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SUPPORTING INFORMATION

Supporting Information for

"Benzyne Adds Across a Closed 5-6 Ring Fusion in C_{70}: Evidence for Bond Delocalization in Fullerenes"

Mark S. Meier,* Guan-Wu Wang, Robert C. Haddon, Carolyn Pratt Brock, Michael L. Lloyd and John P. Selegue.

^{1}H and ^{13}C NMR spectra of **1a - 1d**

Tables and Figures for the Structure Determination of [1,2]benzeno[70]fullerene (**1a**)

Table 1. Crystallographic Data and Experimental Details

Table 2. Positional Parameters and Equivalent U Values for the Refined Atoms

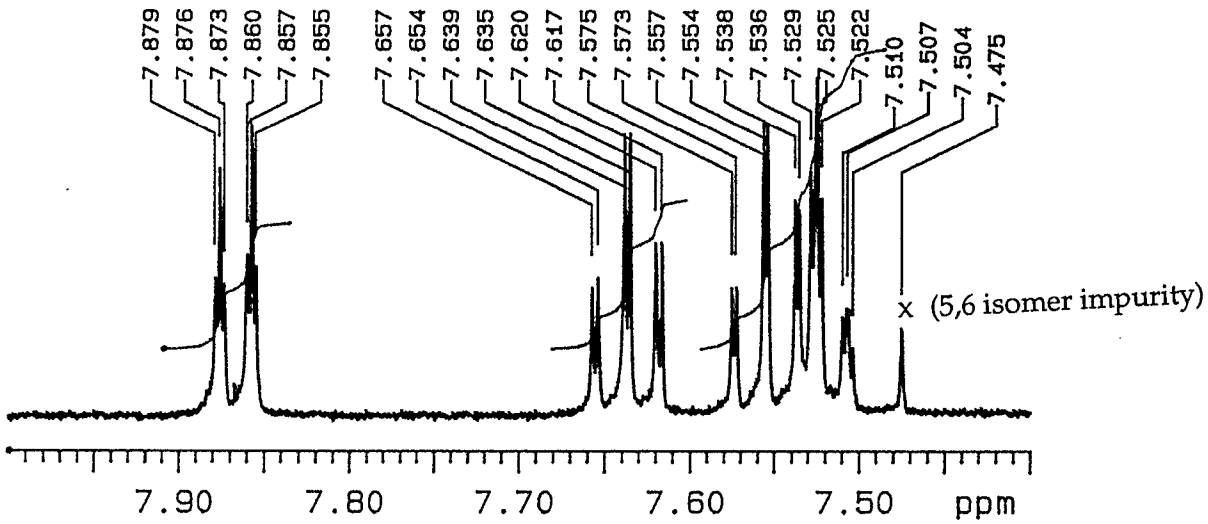
Table 3. Selected Bond Lengths and Angles

Table 4. Positional Parameters and U Values for the Hydrogen Atoms

Figure 1. Line drawing showing the atom-numbering scheme

Figure 3. Stereoscopic view of the unit cell

Figure 4. Perspective drawing of the pseudo-symmetric molecules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7.557
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7.475
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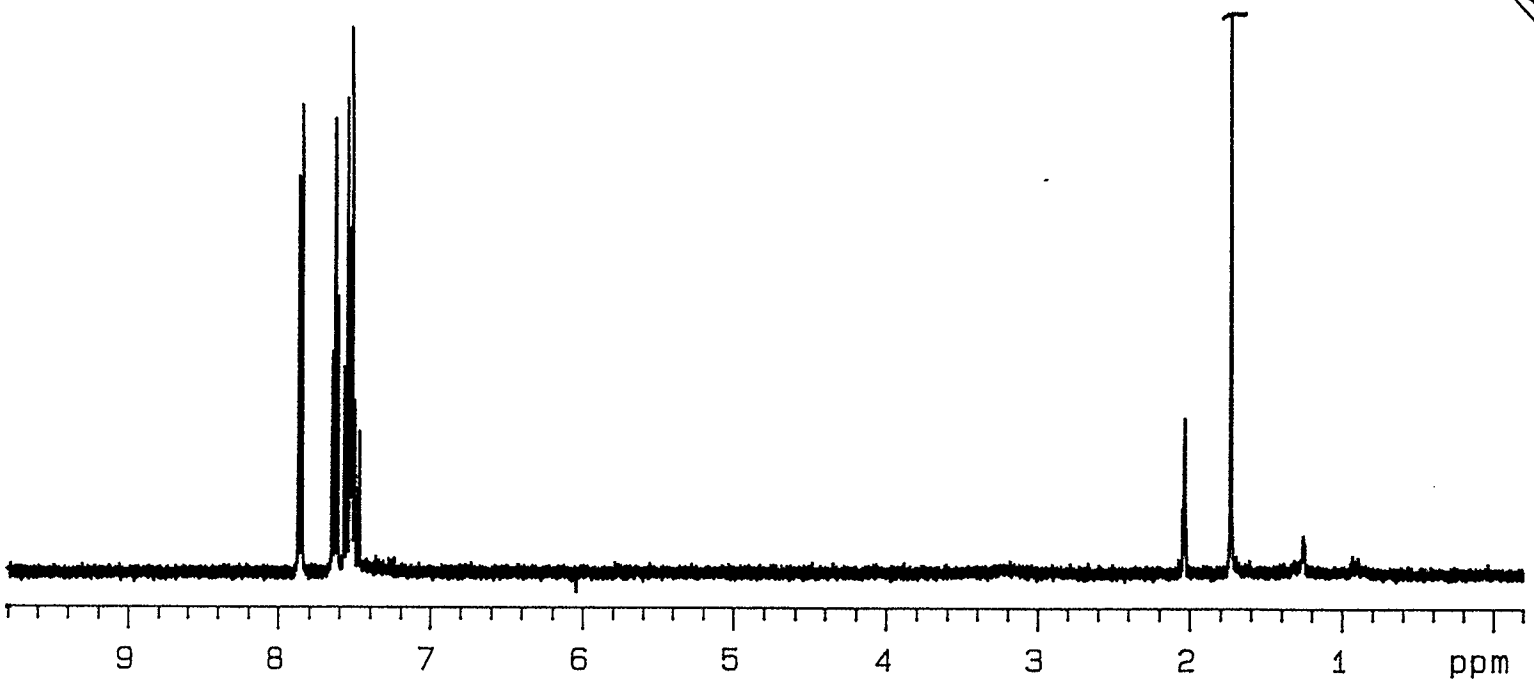
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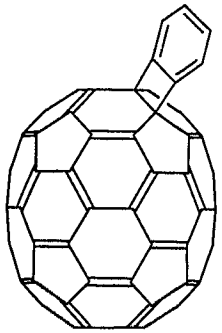

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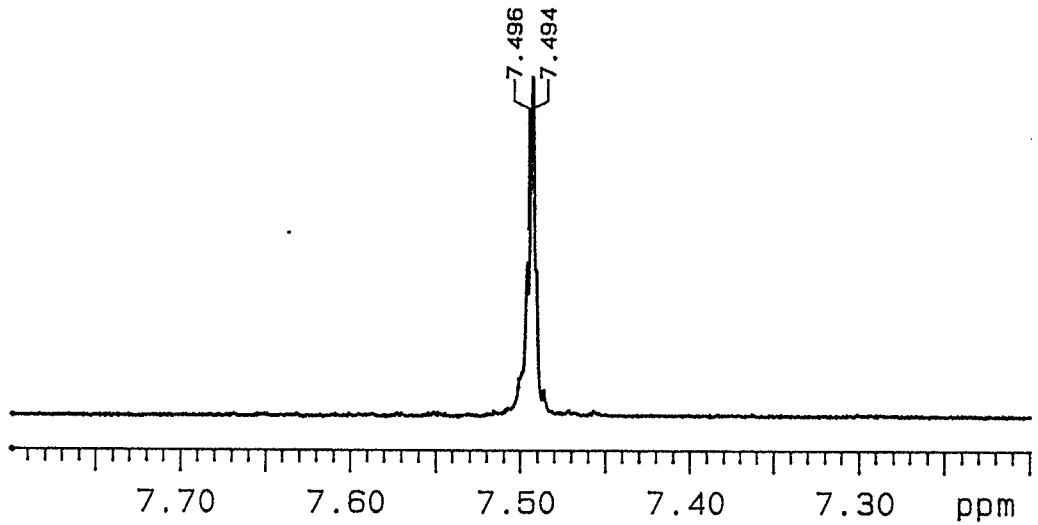

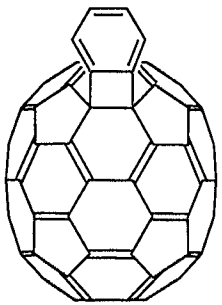

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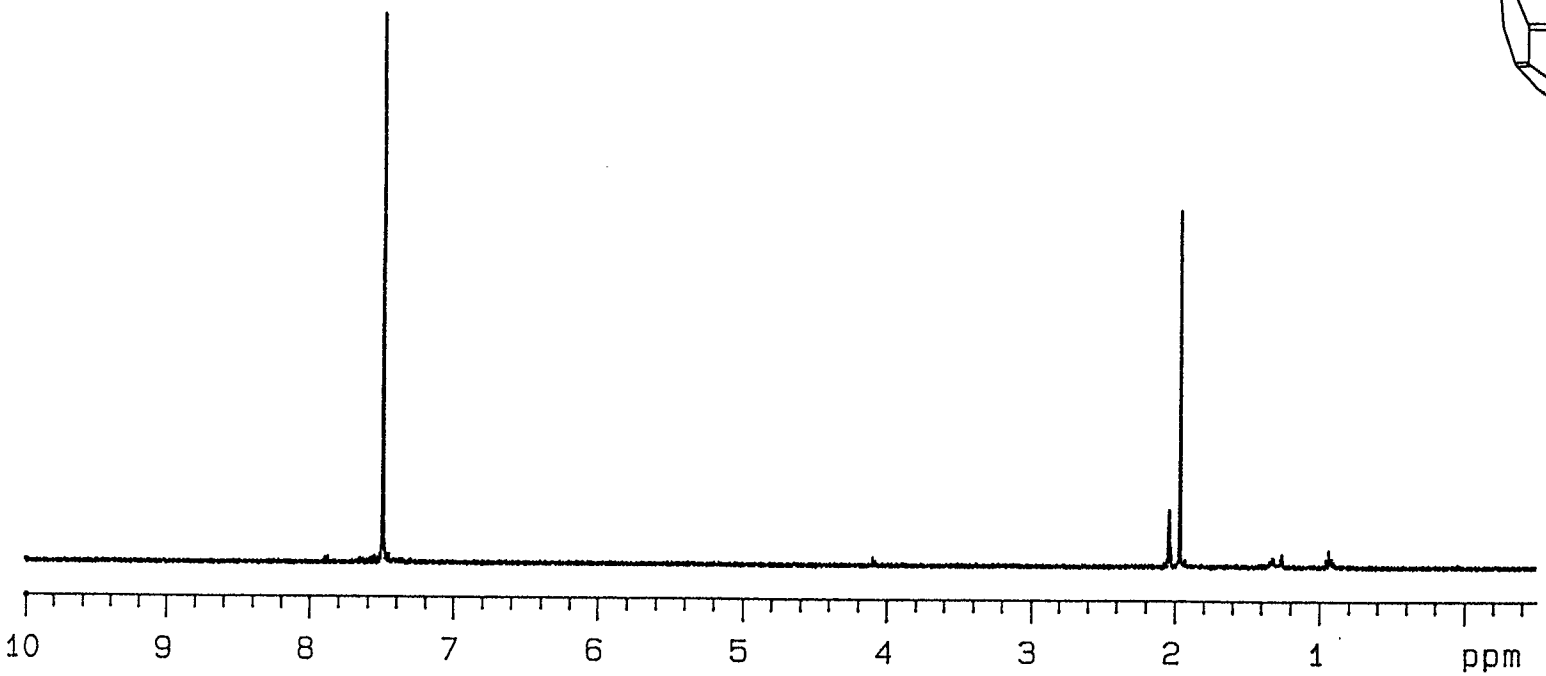

