Supplementary Information

A Reductive-Coupling plus Nazarov Cyclization Sequence in the Asymmetric Synthesis of Five-Membered Carbocycles

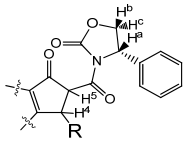
Daniel J. Kerr, Jonathan M. White, Bernard L. Flynn

Medicinal Chemistry and Drug Action, Monash Institute of Pharmaceutical Sciences, Monash University, 381 Royal Parade, Parkville Vic 3052, Australia.

CONTENTS

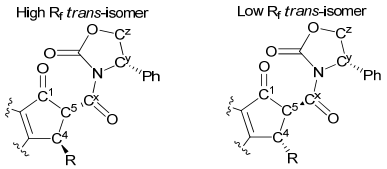
I. NMR Tables for Selected Products 31

Table 5: ^{1}H NMR comparison of *trans*-Nazarov products bearing the (*S*)-4-phenyloxazolidin-2-one auxiliary.



Entry	Product	R_f	H^4 (ppm)	H^5 (ppm)	H^a (ppm)	H^b (ppm)	H^c (ppm)	$J^{4,5}$ (Hz)	$J^{a,b}$ (Hz)	$J^{a,c}$ (Hz)	$J^{b,c}$ (Hz)
1	**31aA**: Minor, *S*-β-*trans*	High	4.92	3.22	5.46	4.71	4.21	br s	9.0	5.9	8.9
	31aC: Major, *S*-α-*trans*	Low	4.89	3.12	5.43	4.76	4.31	3.0	8.3	2.5	8.7
2	**31eA**: Minor, *S*-β-*trans*	High	4.93	3.25	5.49	4.74	4.25	br s	9.0	6.2	8.9
	31eC: Major, *S*-α-*trans*	Low	4.86	3.05	5.43	4.75	4.29	1.8	8.4	2.7	8.7
3	**31fA**: Minor, *S*-β-*trans*	High	5.18	3.85	5.52	4.77	4.29	br s	9.0	5.7	9.0
	31fC: Major, *S*-α-*trans*	Low	5.14	3.74	5.49	4.81	4.35	4.2	8.4	2.8	8.9
4	**31gA**: Major, *S*-β-*trans*	High	5.20	4.41	5.43	4.66	4.21	2.1	9.0	5.7	8.7
	31gC: Minor *S*-α-*trans*	Low	5.18	4.30	5.41	4.75	4.27	3.0	8.4	2.7	8.7
5	**31hA**: Major, *S*-β-*trans*	High	5.16	4.43	5.43	4.66	4.21	br s	8.9	6.2	8.9
	31hC: Minor, *S*-α-*trans*	Low	5.13	4.24	5.42	4.74	4.26	2.7	8.4	2.8	8.7
6	**31iA**: Major, *S*-β-*trans*	High	5.48	5.07	5.48	4.70	4.27	5.7	9.2	5.6	8.9
	31iC: Minor, *S*-α-*trans*	Low	5.46	4.97	5.50	4.79	4.29	5.0	8.4	2.9	8.9
7	**31jA**: Major, *S*-β-*trans*	High	5.62	4.99	5.49	4.67	4.21	3.6	9.2	5.6	8.8
	31jC: Minor, *S*-α-*trans*	Low	5.59	4.88	5.50	4.76	4.28	3.6	8.2	2.7	8.8
8	**37A**: Major**,** *R*-α-*trans*	High	5.48	3.53	5.51	4.73	4.24	3.2	9.2	6.2	8.9
	37C: Minor, *R*-β-*trans*	Low	5.45	3.41	5.47	4.77	4.30	3.2	8.4	2.7	8.7
9	**13D**: Major, *S*-β-*trans*	-	5.40	4.98	5.45	4.71	4.25	5.2	9.0	5.5	8.8

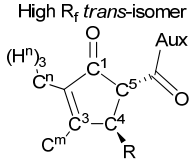
Table 6: ^{13}C NMR comparison of *trans* Nazarov products bearing the (*S*)-4-phenyloxazolidin-2-one auxiliary.



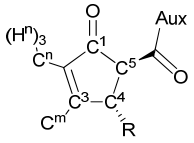
Entry	Product	R_f	C^1 (ppm)	C^5 (ppm)	C^4 (ppm)	C^x (ppm)	C^y (ppm)	C^z (ppm)
1	**31aA** (minor)	High	199.9	56.6	45.5	168.4	58.4	69.7
	31aC (major)	Low	201.1	56.0	46.5	168.8	58.1	69.8
2	**31eA** (minor)	High	191.8	55.4	40.1	168.1	58.7	69.8
	31eC (major)	Low	193.2	54.6	41.7	168.6	58.1	69.9
3	**31fA** (minor)	High	197.6	60.1	40.5	168.0	58.4	69.8
	31fC (major)	Low	198.7	59.6	41.5	168.3	58.1	69.9
4	**31gA** (major)	High	199.7	60.5	51.0	167.4	58.3	69.7
	31gC (minor)	Low	200.9	59.9	52.0	167.9	58.4	70.0
5	**31hA** (major)	High	191.6	58.9	45.7	167.1	58.5	69.8
	31hC (minor)	Low	192.8	58.3	47.0	167.7	58.0	70.0
6	**31iA** (major)	High	196.9	64.0	46.0	166.9	58.3	69.8
	31iC (minor)	Low	198.1	63.4	47.2	167.6	58.3	70.1
7	**31jA** (major)	High	181.9	67.2	41.4	167.9	58.5	69.7
	31jC (minor)	Low	183.0	66.6	42.4	168.3	58.2	69.9
8	**37A** (major)	High	182.7	61.4	43.7	169.1	58.5	69.7
	37C (minor)	Low	183.8	60.8	44.7	169.3	58.1	69.8
9	**13D** (major)*	-	196.9	64.2	45.7	167.0	58.4	69.9

Table 7: NMR comparison of tigloyl-based *trans*-configured Nazarov products.

High R_f *trans*-isomer



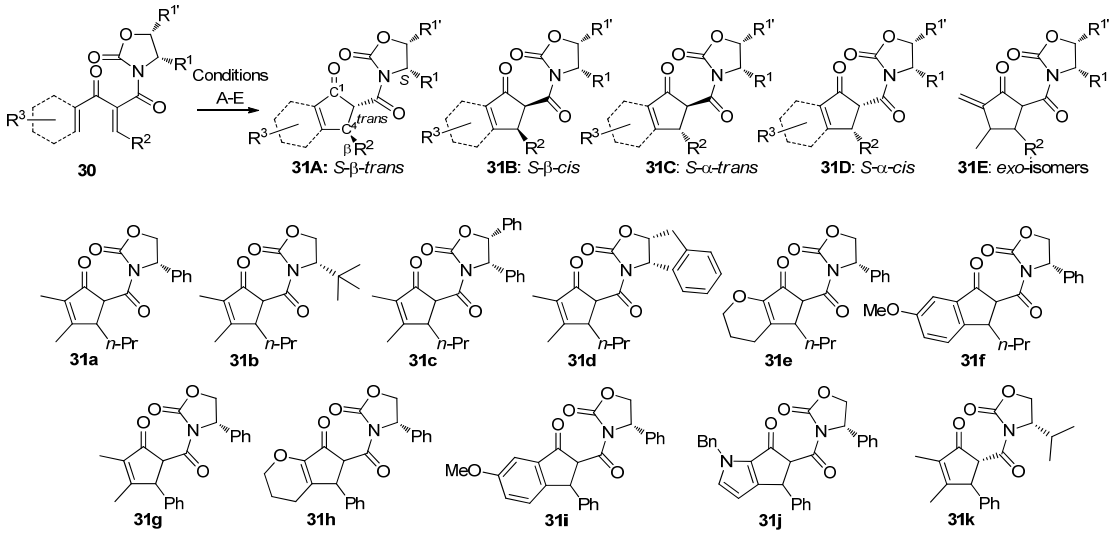
Low R_f *trans*-isomer



Entry	R	Product	R_f	H^n (δ)	C^1 (δ)	C^3 (δ)	C^4 (δ)	C^5 (δ)	C^n (δ)	C^m (δ)
1	*n*-Pr	**31aA** (minor)	Higher	1.97	199.9	172.6	45.5	56.6	15.0	8.2
		31aC (major)	Lower	2.00	201.1	173.2	46.5	56.0	15.0	8.1
2	*n*-Pr	**31bA** (minor)	Higher	2.04	200.7	172.7	45.9	56.4	15.0	8.2
		31bC (major)	Lower	2.05	201.4	173.3	46.4	55.7	15.0	8.1
3	*n*-Pr	**31cA** (minor)	Higher	2.00	200.1	172.5	45.9	56.7	15.0	8.2
		31cC (major)	Lower	2.03	201.0	173.4	46.1	56.3	15.1	8.2
4	*n*-Pr	**31dA** (minor)	Higher	2.05	200.7	172.0	46.9	56.0	15.0	8.2
		31dC (major)	Lower	2.07	201.0	173.2	46.4	56.0	15.1	8.2
5	Ph	**31gA** (major)	Higher	1.79	199.7	170.8	51.0	60.5	15.3	8.5
		31gC (minor)	Lower	1.83	200.9	171.9	52.0	59.9	15.5	8.5
6	Ph	**31kA** (major)	Higher	1.85	200.7	171.0	51.7	60.3	15.4	8.6
		31kC (minor)	Lower	1.86	201.0	171.7	52.2	59.9	15.5	8.5

II. Extended Listing of Reaction Conditions used in the Cyclisation 30 to 31

Table 8: Nazarov Cyclisations of **30** to give **31**.



Entry	Substrate	Conditions[a]	Product, % amount by ^{1}H NMR (isolated yield %)				
			S-β-*trans*	*S*-β-*cis*	*S*-α-*trans*	*S*-α-*cis*	*exo*-isomers
1	**30a**	1.1 Eq. $MeSO_3H$	**31aA**, 21	-	**31aC**, 54	-	**31aE**, 25
2	**30a**	$MeSO_3H$, A	**31aA**, 26 (24)	-	**31aC**, 74 (71)	-	**31aE**, 0
3	**30a**	$Cu(OTf)_2$, B	**31aA**, 31	-	**31aC**, 48	-	**31aE**, 21
4	**30a**	$FeCl_3$, F[b]	**31aA**, 9	-	**31aC**, 19	-	**31aE**, 0
5	**30a**	$SnCl_4$, G[b]	**31aA**, 14	-	**31aC**, 25	-	**31aE**, 2
6	**30b**	$MeSO_3H$, A	**31bA** 22 (21)	-	**31bC** 78 (71)	-	-
7	**30c**	$MeSO_3H$, A	**31cA** 28 (25)	-	**31cC** 72 (71)	-	-
8	**30d**	$MeSO_3H$, A	**31dA** 24 (23)	-	**31dC** 76 (70)	-	-
9	**30e**	2.0 Eq. $MeSO_3H$	**31eA**, 32 (27)	-	**31eC**, 68 (65)	-	-
10	**30e**	$Cu(OTf)_2$, Reflux	**31eA**, 39	-	**31eC**, 61	-	-
11	**30f**	$MeSO_3H$, A[c]	**31fA**, 28 (26)	-	**31fC**, 46 (43)	-	-
12	**30g**	$MeSO_3H$, A	**31gA**, 31	**31gB**, 41	**31gC**, 17	**31gD**, 11	**31gE**, 0
13	**30g**	$MeSO_3H$, C	**31gA**, 0	**31gB**, 63 (70)[d]	**31gC**, 0	**31gD**, 24	**31gE**, 13
14	**30g**	$Cu(OTf)_2$, B	**31gA**, 60 (65)[d]	**31gB**, 8	**31gC**, 16 (10)	**31gD**, 2	**31gE**, 14
15	**30g**	0.1 Eq. $Cu(OTf)_2$, Reflux	**31gA**, 56	**31gB**, 5	**31gC**, 18	**31gD**, 4	**31gE**, 17
16	**30g**	$Cu(OTf)_2$, toluene, 60 °C	**31gA**, 58	**31gB**, 4	**31gC**, 17	**31gD**, 3	**31gE**, 17
17	**30h**	$MeSO_3H$, C	**31hA**, 7	**31hB**, 68 (64)	**31hC**, 2	**31hD**, 23	-
18	**30h**	$Cu(OTf)_2$, B	**31hA**, 70 (48)	**31hB**, 9	**31hC**, 13 (8)	**31hD**, 8	-
19	**30i**	$MeSO_3H$, A	**31iA**, 71 (68)	**31iB**, 15	**31iC**, 13 (5)	**31iD**, 0	-
20	**30i**	$Cu(OTf)_2$, Reflux[c]	**31iA**, 18	**31iB**, 0	**31iC**, 0	**31iD**, 0	-
21	**30j**	$MeSO_3H$, C	**31jA**, 0	**31jB**, 82 (81)	**31jC**, 0	**31jD,** 18 (10)	-
22	**30j**	$Cu(OTf)_2$, Reflux	**31jA**, 56 (51)	**31jB**, 18 (16)	**31jC**, 22 (18)	**31jD,** 4	-
23	**30k**	$MeSO_3H$, A	**31kA**, 40	**31kB**, 14	**31kC**, 23	**31kD**, 16	**31kE**, 7
24	**30k**	$Cu(OTf)_2$, B	**31kA**, 51 (50)	**31kB**, 6	**31kC**, 22 (23)[d]	**31kD**, 5	**31kE**, 16

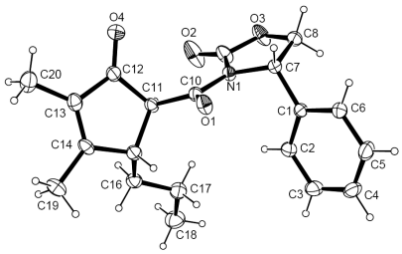
[a]All reactions in CH_2Cl_2 unless stated otherwise, A: $MeSO_3H$ (5-10 equiv.), −78 °C to rt, 1-24 h, then $NaHCO_3$ (aq); B: $Cu(OTf)_2$ (1.1 equiv.), −78 °C to rt, 1-48 h, then $NaHCO_3$ (aq); C: $MeSO_3H$ (10 equiv.), −78 °C to 0 °C, 0.5 – 2 h, then $NaHCO_3$ (aq); F: $FeCl_3$ (1.1 equiv.), −78 °C to rt, 1 h, then $NaHCO_3$ (aq); G: $SnCl_4$ (1.1 equiv.), −78 °C to rt, 1 h, then $NaHCO_3$ (aq). [b]A significant amount of undiscerned product was observed in the ^{1}H NMR of the crude reaction mixture. [c] Starting material **30** represented the majority of remaining product based on ^{1}H NMR of the crude reaction mixture. [d]Some isomerisation of minor isomers during chromatography gives greater yield of isolated product.

III. Crystallographic data for 31aC and 31hA

Crystallography. Diffraction data for compound **31aC** were recorded on an Enraf Nonius CAD4f diffractometer operating in the θ/2θ scan mode at 293 K. Data processing, absorption corrections,[1] structure solution,[2] and refinement[3] were implemented within the WingX suite of programs.[4] Intensity data for **31hA** were collected at 130 K with a Bruker SMART Apex CCD detector using Mo-Kα radiation (graphite crystal monochromator λ = 0.71073).[5] Data were reduced using the program SAINT and corrected for absorption (SADABS).

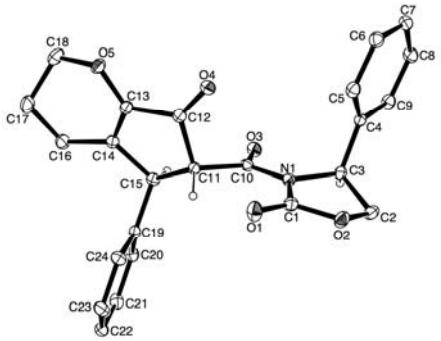
Crystal data for **31aC**. $C_{20}H_{23}NO_4$, *M* = 341.39, *T* = 293(2) K, λ = 1.54180, Orthorhombic, space group $P2_12_12_1$ *a* = 7.793(2*), b* = 13.870(3*), c* =16.681(5*), Å, V* 1803.0(8) $Å^3$, Z = 4, D_c = 1.258 mg M^{-3} μ(Cu-Kα) 0.710 mm^{-1}, F(000) = 728, crystal size 0.4 x 0.3 x 0.2 mm^3. 1971 reflections measured, 1971 independent reflections, the final R was 0.0467 [I > 2σ(I)] and *w*R(F^2) was 0.1238 (all data). This data has been deposited at the Cambridge Crystallographic Data Centre (www.ccdc.cam.ac.uk), depository number: 793956

Figure A: X-Ray crystal structure of **31aC** (ORTEP, numbering different to main text).



Crystal data for **31hA**. $C_{24}H_{21}NO_5$, *M* = 403.42, *T* = 130(2) K, λ = 0.71073, Monoclinic, space group $P2_1$ *a* = 9.483(2*), b* = 22.611(5*), c* =10.386(2*), Å,* β= 114.551(4), *V* 2025.5(8) $Å^3$, Z = 4 (two molecules in asymmetric unit), D_c = 1.323 mg M^{-3} μ(Mo-Kα) 0.093 mm^{-1}, F(000) = 848, crystal size 0.45 x 0.40 x 0.35 mm^3. 16944 reflections measured, 8789 independent reflections (R_{int} = 0.0249), the final R was 0.0515 [I > 2σ(I)] and *w*R(F^2) was 0.1281 (all data). This data has been deposited at the Cambridge Crystallographic Data Centre (www.ccdc.cam.ac.uk), depository number: 793957

Figure B: X-Ray crystal structure **31hA** (ORTEP, numbering different to main text).



[1] A. L. Spek, '*PLATON for windows. A Program for Absorption Corrections' University of Utrecht, The Netherlands*, 1999.
[2] G. M. Sheldrick, '*SHELXS-86; Crystallographic Computing 3*', University of Göttingen, Germany, 1986.
[3] G. M. Sheldrick, '*SHELXL-97; A Program for the Refinement of Crystal Structures*' University of Göttingen, Germany, 1997.
[4] L. J. Farrugia, *J. Appl. Crystallogr.*, **1999**, *32*, 837.
[5] Siemens 1999, SMART, SAINT, SADABS, Siemens Analytical X-ray Instruments Inc. Madison, Wisconsin, USA.

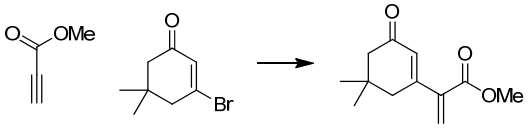
Experimental Details

General Experimental Details

General: All experiments were performed under an anhydrous atmosphere of nitrogen in flame-dried glassware except as indicated. All reaction products were stored in sealed (parafilm) pre-dried vials under an anhydrous atmosphere of nitrogen. Melting points were recorded with an Electrothermal melting point apparatus and are uncorrected. Proton (^{1}H) and carbon (^{13}C) NMR spectra were recorded at 300 MHz for proton and 75 MHz for carbon nuclei. All NMR spectra were recorded in (D)chloroform ($CDCl_3$) at 30 °C. The protonicities of the carbon atoms observed in the carbon NMR were determined using J-modulated spin-echo (JMOD) experiments or APT. Infrared spectra (IR) were obtained on FT-IR spectrometer. Low-resolution mass spectra (LRMS) were recorded on a quadrupole spectrometer using electrospray ionisation (ESI). High-resolution mass spectra (HRMS) were recorded on a time of flight mass spectrometer fitted with either an electrospray (ESI) or atmospheric pressure ionization (APCI) ion source. Tetrahydrofuran and diethyl ether were distilled under nitrogen from sodium benzophenone ketyl. Dichloromethane and 1,2-dichloroethane were distilled from calcium hydride under nitrogen. Analytical thin layer chromatography (TLC) was conducted on aluminium sheets coated with silica gel 60 GF_{254}. Flash chromatography was performed on flash grade silica gel. Experimental methods for the following compounds has been previously reported: **16f-i** and **17f-i** are reported in reference 7 of the main text.

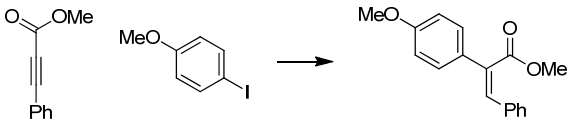
General Procedure A: Reductive-Coupling of 14 (or 29) and 15 to form 16 (or 30).

$Pd(PPh_3)_4$ (0.03 mmol) was added to THF (7 mL) or generated *in situ* by addition $Pd(dba)_2$ (3 mol%) to a solution of PPh_3 (0.12 mmol) in THF followed by stirring for 0.5 h at 18 °C. To this solution alkyne **14** or **29** (1.0 mmol) was added, followed by dropwise addition of Bu_3SnH (1.05 mmol) and then the reaction was left to stir for 10-30 min. Organic halide **15** (1.1 mmol) and CuCl (0.8 mmol) were then added and the reaction stirred at 18 °C for 5-30h, monitoring consumption of intermediate alkenyl stananne by TLC (silica). $KF_{(aq)}$ (30%, 20 mL) was added and the reaction stirred for 2 h. The triphasic (organic, aqueous and solid) mixture was filtered through celite, washing with ethyl acetate (25 mL). The aqueous phase was separated from organic phase and further extracted with ethyl acetate (25mL). The combined organic extracts were dried over $MgSO_4$ and concentrated onto silica gel (1 g) under reduced pressure. The solid residue was subjected to flash chromatography (silica gel) to provide the product (**16** or **30**).



Methyl 2-(5,5-dimethyl-3-oxo-cyclohex-1-enyl)-acrylate (16a)

Alkyne **14a** was reacted with 3-bromo-5,5-dimethyl-2-cyclohexenone (**15a**)[6] in accordance with General Procedure A, giving the product **16a** after flash chromatography (silica gel, hexanes / dichloromethane / diethyl ether 6:2:1) as a clear oil (61%). The ^{1}H NMR of the crude reaction mixture indicated almost quantitative conversion of **14a** to **16a**, however, the product is unstable and the isolated material decomposed at 4 °C over several days. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.25 (s, 1H), 6.16 (t, J = 1.6 Hz, 1H), 5.86 (s, 1H), 3.81 (s, 3H), 2.39 (d, J = 1.6 Hz, 2H), 2.29 (s, 2H), 1.08 (s, 6H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 199.7 (C), 165.6 (C), 154.3 (C), 141.2 (C), 127.1 (CH_2), 126.9 (CH), 52.0 (CH_3), 50.8 (CH_2), 41.7 (CH_2), 33.6 (C), 27.9 (CH_3). IR (NaCl film, cm^{-1}): 2957, 2870, 1726, 1668, 1437, 1369, 1297, 1198, 1158, 994, 906. LRMS (70 eV) *m/z* (%): 208 (M^+, 65), 133 (65), 124 (100), 105 (80), 91 (60). HRMS calcd for $C_{12}H_{16}O_3$: 208.1099. Found: 208.1099.

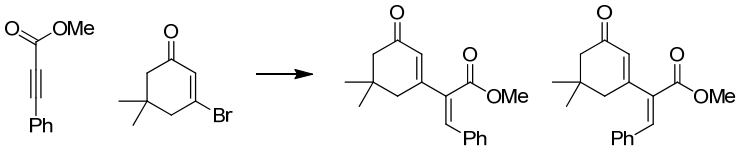


Methyl (*Z*)-2-(4-methoxyphenyl)-3-phenyl-acrylate (16b)

Alkyne **14b** was reacted with 4-iodoanisole (**15b**) in accordance with General Procedure A, giving the product **16b** after flash chromatography (silica gel, hexanes / diethyl ether 9:1) as a slightly tan oil (75%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (d, J = 8.3 Hz, 2H), 7.30-7.20 (m, 5H), 6.89 (s, 1H), 6.84 (d, J = 8.3 Hz, 2H), 3.75 (s, 3H), 3.71 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 170.3 (C), 159.7 (C), 135.8 (C), 134.3 (C), 129.5 (CH), 129.3 (C), 128.4 (CH), 128.0 (CH), 127.9 (CH), 127.6

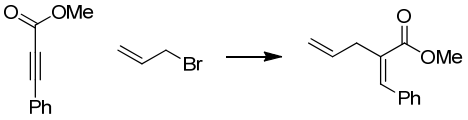
[6] Piers, E.; Grierson, J. R.; Lau, C. K.; Nagakura, I. *Can. J. Chem.* **1982**, *60*, 210.

(CH), 114.0 (CH), 55.2 (CH_3), 52.1 (CH_3). IR (NaCl film, cm^{-1}): 3025, 2952, 2839, 1727, 1606, 1513, 1288, 1249, 1033. LRMS (70 eV) *m/z* (%): 268 ($M^{+\cdot}$, 100), 209 (55), 165 (55), 121 (85), 84 (65). Known compound.[7]



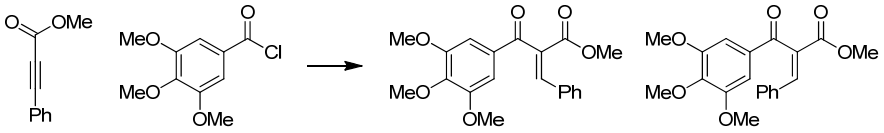
Methyl (*Z*)/(*E*)-2-(5,5-Dimethyl-3-oxo-cyclohex-1-enyl)-3-phenyl-acrylate (16c)

Alkyne **14b** was reacted with 3-bromo-5,5-dimethyl-2-cyclohexenone (**15a**) in accordance with General Procedure A giving the product **16c** after flash chromatography (silica gel, hexanes / dichloromethane / diethyl ether 9:3:1) giving the product as separable (*E/Z*)-isomers of **16c** (93%). **Higher R_f isomer *Z*-16c**, (22%, thick oil) ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (m 3H), 7.12-7.04 (m, 2H), 6.31 (s, 1H), 5.83 (s, 1H), 3.57 (s, 3H), 2.42 (s, 2H), 2.28 (s, 2H), 1.08 (s, 6H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 200.0 (C), 165.8 (C), 156.1 (C), 155.1 (C), 136.2 (C), 130.5 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 120.3 (CH), 51.6 (CH_3), 51.0 (CH_2), 40.6 (CH_2), 33.6 (C), 28.3 (CH_3). IR (NaCl film, cm^{-1}): 3080, 3057, 3026, 2957, 2870, 1731, 1668, 1613, 1590, 1434, 1307, 1276, 1236, 1167, 1118, 1018, 700. LRMS (70 eV) *m/z* (%): 284 ($M^{+\cdot}$, 100), 237 (85), 225 (80), 209 (80), 199 (80), 141 (70). **Lower R_f isomer *E*-16c**, (71%, white solid). Mp = 77-9 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (s, 5H), 7.04 (s, 1H), 6.03 (s, 1H), 3.78 (s, 3H), 2.48 (s, 2H), 2.31 (s, 2H), 1.12 (s, 6H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 199.5 (C), 168.6 (C), 151.6 (C), 134.9 (C), 134.2 (C), 133.8 (CH), 129.3 (CH), 128.6 (CH), 128.4 (CH), 125.5 (CH), 52.3 (CH_3), 50.7 (CH_2), 39.4 (CH_2), 33.1 (C), 28.3 (CH_3). IR (KBr disc, cm^{-1}): 3047, 2955, 2929, 2871, 1725, 1673, 1581, 1465, 1440, 1305, 1277, 1261, 1236, 1219, 1161, 1111, 1003, 759, 688, 520. LRMS (70 eV) *m/z* (%): 284 ($M^{+\cdot}$, 85), 225 (90), 209 (80), 196 (75), 141 (100). HRMS calcd for $C_{18}H_{20}O_3$: 284.1412. Found: 284.1417.



Methyl (*Z*)-2-benzylidine-4-pentenoate (16d)

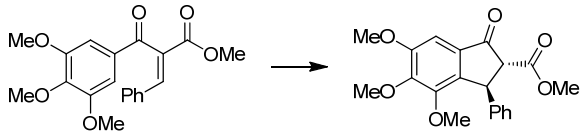
Alkyne **14b** was reacted with allylbromide (**15c**) in accordance with General Procedure A giving the product **16d** after flash chromatography (silica gel, hexanes / diethyl ether 39:1) as a clear oil (83%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.20 (m, 5H), 6.68 (s, 1H), 5.89 (ddt, *J* = 17.0, 10.2, 6.6 Hz, 1H), 5.17 (d, *J* = 17.0 Hz, 1H), 5.13 (d, *J* = 10.2 Hz, 1H), 3.64 (s, 3H), 3.17 (d, *J* = 6.6 Hz, 2H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 169.8 (C), 136.0 (C), 134.5 (CH), 134.2 (CH), 132.5 (C), 128.1 (CH), 128.0 (CH), 127.8 (CH), 117.4 (CH_2), 51.6 (CH_3), 39.2 (CH_2). IR (NaCl film, cm^{-1}): 3081, 3061, 3027, 2951, 1720, 1435, 1215, 1123, 922, 752, 696. LRMS (70 eV) *m/z* (%): 202 ($M^{+\cdot}$, 60), 143 (100).



Methyl (*Z*)/(*E*)-3-phenyl-2-(3,4,5-trimethoxybenzoyl)-acrylate (16e)

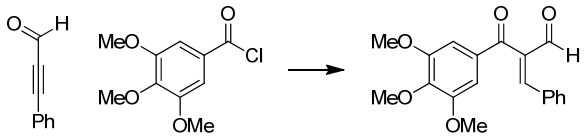
Alkyne **14b** was reacted with 3,4,5-trimethoxybenzoyl chloride (**15d**) in accordance with General Procedure A giving the product **16e** after flash chromatography (silica gel, hexanes / dichloromethane / diethyl ether 10:10:1) as a slightly yellow solid (70%). Mp 69-70 °C. This material was identified as being the (*E*)-isomer, a small amount of (*Z*)-isomer was also isolated (yield not determined). ***Z*-16e**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20-7.50 (m, 6H), 7.08 (s, 2H), 3.93 (s, 3H), 3.88 (s, 6H), 3.79 (s, 3H). ***E*-16e**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.40-7.25 (m, 5H), 7.19 (s, 2H), 3.90 (s, 3H), 3.82 (s, 6H), 3.78 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 193.8 (C), 165.1 (C), 152.9 (C), 143.0 (C), 142.4 (CH), 132.5 (C), 130.6 (C), 130.3 (C), 130.2 (CH), 129.8 (CH), 128.5 (CH), 106.2 (CH), 60.5 (CH_3), 55.9 (CH_3), 52.3 (CH_3). IR (KBr disc, cm^{-1}): 3089, 3061, 3001, 2947, 2836, 1712, 1663, 1619, 1581, 1501, 1329, 1255, 1128, 1000. LRMS (70 eV) *m/z* (%): 356 ($M^{+\cdot}$, 90), 195 (100), 121 (35), 84 (40). HRMS calcd for $C_{20}H_{20}O_6$: 356.1260. Found: 356.1258.

[7] Cacchi, S.; Fabrizi, G; Moro, L.; Pace, P.; *Synlett* **1997**, 1367.



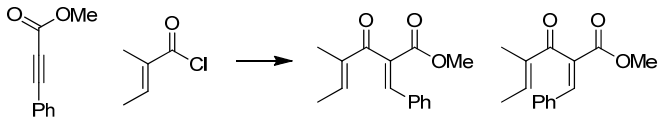
(±)-Methyl *trans*-4,5,6-trimethoxy-1-oxo-3-phenyl-2,3-dihydro-1*H*-indene-2-carboxylate (17e)

Methanesulfonic acid (110 μL, 1.66 mmol) was added to a solution of propenoate **16a** (130 mg, 0.365 mmol) in dry dichloromethane (2 mL). After stirring for 2 h the solution was diluted with diethyl ether (10 mL), washed with distilled water (5 mL), dried over $MgSO_4$ and concentrated onto silica gel (~1 g). Flash chromatography (14:14:1, hexanes / dichloromethane / diethyl ether) returned the desired material **8f** as a discoloured oil (108 mg, 84 %). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.15 (m, 3H), 7.15-7.05 (m, 2H), 7.08 (s, 1H), 4.94 (d, J = 3.2 Hz, 1H), 3.90 (2 × s, 6H), 3.77 (s, 3H), 3.62 (d, J = 3.2 Hz, 1H), 3.33 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 197.7 (C), 168.7 (C), 155.1 (C), 150.2 (C), 149.4 (C), 143.4 (C), 142.5 (C), 130.3 (C), 128.7 (CH), 127.3 (CH), 127.0 (CH), 100.9 (CH), 63.5 (CH), 60.8 (CH_3), 59.9 (CH_3), 56.2 (CH_3), 52.8 (CH_3), 46.1 (CH). IR (KBr film, cm^{-1}): 3084, 3059, 3028, 2952, 2852, 1741, 1710, 1661, 1572, 1471, 1434, 1345, 1313, 1267, 1191, 1126, 1065, 1030, 1004, 966, 841, 702, 559. LRMS (70 eV) *m/z* (%): 356 ($M^{+\cdot}$, 85), 324 (40), 296 (100). HRMS calcd for $C_{20}H_{20}O_6$: 356.1260. Found: 356.1263.



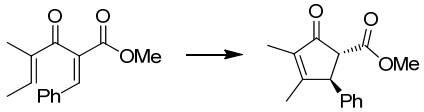
3-Phenyl-2-(3, 4, 5-trimethoxybenzoyl)-2-propenal (16j)

Alkyne **14d** was reacted with **15d** in accordance with General Procedure A giving the product **16j** after flash chromatography (silica gel, hexanes / dichloromethane / diethyl ether 20:20:3) giving the product **16j** as a lightly discolored solid (48%). Mp = 100-2 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.73 (s, 1H), 7.59 (s, 1H), 7.45-7.25 (m, 5H), 7.15 (s, 2H), 3.88 (s, 3H), 3.79 (s, 6H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 194.4 (C), 191.2 (CH), 153.3 (C), 150.1 (CH), 143.7 (C), 140.3 (C), 132.4 (C), 131.6 (CH), 130.6 (CH), 129.1 (CH), 106.6 (CH), 60.9 (CH_3), 56.2 (CH_3). IR (KBr disc, cm^{-1}): 3089, 3014, 2941, 2841, 1678, 1645, 1609, 1580, 1499, 1466, 1413, 1373, 1331, 1234, 1208, 1127, 1000, 860, 761, 684. LRMS (70 eV) *m/z* (%): 326 ($M^{+\cdot}$, 100), 298 (90), 283 (50), 195 (70). HRMS calcd for $C_{19}H_{18}O_5$: 326.1154. Found: 326.1155.



Methyl (2*Z*,4*E*)/(2*Z*,4*E*)-2-benzylidene-4-methyl-3-oxohex-4-enoate (16k).

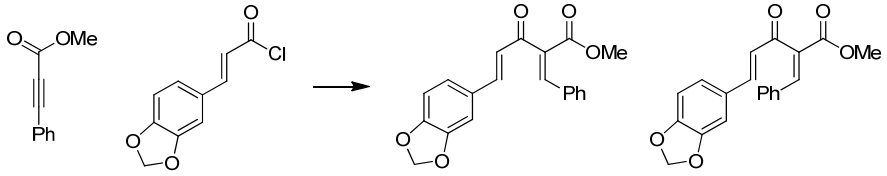
Alkyne **14b** was reacted with **15g** in accordance with General Procedure A giving the product **16k** after flash chromatography (silica gel, hexanes / diethyl ether 4:1) as a slightly tan oil (87%). Proton NMR analysis revealed 91% isomeric purity predominately the *E*,*Z*-isomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.30 (m, 5H), 7.21 (s, 1H), 6.62 (q, J = 6.7, Hz, 1H), 3.77 (s, 3H), 2.89 (s, 3H), 2.88 (d, J = 6.7 Hz, 3H). On standing at ~4 °C for 72 h the (*Z*,*E*)-isomer converted completely to the (*E*,*E*)-isomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.35-7.28 (m, 5H), 6.72 (q, J = 6.7 Hz, 1H), 3.79 (s, 3H), 1.90 (s, 3H), 1.78 (d, J = 6.7 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 197.0 (C), 165.5 (C), 142.9 (CH), 141.6 (CH), 138.1 (C), 133.0 (C), 131.2 (C), 130.0 (CH), 129.8 (CH), 128.6 (CH), 52.3 (CH_3), 14.9 (CH_3), 10.4 (CH_3). IR (NaCl film, cm^{-1}): 3056, 3028, 3000, 2952, 1715 (2 peaks), 1640 (multiple peaks), 1575, 1496, 1434, 1258, 1200. LRMS (70 eV) *m/z* (%): 244 ($M^{+\cdot}$, 85), 211 (65), 185 (65), 121 (70), 83 (100). HRMS calcd for $C_{15}H_{16}O_3$: 244.1099. Found: 244.1098.



(±)-Methyl *trans*-3,4-dimethyl-2-oxo-5-phenylcyclopent-3-enecarboxylate (17k)

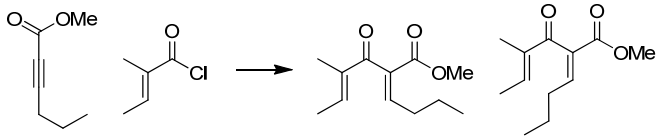
Methanesulfonic acid (16 μL, 0.24 mmol) was added to a solution of **16k** (48.9 mg, 0.200 mmol) in dry dichloromethane (7 mL). After stirring for 4 h the solution was diluted with diethyl ether (20 mL), washed with distilled water (20 mL), dried over $MgSO_4$ and concentrated under reduced pressure to give **17k** as a slightly discolored solid (44.1 mg, 90%) Mp = 70-2

°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.20 (m, 3H), 7.09 (dd, *J* = 6.4, 3.0 Hz, 2H), 4.23 (d, *J* = 2.7 Hz, 1H), 3.76 (s, 3H), 3.39 (d, *J* = 2.7 Hz, 1H), 1.87 (s, 3H), 1.81 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 201.4 (C), 171.8 (C), 169.1 (C), 140.1 (C), 135.4 (C), 129.1 (CH), 127.5 (CH), 60.9 (CH), 53.0 (CH_3 or CH), 52.7 (CH_3 or CH), 15.6 (CH_3), 8.5 (CH_3). IR (KBr disc, cm^{-1}): 3029, 2951, 1735, 1702, 1645, 1599, 1494, 1433, 1162, 1031, 767, 700. LRMS (70 eV) *m/z* (%): 244 ($M^{+\cdot}$, 75), 184 (100), 141 (35). HRMS calcd for $C_{15}H_{16}O_3$: 244.1099. Found: 244.1101.



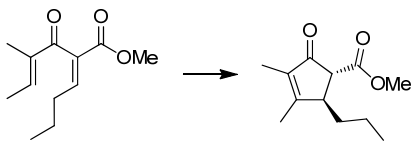
Methyl (2*Z*,4*E*)/(2*E*,4*E*)-5-(benzo[*d*][1,3]dioxol-5-yl)-2-benzylidene-3-oxopent-4-enoate (16l)

Alkyne **14b** was reacted with **15h** in accordance with General Procedure A giving the product **16l** after flash chromatography flash chromatography (silica gel, hexanes / dichloromethane / diethyl ether 10:10:1) as a yellow solid (72%, mixture of (*Z*,*E*)- and (*E*,*E*)-isomers). Crystallization of this material from minimal dichloromethane with hexanes gave ***Z*-16l** (414 mg, 41%), the remaining material contains roughly equal proportions of both isomers. ***Z*-16l**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 15.4 Hz, 1H), 7.72 (s, 1H), 7.50-7.36 (m, 5H), 7.11 (s, 1H), 7.10 (d, *J* = 7.9 Hz, 1H), 7.00 (d, *J* = 15.4 Hz, 1H), 6.84 (d, *J* = 7.9 Hz, 1H), 6.03 (s, 2H), 3.87 (s, 3H). LRMS (70 eV) *m/z* (%): 336 ($M^{+\cdot}$, 100), 277 (75), 175 (95), 145 (65), 89 (75). Complete conversion to the (*E*,*E*)-Isomer ***E*-16l** was observed after storing the material at 4°C for 6 months). Mp = 121-3 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.48-7.22 (m, 6H), 6.99 (s, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.78 (d, *J* = 8.4 Hz, 1H), 6.67 (d, *J* = 16.1 Hz, 1H), 6.00 (s, 2H), 3.82 (s 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 195.4 (C), 165.6 (C), 150.3 (C), 148.4 (C), 146.4 (CH), 142.5 (CH), 132.8 (C), 131.2 (C), 130.5 (CH), 130.2 (CH), 128.8 (CH), 128.5 (C), 125.6 (CH), 125.0 (CH), 108.6 (CH), 106.7 (CH), 101.7 (CH_2), 52.7 (CH_3). IR (KBr disc, cm^{-1}): 3070, 3015, 2949, 2903, 1718, 1642, 1601, 1493, 1363, 1258. LRMS (70 eV) *m/z* (%): 336 ($M^{+\cdot}$, 95), 277 (75), 175 (100), 145 (65), 89 (80). HRMS calcd for $C_{20}H_{16}O_5$: 336.0998. Found: 336.0994.



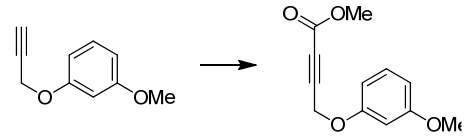
Methyl (*Z*)/(*E*)-2-[(*E*)-2-methylbut-2-enoyl]hex-2-enoate (16m)

Alkyne **14e** was reacted with **15g** in accordance with General Procedure A giving the product **16m** after flash chromatography flash chromatography (silica gel, hexanes / diethyl ether 8:1) as a clear oil (80%). ***Z*-16m**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.51 (q, *J* = 7.1 Hz, 1H), 6.31 (t, *J* = 7.7 Hz, 1H), 3.69 (s, 3H), 2.49 (q, *J* = 7.7 Hz, 2H), 1.82 (d, *J* = 7.1 Hz, 3H), 1.83 (s, 3H), 1.49 (sextet, *J* = 7.7 Hz, 2H), 0.93 (t, *J* = 7.7 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 195.4 (C), 165.7 (C), 149.1 (CH), 140.7 (CH), 138.2 (C), 134.0 (C), 51.7 (CH_3), 31.2 (CH_2), 21.9 (CH_2), 14.7 (CH_3), 13.7 (CH_3), 11.3 (CH_3). IR (NaCl film, cm^{-1}): 2958, 2932, 2871, 1726, 1659, 1642, 1434, 1215, 1158. After standing at 4 °C for 18 months 70% of the initially isolated ***Z*-16m** had converted to the more stable ***E*-16m**, this was purified by flash chromatography (silica gel, hexanes / diethyl ether 8:1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.00 (t, *J* = 7.8 Hz, 1H), 6.64 (m, 1H), 3.71 (s, 3H), 2.01 (dt, *J* = 7.8, 7.4 Hz, 2H), 1.90-1.80 (m, 6H), 1.45 (sextet, *J* = 7.4 Hz, 2H), 0.90 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 196.0 (C), 165.0 (C), 146.6 (CH), 142.8 (CH), 138.8 (C), 133.4 (C), 51.9 (CH_3), 31.4 (CH_2), 21.5 (CH_2), 14.9 (CH_3), 13.6 (CH_3), 10.2 (CH_3). IR (NaCl film, cm^{-1}): 2961, 2932, 2874, 1725, 1660, 1642, 1435, 1281, 1247, 1226, 1054, 1030. LRMS (70 eV) *m/z* (%): 210 ($M^{+\cdot}$, 5), 163 (55), 135 (60), 83 (100), 55 (90). HRMS calcd for $C_{12}H_{18}O_3$: 210.1256. Found: 210.1258.



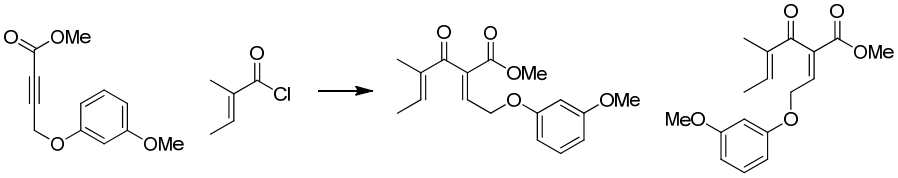
(±)-Methyl *trans*-3,4-dimethyl-2-oxo-5-propylcyclopent-3-enecarboxylate (17m)

Methanesulfonic acid (36 μL, 0.55 mmol) was added to a solution of **16m** (105 mg, 0.500 mmol) in dry dichloromethane (7 mL). After stirring for 24 h the solution was diluted with diethyl ether (10 mL), washed with distilled water (5 mL), dried over $MgSO_4$ and concentrated onto silica gel (0.5 g). Flash chromatography (silica gel, hexanes / diethyl ether 4:1) returned **17m** as a clear oil (92.6 mg, 88%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.74 (s, 3H), 3.08 (d, J = 2.6 Hz, 1H), 3.02 (br. s, 1H), 2.03 (s, 3H), 1.78 (m_c, 1H), 1.69 (s, 3H), 1.42-1.15 (m, 3H), 0.92 (t, J = 7.1 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 201.5 (C), 173.3 (C), 170.2 (C), 134.7 (C), 57.3 (CH), 52.5 (CH_3), 47.2 (CH), 34.3 (CH_2), 20.3 (CH_2), 15.2 (CH_3), 14.0 (CH_3), 8.3 (CH_3). IR (NaCl film, cm^{-1}): 2955, 2929, 2872, 1739, 1702, 1647, 1434, 1158. LRMS (70 eV) *m/z* (%): 210 ($M^{+\cdot}$, 80), 179 (50), 150 (85), 122 (100). HRMS calcd for $C_{12}H_{18}O_3$: 210.1256. Found: 210.1255.



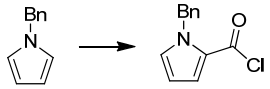
Methyl 4-(3-methoxyphenoxy)-2-butynoate (14f)

n-BuLi (2 M in cyclohexane, 3.10 mL, 6.2 mmol) was added dropwise to 3-methoxyphenyl propargyl ether (1.0 g, 6.17 mmol)[8] in THF (11 mL) at -78 °C. After stirring at this temperature for 15 min, methylchloroformate (0.54 mL, 7.00 mmol) was added and the reaction mixture brought to room temperature. Silica gel (5 g) was added and the solvent removed under reduced pressure. The residue was subject to flash chromatography (silica gel, hexanes / diethyl ether 1:1) giving the product as a colorless oil (1.17 g, 91%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 (t, J = 8.1 Hz, 1H), 6.59-6.50 (m, 3H), 4.78 (s, 2H), 3.78 (s, 3H), 3.76 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 160.7 (C), 158.3 (C), 153.2 (C), 129.9 (CH), 107.5 (CH), 106.5 (CH), 101.3 (CH), 82.0 (C), 78.2 (C), 55.3 (CH_2), 55.2 (CH_3), 52.7 (CH_3). Known compound.[9]



Methyl (2*Z*,4*E*)-2-[2-(3-methoxyphenoxy)ethylidene]-4-methyl-3-oxohex-4-enoate (16n)

Alkyne **14f** was reacted with **15g** in accordance with General Procedure A giving the product **16n** after flash chromatography (silica gel, hexanes / dichloromethane / diethyl ether 5:5:1) as a slightly tan oil (85%). ***Z*-16n** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18 (t, J = 8.1 Hz, 1H), 6.64 (q, J = 6.6 Hz, 1H), 6.55-6.42 (m, 4H), 5.11 (d, J = 4.5 Hz, 1H), 3.77 (s, 3H), 3.76 (s, 3H), 1.86 (d, J = 6.6 Hz, 3H), 1.85 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 194.2 (C), 164.8 (C), 160.8 (C), 159.0 (C), 145.7 (CH), 143.4 (CH), 138.1 (C), 132.9 (C), 129.9 (CH), 106.7 (CH), 106.6 (CH), 101.2 (CH), 65.8 (CH_2), 55.2 (CH_3), 52.2 (CH_3), 15.0 (CH_3), 10.9 (CH_3). IR (KBr film, cm^{-1}): 3000, 2953, 2838, 1722, 1658, 1603, 1492, 1437, 1377, 1334, 1263, 1200, 1152. LRMS (FAB) *m/z* (%): 305 ($M+H^+$, 100), 273 ($M+H^+$ – MeOH, 60). HRMS (FAB) calcd for $C_{17}H_{21}O_5$ ($M+H^+$): 305.1389 Found: 305.1392. Storage of this material at room temperature for extended periods (3-5 days) results in partial isomerization to give some of the (*E,E*)-isomer. This mixture was separated and the (*E,E*)-isomer characterized spectroscopically. ***E*-16n**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19-7.12 (m, 2H), 6.63 (q, J = 6.6 Hz, 1H), 6.52 (dd, J = 8.1 Hz, J = 2.7 Hz, 1H), 6.41-6.35 (m, 2H), 4.56 (d, J = 5.4 Hz, 1H), 3.78 (s, 3H), 3.75 (s, 3H), 1.87 (s, 3H), 1.84 (d, J = 6.6 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 194.9 (C), 164.5 (C), 160.8 (C), 159.1 (C), 143.4 (CH), 140.9 (CH), 138.6 (C), 134.8 (C), 130.0 (CH), 106.9 (CH), 106.2 (CH), 101.1 (CH), 64.6 (CH_2), 55.3 (CH_3), 52.5 (CH_3), 15.1 (CH_3), 10.5 (CH_3). IR (KBr film, cm^{-1}): 2953, 1723, 1658, 1603, 1492, 1436, 1247, 1199, 1153.



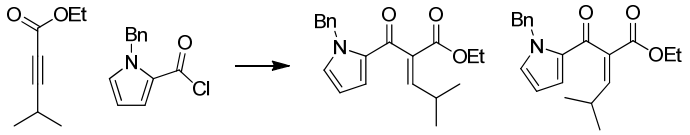
1-Benzyl-1*H*-pyrrole-2-carbonyl chloride (15i)

Trichloroacetyl chloride (4.3 mL, 38 mmol) was added slowly to a stirred solution of *N*-benzylpyrrole (5.0 g, 31.8 mmol) in dry diethyl ether (80 mL). The reaction was allowed to stir for 24 hours before being concentrated under reduced pressure.

[8] Tsutomu, I; Keiko, N.; Naoko, O.; Hisashi, I. *Heterocycles* **1994**, *39*, 371.
[9] Pastine, S. J.; Youn, S. W.; Sames, D. *Tetrahedron* **2003**, *59*, 8859.

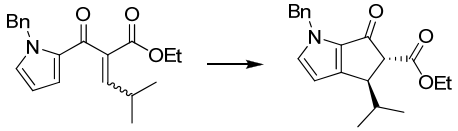
The solid residue was dissolved in ethanol (95%, 160 mL) and aqueous sodium hydroxide (4.0 M, 30 mL, 120 mmol) was added. This solution was refluxed for 5 hours before being reduced in volume to 40 mL under reduced pressure. Diethyl ether (60 mL) and distilled water (50 mL) were added and the aqueous phase was collected, the organic phase was extracted with more distilled water (25 mL) and the combined aqueous extracts were acidified to pH 2 with 5 M hydrochloric acid. This mixture was extracted with diethyl ether (2 × 30 mL) and the organic extracts were dried over magnesium sulphate and concentrated under reduced pressure to give the crude acid that was used without further purification.

Thionyl chloride (23.5 mL, 318 mmol) was added to the above crude acid in dry diethyl ether (160 mL) and the resultant solution was stirred for 4 hours. After this time the solution was concentrated under reduced pressure to give the title compound as a dark solid (6.28 g, 90%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.25 (m, 4H), 7.15-7.05 (m, 3H), 6.27 (dd, *J* = 4.4 Hz, 2.6 Hz, 1H), 5.43 (s, 2H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 157.2 (C), 136.7 (C), 133.8 (CH), 128.9 (CH), 128.1 (CH), 127.3 (CH), 127.2 (CH), 124.7 (C), 110.4 (CH), 52.9 (CH_2). Known compound.[10]



Ethyl (*Z*)/(*E*)-2-(1-benzyl-1*H*-pyrrole-2-carbonyl)-4-methylpent-2-enoate (16o).

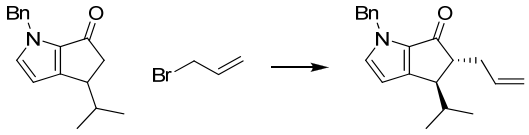
Alkyne **14g** was reacted with **15i** in accordance with General Procedure A giving the product **16o** after flash chromatography (silica gel, 9:1 hexane / diethyl ether) as a tan oil (82%). ***Z*-16o** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.20 (m, 3H), 7.15 (d, *J* = 7.2 Hz, 2H), 6.95 (dd, *J* = 2.6, 1.7 Hz, 1H), 6.82 (dd, *J* = 4.1, 1.7 Hz, 1H), 6.32 (d, *J* = 9.9 Hz, 1H), 6.18 (dd, *J* = 4.1, 2.6 Hz, 1H), 5.60 (s, 2H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.21 (dsept, *J* = 9.9, 6.6 Hz, 1H), 1.20 (t, *J* = 7.2 Hz, 3H), 1.10 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 182.8 (C), 165.6 (C), 154.4 (CH), 138.0 (C), 133.2 (C), 131.0 (CH), 130.0 (C), 128.5 (CH), 127.4 (CH), 127.2 (CH), 122.1 (CH), 108.8 (CH), 61.0 (CH_2), 52.3 (CH_2), 28.6 (CH), 22.0 (CH_3), 13.9 (CH_3). LRMS *m/z* (%): 668.6 (10, 2×M+NH_4^+), 343.2 (10, M+ NH_4^+), 326.2 (100, MH^+). HRMS calcd for $C_{20}H_{23}NNaO_3^+$: 348.1576. Found: 348.1575. IR (cm^{-1}): 3031, 2964, 1718, 1634, 1465, 1407, 1216, 1084, 712. This material isomerised to the *E*-isomer upon standing. ***E*-16o**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.23 (m, 3H), 7.13 (d, *J* = 6.3 Hz, 2H), 6.99 (dd, *J* = 2.4, 1.5 Hz, 1H), 6.79 (dd, *J* = 3.9, 1.5 Hz, 1H), 6.76 (d, *J* = 11.1 Hz, 1H), 6.18 (dd, *J* = 3.9, 2.4 Hz, 1H), 5.66 (s, 2H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.29 (dsept, *J* = 11.1, 6.6 Hz, 1H), 1.15 (t, *J* = 7.2 Hz, 3H), 0.93 (d, *J* = 6.6 Hz, 6H).



(±)-Ethyl *trans*-1-benzyl-4-isopropyl-6-oxo-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carboxylate (17o).

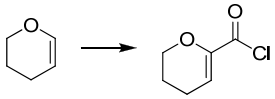
Methanesulfonic acid (1.10 mL, 16.6 mmol) was added dropwise to a stirred solution of **16o** (1.08 g, 3.31 mmol) in dichloromethane (13 mL) at room temperature and the mixture was stirred for 2 hours. After this time the acid was quenched by gradual addition of sodium bicarbonate solution (5% w/v, 60 mL). After stirring for 1 hour the mixture was diluted with dichloromethane (40 mL) and the organic phase was separated, the aqueous phase was then extracted with dichloromethane (2 × 20 mL). The combined organic extracts were dried over $MgSO_4$ and concentrated to give the title compound **17o** as a discoloured solid (1.08 g, 100%). Mp = 68-69 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.20 (m, 5H), 6.99 (d, *J* = 2.1 Hz, 1H), 6.10 (d, *J* = 2.1 Hz, 1H), 5.33-5.17 (m, 2H), 4.24 (q, *J* = 7.1 Hz, 2H), 3.57 (d, *J* = 2.4 Hz, 1H), 3.33 (dd, *J* = 6.3, 2.4 Hz, 1H), 1.93 (app. octet, J_{app} = 6.7 Hz, 1H), 1.30 (t, *J* = 7.1 Hz, 3H), 0.99 (d, *J* = 6.0 Hz, 3H), 0.97 (d, *J* = 5.1 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 184.1 (C), 170.3 (C), 153.9 (C), 136.8 (C), 134.7 (CH), 132.5 (C), 128.7 (CH), 127.9 (CH), 127.8 (CH), 106.4 (CH), 62.9 (CH), 61.1 (CH_2), 50.8 (CH_2), 45.4 (CH), 31.8 (CH), 20.1 (CH_3), 19.7 (CH_3), 14.1 (CH_3). LRMS *m/z* (%): 668.6 (25, 2×M+NH_4^+), 343.2 (10, M+ NH_4^+), 326.2 (100, MH^+). HRMS calcd for $C_{20}H_{24}NO_3^+$: 326.1756. Found: 326.1751. IR (cm^{-1}): 3034, 2980, 1719, 1680, 1407, 1243, 1157, 715.

[10] De Martino, G.; Scalzo, M.; Massa, S.; Giuliano, R. *Farmaco, Edizione Scientifica*, **1973**, *28*, 976



(±)-*trans*-5-Allyl-1-benzyl-4-isopropyl-4,5-dihydrocyclopenta[*b*]pyrrol-6(1*H*)-one (24)

Lithium diisopropylamide (0.5 M in THF / cyclohexane, 0.59 mL, 0.30 mmol) was added slowly to a stirred solution of **23** (57.8 mg, 0.228 mmol) in tetrahydrofuran (2 mL) at −78 °C and the solution was then allowed to warm to room temperature. This solution was then re-cooled to −78 °C and allyl bromide (150 μL, 1.72 mmol) was added. The solution was then allowed to come to room temperature and was stirred for 4 hours. After this time the reaction was taken up in diethyl ether (20 mL) and distilled water (40 mL). The organic phase was separated and the aqueous phase was re-extracted with diethyl ether (2 × 10 mL). The combined organic extracts were dried over $MgSO_4$ and concentrated onto silica (1 g). Flash chromatography (silica gel, 91:9 hexane / diethyl ether) gave the title compound as a viscous oil (55.0 mg, 82%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.20 (m, 5H), 6.98 (d, J = 1.8 Hz, 1H), 6.05 (d, J = 1.8 Hz, 1H), 5.77 (m_c, 1H), 5.33-5.21 (m, 2H), 5.09 (dd, J = 17.1, 1.6 Hz, 1H), 5.00 (dd, J = 9.9, 1.6 Hz, 1H), 2.75 (dd, J = 5.6, 2.0 Hz, 1H), 2.67-2.52 (m, 2H), 2.43 (dt, J = 13.8, 7.5 Hz, 1H), 1.87 (app. octet, J_{app} = 6.5 Hz, 1H), 0.99 (d, J = 6.6 Hz, 3H), 0.88 (d, J = 6.6 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 192.7 (C), 153.0 (C), 138.6 (CH), 137.3 (C), 135.7 (CH), 133.8 (CH), 133.4 (C), 128.7 (CH), 127.8 (CH), 127.6 (CH), 116.8 (CH_2), 106.3 (CH), 56.6 (CH), 50.7 (CH_2), 45.7 (CH), 36.5 (CH_2), 31.6 (CH), 20.8 (CH_3), 19.1 (CH_3). LRMS *m/z* (%): 604.6 (10, 2×M+NH_4^+), 587.5 (25, 2×M+H^+), 294.2.2 (100, MH^+). HRMS calcd for $C_{20}H_{24}NO^+$: 294.1858. Found: 294.1856. IR (cm^{-1}): 3068, 2958, 1672, 1509, 1412, 1067, 913, 722.



3,4-Dihydro-2*H*-pyran-6-carbonyl chloride (15j)

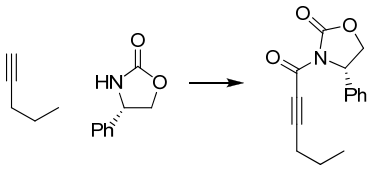
t-Butyllithium (1.70 M in pentane, 15.0 mL, 25.5 mmol) was added dropwise to a stirred solution of dihydropyran (**21**) (2.90 mL, 30.6 mmol) in tetrahydrofuran (40 mL) at −78 °C. After addition was complete the resultant solution was allowed to warm to room temperature before being cooled again to −78 °C. The nitrogen was turned off and carbon dioxide was bubbled slowly through the solution while it was warmed to 0ºC (ice bath) over the course of 30 minutes. The carbon dioxide supply was then removed and the reaction mixture was partitioned between diethyl ether (60 mL) and distilled water (60 mL). The aqueous phase was collected and the organic phase was re-extracted with distilled water (60 mL). The combined aqueous extracts were acidified to pH 2 with 2 M hydrochloric acid. This mixture was extracted with ethyl acetate (4 × 30 mL) and the organic extracts were dried over magnesium sulphate and concentrated under reduced pressure to give 1.74 g of crude acid **22** that was used without further purification.

Crude acid **22** was dissolved in dry dichloromethane (25 mL) and to it was added thionyl chloride (7.0 mL, 95 mmol). After stirring for 24 hours the reaction mixture concentrated under reduced pressure to give the title compound **15j** as a tan oil (1.34 g, 36%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.43 (t, J = 4.4 Hz, 1H), 4.10 (t, J = 5.1 Hz, 2H), 2.27 (td, J = 6.4, 4.4 Hz, 2H), 1.85 (tt, J = 6.4, 5.1 Hz, 2H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 163.6 (C), 146.7 (C), 119.4 (CH), 67.1 (CH_2), 21.4 (CH_2), 20.8 (CH_2). IR (cm^{-1}): 2939, 1755, 1626, 1168, 1055, 934, 746.

General Procedure B: Preparation of *N*-Alkynoyloxazolidinones 29.

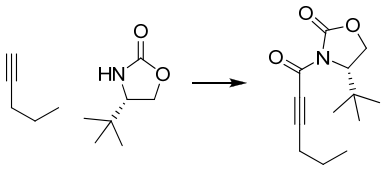
n-Butyllithium (1.82 M in cyclohexane, 5.5 mL, 10.0 mmol) was added dropwise to a stirred solution of alkyne **26** (12.0 mmol) in tetrahydrofuran (30 mL) at −78 °C under nitrogen. After stirring for 30 minutes the nitrogen was turned off and carbon dioxide was bubbled slowly through the solution while it was warmed to 0°C (ice bath) over the course of 30 minutes. The carbon dioxide supply was then removed and replaced with the nitrogen atmosphere. The solution was again cooled to −78 °C and pivaloyl chloride (1.24 mL, 10.0 mmol) was added. The solution was then allowed to warm to room temperature and was stirred for 3 hours. The solution was then cooled again to −78 °C and to it was added *via* cannula a solution of lithiated oxazolidinone (10.0 mmol, generated by addition of 5.5 mL of 1.82 M butyllithium solution to 1.63 g of oxazolidinone **28** in 60 mL of tetrahydrofuran) at −78 °C. The solution was then allowed to warm to room temperature and stirred for 2 hours. After this time the solution was concentrated to a volume of 20 mL under vacuum. Ethyl acetate (50 mL) and distilled water (50 mL) were then added and the organic phase was separated, the aqueous phase was re-extracted with

ethyl acetate (30 mL) and the combined organic extracts were dried over magnesium sulphate and concentrated onto silica gel (5 g) under reduced pressure. The solid residue was subjected to flash chromatography.



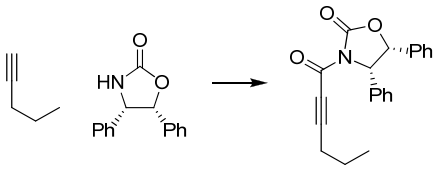
(*S*)-3-Hex-2-ynoyl-4-phenyloxazolidin-2-one (29a)

This material was prepared from alkyne **26a** and oxazolidinone **28a** in accordance with General Procedure B. After flash chromatography (silica gel, 5:5:1 hexane / dichloromethane / diethyl ether) the title compound was obtained as a viscous oil (65%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.27 (m, 5H), 5.43 (dd, J = 8.7, 3.6 Hz, 1H), 4.68 (app. t, J_{app} = 8.7 Hz, 1H), 4.28 (dd, J = 8.7, 3.6 Hz, 1H), 2.41 (t, J = 7.1 Hz, 2H), 1.65 (app. sext, J_{app} = 7.2 Hz, 2H), 1.04 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 152.2 (C), 150.1 (C), 138.6 (C), 129.0 (CH), 128.5 (CH), 125.8 (CH), 98.3 (C), 73.8 (C), 69.8 (CH_2), 57.4 (CH), 21.0 (CH_2), 20.8 (CH_2), 13.3 (CH_3). LRMS *m/z* (%): 537.4 (20), 275.2 (70, M+NH_4^+), 258.2 (100, MH^+). HRMS calcd for $C_{15}H_{16}NO_3^+$: 258.1130. Found: 258.1128. IR (cm^{-1}): 2967, 2238, 2221, 1788, 1662, 1322, 1195, 1088, 1039.



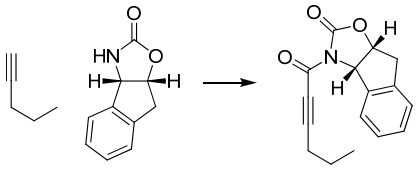
(*S*)-4-*tert*-Butyl-3-hex-2-ynoyloxazolidin-2-one (29b)

This material was prepared from alkyne **26a** and oxazolidinone **28b** in accordance with General Procedure B. After flash chromatography (silica gel, 5:5:1 hexane / dichloromethane / diethyl ether) the title compound was obtained as a viscous oil (43%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.40 (dd, J = 7.4, 1.7 Hz, 1H), 4.29 (dd, J = 9.2, 1.7 Hz, 1H), 4.23 (dd, J = 9.2, 7.4 Hz, 1H), 2.43 (t, J = 7.1 Hz, 2H), 1.67 (app. sext, J_{app} = 7.2 Hz, 2H), 1.06 (t, J = 7.4 Hz, 3H), 0.96 (s, 9H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 153.0 (C), 151.0 (C), 98.5 (C), 73.4 (C), 65.1 (CH_2), 61.2 (CH), 35.7 (C), 25.4 (CH_3), 21.1 (CH_2), 20.9 (CH_2), 13.3 (CH_3). LRMS *m/z* (%): 255.3 (65, M+NH_4^+), 238.2 (100, MH^+). HRMS calcd for $C_{13}H_{19}NNaO_3^+$: 260.1263. Found: 260.1252. IR (cm^{-1}): 2965, 2241, 2219, 1790, 1666, 1324, 1295, 1183, 1090.



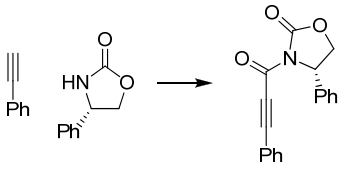
(4*S*,5*R*)-3-Hex-2-ynoyl-4,5-diphenyloxazolidin-2-one (29c)

This material was prepared from alkyne **26a** and oxazolidinone **28c** in accordance with General Procedure B. After flash chromatography (silica gel, 5:5:1 hexane / dichloromethane / diethyl ether) the title compound was obtained as a white solid (43%). Mp = 131-133 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16-7.07 (m, 6H), 7.01-6.94 (m, 2H), 6.91-6.84 (m, 2H), 5.89 (d, J = 7.5 Hz, 1H), 5.67 (d, J = 7.5 Hz, 1H), 2.44 (t, J = 7.1 Hz, 2H), 1.68 (app. sext, J_{app} = 7.3 Hz, 2H), 1.06 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 152.2 (C), 150.0 (C), 133.9 (C), 132.7 (C), 128.4 (CH), 128.23 (CH), 128.18 (CH), 128.0 (CH), 126.6 (CH), 126.1 (CH), 98.8 (C), 80.1 (CH), 73.8 (C), 62.7 (CH), 21.2 (CH_2), 20.9 (CH_2), 13.4 (CH_3). LRMS *m/z* (%): 684.5 (50, 2×M+NH_4^+), 351.3 (60, M+NH_4^+), 334.2 (100, MH^+). HRMS calcd for $C_{21}H_{19}NNaO_3^+$: 356.1263. Found: 356.1257. IR (cm^{-1}): 3035, 2966, 2262, 2214, 1771, 1663, 1362, 1341, 1315, 1192, 1022, 729.



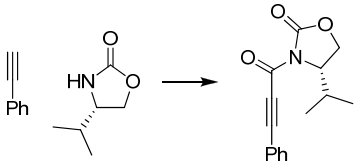
(3a*S*,8a*R*)-3-Hex-2-ynoyl-3,3a,8,8a-tetrahydro-2*H*-indeno[1,2-*d*]oxazol-2-one (29d)

This material was prepared from alkyne **26a** and oxazolidinone **28d** in accordance with General Procedure B. After flash chromatography (silica gel, 5:5:1 hexane / dichloromethane / diethyl ether) the title compound was obtained as a viscous oil (43%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 (d, J = 7.8 Hz, 1H), 7.39-7.23 (m, 3H), 5.93 (d, J = 6.9 Hz, 1H), 5.28 (ddd, J = 6.9, 4.2, 3.0 Hz, 1H), 3.40 (app. d, J_{app} = 3.0 Hz, 2H), 2.43 (t, J = 7.1 Hz, 2H), 1.66 (app. sext, J_{app} = 7.3 Hz, 2H), 1.05 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 151.4 (C), 151.1 (C), 139.3 (C), 138.5 (C), 129.9 (CH), 128.1 (CH), 127.2 (CH), 125.1 (CH), 99.2 (C), 77.9 (CH), 73.5 (C), 62.9 (CH), 37.9 (CH_2), 21.2 (CH_2), 20.9 (CH_2), 13.3 (CH_3). LRMS *m/z* (%): 556.4 (10, 2×M+NH_4^+), 287.3 (70, M+NH_4^+), 270.2 (100, MH^+). HRMS calcd for $C_{16}H_{15}NNaO_3^+$: 292.0950. Found: 292.0944. IR (cm^{-1}): 2966, 2250, 2224, 1787, 1656, 1360, 1324, 1189, 1101, 1032, 753.



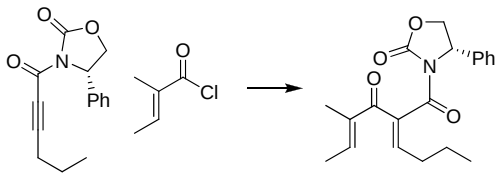
(*S*)-4-Phenyl-3-(3-phenylpropioloyl)oxazolidin-2-one (29e)

This material was prepared from alkyne **26b** and oxazolidinone **28a** in accordance with General Procedure B. After flash chromatography (silica gel, 4:1 hexane / ethyl acetate) the title compound was obtained as a white solid (41%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 (d, J = 7.2 Hz, 2H), 7.49-7.30 (m, 8H), 5.50 (dd, J = 8.7, 3.6 Hz, 1H), 4.73 (app. t, J_{app} = 8.7 Hz, 1H), 4.33 (dd, J = 8.7, 3.6 Hz, 1H). Known compound.[11]



(*S*)-4-Isopropyl-3-(3-phenylpropioloyl)oxazolidin-2-one (29f)

This material was prepared from alkyne **26b** and oxazolidinone **28e** in accordance with General Procedure B. After flash chromatography (silica gel, 83:17 hexanes / ethyl acetate) the title compound was obtained as a white solid (48%). Mp = 91-93 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, J = 7.2 Hz, 2H), 7.49-7.33 (m, 3H), 4.48 (ddd, J = 8.1, 4.2, 3.0 Hz, 1H), 4.30 (app. t, J_{app} = 8.6 Hz, 1H), 4.23 (dd, J = 9.0, 3.0 Hz, 1H), 2.44 (sept.d, J = 6.9, 4.2 Hz, 1H), 0.95 (d, J = 6.9 Hz, 3H), 0.92 (d, J = 6.9 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 152.6 (C), 150.9 (C), 133.3 (CH), 131.0 (CH), 128.6 (CH), 119.9 (C), 94.5 (C), 81.3 (C), 63.4 (CH_2), 58.7 (CH), 28.6 (CH), 18.0 (CH_3), 14.8 (CH_3). LRMS *m/z* (%): 275.2 (45, M+NH_4^+), 258.2 (100, MH^+). HRMS calcd for $C_{15}H_{16}NO_3^+$: 258.1130. Found: 258.1124. IR (cm^{-1}): 2961, 2214, 1778, 1658, 1365, 1322, 1200, 762.

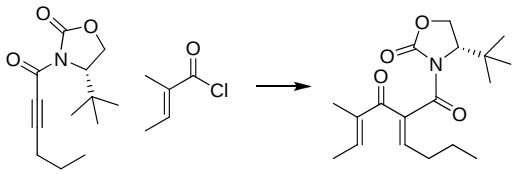


(2*Z*,4*E*)-2-Butylidene-4-methyl-1-[(*S*)-2-oxo-4-phenyloxazolidin-3-yl]hex-4-ene-1,3-dione (30a)

Alkyne **29a** was reacted with acid chloride **15g** in accordance with General Procedure A giving the product **30a** after flash chromatography (silica gel, 10:10:1, hexane / dichloromethane / diethyl ether) as a white crystalline solid (99%). Mp = 104-105 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.30 (m, 5H), 6.62 (t, J = 7.7 Hz, 1H), 6.52 (q, J = 6.6 Hz, 1H), 5.52 (dd, J = 8.9, 4.1 Hz, 1H), 4.73 (app. t, J_{app} = 8.9 Hz, 1H), 4.27 (dd, J = 8.7, 4.1 Hz, 1H), 2.13 (app. q, J_{app} = 7.4 Hz, 2H), 1.86 (s, 3H), 1.82

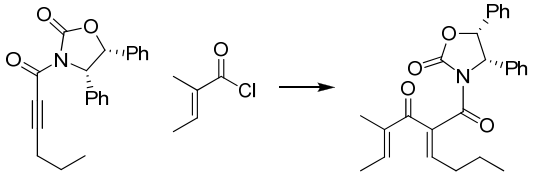
[11] Fonquerna, S.; Moyano, A.; Pericas, M.A.; Riera, A. *Tetrahedron: Asymmetry*, **1997**, *8*, 1685.

(d, J = 6.6 Hz, 3H), 1.47 (app. sext, J_{app} = 7.4 Hz, 2H), 0.89 (t, J = 7.2 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 194.8 (C), 165.7 (C), 153.1 (C), 148.7 (CH), 138.6 (C), 137.5 (CH), 137.3 (C), 135.5 (C), 129.0 (CH), 128.5 (CH), 126.0 (CH), 70.4 (CH_2), 57.4 (CH), 31.7 (CH_2), 21.7 (CH_2), 14.3 (CH_3), 13.7 (CH_3), 12.6 (CH_3). LRMS *m/z* (%): 700.4 (20, 2×M+NH_4^+), 359.3 (20, M+NH_4^+), 342.2 (100, MH^+), 179 (15). HRMS calcd for $C_{20}H_{24}NO_4^+$: 342.1705. Found: 342.1704. IR (cm^{-1}): 2924, 1779, 1718, 1693, 1489, 1332, 1279, 1197, 1023, 699.



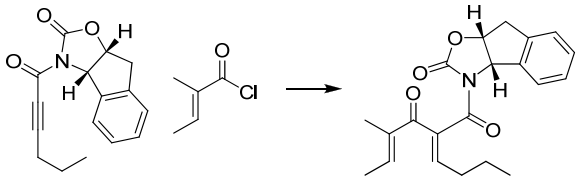
(2*Z*,4*E*)-1-[(*S*)-4-*tert*-Butyl-2-oxo-oxazolidin-3-yl]-2-butylidene-4-methylhex-4-ene-1,3-dione (30b)

Alkyne **29b** was reacted with acid chloride **15g** in accordance with General Procedure A giving the product **30b** after flash chromatography (silica gel, 22:3 hexane / ethyl acetate) as a thick oil (81%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.60 (t, J = 7.8 Hz, 1H), 6.55 (m_c, 1H) 4.48 (dd, J = 6.3, 3.0 Hz, 1H), 4.33-4.26 (m, 2H), 2.31-2.19 (m, 2H), 1.88-1.82 (m, 6H), 1.54 (app. sext, J_{app} = 7.3 Hz, 2H), 1.02 (s, 9H), 0.96 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 195.2 (C), 166.3 (C), 154.1 (C), 147.9 (CH), 137.8 (C), 137.5 (CH), 135.2 (C), 65.6 (CH_2), 61.1 (CH), 35.9 (C), 32.0 (CH_2), 25.5 (CH_3), 21.7 (CH_2), 14.2 (CH_3), 13.7 (CH_3), 12.5 (CH_3). LRMS *m/z* (%): 660.7 (20, 2×M+NH_4^+), 339.3 (25, M+NH_4^+), 322.2 (100, MH^+). HRMS calcd for $C_{18}H_{27}NNaO_4^+$: 344.1838. Found: 344.1830. IR (cm^{-1}): 2964, 1780, 1699, 1370, 1190, 1114.



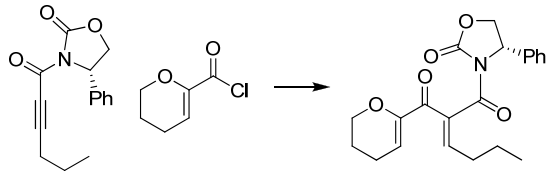
(2*Z*,4*E*)-2-Butylidene-4-methyl-1-[(4*S*,5*R*)-2-oxo-4,5-diphenyloxazolidin-3-yl]hex-4-ene-1,3-dione (30c)

Alkyne **29c** was reacted with acid chloride **15g** in accordance with General Procedure A giving the product **30c** after flash chromatography (silica gel, 87:13 hexane / ethyl acetate) as a tan solid (86%). Mp = 52-54 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.15-7.07 (m, 6H), 6.99-6.92 (m, 4H), 6.68 (t, J = 7.8 Hz, 1H), 6.59 (q, J = 6.9, Hz, 1H), 5.95 (d, J = 7.7 Hz, 1H), 5.77 (d, J = 7.7 Hz, 1H), 2.19 (app. q, J_{app} = 7.6 Hz, 2H), 1.91 (s, 3H), 1.87 (d, J = 6.9 Hz, 3H), 1.52 (app. sext, J_{app} = 7.4 Hz, 2H), 0.93 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 195.0 (C), 165.6 (C), 153.1 (C), 148.8 (CH), 137.7 (CH), 137.6 (C), 135.5 (C), 134.2 (C), 132.9 (C), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 126.8 (CH), 126.2 (CH), 80.7 (CH), 62.5 (CH), 31.9 (CH_2), 21.8 (CH_2), 14.3 (CH_3), 13.8 (CH_3), 12.7 (CH_3). LRMS *m/z* (%): 852.3 (15, 2×M+NH_4^+), 435.3 (40, M+NH_4^+), 418.5 (100, MH^+). HRMS calcd for $C_{26}H_{27}NNaO_4^+$: 440.1838. Found: 440.1830. IR (cm^{-1}): 3036, 2961, 1782, 1694, 1633, 1343, 1187, 697.



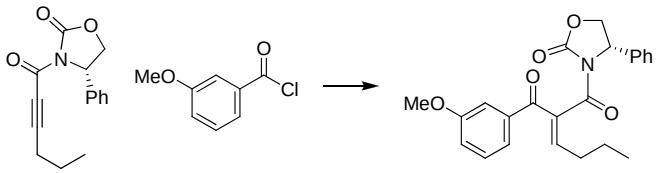
(2*Z*,4*E*)-2-Butylidene-4-methyl-1-[(3a*S*,8a*R*)-2-oxo-*2H*-indeno[1,2-*d*]oxazol-3(3a*H*,8*H*,8a*H*)-yl]hex-4-ene-1,3-dione (30d)

Alkyne **29d** was reacted with acid chloride **15g** in accordance with General Procedure A giving the product **30b** after flash chromatography (silica gel, 21:4 hexane / ethyl acetate) as a thick oil (86%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, J = 7.2 Hz, 1H), 7.39-7.26 (m, 3H), 6.64, (t, J = 7.8 Hz, 1H), 6.50 (q, J = 6.8 Hz, 1H), 6.03 (d, J = 6.9 Hz, 1H), 5.32 (m_c, 1H), 3.47-3.31 (m, 2H), 2.29-2.19 (m, 2H), 1.82 (s, 3H), 1.78 (d, J = 6.8 Hz, 3H), 1.58-1.43 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 194.6 (C), 166.8 (C), 152.4 (C), 148.4 (CH), 139.5 (C), 139.0 (C), 137.8 (CH), 137.5 (C), 135.6 (C), 129.9 (CH), 128.2 (CH), 127.3 (CH), 125.3 (CH), 78.8 (CH), 63.0 (CH), 38.0 (CH_2), 32.0 (CH_2), 21.8 (CH_2), 14.4 (CH_3), 13.9 (CH_3), 12.6 (CH_3). LRMS *m/z* (%): 724.5 (30, 2×M+NH_4^+), 371.3 (15, M+NH_4^+), 354.2 (100, MH^+). HRMS calcd for $C_{21}H_{23}NNaO_4^+$: 376.1525. Found: 376.1518. IR (cm^{-1}): 2962, 1778, 1687, 1633, 1362, 1282, 1191, 1040, 756.



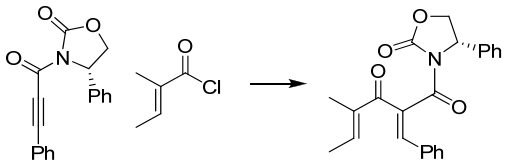
(*S*,*Z*)-2-Butylidene-1-(3,4-dihydro-2*H*-pyran-6-yl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30e)

Alkyne **29a** was reacted with acid chloride **15j** in accordance with General Procedure A giving the product **30e** after flash chromatography (silica gel, 7:3 hexanes / ethyl acetate) as a tan oil (70%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48-7.30 (m, 5H), 7.00 (t, J = 8.0 Hz, 1H), 5.96 (t, J = 4.1 Hz, 1H), 5.51 (dd, J = 8.7, 4.2 Hz, 1H), 4.72 (app. t, J_{app} = 8.7 Hz, 1H), 4.31 (dd, J = 9.0, 4.2 Hz, 1H), 4.00-3.90 (m, 2H), 2.24-2.06 (m, 4H), 1.88−1.80 (m, 2H), 1.47 (app. sext, J_{app} = 7.4 Hz, 2H), 0.88 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 184.7 (C), 165.2 (C), 153.1 (C), 150.3 (C), 149.4 (CH), 138.7 (C), 135.7 (C), 128.9 (CH), 128.5 (CH), 126.2 (CH), 111.8 (CH), 70.3 (CH_2), 66.1 (CH_2), 57.2 (CH), 31.6 (CH_2), 21.6 (CH_2), 21.4 (CH_2), 20.5 (CH_2), 13.7 (CH_3). LRMS *m/z* (%): 756.4 (10, 2×M+ NH_4^+), 387.4 (60, M+NH_4^+), 370.3 (100, MH^+), 164.0 (80). HRMS calcd for $C_{21}H_{24}NNaO_5^+$: 392.1474. Found: 392.1473. IR (cm^{-1}): 2962, 1777, 1696, 1385, 1325, 1202, 1038, 733, 700.



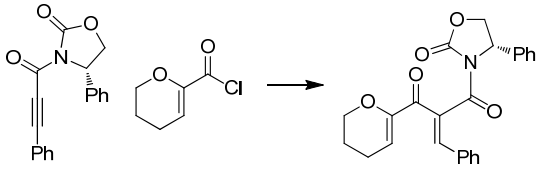
(*S*,*Z*)-2-Butylidene-1-(3-methoxyphenyl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30f)

Alkyne **29a** was reacted with acid chloride **15k** in accordance with General Procedure A giving the product **30f** after flash chromatography (silica gel, 25:25:2 hexane / dichloromethane / diethyl ether as a thick tan oil (91%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.30 (m, 8H), 7.08 (m_c, 1H), 6.65 (t, J = 7.8 Hz, 1H), 5.57 (dd, J = 8.9, 4.4 Hz, 1H), 4.75 (app. t, J_{app} = 8.9 Hz, 1H), 4.28 (dd, J = 8.9, 4.4 Hz, 1H), 3.83 (s, 3H), 2.20 (app. q, J_{app} = 7.5 Hz, 2H), 1.47 (app. sext, J_{app} = 7.4 Hz, 2H), 0.90 (t, J = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 193.4 (C), 165.2 (C), 159.4 (C), 153.4 (C), 150.6 (CH), 138.7 (C), 138.3 (C), 137.9 (C), 129.2 (CH), 129.0 (CH), 128.5 (CH), 126.0 (CH), 122.1 (CH), 118.3 (CH), 114.2 (CH), 70.6 (CH_2), 57.3 (CH), 55.2 (CH_3), 31.8 (CH_2), 21.7 (CH_2), 13.7 (CH_3). LRMS *m/z* (%): 804.5 (20, 2×M+NH_4^+), 411.4 (40, M+NH_4^+), 394.4 (100, MH^+). HRMS calcd for $C_{23}H_{24}NO_5^+$: 394.1654. Found: 394.1661. IR (cm^{-1}): 3034, 2962, 1779, 1696, 1649, 1375, 1285, 1204, 1041, 733, 700.



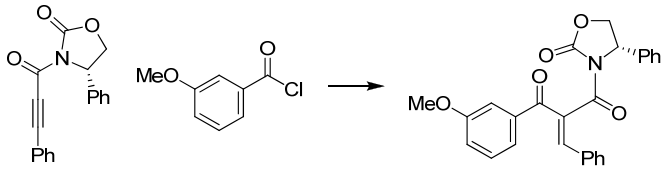
(2*Z*,4*E*)-2-Benzylidene-4-methyl-1-[(*S*)-2-oxo-4-phenyloxazolidin-3-yl]hex-4-ene-1,3-dione (30g)

Alkyne **29e** was reacted with acid chloride **15g** in accordance with General Procedure A giving the product **30g** after flash chromatography (silica gel, 5:5:1 hexanes / dichloromethane / diethyl ether) as a white solid (88%). Mp = 116-117 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (br. s, 5H), 7.35 (s, 1H), 7.27 (m_c, 1H), 7.12 (br. s, 4H), 6.65 (q, J = 6.6 Hz, 1H), 5.55 (dd, J = 8.4 Hz, J = 3.3 Hz, 1H), 4.71 (t, J = 8.4 Hz, 1H), 4.33 (dd, J = 8.4 Hz, J = 3.3 Hz, 1H), 1.91 (s, 3H), 1.75 (d, J = 6.6 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 195.4 (C), 166.2 (C), 152.8 (C), 143.0 (CH), 138.4 (CH), 138.1 (C), 135.4 (C), 135.0 (C), 133.0 (C), 130.0 (CH),129.3 (CH), 129.0 (CH), 128.7 (CH), 128.6 (CH), 126.6 (CH), 70.4 (CH_2), 57.4 (CH), 14.5 (CH_3), 12.8 (CH_3). IR (KBr disc, cm^{-1}): 3043, 2913, 1783, 1699, 1637, 1622, 1393, 1368, 1333, 1282, 1262, 1222, 1120, 1064. LRMS (70 eV) *m/z* (%): 375 ($M^{+\cdot}$, 20), 292 (25), 212 (100). HRMS (FAB) calcd for $C_{23}H_{22}NO_4$ (M+H^+): 376.1549. Found: 376.1552. A NOSEY 2D NMR experiment was performed and is provided in the Supplementary Information.



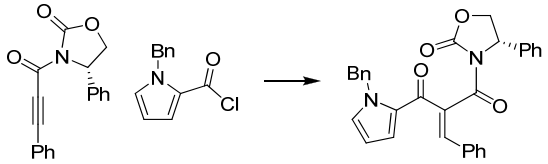
(*S*,*Z*)-2-Benzylidene-1-(3,4-dihydro-2*H*-pyran-6-yl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30h)

Alkyne **29e** was reacted with acid chloride **15j** in accordance with General Procedure A giving the product **30j** after flash chromatography (silica gel, 13:7 hexanes / ethyl acetate) as a white solid (69%). Mp = 70-72 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (s, 1H), 7.43-7.10 (m, 10H), 6.04 (t, J = 4.1 Hz, 1H), 5.50 (dd, J = 8.6, 3.5 Hz, 1H), 4.62 (app. t, J_{app} = 8.7 Hz, 1H), 4.33 (dd, J = 9.0, 3.5 Hz, 1H), 3.97 (br. s, 2H), 2.27-2.19 (m, 2H), 1.92-1.81 (m, 2H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 184.9 (C), 166.0 (C), 152.8 (C), 150.5 (C), 143.5 (CH), 138.2 (C), 133.3 (C), 133.2 (C), 130.1 (CH), 129.5 (CH), 129.0 (CH), 128.64 (CH), 128.55 (CH), 126.8 (CH), 112.4 (CH), 70.3 (CH_2), 66.2 (CH_2), 57.4 (CH), 21.5 (CH_2), 20.6 (CH_2). LRMS *m/z* (%): 824.4 (10, 2×M+NH_4^+), 421.2 (35, M+NH_4^+), 404.2 (100, MH^+). HRMS calcd for $C_{24}H_{21}NNaO_5^+$: 426.1317. Found: 426.1316. IR (cm^{-1}): 2926, 1779, 1692, 1619, 1260, 1200, 756, 696.



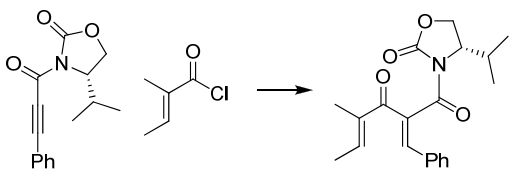
(*S*,*Z*)-2-Benzylidene-1-(3-methoxyphenyl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30i)

Alkyne **29e** was reacted with acid chloride **15k** in accordance with General Procedure A giving the product **30i** after flash chromatography (silica gel, 10:10:1 hexane / dichloromethane / diethyl ether) as a white solid (79%). Mp = 146-148 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.35 (m, 9H), 7.29 (m_c, 1H), 7.22-7.00 (m, 5H), 5.62 (dd, J = 8.7, 3.5 Hz, 1H), 4.76 (app. t, J_{app} = 8.7 Hz, 1H), 4.37 (dd, J = 8.7, 3.5 Hz, 1H), 3.85 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 194.1 (C), 165.8 (C), 159.5 (C), 153.1 (C), 144.8 (CH), 138.4 (C), 138.2 (C), 135.4 (C), 132.7 (C), 130.5 (CH), 129.6 (CH), 129.4 (CH), 129.1 (CH), 128.7 (CH), 126.7 (CH), 122.2 (CH), 118.5 (CH), 114.2 (CH), 70.6 (CH_2), 57.4 (CH), 55.4 (CH_3). LRMS *m/z* (%): 872.4 (30, 2×M+NH_4^+), 445.3 (55, M+NH_4^+), 428.3 (100, MH^+). HRMS calcd for $C_{26}H_{21}NNaO_5^+$: 450.1317. Found: 450.1316. IR (cm^{-1}): 2927, 1777, 1695, 1649, 1577, 1319, 1288, 1215, 1047, 692.



(*S*,*Z*)-1-(1-Benzyl-1*H*-pyrrol-2-yl)-2-benzylidene-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30j)

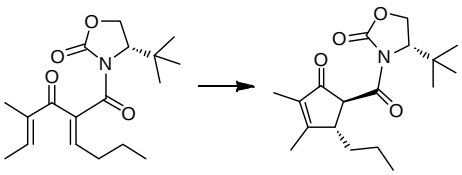
Alkyne **29e** was reacted with acid chloride **15i** in accordance with General Procedure A giving the product **38** after flash chromatography (silica gel, 7:3 hexanes / ethyl acetate) as a yellow solid (72%). Mp = 77-81 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (s, 1H), 7.45-7.00 (m, 16H), 6.95 (s, 1H), 6.21 (s, 1H), 5.62-5.42 (m, 3H), 4.67 (app. t, J_{app} = 8.6 Hz, 1H), 5.30 (m_c, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.9 (C), 165.8 (C), 152.4 (C), 141.8 (CH), 137.9 (C), 137.8 (C), 136.2 (C), 132.8 (C), 130.4 (CH), 129.6 (CH), 129.0 (CH), 128.6 (CH), 128.2 (CH, 3 overlapping resonances), 127.1 (CH), 127.0 (CH), 126.4 (CH), 122.1 (CH), 108.6 (CH), 70.0 (CH_2), 57.0 (CH), 51.5 (CH_2). HRMS calcd for $C_{30}H_{25}N_2O_4^+$: 477.1809. Found: 477.1827.



(2*Z*,4*E*)-2-Benzylidene-1-[(*S*)-4-isopropyl-2-oxooxazolidin-3-yl]-4-methylhex-4-ene-1,3-dione (30k)

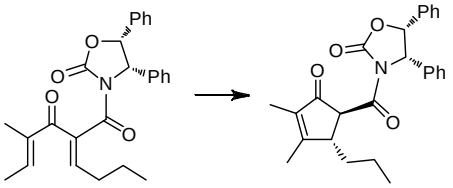
Alkyne **29f** was reacted with acid chloride **15g** in accordance with General Procedure A giving the product **30j** after flash chromatography (silica gel, 83:17 hexanes / ethyl acetate) as a white solid (92%). Mp = 117-119 °C. ^{1}H NMR (300 MHz,

$CDCl_3$) δ 7.41-7.32 (m, 6H), 6.67 (q, *J* = 6.8 Hz, 1H), 4.53 (ddd, *J* = 8.1, 3.8, 3.3 Hz, 1H), 4.28 (app. t, J_{app} = 8.6 Hz, 1H), 4.21 (dd, *J* = 9.0, 3.3 Hz, 1H), 2.51 (sept.d, *J* = 6.8, 3.8 Hz, 1H), 1.91 (s, 3H), 1.89 (d, *J* = 6.8 Hz, 3H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.91 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 195.3 (C), 166.8 (C), 153.4 (C), 142.5 (CH), 138.3 (CH), 136.0 (C), 135.5 (C), 133.6 (C), 130.1 (CH), 129.2 (CH), 128.8 (CH), 63.9 (CH_2), 58.5 (CH), 28.5 (CH), 17.8 (CH_3), 14.7 (CH_3), 14.4 (CH_3), 12.8 (CH_3). LRMS *m/z* (%): 700.6 (20, 2×M+NH_4^+), 359.4 (15, M+NH_4^+), 342.4 (100, MH^+). HRMS calcd for $C_{20}H_{23}NNaO_4^+$: 364.1525. Found: 364.1520. IR (cm^{-1}): 2964, 1776, 1687, 1612, 1381, 1282, 1209, 1146, 1109, 763, 694.



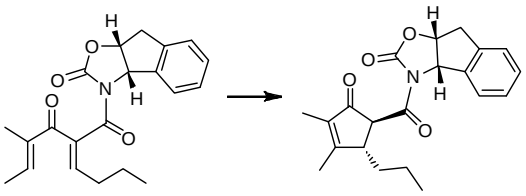
(*S*)-4-*tert*-Butyl-3-[(1*S*,5*S*)-3,4-dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]oxazolidin-2-one (31b*C*)

Conditions A: The reaction was performed as described for **31a*C*** using **30b** giving **31bA** and **31b*C*** after flash chromatography (silica gel, 50:50:6 hexane / dichloromethane / diethyl ether). Major isomer **31b*C*** clear oil (74%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.87 (d, *J* = 3.0 Hz, 1H), 4.37-4.29 (m, 3H), 3.29 (m_c, 1H), 2.05 (s, 3H), 1.85 (m_c, 1H), 1.67 (s, 3H), 1.42-1.29 (m, 3H), 0.96 (s, 9H), 0.96 (t, *J* = 6.6 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 201.4 (C), 173.3 (C), 169.1 (C), 155.1 (C), 133.7 (C), 65.2 (CH_2), 62.3 (CH), 55.7 (CH), 46.4 (CH), 35.5 (C), 34.0 (CH_2), 25.7 (CH_3), 20.6 (CH_2), 15.0 (CH_3), 13.9 (CH_3), 8.1 (CH_3). LRMS *m/z* (%): 660.5 (30, 2×M+NH_4^+), 339.3 (15, M+NH_4^+), 322.3 (100, MH^+). HRMS calcd for $C_{18}H_{27}NNaO_4^+$: 344.1838. Found: 344.1828. IR (cm^{-1}): 2962, 1778, 1690, 1643, 1365, 1182, 1053, 706. Minor isomer **31bA** clear oil (19%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.98 (br s, 1H), 4.52 (dd, *J* = 7.7, 1.9 Hz, 1H), 4.33 (dd, *J* = 9.3, 1.9 Hz, 1H), 4.27 (dd, *J* = 9.3, 7.7 Hz, 1H), 3.28 (m_c, 1H), 2.04 (s, 3H), 1.77 (m_c, 1H), 1.69 (s, 3H), 1.36-1.23 (m, 3H), 0.99-0.90 (m, 12H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 200.7 (C), 172.7 (C), 169.5 (C), 154.3 (C), 133.9 (C), 65.0 (CH_2), 61.2 (CH), 56.4 (CH), 45.9 (CH), 36.2 (C), 33.9 (CH_2), 25.4 (CH_3), 20.4 (CH_2), 15.0 (CH_3), 14.0 (CH_3), 8.2 (CH_3). LRMS *m/z* (%): 660.5 (20, 2×M+NH_4^+), 339.2 (10, M+NH_4^+), 322.3 (100, MH^+). HRMS calcd for $C_{18}H_{27}NNaO_4^+$: 344.1838. Found: 344.1832. IR (cm^{-1}): 2961, 1775, 1690, 1646, 1323, 1184, 1056, 706.



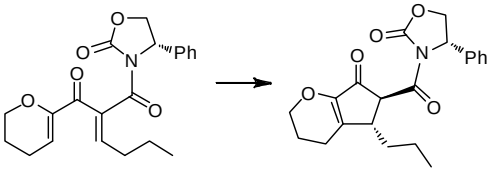
(4*S*,5*R*)-3-[(1*S*,5*S*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-4,5-diphenyloxazolidin-2-one (31c*C*)

Conditions A: The reaction was performed as described for **31a*C*** using **30c** giving **31cA** and **31c*C*** after flash chromatography (silica gel, 50:50:4 hexane / dichloromethane / diethyl ether). Major isomer **31c*C*** white solid (71%). Mp = 143-145 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17-7.07 (m, 6H), 7.02-6.96 (m, 2H), 6.89-6.84 (m, 2H), 6.02 (d, *J* = 7.4 Hz, 1H), 5.63 (d, *J* = 7.4 Hz, 1H), 5.00 (d, *J* = 3.0 Hz, 1H), 3.20 (m_c, 1H), 2.03 (s, 3H), 1.80 (m_c, 1H), 1.71 (s, 3H), 1.43-1.25 (m, 3H), 0.90 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 201.0 (C), 173.4 (C), 168.3 (C), 154.0 (C), 134.5 (C), 133.8 (C), 132.6 (C), 128.3 (CH), 128.2 (CH), 128.0 (CH), 127.9 (CH), 126.4 (CH), 126.1 (CH), 80.3 (CH), 63.5 (CH), 56.3 (CH), 46.1 (CH), 33.9 (CH_2), 20.5 (CH_2), 15.1 (CH_3), 13.9 (CH_3), 8.2 (CH_3). LRMS *m/z* (%): 852.2 (20, 2×M+NH_4^+), 435.5 (25, M+NH_4^+), 418.3 (100, MH^+). HRMS calcd for $C_{26}H_{27}NNaO_4^+$: 440.1838. Found: 440.1833. IR (cm^{-1}): 3035, 2927, 1776, 1707, 1686, 1642, 1331, 1204, 1031, 756. Minor Isomer **31cA** clear oil (26%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16-6.95 (m, 10H), 5.92 (d, *J* = 8.1 Hz, 1H), 5.78 (d, *J* = 8.1 Hz, 1H), 4.95 (br s, 1H), 3.26 (m_c, 1H), 2.00 (s, 3H), 1.80 (m_c, 1H), 1.65 (s, 3H), 1.38-1.22 (m, 3H), 0.95 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 200.1 (C), 172.5 (C), 168.4 (C), 153.4 (C), 133.83 (C), 133.81 (C), 133.1 (C), 128.3 (CH), 128.0 (CH), 127.90 (CH), 127.86 (CH), 126.8 (CH), 126.3 (CH), 79.8 (CH), 63.1 (CH), 56.7 (CH), 45.9 (CH), 34.1 (CH_2), 20.7 (CH_2), 15.0 (CH_3), 14.0 (CH_3), 8.2 (CH_3). LRMS *m/z* (%): 435.5 (10, M+NH_4^+), 418.4 (100, MH^+). HRMS calcd for $C_{26}H_{27}NNaO_4^+$: 440.1838. Found: 440.1833. IR (cm^{-1}): 2928, 1778, 1690, 1643, 1341, 1183, 1041, 729, 697.



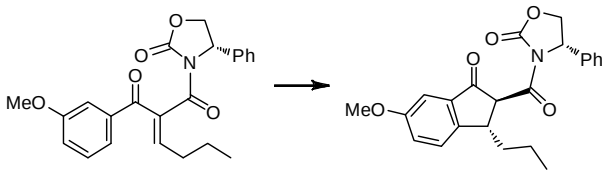
3a*S*,8a*R*)-3-[(1*S*,5*S*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-3,3a,8,8a-tetrahydro-2*H*-indeno[1,2-*d*]oxazol-2-one (31dC)

Conditions A: The reaction was performed as described for **31aC** using **30d** giving **31dA** and **31dC** after flash chromatography (silica gel, 50:50:7 hexane / dichloromethane / diethyl ether). Major isomer **31dC** clear oil (70%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.5 Hz, 1H), 7.40-7.24 (m, 3H), 5.92 (d, *J* = 6.7 Hz, 1H), 5.35 (ddd, *J* = 6.7, 4.5, 2.4 Hz, 1H), 4.86 (d, *J* = 3.3 Hz, 1H), 3.47-3.32 (m, 3H), 2.07 (s, 3H), 1.80 (m_c, 1H), 1.71 (s, 3H), 1.39-1.21 (m, 3H), 0.88 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 201.0 (C), 173.2 (C), 169.6 (C), 153.0 (C), 139.5 (C), 139.2 (C), 133.9 (C), 129.7 (CH), 128.0 (CH), 126.9 (CH), 125.2 (CH), 78.1 (CH), 63.8 (CH), 56.0 (CH), 46.4 (CH), 37.6 (CH_2), 33.8 (CH_2), 20.3 (CH_2), 15.1 (CH_3), 13.8 (CH_3), 8.2 (CH_3). LRMS *m/z* (%): 724.3 (30, 2×M+NH_4^+), 371.2 (10, M+NH_4^+), 354.2 (100, MH^+). HRMS calcd for $C_{21}H_{23}NNaO_4^+$: 376.1525. Found: 376.1520. IR (cm^{-1}): 2928, 1776, 1686, 1643, 1360, 1183, 1043, 755, 727. Minor isomer **31dA** lightly tanned solid (23%). Mp = 130-132 °C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.5 Hz, 1H), 7.36-7.20 (m, 3H), 6.00 (d, *J* = 7.2 Hz, 1H), 5.31 (dt, *J* = 7.2, 3.9 Hz, 1H), 4.81, (br s, 1H), 3.41 (d, *J* = 3.9 Hz, 2H), 3.26 (m_c, 1H), 2.05 (s, 3H), 1.81 (m_c, 1H), 1.68 (s, 3H), 1.44-1.24 (m, 3H), 0.95 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 200.7 (C), 172.0 (C), 170.4 (C), 152.8 (C), 139.1 (C), 138.6 (C), 134.4 (C), 129.8 (CH), 128.2 (CH), 127.2 (CH), 124.9 (CH), 77.9 (CH), 63.1 (CH), 56.0 (CH), 46.9 (CH), 37.9 (CH_2), 33.9 (CH_2), 20.3 (CH_2), 15.0 (CH_3), 14.1 (CH_3), 8.2 (CH_3). LRMS *m/z* (%): 724.5 (25, 2×M+NH_4^+), 371.4 (20, M+NH_4^+), 354.3 (100, MH^+). HRMS calcd for $C_{21}H_{23}NNaO_4^+$: 376.1525. Found: 376.1522. IR (cm^{-1}): 2925, 1757, 1710, 1686, 1643, 1364, 1190, 1034, 753.



