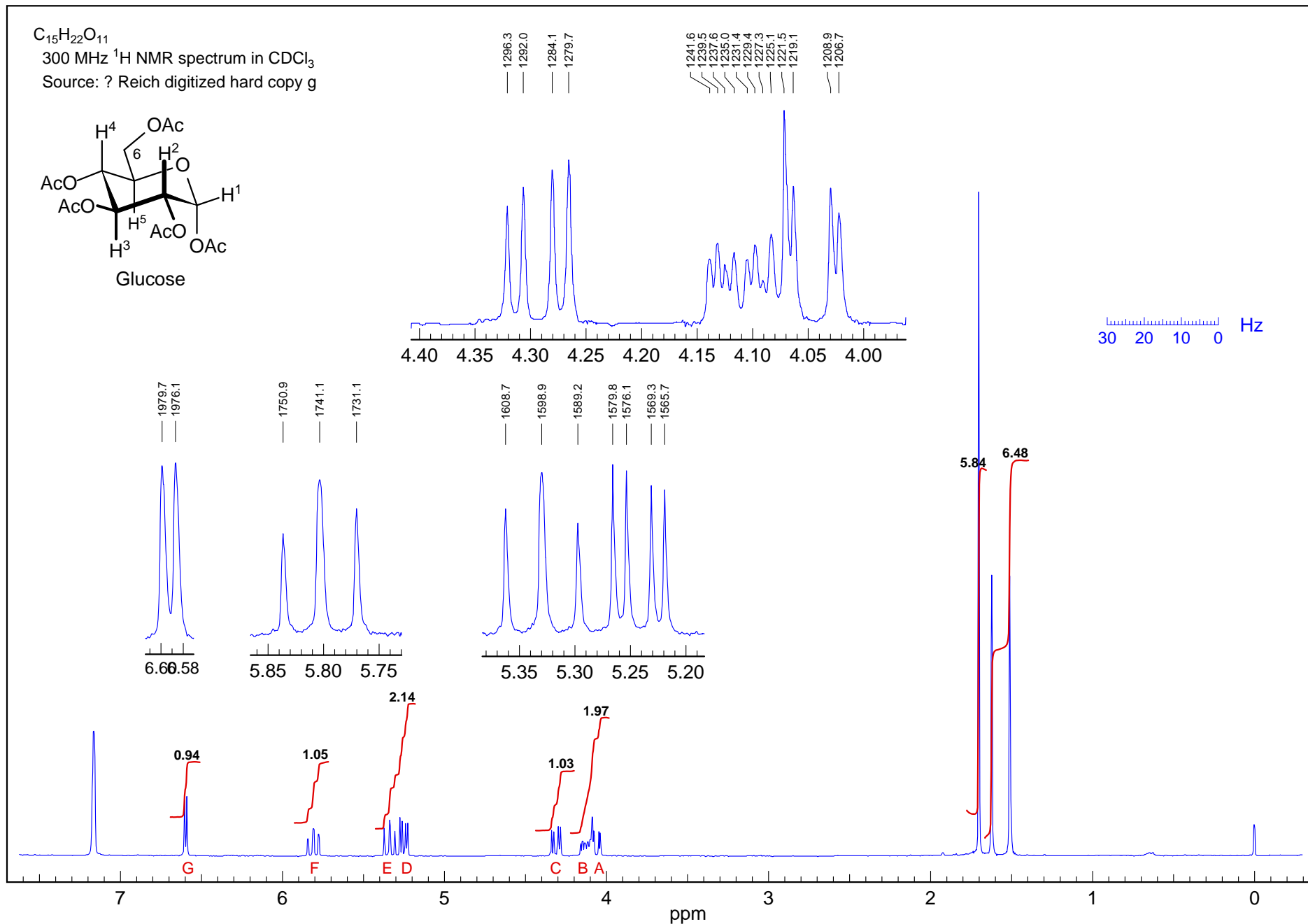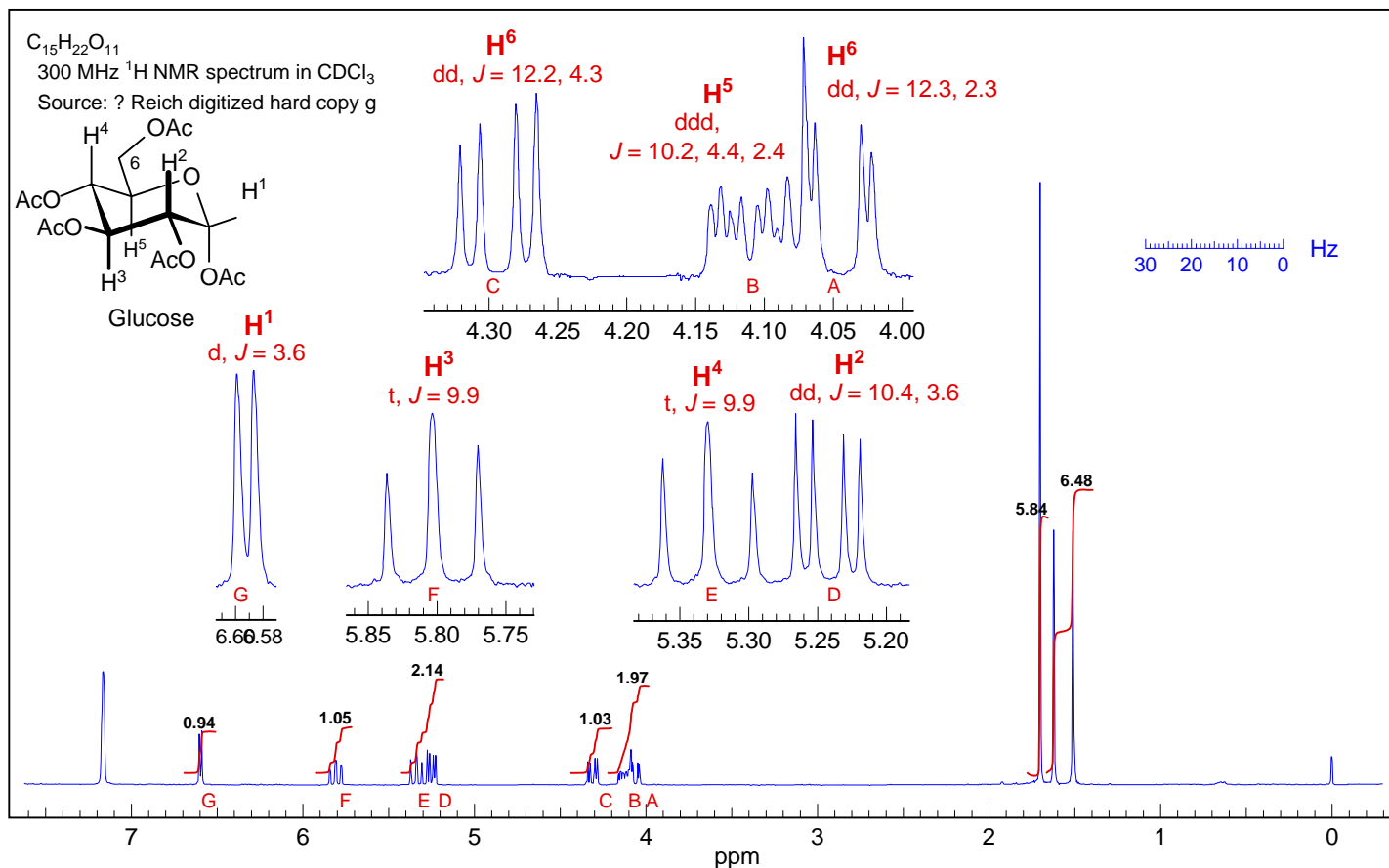**Exercise**: Examine the 300 MHz spectrum of glucose pentaacetate reproduced below. Assume you don't know the stereochemistry and use the spectrum to assign it at each of the carbons.

