Supplementary Material for

"Preparation of Diynes via Selective Bisalkynylation of Zirconacycles"

General procedure for monoalkynylation of zirconacycles. (1*R,2*S**)-1-(3'-phenylprop-2'-yn-1'-yl)-2-methylcyclopentane (6b).** To a solution of **1b**, prepared *in situ* from Cp_2ZrCl_2 (292 mg,1 mmol), BuLi and heptadiene (96 mg, 1 mmol), were added CuCl (10 mg, 0.1 mmol) and phenylethynyl bromide (181 mg, 1 mmol) at 20°C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with 3N HCl and extracted with hexane. GC analysis indicated that **6b** was formed in 80% yield. Column chromatography on silica gel (hexane) afforded 123 mg (62%) of **6b** as a colorless liquid: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 1.03 (d, *J* = 6.5 Hz, 3H), 1.20 (dddd, *J* = 12.4, 8.2, 8.2, 8.2 Hz, 1H), 1.44 (dddd, *J* = 12.0, 8.2, 8.2, 8.2 Hz, 1H), 1.50-1.68 (m, 4H), 1.83-1.94 (m, 2H), 2.34 (dd, *J* = 16.8, 6.8 Hz, 1H), 2.51 (dd, *J* = 16.8, 5.2 Hz, 1H), 7.22-7.39 (m, 5H); ^{13}C NMR ($CDCl_3$, Me_4Si) δ 19.31, 23.48, 23.63, 32.01, 34.88, 39.64, 46.43, 80.87, 89.58, 124.22, 127.36, 128.12 (2C), 131.52 (2C); IR (neat) 2951, 2232, 1599, 1491, 1375, 756, 691 cm^{-1}; UV-vis (Et_2O): 239, 251 nm; HRMS calcd for $C_{15}H_{18}$ 198.1408, found 198.1411.

5-Decyne (6a). GC yield 54%. Isolated yield 34%. A colorless liquid: the spectral data were consistent with those of commercially available authentic sample.

(4*R,5*R**)-1,2-Dibutyl-4-(hept-2'-yn-1'-yl)-5-methylcyclohexene (6c).** GC yield 75%. Isolated yield 57%. A colorless liquid: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 0.79 (d, *J* = 6.9 Hz, 3H), 0.89-0.92 (m, 9H), 1.28-1.40 (m, 10H), 1.40-1.55 (m, 8H), 1.65 (dd, *J* = 16.9, 4.9 Hz, 1H), 1.72-2.15 (m, 7H); ^{13}C NMR ($CDCl_3$, Me_4Si) δ 13.59, 14.10 (2C), 14.60, 18.49, 20.70, 21.93, 22.88 (2C), 30.28, 30.83, 30.86, 31.33, 32.62 (2C), 32.64, 36.62, 37.77, 79.48, 80.61, 128.30, 128.34; HRMS calcd for $C_{22}H_{38}$ 302.2972, found 302.2979. Anal. Calcd for $C_{22}H_{38}$: C, 87.34; H, 12.66. Found: C, 87.37, H, 12.70.

(6*R,7*S**)-1,2,3,4-Tetraethyl-6-(3'-phenylprop-2'-yn-1'-yl)-7-methylcycloocta-1,3-diene (6d).**

Phenylethynyl bromide (181 mg, 1 mmol) and CuCl (99 mg, 1 mmol) were added to a solution of **1d**, prepared *in situ* from Cp_2ZrCl_2 (292 mg,1 mmol), at 0°C and the reaction mixture was stirred for

3h. The reaction mixture was quenched with 3 N HCl and extracted with hexane. GC analysis showed that **6d** was formed in 46% yield. Column chromatography on silica gel (hexane) gave 132 mg (38%) of **6d** as a colorless liquid: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 0.80 (t, J = 7.4 Hz, 6H), 0.99 (d, J = 6.4 Hz, 3H), 1.02 (t, J = 7.7 Hz, 3H), 1.05 (t, J = 7.5 Hz, 3H), 1.73(d, J = 12.9 Hz, 1H), 1.85-2.05 (m, 6H), 2.08-2.38 (m, 8H), 2.61 (dd, J = 16.9, 2.9Hz, 1H), 7.24-7.3(m, 5H); ^{13}C NMR ($CDCl_3$, Me_4Si) d 12.56, 12.61, 13.65, 13.69, 21.67, 21.73, 23.26, 24.25, 24.74, 26.30, 33.94, 36.76, 40.45, 42.10, 81.34, 89.84, 124.20, 127.32, 128.13(2C), 131.42(2C), 134.67, 134.83, 136.08, 136.27; HRMS calcd for $C_{26}H_{36}$ 348.2817, found 348.2830.

General procedure for monoalkynylation of monocyclic zirconacyclopentenes. 3,4-Diethyldec-3-en-5-yne (9a). To a solution of zirconacyclopentene **2a**, prepared *in situ* from Cp_2ZrCl_2 (365 mg,1.25 mmol), EtMgBr (2 equiv) and 3-hexyne (84 mg, 1 mmol), were added CuCl (99 mg, 1 mmol) and hexynyl bromide (161 mg, 1 mmol) 20°C. After stirring at the same temperature for 1h, the reaction mixture was quenched with 3N HCl and extracted with hexane. GC analysis indicated that **9a** was formed in 89% yield. Column chromatography on silica gel (hexane) afforded 129 mg (67%) of **9a** as a colorless liquid: ^{1}H NMR ($CDCl_3$, Me_4Si) d 0.92 (t, J = 7.3 Hz, 3H), 0.96 (t, J = 7.6 Hz, 3H), 1.00 (t, J = 7.6 Hz, 3H), 1.06 (t, J = 7.5 Hz, 3H), 1.44-1.54 (m, 4H), 2.10 (q, J = 7.5 Hz, 2H), 2.12 (q, J = 7.6 Hz, 2H), 2.31 (q, J = 7.6 Hz, 2H), 2.35 (t, J = 6.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, Me_4Si) d 12.83, 13.26, 13.59, 13.69, 19.20, 21.95, 23.67, 25.16, 27.73, 31.26, 80.73, 91.91, 118.59, 148.52; HRMS calcd for $C_{14}H_{24}$ 192.1877, found 192.1879.

(3*E*)-1-Phenyl-3-propyl-4-ethylhept-3-en-1-yne (9b). GC yield 81%. Isolated yield 50%. A colorless liquid: ^{1}H NMR ($CDCl_3$, Me_4Si) d 0.91-0.97 (m, 6H), 1.08 (t, J = 7.5 Hz, 3H), 1.42 (q, J = 7.5 Hz, 2H), 1.61 (q, J = 7.5 Hz, 2H), 2.11-2.17 (m, 2H), 2.19 (t, J = 7.3 Hz, 2H), 2.42 (q, J = 7.5 Hz, 2H), 7.22-7.42 (m, 5H); ^{13}C NMR ($CDCl_3$, Me_4Si) d 13.01, 13.77, 14.30, 21.83, 22.17, 28.36, 33.24, 33.70, 90.50, 91.58, 117.38, 124.51, 127.33, 128.16 (2C), 131.19 (2C), 150.27; IR (neat) 2963, 2201, 1595, 1489, 1462, 754, 691 cm^{-1}; UV-vis (Et_2O): 221, 281, 298 nm; HRMS calcd for $C_{18}H_{24}$ 240.1877, found 240.1870.

(3*E*)-3,4-Diphenyldec-3-en-5-yne (9c). GC yield 95%. Isolated yield 75%. A colorless liquid: ^{1}H NMR ($CDCl_3$, Me_4Si) d 0.94 (t, J = 7.2 Hz, 3H), 1.03 (t, J = 7.6 Hz, 3H), 1.7-1.60 (m, 4H), 2.42 (t, J = 7.0 Hz, 2H), 2.87(q, J = 7.5 Hz, 2H), 6.98-7.12(m, 10H); ^{13}C NMR ($CDCl_3$,

Me_4Si) d 12.47, 13.57, 19.40, 22.01, 31.02, 31.08, 81.23, 94.78, 120.45, 126.23, 126.54, 127.43 (2C), 127.81 (2C), 129.18 (2C), 129.76 (2C), 139.66, 140.99, 150.00; IR (neat) 2963, 2215, 1599, 1460, 770 cm^{-1}; UV -vis (Et_2O): 241, 285 nm; HRMS calcd for $C_{22}H_{24}$ 288.1878, found 288.1875.

(3*E*)-1-Trimethylsilyl-3,4-diphenylhex-3-en-1-yne (9d). GC yield 91%. Isolated yield 77%. A colorless liquid: ^1H NMR ($CDCl_3$, Me_4Si) d 0.33 (s, 9H), 1,13 (t, *J* = 7.5 Hz, 3H), 2.99 (q, *J* = 7.2 Hz, 2H), 7.07-7.22 (m, 10H); ^{13}C NMR ($CDCl_3$, Me_4Si) d 0.07 (3C), 12.25, 31. 37, 98.60, 105.75, 120.18, 126.48, 126.88, 127.54 (2C), 127.92 (2C), 129.02 (2C), 129.80 (2C), 138.57, 140.60, 152.91; HRMS calcd for $C_{21}H_{24}Si$ 304.1647, found 304.1642.

General procedure for monoalkynylation of bicyclic zirconacyclopentenes. (1'*E*)-1-(1'-Hexyl-3'-phenylprop-2'-yn-1'-ylidene)-2-methylcyclohexane (9e). Hexynyl bromide (161 mg, 1 mmol) and CuCl (99 mg, 1 mmol) were added to a solution of **2d**, prepared *in situ* from Cp_2ZrCl_2 (292 mg,1 mmol), BuLi (2 equiv), and tetradec-1-en-7-yne (192 mg, 1 mmol),[7b] at 0°C. The reaction mixture was quenched after 1 h with 3 N HCl and extracted with hexane. GC analysis indicated that **9e** was formed in 75% yield. Column chromatography on silica gel (hexane) gave 174 mg (67%) of **9e** as a colorless liquid: ^1H NMR ($CDCl_3$, Me_4Si) d 0.89 (t, *J* = 6.6 Hz, 3H), 1.12 (d, *J* = 7.2 Hz, 2H), 1.20-1.40 (m, 7H), 1.45-1.70 (m, 7H), 1.80 (d, *J* = 11.5 Hz, 1H), 2.00 (td, *J* = 13.8, 4.4 Hz, 1H), 2.22 (t, *J* = 7.5 Hz, 2H), 2.53 (d, *J* = 13.9 Hz, 1H), 3.44 (m, 1H), 7.23-7.42 (m, 5H); ^{13}C NMR ($CDCl_3$, Me_4Si) d 14.08, 18.26, 20.72, 22.67, 25.23, 27.80, 28.81, 29.08, 31.51, 31.77, 33.15, 35.23, 90.50, 91.50, 114.17, 124.51, 127.28, 128.14(2C), 131.22(2C), 151.43; HRMS calcd for $C_{22}H_{30}$ 294.2348, found 294.2341.

(1'*E*)-1-(1'-Hexylhept-2'-yn-1'-ylidene)-2-methylcyclopentane (9f). GC yield 59%. Isolated yield 53%. A colorless liquid: ^1H NMR ($CDCl_3$, Me_4Si) d 0.88 (t, *J* = 7.0 Hz, 3H), 0.92 (t, *J* = 7.2 Hz, 3H), 1.08 (d, *J* = 7.1 Hz, 3H), 1.28-1.54 (m, 16H), 2.03 (t, *J* = 7.5 Hz, 2H), 2.31-2.36 (m, 4H), 2.83 (t, *J* = 6.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, Me_4Si) d 13.59, 14.07, 19.23, 19.29, 21.95, 22.66, 23.99, 28.29, 28.93, 30.30, 31.27, 31.85, 33.79, 34.34, 38.71, 81.00, 91.77, 114.29, 155.16; HRMS calcd for $C_{19}H_{32}$ 260.2504, found 260.2507.

X-ray Structural Analysis of 14. X-ray data were collected on an Enraf-Nonius CAD4 diffractometer with graphite-monochromated Cu *Ka* radiation(λ=1.54184 Å). A single crystal of

approximate dimensions 0.25 x 0.30 x 0.40 mm was mounted on the top a glass fiber. Crystallographic data and experimental details are summarized in the table. Three standard reflections were monitored after every 2 h of X-ray exposure during the data collection, and no significant crystal decay was detected. The structure was readily solved using direct methods (SHELXS-86[1]), and refinment was performed with Xtal 3.2 software[2] by full-matrix least-squares techniques on *F*. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were located by difference maps and refined isotropically.

1. Sheldrick, G. M. "*SHELXS-86, Program for Crystal Structure Determination*," Univ. of Göttingen, Germany (1986).
2. Hall, S. R.; Flack, H. D. and Stewart, J. M. "*Xtal3.2, Program for X-Ray Crystal Structure Analysis*," Universities of Western Australia, Geneva, and Maryland (1992).

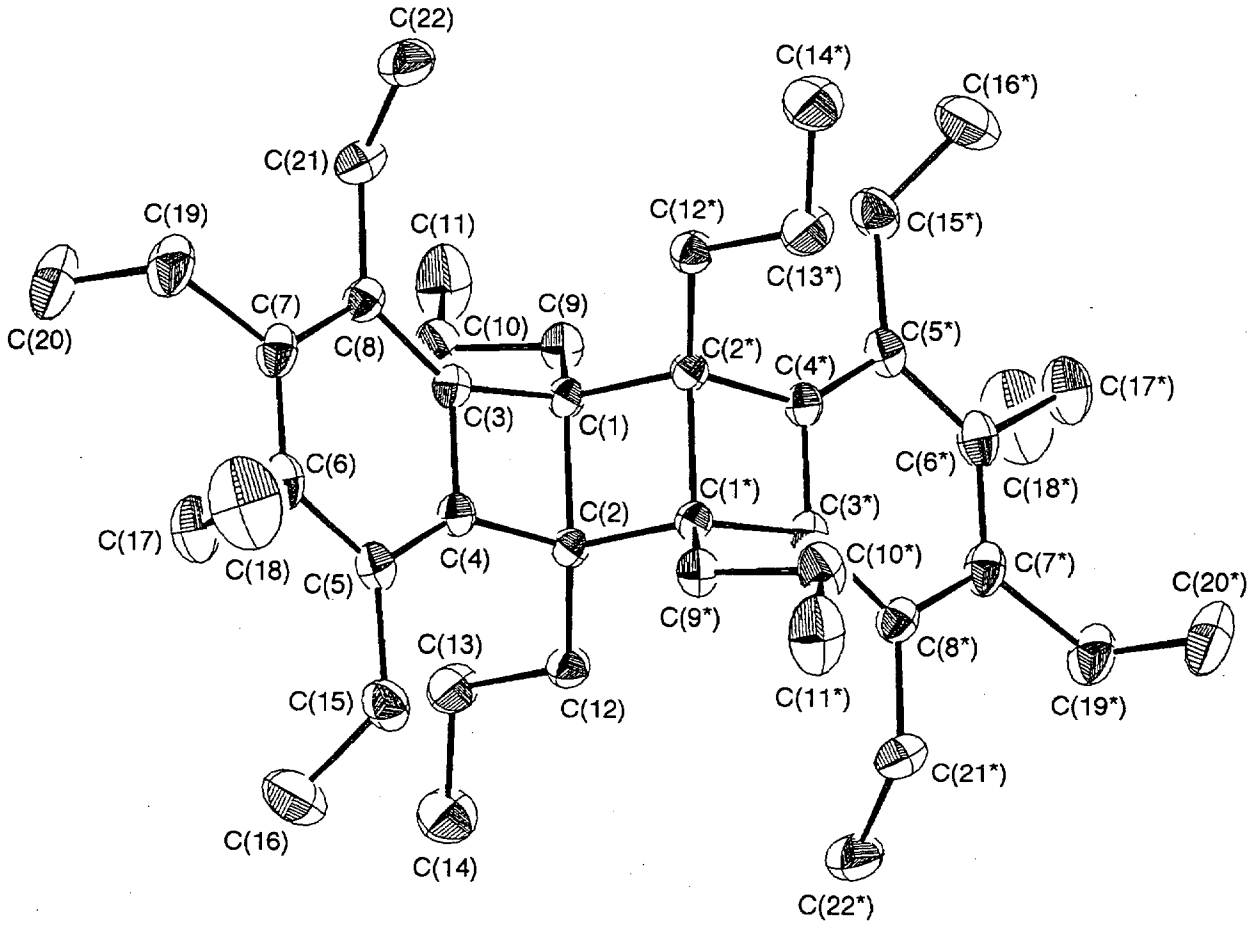


Figure 1. Perspective view of **14**.