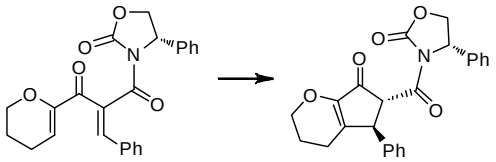
(*S*)-3-[(5*S*,6*S*)-7-Oxo-5-propyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31eC)

Conditions A: The reaction was performed as described for **31aC** using **30e** giving **31eA** and **31eC**, except that only 2 equivalents of $MeSO_3H$ were used and the aqueous $NaHCO_3$ (5% w/v) was added immediately after the reaction was warmed to room temperature over 2 h. After flash chromatography (silica gel, sequential elution 5:5:1 / 5:5:2 hexane / dichloromethane / diethyl ether) the title compound **31eC** was obtained as a white solid (65%). Mp = 178-180 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.22 (m, 5H), 5.43 (dd, *J* = 8.4, 2.7 Hz, 1H), 4.86 (d, *J* = 1.8 Hz, 1H), 4.75 (t, *J* = 8.7 Hz, 1H), 4.29 (dd, *J* = 8.7, 2.7 Hz, 1H), 4.17-3.99 (m, 2H), 3.05 (m_c, 1H), 2.40 (dt, *J* = 18.9, 6.8 Hz, 1H), 2.24 (dt, *J* = 18.9, 5.5 Hz, 1H), 1.93 (quintet, *J* = 5.7 Hz, 2H), 1.70 (m_c, 1H), 1.40-1.10 (m, 3H), 0.83 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 193.2 (C), 168.6 (C), 153.7 (C), 148.9 (C), 148.6 (C), 139.2 (C), 129.1 (CH), 128.7 (CH), 125.8 (CH), 69.9 (CH_2), 66.9 (CH_2), 58.1 (CH), 54.6 (CH), 41.7 (CH), 34.3 (CH_2), 22.3 (CH_2), 21.4 (CH_2), 20.2 (CH_2), 13.9 (CH_3). LRMS *m/z* (%): 756.5 (20, 2×M+NH_4^+), 387.4 (20, M+NH_4^+), 370.3 (100, MH^+). HRMS calcd for $C_{21}H_{23}NNaO_5^+$: 392.1474. Found: 392.1468. IR (cm^{-1}): 2927, 1777, 1712, 1692, 1646, 1350, 1202, 1071, 762, 701. Minor isomer **31eA** waxy solid (27%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.25 (m, 5H), 5.49 (dd, *J* = 9.0, 6.2 Hz, 1H), 4.93 (br s, 1H), 4.74 (t, *J* = 9.0 Hz, 1H), 4.25 (dd, *J* = 8.9, 6.2 Hz, 1H), 4.15-4.00 (m, 2H), 3.25 (m_c, 1H), 2.43 (dt, *J* = 18.6, 6.6 Hz, 1H), 2.24 (dt, *J* = 18.9, 5.6 Hz, 1H), 1.94 (quintet, *J* = 6.0 Hz, 2H), 1.73 (m_c, 1H), 1.42-1.27 (m, 3H), 0.95 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 191.8 (C), 168.1 (C), 153.8 (C), 148.3 (C), 148.2 (C), 137.9 (C), 129.2 (CH), 128.6 (CH), 126.0 (CH), 69.8 (CH_2), 66.8 (CH_2), 58.7 (CH), 55.4 (CH), 40.1 (CH), 34.5 (CH_2), 22.3 (CH_2), 21.4 (CH_2), 20.5 (CH_2), 14.1 (CH_3). LRMS *m/z* (%): 756.3 (15, 2×M+NH_4^+), 387.2 (35, M+NH_4^+), 370.1 (100, MH^+). HRMS calcd for $C_{21}H_{23}NNaO_5^+$: 392.1474. Found: 392.1467. IR (cm^{-1}): 3033, 2930, 1776, 1713, 1694, 1645, 1356, 1200, 1067, 700.



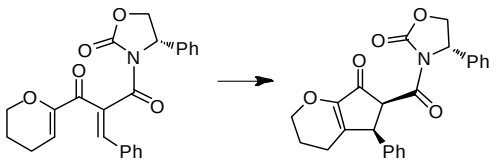
(*S*)-3-[(1*S*,2*S*)-5-Methoxy-3-oxo-1-propyl-2,3-dihydro-1*H*-indene-2-carbonyl]-4-phenyloxazolidin-2-one (31fC)

Conditions A: The reaction was performed as described for **31aC** using **30f** giving **31fA** and **31fC** after flash chromatography (silica gel, sequential elution 20:20:1 / 10:10:1 hexane / dichloromethane / diethyl ether). Major isomer **31fC** white solid (43%). Mp = 166-168 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.30 (m, 6H), 7.20 (dd, *J* = 8.4, 2.6 Hz, 1H), 7.13 (d, *J* = 2.6 Hz, 1H), 5.49 (dd, *J* = 8.4, 2.8 Hz, 1H), 5.14 (d, *J* = 4.2 Hz, 1H), 4.81 (t, *J* = 8.7 Hz, 1H), 4.35 (dd, *J* = 8.9, 2.8 Hz, 1H), 3.83 (s, 3H), 3.74 (m_c, 1H), 1.93 (m_c, 1H), 1.57 (m_c, 1H), 1.37-1.18 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 198.7 (C), 168.3 (C), 159.7 (C), 153.8 (C), 150.2 (C), 139.1 (C), 135.9 (C), 129.1 (CH), 128.6 (CH), 125.8 (CH), 125.7 (CH), 124.4 (CH), 105.3 (CH), 69.9 (CH_2), 59.6 (CH), 58.1 (CH), 55.5 (CH_3), 41.5 (CH), 36.6 (CH_2), 20.7 (CH_2), 13.8 (CH_3). LRMS *m/z* (%): 411.5 (10, M+NH_4^+), 394.5 (100, MH^+). HRMS calcd for $C_{23}H_{24}NO_5^+$: 394.1654. Found: 394.1665. IR (cm^{-1}): 2923, 1778, 1712, 1685, 1490, 1344, 1229, 1201, 759. Minor Isomer **31fA** thick oil (26%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.28 (m, 6H), 7.20 (dd, *J* = 8.4, 2.7 Hz, 1H), 7.11 (d, *J* = 2.7 Hz, 1H), 5.52 (dd, *J* = 9.0, 5.7 Hz, 1H), 5.18 (br s, 1H), 4.77 (t, *J* = 9.0 Hz, 1H), 4.29 (dd, *J* = 9.0, 5.7 Hz, 1H), 3.85 (m_c, 1H), 3.81 (s, 3H), 1.96 (m_c, 1H), 1.58 (m_c, 1H), 1.44-1.31 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 197.6 (C), 168.0 (C), 159.6 (C), 153.7 (C), 149.9 (C), 138.1 (C), 135.8 (C), 129.0 (CH), 128.5 (CH), 125.9 (CH), 125.8 (CH), 124.4 (CH), 105.3 (CH), 69.8 (CH_2), 60.1 (br s, CH), 58.4 (CH), 55.5 (CH_3), 40.5 (CH), 36.8 (CH_2), 20.8 (CH_2), 14.0 (CH_3). LRMS *m/z* (%): 804.2 (25, 2×M+NH_4^+), 411.3 (40, M+NH_4^+), 394.5 (100, MH^+). HRMS calcd for $C_{23}H_{24}NO_5^+$: 394.1654. Found: 394.1641. IR (cm^{-1}): 2925, 1776, 1715, 1691, 1491, 1321, 1277, 1196, 1024, 699.



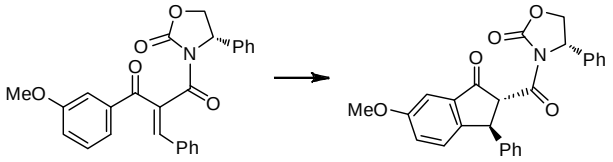
(*S*)-3-[(5*S*,6*R*)-7-Oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31hA)

Conditions B: The reaction was performed as described for **31gA** using **30h** giving **31hA** and **31hC** after flash chromatography (silica gel, sequential elution 50:50:7 / 50:50:12 hexane / dichloromethane / diethyl ether). Major isomer **31hA** white solid (48%). Mp = 102-104 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44-7.26 (m, 8H), 7.18 (d. *J* = 7.8 Hz, 2H), 5.43 (dd, *J* = 8.9, 6.2 Hz, 1H), 5.16 (s, 1H), 4.66 (t, *J* = 9.0 Hz, 1H), 4.43 (s, 1H), 4.21 (dd, *J* = 8.9, 6.2 Hz, 1H), 4.15-4.05 (m, 2H), 2.13-2.05 (m, 2H), 1.93-1.84 (m, 2H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 191.6 (C), 167.1 (C), 153.4 (C), 148.8 (C), 146.9 (C), 139.7 (C), 138.1 (C), 129.2 (CH), 129.1 (CH), 128.6 (CH), 127.7 (CH), 127.6 (CH), 126.0 (CH), 69.8 (CH_2), 67.1 (CH_2), 58.9 (CH), 58.5 (CH), 45.7 (CH), 22.2 (CH_2), 21.3 (CH_2). LRMS *m/z* (%): 421.3 (30, M+NH_4^+), 404.4 (100, MH^+). HRMS calcd for $C_{24}H_{21}NNaO_5^+$: 426.1317. Found: 426.1304. IR (cm^{-1}): 2926, 1778, 1716, 1695, 1648, 1333, 1201, 1109, 1055, 702. Minor isomer **31hC** (8%). Mp = 68-70°C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.22 (m, 8H), 7.10 (dd, *J* = 7.8, 1.5 Hz, 2H), 5.42 (dd, *J* = 8.4, 2.8 Hz, 1H), 5.13 (d, *J* = 2.7 Hz, 1H), 4.74 (t, *J* = 8.6 Hz, 1H), 4.26 (dd, *J* = 8.7, 2.8 Hz, 1H), 4.24 (d, *J* = 2.7 Hz, 1H), 4.17-4.09 (m, 2H), 2.16-2.07 (m, 2H), 1.98-1.86 (m, 2H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 192.8 (C), 167.7 (C), 153.5 (C), 149.2 (C), 147.5 (C), 139.5 (C), 139.0 (C), 129.2 (CH), 129.1 (CH), 128.7 (CH), 127.7 (CH), 127.6 (CH), 125.8 (CH), 70.0 (CH_2), 67.1 (CH_2), 58.3 (CH), 58.0 (CH), 47.0 (CH), 22.3 (CH_2), 21.3 (CH_2). LRMS *m/z* (%): 421.5 (40, M+NH_4^+), 404.4 (100, MH^+). HRMS calcd for $C_{24}H_{21}NNaO_5^+$: 426.1317. Found: 426.1313. IR (cm^{-1}): 3030, 2926, 1777, 1718, 1694, 1647, 1197, 1108, 1072, 700.



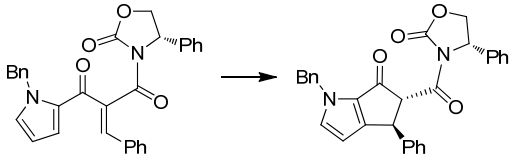
(*S*)-3-[(5*S*,6*S*)-7-Oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31hB)

Conditions C: The reaction was performed as described for **31aC** using **30h** except that the reaction was warmed to 0 °C and stirred at this temperature for 0.5 h, then the aqueous $NaHCO_3$ (5% w/v) added. After flash chromatography (silica gel, 1:1 hexanes / ethyl acetate) the product **31hB** was obtained as a white solid (64%). Mp = 100-102 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 (m_c, 1H), 7.20-6.95 (m, 7H), 6.53 (d, *J* = 7.5 Hz, 2H), 5.20 (dd, *J* = 8.6, 2.9 Hz, 1H), 4.79 (d, *J* = 7.5 Hz, 1H), 4.58-4.48 (m, 2H), 4.20-4.05 (m, 3H), 2.10 (m_c, 1H), 2.01 (m_c, 1H), 1.97-1.80 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8 (C), 166.9 (C), 153.6 (C), 151.3 (C), 147.1 (C), 137.8 (C), 135.7 (C), 129.1 (CH), 128.7 (CH), 128.5 (CH), 127.9 (CH), 127.6 (CH), 125.4 (CH), 70.4 (CH_2), 66.9 (CH_2), 57.5 (CH), 54.8 (CH), 47.7 (CH), 22.1 (CH_2), 21.2 (CH_2). HRMS calcd for $C_{24}H_{22}NO_5^+$: 404.1492. Found: 404.1476.



(*S*)-3-[(1*S*,2*R*)-5-Methoxy-3-oxo-1-phenyl-2,3-dihydro-1*H*-indene-2-carbonyl]-4-phenyloxazolidin-2-one (31iA)

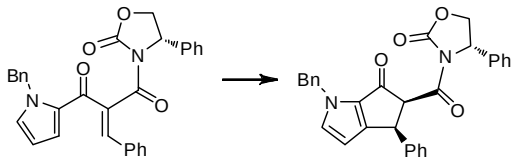
Conditions A: The reaction was performed as described for **31aC** using **30i** except only 5 equivalents of $MeSO_3H$ were used, giving **31iA** and **31iC** after flash chromatography (silica gel, 50:50:6 hexane / dichloromethane / diethyl ether). Major isomer **31iA** white solid (68%). Mp = 80-82 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.05 (m, 13H), 5.48 (dd, *J* = 9.2, 5.6 Hz, 1H), 5.48 (br s, 1), 5.07 (d, *J* = 5.7 Hz, 1H), 4.70 (t, *J* = 9.0 Hz, 1H), 4.27 (dd, *J* = 8.9, 5.6 Hz, 1H), 3.84 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 196.9 (C), 166.9 (C), 159.9 (C), 153.5 (C), 148.6 (C), 141.5 (C), 138.3 (C), 135.7 (C), 129.1 (CH), 128.9 (CH), 128.5 (CH), 128.1 (CH), 127.4 (CH), 127.1 (CH), 125.9 (CH), 124.7 (CH), 105.1 (CH), 69.8 (CH_2), 64.0 (CH), 58.3 (CH), 55.6 (CH_3), 46.0 (CH). LRMS *m/z* (%): 872.4 (25, 2×M+NH_4^+), 445.3 (40, M+NH_4^+), 428.3 (100, MH^+). HRMS calcd for $C_{26}H_{21}NNaO_5^+$: 450.1317. Found: 450.1314. IR (cm^{-1}): 2923, 1778, 1718, 1692, 1489, 1333, 1279, 1023, 699. Minor isomer **31iC** thick oil (5%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.08 (m, 13H), 5.50 (dd, *J* = 8.4, 2.9 Hz, 1H), 5.46 (d, *J* = 5.0 Hz, 1H), 4.97 (d, *J* = 5.0 Hz, 1H), 4.79 (t, *J* = 8.7 Hz, 1H), 4.29 (dd, *J* = 8.9, 2.9 Hz, 1H), 3.86 (s, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 198.1 (C), 167.6 (C), 160.1 (C), 153.7 (C), 149.0 (C), 141.4 (C), 138.9 (C), 136.0 (C), 129.2 (CH), 129.0 (CH), 128.7 (CH), 128.0 (CH), 127.5 (CH), 127.4 (CH), 125.8 (CH), 124.9 (CH), 105.3 (CH), 70.1 (CH_2), 63.4 (CH), 58.3 (CH), 55.7 (CH_3), 47.2 (CH). LRMS *m/z* (%): 450.1 (20, M+Na^+), 428.3 (100, MH^+), 265.2 (40). HRMS calcd for $C_{26}H_{21}NNaO_5^+$: 450.1317. Found: 450.1310. IR (cm^{-1}): 2925, 1779, 1719, 1692, 1490, 1280, 1194, 1024.



(*S*)-3-[(4*S*,5*R*)-1-Benzyl-6-oxo-4-phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (31jA)

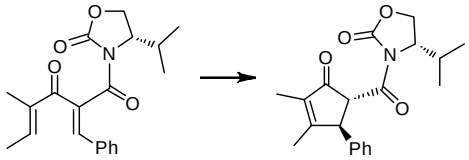
Cupric triflate (76mg, 0.21 mmol) was added to a stirred solution of **30j** (100 mg, 0.21 mmol) in dichloromethane (10 mL), the reaction mixture was then heated at reflux for 4 h. After this time the reaction was cooled to 18 °C and quenched with aqueous $NaHCO_3$ (5% w/v). The phases were separated and the aqueous phase re-extracted with dichloromethane (2 × 10 mL), the combined organic extracts were dried over $MgSO_4$ and concentrated under reduced pressure. The products **31jA**, **31jB** and **31jC** were isolated after flash chromatography (silica gel, 3:1 then 3:2 hexanes / ethyl acetate). Major isomer **31jA**, clear gum (51 mg, 51%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.2 Hz, 2H), 7.40-7.20 (m, 13H), 7.01 (d, *J* = 2.2 Hz, 1H), 5.98 (d, *J* = 2.2 Hz, 1H), 5.62 (br s, 1H), 5.49 (dd, *J* = 9.2, 5.6 Hz, 1H), 5.29 (d, *J* = 14.8 Hz, 1H), 5.25 (d, *J* = 14.8 Hz, 1H), 4.99 (d, *J* = 3.6 Hz, 1H), 4.67 (t, *J* = 9.0 Hz, 1H), 4.21 (dd, *J* = 8.8, 5.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.9 (C), 167.9 (C), 153.5 (C), 153.2 (C), 141.4 (C), 138.3 (C), 136.6 (C), 135.0 (CH), 131.3 (C), 129.0 (CH), 128.8 (CH), 128.7 (CH), 128.4 (CH), 128.0 (CH), 127.8 (CH), 127.5 (CH), 127.1 (CH), 126.0 (CH), 106.1 (CH), 69.7 (CH_2), 67.2 (CH), 58.5 (CH), 50.9 (CH_2), 41.4 (CH). HRMS calcd for $C_{30}H_{25}N_2O_4^+$: 477.1809. Found: 477.1818. *cis*-Isomer **31jB** white solid (16 mg, 16%, characterised in following experiment). Minor Isomer **31jC** thick gum (18 mg, 18%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.22 (m, 13H), 7.14 (dd, *J* = 7.8, 1.4 Hz, 2H), 7.05 (d, *J* = 2.2 Hz, 1H), 6.03 (d, *J* = 2.2 Hz, 1H), 5.59 (d, *J* = 3.6 Hz, 1H), 5.50 (dd, *J* = 8.2, 2.7 Hz, 1H), 5.28 (s, 2H), 4.88 (d, *J* = 3.6 Hz, 1H), 4.76 (t, *J* = 8.6 Hz, 1H), 4.28 (dd, *J* = 8.8, 2.7 Hz,

1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.0 (C), 168.3 (C), 153.60 (C), 153.59 (C), 141.2 (C), 139.1 (C), 136.6 (C), 135.3 (CH), 131.6 (C), 129.2 (CH), 128.9 (CH), 128.7 (CH), 128.6 (CH), 128.1 (CH), 127.9 (CH), 127.3 (CH), 127.1 (CH), 125.7 (CH), 106.3 (CH), 69.9 (CH_2), 66.6 (CH), 58.2 (CH), 51.0 (CH_2), 42.4 (CH). HRMS calcd for $C_{30}H_{25}N_2O_4^+$: 477.1809. Found: 477.1820.



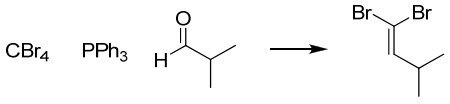
(*S*)-3-[(4*S*,5*S*)-1-Benzyl-6-oxo-4-phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (31jB)

Conditions C: The reaction was performed as described for **31aC** using **30j** except that the reaction was warmed to 0 °C and stirred at this temperature for 2 h, then the aqueous $NaHCO_3$ (5% w/v) added. The products **31jB** and **31jD** were isolated after flash chromatography (silica gel, 3:2 hexanes / ethyl acetate). Major isomer **31jB** white solid (81%). Mp = 88-90 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.28 (m, 5H), 7.16 (t, *J* = 7.4 Hz, 1H), 7.13-7.02 (m, 7H), 7.01 (d, *J* = 2.4 Hz, 1H), 6.53 (d, *J* = 7.6 Hz, 2H), 5.88 (d, *J* = 2.4 Hz, 1H) 5.39 (d, *J* = 7.6 Hz, 1H), 5.35 (d, *J* = 14.8 Hz, 1H), 5.29 (d, *J* = 14.8 Hz, 1H), 5.23 (dd, *J* = 8.8, 3.3 Hz, 1H), 4.99 (d, *J* = 7.6 Hz, 1H) 4.57 (t, *J* = 8.8 Hz, 1H), 4.10 (dd, *J* = 9.0, 3.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.2 (C), 168.1 (C), 154.4 (C), 153.6 (C), 138.2 (C), 138.1 (C), 136.7 (C), 135.2 (CH), 133.9 (C), 129.3 (CH), 128.72 (CH), 128.69 (CH), 128.0 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 127.4 (CH), 125.4 (CH), 104.7 (CH), 70.2 (CH_2), 63.8 (CH), 57.6 (CH), 50.8 (CH_2), 43.5 (CH). HRMS calcd for $C_{30}H_{25}N_2O_4^+$: 477.1809. Found: 477.1807. Minor Isomer **31jD** thick gum (10%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40-7.25 (m, 11H), 7.23-7.17 (m, 2H), 7.14 (d, *J* = 6.6 Hz, 2H), 7.03 (d, *J* = 2.4 Hz, 1H), 5.92 (d, *J* = 2.4 Hz, 1H), 5.59 (d, *J* = 7.2 Hz, 1H), 5.36 (d, *J* = 14.7 Hz, 1H), 5.27 (d, *J* = 14.7 Hz, 1H) 4.87 (d, *J* = 7.2 Hz, 1H) 4.40 (dd, *J* = 8.6, 2.8 Hz, 1H), 3.97 (dd, *J* = 8.4, 2.8 Hz, 1H), 3.77 (t, *J* = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.8 (C), 168.2 (C), 153.4 (C), 153.2 (C), 139.3 (C), 138.6 (C), 136.8 (C), 135.1 (CH), 130.5 (C), 129.3 (CH), 129.1 (CH), 128.9 (CH), 128.6 (CH), 128.3 (CH), 128.12 (CH), 128.06 (CH), 127.6 (CH), 125.8 (CH), 105.8 (CH), 69.8 (CH_2), 64.3 (CH), 57.4 (CH), 51.1 (CH_2), 43.4 (CH). HRMS calcd for $C_{30}H_{25}N_2O_4^+$: 477.1809. Found: 477.1822.



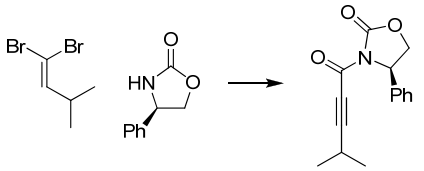
(*S*)-3-[(1*R*,5*S*)-3,4-Dimethyl-2-oxo-5-phenylcyclopent-3-enecarbonyl]-4-isopropyloxazolidin-2-one (31kA)

Conditions B: The reaction was performed as described for **31gA** using **30j** giving **31kA** and **31kC** after flash chromatography (silica gel, 50:50:4 hexane / dichloromethane / diethyl ether). Major isomer **31kA** thick oil (50%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.25 (m, 3H), 7.16 (d, *J* = 7.5 Hz, 2H), 5.23 (d, *J* = 3.3 Hz, 1H), 4.51-4.42 (m, 2H), 4.27-4.19 (m, 2H), 2.35 (sept.d, *J* = 6.9, 3.8 Hz, 1H), 1.85 (s, 3H), 1.79 (s, 3H), 1.01 (d, *J* = 6.9 Hz, 3H), 0.92 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 200.7 (C), 171.0 (C), 168.4 (C), 153.7 (C), 140.2 (C), 134.6 (C), 129.1 (CH), 128.0 (CH), 127.5 (CH), 63.1 (CH_2), 60.3 (CH), 58.7 (CH), 51.7 (CH), 28.3 (CH), 17.9 (CH_3), 15.4 (CH_3), 14.5 (CH_3), 8.6 (CH_3). LRMS *m/z* (%): 700.4 (15, 2×M+NH_4^+), 359.4 (10, M+NH_4^+), 342.2 (100, MH^+). HRMS calcd for $C_{20}H_{23}NNaO_4^+$: 364.1525. Found: 364.1521. IR (cm^{-1}): 2964, 1778, 1707, 1687, 1647, 1371, 1203, 1083, 702.. Minor isomer thick oil **31kC** (23%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38-7.24 (m, 3H), 7.15 (d, *J* = 7.5 Hz, 2H), 5.16 (d, *J* = 3.3 Hz, 1H), 4.44 (br s, 1H), 4.39-4.15 (m, 3H), 2.32 (m_c, 1H), 1.86 (s, 3H), 1.78 (s, 3H), 0.92 (d, *J* = 6.9 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 201.0 (C), 171.7 (C), 168.2 (C), 154.2 (C), 140.1 (C), 134.6 (C), 129.1 (CH), 127.9 (CH), 127.6 (CH), 63.8 (CH_2), 59.9 (CH), 59.8 (CH), 52.0 (CH), 29.3 (CH), 18.1 (CH_3), 15.5 (CH_3), 15.2 (CH_3), 8.5 (CH_3). LRMS *m/z* (%): 700.3 (20, 2×M+NH_4^+), 359.3 (15, M+NH_4^+), 342.2 (100, MH^+). HRMS calcd for $C_{20}H_{23}NNaO_4^+$: 364.1525. Found: 364.1520. IR (cm^{-1}): 2964, 1778, 1708, 1688, 1646, 1370, 1200, 1078, 703.



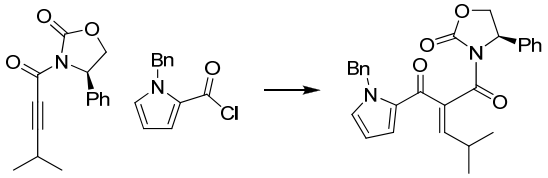
1,1-Dibromo-3-methylbut-1-ene (33)

Triphenylphosphine (28.9 g, 109 mmol) was added to a stirred solution of carbon tetrabromide (36.6 g, 109 mmol) in dry dichloromethane (350 mL) at 0 °C. Zinc dust (14.6 g, 219 mmol) was then added and the resultant mixture was allowed to warm to room temperature and was stirred under N_2 for 18 hours. After this time isobutyrylaldehyde (5.1 mL, 55 mmol) was added and the mixture was stirred for 4 hours. An additional portion of isobutyrylaldehyde (5.1 mL, 55 mmol) was then added with stirring continued for a further 2 hours. After this time the mixture was filtered through celite and the liquid collected was concentrated under reduced pressure. The crude product was then extracted from the resultant sludge with a 1:1 mixture of dichloromethane and hexane, (5 × 50 mL, dichloromethane added first). The combined extracts were concentrated under reduced pressure and flash chromatographed (silica gel, 17:3 hexane / dichloromethane) to return the title compound as a clear liquid (14.6 g, 59%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.22 (d, J = 9.0 Hz, 1H), 2.58 (dsept., J = 9.0, 6.9 Hz, 1H), 1.03 (d, J = 6.9 Hz, 6H).



(*R*)-3-(4-Methylpent-2-ynoyl)-4-phenyloxazolidin-2-one (35)

Butyllithium (2.0 M in cyclohexane, 5.0 mL, 10.0 mmol) was added dropwise to a stirred solution of dibromo-olefin **33** (1.14 g, 5.00 mmol) in tetrahydrofuran (15 mL) at −78 °C under nitrogen. After stirring for 30 minutes the nitrogen was turned off and carbon dioxide was bubbled slowly through the solution while it was warmed to 0ºC (ice bath) over the course of 30 minutes. The carbon dioxide supply was then removed and replaced with the nitrogen atmosphere. The solution was again cooled to -78 °C and pivaloyl chloride (635 µL, 5.00 mmol) was added. The solution was then allowed to warm to room temperature and was stirred for 3 hours. The solution was then cooled again to -78 °C and to it was added *via* cannula a solution of lithiated oxazolidinone (5.00 mmol, generated by addition of 2.5 mL of 2.0 M butyllithium solution to 816 mg of *R*-phenyloxazolidinone **34** in 30 mL of tetrahydrofuran) at −78ºC. The solution was then allowed to warm to room temperature and stirred for 2 hours. After this time the solution was concentrated to a volume of 10 mL under vacuum. Ethyl acetate (30 mL) and distilled water (30 mL) were then added and the organic phase was separated, the aqueous phase was re-extracted with ethyl acetate (2 × 20 mL) and the combined organic extracts were dried over $MgSO_4$ and concentrated onto silica gel (3 g) under reduced pressure. The solid residue was subjected to flash chromatography (silica gel, 83:17 hexane / ethyl acetate) giving the title compound as a thick oil (887 mg, 69%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.25 (m, 5H), 5.43 (dd, J = 8.7, 3.6 Hz, 1H), 4.67 (app. t, J_{app} = 8.7 Hz, 1H), 4.27 (dd, J = 9.0, 3.6 Hz, 1H), 2.77 (sept, J = 6.9 Hz, 1H), 1.26 (d, J = 6.9 Hz, 6H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 152.2 (C), 150.4 (C), 138.4 (C), 129.2 (CH), 128.9 (CH), 126.0 (CH), 103.2 (C), 72.8 (C), 69.8 (CH_2), 57.6 (CH), 21.5 (2×CH_3), 21.1 (CH). LRMS *m/z* (%): 532.2 (10, 2×M+NH_4^+), 275.2 (60, M+NH_4^+), 258.2 (100, MH^+). HRMS calcd for $C_{15}H_{15}NNaO_3^+$: 280.0950. Found: 280.0941. IR (cm^{-1}): 3034, 2976, 2231, 1789, 1664, 1319, 1195, 1056, 712.



(*R*,*Z*)-1-(1-Benzyl-1*H*-pyrrol-2-yl)-2-(2-methylpropylidene)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (36)

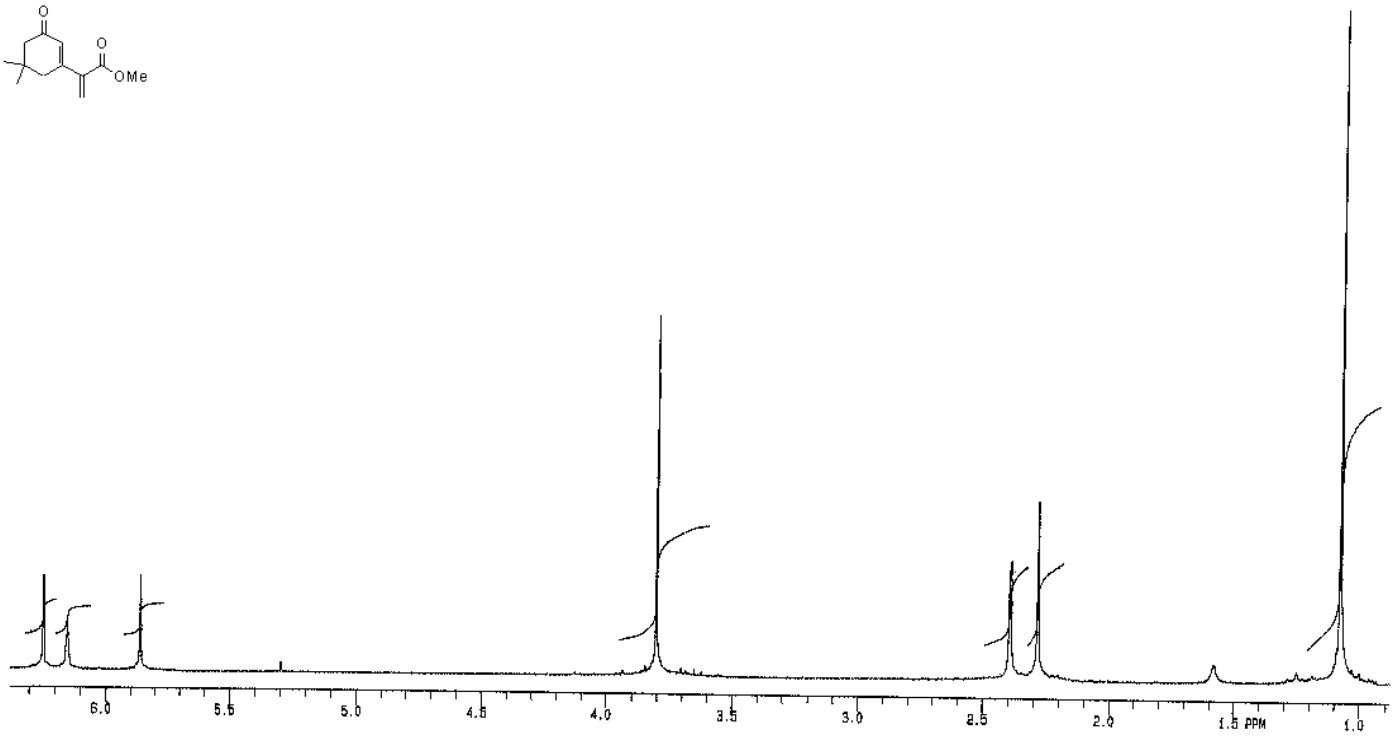
Alkyne **35** was reacted with acid chloride **15i** in accordance with General Procedure A giving the product **36** after flash chromatography (silica gel, 4:1 hexane / ethyl acetate) as a tan solid (91%). Mp = 60-62 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.20 (m, 8H), 7.15 (d, J = 6.9 Hz, 2H), 7.94-6.85 (m, 2H), 6.58 (d, J = 10.5 Hz, 1H), 6.17 (m_c, 1H), 5.55-5.42 (m, 3H), 4.69 (app. t, J_{app} = 8.7 Hz, 1H), 4.22 (dd, J = 8.7, 4.2 Hz, 1H), 2.47 (m_c, 1H), 1.07 (d, J = 6.6 Hz, 3H), 1.02 (d, J = 6.3 Hz, 3H). ^{13}C NMR (JMOD, 75 MHz, $CDCl_3$) δ 182.2 (C), 165.7 (C), 153.9 (CH), 153.0 (C), 138.7 (C), 138.2 (C), 136.3 (C),

130.1 (CH), 129.3 (C), 129.0 (CH), 128.6 (CH), 128.5 (CH), 127.42 (CH), 127.35 (CH), 126.0 (CH), 122.0 (CH), 108.7 (CH), 70.3 (CH_2), 57.5 (CH), 51.9 (CH_2), 29.7 (CH), 21.94 (CH_3), 21.91 (CH_3). LRMS *m/z* (%): 902.5 (10, $2\times M+NH_4^+$), 443.4 (100, MH^+). HRMS calcd for $C_{27}H_{27}N_2O_4^+$: 443.1971. Found: 443.1983. IR (cm^{-1}): 2964, 1782, 1692, 1614, 1385, 1319, 1201, 1083, 699.

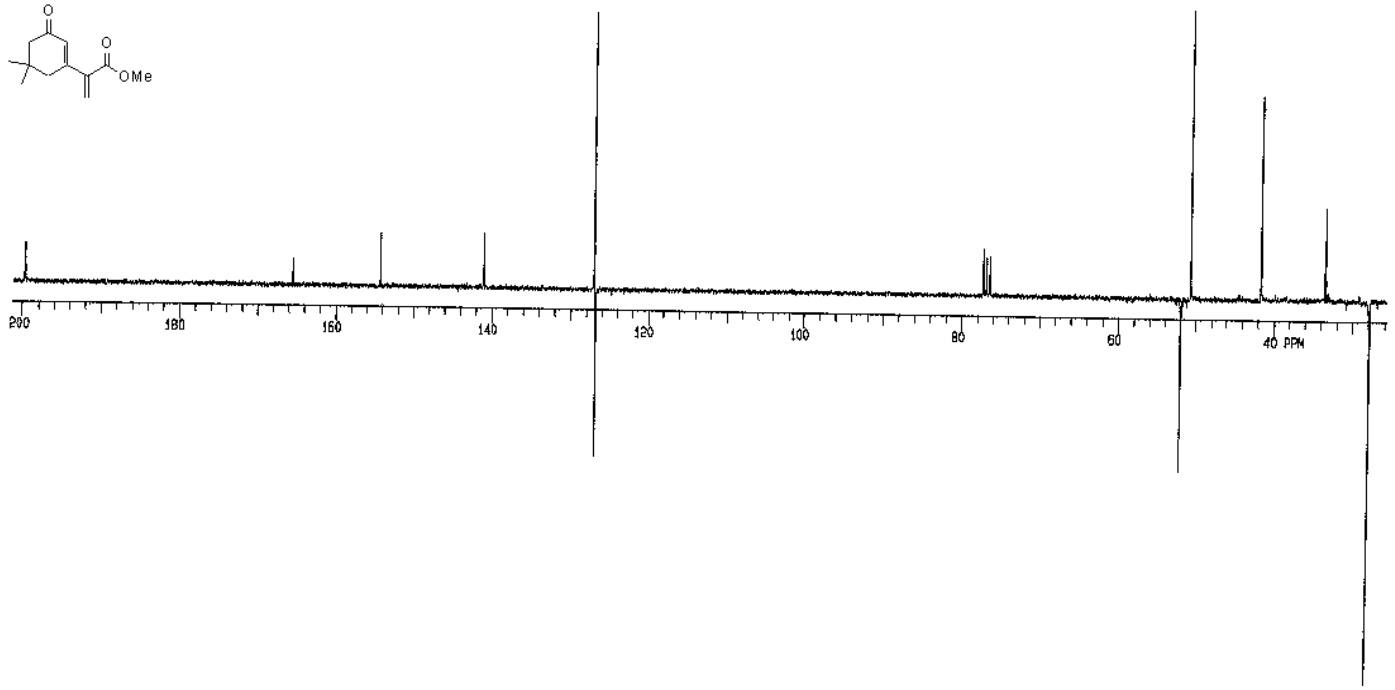
IV. ^{1}H and ^{13}C (JMOD or APT) NMR Spectra

Methyl 2-(5,5-dimethyl-3-oxo-cyclohex-1-enyl)-acrylate (16a)

^{1}H NMR (300 MHz, $CDCl_3$)

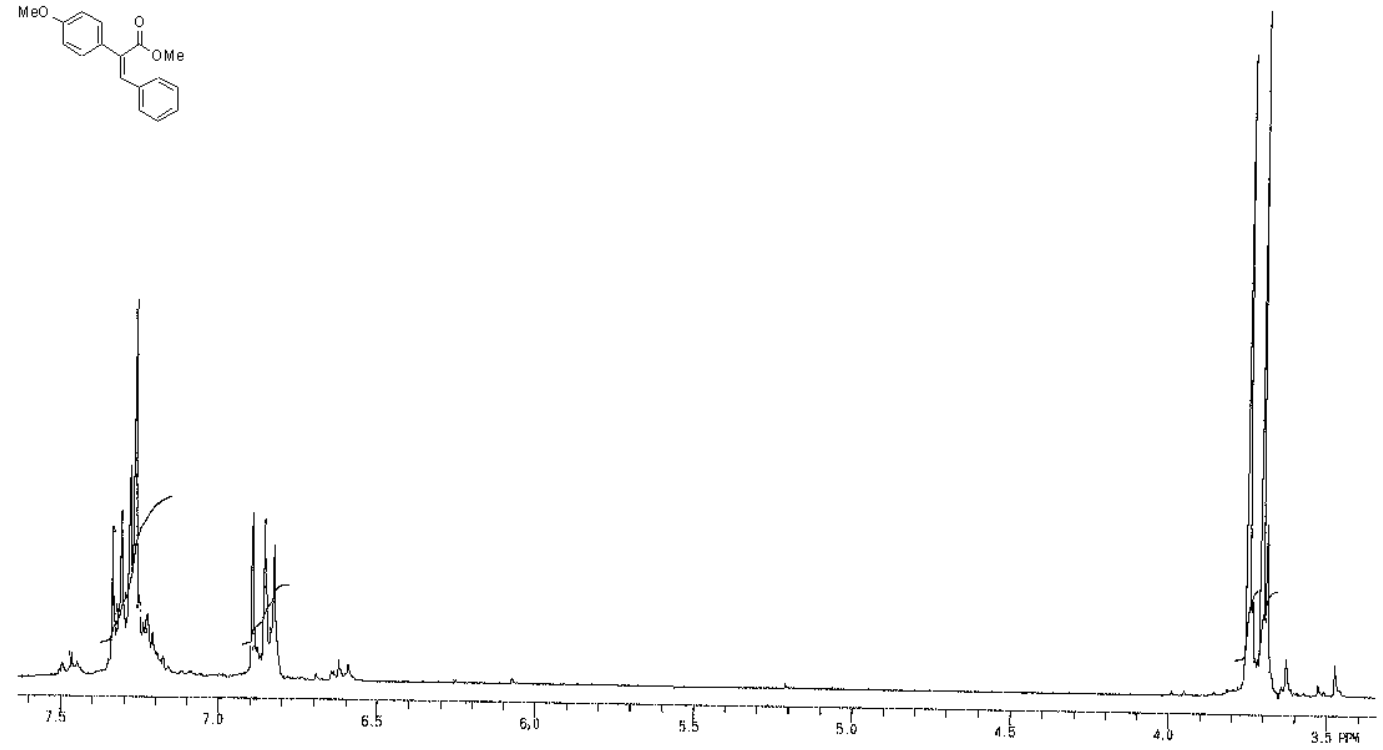


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

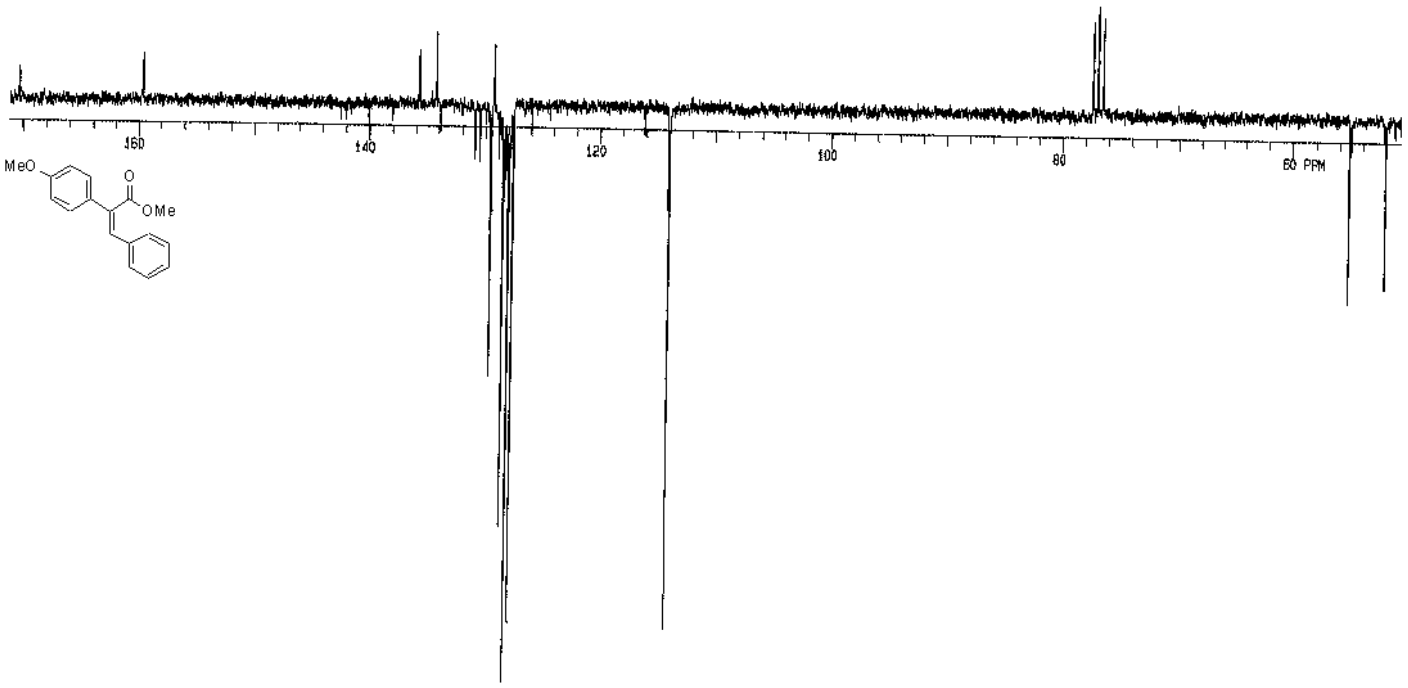


Methyl (*Z*)-2-(4-methoxyphenyl)-3-phenyl-acrylate (16b)

^{1}H NMR (300 MHz, $CDCl_3$)

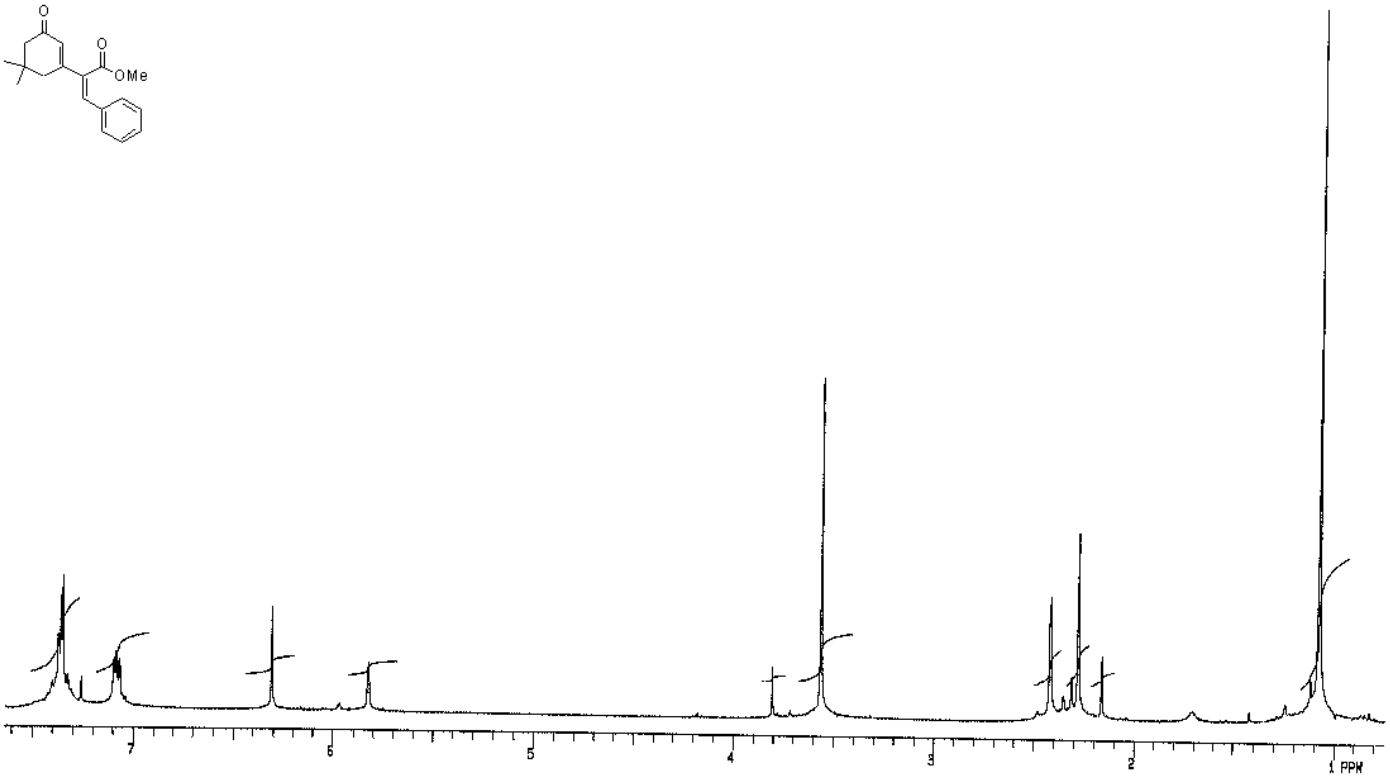


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

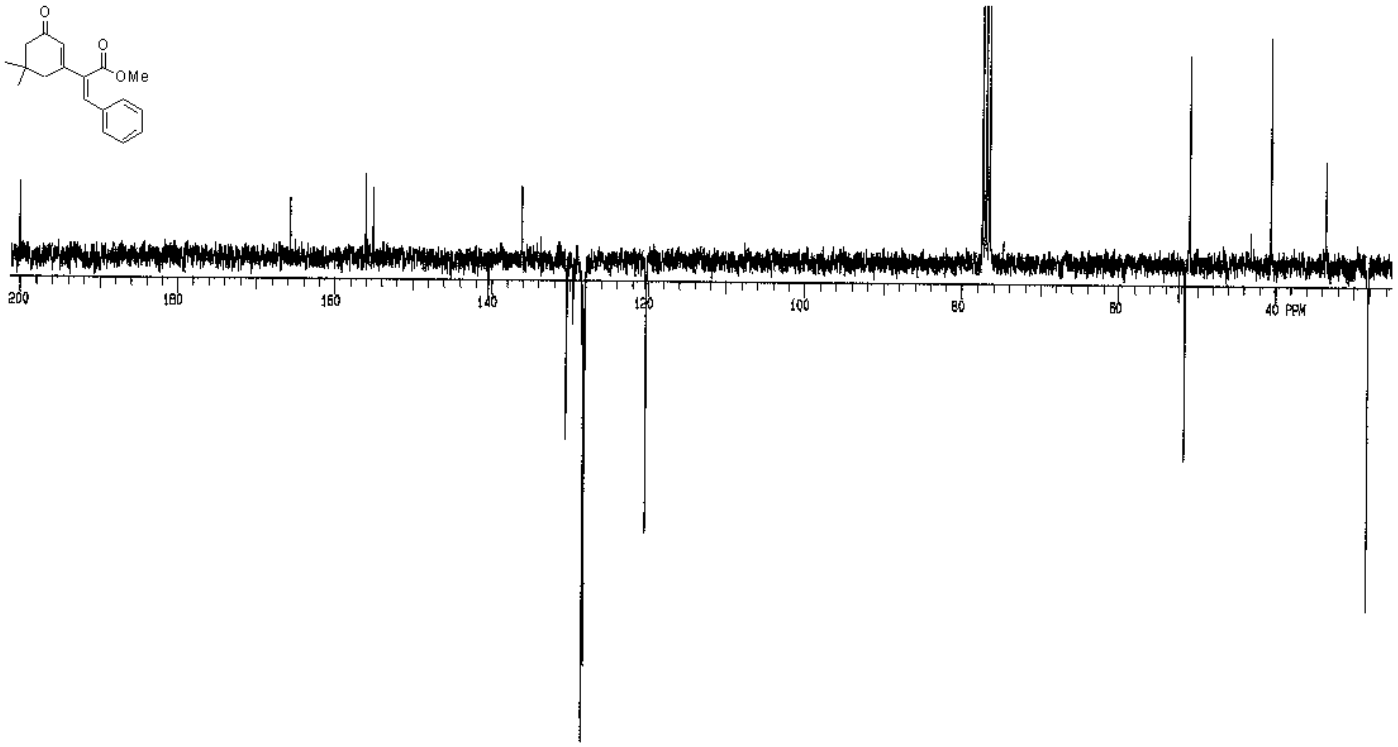


Methyl (*Z*)-2-(5,5-Dimethyl-3-oxo-cyclohex-1-enyl)-3-phenyl-acrylate (*Z*-16c)

^{1}H NMR (300 MHz, $CDCl_3$)

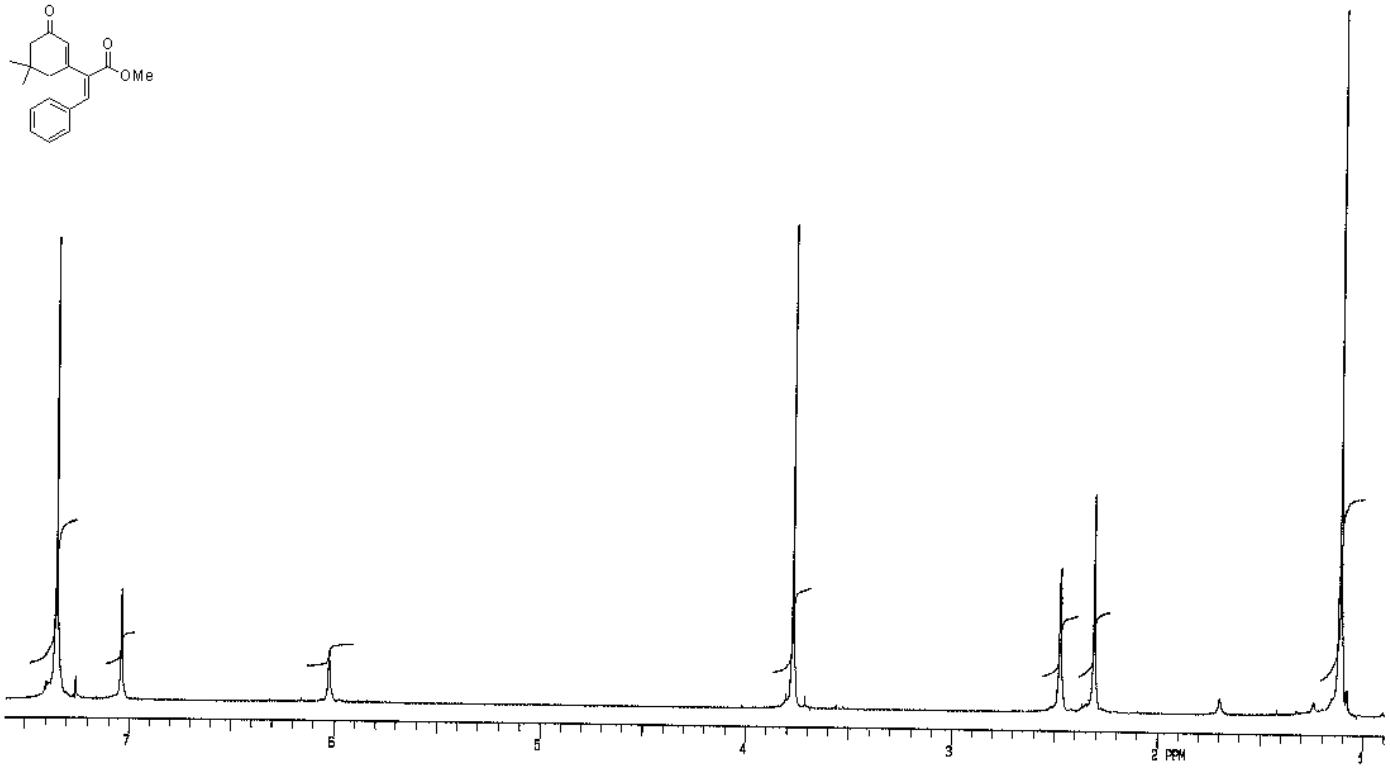


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

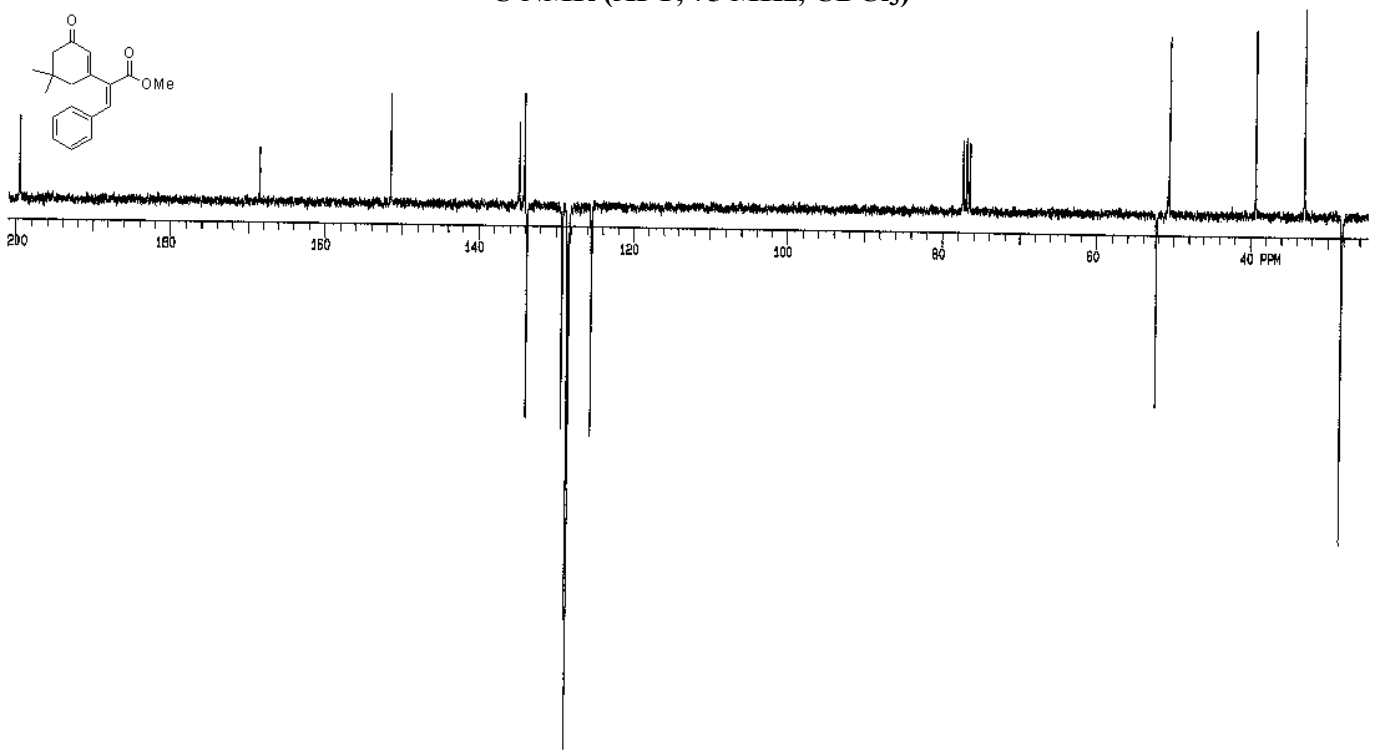


Methyl (*E*)-2-(5,5-Dimethyl-3-oxo-cyclohex-1-enyl)-3-phenyl-acrylate (*E*-16c)

^{1}H NMR (300 MHz, $CDCl_3$)

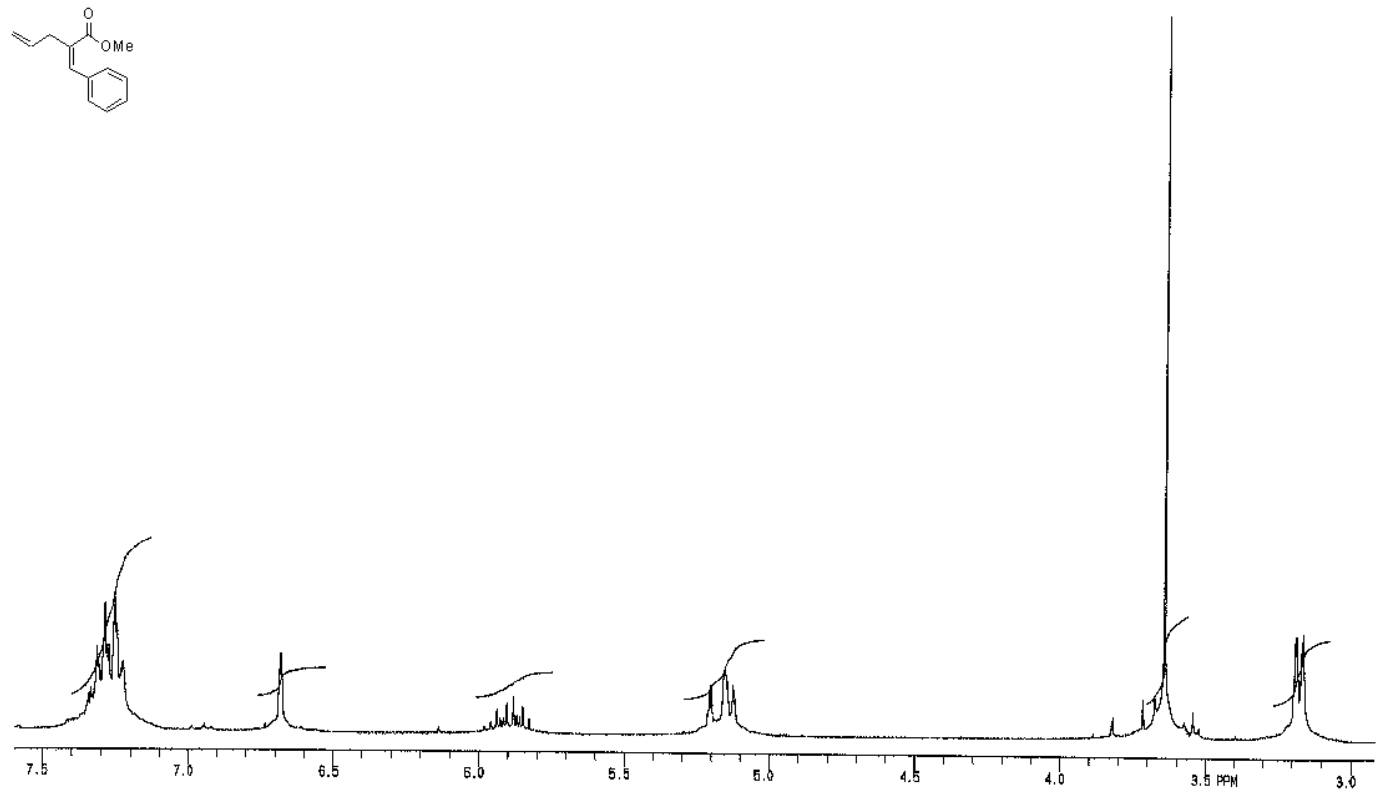


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

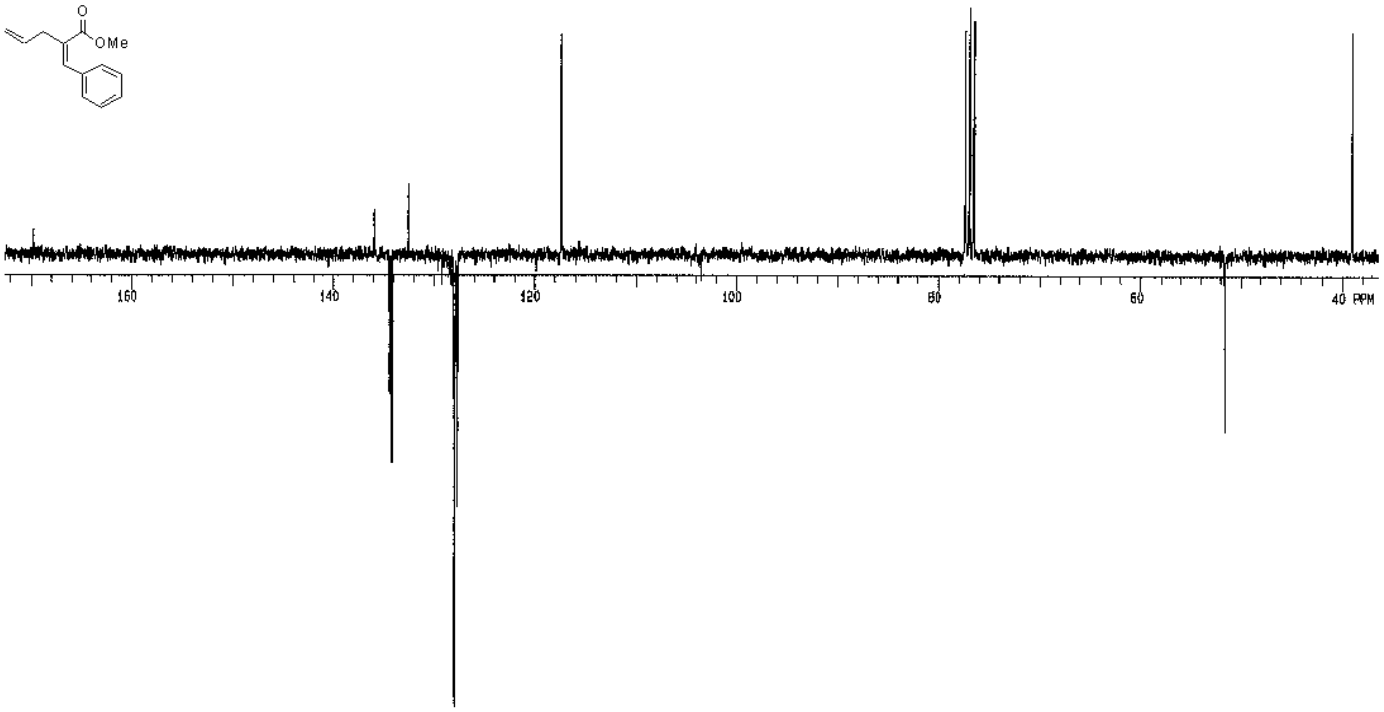


Methyl (*Z*)-2-benzylidine-4-pentenoate (16d)

^{1}H NMR (300 MHz, $CDCl_3$)

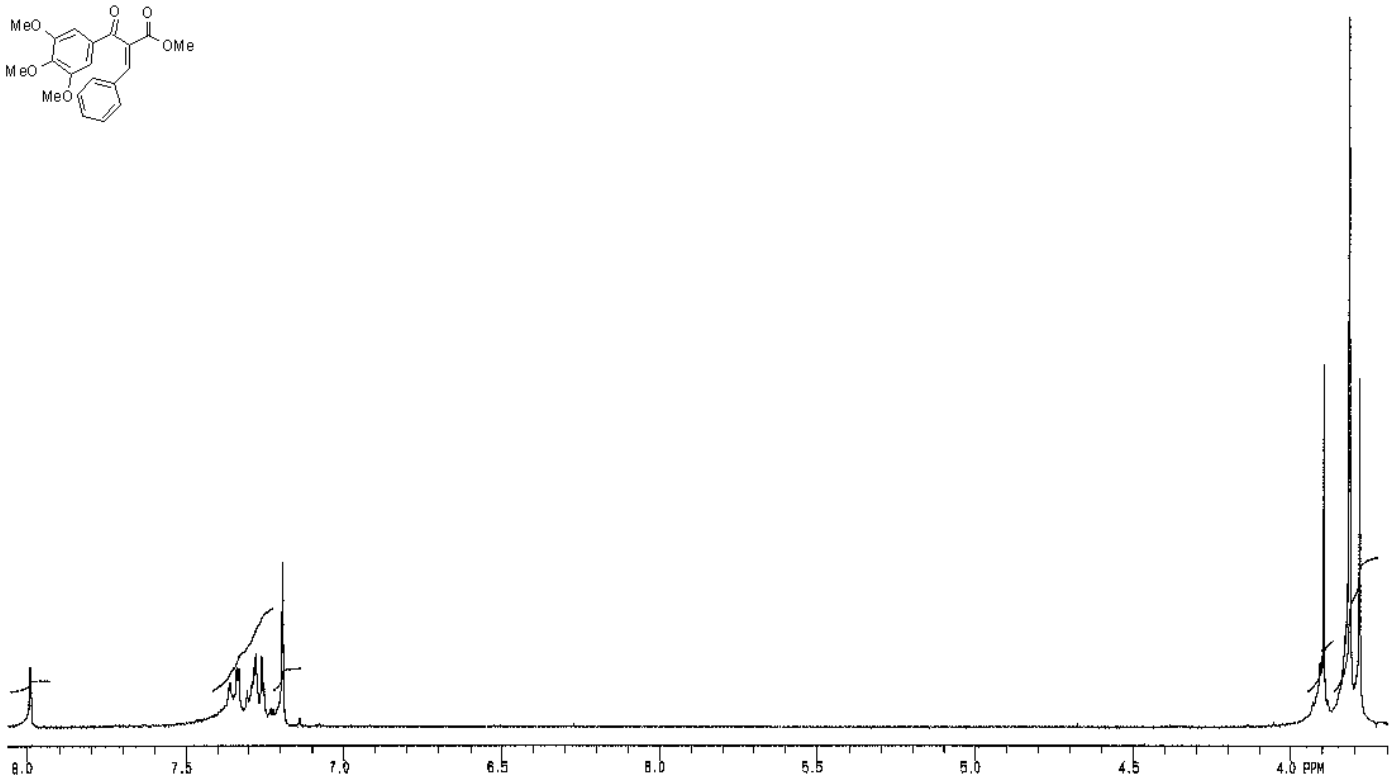


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

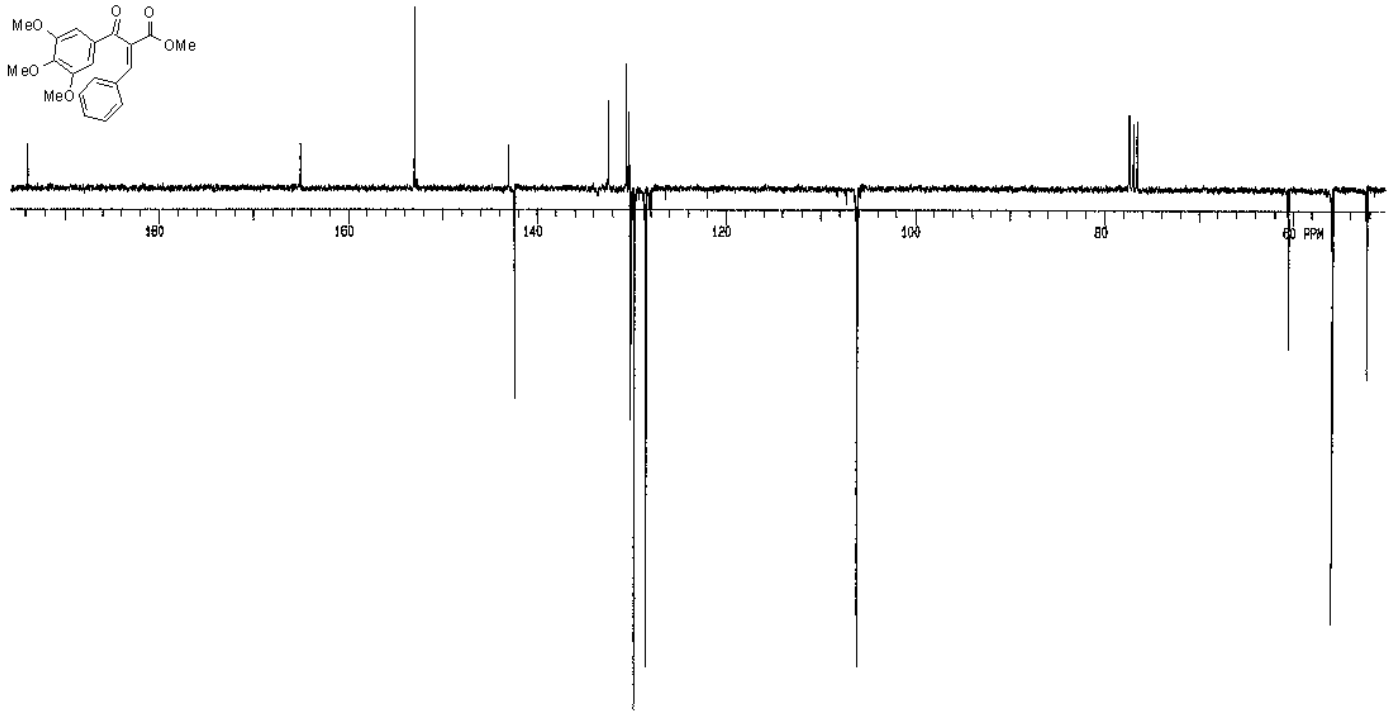


Methyl (*E*)-3-phenyl-2-(3,4,5-trimethoxybenzoyl)-acrylate (*E*-16e)

^{1}H NMR (300 MHz, $CDCl_3$)

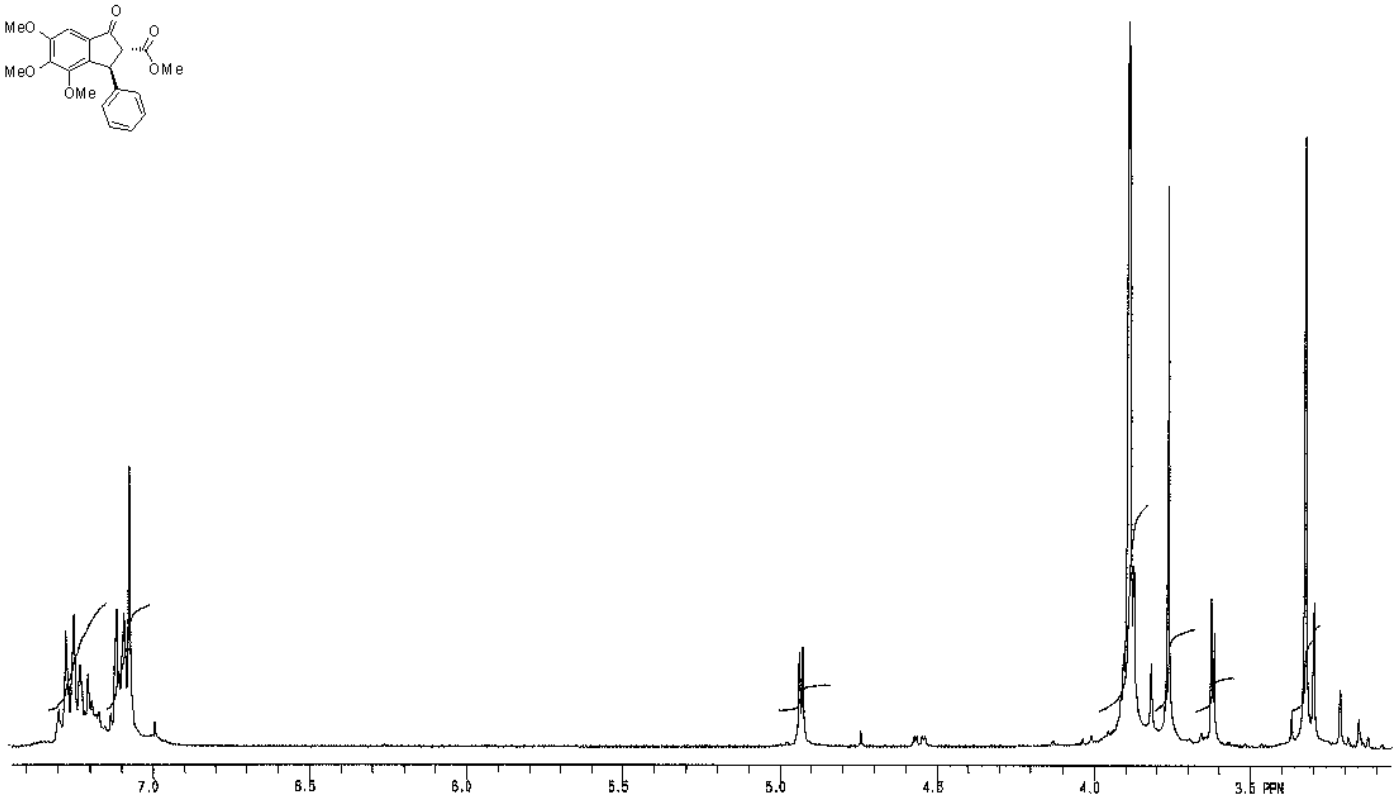


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

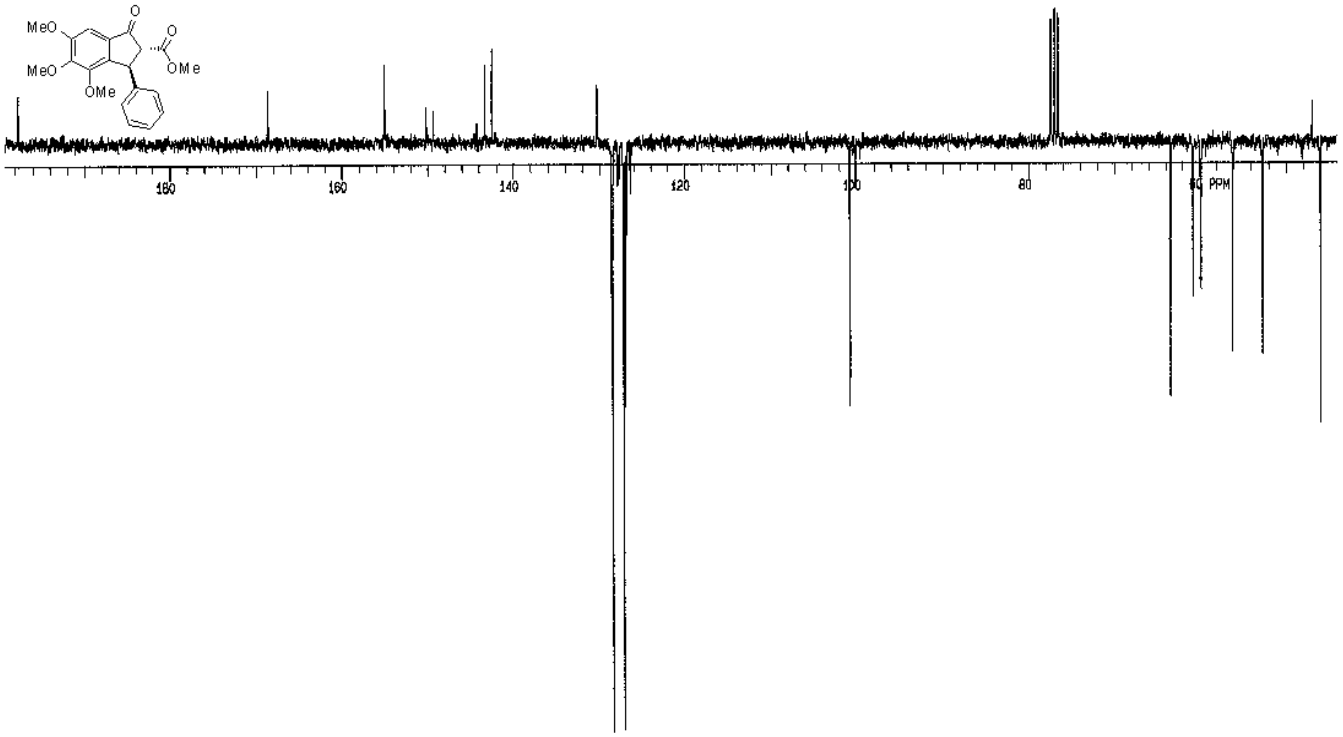


(±)-Methyl *trans*-4,5,6-trimethoxy-1-oxo-3-phenyl-2,3-dihydro-1*H*-indene-2-carboxylate (17e)

^{1}H NMR (300 MHz, $CDCl_3$)

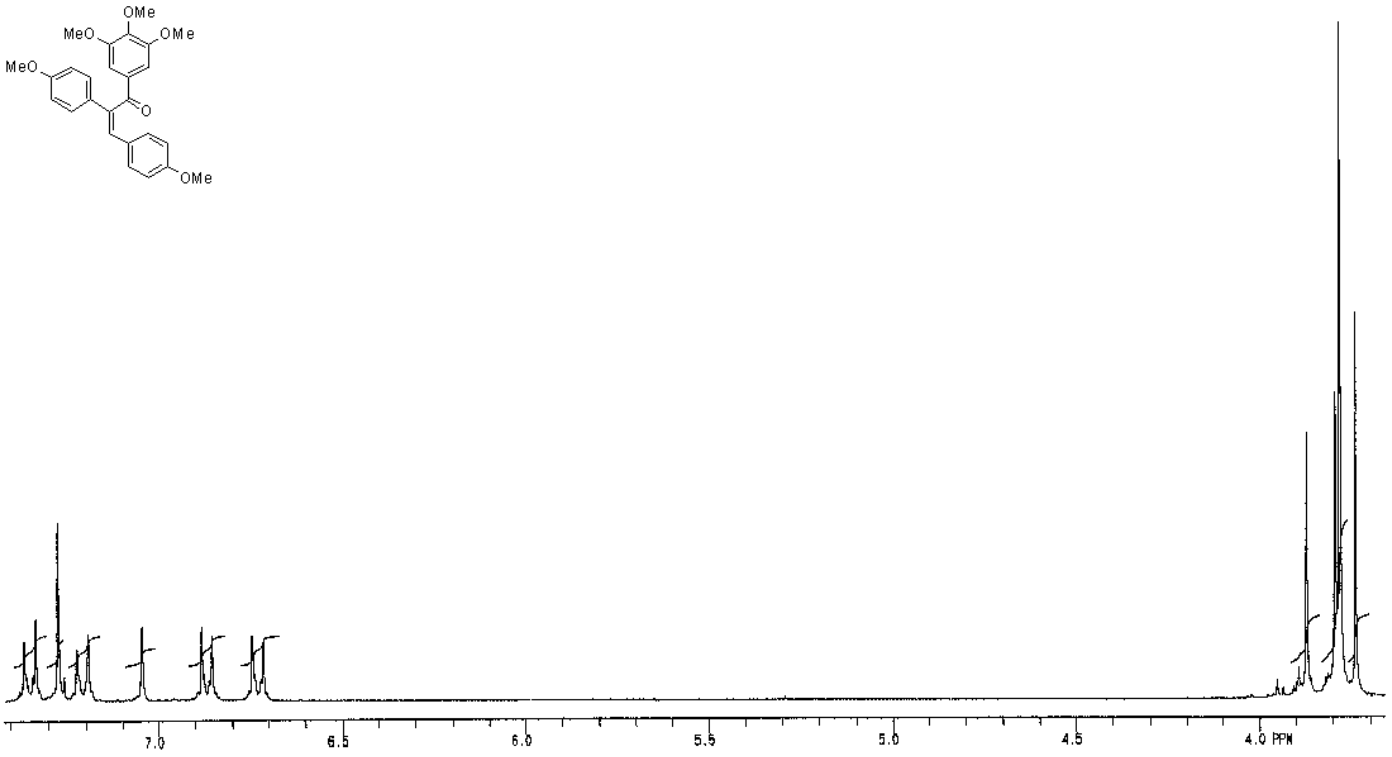


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

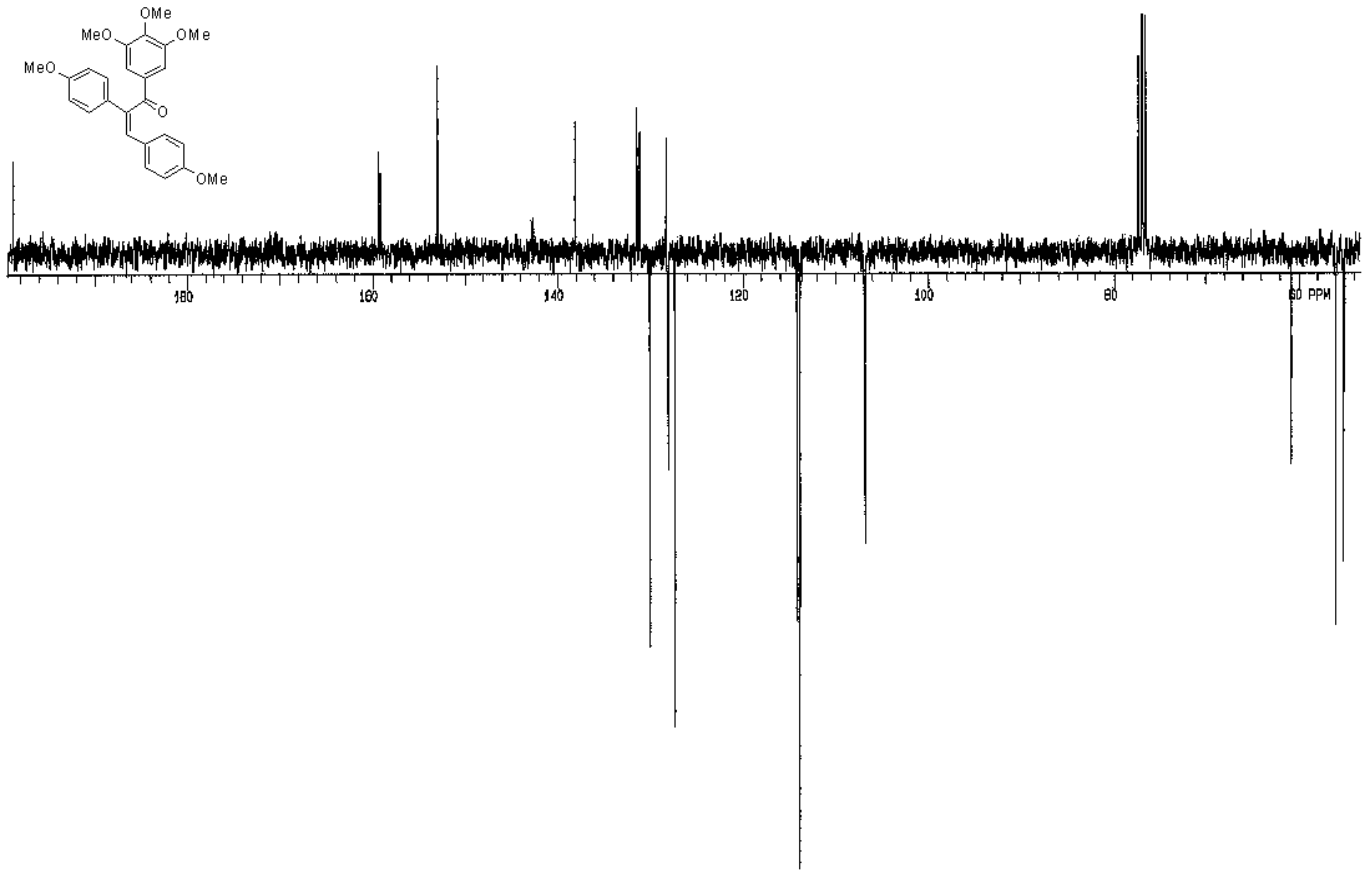


(*Z*)-2,3-Bis(4-methoxyphenyl)-1-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (*Z*-16f)

^{1}H NMR (300 MHz, $CDCl_3$)

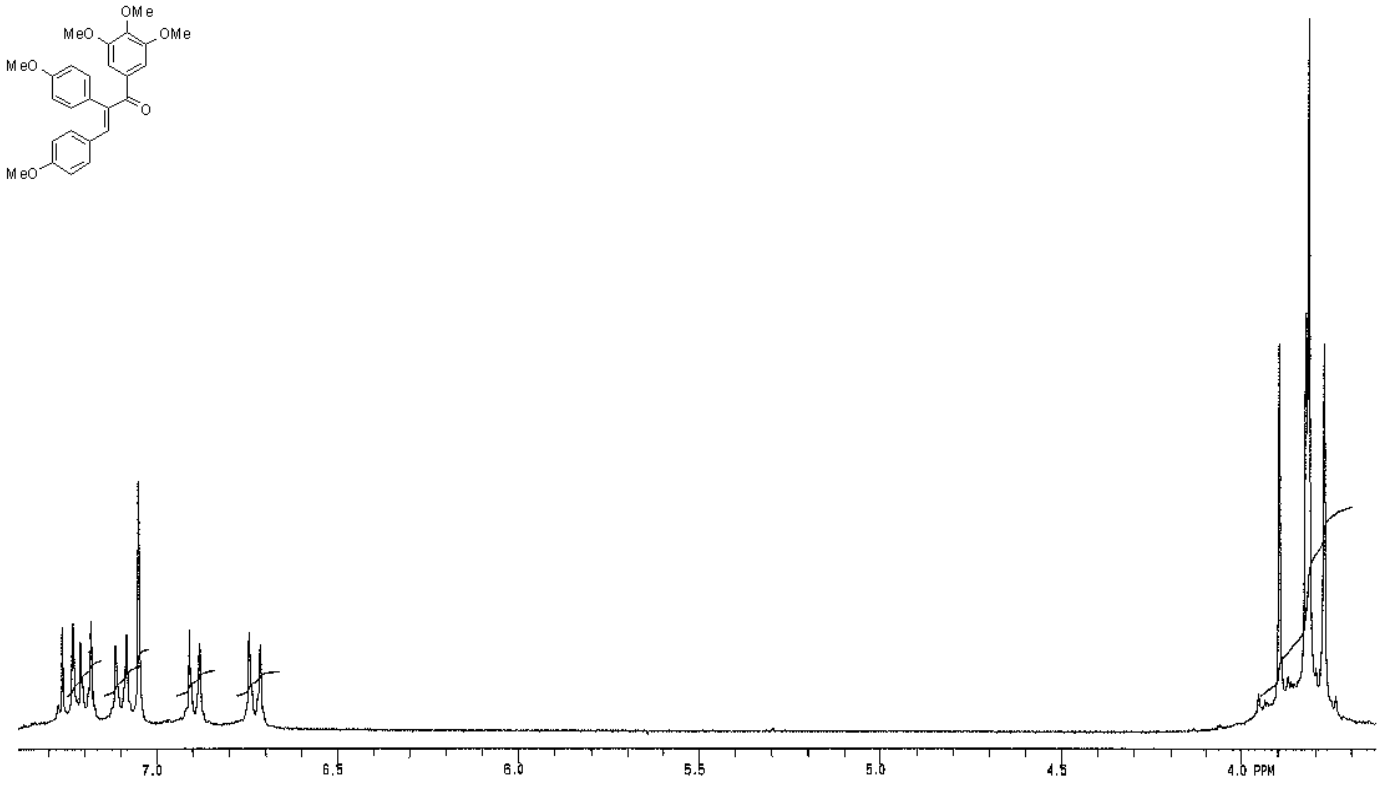


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

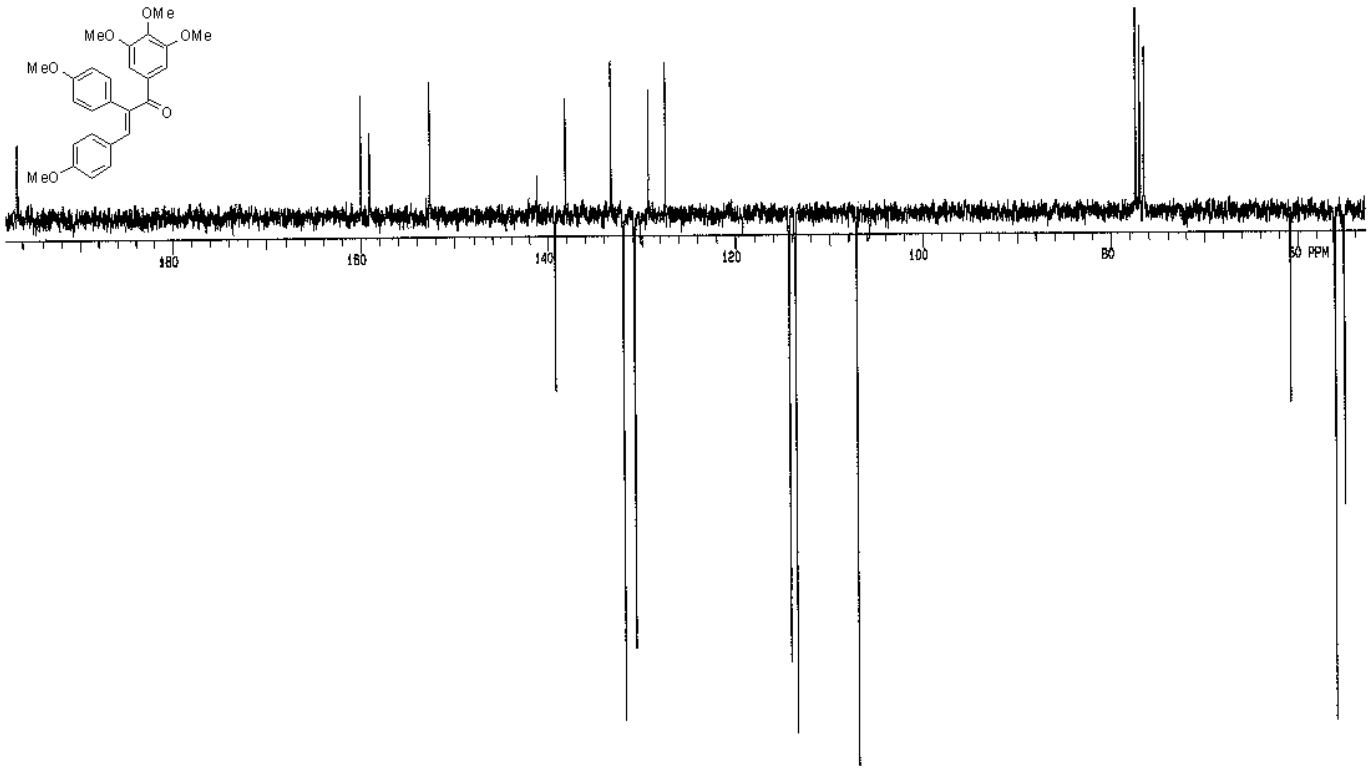


(*E*)-2,3-Bis(4-methoxyphenyl)-1-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (*E*-16f)

^{1}H NMR (300 MHz, $CDCl_3$)

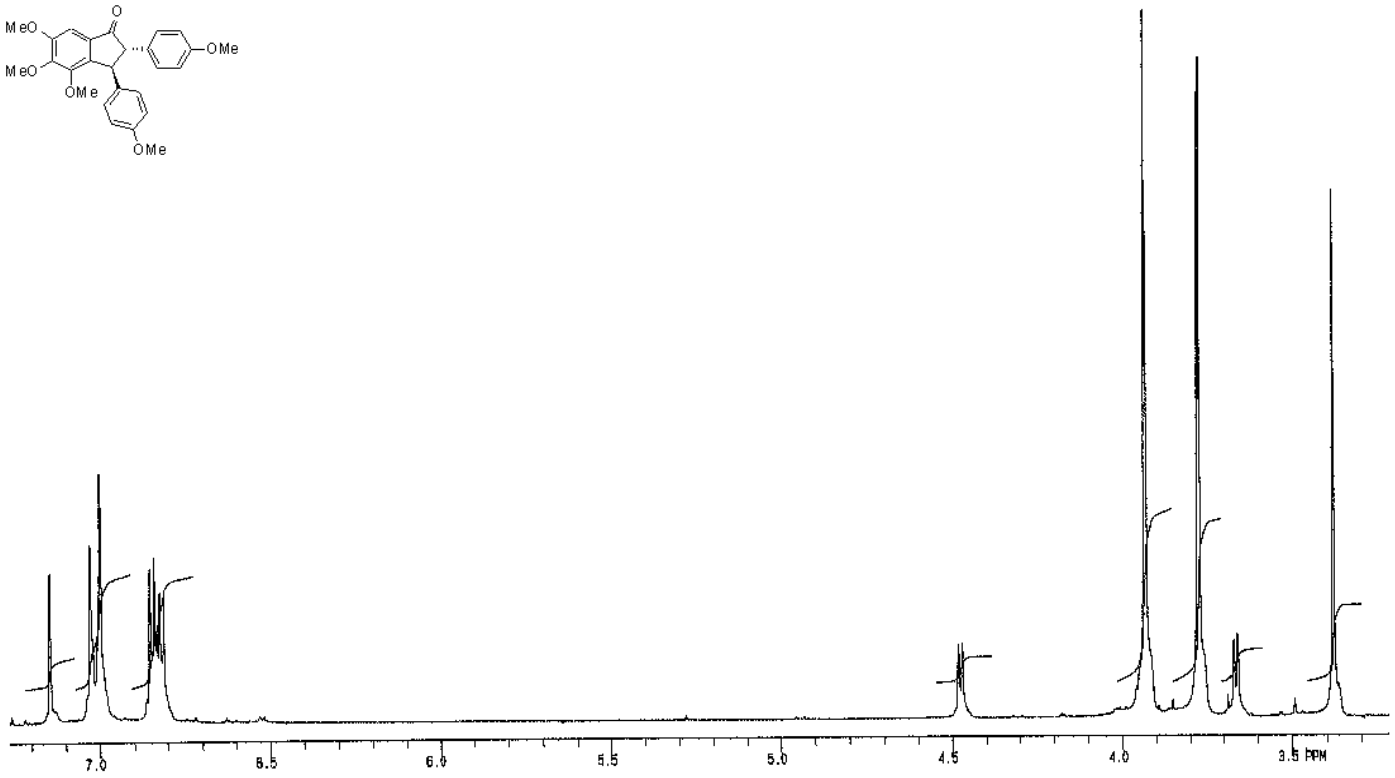


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

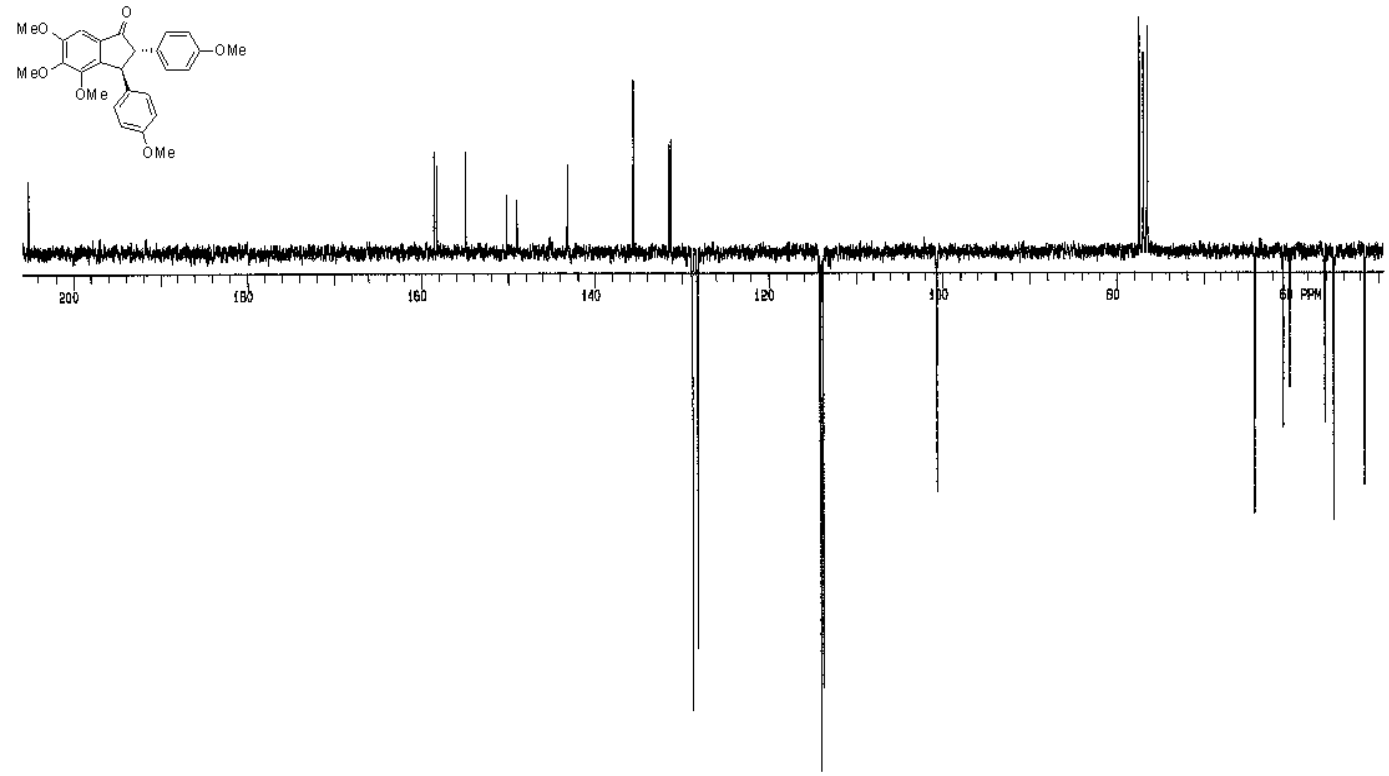


(±)-*trans*-4,5,6-Trimethoxy-2,3-bis(4-methoxyphenyl)-2,3-dihydro-1*H*-inden-1-one (17f)

^{1}H NMR (300 MHz, $CDCl_3$)

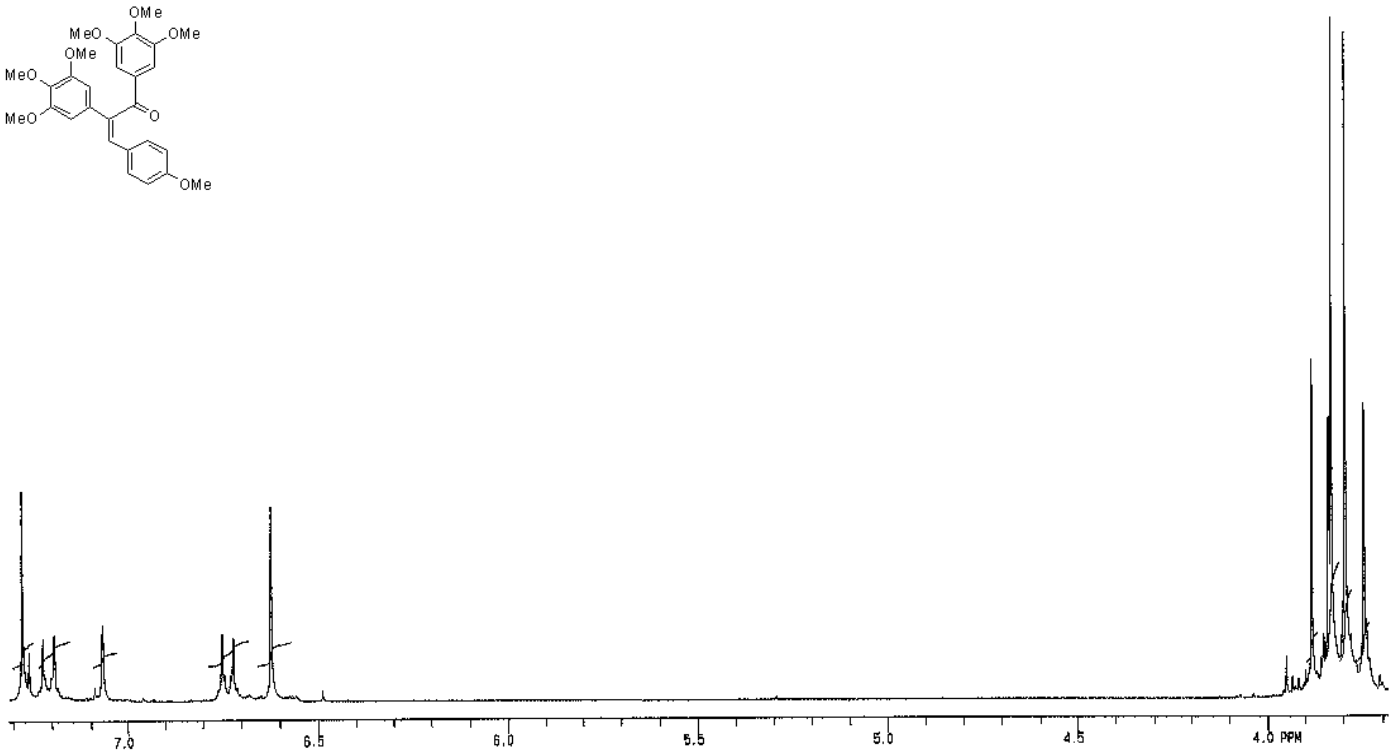


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

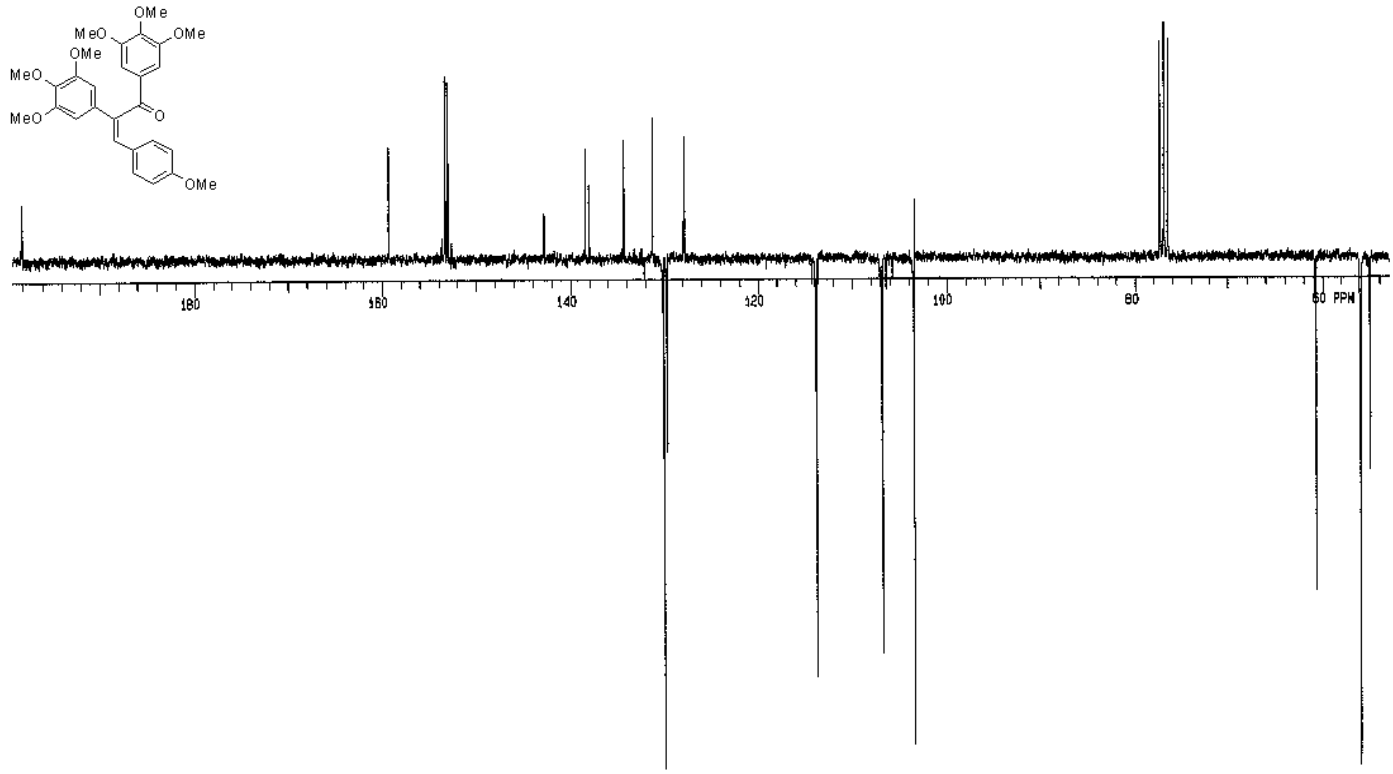


(*Z*)-3-(4-Methoxyphenyl)-1,2-bis(3,4,5-trimethoxyphenyl)prop-2-en-1-one (*Z*-16g)

