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Supporting Information

A New Method for the Synthesis of Polyether Bridged Azulenes; Reactions of Conjugated Keto-carbenes Generated from the Corresponding Azulenoquinone Diazides

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(1) Summary of Crystallographic data (bond distances, angles and torsional angles) for **5a-2THF, 5a-3THF, 5b-2THP, 5a-2DXN, 5b-2DXN** ; Table 2-6.

(2) X-ray structure (ORTEP drawings) of **5b-2THP, 5a-2DXN, 5b-2DXN** ; Figure (2a) - Figure(2c).

(3) ^{1}H NMR spectra of **3, 4, 5, 6, 7** ; Figure 3 - Figure 16

Experimental

The structures of **5a-2THF, 5a-3THF, 5b-2THP, 5a-2DXN, 5b-2DXN** were completely characterized by single crystal X-ray analysis, crystals of these compounds were obtained by crystallization from dichloromethane/ n-hexane 10 / 1). All the crystal structures were solved by direct methods (NRCVAX). Crystallographic calculations were performed on MicrovaxIII using NRCVAX structure determination package. We collected the following collection of the X-ray diffraction data;

5a-2THF-----$C_{21}H_{23}NO_5$, triclinic in the space group P -1, with Z = 4, a = 8.0669 (12), b = 10.9852(6), c = 21.770(3) Å, V = 1849.5(4) Å^3, β =

85.953(12)° (from 25 orentation reflections , 29.46° < 2θ < 44.42°), Density (calc.) =1.327 g / cm^3, 5483 uniqure reflections measured with M_oK_α radiation (λ = 1.5418 Å), and 4451 obserbed with I > 2σ(I). Absorption correction was not applied .Refinement was carried out to converge on R_f = 0.060, R_w = 0.058.

5a-3THF-----$C_{25}H_{31}NO_6$, orthorhombic in the space group P 212121, with Z = 4, a = 6.2781(18), b = 15.252(4), c = 23.994(3) Å, V = 2297.4(9) $Å^3$, β = 90.00° (from 25 orentation reflections , 17.40° < 2θ < 22.38°), Density (calc.) =1.277g / cm^3, 2352 uniqure reflections measured with M_oK_α radiation (λ = 0.7107 Å), and 1168 obserbed with I > 2σ(I). Absorption correction was not applied .Refinement was carried out to converge on R_f = 0.240, Rw = 0.058.

5b-2THP-----$C_{22}H_{24}N_2O_3$, monoclinic in the space group P 21/c, with Z = 8, a = 17.428(3), b = 15.9095(13), c = 15.0070(13) Å, V = 3889.7(8) $Å^3$, β = 110.809(10)°(from 25 orentation reflections , 34.90° < 2θ < 47.48°), Density (calc.) =1.245 g / cm^3, 5768 uniqure reflections measured with M_oK_α radiation (λ = 1.5418 Å), and 3914 obserbed with I > 2σ(I). Absorption correction was not applied .Refinement was carried out to converge on R_f = 0.062, R_w = 0.055.

5a-2DXN-----$C_{21}H_{23}NO_7$, monoclinic in the space group P 21/c, with Z = 4, a = 2.3496(19), b = 12.329(3), c = 13.8677(18) Å, V = 2076.3(6) $Å^3$, β = 100.466(12)°(from 25 orentation reflections , 39.48° < 2θ < 60.98°),

Density (calc.) =1.335 g / cm^3, 3540 uniqure reflections measured with M_oK_α radiation (λ = 1.5418 Å), and 2969 obserbed with I > 2σ(I). Absorption correction was not applied .Refinement was carried out to converge on R_f = 0.043, R_w = 0.038.

5b-2DXN-----$C_{20}H_{20}N_2O_5$, monoclinic in the space group P 21/n, with Z = 4, a = 7.2362(9), b = 12.0551(11), c = 21.279(3) Å, V = 1832.3(4) $Å^3$, β = 99.228(11)°(from 25 orentation reflections , 34.76° < 2θ < 43.70°), Density (calc.) = 1.335 g / cm^3, 2713 uniqure reflections measured with M_oK_α radiation (λ = 1.5418 Å), and 2227 obserbed with I > 2σ(I). Absorption correction was not applied .Refinement was carried out to converge on R_f = 0.037, R_w = 0.037.

Table 2 The structure parameters (bond distances, angles and torsional angles) of **5a-2THF**.

O1-C2	1.345(4)	O1'-C2'	1.348(4)	C4-C10-C9	127.7(3)	C4'-C10'-C9'	127.8(3)
O1-C14	1.458(4)	O1'-C14'	1.461(6)	O4-C11-O5	122.0(3)	O4'-C11'-O5'	121.5(3)
O2-C17	1.416(5)	O2'-C17'	1.459(7)	O4-C11-C1	125.3(3)	O4'-C11'-C1'	125.4(3)
O2-C18	1.415(5)	O2'-C18'	1.407(6)	O5-C11-C1	112.7(3)	O5'-C11'-C1'	113.1(3)
O3-C4	1.352(4)	O3'-C4'	1.360(4)	N-C13-C3	176.4(4)	N'-C13'-C3'	176.0(5)
O3-C21	1.453(5)	O3'-C21'	1.450(5)	O1-C14-C15	110.7(3)	O1'-C14'-C15'	110.8(3)
O4-C11	1.204(4)	O4'-C11'	1.206(4)	C14-C15-C16	113.5(3)	C14'-C15'-C16'	116.1(4)
O5-C11	1.337(4)	O5'-C11'	1.336(5)	C15-C16-C17	113.9(3)	C15'-C16'-C17'	114.8(5)
O5-C12	1.453(5)	O5'-C12'	1.444(5)	O2-C17-C16	109.7(4)	O2'-C17'-C16'	109.7(5)
N-C13	1.144(5)	N'-C13'	1.138(5)	O2-C18-C19	107.5(3)	O2'-C18'-C19'	108.3(4)
C1-C2	1.407(5)	C1'-C2'	1.408(5)	C18-C19-C20	115.3(3)	C18'-C19'-C20'	123.4(6)
C1-C9	1.421(5)	C1'-C9'	1.422(5)	C19-C20-C21	110.7(3)	C19'-C20'-C21'	120.7(5)
C1-C11	1.465(5)	C1'-C11'	1.466(5)	O3-C21-C20	106.7(3)	O3'-C21'-C20'	111.0(4)
C2-C3	1.412(5)	C2'-C3'	1.409(5)				
C3-C10	1.416(5)	C3'-C10'	1.413(5)				
C3-C13	1.423(5)	C3'-C13'	1.422(5)				
C4-C5	1.402(5)	C4'-C5'	1.402(5)				
C4-C10	1.396(5)	C4'-C10'	1.386(5)				
C5-C6	1.377(6)	C5'-C6'	1.373(6)				
C6-C7	1.382(6)	C6'-C7'	1.379(6)				
C7-C8	1.373(5)	C7'-C8'	1.375(5)				
C8-C9	1.399(5)	C8'-C9'	1.396(5)				
C9-C10	1.459(5)	C9'-C10'	1.466(5)				
C14-C15	1.496(5)	C14'-C15'	1.569(8)				
C15-C16	1.513(6)	C15'-C16'	1.459(8)				
C16-C17	1.492(7)	C16'-C17'	1.500(9)				
C18-C19	1.511(6)	C18'-C19'	1.473(8)				
C19-C20	1.533(6)	C19'-C20'	1.408(8)				
C20-C21	1.504(6)	C20'-C21'	1.455(7)				

C2-O1-C14	117.0(3)	C2'-O1'-C14'	116.3(3)
C17-O2-C18	114.1(3)	C17'-O2'-C18'	109.2(4)
C4-O3-C21	120.4(3)	C4'-O3'-C21'	119.6(3)
C11-O5-C12	115.6(3)	C11'-O5'-C12'	114.8(3)
C2-C1-C9	107.7(3)	C2'-C1'-C9'	107.5(3)
C2-C1-C11	127.0(3)	C2'-C1'-C11'	128.0(3)
C9-C1-C11	125.2(3)	C9'-C1'-C11'	124.5(3)
O1-C2-C1	123.7(3)	O1'-C2'-C1'	124.1(3)
O1-C2-C3	126.6(3)	O1'-C2'-C3'	126.1(3)
C1-C2-C3	109.6(3)	C1'-C2'-C3'	109.6(3)
C2-C3-C10	108.1(3)	C2'-C3'-C10'	108.5(3)
C2-C3-C13	124.5(3)	C2'-C3'-C13'	124.7(3)
C10-C3-C13	126.8(3)	C10'-C3'-C13'	126.0(3)
O3-C4-C5	118.9(3)	O3'-C4'-C5'	118.6(3)
O3-C4-C10	113.5(3)	O3'-C4'-C10'	113.4(3)
C5-C4-C10	127.6(3)	C5'-C4'-C10'	127.9(3)
C4-C5-C6	128.8(4)	C4'-C5'-C6'	128.8(4)
C5-C6-C7	130.1(3)	C5'-C6'-C7'	129.9(4)
C6-C7-C8	128.8(3)	C6'-C7'-C8'	128.9(3)
C7-C8-C9	128.8(3)	C7'-C8'-C9'	129.2(3)
C1-C9-C8	125.0(3)	C1'-C9'-C8'	125.7(3)
C1-C9-C10	107.5(3)	C1'-C9'-C10'	107.7(3)
C8-C9-C10	127.5(3)	C8'-C9'-C10'	126.6(3)
C3-C10-C4	124.9(3)	C3'-C10'-C4'	125.3(3)
C3-C10-C9	107.1(3)	C3'-C10'-C9'	106.5(3)

C14	O1	C2	C1	-136.8(	4)	C14	O1	C2	C3	48.0(	3)
C2	O1	C14	C15	71.4(	3)	C18	O2	C17	C16	-168.5(	6)
C17	O2	C18	C19	166.6(	6)	C21	O3	C4	C5	44.9(	3)
C21	O3	C4	C10	-137.4(	4)	C4	O3	C21	C20	131.6(	5)
C12	O5	C11	O4	0.5(	3)	C12	O5	C11	C1	179.8(	5)
C9	C1	C2	O1	-175.3(	5)	C9	C1	C2	C3	0.6(	2)
C11	C1	C2	O1	6.9(	2)	C11	C1	C2	C3	-177.2(	5)
C2	C1	C9	C8	177.9(	5)	C2	C1	C9	C10	-2.3(	2)
C11	C1	C9	C8	-4.3(	2)	C11	C1	C9	C10	175.5(	5)
C2	C1	C11	O4	-163.3(	5)	C2	C1	C11	O5	17.5(	2)
C9	C1	C11	O4	19.3(	2)	C9	C1	C11	O5	-159.9(	5)
O1	C2	C3	C10	177.2(	5)	O1	C2	C3	C13	5.8(	2)
C1	C2	C3	C10	1.4(	2)	C1	C2	C3	C13	-170.0(	5)
C2	C3	C10	C4	171.4(	5)	C2	C3	C10	C9	-2.8(	2)
C13	C3	C10	C4	-17.4(	2)	C13	C3	C10	C9	168.4(	5)
C2	C3	C13	N	34.7(	3)	C10	C3	C13	N	-135.0(	5)
O3	C4	C5	C6	-179.0(	5)	C10	C4	C5	C6	3.7(	2)
O3	C4	C10	C3	-0.9(	2)	O3	C4	C10	C9	172.0(	5)
C5	C4	C10	C3	176.5(	5)	C5	C4	C10	C9	-10.6(	2)
C4	C5	C6	C7	2.0(	2)	C5	C6	C7	C8	0.2(	3)
C6	C7	C8	C9	-3.6(	2)	C7	C8	C9	C1	179.1(	5)
C7	C8	C9	C10	-0.6(	2)	C1	C9	C10	C3	3.2(	2)
C1	C9	C10	C4	-170.8(	5)	C8	C9	C10	C3	-177.1(	5)
C8	C9	C10	C4	9.0(	2)	O1	C14	C15	C16	-173.1(	5)
C14	C15	C16	C17	59.7(	4)	C15	C16	C17	O2	51.3(	3)
O2	C18	C19	C20	-158.2(	6)	C18	C19	C20	C21	147.8(	6)
C19	C20	C21	O3	-65.3(	3)	C14'	O1'	C2'	C1'	-134.2(	5)
C14'	O1'	C2'	C3'	49.9(	3)	C2'	O1'	C14'	C15'	71.5(	4)
C18'	O2'	C17'	C16'	-165.8(	8)	C17'	O2'	C18'	C19'	147.4(	7)
C21'	O3'	C4'	C5'	46.4(	3)	C21'	O3'	C4'	C10'	-135.3(	5)
C4'	O3'	C21'	C20'	115.7(	5)	C12'	O5'	C11'	O4'	3.1(	3)
C12'	O5'	C11'	C1'	-177.9(	5)	C9'	C1'	C2'	O1'	-177.2(	5)
C9'	C1'	C2'	C3'	-0.7(	2)	C11'	C1'	C2'	O1'	5.1(	2)
C11'	C1'	C2'	C3'	-178.4(	5)	C2'	C1'	C9'	C8'	178.2(	5)
C2'	C1'	C9'	C10'	-2.2(	2)	C11'	C1'	C9'	C8'	-4.0(	2)
C11'	C1'	C9'	C10'	175.6(	5)	C2'	C1'	C11'	O4'	-174.4(	6)
C2'	C1'	C11'	O5'	6.7(	2)	C9'	C1'	C11'	O4'	8.2(	2)
C9'	C1'	C11'	O5'	-170.7(	5)	O1'	C2'	C3'	C10'	179.9(	5)
O1'	C2'	C3'	C13'	9.8(	2)	C1'	C2'	C3'	C10'	3.5(	2)
C1'	C2'	C3'	C13'	-166.6(	5)	C2'	C3'	C10'	C4'	169.2(	5)
C2'	C3'	C10'	C9'	-4.8(	2)	C13'	C3'	C10'	C4'	-20.8(	3)
C13'	C3'	C10'	C9'	165.2(	5)	C2'	C3'	C13'	N'	44.9(	4)
C10'	C3'	C13'	N'	-123.5(	6)	O3'	C4'	C5'	C6'	-177.6(	6)
C10'	C4'	C5'	C6'	4.4(	3)	O3'	C4'	C10'	C3'	-2.4(	2)
O3'	C4'	C10'	C9'	170.2(	5)	C5'	C4'	C10'	C3'	175.7(	5)
C5'	C4'	C10'	C9'	-11.7(	2)	C4'	C5'	C6'	C7'	1.5(	3)
C5'	C6'	C7'	C8'	0.8(	3)	C6'	C7'	C8'	C9'	-3.8(	3)
C7'	C8'	C9'	C1'	178.3(	5)	C7'	C8'	C9'	C10'	-1.2(	2)
C1'	C9'	C10'	C3'	4.3(	2)	C1'	C9'	C10'	C4'	-169.4(	5)
C8'	C9'	C10'	C3'	-176.1(	5)	C8'	C9'	C10'	C4'	10.1(	2)
O1'	C14'	C15'	C16'	-175.6(	7)	C14'	C15'	C16'	C17'	70.0(	5)
C15'	C16'	C17'	O2'	50.1(	4)	O2'	C18'	C19'	C20'	-159.0(	8)
C18'	C19'	C20'	C21'	155.3(	8)	C19'	C20'	C21'	O3'	-54.9(	4)

