Supplementary Material

Ring-closing metathesis studies in the context of the formal synthesis of the marine macrolide (–)-callyspongiolide

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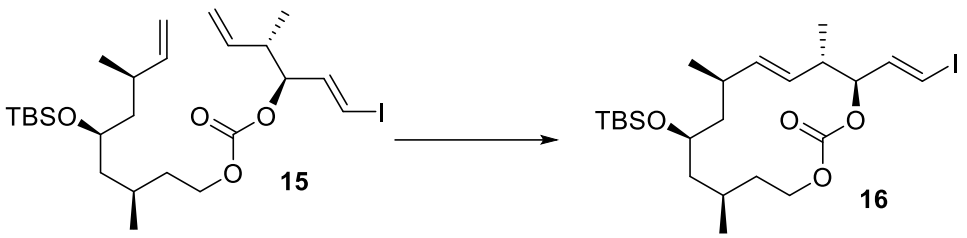

Entry	Catalyst	Solvent	Dilution [mM]	t (ºC)	T (h)	Yield (%) of 16
1	G II (20 mol%)	CH_2Cl_2	2.50	45	24	-
2	G II (30 mol%)	Toluene	2.50	80	15	5
3	HG II (20 mol%)	Toluene	0.42	80	24	22
4	HG II (30 mol%)	Toluene	0.42	110	48	25
5	HG II (40 mol%)	Toluene	0.25	110	96	-
6	HG II (30 mol%)	Toluene	2,50	80	15	20
7	HG II (30 mol%)	Toluene	0.88[a]	80	21	20
8	HG II (30 mol%)	Toluene	0.88[b]	80	22	13
9	HG II (50 mol%)	Toluene	0.88[c]	80	70	30

Table S1. *a*) Initial dilution: 2.5 mM; addition of 30 mol% HG II catalyst dissolved in 20 mL of toluene for 7 h (3 mL/h syringe pump). *b*) Initial dilution: 1.0 mM; addition of 30 mol% HG II catalyst dissolved in 20 mL of toluene for 10 h (2 mL/h syringe pump). *c*) Initial dilution: 1.0 mM; addition of 30 mol% HG II catalyst dissolved in 10 mL of toluene for 10 h (2 mL/h syringe pump); after 12 and 22 h: addition of 10 mol% HG II dissolved in 5 mL of toluene for 2.5 h (2 mL/h).

S1. ^{1}H- and ^{13}C-NMR spectra of (3*R*,5*R*,7*R*)-5-[(*tert*-butyldimethylsilyl)oxy]-3,7-dimethyl-8-nonen-1-yl (3*S*,4*S*,*E*)-1-iodo-4-methyl-1,5-hexadien-3-yl carbonate (**15**)