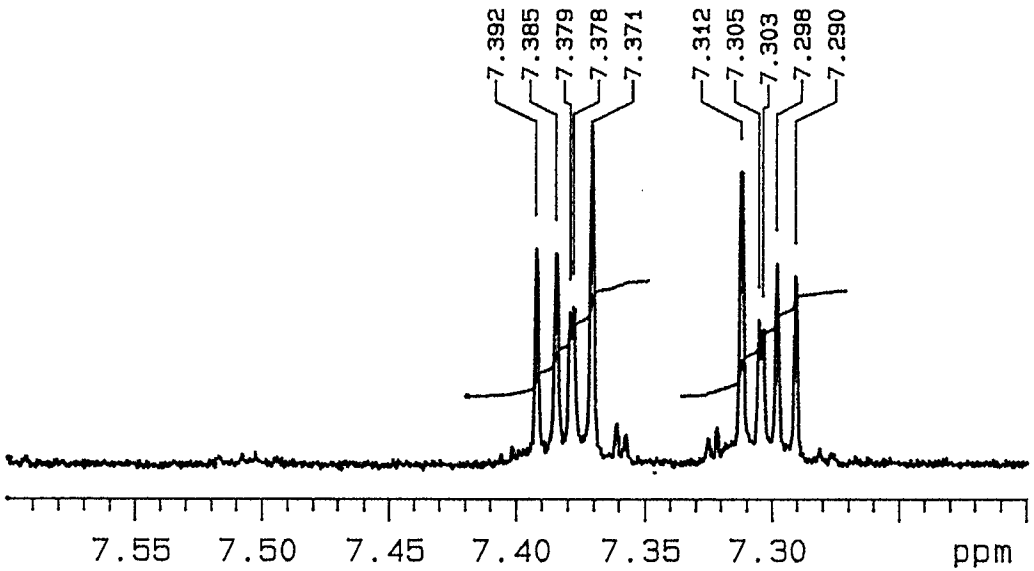
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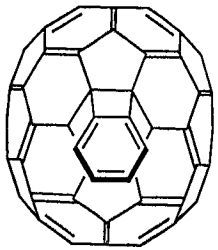

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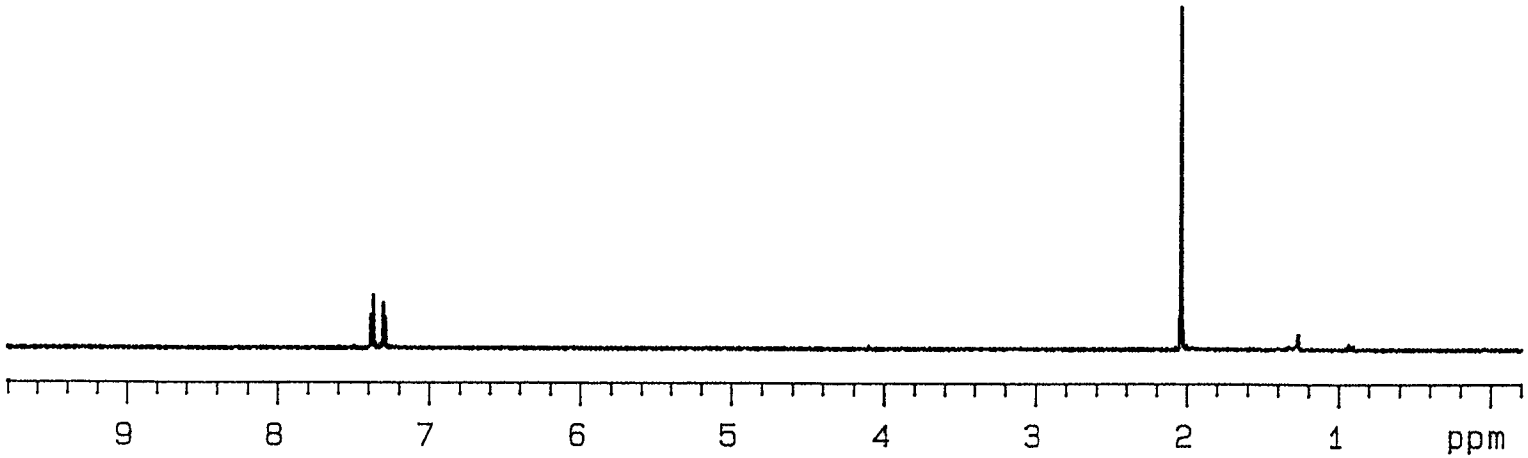
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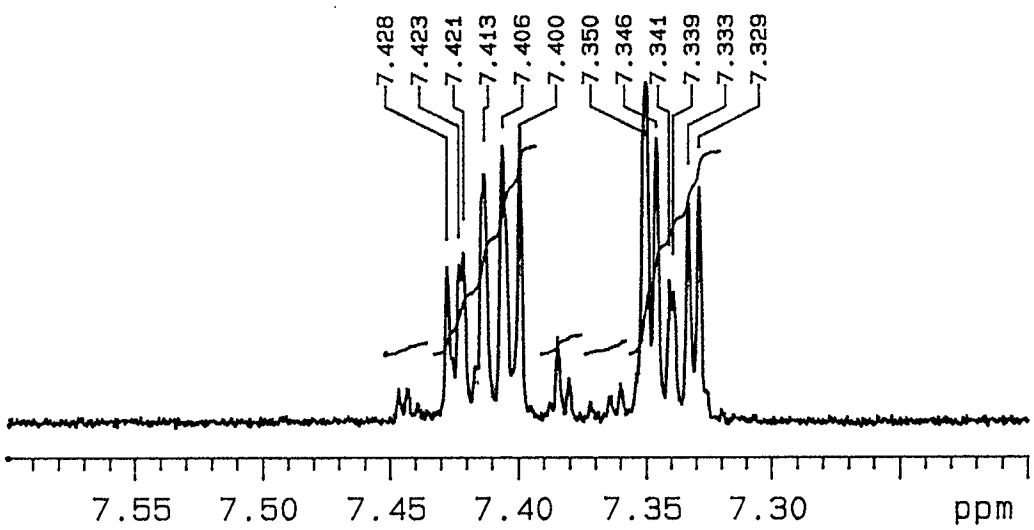
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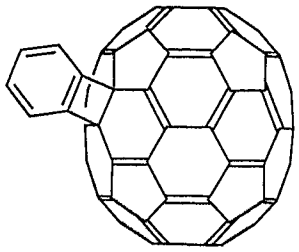