Table 3 The structure parameters (bond distances, angles and torsional angles) of **5a-3THF**.

C1-C2	1.400(6)	C12-C13	1.507(7)
C1-C9	1.408(7)	C13-C14	1.493(8)
C1-C24	1.461(7)	C14-O3	1.420(6)
C2-C3	1.413(6)	C15-C16	1.511(9)
C2-O1	1.339(6)	C15-O3	1.425(7)
C3-C10	1.413(7)	C16-C17	1.521(7)
C3-C23	1.425(7)	C17-C18	1.508(8)
C4-C5	1.407(7)	C18-O2	1.404(6)
C4-C10	1.412(7)	C19-C20	1.515(7)
C4-O4	1.349(6)	C19-O2	1.415(7)
C5-C6	1.373(8)	C20-C21	1.514(7)
C6-C7	1.379(8)	C21-C22	1.501(7)
C7-C8	1.378(8)	C22-O1	1.424(6)
C8-C9	1.394(7)	C23-N	1.139(6)
C9-C10	1.462(6)	C24-O5	1.193(6)
C11-C12	1.498(8)	C24-O6	1.312(7)
C11-O4	1.442(6)	C25-O6	1.433(6)

C2-C1-C9	107.4(4)	C12-C11-O4	105.1(4)
C2-C1-C24	127.5(5)	C11-C12-C13	113.1(4)
C9-C1-C24	125.0(4)	C12-C13-C14	113.8(5)
C1-C2-C3	110.4(4)	C13-C14-O3	108.5(4)
C1-C2-O1	121.5(4)	C16-C15-O3	114.2(4)
C3-C2-O1	128.2(4)	C15-C16-C17	115.2(5)
C2-C3-C10	107.2(4)	C16-C17-C18	111.1(5)
C2-C3-C23	126.4(4)	C17-C18-O2	110.7(4)
C10-C3-C23	126.2(4)	C20-C19-O2	115.0(4)
C5-C4-C10	128.3(4)	C19-C20-C21	114.9(4)
C5-C4-O4	120.1(4)	C20-C21-C22	112.4(4)
C10-C4-O4	111.5(4)	C21-C22-O1	107.1(4)
C4-C5-C6	128.4(5)	C3-C23-N	176.8(5)
C5-C6-C7	130.1(5)	C1-C24-O5	124.8(5)
C6-C7-C8	129.3(5)	C1-C24-O6	112.6(4)
C7-C8-C9	129.3(5)	O5-C24-O6	122.5(5)
C1-C9-C8	125.1(4)	C2-O1-C22	121.8(4)
C1-C9-C10	107.9(4)	C18-O2-C19	114.0(4)
C8-C9-C10	127.0(5)	C14-O3-C15	113.9(4)
C3-C10-C4	125.3(4)	C4-O4-C11	124.3(4)
C3-C10-C9	107.1(4)	C24-O6-C25	115.9(4)
C4-C10-C9	127.5(4)		

C9	C1	C2	C3	1.6(| 3)	C9	C1	C2	O1	-179.5(| 6)
C24	C1	C2	C3	-175.8(| 6)	C24	C1	C2	O1	3.1(| 2)
C2	C1	C9	C8	-178.5(| 6)	C2	C1	C9	C10	-0.7(| 3)
C24	C1	C9	C8	-1.1(| 3)	C24	C1	C9	C10	176.8(| 6)
C2	C1	C24	O5	-179.0(| 7)	C2	C1	C24	O6	0.0(| 3)
C9	C1	C24	O5	4.0(| 3)	C9	C1	C24	O6	-176.9(| 6)
C1	C2	C3	C10	-1.9(| 3)	C1	C2	C3	C23	174.9(| 6)
O1	C2	C3	C10	179.3(| 6)	O1	C2	C3	C23	-3.9(| 2)
C1	C2	O1	C22	170.8(| 5)	C3	C2	O1	C22	-10.5(| 3)
C2	C3	C10	C4	-178.5(| 6)	C2	C3	C10	C9	1.4(| 3)
C23	C3	C10	C4	4.8(| 3)	C23	C3	C10	C9	-175.3(| 6)
C2	C3	C23	N	-35.7(| 4)	C10	C3	C23	N	140.4(| 6)
C10	C4	C5	C6	0.2(| 3)	O4	C4	C5	C6	-178.6(| 7)
C5	C4	C10	C3	-177.7(| 6)	C5	C4	C10	C9	2.4(| 3)
O4	C4	C10	C3	1.2(| 2)	O4	C4	C10	C9	-178.6(| 6)
C5	C4	O4	C11	0.8(| 3)	C10	C4	O4	C11	-178.2(| 5)
C4	C5	C6	C7	-1.6(| 3)	C5	C6	C7	C8	0.2(| 3)
C6	C7	C8	C9	1.0(| 3)	C7	C8	C9	C1	178.0(| 7)
C7	C8	C9	C10	0.5(| 3)	C1	C9	C10	C3	-0.5(| 3)
C1	C9	C10	C4	179.4(| 6)	C8	C9	C10	C3	177.4(| 6)
C8	C9	C10	C4	-2.8(| 3)	O4	C11	C12	C13	66.3(| 4)
C12	C11	O4	C4	-177.8(| 6)	C11	C12	C13	C14	-165.0(| 7)
C12	C13	C14	O3	174.4(| 7)	C13	C14	O3	C15	-167.1(| 6)
O3	C15	C16	C17	58.4(| 4)	C16	C15	O3	C14	75.7(| 4)
C15	C16	C17	C18	-178.2(| 7)	C16	C17	C18	O2	-176.3(| 6)
C17	C18	O2	C19	171.2(| 6)	O2	C19	C20	C21	61.3(| 4)
C20	C19	O2	C18	77.3(| 4)	C19	C20	C21	C22	-81.3(| 5)
C20	C21	C22	O1	-171.9(| 6)	C21	C22	O1	C2	-175.8(| 6)
C1	C24	O6	C25	177.7(| 6)	O5	C24	O6	C25	-3.2(| 3)

Table 4 The structure parameters (bond distances, angles and torsional angles) of **5b-2THP**.

O1-C2	1.351(4)	O1'-C2'	1.331(5)	O1-C11-C12	110.3(3)	O1'-C11'-C12'	109.3(4)
O1-C11	1.453(5)	O1'-C11'	1.430(5)	C11-C12-C13	114.6(3)	C11'-C12'-C13'	116.5(4)
O2-C15	1.407(5)	O2'-C15'	1.410(6)	C12-C13-C14	114.8(4)	C12'-C13'-C14'	115.5(4)
O2-C16	1.412(6)	O2'-C16'	1.405(7)	C13-C14-C15	113.4(4)	C13'-C14'-C15'	112.9(4)
O3-C4	1.332(4)	O3'-C4'	1.350(5)	O2-C15-C14	110.0(4)	O2'-C15'-C14'	109.5(4)
O3-C20	1.454(5)	O3'-C20'	1.434(6)	O2-C16-C17	108.7(4)	O2'-C16'-C17'	109.1(4)
N1-C21	1.138(5)	N1'-C21'	1.135(5)	C16-C17-C18	112.3(4)	C16'-C17'-C18'	113.0(5)
N2-C22	1.137(5)	N2'-C22'	1.115(6)	C17-C18-C19	114.4(4)	C17'-C18'-C19'	114.6(5)
C1-C2	1.379(5)	C1'-C2'	1.405(6)	C18-C19-C20	112.9(4)	C18'-C19'-C20'	114.1(4)
C1-C9	1.435(5)	C1'-C10'	1.386(6)	O3-C20-C19	106.3(3)	O3'-C20'-C19'	107.3(4)
C1-C21	1.425(5)	C1'-C21'	1.409(5)	N1-C21-C1	177.3(4)	N1'-C21'-C1'	176.0(5)
C2-C3	1.412(5)	C2'-C3'	1.345(6)	N2-C22-C3	175.5(4)	N2'-C22'-C3'	177.3(5)
C3-C10	1.406(5)	C3'-C9'	1.479(6)				
C3-C22	1.423(5)	C3'-C22'	1.416(6)				
C4-C5	1.404(5)	C4'-C5'	1.439(6)				
C4-C10	1.403(5)	C4'-C10'	1.364(6)				
C5-C6	1.359(6)	C5'-C6'	1.310(7)				
C6-C7	1.388(6)	C6'-C7'	1.422(7)				
C7-C8	1.386(6)	C7'-C8'	1.430(7)				
C8-C9	1.380(5)	C8'-C9'	1.362(5)				
C9-C10	1.447(5)	C9'-C10'	1.434(6)				
C11-C12	1.500(6)	C11'-C12'	1.476(7)				
C12-C13	1.495(6)	C12'-C13'	1.489(8)				
C13-C14	1.512(6)	C13'-C14'	1.517(7)				
C14-C15	1.499(7)	C14'-C15'	1.482(8)				
C16-C17	1.499(7)	C16'-C17'	1.481(8)				
C17-C18	1.514(7)	C17'-C18'	1.503(8)				
C18-C19	1.507(7)	C18'-C19'	1.491(8)				
C19-C20	1.498(6)	C19'-C20'	1.493(7)				

C2-O1-C11	118.3(3)	C2'-O1'-C11'	120.0(3)
C15-O2-C16	112.7(3)	C15'-O2'-C16'	112.0(4)
C4-O3-C20	121.8(3)	C4'-O3'-C20'	120.7(4)
C2-C1-C9	108.4(3)	C2'-C1'-C10'	106.6(3)
C2-C1-C21	127.5(4)	C2'-C1'-C21'	120.2(3)
C9-C1-C21	124.1(3)	C10'-C1'-C21'	133.2(4)
O1-C2-C1	129.0(3)	O1'-C2'-C1'	119.4(3)
O1-C2-C3	121.3(3)	O1'-C2'-C3'	131.5(4)
C1-C2-C3	109.6(3)	C1'-C2'-C3'	109.0(4)
C2-C3-C10	107.9(3)	C2'-C3'-C9'	108.9(4)
C2-C3-C22	121.5(3)	C2'-C3'-C22'	126.7(4)
C10-C3-C22	130.7(3)	C9'-C3'-C22'	124.4(4)
O3-C4-C5	121.3(3)	O3'-C4'-C5'	120.5(4)
O3-C4-C10	112.2(3)	O3'-C4'-C10'	114.3(4)
C5-C4-C10	126.5(3)	C5'-C4'-C10'	125.2(4)
C4-C5-C6	129.8(4)	C4'-C5'-C6'	132.8(5)
C5-C6-C7	130.6(4)	C5'-C6'-C7'	128.1(4)
C6-C7-C8	128.1(4)	C6'-C7'-C8'	129.3(4)
C7-C8-C9	127.3(4)	C7'-C8'-C9'	123.0(4)
C1-C9-C8	123.4(3)	C3'-C9'-C8'	119.7(4)
C1-C9-C10	106.3(3)	C3'-C9'-C10'	105.6(3)
C8-C9-C10	130.2(3)	C8'-C9'-C10'	134.7(4)
C3-C10-C4	125.0(3)	C1'-C10'-C4'	123.5(4)
C3-C10-C9	107.8(3)	C1'-C10'-C9'	109.8(4)
C4-C10-C9	127.0(3)	C4'-C10'-C9'	126.4(4)