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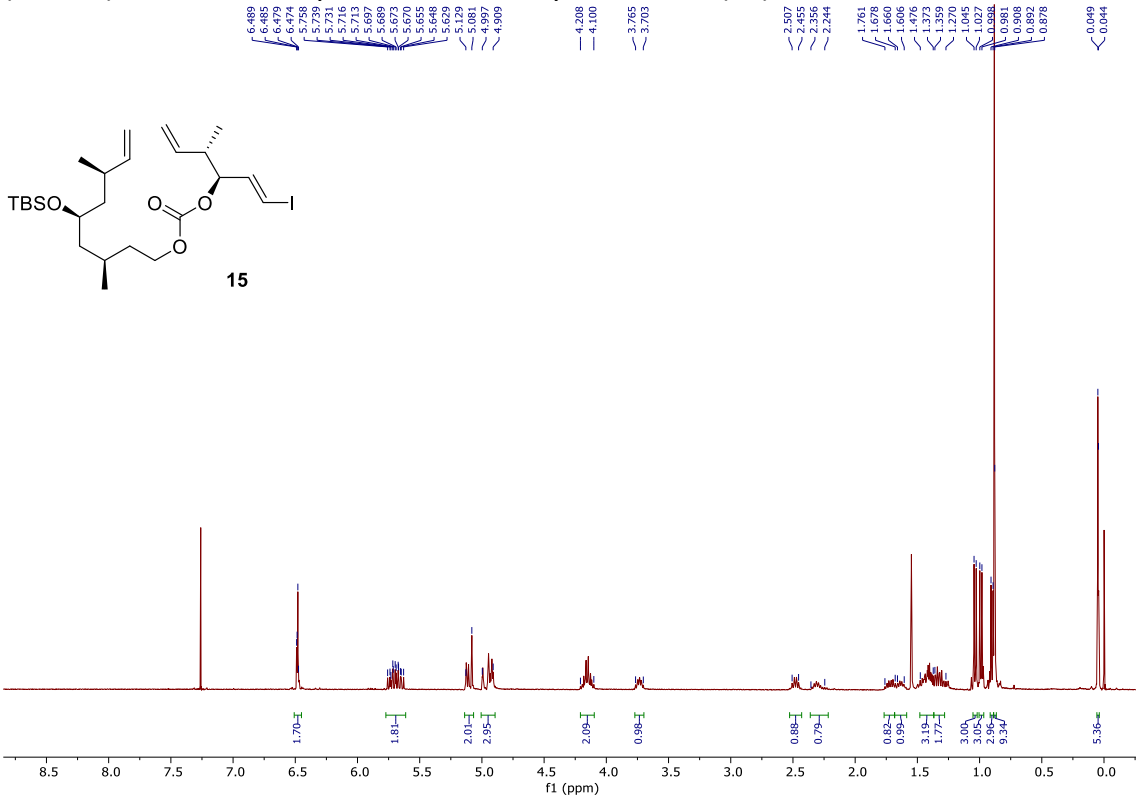
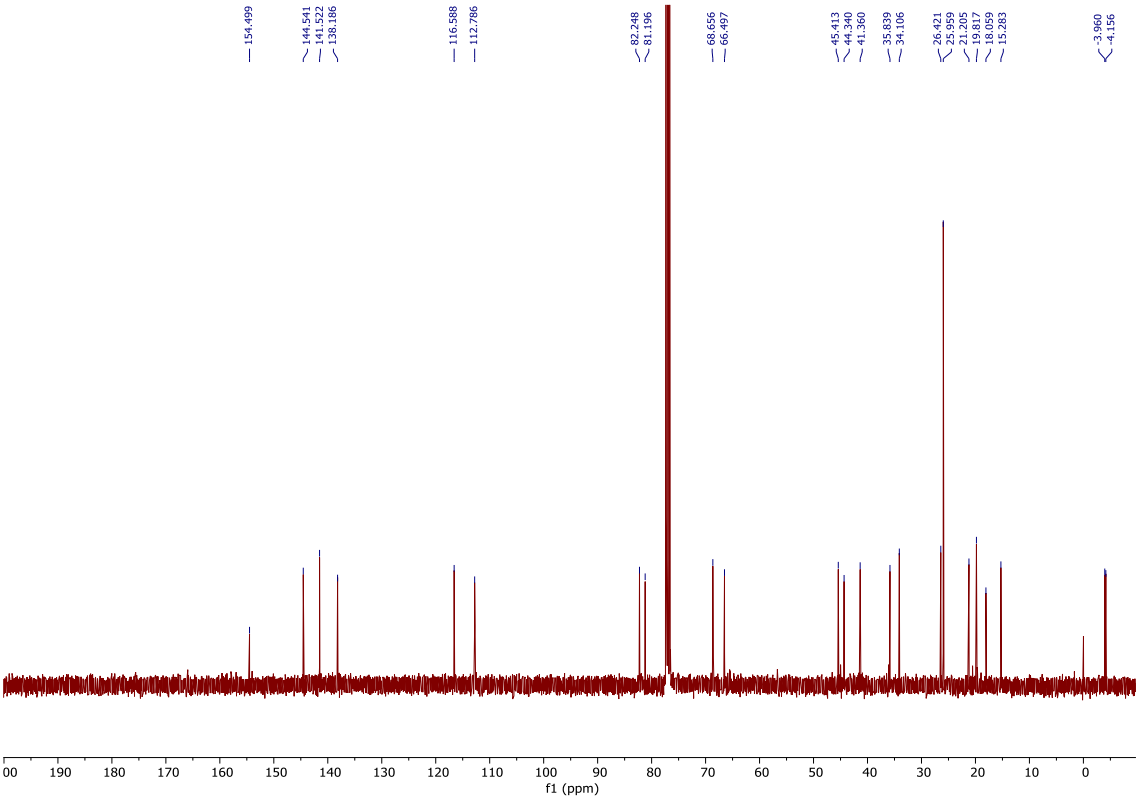
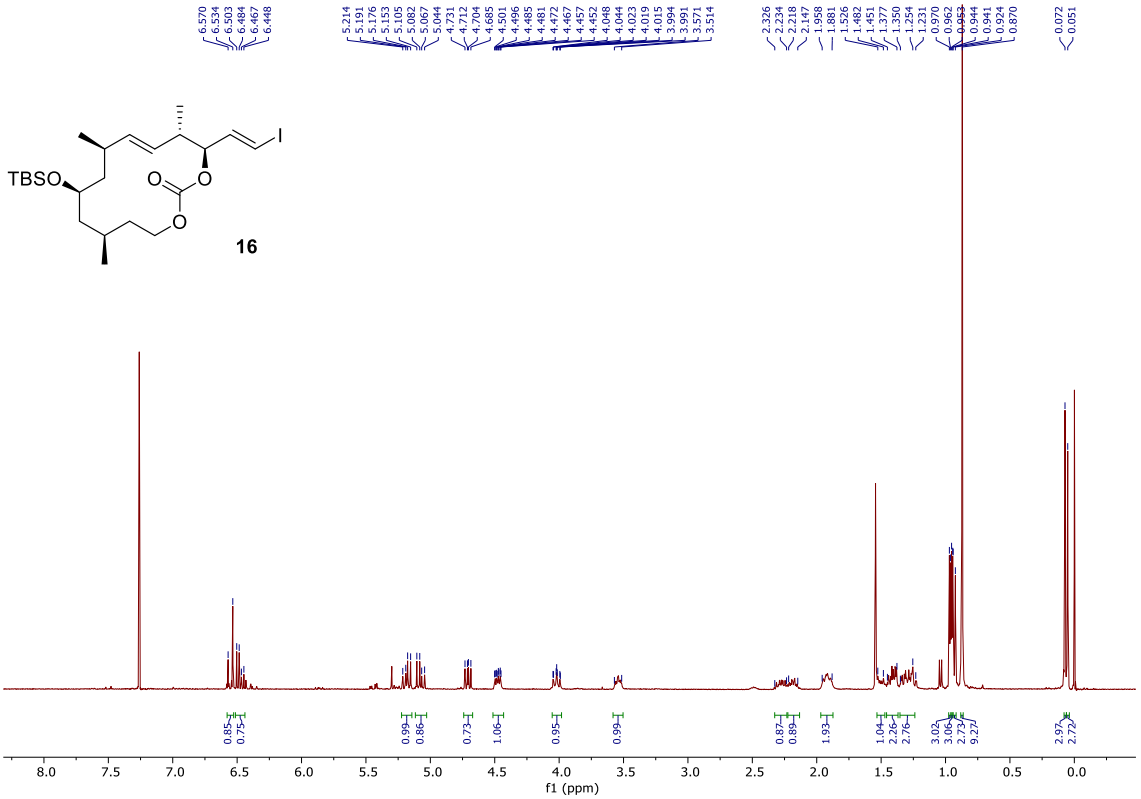
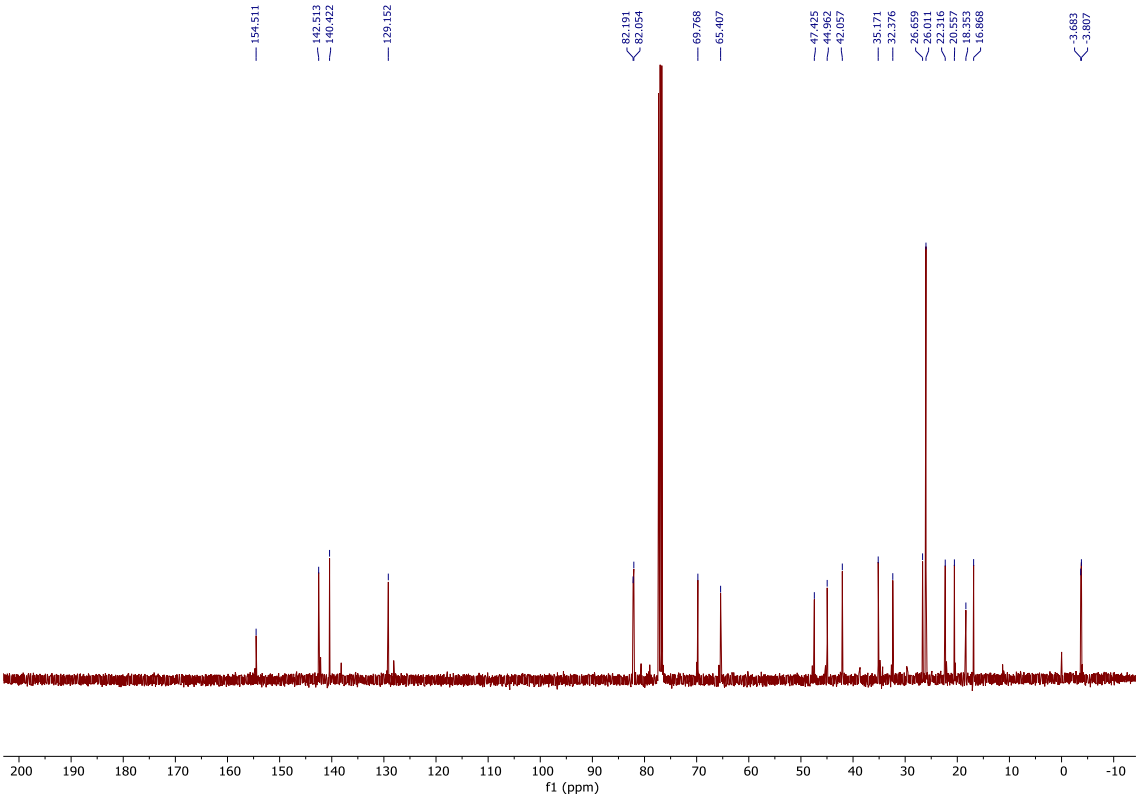


