^{13}C NMR Spectrum of 21

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BSERVE

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          C13    dpwr            30
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        39296    lb            3.00
      30007.5    wtfile
        16500    proc            ft
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         10.0
        3.000    werr
            0    wexp
         1024    wbs
          684    wnt
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     not used    sp         -1005.9
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            y    vs             107
            n    sc               0
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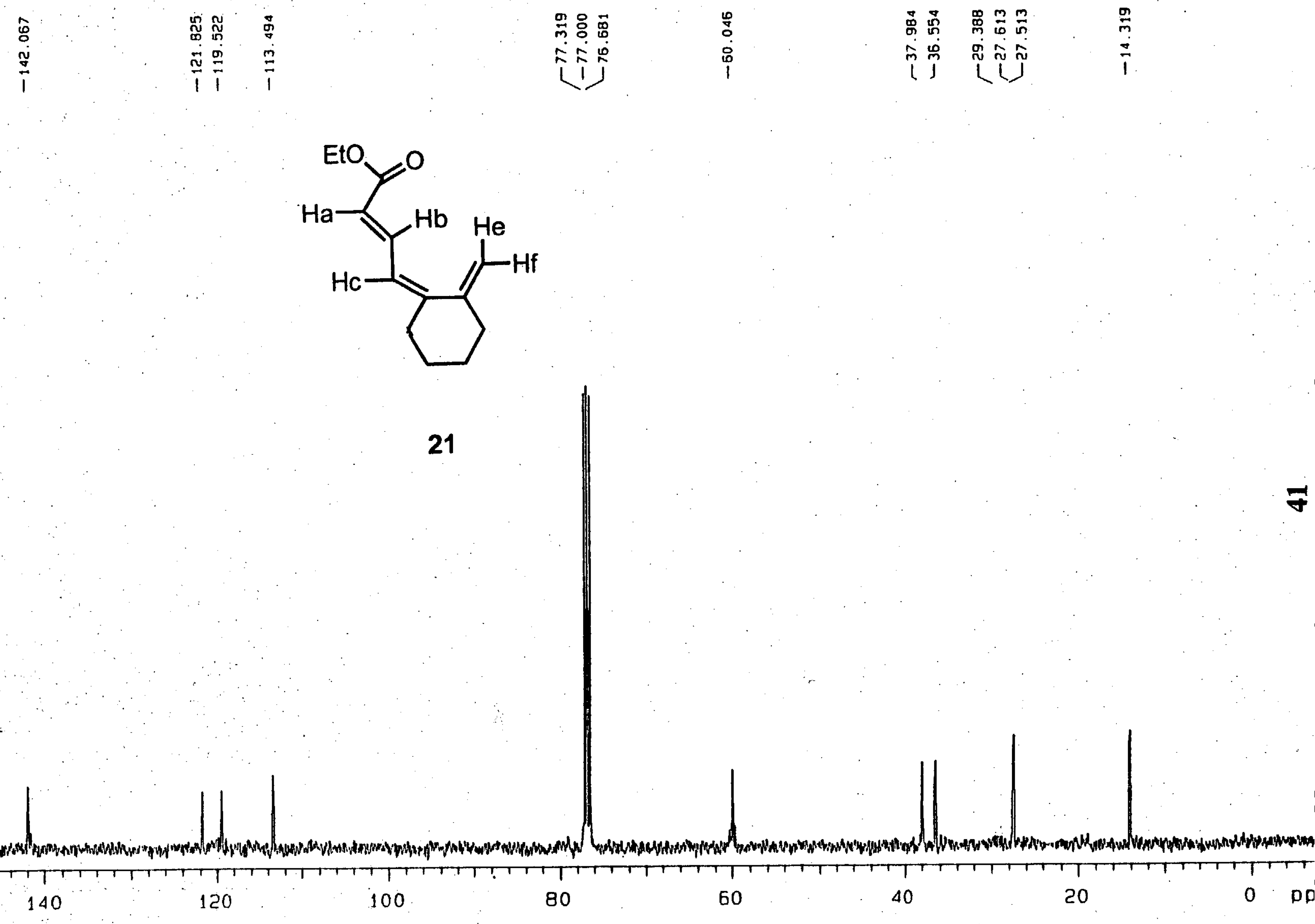
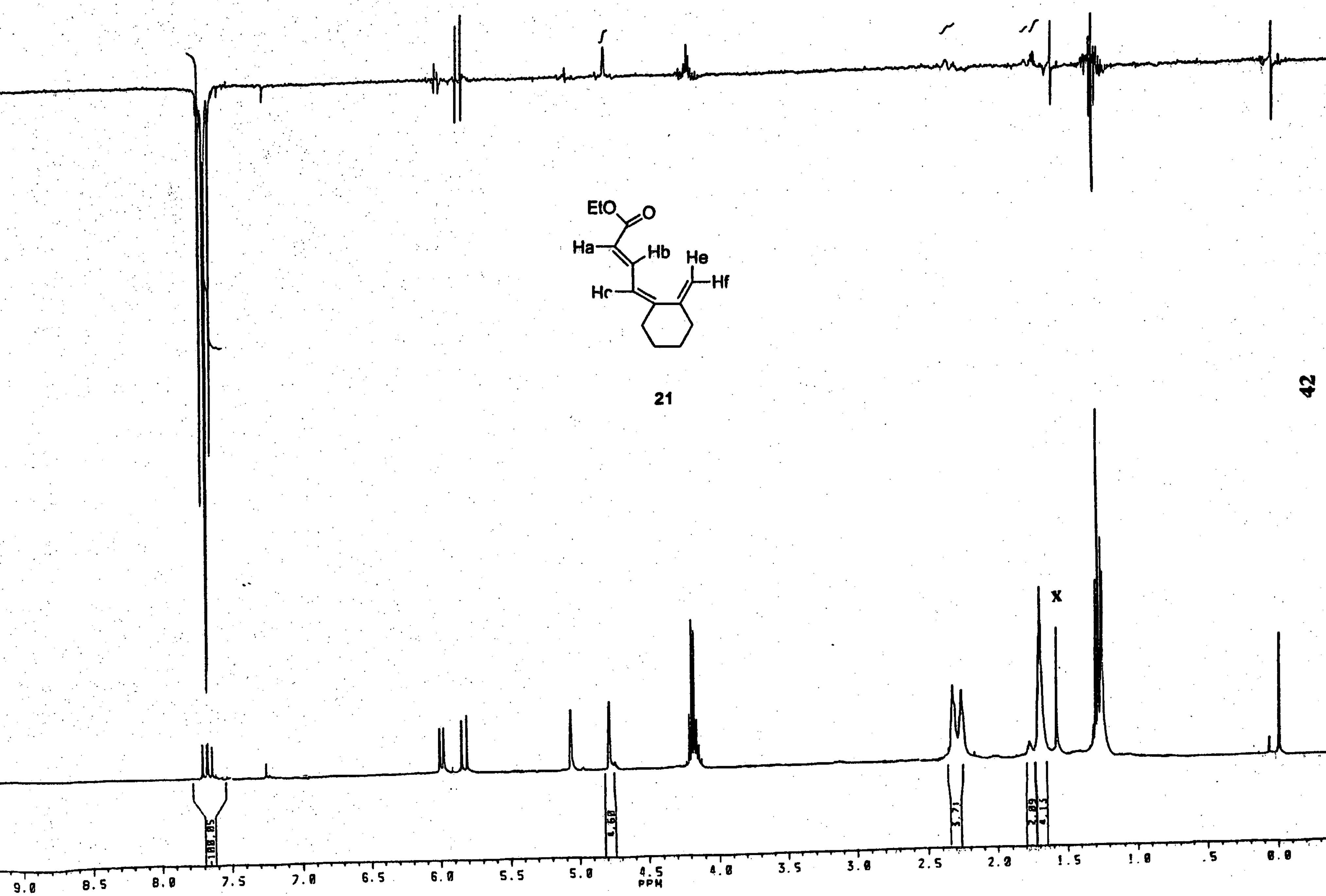


