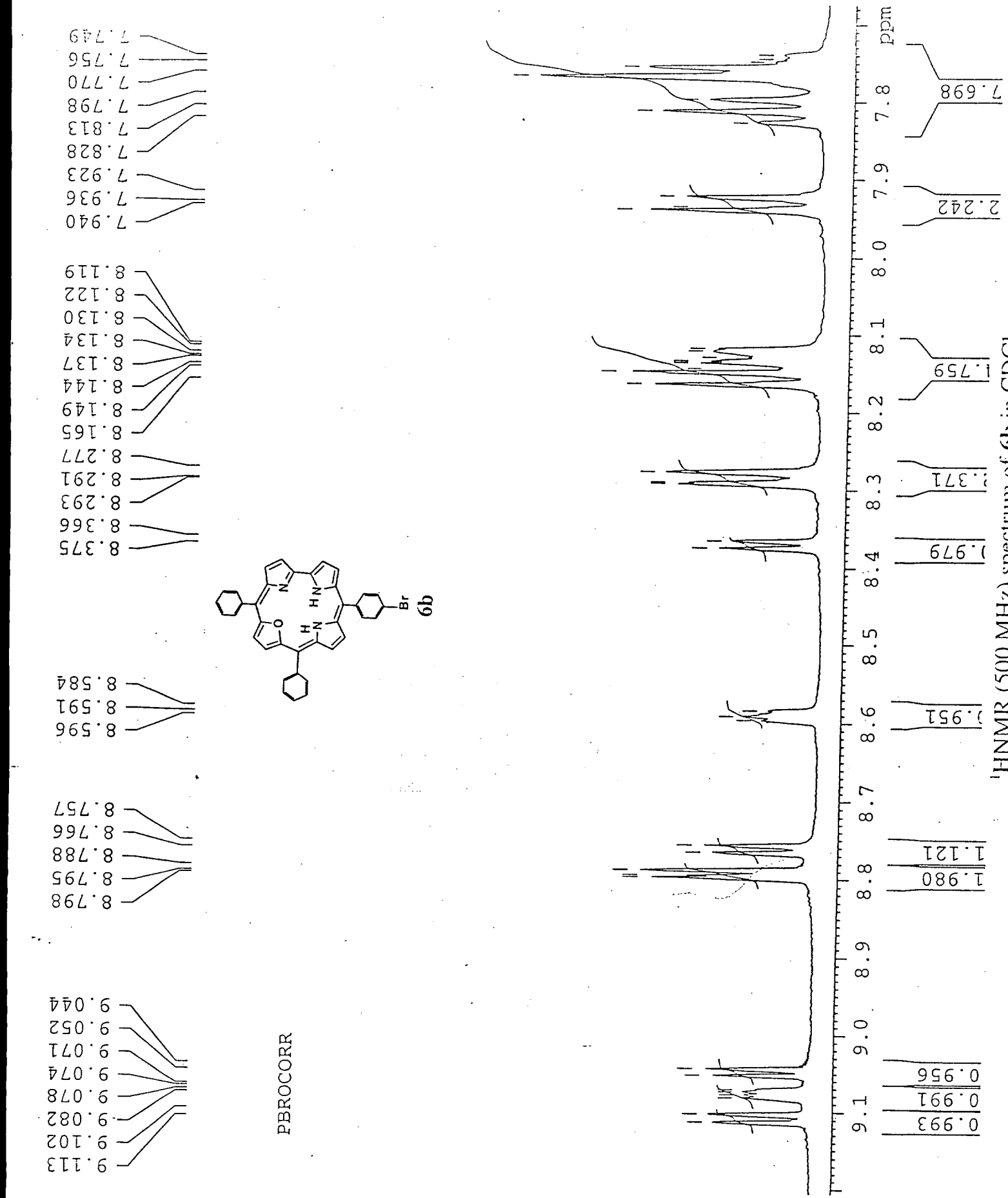

^{1}HNMR (500 MHz) spectrum of **6b** in CDCl

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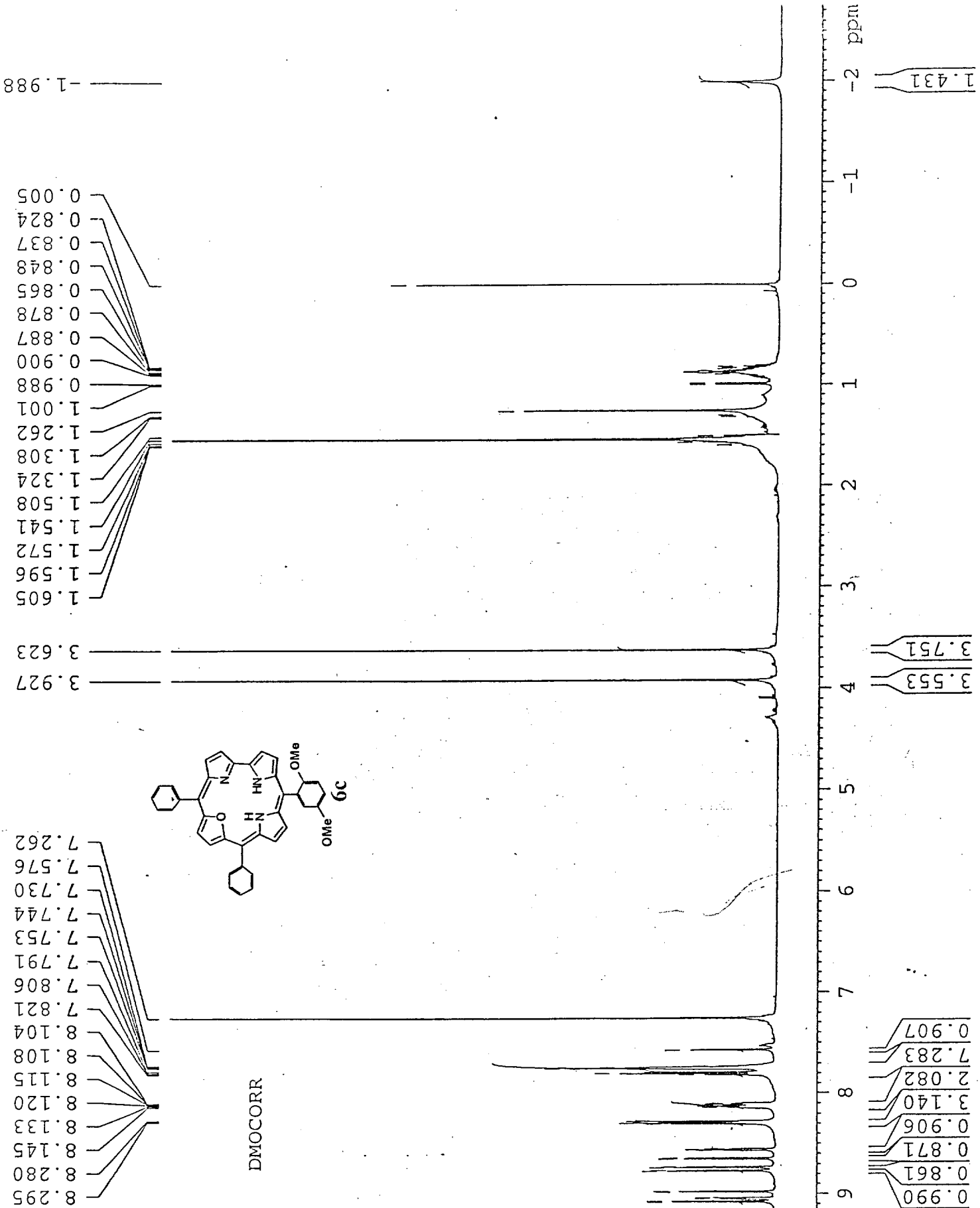

^{1}HNMR (500 MHz) spectrum of **6c** in $CDCl_3$.

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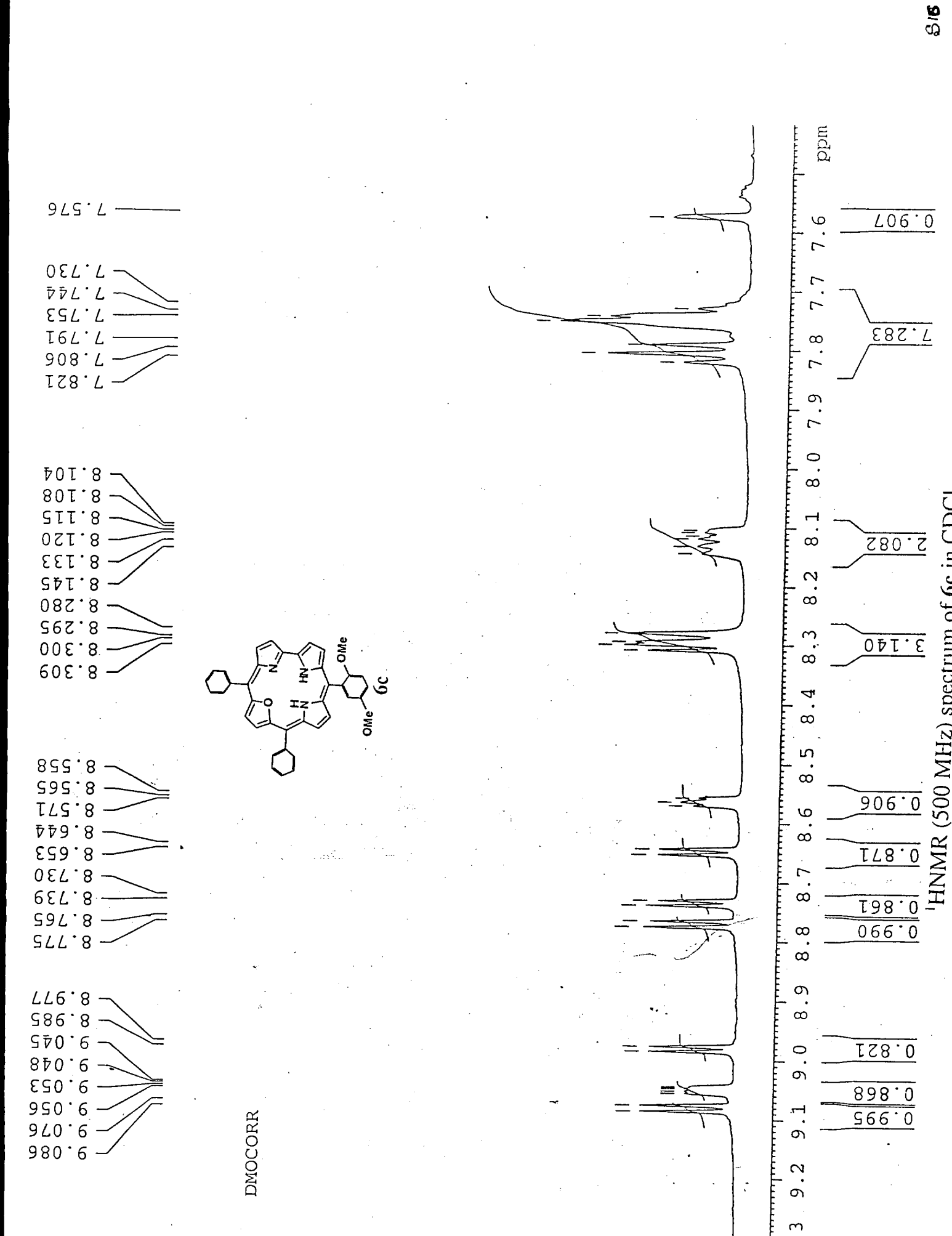

^{1}HNMR (500 MHz) spectrum of **6c** in $CDCl_3$.