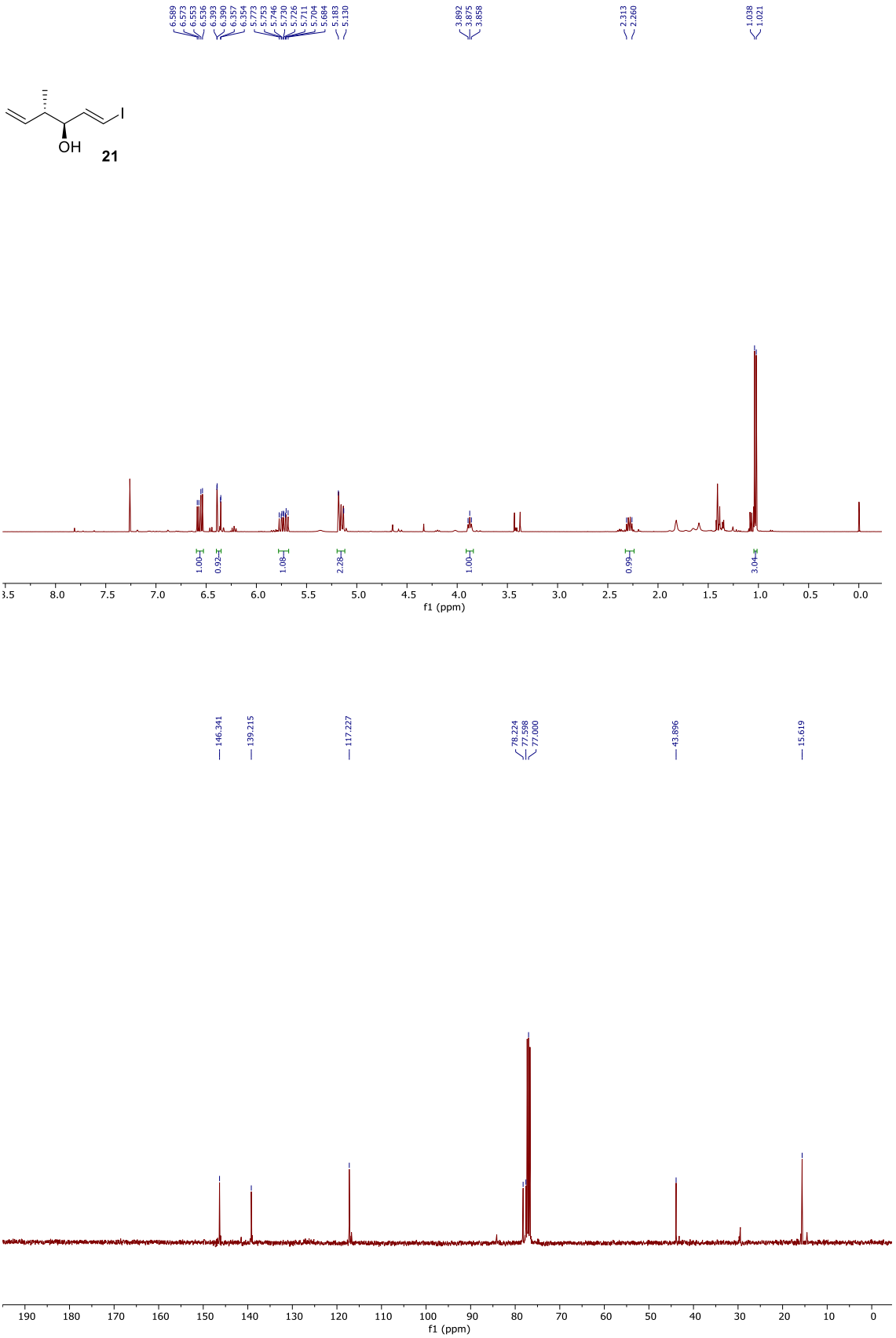


S2. ^{1}H- and ^{13}C-NMR spectra of (4*S*,5*S*,8*R*,10*R*,12*R*,6*E*)-10-[(*tert*-butyldimethylsilyl)oxy]-4-[(*E*)-2-iodovinyl]-5,8,12-trimethyl-1,3-dioxacyclotetradec-6-en-2-one (**16**)