Crystallographic Data and Experimental Details

Compound	**14**
Formula	$C_{44}H_{68}$
M	597.02
Crystal system	monoclinic
Space group	$P2_1/n$ (No. 14)
a, (Å)	10.7689(5)
b, (Å)	9.8235(4)
c, (Å)	18.4390(7)
β, (°)	100.456(4)
Z	2
V, (Å^3)	1918.2(1)
μ(Cu $K\alpha$), (cm^{-1})	3.9
Crystal color	colorless
Crystal habit	prismatic
Crystal size, (mm^3)	0.25x0.30x0.40
D_{calcd}, (g cm^{-3})	1.03
F(000)	664
Diffractometer	Enraf-Nonius CAD4
λ(Cu $K\alpha$), (Å)	1.54184
T, K	298
Scan range, (°)	0.69+0.15tanθ
Scan mode	ω-2θ
Scan speed, (° min^{-1})	variable
$2\theta_{max}$, (°)	148.52
Reflections measd	$-13 \leq h \leq 0$ $-12 \leq k \leq 0$ $-22 \leq l \leq 23$
No. of reflections measd	4100
No. of reflections obsd [|Fo|>3σ(|Fo|)]	3360
No. of parameters refined	335
R	0.056
R_w	0.072
S, goodness of fit	3.07
$(\Delta/\delta)_{max}$	0.34
Largest diff peak, (e Å^{-3})	0.20
Largest diff hole, (e Å^{-3})	-0.30

$R=\Sigma||Fo|-|Fc||/\Sigma|Fo|$.
$R_w=[\Sigma w||Fo|-|Fc||^2/\Sigma w|Fo|^2]^{1/2}$, $w=[\sigma^2(Fo)+\{0.015(Fo)\}^2]^{-1}$.
$S=[\Sigma w||Fo|-|Fc||^2/(m-n)]^{1/2}$ (m=no. of used reflections, n=no. of refined parameters).

Atomic Coordinates and Equivalent Isotropic Displacement Parameters ($Å^2$)

atom	x	y	z	U_{eq}
C(1)	0.0095(1)	0.4522(1)	0.55660(6)	0.0302(3)
C(2)	-0.0036(1)	0.3970(1)	0.47297(6)	0.0302(3)
C(3)	0.1442(1)	0.3946(1)	0.56766(7)	0.0327(4)
C(4)	0.1325(1)	0.3488(1)	0.49591(7)	0.0327(4)
C(5)	0.2309(1)	0.2877(1)	0.46852(7)	0.0382(4)
C(6)	0.3444(1)	0.2718(1)	0.52004(9)	0.0432(4)
C(7)	0.3552(1)	0.3153(2)	0.59386(8)	0.0434(4)
C(8)	0.2545(1)	0.3809(1)	0.61958(7)	0.0379(4)
C(9)	-0.0758(1)	0.4206(1)	0.61201(7)	0.0381(4)
C(10)	-0.0581(2)	0.2858(2)	0.6528(1)	0.0519(5)
C(11)	-0.1380(3)	0.2719(3)	0.7105(1)	0.0796(9)
C(12)	-0.1025(1)	0.2996(1)	0.43345(8)	0.0381(4)
C(13)	-0.1036(2)	0.1572(2)	0.4646(1)	0.0593(6)
C(14)	-0.1886(3)	0.0595(2)	0.4169(1)	0.0768(8)
C(15)	0.2144(2)	0.2411(2)	0.38900(9)	0.0522(5)
C(16)	0.1906(3)	0.0889(2)	0.3770(2)	0.0798(9)
C(17)	0.4580(2)	0.2115(2)	0.4930(1)	0.0650(7)
C(18)	0.5389(3)	0.3219(4)	0.4660(3)	0.107(1)
C(19)	0.4766(2)	0.2863(2)	0.6486(1)	0.0608(6)
C(20)	0.4787(2)	0.1451(3)	0.6826(2)	0.0834(9)
C(21)	0.2638(2)	0.4316(2)	0.69809(8)	0.0503(5)
C(22)	0.3406(2)	0.5618(2)	0.7166(1)	0.0664(7)

$U_{eq}=1/3\{\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \boldsymbol{a}_i \boldsymbol{a}_j\}$

Atomic Coordinates and Equivalent Isotropic Displacement Parameters ($Å^2$)

atom	x	y	z	U_{iso}
H(9A)	-0.169(2)	0.437(2)	0.585(1)	0.058(5)
H(9B)	-0.057(2)	0.494(2)	0.655(1)	0.066(5)
H(10A)	-0.067(3)	0.209(3)	0.623(2)	0.111(9)
H(10B)	0.040(3)	0.274(3)	0.677(1)	0.110(9)
H(11A)	-0.131(3)	0.185(3)	0.736(2)	0.108(9)
H(11B)	-0.208(3)	0.320(3)	0.706(2)	0.12(1)
H(11C)	-0.100(4)	0.362(5)	0.750(2)	0.19(2)
H(12A)	-0.091(2)	0.293(2)	0.382(1)	0.053(5)
H(12B)	-0.187(2)	0.340(2)	0.430(1)	0.061(5)
H(13A)	-0.140(3)	0.168(3)	0.510(2)	0.114(9)
H(13B)	-0.025(3)	0.126(3)	0.489(2)	0.101(8)
H(14A)	-0.194(3)	-0.026(3)	0.438(2)	0.110(9)
H(14B)	-0.270(3)	0.101(3)	0.389(2)	0.12(1)
H(14C)	-0.143(3)	0.038(4)	0.373(2)	0.15(1)
H(15A)	0.290(2)	0.266(2)	0.368(1)	0.075(6)
H(15B)	0.140(2)	0.295(2)	0.358(1)	0.062(5)
H(16A)	0.256(3)	0.024(4)	0.402(2)	0.12(1)
H(16B)	0.118(4)	0.062(3)	0.394(2)	0.14(1)
H(16C)	0.184(3)	0.068(3)	0.322(2)	0.13(1)
H(17A)	0.510(2)	0.151(3)	0.531(1)	0.086(7)
H(17B)	0.427(2)	0.140(2)	0.448(1)	0.071(6)
H(18A)	0.576(3)	0.388(4)	0.509(2)	0.15(1)
H(18B)	0.610(3)	0.280(3)	0.452(2)	0.13(1)
H(18C)	0.480(4)	0.380(4)	0.427(2)	0.15(1)
H(19A)	0.549(2)	0.301(2)	0.623(1)	0.083(7)
H(19B)	0.491(2)	0.360(2)	0.693(1)	0.077(6)
H(20A)	0.562(3)	0.136(3)	0.724(2)	0.13(1)
H(20B)	0.470(3)	0.067(3)	0.642(2)	0.12(1)
H(20C)	0.412(3)	0.139(3)	0.711(2)	0.106(9)
H(21A)	0.297(2)	0.360(2)	0.738(1)	0.069(6)
H(21B)	0.176(2)	0.446(2)	0.711(1)	0.059(5)
H(22A)	0.301(3)	0.646(3)	0.692(2)	0.113(9)
H(22B)	0.430(3)	0.551(3)	0.711(2)	0.11(1)
H(22C)	0.345(3)	0.587(3)	0.767(2)	0.109(9)

Atomic Anisotropic Displacement Parameters ($Å^2$)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	.0303(6)	.0307(6)	.0296(6)	.0001(4)	.0055(4)	.0015(4)
C(2)	.0312(6)	.0304(6)	.0293(5)	-.0001(4)	.0059(4)	.0006(4)
C(3)	.0307(6)	.0322(6)	.0355(6)	-.0003(5)	.0067(5)	.0033(5)
C(4)	.0328(6)	.0310(6)	.0347(6)	.0010(5)	.0076(5)	.0027(5)
C(5)	.0411(7)	.0326(6)	.0437(7)	.0035(5)	.0150(5)	.0038(5)
C(6)	.0356(7)	.0386(7)	.0581(8)	.0053(5)	.0156(6)	.0069(6)
C(7)	.0307(6)	.0422(7)	.0553(8)	.0004(5)	.0028(6)	.0077(6)
C(8)	.0341(6)	.0393(7)	.0388(7)	-.0025(5)	.0027(5)	.0037(5)
C(9)	.0384(7)	.0400(7)	.0380(7)	.0027(5)	.0131(5)	.0052(5)
C(10)	.067(1)	.0430(8)	.0519(8)	.0035(7)	.0259(8)	.0115(7)
C(11)	.088(2)	.082(2)	.082(1)	.016(1)	.050(1)	.038(1)
C(12)	.0396(7)	.0357(7)	.0373(7)	-.0042(5)	.0021(5)	-.0006(5)
C(13)	.074(1)	.0398(8)	.0566(9)	-.0175(8)	-.0085(9)	.0066(7)
C(14)	.098(2)	.044(1)	.079(1)	-.025(1)	-.011(1)	-.0027(9)
C(15)	.065(1)	.0483(8)	.0487(8)	.0109(7)	.0236(7)	-.0008(6)
C(16)	.112(2)	.059(1)	.072(1)	-.002(1)	.026(1)	-.019(1)
C(17)	.0442(9)	.069(1)	.087(1)	.0171(8)	.0243(9)	.004(1)
C(18)	.068(1)	.116(2)	.154(3)	.003(2)	.068(2)	.015(2)
C(19)	.0345(8)	.066(1)	.077(1)	.0040(7)	-.0052(7)	.0075(9)
C(20)	.065(1)	.083(2)	.092(2)	.013(1)	-.012(1)	.030(1)
C(21)	.0491(8)	.0592(9)	.0387(7)	-.0048(7)	-.0024(6)	-.0013(6)
C(22)	.074(1)	.060(1)	.056(1)	-.0101(9)	-.0119(9)	-.0069(8)

Expression for displacement parameters is

$\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)]$

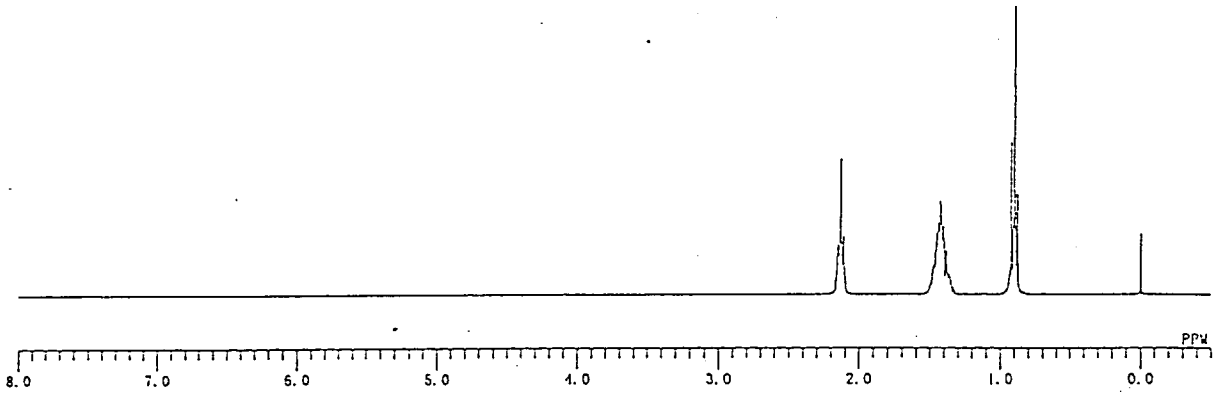
Intramolecular Distances (Å) with e.s.d. in parentheses

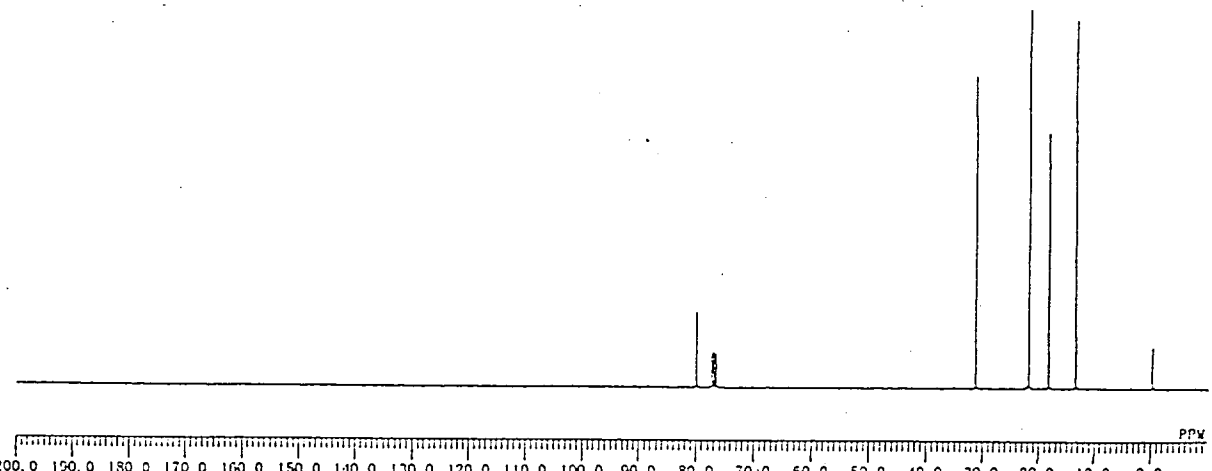
C(1)–C(2)	1.616(2)	C(6)–C(17)	1.523(3)
C(1)–C(3)	1.536(2)	C(7)–C(8)	1.415(2)
C(1)–C(9)	1.525(2)	C(7)–C(19)	1.526(2)
C(1)–C(2*)	1.575(2)	C(8)–C(21)	1.517(2)
C(2)–C(4)	1.525(2)	C(9)–C(10)	1.518(2)
C(2)–C(12)	1.517(2)	C(10)–C(11)	1.490(4)
C(3)–C(4)	1.381(2)	C(12)–C(13)	1.513(2)
C(3)–C(8)	1.390(2)	C(13)–C(14)	1.497(3)
C(4)–C(5)	1.390(2)	C(15)–C(16)	1.526(3)
C(5)–C(6)	1.414(2)	C(17)–C(18)	1.530(4)
C(5)–C(15)	1.516(2)	C(19)–C(20)	1.521(3)
C(6)–C(7)	1.411(2)	C(21)–C(22)	1.528(3)

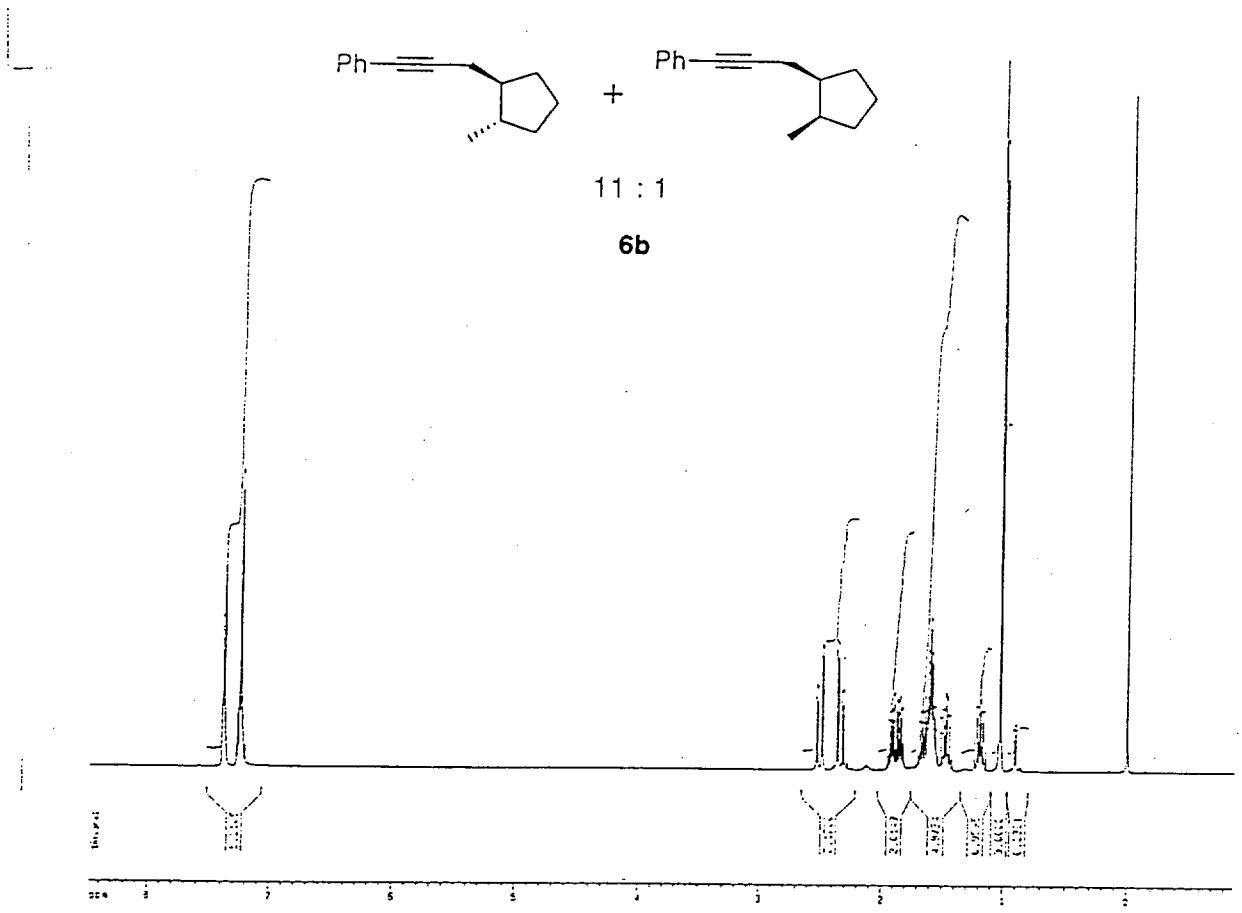
Intramolecular Angles (degrees) with e.s.d. in parentheses

