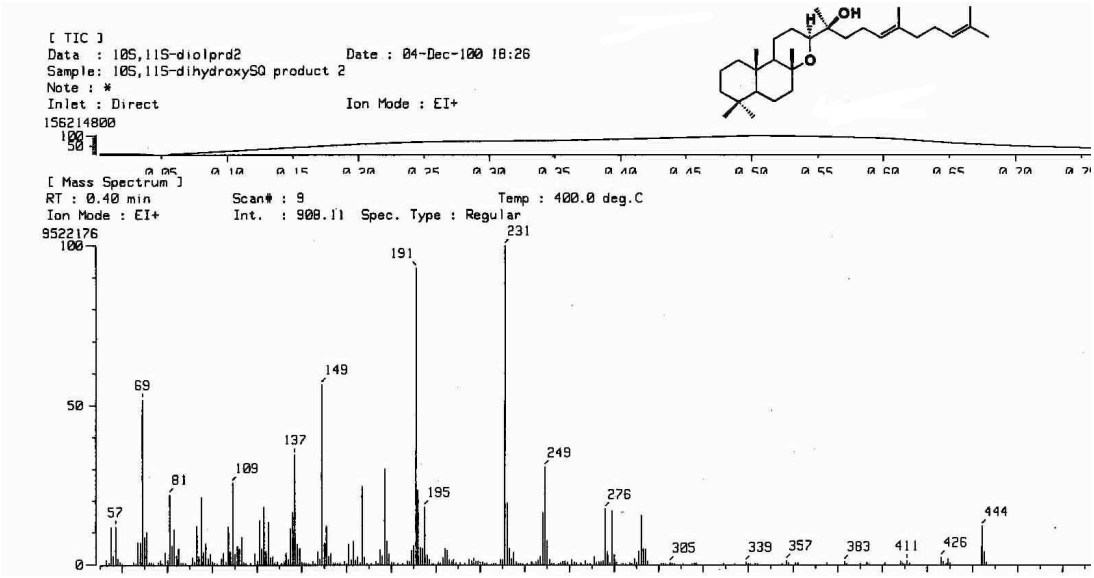
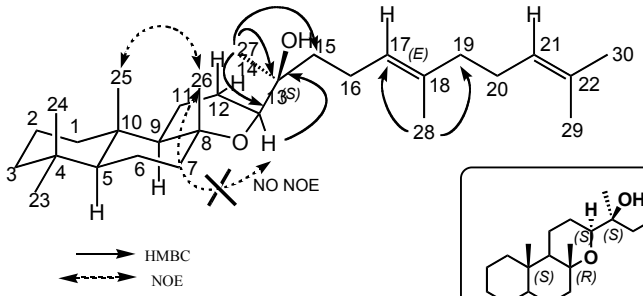
Product 29

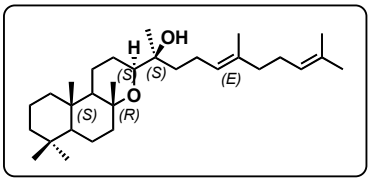


Product **29** (oil) from (10*S*,11*S*)-10,11-DihydroxySQ

$[\alpha]_D^{25} = +14.7$
(c = 0.16, in $CHCl_3$)