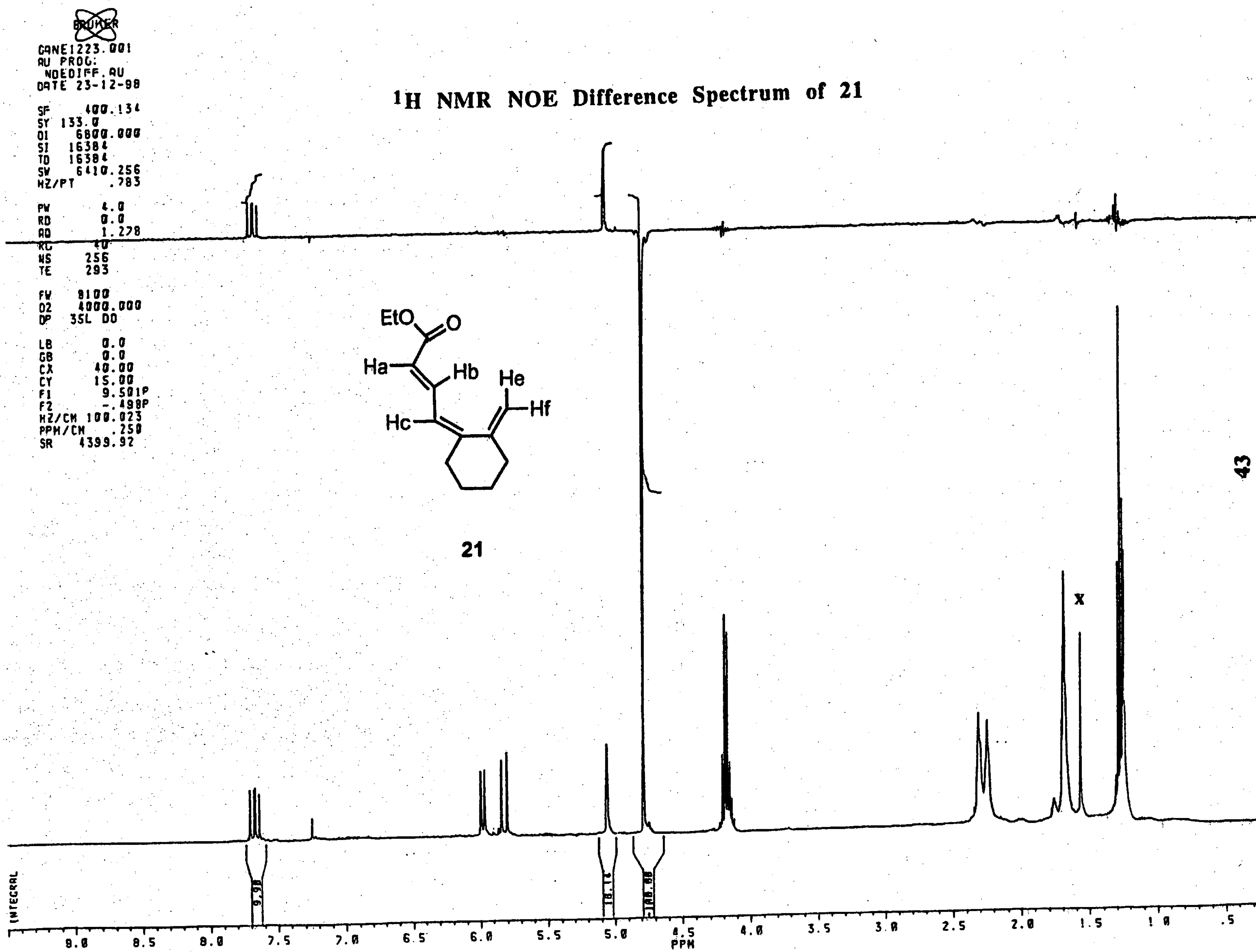
1H NMR NOE Difference Spectrum of 21



21

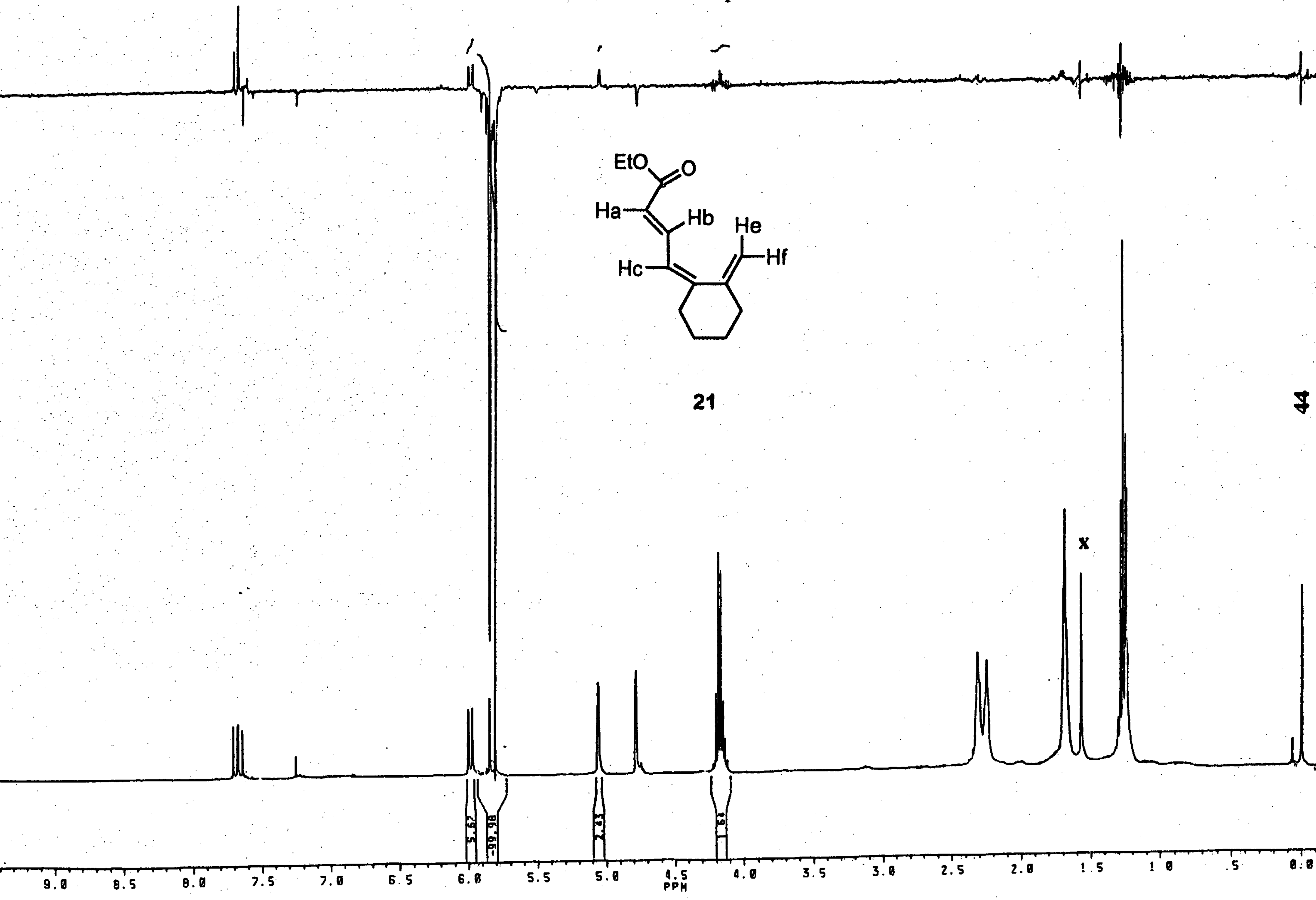
43