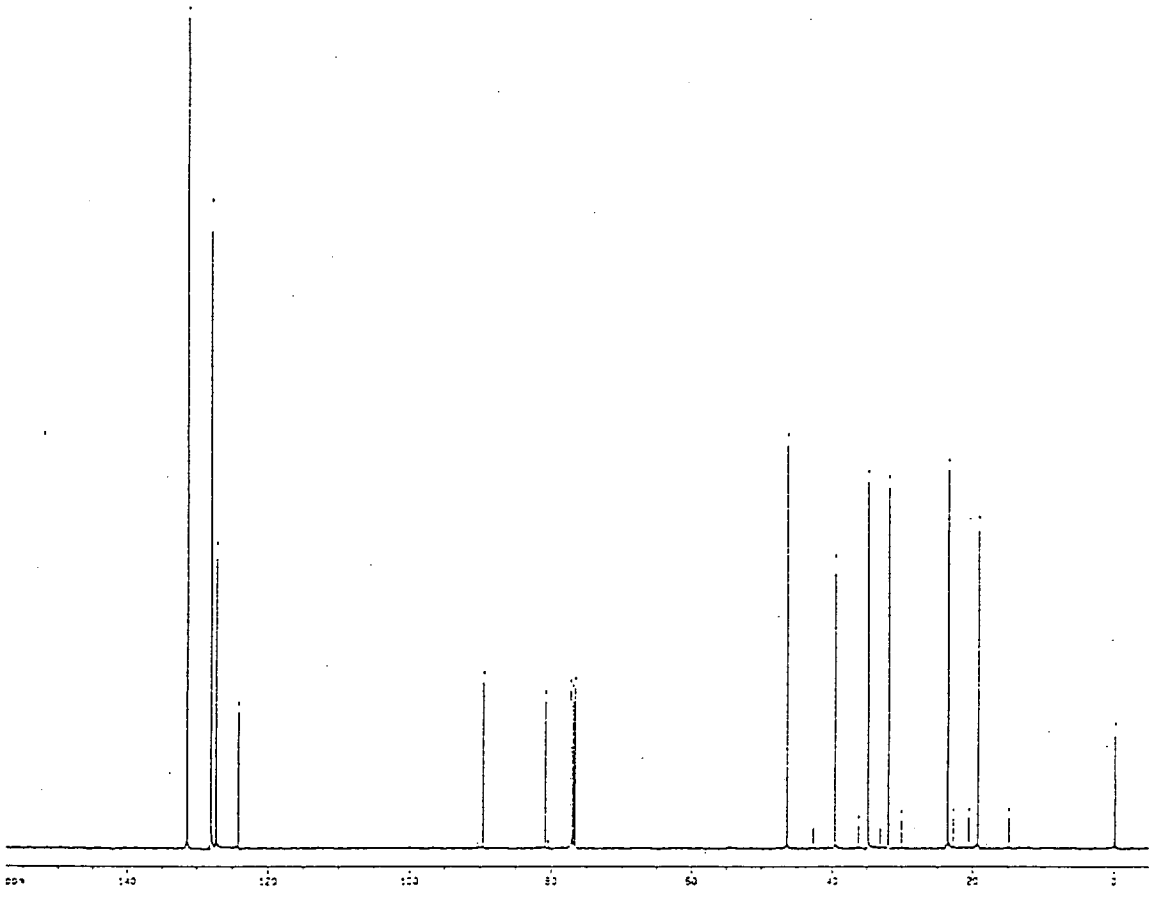
C(2)–C(1)–C(3)	85.40(9)	C(4)–C(5)–C(15)	121.3(1)
C(2)–C(1)–C(9)	127.7(1)	C(6)–C(5)–C(15)	123.5(1)
C(2)–C(1)–C(2*)	89.66(8)	C(5)–C(6)–C(7)	121.6(1)
C(3)–C(1)–C(9)	120.4(1)	C(5)–C(6)–C(17)	118.1(1)
C(3)–C(1)–C(2*)	111.7(1)	C(7)–C(6)–C(17)	120.2(1)
C(9)–C(1)–C(2*)	115.6(1)	C(6)–C(7)–C(8)	121.8(1)
C(1)–C(2)–C(4)	85.79(8)	C(6)–C(7)–C(19)	119.5(1)
C(1)–C(2)–C(12)	127.0(1)	C(8)–C(7)–C(19)	118.6(1)
C(1)–C(2)–C(1*)	90.34(8)	C(3)–C(8)–C(7)	115.1(1)
C(4)–C(2)–C(12)	119.6(1)	C(3)–C(8)–C(21)	121.7(1)
C(4)–C(2)–C(1*)	111.4(1)	C(7)–C(8)–C(21)	123.2(1)
C(12)–C(2)–C(1*)	116.48(9)	C(1)–C(9)–C(10)	118.2(1)
C(1)–C(3)–C(4)	94.19(9)	C(9)–C(10)–C(11)	113.4(2)
C(1)–C(3)–C(8)	142.7(1)	C(2)–C(12)–C(13)	117.0(1)
C(4)–C(3)–C(8)	123.1(1)	C(12)–C(13)–C(14)	114.6(2)
C(2)–C(4)–C(3)	94.6(1)	C(5)–C(15)–C(16)	115.1(2)
C(2)–C(4)–C(5)	142.3(1)	C(6)–C(17)–C(18)	111.7(2)
C(3)–C(4)–C(5)	123.1(1)	C(7)–C(19)–C(20)	112.9(2)
C(4)–C(5)–C(6)	115.3(1)	C(8)–C(21)–C(22)	115.3(1)

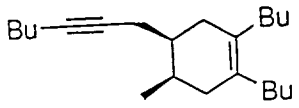
6a






Ph
+
Ph
11 : 1
6b





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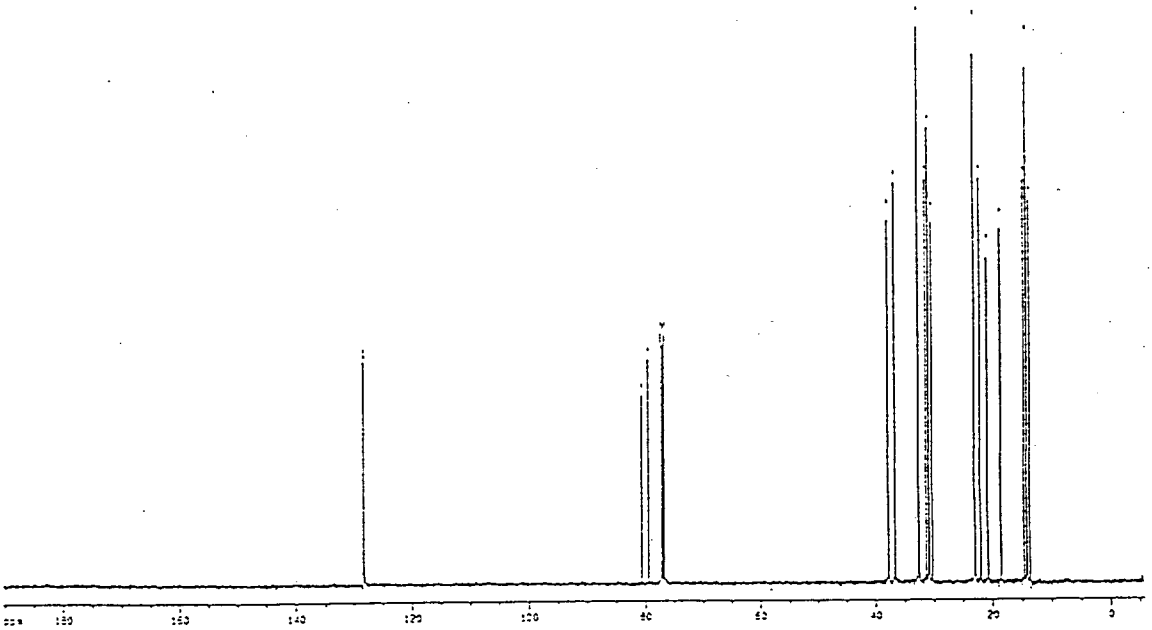
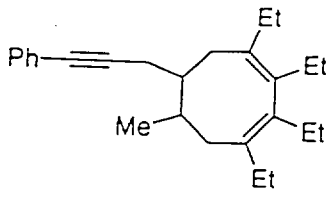
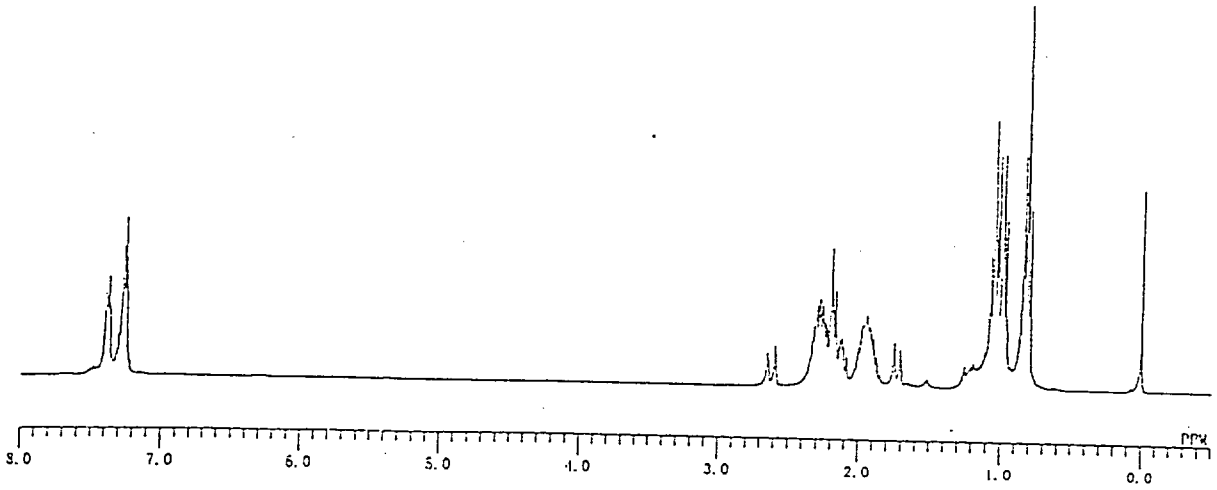



Et
Ph
Et
Me
Et
Et

6d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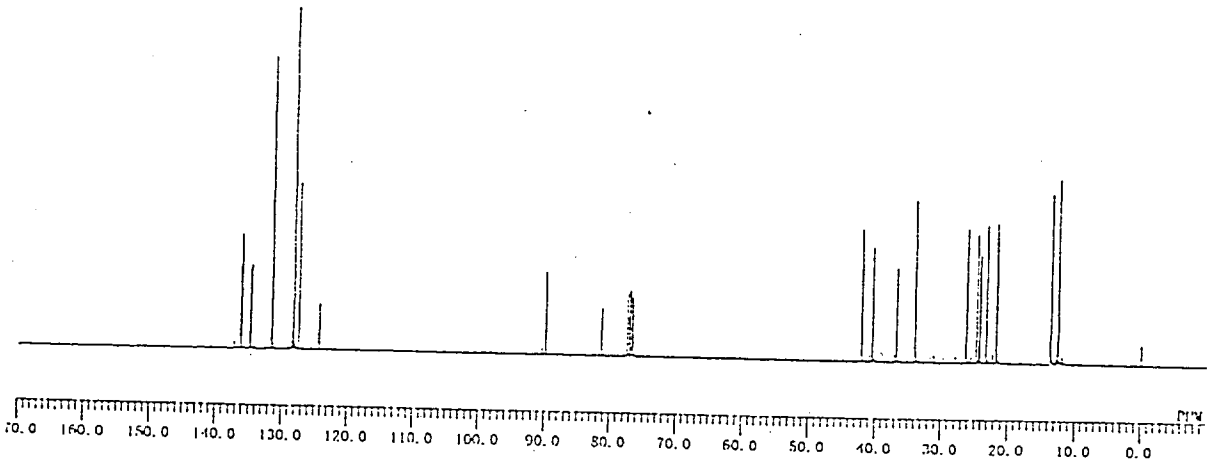
PPM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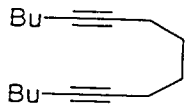

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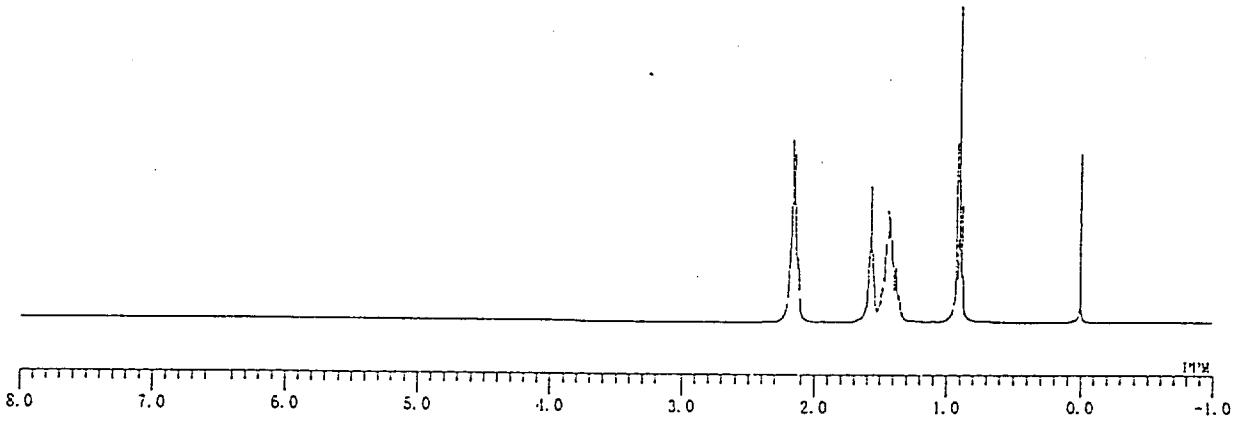

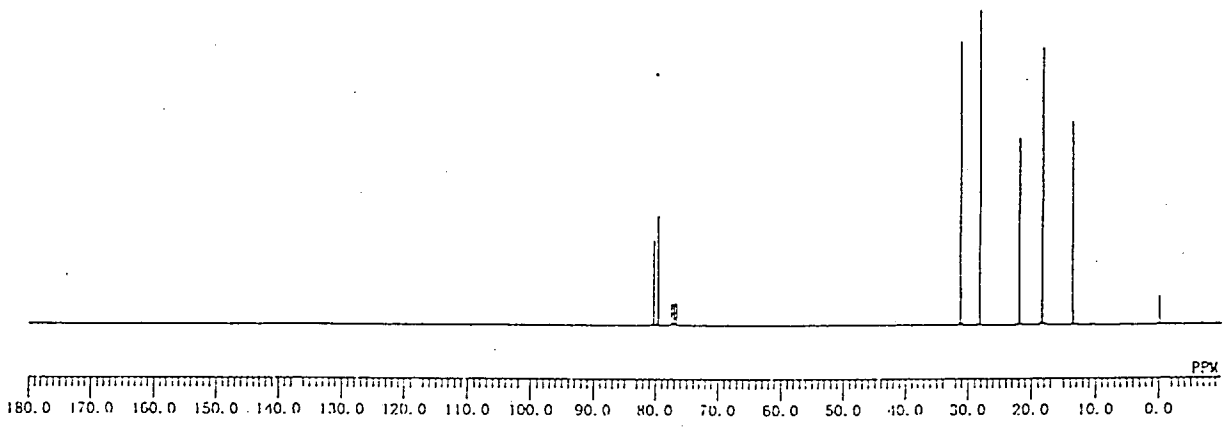

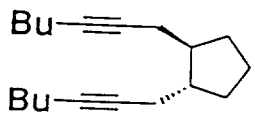

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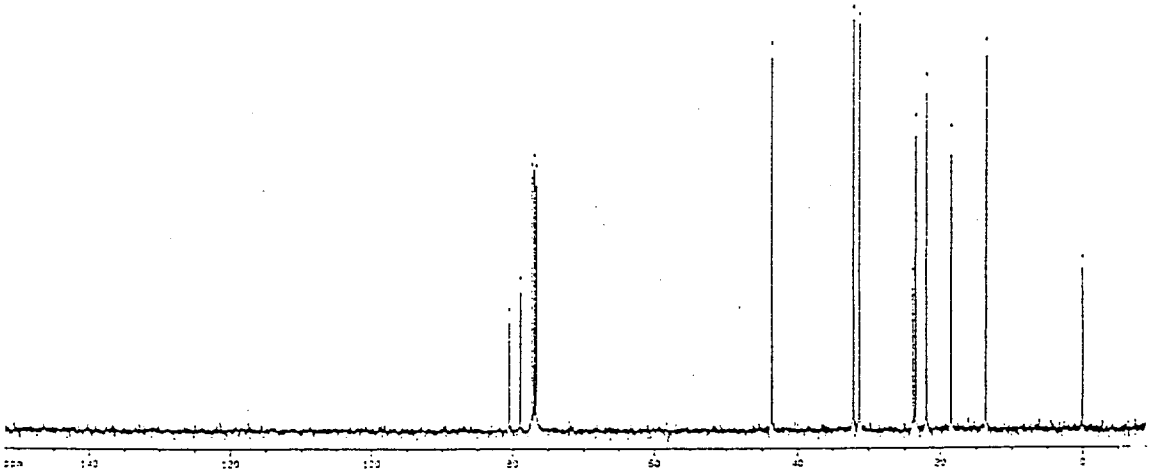