C11	O1	C2	C1	-52.4(	2)	C11	O1	C2	C3	131.5(	4)
C2	O1	C11	C12	-106.1(	4)	C16	O2	C15	C14	-179.1(	5)
C15	O2	C16	C17	-172.4(	5)	C20	O3	C4	C5	26.2(	2)
C20	O3	C4	C10	-154.6(	4)	C4	O3	C20	C19	138.6(	4)
C9	C1	C2	O1	-177.4(	4)	C9	C1	C2	C3	-0.9(	2)
C21	C1	C2	O1	-0.1(	2)	C21	C1	C2	C3	176.4(	4)
C2	C1	C9	C8	179.1(	4)	C2	C1	C9	C10	-0.5(	2)
C21	C1	C9	C8	1.7(	2)	C21	C1	C9	C10	-177.9(	4)
C2	C1	C21	N1	-135.7(	5)	C9	C1	C21	N1	41.2(	3)
O1	C2	C3	C10	178.8(	4)	O1	C2	C3	C22	-0.5(	2)
C1	C2	C3	C10	2.0(	2)	C1	C2	C3	C22	-177.4(	4)
C2	C3	C10	C4	172.7(	4)	C2	C3	C10	C9	-2.2(	2)
C22	C3	C10	C4	-8.0(	2)	C22	C3	C10	C9	177.0(	4)
C2	C3	C22	N2	14.6(	2)	C10	C3	C22	N2	-164.6(	5)
O3	C4	C5	C6	-179.5(	5)	C10	C4	C5	C6	1.4(	2)
O3	C4	C10	C3	-0.6(	2)	O3	C4	C10	C9	173.4(	4)
C5	C4	C10	C3	178.5(	4)	C5	C4	C10	C9	-7.5(	2)
C4	C5	C6	C7	2.6(	2)	C5	C6	C7	C8	0.4(	2)
C6	C7	C8	C9	-3.5(	2)	C7	C8	C9	C1	-180.0(	4)
C7	C8	C9	C10	-0.5(	2)	C1	C9	C10	C3	1.7(	2)
C1	C9	C10	C4	-173.1(	4)	C8	C9	C10	C3	-177.9(	4)
C8	C9	C10	C4	7.3(	2)	O1	C11	C12	C13	64.3(	3)
C11	C12	C13	C14	171.4(	5)	C12	C13	C14	C15	179.5(	5)
C13	C14	C15	O2	-60.1(	3)	O2	C16	C17	C18	69.4(	3)
C16	C17	C18	C19	-169.3(	5)	C17	C18	C19	C20	-175.9(	5)
C18	C19	C20	O3	-67.5(	3)	C11'	O1'	C2'	C1'	131.7(	4)
C11'	O1'	C2'	C3'	-50.7(	3)	C2'	O1'	C11'	C12'	-108.3(	4)
C16'	O2'	C15'	C14'	-179.0(	5)	C15'	O2'	C16'	C17'	-171.4(	5)
C20'	O3'	C4'	C5'	28.5(	3)	C20'	O3'	C4'	C10'	-152.5(	4)
C4'	O3'	C20'	C19'	137.9(	5)	C10'	C1'	C2'	O1'	179.9(	5)
C10'	C1'	C2'	C3'	1.9(	2)	C21'	C1'	C2'	O1'	-0.7(	2)
C21'	C1'	C2'	C3'	-178.8(	5)	C2'	C1'	C10'	C4'	172.1(	5)
C2'	C1'	C10'	C9'	-2.4(	2)	C21'	C1'	C10'	C4'	-7.1(	2)
C21'	C1'	C10'	C9'	178.4(	4)	C2'	C1'	C21'	N1'	5.8(	2)
C10'	C1'	C21'	N1'	-175.0(	5)	O1'	C2'	C3'	C9'	-178.4(	5)
O1'	C2'	C3'	C22'	2.5(	2)	C1'	C2'	C3'	C9'	-0.6(	2)
C1'	C2'	C3'	C22'	-179.8(	5)	C2'	C3'	C9'	C8'	179.0(	5)
C2'	C3'	C9'	C10'	-0.8(	2)	C22'	C3'	C9'	C8'	-1.9(	2)
C22'	C3'	C9'	C10'	178.3(	5)	C2'	C3'	C22'	N2'	-174.8(	6)
C9'	C3'	C22'	N2'	6.2(	2)	O3'	C4'	C5'	C6'	179.8(	5)
C10'	C4'	C5'	C6'	1.0(	2)	O3'	C4'	C10'	C1'	-0.1(	2)
O3'	C4'	C10'	C9'	173.5(	5)	C5'	C4'	C10'	C1'	178.8(	5)
C5'	C4'	C10'	C9'	-7.6(	2)	C4'	C5'	C6'	C7'	3.9(	2)
C5'	C6'	C7'	C8'	-1.4(	2)	C6'	C7'	C8'	C9'	-1.8(	2)
C7'	C8'	C9'	C3'	178.6(	4)	C7'	C8'	C9'	C10'	-1.7(	2)
C3'	C9'	C10'	C1'	2.0(	2)	C3'	C9'	C10'	C4'	-172.3(	5)
C8'	C9'	C10'	C1'	-177.7(	5)	C8'	C9'	C10'	C4'	8.0(	2)
O1'	C11'	C12'	C13'	65.3(	3)	C11'	C12'	C13'	C14'	172.2(	6)
C12'	C13'	C14'	C15'	177.5(	6)	C13'	C14'	C15'	O2'	-61.0(	3)
O2'	C16'	C17'	C18'	67.4(	4)	C16'	C17'	C18'	C19'	-173.5(	6)
C17'	C18'	C19'	C20'	-177.4(	6)	C18'	C19'	C20'	O3'	-64.5(	4)

Table 5 The structure parameters (bond distances, angles and torsional angles) of **5a-2DXN.**

O1-C2	1.3556(24)	C2-C3	1.402(3)
O1-C11	1.447(3)	C3-C10	1.413(3)
O2-C12	1.403(3)	C3-C19	1.424(3)
O2-C13	1.424(3)	C4-C5	1.395(3)
O3-C14	1.403(4)	C4-C10	1.399(3)
O3-C15	1.423(3)	C5-C6	1.376(3)
O4-C16	1.421(3)	C6-C7	1.385(4)
O4-C17	1.416(3)	C7-C8	1.378(3)
O5-C4	1.346(3)	C8-C9	1.395(3)
O5-C18	1.448(3)	C9-C10	1.454(3)
O6-C20	1.201(3)	C11-C12	1.487(4)
O7-C20	1.336(3)	C13-C14	1.493(4)
O7-C21	1.450(3)	C15-C16	1.485(5)
N-C19	1.144(3)	C17-C18	1.490(4)
C1-C2	1.401(3)	C22-O8	1.532(7)
C1-C9	1.431(3)	O8-C22	1.532(7)
C1-C20	1.459(3)		

C2-O1-C11	118.33(16)	C7-C8-C9	129.34(21)
C12-O2-C13	111.95(18)	C1-C9-C8	125.17(20)
C14-O3-C15	114.25(22)	C1-C9-C10	107.37(17)
C16-O4-C17	112.87(20)	C8-C9-C10	127.38(19)
C4-O5-C18	121.23(17)	C3-C10-C4	125.17(19)
C20-O7-C21	115.01(20)	C3-C10-C9	107.15(17)
C2-C1-C9	107.11(18)	C4-C10-C9	127.59(18)
C2-C1-C20	128.41(19)	O1-C11-C12	110.75(20)
C9-C1-C20	124.37(19)	O2-C12-C11	110.03(19)
O1-C2-C1	129.42(19)	O2-C13-C14	109.86(20)
O1-C2-C3	120.35(18)	O3-C14-C13	108.20(22)
C1-C2-C3	110.21(18)	O3-C15-C16	108.54(24)
C2-C3-C10	108.10(18)	O4-C16-C15	109.12(22)
C2-C3-C19	121.84(19)	O4-C17-C18	108.45(19)
C10-C3-C19	130.03(19)	O5-C18-C17	105.90(19)
O5-C4-C5	120.24(19)	N-C19-C3	175.81(24)
O5-C4-C10	111.77(18)	O6-C20-O7	121.56(21)
C5-C4-C10	127.96(20)	O6-C20-C1	126.11(21)
C4-C5-C6	129.10(21)	O7-C20-C1	112.31(19)
C5-C6-C7	129.65(20)	C22-O8-C22	180.0
C6-C7-C8	128.40(21)		

C11	O1	C2	C1	-57.1(	2)	C11	O1	C2	C3	124.9(	2)
C2	O1	C11	C12	-104.8(	2)	C13	O2	C12	C11	175.4(	3)
C12	O2	C13	C14	166.5(	3)	C15	O3	C14	C13	-166.5(	3)
C14	O3	C15	C16	-178.3(	3)	C17	O4	C16	C15	-173.4(	3)
C16	O4	C17	C18	176.8(	3)	C18	O5	C4	C5	29.0(	1)
C18	O5	C4	C10	-153.0(	2)	C4	O5	C18	C17	141.1(	3)
C21	O7	C20	O6	-3.2(	1)	C21	O7	C20	C1	178.6(	3)
C9	C1	C2	O1	179.6(	2)	C9	C1	C2	C3	-2.3(	1)
C20	C1	C2	O1	-4.1(	1)	C20	C1	C2	C3	173.9(	2)
C2	C1	C9	C8	-174.4(	3)	C2	C1	C9	C10	2.4(	1)
C20	C1	C9	C8	9.2(	1)	C20	C1	C9	C10	-174.0(	2)
C2	C1	C20	O6	167.9(	3)	C2	C1	C20	O7	-14.0(	1)
C9	C1	C20	O6	-16.5(	1)	C9	C1	C20	O7	161.6(	3)
O1	C2	C3	C10	179.5(	2)	O1	C2	C3	C19	-2.2(	1)
C1	C2	C3	C10	1.2(	1)	C1	C2	C3	C19	179.5(	3)
C2	C3	C10	C4	177.2(	2)	C2	C3	C10	C9	0.3(	1)
C19	C3	C10	C4	-0.9(	1)	C19	C3	C10	C9	-177.7(	2)
C2	C3	C19	N	22.6(	1)	C10	C3	C19	N	-159.6(	3)
O5	C4	C5	C6	-174.2(	3)	C10	C4	C5	C6	8.1(	1)
O5	C4	C10	C3	0.0(	1)	O5	C4	C10	C9	176.2(	2)
C5	C4	C10	C3	177.8(	3)	C5	C4	C10	C9	-6.0(	1)
C4	C5	C6	C7	-0.8(	1)	C5	C6	C7	C8	-5.6(	1)
C6	C7	C8	C9	2.4(	1)	C7	C8	C9	C1	179.7(	3)
C7	C8	C9	C10	3.6(	1)	C1	C9	C10	C3	-1.7(	1)
C1	C9	C10	C4	-178.4(	2)	C8	C9	C10	C3	175.0(	2)
C8	C9	C10	C4	-1.8(	1)	O1	C11	C12	O2	78.4(	2)
O2	C13	C14	O3	-68.5(	2)	O3	C15	C16	O4	76.2(	2)
O4	C17	C18	O5	-71.1(	2)						

Table 6 The structure parameters (bond distances, angles and torsional angles) of **5b-2DXN**.

