Problem Set 6 **2D NMR Spectroscopy**
CHEM 393
Dr. H.M. Muchall

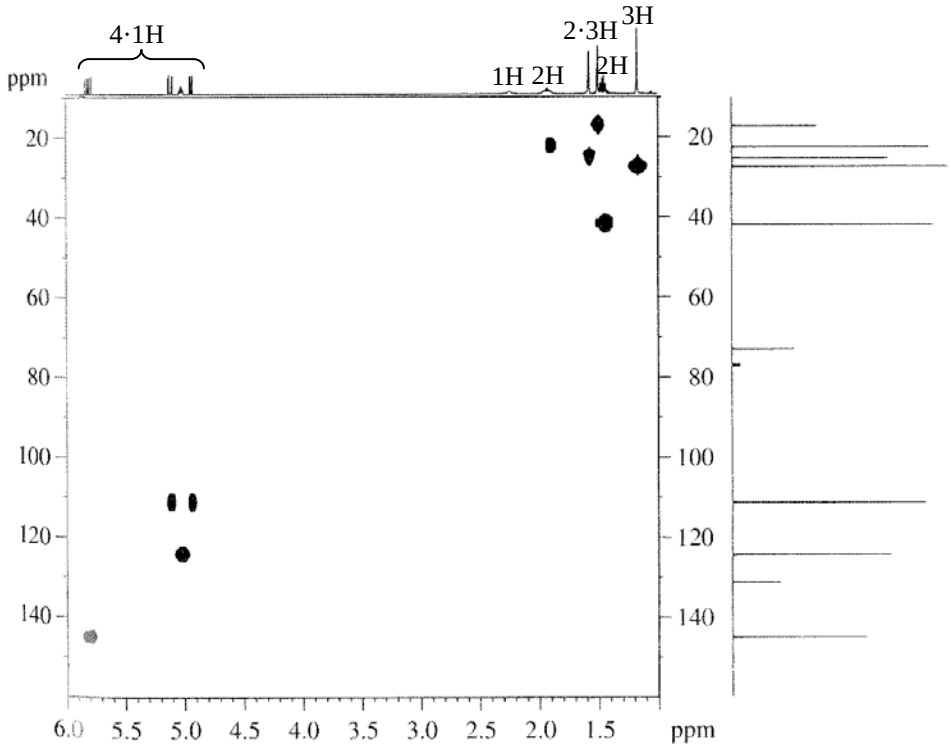
1. The HMQC (HETCOR-equivalent) spectrum below belongs to a compound $C_{10}H_{18}O$.

   a) (2 points) Why is there no correlation for the ^{1}H signal at 2.2 ppm? Which structural feature follows from this observation?

   b) (2 points) Why is there no correlation for the ^{13}C signal at 130 ppm? Which structural feature follows from this observation?

   c) (2 points) What can you conclude for the connectivity (and corresponding structural feature) for the ^{13}C signal at 110 ppm?

   d) (2 points) The ^{13}C signal at 73 ppm also does not show a correlation peak. Why? With this information, the information from a), its chemical shift and the integrations given, provide a guess for the partial structural information for this carbon atom.



2. The data below (expansions of the aromatic region of a ^{1}H NMR spectrum at 600 MHz, and a COSY spectrum) belong to a di-substituted aromatic compound.

   a) (6 points) From the ^{1}H NMR signals, determine all coupling constants. Then, to determine the "smoothed" signal multiplicity, ignore 1 Hz and 2 Hz couplings.

   b) (1 points) From the "smoothed" signals from a), determine the substitution pattern: o, m or p.

   c) (5 points) From the COSY, it is obvious that "1.3 ppm couples with 4.3 ppm", and from that the connectivity is CH_3-CH_2. In a similar fashion, determine the connectivity in the aromatic system. The connectivity should fit your result from b). (Hint: When you determine the coupling, keep in mind the "smoothed" signals that you determined in a), i.e., initially ignore the long-range couplings – the smaller dots-, then put them back into the analysis.)

   d) Study the COSY again. Did you really need the ^{1}H expansions to determine the substitution pattern?