1H NMR NOE Difference Spectrum of 21

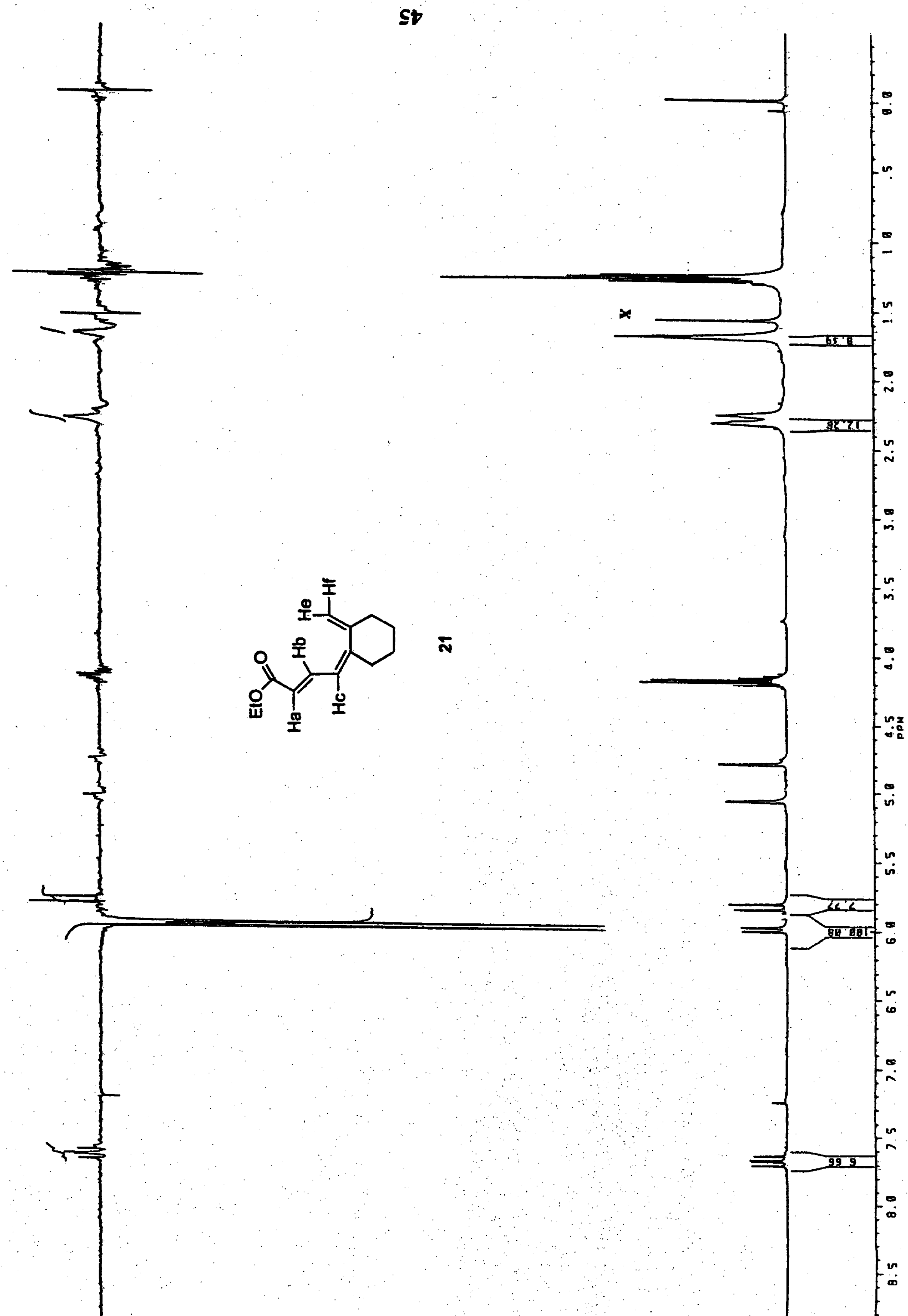


21

^{1}H NMR NOE Difference Spectrum of 21



1H NMR NOE Difference Spectrum of 21