O1-C2	1.348(3)	C2-C3	1.400(3)
O1-C11	1.447(3)	C3-C4	1.418(3)
O2-C12	1.417(3)	C3-C20	1.416(3)
O2-C13	1.415(3)	C4-C5	1.393(3)
O3-C14	1.413(3)	C4-C10	1.452(3)
O3-C15	1.413(3)	C5-C6	1.402(3)
O4-C16	1.420(3)	C6-C7	1.374(3)
O4-C17	1.414(3)	C7-C8	1.383(4)
O5-C5	1.350(3)	C8-C9	1.381(3)
O5-C18	1.431(3)	C9-C10	1.391(3)
N1-C19	1.140(4)	C11-C12	1.488(4)
N2-C20	1.140(3)	C13-C14	1.490(4)
C1-C2	1.393(3)	C15-C16	1.493(4)
C1-C10	1.425(3)	C17-C18	1.493(4)
C1-C19	1.405(3)		

C2-O1-C11	119.17(17)	C4-C5-C6	127.58(21)
C12-O2-C13	111.48(17)	C5-C6-C7	129.37(22)
C14-O3-C15	112.91(18)	C6-C7-C8	130.65(21)
C16-O4-C17	111.80(19)	C7-C8-C9	127.54(21)
C5-O5-C18	121.55(18)	C8-C9-C10	128.49(21)
C2-C1-C10	107.98(19)	C1-C10-C4	106.86(18)
C2-C1-C19	127.59(21)	C1-C10-C9	123.63(20)
C10-C1-C19	124.41(21)	C4-C10-C9	129.43(20)
O1-C2-C1	128.50(21)	O1-C11-C12	108.86(20)
O1-C2-C3	121.34(20)	O2-C12-C11	109.07(18)
C1-C2-C3	110.01(19)	O2-C13-C14	110.83(19)
C2-C3-C4	107.82(18)	O3-C14-C13	108.84(20)
C2-C3-C20	122.40(19)	O3-C15-C16	109.90(20)
C4-C3-C20	129.77(20)	O4-C16-C15	110.56(20)
C3-C4-C5	125.88(20)	O4-C17-C18	107.98(20)
C3-C4-C10	107.32(18)	O5-C18-C17	107.27(19)
C5-C4-C10	126.79(19)	N1-C19-C1	178.9(3)
O5-C5-C4	112.28(19)	N2-C20-C3	176.40(25)
O5-C5-C6	120.11(20)		

C11	O1	C2	C1	-51.4(2)	C11	O1	C2	C3	133.6(2)
C2	O1	C11	C12	-106.3(2)	C13	O2	C12	C11	-173.9(3)
C12	O2	C13	C14	168.9(3)	C15	O3	C14	C13	-165.9(3)
C14	O3	C15	C16	175.4(3)	C17	O4	C16	C15	-171.8(3)
C16	O4	C17	C18	172.3(3)	C18	O5	C5	C4	-149.5(2)
C18	O5	C5	C6	32.3(1)	C5	O5	C18	C17	135.3(3)
C10	C1	C2	O1	-175.5(3)	C10	C1	C2	C3	-0.1(1)
C19	C1	C2	O1	2.9(1)	C19	C1	C2	C3	178.3(3)
C2	C1	C10	C4	0.0(1)	C2	C1	C10	C9	-177.1(3)
C19	C1	C10	C4	-178.5(3)	C19	C1	C10	C9	4.4(1)
C2	C1	C19	N1	-179.3(3)	C10	C1	C19	N1	-1.2(2)
O1	C2	C3	C4	176.0(3)	O1	C2	C3	C20	-5.0(1)
C1	C2	C3	C4	0.1(1)	C1	C2	C3	C20	179.1(3)
C2	C3	C4	C5	178.8(3)	C2	C3	C4	C10	-0.1(1)
C20	C3	C4	C5	-0.1(1)	C20	C3	C4	C10	-179.0(3)
C2	C3	C20	N2	11.5(1)	C4	C3	C20	N2	-169.7(3)
C3	C4	C5	O5	1.2(1)	C3	C4	C5	C6	179.2(3)
C10	C4	C5	O5	179.9(3)	C10	C4	C5	C6	-2.1(1)
C3	C4	C10	C1	0.1(1)	C3	C4	C10	C9	177.0(3)
C5	C4	C10	C1	-178.8(3)	C5	C4	C10	C9	-1.9(1)
O5	C5	C6	C7	-178.4(3)	C4	C5	C6	C7	3.8(1)
C5	C6	C7	C8	-0.3(1)	C6	C7	C8	C9	-3.3(1)
C7	C8	C9	C10	1.7(1)	C8	C9	C10	C1	178.5(3)
C8	C9	C10	C4	2.1(1)	O1	C11	C12	O2	72.1(2)
O2	C13	C14	O3	-68.8(2)	O3	C15	C16	O4	76.4(2)
O4	C17	C18	O5	-68.2(2)					

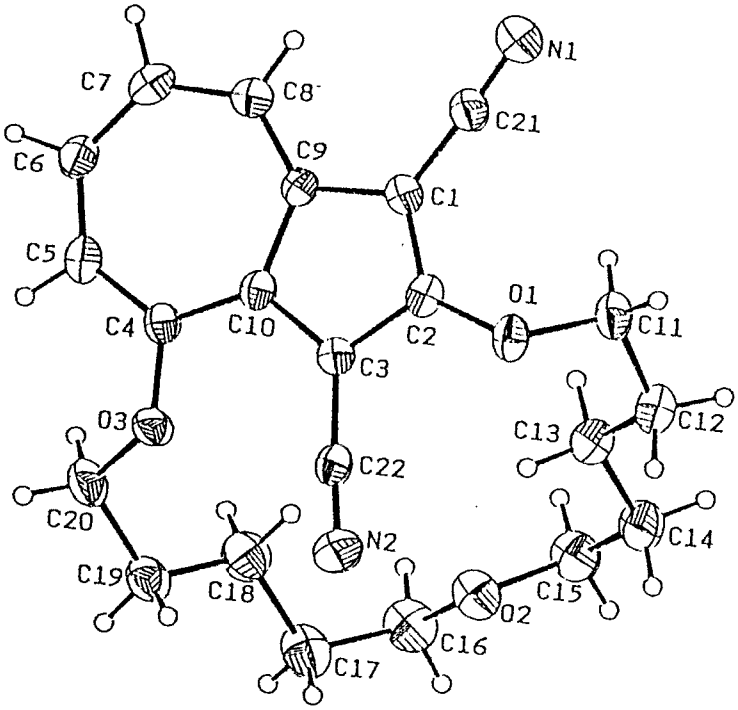


Figure 2a. The X-ray structure (ORTEP drawings) for **5b-2TIIP.**

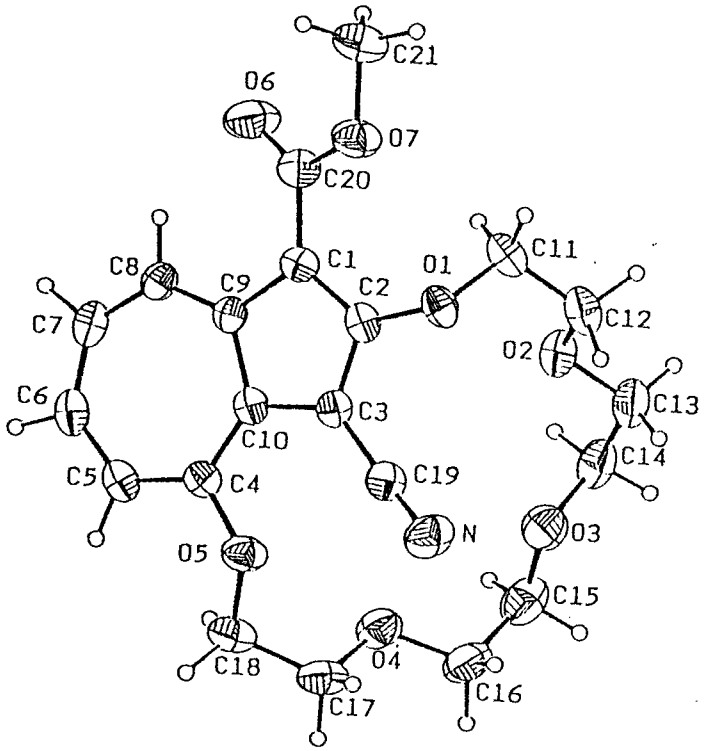


Figure 2b. The X-ray structure (ORTEP drawings) for **5a-2DXN.**

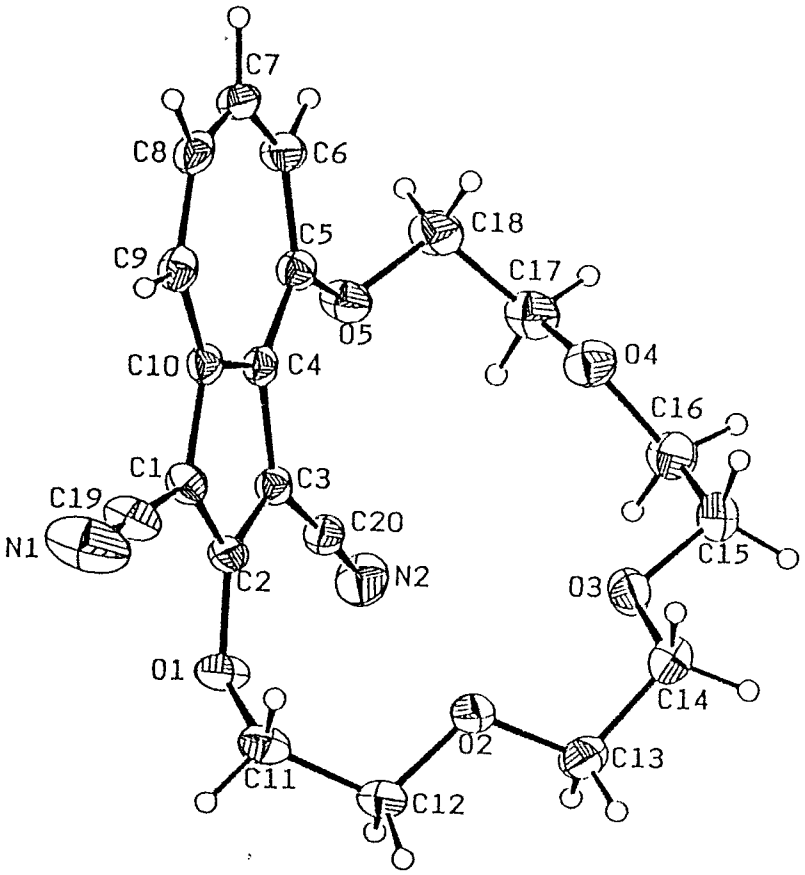
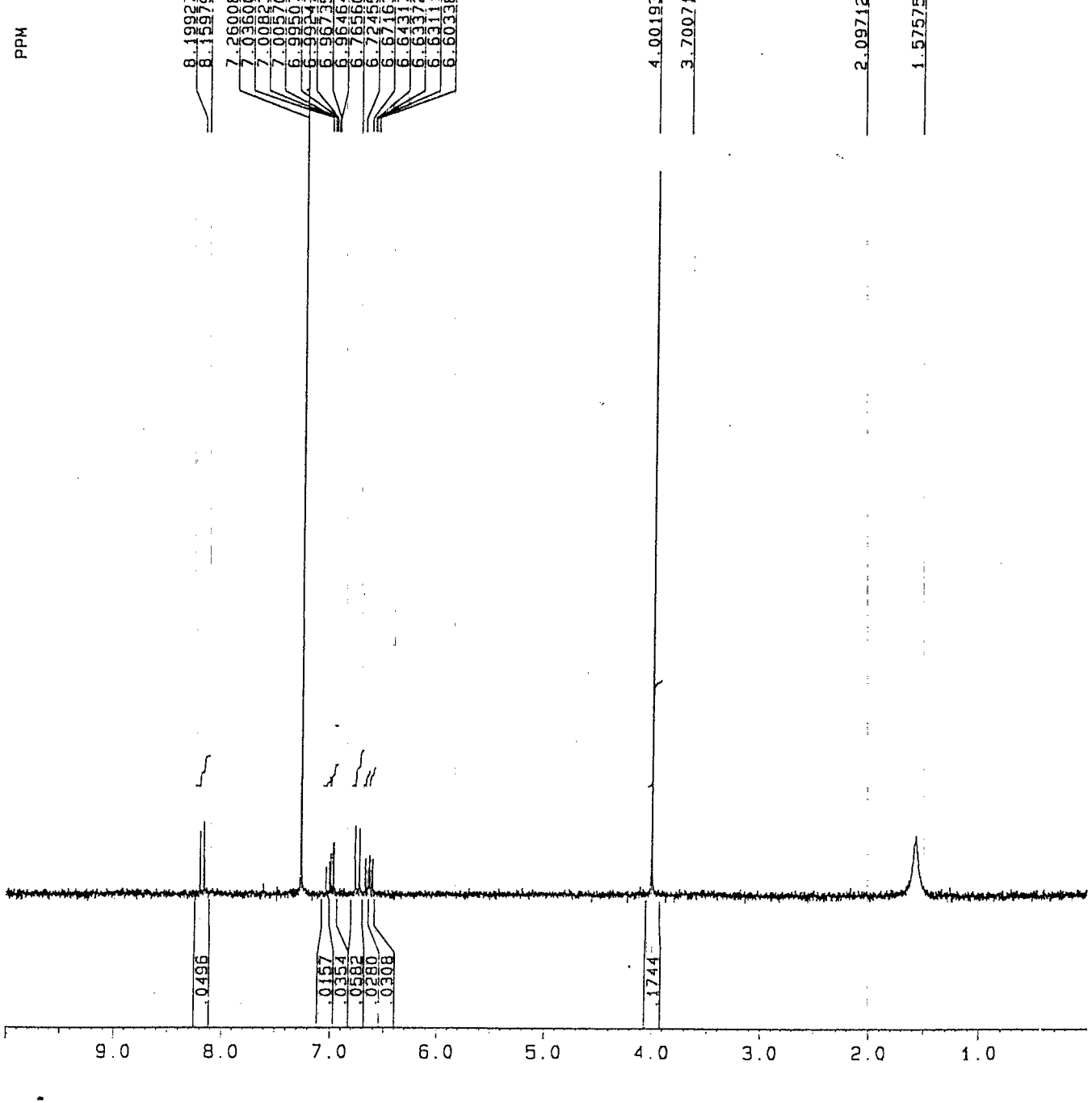