^{1}H NMR (300 MHz, $CDCl_3$)

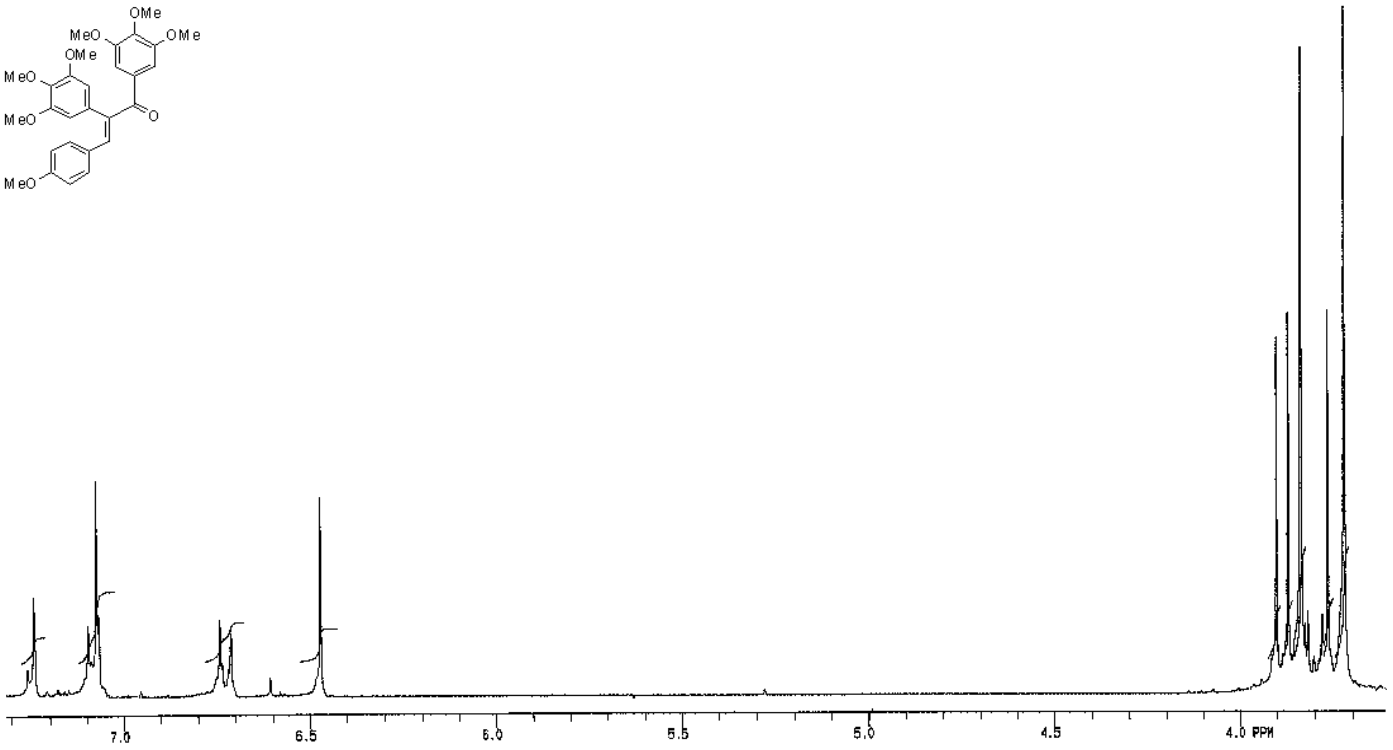


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

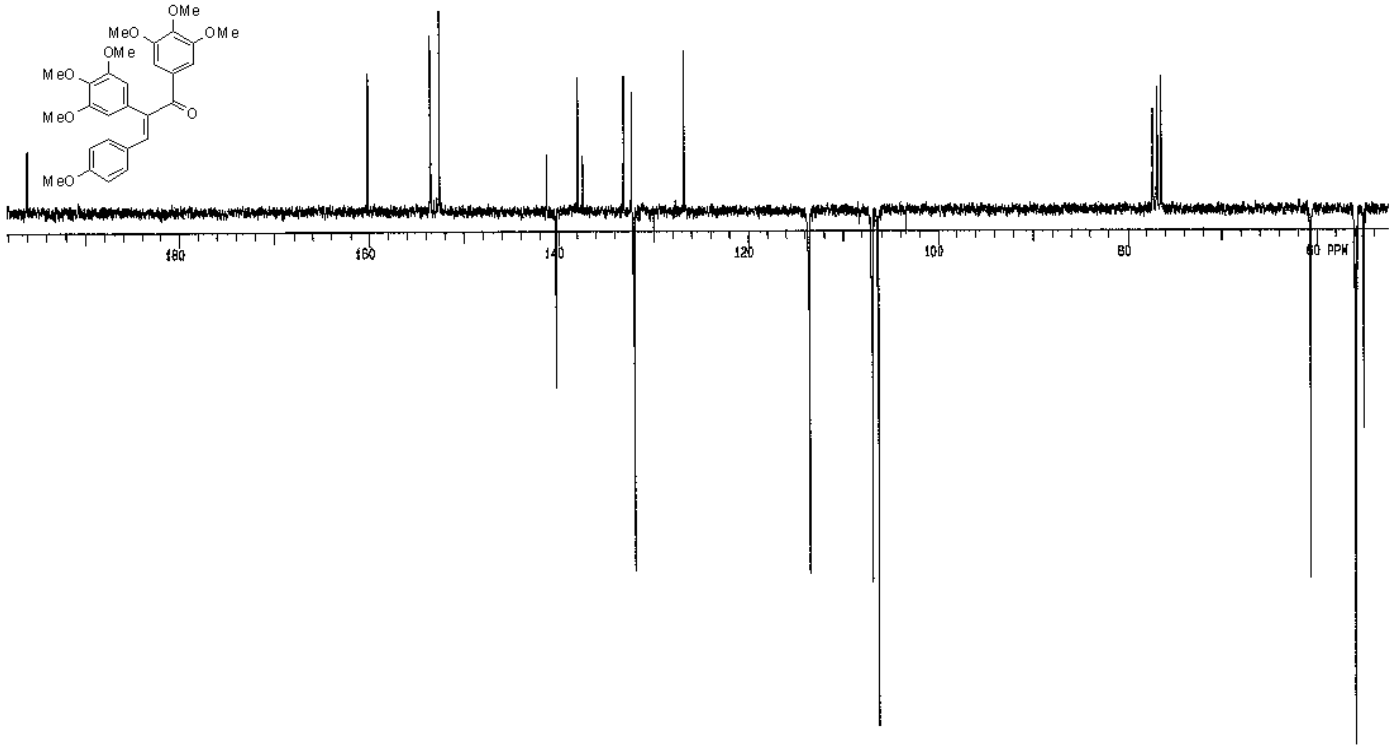


(*E*)-3-(4-Methoxyphenyl)-1,2-bis(3,4,5-trimethoxyphenyl)prop-2-en-1-one (*E*-16g)

^{1}H NMR (300 MHz, $CDCl_3$)

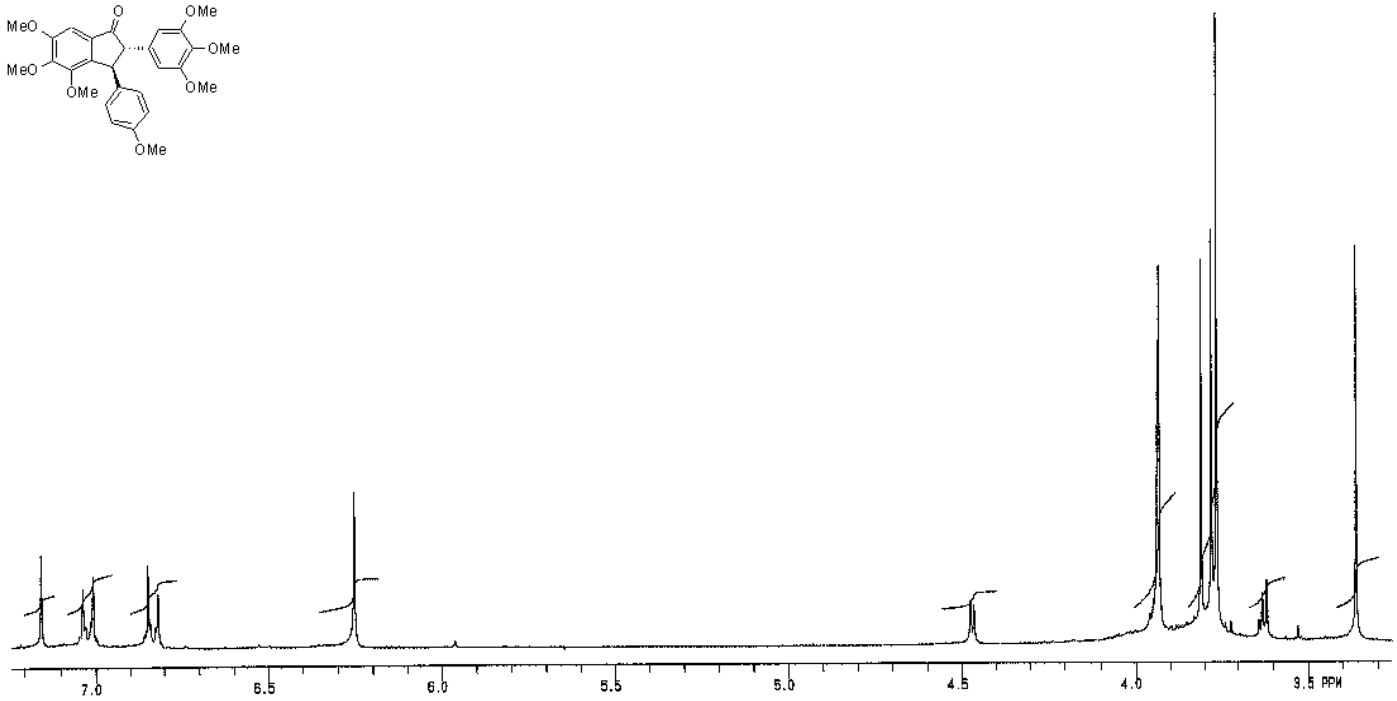


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

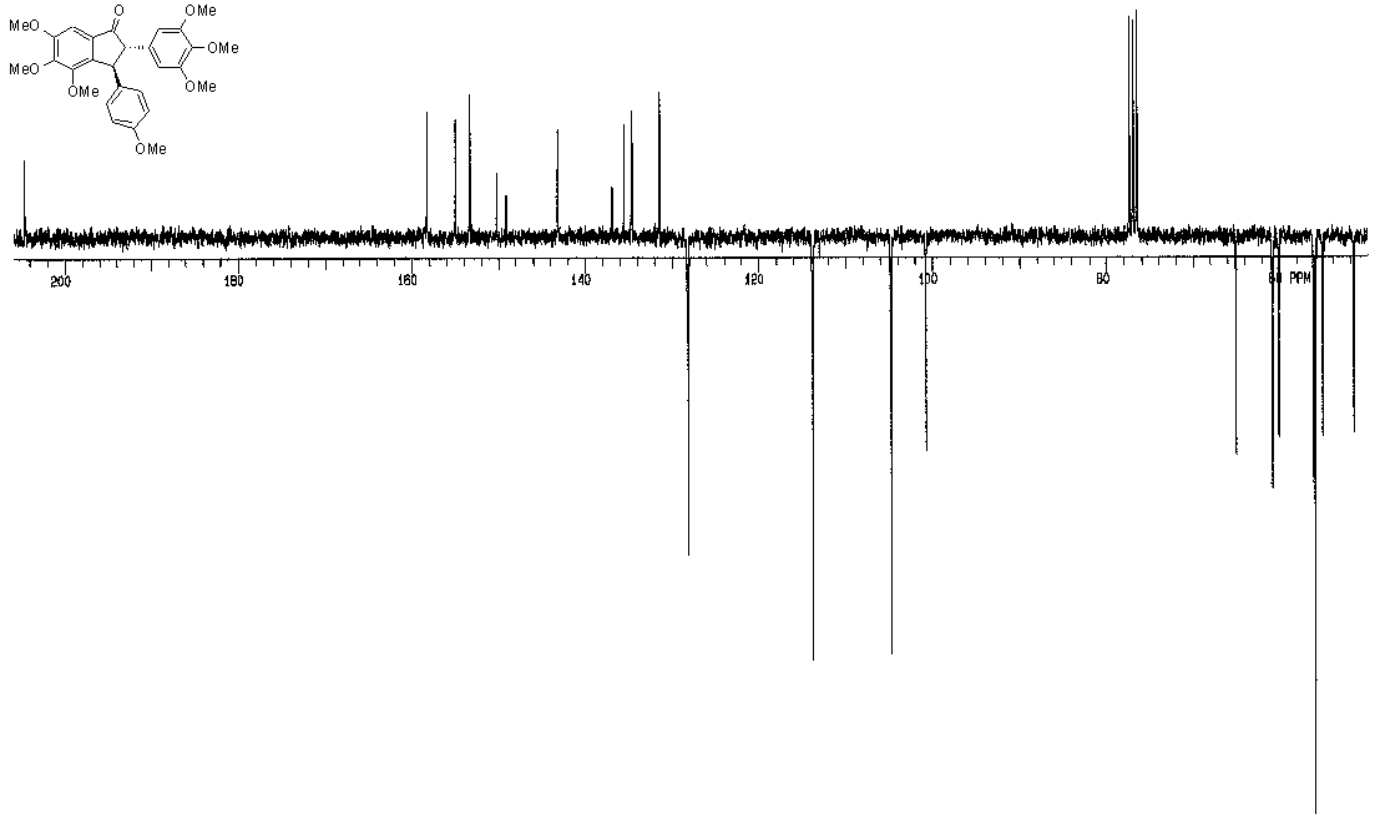


(±)-*trans*-4,5,6-trimethoxy-3-(4-methoxyphenyl)-2-(3,4,5-trimethoxyphenyl)-2,3-dihydro-1*H*-inden-1-one (17g)

^{1}H NMR (300 MHz, $CDCl_3$)

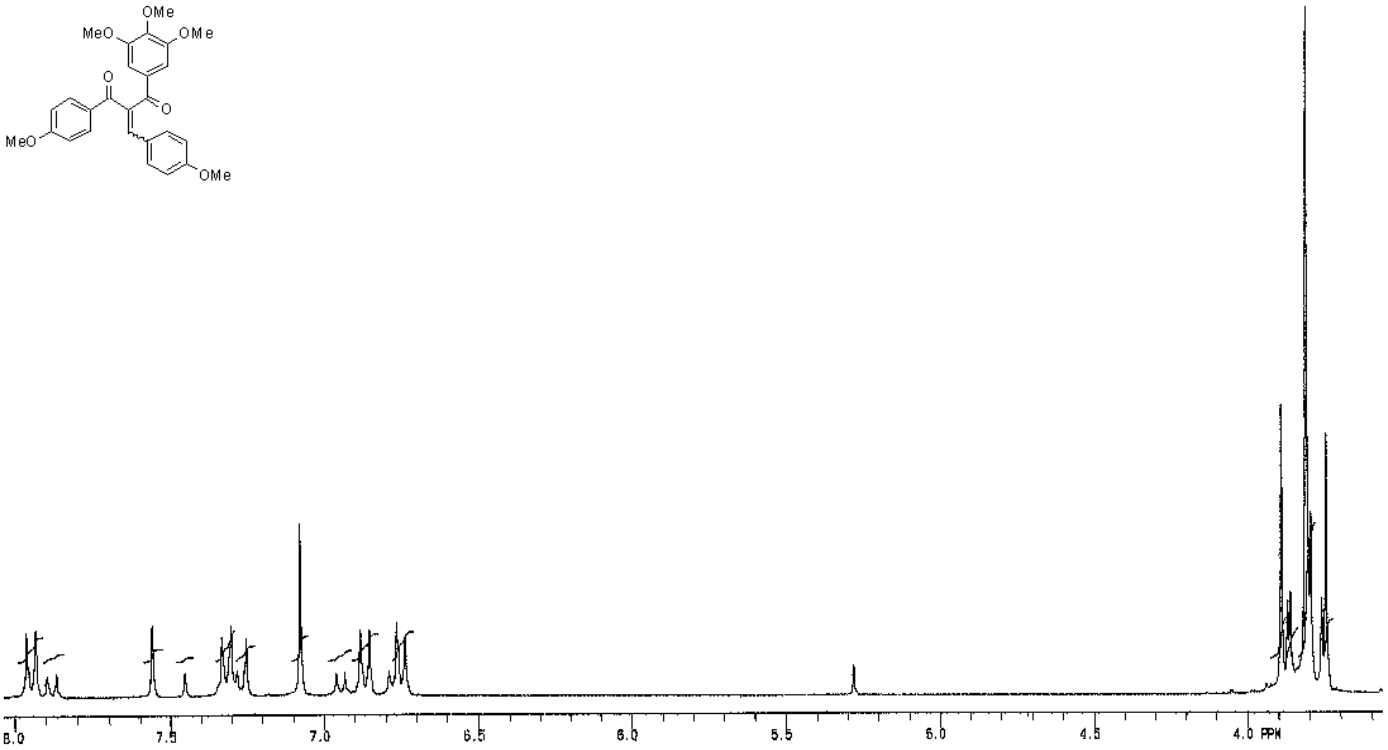


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

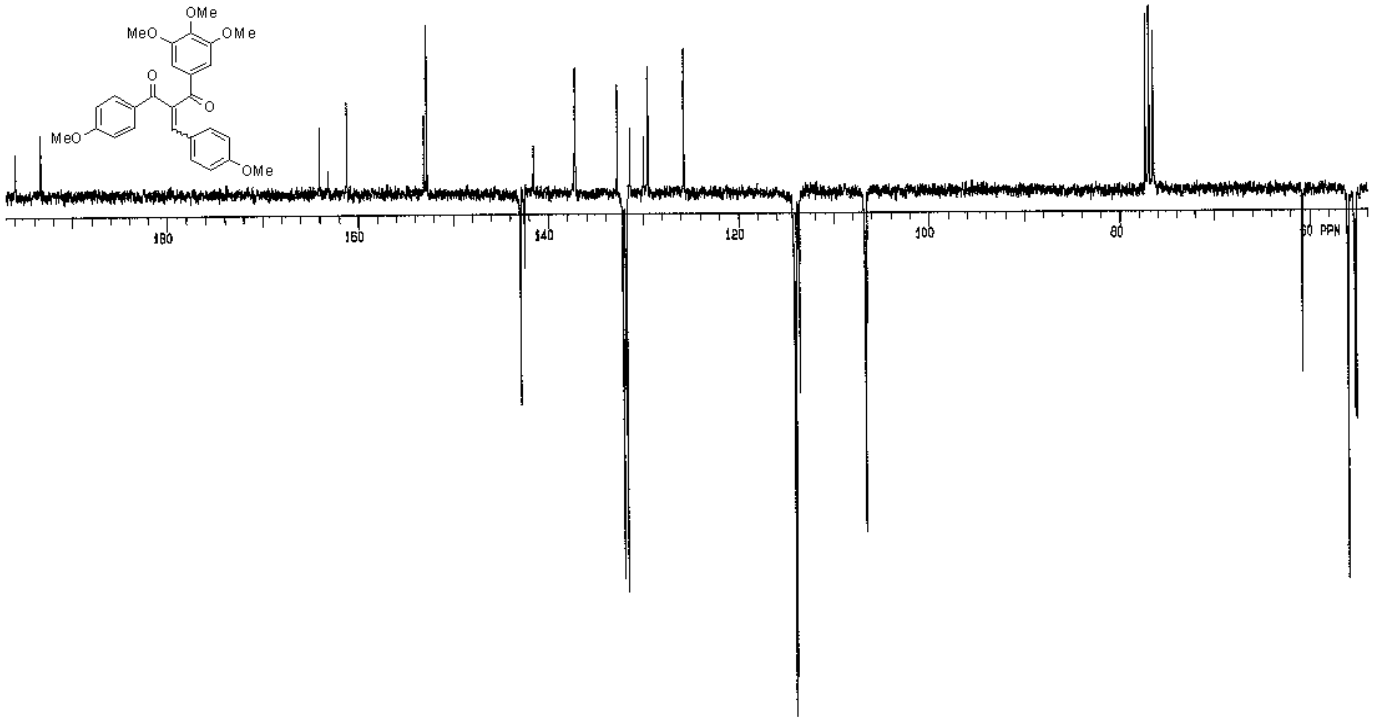


2-(4-Methoxybenzylidene)-1-(4-methoxyphenyl)-3-(3,4,5-trimethoxyphenyl)propane-1,3-dione (16h)

^{1}H NMR (300 MHz, $CDCl_3$)

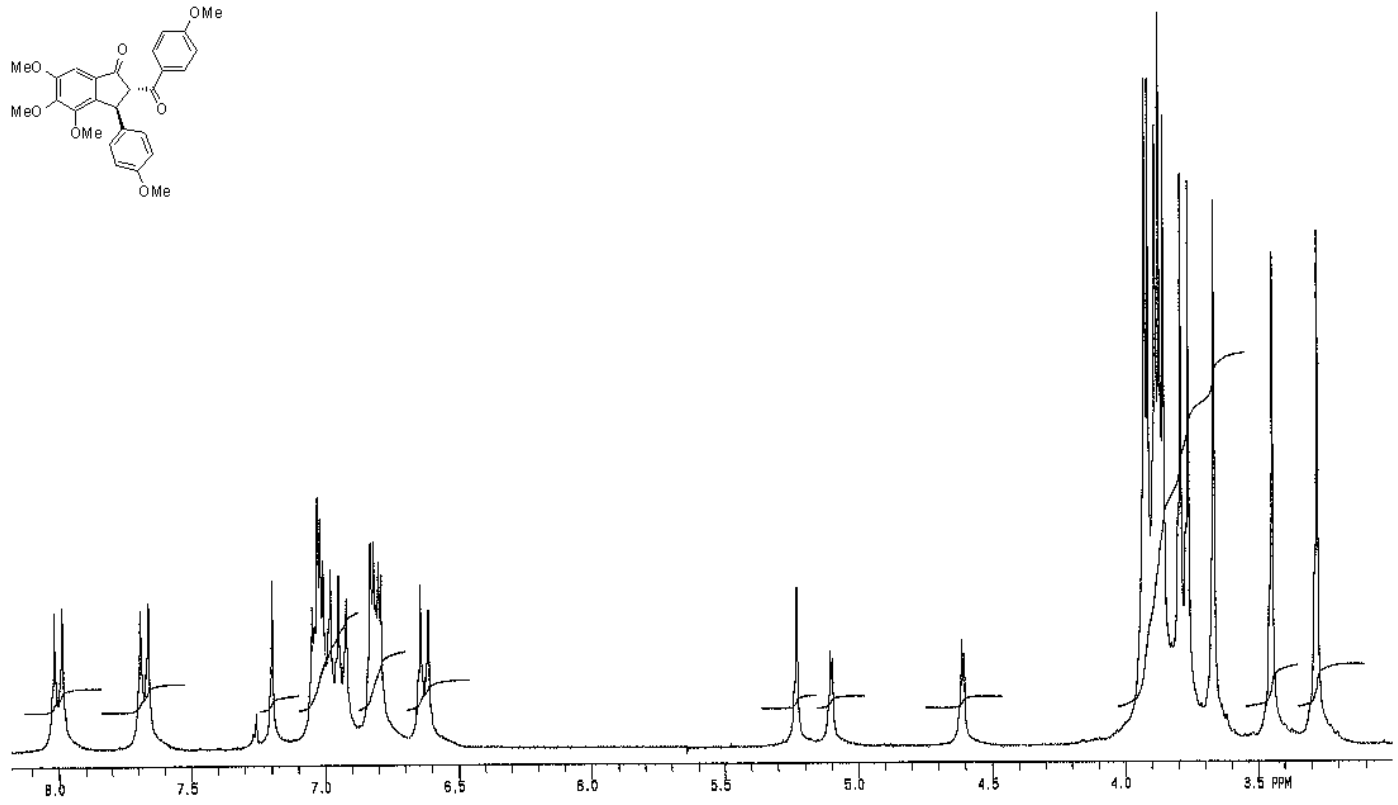


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

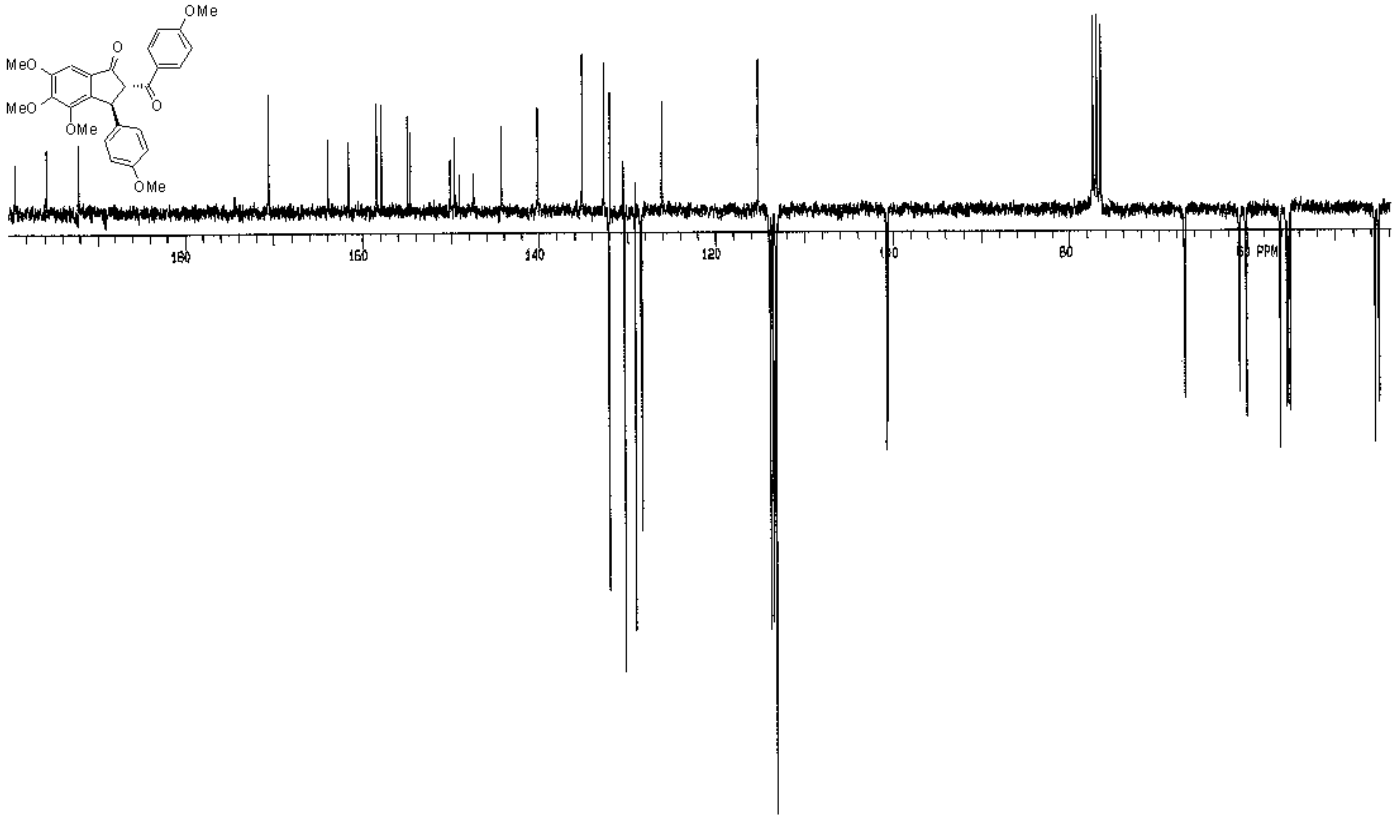


(±)-*trans*-4,5,6-Trimethoxy-2-(4-methoxybenzoyl)-3-(4-methoxyphenyl)-2,3-dihydro-1*H*-inden-1-one (17h)

^{1}H NMR (300 MHz, $CDCl_3$)

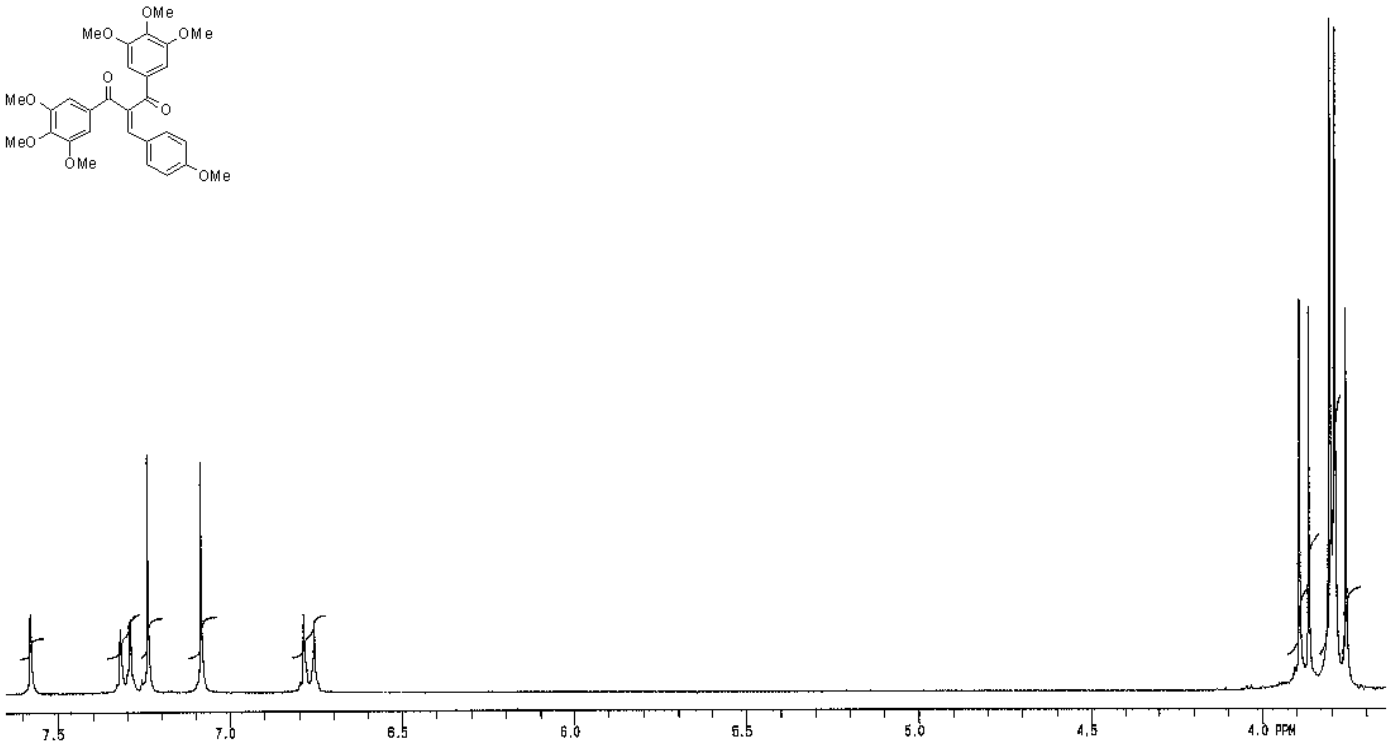


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

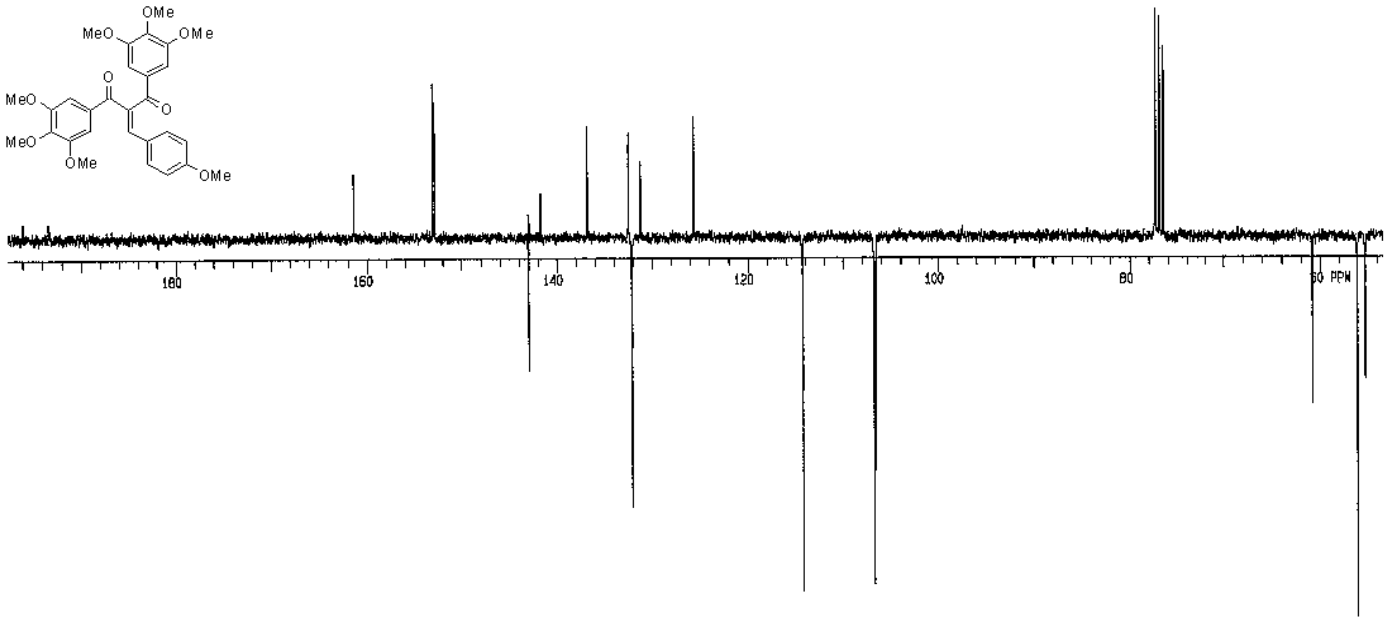


2-(4-Methoxybenzylidene)-1,3-bis(3,4,5-trimethoxyphenyl)propane-1,3-dione (16i)

^{1}H NMR (300 MHz, $CDCl_3$)

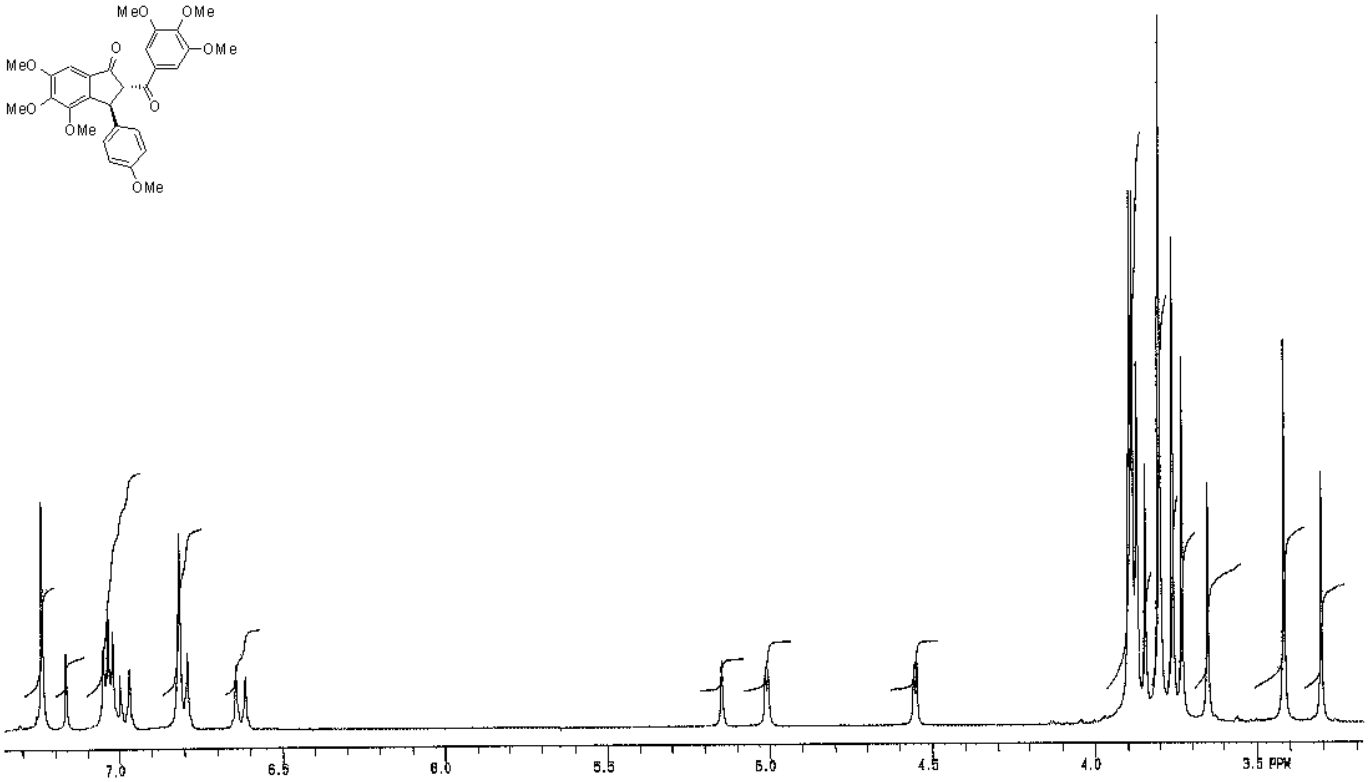


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

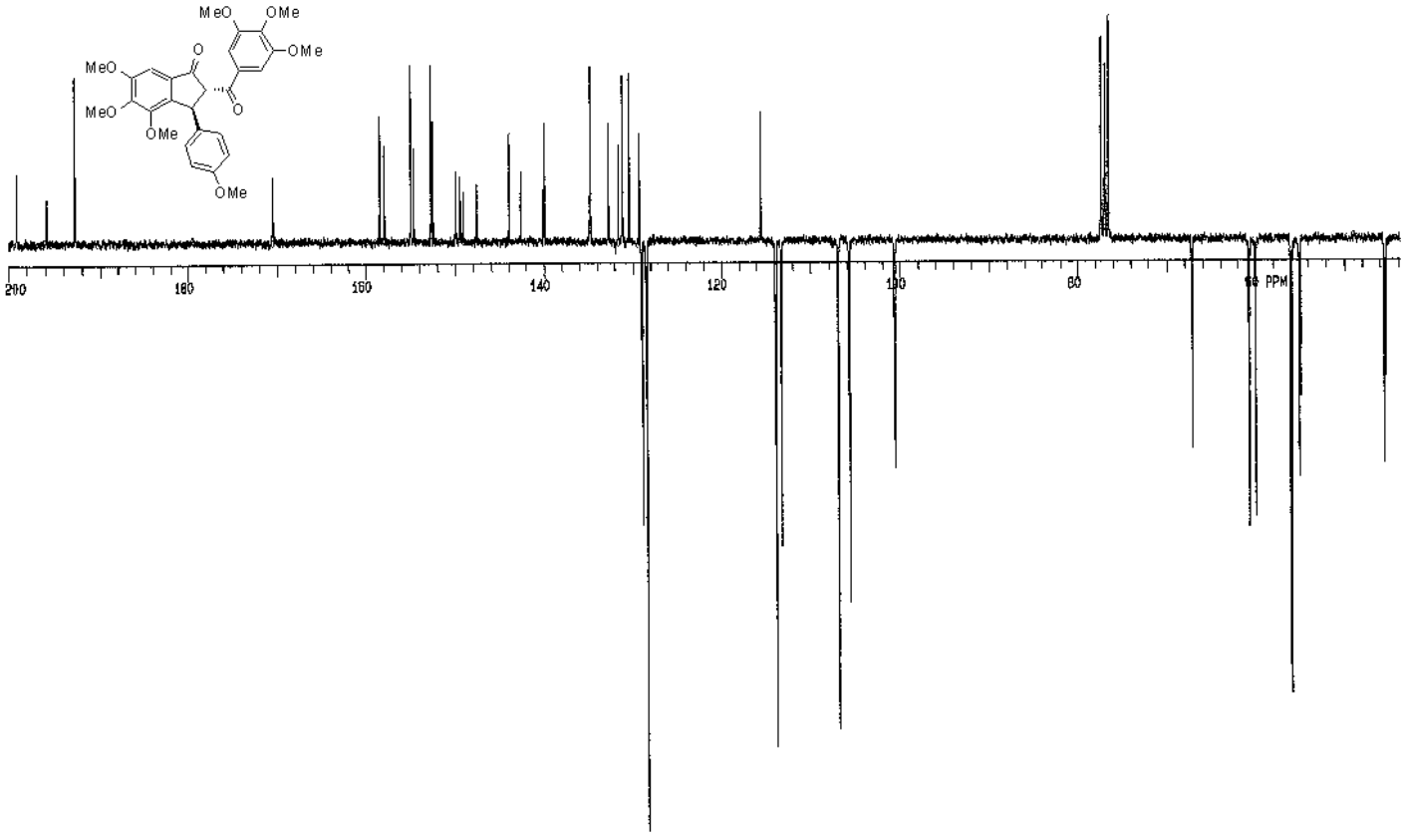


(±)-*trans*-4,5,6-Trimethoxy-3-(4-methoxyphenyl)-2-(3,4,5-trimethoxybenzoyl)-2,3-dihydro-1*H*-inden-1-one (17i)

^{1}H NMR (300 MHz, $CDCl_3$)

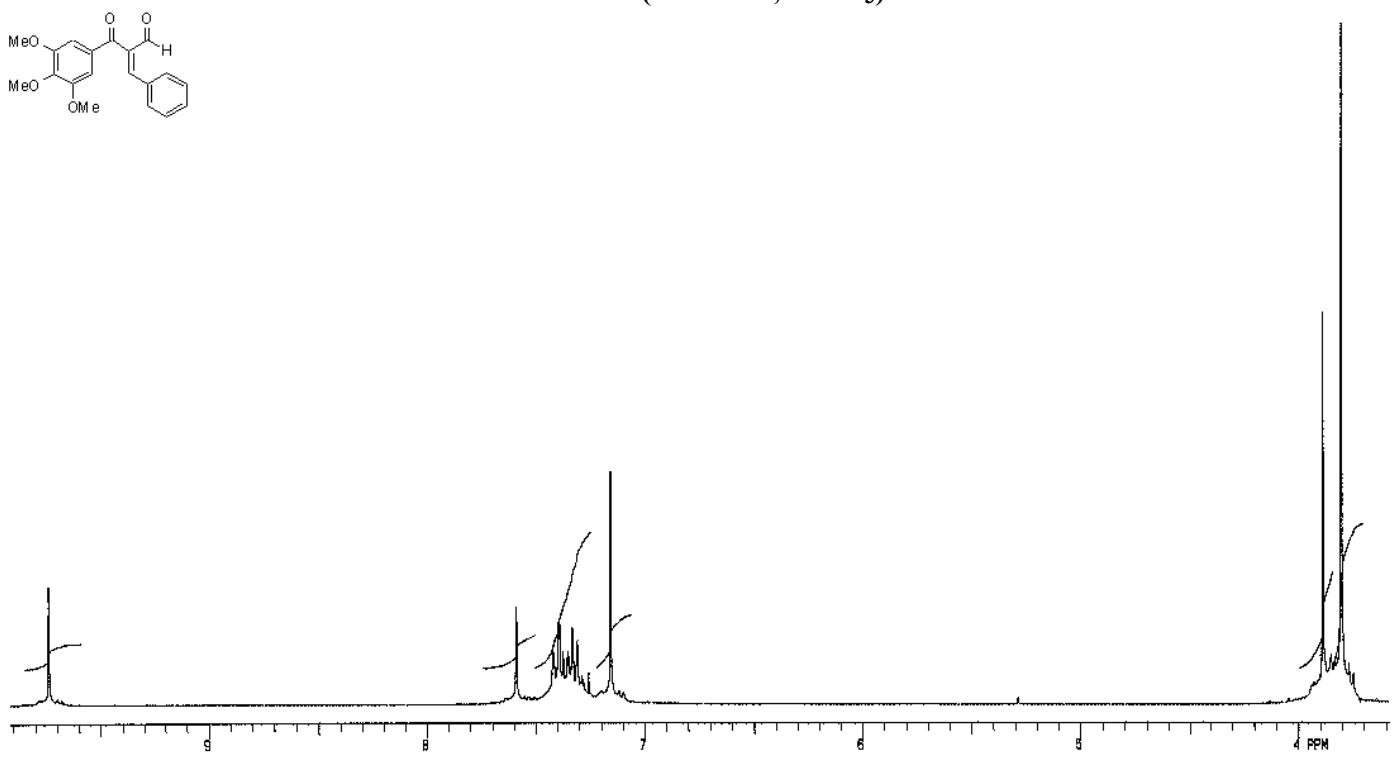


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

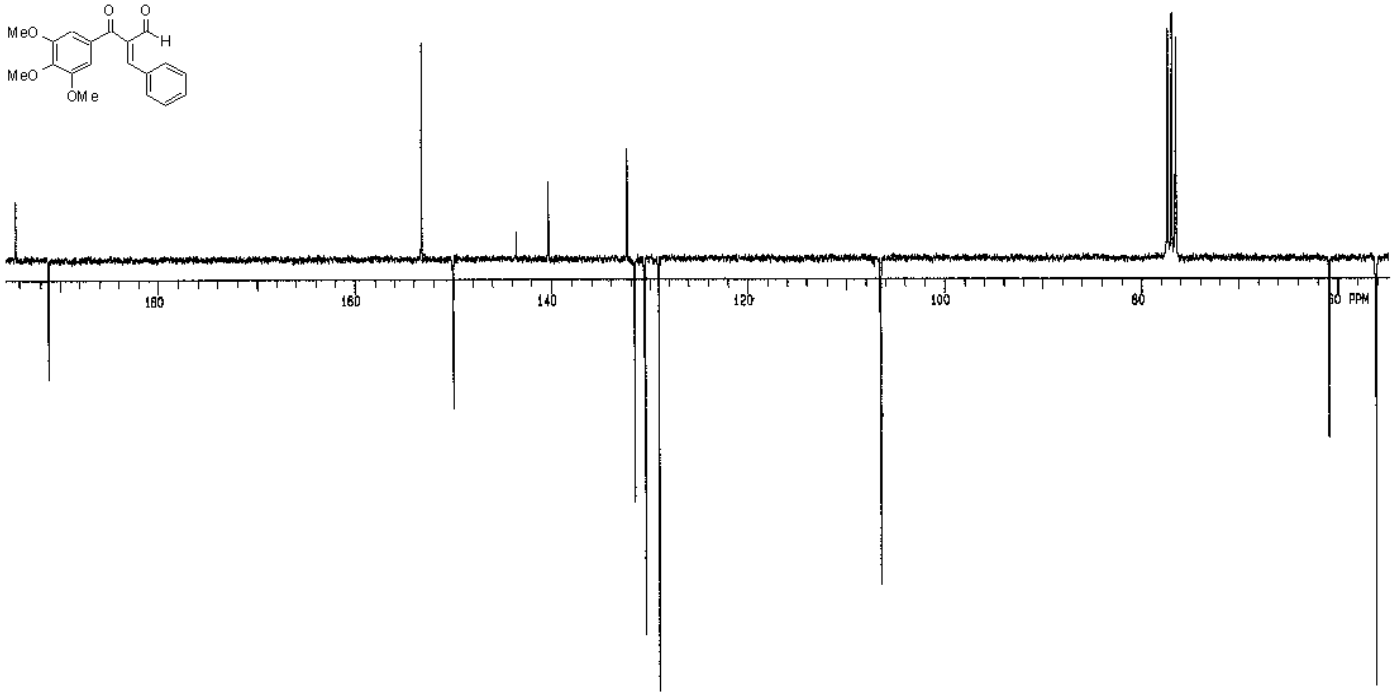


(*E*)-3-Phenyl-2-(3,4,5-trimethoxybenzoyl)-2-propenal (16j)

^{1}H NMR (300 MHz, $CDCl_3$)

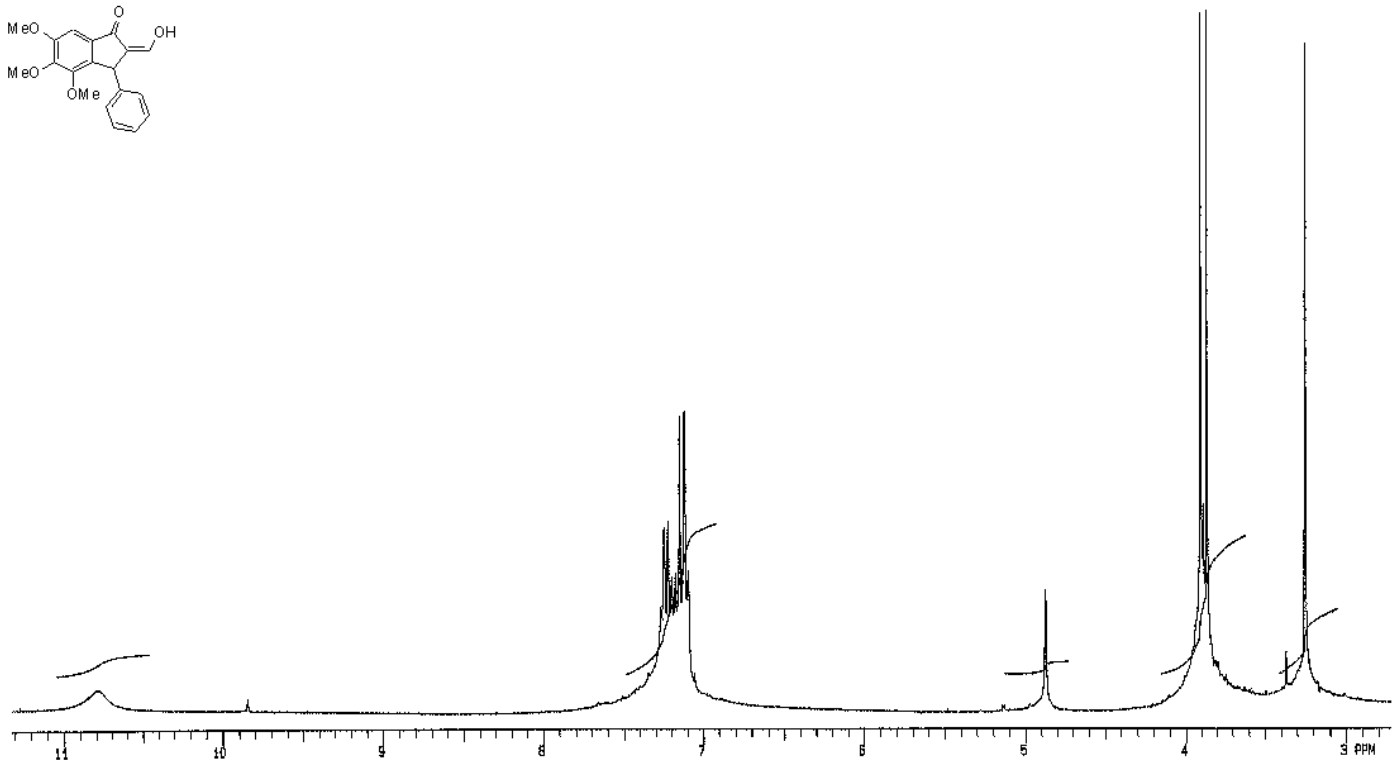


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

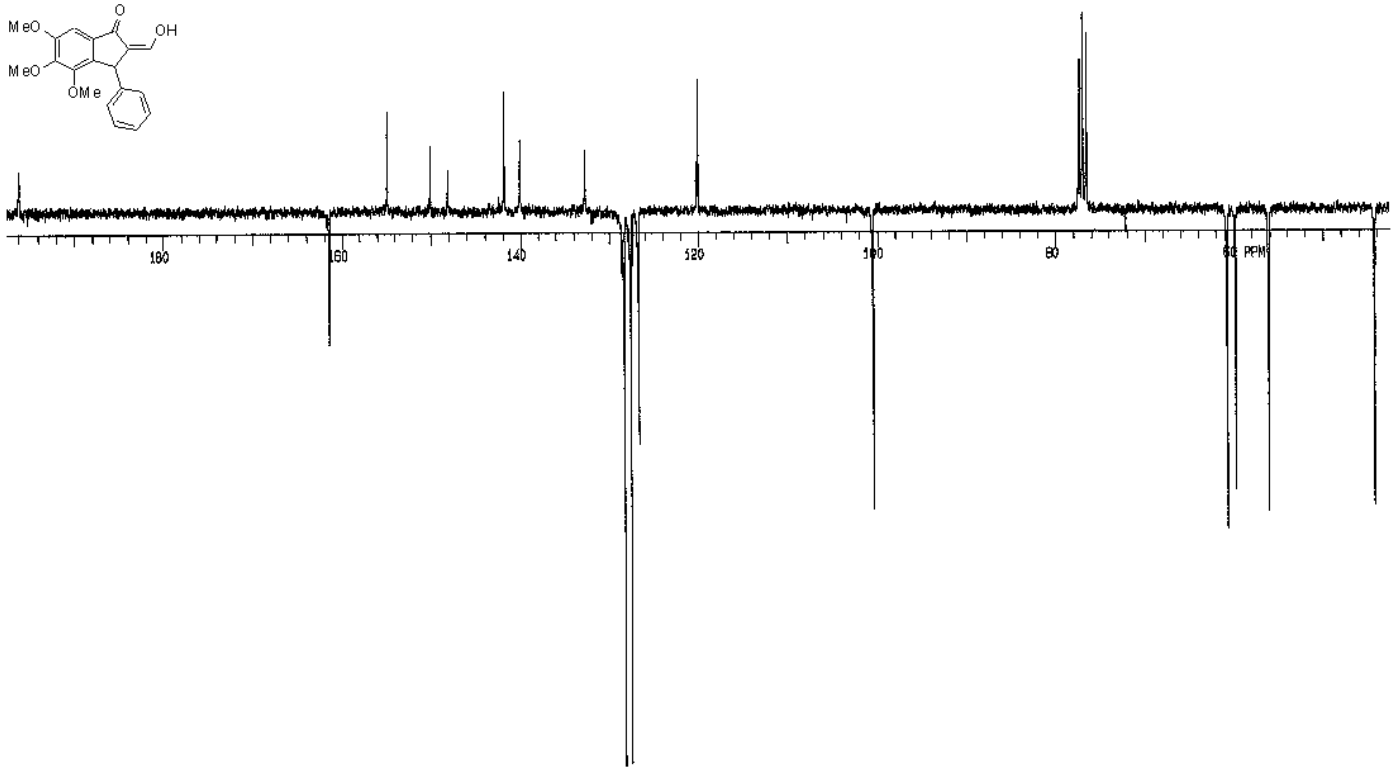


(±)-2-Hydroxymethylene-4,5,6-trimethoxy-3-phenyl-indan-1-one (17j)

^{1}H NMR (300 MHz, $CDCl_3$)

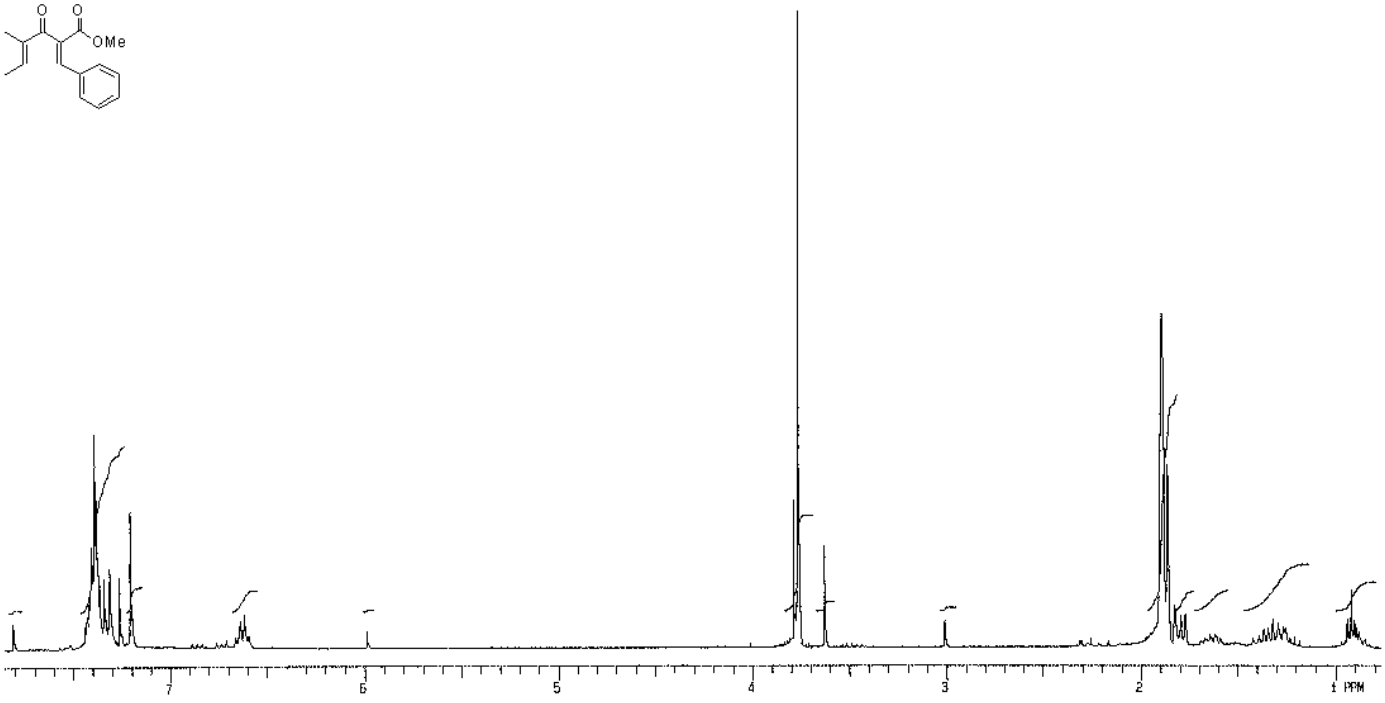


^{13}C NMR (APT, 75 MHz, $CDCl_3$)



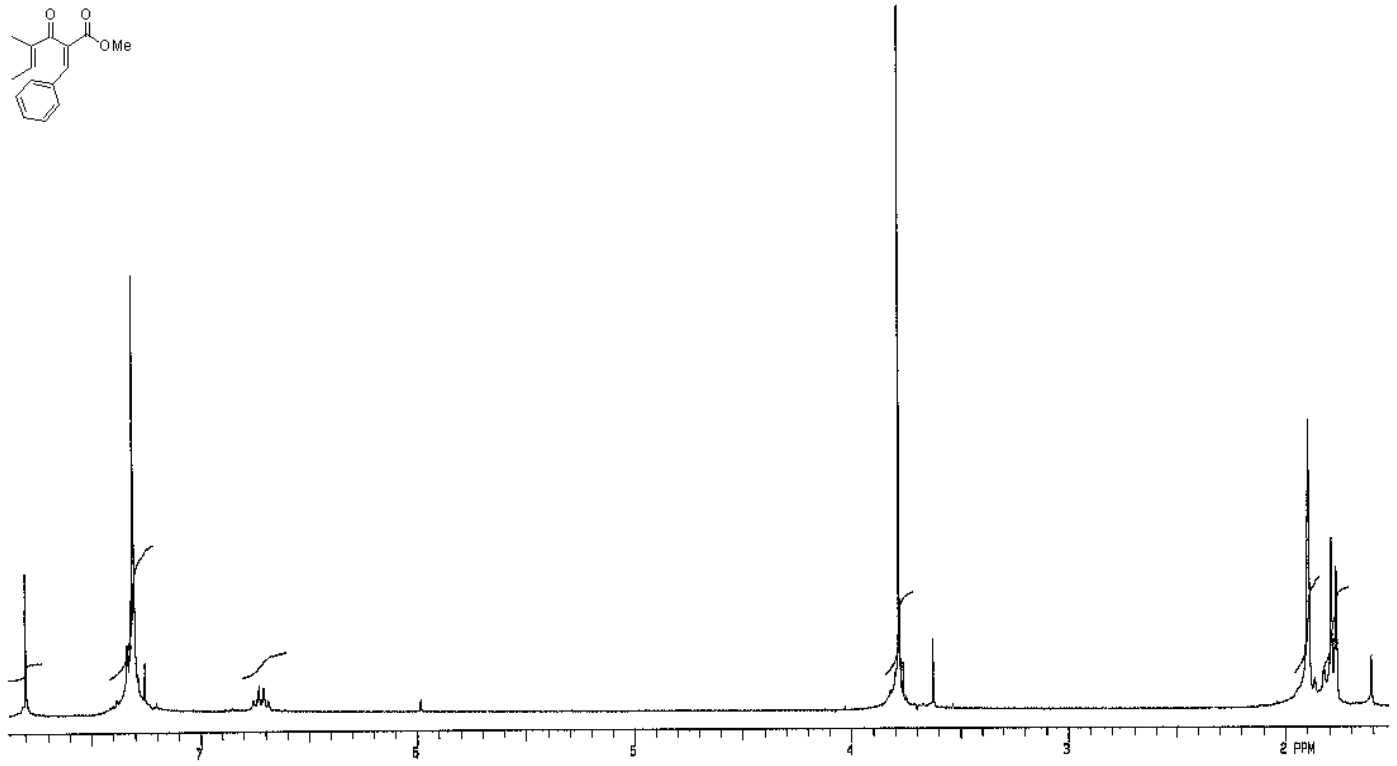
Methyl (2*Z*,4*E*)-2-benzylidene-4-methyl-3-oxohex-4-enoate (*Z*-16k)

^{1}H NMR (300 MHz, $CDCl_3$)

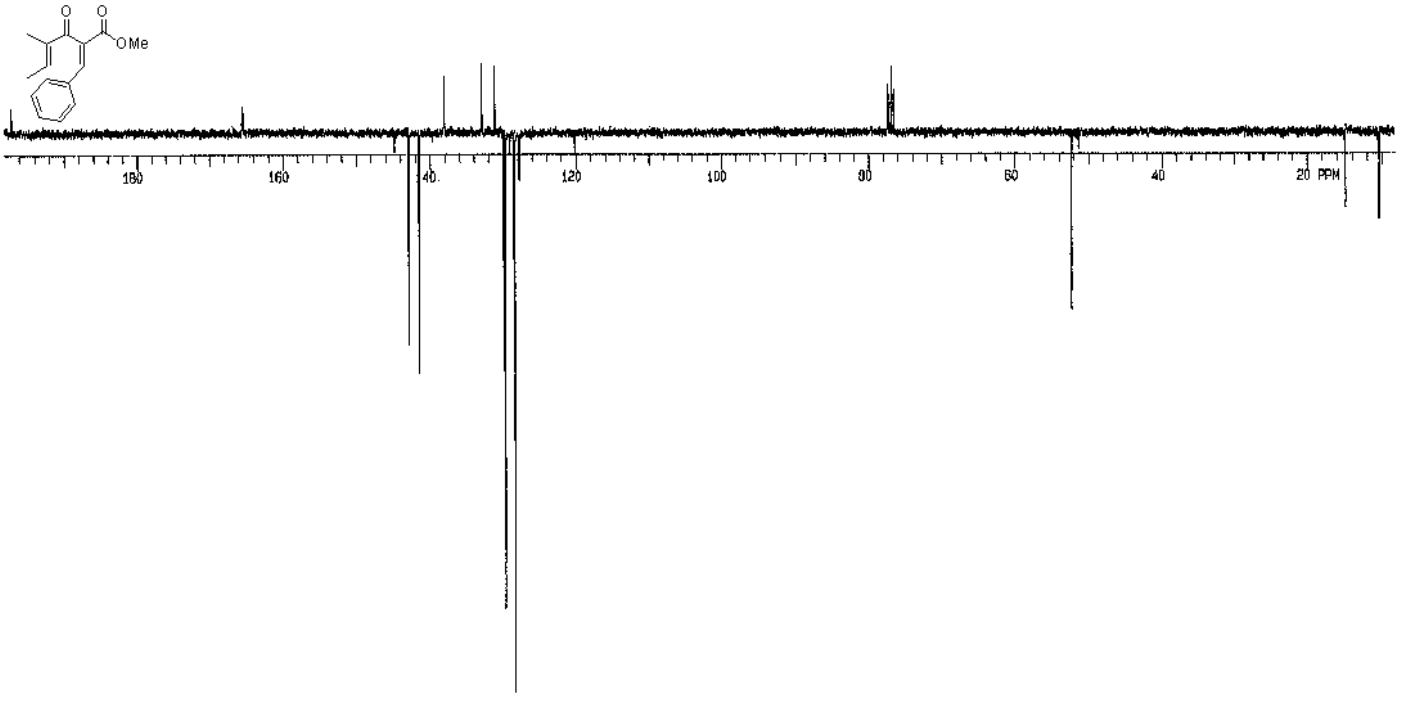


Methyl (2*E*,4*E*)-2-benzylidene-4-methyl-3-oxohex-4-enoate (*E*-16k)

^{1}H NMR (300 MHz, $CDCl_3$)

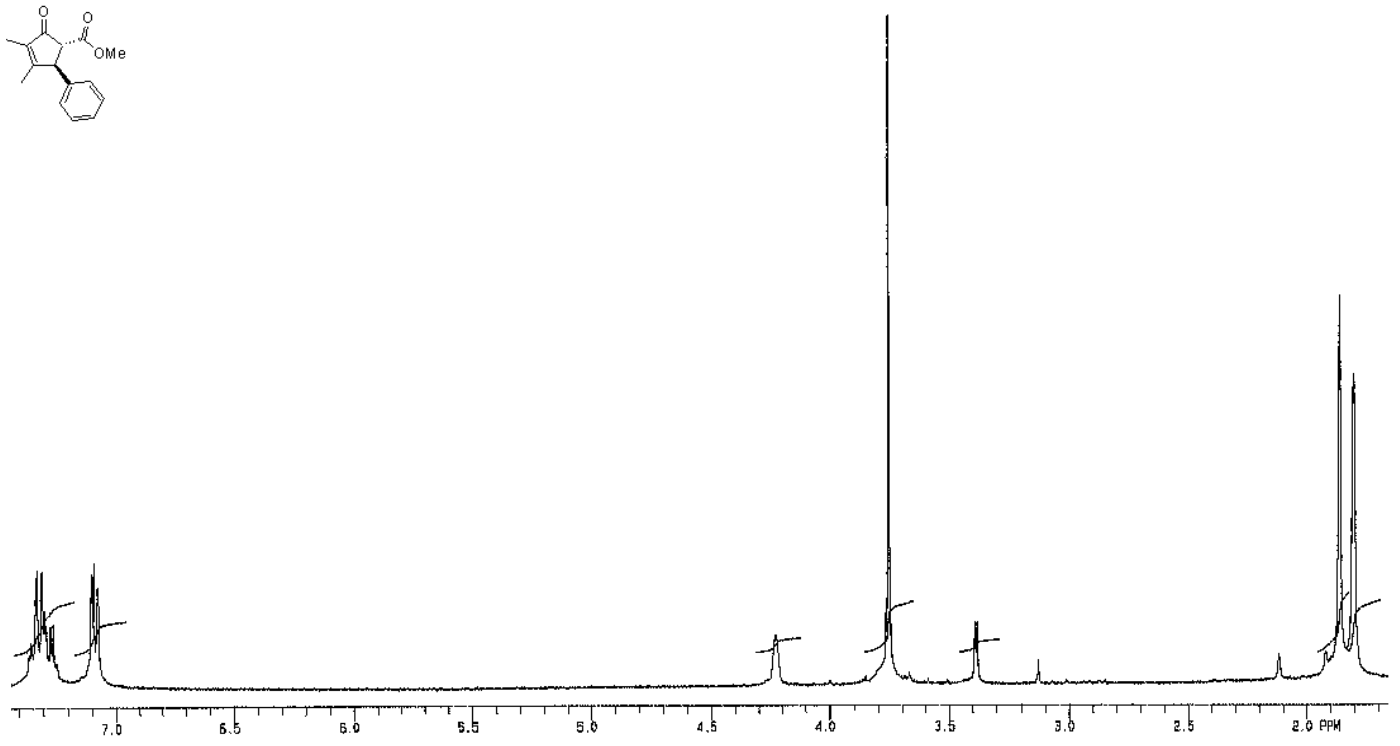


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

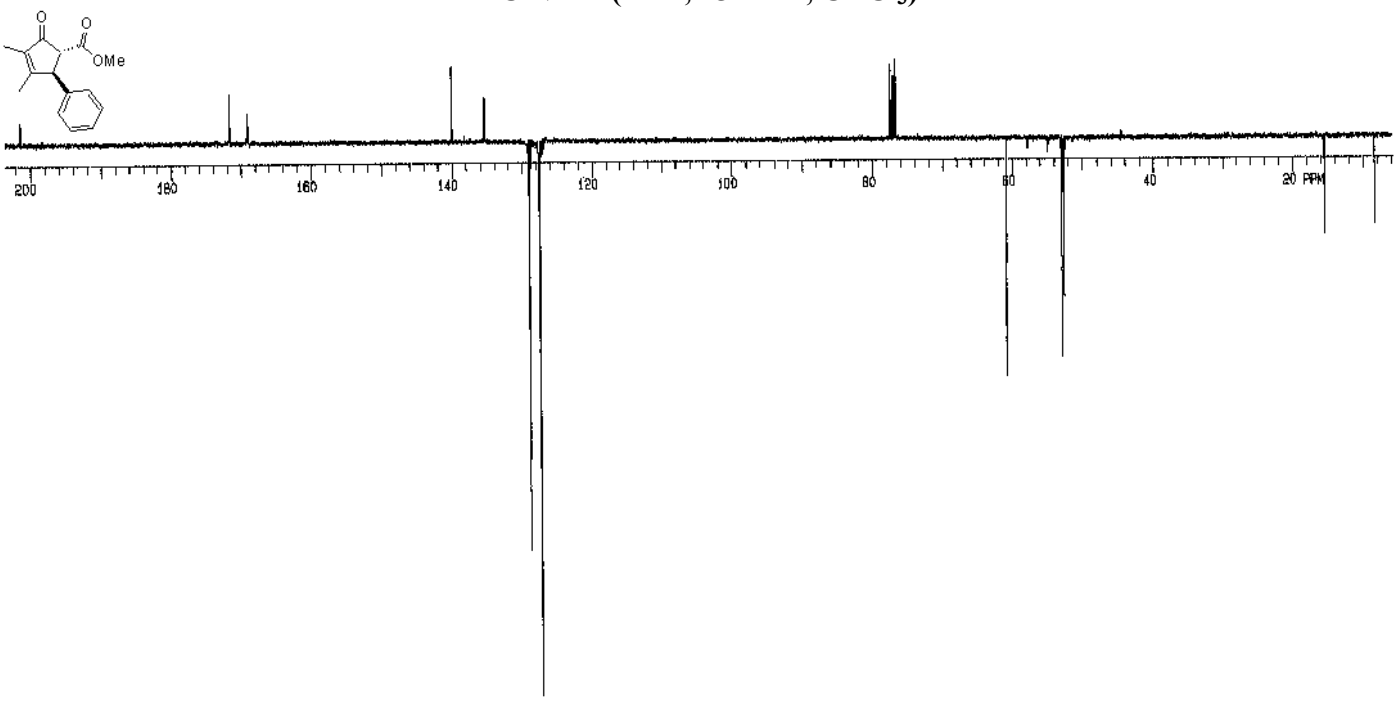


(±)-Methyl *trans*-3,4-dimethyl-2-oxo-5-phenylcyclopent-3-enecarboxylate (17k)

^{1}H NMR (300 MHz, $CDCl_3$)

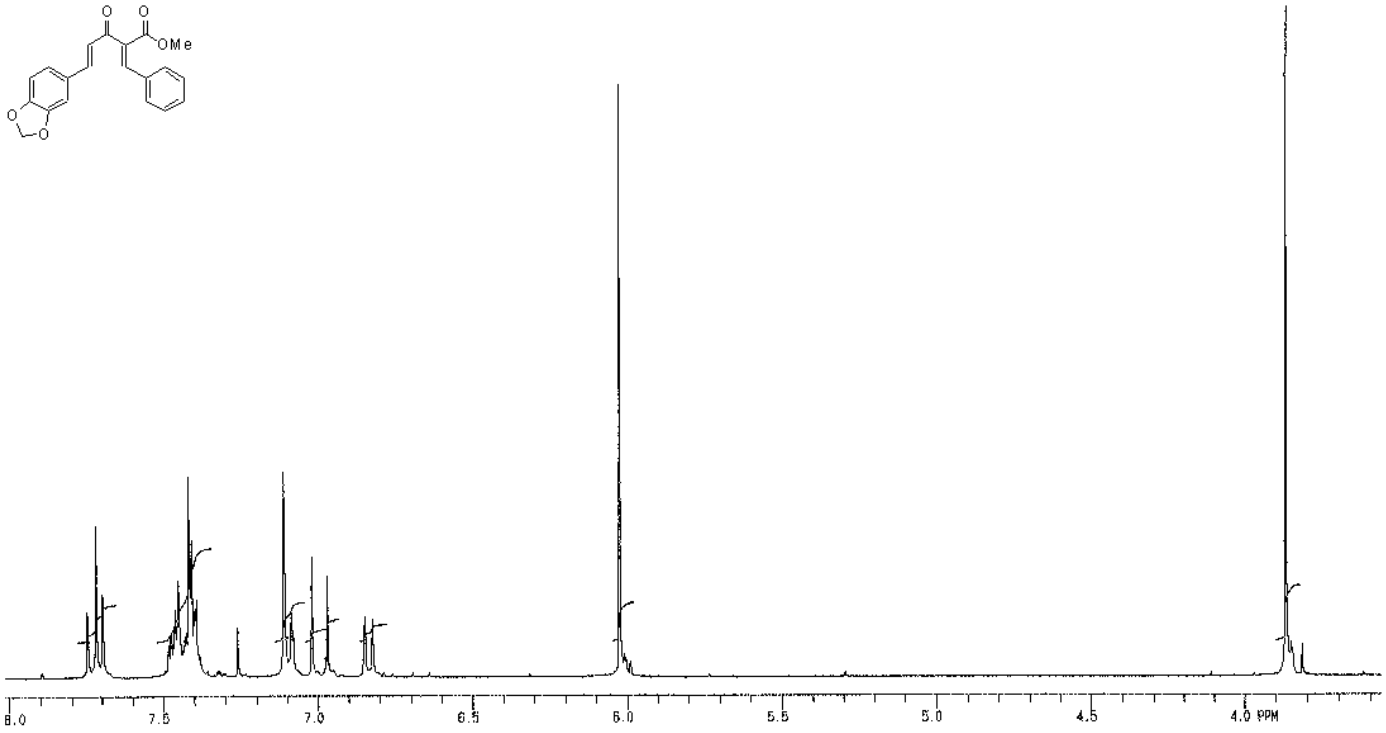


^{13}C NMR (APT, 75 MHz, $CDCl_3$)



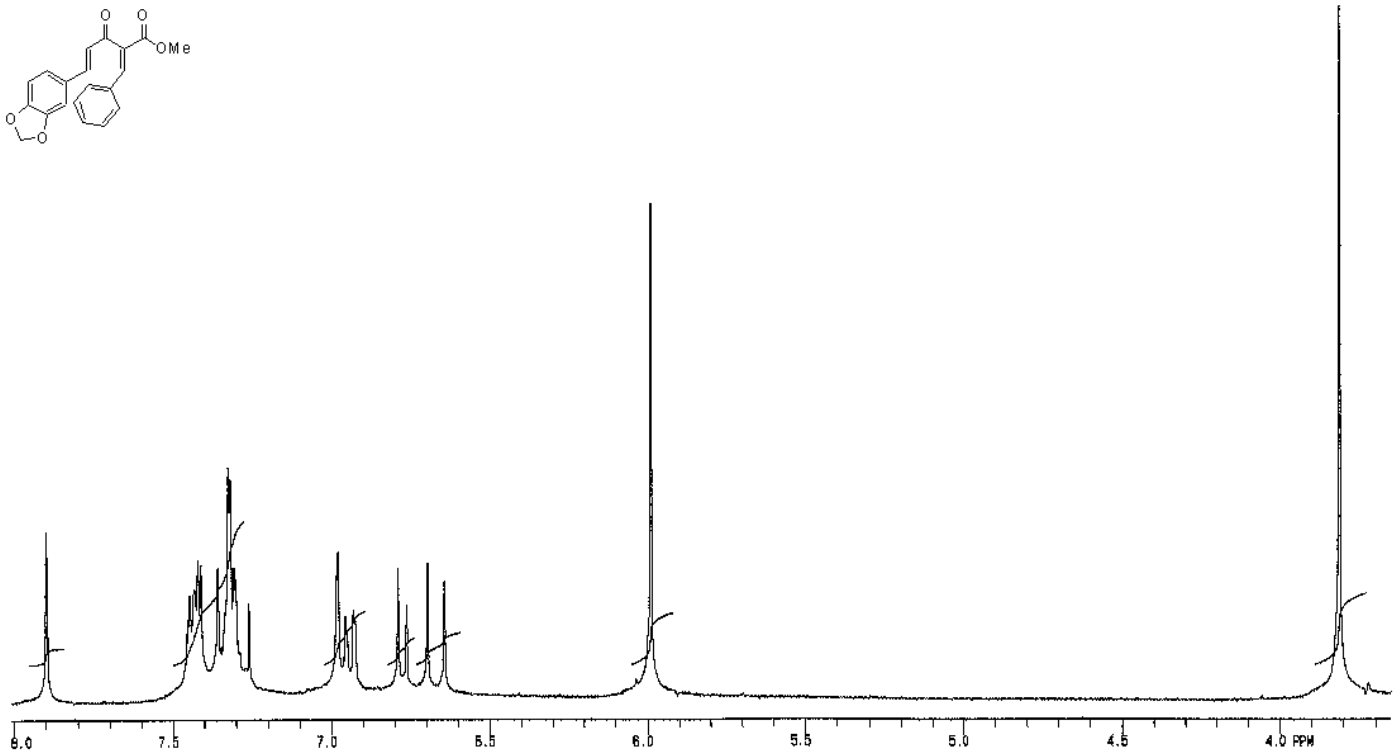
Methyl (2*Z*,4*E*)-5-(benzo[*d*][1,3]dioxol-5-yl)-2-benzylidene-3-oxopent-4-enoate (*Z*-16l)

^{1}H NMR (300 MHz, $CDCl_3$)

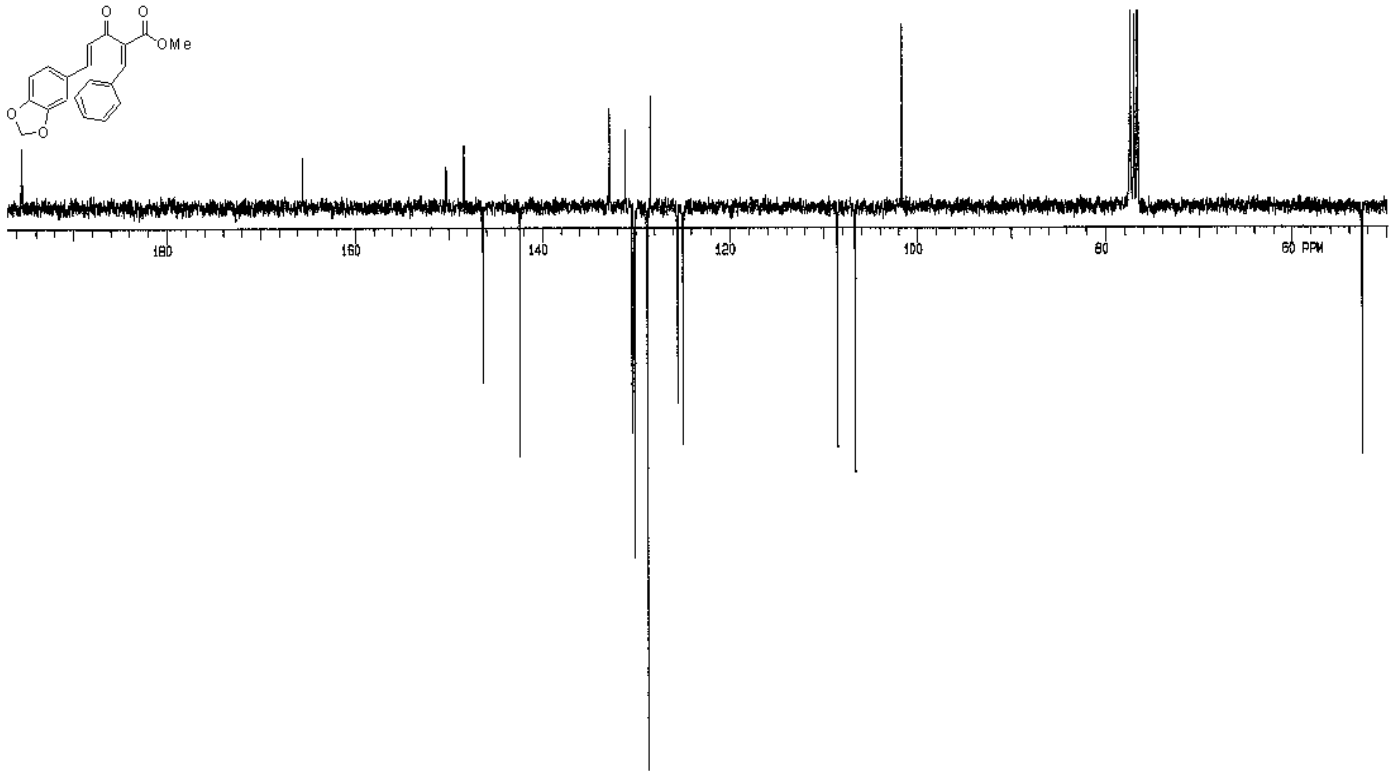


Methyl (2*E*,4*E*)-5-(benzo[*d*][1,3]dioxol-5-yl)-2-benzylidene-3-oxopent-4-enoate (*E*-16l)

^{1}H NMR (300 MHz, $CDCl_3$)

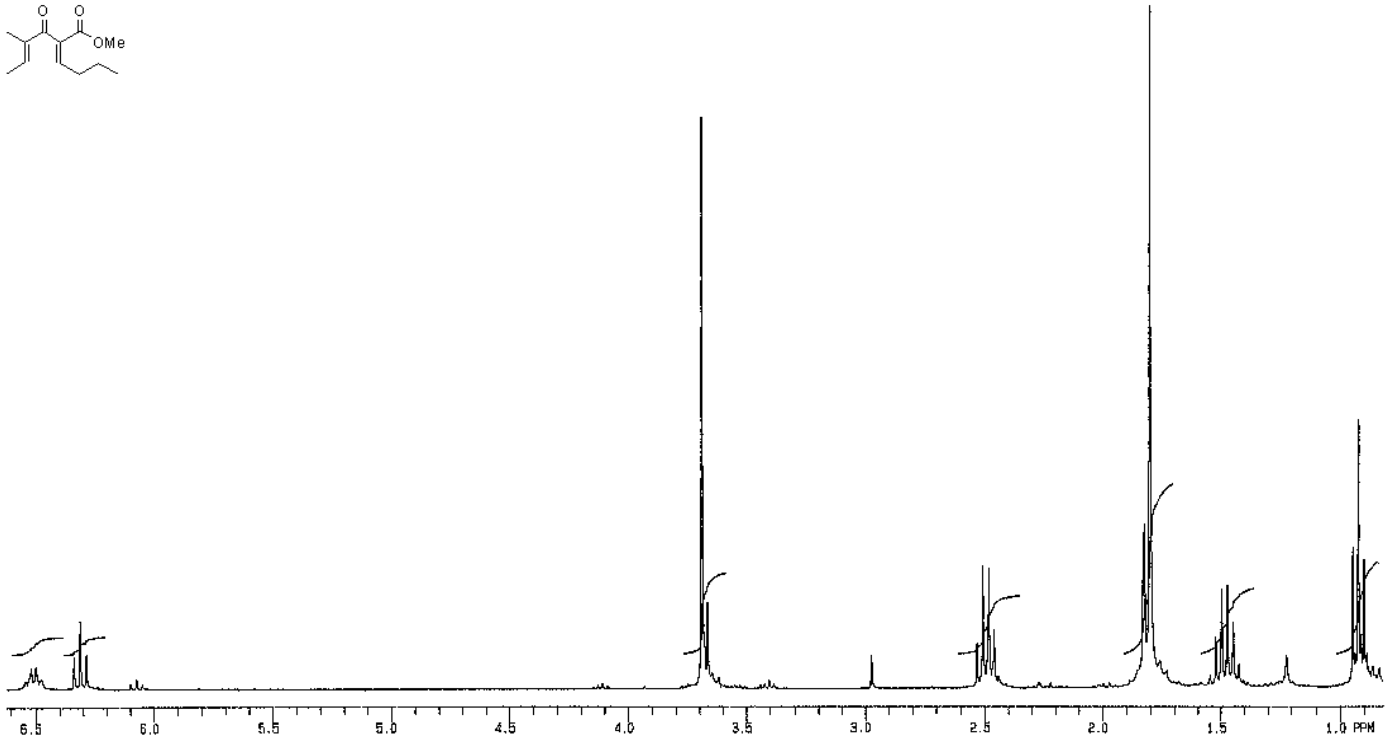


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

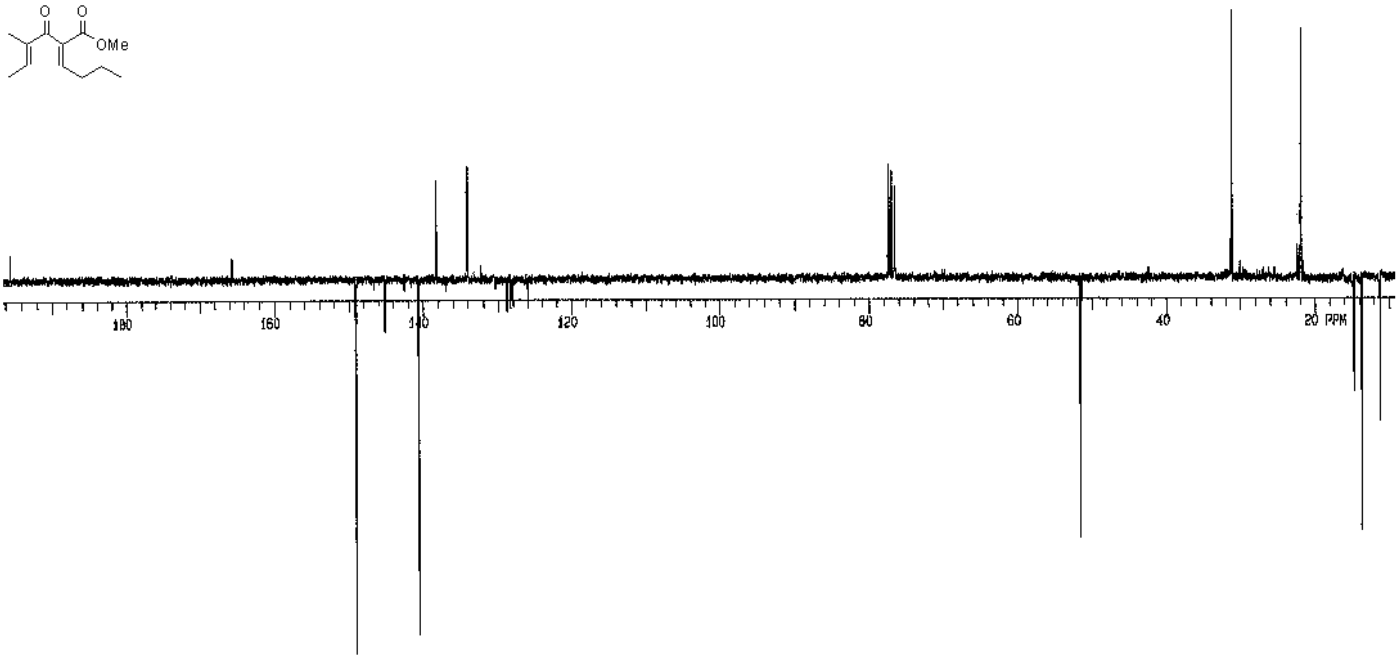


Methyl (Z)-2-[(*E*)-2-methylbut-2-enoyl]hex-2-enoate (*Z*-16m)

^{1}H NMR (300 MHz, $CDCl_3$)

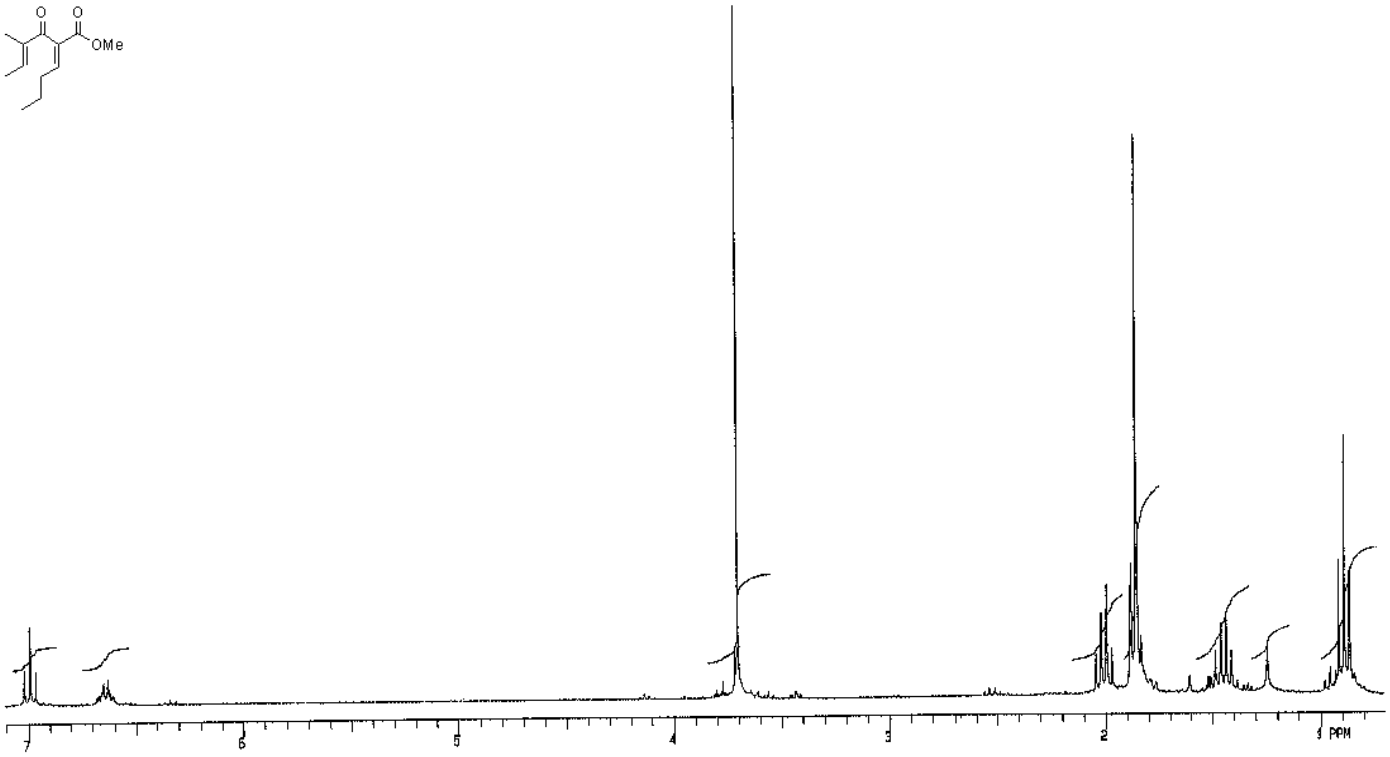


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

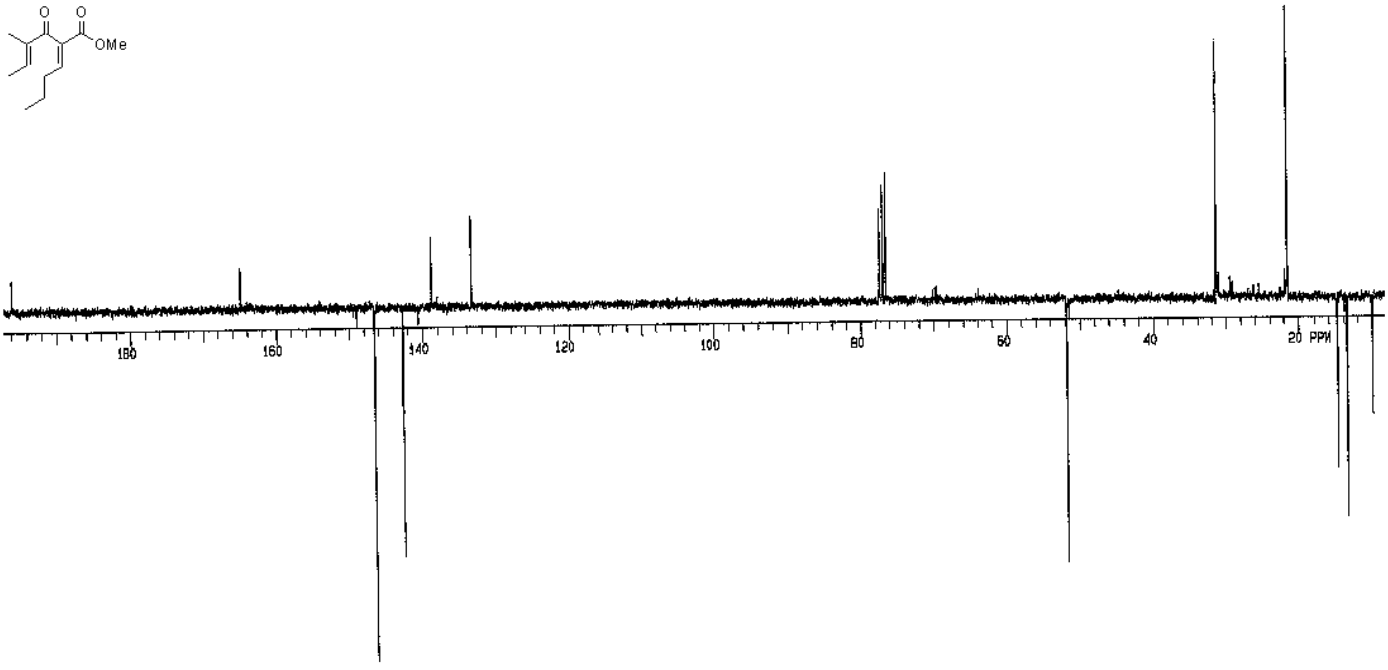


Methyl (*E*)-2-[(*E*)-2-methylbut-2-enoyl]hex-2-enoate (*E*-16m)

^{1}H NMR (300 MHz, $CDCl_3$)

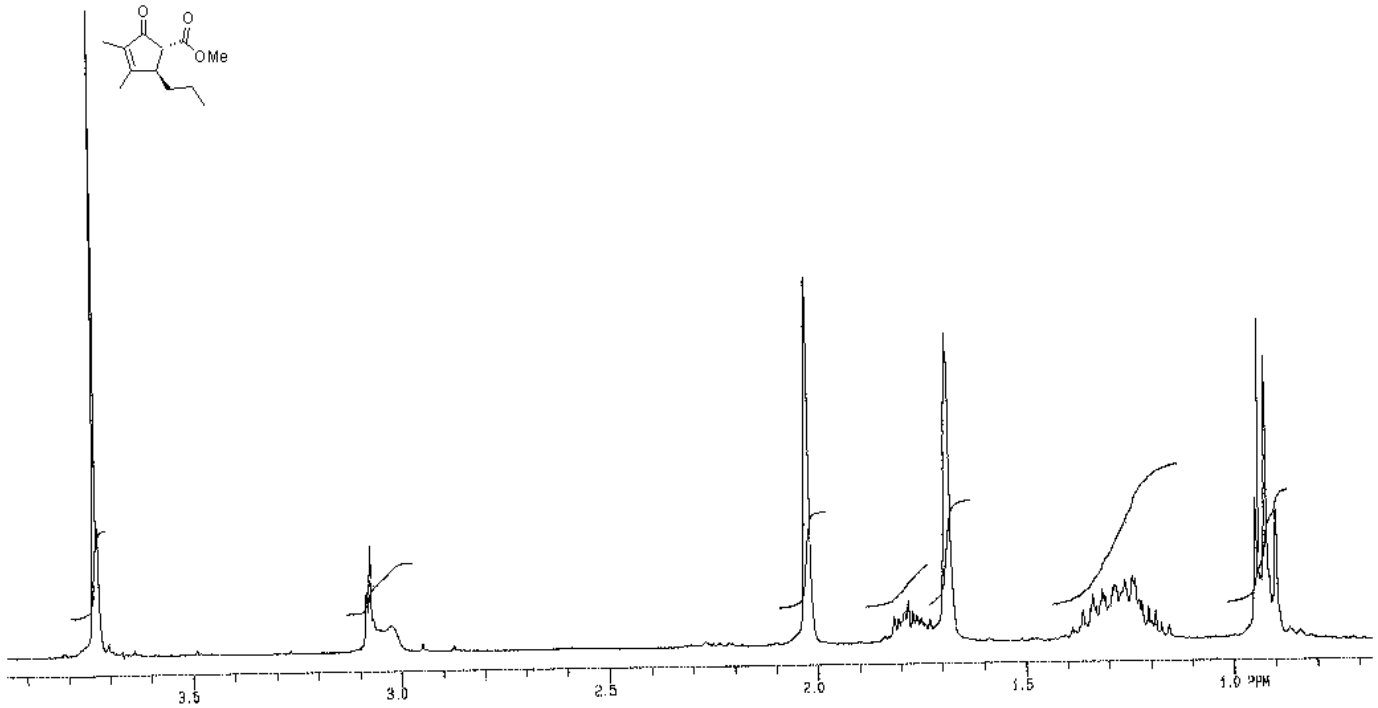


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

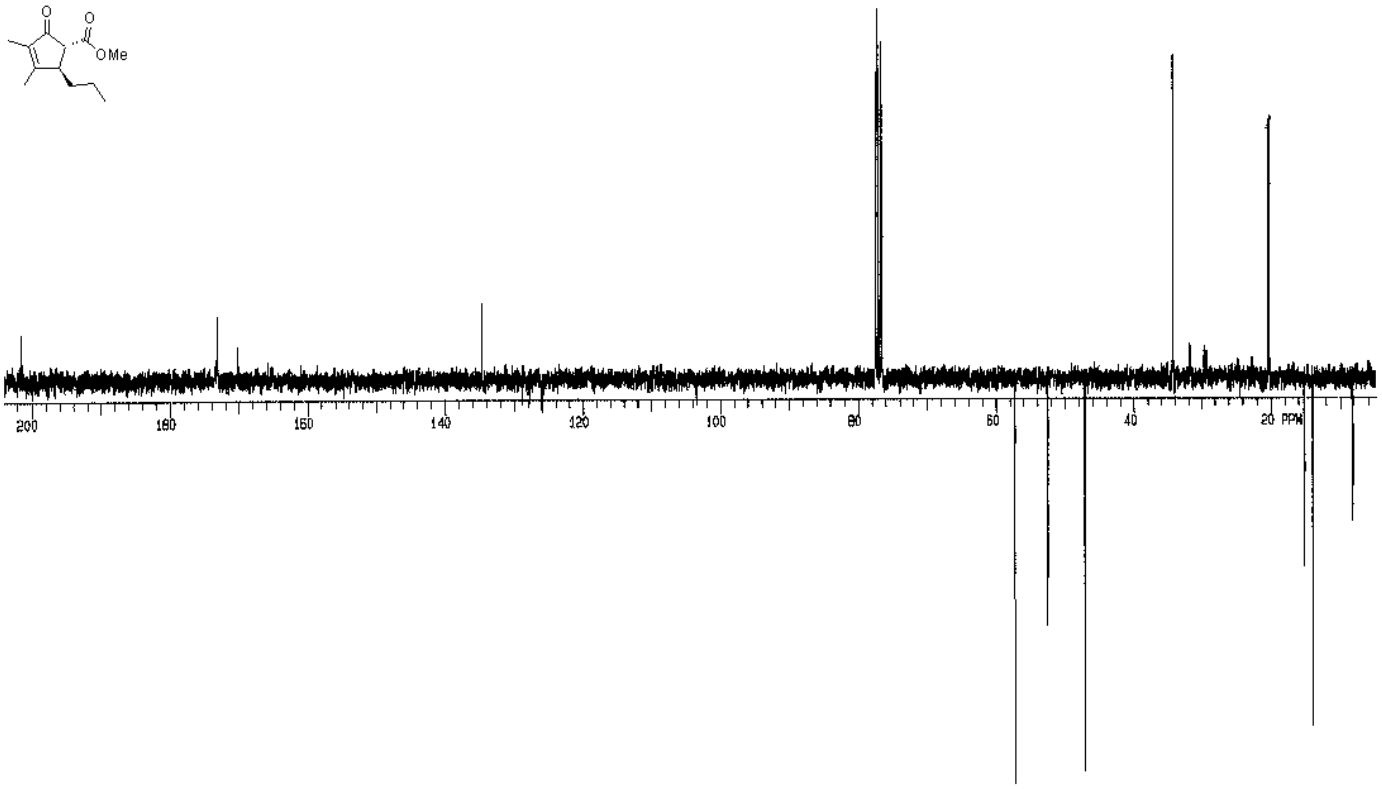


(±)-Methyl *trans*-3,4-dimethyl-2-oxo-5-propylcyclopent-3-enecarboxylate (17m)

^{1}H NMR (300 MHz, $CDCl_3$)