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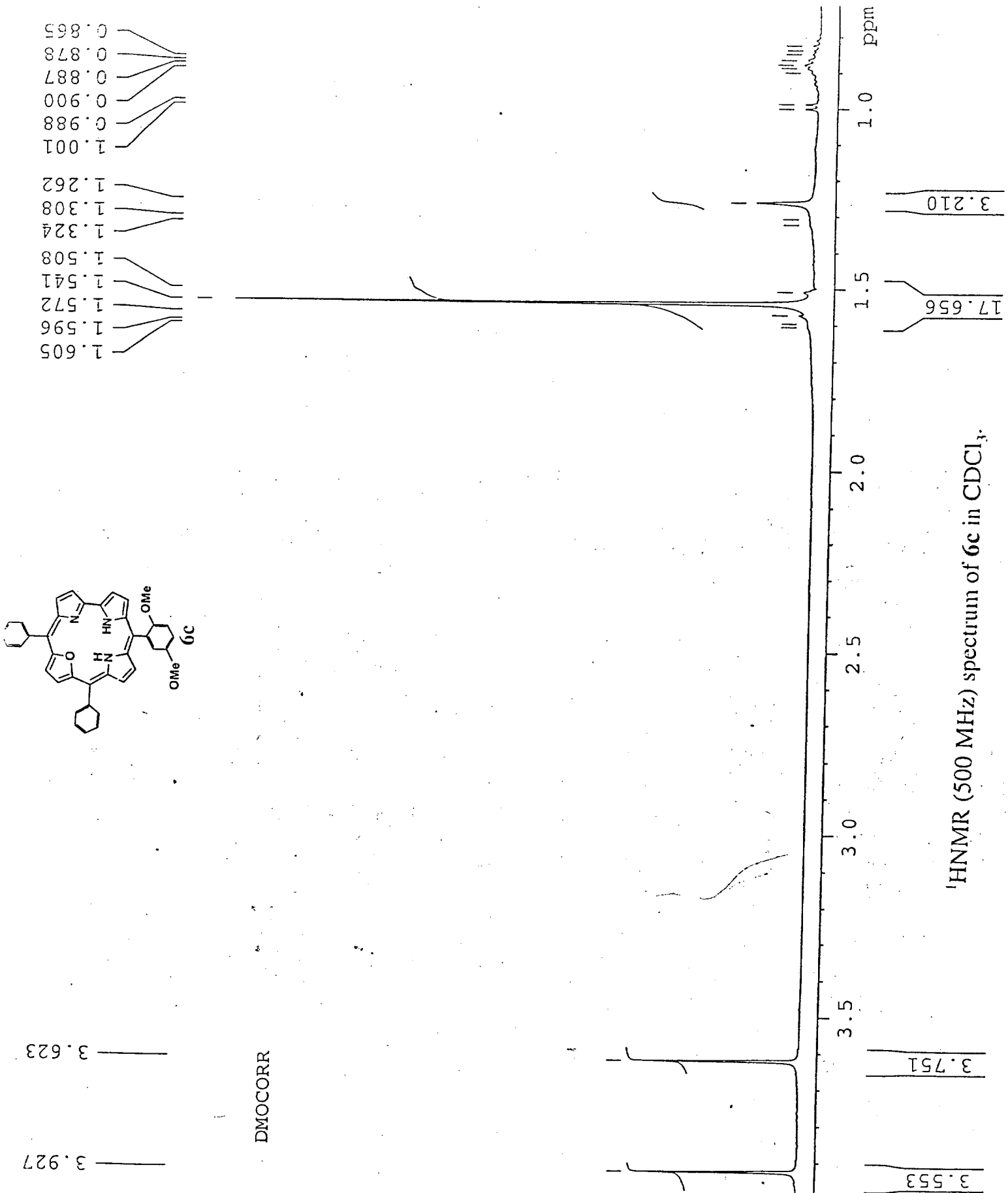

^{1}HNMR (500 MHz) spectrum of **6c** in $CDCl_3$.

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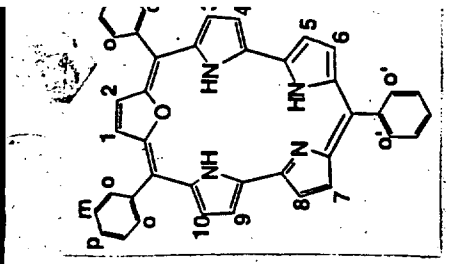

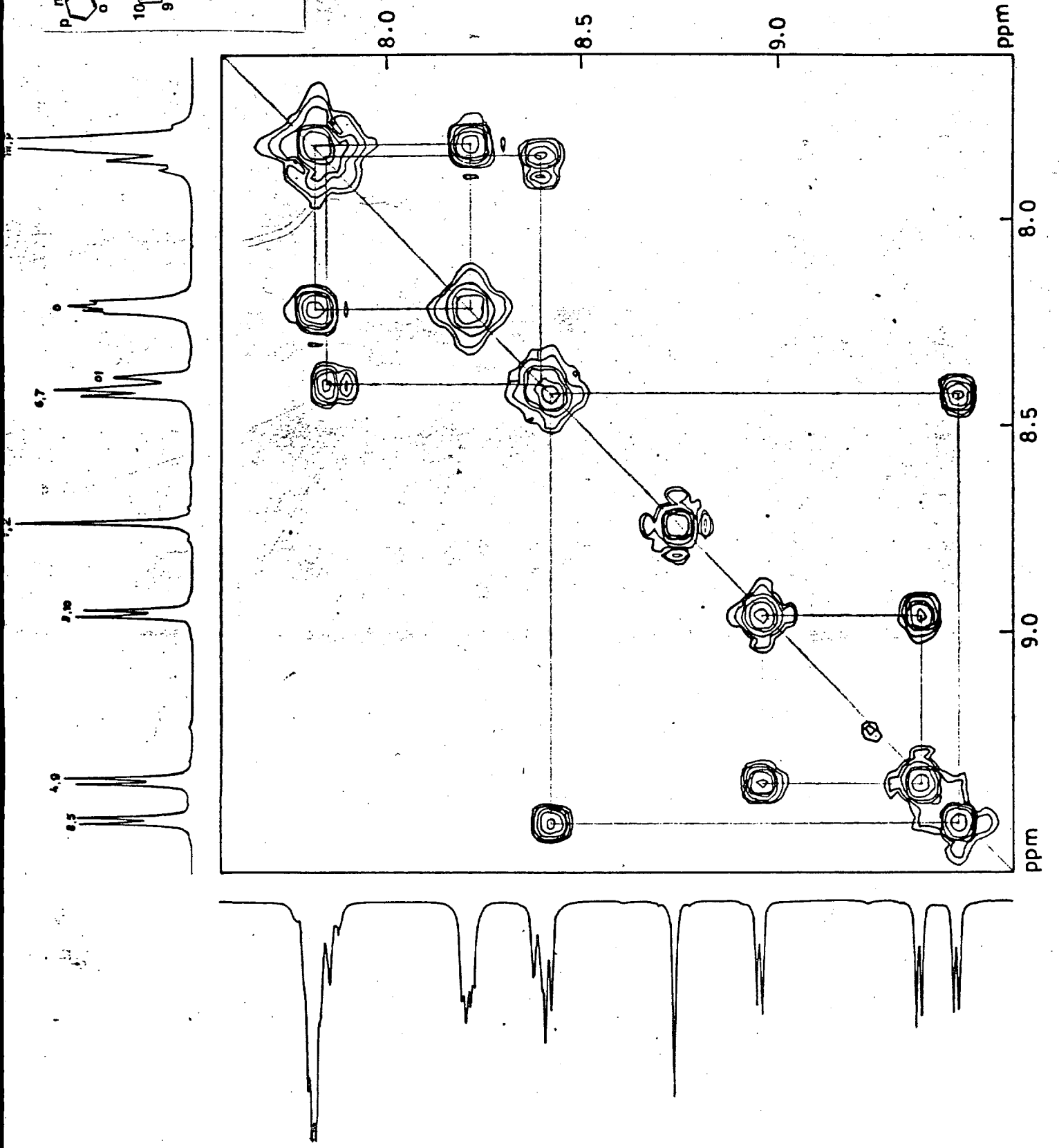

2D-COSY (300 MHz) spectrum of 5a in $CDCl_3$

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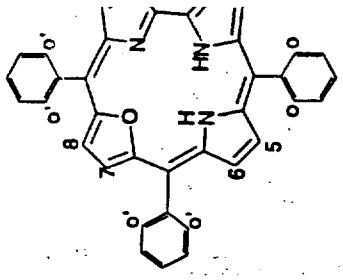