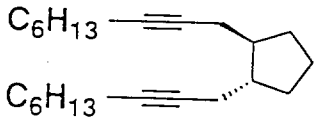

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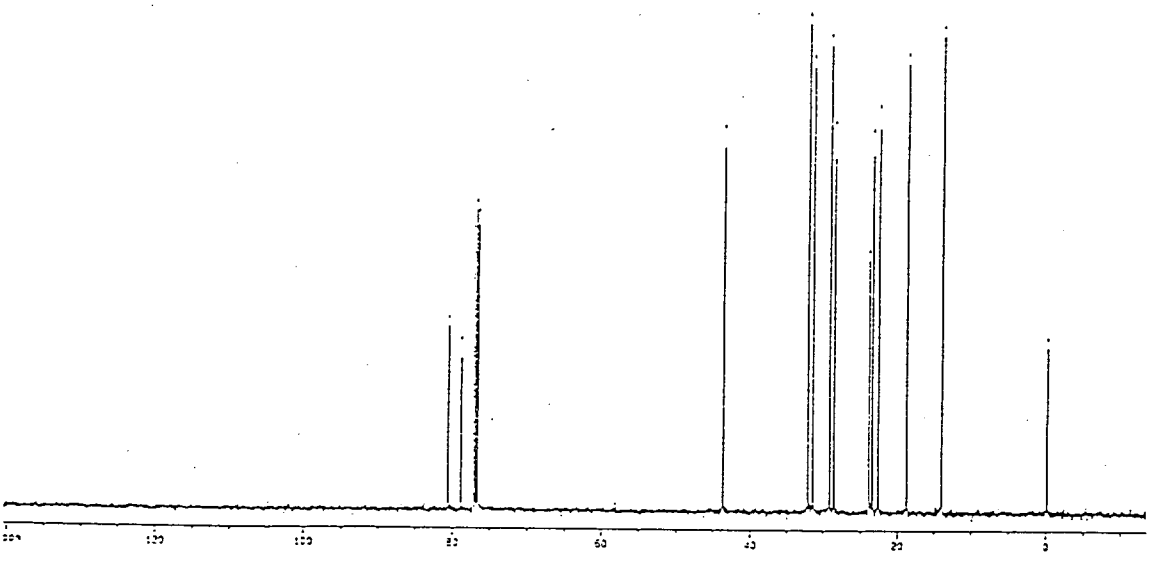
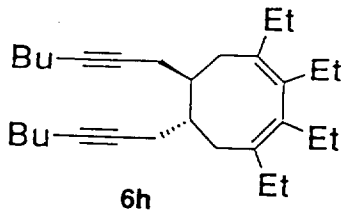
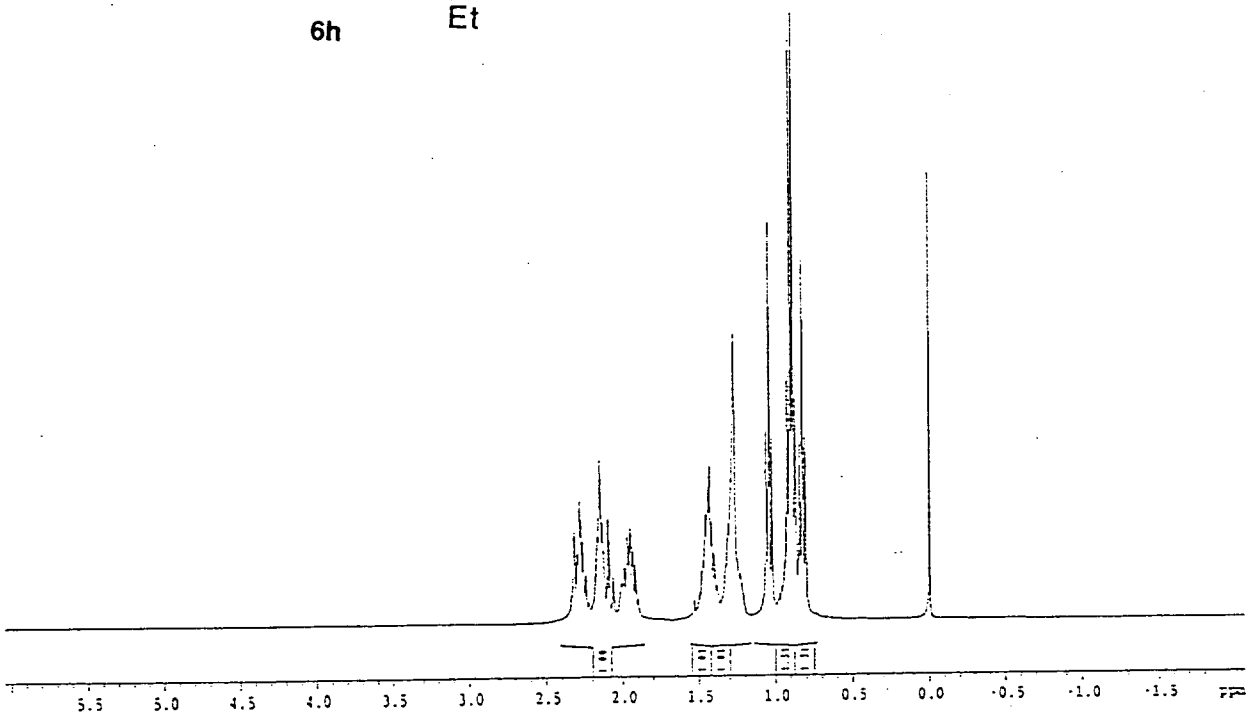



Et
Bu
Et
Bu
Et
Et

6h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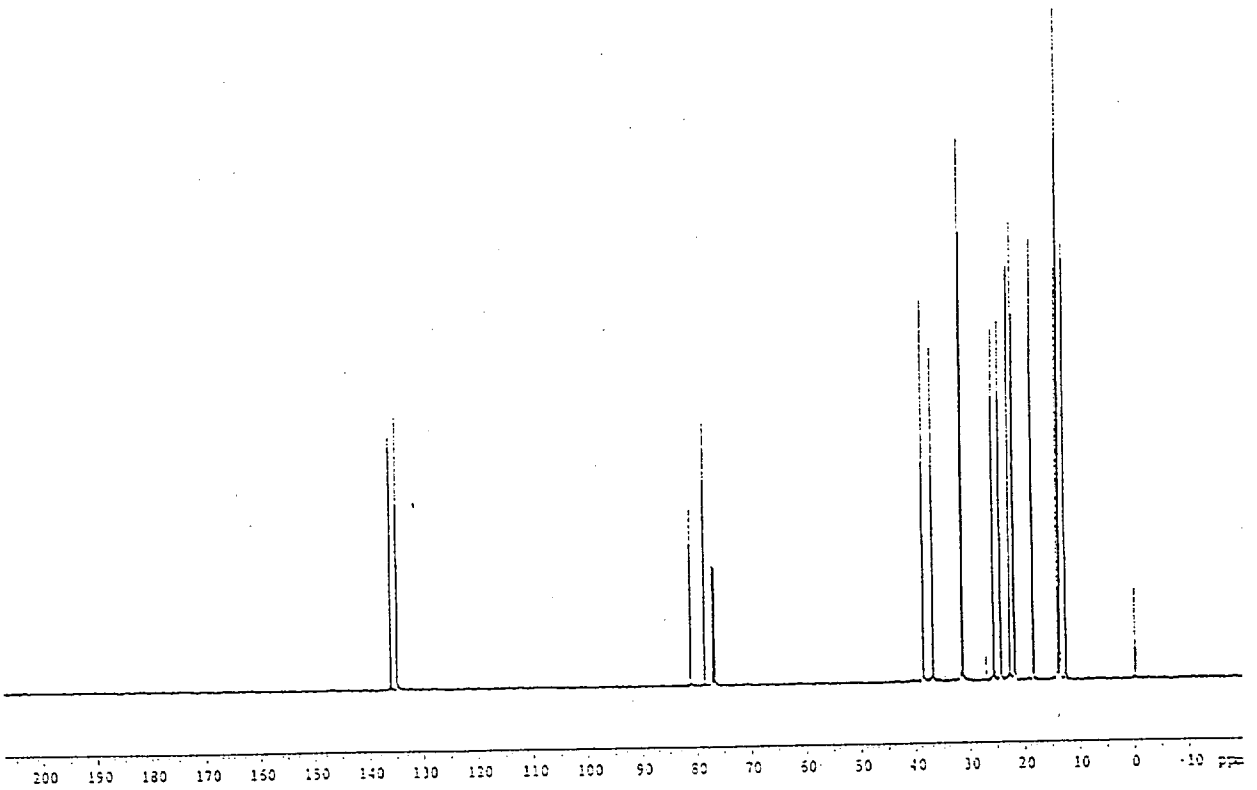
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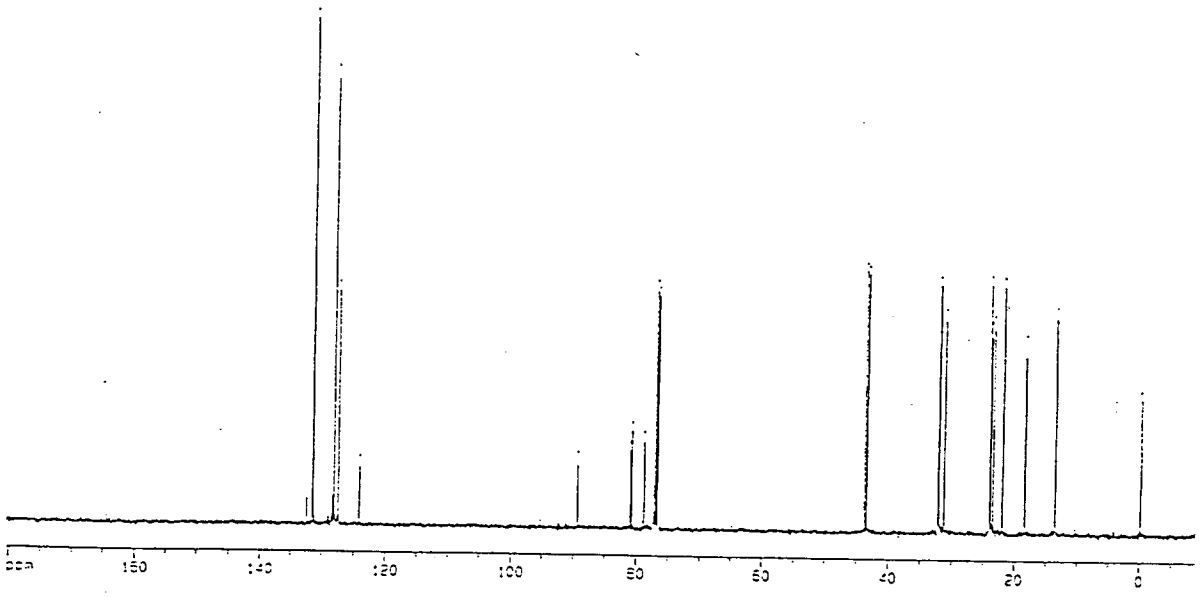
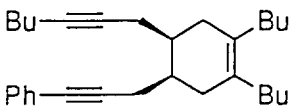
PPM

6i





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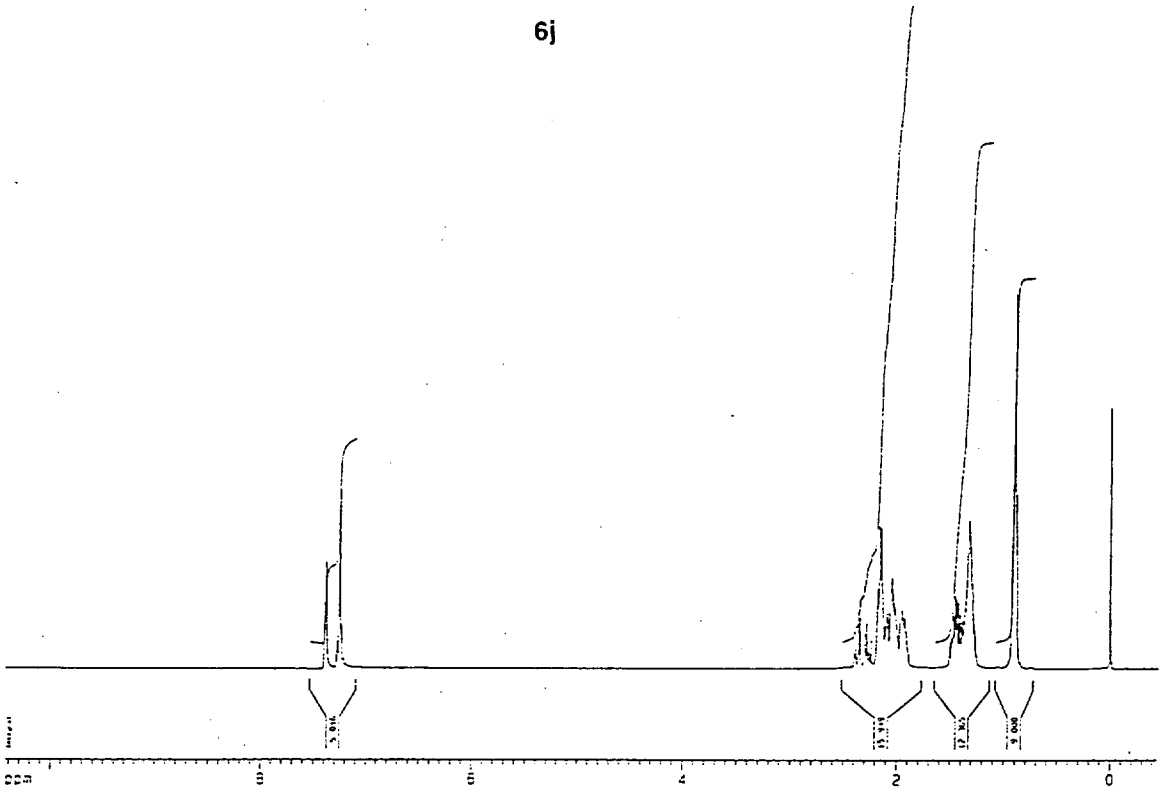

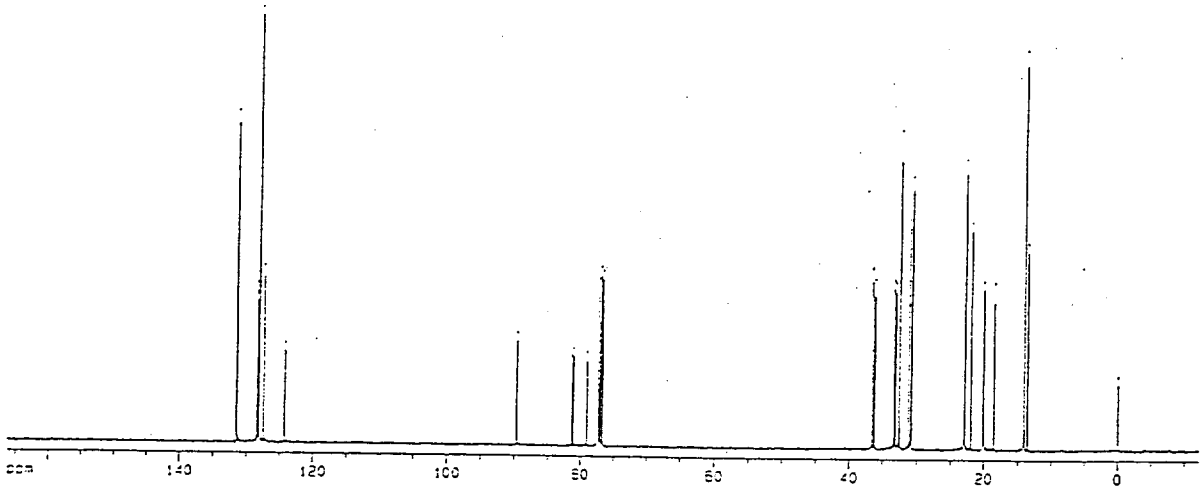

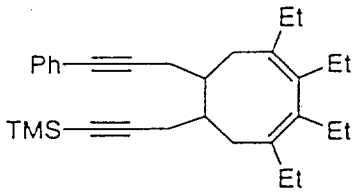

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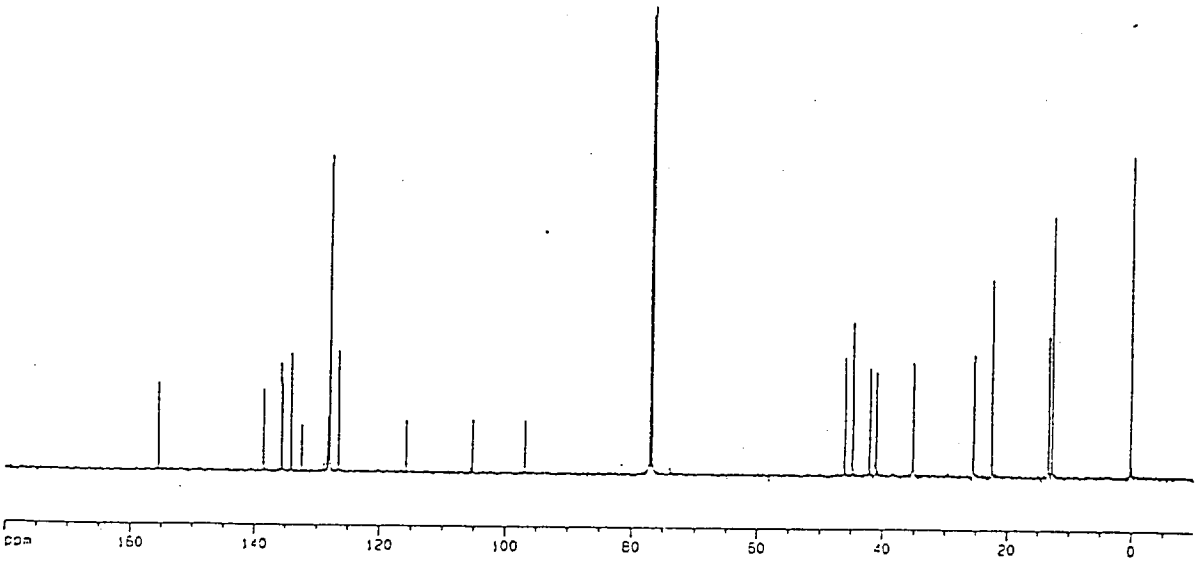