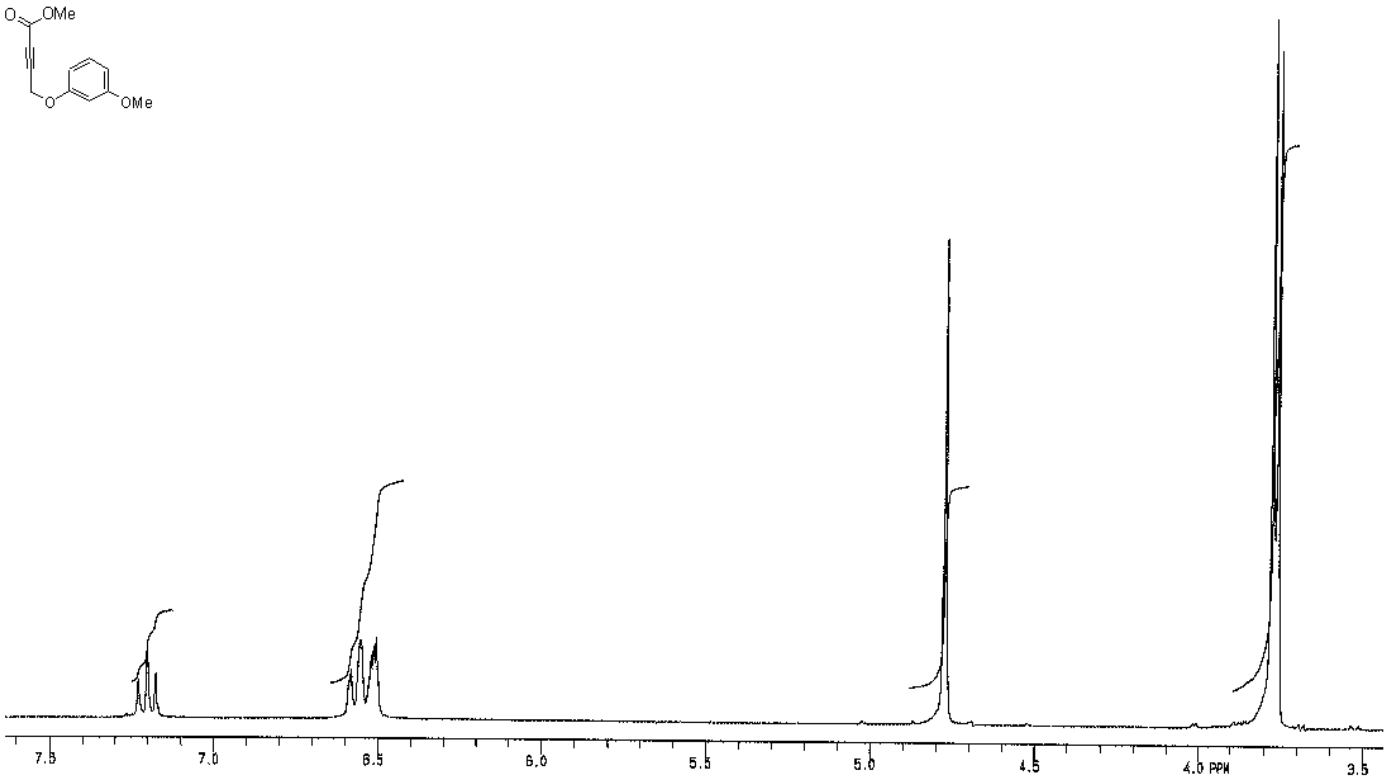


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

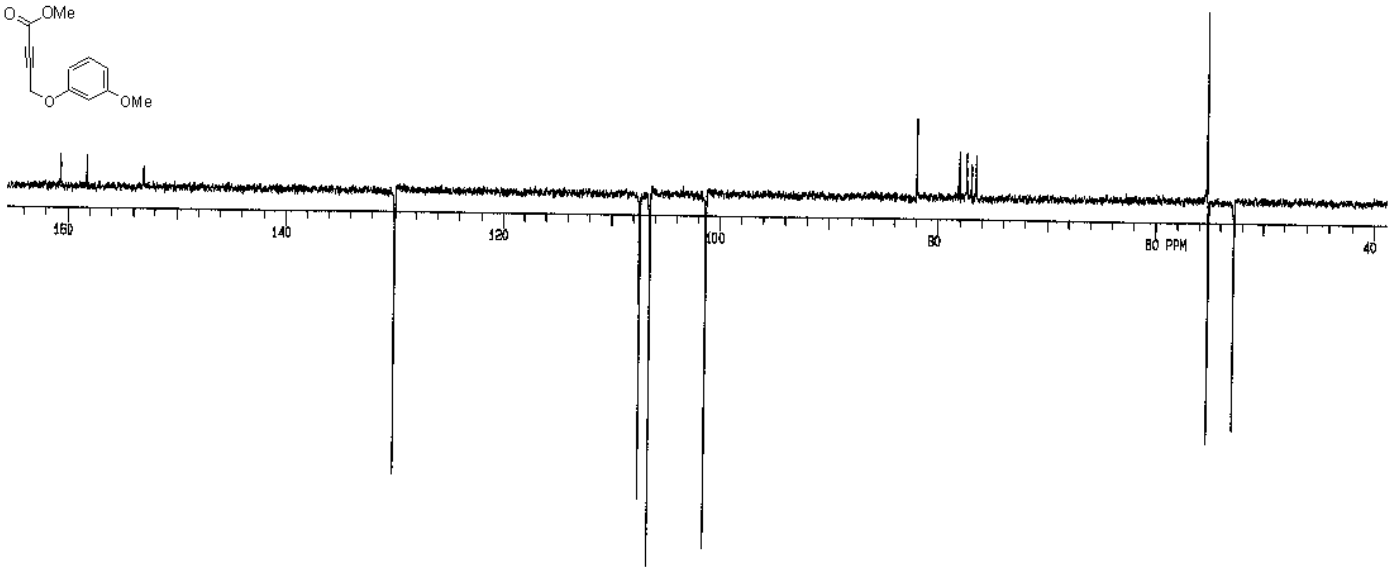


Methyl 4-(3-methoxyphenoxy)-2-butynoate (14f)

^{1}H NMR (300 MHz, $CDCl_3$)

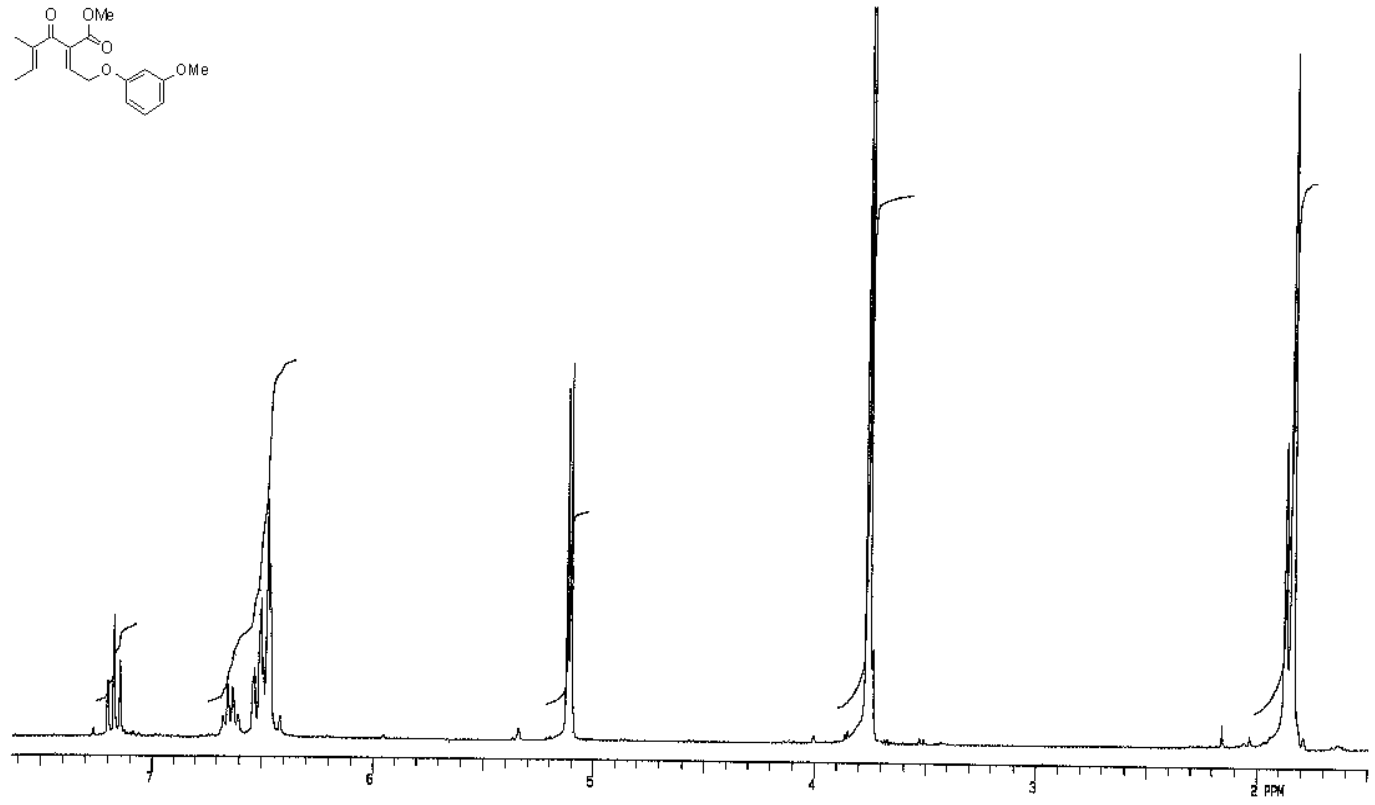


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

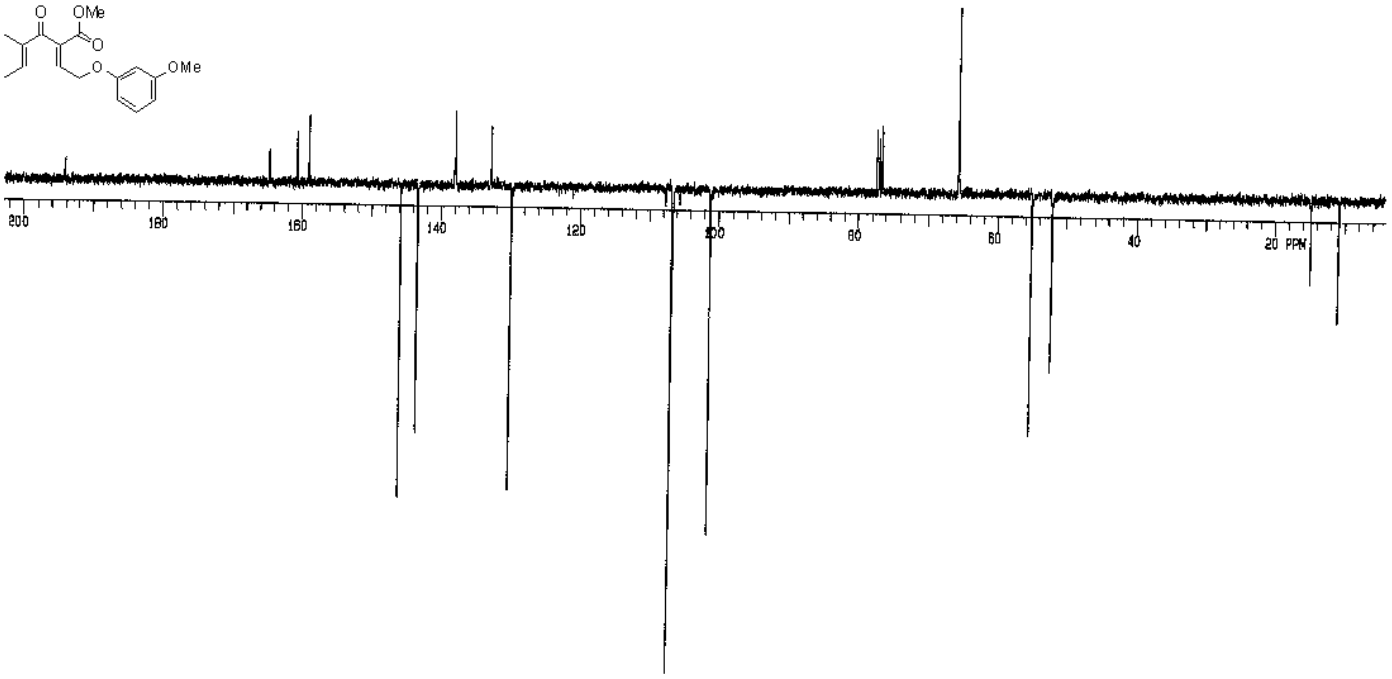


Methyl (2*Z*,4*E*)-2-[2-(3-methoxyphenoxy)ethylidene]-4-methyl-3-oxohex-4-enoate (*Z*-16n)

^{1}H NMR (300 MHz, $CDCl_3$)

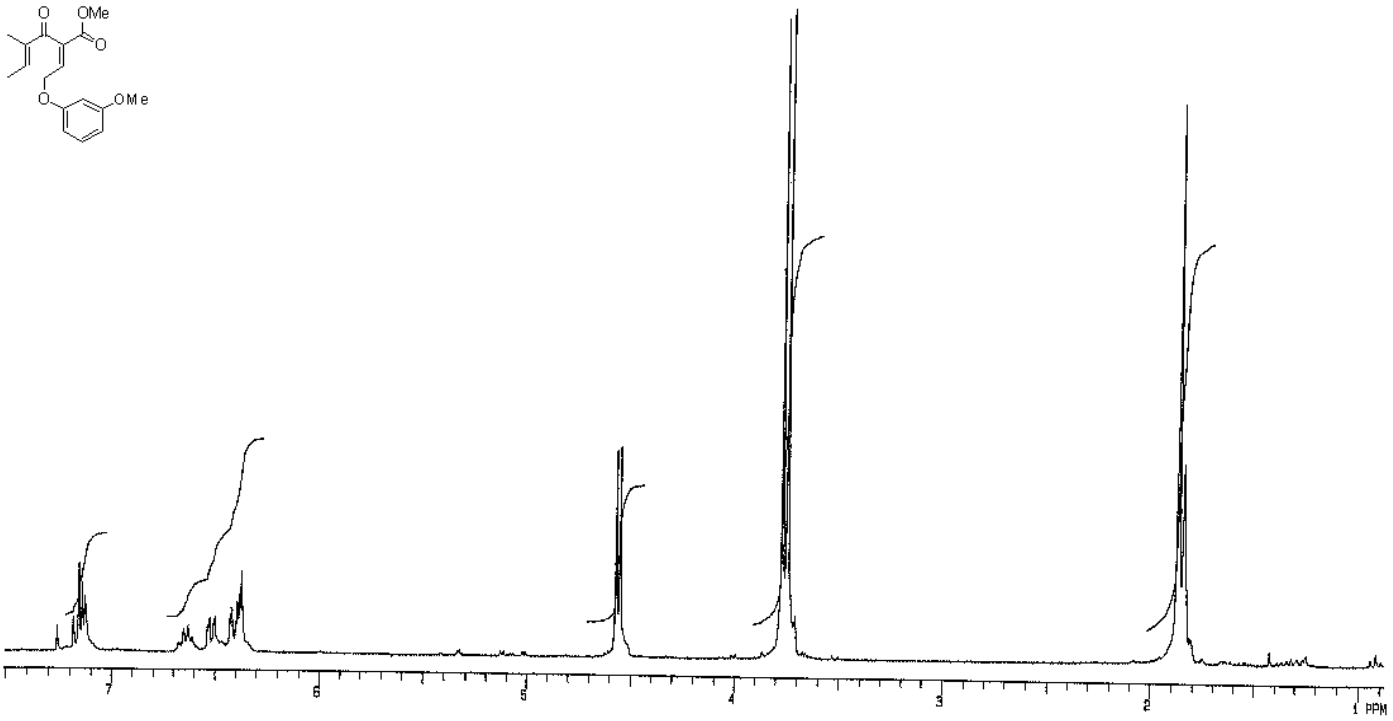


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

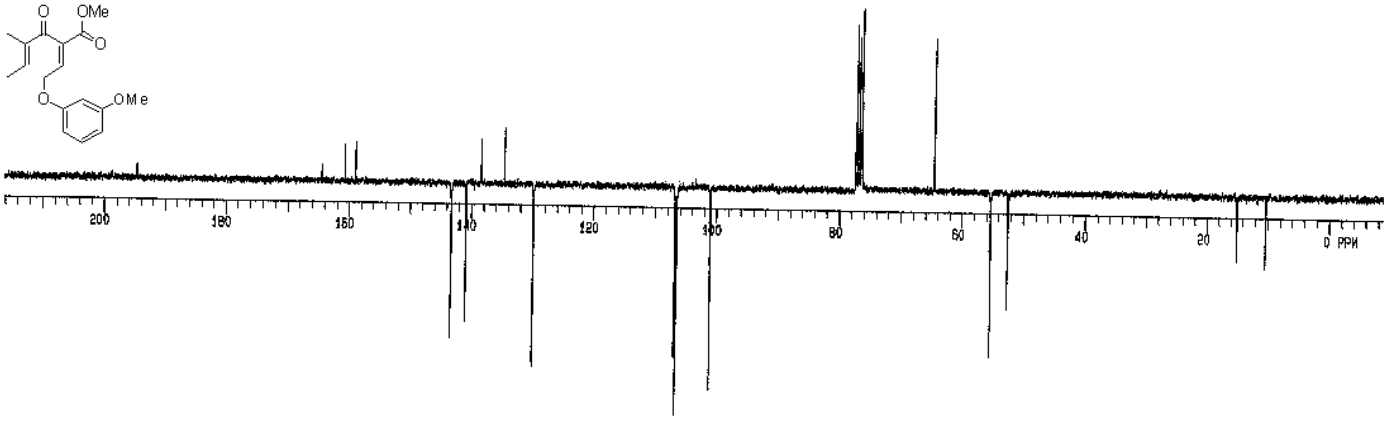


Methyl (2*E*,4*E*)-2-[2-(3-methoxyphenoxy)ethylidene]-4-methyl-3-oxohex-4-enoate (*E*-16n)

^{1}H NMR (300 MHz, $CDCl_3$)

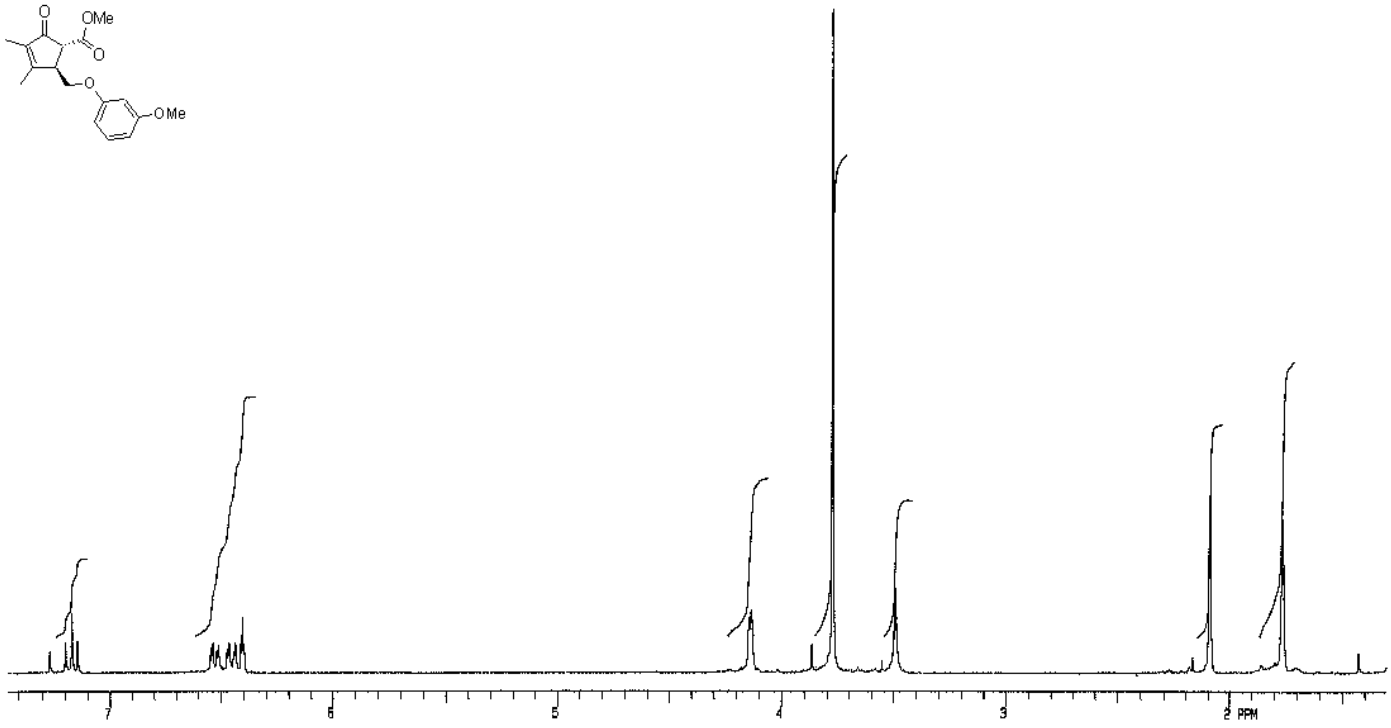


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

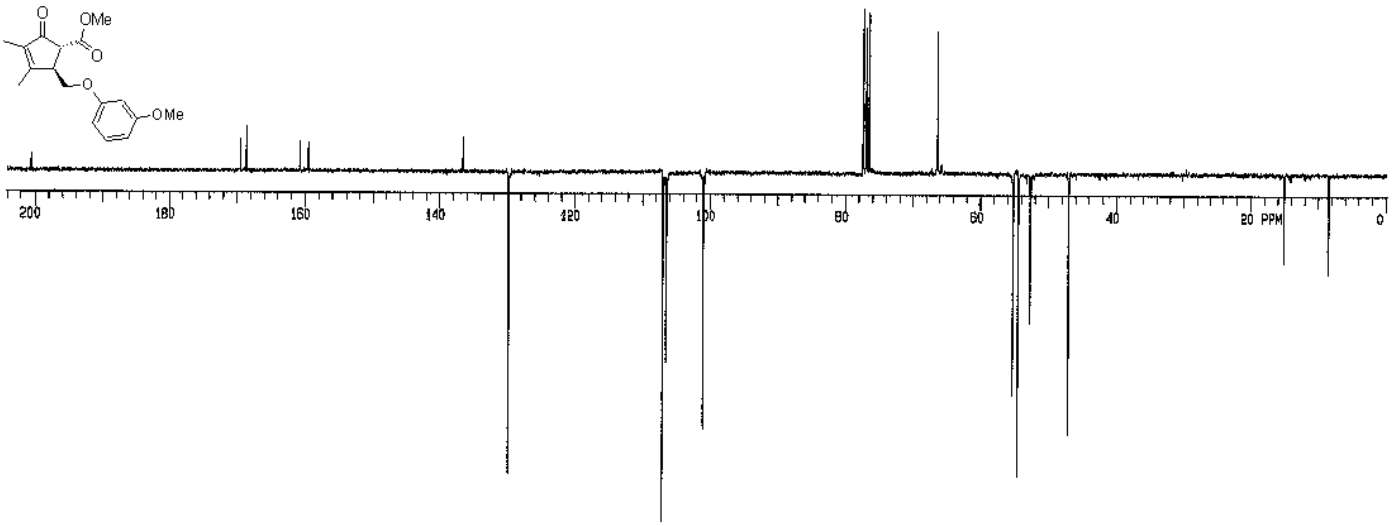


(±)-Methyl *trans*-2-[(3-methoxyphenoxy)methyl]-3,4-dimethyl-5-oxocyclopent-3-enecarboxylate (17n)

^{1}H NMR (300 MHz, $CDCl_3$)

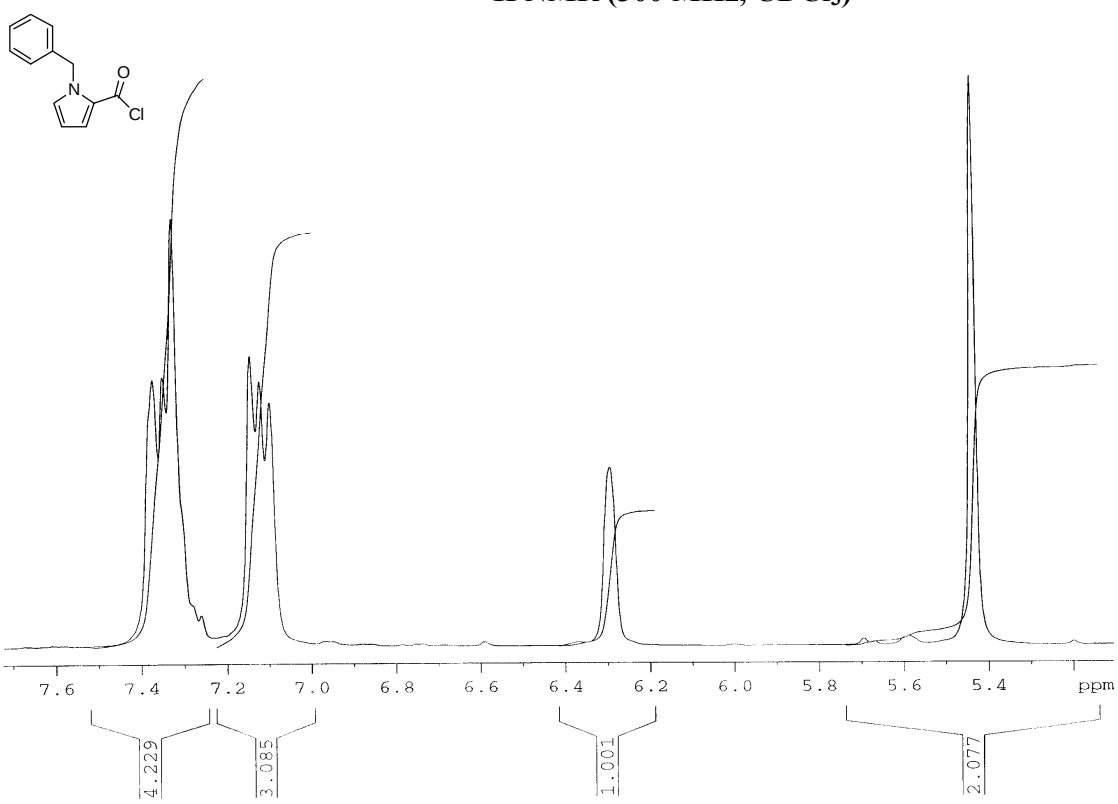


^{13}C NMR (APT, 75 MHz, $CDCl_3$)



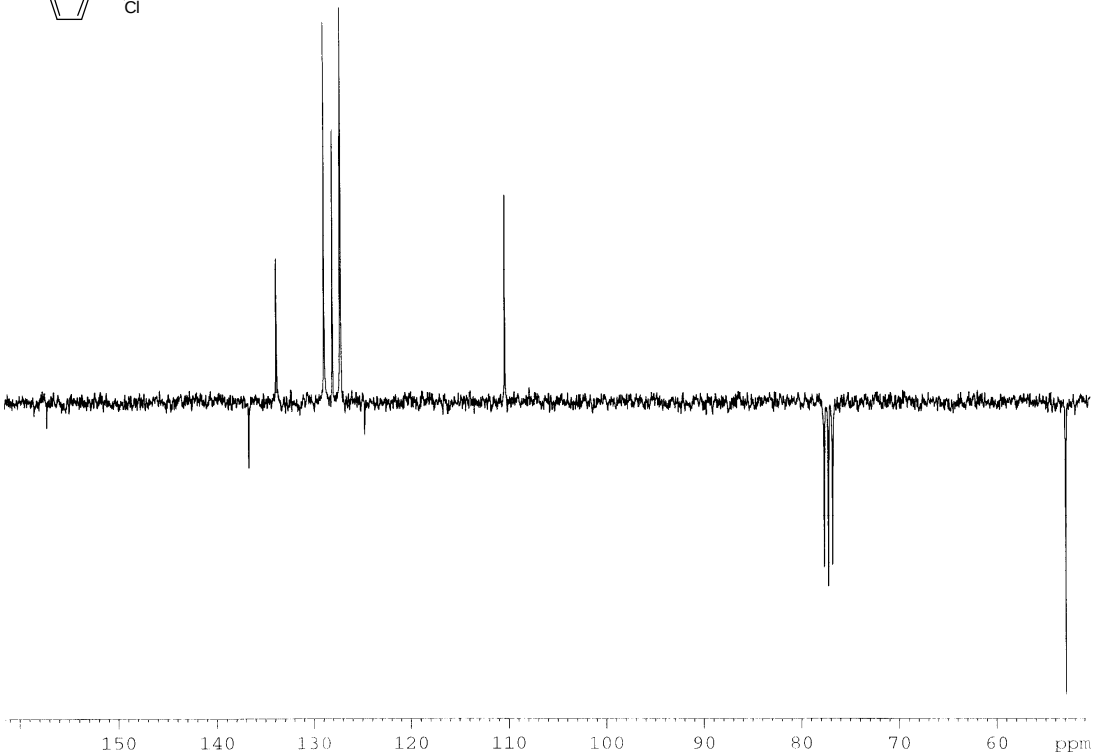
1-Benzyl-1*H*-pyrrole-2-carbonyl chloride (15i)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          djsedj
EXPNO            146
PROCNO             1

F2 - Acquisition Parameters
Date_        20100113
Time             19.31
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                40.3
DW             111.200 usec
DE               6.00 usec
TE              295.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.2300185 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW               no
SSB                2
LB              0.30 Hz
```

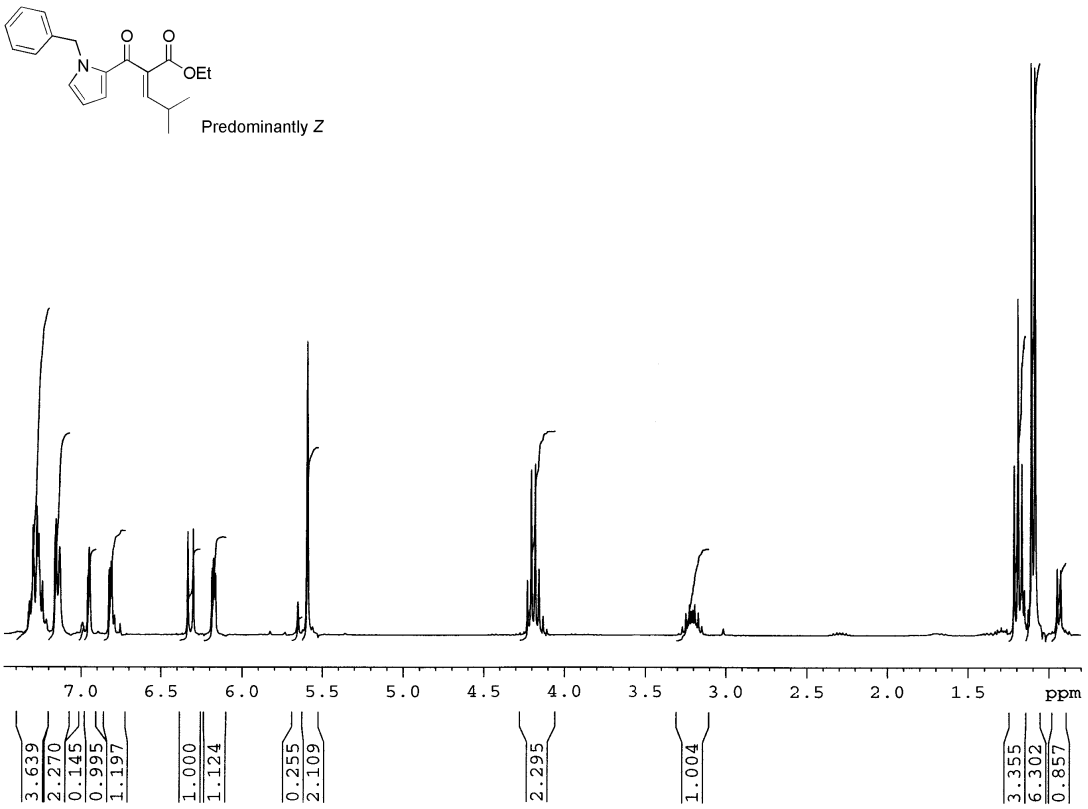
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                djsedj
EXPNO                  147
PROCNO                   1

F2 - Acquisition Parameters
Date_           20100113
Time                19.36
INSTRUM             spect
PROBHD    5 mm Multinucl
PULPROG              jmod
TD                  32768
SOLVENT             CDCl3
NS                    153
DS                      4
SWH             18115.941 Hz
FIDRES            0.552855 Hz
AQ                0.9044468 sec
RG                   1824.6
DW                   27.600 usec
DE                    6.00 usec
TE                   295.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

========= CHANNEL f1 ========
NUC1                   13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1           75.5018965 MHz

========= CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2           300.2320007 MHz

F1 - Acquisition parameters
ND0                     3
TD                    512
SFO1           300.1314 MHz
FIDRES           0.586194 Hz
SW                1.000 ppm
FnMODE          undefined

F2 - Processing parameters
SI                  32768
SF             75.4928940 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00

F1 - Processing parameters
```

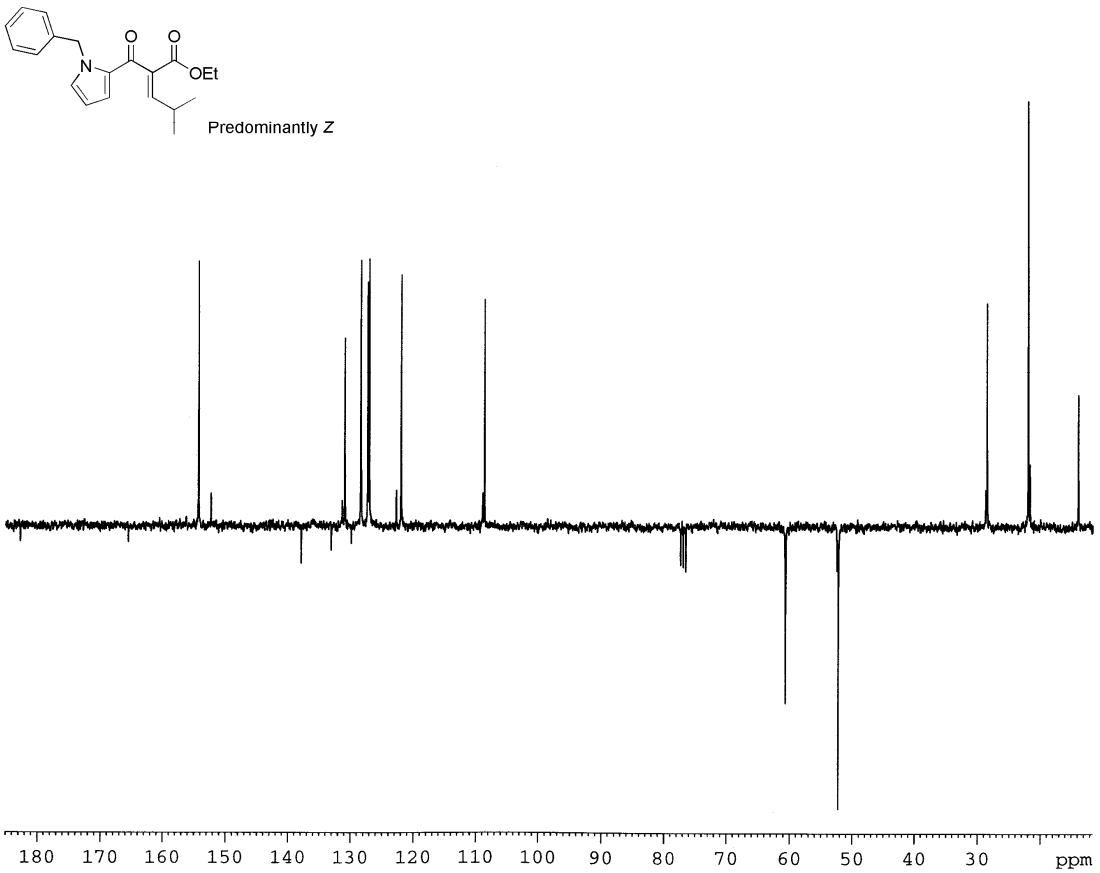
Ethyl (*Z*)-2-(1-benzyl-1*H*-pyrrole-2-carbonyl)-4-methylpent-2-enoate (*Z*-16o)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20050607
Time             17.17
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           4496.403 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                 35.9
DW              111.200 usec
DE                6.00 usec
TE               303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1      300.1314 MHz
FIDRES      7.015535 Hz
SW          11.968 ppm
FnMODE      undefined

F2 - Processing parameters
SI               16384
SF        300.1300051 MHz
WDW                EM
SSB                 0
LB           0.30 Hz
GB                  0
PC                2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF        300.1300000 MHz
WDW                no
SSB                 2
LB           0.30 Hz
```

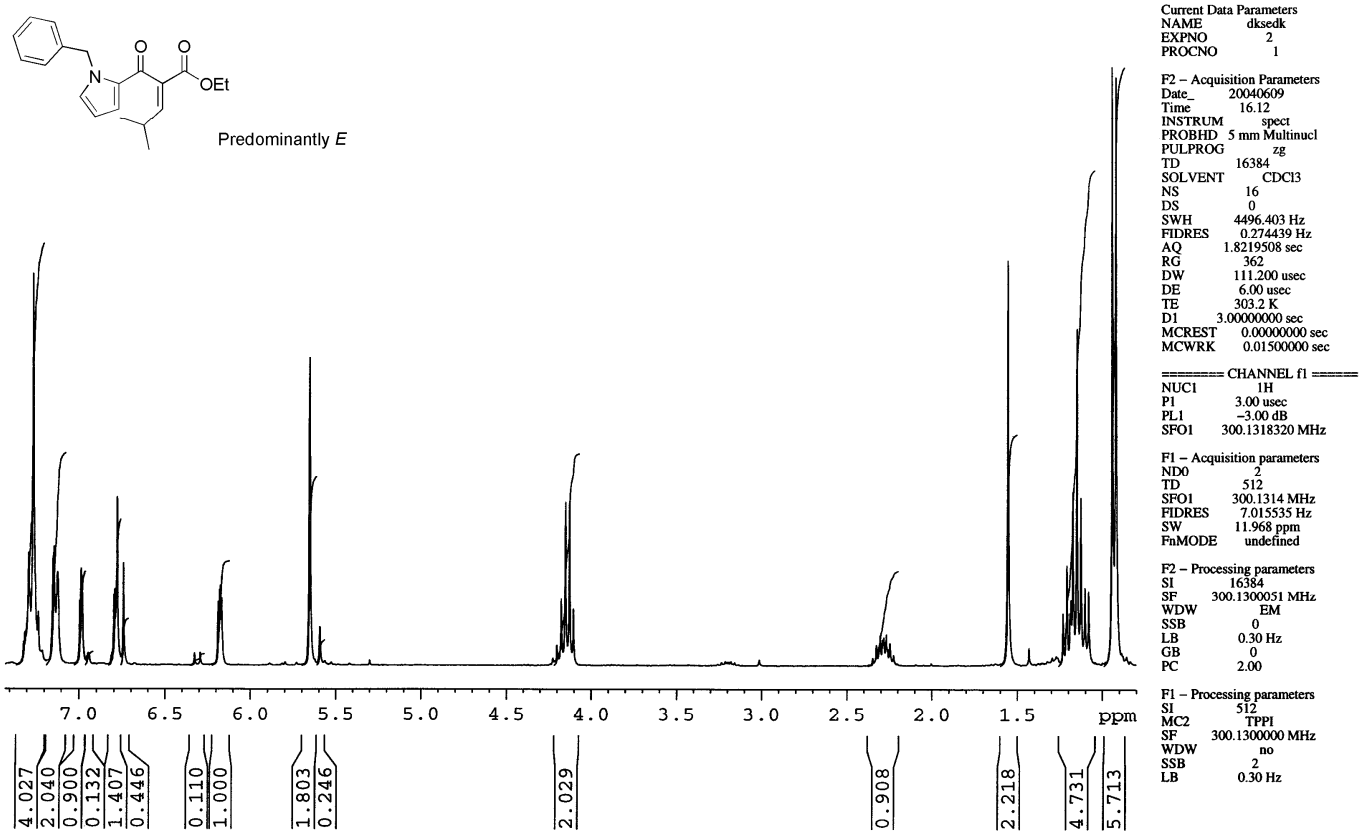
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050607
Time                  17.26
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT             CDCl3
NS                      539
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                 0.8061428 sec
RG                    1149.4
DW                    24.600 usec
DE                    20.00 usec
TE                    303.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 2.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1              75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2             300.1320000 MHz

F2 - Processing parameters
SI                    32768
SF              75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

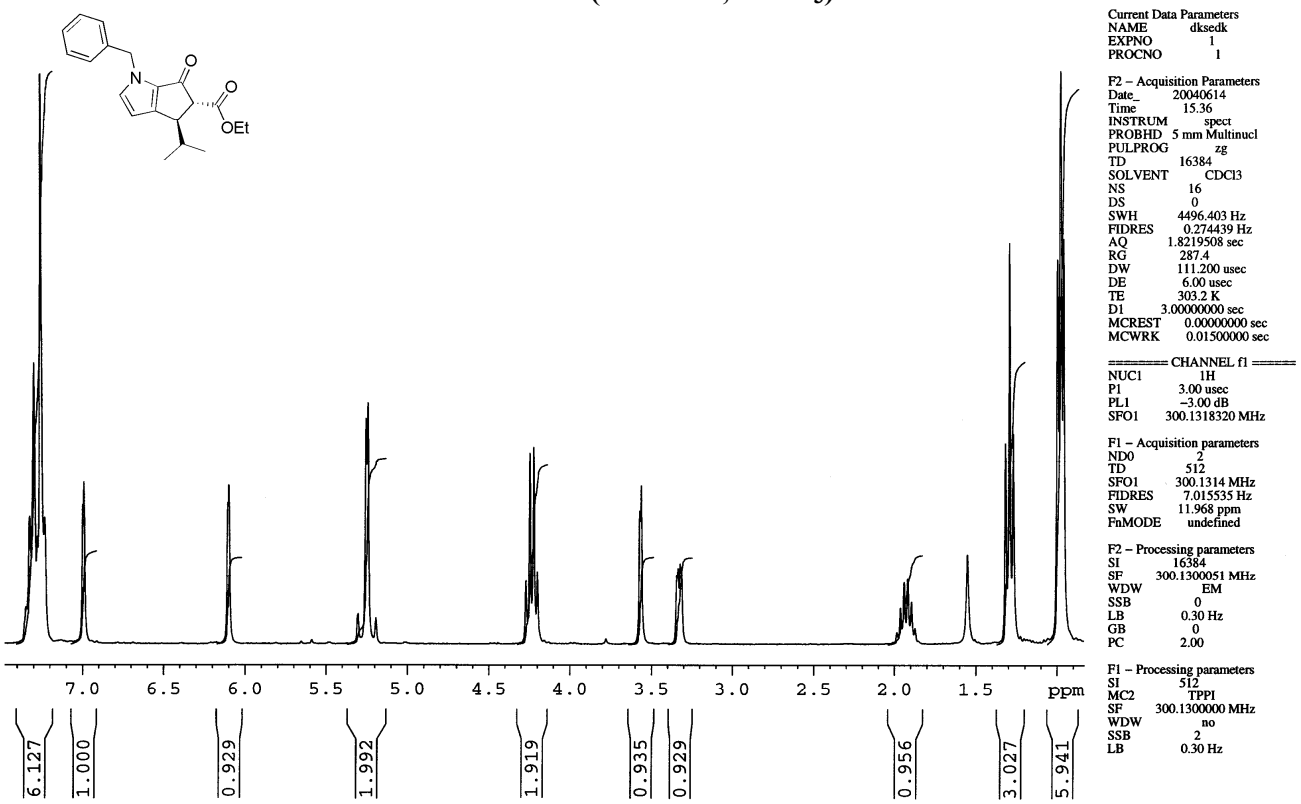
Ethyl (*E*)-2-(1-benzyl-1*H*-pyrrole-2-carbonyl)-4-methylpent-2-enoate (*E*-16o)

^{1}H NMR (300 MHz, $CDCl_3$)



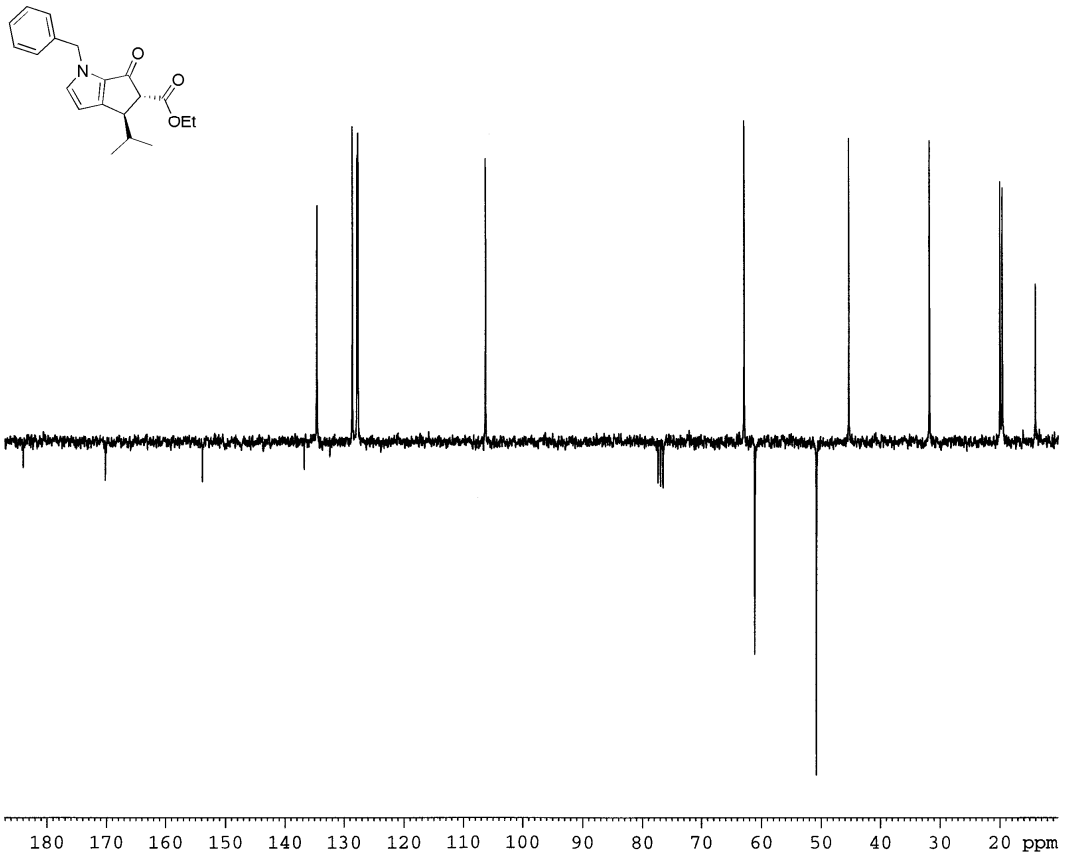
(±)-Ethyl *trans*-1-benzyl-4-isopropyl-6-oxo-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carboxylate (17o)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20040614
Time             15.36
INSTRUM           spect
PROBHD  5 mm Multinucl
PULPROG             zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           4496.403 Hz
FIDRES          0.274439 Hz
AQ            1.8219508 sec
RG                287.4
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0                   2
TD                  512
SFO1        300.1314 MHz
FIDRES        7.015535 Hz
SW             11.968 ppm
FnMODE        undefined

F2 - Processing parameters
SI                16384
SF        300.1300051 MHz
WDW                EM
SSB                   0
LB             0.30 Hz
GB                    0
PC                 2.00

F1 - Processing parameters
SI                  512
MC2                TPPI
SF        300.1300000 MHz
WDW                   no
SSB                   2
LB             0.30 Hz
```

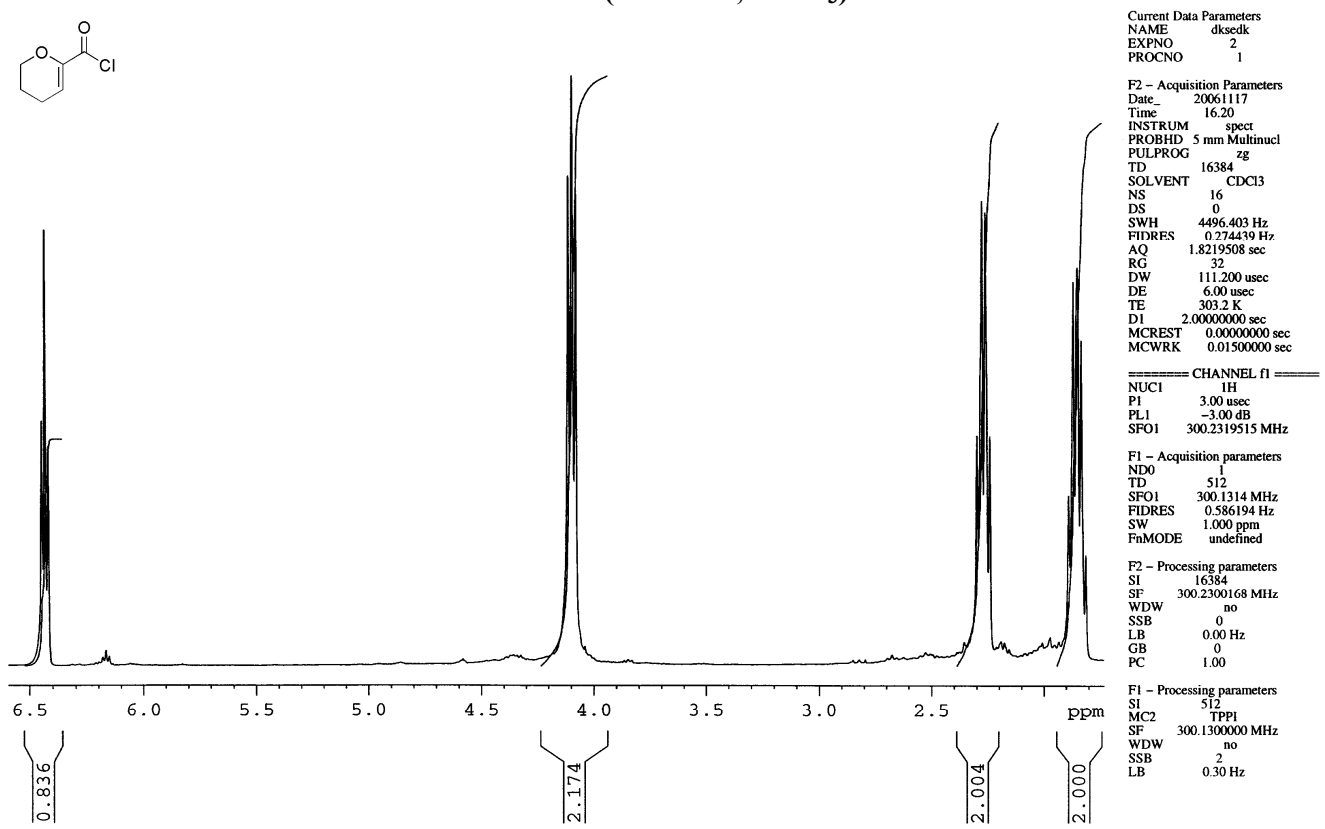
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050119
Time                  13.26
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     466
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                 24.600 usec
DE                 20.00 usec
TE                 303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              2.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               19.40 dB
SFO2          300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF             75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

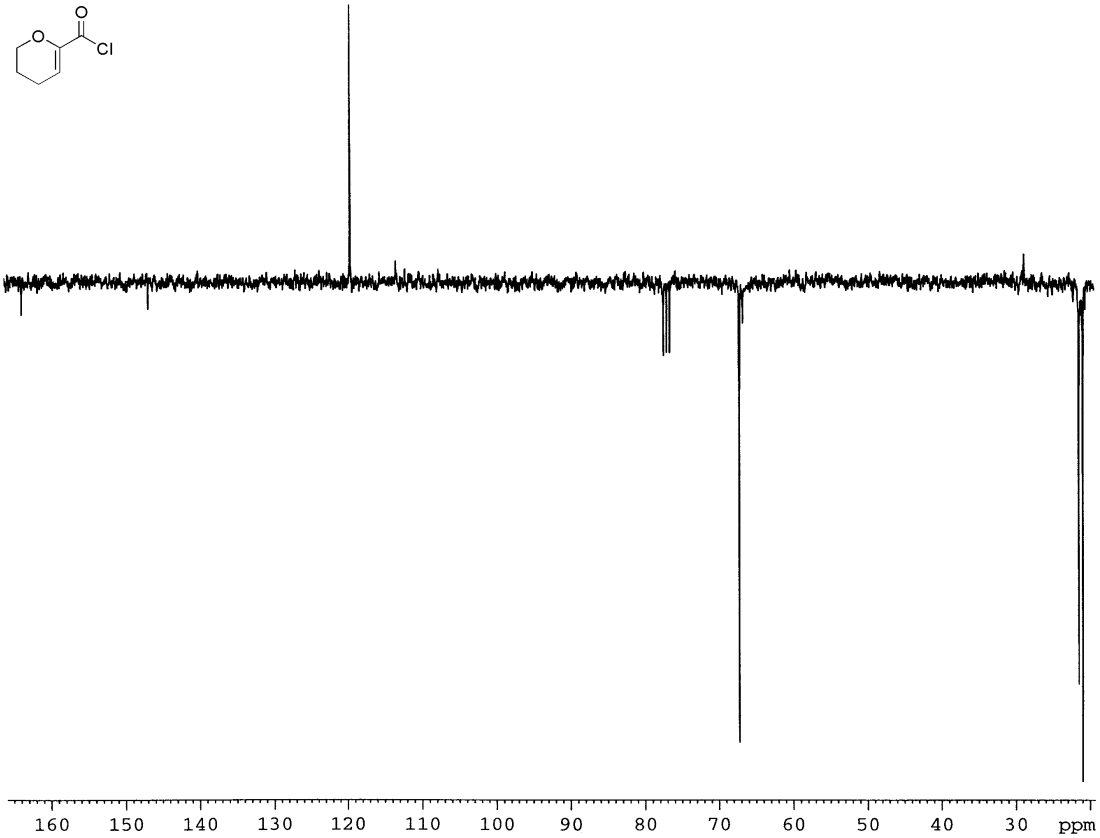
3,4-Dihydro-2*H*-pyran-6-carbonyl chloride (15j)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20061117
Time             16.20
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                 32
DW              111.200 usec
DE                6.00 usec
TE              303.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.2300168 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW               no
SSB                2
LB              0.30 Hz
```

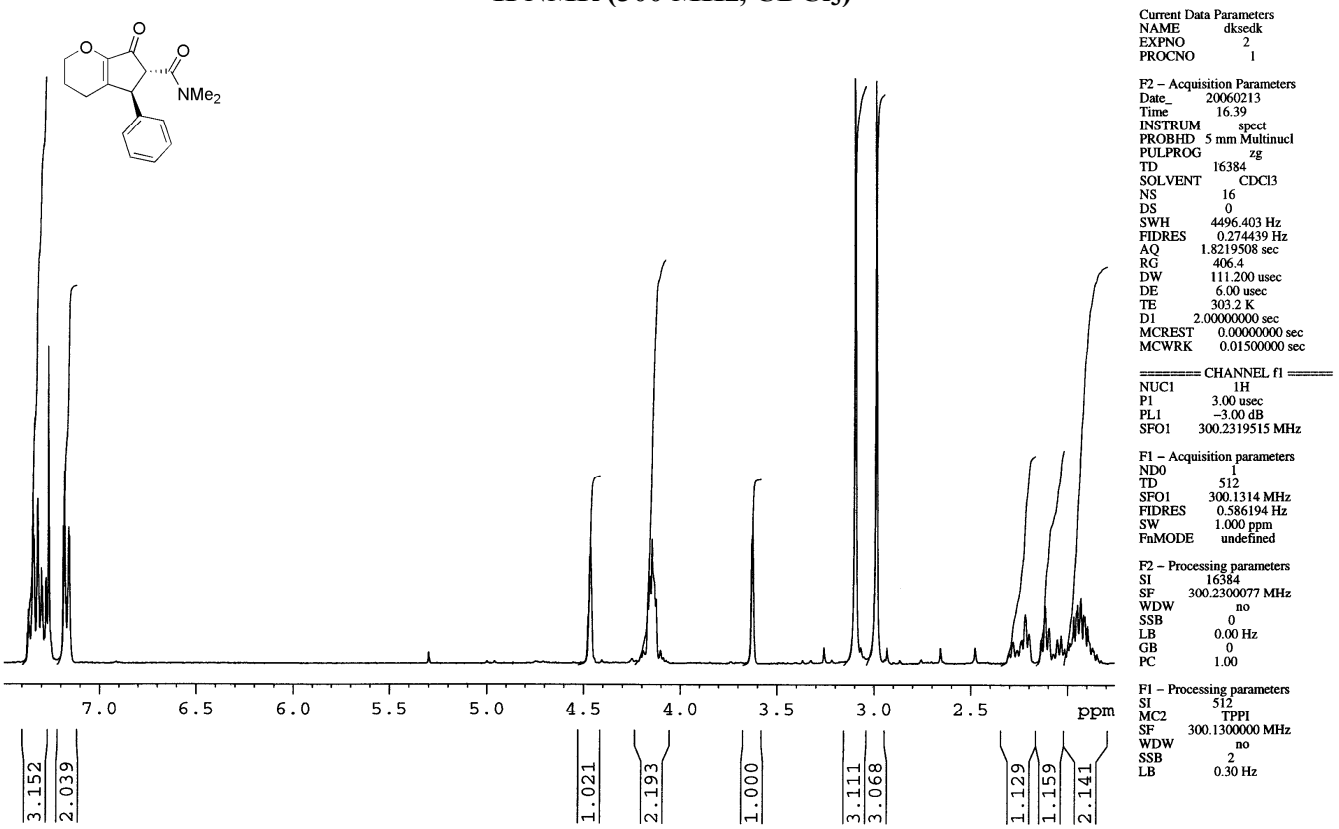
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20061117
Time                  16.27
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                      262
DS                        4
SWH               18115.941 Hz
FIDRES             0.552855 Hz
AQ                 0.9044468 sec
RG                    1625.5
DW                    27.600 usec
DE                     6.00 usec
TE                    303.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1              75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2             300.2320007 MHz

F1 - Acquisition parameters
ND0                       3
TD                      512
SFO1              300.1314 MHz
FIDRES              0.586194 Hz
SW                  1.000 ppm
FnMODE              undefined

F2 - Processing parameters
SI                    32768
SF                75.4929074 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```

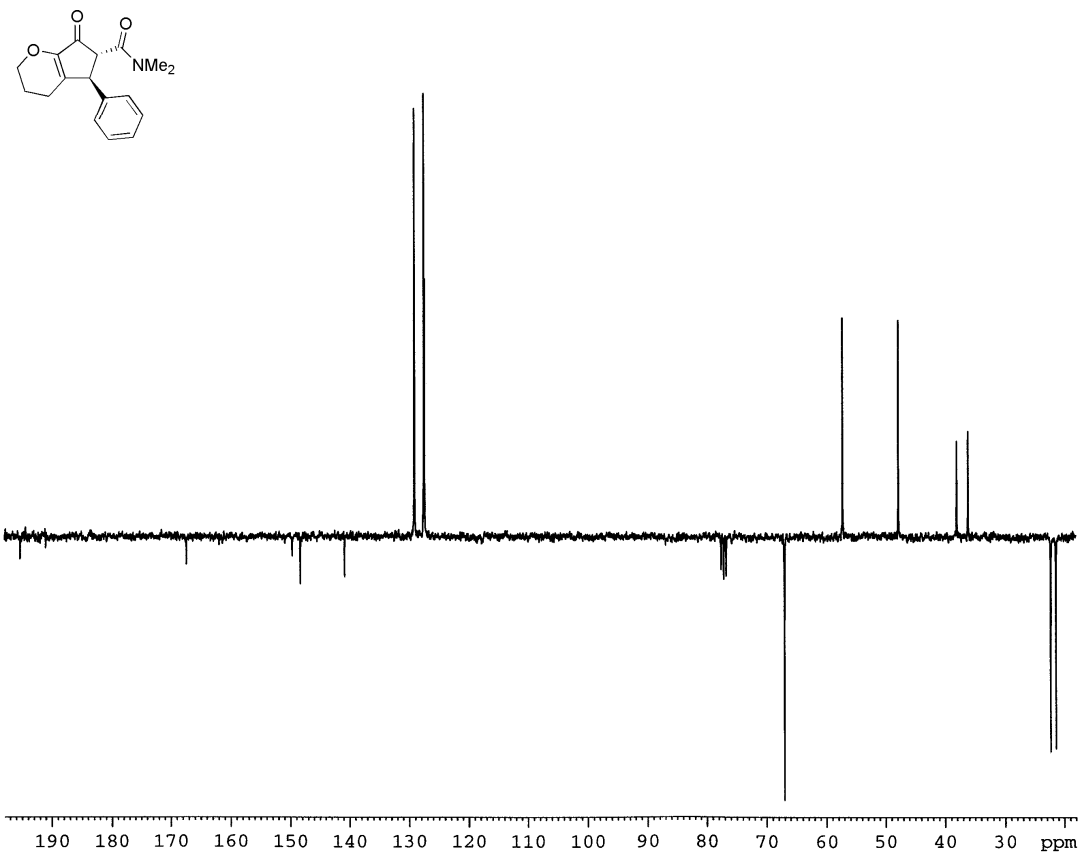
(±)-*trans*-*N*,*N*-Dimethyl-7-oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carboxamide (17p)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20060213
Time            16.39
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG               406.4
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.2300077 MHz
WDW                no
SSB                 0
LB           0.00 Hz
GB                  0
PC               1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW                no
SSB                 2
LB           0.30 Hz
```

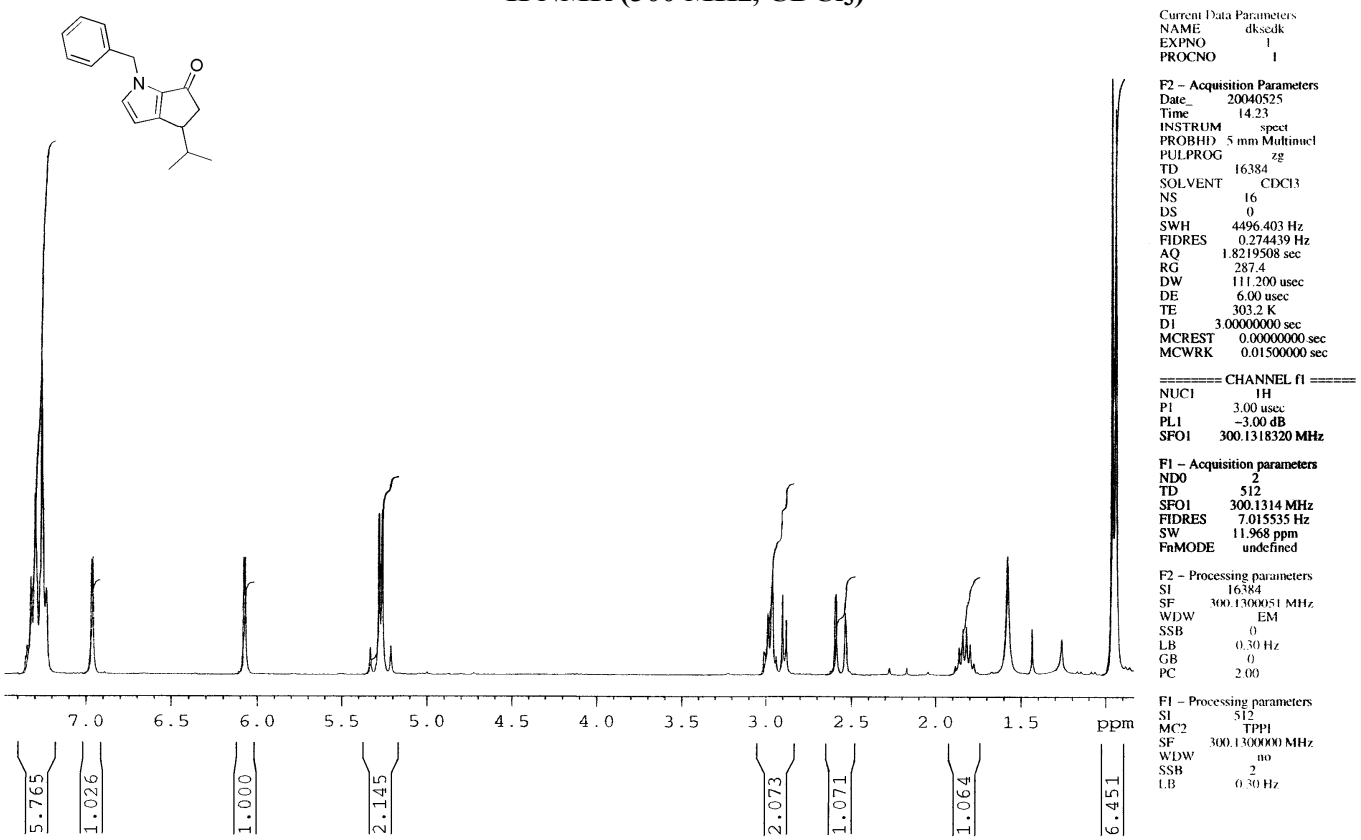
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



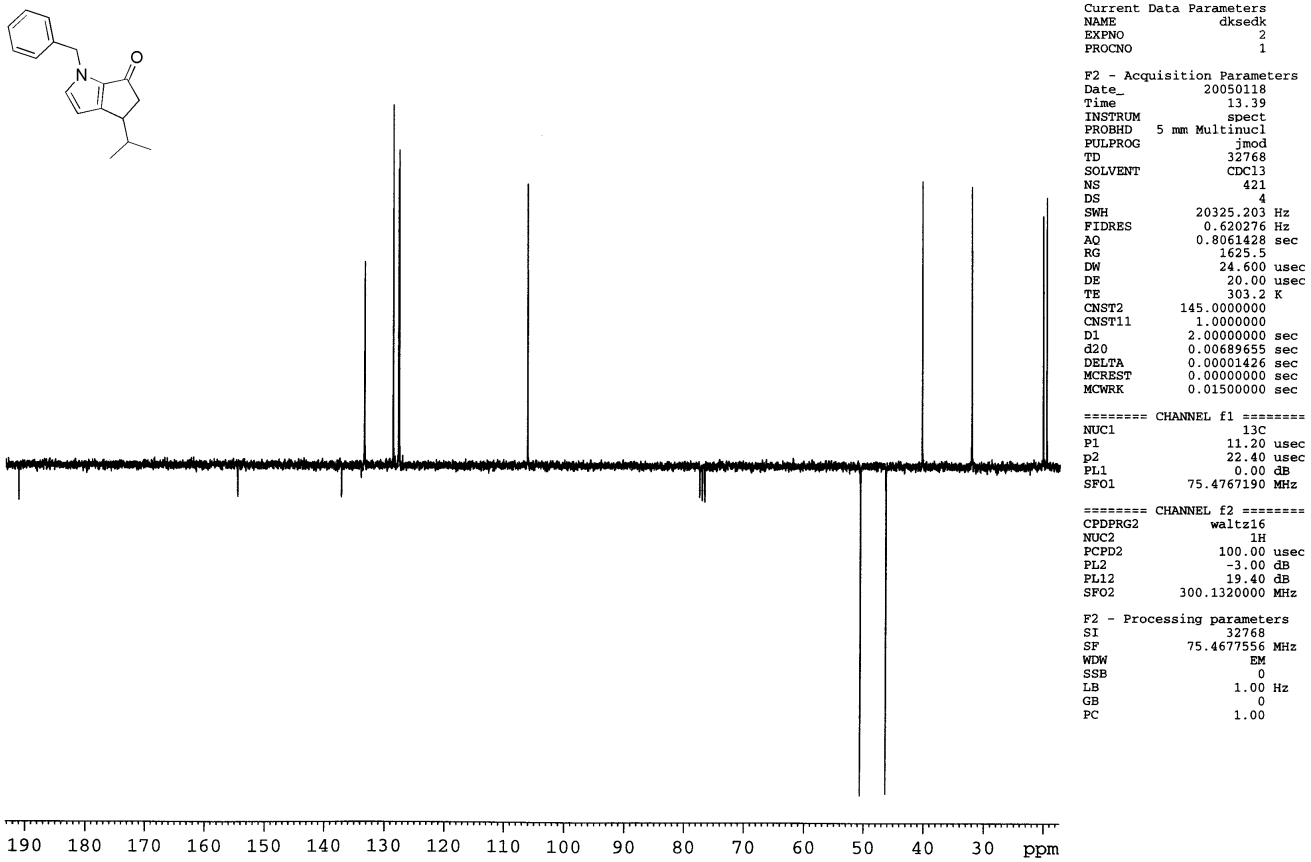

```
Current Data Parameters
NAME          dksedk
EXPNO              7
PROCNO             1

F2 - Acquisition Parameters
Date_        20070523
Time            17.59
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG          jmod
TD              32768
SOLVENT         CDCl3
NS                395
DS                  4
SWH         18115.941 Hz
FIDRES       0.552855 Hz
AQ           0.9044468 sec
RG              1149.4
DW             27.600 usec
DE               6.00 usec
TE              303.2 K
CNST2       145.0000000
CNST11        1.0000000
D1          2.00000000 sec
d20         0.00689655 sec
DELTA       0.00001426 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1              13C
P1             11.20 usec
p2             22.40 usec
PL1             0.00 dB
SFO1       75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2         100.00 usec
PL2             -3.00 dB
PL12            19.40 dB
SFO2      300.2320007 MHz

F1 - Acquisition parameters
ND0                3
TD               512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              32768
SF       75.4928940 MHz
WDW                EM
SSB                 0
LB           3.00 Hz
GB                  0
PC               1.00

F1 - Processing parameters
```

(±)-1-Benzyl-4-isopropyl-4,5-dihydrocyclopenta[*b*]pyrrol-6(1*H*)-one (21)

^{1}H NMR (300 MHz, $CDCl_3$)

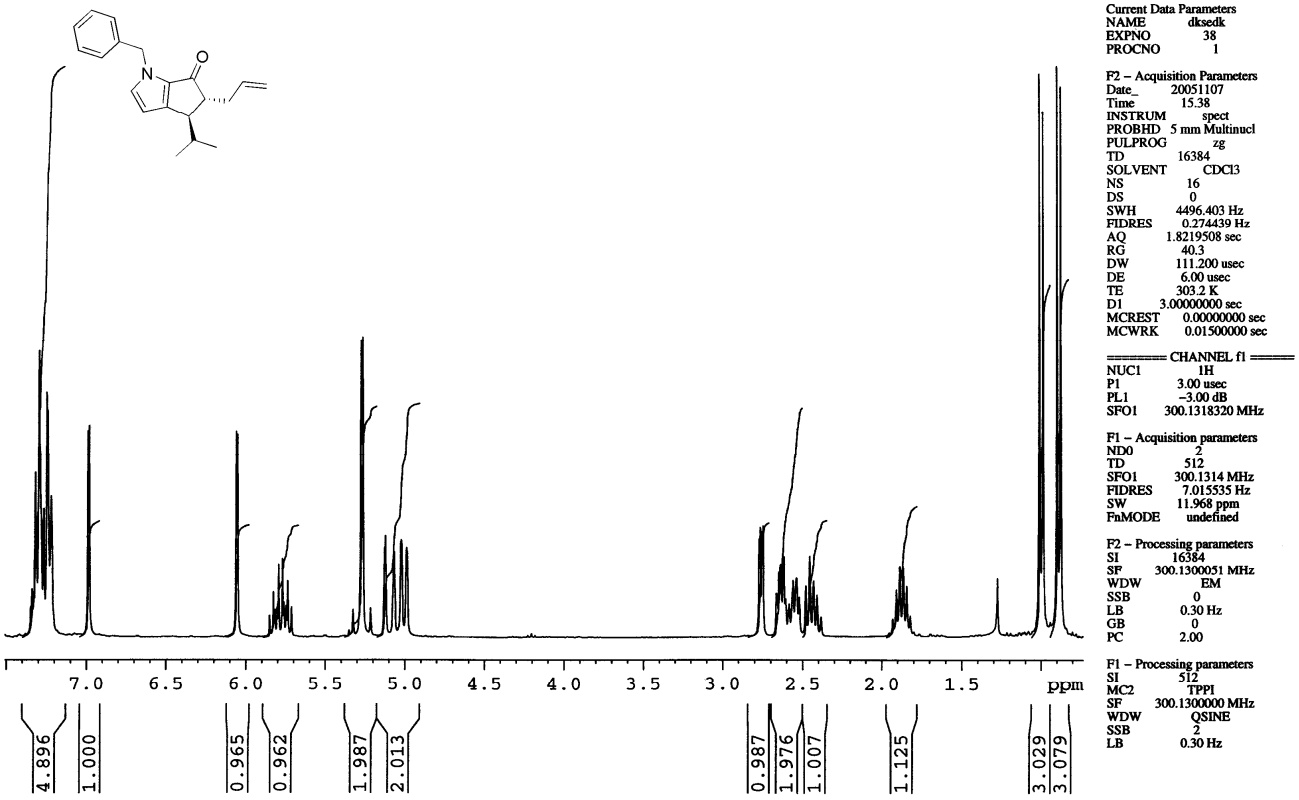


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

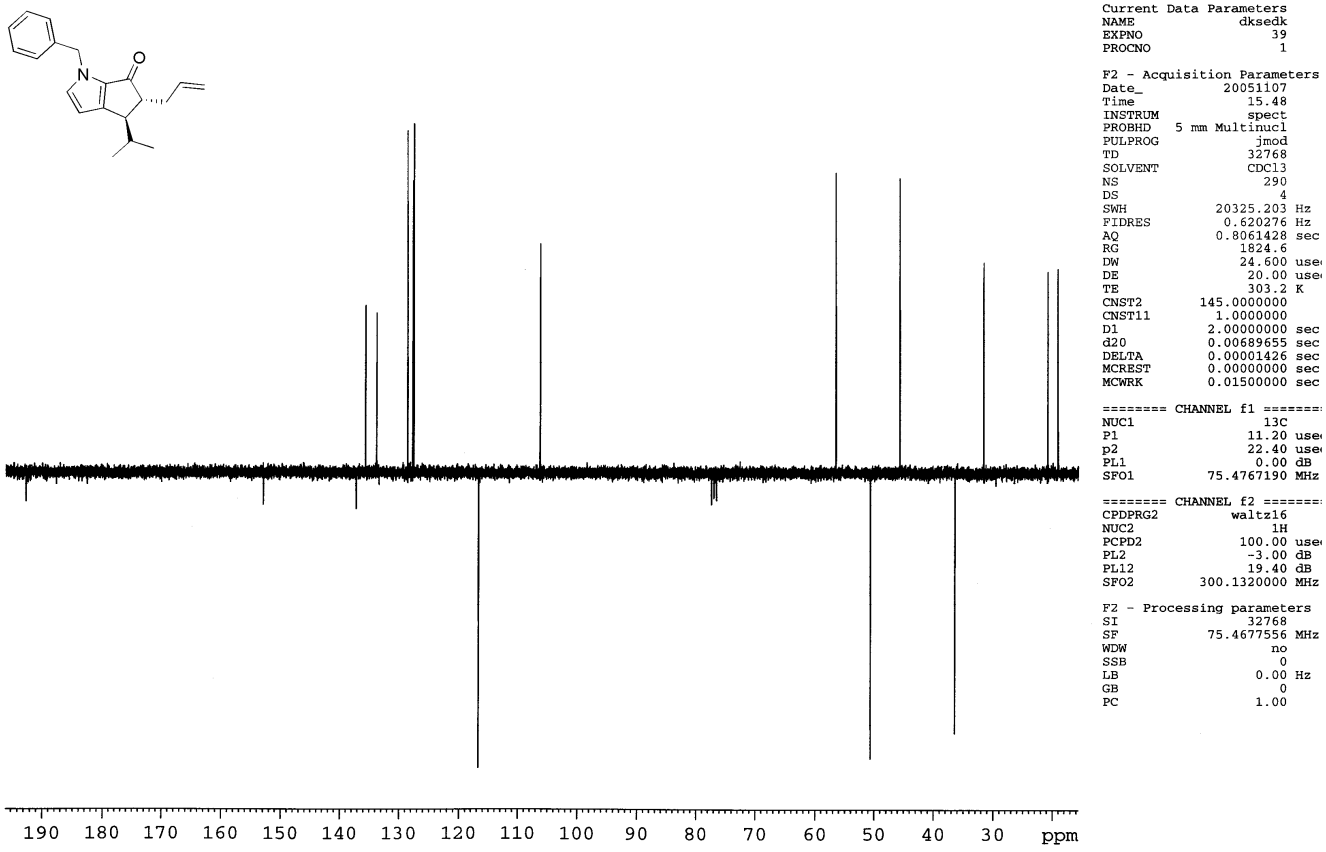


(±)-*trans*-5-Allyl-1-benzyl-4-isopropyl-4,5-dihydrocyclopenta[*b*]pyrrol-6(1*H*)-one (24)

^{1}H NMR (300 MHz, $CDCl_3$)

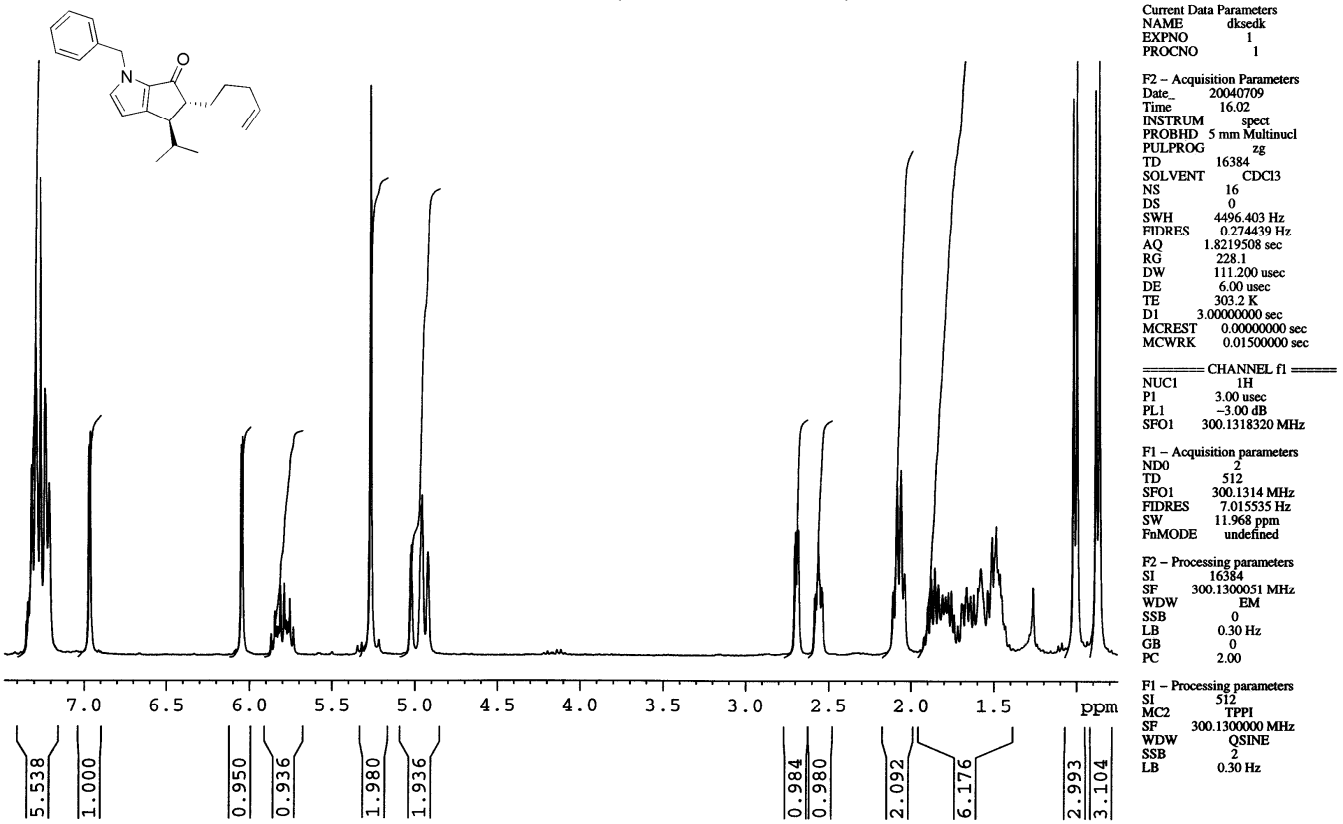


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

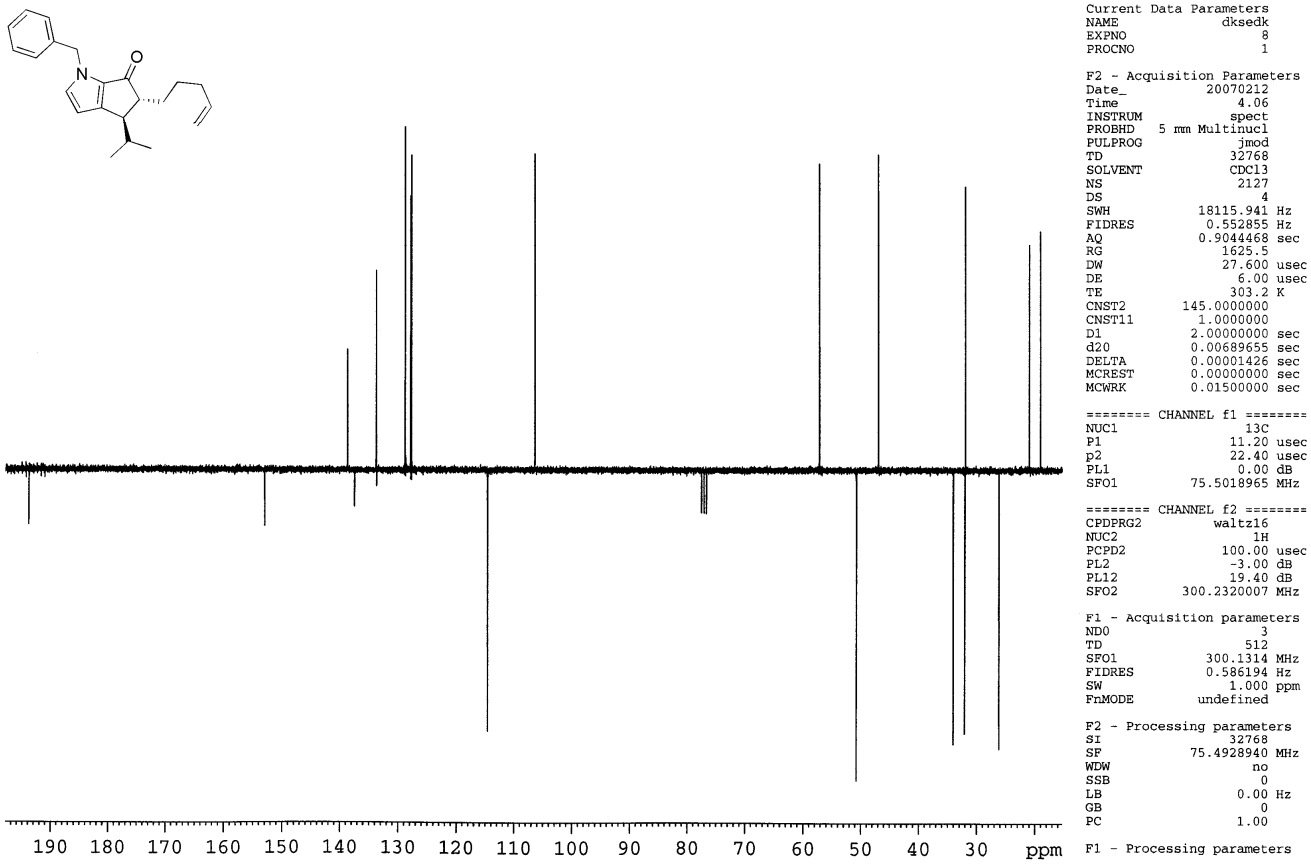


(±)-*trans*-1-Benzyl-4-isopropyl-5-(pent-4-enyl)-4,5-dihydrocyclopenta[*b*]pyrrol-6(1*H*)-one (25)

^{1}H NMR (300 MHz, $CDCl_3$)

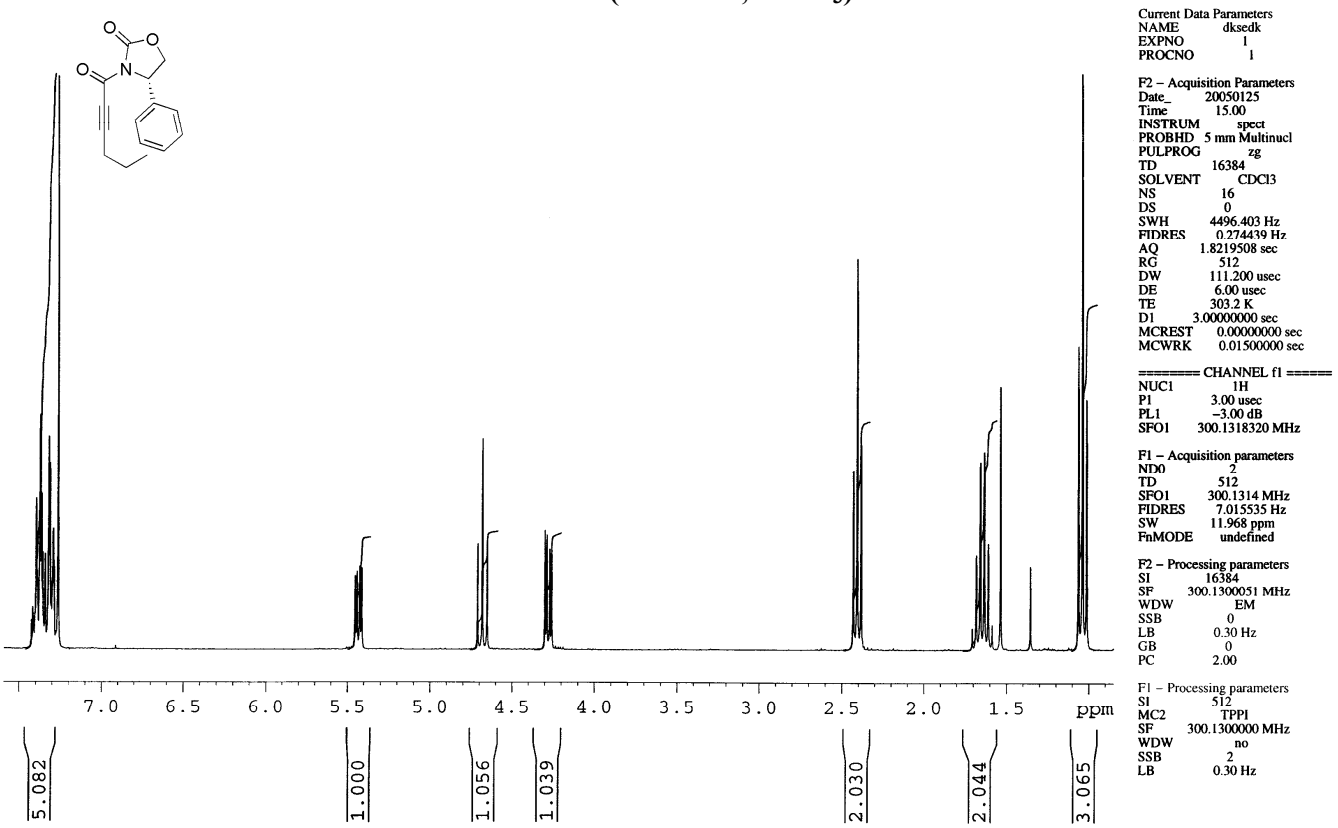


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



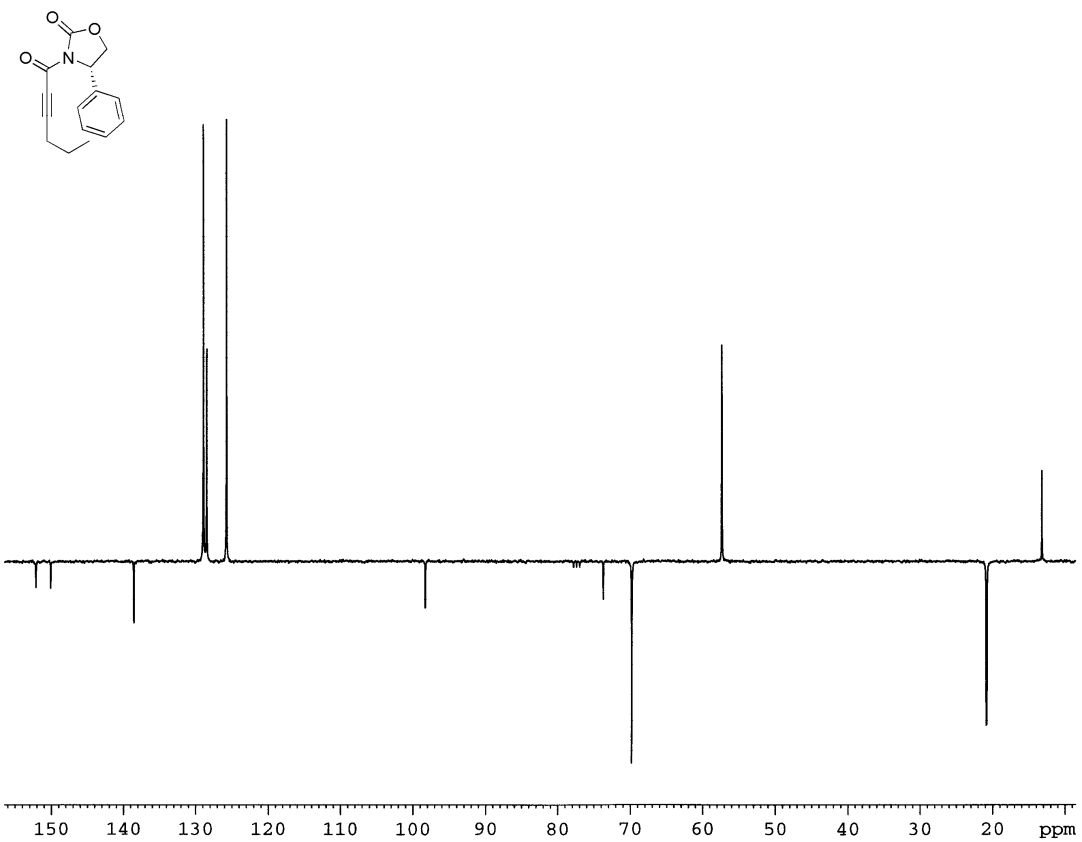
(*S*)-3-Hex-2-ynoyl-4-phenyloxazolidin-2-one (29a)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20050125
Time            15.00
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                512
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW              no
SSB               2
LB              0.30 Hz
```

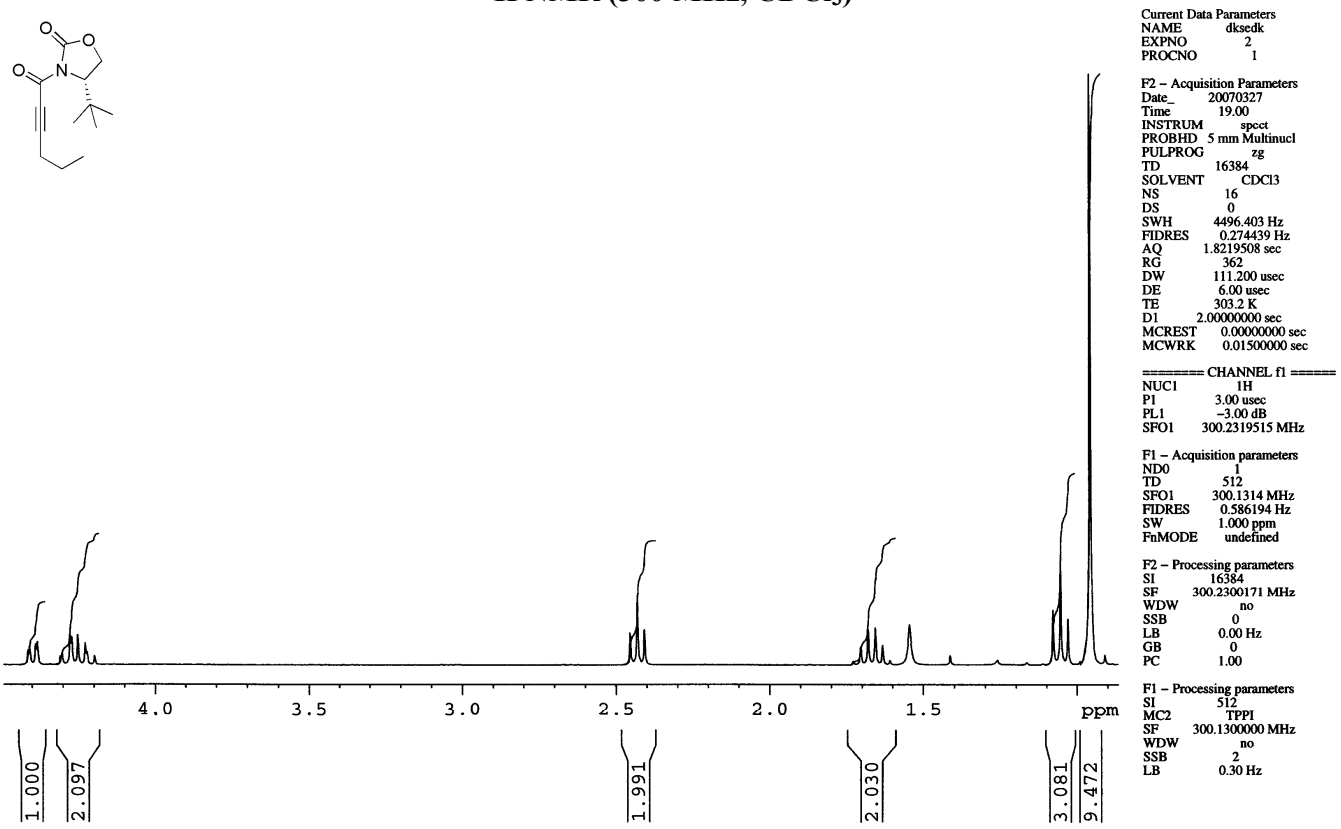
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050426
Time                  15.10
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     107
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1149.4
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              2.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               19.40 dB
SFO2          300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1           300.1314 MHz
FIDRES           7.015535 Hz
SW                 11.968 ppm
FnMODE           undefined

F2 - Processing parameters
SI                     32768
SF            75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

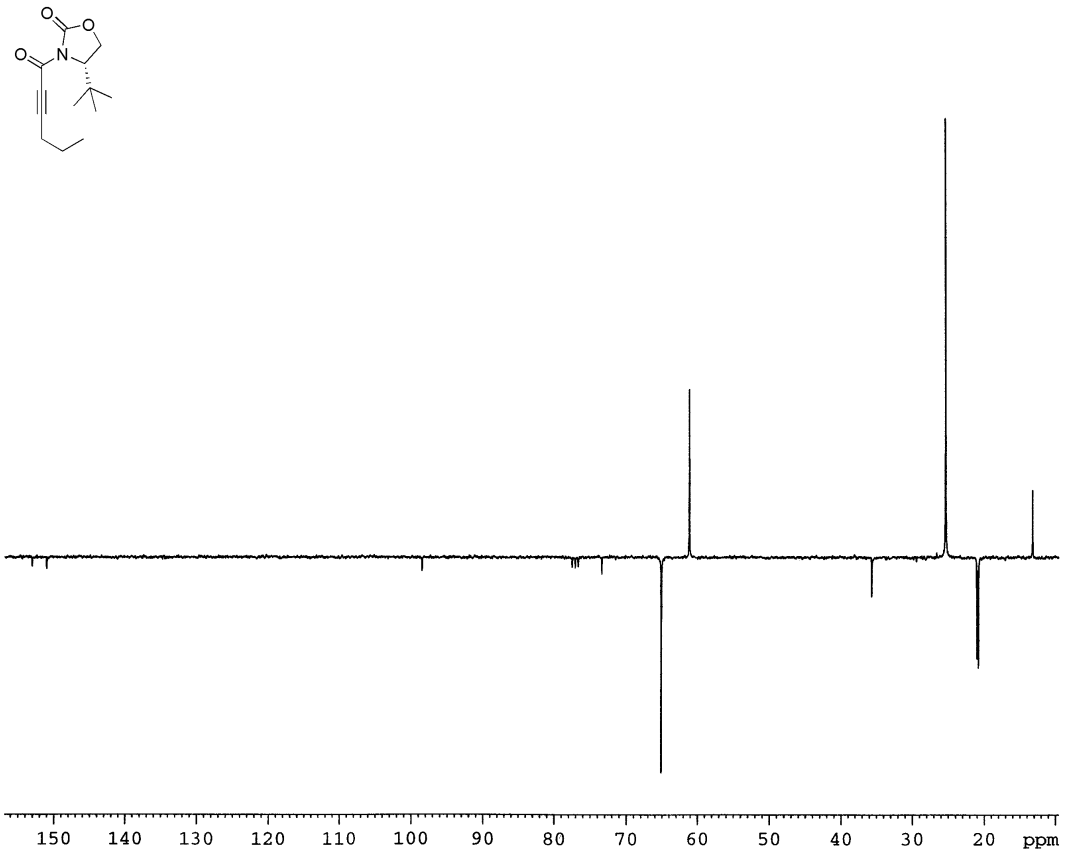
(*S*)-4-*tert*-Butyl-3-hex-2-ynoyloxazolidin-2-one (29b)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20070327
Time            19.00
INSTRUM          spect
PROBHD   5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                362
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1      300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1      300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF        300.2300171 MHz
WDW                no
SSB                 0
LB           0.00 Hz
GB                  0
PC           1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF        300.1300000 MHz
WDW                no
SSB                 2
LB           0.30 Hz
```

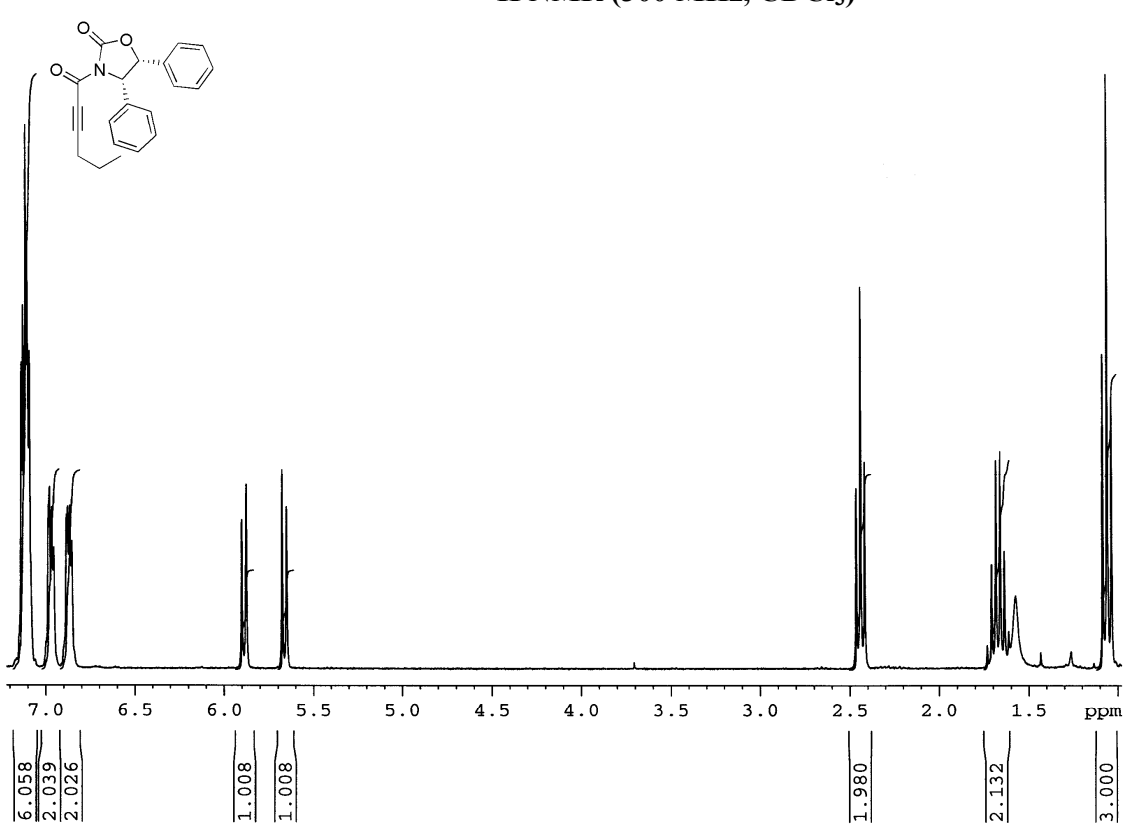
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_        20050404
Time            15.29
INSTRUM          spect
PROBHD   5 mm Multinucl
PULPROG            jmod
TD              32768
SOLVENT         CDCl3
NS                285
DS                  4
SWH          20325.203 Hz
FIDRES        0.620276 Hz
AQ            0.8061428 sec
RG               1625.5
DW              24.600 usec
DE              20.00 usec
TE              303.2 K
CNST2       145.0000000
CNST11        1.0000000
D1          2.00000000 sec
d20         0.00689655 sec
DELTA       0.00001426 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1              13C
P1              11.20 usec
p2              22.40 usec
PL1              0.00 dB
SFO1       75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          100.00 usec
PL2             -3.00 dB
PL12            19.40 dB
SFO2       300.1320000 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1      300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              32768
SF         75.4677556 MHz
WDW                EM
SSB                 0
LB           3.00 Hz
GB                  0
PC           1.00

F1 - Processing parameters
```

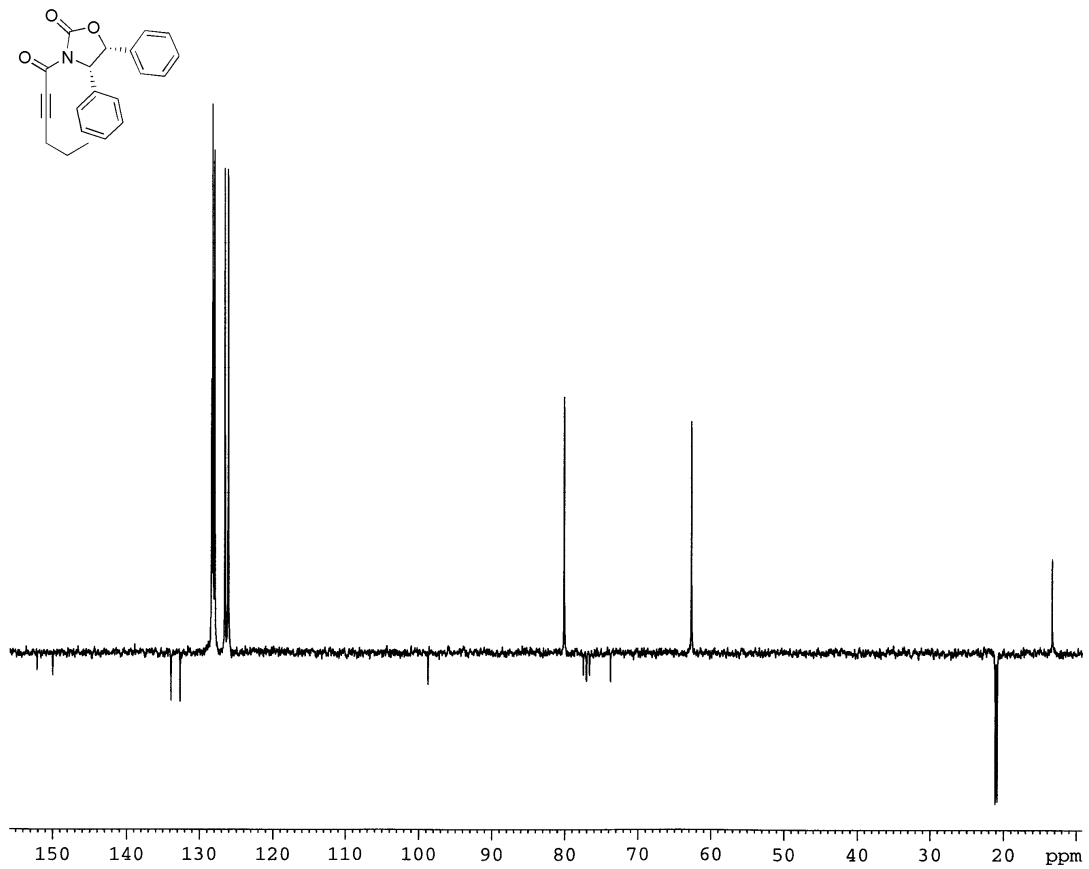
(4*S*,5*R*)-3-Hex-2-ynoyl-4,5-diphenyloxazolidin-2-one (29c)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20070327
Time            18.51
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG               406.4
DW             111.200 usec
DE               6.00 usec
TE             303.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1     300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1     300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.2300171 MHz
WDW                no
SSB                 0
LB           0.00 Hz
GB                  0
PC           1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW                no
SSB                 2
LB           0.30 Hz
```