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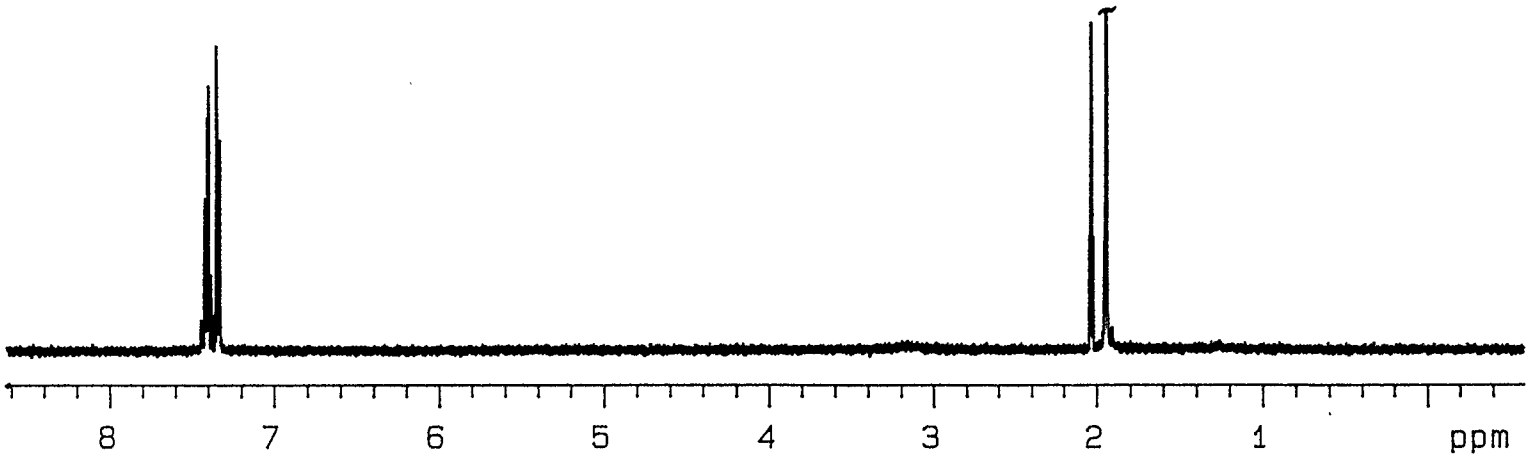
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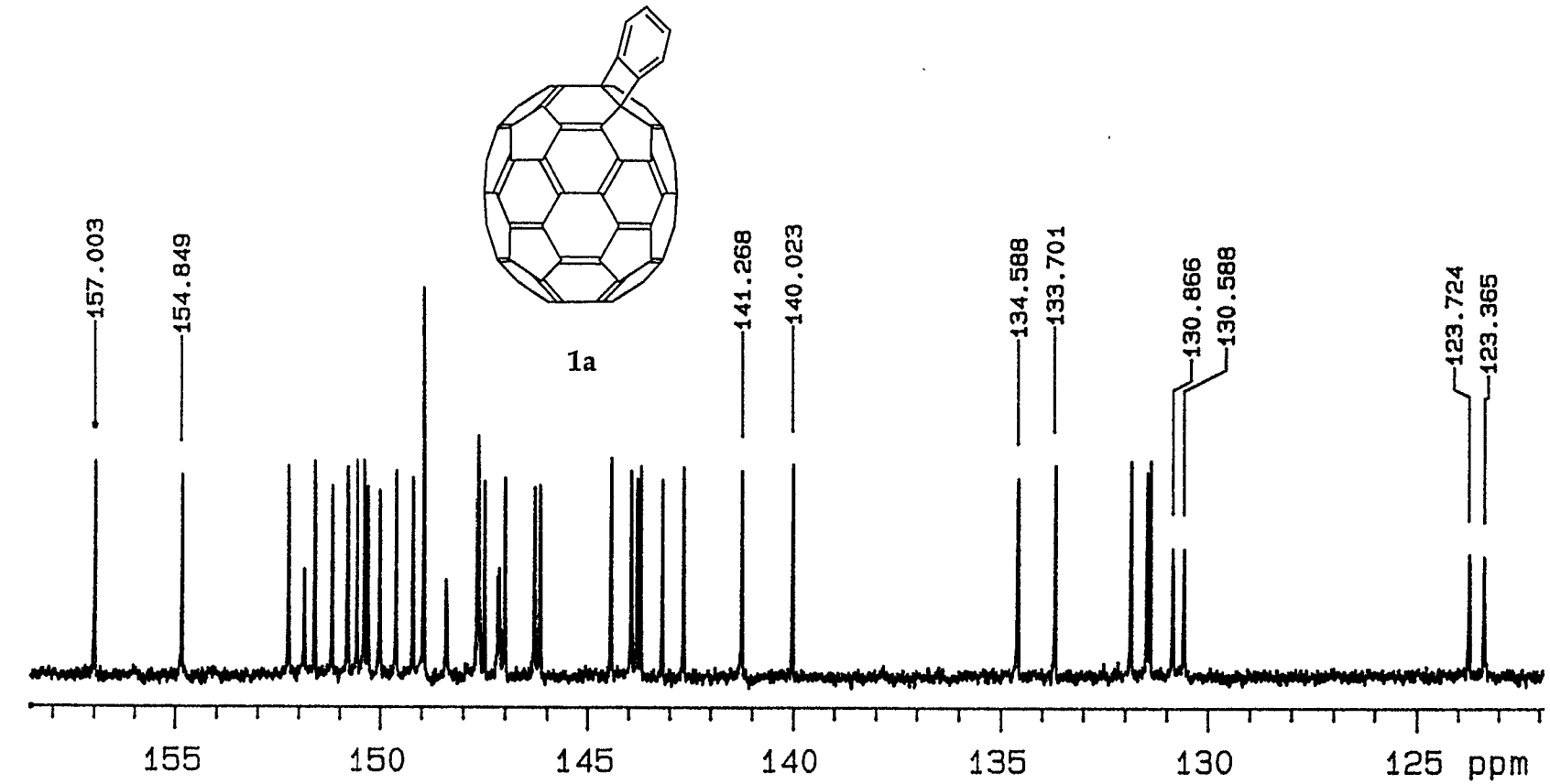
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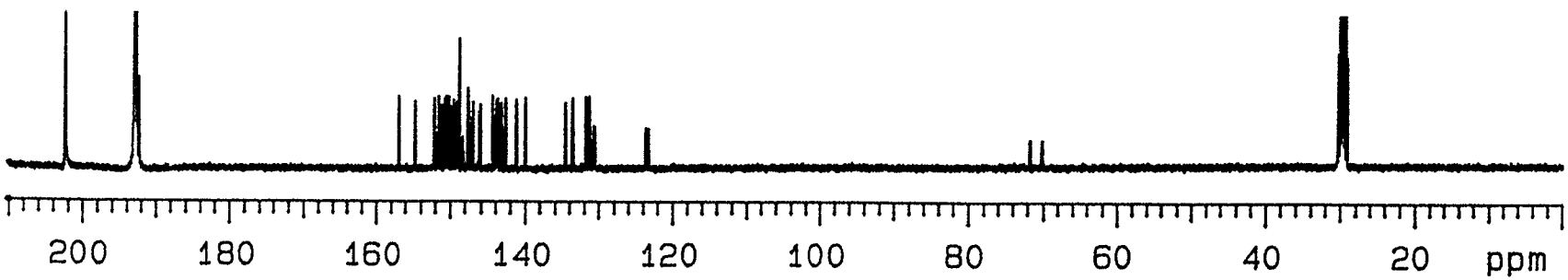
1a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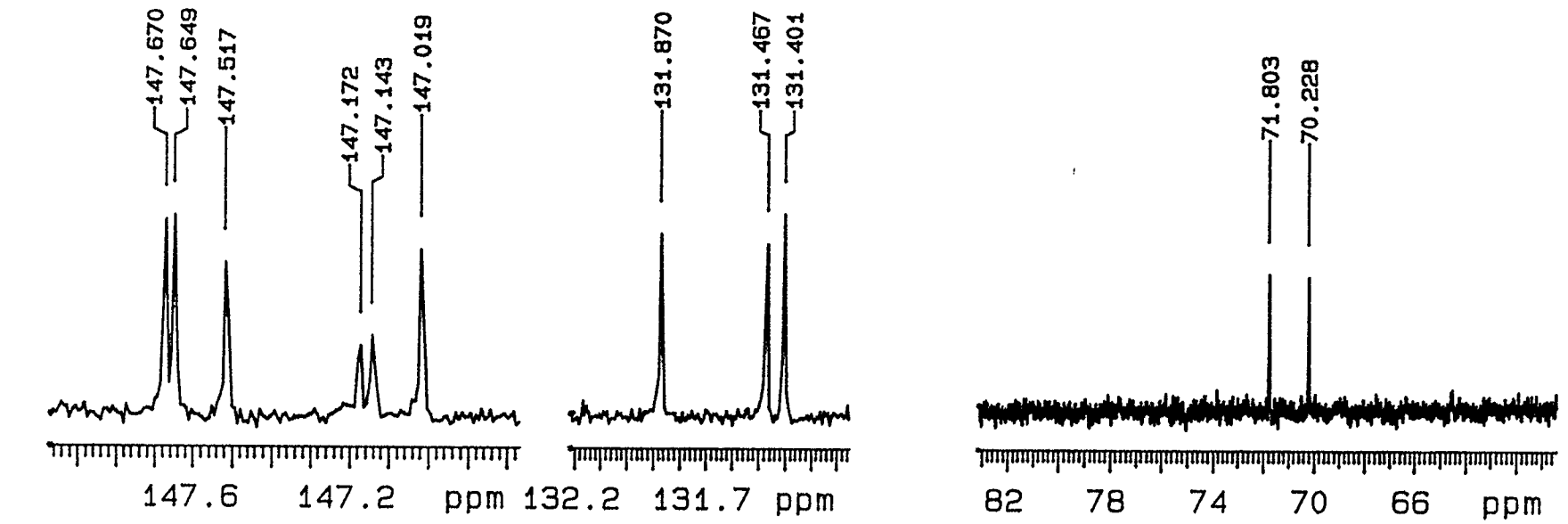
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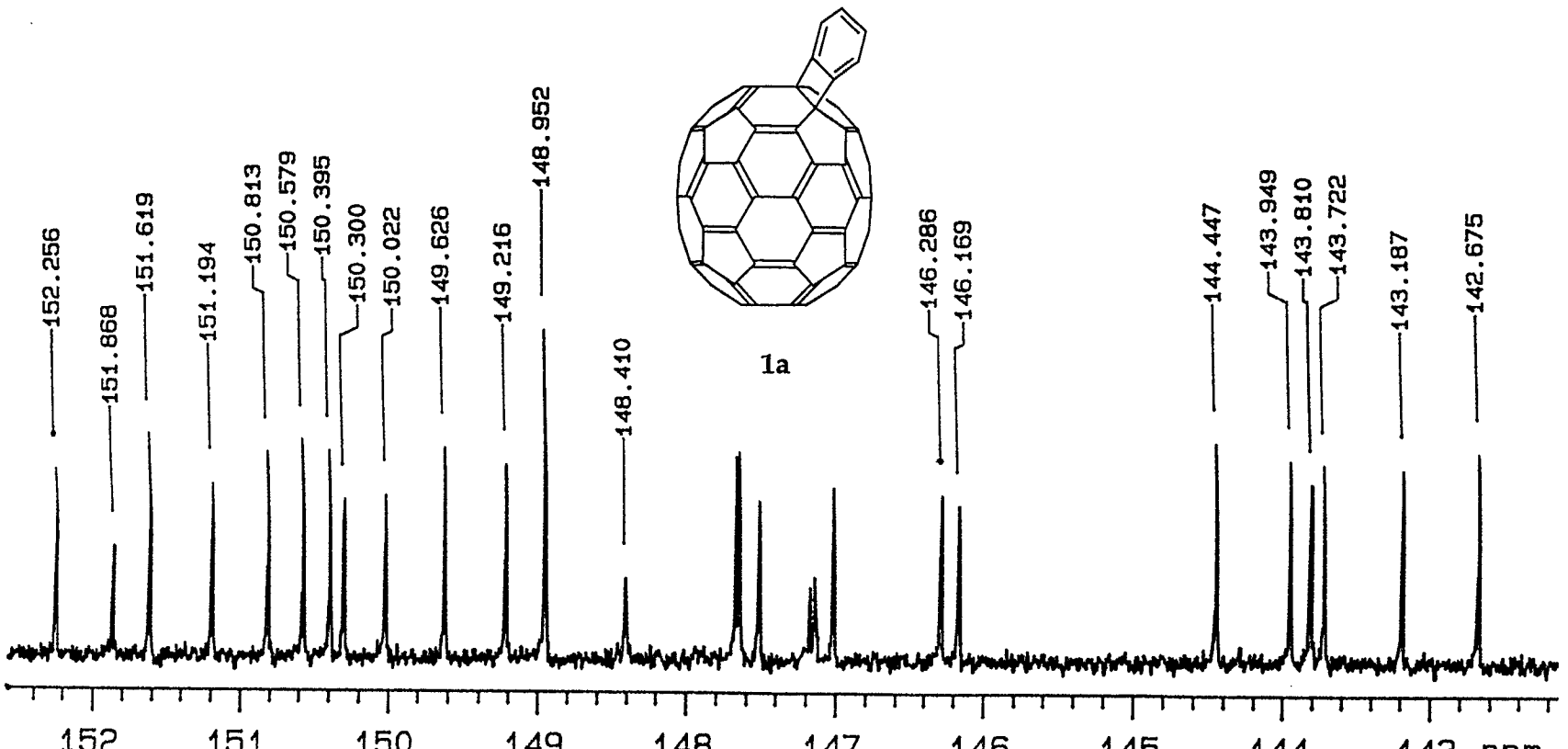
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146.169
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150
149
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144
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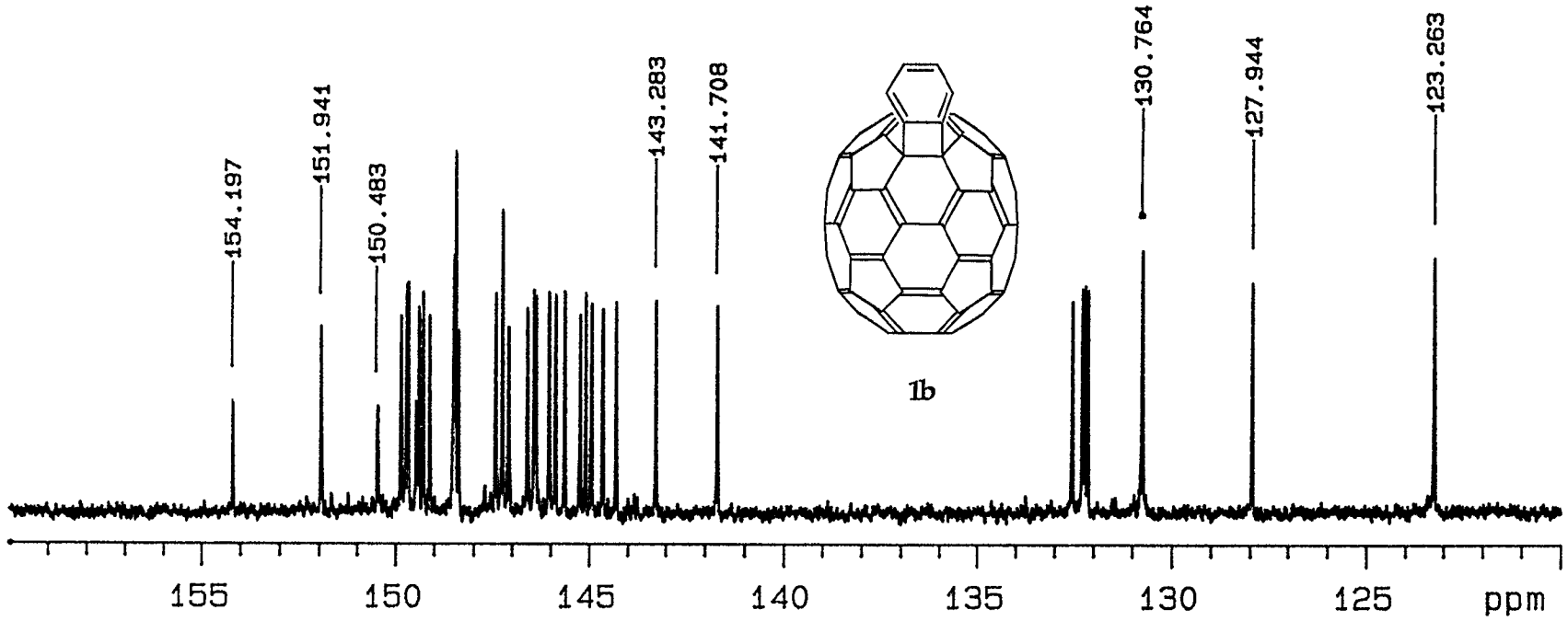
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130
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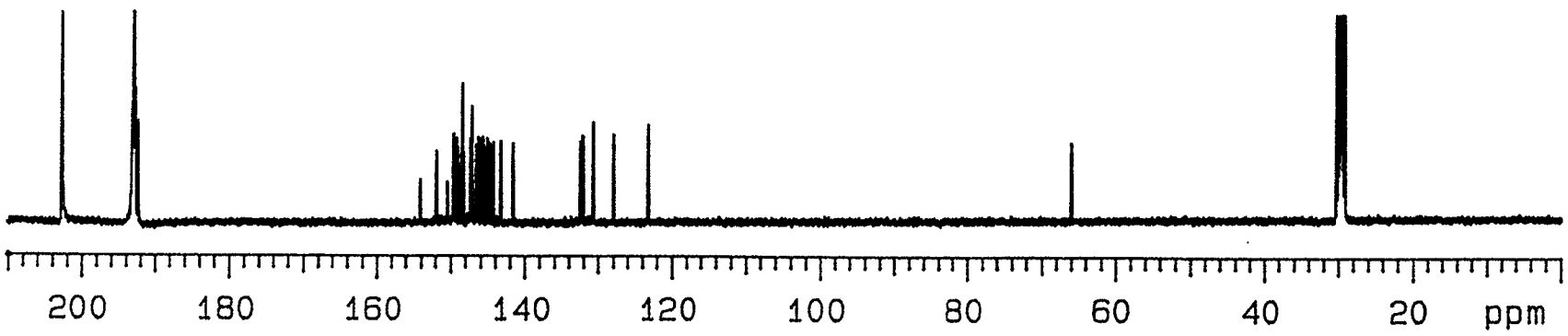
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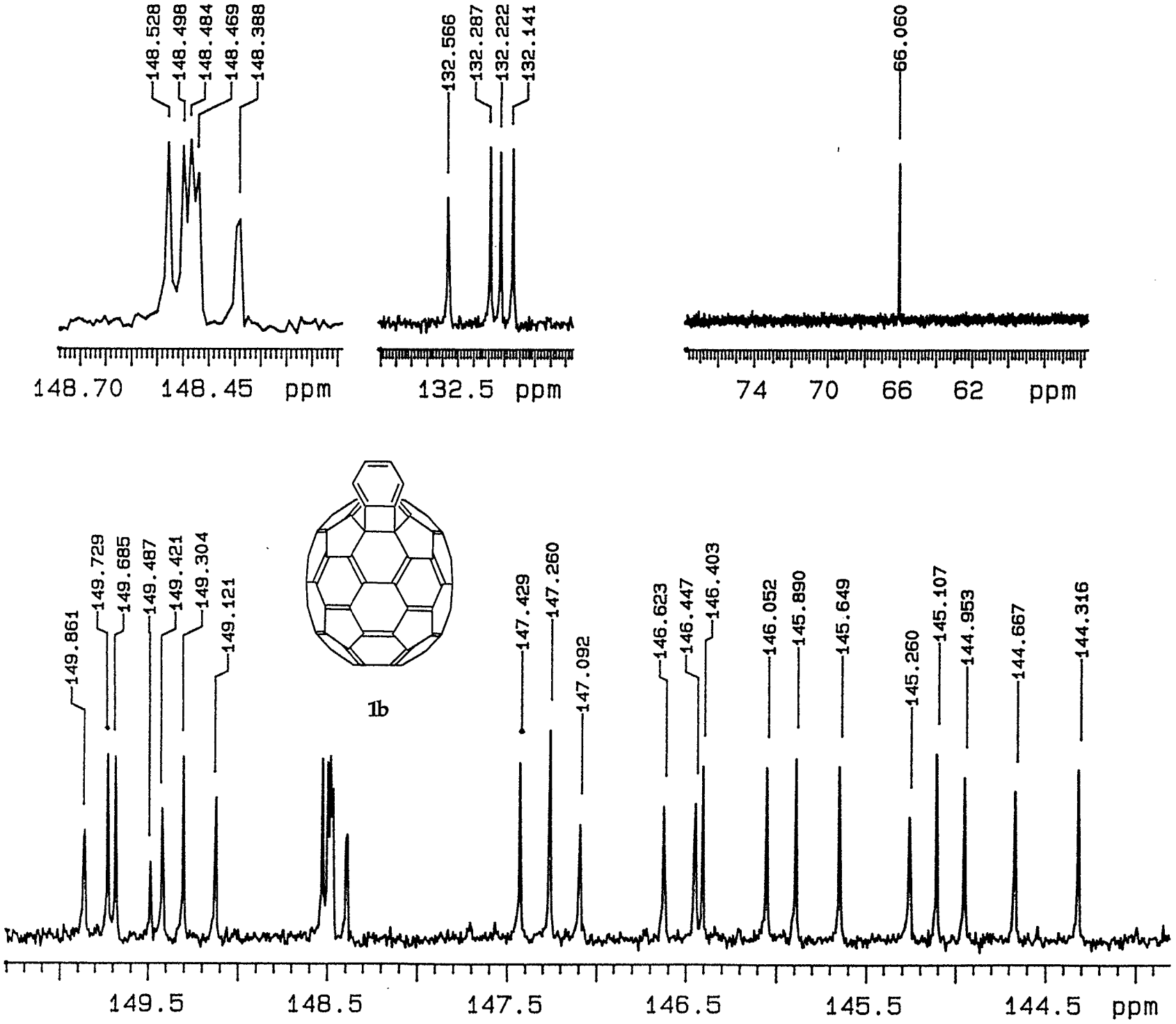
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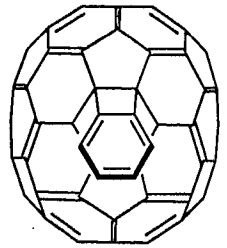

