Assignment Methodology for Carbons (moderately complex)

1. Assign all protons as much as possible from 1H homonuclear experiments

2. List all carbon chemical shifts and approximate intensity (1C protonated, 2C protonated, 1C quat, etc.)
    a) obtain Dept-135, and Dept-90 if needed, presuming sufficient concentration for 13C 1d

    A 13C 1d spectrum (and Dept's) is completely unnecessary, unless quaternary carbon assignments are particularly relevant, since HSQC is at least 10x more sensitivity
    - in other words, HSQC can provide protonated carbon chemical shifts/assignment on samples having 1/10th the concentration needed for a 13C 1d
    - HMBC provides quaternary 13C chemical shifts, but is only ~3x more sensitive than 13C 1d and is considerably more difficult to interpret

    --> then again, the additional information from an HMBC often completes a complex assignment (as we'll see for this compound)!!

3. For this compound, all protons were assigned from the 1H experiments. Often, gHSQC will assist in the assignment of overlapping protons (spreads the overlap out along the 13C dimension).
    a) Assign all protonated carbons directly from the gHSQC data.
    b) The edited-gHSQC (like a DEPT-135, -CH2- are inverted) is significantly less sensitive than gHSQC
    c) Some carbons may be difficult to resolve in an HSQC/HMBC; 13C detected experiments can overcome this problem with resolution, but with similar sensitivity as a DEPT
    d) 2 or 4x linear prediction of the indirect data is recommended; improves sensitivity and resolution

4. Make a grid of gHMBC 1H to 13C long-range correlations/crosspeaks.
    a) Provide an indication of strong (s), moderate(m), weak(w) and very weak (vw) crosspeaks, which will indicate how close the J(CH) matches the J selected in the experiment (J=8Hz here)
    b) In aromatic systems, 3-bond J(CH) ~ 7-10Hz, whereas 2-bond J(CH) ~ 1 Hz providing a simple method for assigning the aromatic quaternary carbons:
        i) C11 shows J(CH) to H13, H15 and H17 protons: C11 is easily identified at at 131.7ppm
        II) C16 correlates to H14, H18, and H12: 134.1ppm is found
        iii) C3 correlates to H18 and H12 protons, similar to C16 but without the H14 correlation at 132.7ppm
        iv) C4 correlates to H17 and H6, at 129.6ppm; expect correlations to H2 as observed
    c) The rest of the quaternary carbons are assigned easily once the grid is finished.