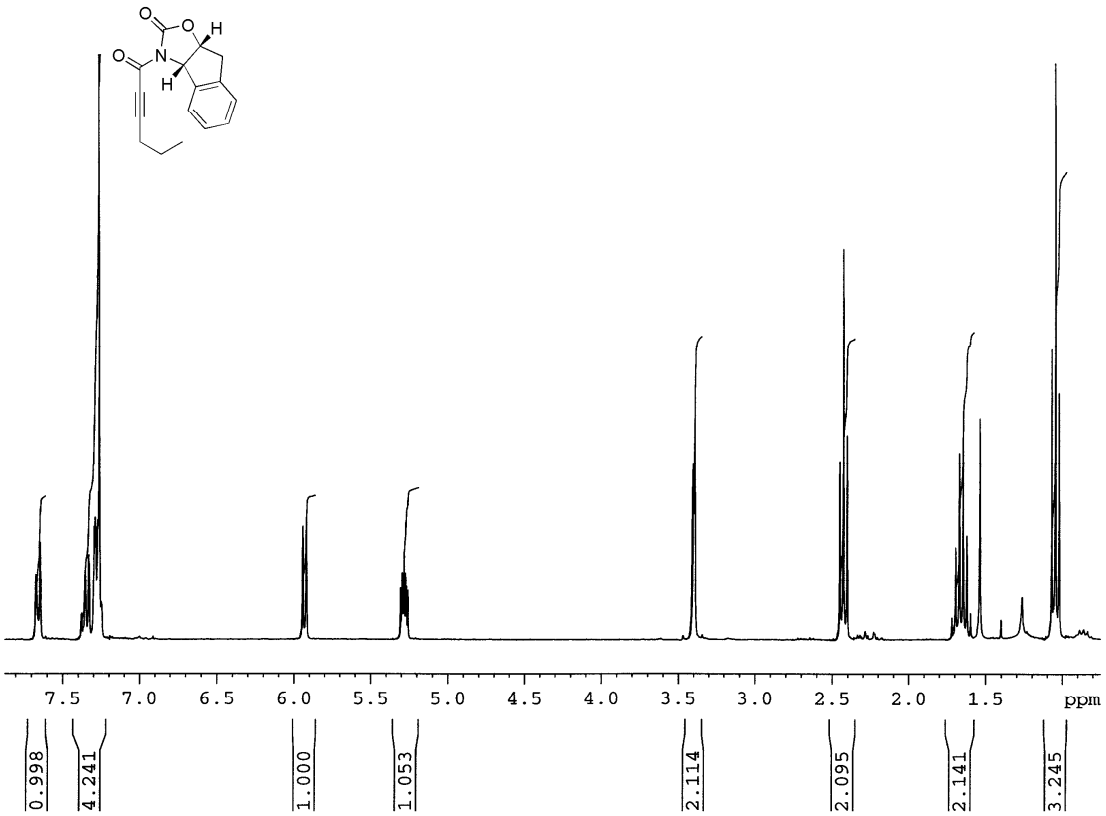
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050405
Time                 14.53
INSTRUM              spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     192
DS                       4
SWH             20325.203 Hz
FIDRES           0.620276 Hz
AQ               0.8061428 sec
RG                  1824.6
DW                 24.600 usec
DE                 20.00 usec
TE                 303.2 K
CNST2          145.0000000
CNST11           1.0000000
D1               2.00000000 sec
d20              0.00689655 sec
DELTA            0.00001426 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               19.40 dB
SFO2          300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF            75.4677556 MHz
WDW                     EM
SSB                      0
LB                  3.00 Hz
GB                       0
PC                    1.00
```

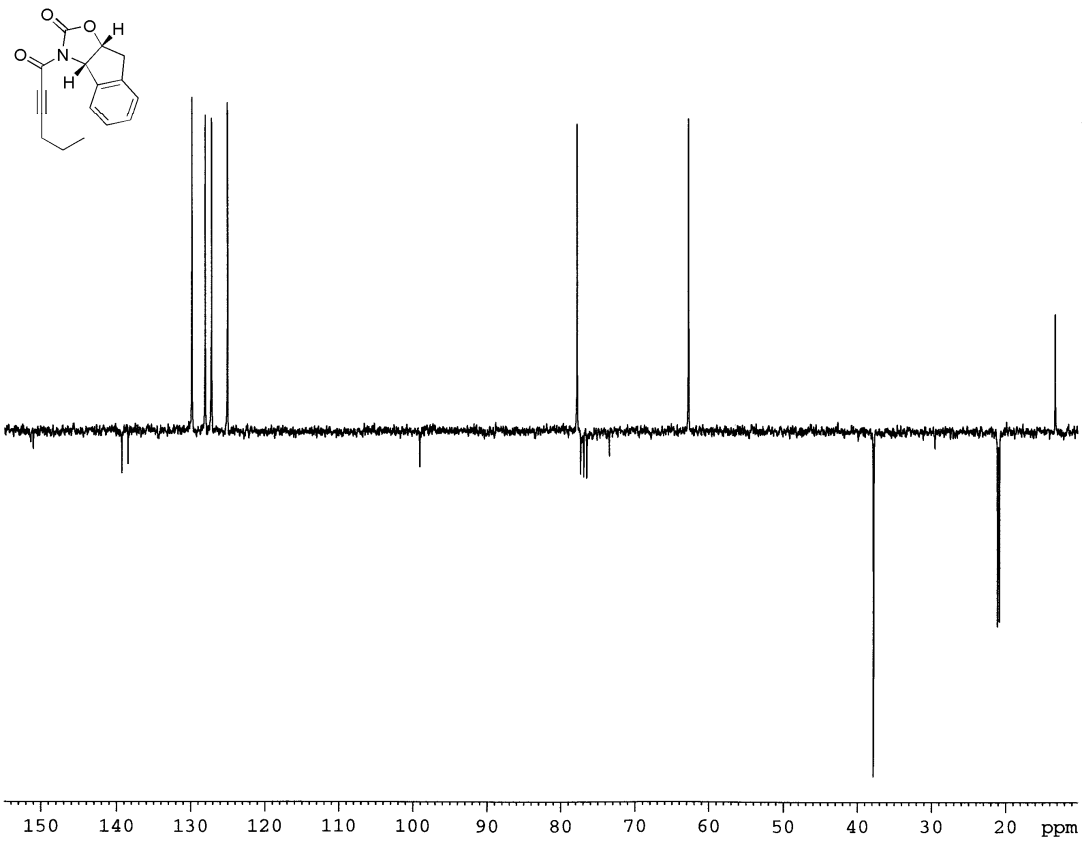
(3a*S*,8a*R*)-3-Hex-2-ynoyl-3,3a,8,8a-tetrahydro-2*H*-indeno[1,2-*d*]oxazol-2-one (29d)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20050323
Time            11.59
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG               406.4
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1               3.00 usec
PL1              -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW            QSINE
SSB                2
LB              0.30 Hz
```

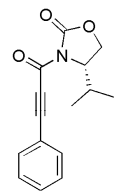
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     5
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050404
Time                  15.50
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                      467
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                 0.8061428 sec
RG                    1625.5
DW                   24.600 usec
DE                   20.00 usec
TE                    303.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 2.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1              75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2             300.1320000 MHz

F2 - Processing parameters
SI                    32768
SF               75.4677556 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00
```

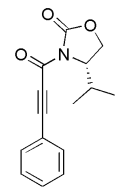
(*S*)-4-Isopropyl-3-(3-phenylpropioloyl)oxazolidin-2-one (29f)

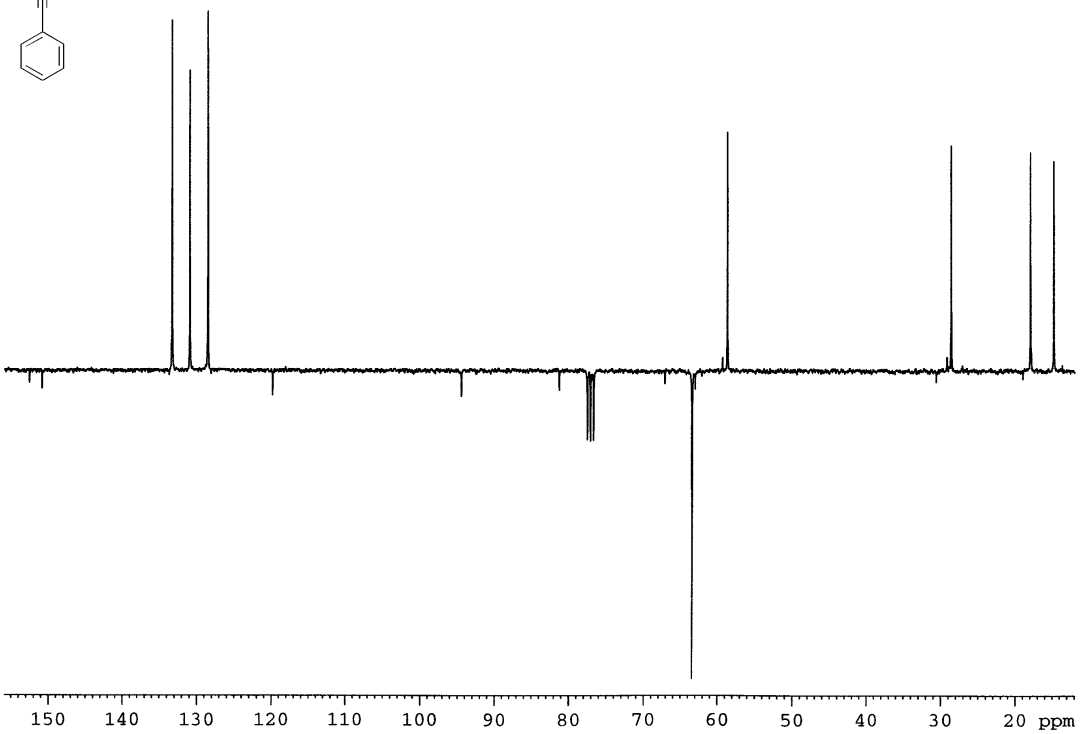
^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              5
PROCNO            22

F2 - Acquisition Parameters
Date_        20060528
Time            20.50
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG                 64
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1               3.00 usec
PL1             -3.00 dB
SFO1        300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1        300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF          300.2300165 MHz
WDW                no
SSB                 0
LB              0.00 Hz
GB                  0
PC               1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF          300.1300000 MHz
WDW                no
SSB                 2
LB              0.30 Hz
```

^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

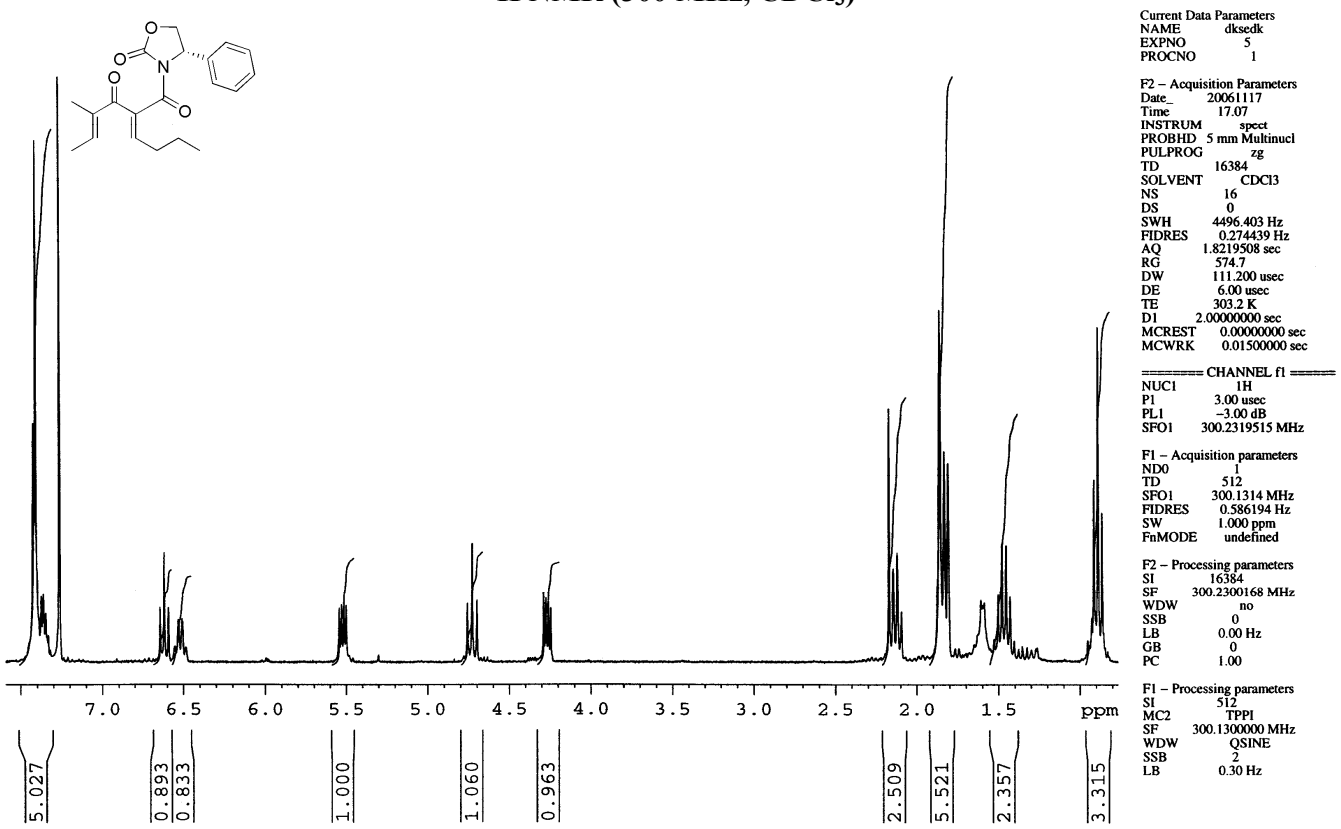




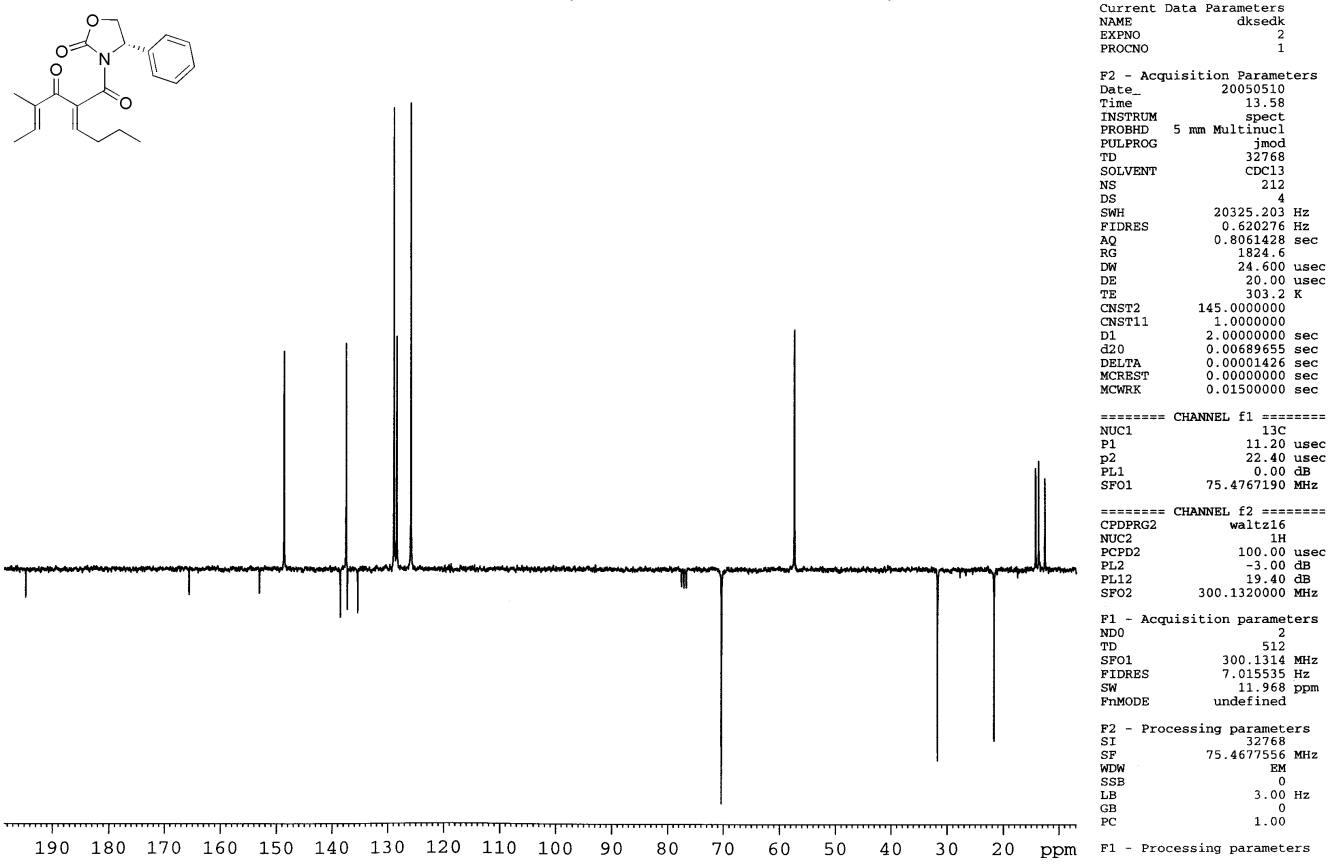

```
Current Data Parameters
NAME                 dksedk
EXPNO                     6
PROCNO                   22

F2 - Acquisition Parameters
Date_              20060528
Time                  20.57
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDC13
NS                    3750
DS                       4
SWH              18115.941 Hz
FIDRES            0.552855 Hz
AQ                0.9044468 sec
RG                   1149.4
DW                   27.600 usec
DE                     6.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.50000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1           75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2          300.2320007 MHz

F1 - Acquisition parameters
ND0                      3
TD                     512
SFO1           300.1314 MHz
FIDRES          0.586194 Hz
SW                1.000 ppm
FnMODE          undefined

F2 - Processing parameters
SI                    32768
SF             75.4928940 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```

(2*Z*,4*E*)-2-Butylidene-4-methyl-1-[(*S*)-2-oxo-4-phenyloxazolidin-3-yl]hex-4-ene-1,3-dione (30a)

^{1}H NMR (300 MHz, $CDCl_3$)

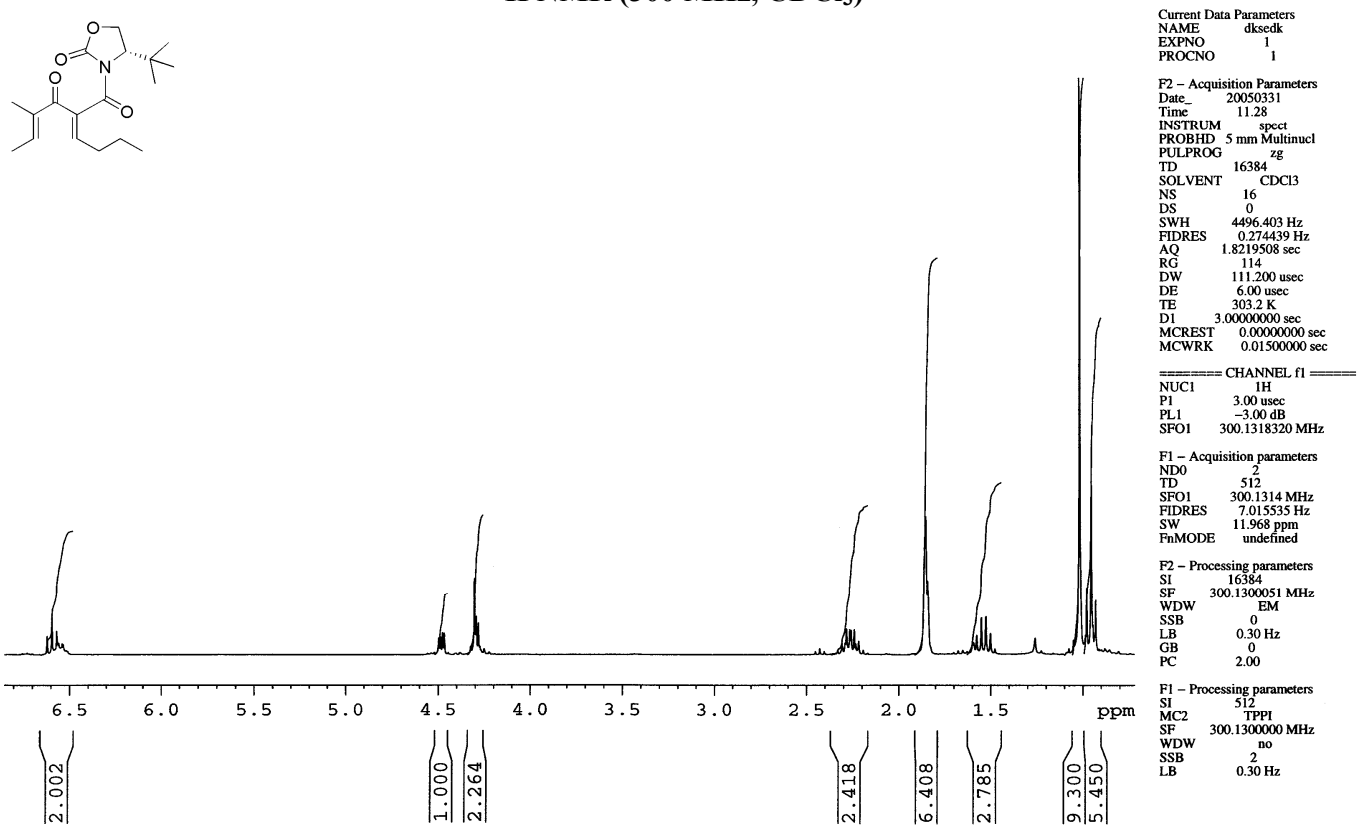


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

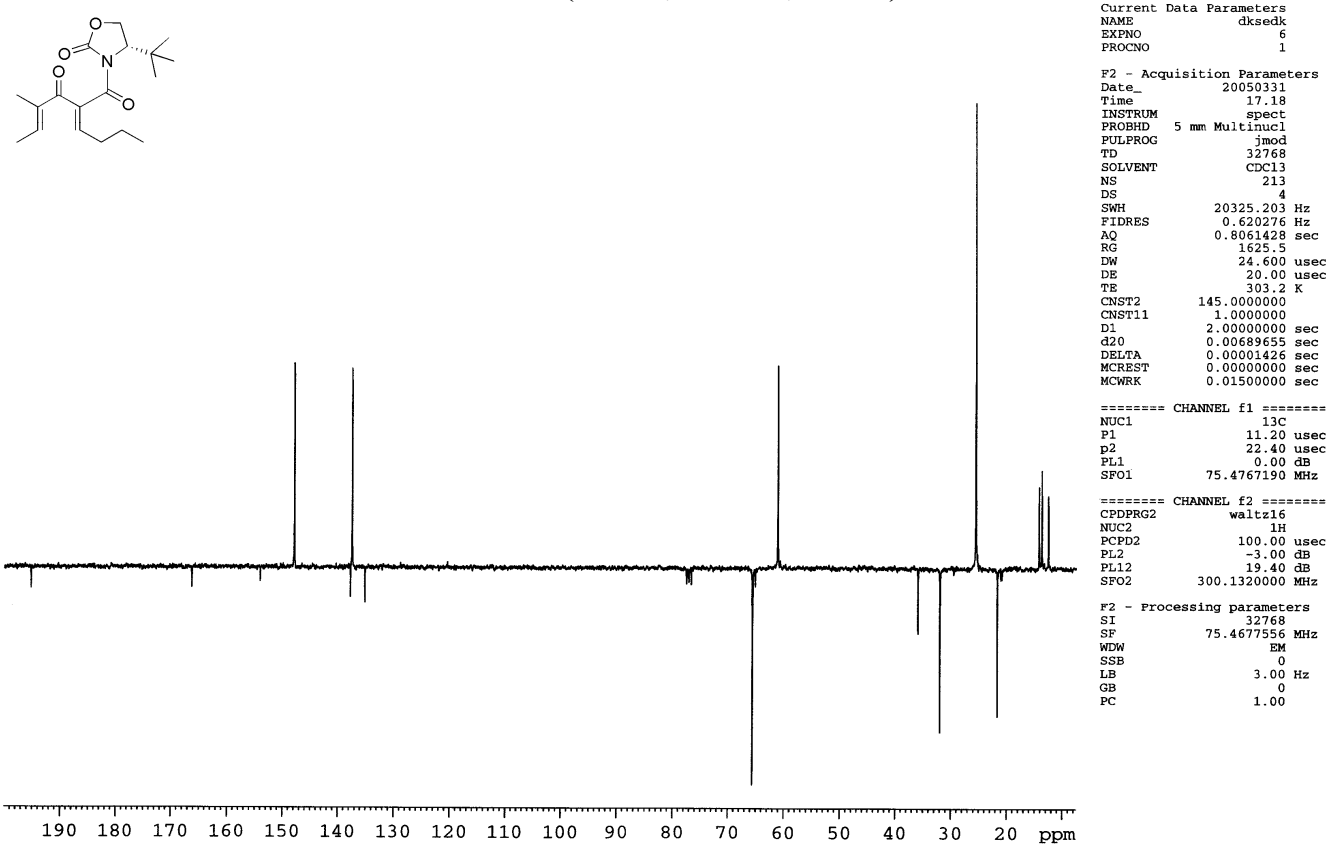


(2*Z*,4*E*)-1-[(*S*)-4-*tert*-Butyl-2-oxo-oxazolidin-3-yl]-2-butylidene-4-methylhex-4-ene-1,3-dione (30b)

^{1}H NMR (300 MHz, $CDCl_3$)

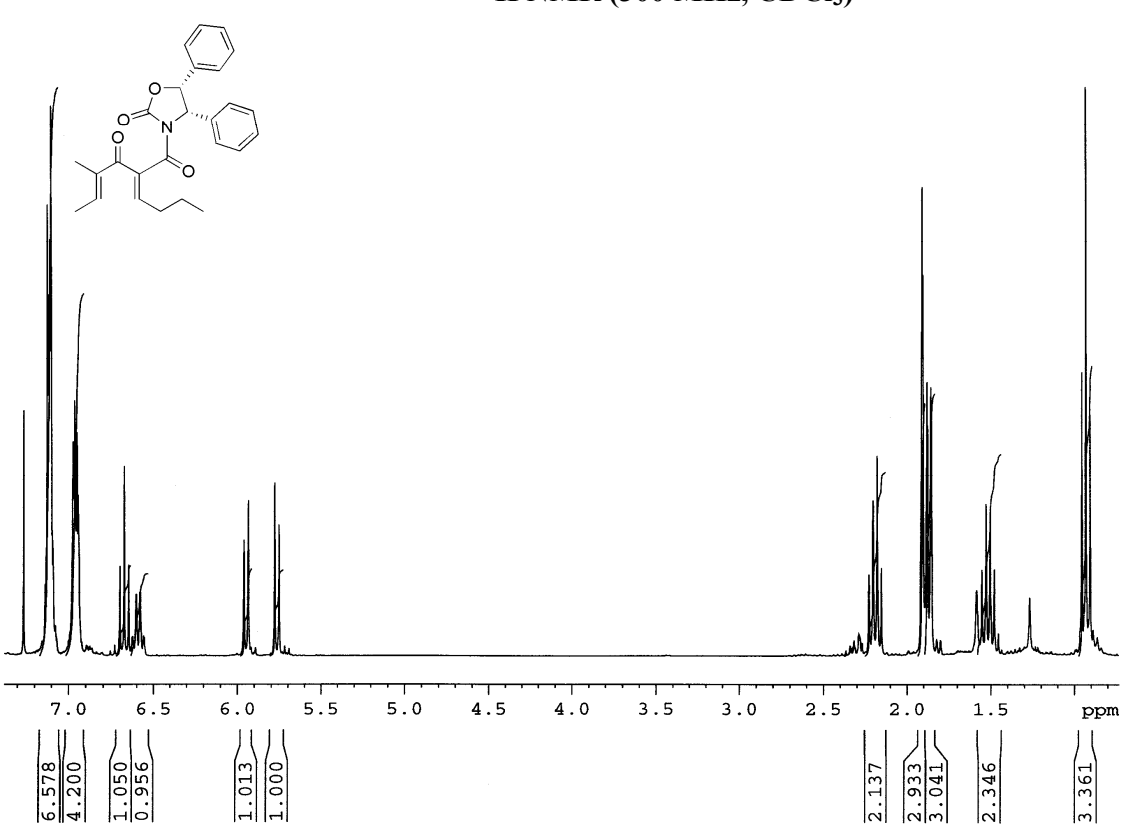


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



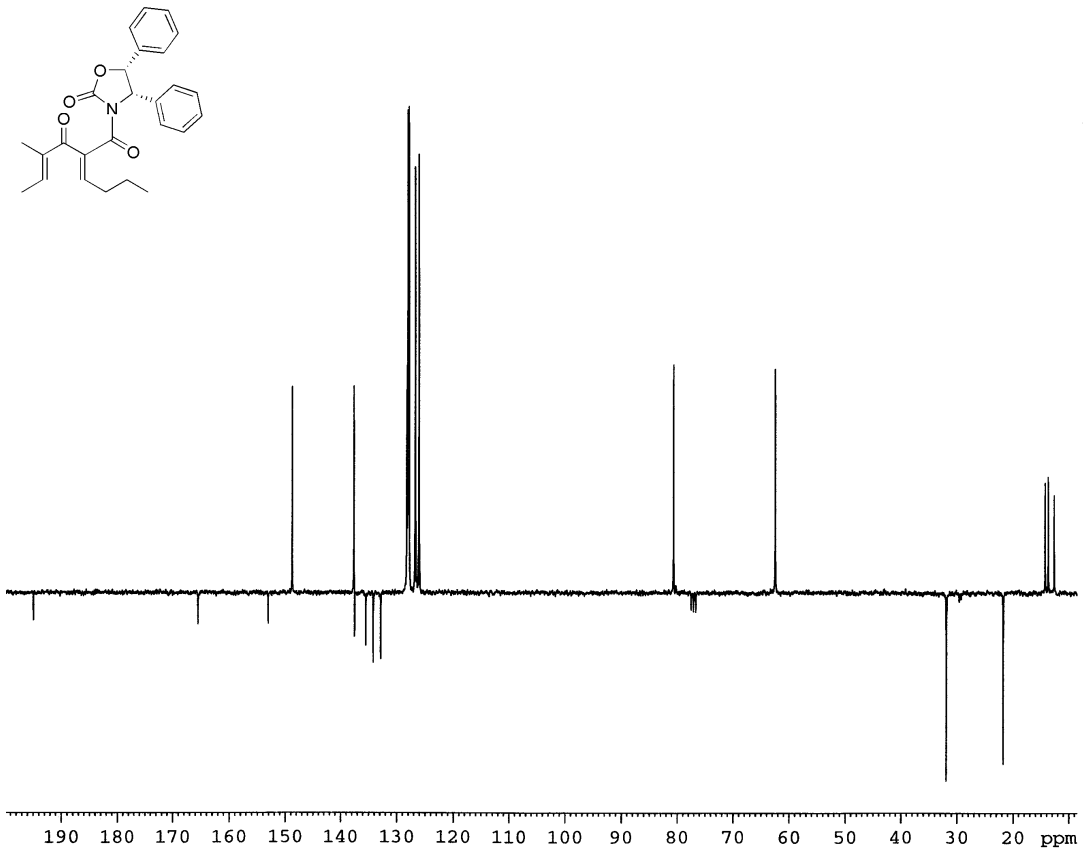
(2*Z*,4*E*)-2-Butylidene-4-methyl-1-[(4*S*,5*R*)-2-oxo-4,5-diphenyloxazolidin-3-yl]hex-4-ene-1,3-dione (30c)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_        20050331
Time            11.50
INSTRUM          spect
PROBHD   5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                 114
DW             111.200 usec
DE               6.00 usec
TE               303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1     300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1     300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW              no
SSB                2
LB              0.30 Hz
```

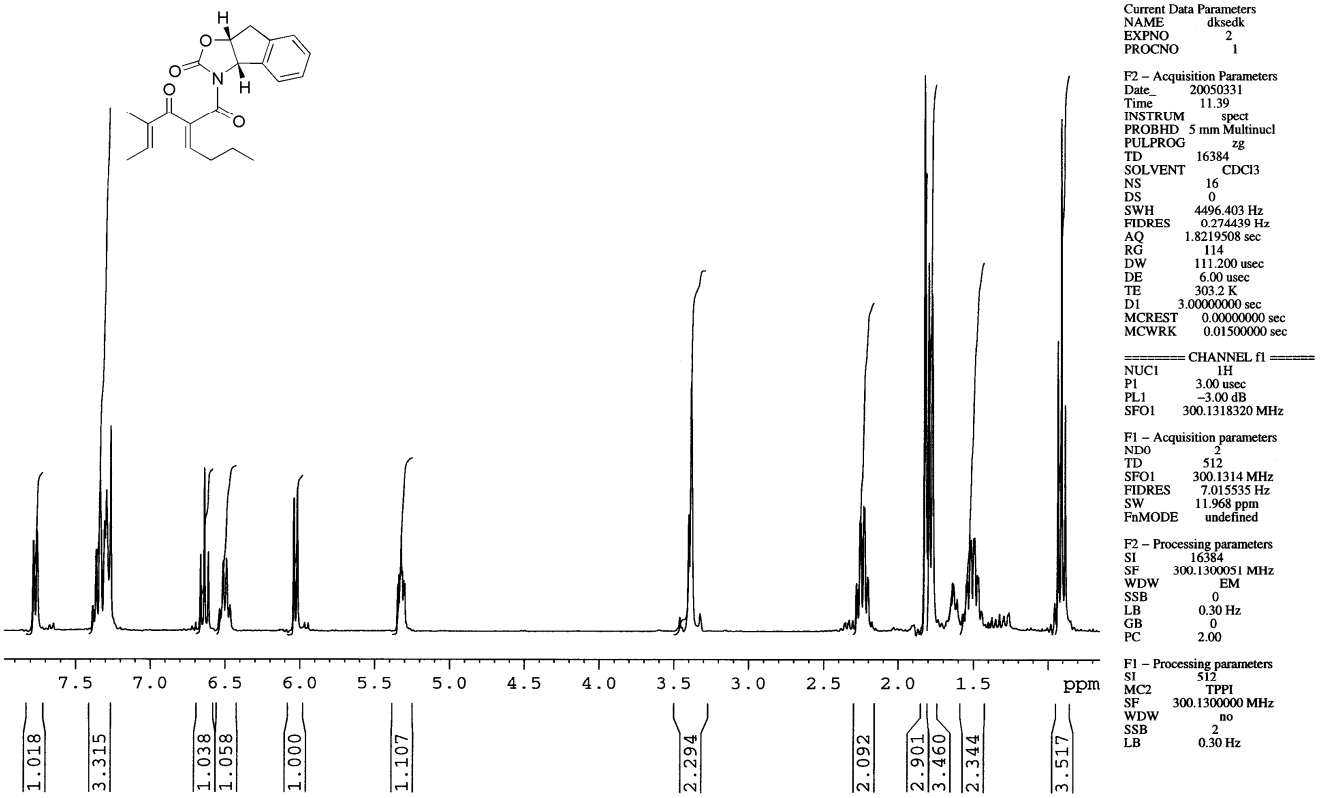
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     5
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050331
Time                  16.52
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     360
DS                       4
SWH             20325.203 Hz
FIDRES           0.620276 Hz
AQ               0.8061428 sec
RG                  1625.5
DW                 24.600 usec
DE                 20.00 usec
TE                 303.2 K
CNST2          145.0000000
CNST11           1.0000000
D1               2.00000000 sec
d20              0.00689655 sec
DELTA            0.00001426 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1          75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               19.40 dB
SFO2          300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF            75.4677556 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

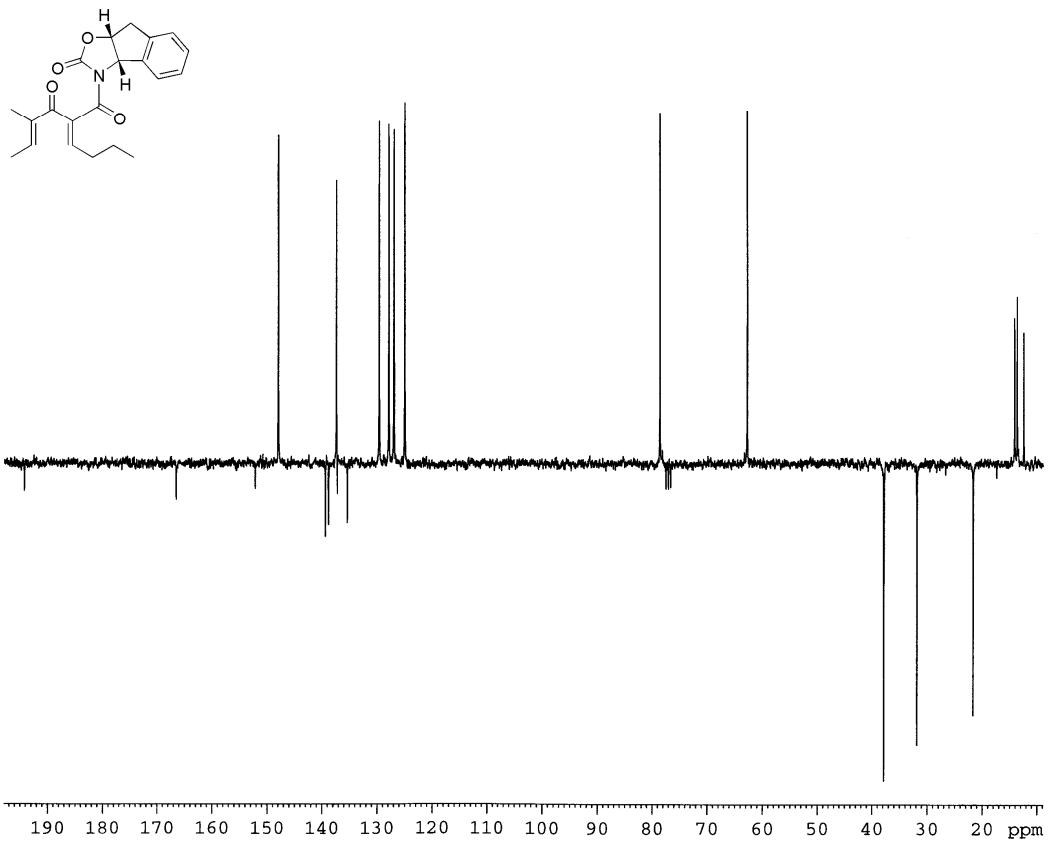
(2*Z*,4*E*)-2-Butylidene-4-methyl-1-[(3a*S*,8a*R*)-2-oxo-*2H*-indeno[1,2-*d*]oxazol-3(3a*H*,8*H*,8a*H*)-yl]hex-4-ene-1,3-dione (30d)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20050331
Time             11.39
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH            4496.403 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                  114
DW              111.200 usec
DE                6.00 usec
TE                303.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ======
NUC1                1H
P1               3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                  2
TD                 512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE         undefined

F2 - Processing parameters
SI                16384
SF        300.1300051 MHz
WDW                 EM
SSB                  0
LB              0.30 Hz
GB                   0
PC                2.00

F1 - Processing parameters
SI                 512
MC2               TPPI
SF        300.1300000 MHz
WDW                 no
SSB                  2
LB              0.30 Hz
```

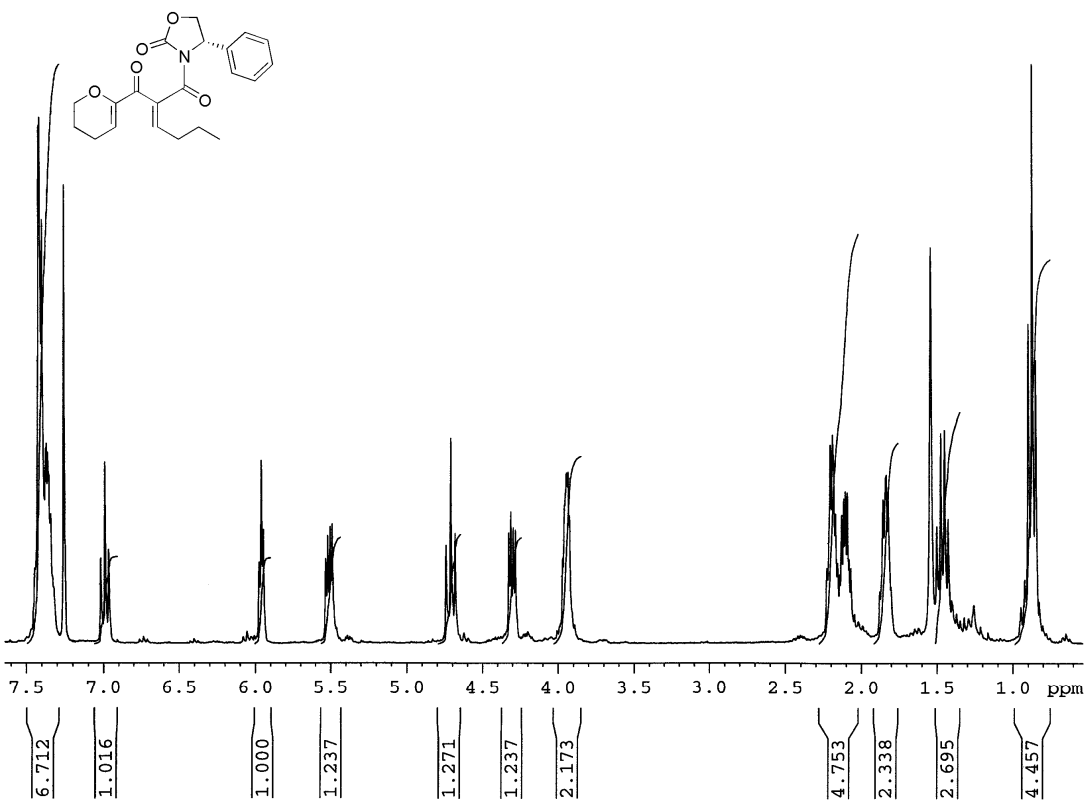
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050331
Time                  16.31
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                      193
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2          300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1           300.1314 MHz
FIDRES           7.015535 Hz
SW               11.968 ppm
FnMODE             undefined

F2 - Processing parameters
SI                     32768
SF             75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

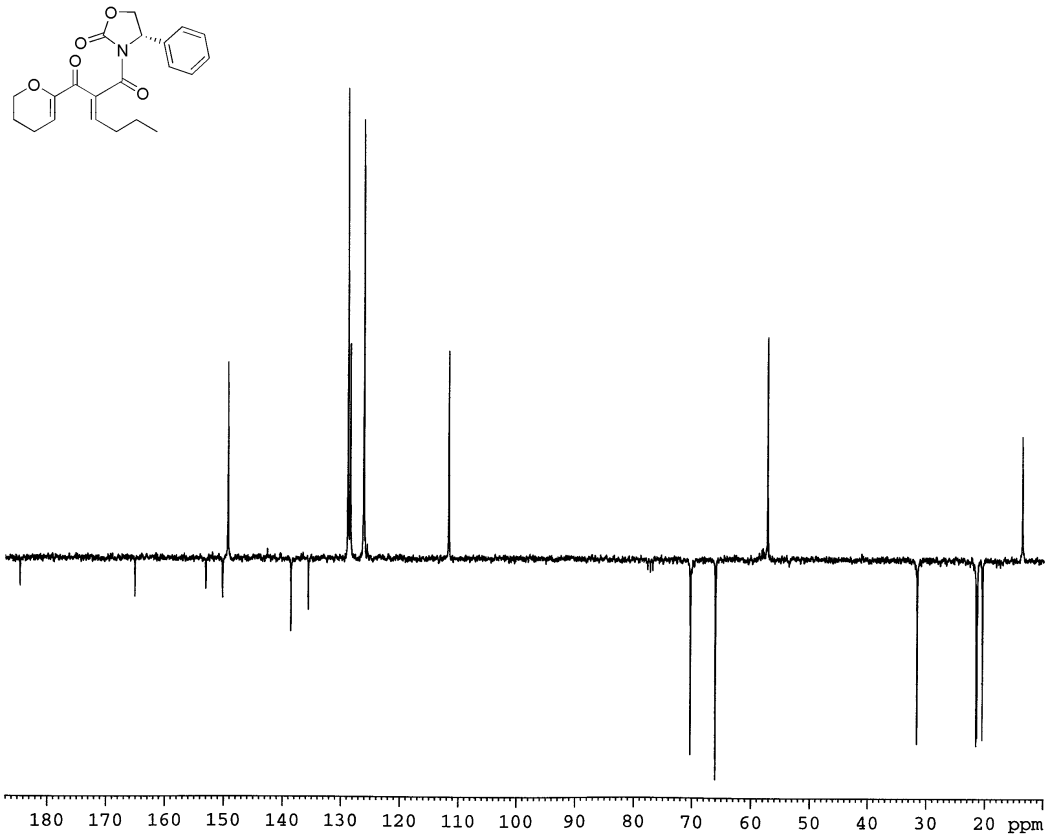
(*S*,*Z*)-2-Butylidene-1-(3,4-dihydro-2*H*-pyran-6-yl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30e)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO             73
PROCNO             1

F2 - Acquisition Parameters
Date_       20051206
Time            16.40
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG           zg
TD             16384
SOLVENT        CDCl3
NS                16
DS                 0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG              322.5
DW          111.200 usec
DE            6.00 usec
TE            303.2 K
D1        3.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ======
NUC1              1H
P1              3.00 usec
PL1            -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0               2
TD              512
SFO1      300.1314 MHz
FIDRES      7.015535 Hz
SW          11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI            16384
SF      300.1300051 MHz
WDW             EM
SSB              0
LB            0.30 Hz
GB               0
PC            2.00

F1 - Processing parameters
SI              512
MC2            TPPI
SF      300.1300000 MHz
WDW              no
SSB               2
LB            0.30 Hz
```

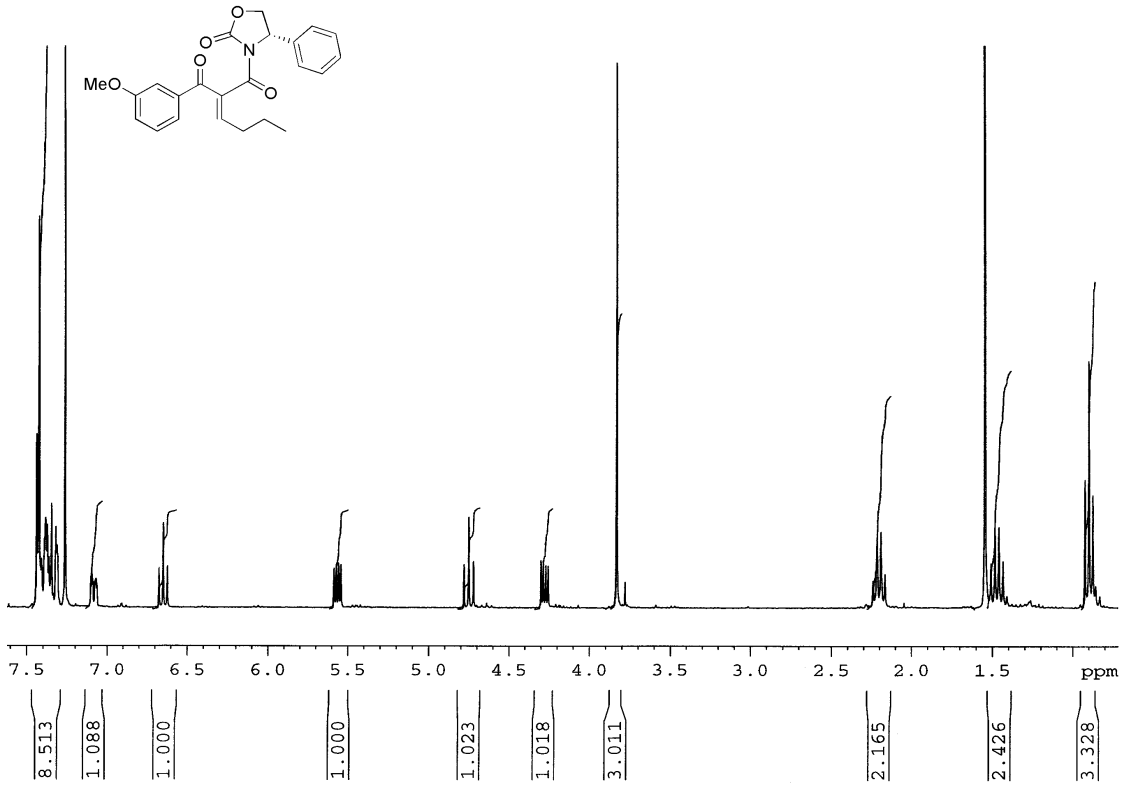
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME              dksedk
EXPNO                 74
PROCNO                 1

F2 - Acquisition Parameters
Date_           20051206
Time               15.55
INSTRUM            spect
PROBHD   5 mm Multinucl
PULPROG             jmod
TD                 32768
SOLVENT            CDCl3
NS                   107
DS                     4
SWH            20325.203 Hz
FIDRES          0.620276 Hz
AQ              0.8061428 sec
RG                1149.4
DW                24.600 usec
DE                20.00 usec
TE                303.2 K
CNST2         145.0000000
CNST11          1.0000000
D1            2.00000000 sec
d20           0.00689655 sec
DELTA         0.00001426 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               11.20 usec
p2               22.40 usec
PL1               0.00 dB
SFO1          75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2           100.00 usec
PL2              -3.00 dB
PL12             19.40 dB
SFO2         300.1320000 MHz

F1 - Acquisition parameters
ND0                   2
TD                  512
SFO1          300.1314 MHz
FIDRES          7.015535 Hz
SW                11.968 ppm
FnMODE         undefined

F2 - Processing parameters
SI                 32768
SF            75.4677556 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00

F1 - Processing parameters
```

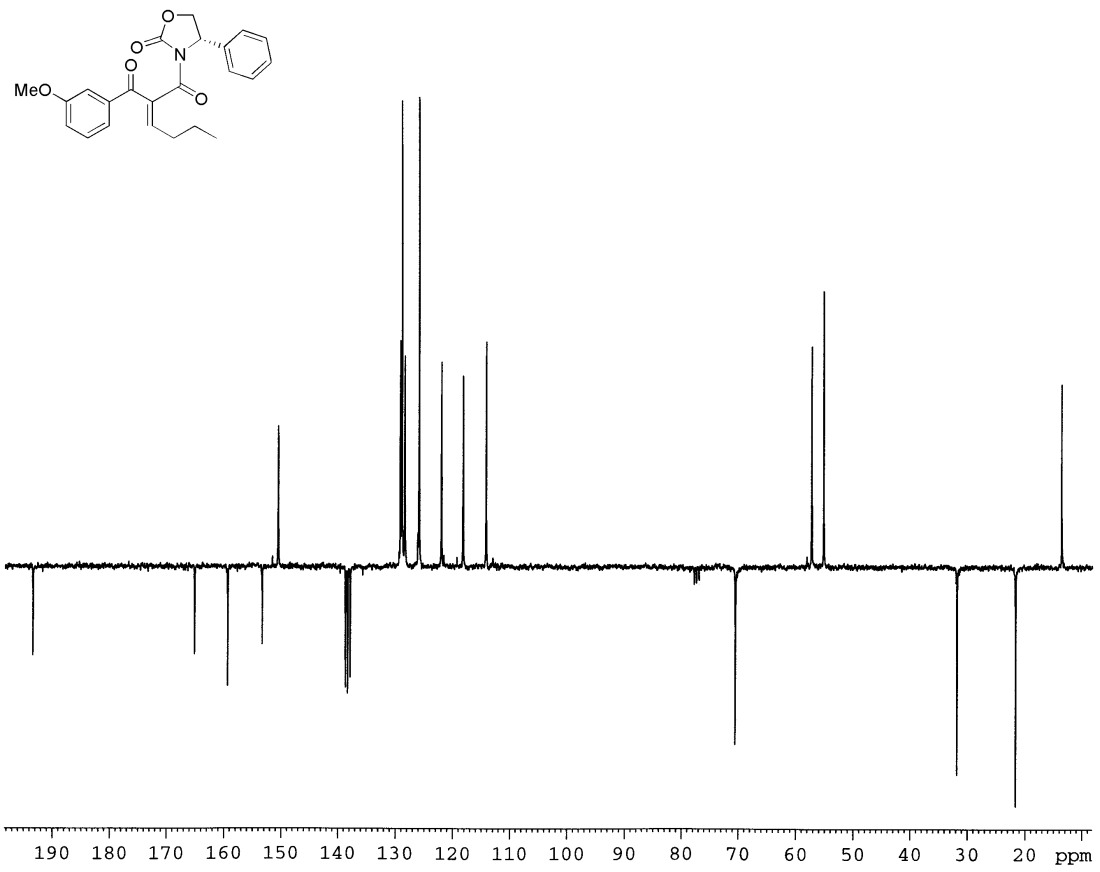
(*S*,*Z*)-2-Butylidene-1-(3-methoxyphenyl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30f)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              6
PROCNO             1

F2 - Acquisition Parameters
Date_         20050201
Time             16.36
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           4496.403 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                  512
DW              111.200 usec
DE                6.00 usec
TE                303.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ======
NUC1                1H
P1               3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                   2
TD                  512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW             11.968 ppm
FnMODE        undefined

F2 - Processing parameters
SI                16384
SF        300.1300051 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               2.00

F1 - Processing parameters
SI                  512
MC2                TPPI
SF        300.1300000 MHz
WDW                no
SSB                 2
LB               0.30 Hz
```

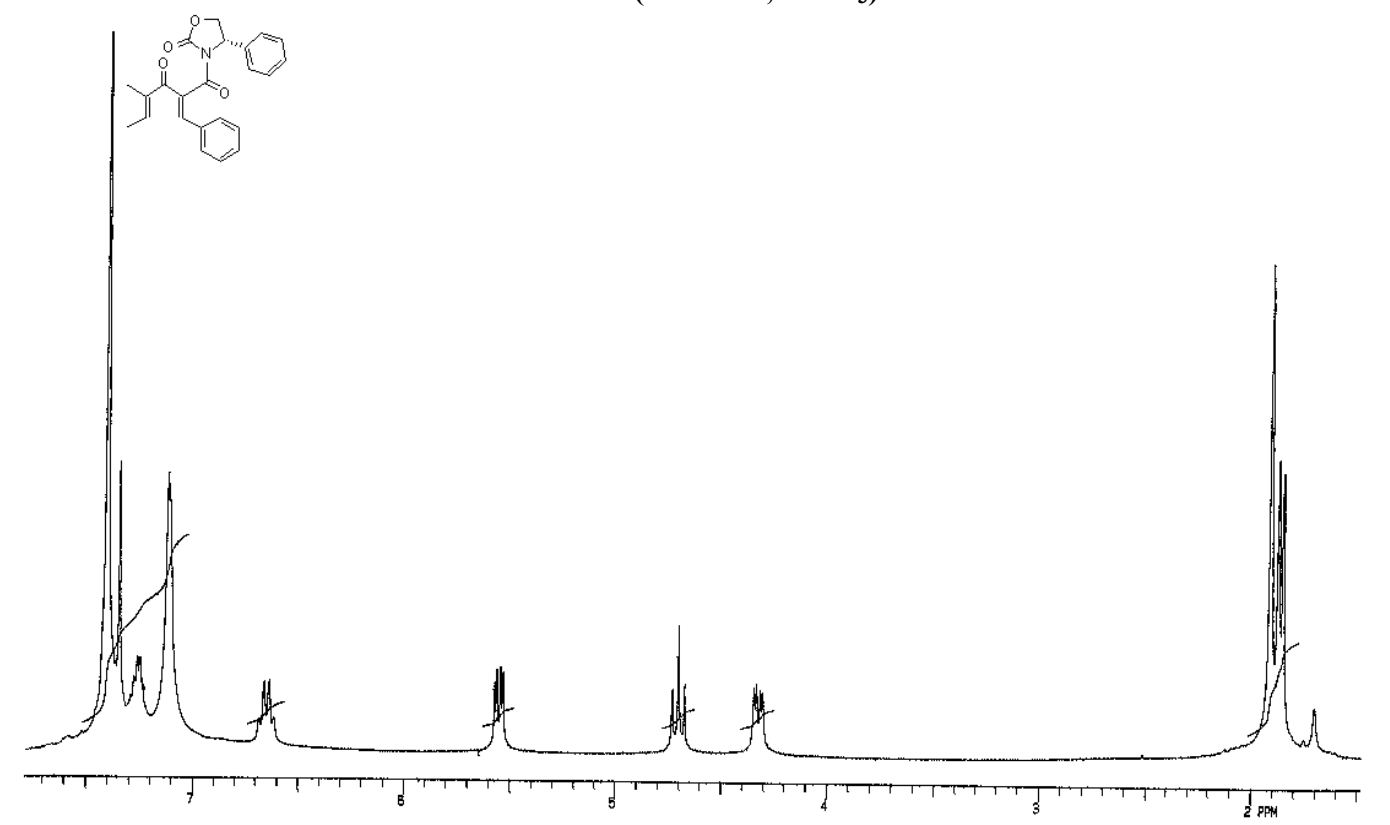
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



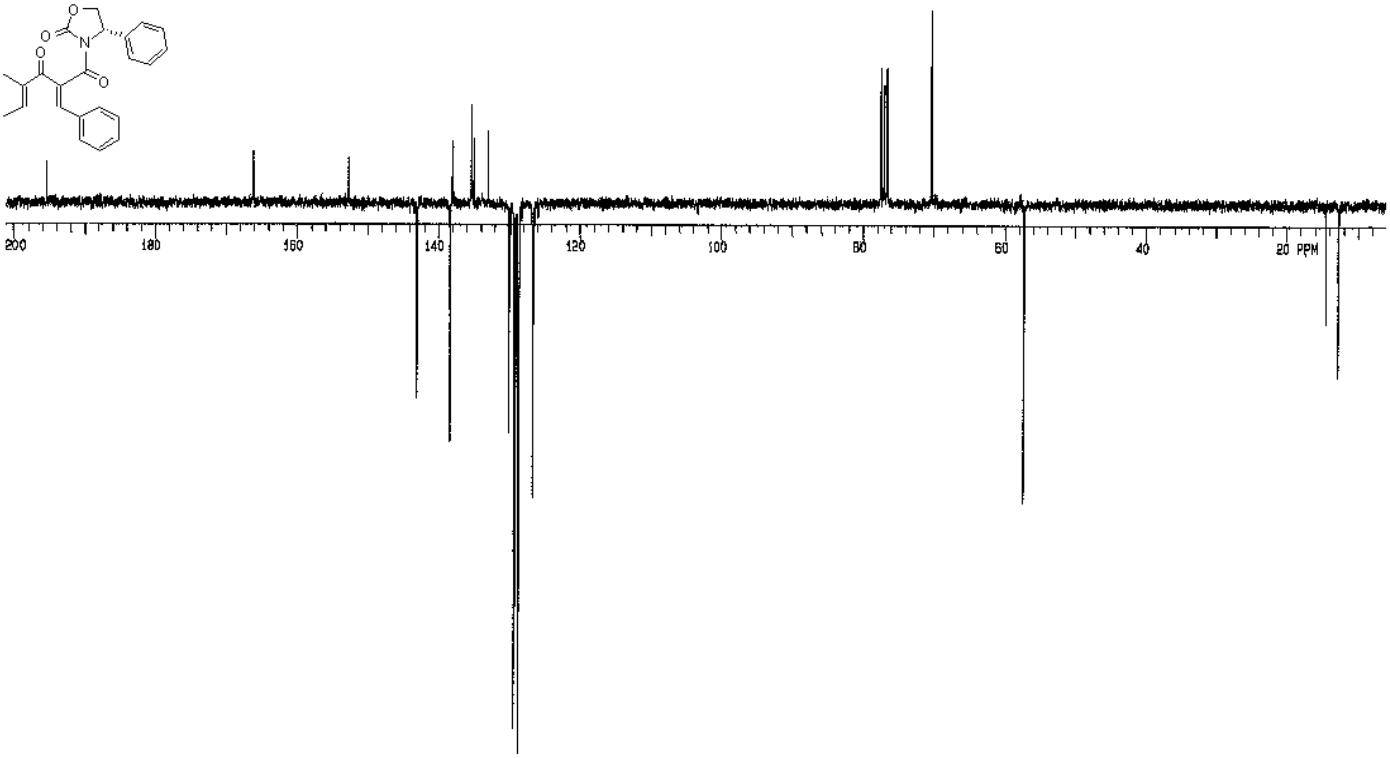

```
Current Data Parameters
NAME                 dksedk
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050427
Time                  15.43
INSTRUM               spect
PROBHD    5 mm Multinuc1
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     108
DS                       4
SWH              20325.203 Hz
FIDRES             0.620276 Hz
AQ                 0.8061428 sec
RG                    1824.6
DW                   24.600 usec
DE                   20.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2          300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1           300.1314 MHz
FIDRES           7.015535 Hz
SW                 11.968 ppm
FnMODE            undefined

F2 - Processing parameters
SI                    32768
SF             75.4677556 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```

(2*Z*,4*E*)-2-Benzylidene-4-methyl-1-[(*S*)-2-oxo-4-phenyloxazolidin-3-yl]hex-4-ene-1,3-dione (30g)

^{1}H NMR (300 MHz, $CDCl_3$)

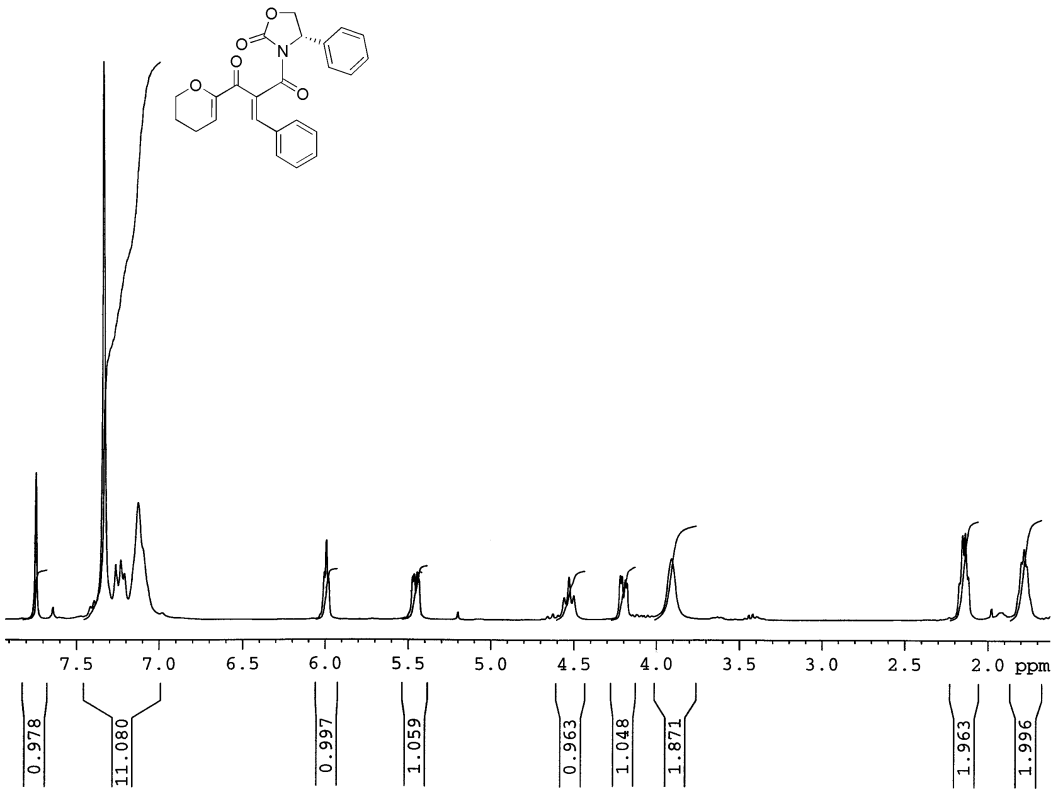


^{13}C NMR (APT, 75 MHz, $CDCl_3$)



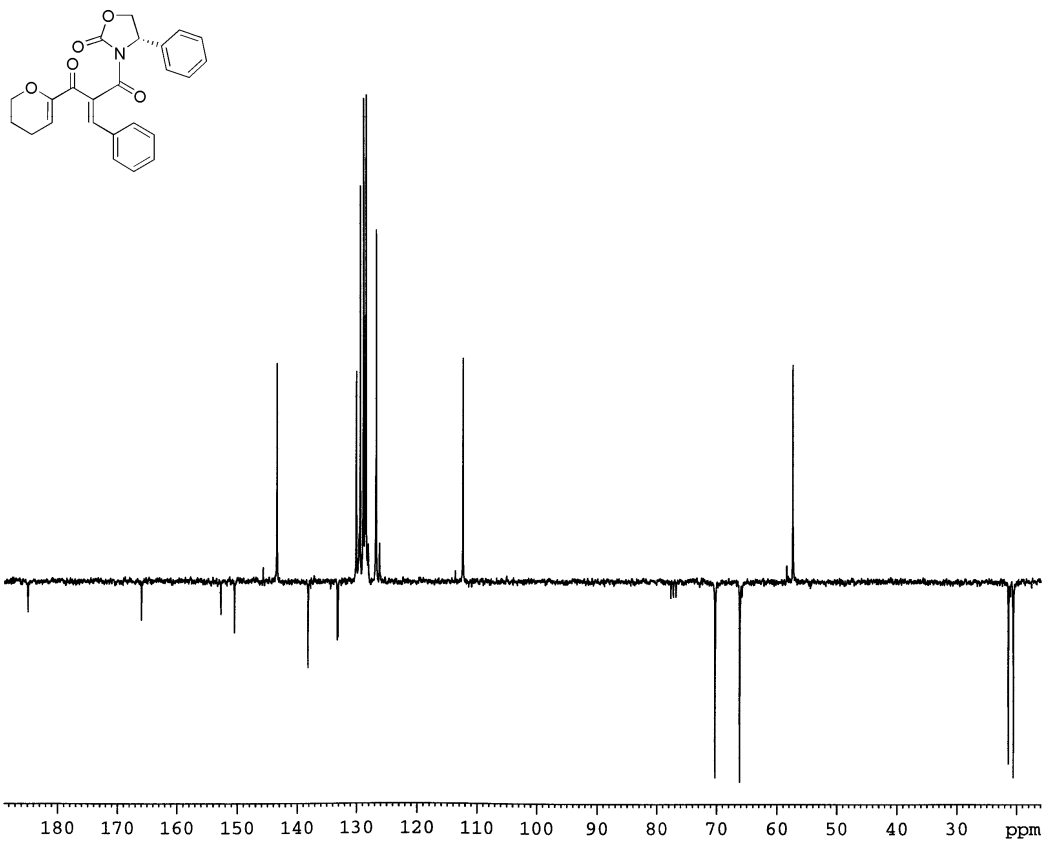
(*S*,*Z*)-2-Benzylidene-1-(3,4-dihydro-2*H*-pyran-6-yl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30h)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO             71
PROCNO             1

F2 - Acquisition Parameters
Date_        20051206
Time             15.20
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                20.2
DW           111.200 usec
DE             6.00 usec
TE             303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW            11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW              no
SSB               2
LB              0.30 Hz
```

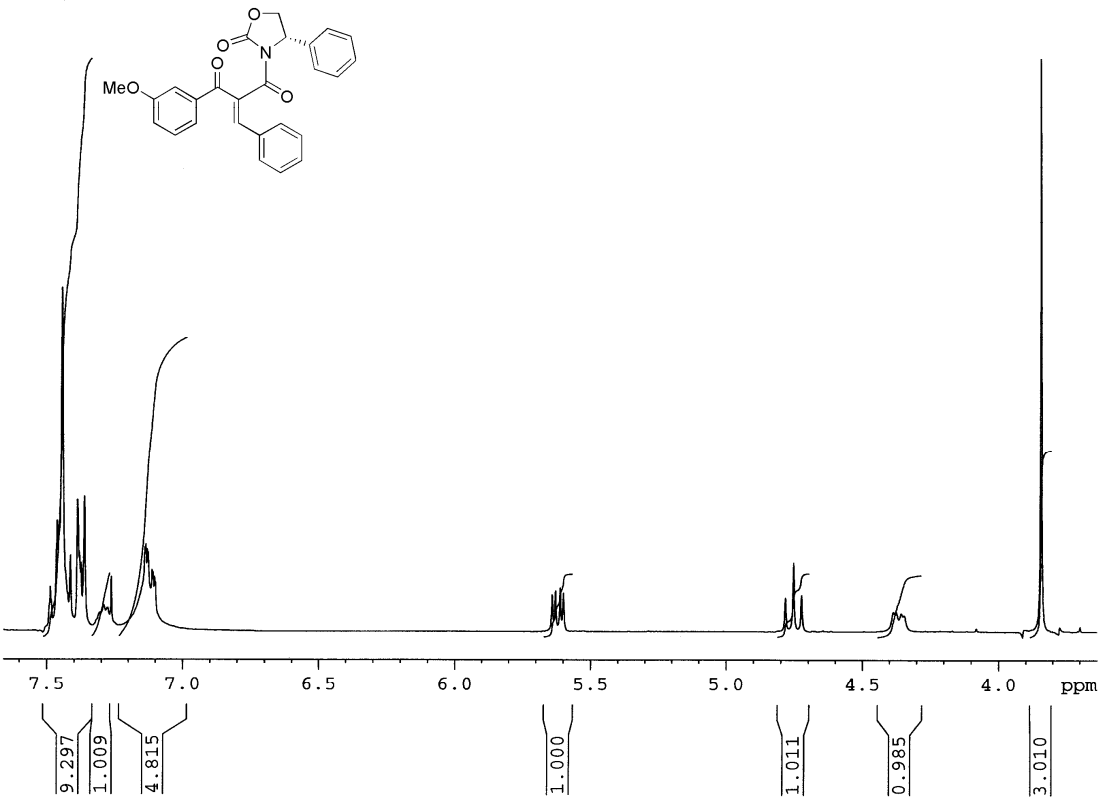
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                    72
PROCNO                    1

F2 - Acquisition Parameters
Date_             20051206
Time                 15.30
INSTRUM              spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     160
DS                       4
SWH              20325.203 Hz
FIDRES           0.620276 Hz
AQ               0.8061428 sec
RG                  1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2           300.1320000 MHz

F1 - Acquisition parameters
ND0                      2
TD                     512
SFO1             300.1314 MHz
FIDRES           7.015535 Hz
SW                11.968 ppm
FnMODE           undefined

F2 - Processing parameters
SI                    32768
SF               75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00

F1 - Processing parameters
```

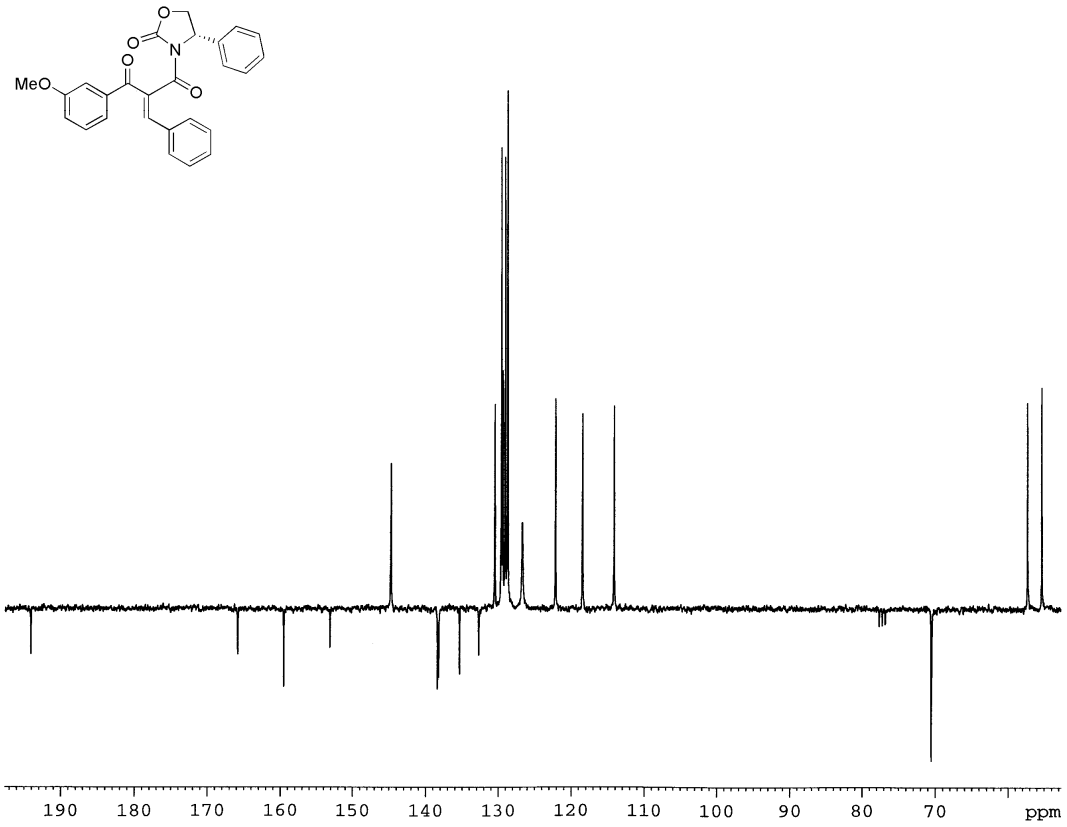
(*S*,*Z*)-2-Benzylidene-1-(3-methoxyphenyl)-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30i)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20050427
Time             15.19
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                71.8
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1           3.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
NDO                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW          11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW               no
SSB                2
LB              0.30 Hz
```

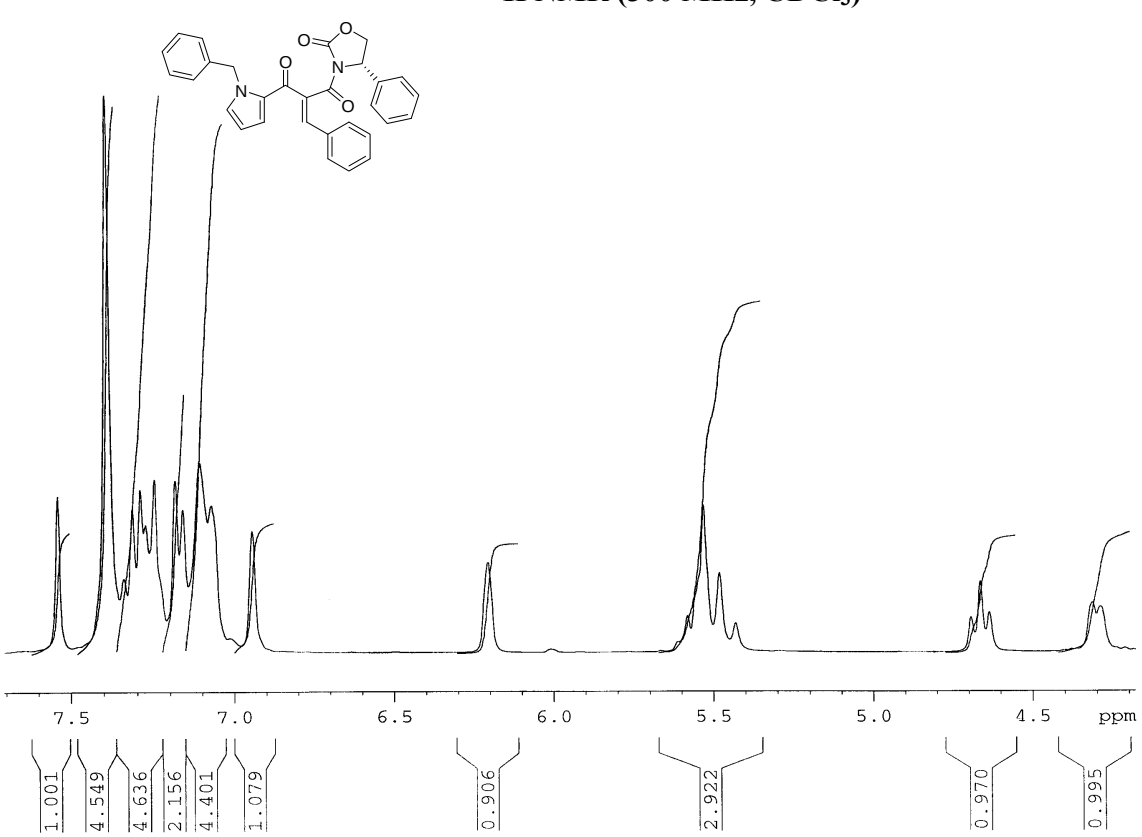
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050427
Time                  15.54
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     157
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2           300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

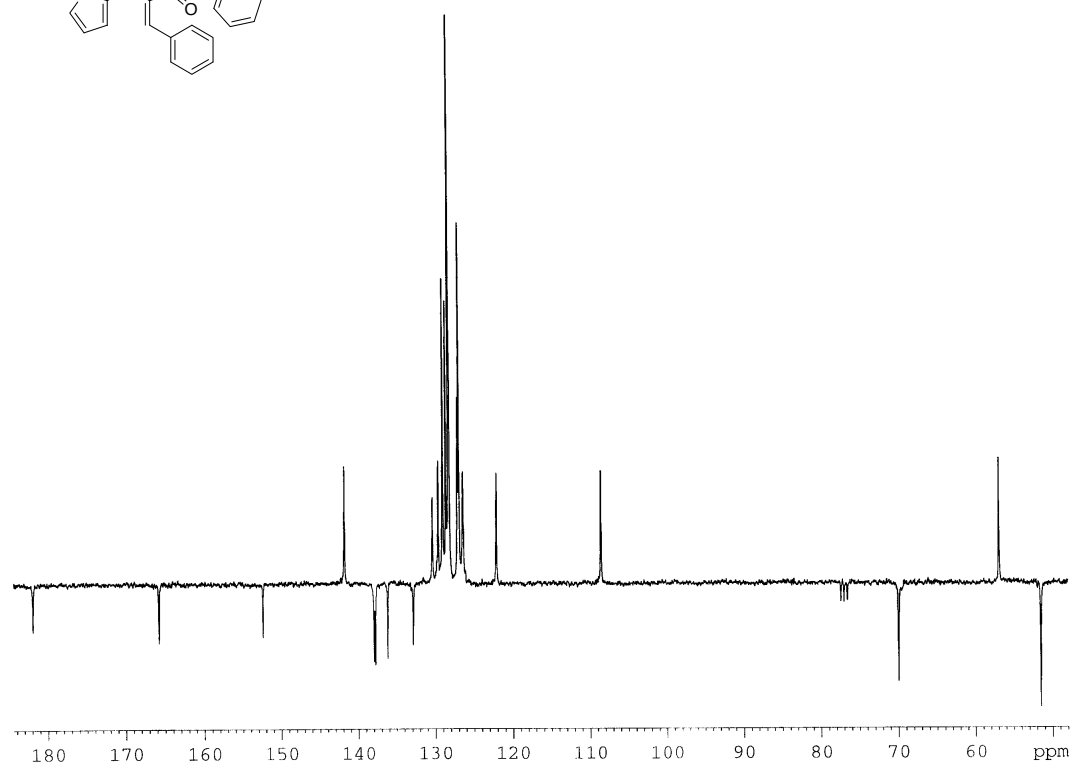
(*S*,*Z*)-1-(1-Benzyl-1*H*-pyrrol-2-yl)-2-benzylidene-3-(2-oxo-4-phenyloxazolidin-3-yl)propane-1,3-dione (30j)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          djsedj
EXPNO            152
PROCNO             1

F2 - Acquisition Parameters
Date_        20100118
Time            18.39
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG                 57
DW             111.200 usec
DE               6.00 usec
TE              294.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1      300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1      300.1314 MHz
FIDRES      0.586194 Hz
SW          1.000 ppm
FnMODE      undefined

F2 - Processing parameters
SI              16384
SF      300.2300226 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF      300.1300000 MHz
WDW               no
SSB                2
LB              0.30 Hz
```

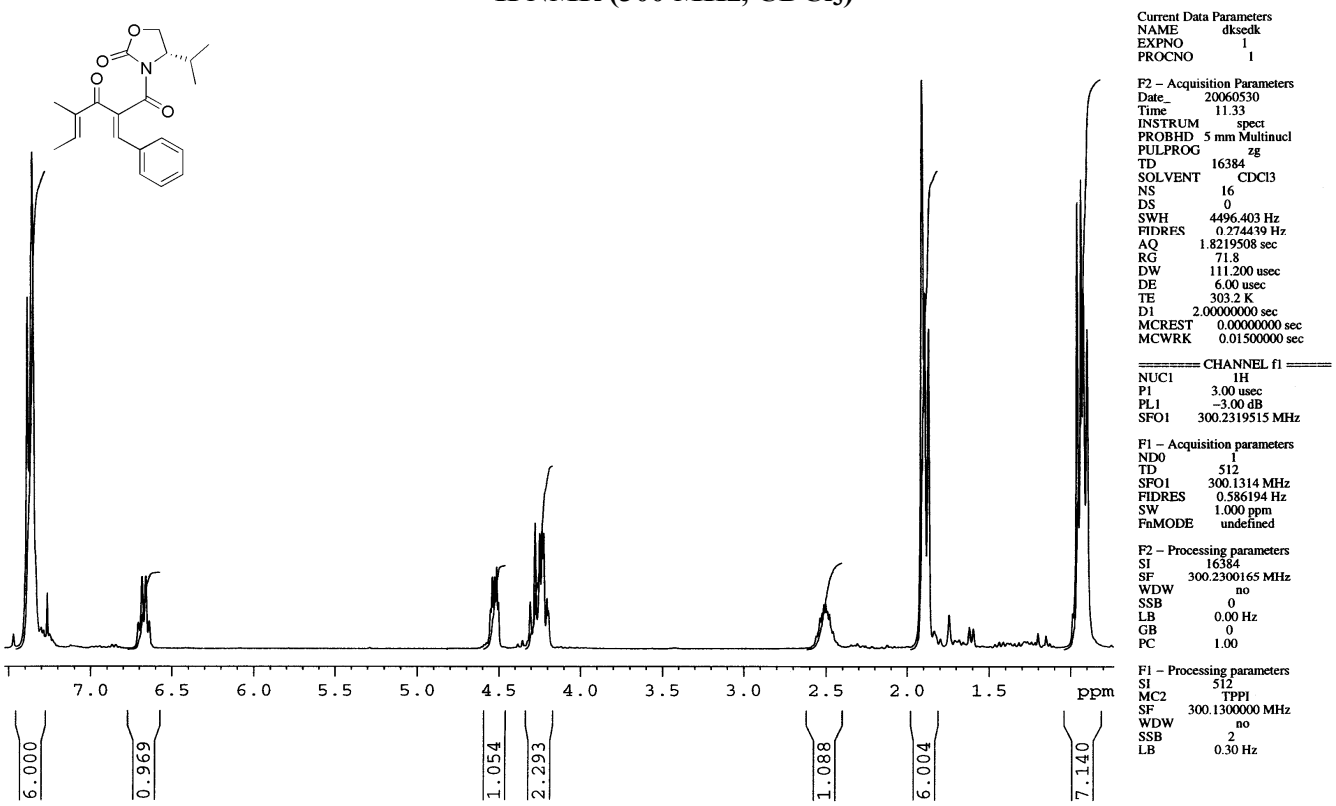
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          djsedj
EXPNO            153
PROCNO             1

F2 - Acquisition Parameters
Date_        20100118
Time            18.46
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG          jmod
TD              32768
SOLVENT         CDCl3
NS                 99
DS                  4
SWH         18115.941 Hz
FIDRES        0.552855 Hz
AQ          0.9044468 sec
RG             1625.5
DW              27.600 usec
DE               6.00 usec
TE              294.2 K
CNST2       145.0000000
CNST11        1.0000000
D1          2.00000000 sec
d20         0.00689655 sec
DELTA       0.00001426 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1              13C
P1              11.20 usec
p2              22.40 usec
PL1              0.00 dB
SFO1      75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          100.00 usec
PL2             -3.00 dB
PL12            19.40 dB
SFO2      300.2320007 MHz

F1 - Acquisition parameters
ND0                3
TD               512
SFO1      300.1314 MHz
FIDRES      0.586194 Hz
SW          1.000 ppm
FnMODE      undefined

F2 - Processing parameters
SI              32768
SF      75.4930097 MHz
WDW               EM
SSB                0
LB              3.00 Hz
GB                 0
PC              1.00

F1 - Processing parameters
```

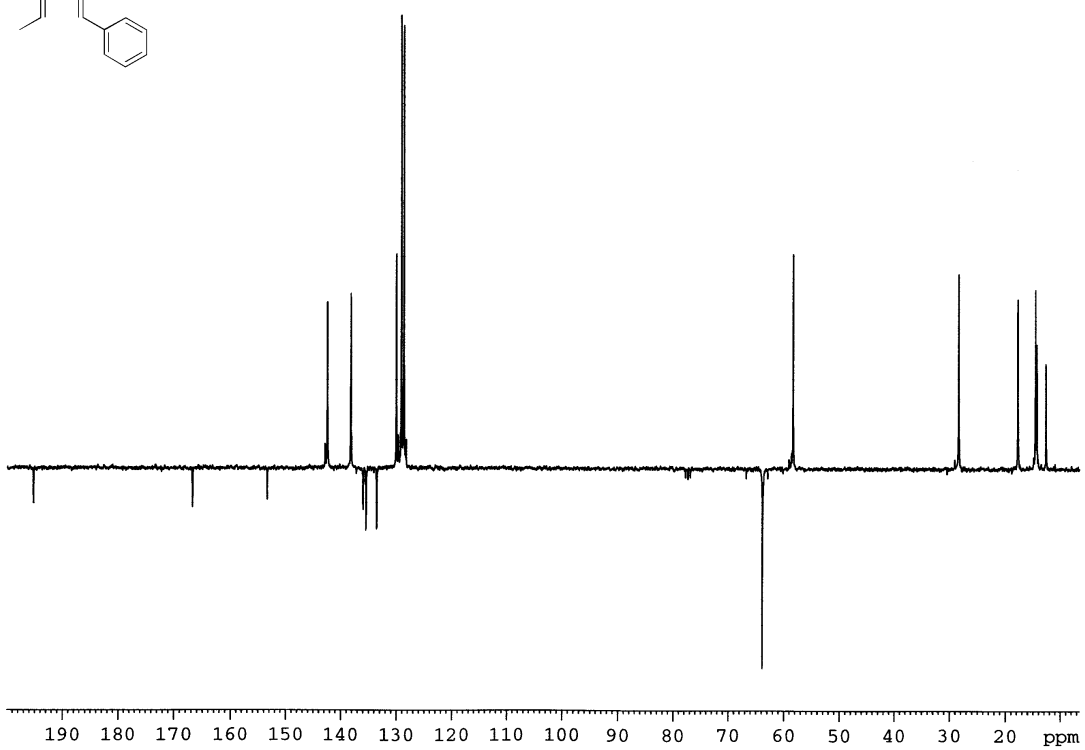
(2*Z*,4*E*)-2-Benzylidene-1-[(*S*)-4-isopropyl-2-oxooxazolidin-3-yl]-4-methylhex-4-ene-1,3-dione (30k)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20060530
Time            11.33
INSTRUM          spect
PROBHD   5 mm Multinucl
PULPROG             zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           4496.403 Hz
FIDRES        0.274439 Hz
AQ           1.8219508 sec
RG                 71.8
DW             111.200 usec
DE               6.00 usec
TE               303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1               3.00 usec
PL1             -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                   1
TD                  512
SFO1        300.1314 MHz
FIDRES        0.586194 Hz
SW              1.000 ppm
FnMODE          undefined

F2 - Processing parameters
SI                16384
SF        300.2300165 MHz
WDW                  no
SSB                   0
LB             0.00 Hz
GB                    0
PC                 1.00

F1 - Processing parameters
SI                  512
MC2                TPPI
SF        300.1300000 MHz
WDW                  no
SSB                   2
LB             0.30 Hz
```

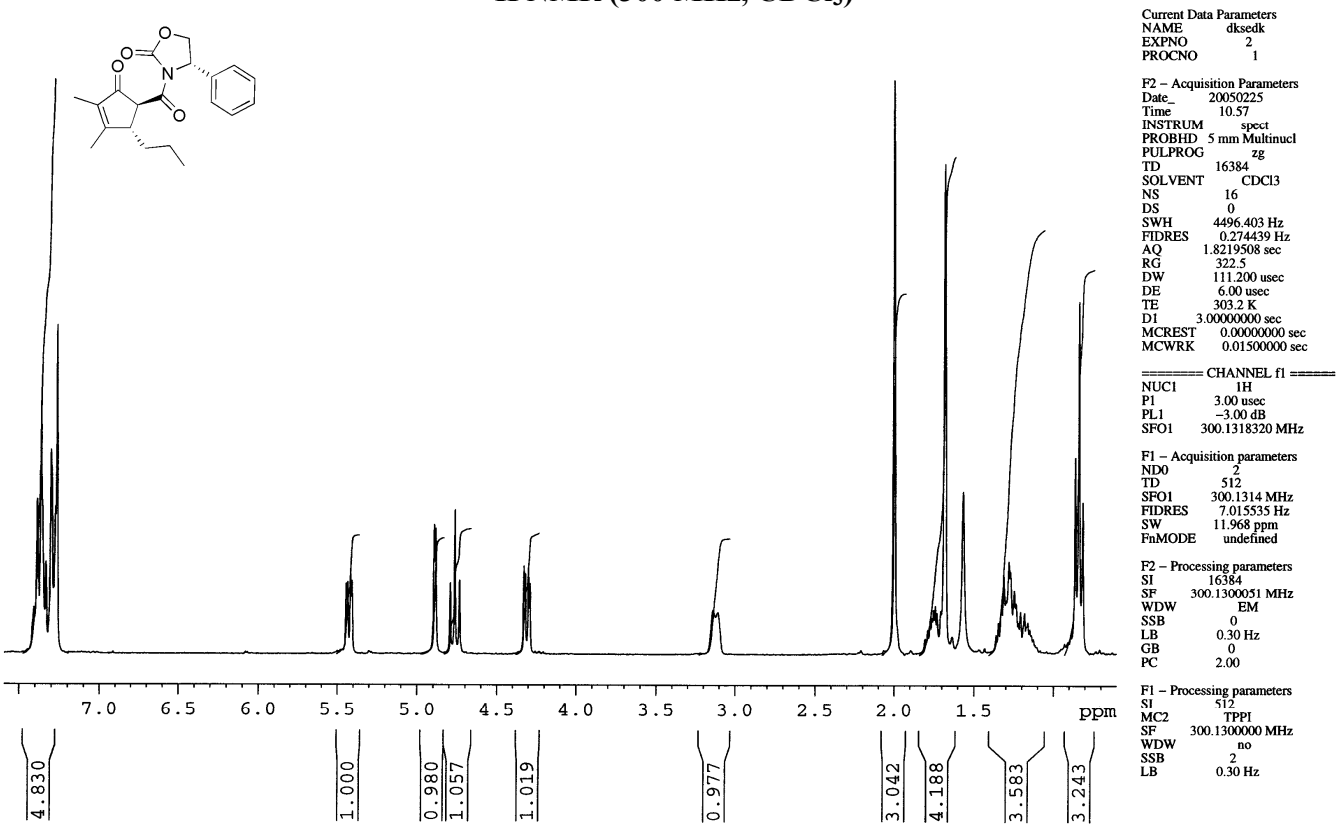
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060530
Time                  11.43
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     215
DS                       4
SWH              18115.941 Hz
FIDRES             0.552855 Hz
AQ                0.9044468 sec
RG                   1149.4
DW                   27.600 usec
DE                     6.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1            75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2           300.2320007 MHz

F1 - Acquisition parameters
ND0                       3
TD                      512
SFO1            300.1314 MHz
FIDRES            0.586194 Hz
SW                  1.000 ppm
FnMODE            undefined

F2 - Processing parameters
SI                    32768
SF              75.4928940 MHz
WDW                      EM
SSB                       0
LB                   3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```

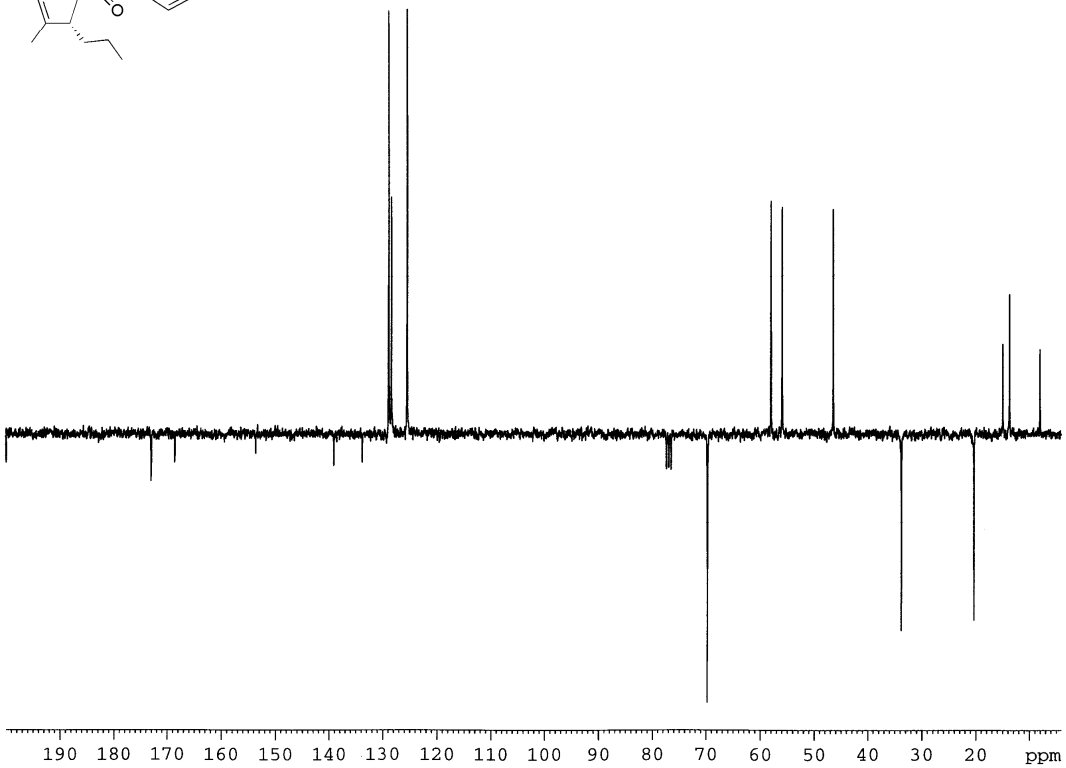
(*S*)-3-[(1*S*,5*S*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-4-phenyloxazolidin-2-one (31aC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20050225
Time            10.57
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG               322.5
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1      300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF        300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF        300.1300000 MHz
WDW              no
SSB                2
LB              0.30 Hz
```

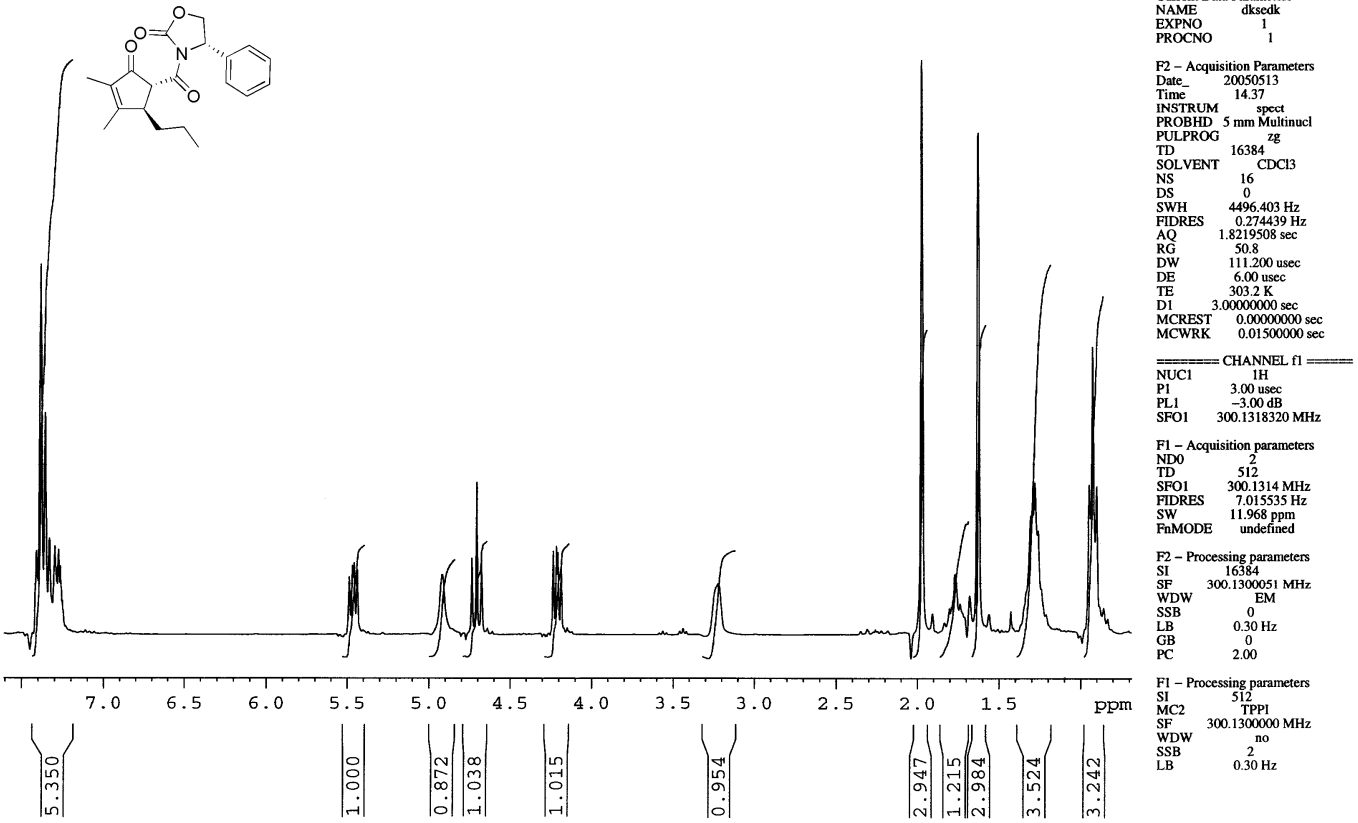
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



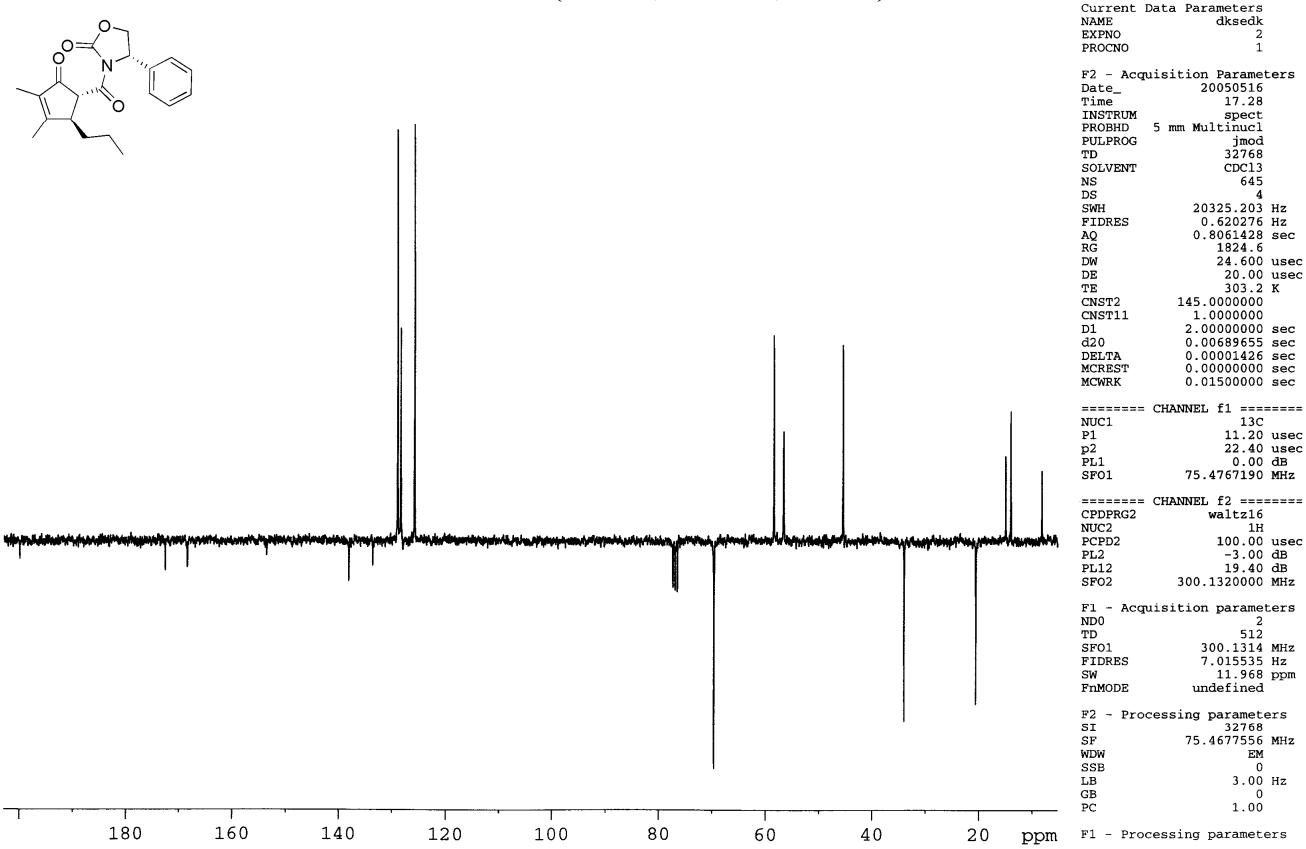

```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050428
Time                  18.23
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     331
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1824.6
DW                  24.600 usec
DE                  20.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1             75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2            300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1            300.1314 MHz
FIDRES           7.015535 Hz
SW                  11.968 ppm
FnMODE           undefined

F2 - Processing parameters
SI                     32768
SF               75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

(*S*)-3-[(1*R*,5*R*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-4-phenyloxazolidin-2-one (31aA)

^{1}H NMR (300 MHz, $CDCl_3$)

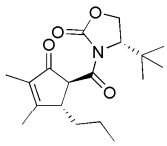


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



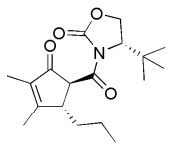
(*S*)-4-*tert*-Butyl-3-[(1*S*,5*S*)-3,4-dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]oxazolidin-2-one (31bC)

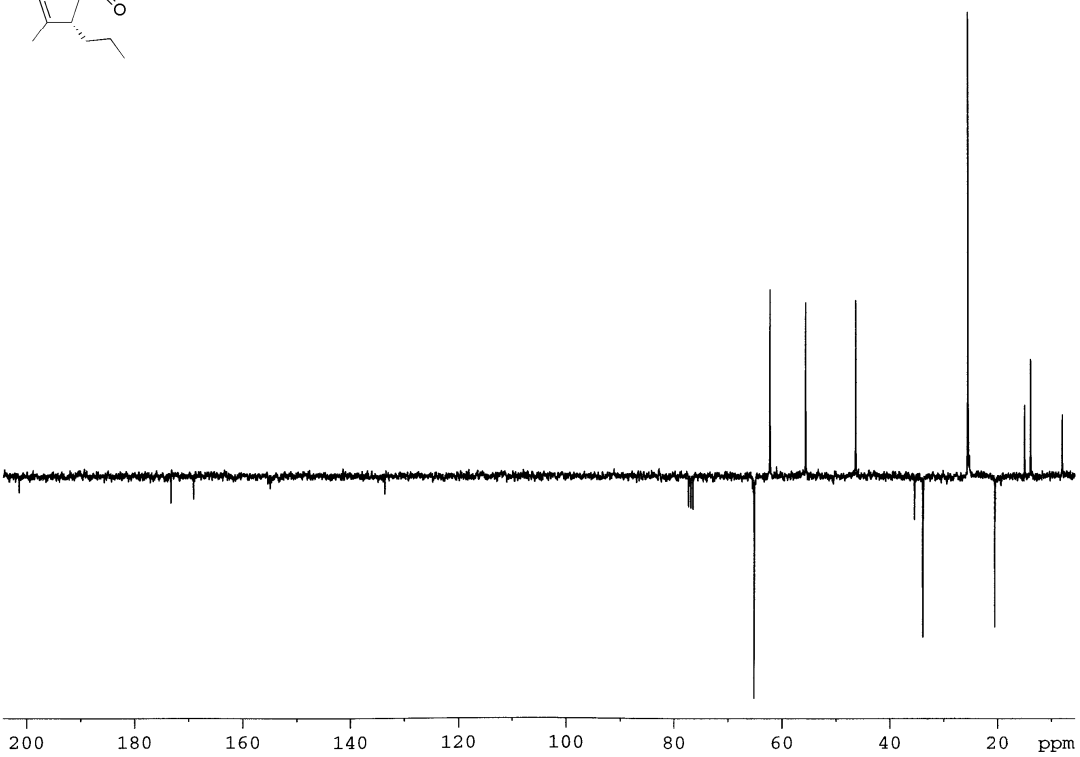
^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_        20050420
Time            16.14
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG                406.4
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW          11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW               no
SSB                2
LB              0.30 Hz
```