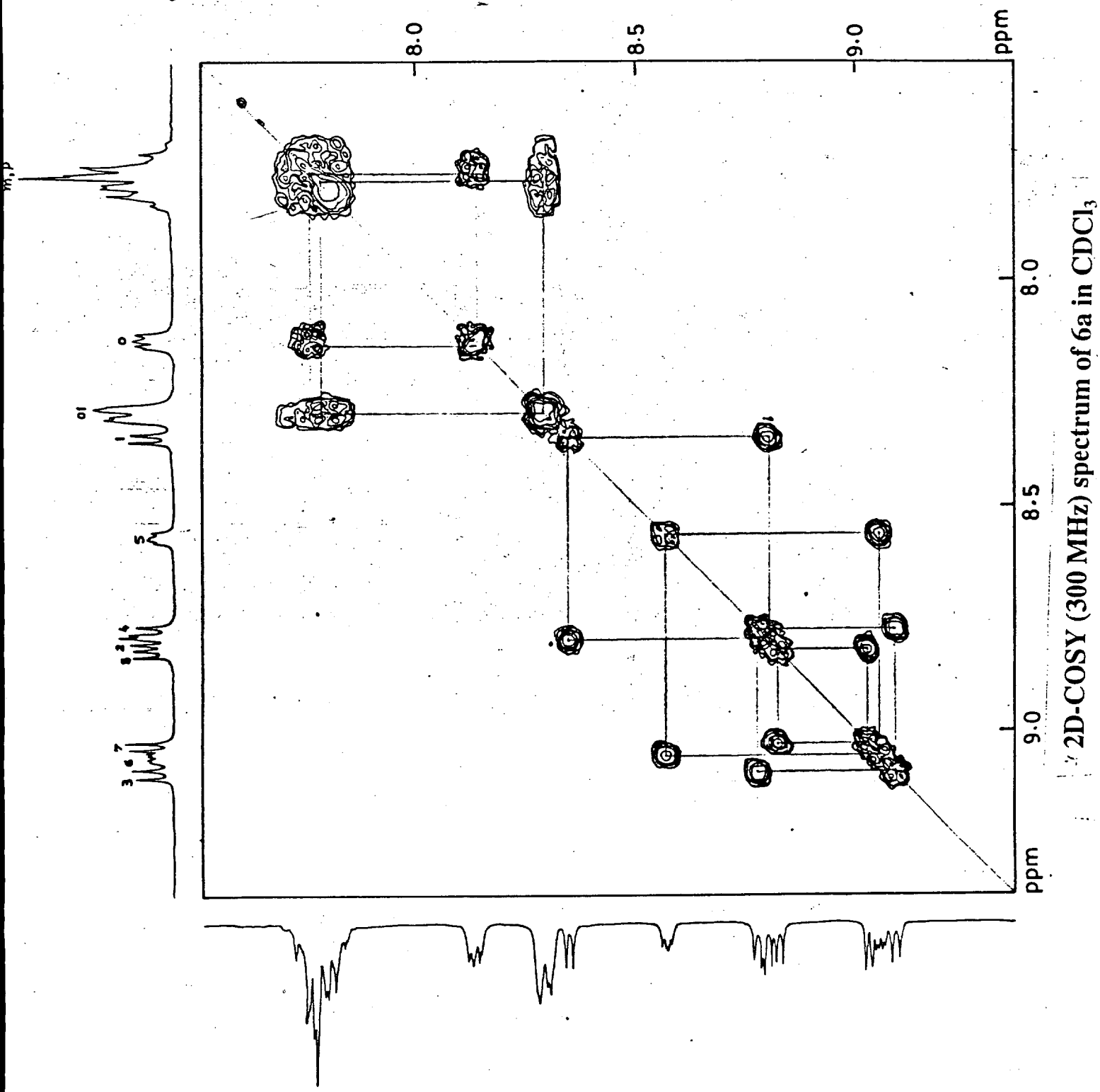

2D-COSY (300 MHz) spectrum of 6a in $CDCl_3$

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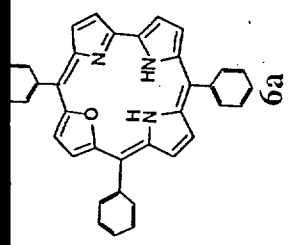

6a

2D-Total COSY (300 MHz) spectrum of **6a** in $CDCl_3$.

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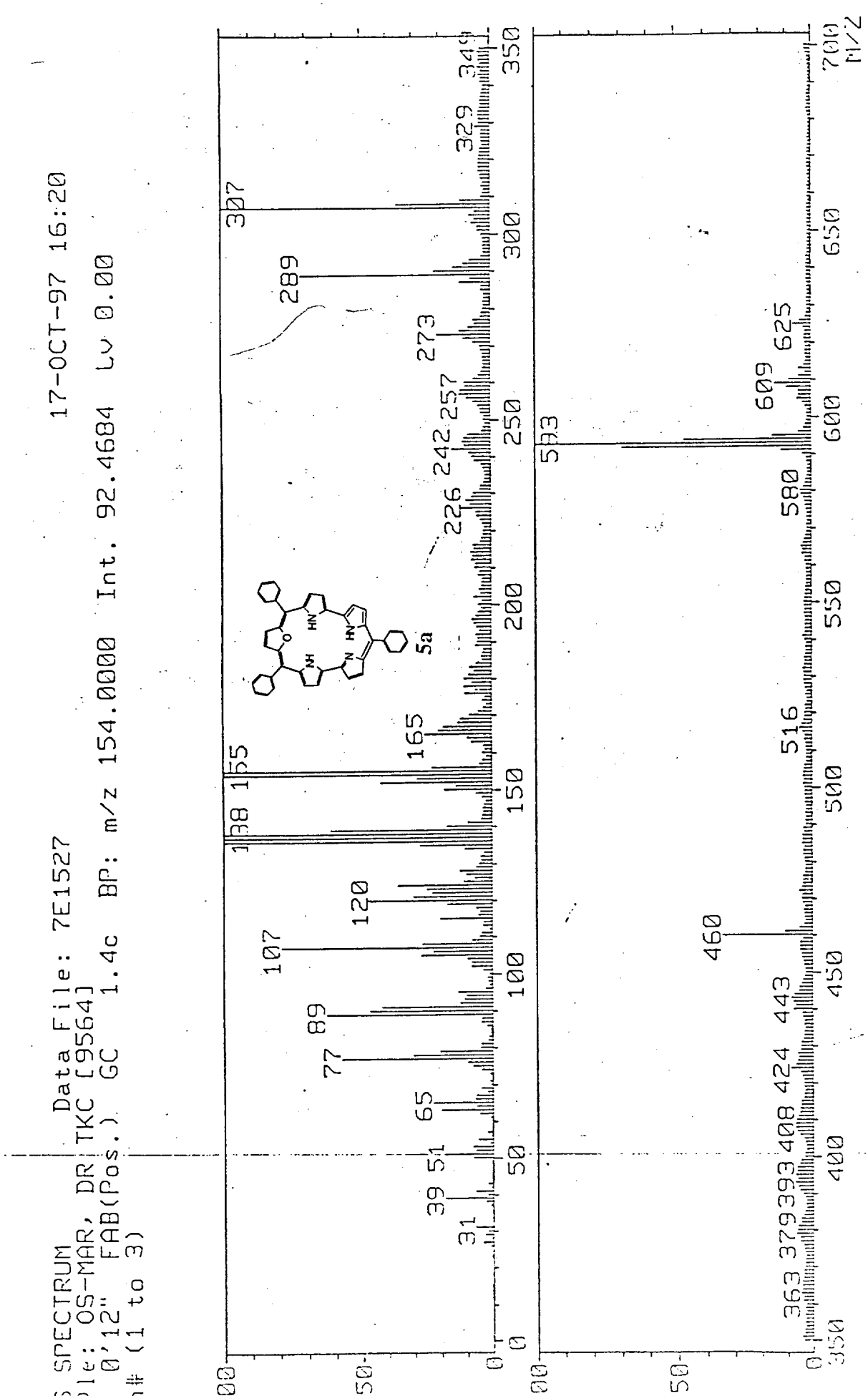
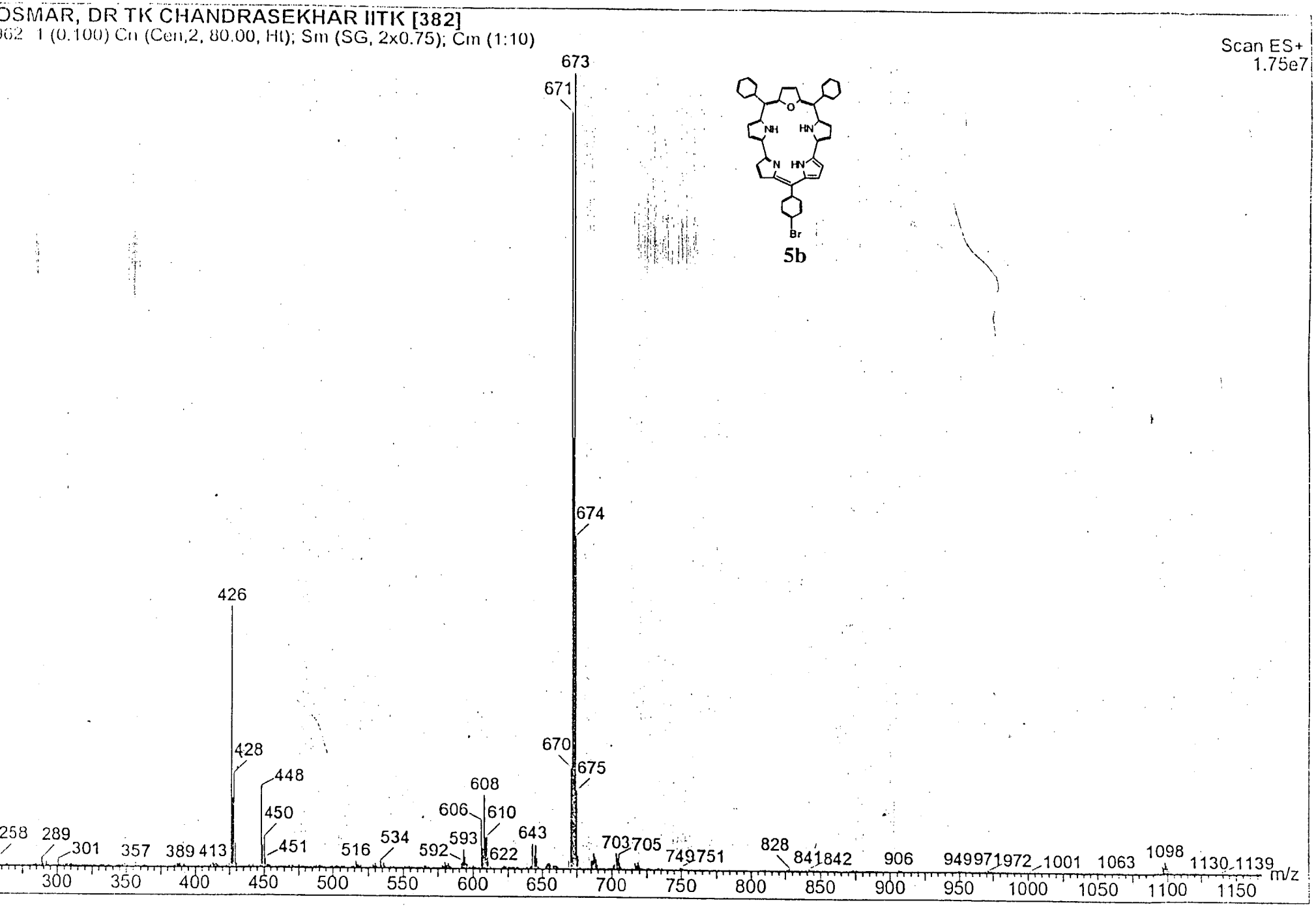


