Interpretation of NMR spectra of steroids

Strategy and Conclusions

Sum formula :   Calculate the double-bond equivalents from the sum formula $C_{27}H_{44}O_7$.

$$DBE = \frac{(2 * 27 + 2) - 44}{2} = 6$$

⟶   DBE = 6 :
„backbone" of steroids has already 4 DBE . So there are just 2 additional DBE

[1]H :   The angular CH_3 groups at 0.89 ppm and 0.96 ppm

[13]C{[1]H} :   Number the ^{13}C signals sequentially, starting with No. 1 for the most high field signal.

27 Carbons

27 Carbons: does agree with the sum formula $C_{27}H_{44}O_7$.

HSQC :   Assign the ^{1}H signals to the corresponding ^{13}C signals.

[1]H :   Copy the numeration of the ^{1}H signals in the HSQC to the ^{1}H spectra.

Use the integrals to define the signals or some integral regions. And use the DEPT experiments to assign the CH_3, CH_2, CH and C groups.

1	CH_3	15	CH_2	
2	CH_3	16	C	
3	CH_2	17	CH	
4	CH_2	18	CH	
5	CH_3	19	CH	
6	CH_2	20	CH	
7	CH_3	21	C	C - OH
8	CH_3	22	C	
9	CH_2	23	CH	
10	CH_2	24	C	
11	CH_2	25	CH	Double bond
12	CH	26	C	region
13	CH_2	27	C	Ketone
14	C			

HMBC : Use the HMBC to put the fragment around the angular CH₃ together.
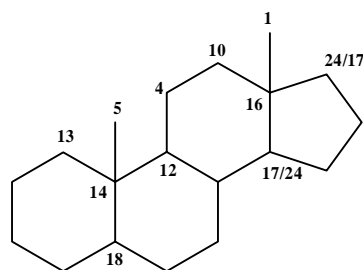
H(5) = C(12), C(13), C(14), C(18), [C(20)]
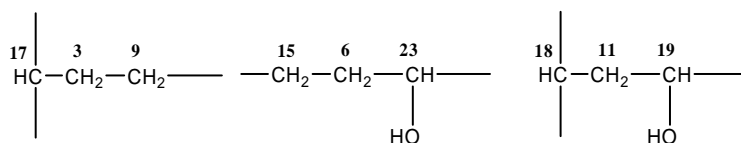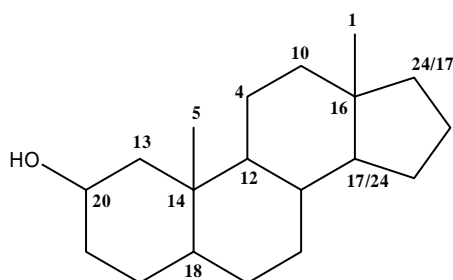H(1) = C(10), C(16), C(17), C(24)
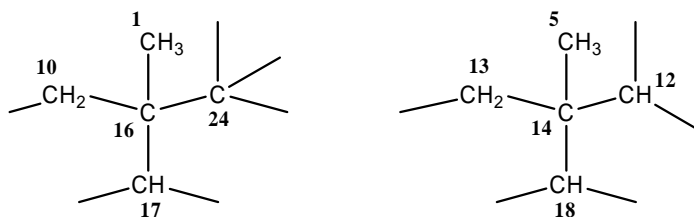
HSQC-TOCSY : Use the HSQC-TOCSY instead of the DQF-COSY to put several fragments together, because in the DQF-COSY-Spectra the peak overlapping is enormous.

C(4) = H(10), H(12)
Link between the two angular CH₃ fragments.

The exact position of C(24) and C(17) is not defined yet.

C(20) = H(15/13), H(20)   C(15) = H(6), H(15)
C(13) = H(20), H(13)   C(3) = H(9), H(17), H(3)
C(18) = H(11), H(18)   C(23) = H(6), H(23)
C(17) = H(3), H(17)   C(11) = H(19), H(11)

HMBC :   Use the HMBC to put more fragments together.

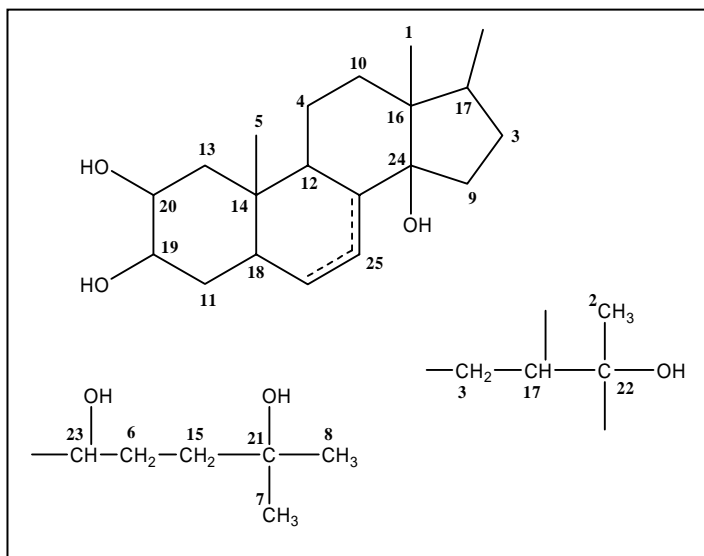H(13) = C(5), C(12), C(14, C(18), [C(26)], C(19/20)
C(19) = H(20)
H(25) = C(12), C(18), C(24)
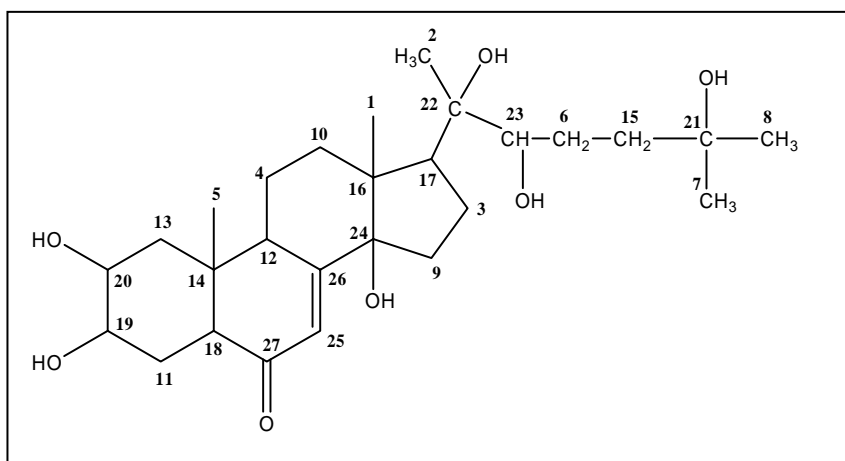

H(8) = C(7), C(15), C(21)
H(7) = C(8), C(15), C(21)
H(2) = C(17), C(22), C(23)
C(22) = H(3a)

1	CH₃
2	CH₃
3	CH₃
4	CH₂
5	CH₃
6	CH₂
7	CH₃
8	CH₃
9	CH₃
10	CH₂
11	CH₂
12	CH
13	CH₂
14	C
15	CH₂
16	C
17	CH
18	CH
19	CH
20	CH
21	C
22	C
23	CH
24	C
25	CH
26	C
27	C

There is just C(26) and C(27) left to close the ring
at C(18), C(12), C(24).

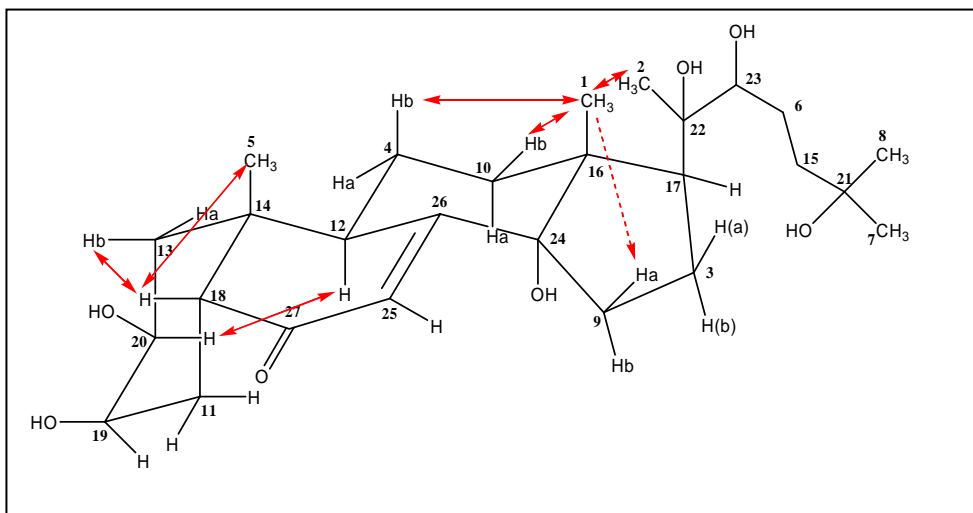**NOESY :**   The configuration at the individual carbons are determined by using the NOESY .

H(12) = H(20)
H(1) = H(9a), H(4b), H(10b)
H(5) = H(18)
H(19) = H(11)
H(17) = H(10a)
H(18) = H(13b)

¹H :   The ¹H signal of H(19) has only small couplings no big ones. Therefore H(19) has no axial-axial coupling to the axial H(20). So H(19) is equatorial.

The configuration at C(11) and C(3) can not be determined exactly because the relevant signals are overlapping.

It's **β- Ecdyson**