^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

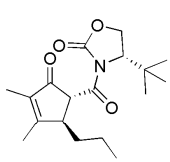





```
Current Data Parameters
NAME                 dksedk
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050509
Time                  11.43
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     413
DS                       4
SWH              20325.203 Hz
FIDRES             0.620276 Hz
AQ                 0.8061428 sec
RG                    1824.6
DW                   24.600 usec
DE                   20.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              2.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1            75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2           300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1              300.1314 MHz
FIDRES              7.015535 Hz
SW                 11.968 ppm
FnMODE           undefined

F2 - Processing parameters
SI                     32768
SF             75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

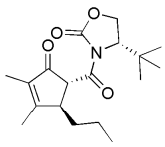
(*S*)-4-*tert*-Butyl-3-[(1*R*,5*R*)-3,4-dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]oxazolidin-2-one (31bA)

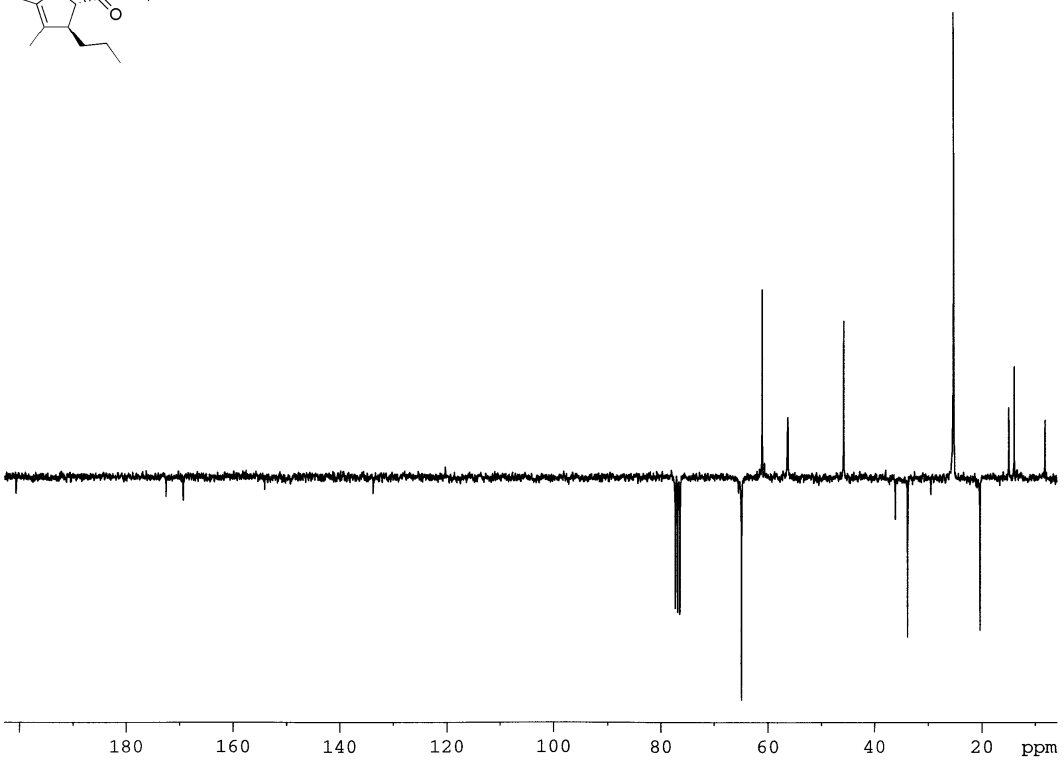
^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20050509
Time            11.04
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                114
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1     300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1     300.1314 MHz
FIDRES     7.015535 Hz
SW          11.968 ppm
FnMODE      undefined

F2 - Processing parameters
SI              16384
SF       300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW           QSINE
SSB                2
LB              0.30 Hz
```

^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

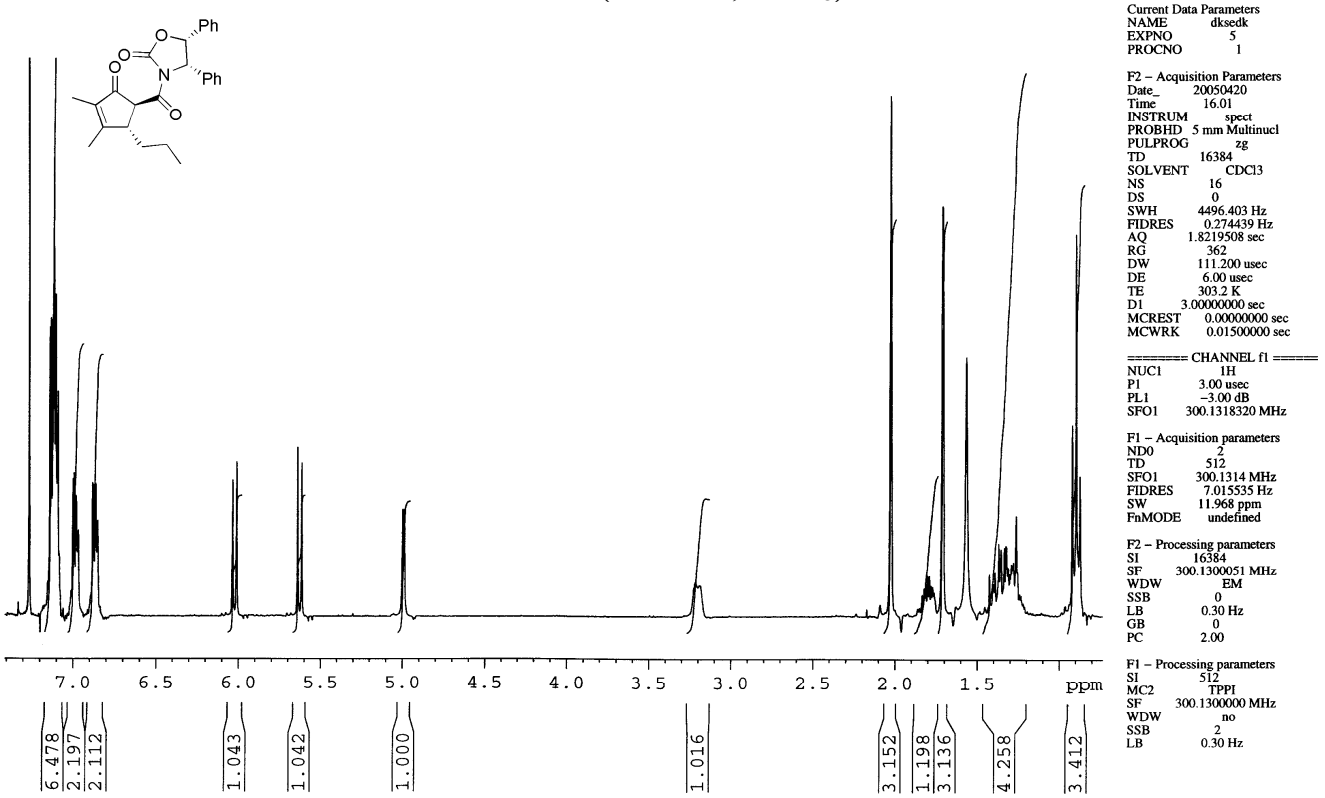





```
Current Data Parameters
NAME                 dksedk
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050517
Time                   2.02
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                   10000
DS                       4
SWH                20325.203 Hz
FIDRES              0.620276 Hz
AQ                  0.8061428 sec
RG                     1625.5
DW                    24.600 usec
DE                    20.00 usec
TE                    303.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                  2.00000000 sec
d20                 0.00689655 sec
DELTA               0.00001426 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.20 usec
p2                     22.40 usec
PL1                     0.00 dB
SFO1               75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2              300.1320000 MHz

F2 - Processing parameters
SI                       32768
SF                75.4677556 MHz
WDW                        EM
SSB                         0
LB                       3.00 Hz
GB                          0
PC                       1.00
```

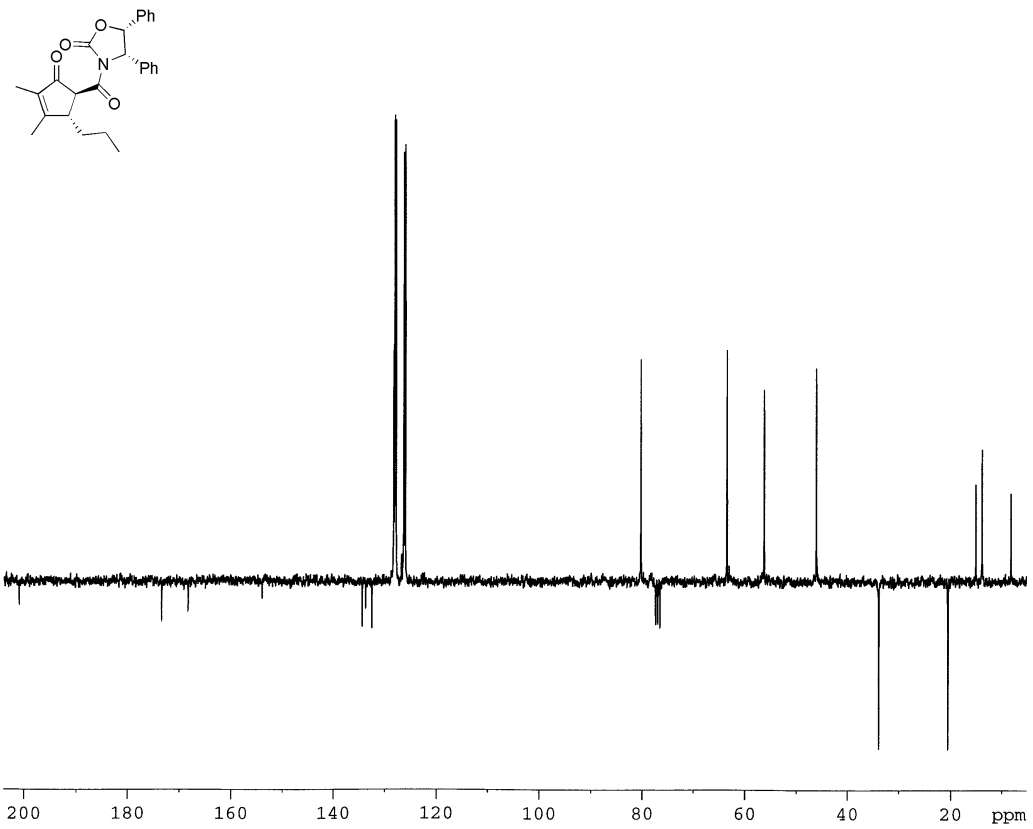
(4*S*,5*R*)-3-[(1*S*,5*S*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-4,5-diphenyloxazolidin-2-one (31cC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              5
PROCNO             1

F2 - Acquisition Parameters
Date_         20050420
Time             16.01
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                362
DW            111.200 usec
DE              6.00 usec
TE             303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1               3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW              no
SSB                2
LB               0.30 Hz
```

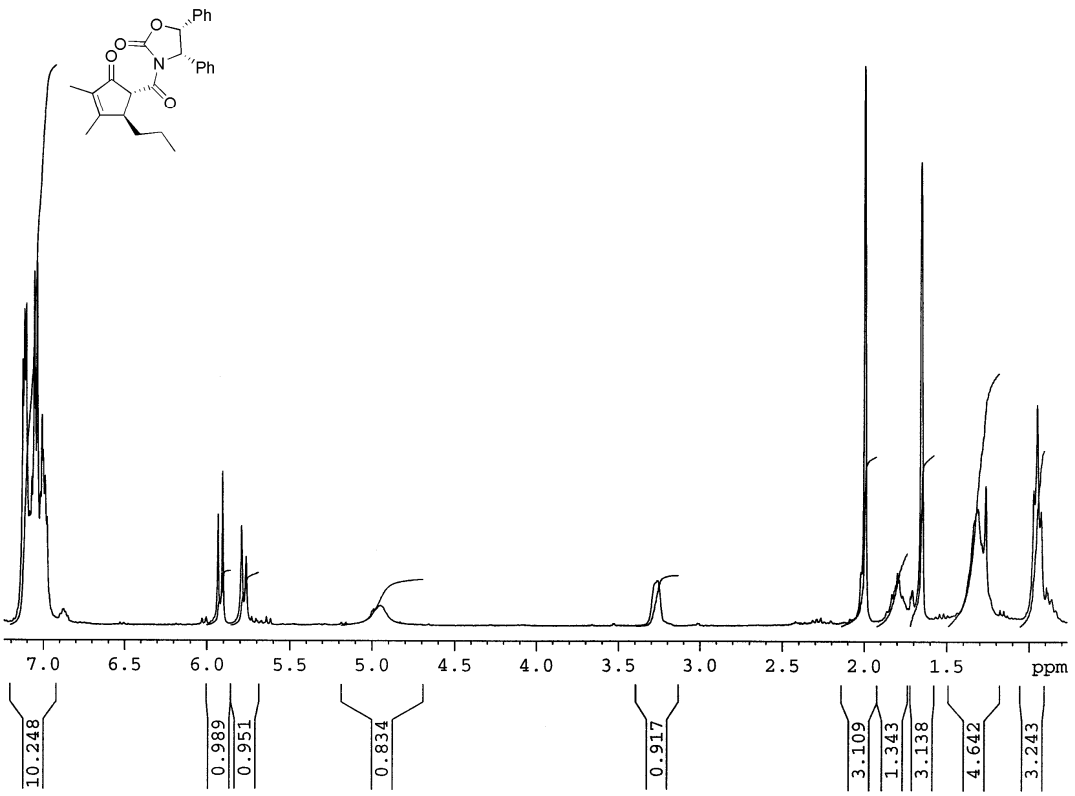
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     5
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050509
Time                  15.36
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     567
DS                       4
SWH           20325.203 Hz
FIDRES         0.620276 Hz
AQ             0.8061428 sec
RG                1824.6
DW              24.600 usec
DE              20.00 usec
TE              303.2 K
CNST2        145.0000000
CNST11         1.0000000
D1           2.00000000 sec
d20          0.00689655 sec
DELTA        0.00001426 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                11.20 usec
p2                22.40 usec
PL1                0.00 dB
SFO1          75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2               -3.00 dB
PL12              19.40 dB
SFO2         300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1            300.1314 MHz
FIDRES            7.015535 Hz
SW                  11.968 ppm
FnMODE          undefined

F2 - Processing parameters
SI                     32768
SF           75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

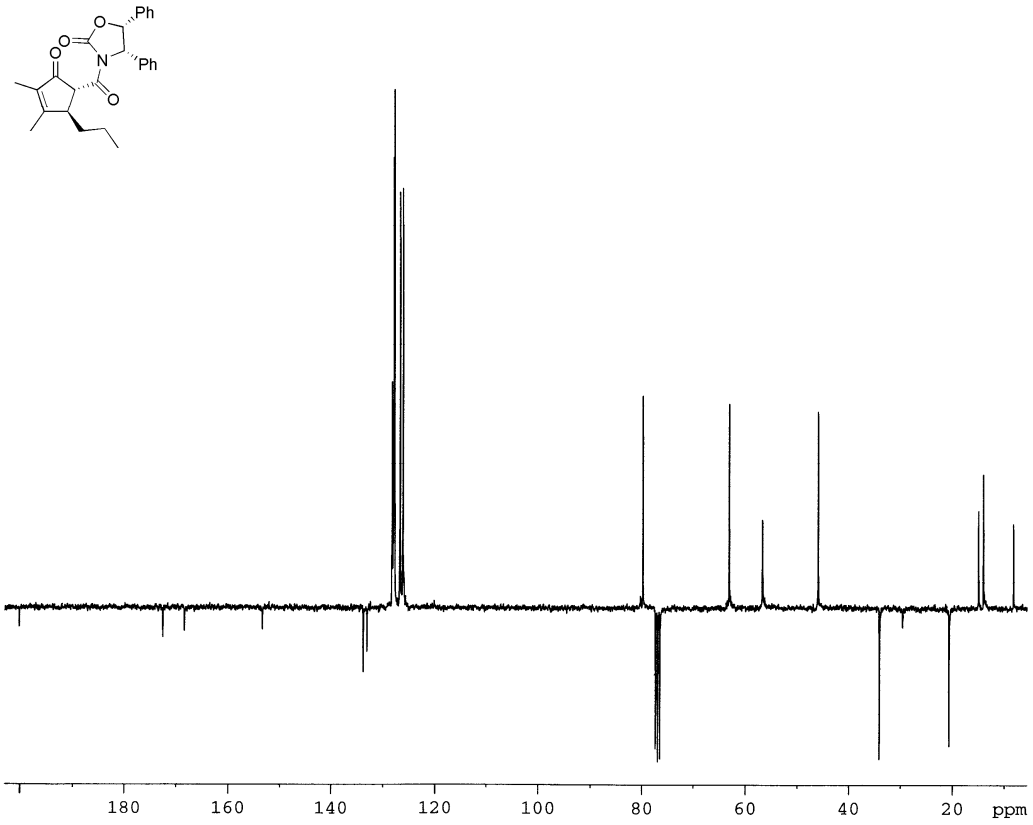
(4*S*,5*R*)-3-[(1*R*,5*R*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-4,5-diphenyloxazolidin-2-one (31cA)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO             10
PROCNO             1

F2 - Acquisition Parameters
Date_       20070212
Time           18.40
INSTRUM         spect
PROBHD  5 mm Multinucl
PULPROG           zg
TD             16384
SOLVENT        CDCl3
NS                16
DS                 0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG              90.5
DW            111.200 usec
DE              6.00 usec
TE             303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1              1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0               1
TD              512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.2300171 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00

F1 - Processing parameters
SI              512
MC2            TPPI
SF         300.1300000 MHz
WDW               no
SSB                2
LB              0.30 Hz
```

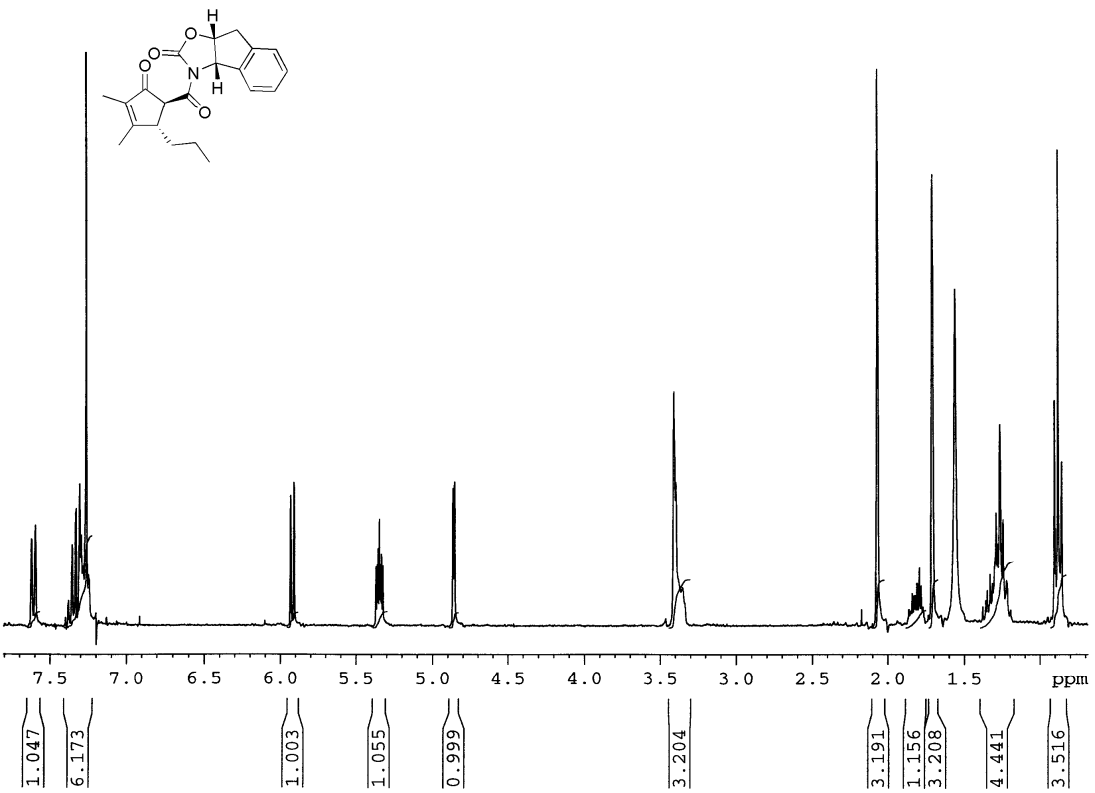
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050530
Time                  18.51
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                   13314
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1824.6
DW                  24.600 usec
DE                  20.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                3.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1          75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2          300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

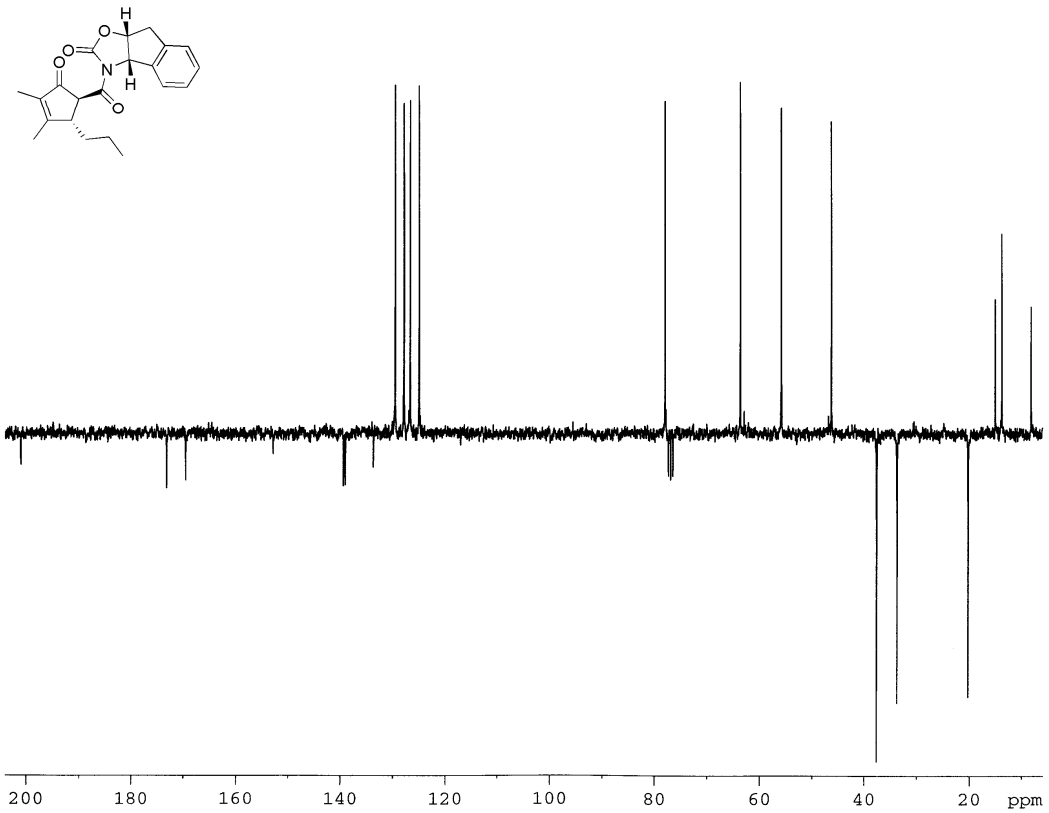
(3a*S*,8a*R*)-3-[(1*S*,5*S*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-3,3a,8,8a-tetrahydro-2*H*-indeno[1,2-*d*]oxazol-2-one (31dC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_       20050420
Time           16.30
INSTRUM         spect
PROBHD  5 mm Multinucl
PULPROG           zg
TD             16384
SOLVENT        CDCl3
NS                16
DS                 0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG              456.1
DW            111.200 usec
DE              6.00 usec
TE             303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW          11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI             16384
SF        300.1300051 MHz
WDW              EM
SSB               0
LB             0.30 Hz
GB                0
PC             2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF        300.1300000 MHz
WDW              no
SSB               2
LB             0.30 Hz
```

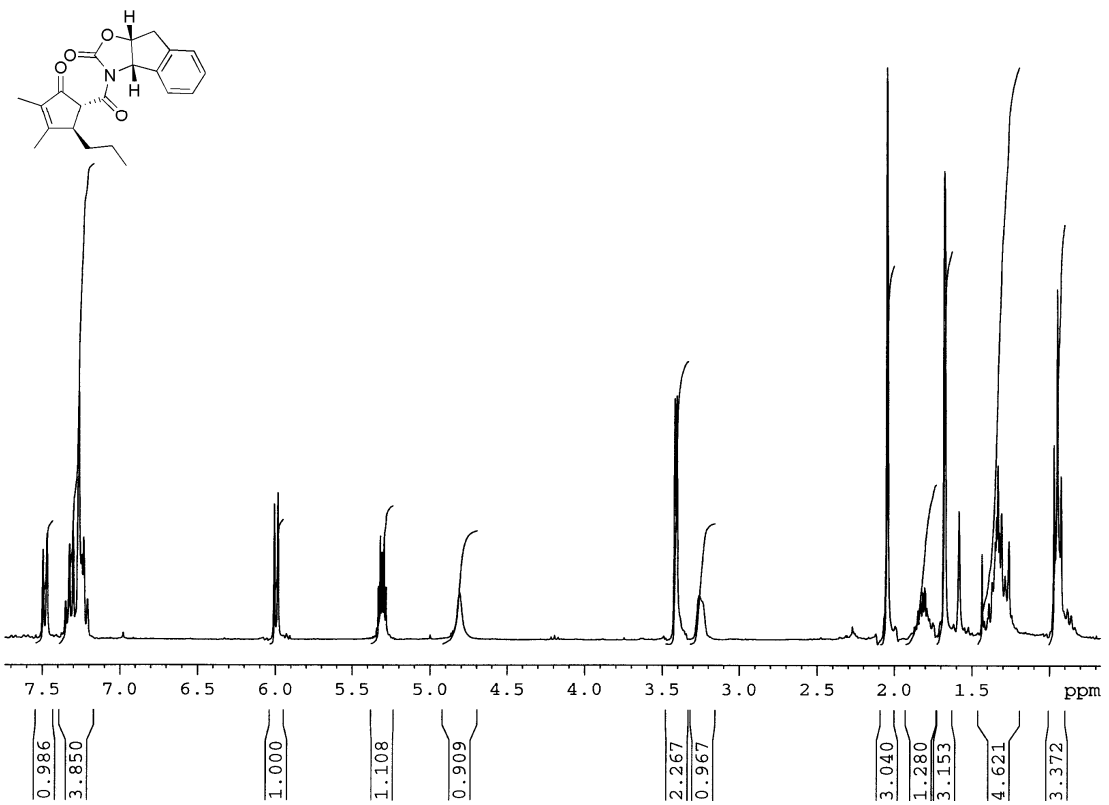
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     6
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050509
Time                  16.18
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     372
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              2.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2          300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF             75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

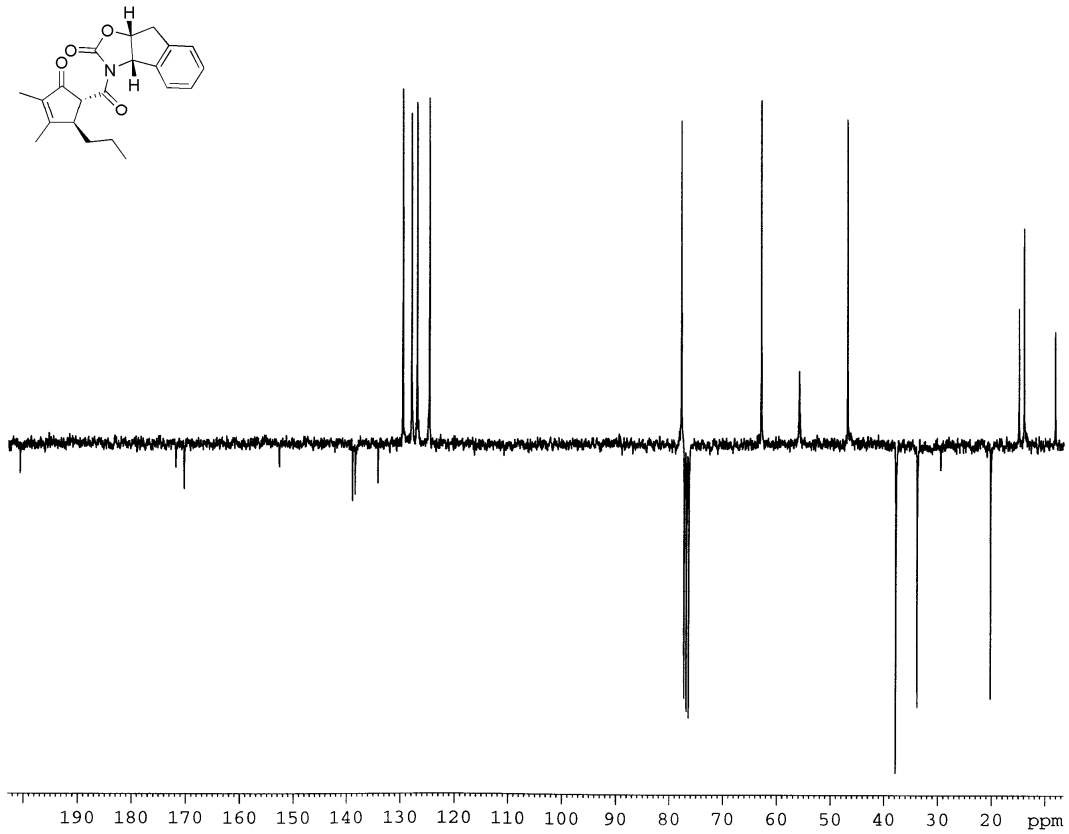
(3a*S*,8a*R*)-3-[(1*R*,5*R*)-3,4-Dimethyl-2-oxo-5-propylcyclopent-3-enecarbonyl]-3,3a,8,8a-tetrahydro-2*H*-indeno[1,2-*d*]oxazol-2-one (31dA)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_         20050509
Time             11.23
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                90.5
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW          11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.1300051 MHz
WDW              EM
SSB               0
LB             0.30 Hz
GB                0
PC             2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW              no
SSB               2
LB             0.30 Hz
```

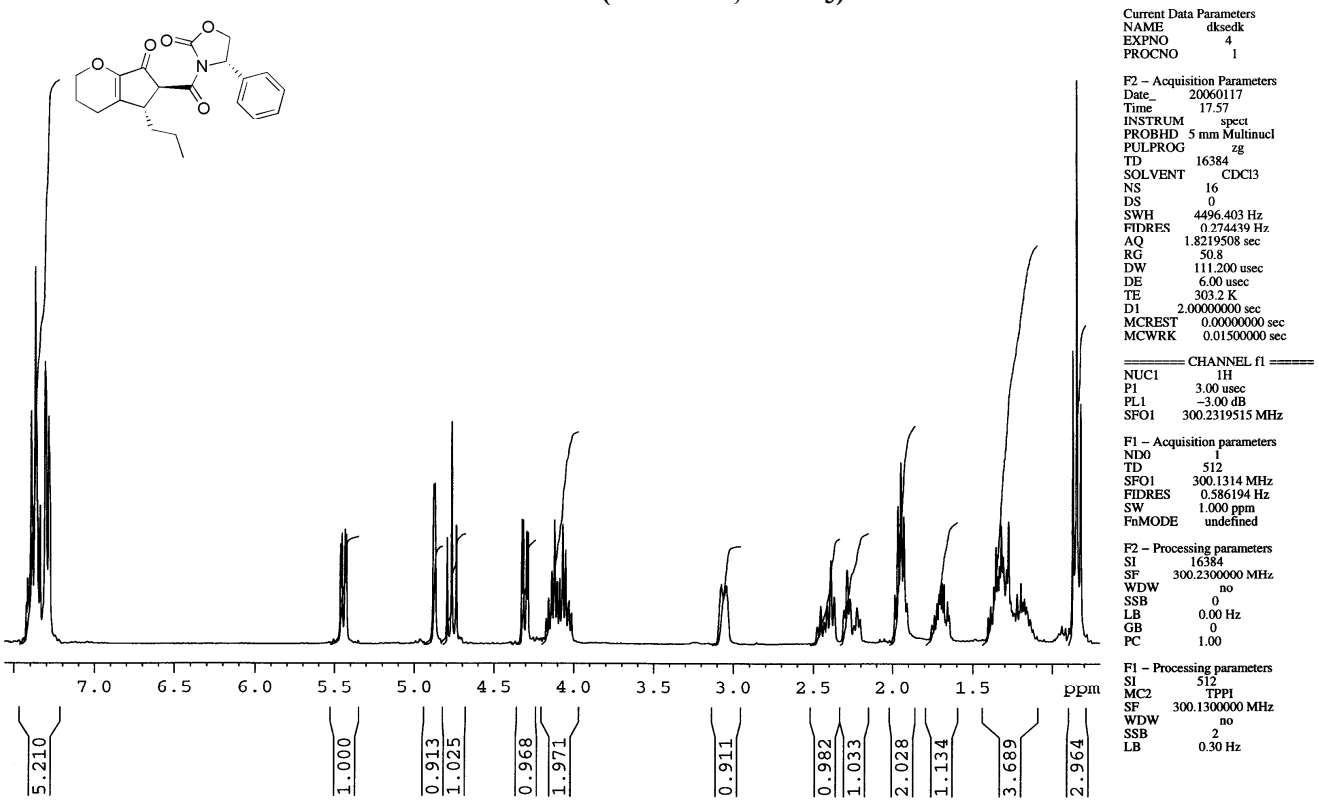
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



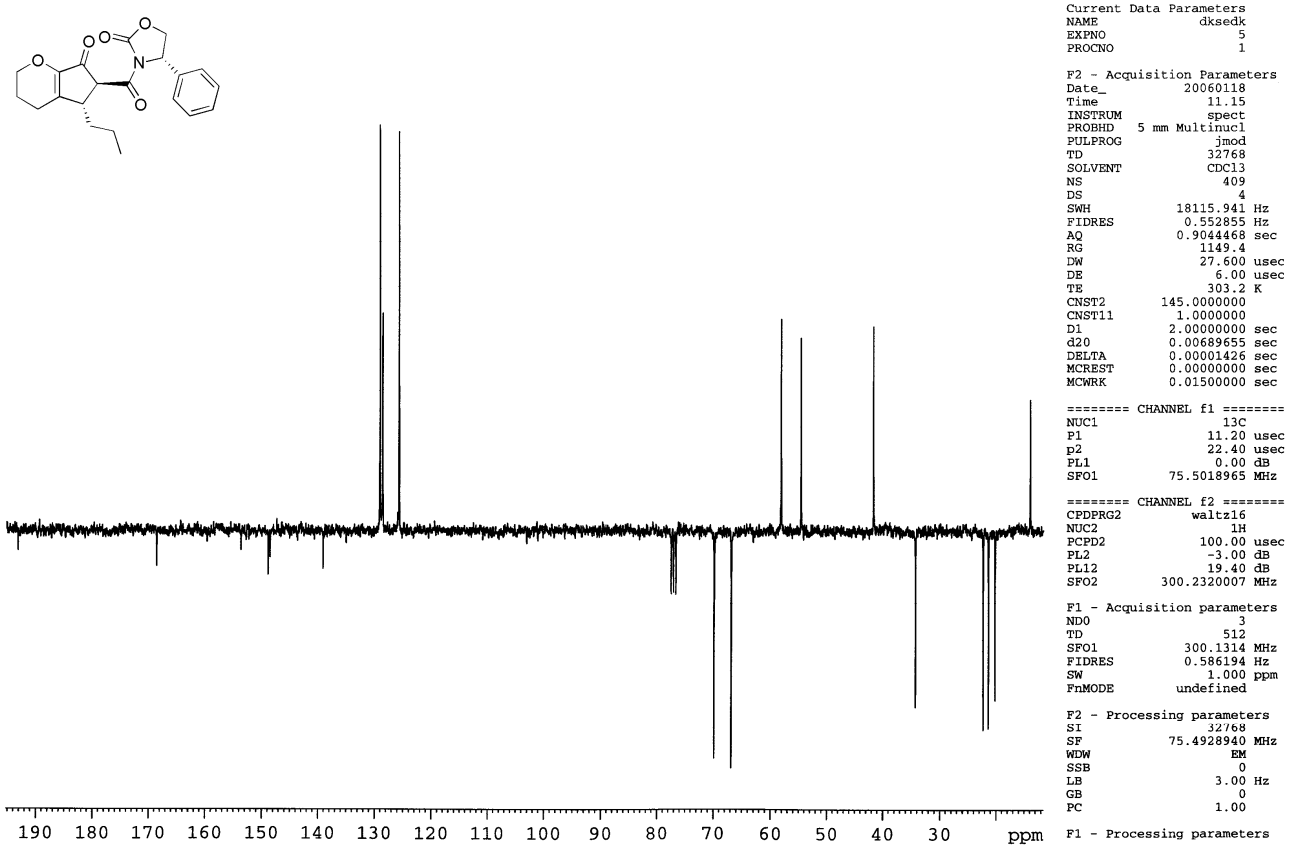

```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20050609
Time                 18.43
INSTRUM              spect
PROBHD   5 mm Multinucl
PULPROG              jmod
TD                  32768
SOLVENT             CDCl3
NS                  13405
DS                      4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1149.4
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              3.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               19.40 dB
SFO2          300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1            300.1314 MHz
FIDRES            7.015535 Hz
SW                   11.968 ppm
FnMODE            undefined

F2 - Processing parameters
SI                     32768
SF           75.4677556 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00

F1 - Processing parameters
```

(*S*)-3-[(5*S*,6*S*)-7-Oxo-5-propyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31eC)

^{1}H NMR (300 MHz, $CDCl_3$)

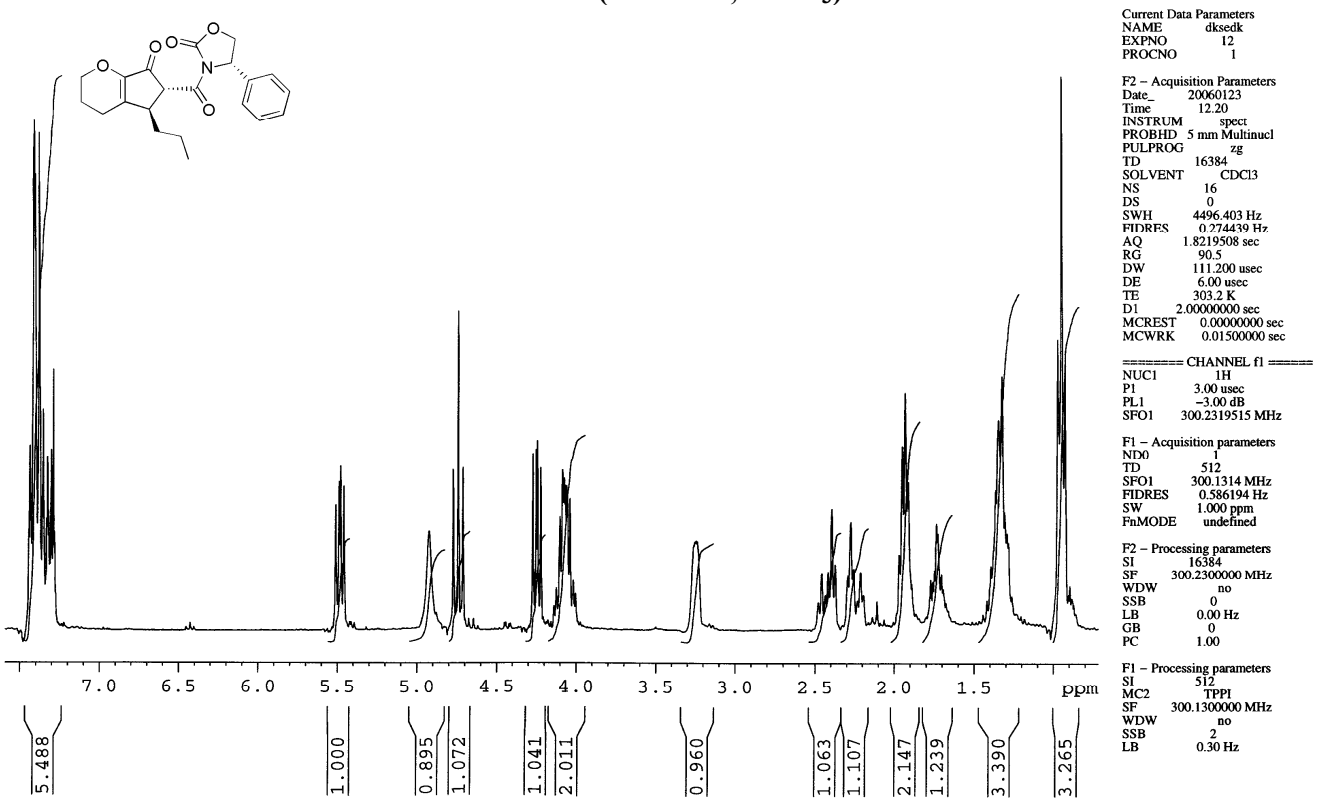


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

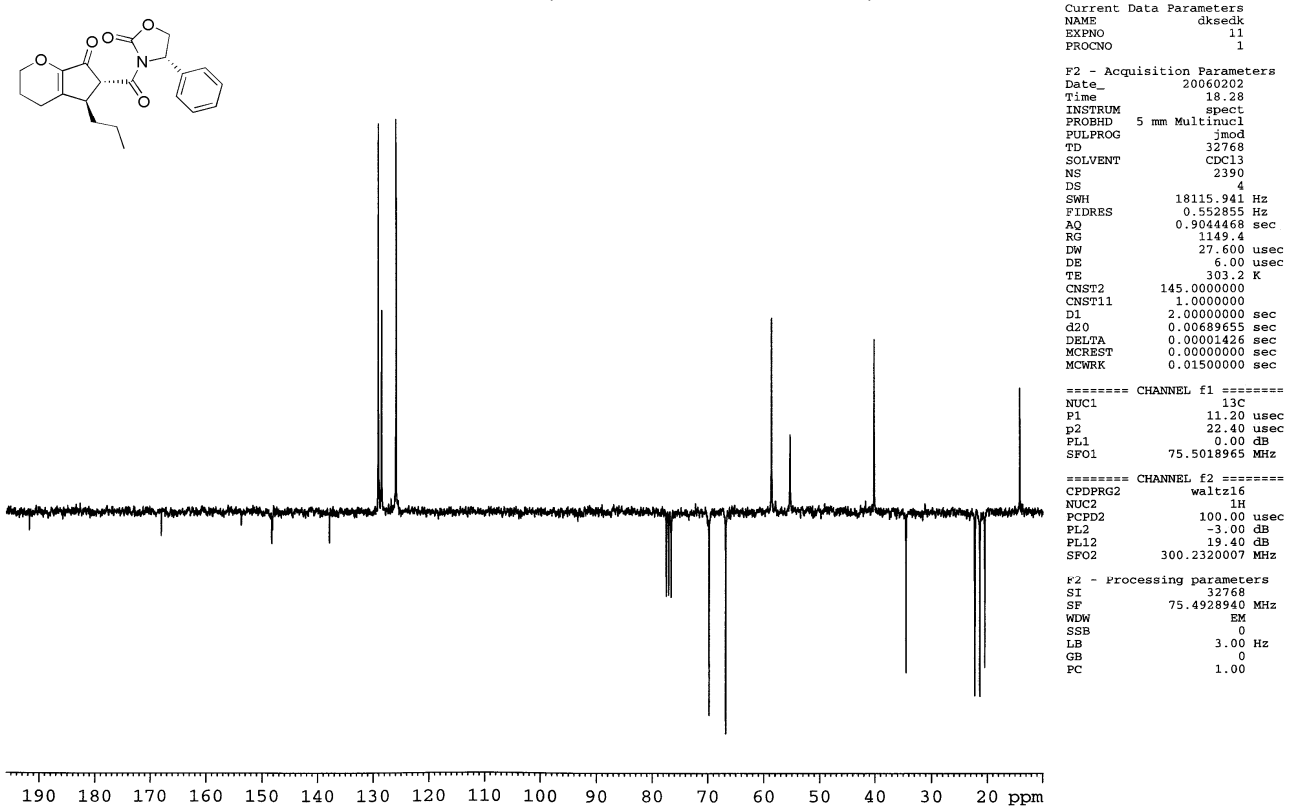


(*S*)-3-[(5*R*,6*R*)-7-Oxo-5-propyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31eA)

^{1}H NMR (300 MHz, $CDCl_3$)

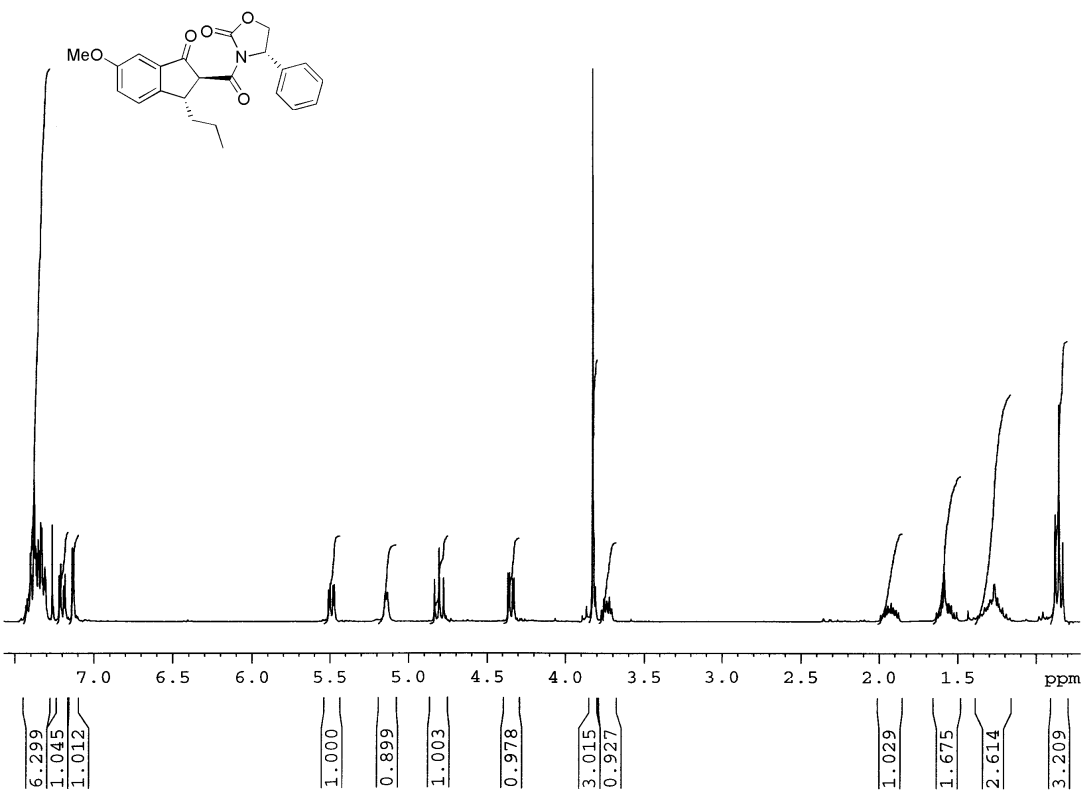


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



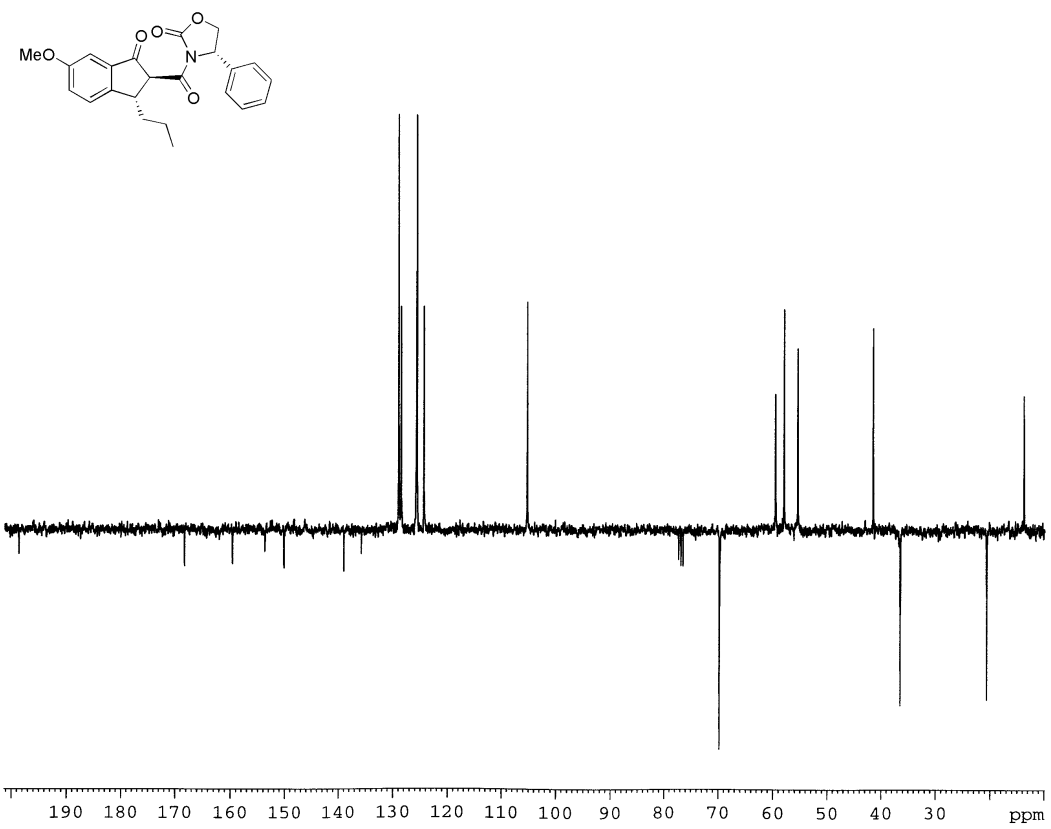
(*S*)-3-[(1*S*,2*S*)-5-Methoxy-3-oxo-1-propyl-2,3-dihydro-1*H*-indene-2-carbonyl]-4-phenyloxazolidin-2-one (31fC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME            dksedk
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_         20050601
Time             12.19
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG             zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           4496.403 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                161.3
DW              111.200 usec
DE                6.00 usec
TE                303.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ======
NUC1                 1H
P1                3.00 usec
PL1               -3.00 dB
SFO1        300.1318320 MHz

F1 - Acquisition parameters
ND0                  2
TD                 512
SFO1        300.1314 MHz
FIDRES        7.015535 Hz
SW             11.968 ppm
FnMODE        undefined

F2 - Processing parameters
SI                16384
SF          300.1300051 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                2.00

F1 - Processing parameters
SI                 512
MC2               TPPI
SF          300.1300000 MHz
WDW                 no
SSB                  2
LB                0.30 Hz
```

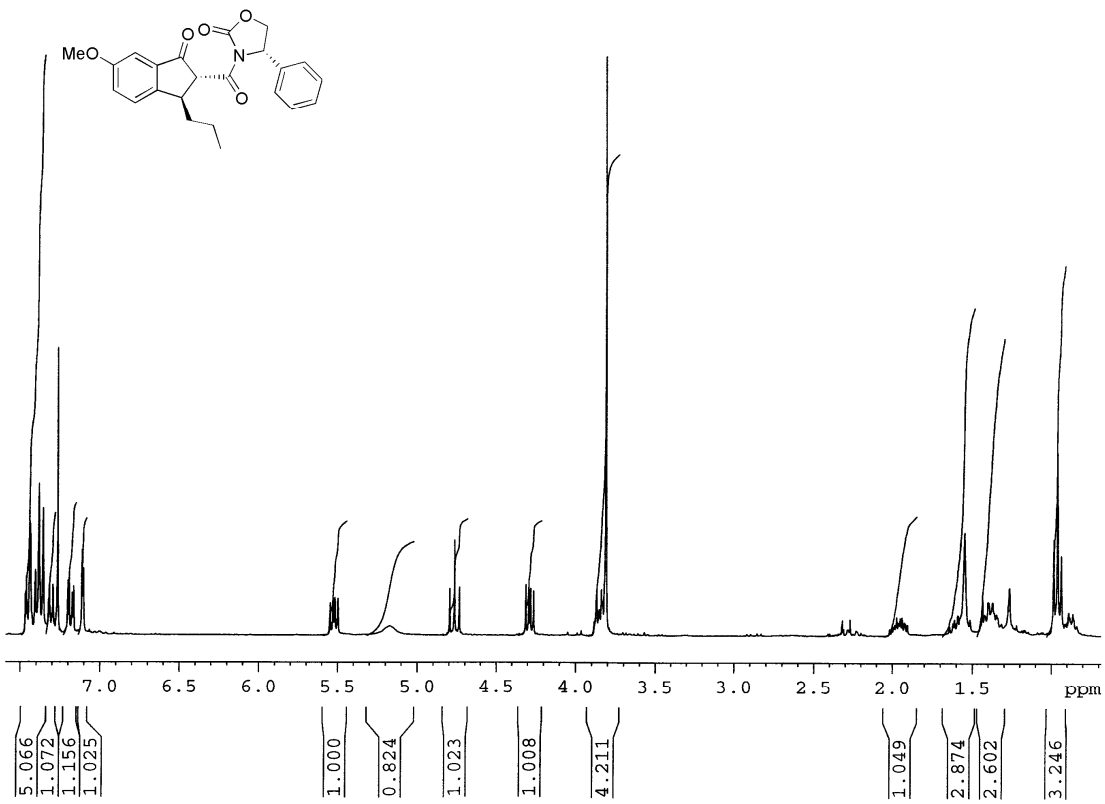
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                dksedk
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20050601
Time                 15.53
INSTRUM              spect
PROBHD    5 mm Multinucl
PULPROG              jmod
TD                  32768
SOLVENT             CDCl3
NS                    266
DS                      4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              2.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2           300.1320000 MHz

F1 - Acquisition parameters
ND0                      2
TD                     512
SFO1            300.1314 MHz
FIDRES            7.015535 Hz
SW                 11.968 ppm
FnMODE           undefined

F2 - Processing parameters
SI                    32768
SF              75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00

F1 - Processing parameters
```

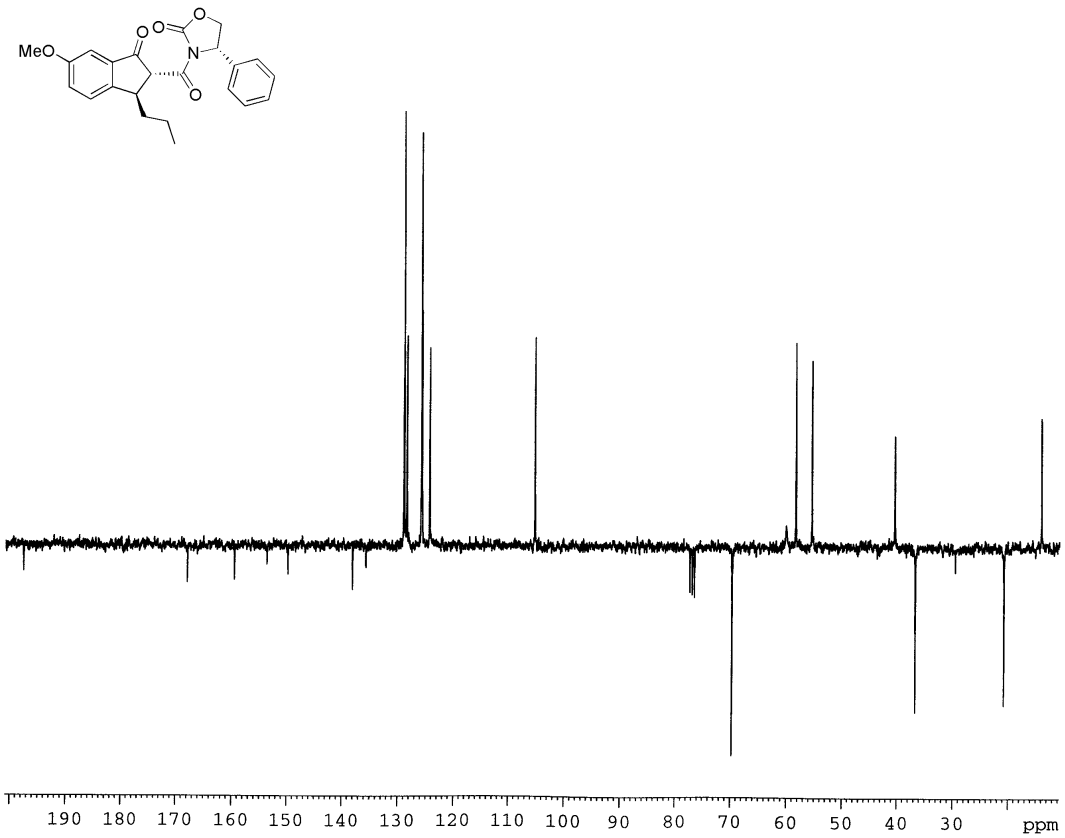
(*S*)-3-[(1*R*,2*R*)-5-Methoxy-3-oxo-1-propyl-2,3-dihydro-1*H*-indene-2-carbonyl]-4-phenyloxazolidin-2-one (31fA)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20050601
Time             12.10
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ         1.8219508 sec
RG               322.5
DW           111.200 usec
DE             6.00 usec
TE             303.2 K
D1         3.00000000 sec
MCREST     0.00000000 sec
MCWRK      0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW                EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW              no
SSB                2
LB              0.30 Hz
```

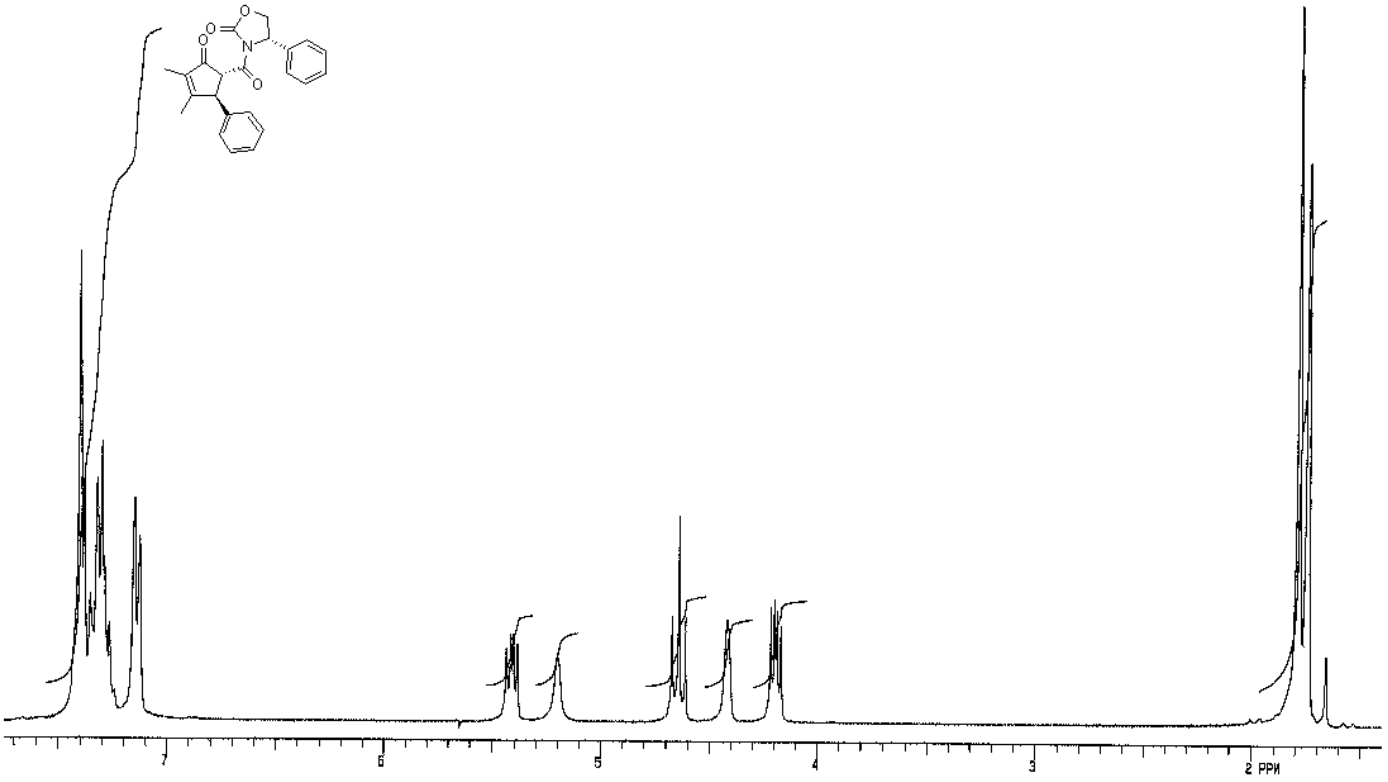
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



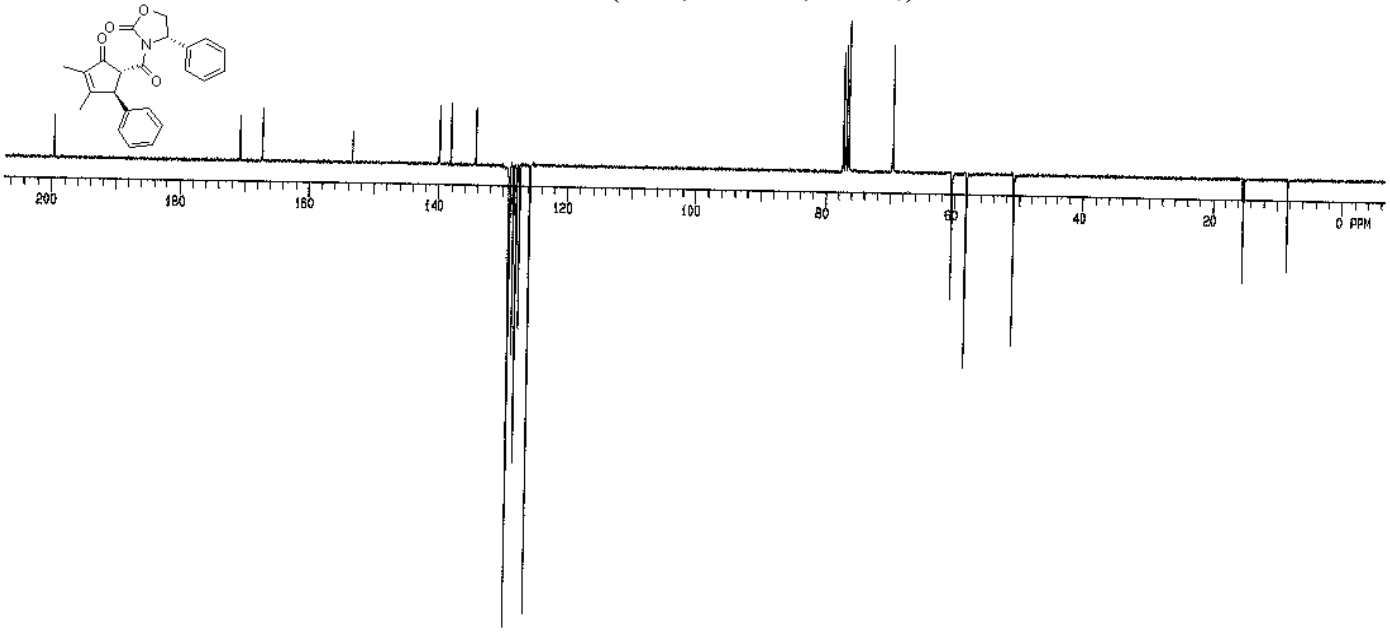

```
Current Data Parameters
NAME               dksedk
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20050602
Time                10.23
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG              jmod
TD                  32768
SOLVENT             CDCl3
NS                    700
DS                      4
SWH             20325.203 Hz
FIDRES           0.620276 Hz
AQ               0.8061428 sec
RG                  574.7
DW                 24.600 usec
DE                 20.00 usec
TE                 303.2 K
CNST2          145.0000000
CNST11           1.0000000
D1               2.00000000 sec
d20              0.00689655 sec
DELTA            0.00001426 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2          300.1320000 MHz

F2 - Processing parameters
SI                  32768
SF             75.4677556 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

(*S*)-3-[(1*R*,5*S*)-3,4-Dimethyl-2-oxo-5-phenylcyclopent-3-enecarbonyl]-4-phenyloxazolidin-2-one (31gA)

^{1}H NMR (300 MHz, $CDCl_3$)

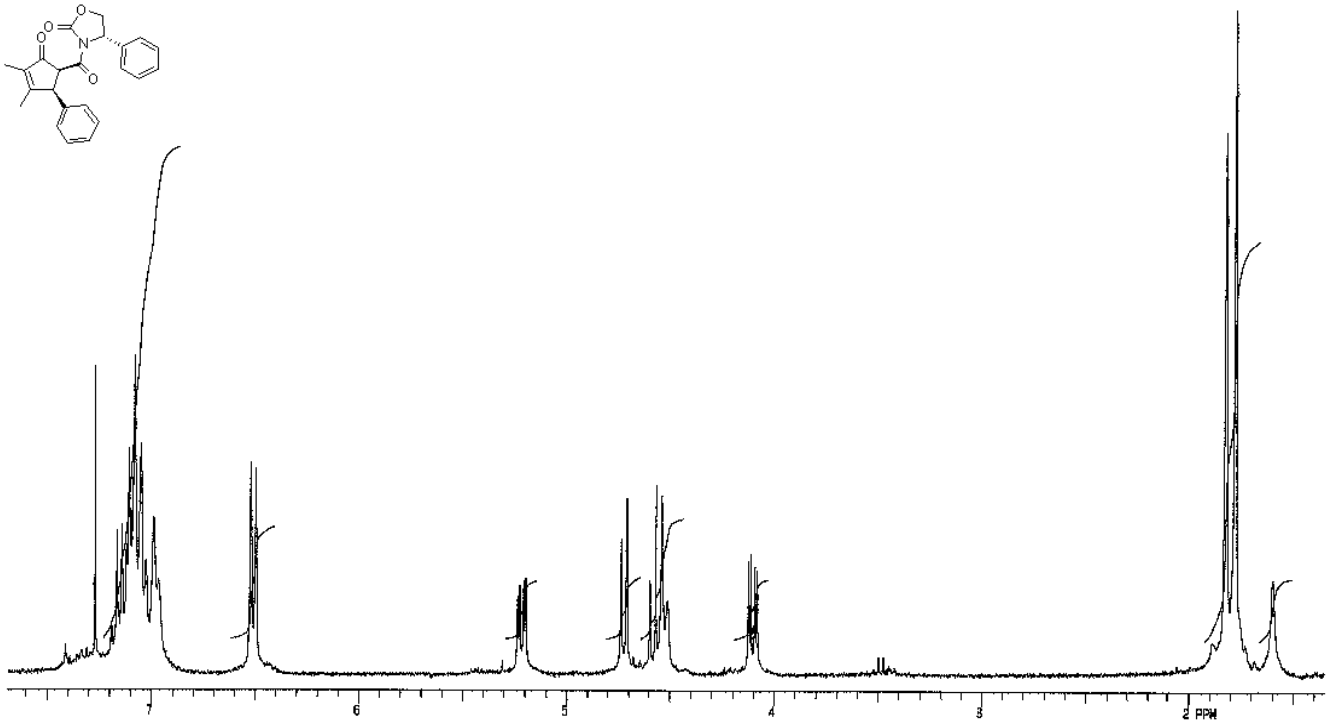


^{13}C NMR (APT, 75 MHz, $CDCl_3$)

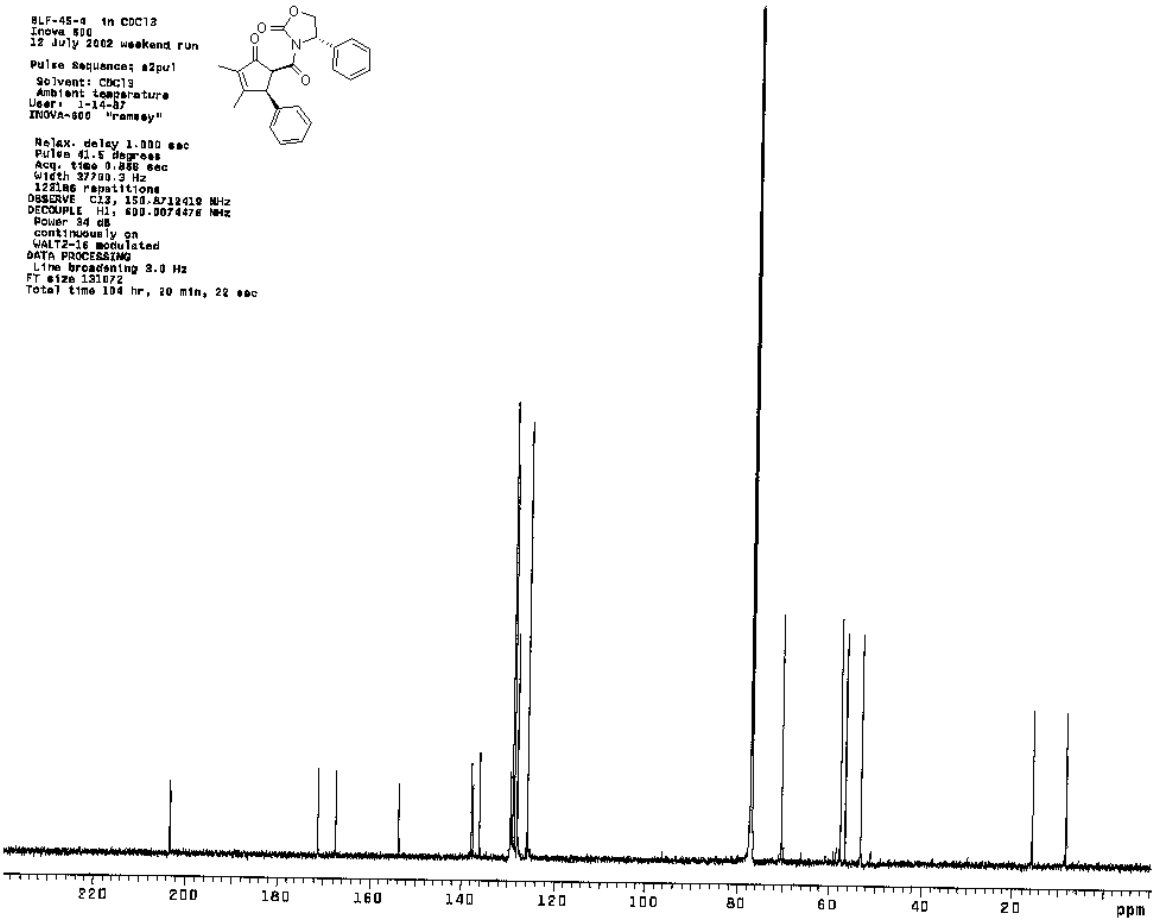


(*S*)-3-[(1*S*,5*S*)-3,4-Dimethyl-2-oxo-5-phenylcyclopent-3-enecarbonyl]-4-phenyloxazolidin-2-one (31gB)

^{1}H NMR (300 MHz, $CDCl_3$)

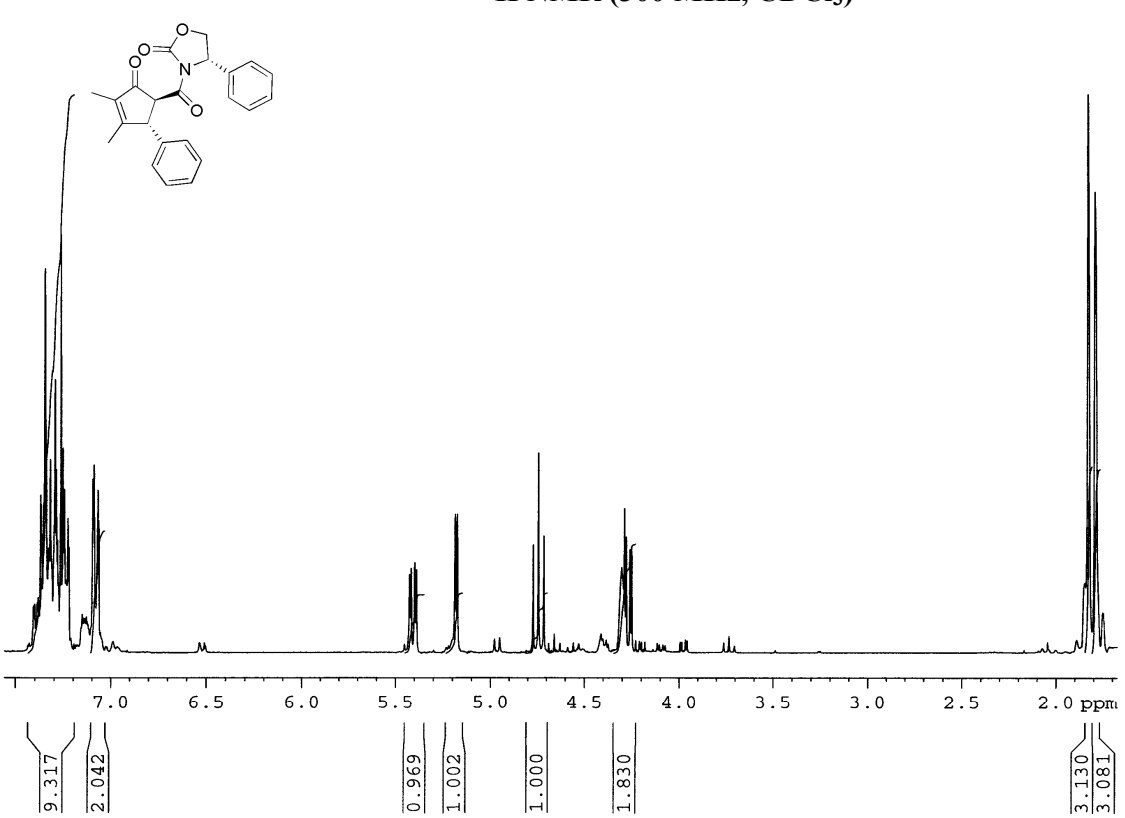


^{13}C NMR (150 MHz, $CDCl_3$)



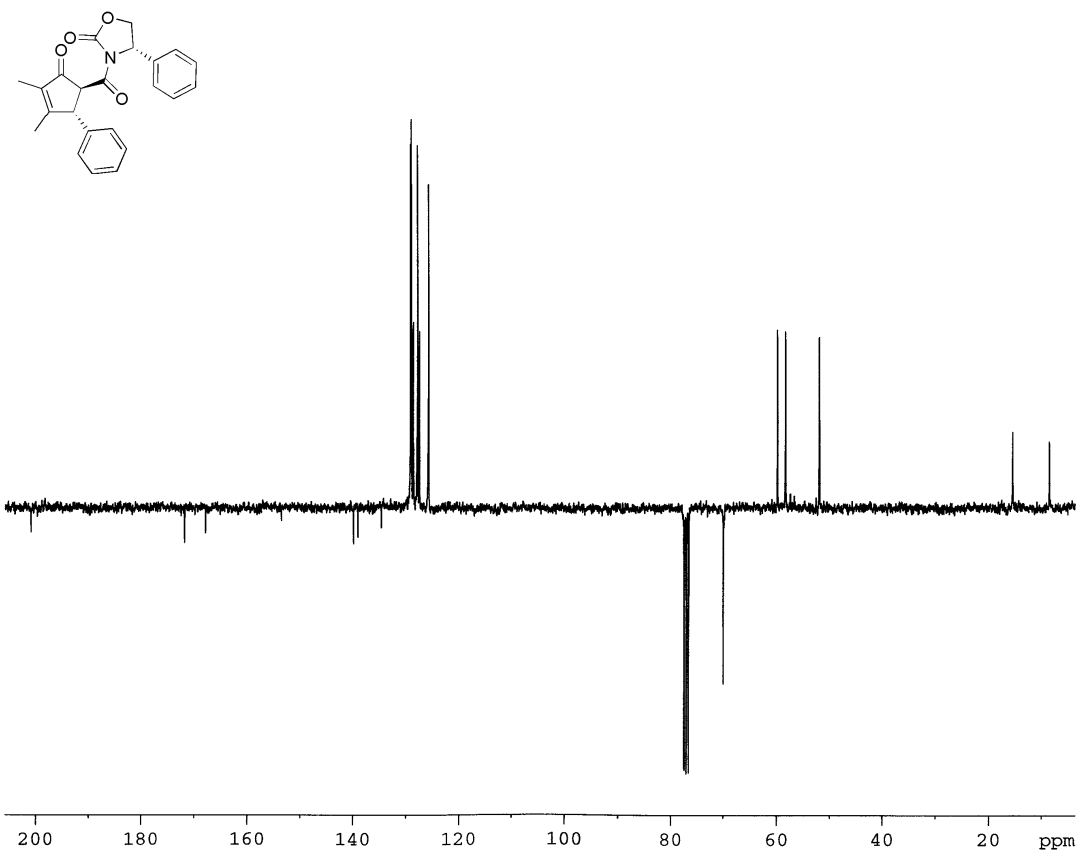
(*S*)-3-[(1*S*,5*R*)-3,4-Dimethyl-2-oxo-5-phenylcyclopent-3-enecarbonyl]-4-phenyloxazolidin-2-one (31gC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO             13
PROCNO             1

F2 - Acquisition Parameters
Date_       20050721
Time           10.30
INSTRUM         spect
PROBHD  5 mm Multinucl
PULPROG           zg
TD             16384
SOLVENT        CDCl3
NS                16
DS                 0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG               181
DW           111.200 usec
DE             6.00 usec
TE            303.2 K
D1        3.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

======== CHANNEL f1 ======
NUC1              1H
P1              3.00 usec
PL1             -3.00 dB
SFO1      300.1318320 MHz

F1 - Acquisition parameters
ND0               2
TD              512
SFO1      300.1314 MHz
FIDRES      7.015535 Hz
SW         11.968 ppm
FnMODE      undefined

F2 - Processing parameters
SI             16384
SF        300.1300051 MHz
WDW              EM
SSB               0
LB             0.30 Hz
GB                0
PC             2.00

F1 - Processing parameters
SI              512
MC2            TPPI
SF        300.1300000 MHz
WDW            QSINE
SSB               2
LB             0.30 Hz
```

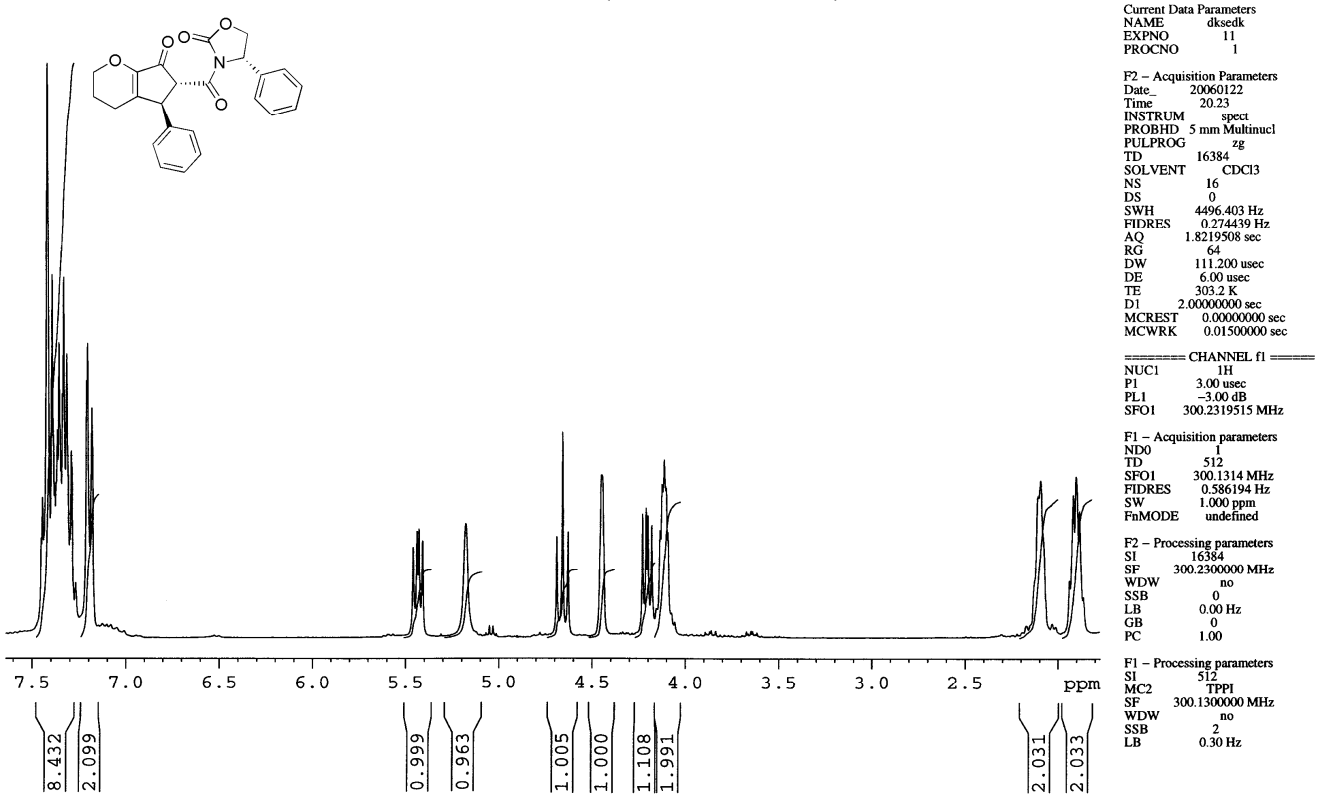
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060202
Time                  20.20
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                   11151
DS                       4
SWH              18115.941 Hz
FIDRES             0.552855 Hz
AQ                0.9044468 sec
RG                   1149.4
DW                  27.600 usec
DE                    6.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1             3.00000000 sec
d20            0.00689655 sec
DELTA          0.00001426 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1           75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2          300.2320007 MHz

F2 - Processing parameters
SI                   32768
SF             75.4928940 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

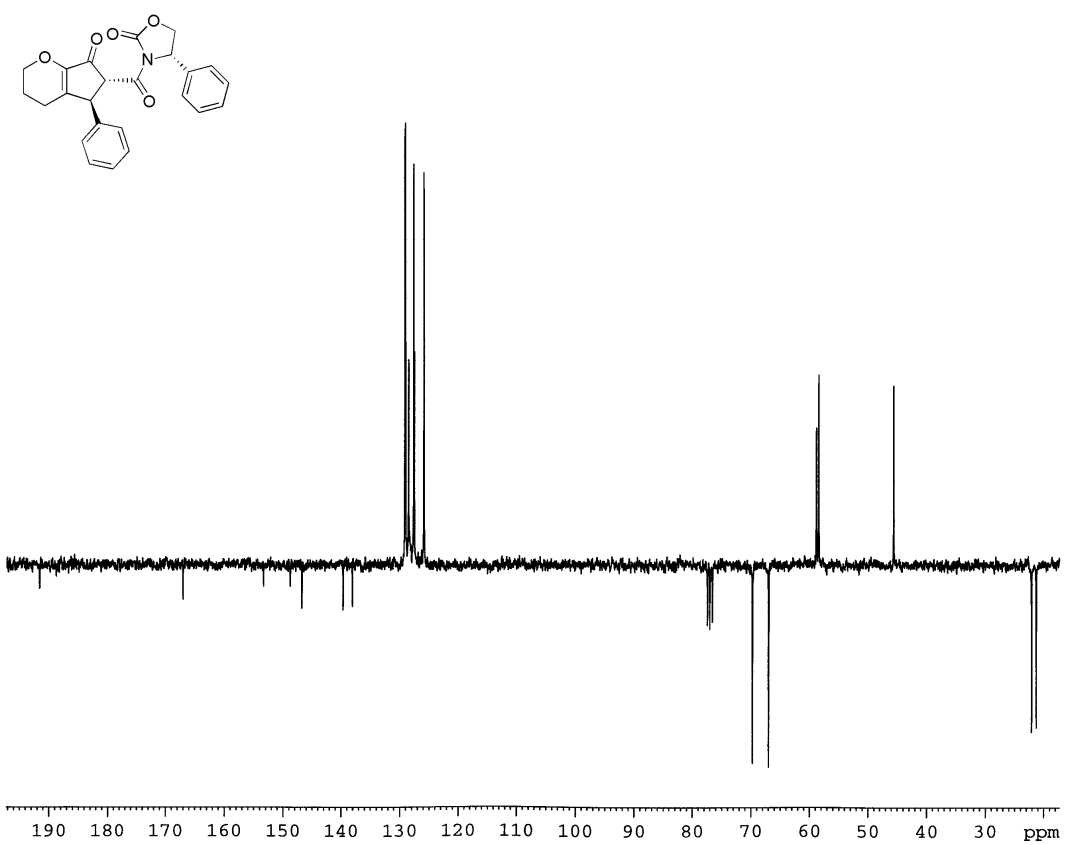
(*S*)-3-[(5*S*,6*R*)-7-Oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31hA)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME            dksedk
EXPNO               11
PROCNO               1

F2 - Acquisition Parameters
Date_         20060122
Time             20.23
INSTRUM          spect
PROBHD   5 mm Multinucl
PULPROG             zg
TD                16384
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            4496.403 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                   64
DW               111.200 usec
DE                 6.00 usec
TE                303.2 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ======
NUC1                 1H
P1                3.00 usec
PL1              -3.00 dB
SFO1         300.2319515 MHz

F1 - Acquisition parameters
ND0                  1
TD                 512
SFO1         300.1314 MHz
FIDRES         0.586194 Hz
SW             1.000 ppm
FnMODE         undefined

F2 - Processing parameters
SI                16384
SF         300.2300000 MHz
WDW                  no
SSB                   0
LB             0.00 Hz
GB                    0
PC             1.00

F1 - Processing parameters
SI                 512
MC2               TPPI
SF         300.1300000 MHz
WDW                  no
SSB                   2
LB             0.30 Hz
```

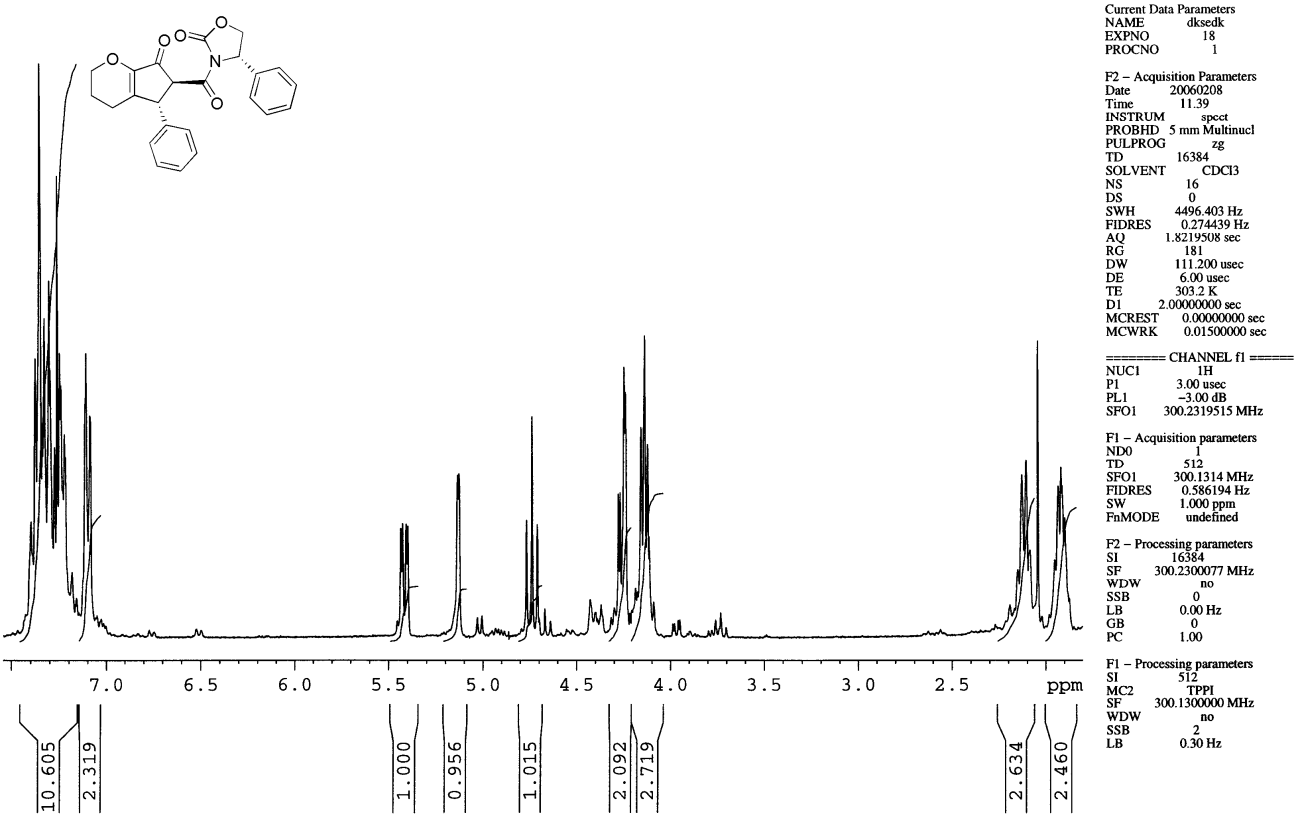
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                    11
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060122
Time                  20.33
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                      554
DS                        4
SWH               18115.941 Hz
FIDRES             0.552855 Hz
AQ                 0.9044468 sec
RG                    1149.4
DW                    27.600 usec
DE                     6.00 usec
TE                    303.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 2.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1              75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2             300.2320007 MHz

F1 - Acquisition parameters
ND0                       3
TD                      512
SFO1              300.1314 MHz
FIDRES             0.586194 Hz
SW                     1.000 ppm
FnMODE             undefined

F2 - Processing parameters
SI                    32768
SF              75.4928940 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```

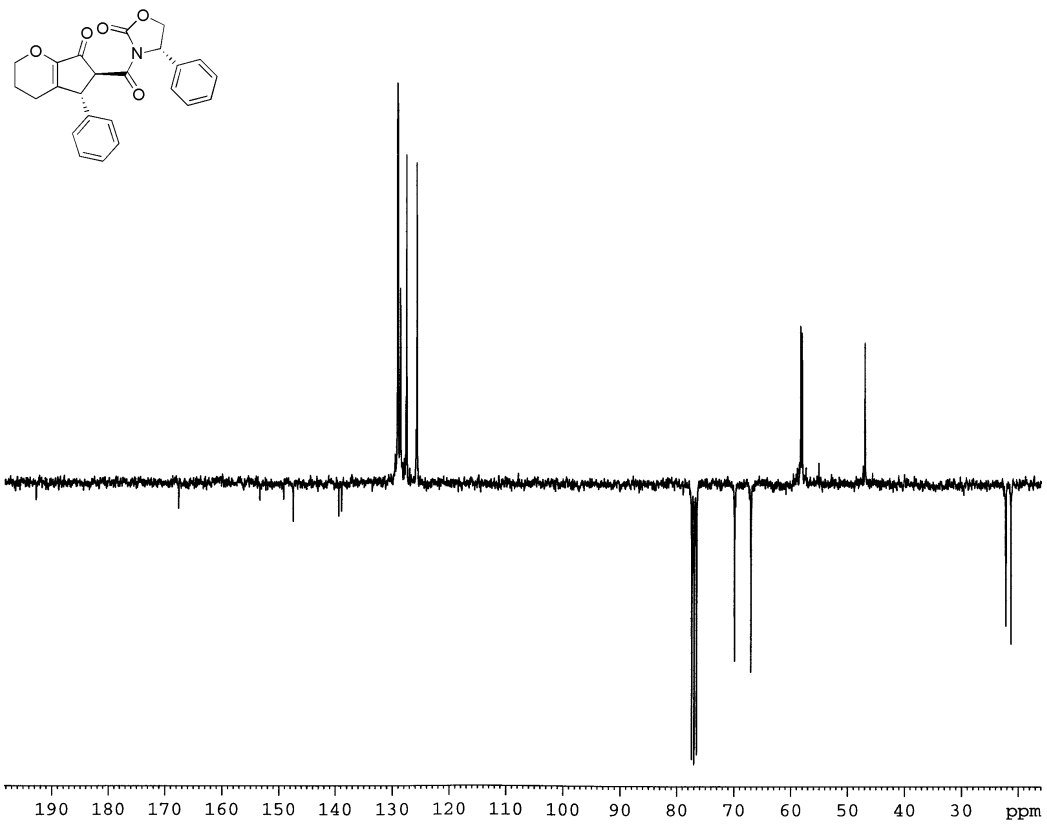
(*S*)-3-[(5*R*,6*S*)-7-Oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31hC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO             18
PROCNO             1

F2 - Acquisition Parameters
Date          20060208
Time             11.39
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                181
DW            111.200 usec
DE              6.00 usec
TE             303.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI               16384
SF         300.2300077 MHz
WDW                no
SSB                 0
LB           0.00 Hz
GB                  0
PC           1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW                no
SSB                 2
LB           0.30 Hz
```

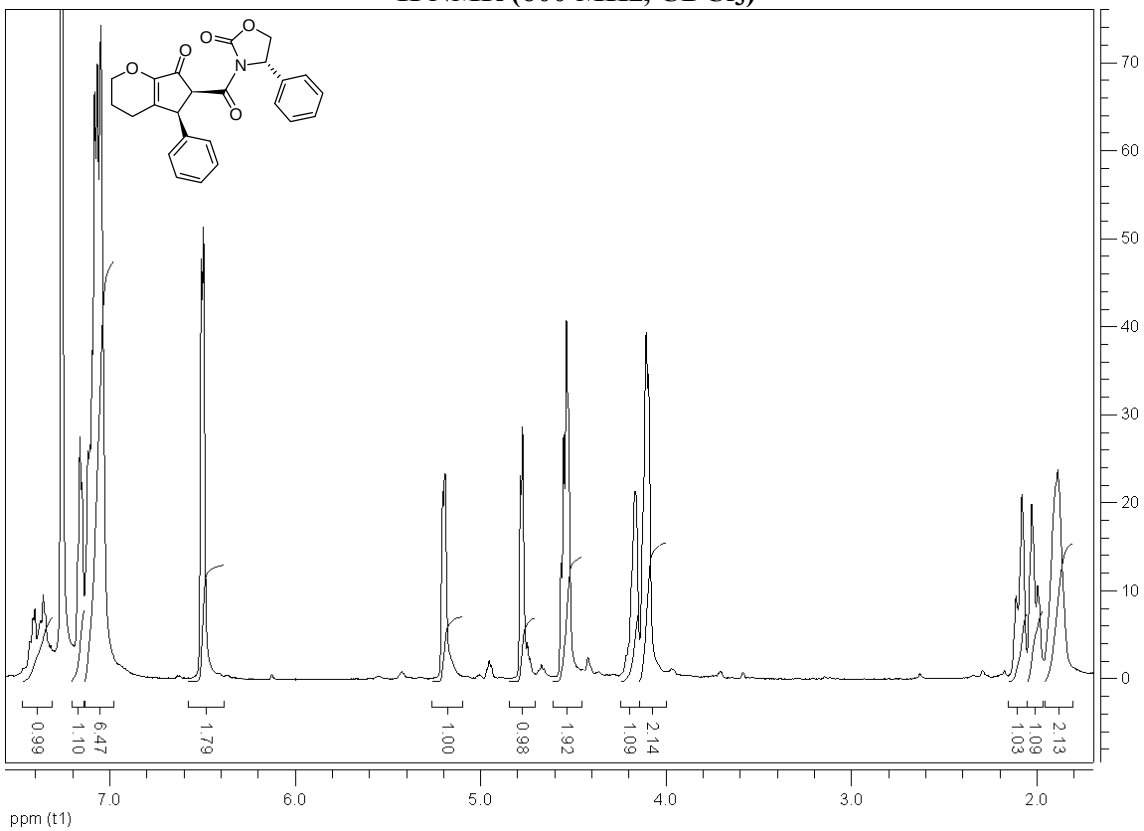
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



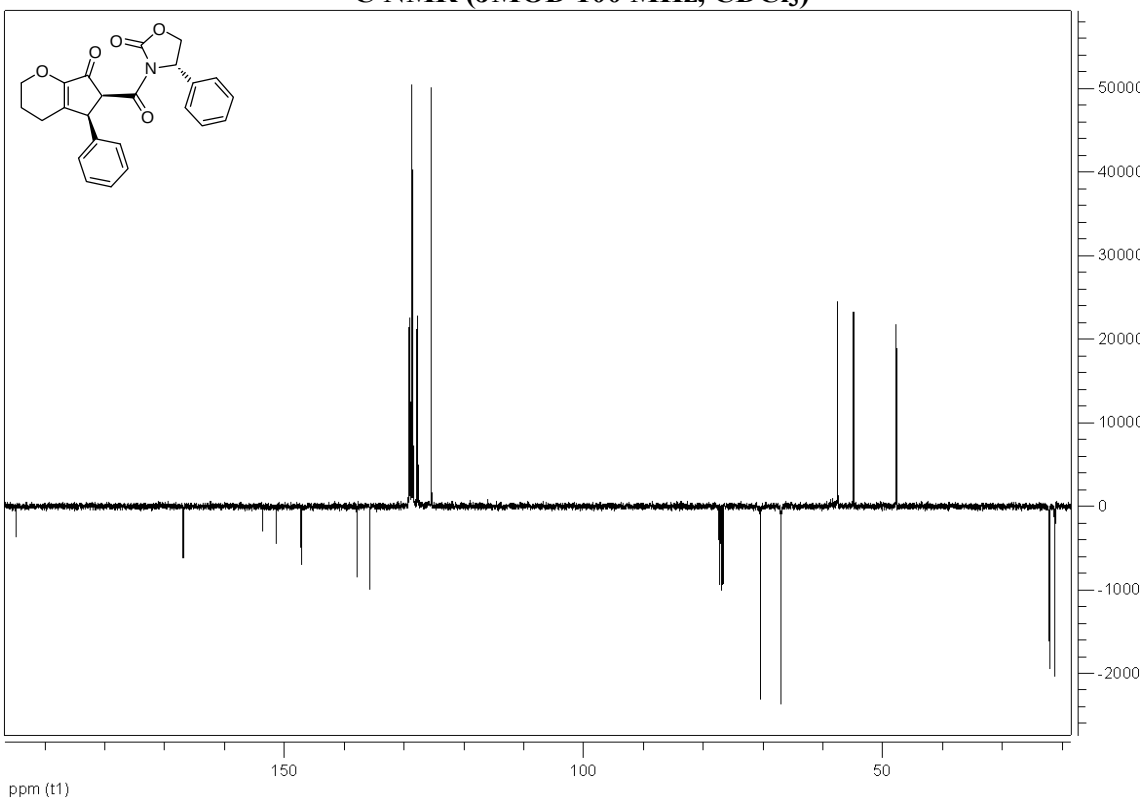

```
Current Data Parameters
NAME                 dksedk
EXPNO                     6
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060215
Time                  17.15
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                   14139
DS                       4
SWH             18115.941 Hz
FIDRES           0.552855 Hz
AQ               0.9044468 sec
RG                  1625.5
DW                 27.600 usec
DE                   6.00 usec
TE                  303.2 K
CNST2          145.0000000
CNST11           1.0000000
D1             3.00000000 sec
d20            0.00689655 sec
DELTA          0.00001426 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.20 usec
p2                 22.40 usec
PL1                 0.00 dB
SFO1          75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               19.40 dB
SFO2          300.2320007 MHz

F1 - Acquisition parameters
ND0                  1
TD                 512
SFO1          300.1314 MHz
FIDRES          0.586194 Hz
SW                1.000 ppm
FnMODE         undefined

F2 - Processing parameters
SI                 32768
SF            75.4928940 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00