Figure 2c. The X-ray structure (ORTEP drawings) for **5b-2DXN.**



D1012.002
DATE 12-10-95

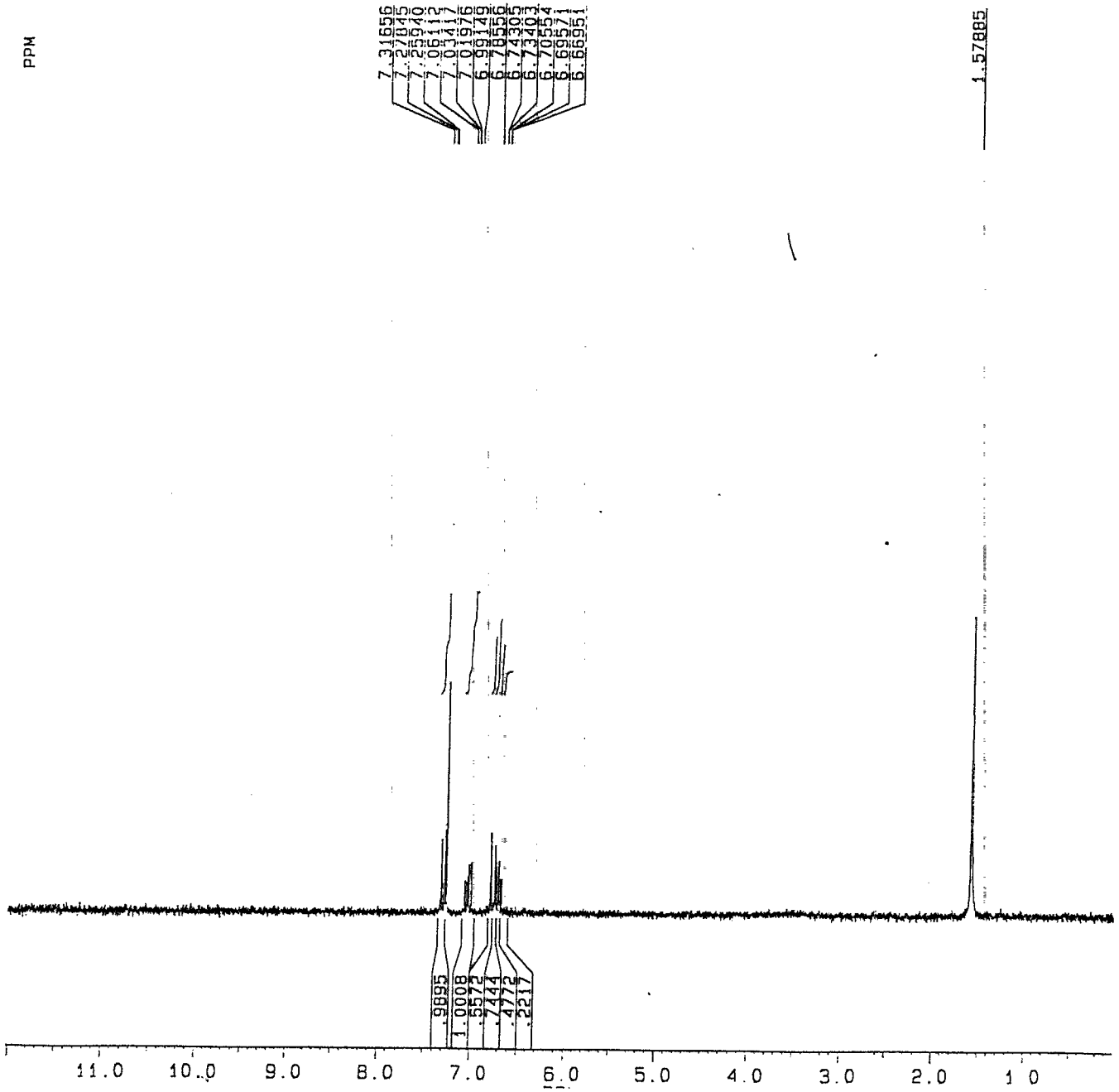
SF 300.133
SY 299.0
O1 5168.181
SI 16384
TD 16384
SW 3597.122
HZ/PT .439

PW 1.0
RD 1.000
AQ 2.277
RG 800
NS 64
TE 297

FW 4500
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 10.000P
F2 .000P
HZ/CM 142.917
PPM/CM .476
SR 3367.28

Figure 3. ^{1}H NMR spectra of **3a**



D0809.007
DATE 9-8-96

SF 300.133
SY 299.0
O1 5018.860
SI 16384
TD 16384
SW 3906.250
HZ/PT .477

PW 1.0
RD 1.000
AQ 2.097
RG 800
NS 64
TE 297

FW 4900
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 12.000P
F2 .001P
HZ/CM 171.502
PPM/CM .571
SR 3367.88

Figure 4. ^{1}H NMR spectra of **3b**

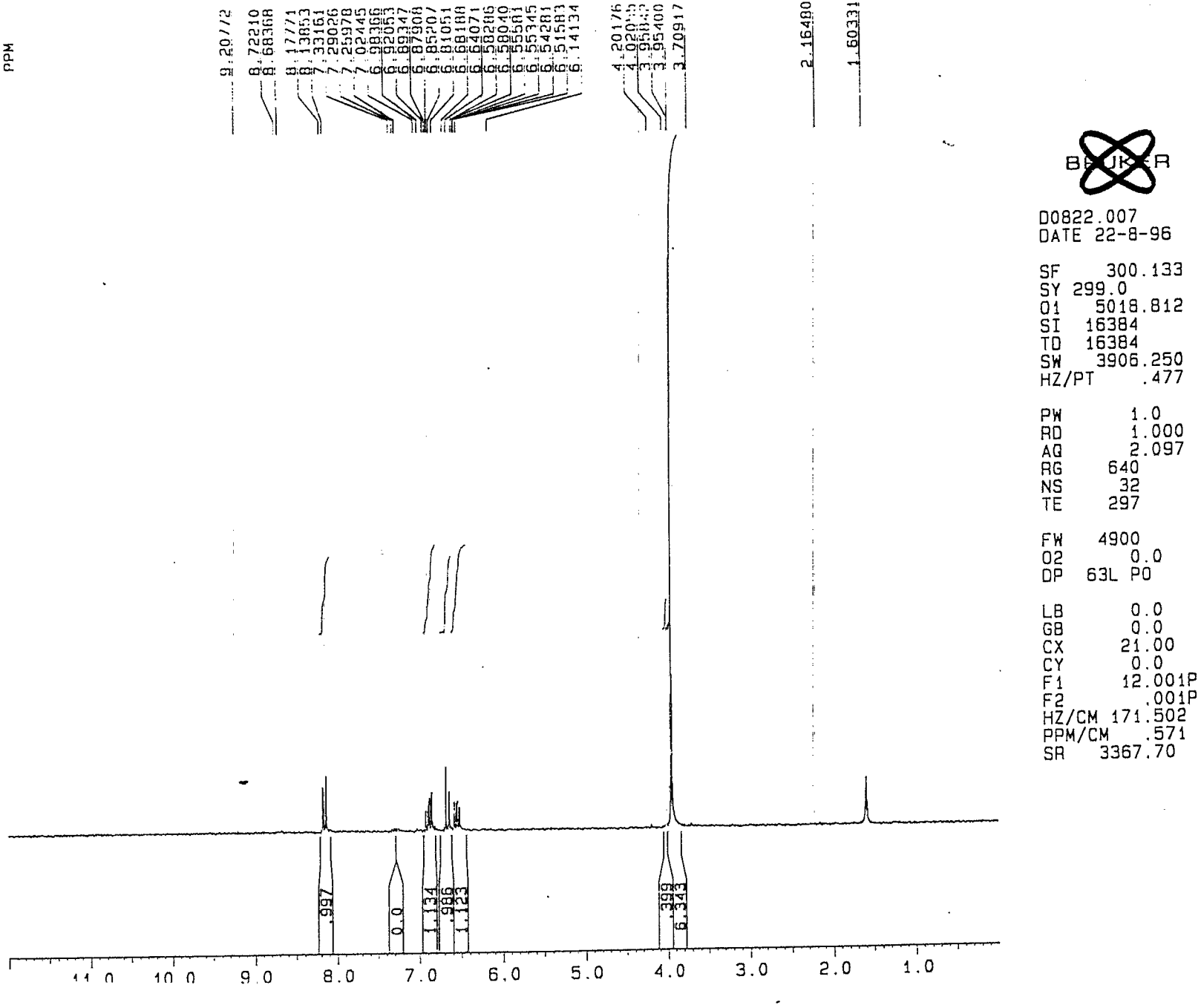
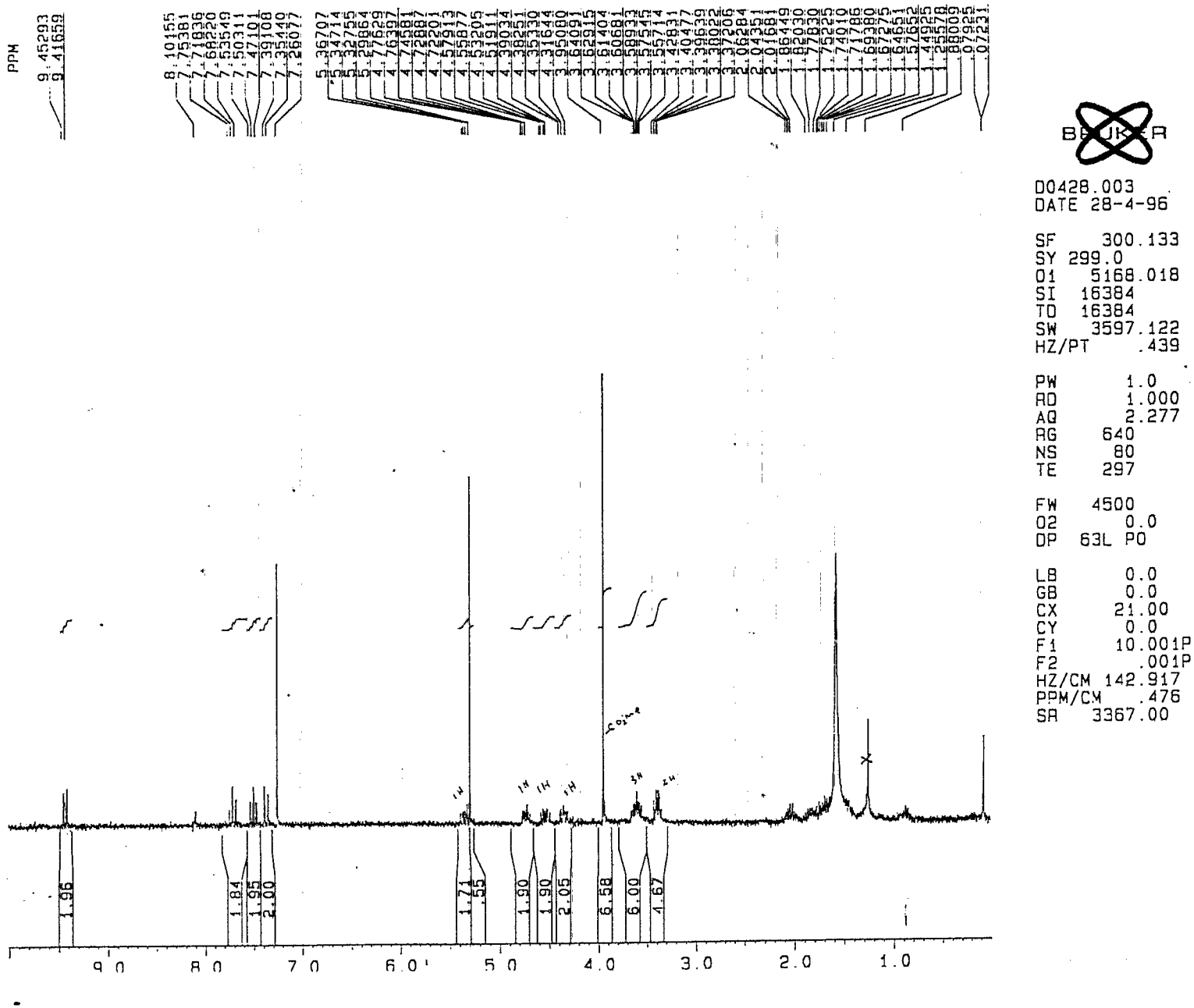