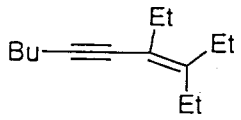

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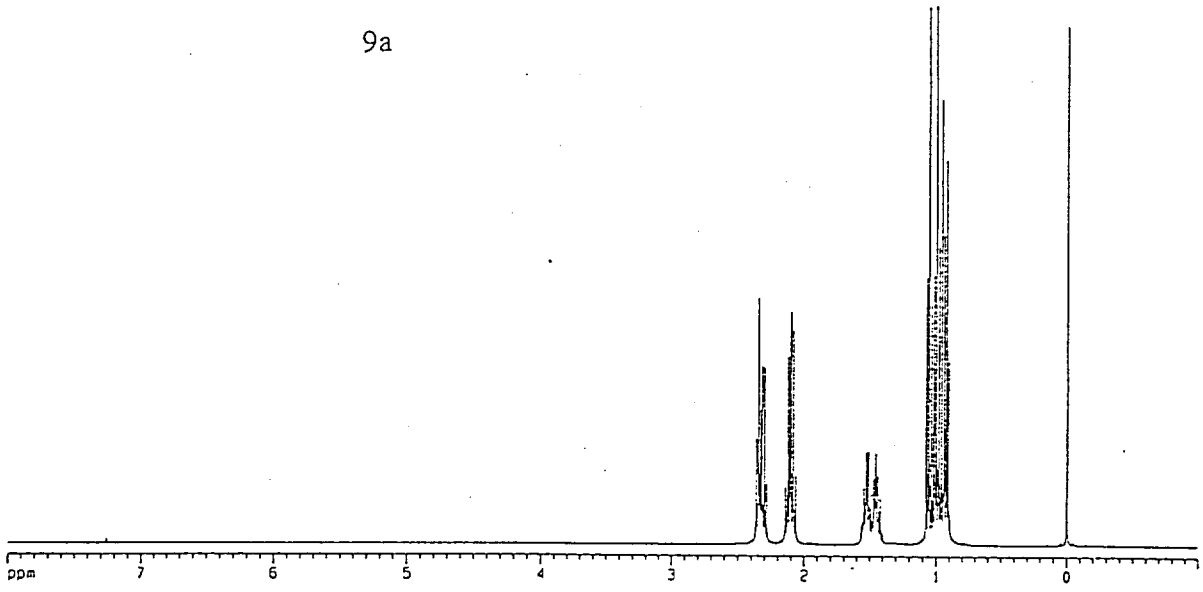

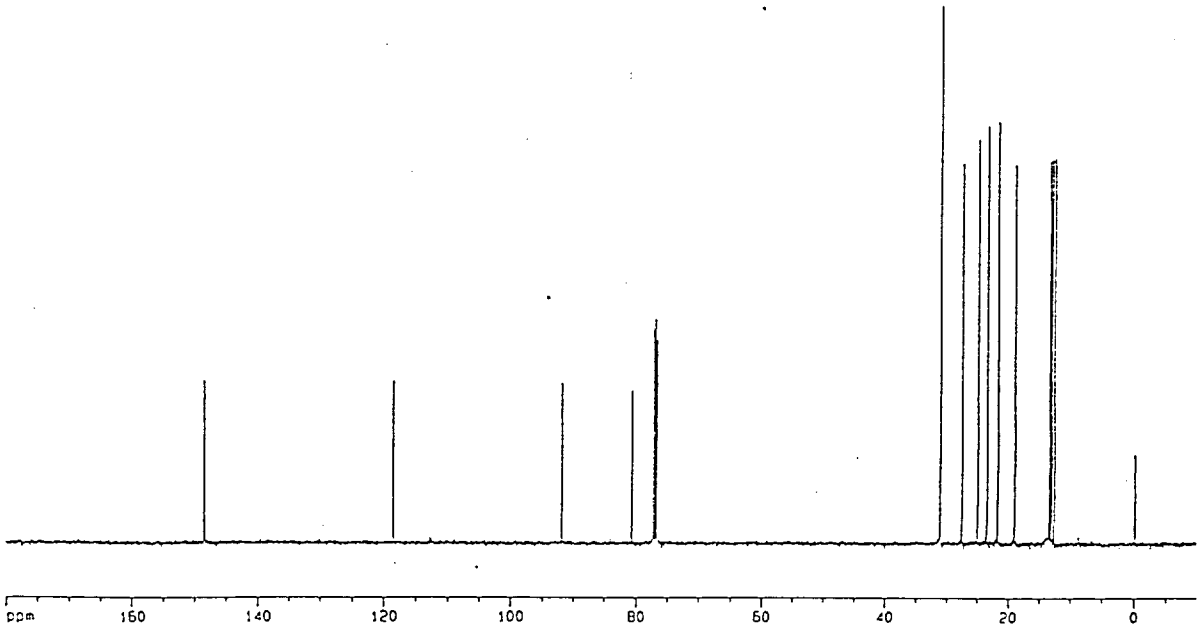

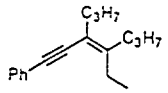

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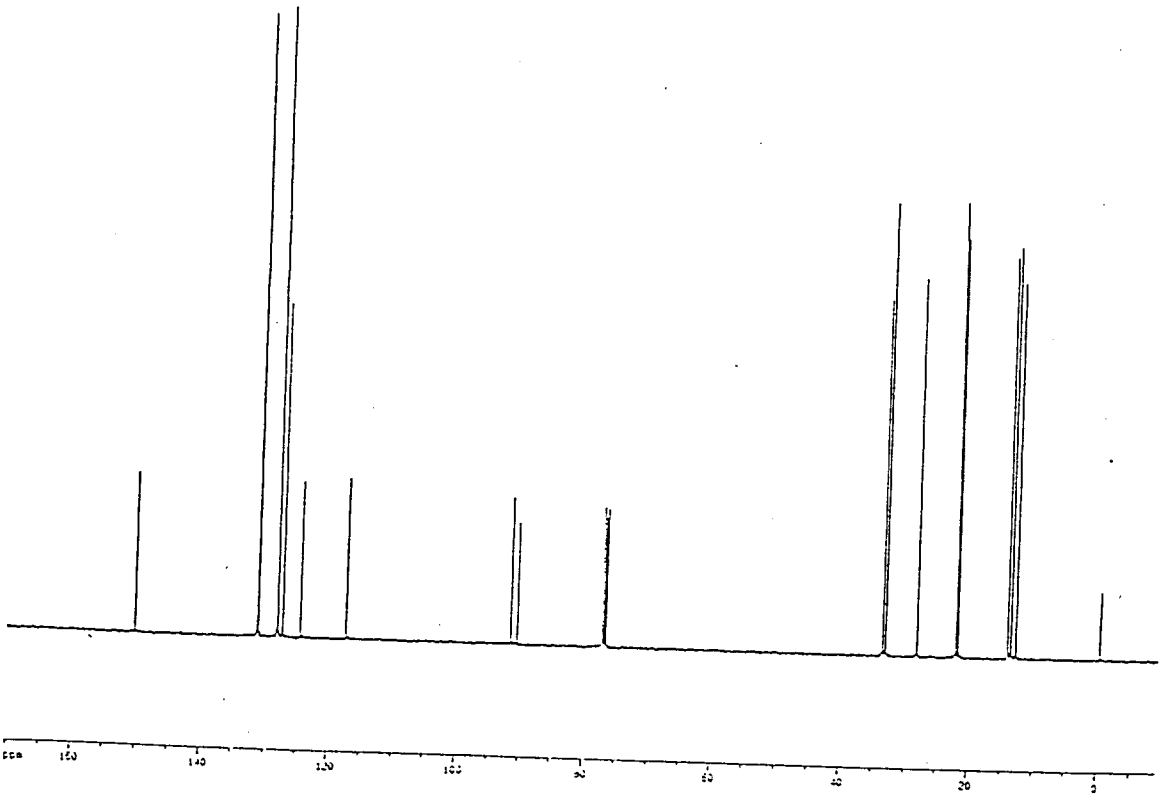

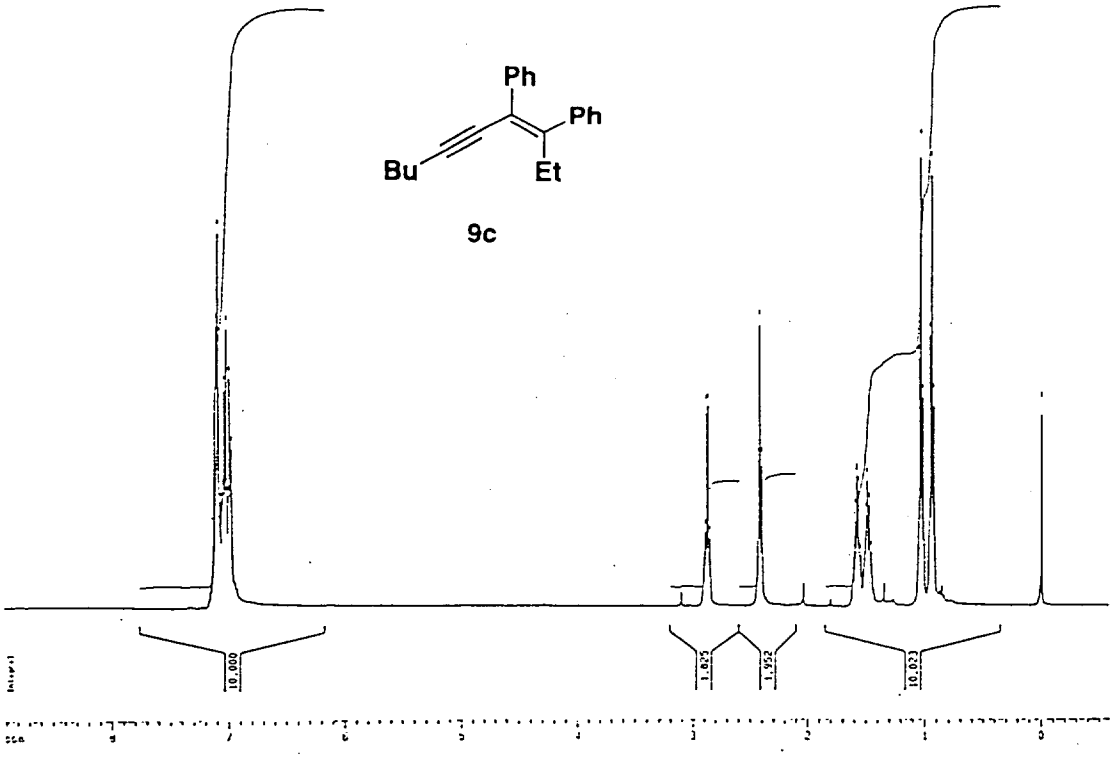
Ph
Ph
Bu
Et
9c
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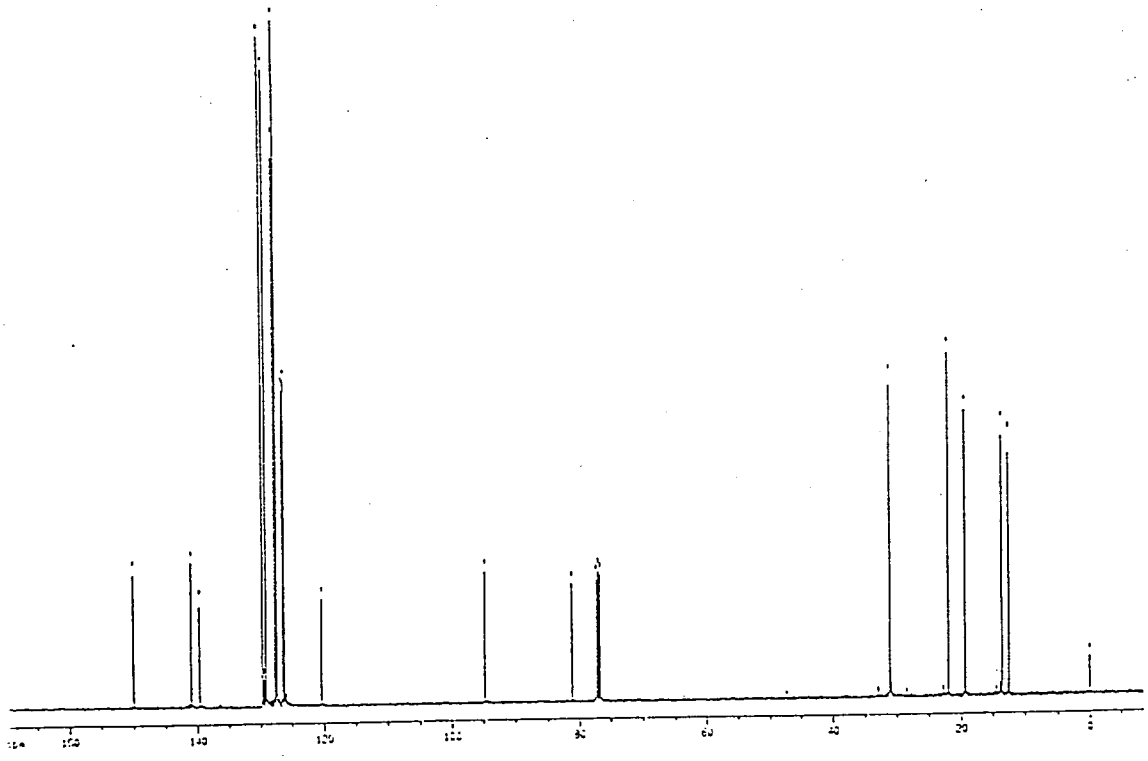
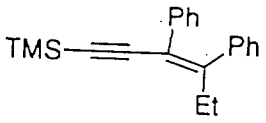
10.023