F1 - Processing parameters
```

(*S*)-3-[(5*S*,6*S*)-7-Oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carbonyl]-4-phenyloxazolidin-2-one (31hB)

^{1}H NMR (600 MHz, $CDCl_3$)

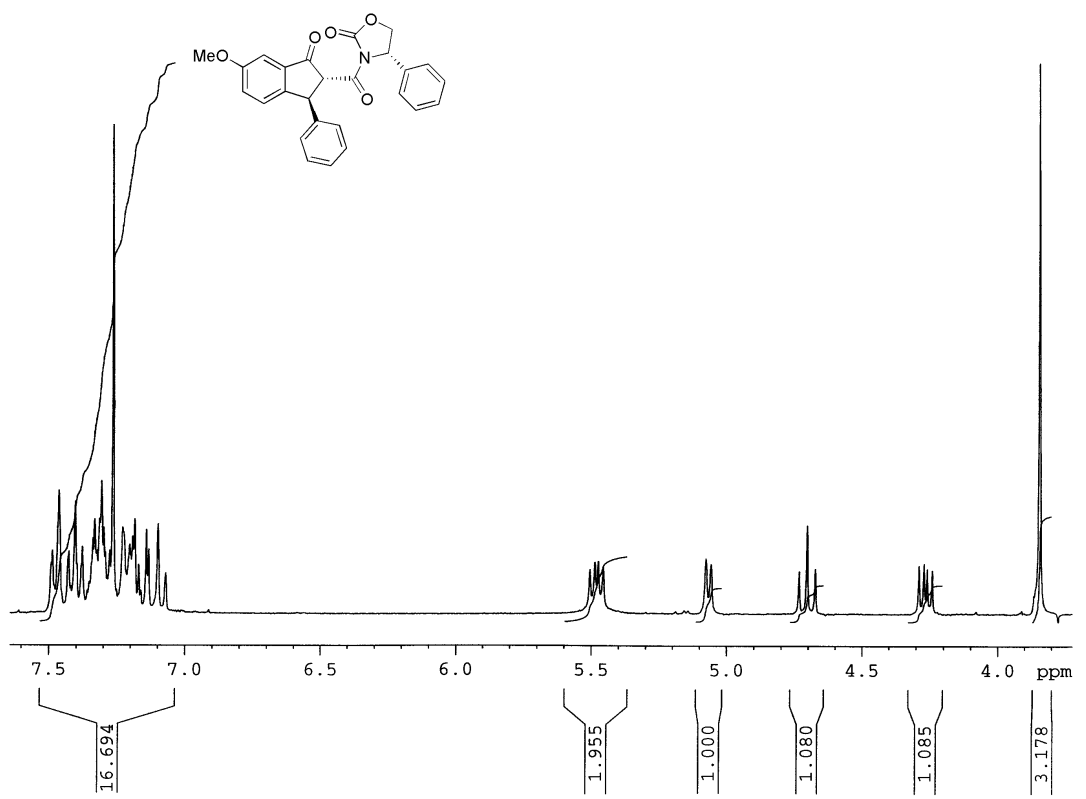


^{13}C NMR (JMOD 100 MHz, $CDCl_3$)



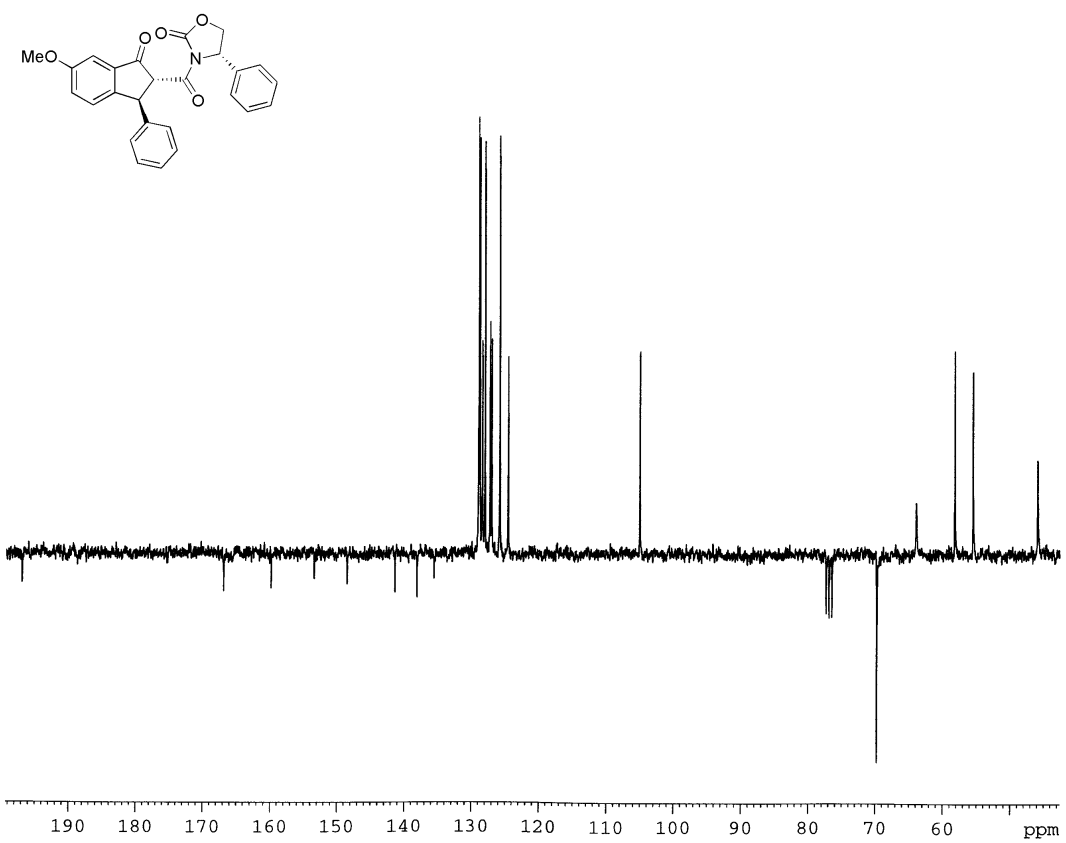
(*S*)-3-[(1*S*,2*R*)-5-Methoxy-3-oxo-1-phenyl-2,3-dihydro-1*H*-indene-2-carbonyl]-4-phenyloxazolidin-2-one (31iA)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_         20050520
Time             12.41
INSTRUM          spect
PROBHD   5 mm Multinucl
PULPROG            zg
TD                16384
SOLVENT          CDCl3
NS                   16
DS                    0
SWH           4496.403 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                  512
DW              111.200 usec
DE                6.00 usec
TE                303.2 K
D1            3.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ======
NUC1                1H
P1               3.00 usec
PL1              -3.00 dB
SFO1        300.1318320 MHz

F1 - Acquisition parameters
ND0                  2
TD                 512
SFO1        300.1314 MHz
FIDRES        7.015535 Hz
SW            11.968 ppm
FnMODE         undefined

F2 - Processing parameters
SI                16384
SF          300.1300051 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                2.00

F1 - Processing parameters
SI                 512
MC2               TPPI
SF          300.1300000 MHz
WDW                 no
SSB                  2
LB                0.30 Hz
```

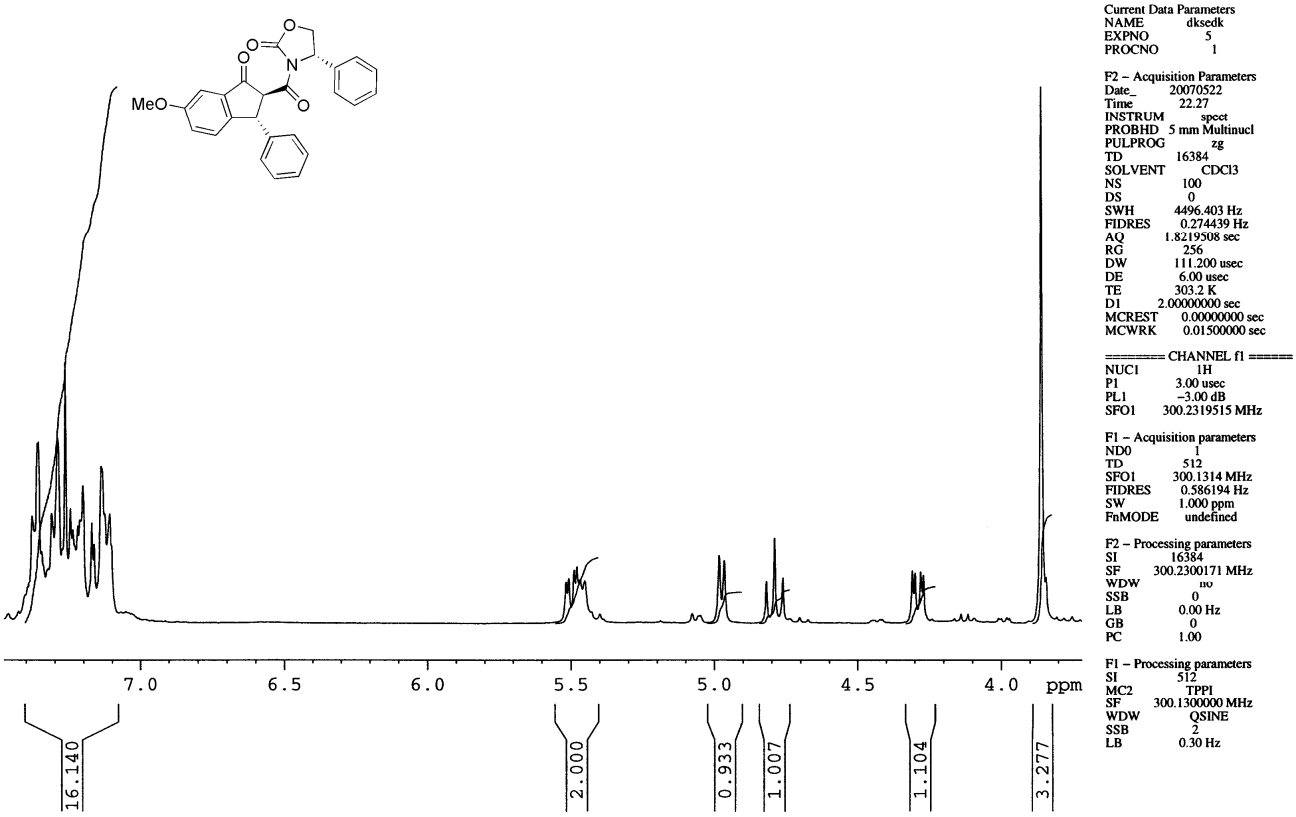
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050525
Time                  15.04
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     574
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1149.4
DW                  24.600 usec
DE                  20.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1                2.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1             75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2            300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1             300.1314 MHz
FIDRES             7.015535 Hz
SW                    11.968 ppm
FnMODE             undefined

F2 - Processing parameters
SI                     32768
SF               75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

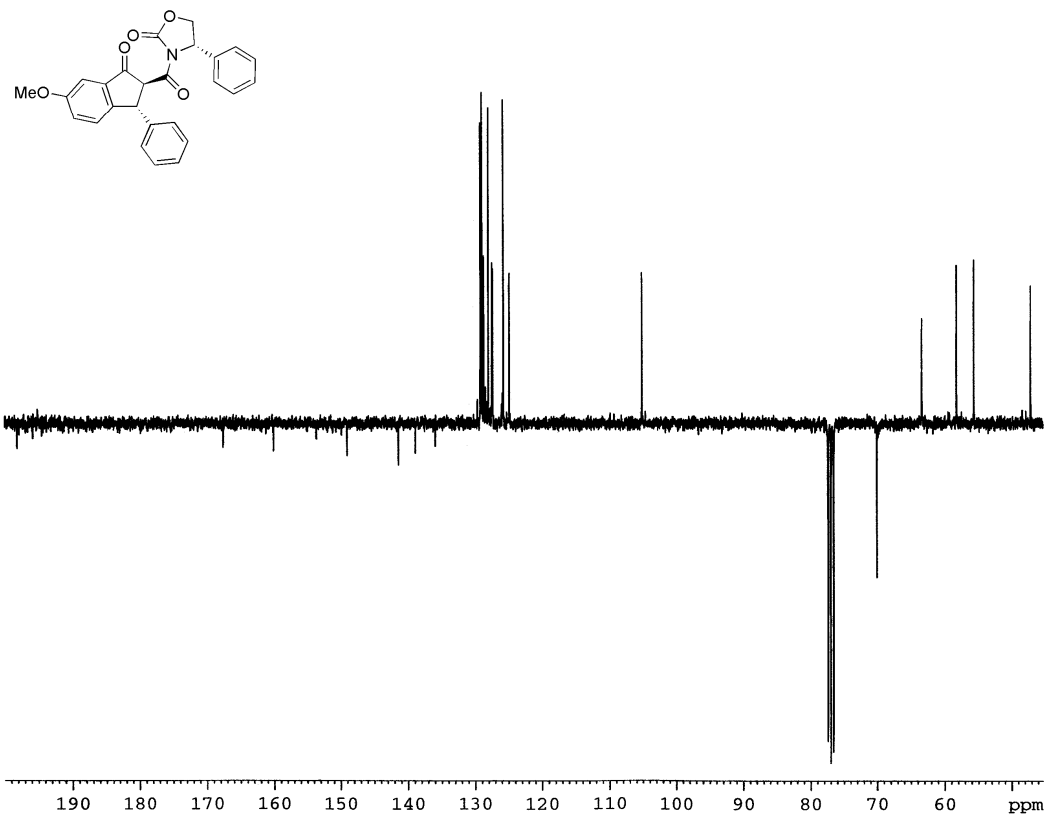
(*S*)-3-[(1*R*,2*S*)-5-Methoxy-3-oxo-1-phenyl-2,3-dihydro-1*H*-indene-2-carbonyl]-4-phenyloxazolidin-2-one (31iC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              5
PROCNO             1

F2 - Acquisition Parameters
Date_        20070522
Time            22.27
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                100
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                256
DW            111.200 usec
DE              6.00 usec
TE             303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                1
TD               512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF       300.2300171 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF       300.1300000 MHz
WDW             QSINE
SSB                2
LB              0.30 Hz
```

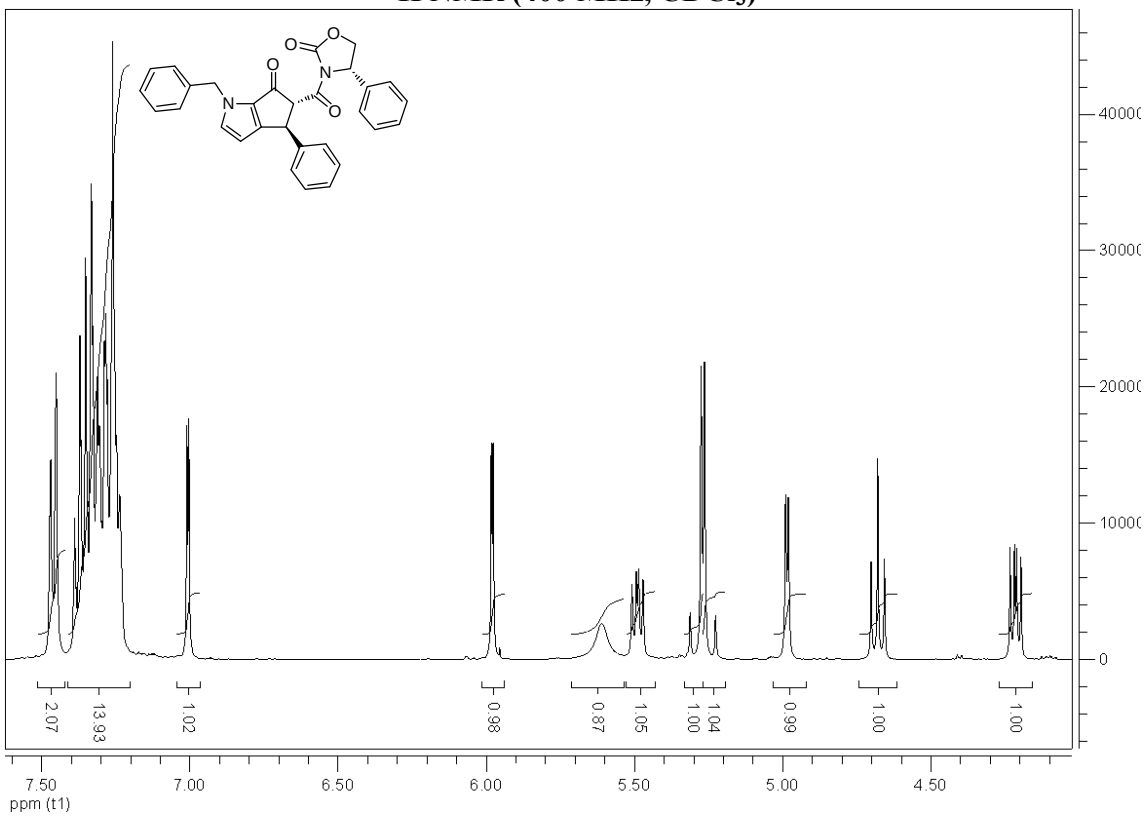
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



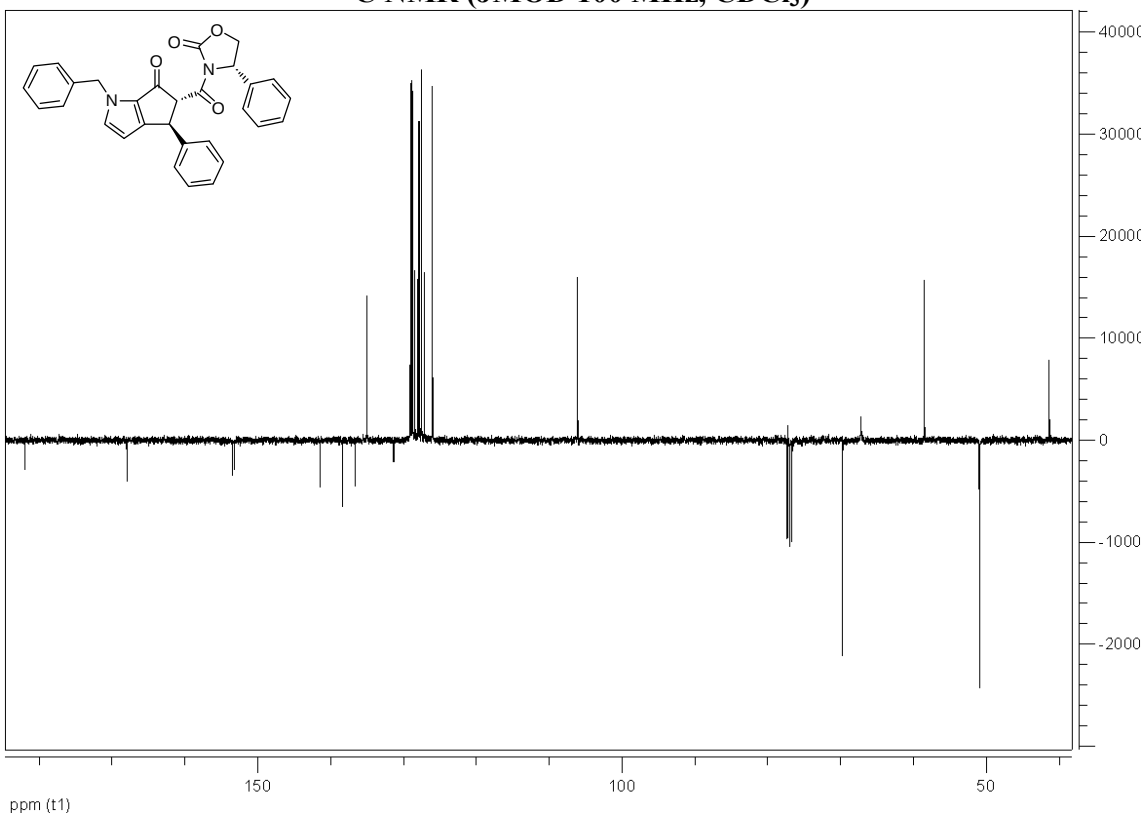

```
Current Data Parameters
NAME                 dksedk
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20070527
Time                  17.56
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                    16769
DS                        4
SWH               18115.941 Hz
FIDRES             0.552855 Hz
AQ                 0.9044468 sec
RG                    1149.4
DW                   27.600 usec
DE                    6.00 usec
TE                   303.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                2.50000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1              75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2             300.2320007 MHz

F1 - Acquisition parameters
ND0                       3
TD                      512
SFO1             300.1314 MHz
FIDRES             0.586194 Hz
SW                 1.000 ppm
FnMODE             undefined

F2 - Processing parameters
SI                     32768
SF               75.4928940 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

(*S*)-3-[(4*S*,5*R*)-1-Benzyl-6-oxo-4-phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (31jA)

^{1}H NMR (400 MHz, $CDCl_3$)

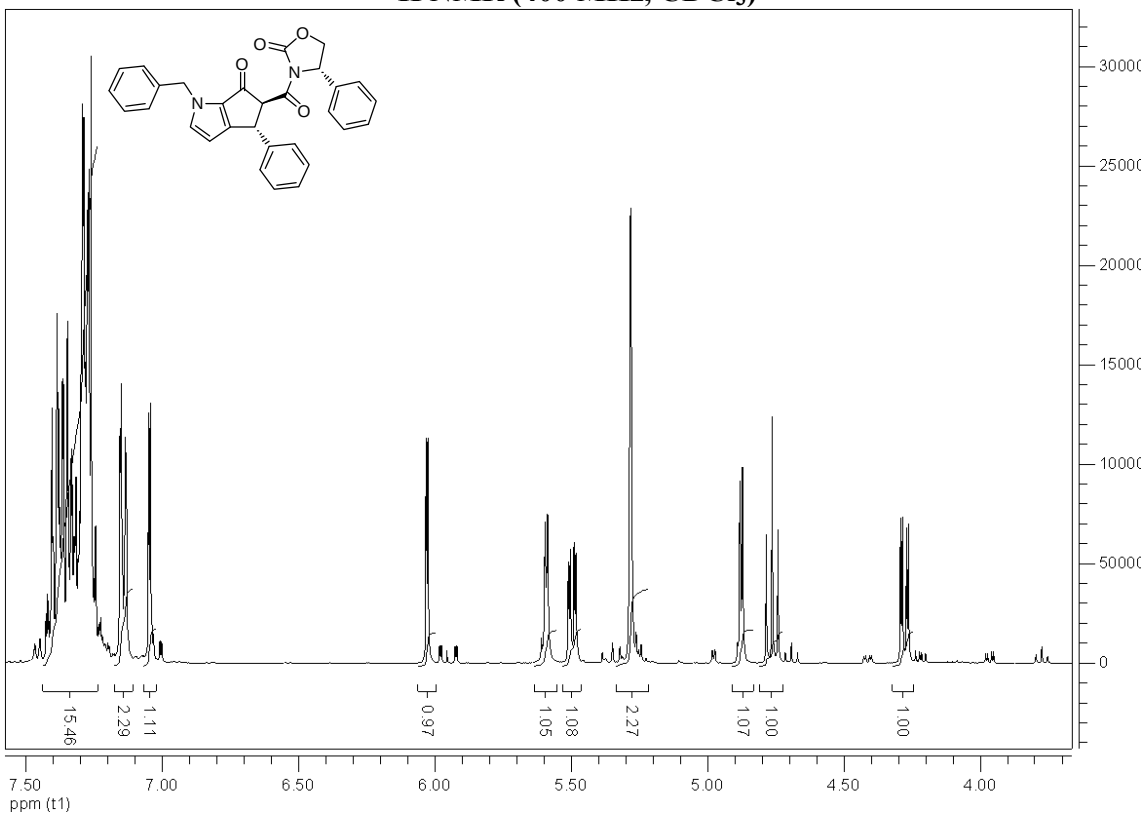


^{13}C NMR (JMOD 100 MHz, $CDCl_3$)

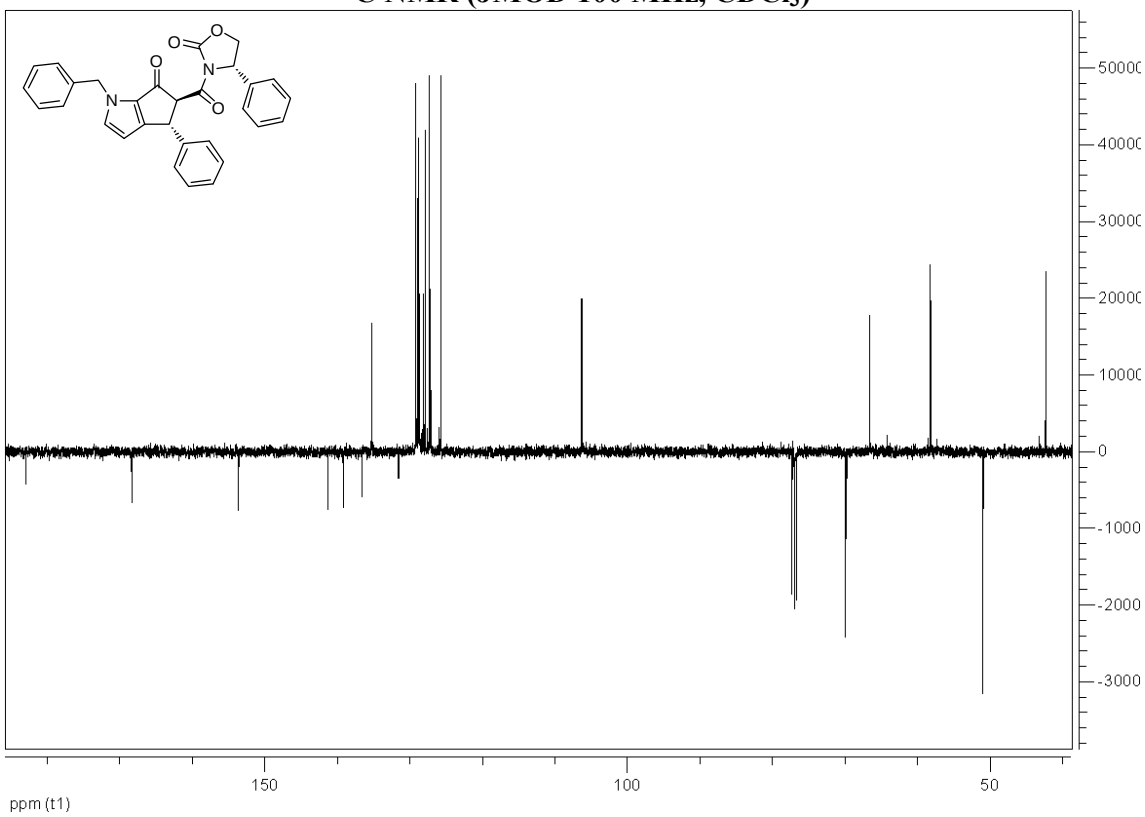


(*S*)-3-[(4*R*,5*S*)-1-Benzyl-6-oxo-4-phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (31jC)

^{1}H NMR (400 MHz, $CDCl_3$)

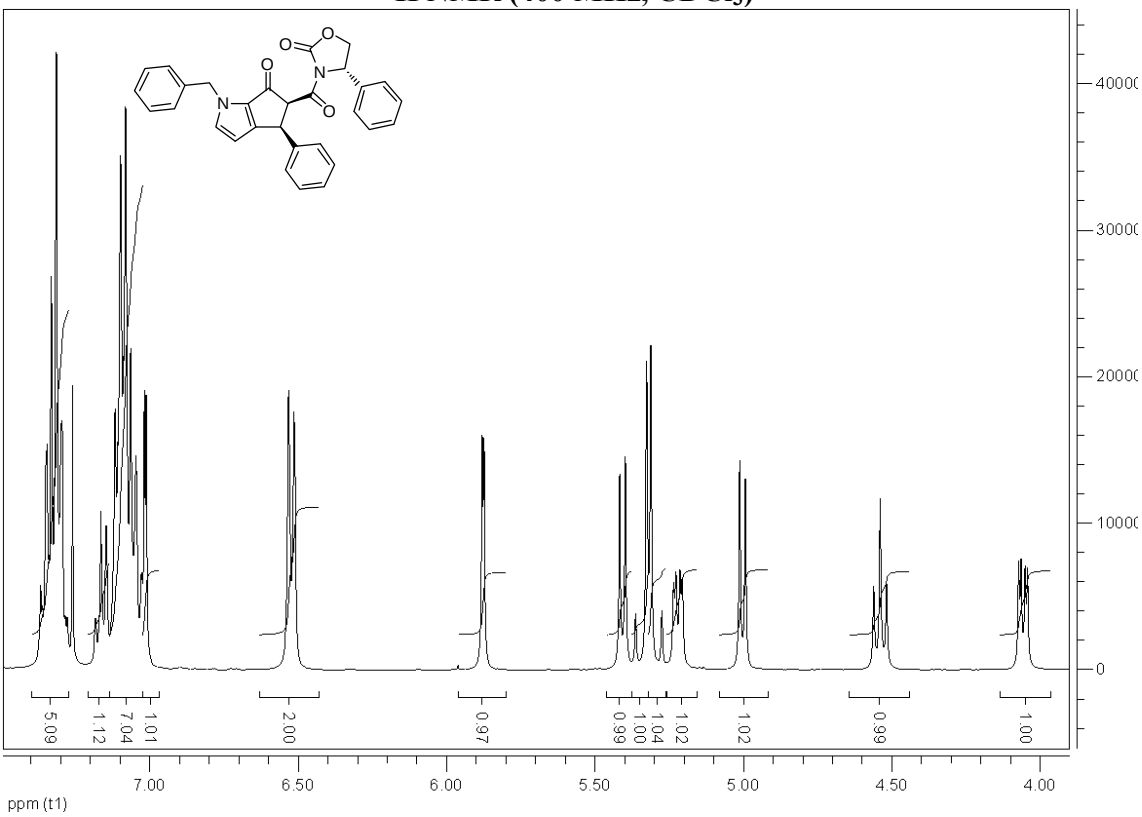


^{13}C NMR (JMOD 100 MHz, $CDCl_3$)

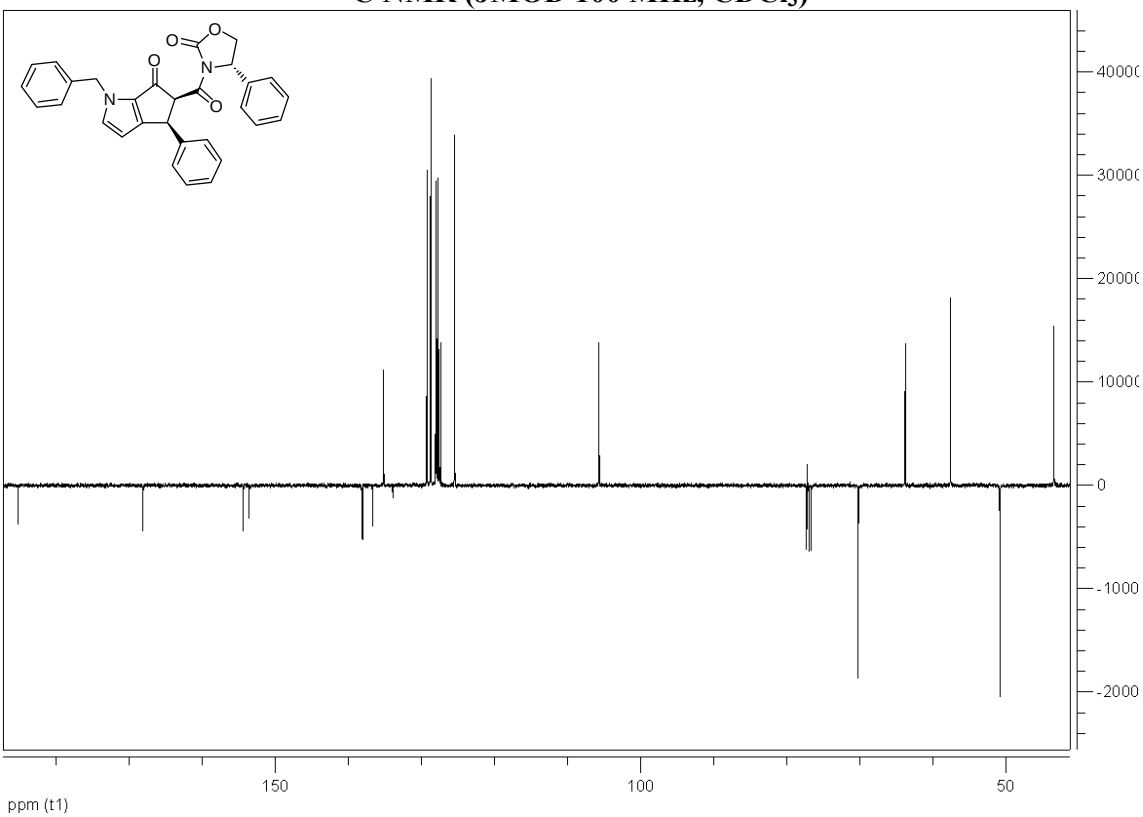


(*S*)-3-[(4*S*,5*S*)-1-Benzyl-6-oxo-4-phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (31jB)

^{1}H NMR (400 MHz, $CDCl_3$)

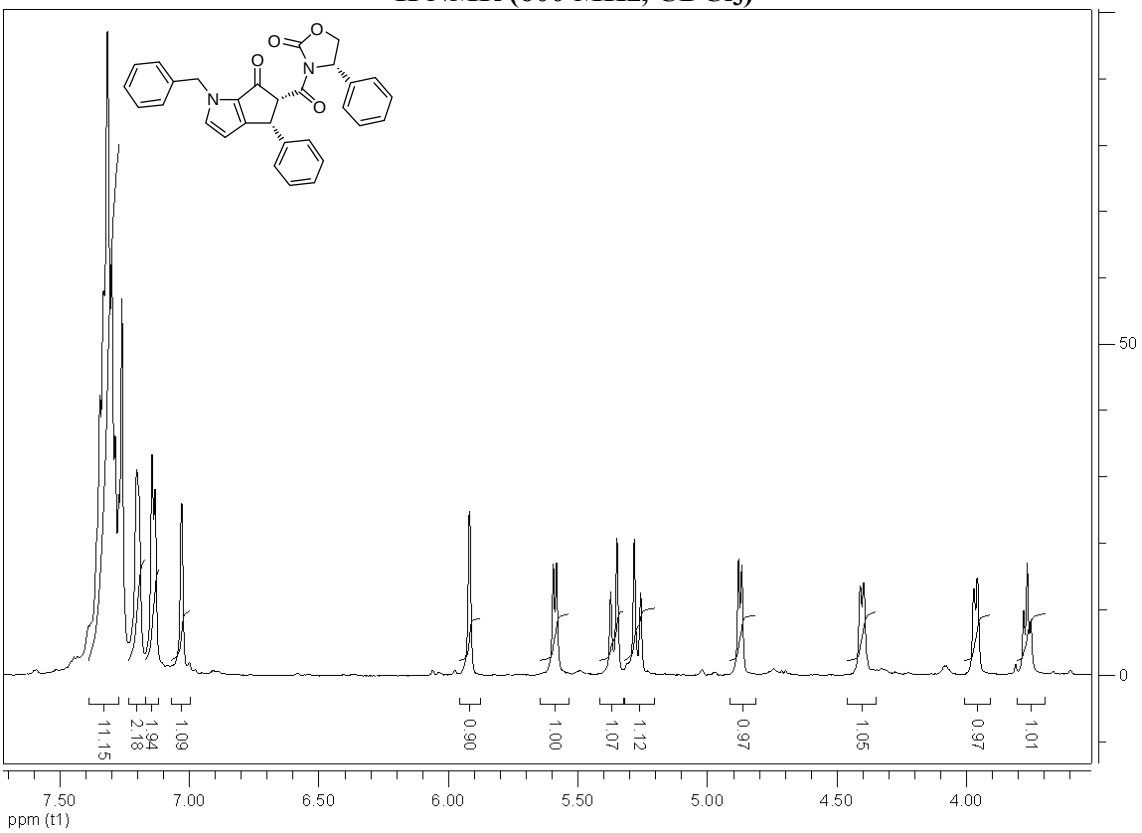


^{13}C NMR (JMOD 100 MHz, $CDCl_3$)

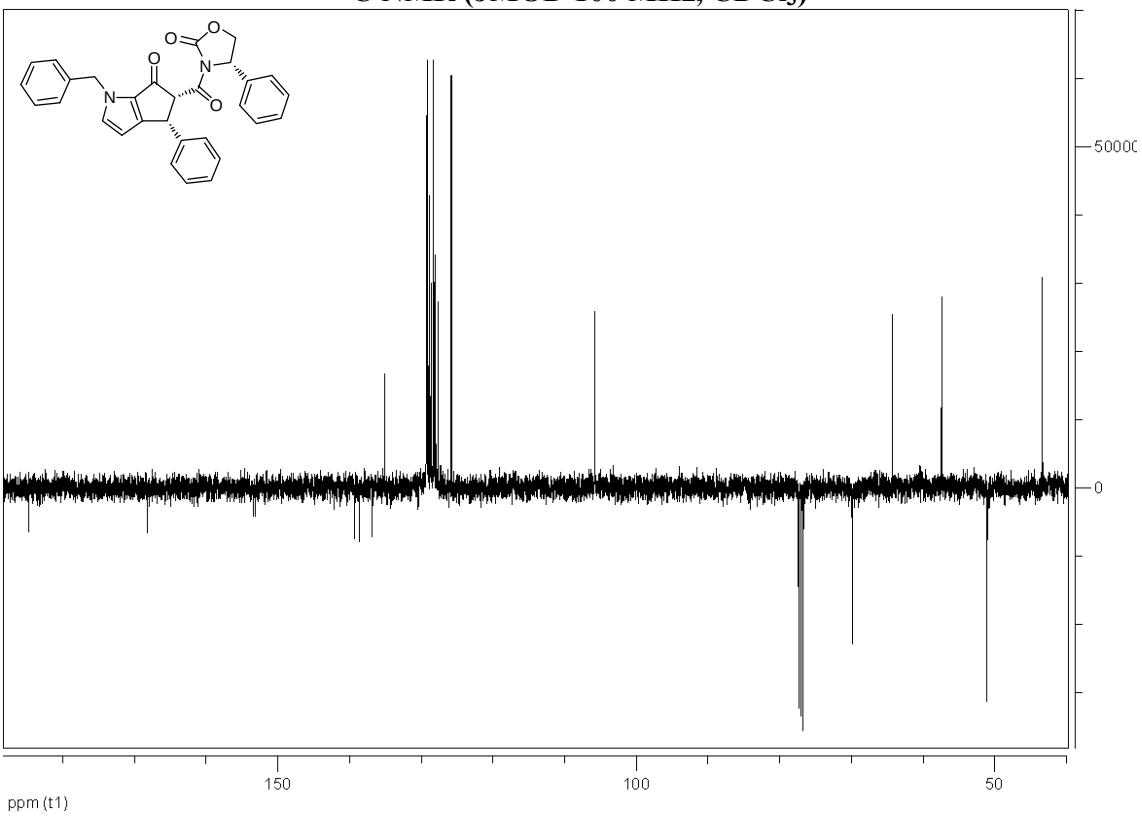


(*S*)-3-[(4*R*,5*R*)-1-benzyl-6-oxo-4-phenyl-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (31jD)

^{1}H NMR (600 MHz, $CDCl_3$)

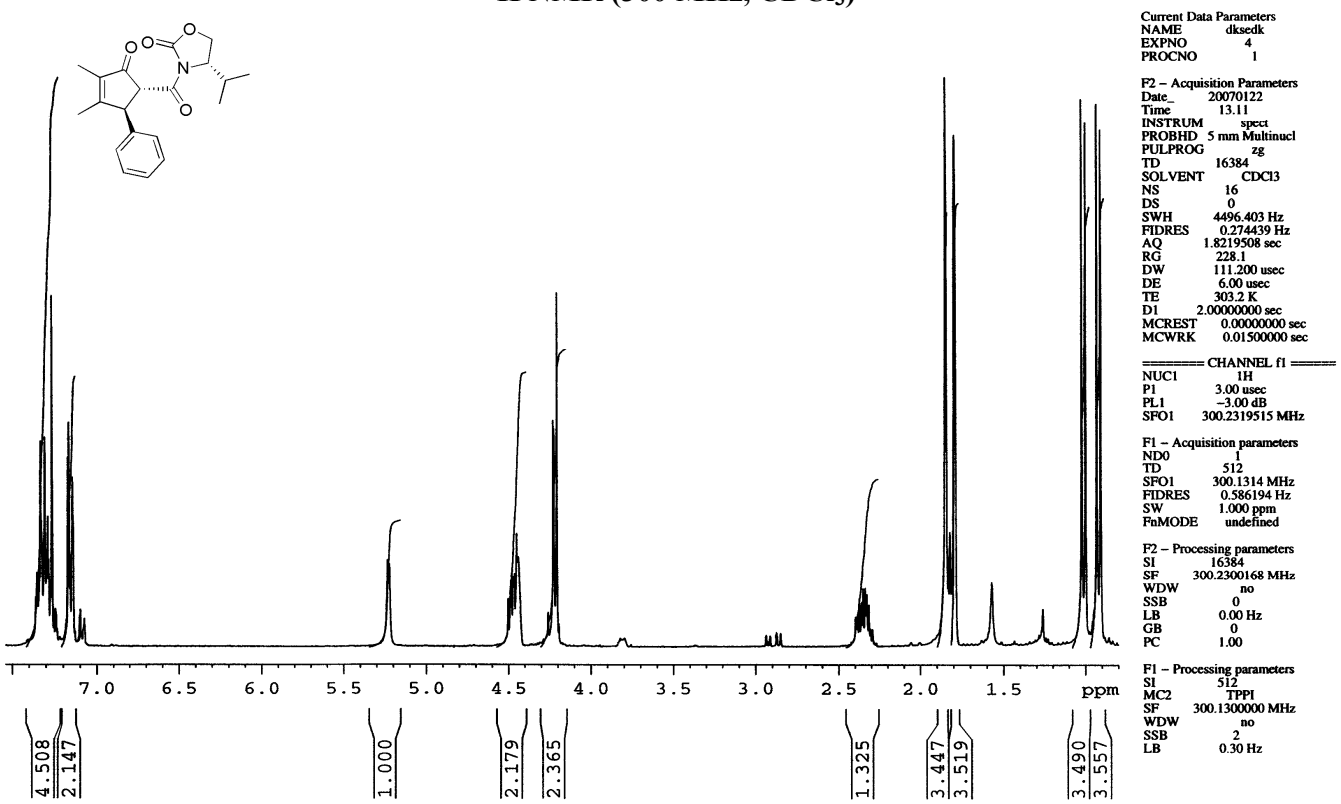


^{13}C NMR (JMOD 100 MHz, $CDCl_3$)

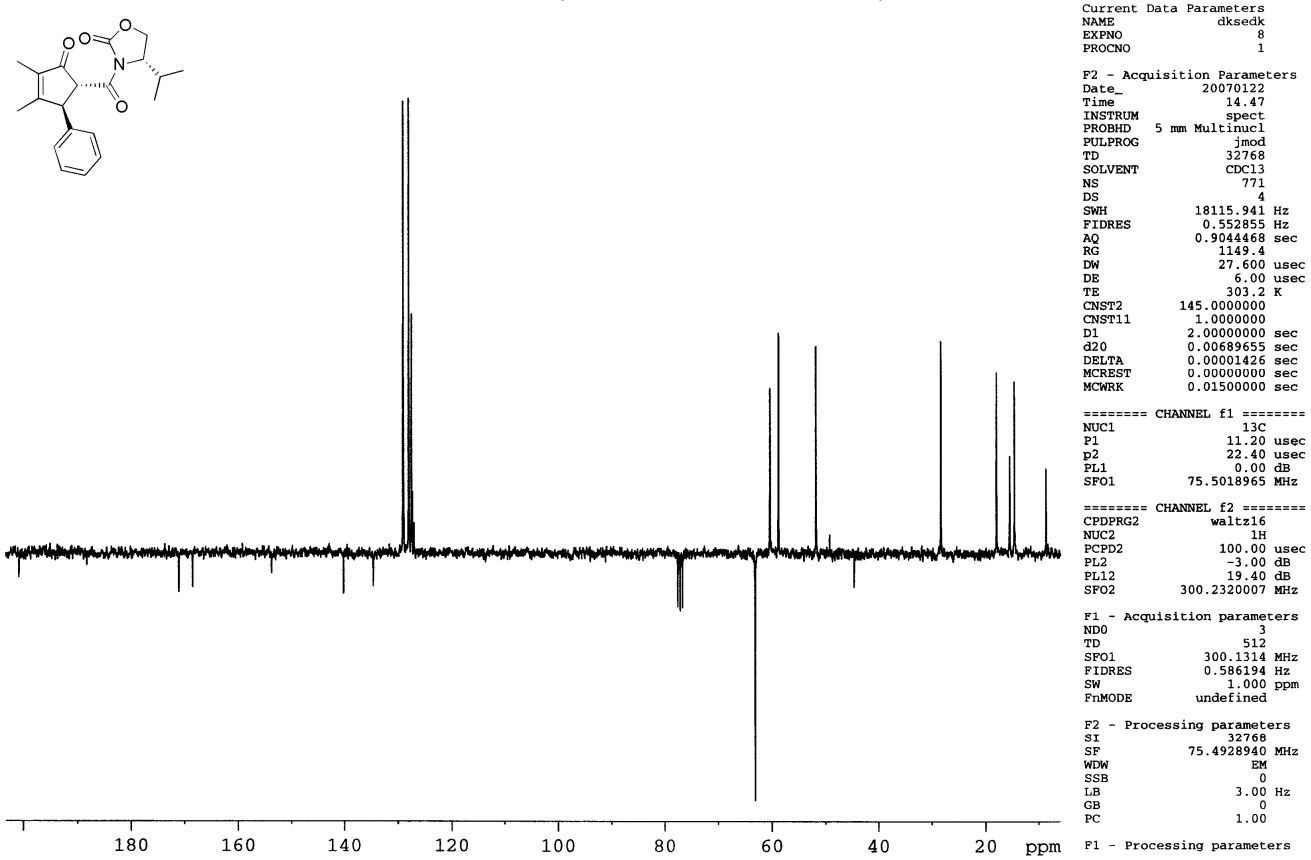


(*S*)-3-[(1*R*,5*S*)-3,4-Dimethyl-2-oxo-5-phenylcyclopent-3-enecarbonyl]-4-isopropyloxazolidin-2-one (31kA)

^{1}H NMR (300 MHz, $CDCl_3$)

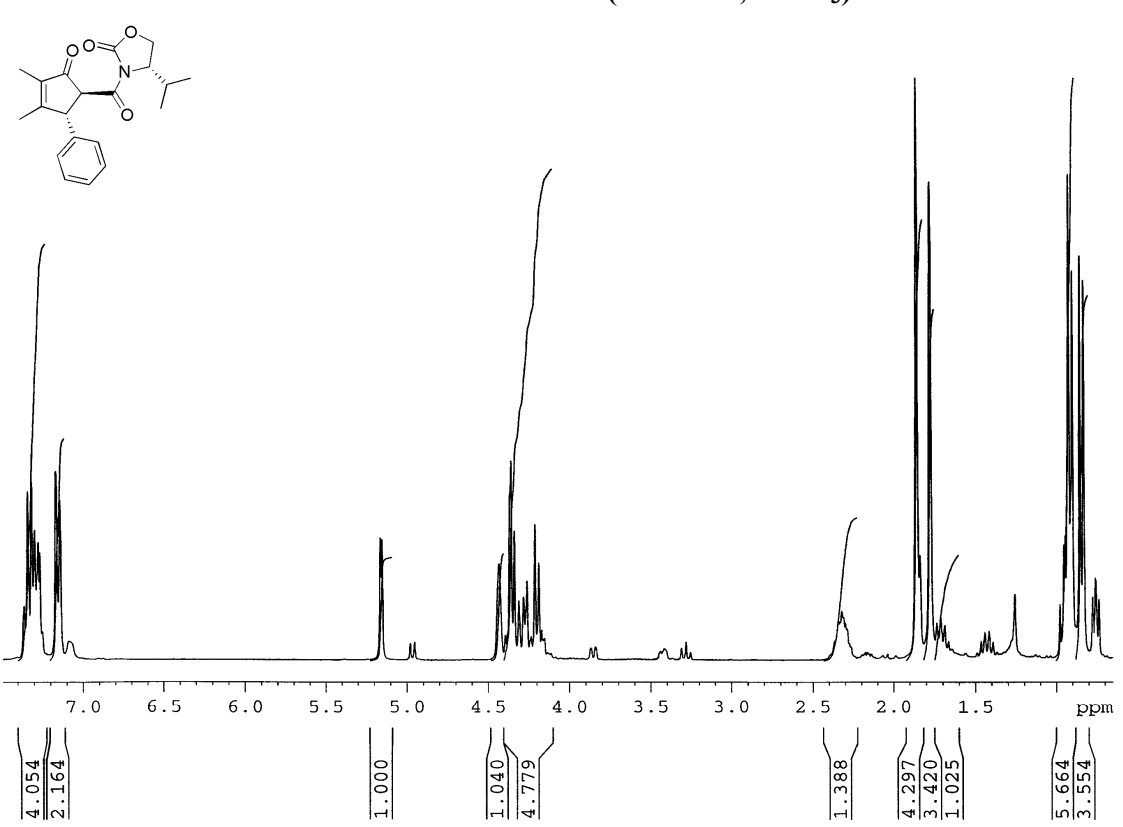


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



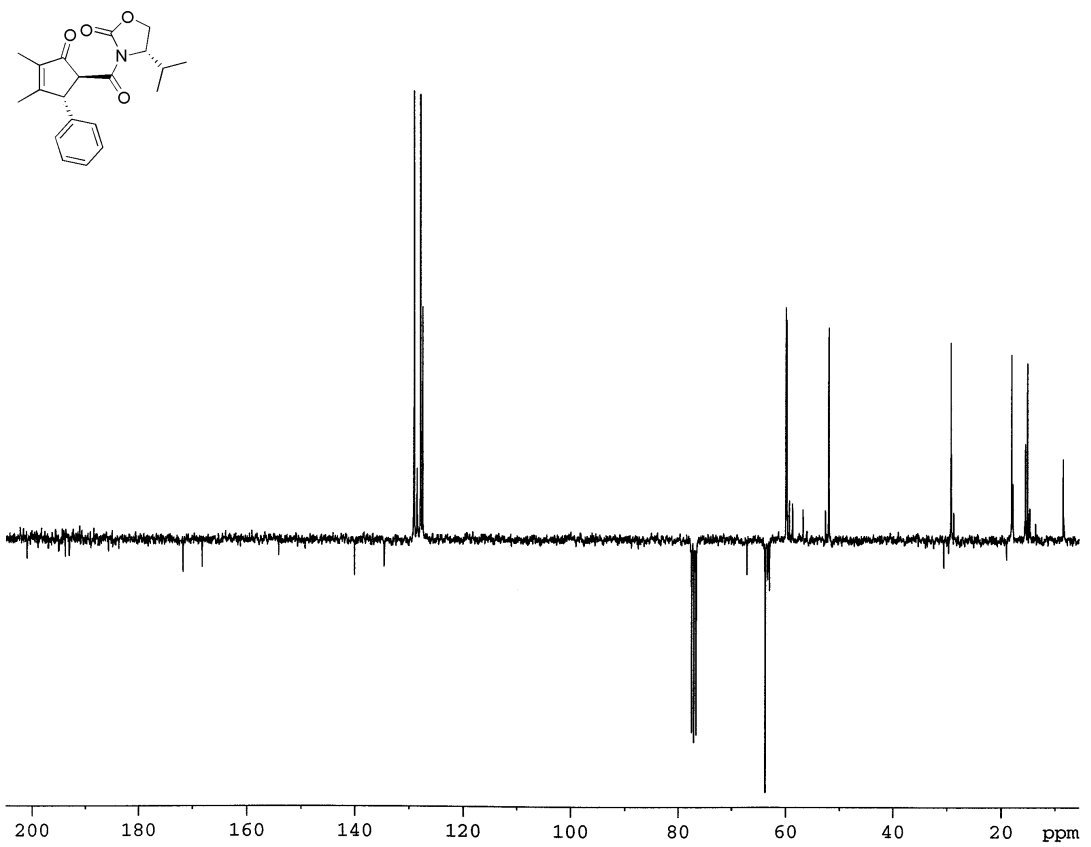
(*S*)-3-[(1*S*,5*R*)-3,4-Dimethyl-2-oxo-5-phenylcyclopent-3-enecarbonyl]-4-isopropyloxazolidin-2-one (31kC)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              5
PROCNO             1

F2 - Acquisition Parameters
Date_        20070206
Time             2.19
INSTRUM         spect
PROBHD  5 mm Multinucl
PULPROG           zg
TD               16384
SOLVENT         CDCl3
NS                  16
DS                   0
SWH          4496.403 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG                101.6
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          2.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.2319515 MHz

F1 - Acquisition parameters
ND0                  1
TD                 512
SFO1       300.1314 MHz
FIDRES       0.586194 Hz
SW           1.000 ppm
FnMODE       undefined

F2 - Processing parameters
SI                16384
SF         300.2300168 MHz
WDW                  no
SSB                   0
LB             0.00 Hz
GB                    0
PC             1.00

F1 - Processing parameters
SI                  512
MC2                TPPI
SF         300.1300000 MHz
WDW                  no
SSB                   2
LB             0.30 Hz
```

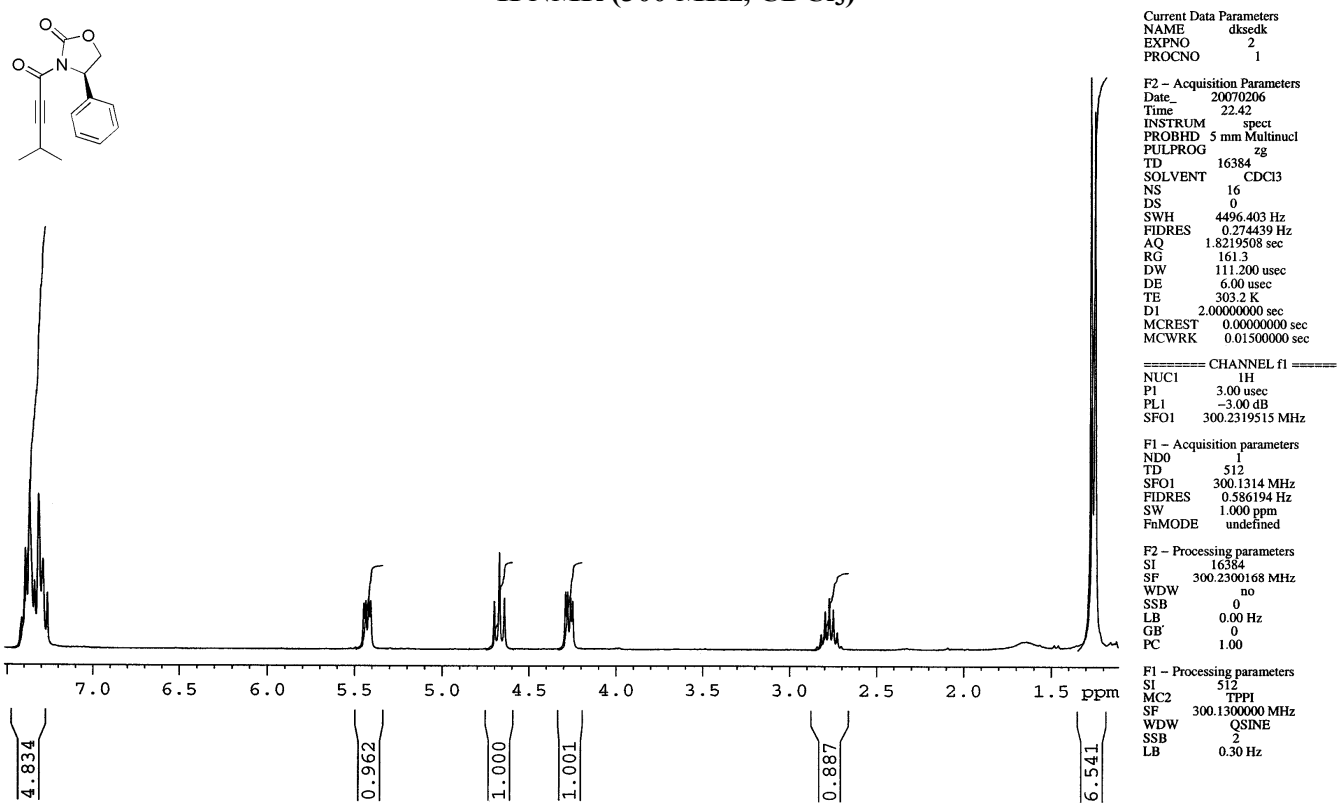
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



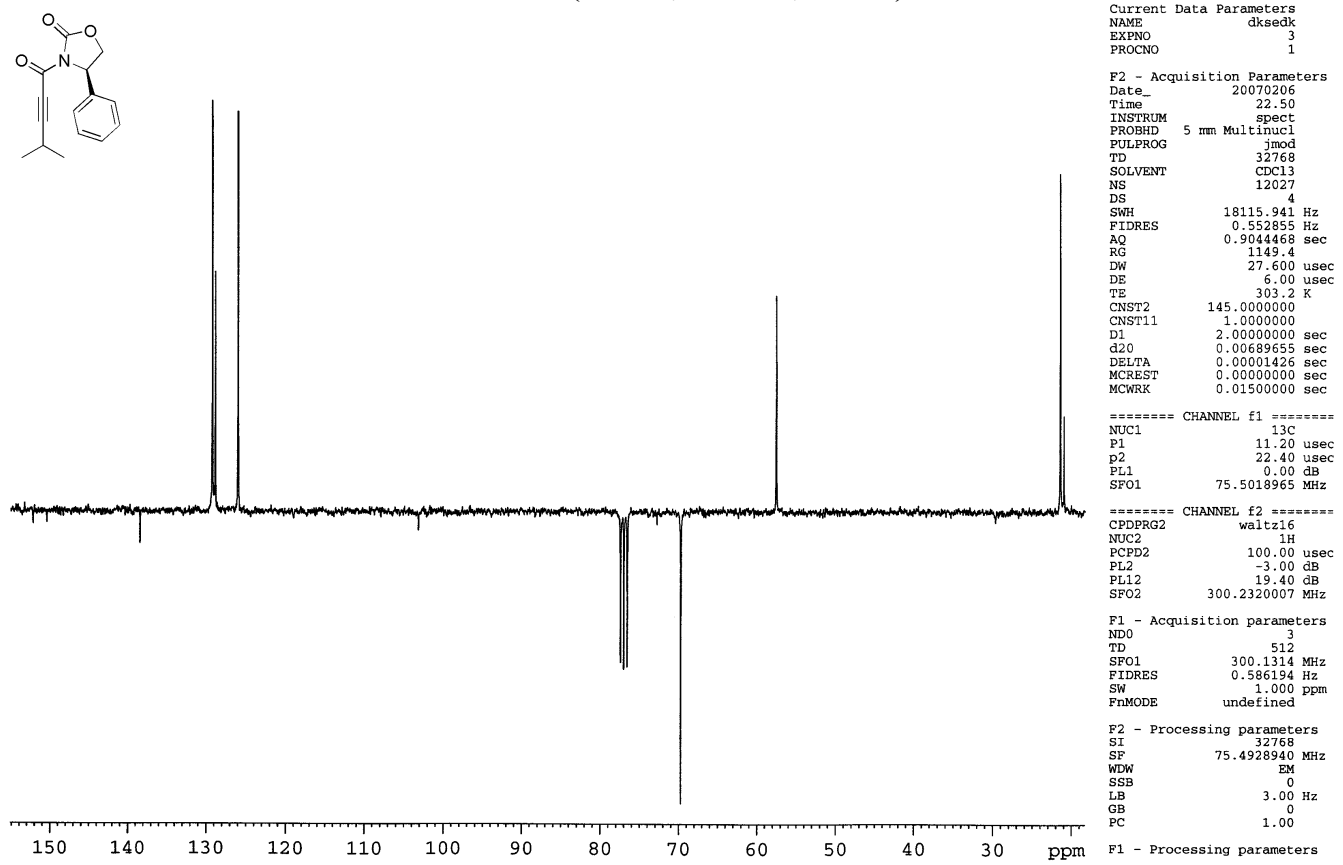

```
Current Data Parameters
NAME                 dksedk
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_               20070208
Time                    0.01
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                    10946
DS                        4
SWH                18115.941 Hz
FIDRES              0.552855 Hz
AQ                  0.9044468 sec
RG                     1149.4
DW                    27.600 usec
DE                     6.00 usec
TE                     303.2 K
CNST2              145.0000000
CNST11               1.0000000
D1                 2.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.20 usec
p2                     22.40 usec
PL1                     0.00 dB
SFO1               75.5018965 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                    -3.00 dB
PL12                   19.40 dB
SFO2               300.2320007 MHz

F1 - Acquisition parameters
ND0                        3
TD                       512
SFO1               300.1314 MHz
FIDRES               0.586194 Hz
SW                   1.000 ppm
FnMODE               undefined

F2 - Processing parameters
SI                       32768
SF                 75.4928940 MHz
WDW                         EM
SSB                          0
LB                      3.00 Hz
GB                           0
PC                      1.00

F1 - Processing parameters
```

(*R*)-3-(4-Methylpent-2-ynoyl)-4-phenyloxazolidin-2-one (35)

^{1}H NMR (300 MHz, $CDCl_3$)

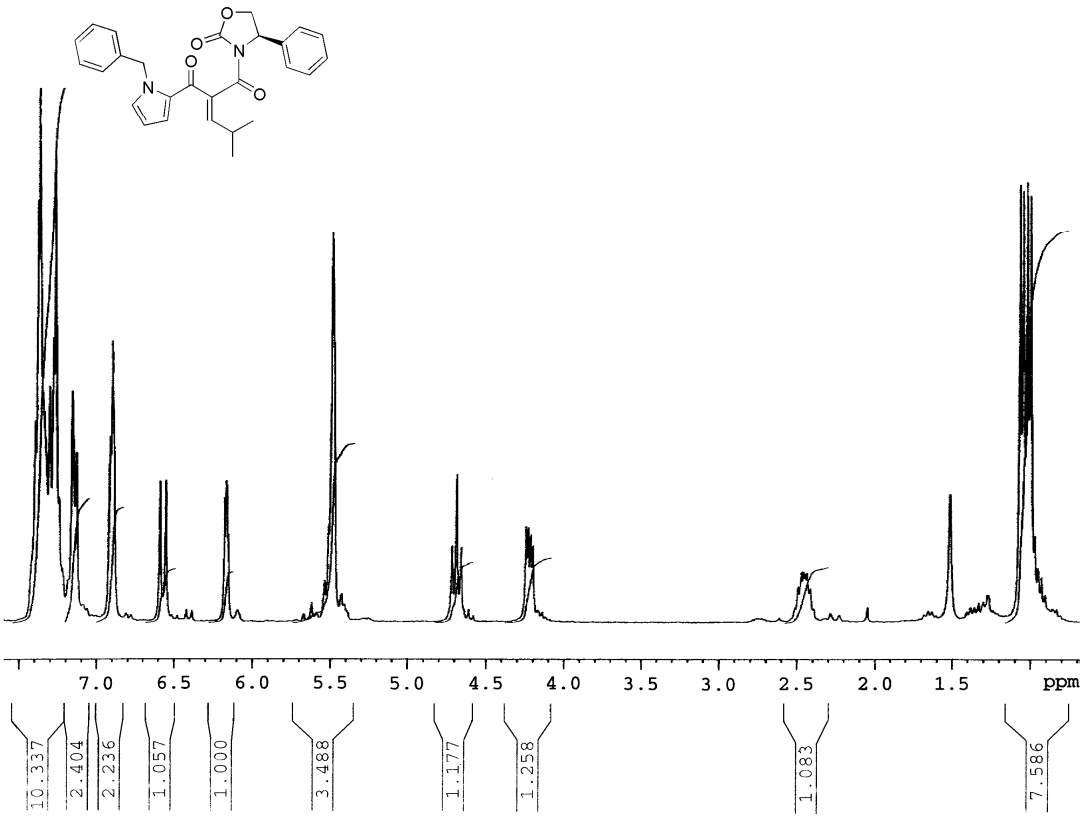


^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)



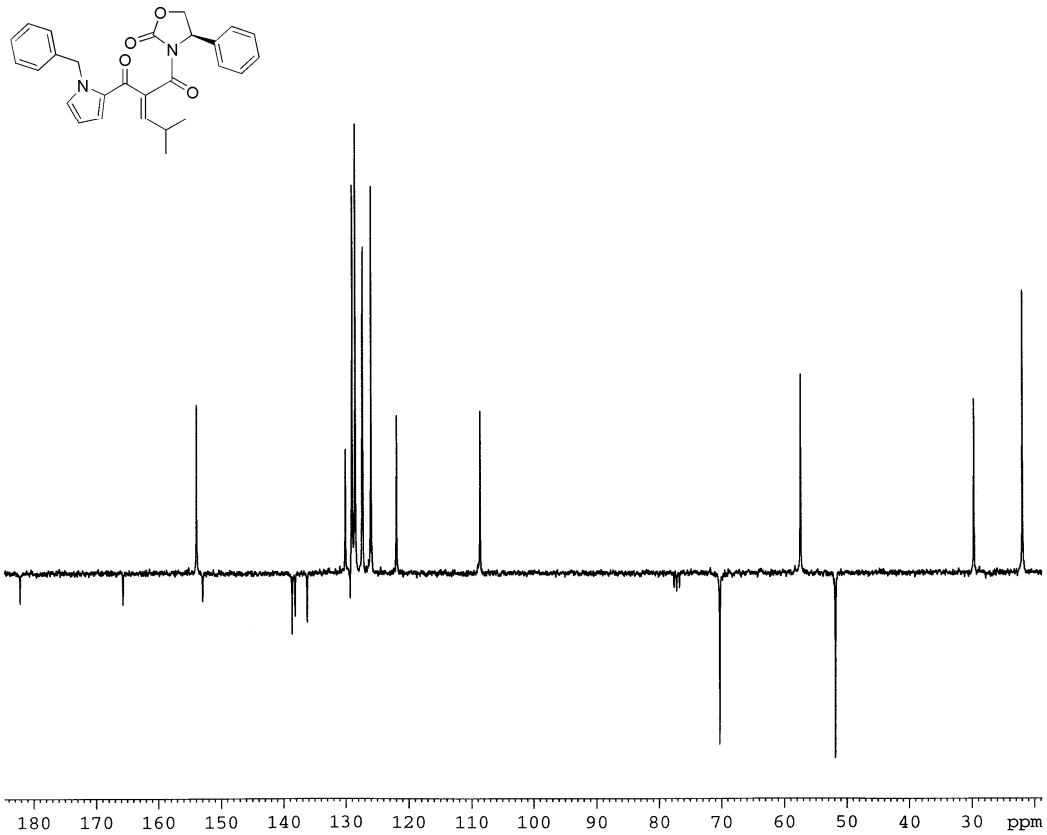
(*R*,*Z*)-1-(1-Benzyl-1*H*-pyrrol-2-yl)-2-(2-methylpropylidene)-3-(2-oxo-4-phenyloxazolidin-3-yl)-propane-1,3-dione (36)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_        20040429
Time            12.21
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG               287.4
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1               3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW                no
SSB                 2
LB               0.30 Hz
```

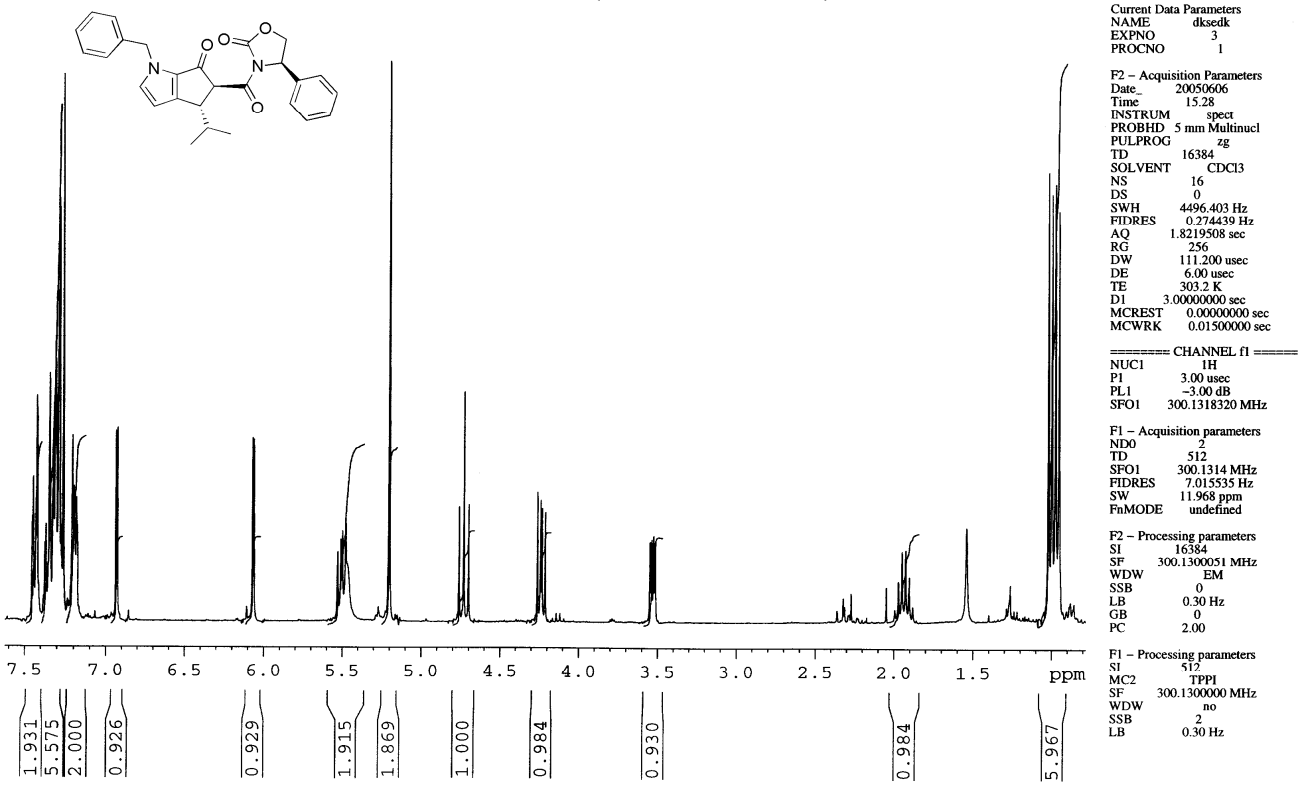
^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050530
Time                  18.01
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     306
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1824.6
DW                  24.600 usec
DE                  20.00 usec
TE                   303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              2.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1            75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2           300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1           300.1314 MHz
FIDRES           7.015535 Hz
SW                11.968 ppm
FnMODE            undefined

F2 - Processing parameters
SI                     32768
SF             75.4677556 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

F1 - Processing parameters
```

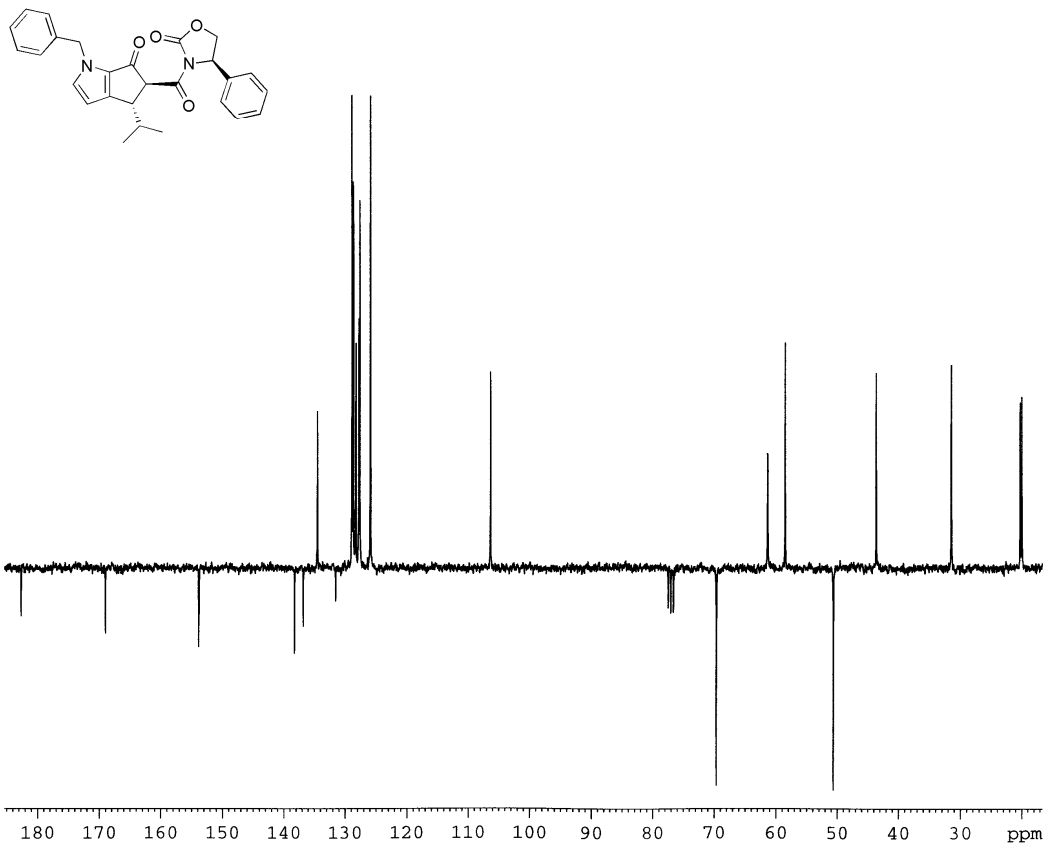
(*R*)-3-[(4*S*,5*S*)-1-Benzyl-4-isopropyl-6-oxo-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (37A)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              3
PROCNO             1

F2 - Acquisition Parameters
Date_        20050606
Time            15.28
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                256
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW                EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW                no
SSB                2
LB              0.30 Hz
```

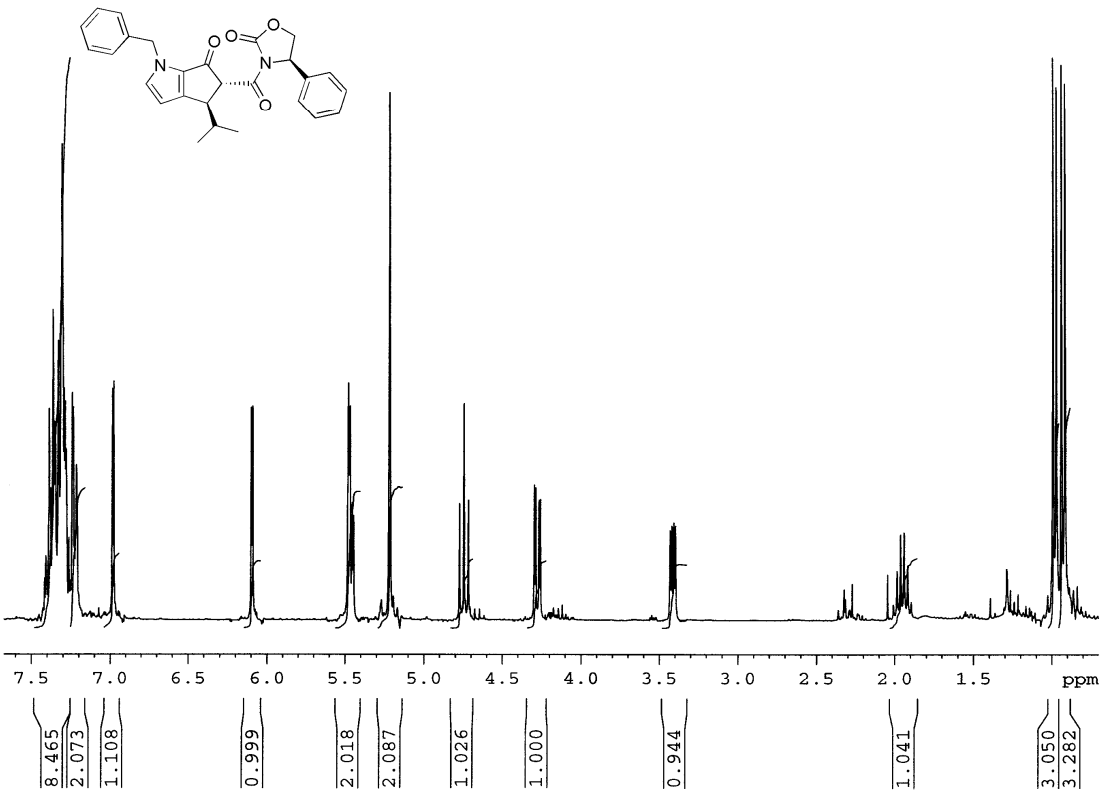
^{13}C NMR (JMOD, 75 MHz)




```
Current Data Parameters
NAME                 dksedk
EXPNO                     3
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050607
Time                  18.05
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                     186
DS                       4
SWH              20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1               4.00000000 sec
d20              0.00689655 sec
DELTA            0.00001426 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1            75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2           300.1320000 MHz

F2 - Processing parameters
SI                   32768
SF              75.4677556 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

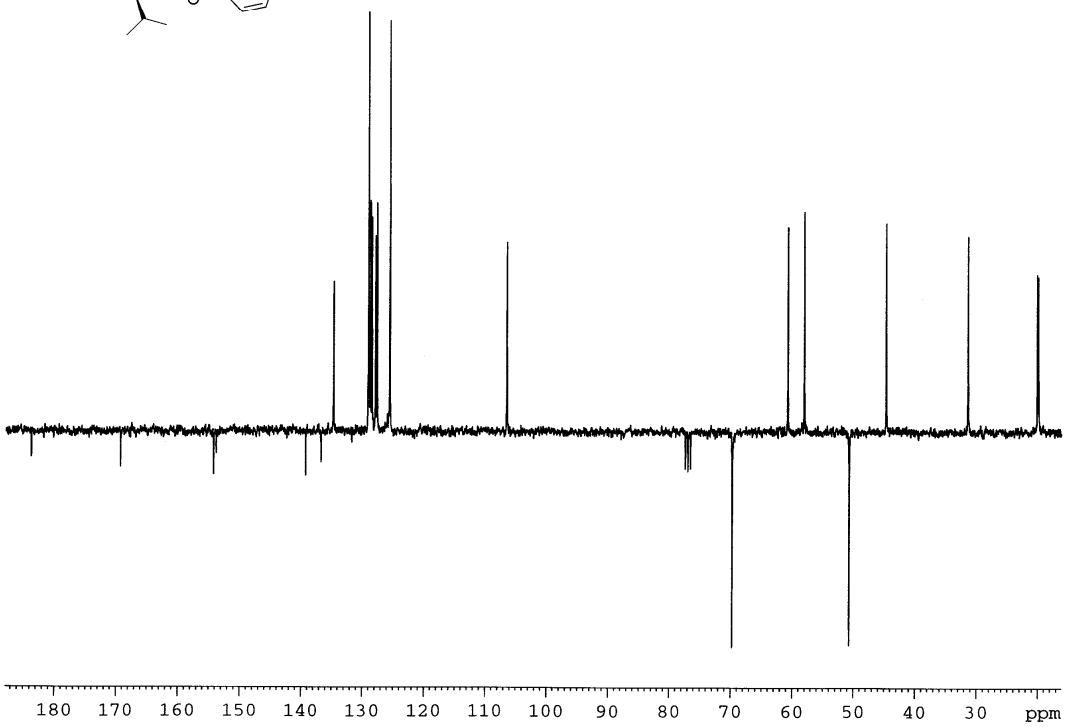
(*R*)-3-[(4*R*,5*R*)-1-Benzyl-4-isopropyl-6-oxo-1,4,5,6-tetrahydrocyclopenta[*b*]pyrrole-5-carbonyl]-4-phenyloxazolidin-2-one (37C)

^{1}H NMR (300 MHz, $CDCl_3$)




```
Current Data Parameters
NAME          dksedk
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_        20050622
Time            14.07
INSTRUM         spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCL3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG                35.9
DW             111.200 usec
DE               6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW          11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW             QSINE
SSB                 2
LB              0.30 Hz
```

^{13}C NMR (JMOD, 75 MHz, $CDCl_3$)

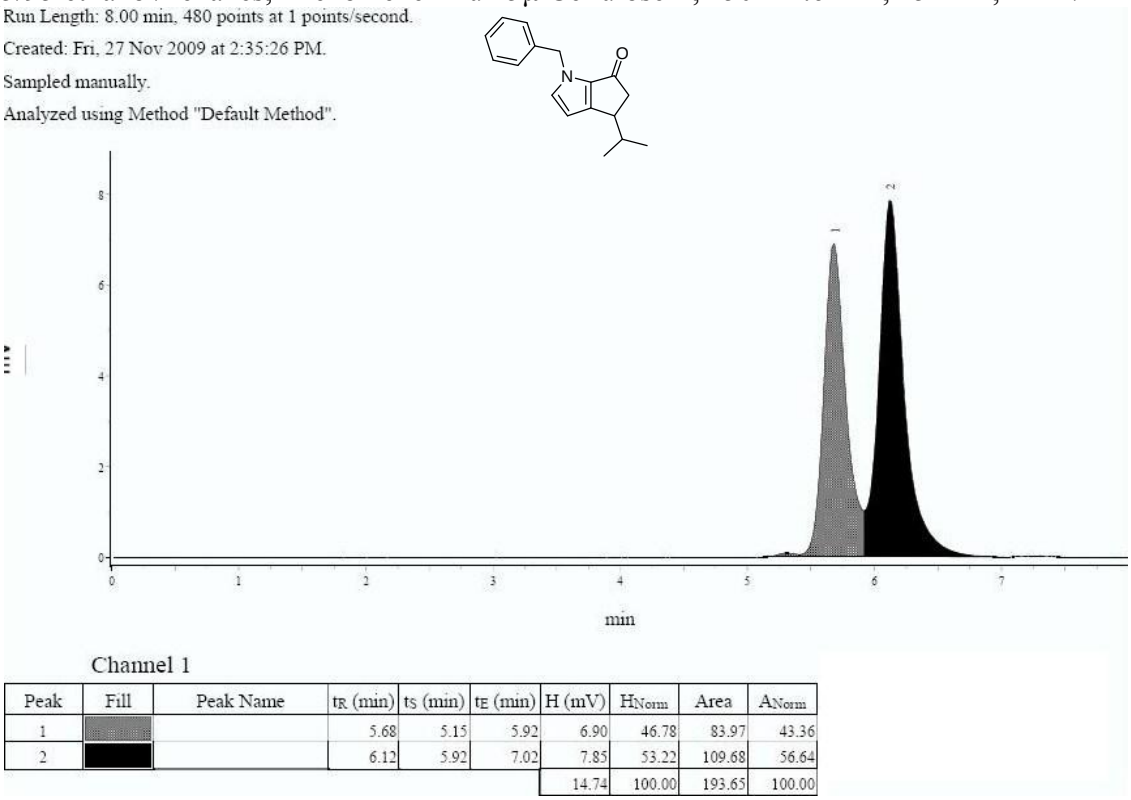



```
Current Data Parameters
NAME                 dksedk
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20050622
Time                  14.20
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                      398
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                 0.8061428 sec
RG                    1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 2.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   11.20 usec
p2                   22.40 usec
PL1                   0.00 dB
SFO1             75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                  -3.00 dB
PL12                 19.40 dB
SFO2            300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1             300.1314 MHz
FIDRES             7.015535 Hz
SW                11.968 ppm
FnMODE             undefined

F2 - Processing parameters
SI                    32768
SF               75.4677556 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```

V. Enantiopurity Determination of Hydrolysis Product *R*-21 by Chiral HPLC

Chiral HPLC (±)-21

(5/95 ethanol/hexanes, Phenomenex Lux 5µ Cellulose-1, 150 × 4.6 mm, 254 nm, 1 mL/min)

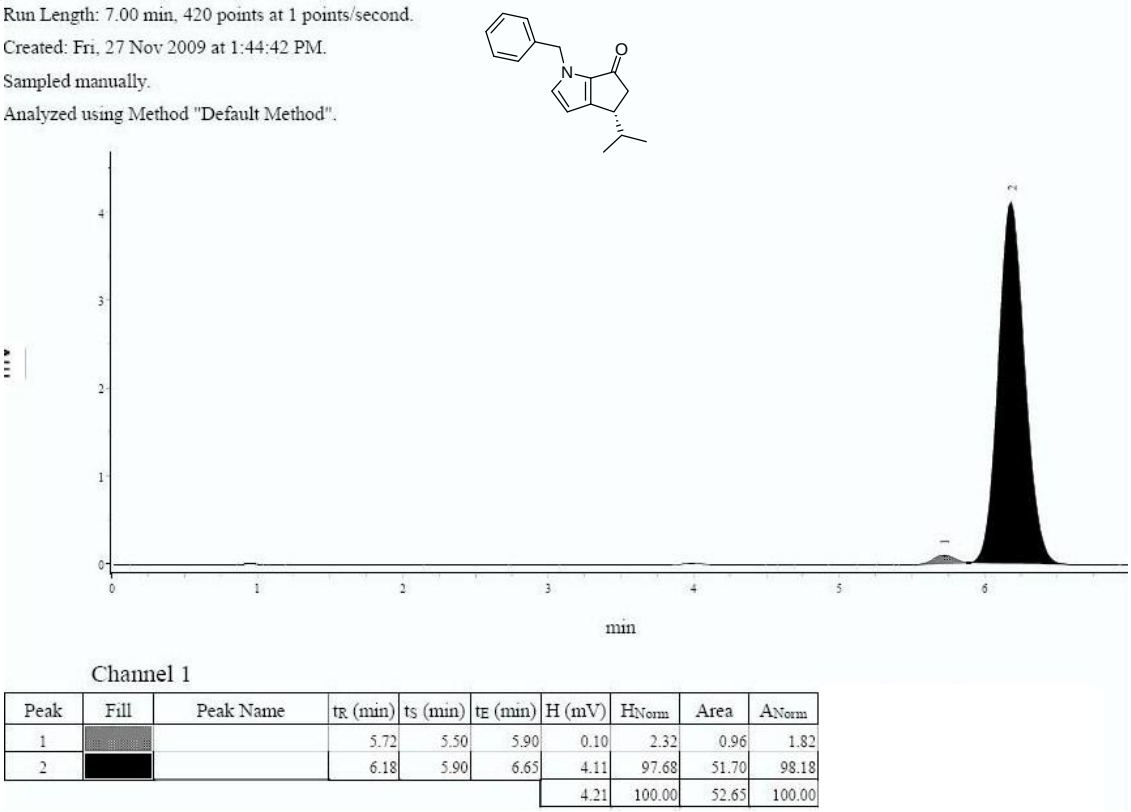


Channel 1

Peak	Fill	Peak Name	t_R (min)	t_S (min)	t_E (min)	H (mV)	H_{Norm}	Area	A_{Norm}
1			5.68	5.15	5.92	6.90	46.78	83.97	43.36
2			6.12	5.92	7.02	7.85	53.22	109.68	56.64
						14.74	100.00	193.65	100.00

Chiral HPLC *R*-21

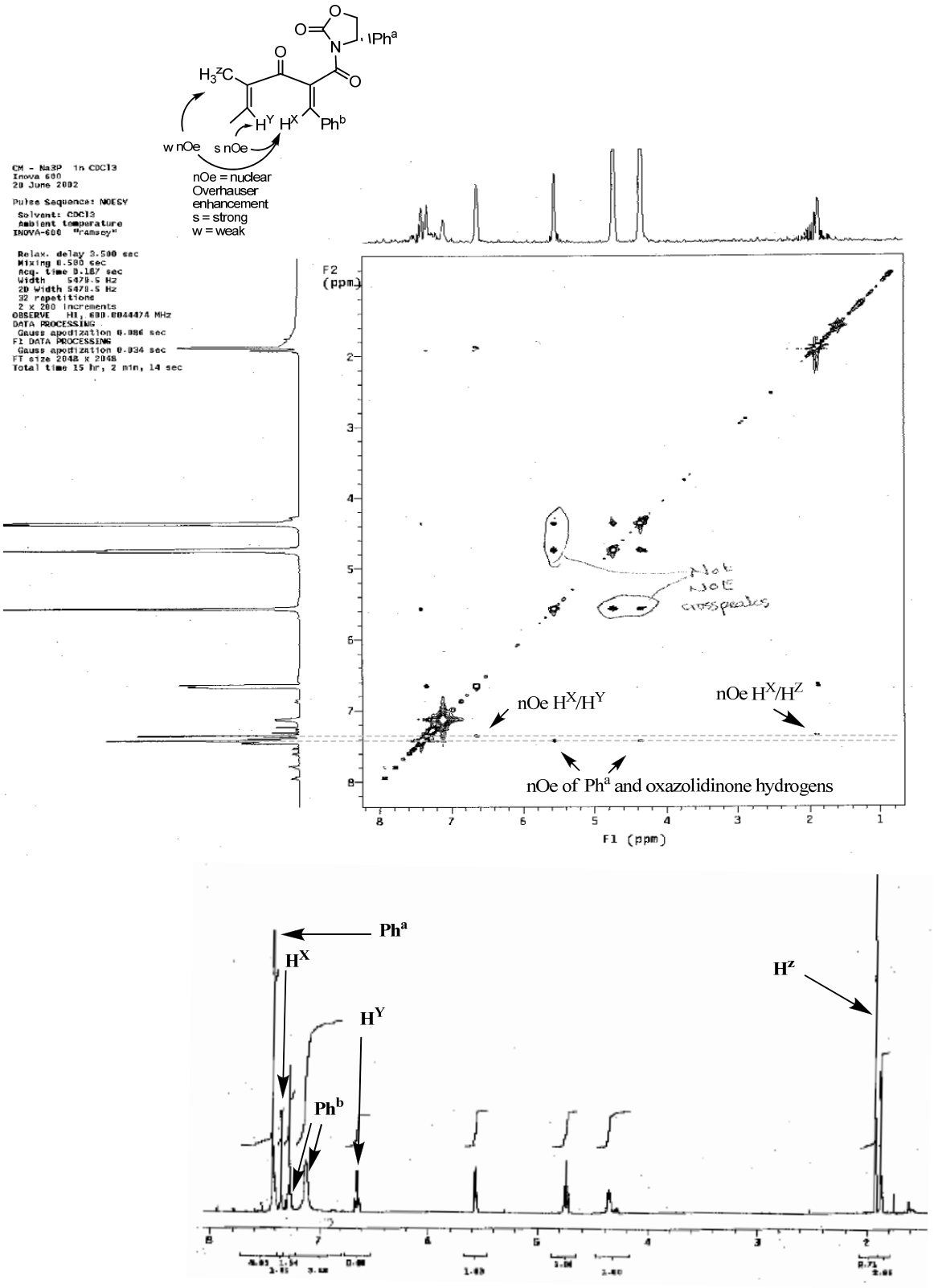
(5/95 ethanol/hexanes, Phenomenex Lux 5µ Cellulose-1, 150 × 4.6 mm, 254 nm, 1 mL/min)



Channel 1

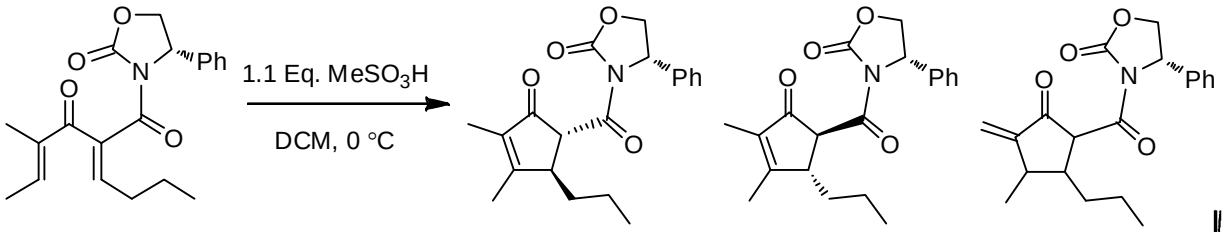
Peak	Fill	Peak Name	t_R (min)	t_S (min)	t_E (min)	H (mV)	H_{Norm}	Area	A_{Norm}
1			5.72	5.50	5.90	0.10	2.32	0.96	1.82
2			6.18	5.90	6.65	4.11	97.68	51.70	98.18
						4.21	100.00	52.65	100.00

VI. Determination of 30g Stereochemistry by NOESY NMR Spectroscopy

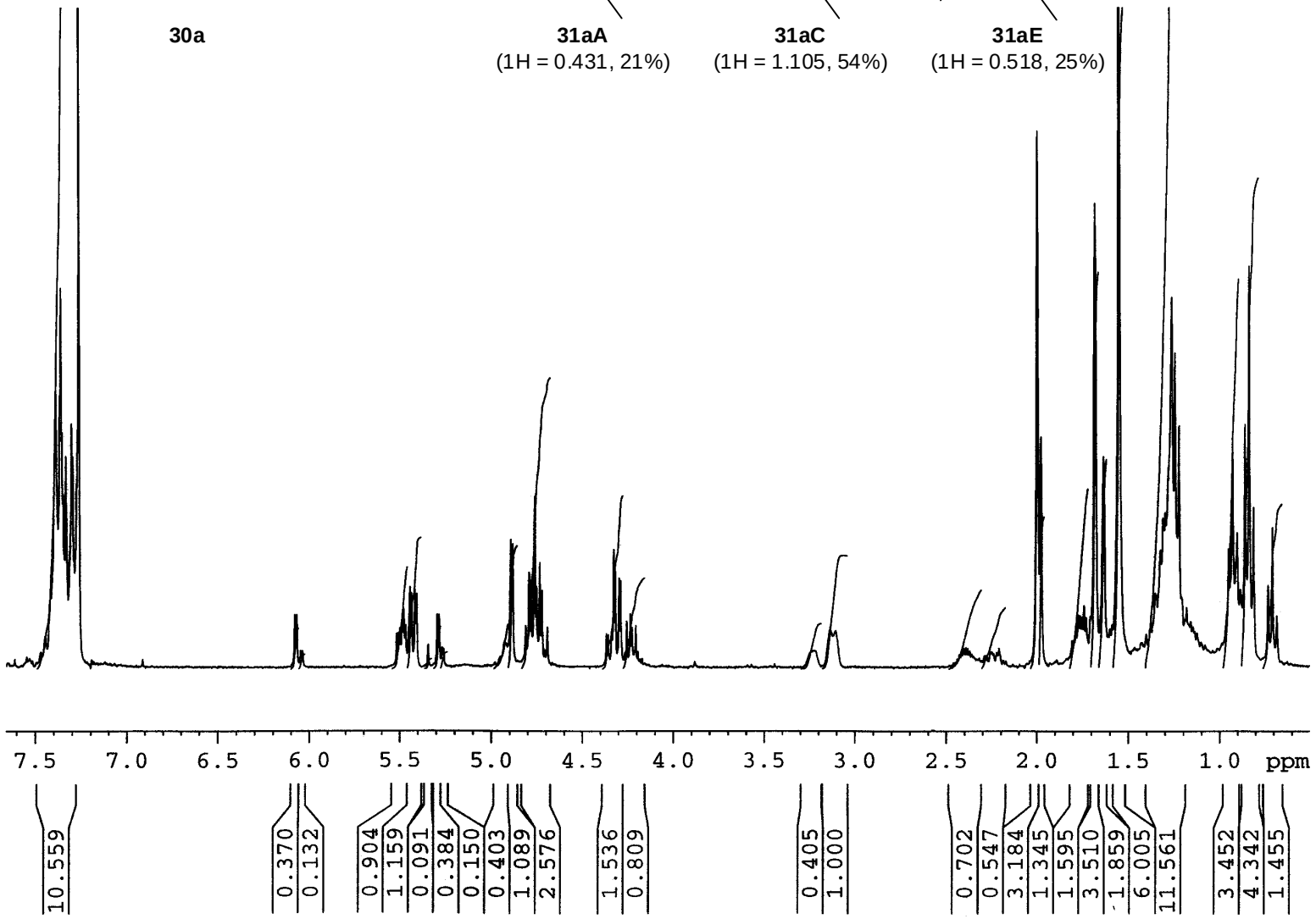


VII. Asymmetric Nazarov Reaction Crude NMR Spectra (Determination of Product Distribution for Tables 4 and 8)

Table 8: Entry 1

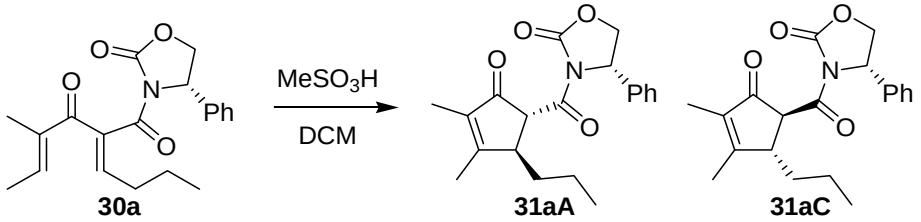


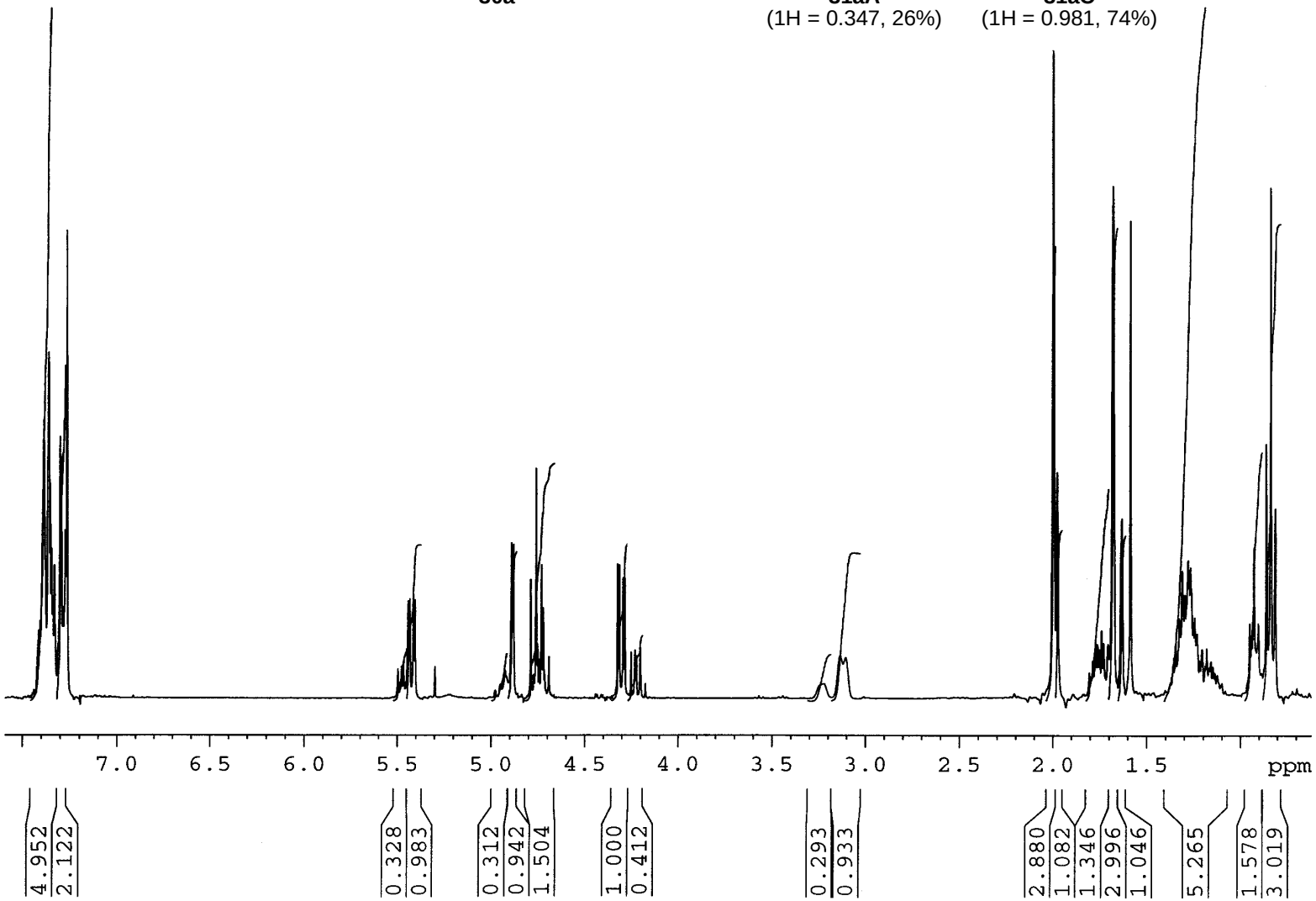
30a → **31aA** (1H = 0.431, 21%) + **31aC** (1H = 1.105, 54%) + **31aE** (1H = 0.518, 25%)




```
Current Data Parameters
NAME          dksedk
EXPNO              2
PROCNO             1

F2 - Acquisition Parameters
Date_         20050215
Time             12.52
INSTRUM          spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH          4496.403 Hz
FIDRES       0.274439 Hz
AQ           1.8219508 sec
RG               456.1
DW           111.200 usec
DE             6.00 usec
TE             303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1             -3.00 dB
SFO1       300.1318320 MHz

F1 - Acquisition parameters
ND0                2
TD               512
SFO1       300.1314 MHz
FIDRES       7.015535 Hz
SW           11.968 ppm
FnMODE       undefined

F2 - Processing parameters
SI              16384
SF         300.1300051 MHz
WDW                EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 - Processing parameters
SI               512
MC2             TPPI
SF         300.1300000 MHz
WDW              no
SSB                2
LB              0.30 Hz
```

Table 8: Entry 2





Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050512
Time 16.33
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 143.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

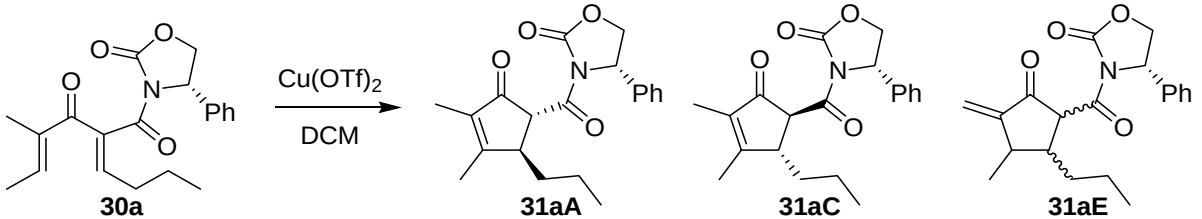
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 3

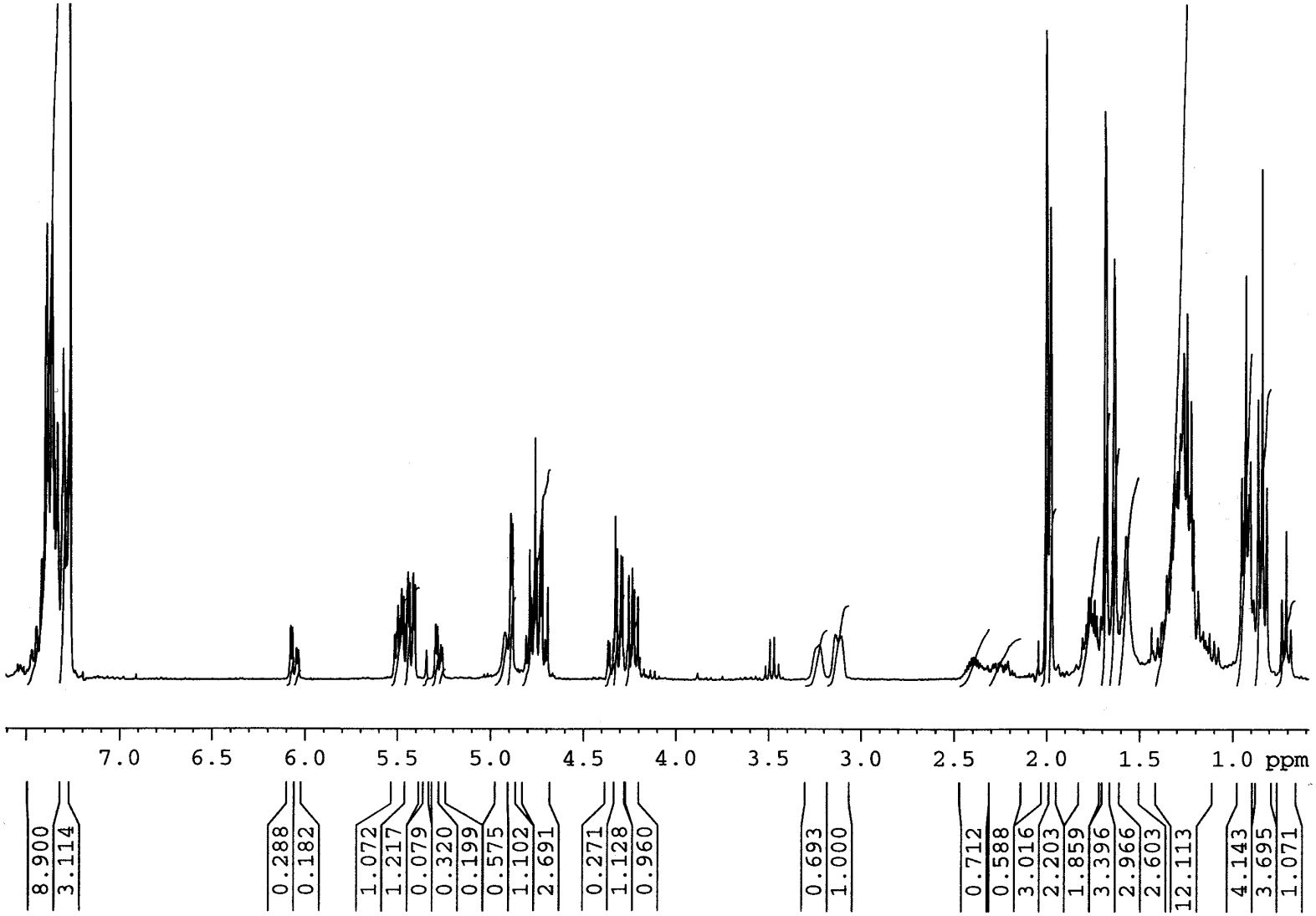


30a

31aA (1H = 0.694, 31%)

31aC (1H = 1.086, 48%)

31aE (1H = 0.495, 21%)



Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050216
Time 12.09
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 287.4
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

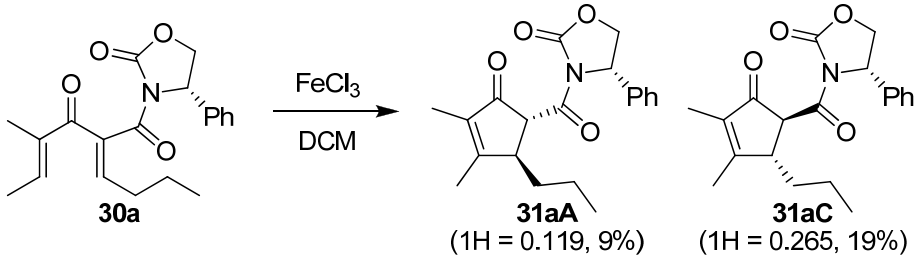
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