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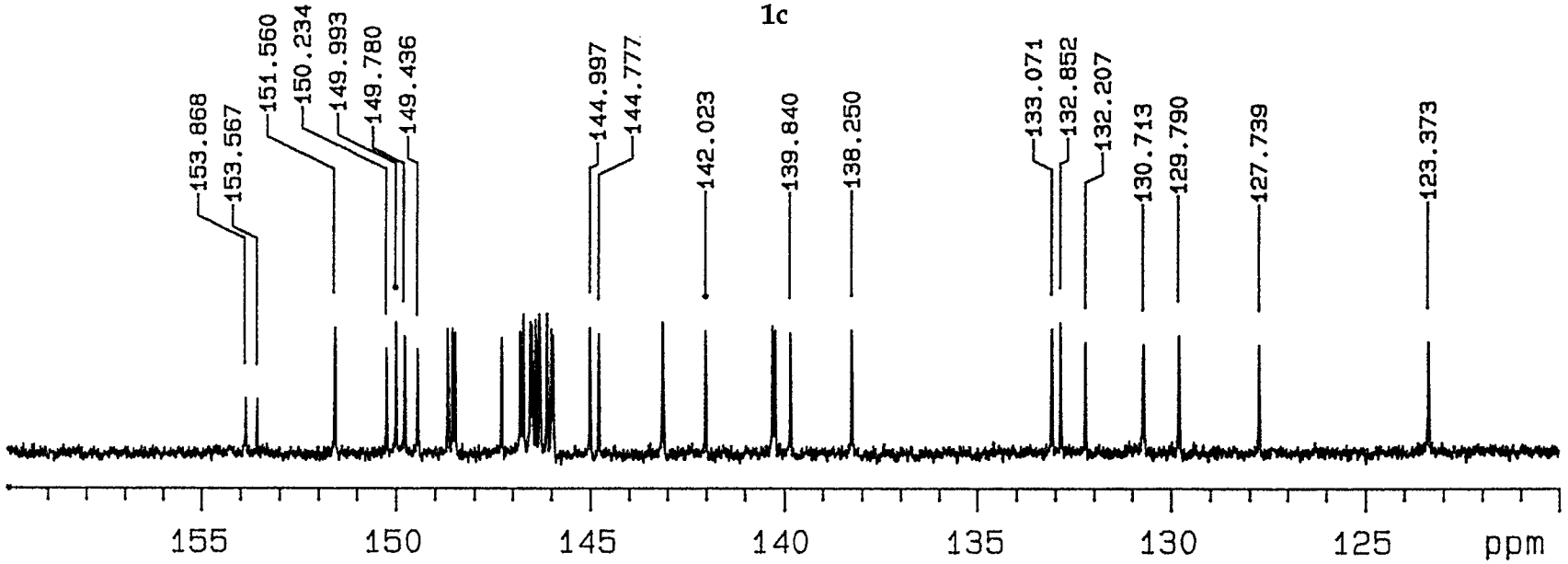
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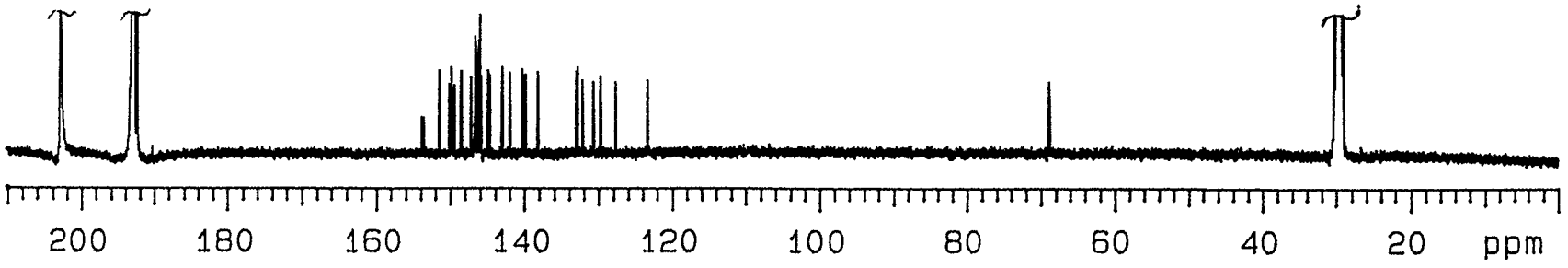
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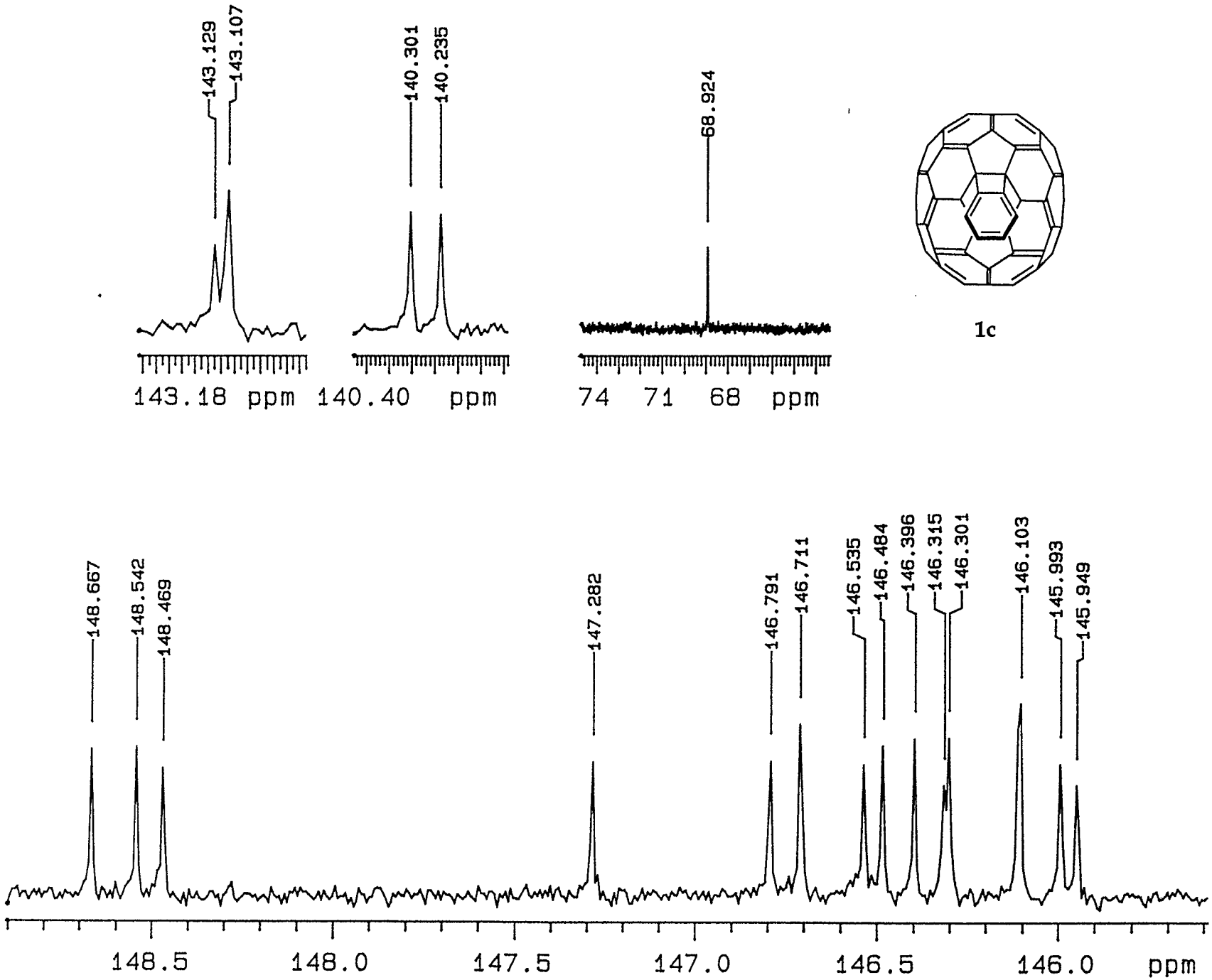
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74 71 68 ppm
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146.484
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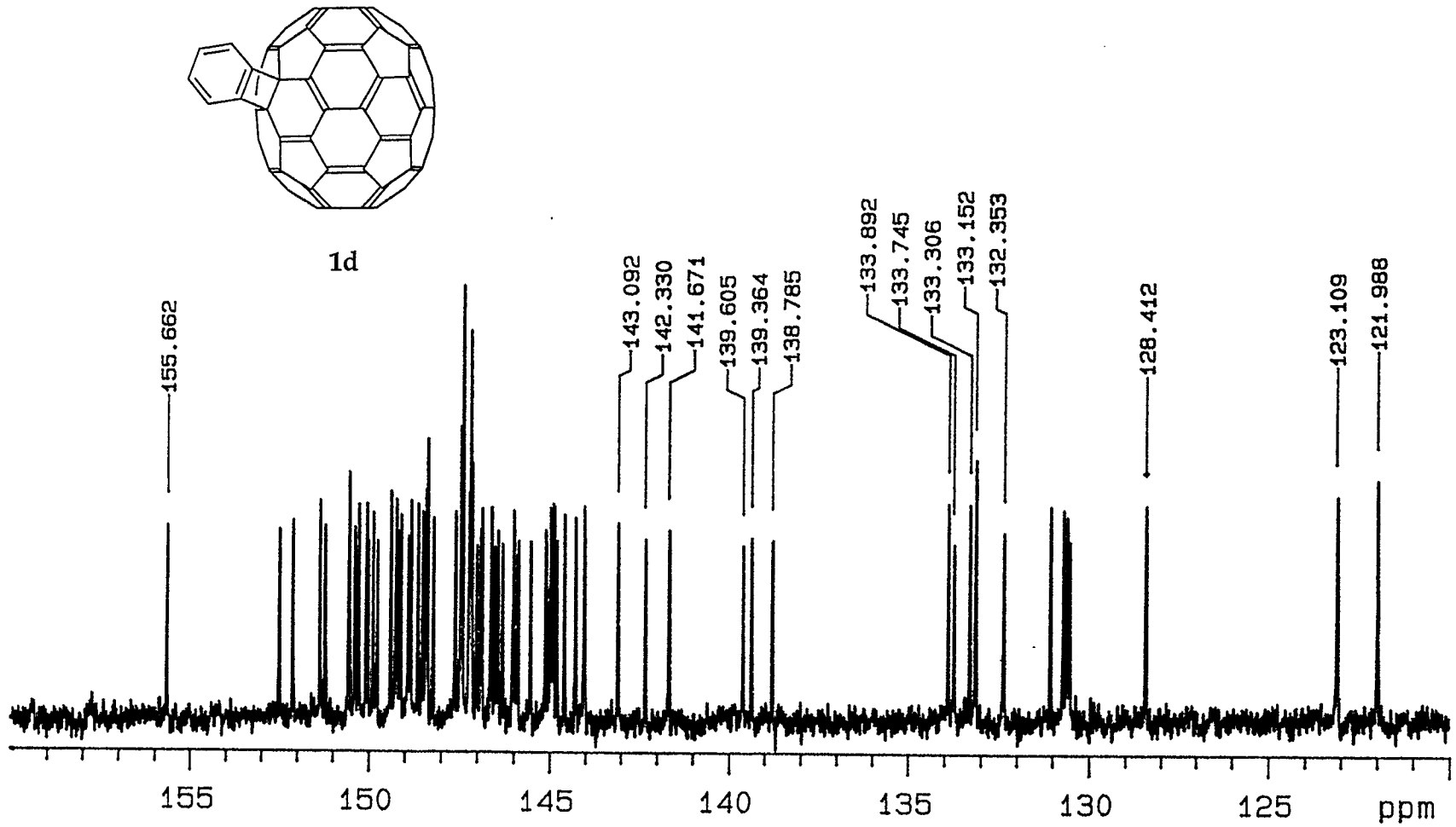
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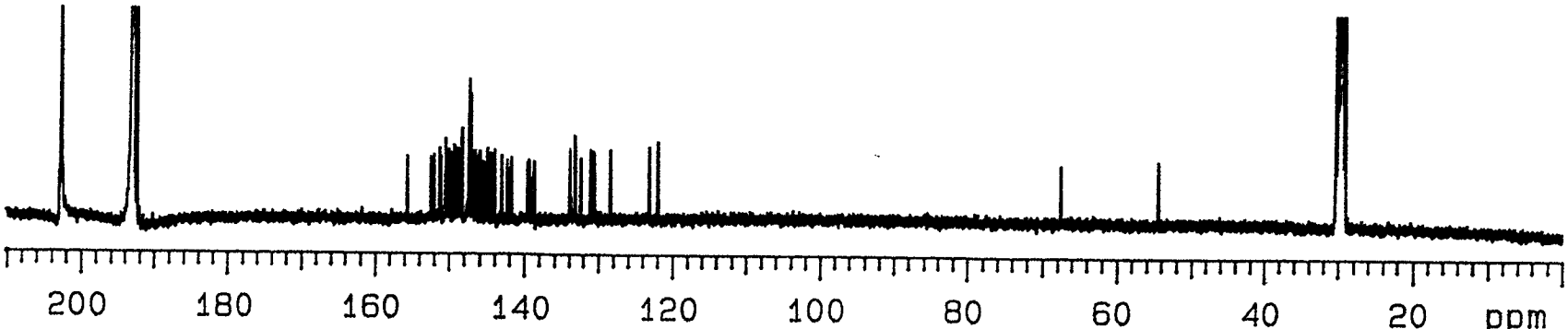
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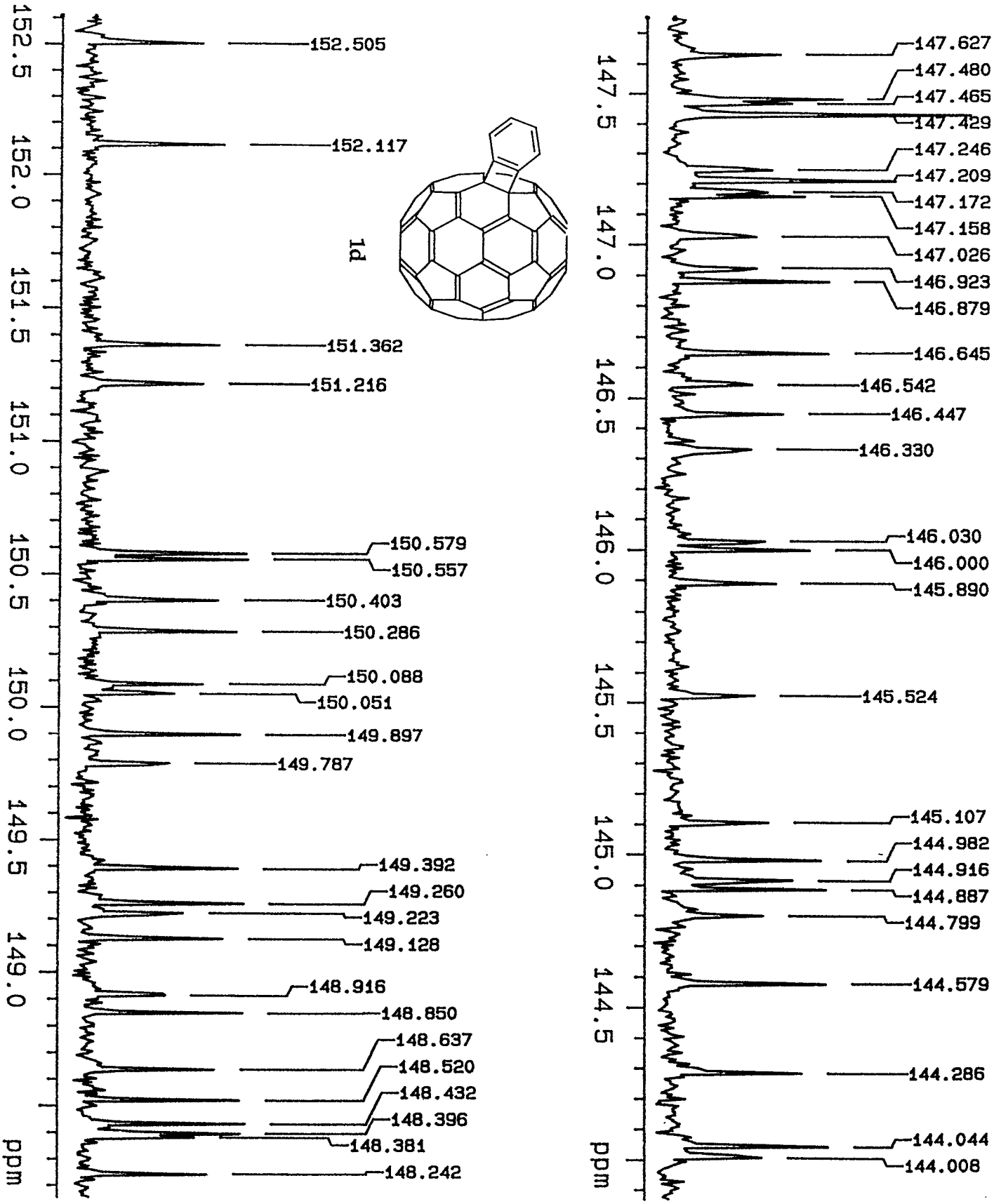
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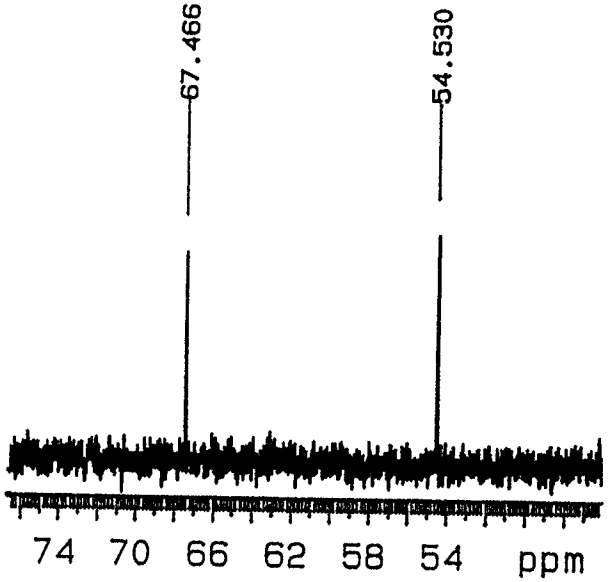
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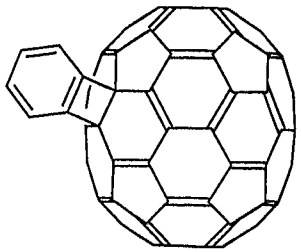