$C_{15}H_{22}O_{11}$
300 MHz ^{1}H NMR spectrum in $CDCl_3$
Source: ? Reich digitized hard copy g

Glucose

1296.3 1292.0 1284.1 1279.7
1241.6 1239.5 1237.6 1235.0 1231.4 1229.4 1227.3 1225.1 1221.5 1219.1
1208.9 1206.7

4.40 4.35 4.30 4.25 4.20 4.15 4.10 4.05 4.00

30 20 10 0 Hz

1979.7 1976.1
1750.9 1741.1 1731.1
1608.7 1598.9 1589.2 1579.8 1576.1 1569.3 1565.7

5.84 6.48

6.66.58
5.85 5.80 5.75
5.35 5.30 5.25 5.20

2.14
0.94 1.05 1.97
1.03

G F E D C B A

7 6 5 4 3 2 1 0
ppm

PLT NMR-glucose-pentaacetate-g.plt

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$C_{15}H_{22}O_{11}$
300 MHz ^1H NMR spectrum in $CDCl_3$
Source: ? Reich digitized hard copy g

Glucose

H^6 dd, J = 12.2, 4.3
H^5 ddd, J = 10.2, 4.4, 2.4
H^6 dd, J = 12.3, 2.3

H^1 d, J = 3.6
H^3 t, J = 9.9
H^4 t, J = 9.9
H^2 dd, J = 10.4, 3.6

1. Stereochemical determinations like this usually start with the identification of one or more protons that can be assigned from considerations of δ or J values. In this case G can be assigned to H^1, the anomeric proton, since it the most downfield one (two α-O substituents) and it is the only one coupled to just one other proton.

2. Multiplet B can be assigned to H^5, both from its chemical shift (it has an α-ether, rather than an α-acetoxy substituent, which has a much larger Δδ effect) and from the coupling, it is a ddd (J = 10.2, 4.4, 2.4 Hz), the only proton in the molecule coupled to 3 others.

3. Multiplets A and C can be assigned to the diastereotopic protons H^6, again using a chemical shift argument (secondary protons are upfield of tertiary ones), as well as the observation that these are both coupled to H^5 (B) from a consideration of both the size of J and the leaning.

4. Starting with H^1 (G): the small 3.4 Hz coupling must be either an eq-eq coupling (part structure **1**, requires an axial substituent at C^2) or an eq-ax one (part structures **2** and **3**).

Three possibilities from analysis of H^1 (G)

1   **2**   **3**   **4**   **5**
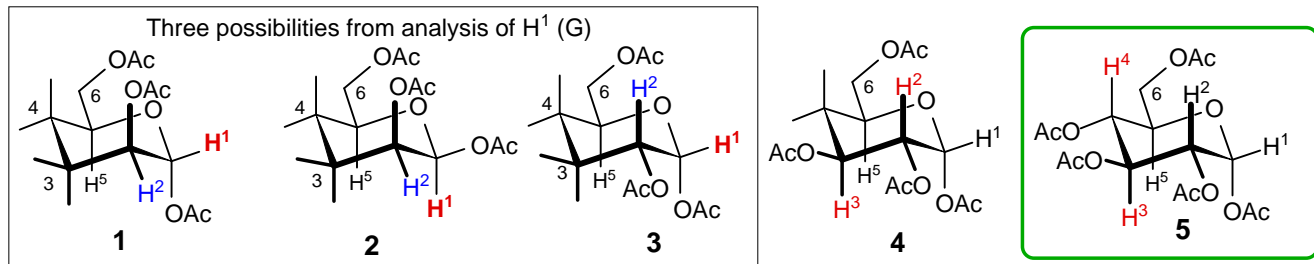
5. We can assign H^2 to multiplet D, a dd with J = 12.2, 3.6 Hz, since it the only signal that shows the 3.6 Hz coupling to H^1 (G). Since the second coupling to H^2 is an ax-ax one, this *requires* that *both* H^2 and its neighbor H^3 be axial. Thus part structure **3** is correct, and we know the details shown in structure **4**.

6. This leaves only two multiplets unassigned, E and F. Both are triplets with J = 9.9 Hz, thus both *must* be axial, and the *neighbors on both sides (H^2 and H^5) must also be axial*, so we know the full spereochemistry (**5**).

7. The assignment of H^3 and H^4 can be done as follows: we know that multiplets D and E canot be coupled to each other with J = 9.9, because there is not nearly enough leaning. So E cannot be H^3, so must be H^4, and F is then H^3.