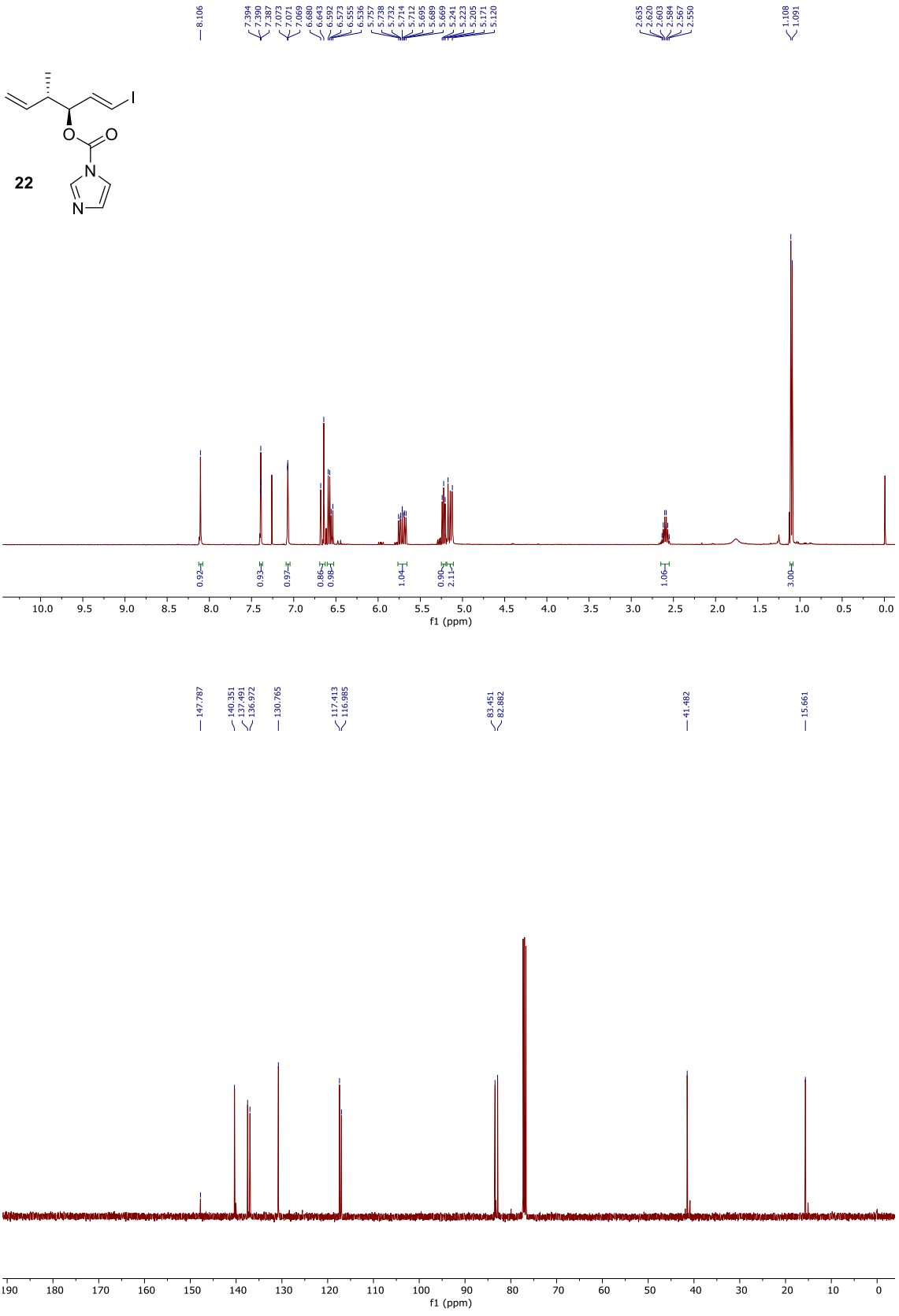




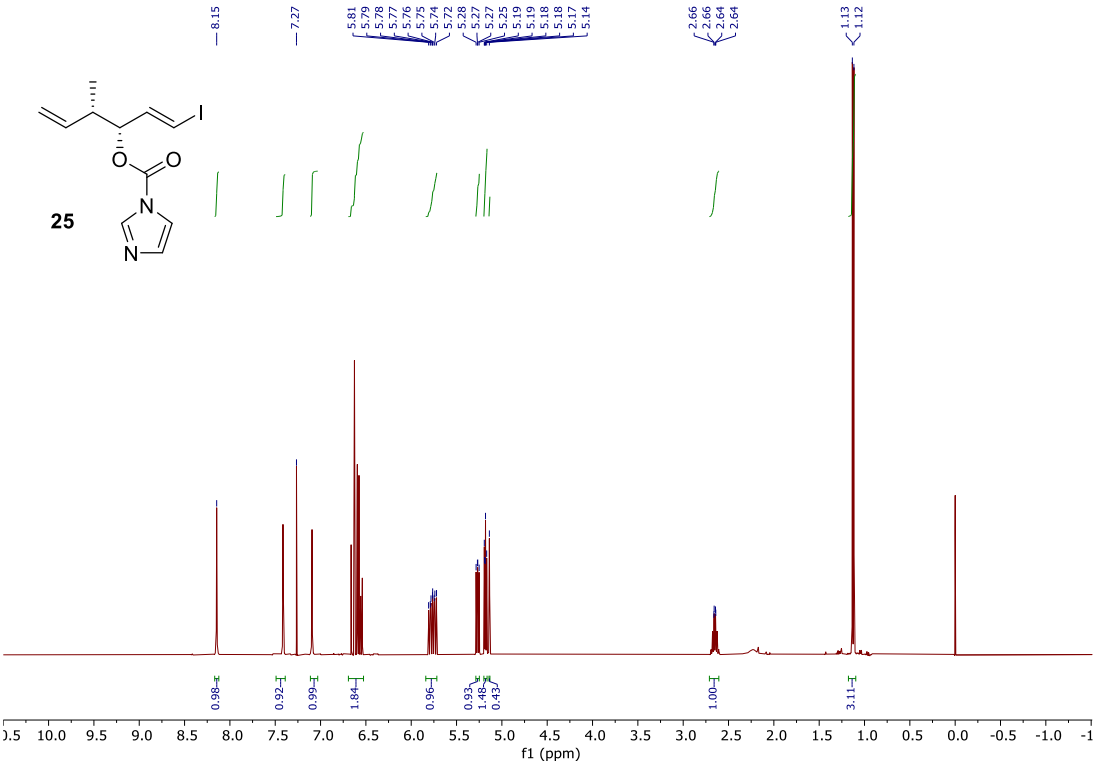
S3. ^{1}H- and ^{13}C-NMR spectra of (3*S*,4*S*,1*E*)-1-iodo-4-methyl-1,5-hexadien-3-ol (**21**)