FAB Mass spectrum of **5a**

5a



Electron Spray Mass spectrum of **5b**

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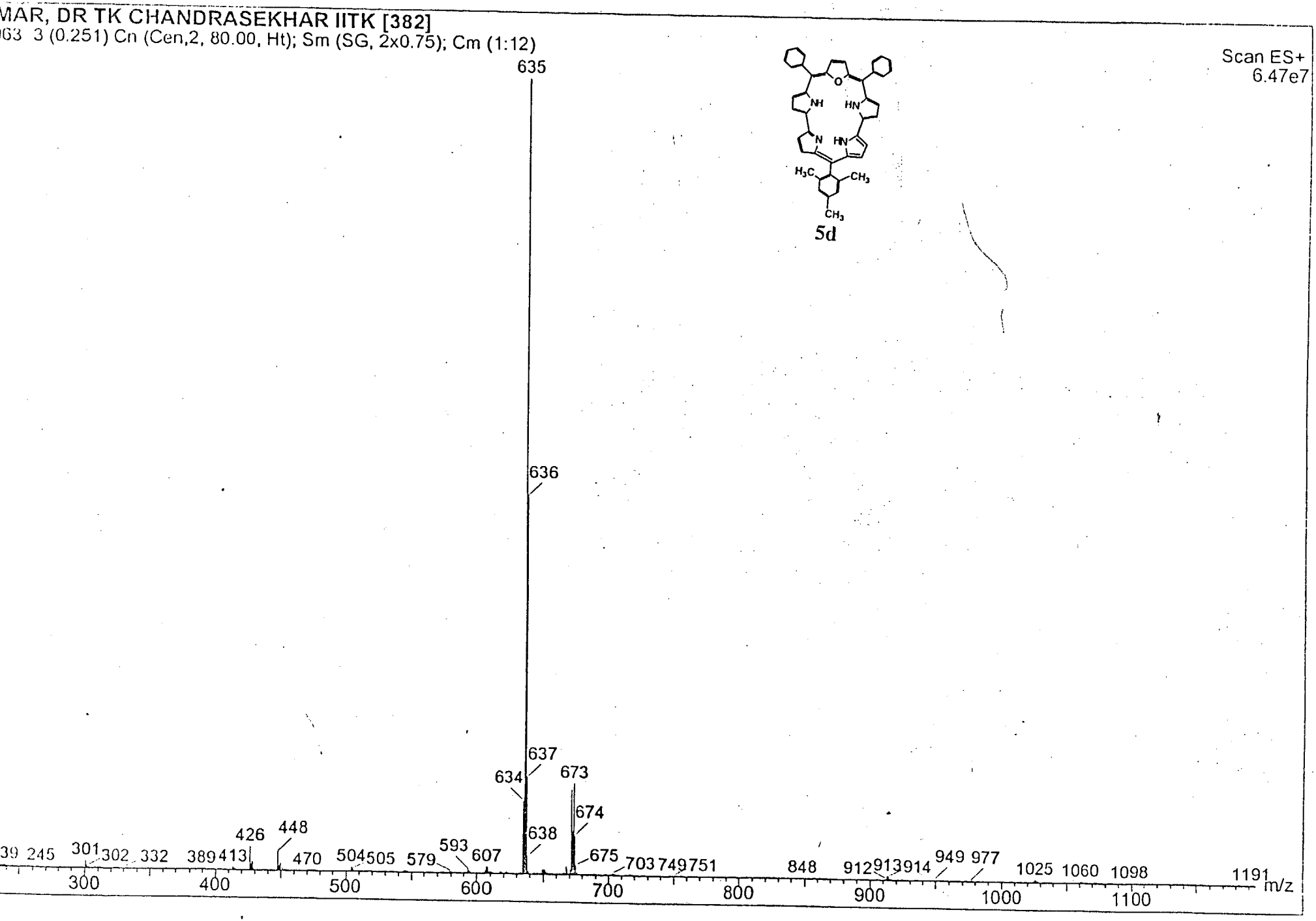

Electron Spray Mass spectrum of **5d**

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SMAR, PROF TK CHANDRASHEKAR [638]
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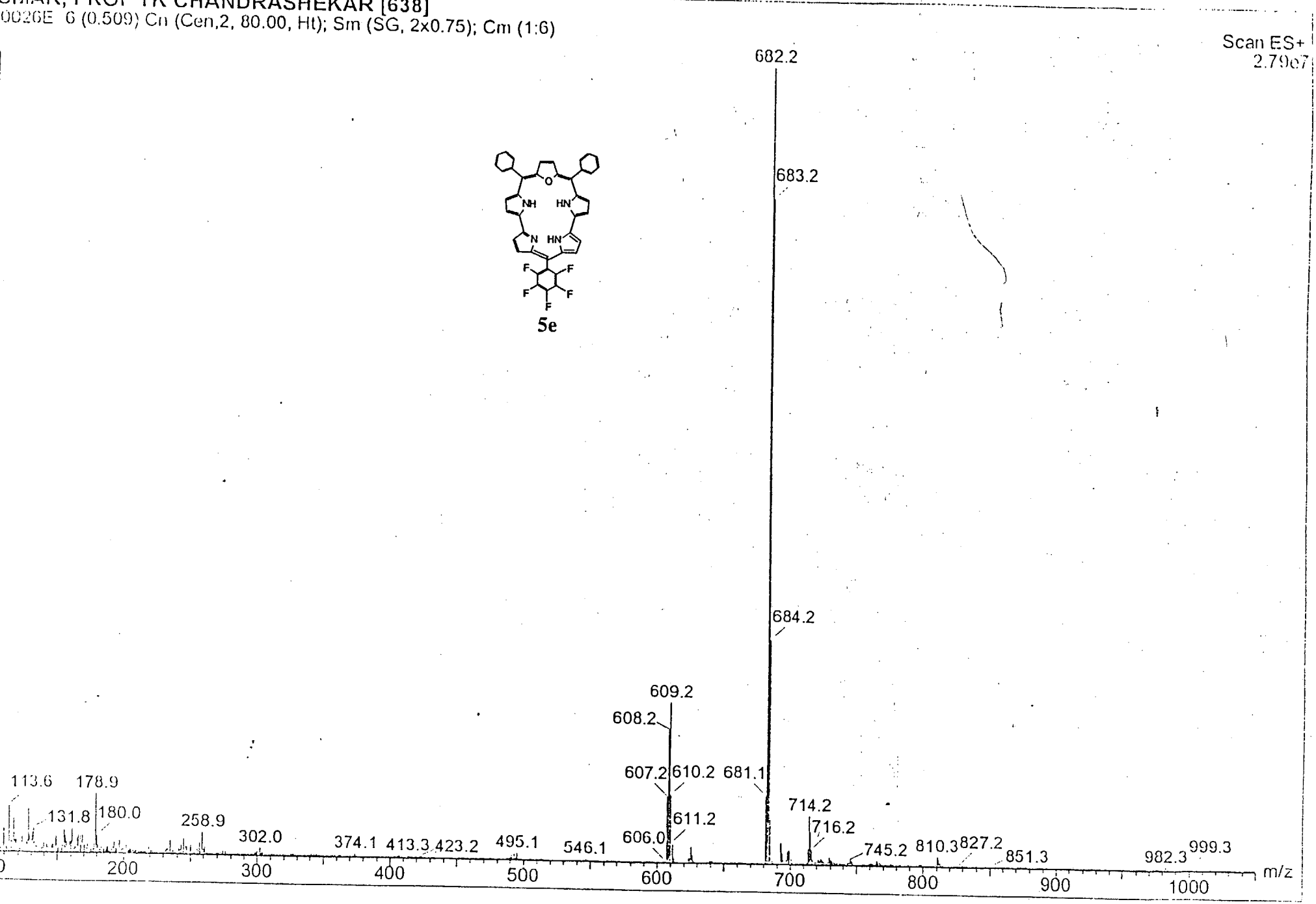