HRFABMS (glycerol)
M+H, Found: 445.4070
Calcd: 445.4046



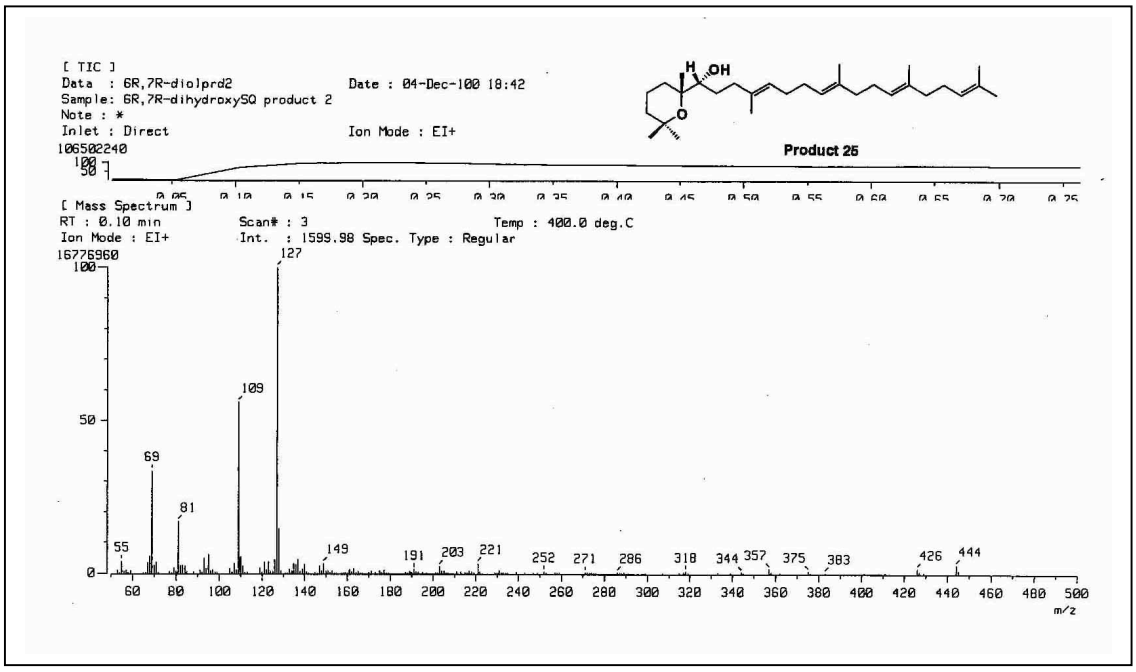


NMR data in C_6D_6, solvent peak : ^1H; 7.28 ppm , ^{13}C; 128.0 ppm

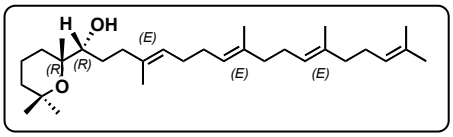
NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C
1	0.82 (m) ; 1.51(m)	**39.16**	9	1.53 (m)	**50.69**	17	5.52 (t, 6.8 Hz)	**125.67**	25	0.792 (3H, s)	**14.98**
2	1.47(m) ; 1.66(m)	**18.82**	10	—	**37.24**	18	—	**134.79**	26	1.176 (3H, s)	**26.54**
3	1.22(m) ; 1.46(m)	**42.23**	11	1.36 (m) ; 1.54(m)	**15.82**	19	2.24 (2H, t, 7.2 Hz)	**40.24**	27	1.291 (3H, s)	**21.74**
4	—	**33.18**	12	1.60(m) ; 1.69(m)	**21.31**	20	2.32 (2H,m)	**27.24**	28	1.814 (3H, s)	**16.12**
5	0.89 (m)	**56.30**	13	3.76(dd, 12.4 , 2.8Hz)	**74.72**	21	5.38 (t, 6.8Hz)	**124.99**	29	1.690 (3H, s)	**17.73**
6	1.13 (m) ; 1.58(m)	**20.42**	14	—	**72.98**	22	—	**131.08**	30	1.803 (3H, s)	**25.84**
7	1.57(m) ; 1.89(m)	**43.40**	15	1.84 (2H,m)	**39.75**	23	0.958 (3H, s)	**33.50**			
8	—	**74.91**	16	2.514 (2H,m)	**22.83**	24	0.888 (3H, s)	**21.63**			

The coupling constats of H-13 (*J*=12.4 Hz and 2.8 Hz) suggest that the dihedral angles between H12 and H13 are ca 170° and 53°, indicating that the C-ring is boat structure.

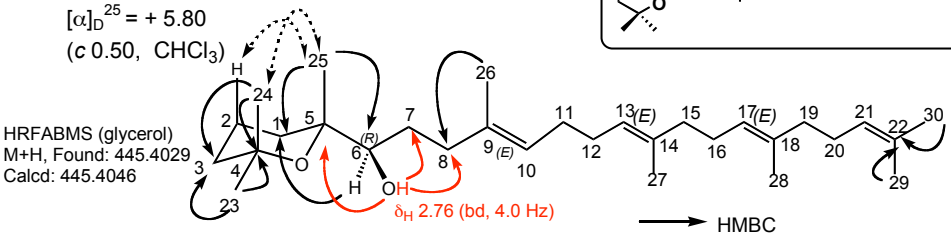
Product 30



Product **30** (oil) from (6*R*,7*R*)-DihydroxySQ



$[\alpha]_D^{25}$ = + 5.80
(*c* 0.50, $CHCl_3$)