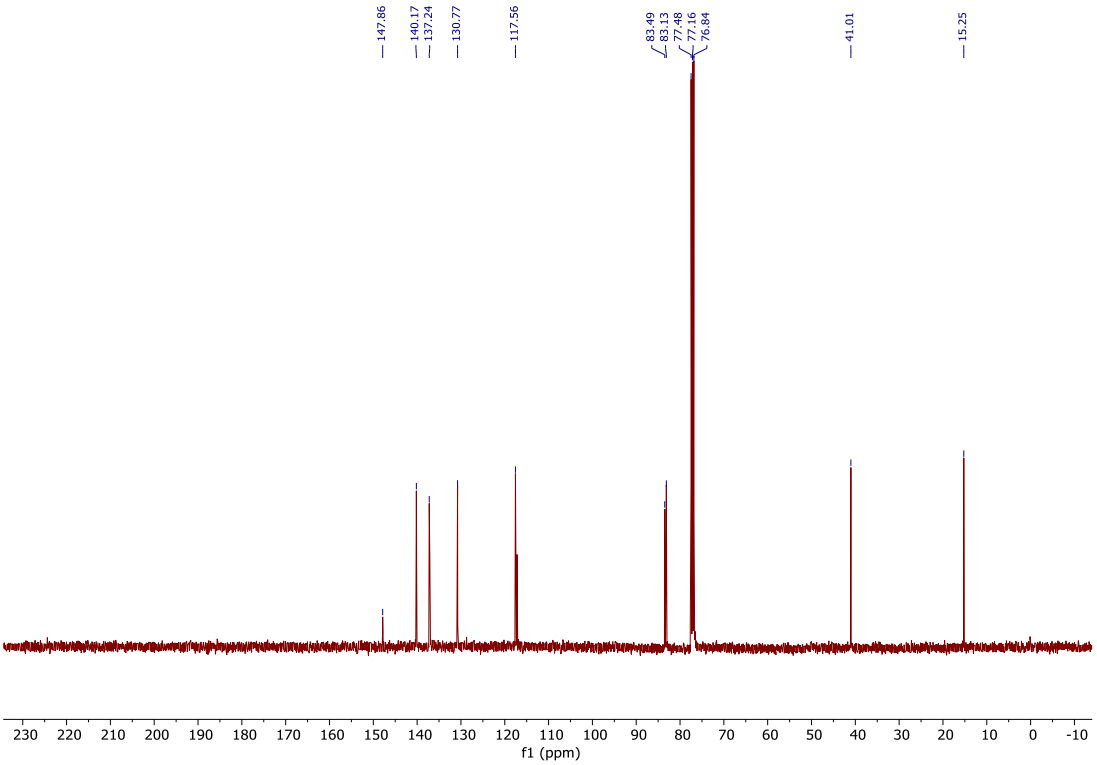


S4. ^{1}H- and ^{13}C-NMR spectra of (3*S*,4*S*,*E*)-3-[(1-imidazolylcarbonyl)oxy]-1-iodo-4-methyl-1,5-hexadiene (**22**)

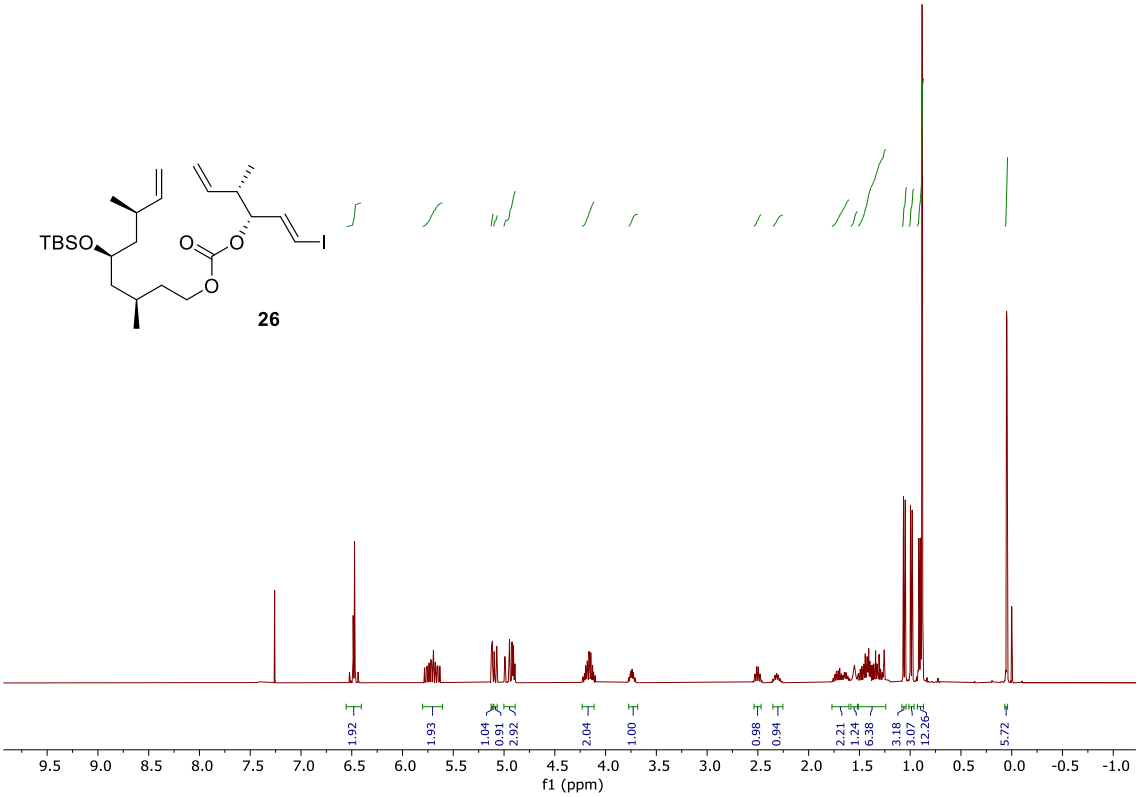


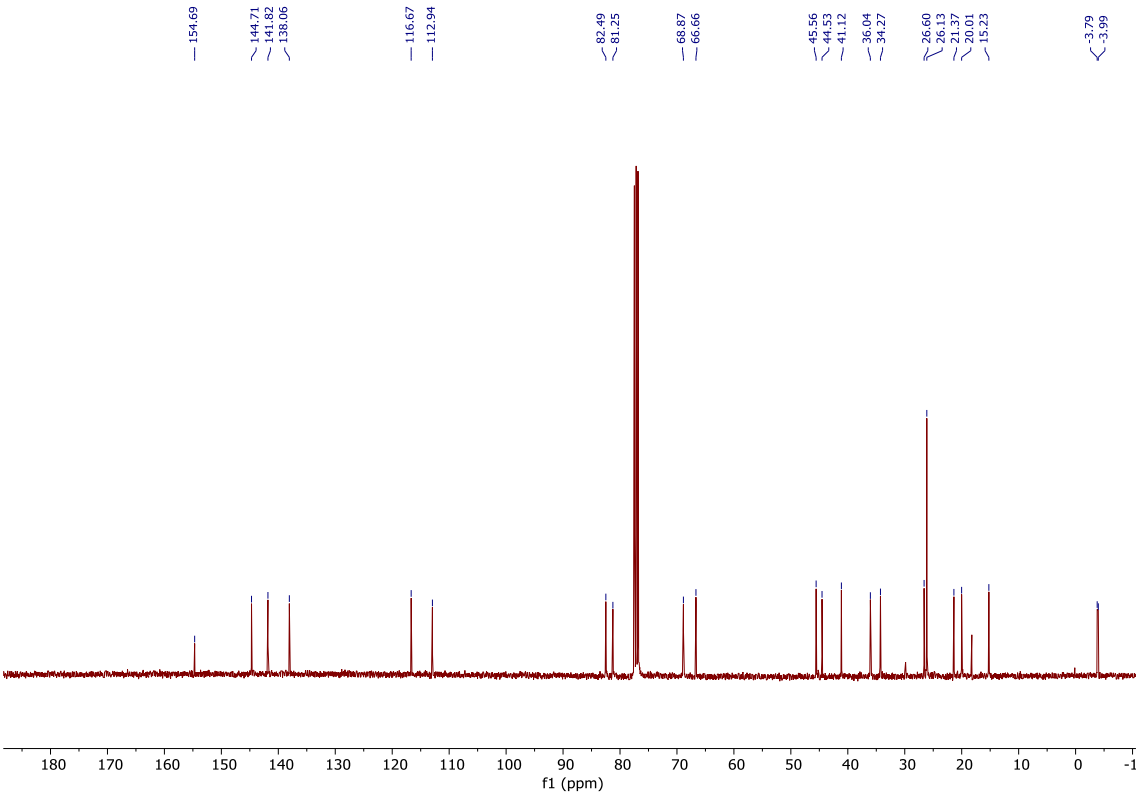
S5. ^{1}H- and ^{13}C-NMR spectra of (3*R*,4*S*,*E*)-3-[(1-imidazolylcarbonyl)oxy]-1-iodo-4-methyl-1,5-hexadiene (**25**)