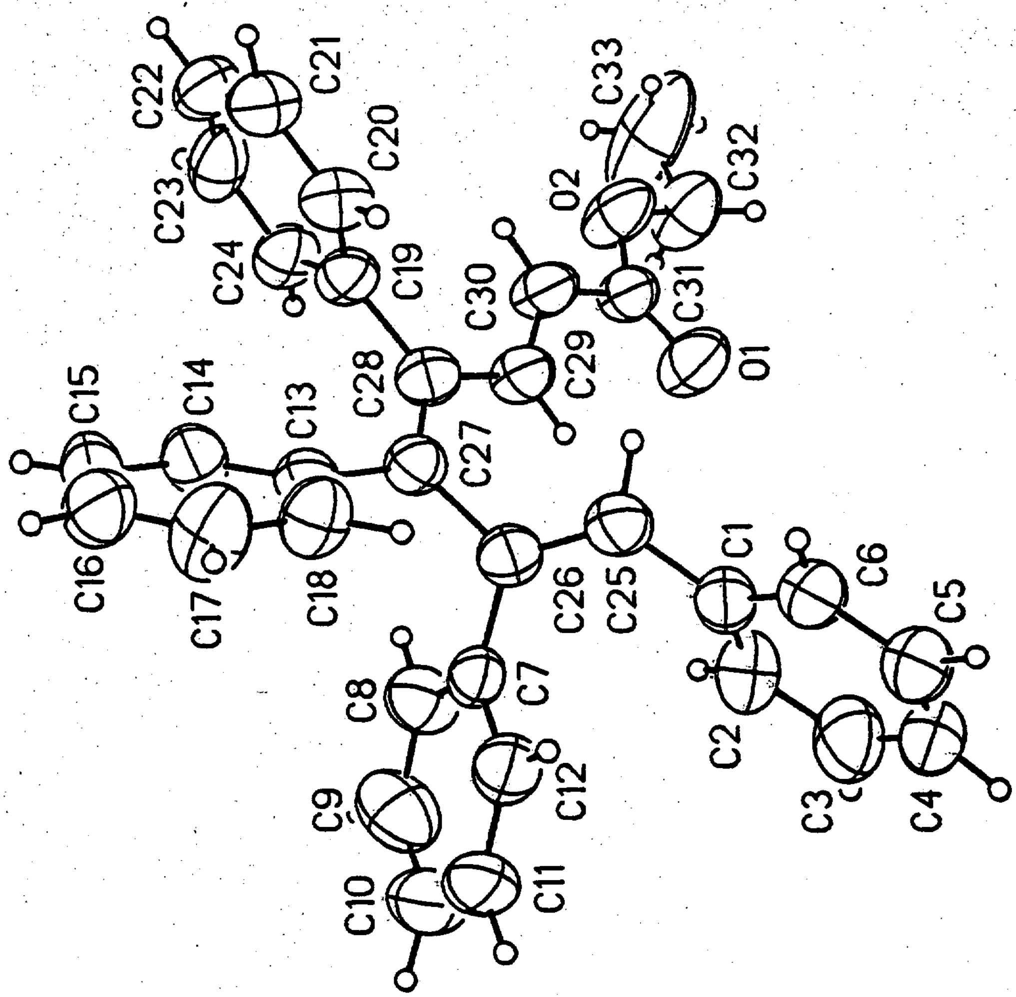


Figure 1. Crystal structure of **17b**

Summary of Crystal Data and Intensity Collection 98FB06 (980027)