5e

Electron Spray Mass spectrum of **5e**

S24

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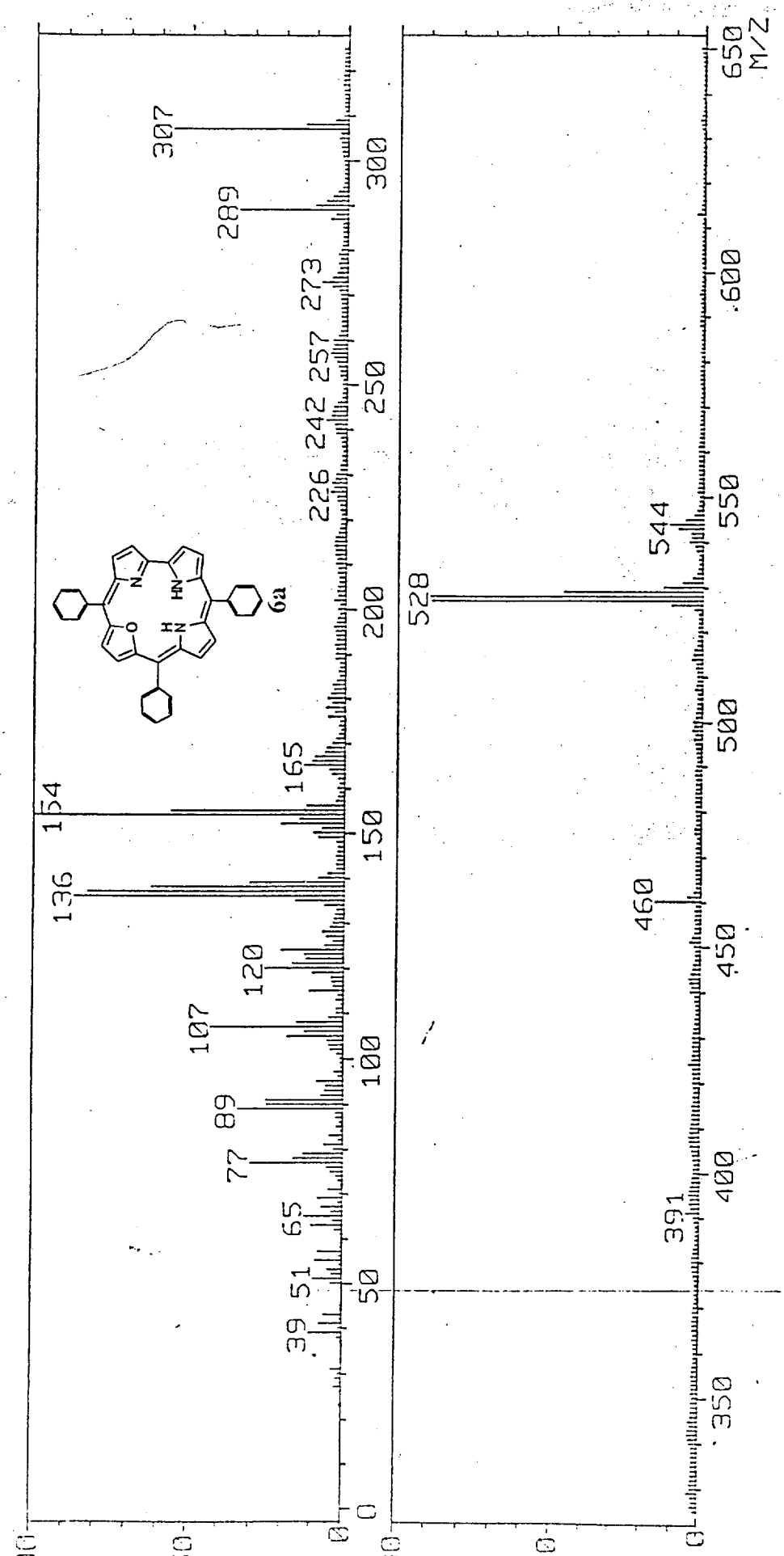
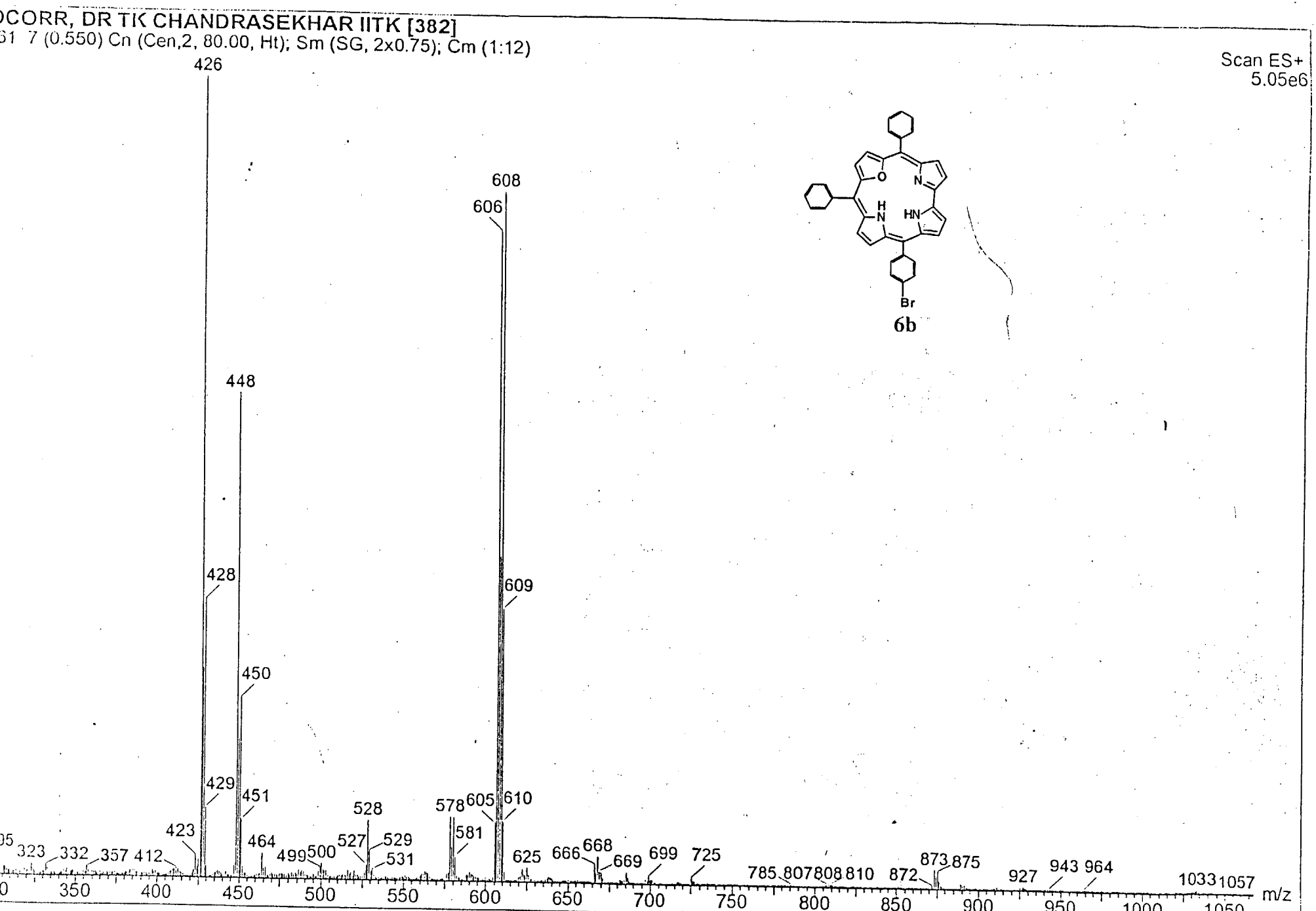


FAB Mass spectrum of **6a**



Electron Spray Mass spectrum of **6b**

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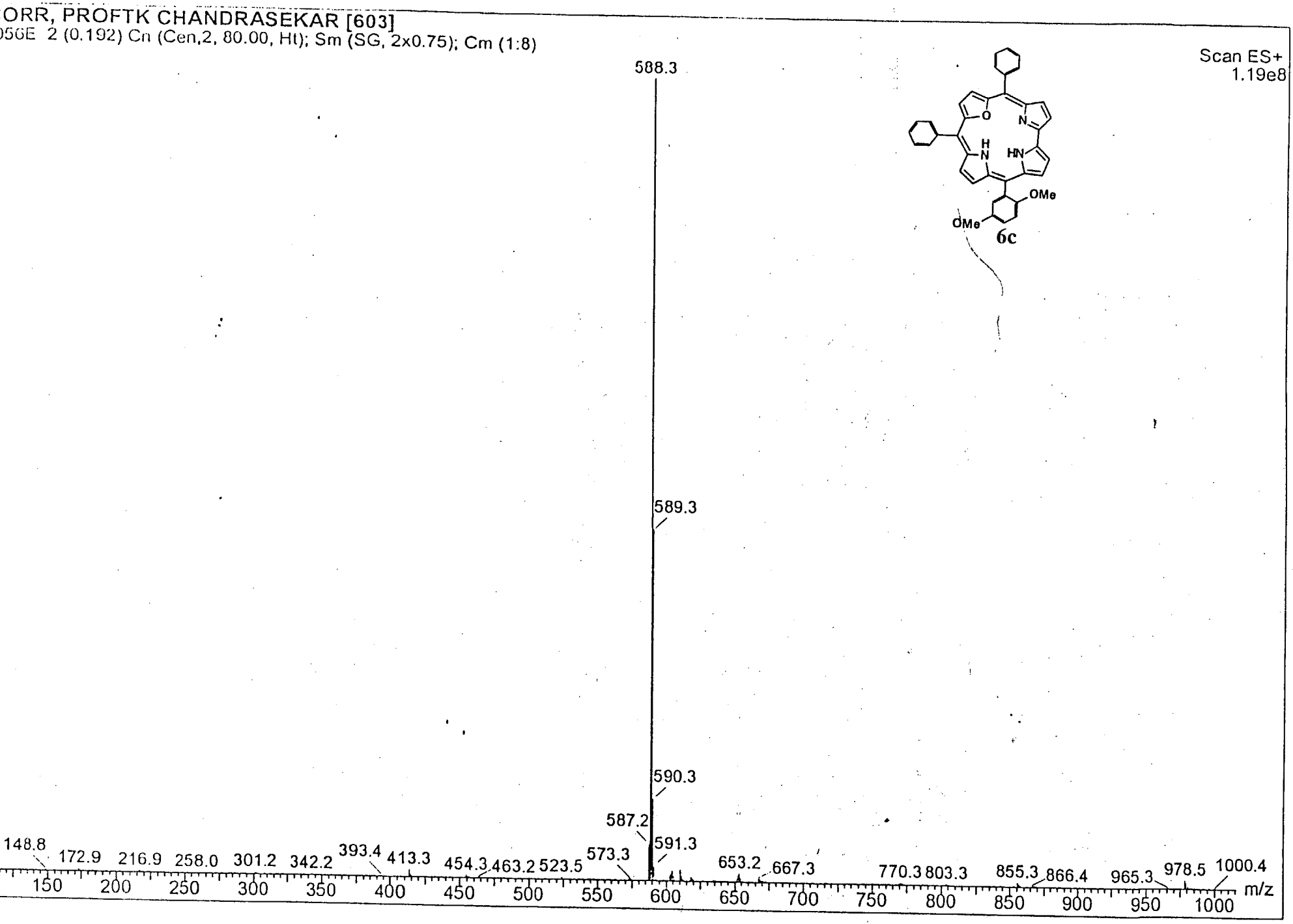