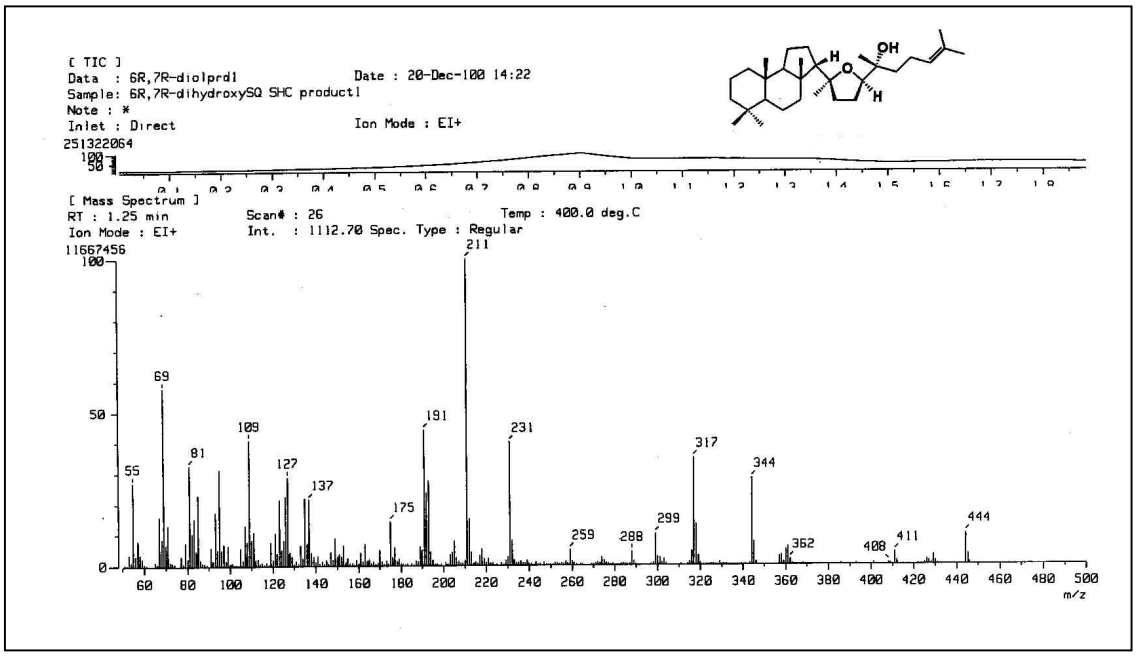
HRFABMS (glycerol)
M+H, Found: 445.4029
Calcd: 445.4046

NMR data in C_6D_6, solvent peak : ^1H; 7.28 ppm , ^{13}C; 128.0 ppm

NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C
1	1.29(m) ; 1.49(m)	**31.77**	9	—	**135.62**	17	5.42(m)	**124.91** [d]	25	1.234 (3H, s)	**21.22**
2	1.45(m) ; 1.58(m)	**16.45**	10	5.55 (bt, 6.8Hz)	**124.80** [d]	18	—	**135.10** [e]	26	1.788 (3H,s)	**16.30**
3	1.26(m); 1.35(m)	**36.62**	11	2.27 (2H, m)	**28.79** [c]	19	2.21 (2H, bt, 6.8Hz)	**40.21**	27	1.731 (3H, s)	**16.10** [a]
4	—	**71.47**	12	2.27 (2H, m)	**28.77** [c]	20	2.30 (2H,m)	**27.10** [b]	28	1.731 (3H, s)	**16.15** [a]
5	—	**75.81**	13	5.45(m)	**124.83** [d]	21	5.37(bt, 6.8 Hz)	**124.95** [d]	29	1.691 (3H, s)	**17.73**
6	3.48 (bd, 10.6Hz)	**78.94**	14	—	**134.93** [e]	22	—	**131.07**	30	1.804 (3H, s)	**25.85**
7	1.69 (2H,m)	**29.79**	15	2.21 (2H, m)	**40.21**	23	1.193 (3H,s)	**32.79**			
8	2.40(m) ; 2.72(m)	**37.34**	16	2.30 (2H, m)	**27.22** [b]	24	1.193 (3H,s)	**28.09**			

a-e) These carbon signals may be exchangeable between the same character, due to very close values.