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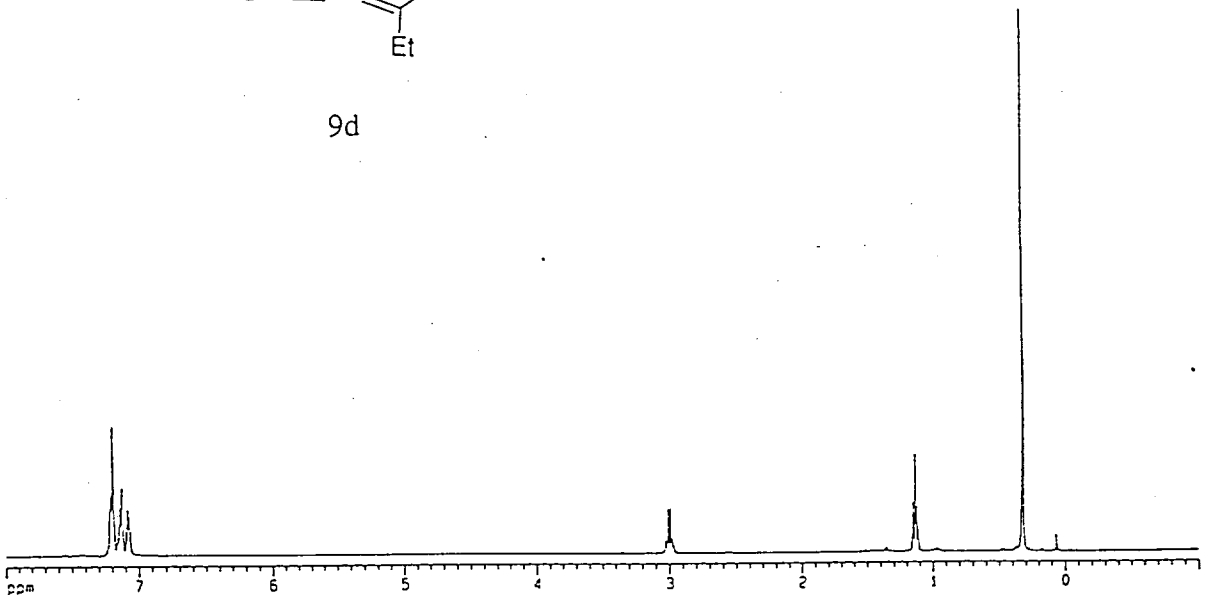
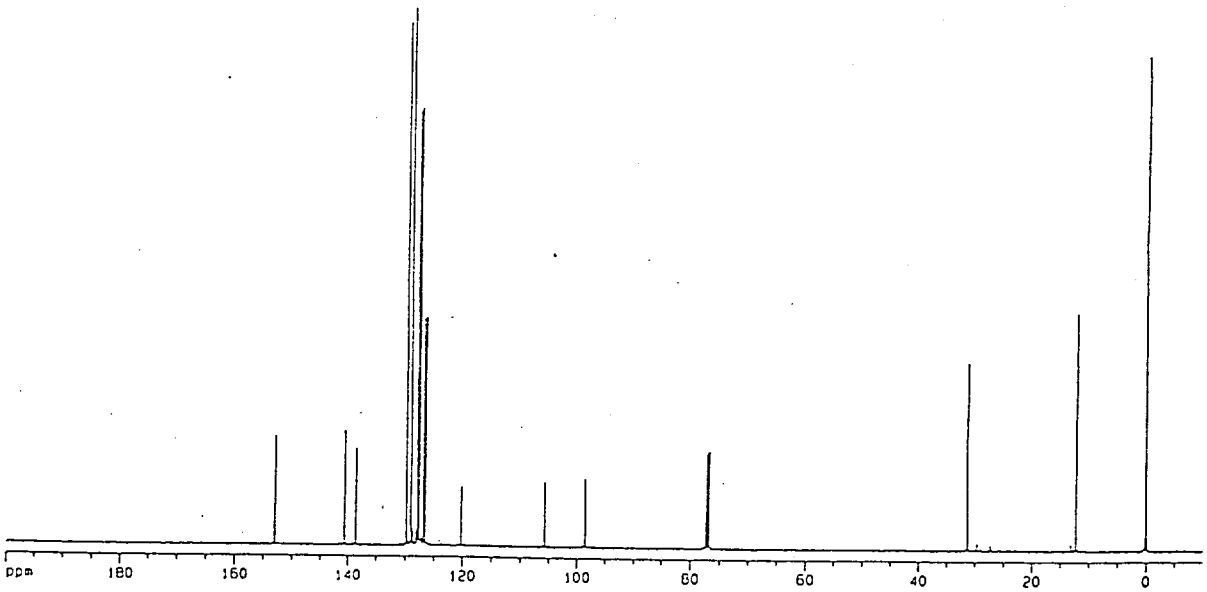
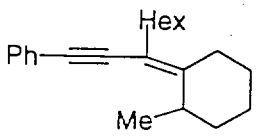
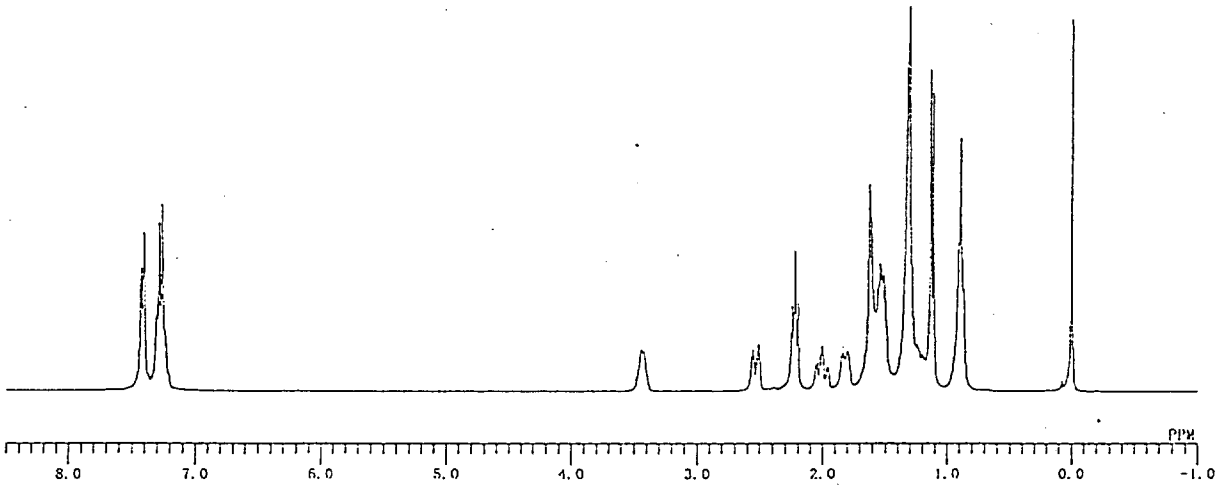





Hex
Ph
Me

9e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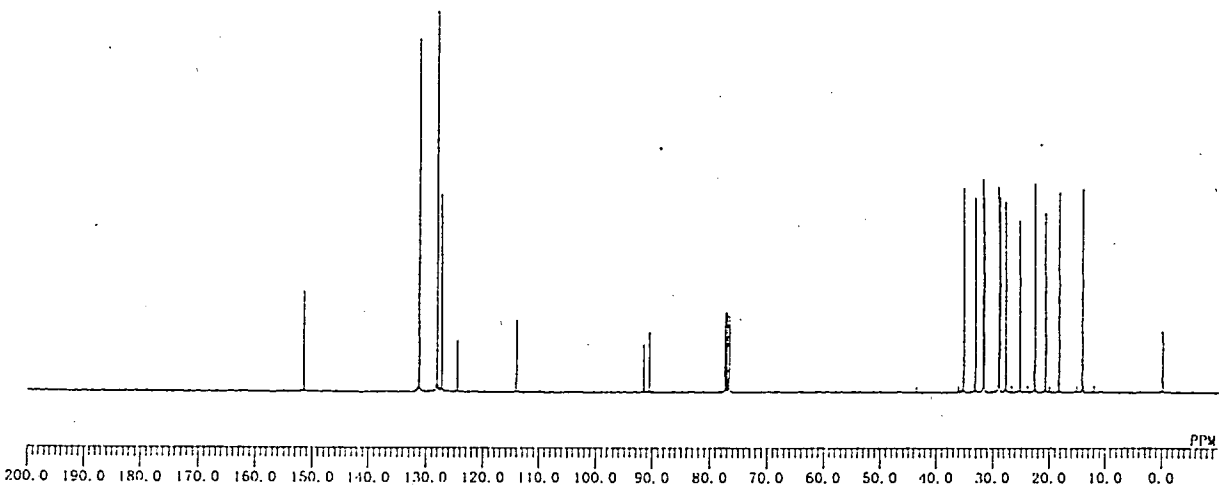
PPM
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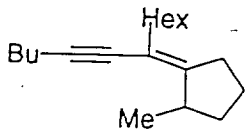

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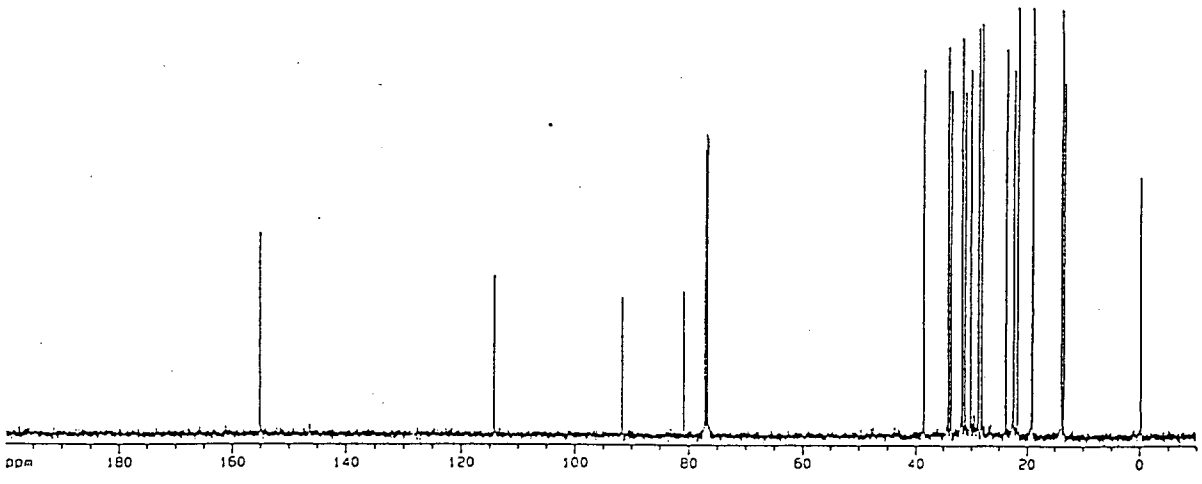
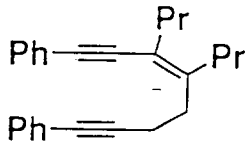
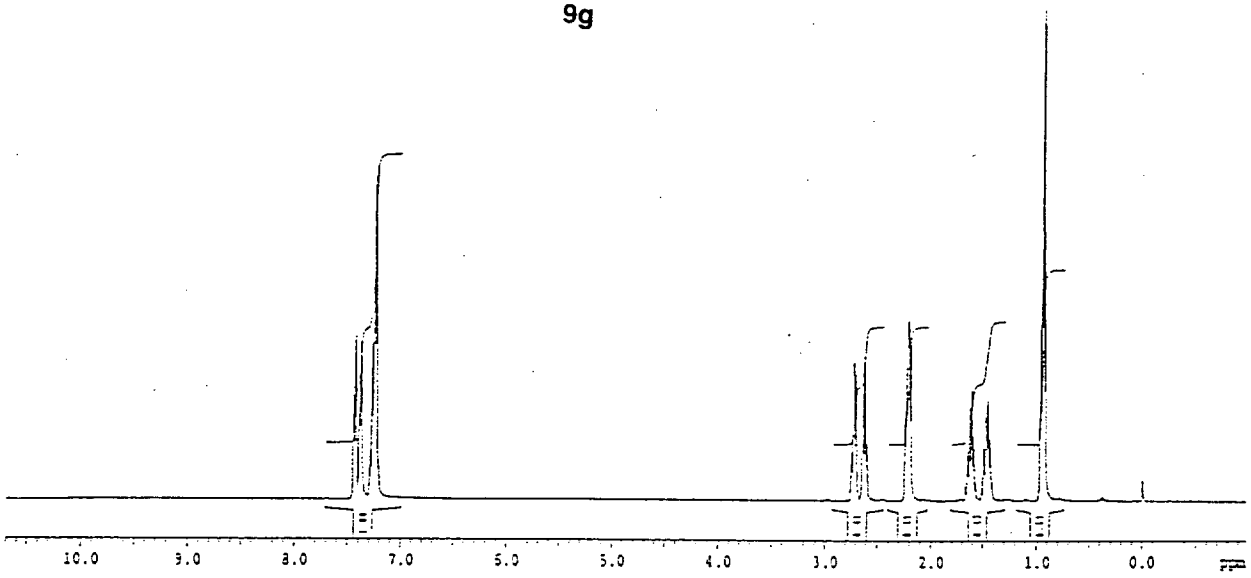



Pr
Ph
Pr
Ph

9g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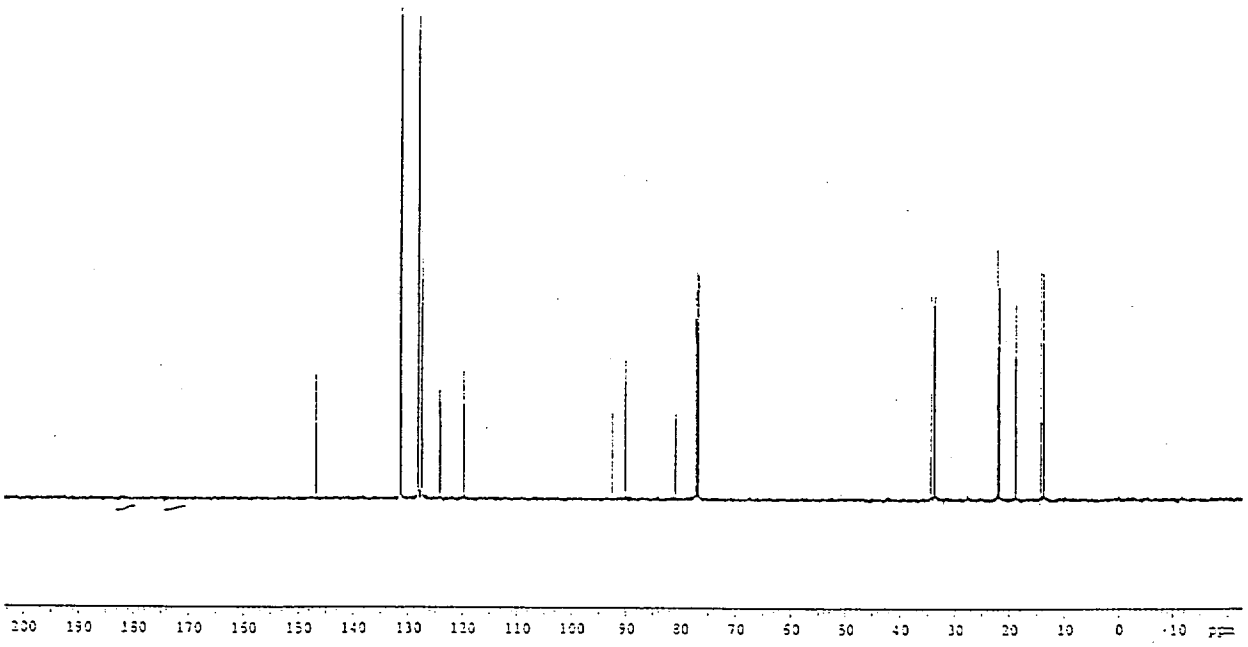
ppm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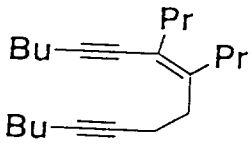
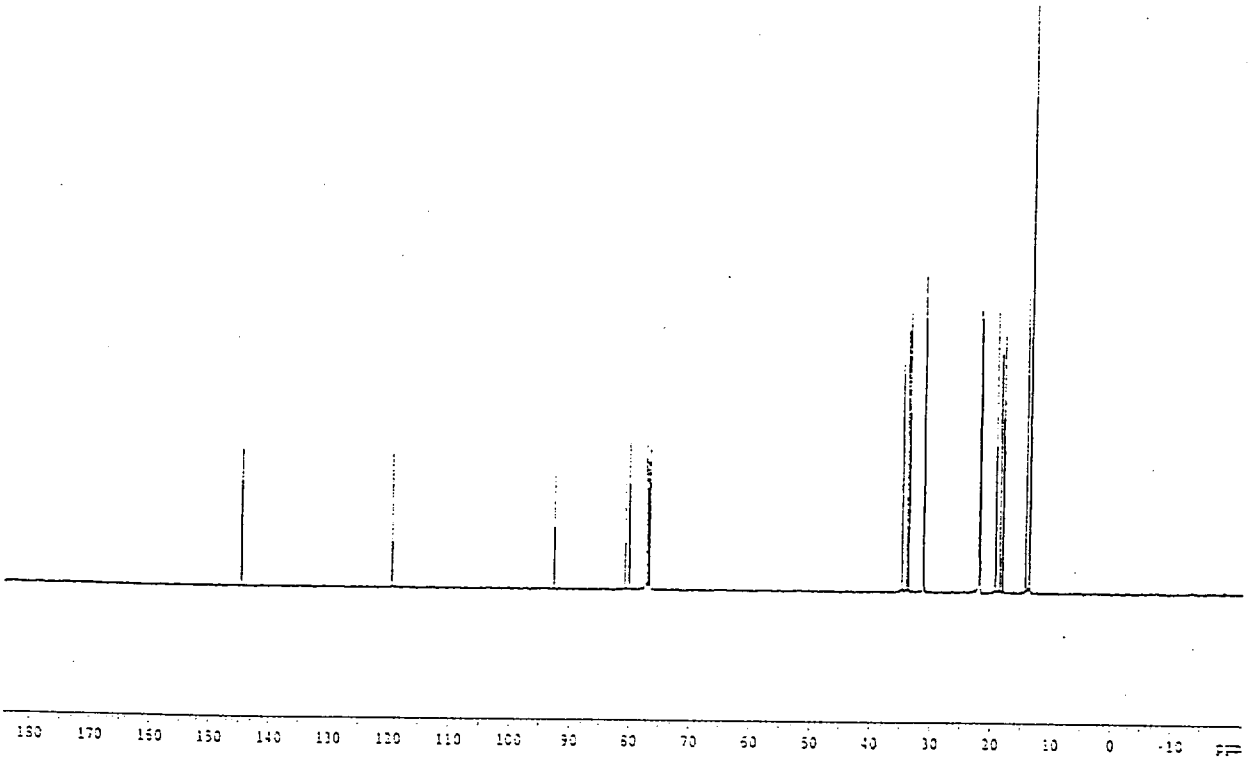
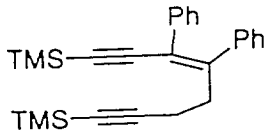
-10
ppm