Electron Spray Mass spectrum of **6c**

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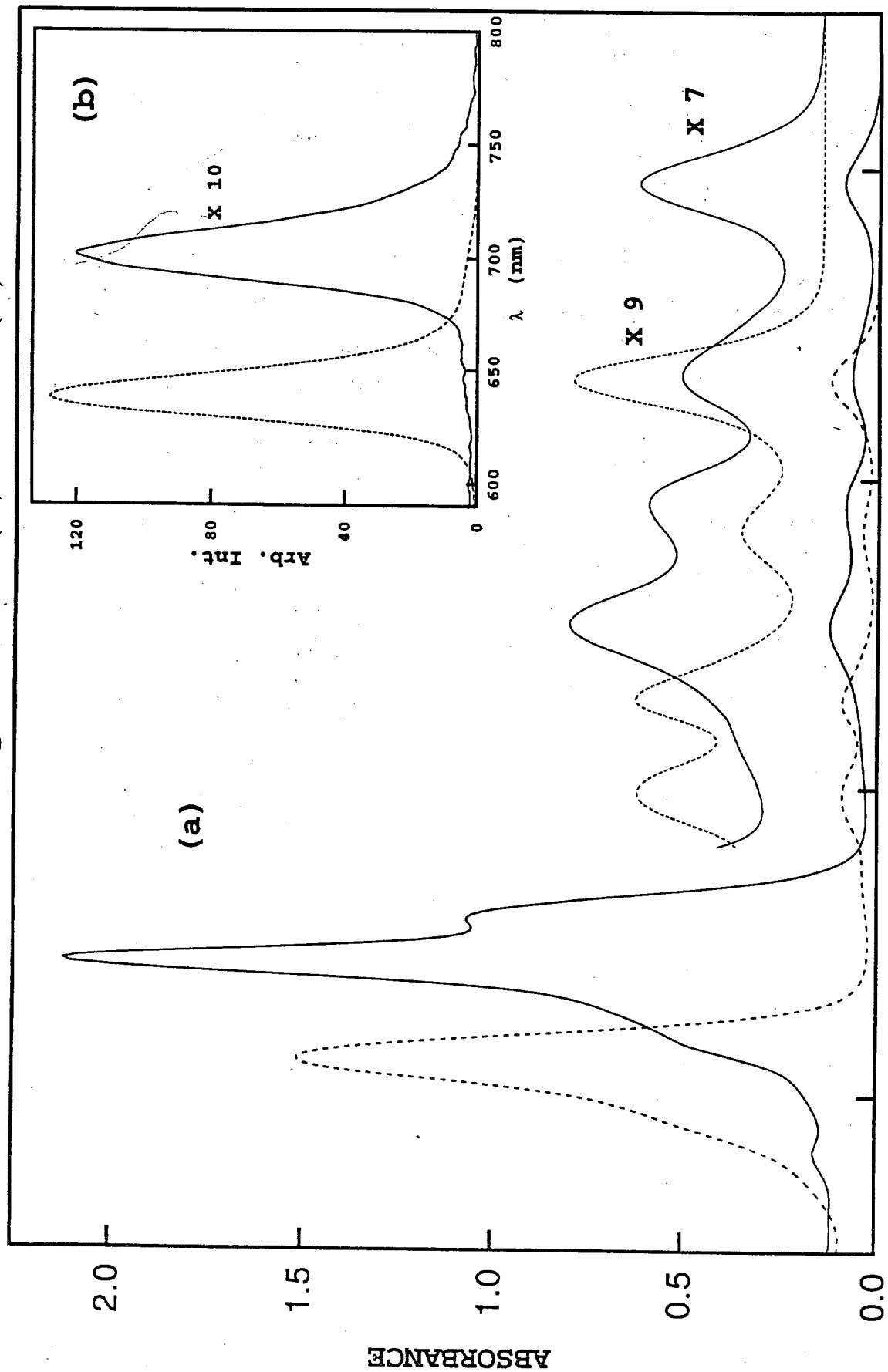

The electronic (a) and fluorescence (b) spectra of 5a (solid line) and 6a (dashed line) in CH_2Cl_2. Excitation wavelengths are 443nm (5a) and 411nm (6a).

Selected physical data for the new compounds :

5a: ^{1}HNMR (300 MHz, $CDCl_3$, 25°C, TMS): δ=7.83 (m, 9H), 8.21 (m, 4H), 8.38 (m, 2H), 8.42 (d, 2H, J=4.2Hz), 8.74 (s, 2H), 8.95 (d, 2H, J=4.2 Hz), 9.36 (d, 2H, J=4.2 Hz), 9.46 (d, 2H, J=4.2 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 443 (33.0), 456sh (16.0), 552 (2.0), 591 (1.4), 633 (1.0), 696nm (1.4); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 450 (27.9), 482 (13.5), 605 (1.8), 657 (2.4), 720nm (4.6); Emission (CH_2Cl_2): λ=701nm; MS(FAB): m/z (%): 593 (100) [M$^+$]

5b: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=7.82 (m, 6H), 7.99 (d, 2H, J=7.5 Hz), 8.22 (m, 4H), 8.26 (d, 2H, J=7.5Hz), 8.47 (d, 2H, J=4.0Hz), 8.77 (s, 2H), 8.94 (d, 2H, J=4.0 Hz), 9.42 (d, 2H, J=4.0 Hz); 9.52 (d, 2H, J=4.0 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 444 (24.0), 457sh (11.9), 554 (1.46), 594 (1.17), 637 (1.0), 699nm (1.17); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 452 (19.6), 483 (8.9), 606 (1.17), 662 (1.84), 724nm (3.5); Emission (CH_2Cl_2): λ=706nm; MS(Electron Spray): m/z (%): 673 (100) [M +1]

5c: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=3.67 (s,3H), 3.97 (s,3H), 7.71 (s, 1H), 7.80 (m, 8H), 8.22 (m, 4H), 8.44 (d, 2H, J=4.0 Hz), 8.74 (s, 2H), 8.87 (d, 2H, J=4.5 Hz), 9.35 (d, 2H, J=4.0 Hz), 9.48 (d, 2H, J=4.0 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 444 (32.0), 552 (2.6), 592 (2.0), 634 (1.8), 696nm (1.8); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 450 (28.0), 479 (16.6), 605 (3.1), 658 (4.4), 712nm (5.9); Emission (CH_2Cl_2): λ=701nm; MS (Electron Spray): m/z (%): 653 (100) [M$^+$]

5d: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=2.04 (s,3H), 2.64 (s, 6H), 7.31 (s, 2H), 7.80 (m, 6H), 8.20 (m, 4H), 8.38 (d, 2H, J=4.0 Hz), 8.66 (d, 2H, J=4.5 Hz), 8.69 (s, 2H), 9.28 (d, 2H, J=4.0 Hz), 9.42 (d, 2H, J=4.5 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 442 (14.88), 456sh (7.71), 551 (0.88), 590 (0.62), 631 (0.54), 692nm (0.56); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 448 (13.09), 477 (6.19), 600 (0.97), 656 (1.38), 707nm (2.24); Emission (CH_2Cl_2): λ=697nm ;MS(FAB) : m/z (%): 635 (100) [M$^+$]

5e: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=-4.1 (s,1H), 7.86 (m, 6H), 8.28 (m, 4H), 8.71 (d, 2H, J=4.5 Hz), 8.98 (d, 2H, J=4.0 Hz), 9.00 (s,2H), 9.64 (d, 2H, J=4.0 Hz), 9.75 (d, 2H, J=4.5 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 445 (23.7), 459sh (12.8), 558 (1.30), 600 (1.20), 645 (0.90), 710nm (1.65); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 450 (20.6), 481 (9.9), 611 (1.3), 667 (2.2), 714nm (3.6); Emission (CH_2Cl_2): λ=716nm; MS(Electron Spray): m/z (%): 682 (100) [M$^+$]

5f: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=7.85 (m, 9H), 8.32 (m, 4H), 8.35 (m, 2H), 8.63 (d, 2H, J=1.0 Hz), 8.91 (d, 2H, J=4.0 Hz), 9.22 (s, 2H), 9.36 (d, 2H, J=4.0 Hz), 9.38 (d, 2H, J=4.5 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 453 (12.6), 557 (1.11), 590 (0.77), 643 (0.57), 710 (0.46), 823nm (0.19); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 478 (10.85), 616 (0.86), 671 (0.86), 878sh (0.95), 927nm (1.34); Emission (CH_2Cl_2): λ=725nm; MS(Electron Spray): m/z (%): 609 (85) [M$^+$]

6a: ^{1}HNMR (300 MHz, $CDCl_3$, 25°C, TMS): δ=-1.91 (s,1H), 7.77 (m, 9H), 8.13 (m, 2H), 8.30 (m, 4H), 8.35 (d, 1H, J=4.5 Hz), 8.57 (q, 1H), 8.78 (d, 1H, J=5.1 Hz), 8.80 (d, 1H, J=4.8 Hz), 8.82 (d, 1H,

J=4.2 Hz), 9.03 (d, 1H, J=4.2 Hz), 9.05 (q, 1H), 9.09 (d, 1H, J=5.1 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 411 (27.0), 497 (1.6), 528 (1.6), 583 (0.7), 632nm (2.1); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 412 (24.3), 431sh (13.7), 523 (3.2), 583 (0.3), 635nm (5.4); Emission (CH_2Cl_2): λ=640nm; Emission (CH_2Cl_2/HCl): λ=652nm; MS(FAB) : m/z (%): 528 (100) [M^+]

6b: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=-2.01 (s,1H), 7.79 (m, 6H), 7.93 (m, 2H), 8.18 (m, 4H), 8.29 (m, 2H), 8.37 (d, 1H, J=4.5 Hz), 8.59 (q, 1H), 8.76(d, 1H, J=4.5 Hz), 8.79 (d, 1H, J=3.5 Hz), 8.79 (d, 1H, J=5.0 Hz), 9.05 (d, 1H, J=4.0 Hz), 9.08 (d, 1H, J=5.5 Hz), 9.11 (d, 1H, J=5.5 Hz); UV/Vis (CH_2Cl_2) : λ_{max} ($\epsilon x10^{-4}$); 412 (38.0), 498 (2.5), 529 (2.2), 584 (1.1), 633nm (3.6); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 414 (36.9), 526 (2.7), 563 (2.5), 586 (2.7), 637nm (7.5); Emission (CH_2Cl_2): λ=641nm; Emission (CH_2Cl_2/HCl): λ=651nm; MS(Electron Spray): m/z (%): 608 (100) [M+1]