Empirical Formula	$C_{33}H_{28}O_2$
Color; Habit	Colorless; Columnar
Crystal Size (mm)	0.18 x 0.25 x 0.40
Space Group	$P2_1$; Monoclinic
Unit Cell Dimensions	$\underline{a}$ = 6.3421(2) Å
	$\underline{b}$ = 17.9336(4) Å; β = 91.342(1)^{o}
	$\underline{c}$ = 11.5250(1) Å
Volume	1310.5(5) $Å^3$
Z	2
Formula Weight	456.6
Density(calc.)	1.157 Mg/m^3
Absorption Coefficient	0.071 mm^{-1}
F(000)	484
Diffractometer Used	Siemens SMART CCD
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	296
Monochromator	Highly oriented graphite crystal
2θ Range	2.5 to 56.0^{o}
Scan Speed	10.00 seconds/ frame
Scan Range (ω)	0.30^{o}/ frame
Index Ranges	$-8 \le h \le 8, -14 \le k \le 23, -14 \le \ell \le 14$
Reflections Collected	7622(5583≥3.0σ(I))
Independent Reflections	4403(2326≥3.0σ(I))(R_{int}=3.77%)
System Used	Siemens SHELXTL PLUS (VMS)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Absolute Structure	η = 3(3)
Extinction Correction	χ = 0.0047(5), where
	$F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Located on difference map
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0006F^2$
Number of Parameters Refined	317
Final R Indices (obs. data)	R = 0.0360, Rw = 0.0383
Goodness-of-Fit	1.09
Largest and Mean Δ/σ	0.001, 0.000
Data-to-Parameter Ratio	7.3:1
Largest Difference Peak/Hole	0.17/-0.18 $eÅ^{-3}$

A crystal of dimensions 0.18 x 0.25 x 0.40 mm for $C_{33}H_{28}O_2$ was selected for indexing and intensity data collection on a Siemens Smart-CCD diffractometer equipped with a normal focus, 3 KW sealed tube X-ray source. Intensity data were collected in 1271 frames with increasing ω (width of 0.3 deg per frame). Unit cell dimensions were determined by a least-squares fit of 3695 reflections with $5 < 2\theta < 50$ deg. Absorption correction was based on 4604 symmetry-equivalent reflections using the SHELXTL-PC program package (Tmin,max = 0.177, 0.976). On the basis of systematic absences, statistics of intensity distribution, and successful solution and refinement of the structure, the space group was determined to be $P2_1$ (No. 4).

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
O(1)	5830(4)	5694	10113(2)	84(1)
O(2)	5025(4)	4679(2)	11105(2)	87(1)
C(1)	12259(5)	6579(2)	7758(3)	60(1)
C(2)	10596(6)	7011(3)	7356(3)	75(1)
C(3)	10743(7)	7781(3)	7343(4)	90(2)
C(4)	12527(8)	8125(3)	7731(3)	83(2)
C(5)	14168(7)	7709(3)	8149(3)	83(2)
C(6)	14053(6)	6941(3)	8170(3)	71(1)
C(7)	10741(5)	5565(2)	5781(2)	52(1)
C(8)	8801(5)	5389(3)	5307(3)	72(1)
C(9)	8203(7)	5673(3)	4226(3)	96(2)
C(10)	9515(9)	6126(3)	3635(3)	96(2)
C(11)	11468(7)	6296(3)	4082(3)	84(2)
C(12)	12079(5)	6017(2)	5156(3)	66(1)
C(13)	12277(5)	3971(2)	6444(2)	52(1)
C(14)	11449(5)	3312(3)	6013(3)	66(1)
C(15)	12626(7)	2846(3)	5314(3)	80(1)
C(16)	14616(7)	3046(3)	5030(3)	82(2)
C(17)	15461(6)	3700(3)	5420(4)	87(2)
C(18)	14288(5)	4163(2)	6117(3)	68(1)
C(19)	9914(5)	3435(2)	8496(2)	54(1)
C(20)	11726(6)	3127(3)	8973(3)	69(1)
C(21)	11759(7)	2405(3)	9377(3)	81(2)
C(22)	9982(8)	1982(3)	9322(3)	80(2)
C(23)	8144(7)	2270(2)	8858(3)	76(1)
C(24)	8115(5)	2997(2)	8431(3)	64(1)
C(25)	12098(5)	5753(2)	7805(3)	63(1)
C(26)	11347(4)	5296(2)	6974(2)	53(1)
C(27)	11093(4)	4486(2)	7217(2)	52(1)
C(28)	9890(5)	4233(2)	8104(2)	53(1)
C(29)	8541(5)	4733(2)	8756(2)	56(1)
C(30)	7534(5)	4545(2)	9715(3)	63(1)
C(31)	6103(5)	5047(2)	10312(3)	59(1)
C(32)	3417(6)	5082(3)	11725(3)	91(2)
C(33)	2245(10)	4559(4)	12361(7)	193(4)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