Pr
Bu
Pr
Bu

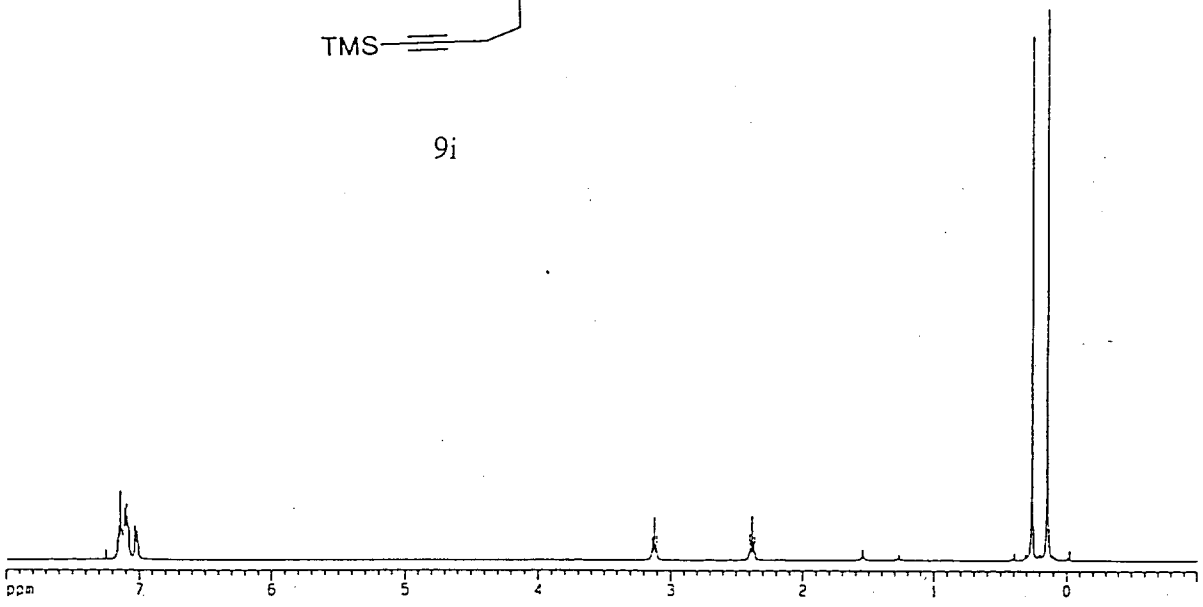
9h

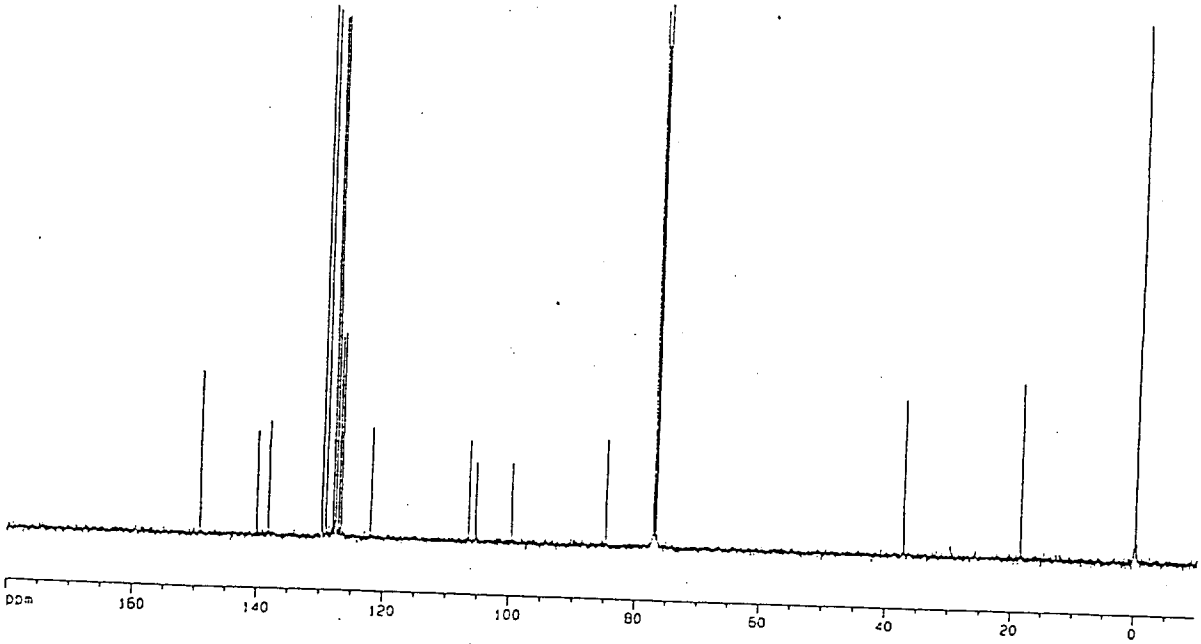
4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2 0.0 -0.2 ppm

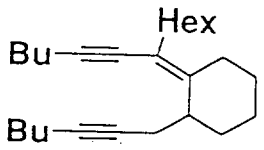

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm



9i

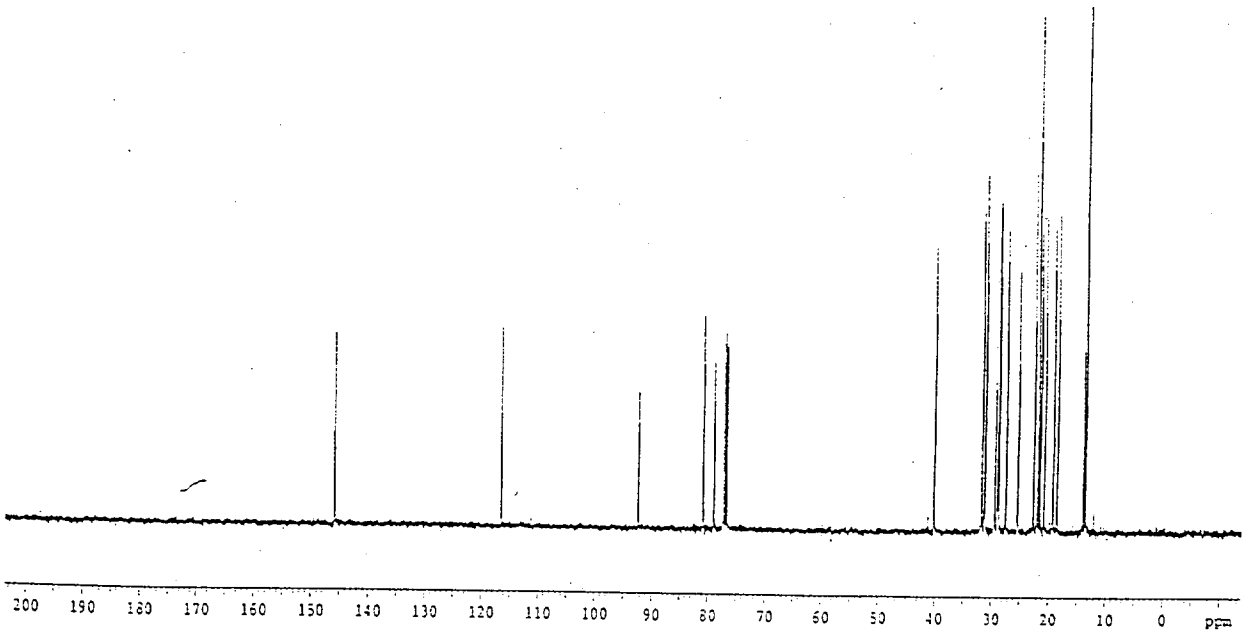


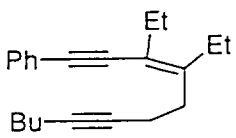



Hex
Bu
Bu

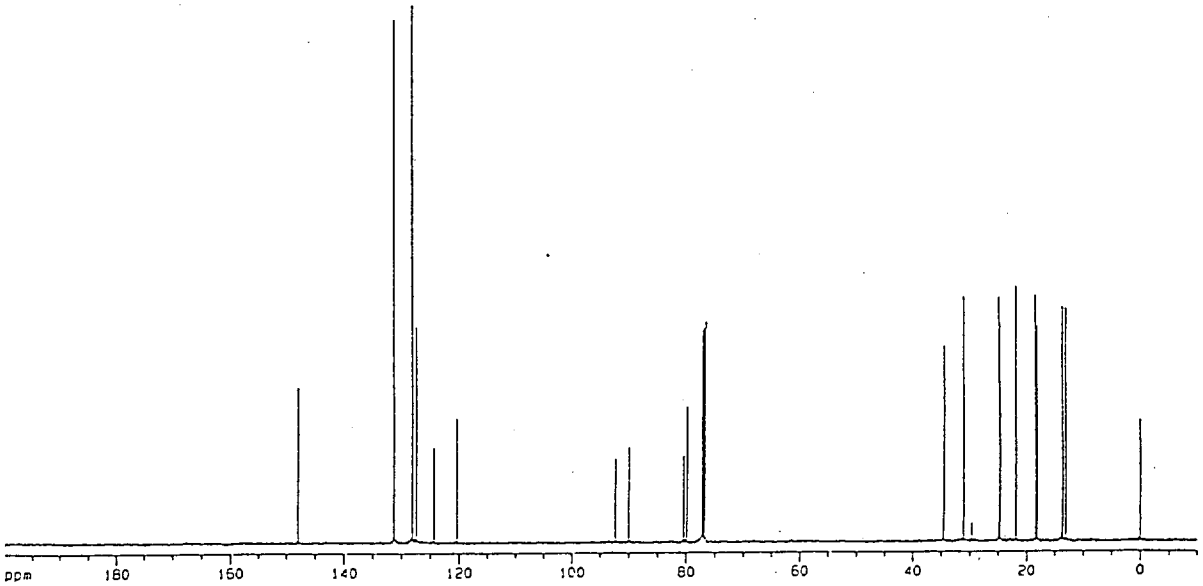
9j

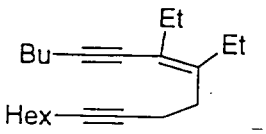
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 ppm