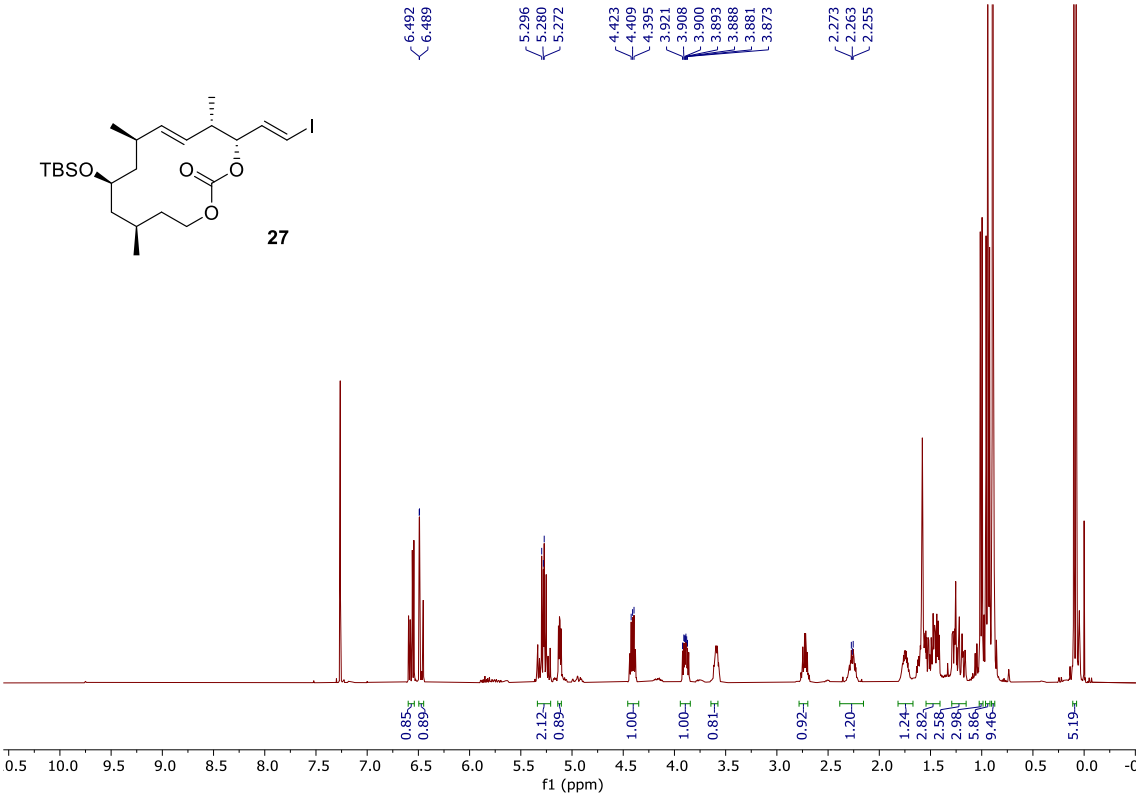


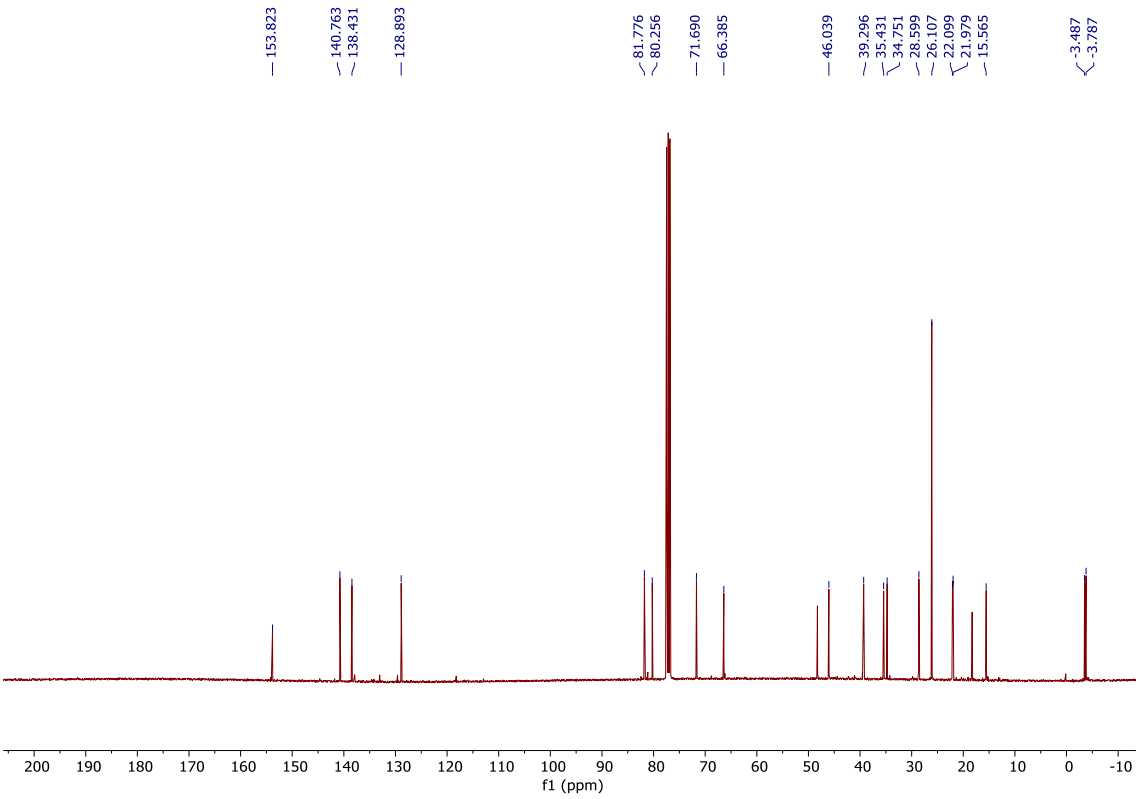
S6. ^{1}H- and ^{13}C-NMR spectra of (3*R*,5*R*,7*R*)-5-[(*tert*-butyldimethylsilyl)oxy]-3,7-dimethyl-8-nonen-1-yl (3*R*,4*S*,*E*)-1-iodo-4-methyl-1,5-hexadien-3-yl carbonate (**26**)



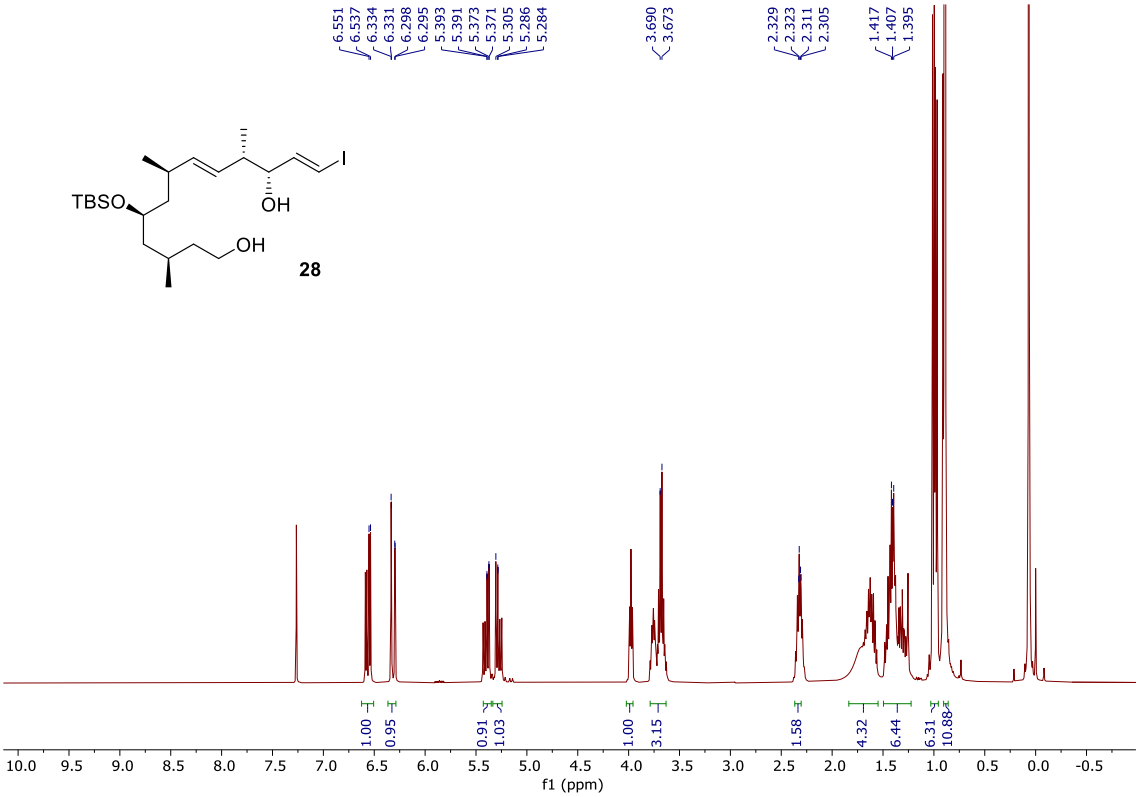


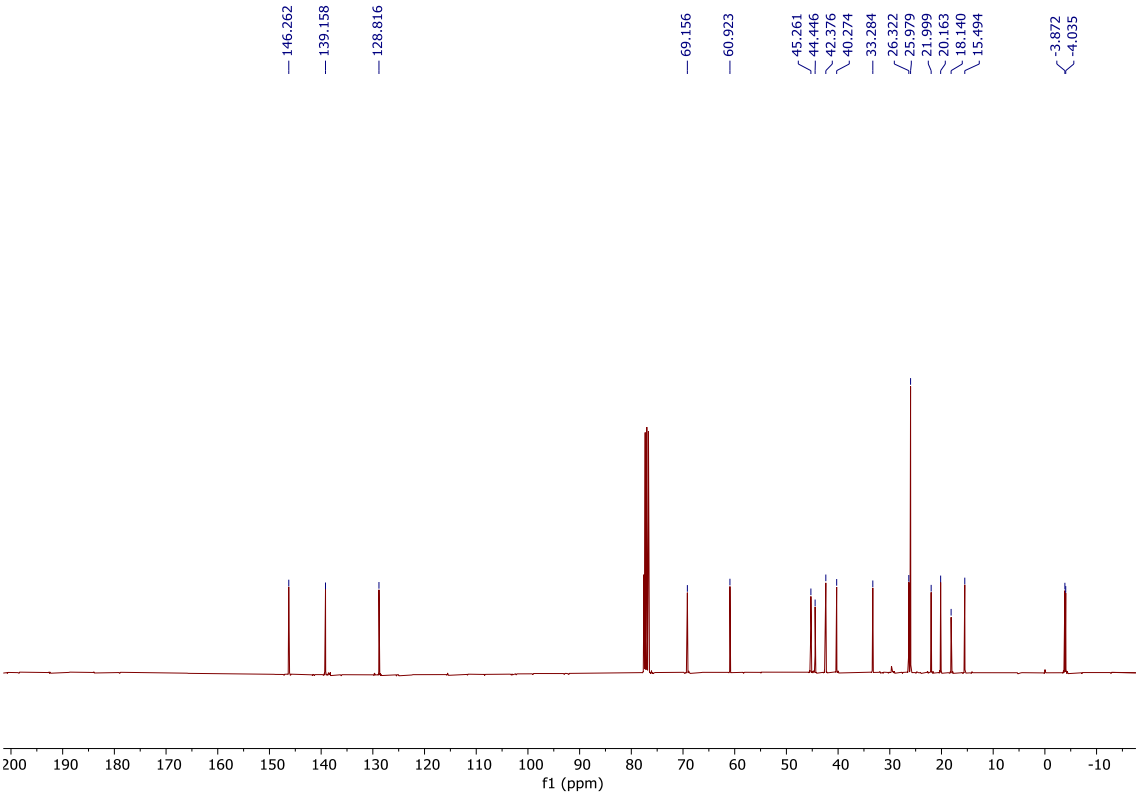
S7. ^{1}H- and ^{13}C-NMR spectra of (4*R*,5*S*,8*R*,10*R*,12*R*,6*E*)-10-[(*tert*-butyldimethylsilyl)oxy]-4-[(*E*)-2-iodovinyl]-5,8,12-trimethyl-1,3-dioxacyclotetradec-6-en-2-one (**27**)