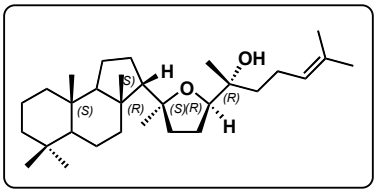
Product 31

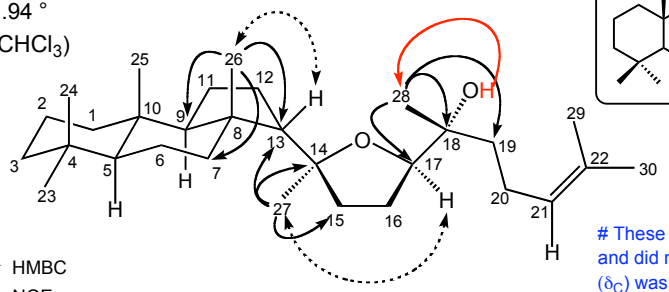


Product **31** (solid) from (6*R*,7*R*)-DihydroxySQ

$[\alpha]_D^{25}$ = + 1.94 °
(*c* 0.283, $CHCl_3$)

HMBC between Me-28 and O*H*





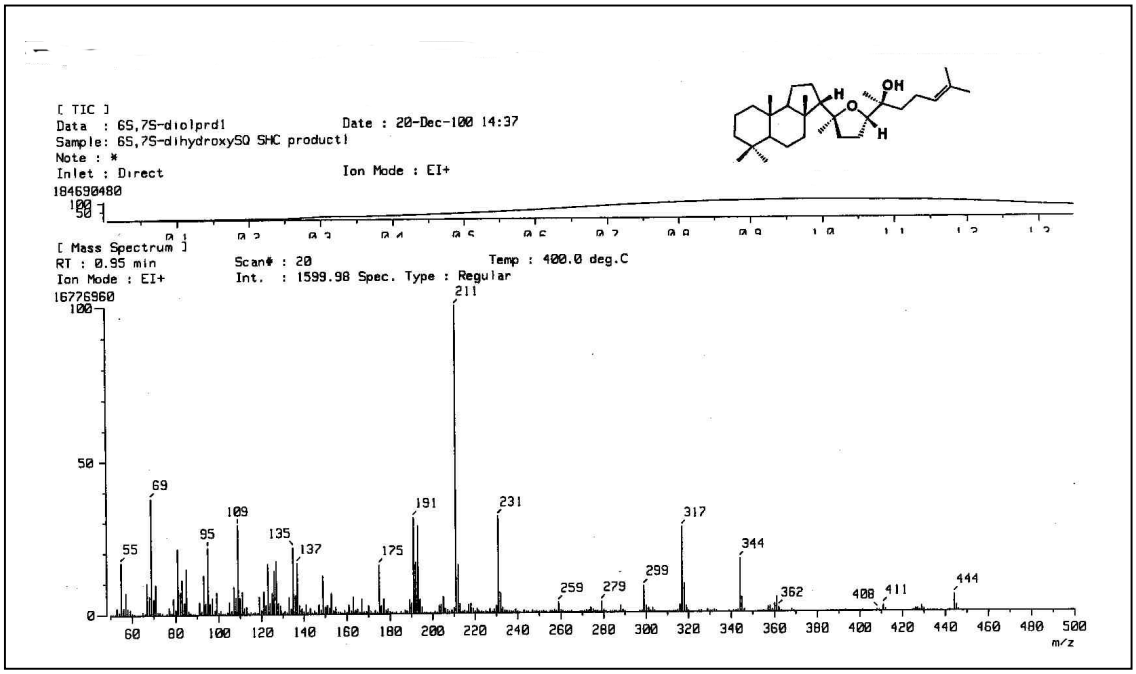
HMBC
NOE

These carbon signals are significantly small and did not clearly appear. The chemical shifts (δ_C) was determined by HMQC or HMBC spectra.