6c: ^{1}HNMR (500 MHz, $CDCl_3$, 25°C, TMS): δ=-1.99 (s, 1H), 3.62 (s, 3H), 3.93 (s, 3H), 7.58 (s, 1H), 7.74 (m, 6H), 7.81 (m, 2H), 8.12 (m, 2H), 8.29 (m, 2H), 8.30 (d, 1H, J=4.5 Hz), 8.56 (q, 1H), 8.65 (d, 1H, J=4.5 Hz), 8.73 (d, 1H, J=4.5 Hz), 8.77 (d, 1H, J=5.0 Hz), 8.98 (d, 1H, J=4.0 Hz), 9.05 (q, 1H), 9.08 (d, 1H, J=5.0 Hz); UV/Vis (CH_2Cl_2): λ_{max} ($\epsilon x10^{-4}$); 410 (22.65), 498 (1.42), 529 (1.67), 582 (0.64), 630nm (1.94); UV/Vis (CH_2Cl_2/HCl): λ_{max} ($\epsilon x10^{-4}$); 411 (15.82), 530 (1.04), 573 (1.19), 629nm (2.61); Emission (CH_2Cl_2): λ=638nm; Emission (CH_2Cl_2/ HCl): λ=646nm; MS(Electron Spray): m/z (%): 588 (100) [M^+]

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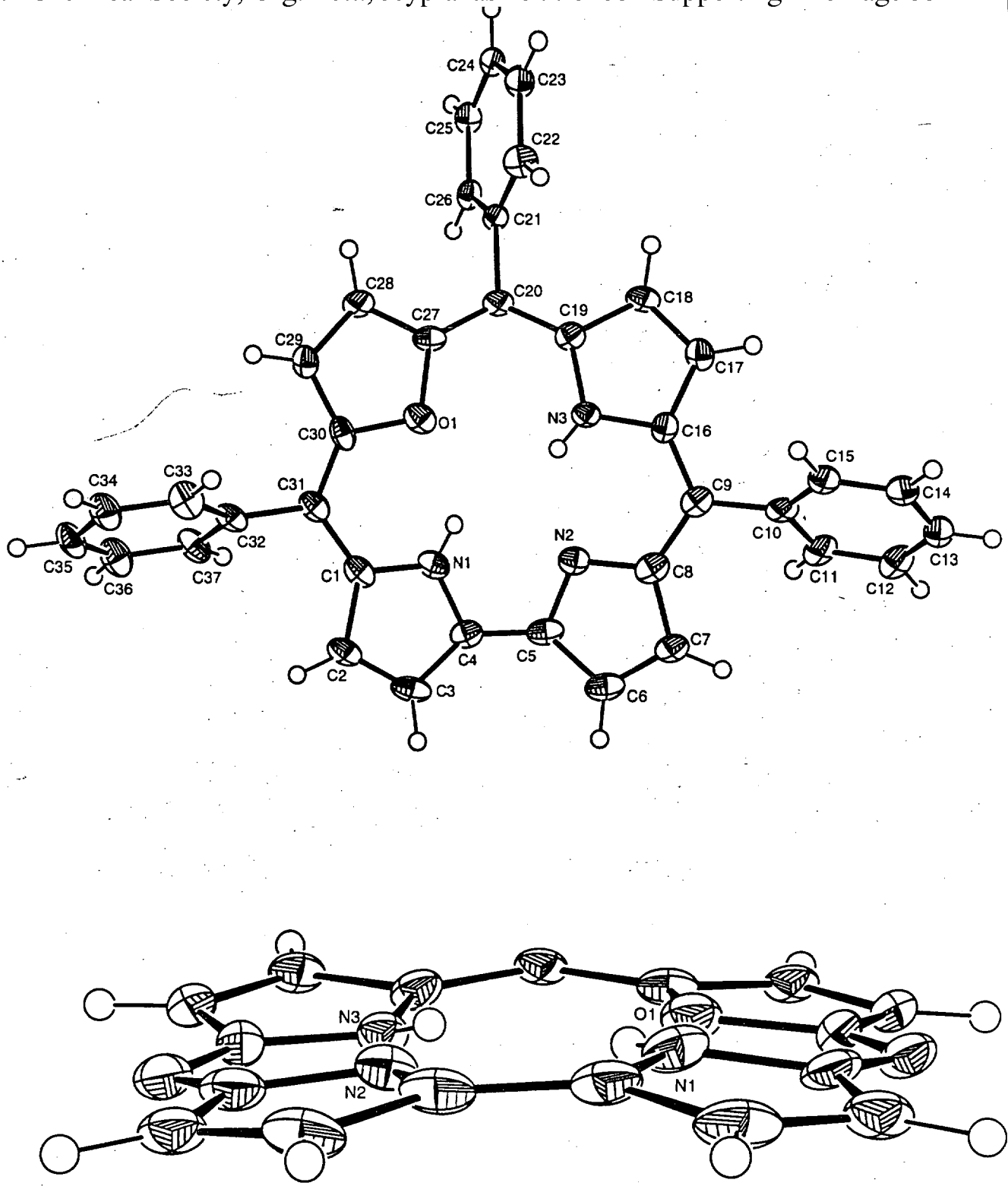
General synthetic procedure :

Tripyrrane **3a** (1.263 mmol) and dipyrromethane **4a** (1.263 mmol) was dissolved in 500 ml dry dichloromethane and stirred under nitrogen atmosphere for 5 min. TFA (0.1263 mmol) was added and the stirring continued for further 90 min. Then chloranil (3.789 mmol) was added and the reaction mixture heated at reflux for an additional 90 min. After removal of the solvent the residue was purified by chromotography on basic alumina (grade 3). The pink fraction which eluted with petroleum ether/dichloromethane (5/1) was identified as **6a** (19mg, 3%); smaragdyrin **5a** (380mg, 51%) eluted as a green band when the eluent was dichloromethane. The same procedure was followed for other reactions with appropriate precursors.

Yield of Smaragdyrins and Corroles

Substituents on the phenyl rings	Equivalents of TFA added	Smaragdyrins		Corroles	
		Compound No.	Yield (%)	Compound No.	Yield (%)
No substituents	0.1	5a	51	6a	3
No substituents	0.5	5a	48	6a	4
No substituents	1	5a	33	6a	6
p-Br	0.5	5b	34	6b	3
2,5- dimethoxy	0.5	5c	13	6c	6
2,4,6-trimethyl (mesityl)	0.3	5d	6	6d	< 1
pentafluoro-	0.5	5e	28	6e	< 1
No substituents	0.1	5f	5	6f	< 1



Cystal structure of Monooxa Corrole. Top: plane view; Bottom: side view (meso phenyl rings are omitted for clarity)

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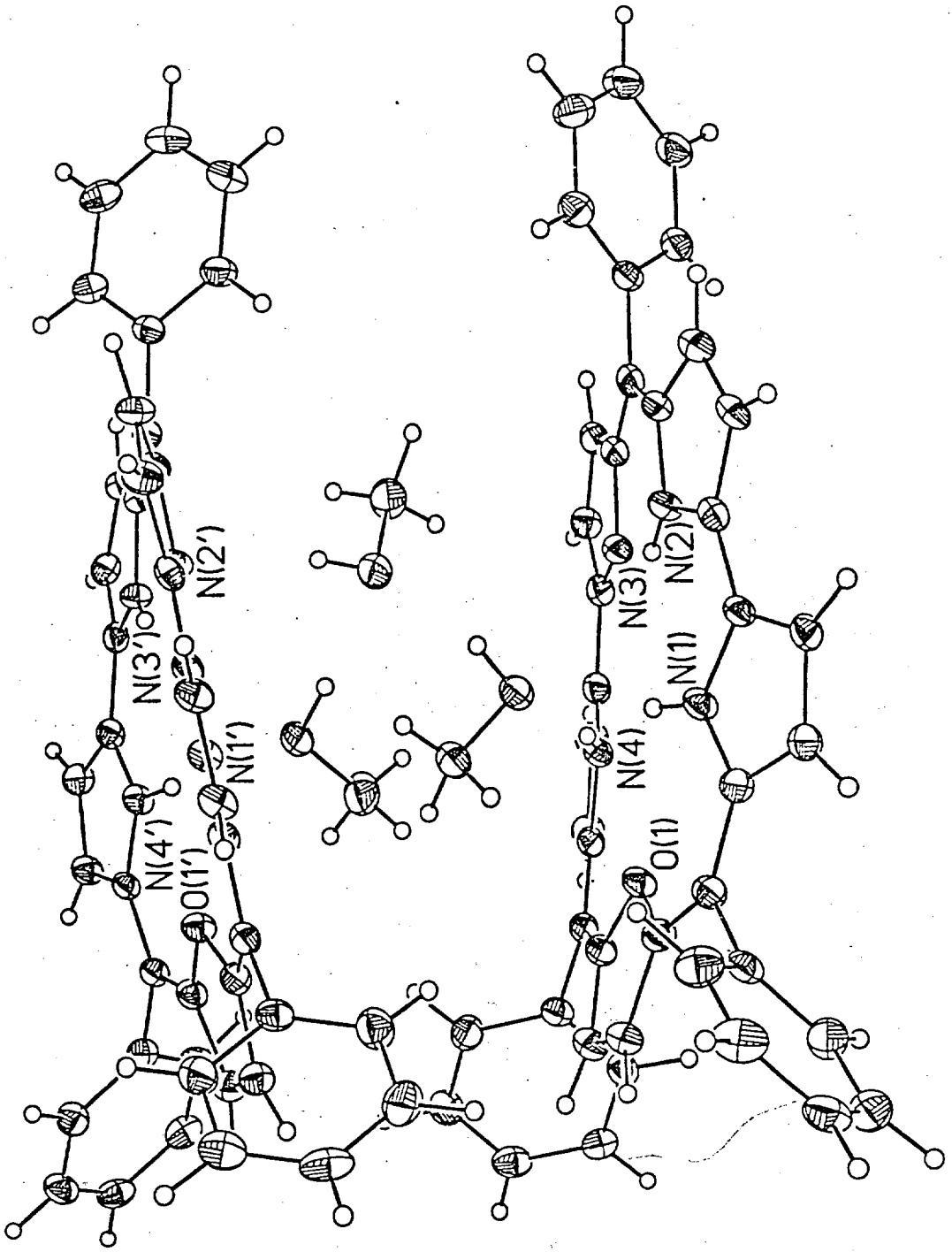
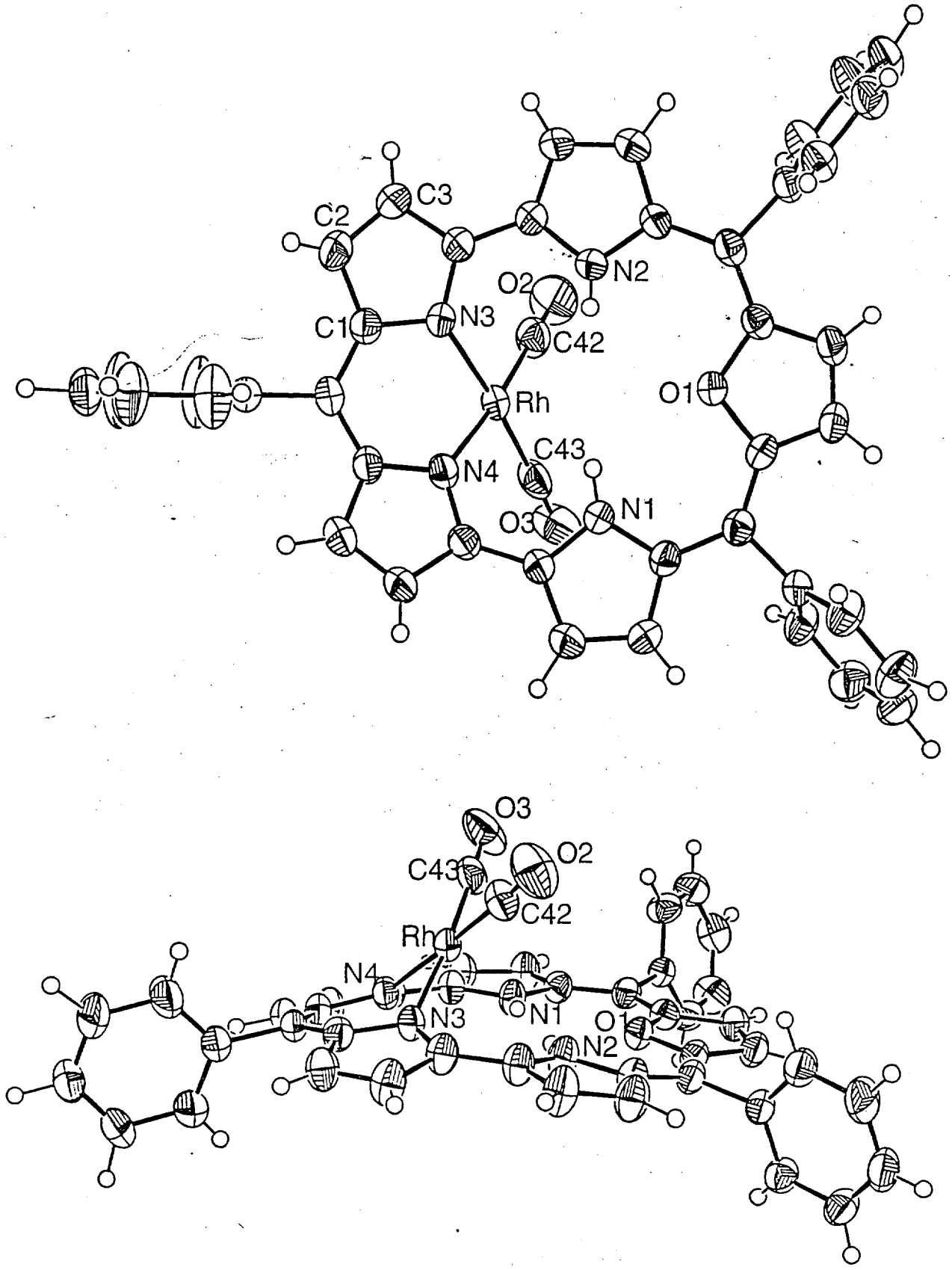


The ORTEP plot showing two independent molecules of 5a trapping three solvent methanol molecules with weak interactions in the unit cell.



ORTEP Plots of $Rh(CO)_2$ Oxa Smaragdyrin

Selected bond lengths and bond angles :
Rh-N3 2.034, Rh-N4 2.042, Rh-C42 1.871, Rh-C43 1.856, C42-O2 1.126, C43-O3 1.127
N3-Rh-N4 82.45, N4-Rh-C43 94.27, N3-Rh-C42 93.73, C42-Rh-C43 89.32

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Table 1. Crystal data and structure refinement for OSMAR.

Identification code	OSMAR
Empirical formula	$C_{85}H_{68}N_8O_5$
Crystal color, habit	deep purple block
Crystal size	0.4 x 0.2 x 0.2 mm
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 11.7055(2) Å α = 80.1370(10)° b = 14.83020(10) Å β = 79.2780(10)° c = 19.6421(4) Å γ = 80.5760(10)°
Volume	3269.95(9) $Å^3$
Peaks to determine cell	6492 with I > 10σ(I)
Z	2
Formula weight	1281.47
Density (calculated)	1.302 g/cm^3
Absorption coefficient	0.082 mm^{-1}
F(000)	1348

Data Collection

Diffractometer	CCD area detector
θ range for data collection	1.79 to 28.31°
Index ranges	$-14 \le h \le 15$, $-9 \le k \le 19$, $-26 \le l \le 25$
Scan Type	phi and omega scans
Scan Time	30sec / frame
Scan Range	0.3° in phi and omega scans
Temperature	98(2) K
Wavelength	0.71073 Å
Detector-to-sample distance	4.956 cm
Reflections collected	21116
Independent reflections	14489 (R_{int} = 0.0308)
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.98 and 0.84

The data collection nominally covered over a hemisphere of reciprocal space, by a combination of three sets of exposures; each set had a different ϕ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 4.956 cm. Coverage of the unique set is over 97% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analyzing the duplicate reflections. No decay was observed.

Solution and Refinement

Solution	direct methods
Refinement method	Full-matrix least-squares on F^2
Hydrogen atoms	

Positions of all hydrogen atoms were found in difference Fourier maps and the atoms were refined free in their positional and displacement parameters. No constraints were applied.

Weighting scheme

$$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.0P]$$

$$\text{where } P = [F_o^2 + 2F_c^2]/3$$

Data / restraints / parameters	14489 / 0 / 1156
Goodness-of-fit on F^2	0.946
Final R indices [$I>2\sigma(I)$]	R1 = 0.0541, wR2 = 0.1034
R indices (all data)	R1 = 0.1134, wR2 = 0.1232
Observed data [$I>2\sigma(I)$]	8571
Extinction coefficient	0.0024(3)
Largest diff. peak and hole	0.337 and -0.298 $eÅ^{-3}$
Largest and mean Δ / esd	0.001 and 0.000

$$wR2 = [\sum[w(F_o^2 - F_c^2)^2] / \sum[w(F_o^2)^2]]^{1/2}$$

$$R1 = \sum||F_o| - |F_c|| / \sum|F_o|$$

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Data Collection:

SMART Software Reference Manual (1994). Siemens Analytical X-ray Instruments, 6300 Enterprise Dr., Madison, WI 53719-1173, USA.

Data Reduction:

SAINT Version 4 Software Reference Manual (1995). Siemens Analytical X-ray Instruments, 6300 Enterprise Dr., Madison, WI 53719-1173, USA.

Structure Solution, Refinement and Graphics:

G. M. Sheldrick (1994). SHELXTL Version 5 Reference Manual. Siemens Analytical X-ray Instruments, 6300 Enterprise Dr., Madison, WI 53719-1173, USA.

Neutral atom scattering factors were taken from:

International Tables for Crystallography, Vol C, Tables 6.1.1.4, 4.2.6.8, and 4.2.4.2, Kluwer: Boston.

Acknowledgement

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Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for OSMAR. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	3435(1)	1868(1)	1772(1)	21(1)
N(1)	4363(2)	1152(1)	3114(1)	20(1)
N(2)	3432(2)	2194(1)	4302(1)	20(1)
N(3)	2213(1)	3697(1)	3797(1)	21(1)
N(4)	2096(2)	3606(1)	2355(1)	20(1)
C(1)	5044(2)	588(1)	2661(1)	20(1)
C(2)	5981(2)	113(2)	2998(1)	23(1)
C(3)	5834(2)	385(2)	3652(1)	22(1)
C(4)	4815(2)	1017(1)	3730(1)	19(1)
C(5)	4292(2)	1463(1)	4320(1)	19(1)
C(6)	4557(2)	1265(2)	5008(1)	22(1)
C(7)	3852(2)	1895(2)	5390(1)	23(1)
C(8)	3153(2)	2512(1)	4944(1)	20(1)
C(9)	2449(2)	3367(1)	5026(1)	20(1)
C(10)	2234(2)	3665(2)	5738(1)	21(1)
C(11)	1807(2)	3082(2)	6330(1)	27(1)
C(12)	1621(2)	3358(2)	6990(1)	33(1)
C(13)	1857(2)	4216(2)	7062(1)	34(1)
C(14)	2280(2)	4803(2)	6478(1)	29(1)
C(15)	2473(2)	4527(2)	5817(1)	24(1)
C(16)	2019(2)	3953(1)	4462(1)	20(1)
C(17)	1340(2)	4856(2)	4450(1)	22(1)
C(18)	1142(2)	5146(2)	3783(1)	22(1)
C(19)	1695(2)	4412(2)	3390(1)	21(1)
C(20)	1699(2)	4389(2)	2664(1)	21(1)
C(21)	1345(2)	5092(2)	2149(1)	24(1)
C(22)	1502(2)	4721(2)	1530(1)	24(1)
C(23)	1961(2)	3788(2)	1656(1)	19(1)
C(24)	2198(2)	3138(2)	1175(1)	20(1)
C(25)	1737(2)	3472(1)	495(1)	20(1)
C(26)	593(2)	3397(2)	432(1)	30(1)
C(27)	185(2)	3717(2)	-199(1)	32(1)
C(28)	913(2)	4110(2)	-773(1)	28(1)
C(29)	2053(2)	4184(2)	-718(1)	25(1)
C(30)	2460(2)	3869(2)	-84(1)	22(1)
C(31)	2812(2)	2267(2)	1222(1)	22(1)
C(32)	3002(2)	1631(2)	740(1)	24(1)
C(33)	3745(2)	884(2)	961(1)	23(1)
C(34)	4045(2)	1023(1)	1599(1)	20(1)
C(35)	4826(2)	478(1)	1994(1)	21(1)
C(36)	5572(2)	-314(2)	1675(1)	22(1)
C(37)	6474(2)	-144(2)	1119(1)	28(1)
C(38)	7220(2)	-864(2)	841(1)	30(1)
C(39)	7074(2)	-1768(2)	1110(1)	29(1)
C(40)	6178(2)	-1947(2)	1660(1)	34(1)
C(41)	5433(2)	-1229(2)	1946(1)	30(1)