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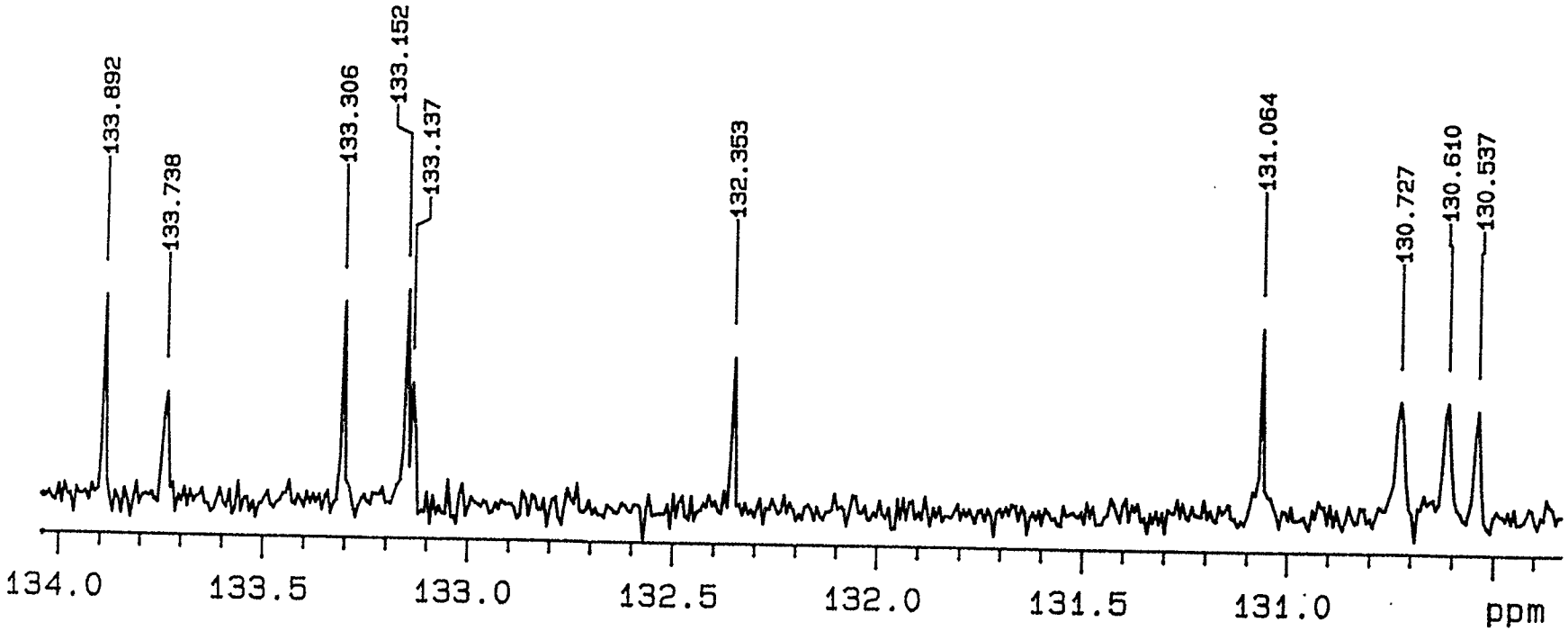
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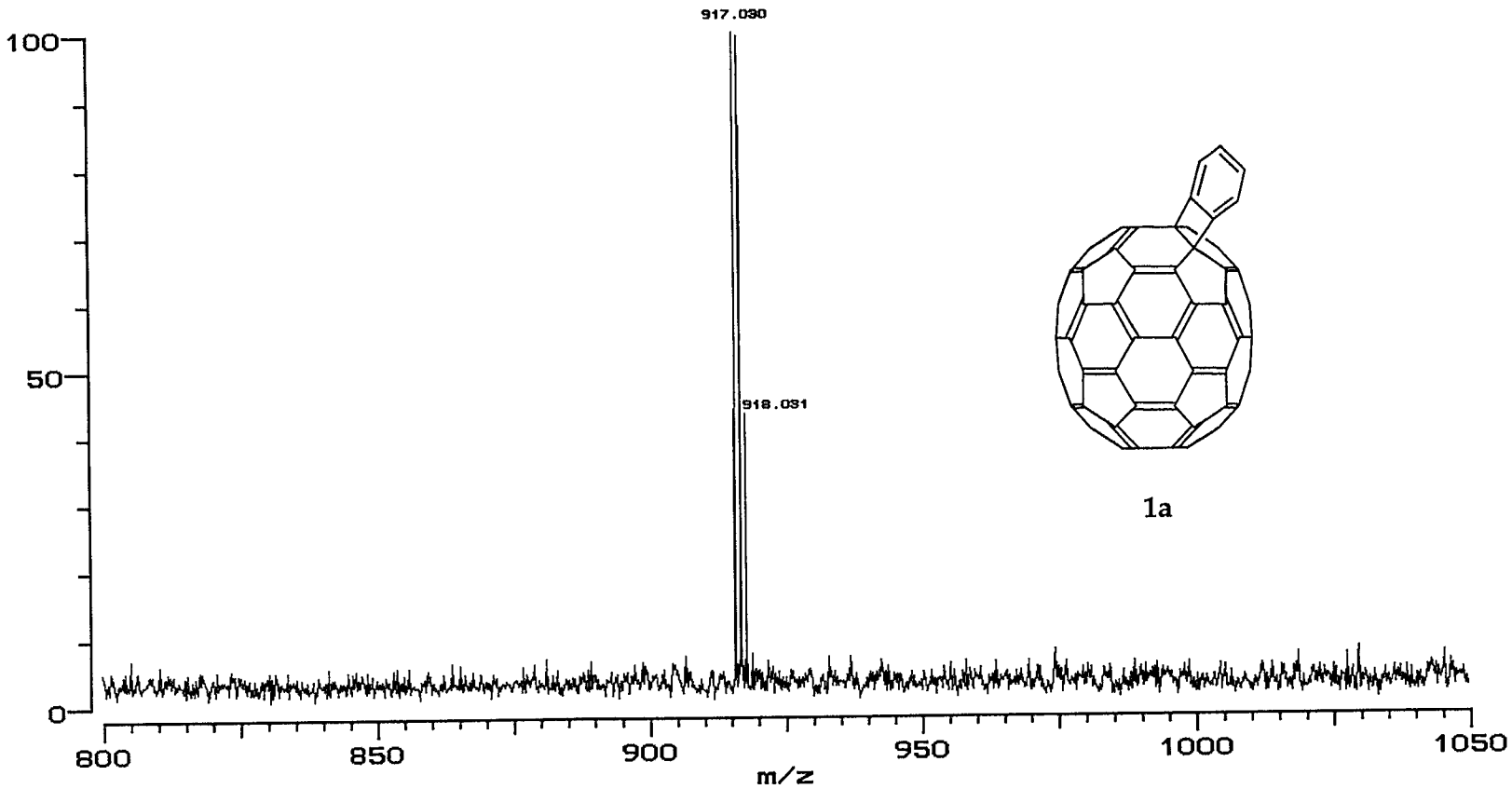
MALDI Mass Spectrum of **1a** (9-nitroanthracene matrix, positive ions)



1a

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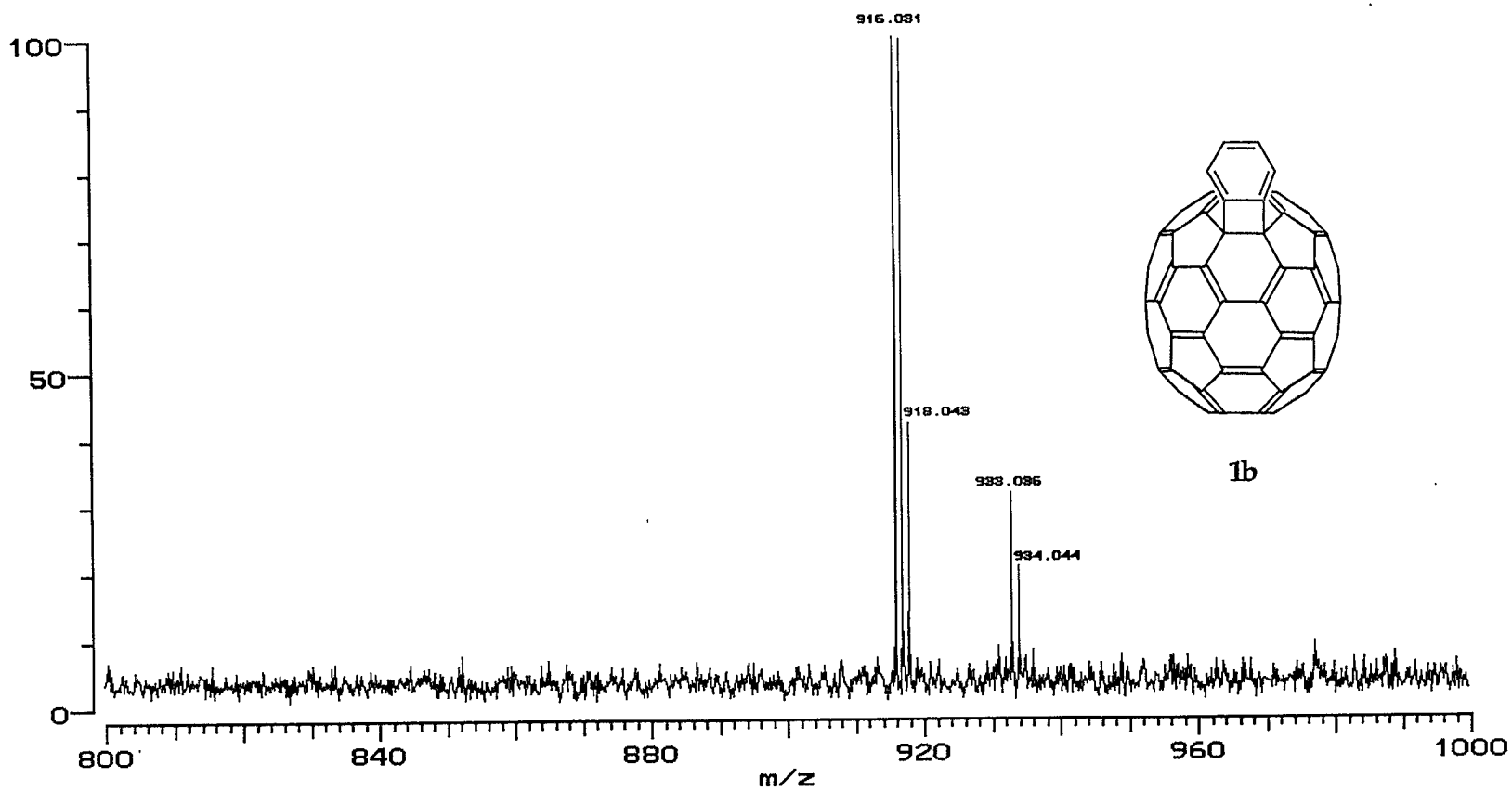

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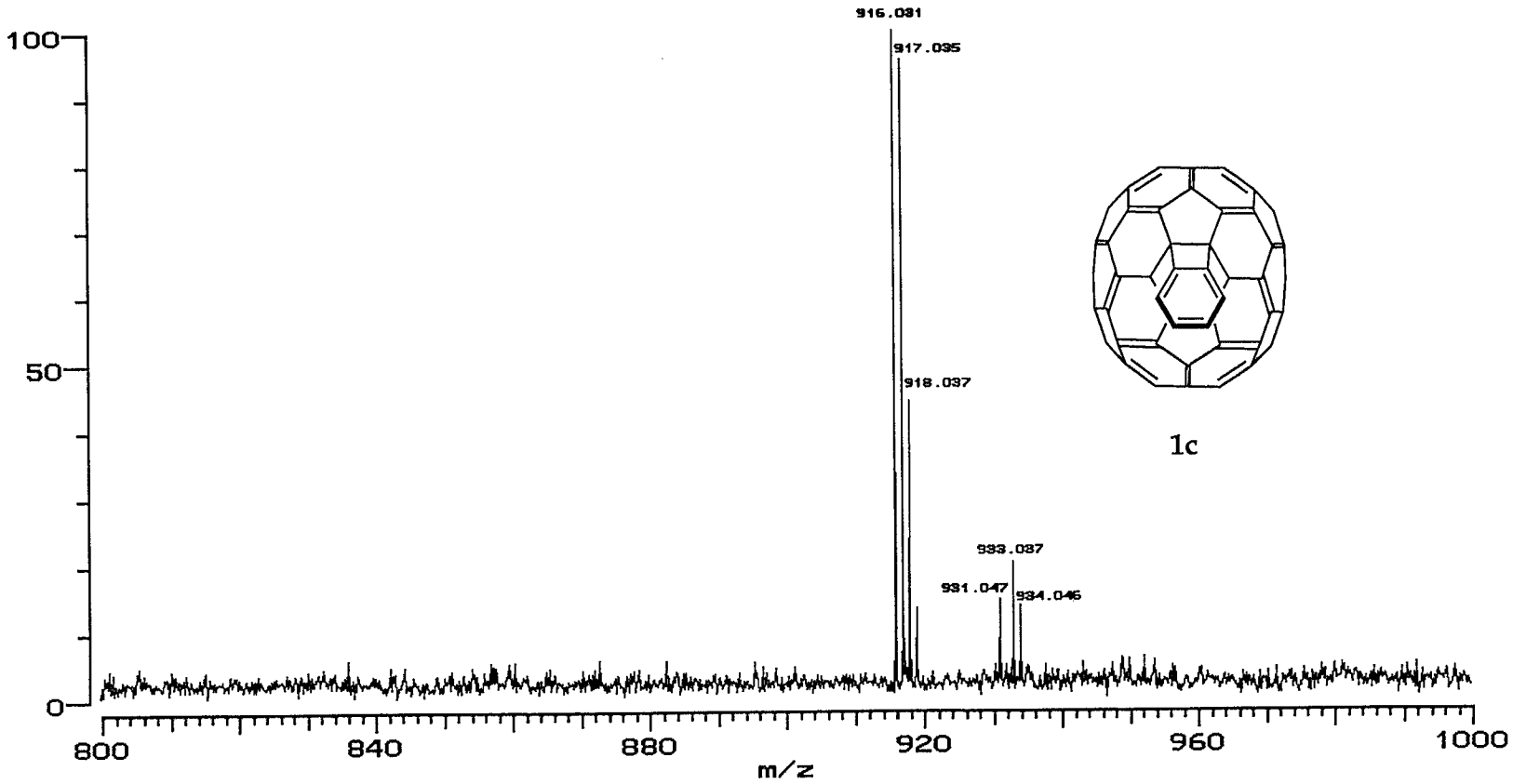

(Masses at M^{+}+16 indicate that small amounts of oxygen were incorporated during MS sample preparation)

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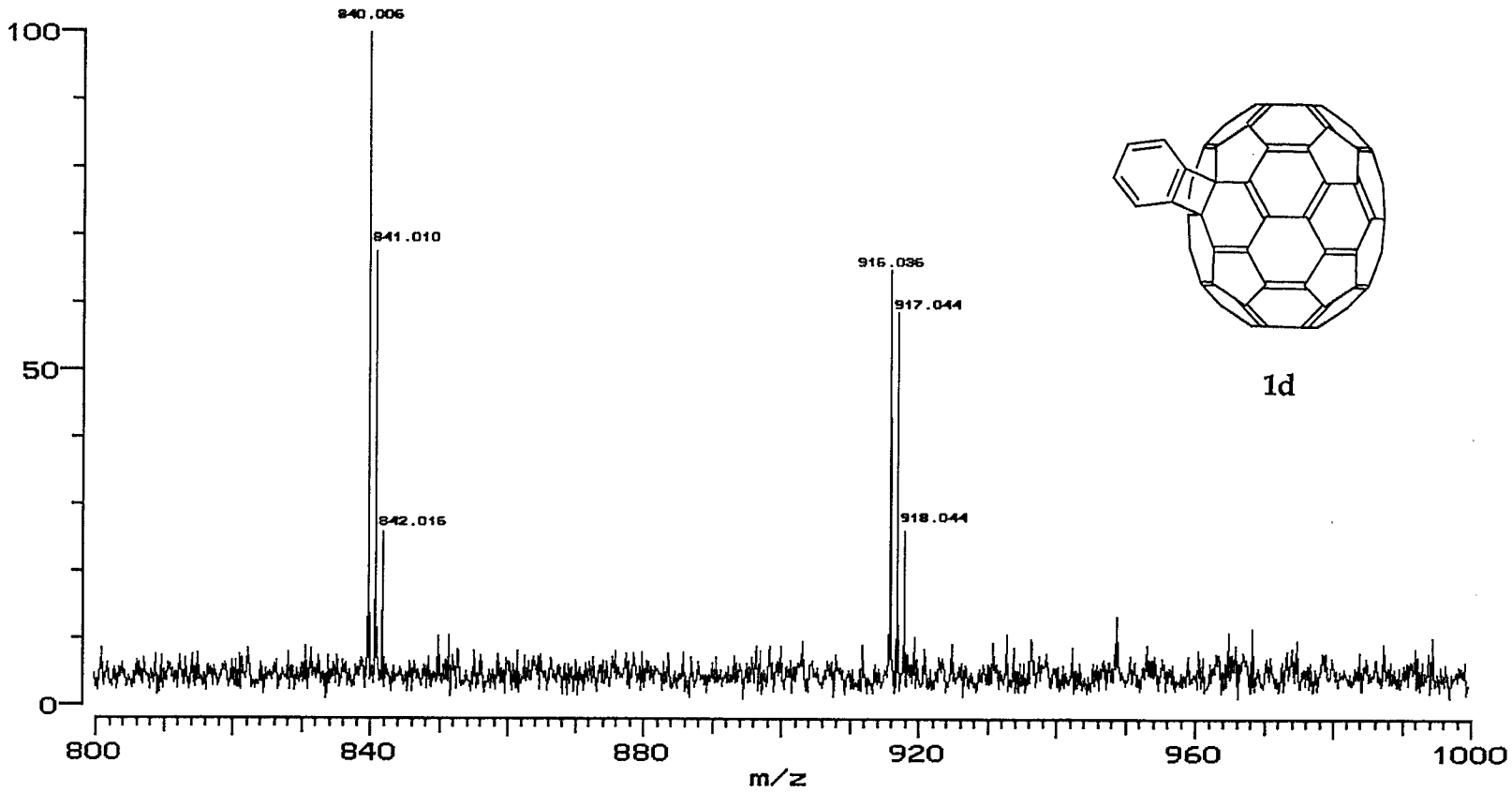


1d

Table 1. Crystallographic Data for C70BZ and Experimental Details of the Structure Determination

formula	$C_{76}H_4 \cdot C_7H_8$
formula mass	1009.01 amu
color	metallic black
melting point	>400° C
space group	$Pna2_1$ (#33)
cell dimensions	a = 19.434(2) Å
	b = 13.628(1) Å
	c = 15.491(1) Å
	α = 90.00(0)°
	β = 90.000(0)°
	γ = 90.00(0)°
volume	4102.1(7) $Å^3$
temperature	296(2) K
Z	4
imposed symmetry	none
density (calcd)	1.634 g cm^{-3}
F(000)	2040 e
crystallization conditions	grown at room temperature from solution in toluene and CS_2
crystal habit	irregular prisms
crystal dimensions	0.2 x 0.3 x 0.55 mm
radiation	Mo Kα (λ = 0.7107 Å)
monochromator	graphite
absorption coeff μ (Mo Kα)	0.09 mm^{-1}
diffractometer	Enraf-Nonius CAD-4
2θ range	4 – 50°
$(\sin\theta/\lambda)_{max}$	0.60 $Å^{-1}$

scan type	$\omega/2\theta$
scan range	$0.70 + 0.35\tan\theta$, deg
maximum counting time	100 s
index range	h: 0 – 23
	k: 0 – 16
	l: 0 – 18
X-ray exposure	77.6 hrs
number of standard reflections	3
decay	0.043 % (linear correction)
absorption correction procedure	none, $\mu R < 0.05$
total data collected	4041
unique data collected	3766
R factor for averaging	0.000
data with $I>2\sigma(I)$	1376
number of variables	138
number and type of restraints	7; 6 bond lengths and the floating origin restraint.
extinction coefficient	none
R [on F; $I > 2\sigma(I)$]	0.089
wR [on F^2; all data]	0.348
S	1.22
$(\Delta/\sigma)_{max}$	0.009
$\Delta(\rho)_{max}$	+0.48 e Å^{-3}
$\Delta(\rho)_{min}$	-0.38 e Å^{-3}

Table 2. Positional Parameters and Equivalent U Values for the Refined Atoms of C70BZ

Atom	x	y	z	$U_{iso}(A^2)$
C1'	0.2635(9)	0.7196(9)	0.8932(27)	0.040(4)
C2'	0.1943(8)	0.7393(9)	0.8883(32)	0.039(4)
C3'	0.1439(8)	0.6766(9)	0.8808(25)	0.034(4)
C4'	0.1698(9)	0.5925(10)	0.8890(32)	0.043(4)
C5'	0.2387(9)	0.5728(10)	0.8959(23)	0.040(5)
C6'	0.2887(9)	0.6364(10)	0.8992(21)	0.041(5)
C01	0.2888(7)	0.8148(8)	0.8977(14)	0.026(4)
C02	0.2060(8)	0.8387(9)	0.8845(18)	0.037(4)
C03	0.1818(9)	0.8967(11)	0.9677(12)	0.013(5)
C06	0.3272(8)	0.8542(12)	0.9834(13)	0.021(6)
C09	0.3294(8)	0.8454(12)	0.8111(13)	0.022(6)
C12	0.1861(12)	0.8887(14)	0.7943(16)	0.045(8)
C04	0.2217(3)	0.9184(3)	1.0500(3)	0.055(8)
C05	0.2944(3)	0.8939(3)	1.0593(3)	0.047(7)
C07	0.3936(2)	0.8816(2)	0.9475(4)	0.033(6)
C08	0.3960(2)	0.8773(2)	0.8438(4)	0.031(6)
C10	0.3035(2)	0.8852(3)	0.7284(3)	0.032(5)
C11	0.2290(2)	0.9061(3)	0.7196(3)	0.015(4)
C13	0.1402(2)	0.9580(3)	0.8301(4)	0.020(5)
C14	0.1329(2)	0.9604(3)	0.9342(5)	0.044(8)
C15	0.1271(2)	1.0387(3)	0.9831(5)	0.029(6)
C16	0.1678(2)	1.0580(3)	1.0690(4)	0.026(5)
C17	0.2141(3)	0.9981(4)	1.1043(3)	0.031(5)
C18	0.2811(3)	1.0260(4)	1.1384(3)	0.042(6)
C19	0.3303(3)	0.9624(3)	1.1103(3)	0.046(6)
C20	0.3986(3)	0.9851(3)	1.0861(3)	0.048(7)
C21	0.4313(2)	0.9428(3)	1.0023(4)	0.030(5)
C22	0.4818(2)	0.9886(3)	0.9488(4)	0.057(8)
C23	0.4841(2)	0.9857(3)	0.8442(4)	0.018(5)
C24	0.4358(2)	0.9371(3)	0.7898(4)	0.035(6)
C25	0.4068(2)	0.9745(3)	0.7004(3)	0.025(5)
C26	0.3395(3)	0.9504(3)	0.6721(3)	0.029(5)
C27	0.2918(3)	1.0122(4)	0.6352(2)	0.034(5)
C28	0.2234(3)	0.9861(3)	0.6653(3)	0.056(8)
C29	0.1759(2)	1.0476(3)	0.6923(4)	0.051(7)
C30	0.1316(2)	1.0330(3)	0.7758(5)	0.048(7)
C31	0.1240(2)	1.1148(3)	0.8258(5)	0.039(6)
C32	0.1219(1)	1.1175(3)	0.9268(5)	0.028(6)
C33	0.1582(2)	1.1850(3)	0.9789(5)	0.041(6)
C34	0.1864(2)	1.1485(3)	1.0661(5)	0.039(6)
C35	0.2494(3)	1.1781(3)	1.1055(4)	0.056(7)

C36	0.2985(3)	1.1146(4)	1.1431(3)	0.035(5)
C37	0.3695(3)	1.1338(4)	1.1385(3)	0.056(7)
C38	0.4189(3)	1.0701(4)	1.1104(3)	0.041(6)
C39	0.4743(2)	1.1144(3)	1.0622(4)	0.040(6)
C40	0.5071(2)	1.0764(3)	0.9835(4)	0.021(4)
C41	0.5227(1)	1.1217(3)	0.8935(4)	0.039(4)
C42	0.5067(2)	1.0646(3)	0.8126(4)	0.034(5)
C43	0.4806(2)	1.1007(3)	0.7279(4)	0.026(5)
C44	0.4281(3)	1.0581(3)	0.6720(3)	0.037(6)
C45	0.3803(3)	1.1200(4)	0.6353(3)	0.044(6)
C46	0.3095(3)	1.1004(4)	0.6259(3)	0.051(7)
C47	0.2591(3)	1.1657(3)	0.6544(4)	0.038(6)
C48	0.1944(2)	1.1382(3)	0.6903(4)	0.047(6)
C49	0.1626(2)	1.1793(3)	0.7722(5)	0.051(7)
C50	0.1943(2)	1.2494(2)	0.8209(5)	0.058(7)
C51	0.1922(2)	1.2524(2)	0.9282(6)	0.032(5)
C52	0.2468(2)	1.2927(2)	0.9789(5)	0.049(7)
C53	0.2750(2)	1.2563(3)	1.0662(5)	0.041(6)
C54	0.3476(2)	1.2809(3)	1.0674(4)	0.037(5)
C55	0.3926(3)	1.2193(3)	1.1020(3)	0.037(5)
C56	0.4576(2)	1.2050(3)	1.0532(4)	0.047(7)
C57	0.4754(2)	1.2546(3)	0.9723(4)	0.056(8)
C58	0.5075(2)	1.2091(3)	0.8909(4)	0.051(4)
C59	0.4796(2)	1.2440(3)	0.8003(4)	0.047(7)
C60	0.4659(2)	1.1919(3)	0.7198(4)	0.040(6)
C61	0.4028(3)	1.2002(3)	0.6645(3)	0.041(6)
C62	0.3568(3)	1.2638(3)	0.6909(4)	0.040(6)
C63	0.2830(3)	1.2459(3)	0.6903(4)	0.043(6)
C64	0.2511(2)	1.2871(2)	0.7723(5)	0.043(6)
C65	0.3038(2)	1.3318(2)	0.8212(5)	0.034(5)
C66	0.3029(2)	1.3388(2)	0.9220(5)	0.038(6)
C67	0.3645(2)	1.3297(2)	0.9803(5)	0.052(7)
C68	0.4261(2)	1.3154(2)	0.9318(5)	0.053(8)
C69	0.4301(2)	1.3105(2)	0.8256(5)	0.049(8)
C70	0.3694(2)	1.3203(2)	0.7733(5)	0.046(6)
C71	0.4766(7)	0.6147(6)	0.9415(7)	0.050
C72	0.4626(9)	0.5382(5)	0.8922(9)	0.050
C73	0.4607(10)	0.5370(7)	0.7902(9)	0.050
C74	0.4729(10)	0.6123(9)	0.7376(7)	0.050
C75	0.4869(11)	0.6888(7)	0.7869(9)	0.050
C76	0.4888(9)	0.6901(5)	0.8888(10)	0.050
C77	0.4791(12)	0.6159(11)	1.0494(7)	0.050