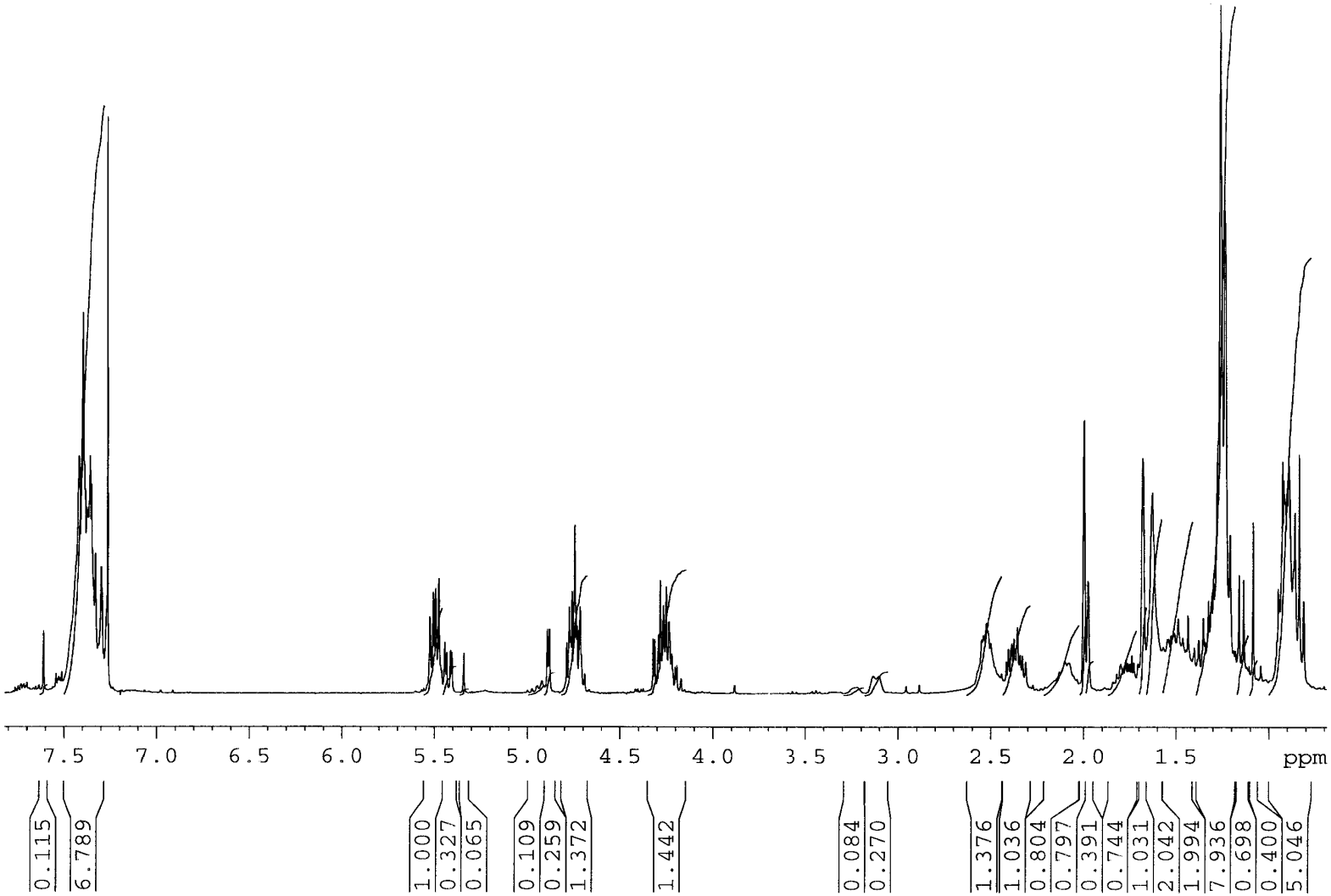
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 4





Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050215
Time 12.40
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

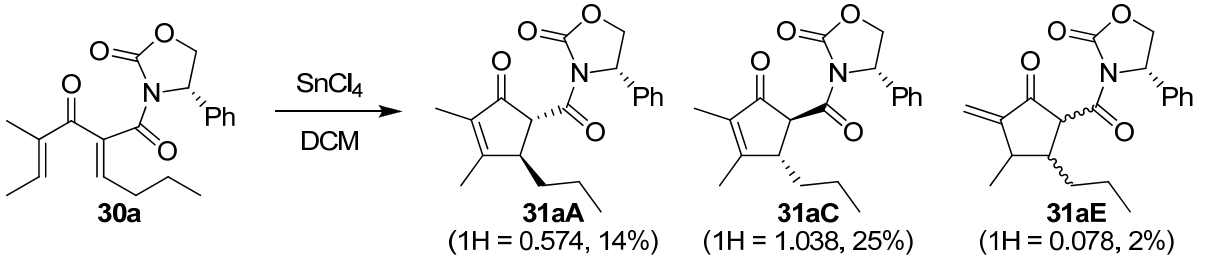
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

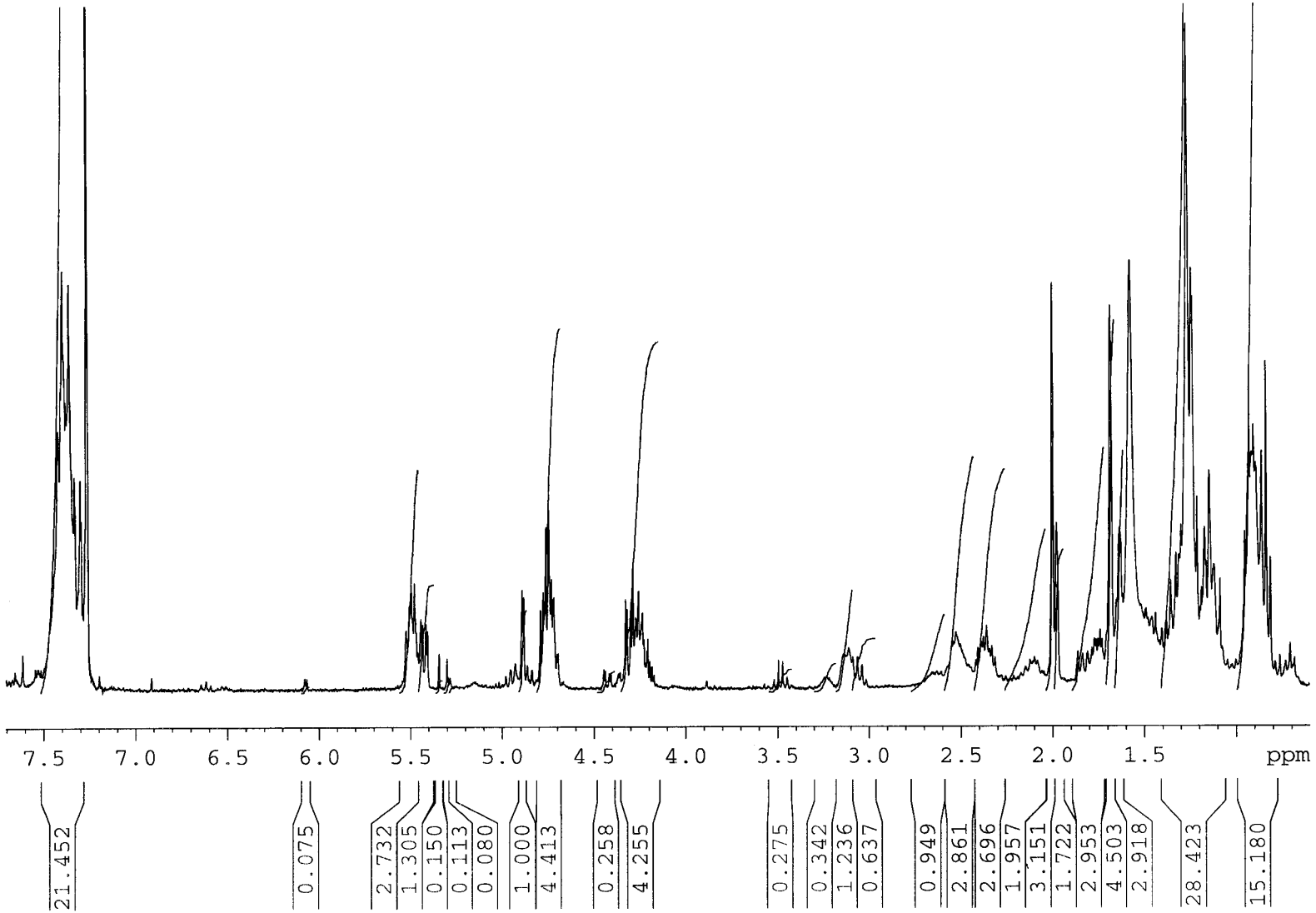
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 5





Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050218
Time 11.55
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

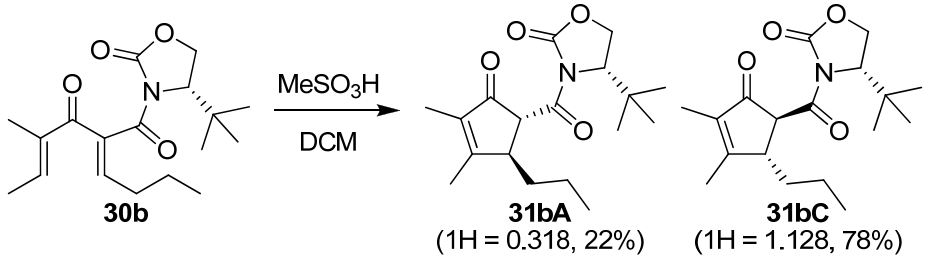
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

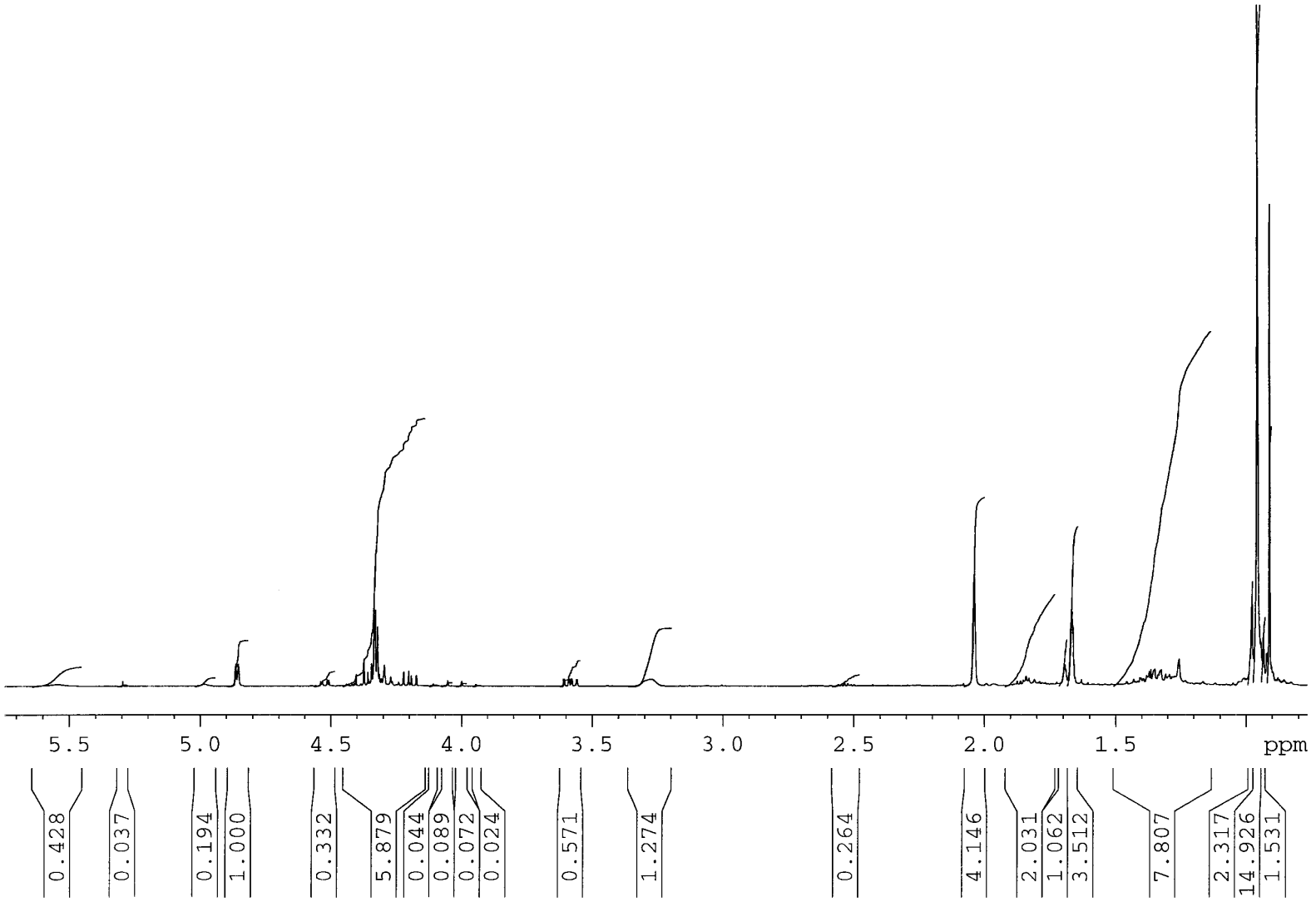
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 6





Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050504
Time 14.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 143.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

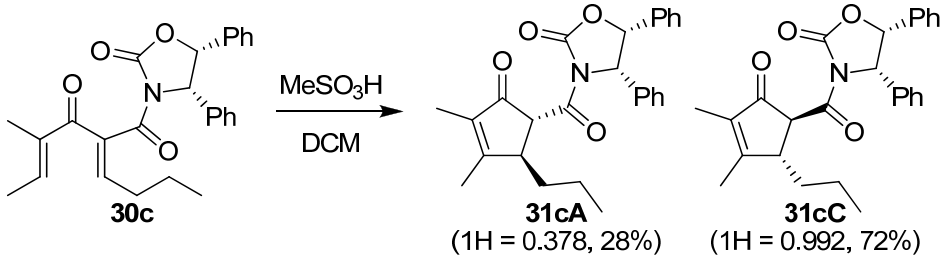
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

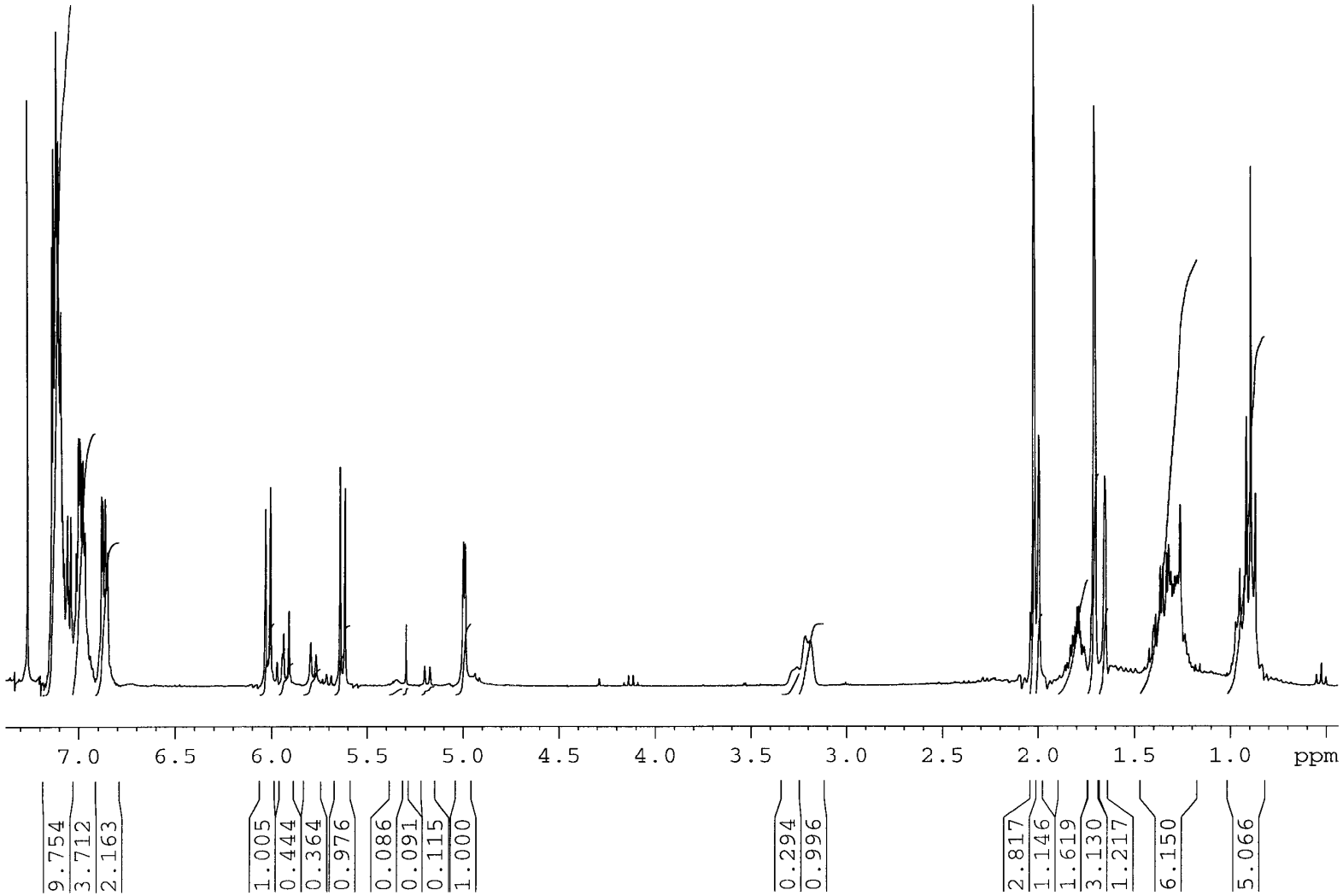
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 7





Current Data Parameters
NAME dksedk
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050504
Time 14.54
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 161.3
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

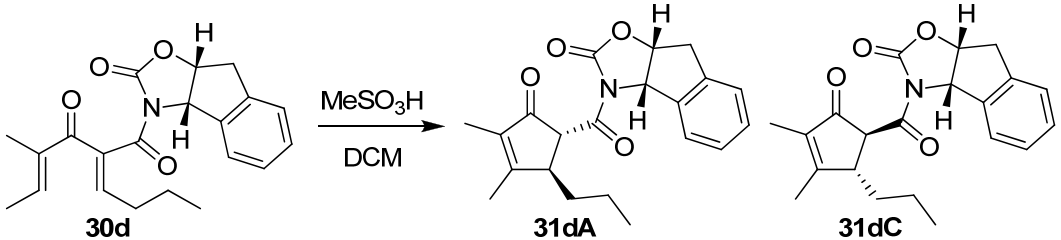
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

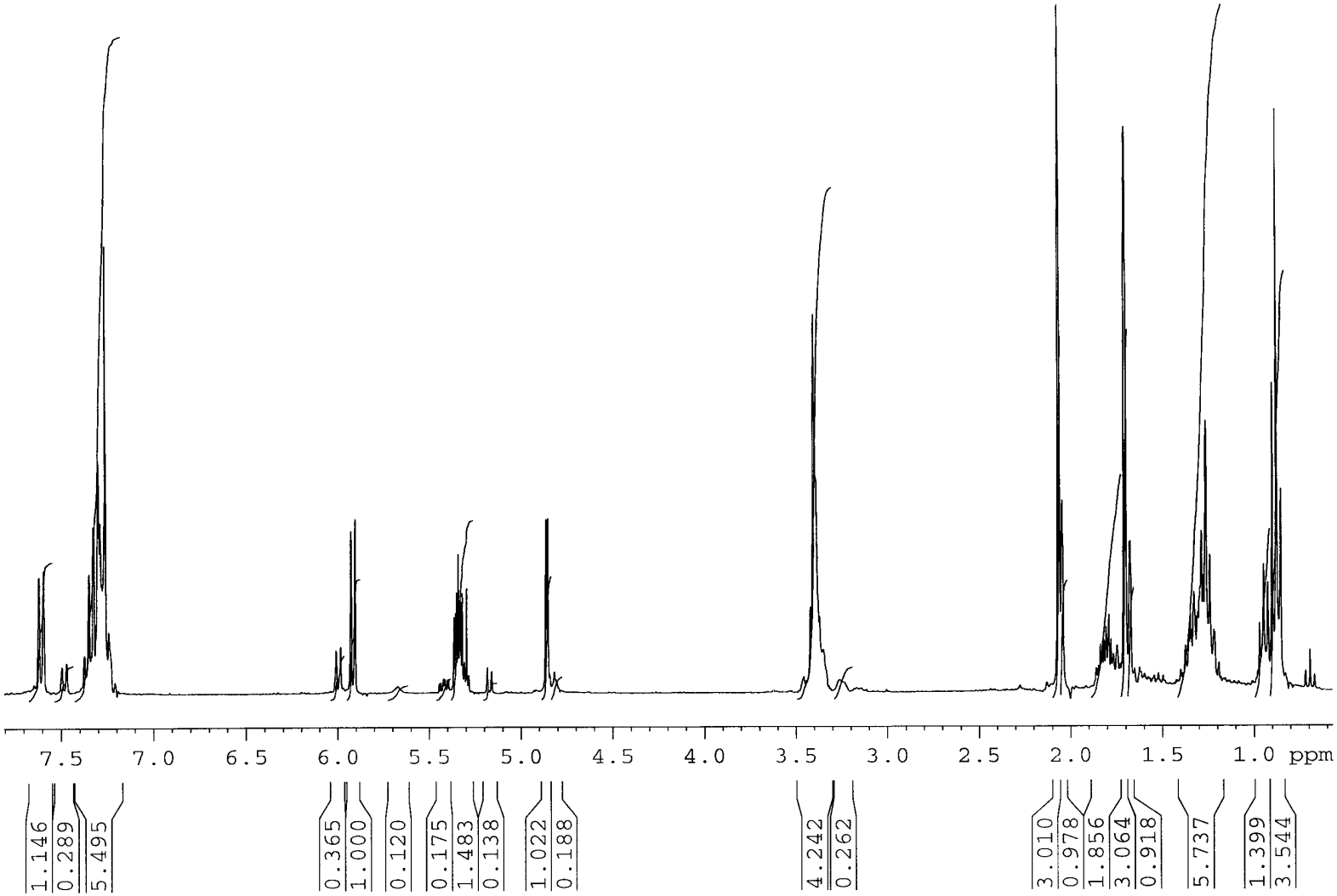
Table 8: Entry 8



30d

31dA
(1H = 0.338, 24%)

31dC
(1H = 1.066, 76%)



Current Data Parameters
NAME dksedk
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050504
Time 15.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

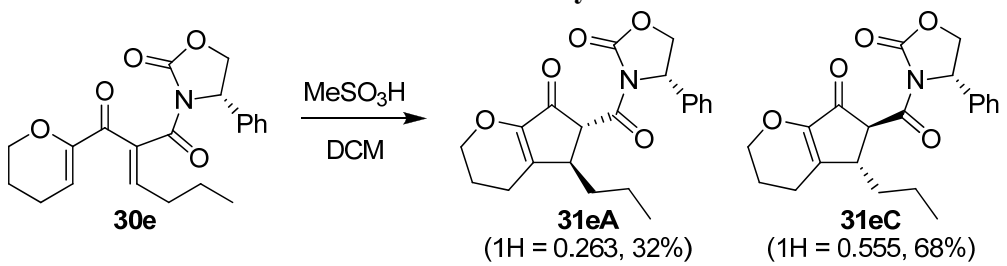
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

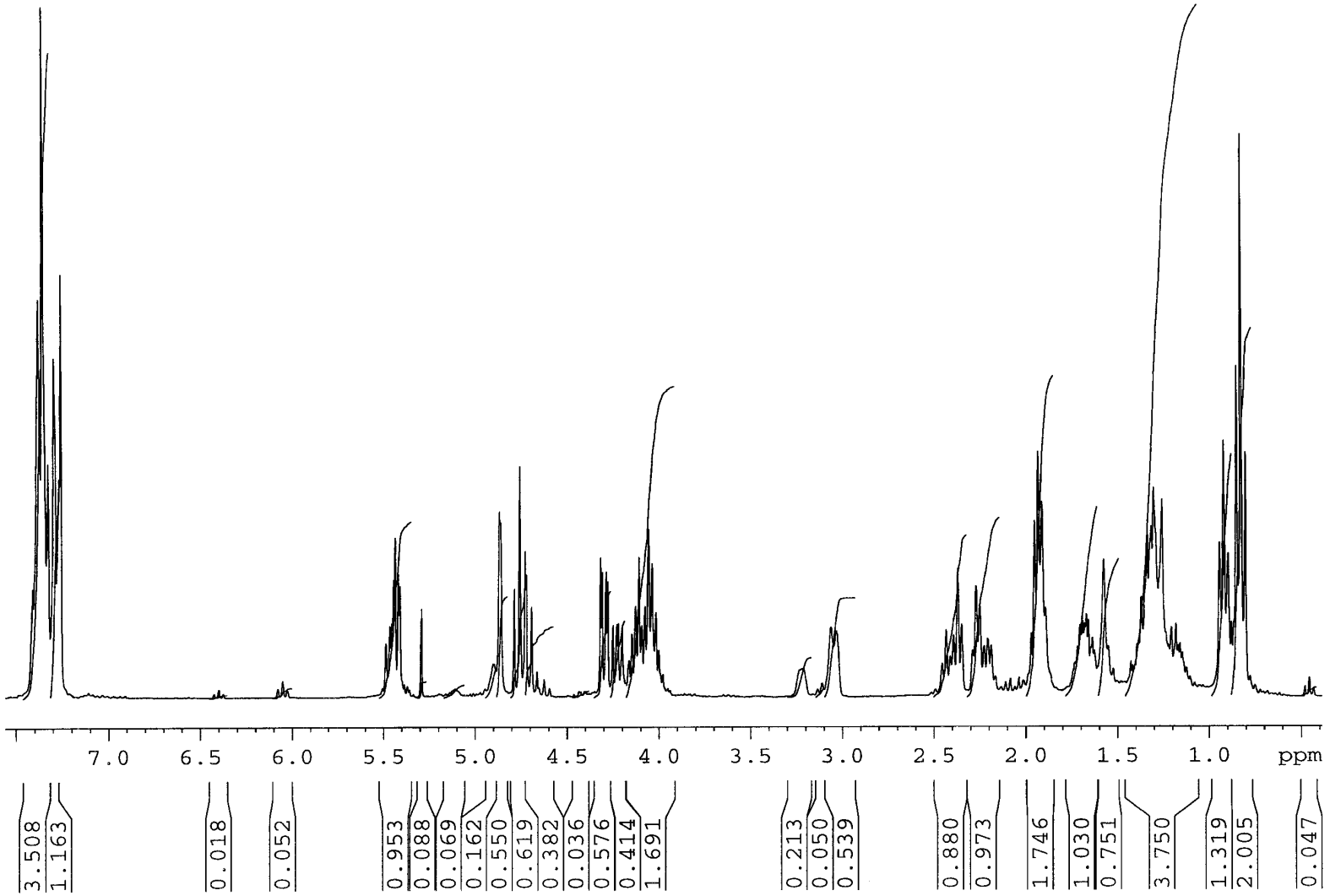
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 9





Current Data Parameters
NAME dksedk
EXPNO 86
PROCNO 1

F2 – Acquisition Parameters
Date_ 20051213
Time 15.42
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 143.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

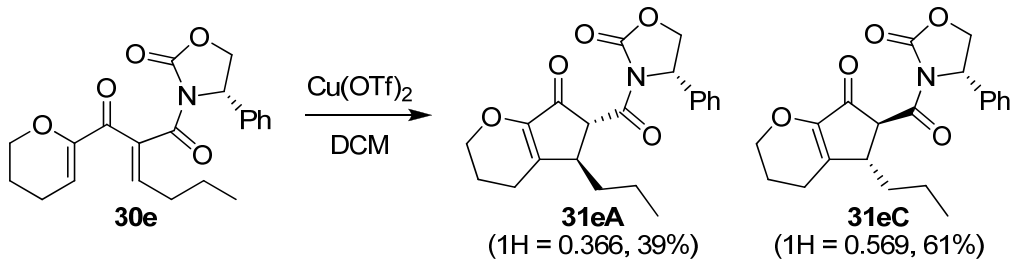
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

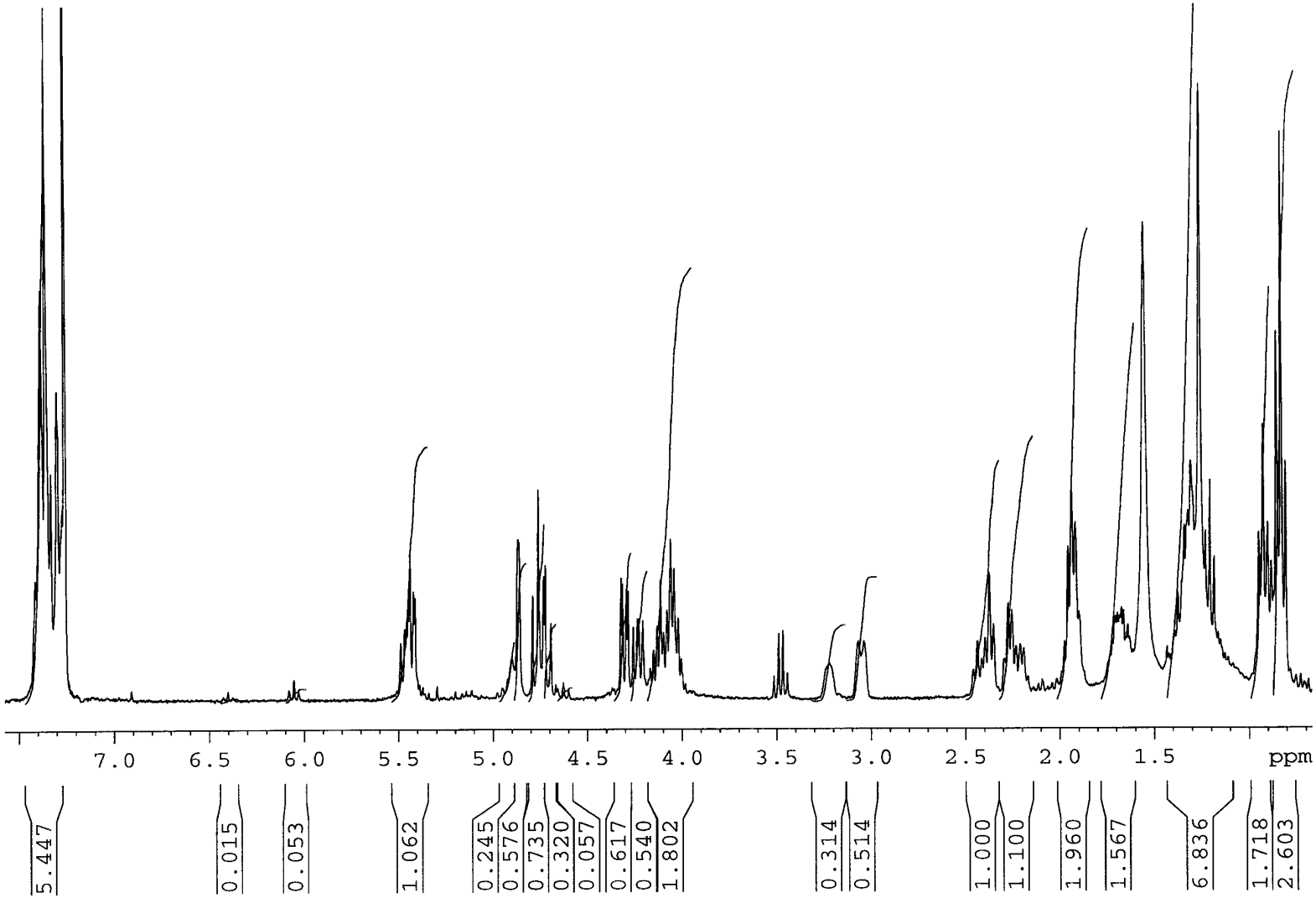
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 10





Current Data Parameters
NAME dksedk
EXPNO 88
PROCNO 1

F2 – Acquisition Parameters
Date_ 20051214
Time 10.34
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 456.1
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

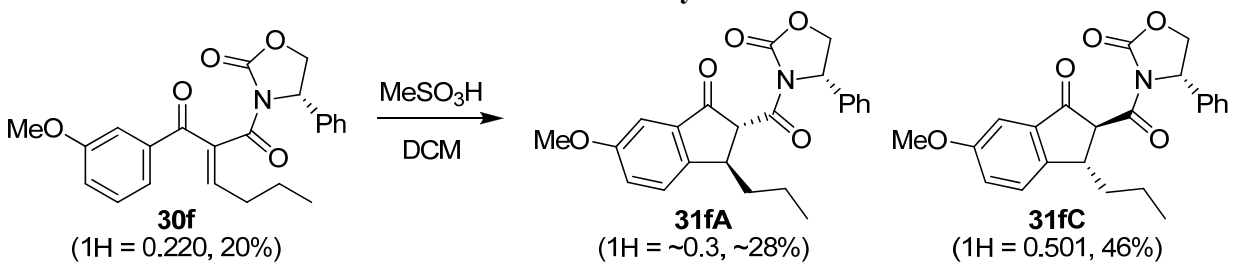
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

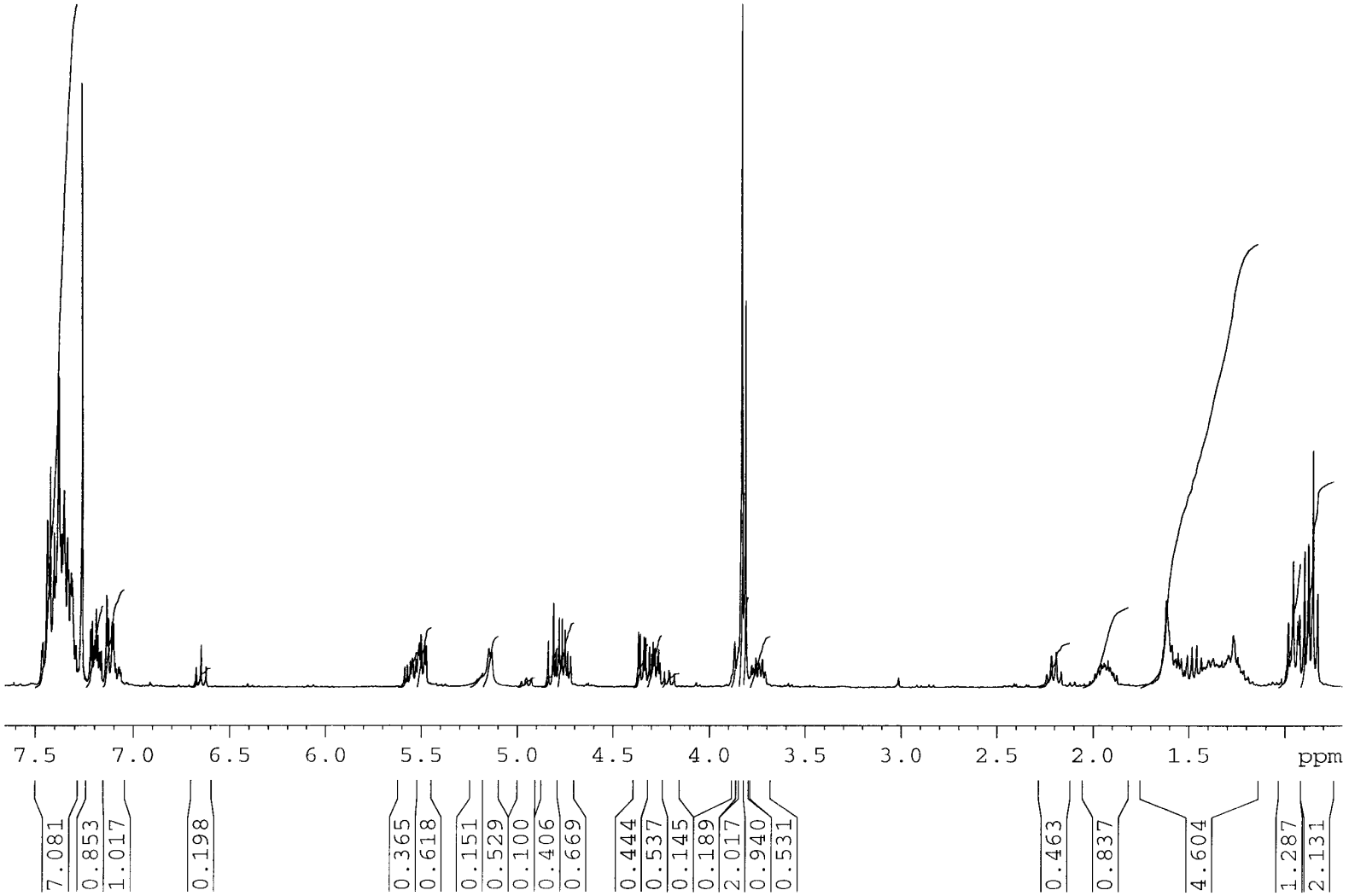
Table 8: Entry 11



30f
(1H = 0.220, 20%)

31fA
(1H = ~0.3, ~28%)

31fC
(1H = 0.501, 46%)



Current Data Parameters
NAME dksedk
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050520
Time 12.58
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 322.5
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

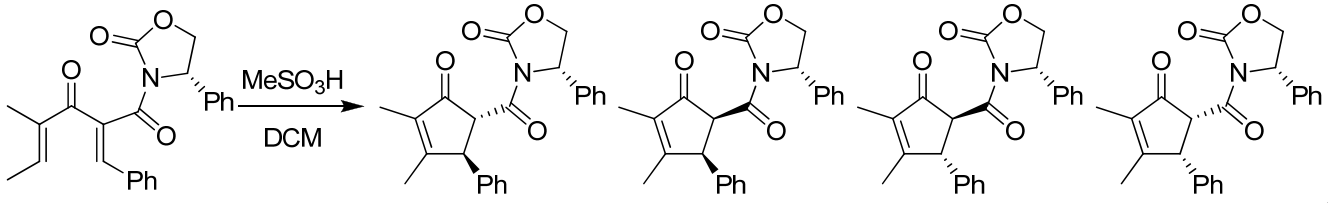
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

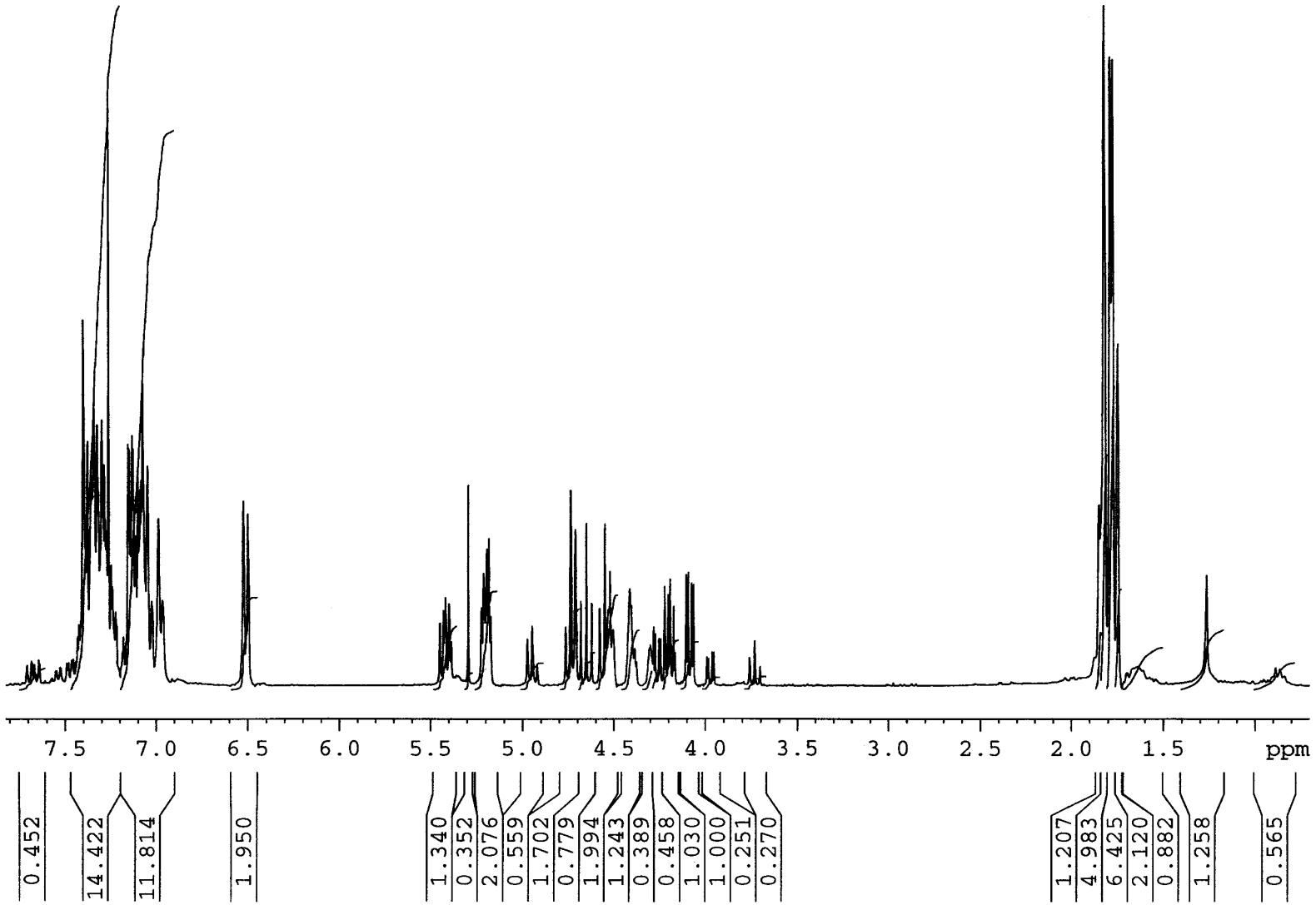
F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 12



30g → **31gA** (1H = 0.725, 31%) + **31gB** (1H = 0.989, 41%) + **31gC** (1H = 0.397, 17%) + **31gD** (1H = 0.261, 11%)



Current Data Parameters
NAME dksedk
EXPNO 4
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050517
Time 14.06
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 114
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 13

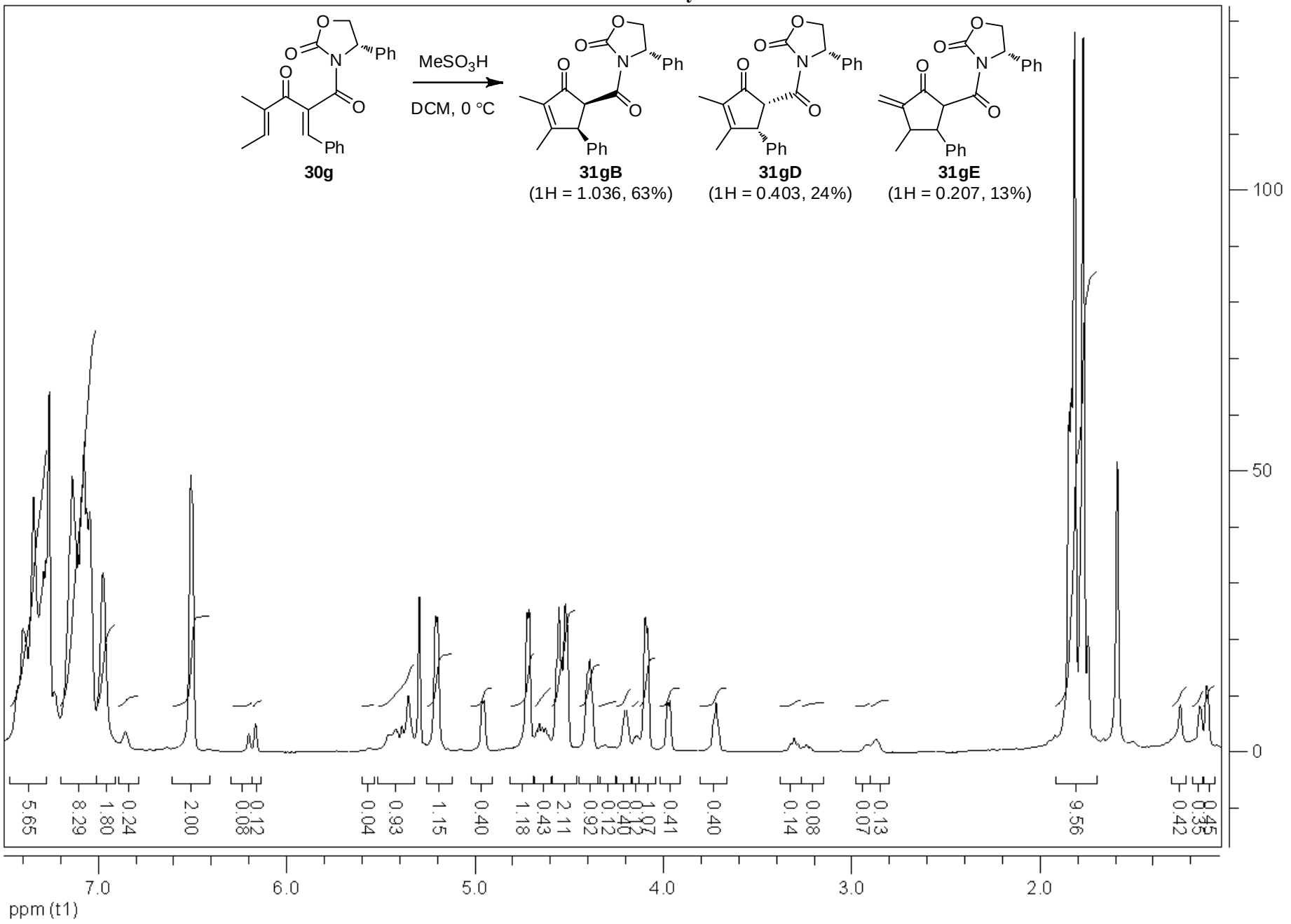
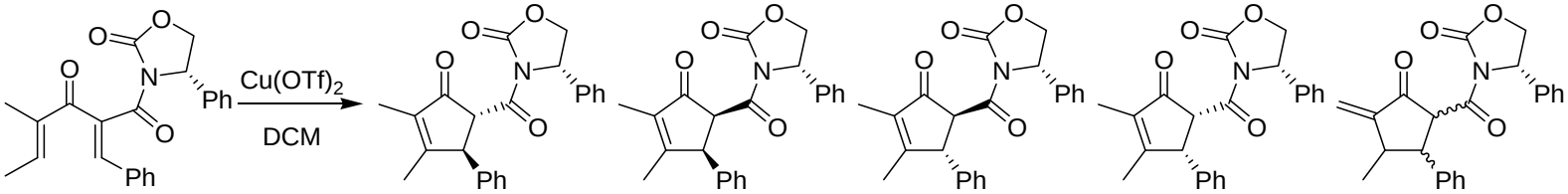
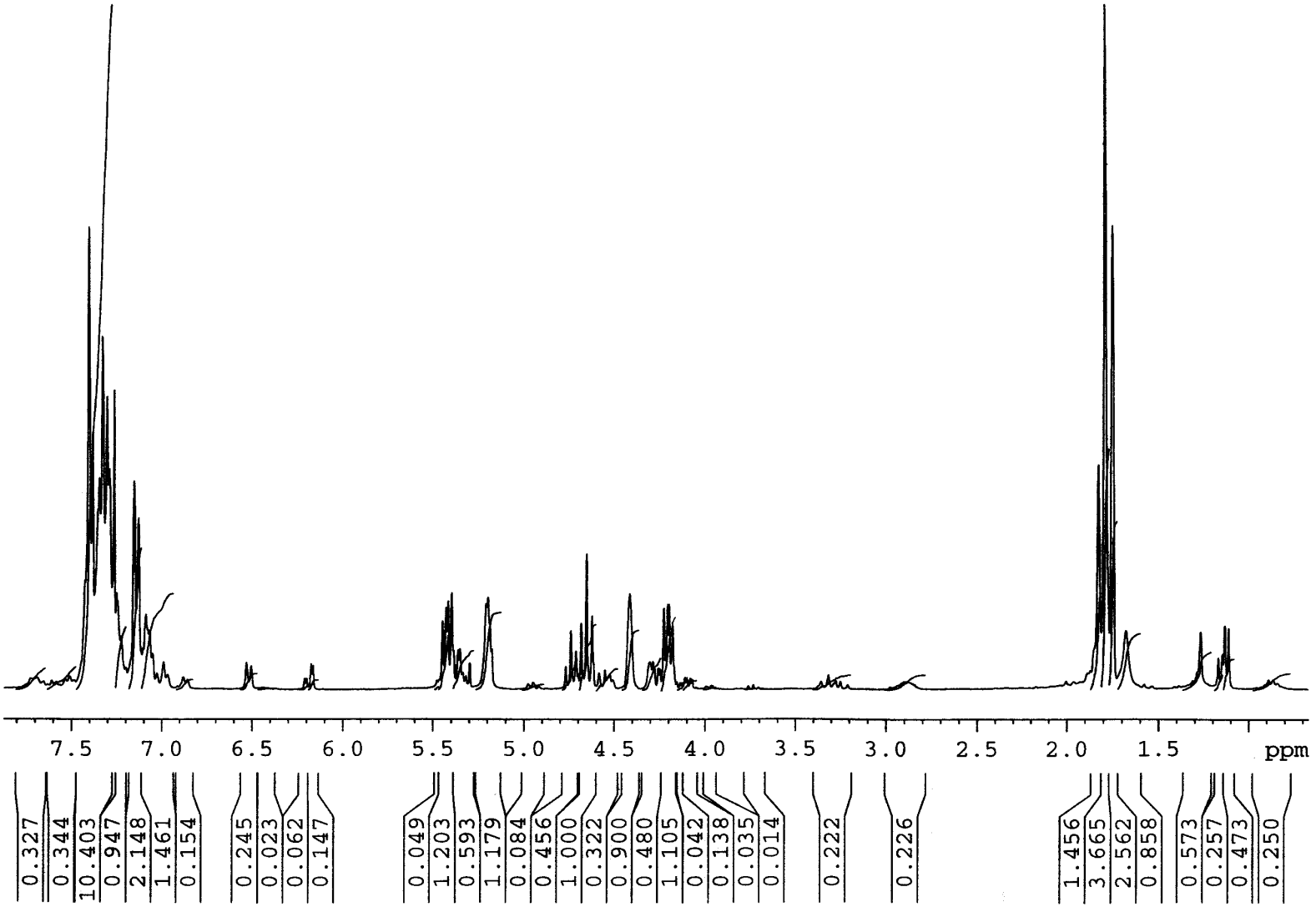


Table 8: Entry 14



30g

31gA (1H = 0.900, 60%) **31gB** (1H = 0.128, 8%) **31gC** (1H = 0.240, 16%) **31gD** (1H = 0.025, 2%) **31gE** (1H = 0.219, 14%)



Current Data Parameters
NAME dksedk
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050705
Time 14.29
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 181
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

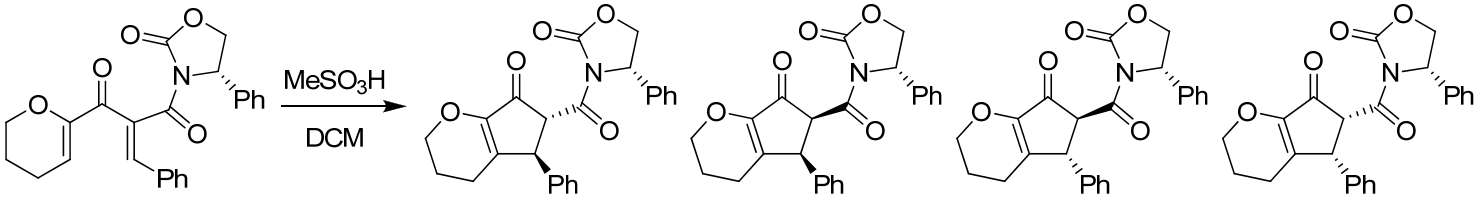
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 17



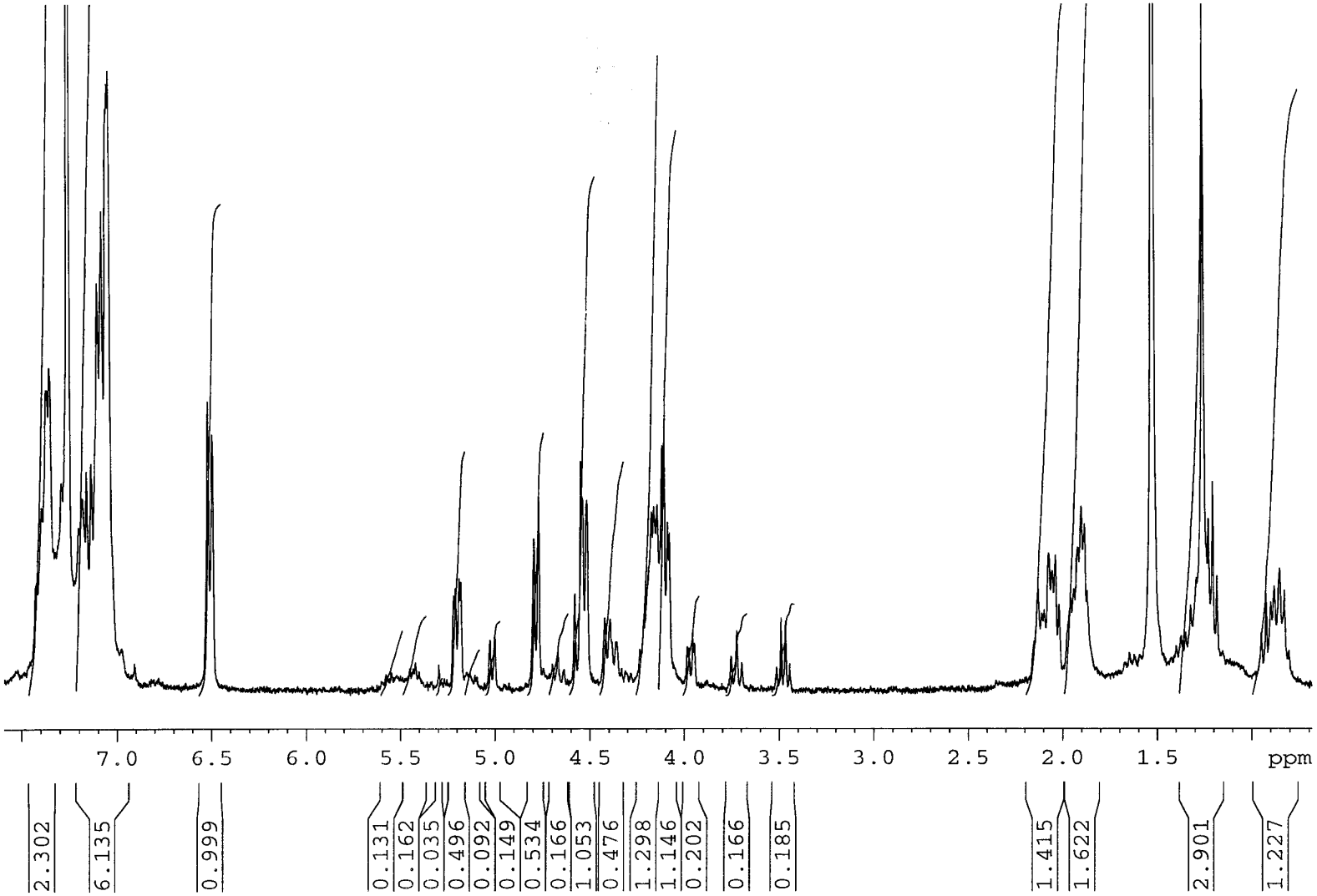
30h

31hA
(1H = 0.055, 7%)

31hB
(1H = 0.514, 68%)

31hC
(1H = 0.018, 2%)

31hD
(1H = 0.172, 23%)



Current Data Parameters
NAME dksedk
EXPNO 77
PROCNO 1

F2 – Acquisition Parameters
Date_ 20051207
Time 16.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

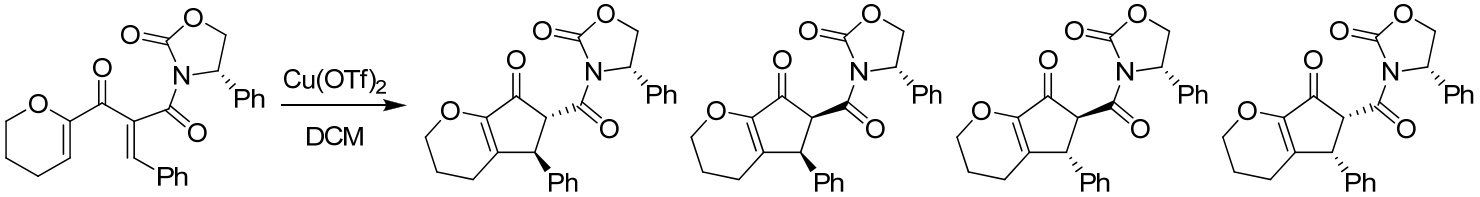
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

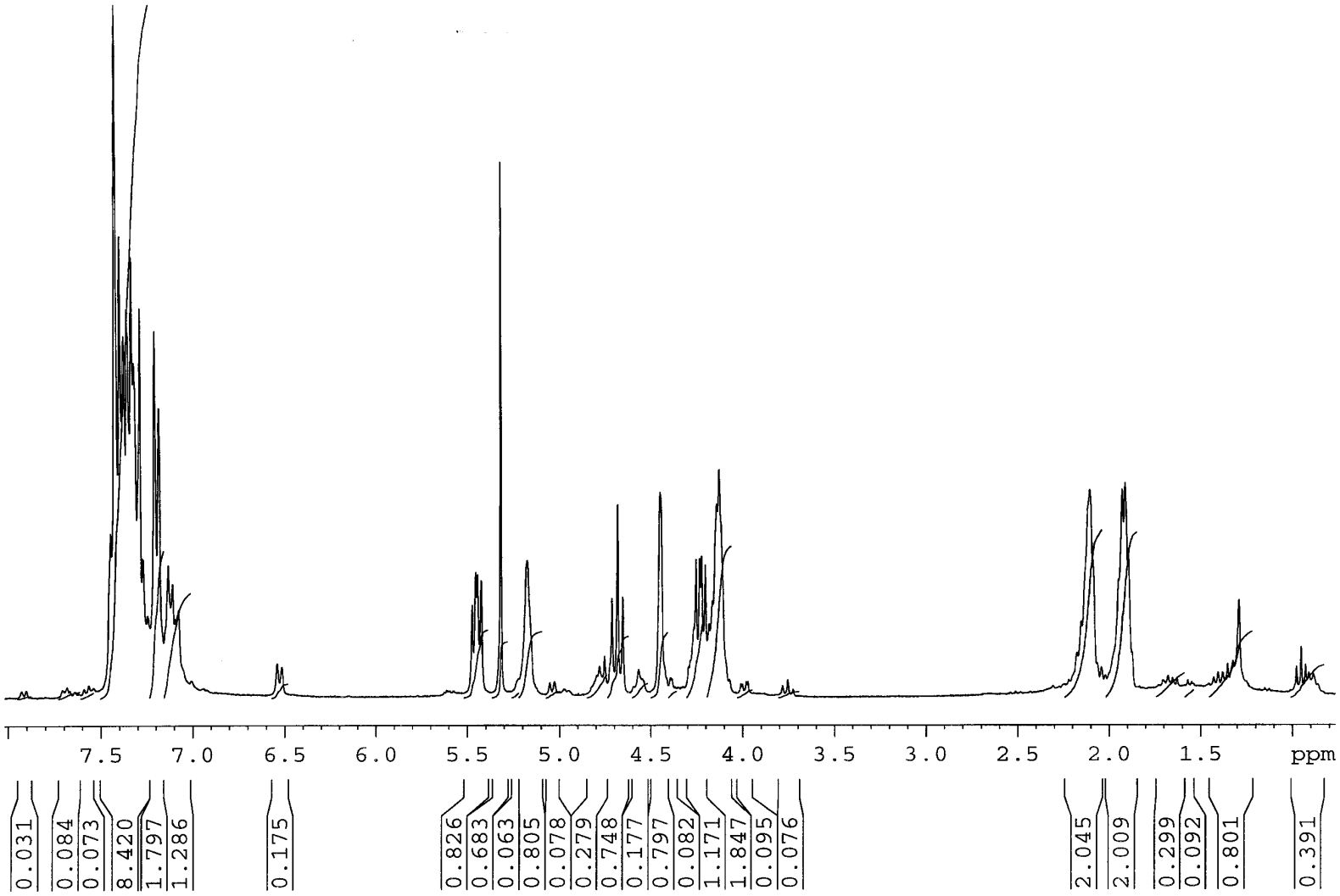
F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 18



30h → **31hA** (1H = 0.713, 70%) | **31hB** (1H = 0.088, 9%) | **31hC** (1H = 0.135, 13%) | **31hD** (1H = 0.083, 8%)



Current Data Parameters
NAME dksedk
EXPNO 9
PROCNO 1

F2 – Acquisition Parameters
Date_ 20060119
Time 17.07
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 101.6
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

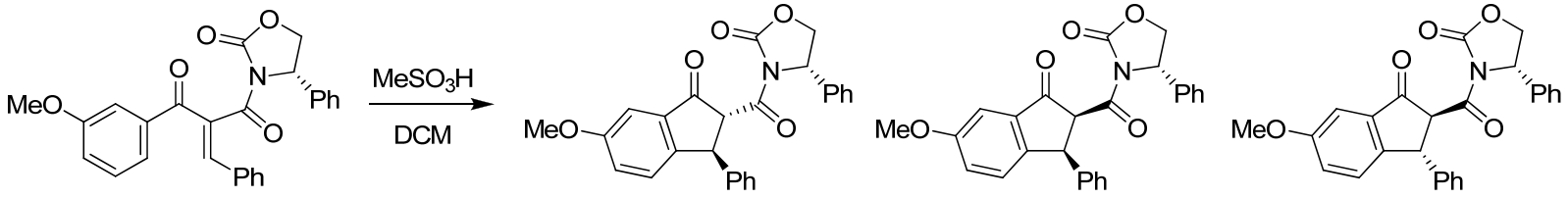
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.2319515 MHz

F1 – Acquisition parameters
ND0 1
TD 512
SFO1 300.1314 MHz
FIDRES 0.586194 Hz
SW 1.000 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.2300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 19

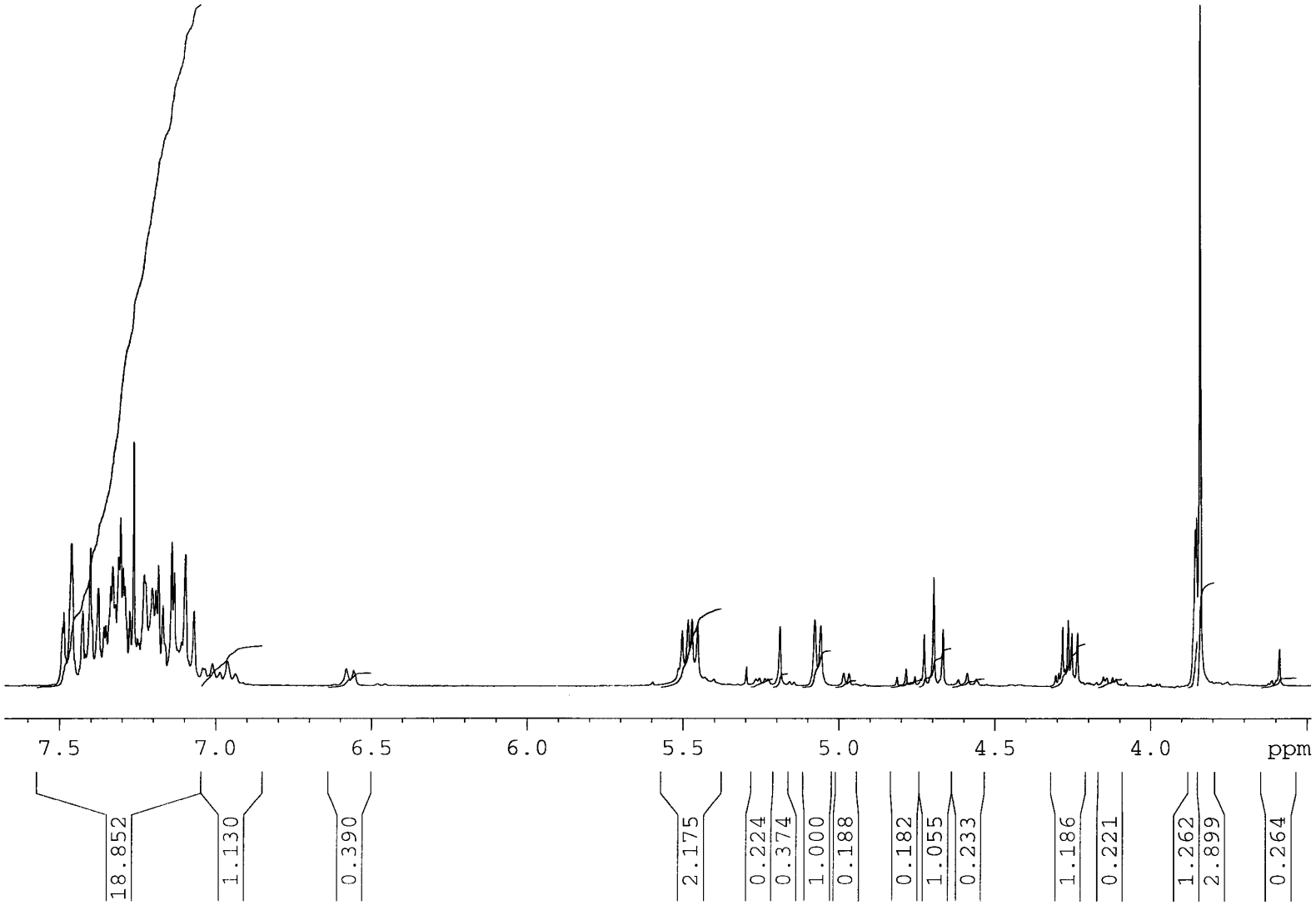


30i

31iA
(1H = 1.005, 71%)

31iB
(1H = 0.212, 15%)

31iC
(1H = 0.185, 13%)



Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050518
Time 14.06
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 143.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

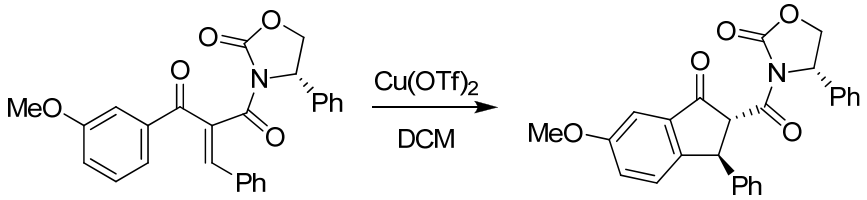
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

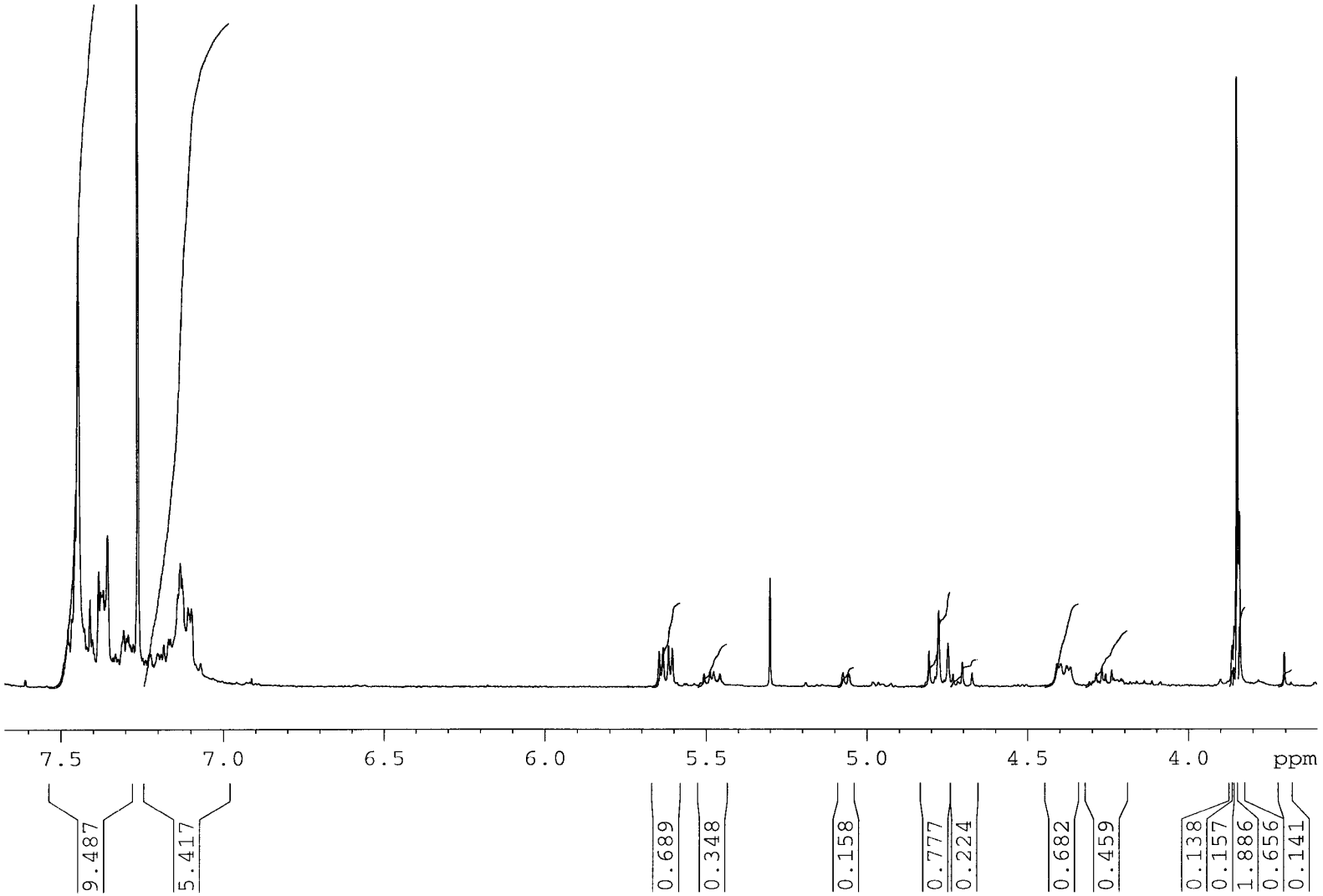
F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 20



30i
(1H = 0.716, 72%)

31iA
(1H = 0.183, 18%)



Current Data Parameters
NAME dksedk
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050525
Time 13.50
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

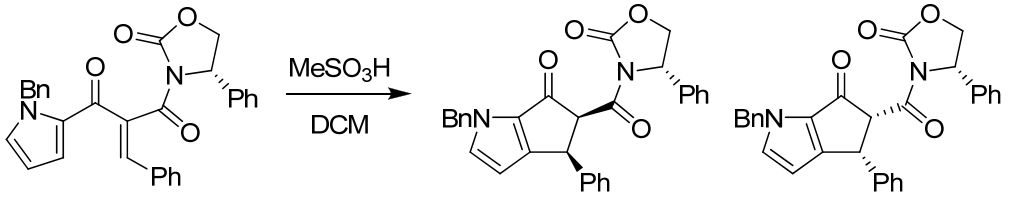
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

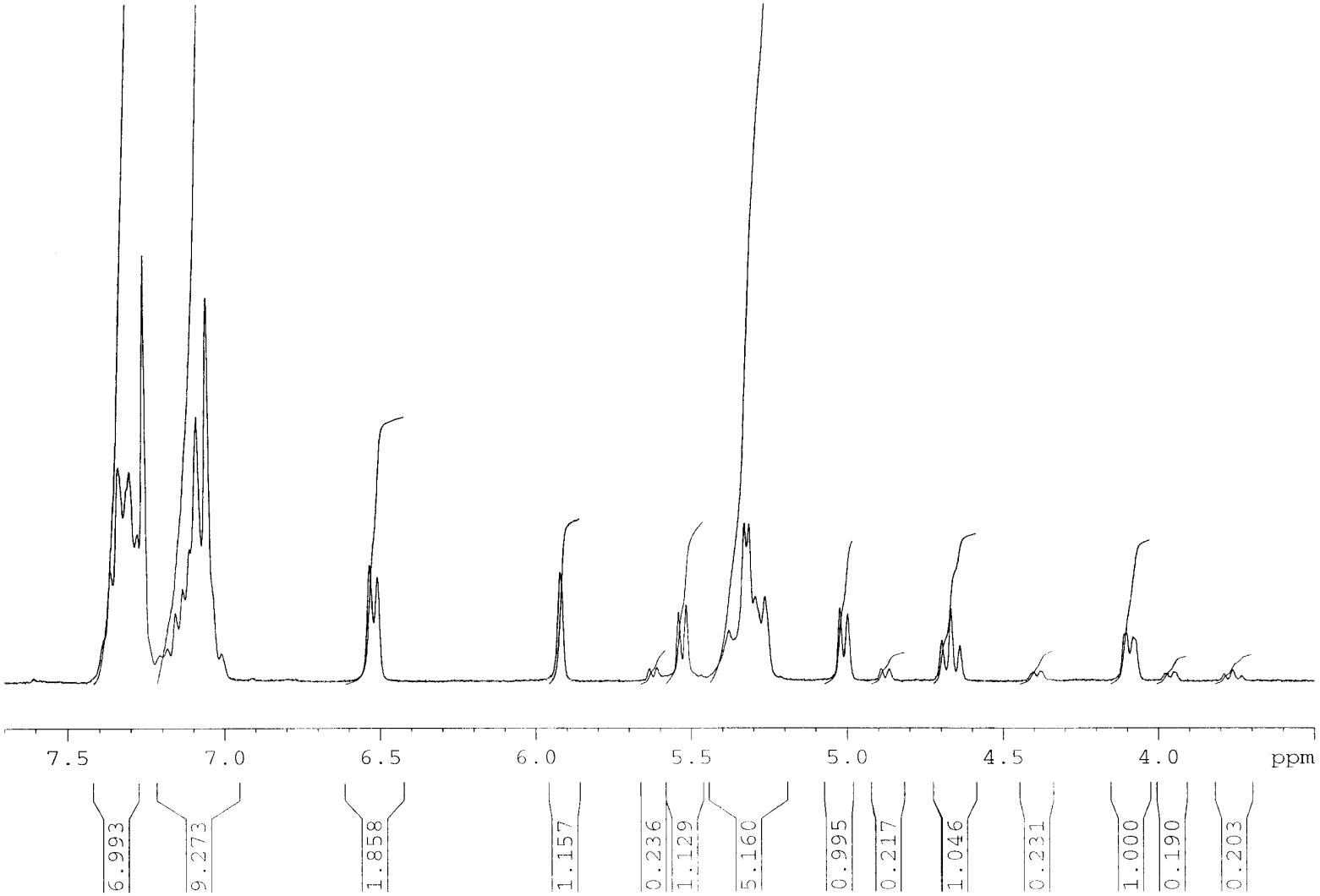
F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 21



30j | **31jB** (1H = 1.009, 82%) | **31jD** (1H = 0.215, 18%)



Current Data Parameters
NAME djsedj
EXPNO 154
PROCNO 1

F2 – Acquisition Parameters
Date_ 20100119
Time 14.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 406.4
DW 111.200 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.2319515 MHz

F1 – Acquisition parameters
ND0 1
TD 512
SFO1 300.1314 MHz
FIDRES 0.586194 Hz
SW 1.000 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.2300183 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.30 Hz

Table 8: Entry 22

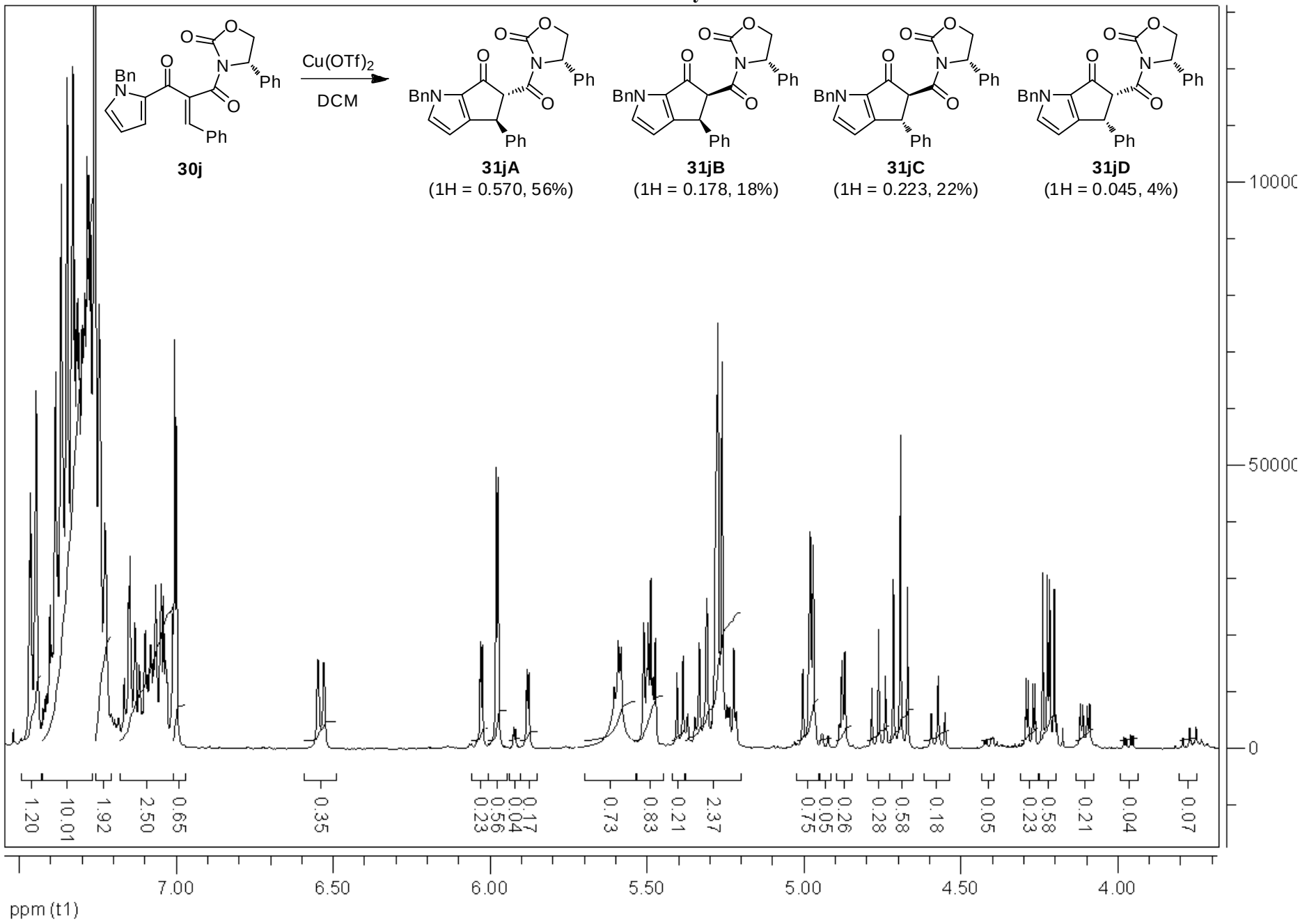
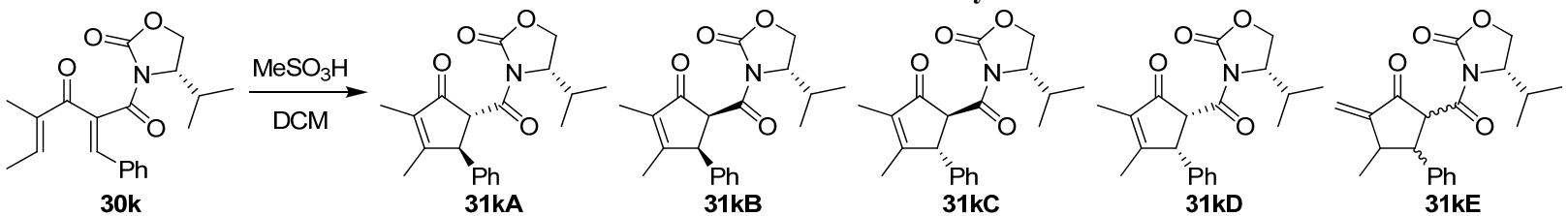


Table 8: Entry 23



30k

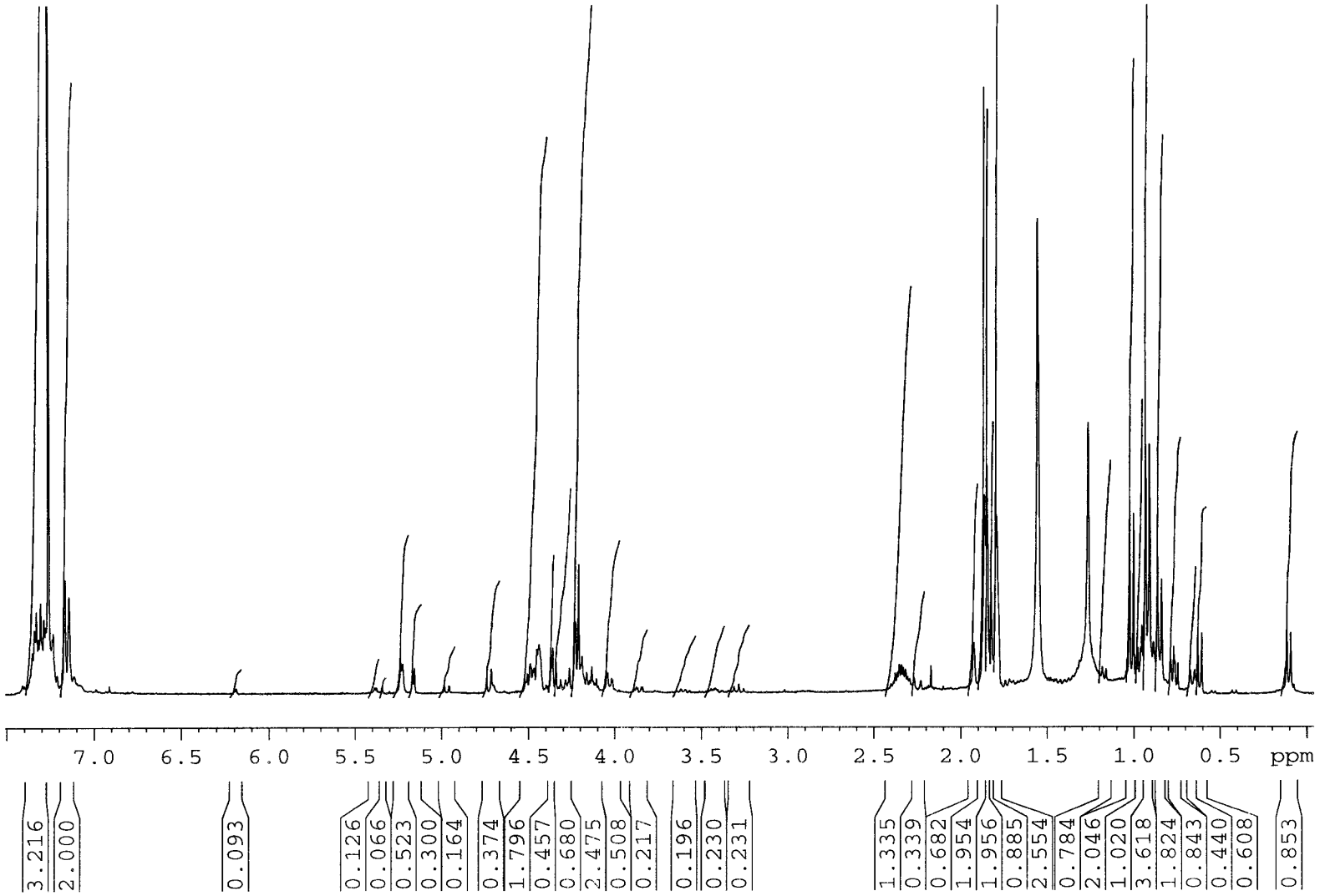
31kA (1H = 0.523, 40%)

31kB (1H = 0.187, 14%)

31kC (1H = 0.300, 23%)

31kD (1H = 0.211, 16%)

31kE (1H = 0.093, 7%)



Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20060531
Time 11.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 106
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 574.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

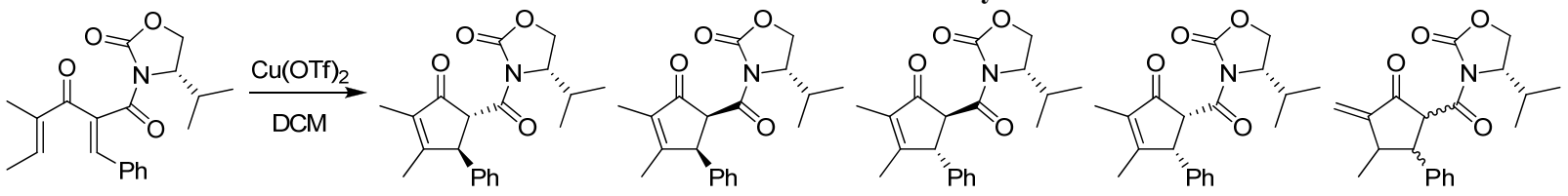
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.2319515 MHz

F1 – Acquisition parameters
ND0 1
TD 512
SFO1 300.1314 MHz
FIDRES 0.586194 Hz
SW 1.000 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.2300165 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 8: Entry 24



30k

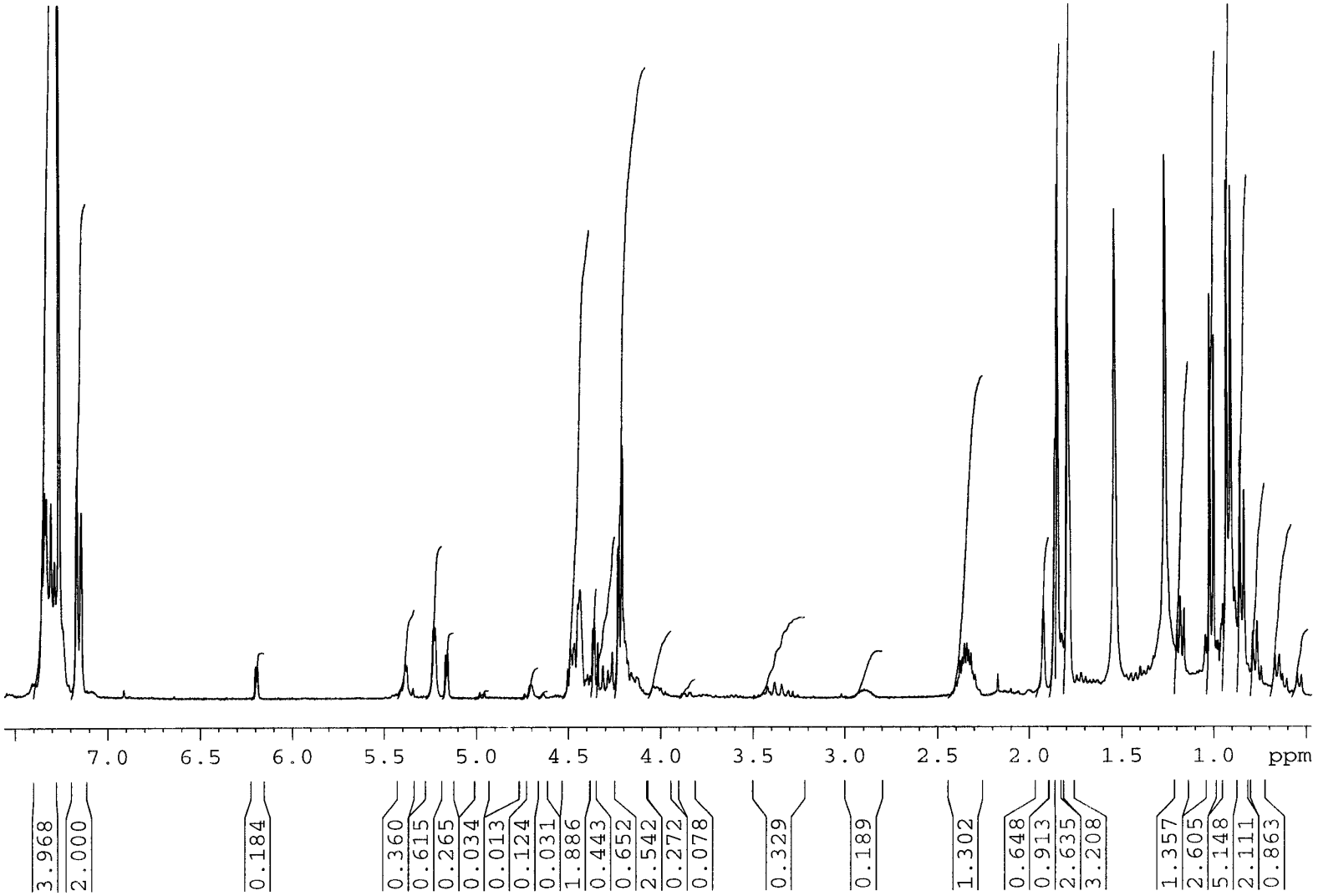
31kA (1H = 0.615, 51%)

31kB (1H = 0.069, 6%)

31kC (1H = 0.265, 22%)

31kD (1H = 0.056, 5%)

31kE (1H = 0.187, 16%)



Current Data Parameters
NAME dksedk
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20060531
Time 11.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 133
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

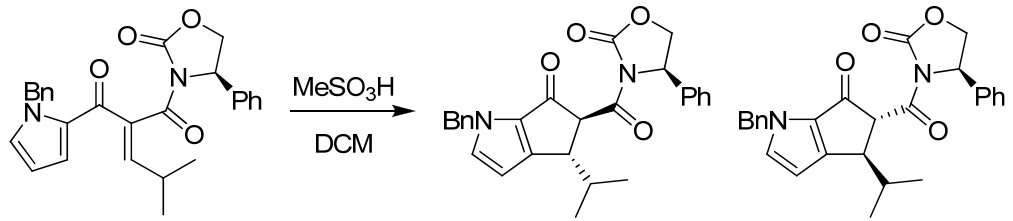
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.2319515 MHz

F1 – Acquisition parameters
ND0 1
TD 512
SFO1 300.1314 MHz
FIDRES 0.586194 Hz
SW 1.000 ppm
FnMODE undefined

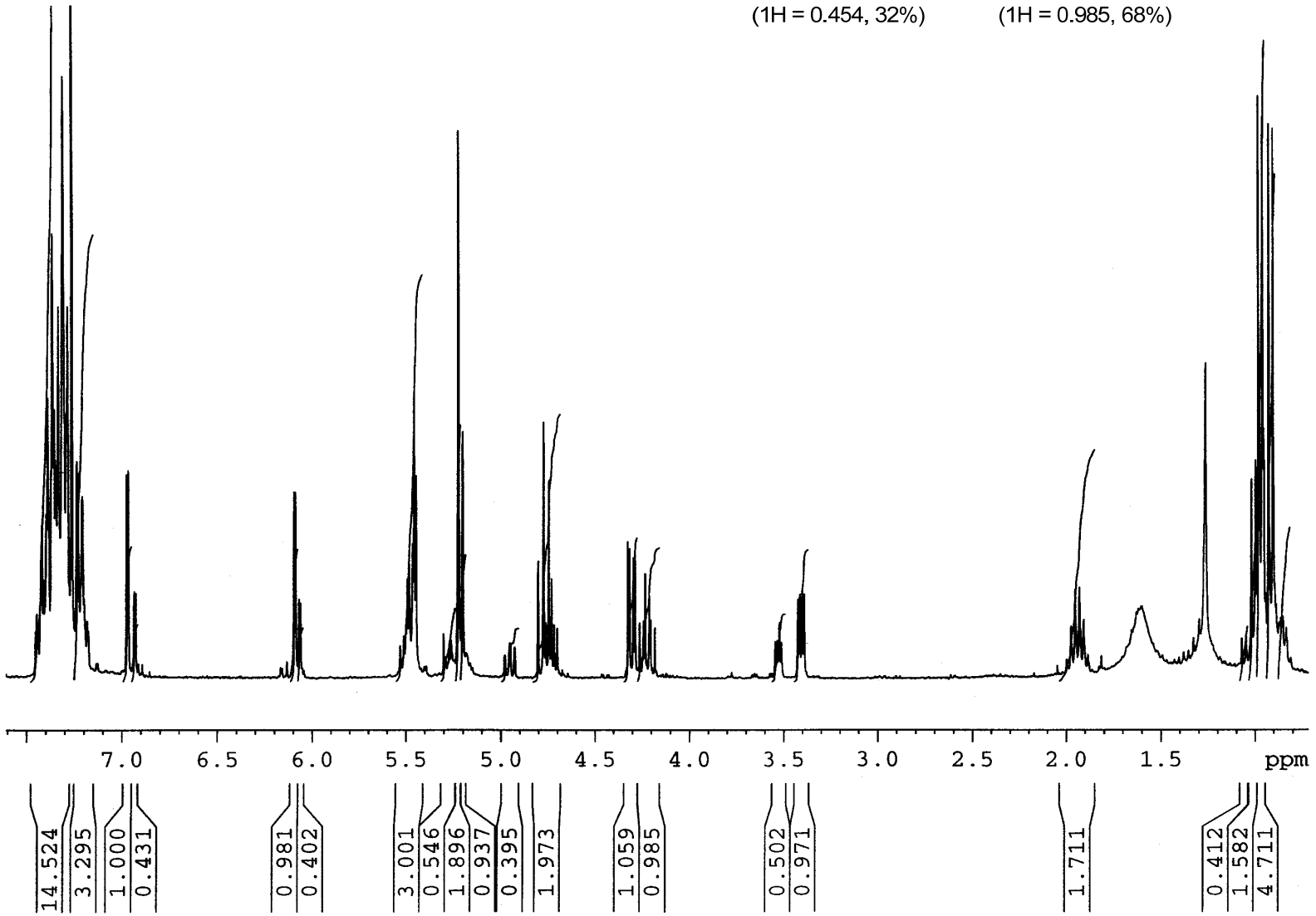
F2 – Processing parameters
SI 16384
SF 300.2300168 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 4: Entry 1



36 → **37A** (1H = 0.454, 32%) + **37C** (1H = 0.985, 68%)



Current Data Parameters
NAME dksedk
EXPNO 4
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050707
Time 13.29
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 256
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

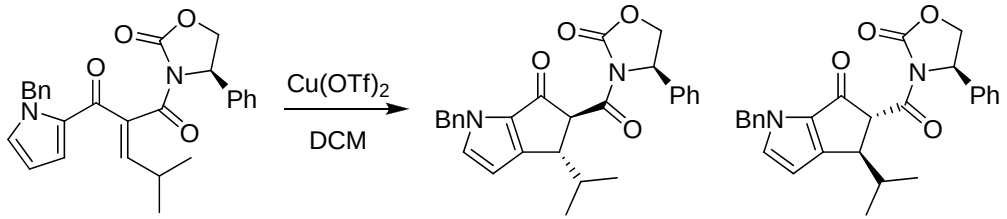
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

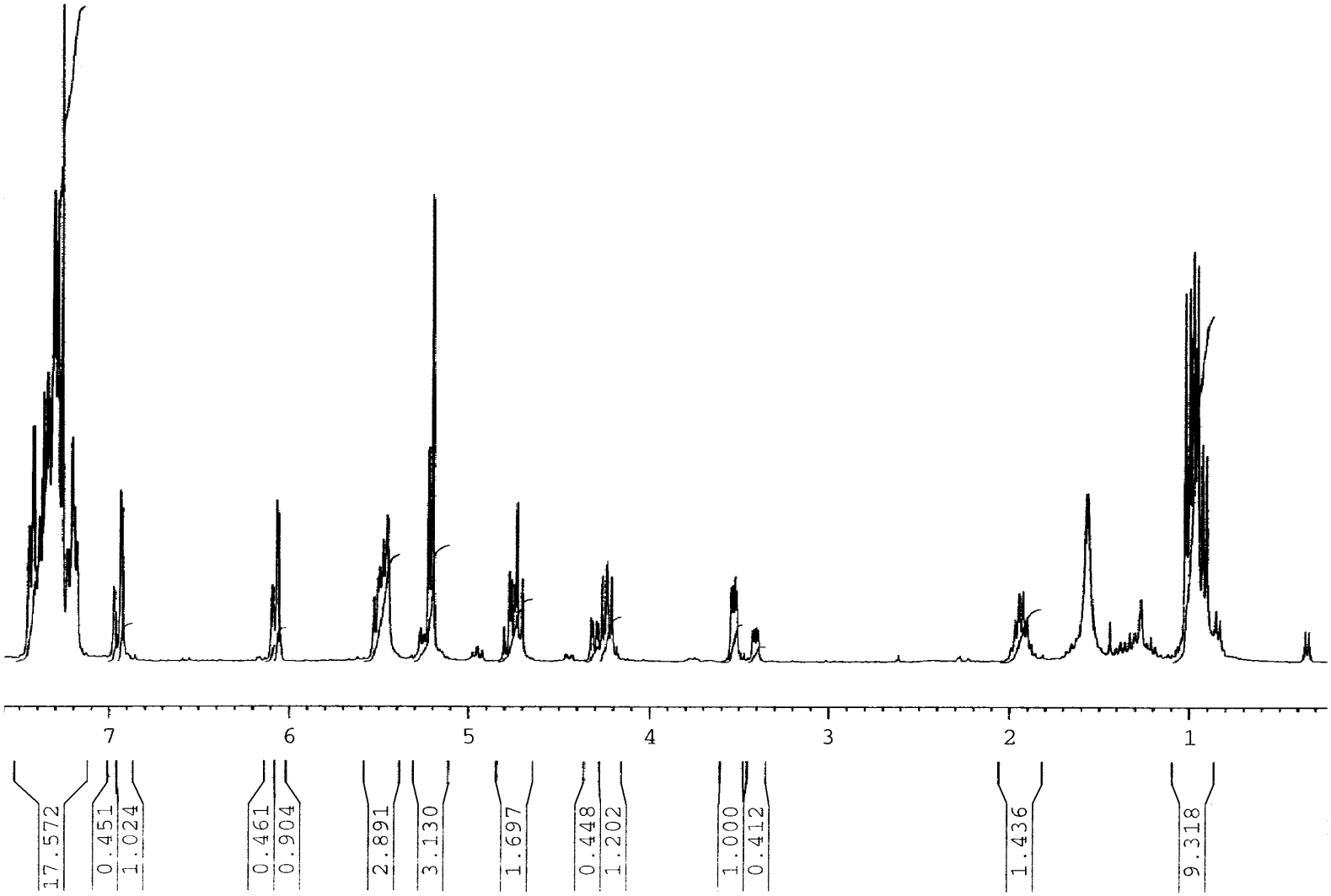
F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 4: Entry 2



36 | **37A** (1H = 1.033, 70%) | **37C** (1H = 0.443, 30%)



Current Data Parameters
NAME dksedk
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date_ 20040429
Time 12.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 287.4
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

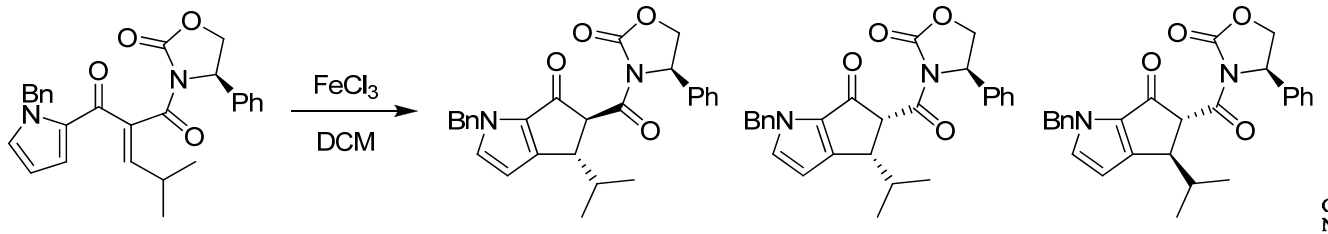
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

Table 4: Entry 3

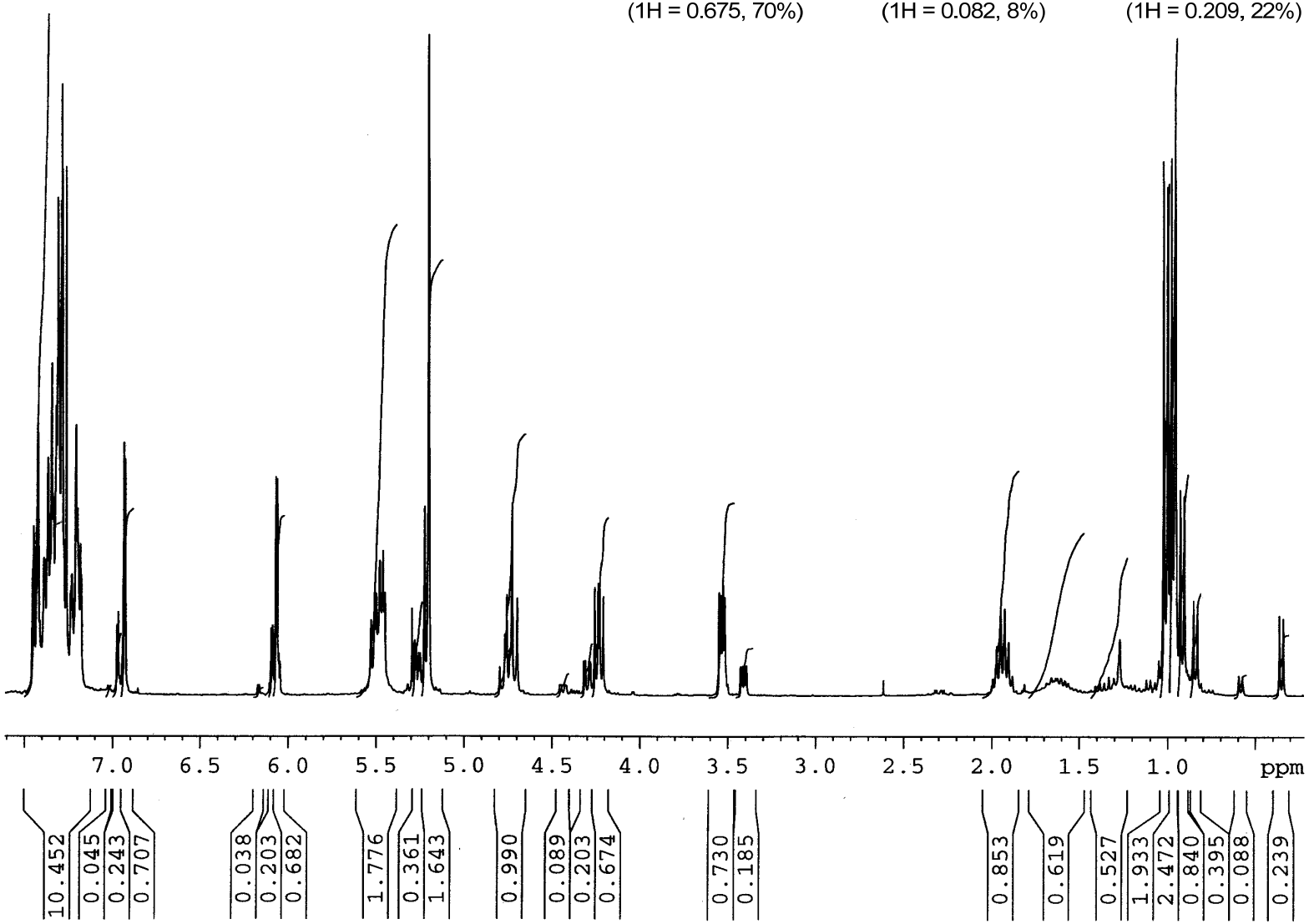


36

37A
(1H = 0.675, 70%)

37B
(1H = 0.082, 8%)

37C
(1H = 0.209, 22%)



Current Data Parameters
NAME dksedk
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050603
Time 13.36
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 114
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz