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REVISED

1

Supporting Material

To accompany "The Reaction of Resorcinol With α,β -Unsaturated Ketones" by P. Livant
and W. Xu; MS No. JO9715478

(a) More Complete Lists of Mass Spectral Peaks for 9 - 12, 13b - 17

4-(3,4-dihydro-7-hydroxy-2-methyl-2H-1-benzopyran-2-yl)-1,3-benzenediol, 9. MS (EI): m/z (rel intens) 272 (M+, 17), 257(11), 150(45), 149(12), 147(21), 137(28), 135(22), 123(100), 107(16), 91(10), 84(11), 79(10), 77(21), 69(14).

4-(3,4-dihydro-7-hydroxy-2-ethyl-2H-1-benzopyran-2-yl)-1,3-benzenediol, 10. MS (EI): m/z (rel intens) 286 (M+, 6), 257(21), 164(7), 149(15), 147(22), 123(100), 107(9), 91(7), 77(12), 69(10).

4-(3,4-Dihydro-7-hydroxy-2-methyl-4-phenyl-2H-1-benzopyran-2-yl)-1,3-benzenediol, 11. MS (EI): m/z (rel intens) 348 (M+, 1), 300(13), 285(7), 257(8), 238(11), 223(9), 199(34), 197(12), 175(25), 163(14), 152(14), 151(100), 150(43), 149(13), 137(26), 135(44), 123(31), 115(11), 107(24), 91(14), 79(15), 77(28), 69(18), 65(11), 55(12), 43(16).

2',4',7-Trihydroxy-2,3-norbornanoflavan, 12. MS (EI): m/z (rel intens) 325 (M+1, 4), 324 (M⁺, 20), 271(10), 255(3), 202(17), 201(100), 199(10), 173(12), 147(21), 123(69), 110(18), 77(10), 67(24), 55(11).

11,11-Dimethyl-2',4',7-trihydroxy-2,4-propanoflavan, 13b. MS(EI): m/z (rel intens) 327(M+1, 5), 326(M, 27), 271(10), 270(52), 269(21), 256(16), 255(100), 164(10), 161(29), 160(9), 148(15), 147(13), 127(9), 123(38), 110(6), 107(8), 91(8), 77(12), 69(12), 55(9), 43(10).

2',4',7-Trihydroxy-2,4-(1,3-cholestano)flavan, 14. MS (EI): m/z (rel intens) 587(M+1, 2), 586(M, 7), 476(2), 309(4), 296(3), 283(4), 271(21), 270(100), 269(15), 257(26), 256(17), 255(97), 178(9), 163(10), 161(36), 160(11), 148(14),

147(14), 123(79), 107(12), 95(15), 93(9), 91(9), 81(17), 79(10), 71(10), 69(17), 57(19), 55(22), 43(27), 41(12).

2,2'-Spirobi(7-hydroxy-4-phenylchroman), 17. MS (EI): *m/z* (rel intens) 437(M+1, 2), 436(M, 4), 238(4), 237(10), 236(3), 223(6), 200(13), 270(100), 199(100), 198(4), 197(18), 196(2), 181(3), 165(4), 161(6), 152(6), 141(5), 131(3), 128(5), 115(9), 91(4), 69(4).

(b) MS, $^1\text{H-NMR}$, and $^{13}\text{C-NMR}$ Data for Acetate Derivatives

Triacetate from 12: MS (EI): *m/z* (rel intens) 450(M⁺, 2), 408(12), 366(3), 243(12), 202(17), 201(100), 160(25), 147(16), 123(55), 79(14), 67(14), 43(54). $^1\text{H-NMR}$ (CD₃CN, 400 MHz): 7.401 (d, *J* = 9.2, 1H), 6.995 (d, *J* = 8.0, 1H), 6.89-6.86 (m, 2H), 6.504 (dd, *J* = 7.9, 1.8, 1H), 6.446 (d, *J* = 1.9, 1H), 3.324 (m, approx. d, *J* = 4.8, 1H), 3.00-2.87 (m, 2H), 2.563 (m, approx. quintet *J* = 1.8, 1H), 2.436 (m, approx. quintet *J* = 1.8, 1H), 2.221 (s, 3H), 2.214 (s, 3H), 2.206 (s, 3H), 1.799 (m, approx. d, *J* = 10.2, 1H), 1.54-1.27 (m, 4H), 0.81-0.73 (m, 1H). $^{13}\text{C-NMR}$ (CD₃CN, 100.6 MHz): 170.48 (C=O), 170.35 (C=O), 170.10 (C=O), 162.47 (quat), 152.32 (quat), 150.98 (quat), 150.78 (quat), 134.04 (CH), 127.83 (quat), 126.73 (quat), 126.40 (CH), 119.96 (CH), 117.25 (CH), 114.31 (CH), 104.32 (CH), 99.57 (quat), 54.15 (CH), 46.60(CH), 42.34 (CH), 39.48 (CH₂), 38.68 (CH₂), 24.17 (CH₂), 22.754 (CH₂), 21.35 (2CH₃), 21.31(CH₃).

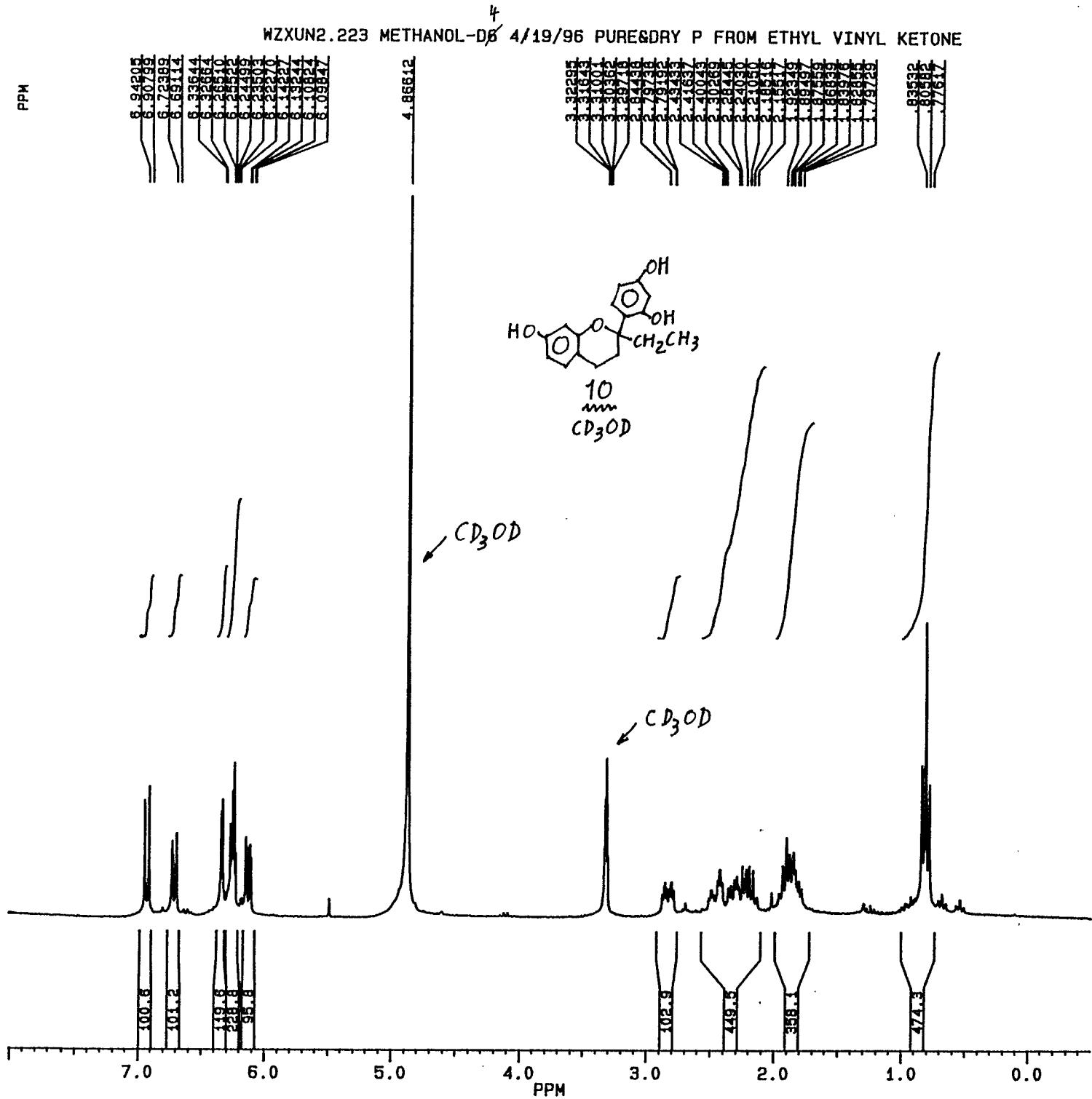
Triacetate from 13b: MS (EI): *m/z* (rel intens) 452(M⁺, 4), 396(17), 354(13), 311(10), 270(9), 269(12), 255(18), 164(15), 161(17), 149(8), 147(15), 135(8), 123(40), 79(22), 69(10), 52(14), 43(100) $^1\text{H-NMR}$ (CDCl₃, 250 MHz): 7.574 (d, *J* = 8.7, 1H), 6.96-7.02 (m, 2H), 6.887 (d, *J* = 2.4, 1H), 6.53-6.57 (m, 2H), 2.544 (m, approx. quintet *J* = 3.3, 1H), 2.445 (dd, *J* = 13.4, 2.8, 1H), 2.0-2.3 (m, 3H), 1.2-1.5 (m, 2H), 2.284 (s, 3H), 2.267 (s, 3H), 2.047 (s, 3H), 1.189 (s, 3H), 0.872 (s, 3H). $^{13}\text{C-NMR}$ (CD₃CN, 63 MHz): 169.35 (C=O), 169.08 (C=O), 168.94 (C=O), 156.05 (quat), 150.27 (quat),

150.14 (quat), 149.03 (quat), 134.00 (quat), 130.67 (CH), 127.18 (CH), 122.63 (quat), 118.59 (CH), 117.95 (CH), 112.05 (CH), 108.89 (CH), 77.33 (quat), 43.41 (CH), 33.87 (quat), 33.65 (CH₂), 31.42 (CH₂), 30.12 (CH), 29.51 (CH₃), 25.28 (CH₃), 21.16 (CH₃), 21.07 (CH₃).

Diacetate from 17: MS (EI): *m/z* (rel intens) 520(M, 1), 461(3), 279(3), 265(2), 242(4), 241(22), 238(11), 237(16), 236(4), 223(8), 199(100), 198(6), 197(24), 196(4), 181(4), 165(6), 161(7), 152(9), 141(6), 115(10), 91(5), 69(4). ¹H-NMR (CDCl₃, 250 MHz): 7.40-7.25 (m, 6H), 6.756 (dd, *J* = 0.8, 8.4, 1H), 6.638 (d, *J* = 2.3, 1H), 6.557 (dd, *J* = 2.3, 8.4, 1H), 4.554 (dd, *J* = 5.5, 12.9 Hz, 1H) 2.408 (dd, *J* = 5.6, 13.4, 1H), 2.235 (s, 3H), 2.150 (app. t, *J* = 13.2, 1H) ¹³C-NMR (CDCl₃, 63 MHz): 169.29 (quat), 152.37 (quat), 149.92 (quat), 143.36 (quat), 129.73 (CH), 128.92 (CH), 128.79 (CH), 127.03 (CH), 123.50 (quat), 114.37 (CH), 110.30 (CH), 96.85 (quat), 40.60 (CH₂), 37.41 (CH), 21.07 (CH₃).

(c) ¹³C NMR of 13b

11,11-Dimethyl-2',4',7-trihydroxy-2,4-propanoflavan, 13b. ¹³C NMR (CD₃CN, 100.6 MHz): 158.96 (quat), 158.51 (quat), 158.13 (quat), 156.57 (quat), 132.11 (CH), 127.90 (CH), 124.66 (quat), 118.62 (quat), 107.49 (CH), 107.41 (CH), 104.86 (CH), 103.03 (CH), 80.30 (quat; C2), 44.57 (CH; C4), 35.13 (CH₂; C9), 34.96 (quat; C11), 33.12 (CH₂; C10), 31.67 (CH₂; C3), 30.43 (CH₃), 25.94 (CH₃).



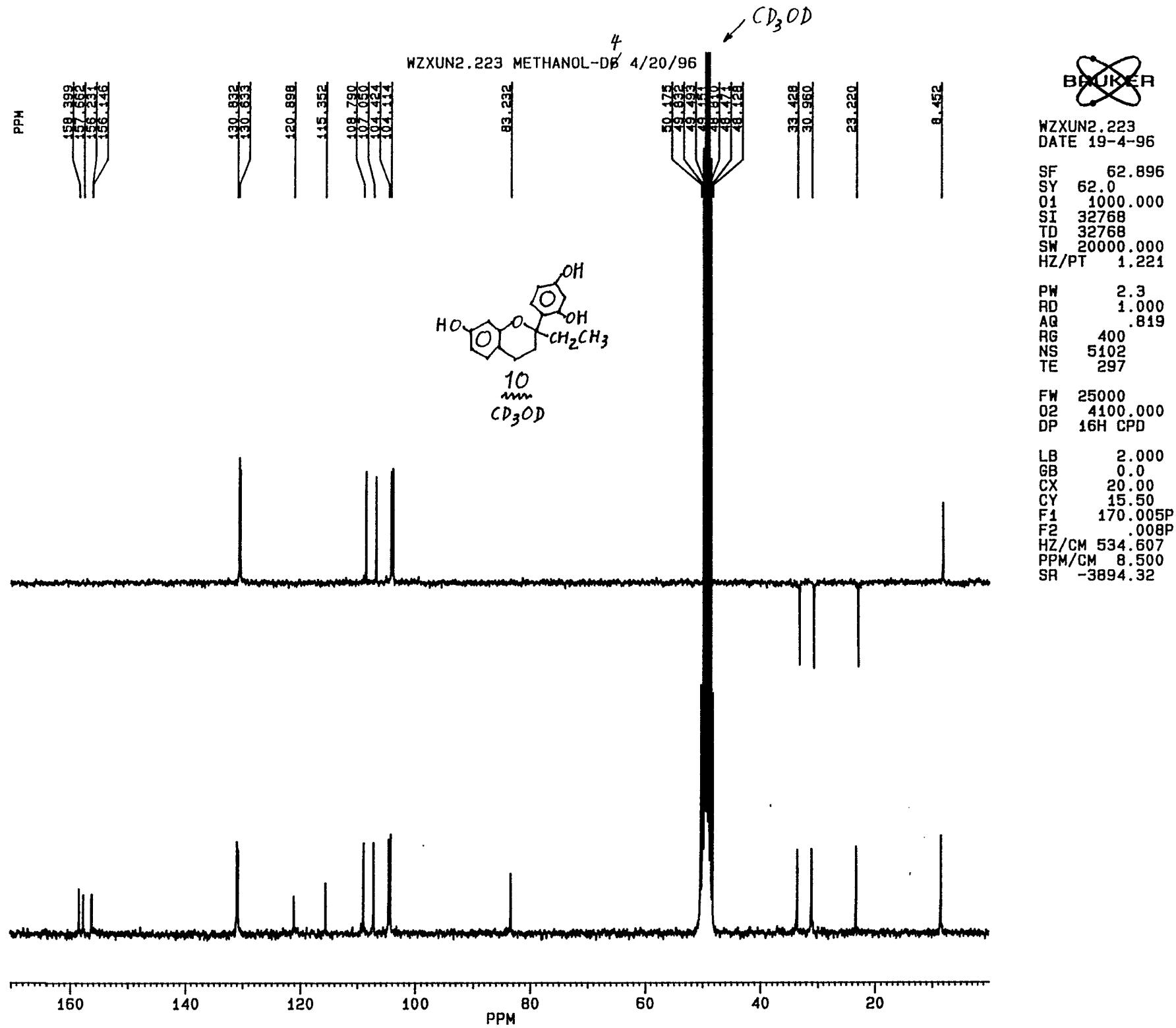
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AQ 3.850
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NS 24
TE 297

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GB 0.0
CX 20.00
CY 13.00
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PPM/CM .425
SR 3838.50



WZXUN3.006 CD₃CN 5/22/96 3RD SPOT



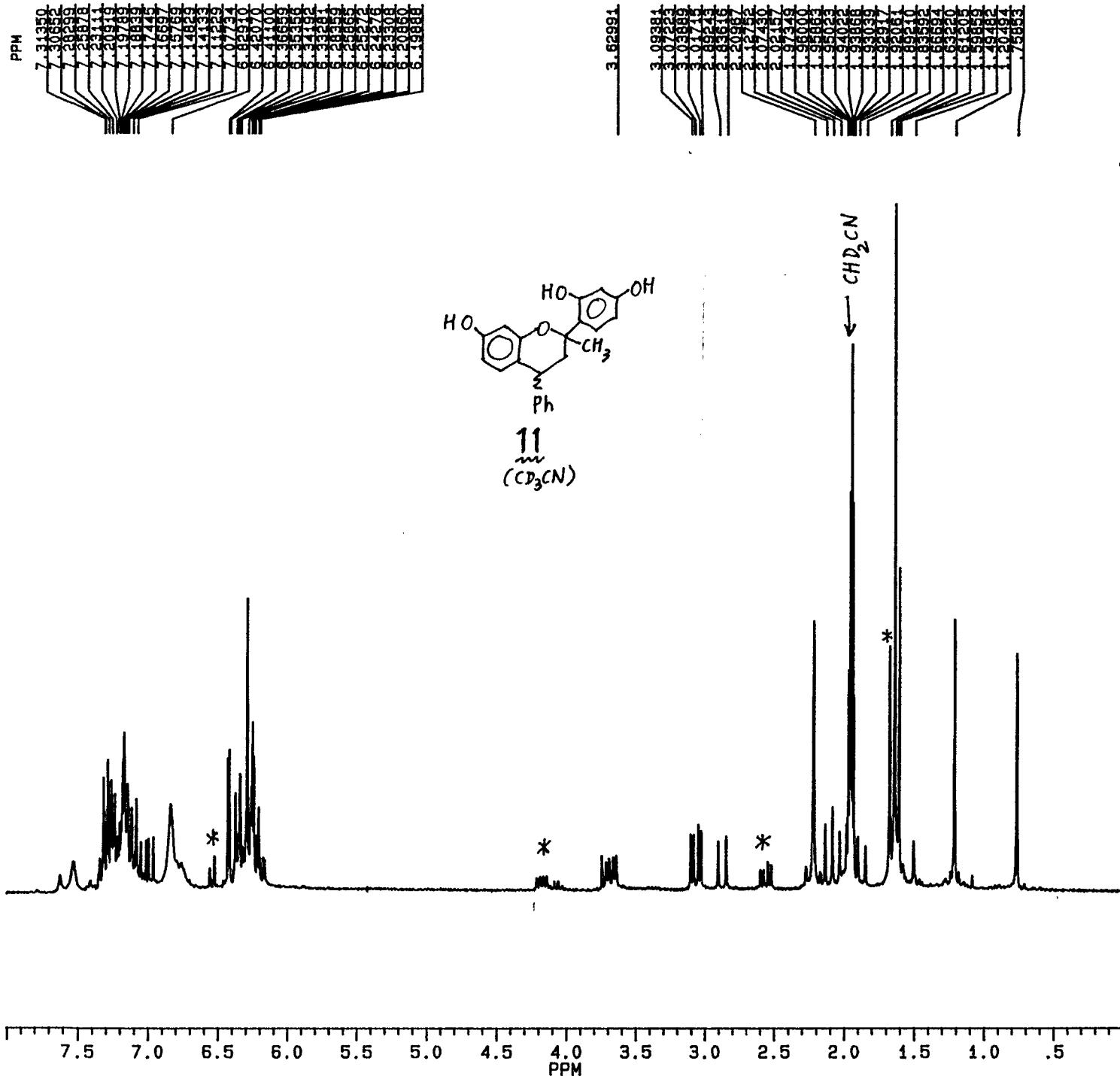
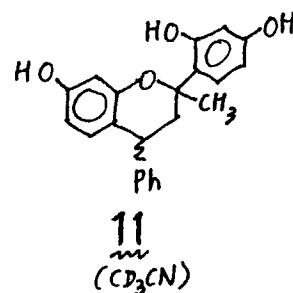
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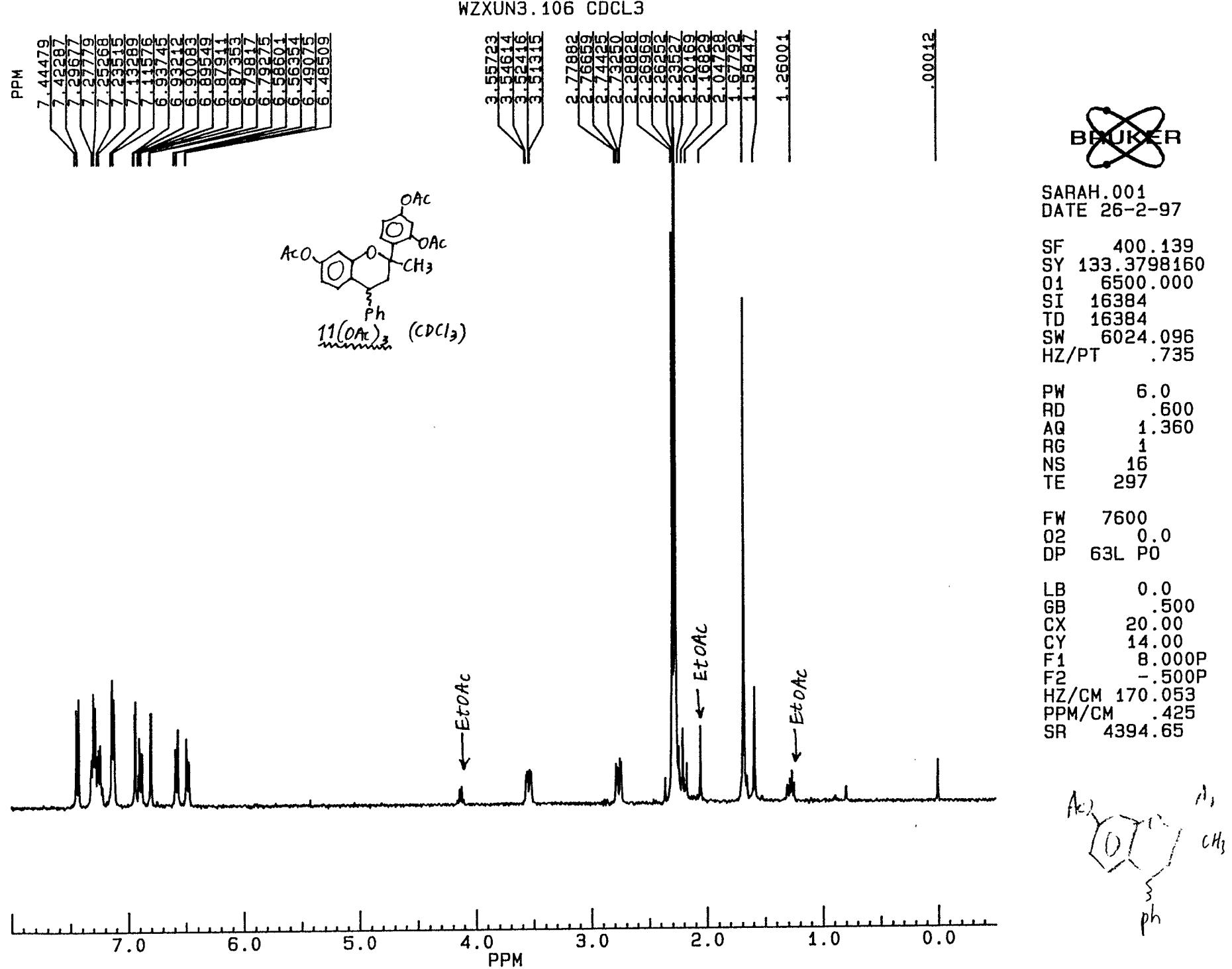
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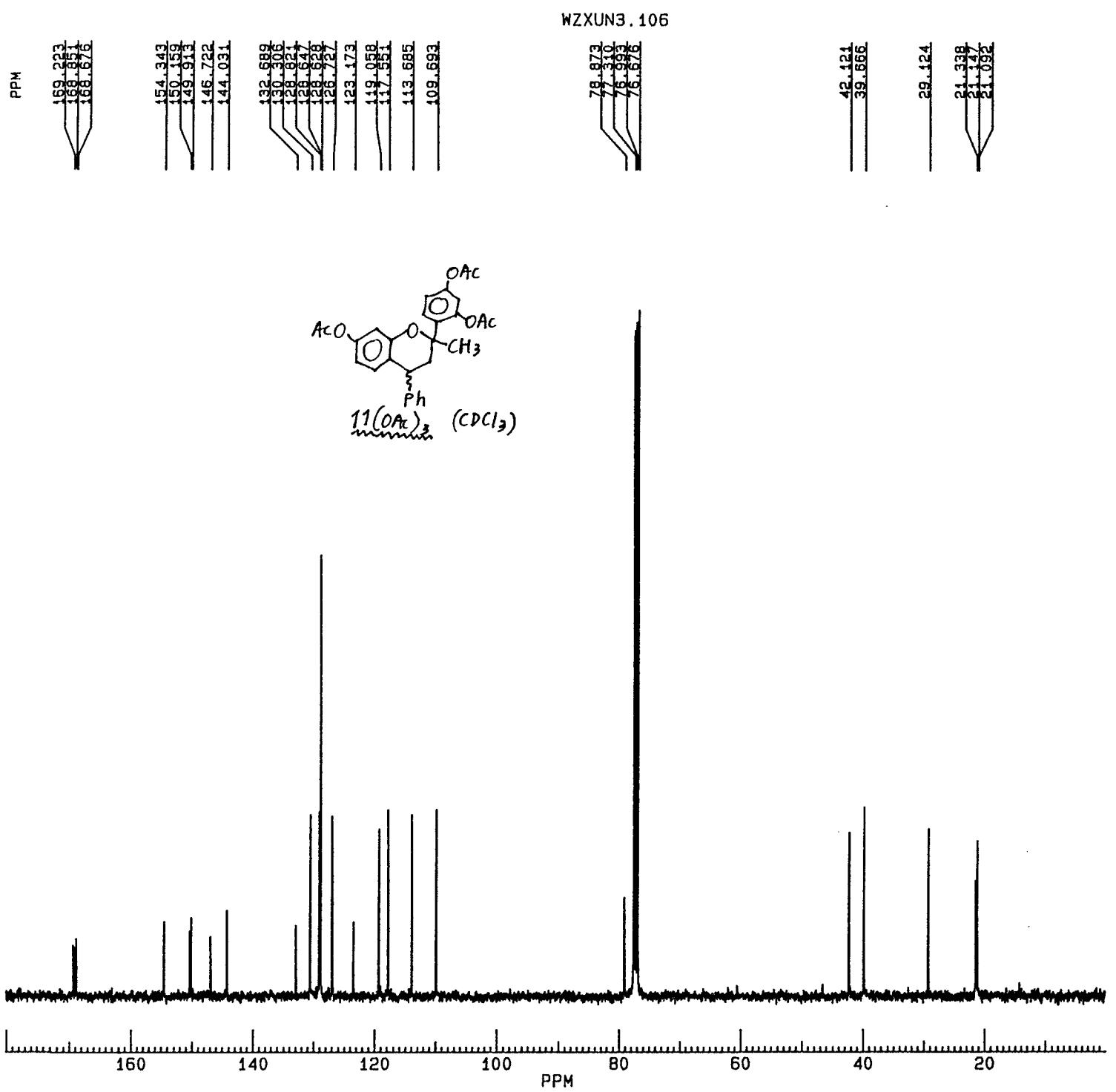
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PPM/CM .400
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* = contamination by
previous fraction





In 53-11



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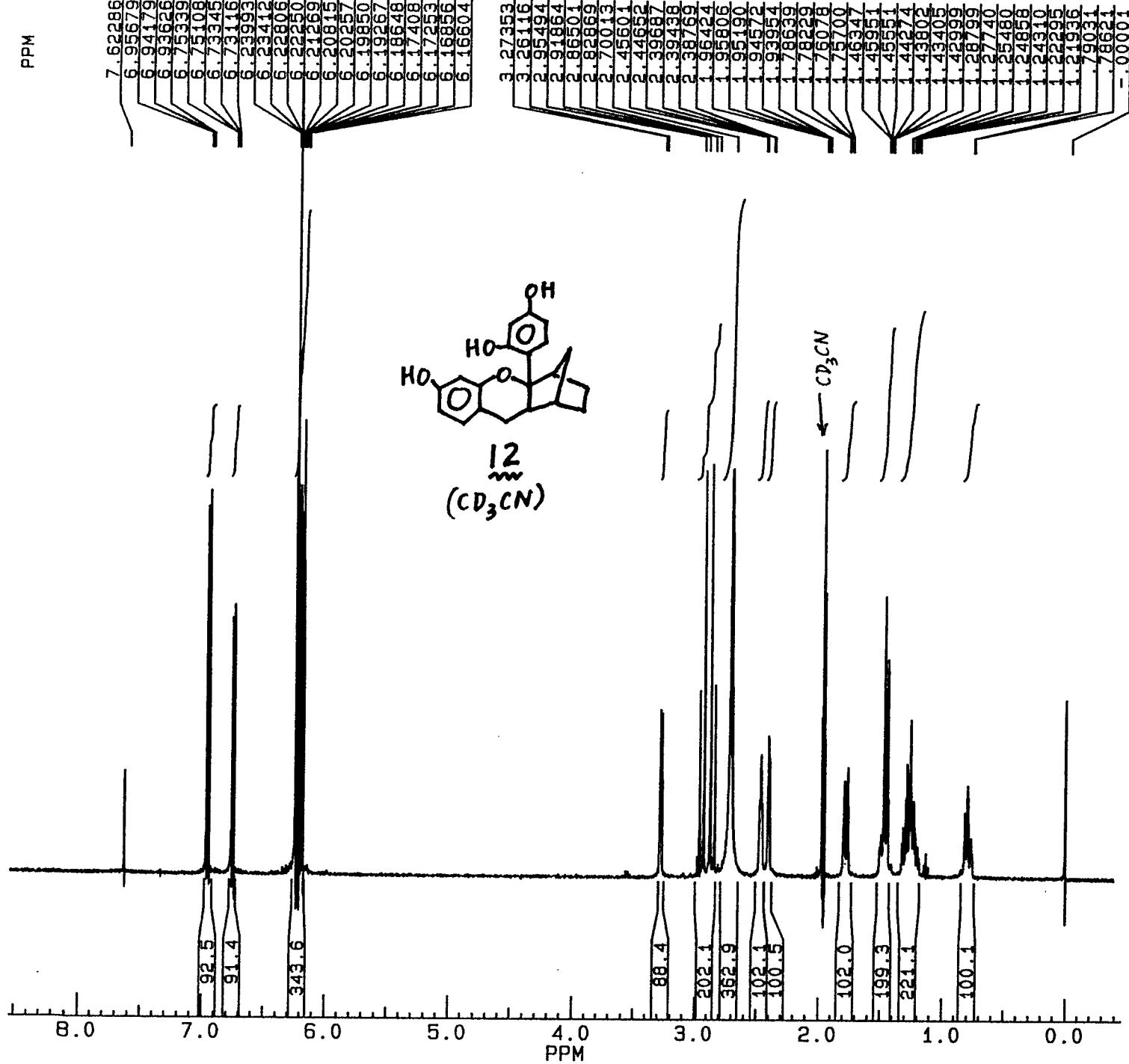
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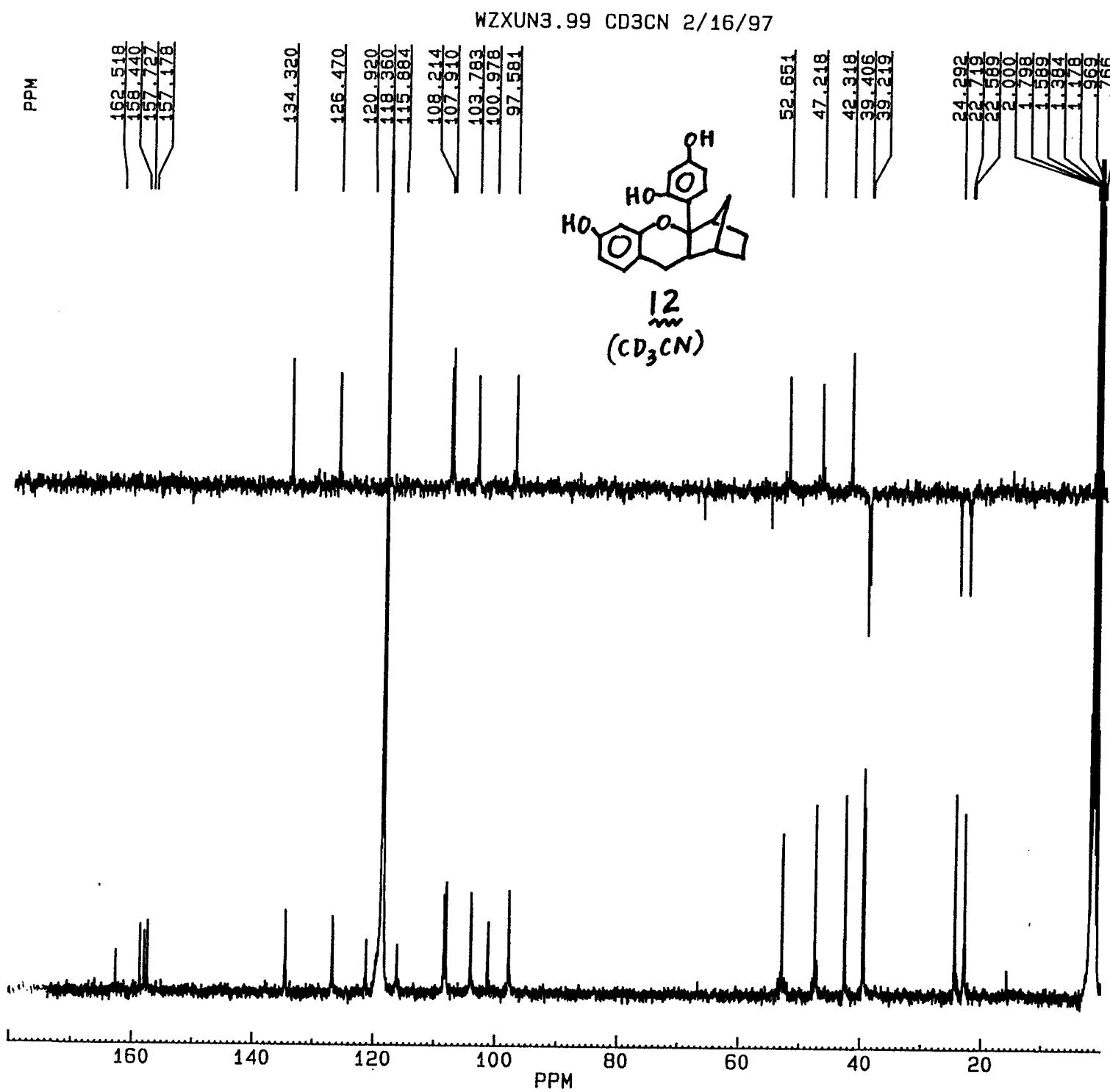
METHYLENE NORBORNANONE + RESORCINOL PRODUCT

PPM



12

N3-P53



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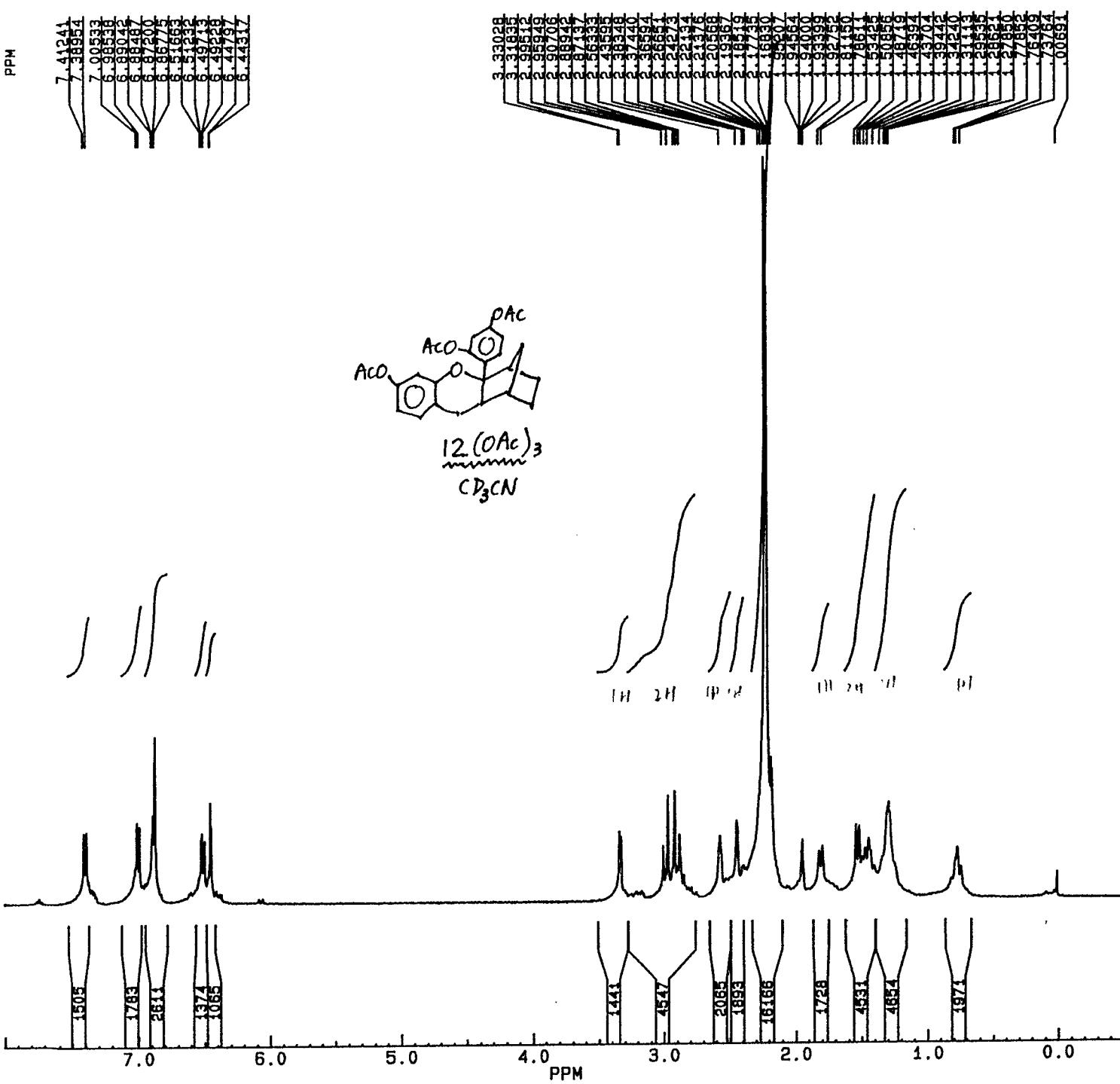
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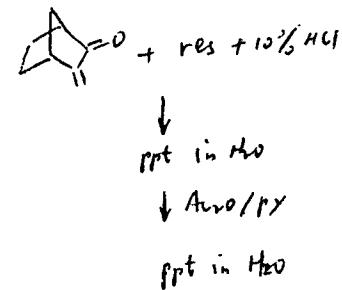
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DATE 16-2-97

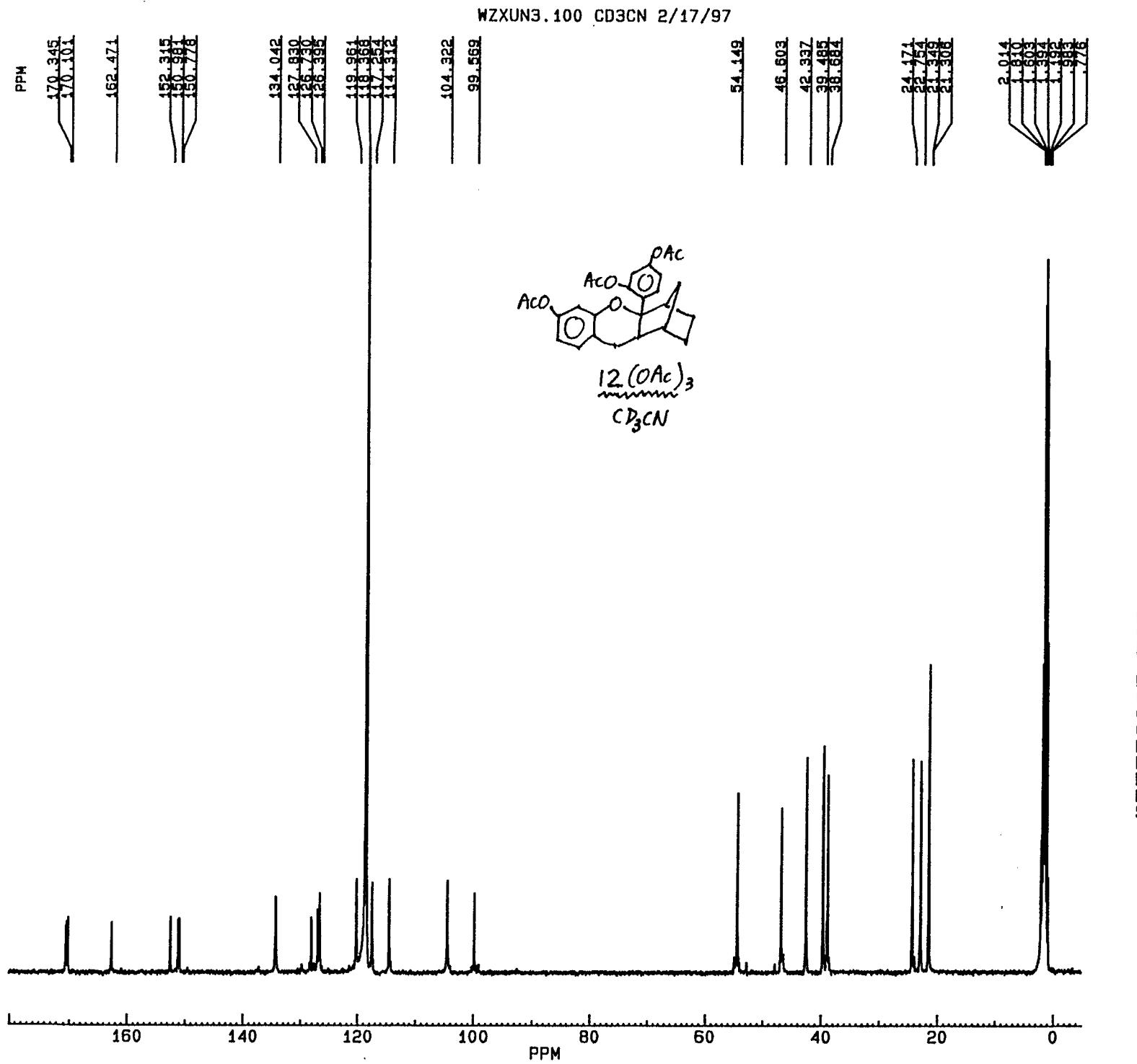
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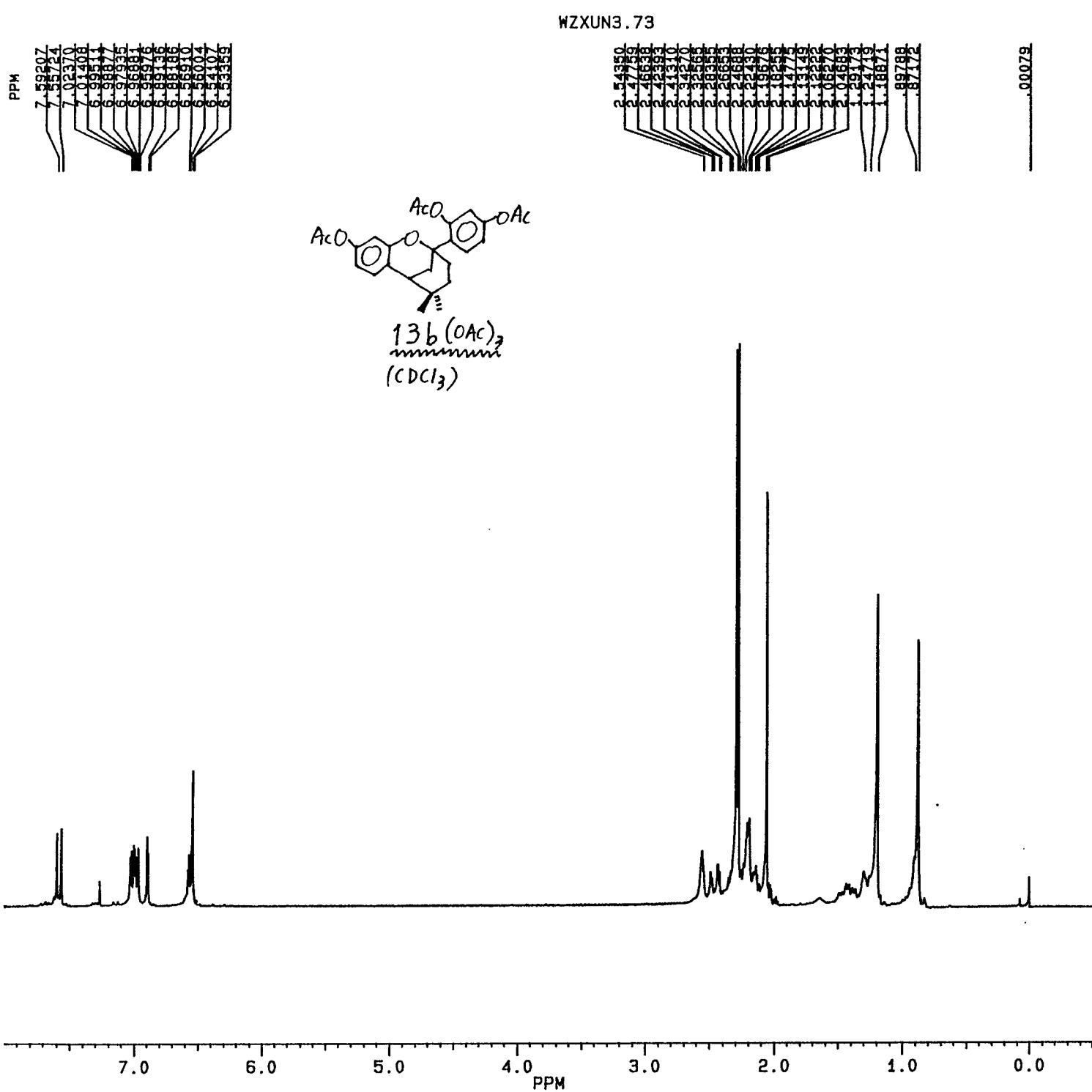
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SR 6521.45







BRUKER

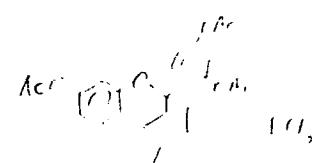
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DATE 7-1-97

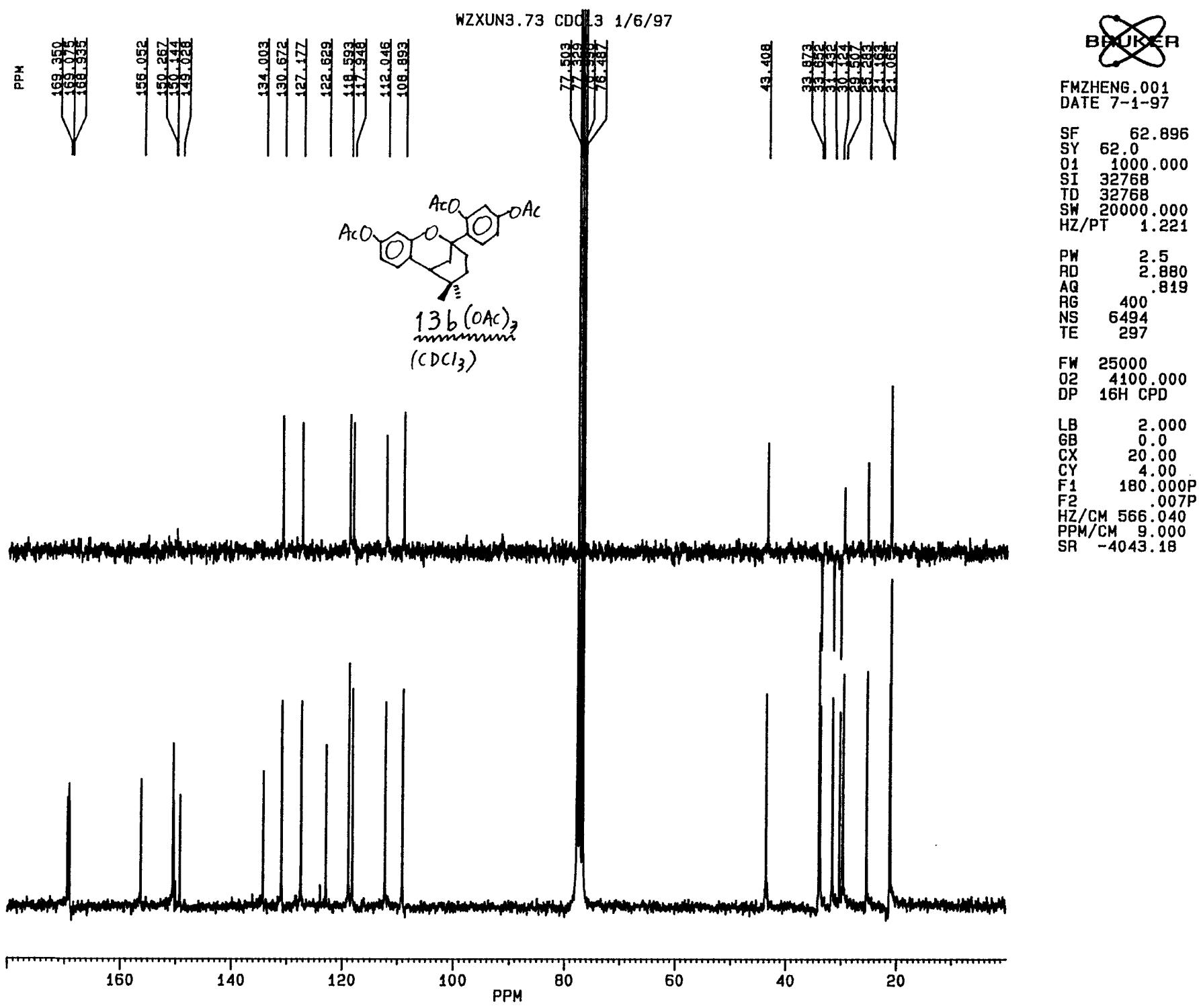
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LB 0.0
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CX 20.00
CY 10.00
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SR 2854.69





WZXUAU.011 ACET-D6

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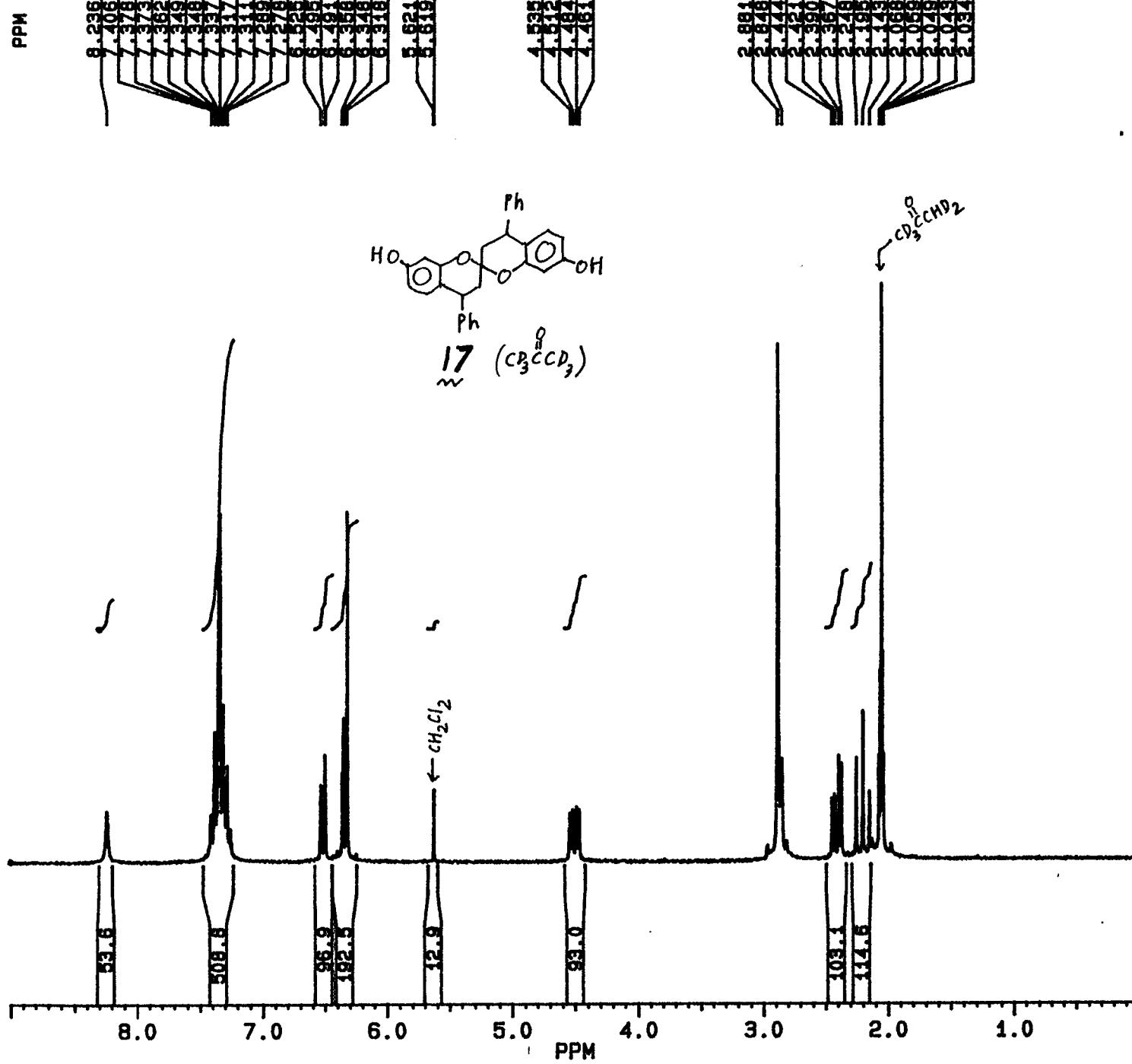
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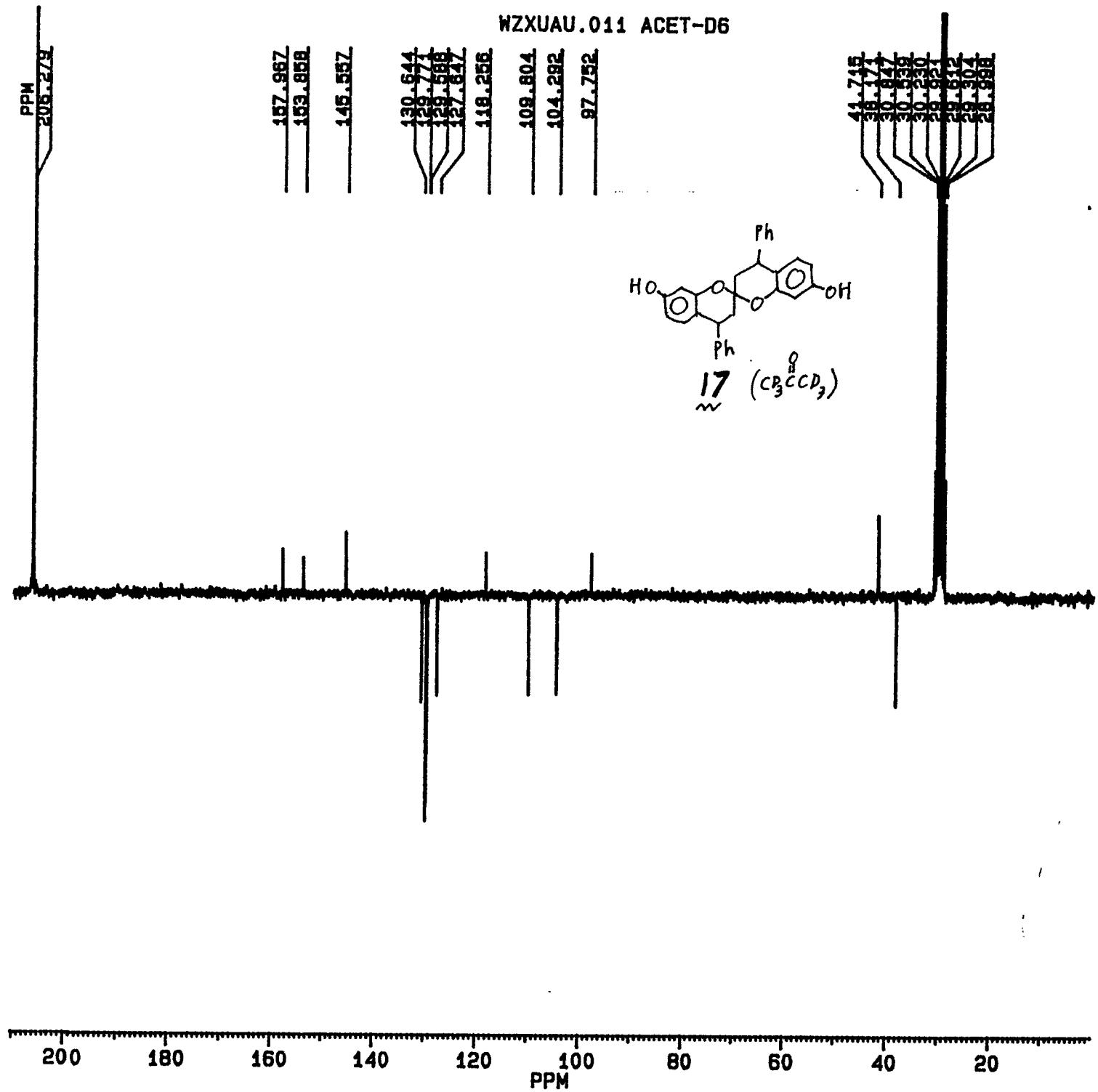
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SR 4152.76





WXAUJ011.001
AU PROG:
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DATE 19-6-97

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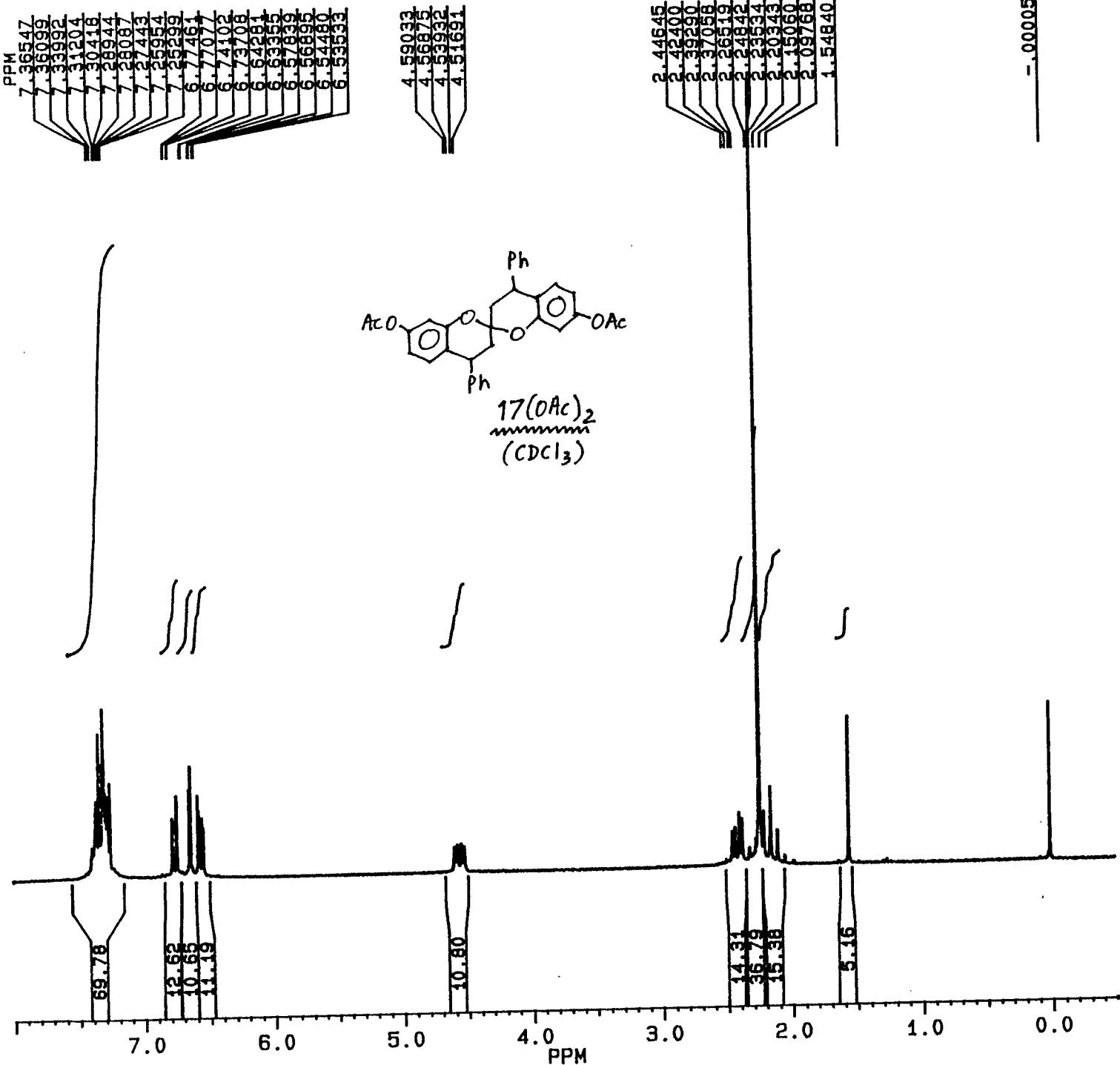
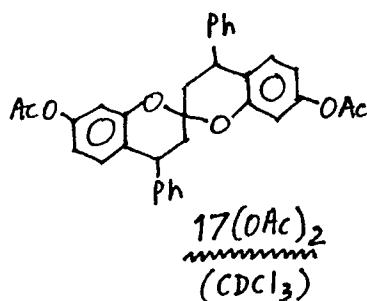
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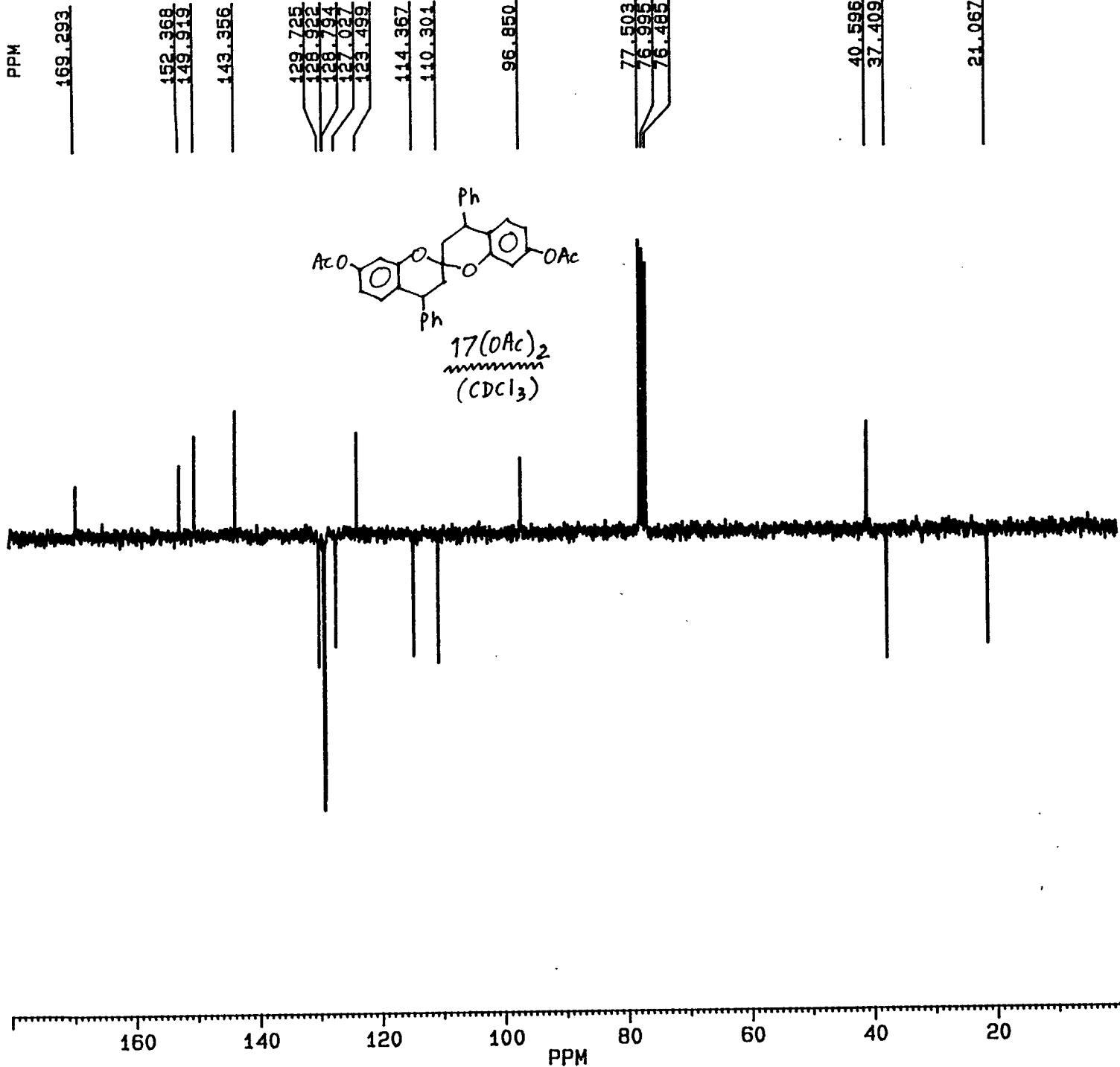
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WZXUAU.009
DATE 10-6-97

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DATE 10-6-97

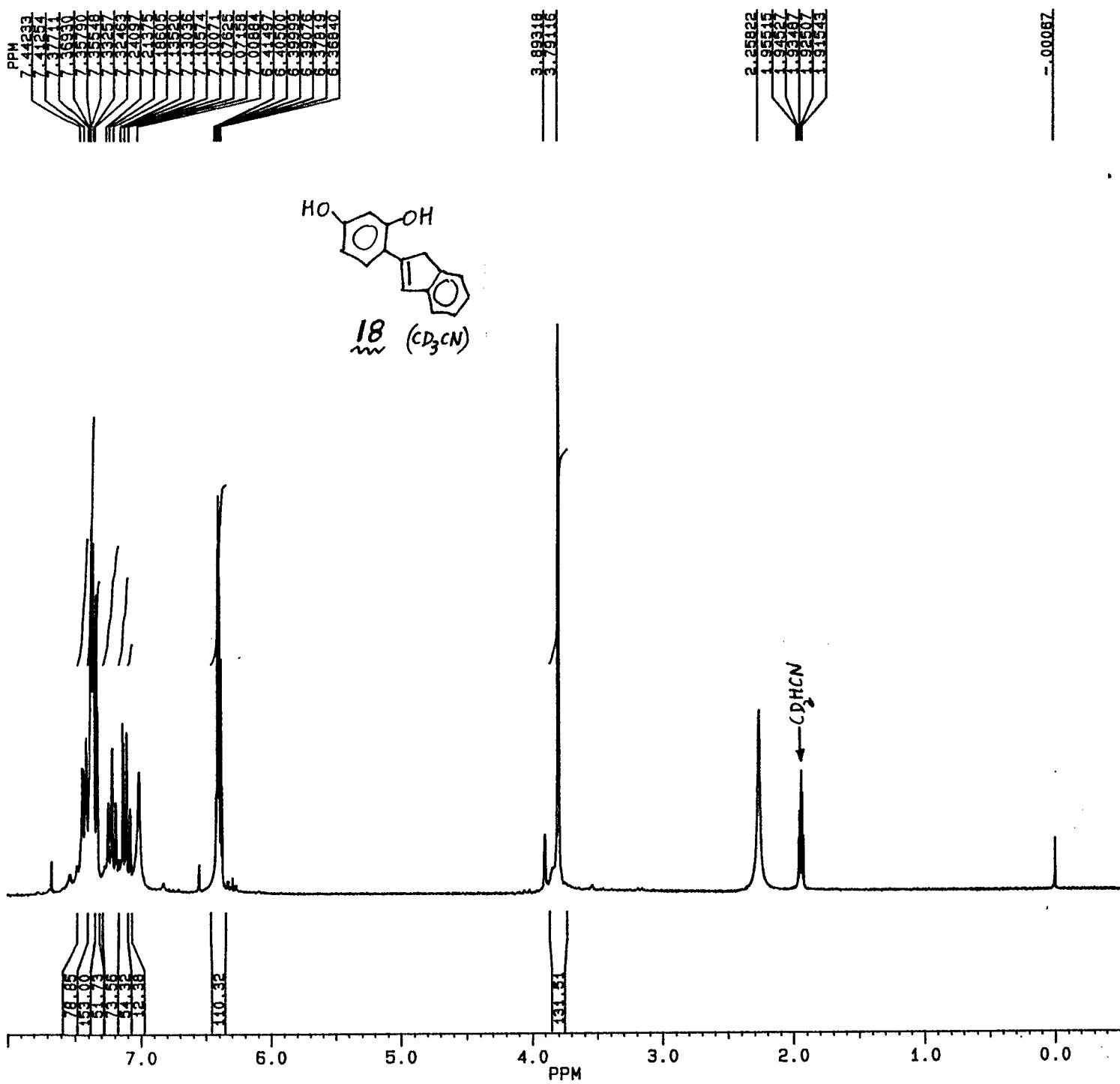
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WZXUN3.107 CD₃CN 2ND RECRY BY CHCl₃

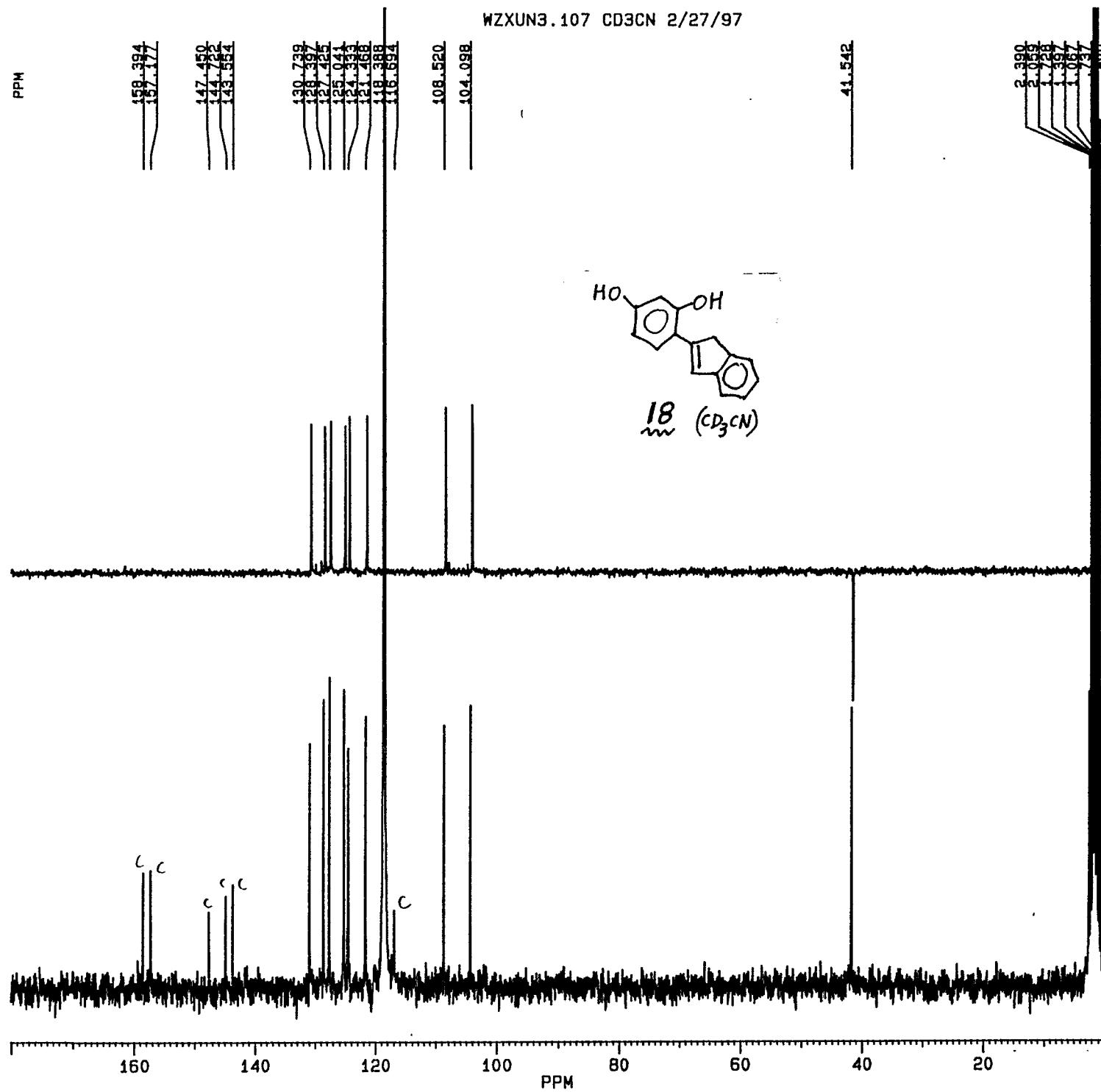


PANHNR02.001
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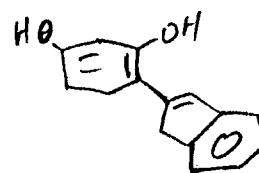


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1 cH

WZXUN3.122 CDCL₃ 3/6/97



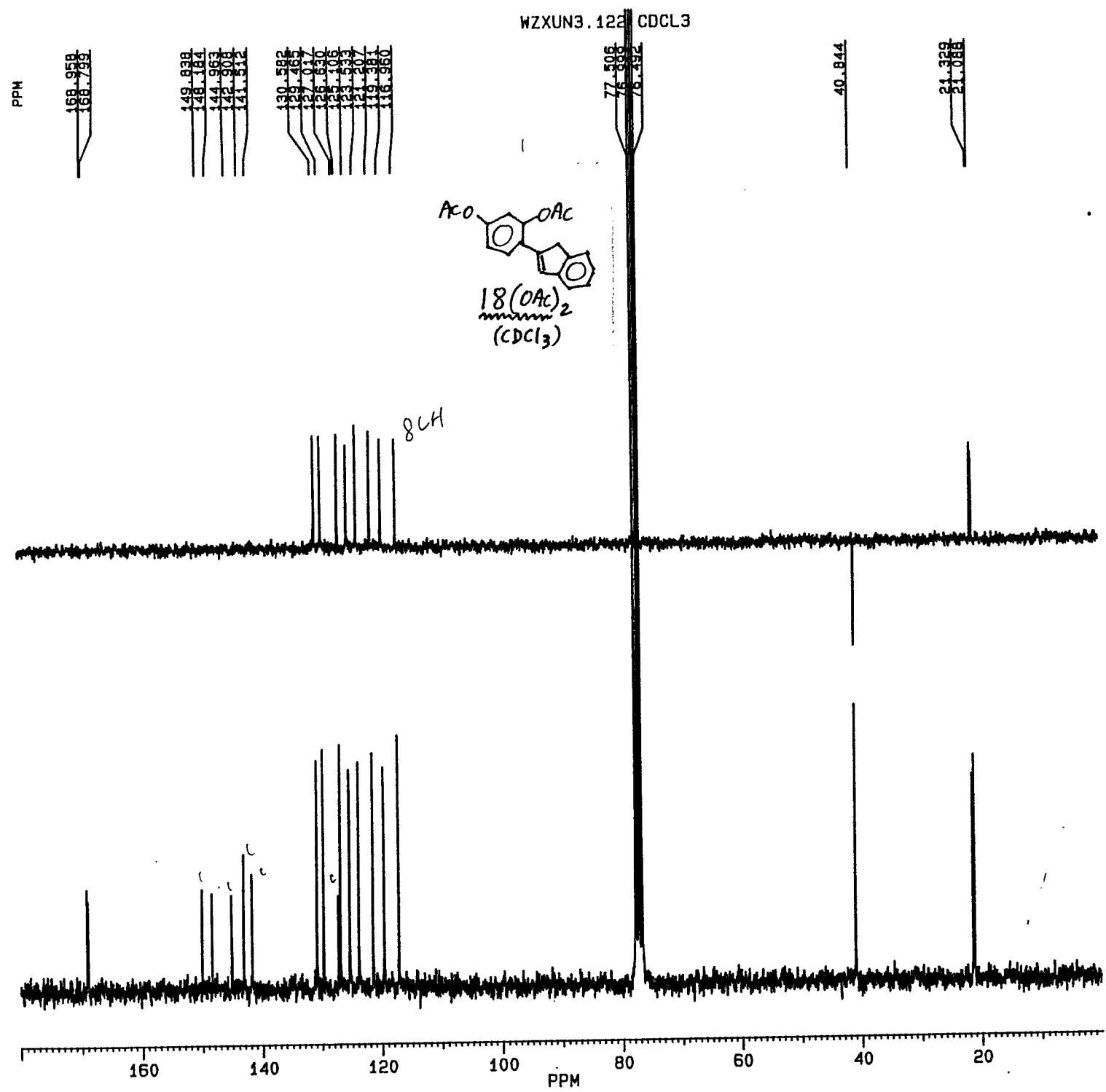
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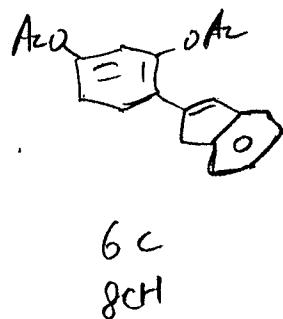
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DATE 6-3-97

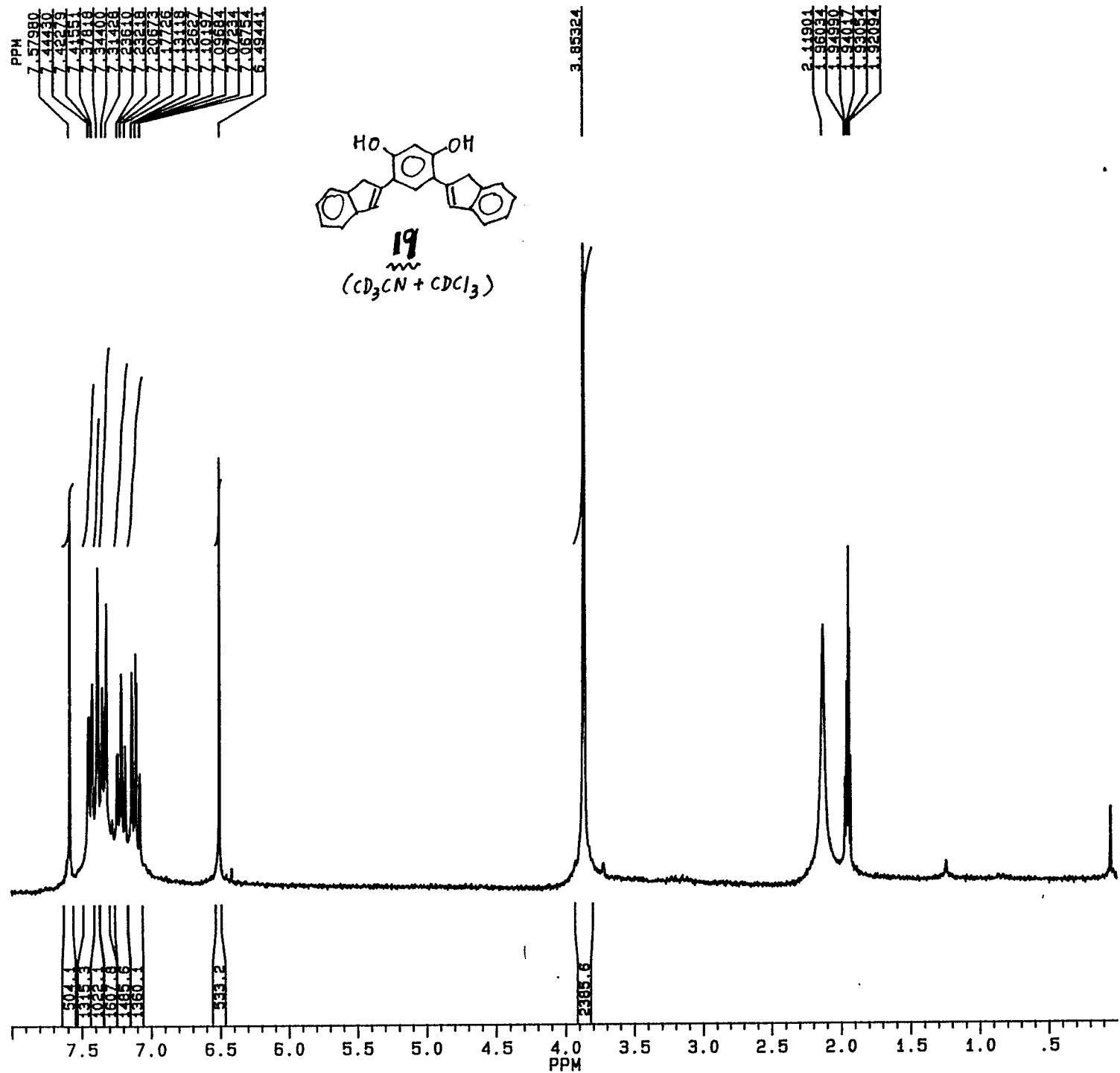
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TE 297

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CY 5.00
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WZXUN3.129 CD₃CN+CDCl₃ 4TH SPOT IN P (2:1)



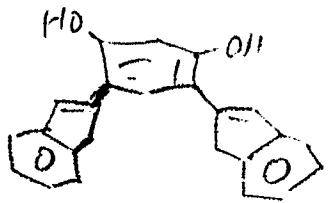
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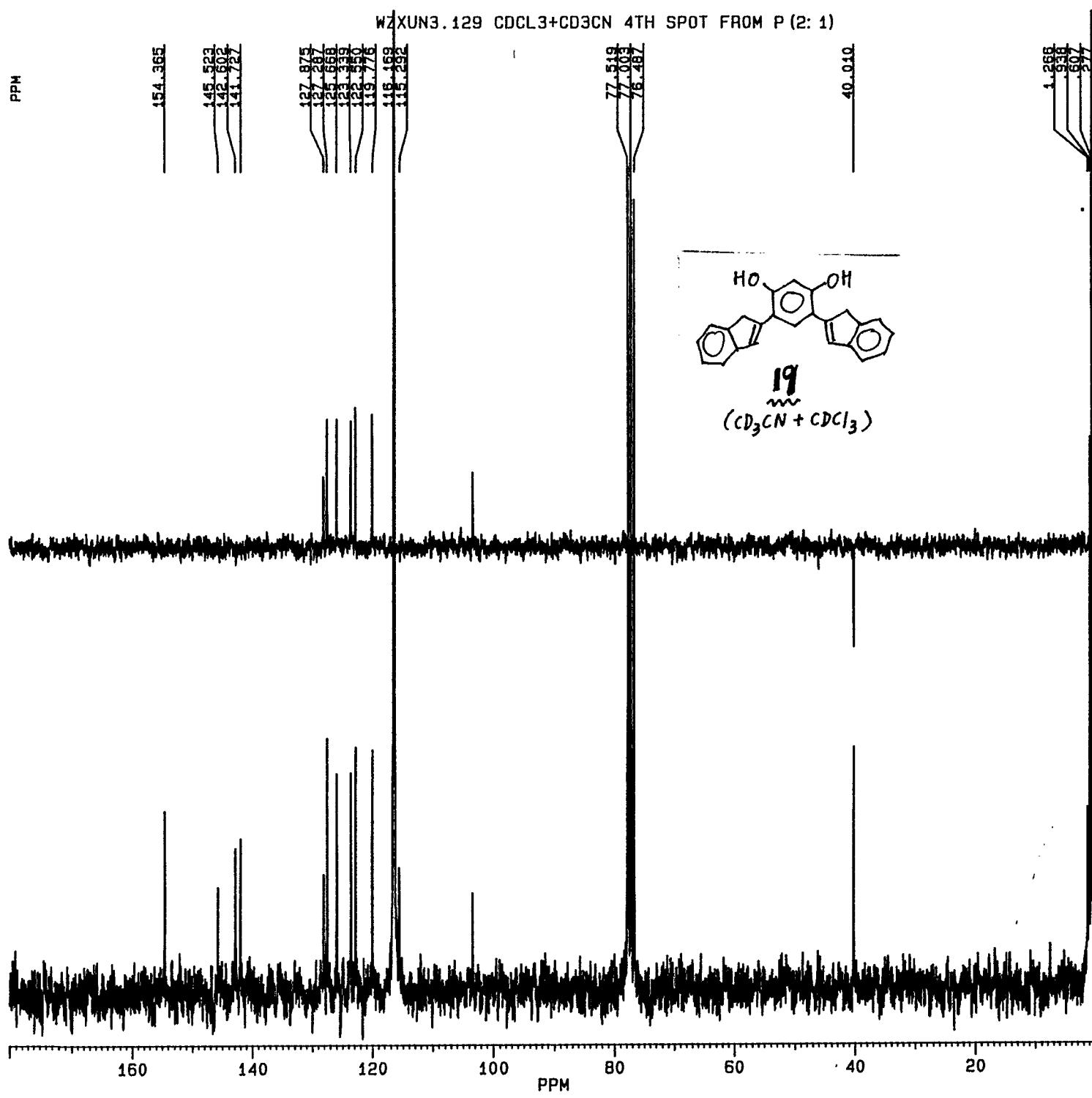
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TE 297

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LB 0.0
GB 0.0
CX 20.00
CY 11.00
F1 8.000P
F2 .001P
HZ/CM 100.050
PPM/CM .400
SR 4183.68





~~BRUKER~~

WZXUN3.129
DATE 8-3-97

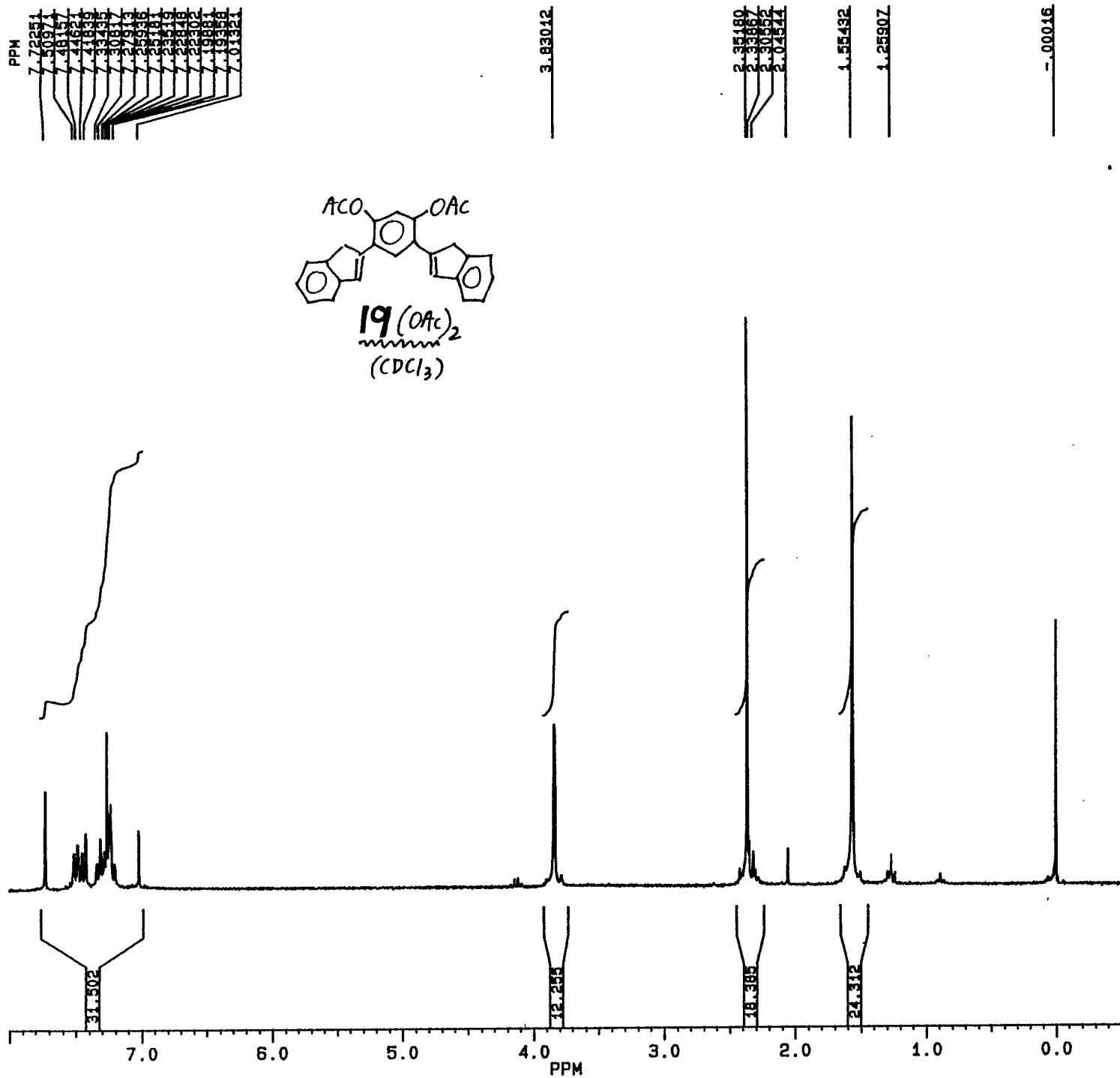
SF 62.896
SY 62.0
O1 1000.000
SI 32768
TD 32768
SW 20000.000
HZ/PT 1.221

PW 2.5
RD 2.880
AQ .819
RG 400
NS 515
TE 297

FW 25000
O2 4100.000
DP 16H, CPD

LB 2.000
GB .200
CX 20.00
CY 4.50
F1 179.999P
F2 .008P
HZ/CM 566.040
PPM/CM 9.000
SR -3664.80

WZXUN3.70 CDCL₃ 1/3/97



BRUKER

WZXUN3H.070
DATE 3-1-97

SF 250.133
SY 250.0
O1 4300.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 8.0
RD 0.0
AQ 2.048
RG 100
NS 48
TE 297

FW 5000
O2 0.0
DP 63L.P0

LB 0.0
GB 0.0
CX 20.00
CY 10.00
F1 8.000P
F2 -.500P
HZ/CM 106.299
PPM/CM .425
SR 2854.20