Table 3. Selected Bond Lengths (Å) and Angles (deg) in C70BZ

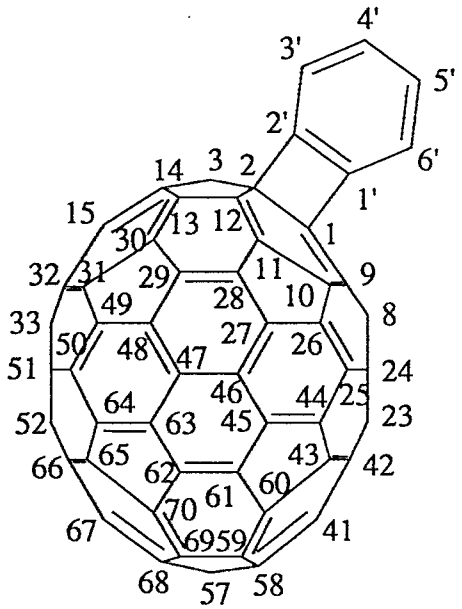
Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
C1'	C6'	1.38(2)	C1'	C2'	1.38(2)
C2'	C3'	1.38(2)	C3'	C4'	1.40(2)
C4'	C5'	1.38(2)	C5'	C6'	1.38(2)
C1'	C01	1.56(2)	C2'	C02	1.56(2)
C01	C09	1.50(2)	C01	C06	1.51(2)
C01	C02	1.66(2)	C02	C12	1.50(2)
C02	C03	1.52(2)	C03	C04	1.40(2)
C03	C14	1.44(2)	C06	C05	1.36(2)
C06	C07	1.442(14)	C09	C10	1.38(2)
C09	C08	1.46(2)	C12	C11	1.34(2)
C12	C13	1.48(2)			

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
C6'	C1'	C2'	123.7(14)	C6'	C1'	C01	140.4(14)
C2'	C1'	C01	95.7(12)	C3'	C2'	C1'	122.6(14)
C3'	C2'	C02	143(2)	C1'	C2'	C02	94.5(12)
C2'	C3'	C4'	113(2)	C5'	C4'	C3'	124.2(14)
C4'	C5'	C6'	122(2)	C1'	C6'	C5'	115(2)
C09	C01	C06	102.7(11)	C09	C01	C1'	116(2)
C06	C01	C1'	125(2)	C09	C01	C02	111(2)
C06	C01	C02	118.2(14)	C1'	C01	C02	84.4(10)
C12	C02	C03	103.0(12)	C12	C02	C2'	120(2)
C03	C02	C2'	121(2)	C12	C02	C01	117(2)
C03	C02	C01	111(2)	C2'	C02	C01	85.2(10)
C04	C03	C14	116.9(10)	C04	C03	C02	124.5(14)
C14	C03	C02	111.8(11)	C05	C06	C07	122.9(13)
C05	C06	C01	122.4(13)	C07	C06	C01	107.4(11)
C10	C09	C08	115.1(11)	C10	C09	C01	126.3(13)
C08	C09	C01	109.6(12)	C11	C12	C13	118.7(14)
C11	C12	C02	124(2)	C13	C12	C02	105.1(12)
C03	C04	C17	123.8(6)	C03	C04	C05	122.5(7)
C17	C04	C05	106.0	C06	C05	C19	118.0(7)
C06	C05	C04	120.2(8)	C19	C05	C04	108.4
C21	C07	C08	122.8	C21	C07	C06	118.9(7)
C08	C07	C06	110.8(8)	C24	C08	C07	120.5
C24	C08	C09	123.3(7)	C07	C08	C09	107.0(7)
C09	C10	C26	124.6(6)	C09	C10	C11	121.2(7)
C26	C10	C11	106.0	C12	C11	C28	120.8(8)
C12	C11	C10	120.0(10)	C28	C11	C10	107.5
C30	C13	C14	119.8	C30	C13	C12	120.3(8)
C14	C13	C12	114.0(9)	C15	C14	C13	120.5
C15	C14	C03	119.9(6)	C13	C14	C03	103.4(7)

Table 4. Positional Parameters and U Values for the Hydrogen Atoms of C70BZ

Atom	x	y	z	U_{iso} (Å^2)
H3'	0.0975(8)	0.6890(9)	0.8713(25)	0.041
H4'	0.1384(9)	0.5472(10)	0.8899(32)	0.051
H5'	0.2521(9)	0.5151(10)	0.8985(23)	0.048
H6'	0.3353(9)	0.6241(10)	0.9049(21)	0.050
H72	0.4544(13)	0.4878(6)	0.9274(11)	0.060
H73	0.4513(14)	0.4857(9)	0.7572(11)	0.060
H74	0.4716(14)	0.6114(12)	0.6694(7)	0.060
H75	0.4951(15)	0.7392(9)	0.7517(12)	0.060
H76	0.4982(12)	0.7413(6)	0.9218(12)	0.060
H77A	0.4708(14)	0.6736(12)	1.0725(9)	0.060
H77A	0.5236(14)	0.5970(13)	1.0711(10)	0.060
H77C	0.4445(14)	0.5780(13)	1.0751(8)	0.060

Figure 1. Line drawing of C70BZ showing the atom-numbering scheme.



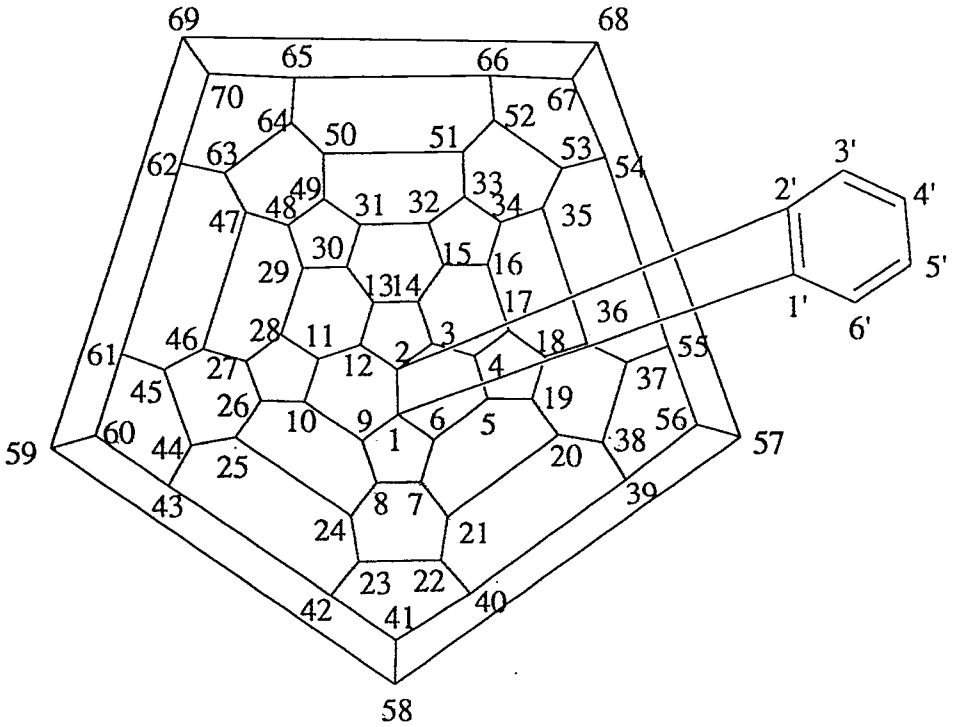


Figure 3. Stereoscopic view of the unit cell of C70BZ.

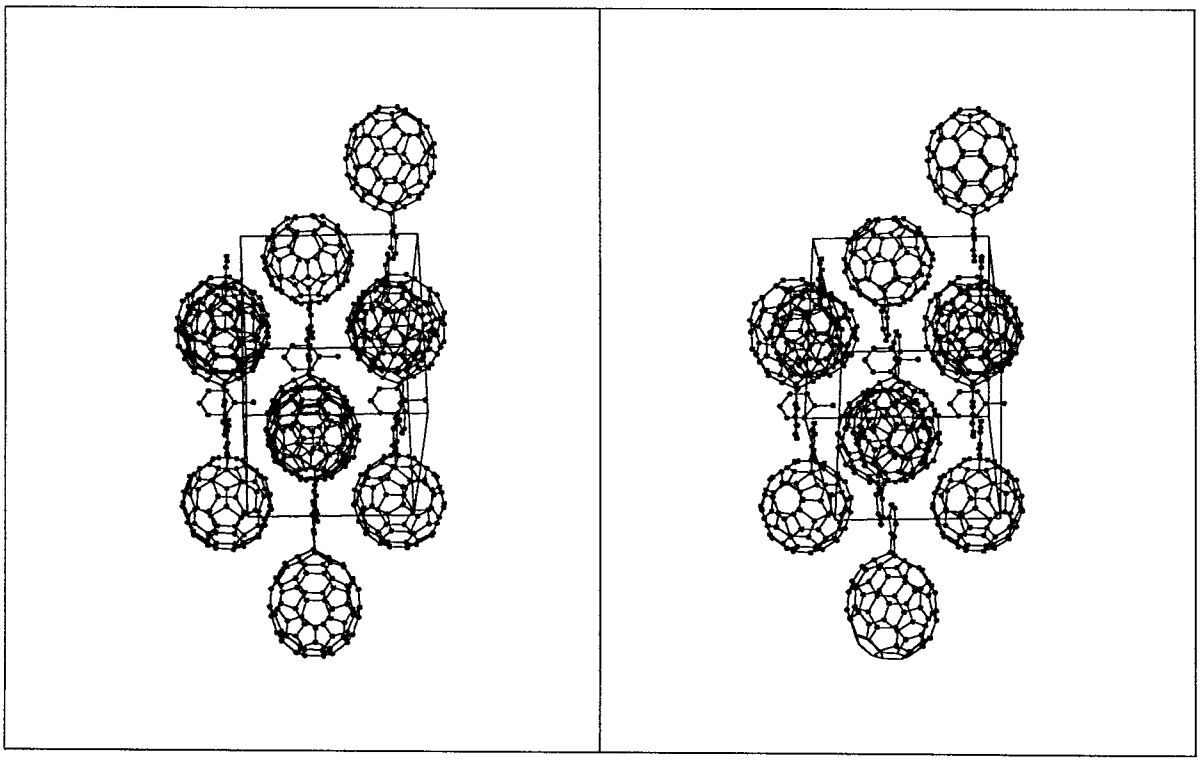
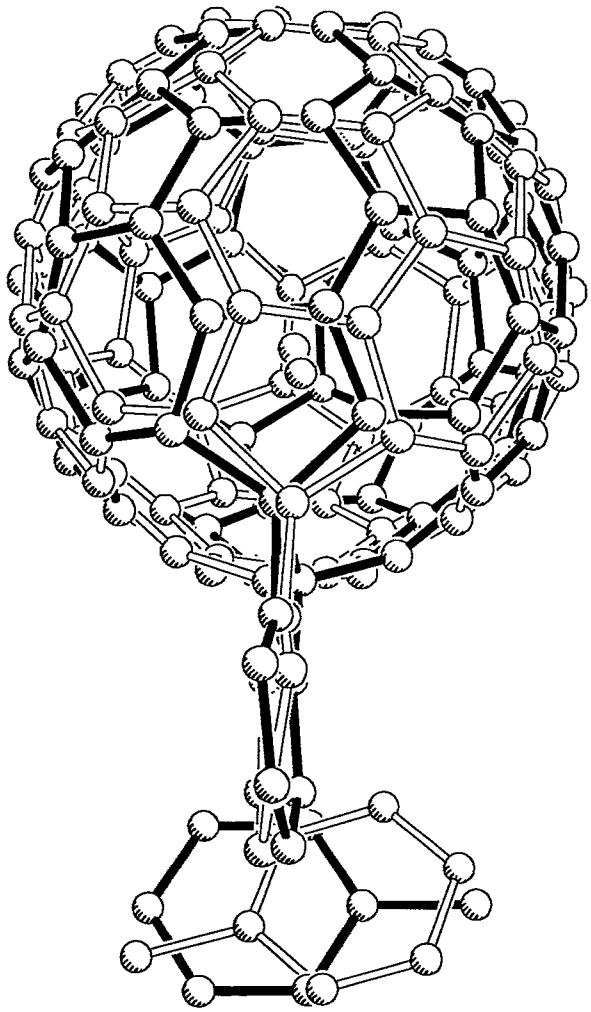



Figure 4. Perspective drawing of the pseudo-symmetric molecules, oriented with the pseudo-mirror perpendicular to the page; hydrogen atoms have been omitted for the sake of clarity.