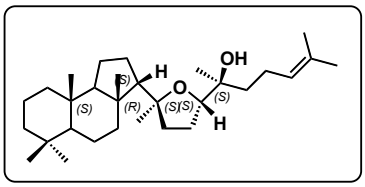
NMR data in C_6D_6, solvent peak : ^1H; 7.28 ppm , ^{13}C; 128.0 ppm

NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C
1	1.02(m) ; 1.59(m)	**41.32**	9	1.55(m)	**59.23(#)**	17	3.72 (dd, 8.4 ; 6.4Hz)	**82.03(#)**	25	0.957 (3H, s)	**16.25**
2	1.46(m) ; 1.74(m)	**18.84**	10	—	**37.40#**	18	—	**72.48**	26	0.999(3H, s)	**26.30**
3	1.32(m) ; 1.53(m)	**42.75**	11	1.41(m) ; 1.59(m)	**21.33**	19	1.78(m) ; 1.93(m)	**41.58(#)**	27	1.293 (3H,s)	**24.50(#)**
4	—	**33.18**	12	1.96(2H, m)	**24.50(#)**	20	2.47(m); 2.35(m)	**23.27**	28	1.223 (3H, s)	**21.99**
5	0.96(m)	**57.46**	13	1.78(m)	**60.83(#)**	21	5.45 (t, 6.8Hz)	**125.68**	29	1.767 (3H, s)	**17.68**
6	1.49(m) ; 1.70(m)	**20.03**	14	—	**85.95**	22	—	**130.94**	30	1.832 (3H,s)	**25.86**
7	1.79 (2H, m)	**38.00(#)**	15	1.53(m); 1.82 (m)	**38.49**	23	1.045 (3H, s)	**33.73**	*OH*	2.13 (bs)	
8	—	**44.66(#)**	16	1.68(m) ; 1.99(m)	**26.45(#)**	24	0.980 (3H, s)	**21.52**			

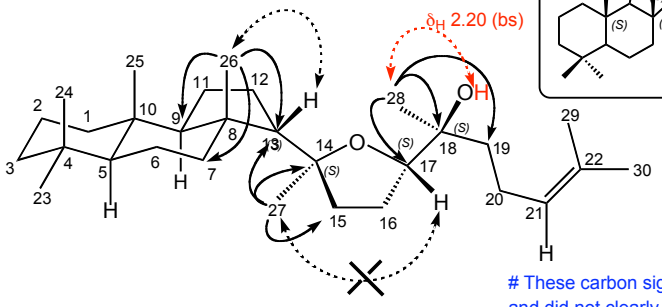
Product 32



Product **32** (solid) from (6*S*,7*S*)-DihydroxySQ



$[\alpha]_D^{25}$ = + 4.35
(c = 0.092, $CHCl_3$)



HMBC
NOE

These carbon signals are significantly small and did not clearly appear. The chemical shifts (δ_C) was determined by HMQC or HMBC spectra.

Chemical shift in C_6D_6 Solven peak: ^1H ; 7.28ppm , ^{13}C ; 128.0ppm

NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C	NO.	^1H	^{13}C
1	1.02(m) ; 1.62(m)	**41.16**	9	1.47(m)	**59.56**	17	3.71 (dd, 10.4 ; 4.6Hz)	**83.96(#)**	25	0.985 (3H, s)	**16.37**
2	1.49(m) ; 1.77(m)	**18.86**	10	—	**37.41**	18	—	**71.61**	26	1.063 (3H, s)	**26.53**
3	1.33 (m); 1.53(m)	**42.70**	11	1.46(m) ; 1.62(m)	**21.41**	19	1.74(m) ; 1.91(m)	**41.75**	27	1.231 (3H, s)	**25.19(#)**
4	—	**33.20**	12	1.96(2H, m)	**24.50(#)**	20	2.40 (2H, m)	**23.29**	28	1.211 (3H, s)	**21.92**
5	0.96 (m)	**57.46**	13	1.87(m)	**60.31(#)**	21	5.44 (t, 6.8Hz)	**125.72**	29	1.765 (3H, s)	**17.72**
6	1.49(m) ; 1.73(m)	**20.04**	14	—	**85.84(#)**	22	—	**130.89**	30	1.832 (3H, s)	**25.86**
7	1.79 (2H, m)	**37.87(#)**	15	1.53(m);1.70(m)	**39.10(#)**	23	1.050 (3H, s)	**33.75**	*OH*	2.20 (bs)	
8	—	**44.26(#)**	16	1.60(m) ; 2.02(m)	**26.54(#)**	24	0.998 (3H, s)	**21.54**			