Figure 5. ^{1}H NMR spectra of **3c**



D0428.003
DATE 28-4-96

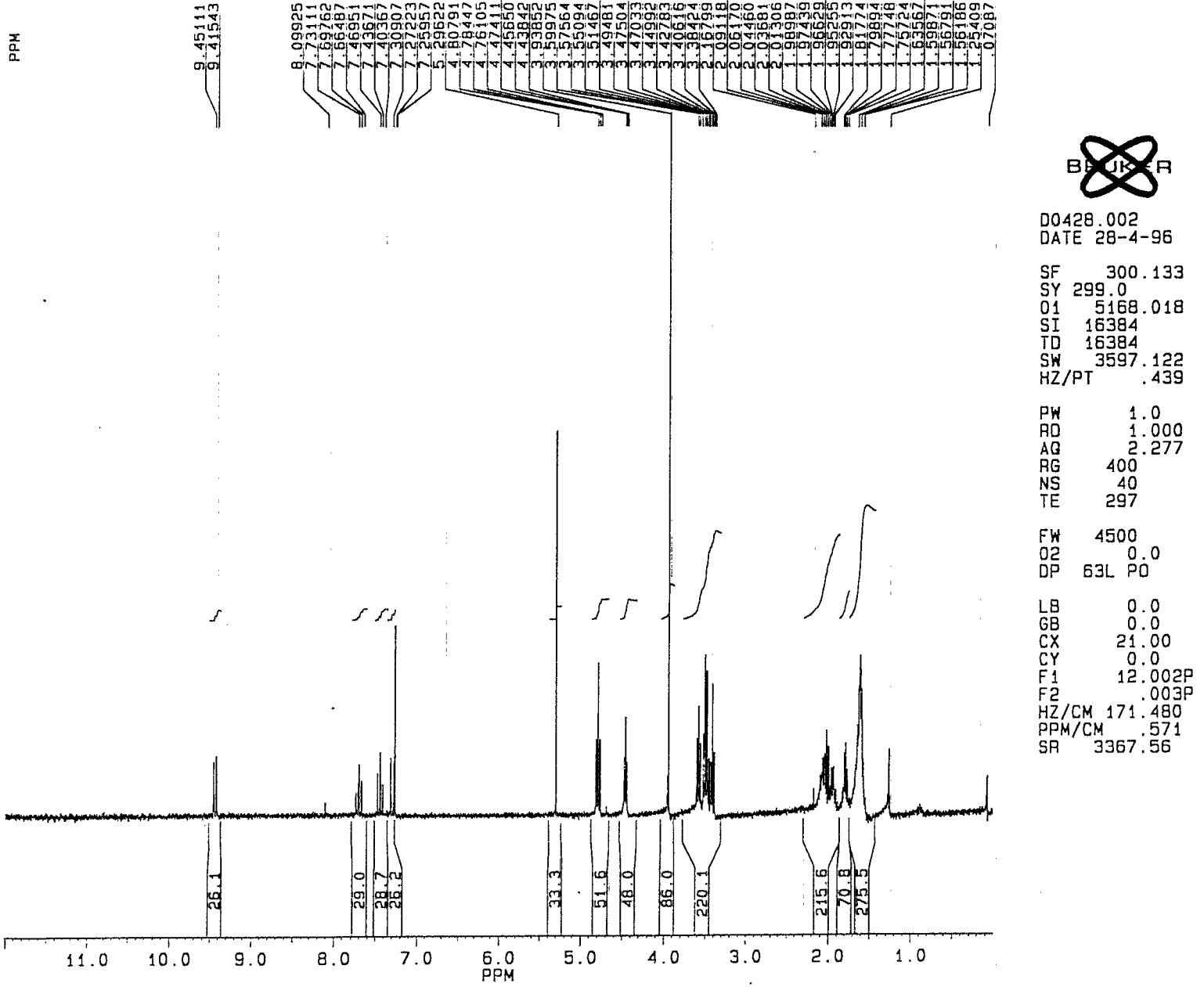
SF 300.133
SY 299.0
O1 5168.018
SI 16384
TD 16384
SW 3597.122
HZ/PT .439

PW 1.0
RD 1.000
AQ 2.277
RG 640
NS 80
TE 297

FW 4500
O2 0.0
OP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 10.001P
F2 .001P
HZ/CM 142.917
PPM/CM .476
SR 3367.00

Figure 6. ^{1}H NMR spectra of **5a-2THF**



BRUKER

D0428.002
DATE 28-4-96

SF 300.133
SY 299.0
O1 5168.018
SI 16384
TD 16384
SW 3597.122
HZ/PT .439

PW 1.0
RD 1.000
AQ 2.277
RG 400
NS 40
TE 297

FW 4500
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 12.002P
F2 .003P
HZ/CM 171.480
PPM/CM .571
SR 3367.56

Figure 7. ^{1}H NMR spectra of **5a-3THF**

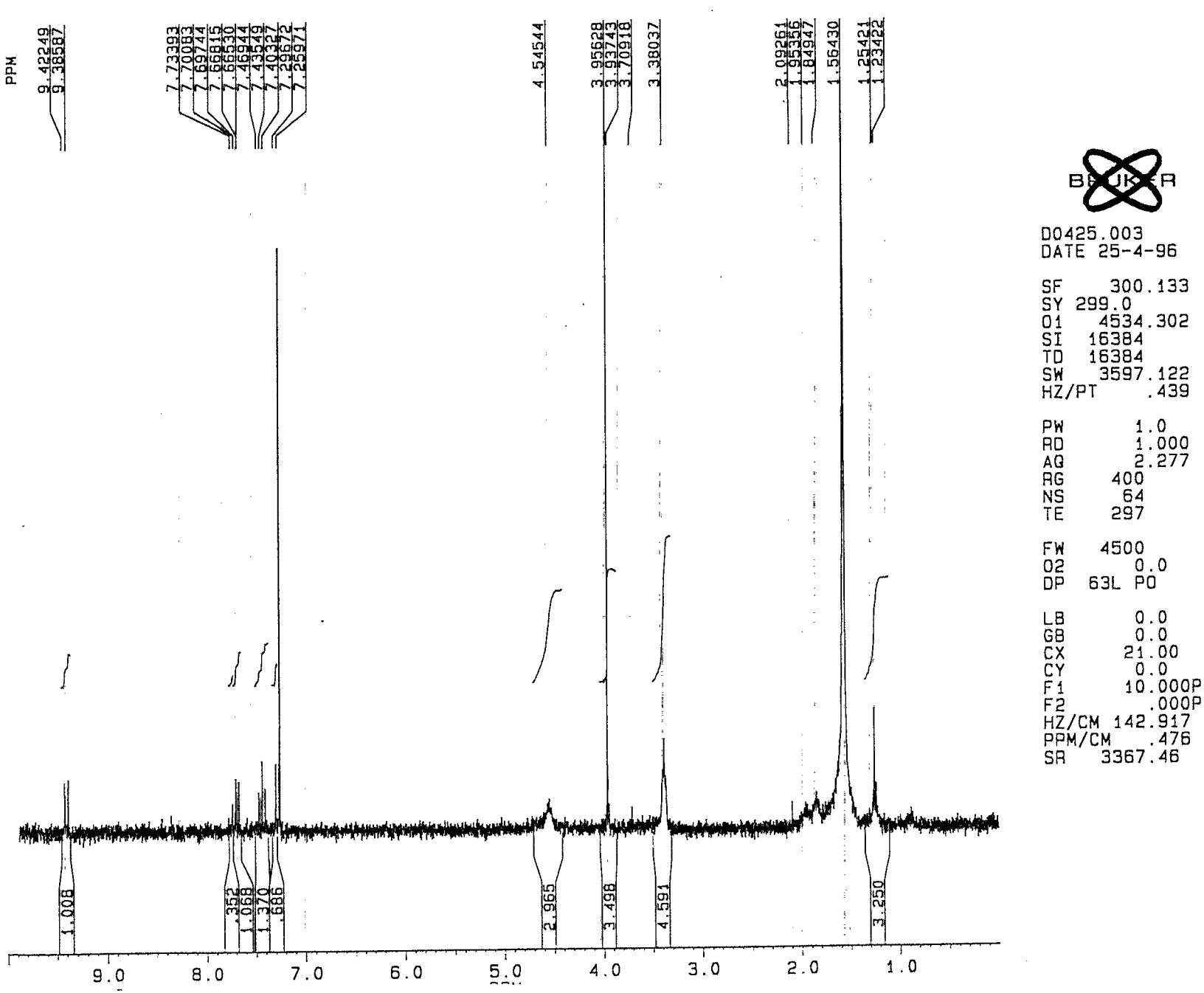


Figure 8. ^{1}H NMR spectra of **5a-2THP**

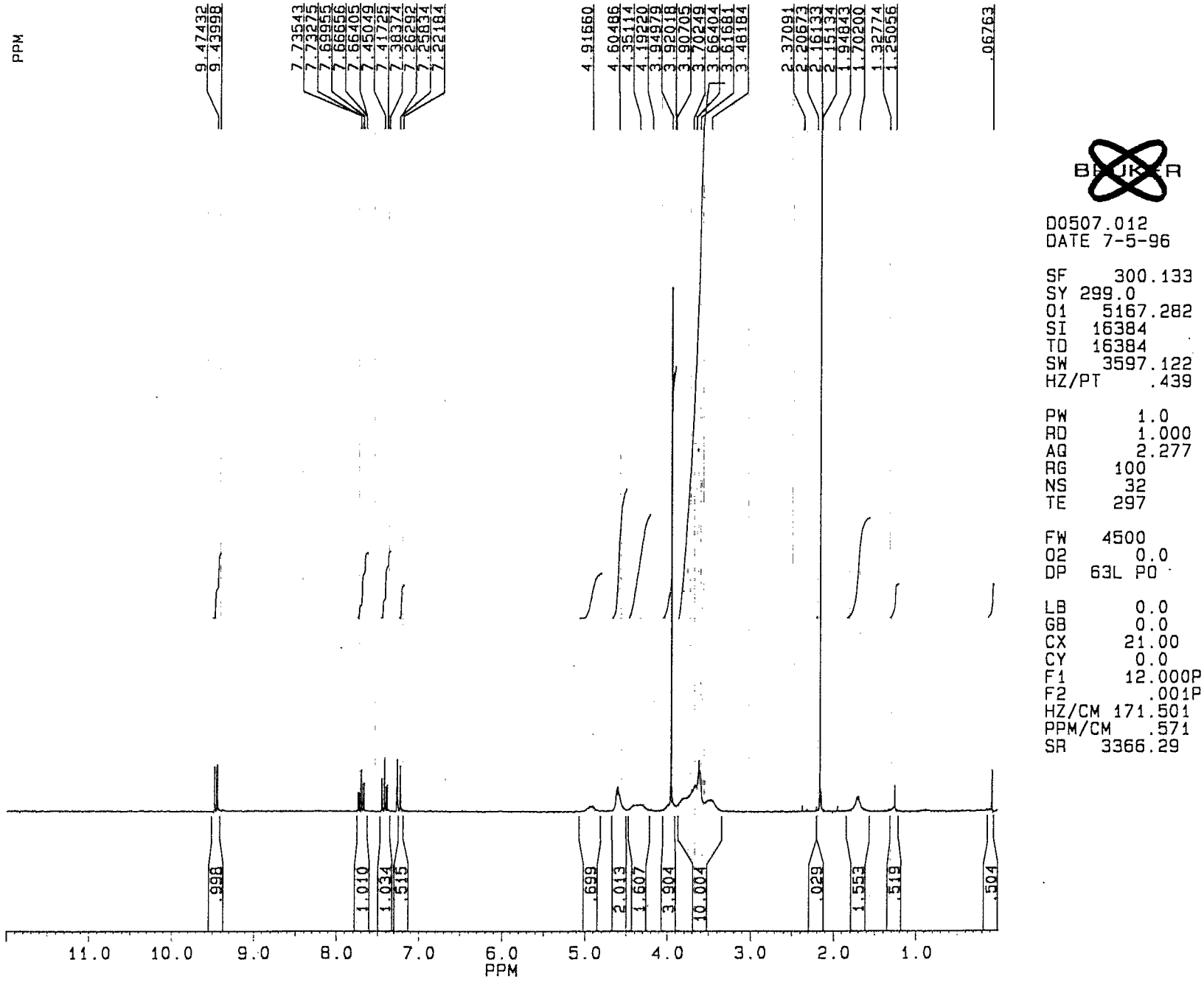
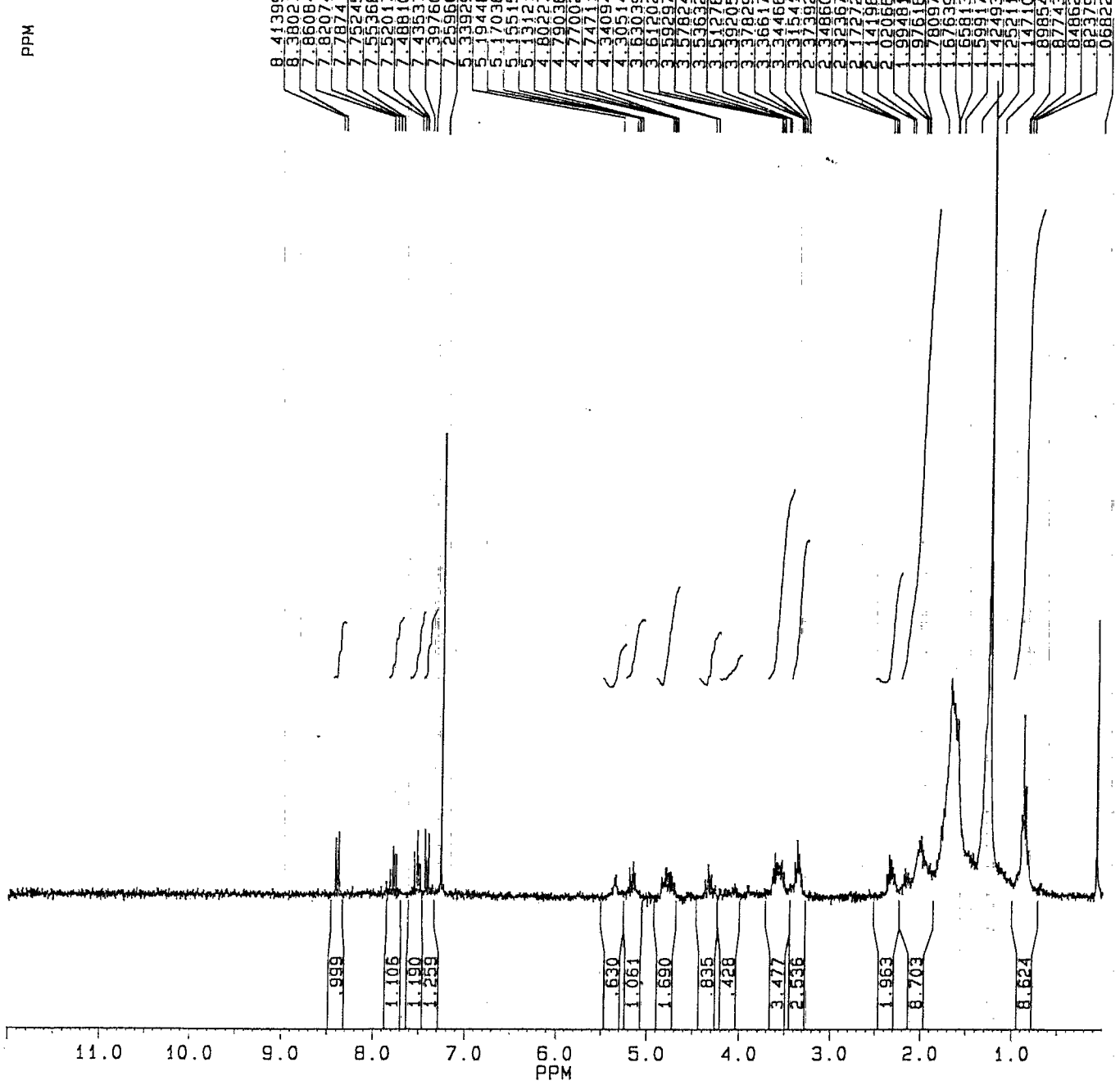