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

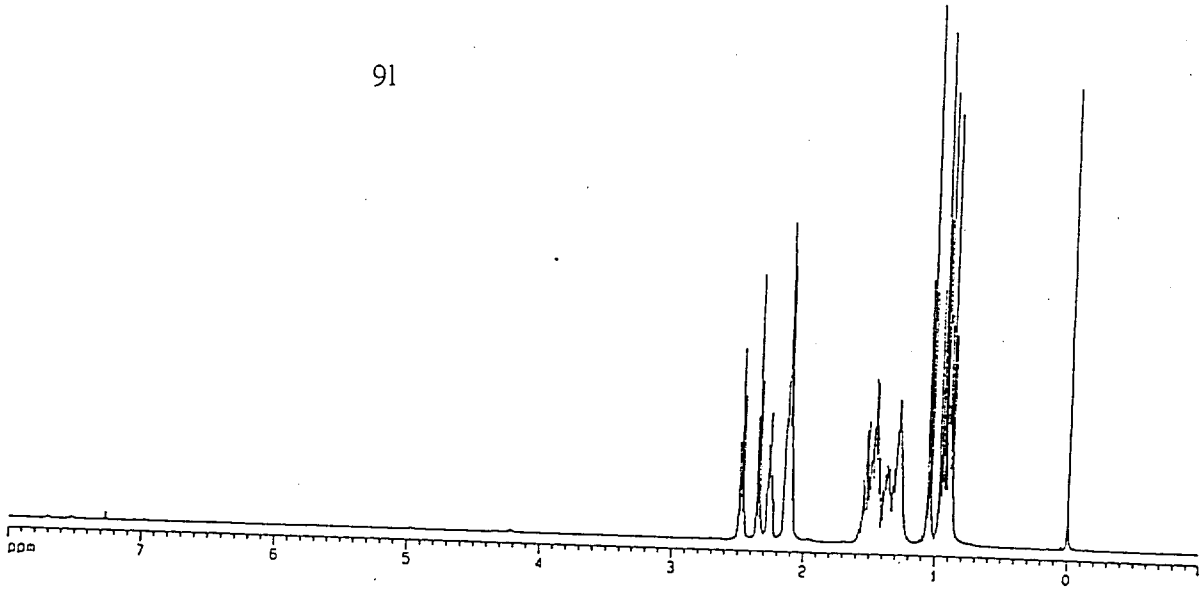


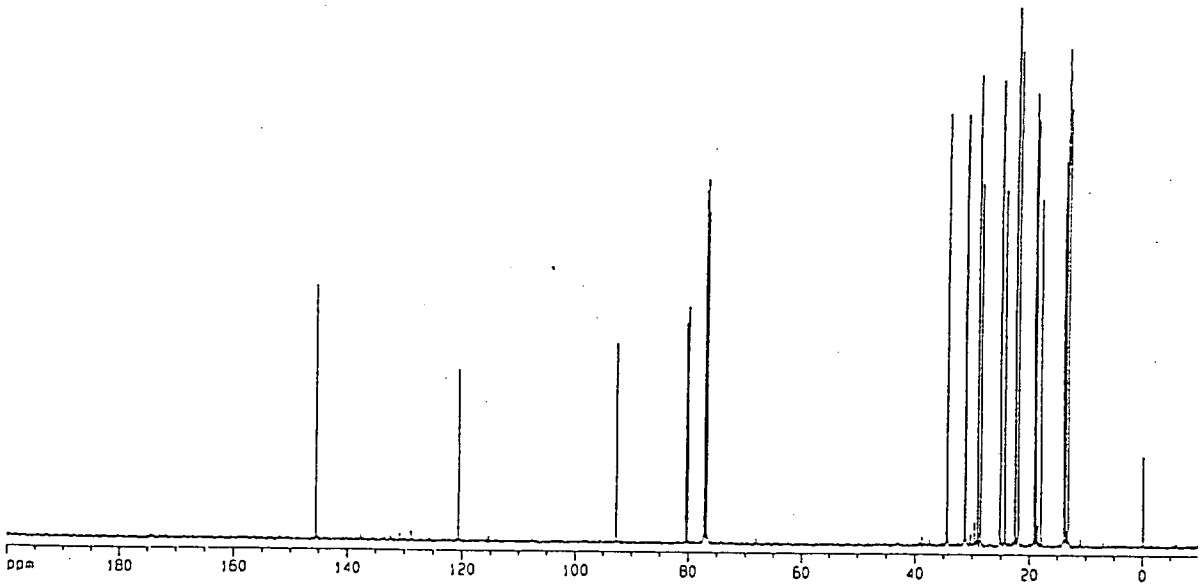
9k

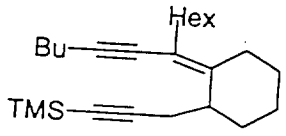




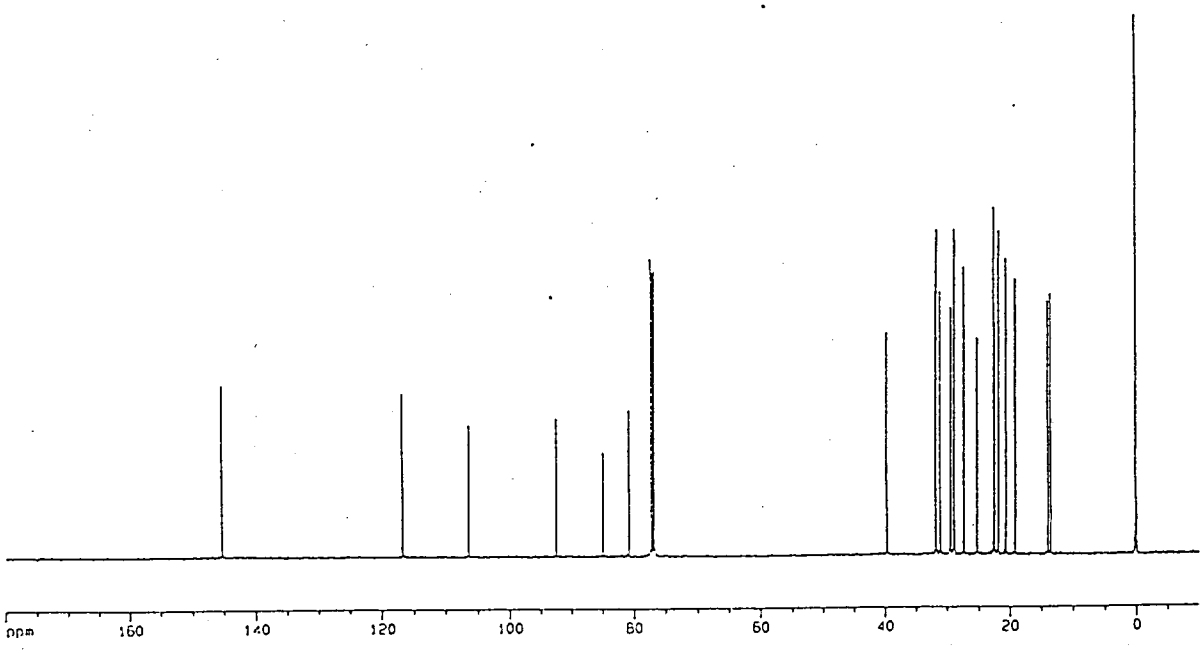
91

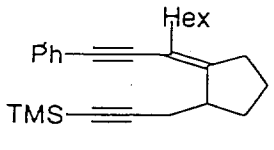




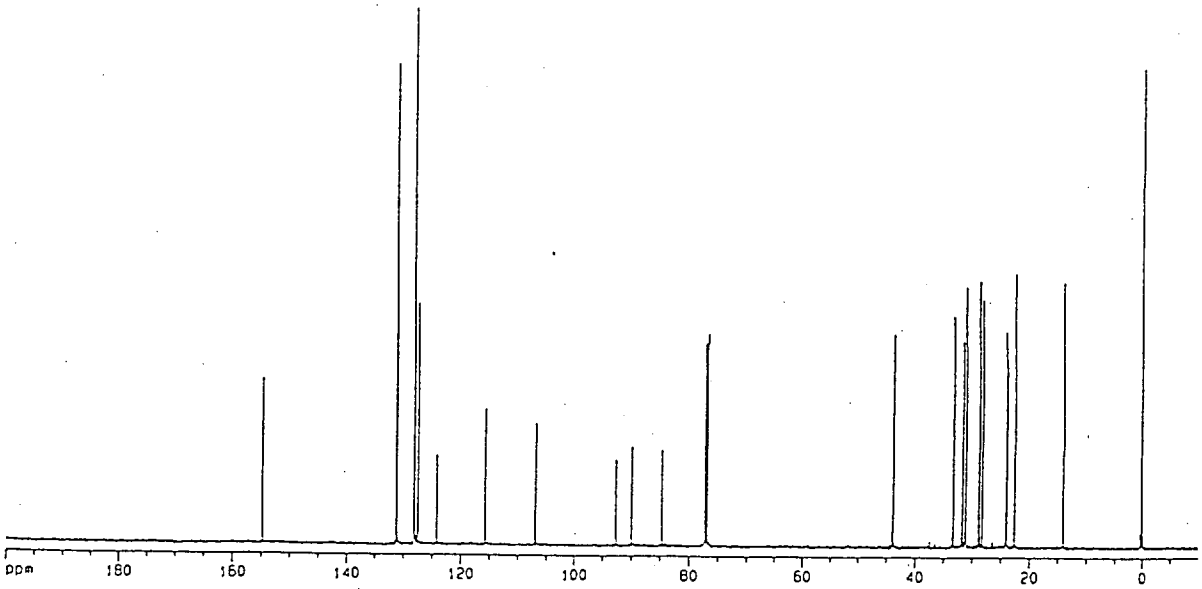


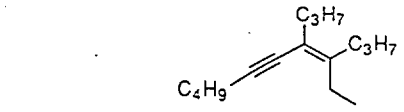
9m





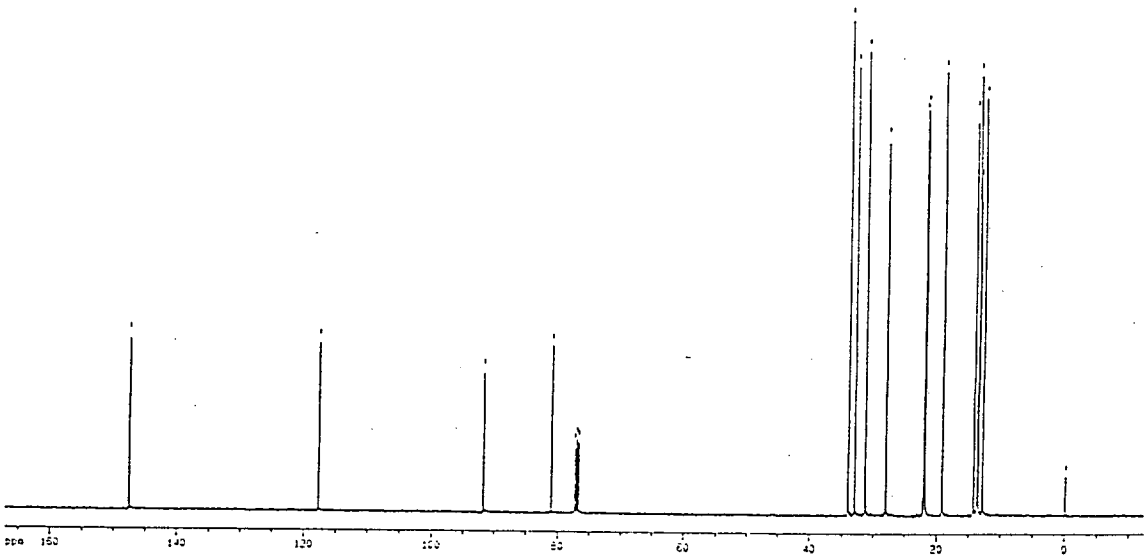
9n

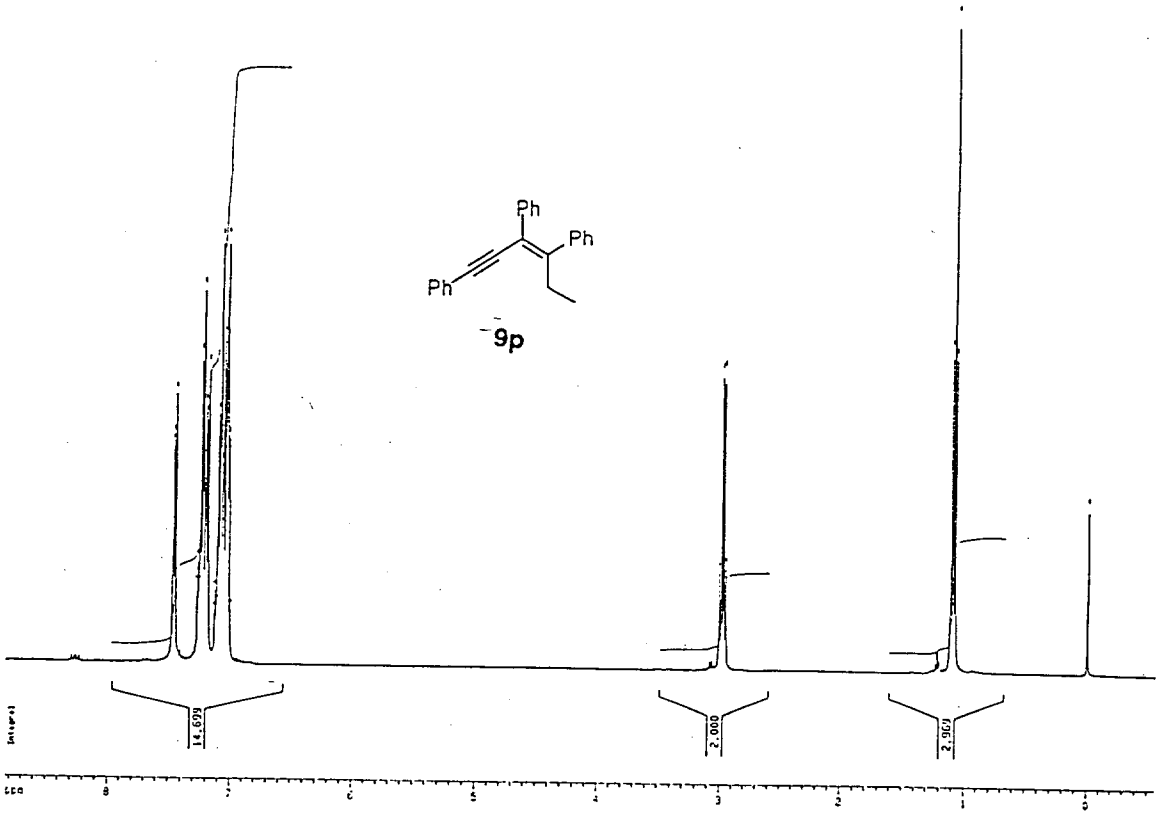



C_3H_7
C_3H_7
C_4H_9

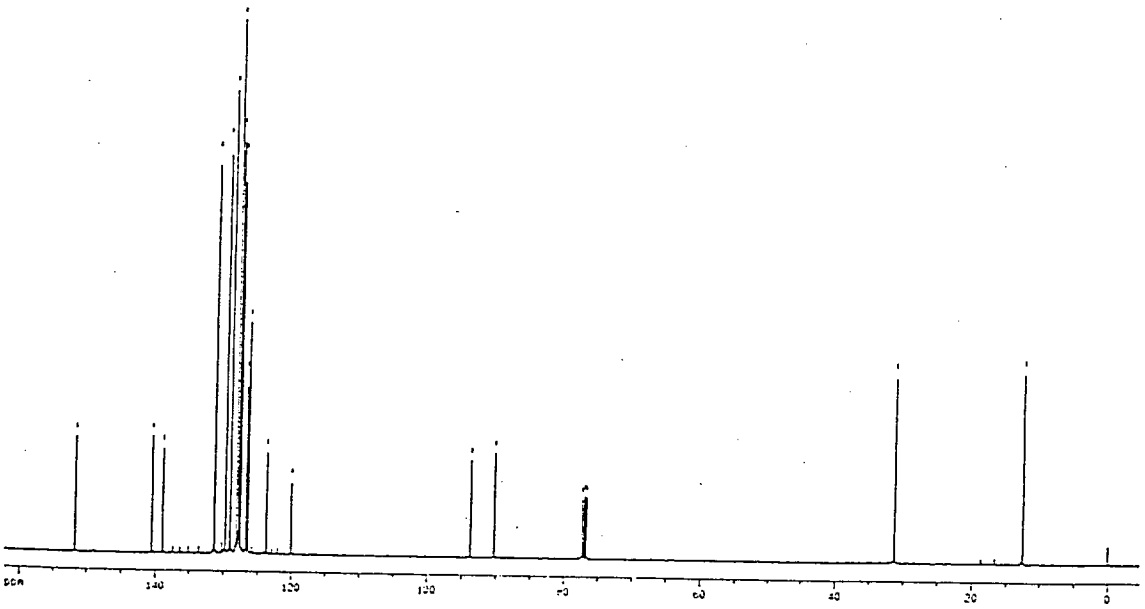
9o

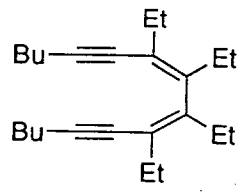
8.000
8.072
12.141
ppm
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


ppm
160
140
120
100
80
60
40
20
0

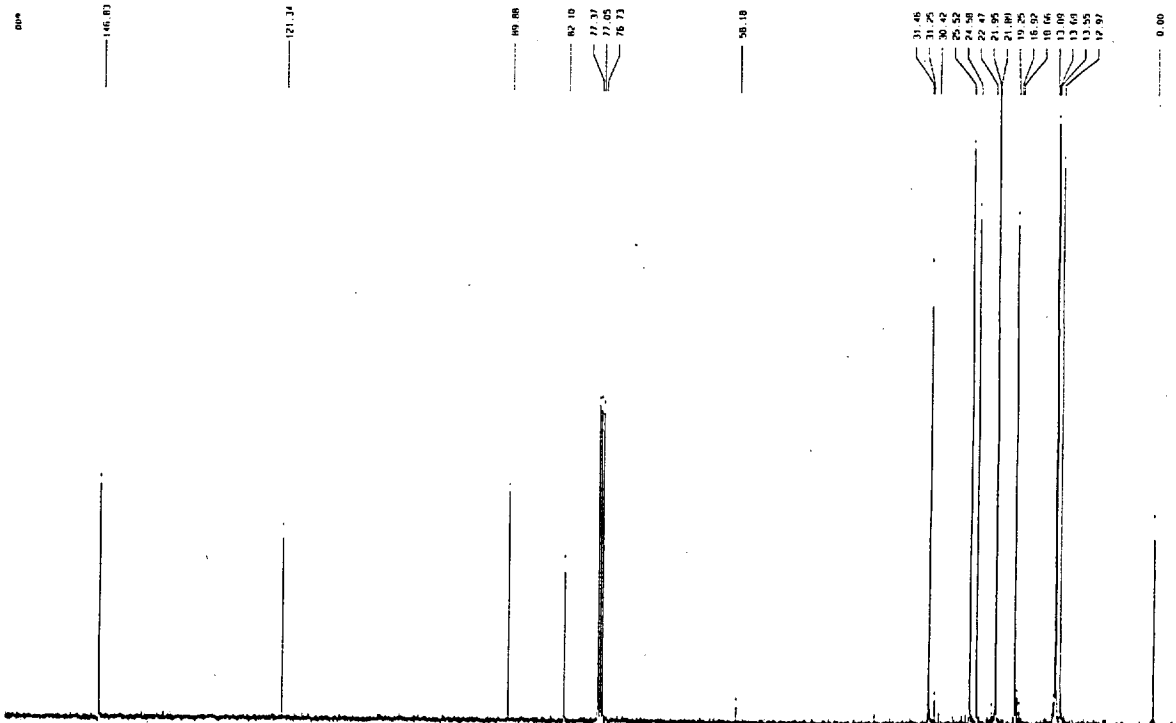


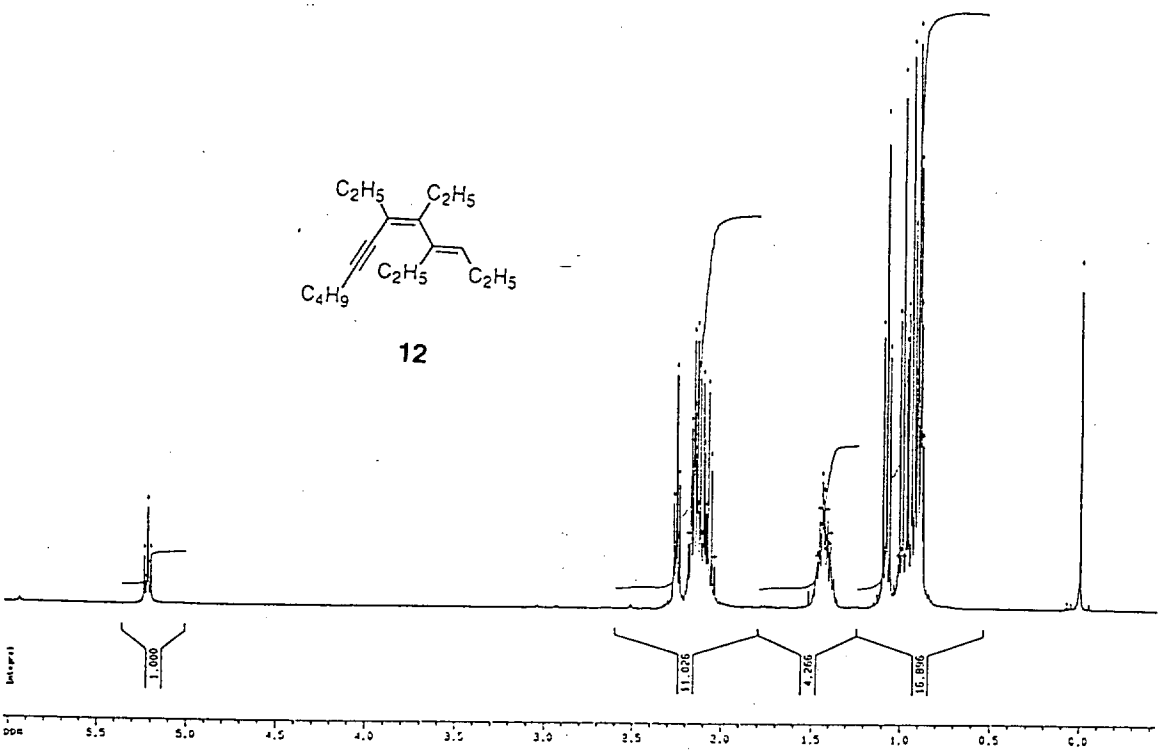
9p

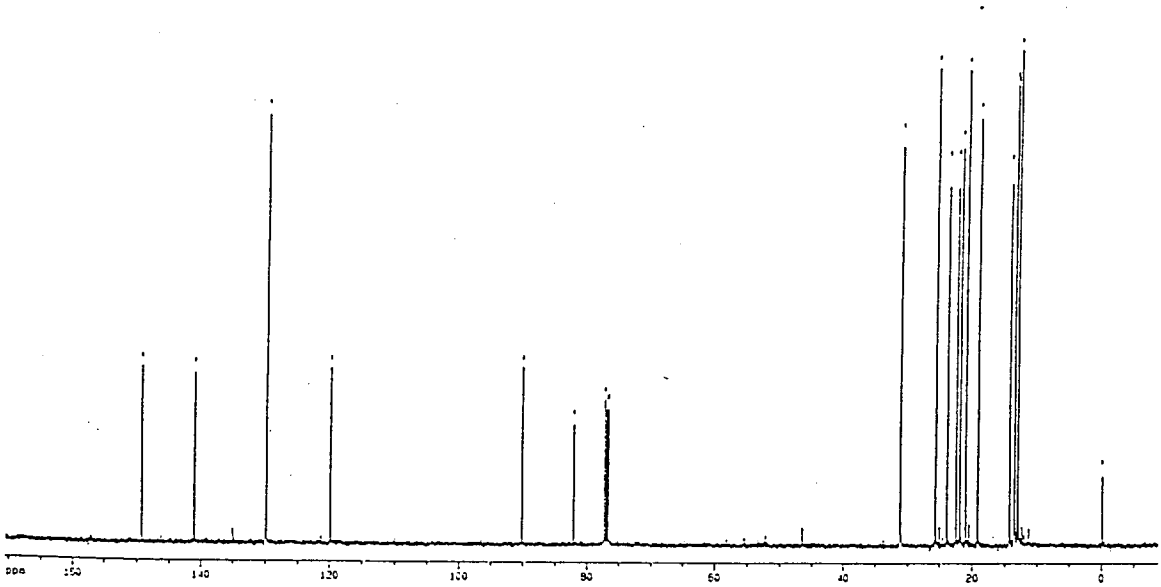




13b




C_2H_5
C_2H_5
C_2H_5
C_2H_5
C_4H_9
12
1.000
11.026
4.266
16.896
ppm
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


ppm
150
140
130
120
110
90
60
40
20
0