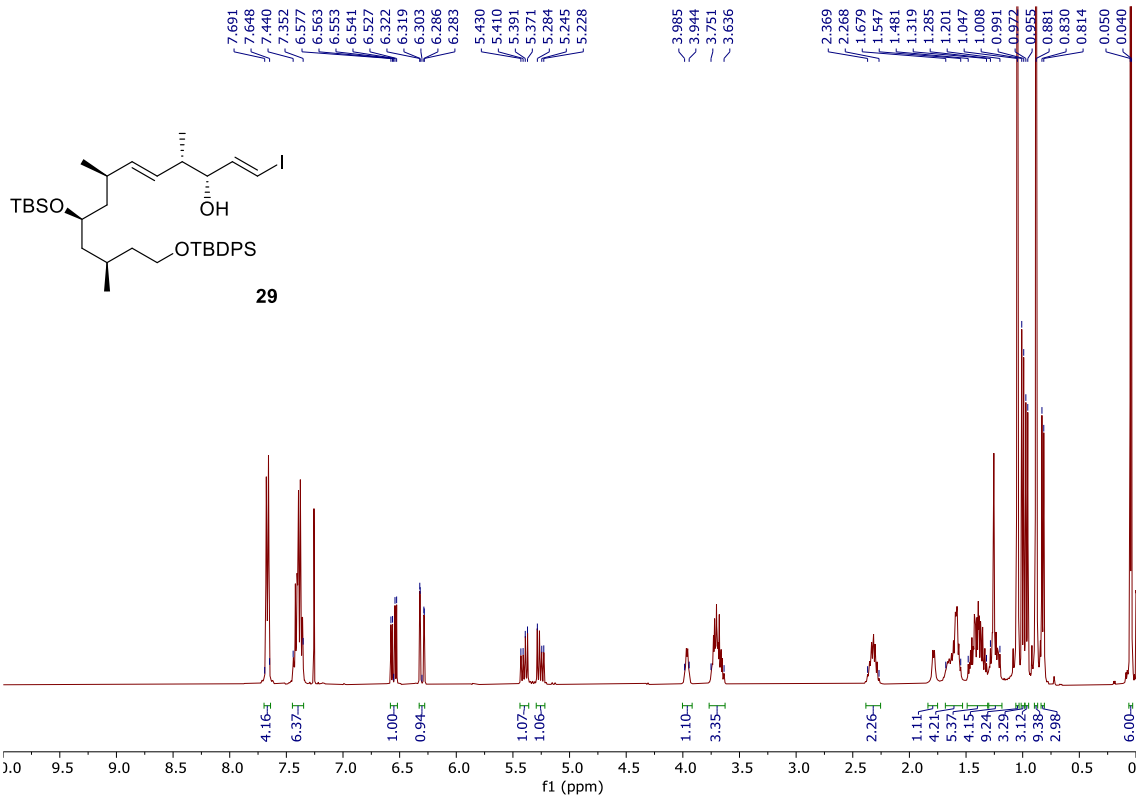


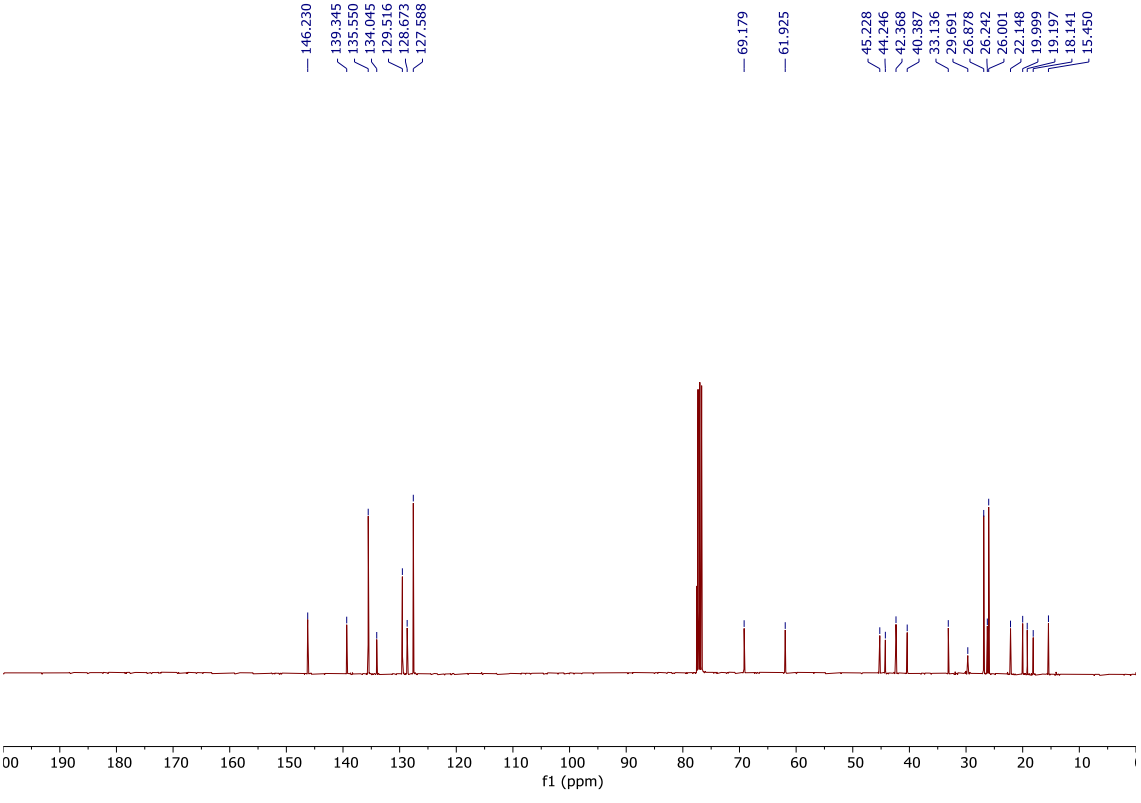
S8. ^{1}H- and ^{13}C-NMR spectra of (3*R*,5*R*,7*R*,8*E*,10*S*,11*R*,12*E*)-5-[(*tert*-butyldimethylsilyl)oxy]-13-iodo-3,7,10-trimethyl-8,12-tridecadiene-1,11-diol (**28**)





S9. ^{1}H- and ^{13}C-NMR spectra of (1*E*,3*R*,4*S*,5*E*,7*R*,9*R*,11*R*)-9-[(*tert*-butyldimethylsilyl)oxy]-13-[(*tert*-butyldiphenylsilyl)oxy]-1-iodo-4,7,11-trimethyl-1,5-tridecadien-3-ol (**29**)





S10. ^{1}H-NMR spectrum of (1*E*,3*S*,4*S*,5*E*,7*R*,9*R*,11*R*)-9-[(*tert*-butyldimethylsilyl)oxy]-13-[(*tert*-butyldiphenylsilyl)oxy]-1-iodo-4,7,11-trimethyl-1,5-tridecadien-3-ol (**30**)