O(1)-C(31)	1.194 (4)	O(2)-C(31)	1.330 (4)
O(2)-C(32)	1.452 (5)	C(1)-C(2)	1.380 (5)
C(1)-C(6)	1.384 (5)	C(1)-C(25)	1.487 (6)
C(2)-C(3)	1.385 (7)	C(3)-C(4)	1.355 (6)
C(4)-C(5)	1.360 (6)	C(5)-C(6)	1.379 (7)
C(7)-C(8)	1.371 (4)	C(7)-C(12)	1.386 (5)
C(7)-C(26)	1.499 (4)	C(8)-C(9)	1.391 (5)
C(9)-C(10)	1.358 (7)	C(10)-C(11)	1.365 (7)
C(11)-C(12)	1.383 (5)	C(13)-C(14)	1.382 (6)
C(13)-C(18)	1.381 (5)	C(13)-C(27)	1.497 (5)
C(14)-C(15)	1.391 (5)	C(15)-C(16)	1.359 (6)
C(16)-C(17)	1.361 (7)	C(17)-C(18)	1.385 (6)
C(19)-C(20)	1.377 (5)	C(19)-C(24)	1.387 (5)
C(19)-C(28)	1.500 (5)	C(20)-C(21)	1.377 (7)
C(21)-C(22)	1.359 (7)	C(22)-C(23)	1.373 (6)
C(23)-C(24)	1.394 (6)	C(25)-C(26)	1.340 (5)
C(26)-C(27)	1.489 (6)	C(27)-C(28)	1.367 (4)
C(28)-C(29)	1.460 (5)	C(29)-C(30)	1.334 (4)
C(30)-C(31)	1.462 (5)	C(32)-C(33)	1.413 (8)

Table 3. Bond angles (°)

C(31)-O(2)-C(32)	118.0(4)	C(2)-C(1)-C(6)	117.9(4)
C(2)-C(1)-C(25)	121.3(3)	C(6)-C(1)-C(25)	120.8(3)
C(1)-C(2)-C(3)	120.8(4)	C(2)-C(3)-C(4)	120.4(4)
C(3)-C(4)-C(5)	119.6(5)	C(4)-C(5)-C(6)	120.9(4)
C(1)-C(6)-C(5)	120.4(4)	C(8)-C(7)-C(12)	118.9(3)
C(8)-C(7)-C(26)	119.9(3)	C(12)-C(7)-C(26)	121.2(3)
C(7)-C(8)-C(9)	119.7(4)	C(8)-C(9)-C(10)	120.7(4)
C(9)-C(10)-C(11)	120.4(4)	C(10)-C(11)-C(12)	119.3(4)
C(7)-C(12)-C(11)	120.9(3)	C(14)-C(13)-C(18)	117.4(3)
C(14)-C(13)-C(27)	123.3(3)	C(18)-C(13)-C(27)	119.2(3)
C(13)-C(14)-C(15)	121.2(3)	C(14)-C(15)-C(16)	119.6(4)
C(15)-C(16)-C(17)	120.6(4)	C(16)-C(17)-C(18)	119.7(4)
C(13)-C(18)-C(17)	121.4(4)	C(20)-C(19)-C(24)	118.2(4)
C(20)-C(19)-C(28)	120.3(3)	C(24)-C(19)-C(28)	121.5(3)
C(19)-C(20)-C(21)	121.2(4)	C(20)-C(21)-C(22)	120.3(4)
C(21)-C(22)-C(23)	120.2(4)	C(22)-C(23)-C(24)	119.6(4)
C(19)-C(24)-C(23)	120.5(3)	C(1)-C(25)-C(26)	127.4(3)
C(7)-C(26)-C(25)	122.5(4)	C(7)-C(26)-C(27)	117.5(3)
C(25)-C(26)-C(27)	120.0(3)	C(13)-C(27)-C(26)	115.5(3)
C(13)-C(27)-C(28)	122.5(4)	C(26)-C(27)-C(28)	122.0(3)
C(19)-C(28)-C(27)	122.7(3)	C(19)-C(28)-C(29)	115.6(3)
C(27)-C(28)-C(29)	121.7(3)	C(28)-C(29)-C(30)	124.7(4)
C(29)-C(30)-C(31)	123.3(4)	O(1)-C(31)-O(2)	122.7(3)
O(1)-C(31)-C(30)	126.6(3)	O(2)-C(31)-C(30)	110.6(3)
O(2)-C(32)-C(33)	108.0(4)		

Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	102(2)	55(2)	95(2)	13(1)	24(1)	10(1)
O(2)	105(2)	57(2)	101(2)	22(1)	53(1)	15(1)
C(1)	72(2)	54(2)	54(2)	-10(2)	9(2)	-6(2)
C(2)	70(2)	64(3)	90(2)	0(2)	3(2)	-19(2)
C(3)	101(3)	72(3)	98(3)	19(3)	-3(2)	-18(2)
C(4)	117(3)	58(2)	75(2)	-14(2)	10(2)	-7(2)
C(5)	101(3)	73(3)	74(2)	-31(3)	-4(2)	-1(2)
C(6)	88(3)	62(2)	63(2)	-20(2)	-4(2)	3(2)
C(7)	62(2)	41(2)	54(2)	-1(2)	14(1)	-3(1)
C(8)	70(2)	82(3)	64(2)	-8(2)	1(2)	4(2)
C(9)	87(3)	122(4)	78(3)	14(3)	-15(2)	-6(3)
C(10)	139(4)	85(3)	64(2)	31(3)	15(3)	14(2)
C(11)	123(4)	70(3)	61(2)	9(2)	32(2)	8(2)
C(12)	74(2)	59(2)	66(2)	-5(2)	18(2)	-3(2)
C(13)	53(2)	52(2)	50(2)	-4(2)	4(1)	3(1)
C(14)	71(2)	68(2)	59(2)	-20(2)	12(2)	-5(2)
C(15)	110(3)	66(3)	65(2)	-23(2)	24(2)	-14(2)
C(16)	101(3)	67(3)	79(2)	10(2)	29(2)	2(2)
C(17)	66(2)	78(3)	118(3)	4(2)	28(2)	-9(3)
C(18)	58(2)	61(2)	87(2)	-11(2)	12(2)	-4(2)
C(19)	61(2)	50(2)	52(2)	4(2)	17(1)	7(2)
C(20)	78(2)	66(2)	63(2)	-1(2)	3(2)	11(2)
C(21)	108(3)	71(3)	65(2)	16(3)	2(2)	6(2)
C(22)	124(4)	51(2)	67(2)	9(3)	30(2)	9(2)
C(23)	94(3)	53(2)	81(2)	-15(2)	35(2)	-9(2)
C(24)	66(2)	57(2)	70(2)	-8(2)	21(2)	5(2)
C(25)	69(2)	62(2)	56(2)	-10(2)	1(2)	6(2)
C(26)	53(2)	53(2)	54(2)	-9(2)	8(1)	2(2)
C(27)	53(2)	51(2)	52(2)	-7(2)	3(1)	4(2)
C(28)	54(2)	53(2)	54(2)	-3(2)	5(1)	7(2)
C(29)	61(2)	49(2)	58(2)	-2(2)	5(1)	8(1)
C(30)	74(2)	50(2)	65(2)	11(2)	16(2)	14(2)
C(31)	67(2)	51(2)	59(2)	4(2)	10(2)	8(2)
C(32)	97(3)	67(2)	112(3)	12(2)	55(2)	-5(2)
C(33)	186(6)	85(4)	315(8)	0(4)	169(6)	-29(5)

The anisotropic displacement factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2x10^3$)

	x	y	z	U
H(2A)	9363	6781	7148	80
H(3A)	9769	8019	6959	80
H(4A)	12784	8641	7662	80
H(5A)	15465	7917	8441	80
H(6A)	15152	6640	8461	80
H(8A)	7937	5050	5760	80
H(9A)	6932	5485	3958	80
H(10A)	9006	6301	2839	80
H(11A)	12313	6627	3662	80
H(12A)	13414	6129	5495	80
H(14A)	10170	3169	6209	80
H(15A)	11821	2415	5009	80
H(16A)	15310	2775	4536	80
H(17A)	16861	3826	5188	80
H(18A)	14967	4636	6435	80
H(20A)	12899	3420	8969	80
H(21A)	12842	2109	9772	80
H(22A)	9887	1491	9579	80
H(23A)	6845	2009	8777	80
H(24A)	6836	3206	8103	80
H(25A)	12534	5525	8547	80
H(29A)	8361	5182	8489	80
H(30A)	7825	4079	10013	80
H(32A)	2038	5116	11140	80
H(32B)	4057	5487	12091	80
H(33A)	1591	4163	12076	80
H(33B)	3336	4359	12746	80
H(33C)	1077	4805	12910	80