Figure 9. ^{1}H NMR spectra of **5a-2DXN**



D0809.026
DATE 9-8-96

SF 300.133
SY 299.0
O1 4868.111
SI 16384
TD 16384
SW 6024.096
HZ/PT .735

PW 1.0
RD 1.000
AQ 1.360
RG 640
NS 350
TE 297

FW 7600
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 12.001F
F2 .003F
HZ/CM 171.480
PPM/CM .571
SR 3367.88

Figure 10. ^{1}H NMR spectra of **5b-2THF**

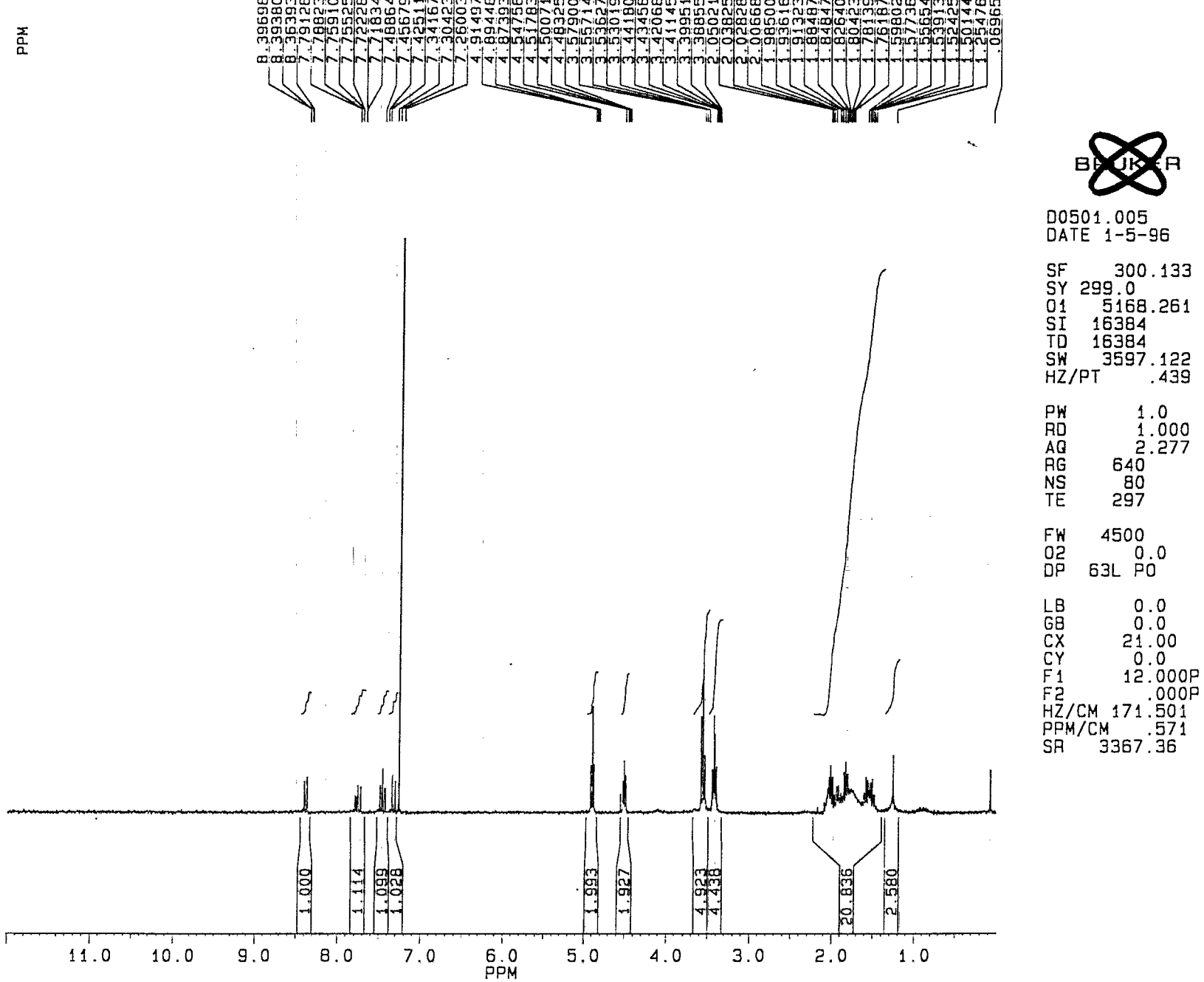


Figure 11. ^{1}H NMR spectra of **5b-3THF**

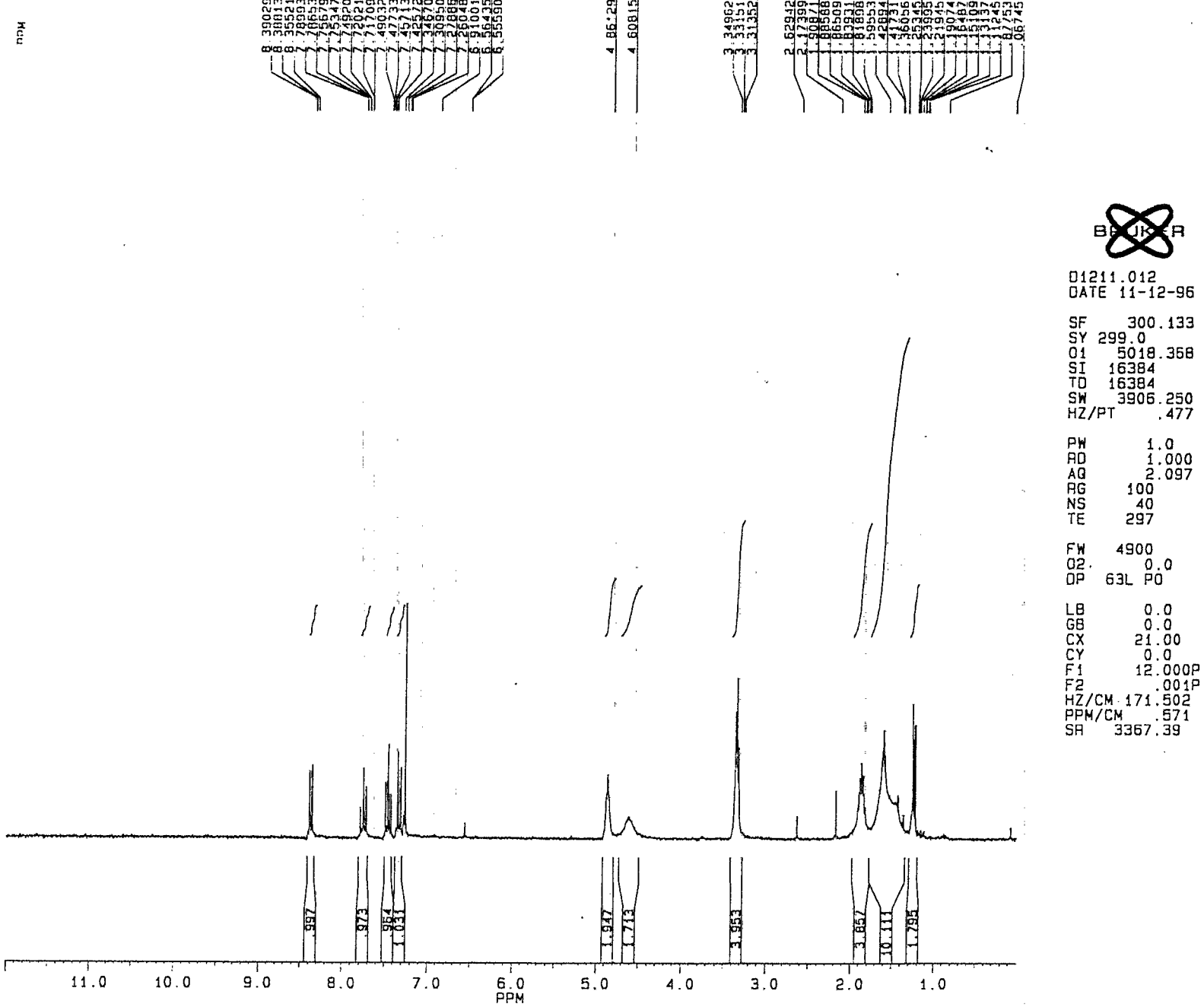
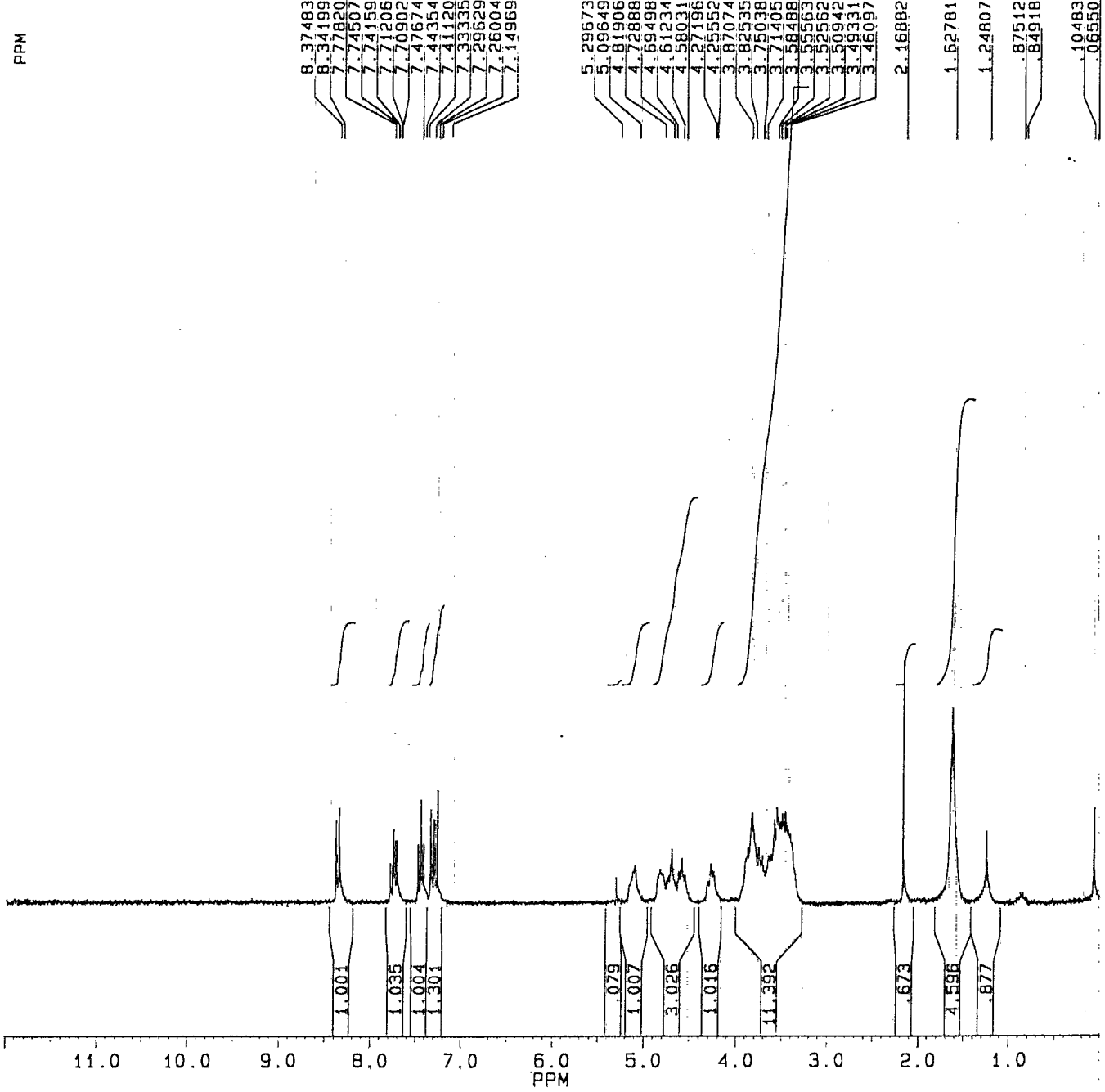


Figure 12. ^{1}H NMR spectra of **5b-2THP**



D0527.014
DATE 27-5-96

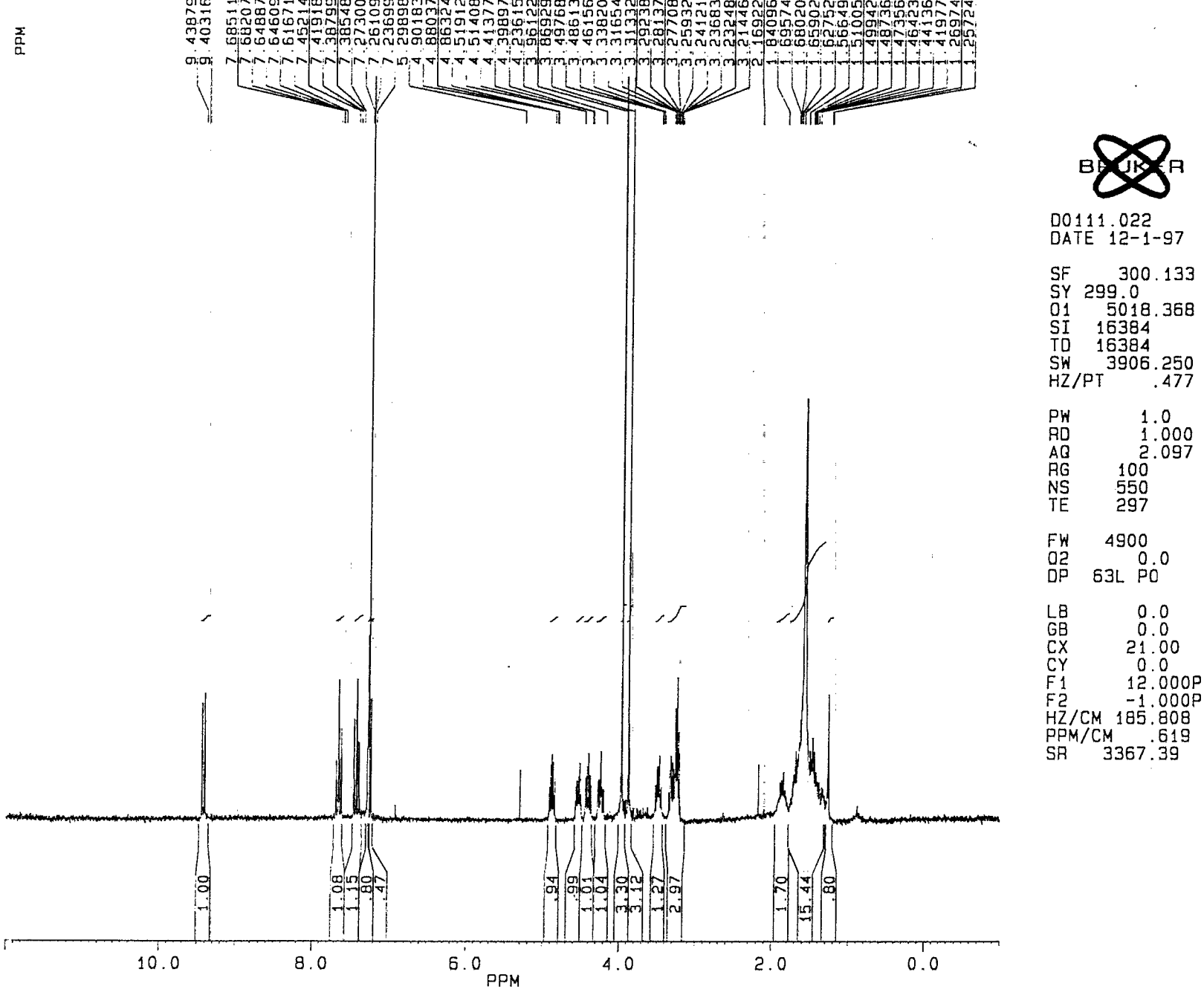
SF 300.133
SY 299.0
O1 5169.488
SI 16384
TD 16384
SW 3597.122
HZ/PT .439

PW 1.0
RD 1.000
AQ 2.277
RG 320
NS 150
TE 297

FW 4500
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 12.000P
F2 .001P
HZ/CM 171.501
PPM/CM .571
SR 3368.50

Figure 13. ^{1}H NMR spectra of **5b-2DXN**



D0111.022
DATE 12-1-97

SF 300.133
SY 299.0
O1 5018.368
SI 16384
TD 16384
SW 3906.250
HZ/PT .477

PW 1.0
RD 1.000
AQ 2.097
RG 100
NS 550
TE 297

FW 4900
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 12.000P
F2 -1.000P
HZ/CM 185.808
PPM/CM .619
SR 3367.39

Figure 14. ^{1}H NMR spectra of **5c-2THF**

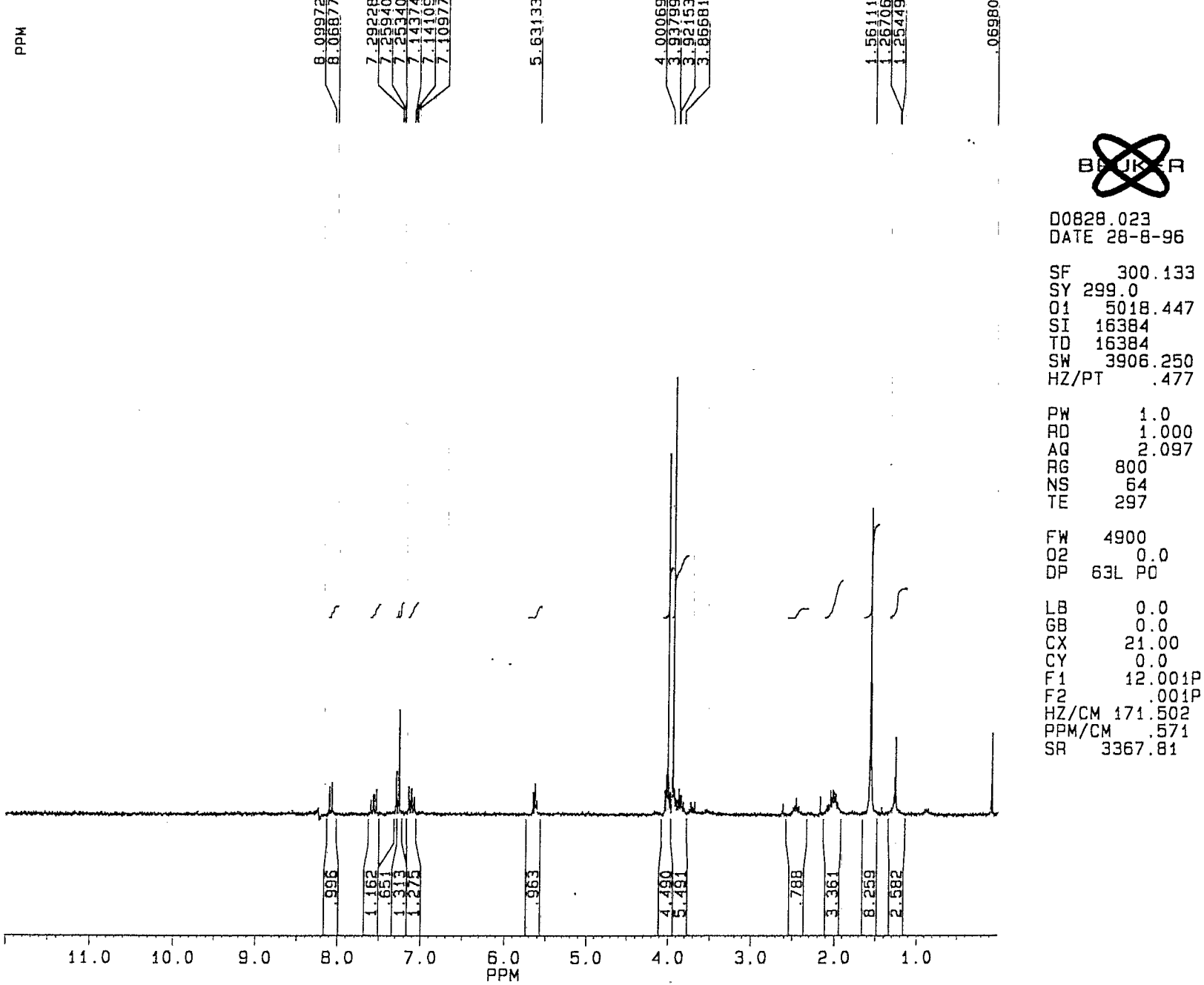
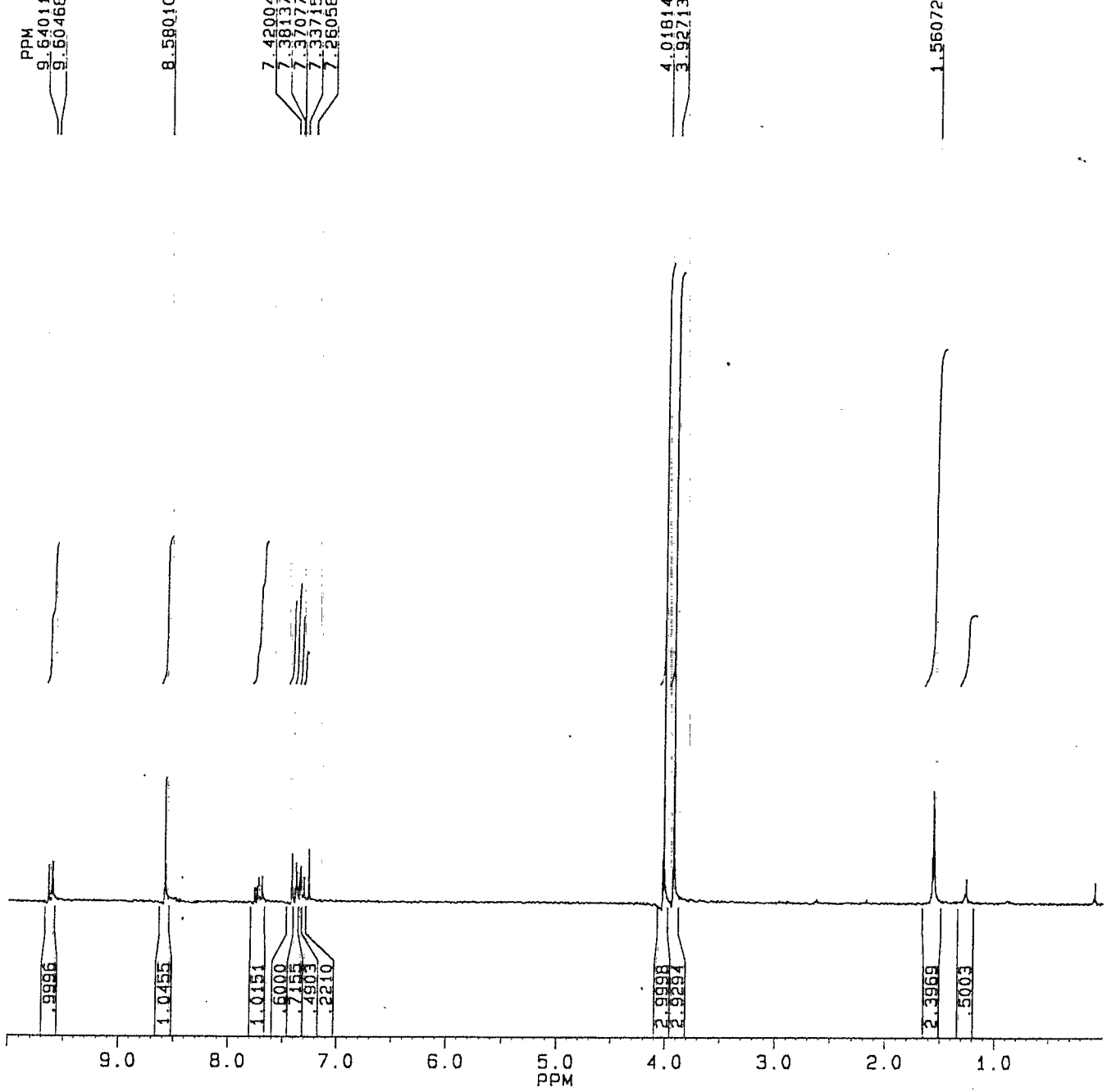


Figure 15. ^{1}H NMR spectra of **6**



D0826.009
DATE 26-8-96

SF 300.133
SY 299.0
O1 5018.447
SI 16384
TD 16384
SW 3906.250
HZ/PT .477

PW 1.0
RD 1.000
AQ 2.097
RG 800
NS 64
TE 297

FW 4900
O2 0.0
DP 63L PO

LB 0.0
GB 0.0
CX 21.00
CY 0.0
F1 10.000P
F2 .000P
HZ/CM 142.915
PPM/CM .476
SR 3367.60

Figure 16. ^{1}H NMR spectra of 7