Problem 1

Question:

The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of 1-iodopropane (C_3H_7I) recorded in $CDCl_3$ solution at 298 K and 400 MHz are given below.

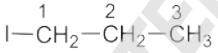
The ^{1}H NMR spectrum has signals at δ 0.99 (H_3), 1.84 (H_2) and 3.18 (H_1) ppm.

The $^{13}C\{^{1}H\}$ NMR spectrum has signals at δ 9.6 (C_1), 15.3 (C_3) and 26.9 (C_2) ppm.

Also given on the following pages are the ^{1}H–^{1}H COSY, ^{1}H–^{13}C me-HSQC, ^{1}H–^{13}C HMBC and INADEQUATE spectra. For each 2D spectrum, indicate which correlation gives rise to each cross-peak by placing an appropriate label in the box provided (*e.g.* $H_1 \rightarrow H_2$, $H_1 \rightarrow C_1$).

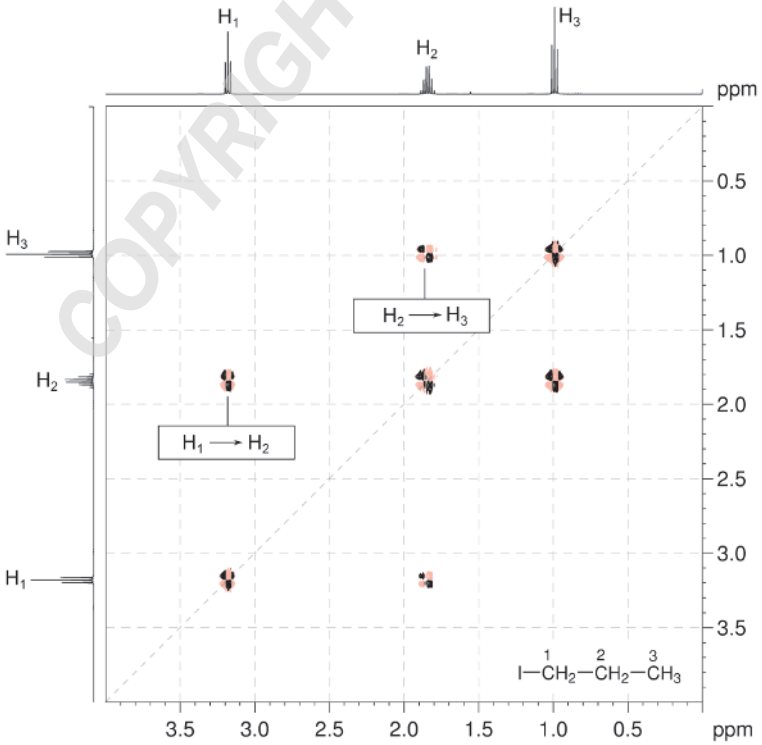
Solution:

1-Iodopropane



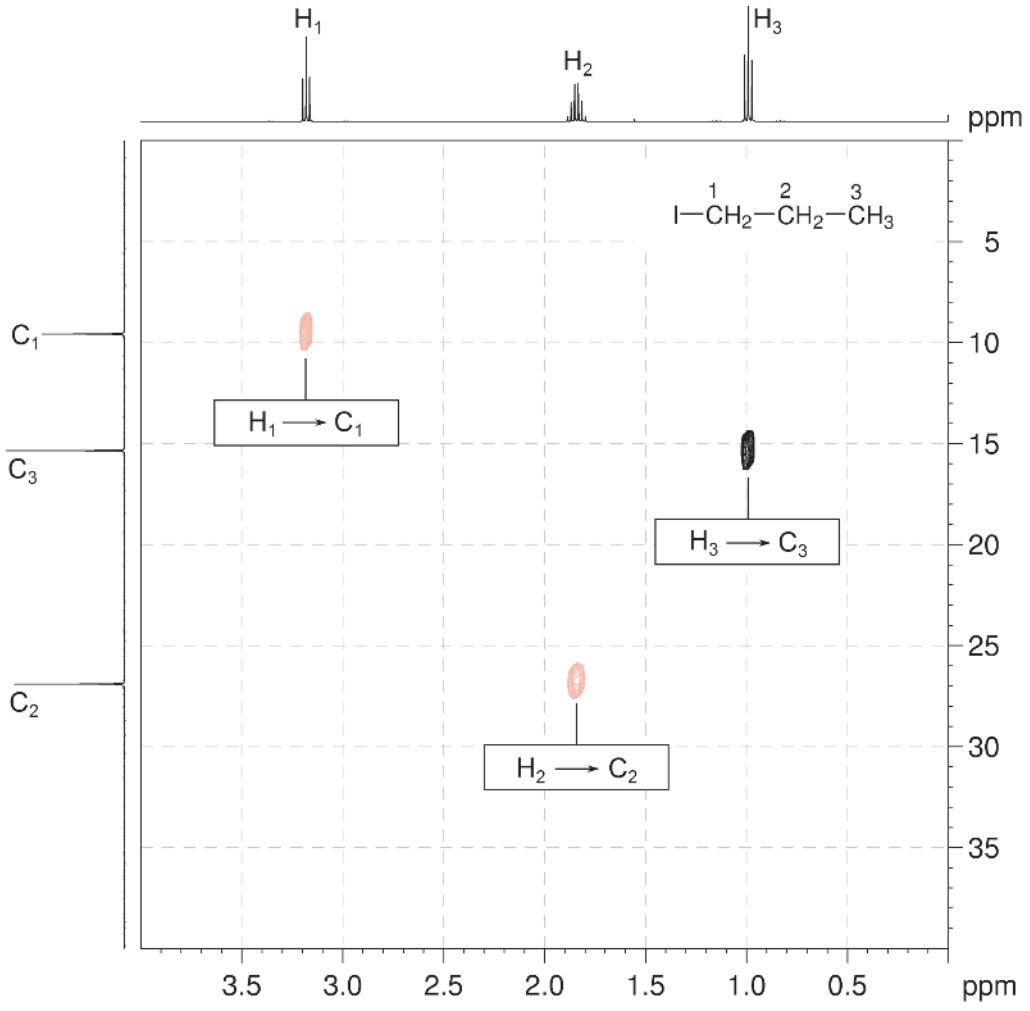
1. ^{1}H–^{1}H COSY spectra show which pairs of protons are coupled to each other. The COSY spectrum is always symmetrical about a diagonal. In the COSY spectrum, there are two $^{3}J_{H-H}$ correlations above the diagonal ($H_1 \rightarrow H_2$ and $H_2 \rightarrow H_3$). There are no long-range correlations.

^{1}H–^{1}H COSY spectrum of 1-iodopropane ($CDCl_3$, 400 MHz)



2. The ^{1}H–^{13}C me-HSQC spectrum shows direct (one-bond) correlations between proton and carbon nuclei, so there will be cross-peaks between H_1 and C_1, H_2 and C_2 and also between H_3 and C_3. As the spectrum is multiplicity edited, the cross-peaks corresponding to CH_2 groups are shown in red and are of opposite phase to those for CH_3 groups.

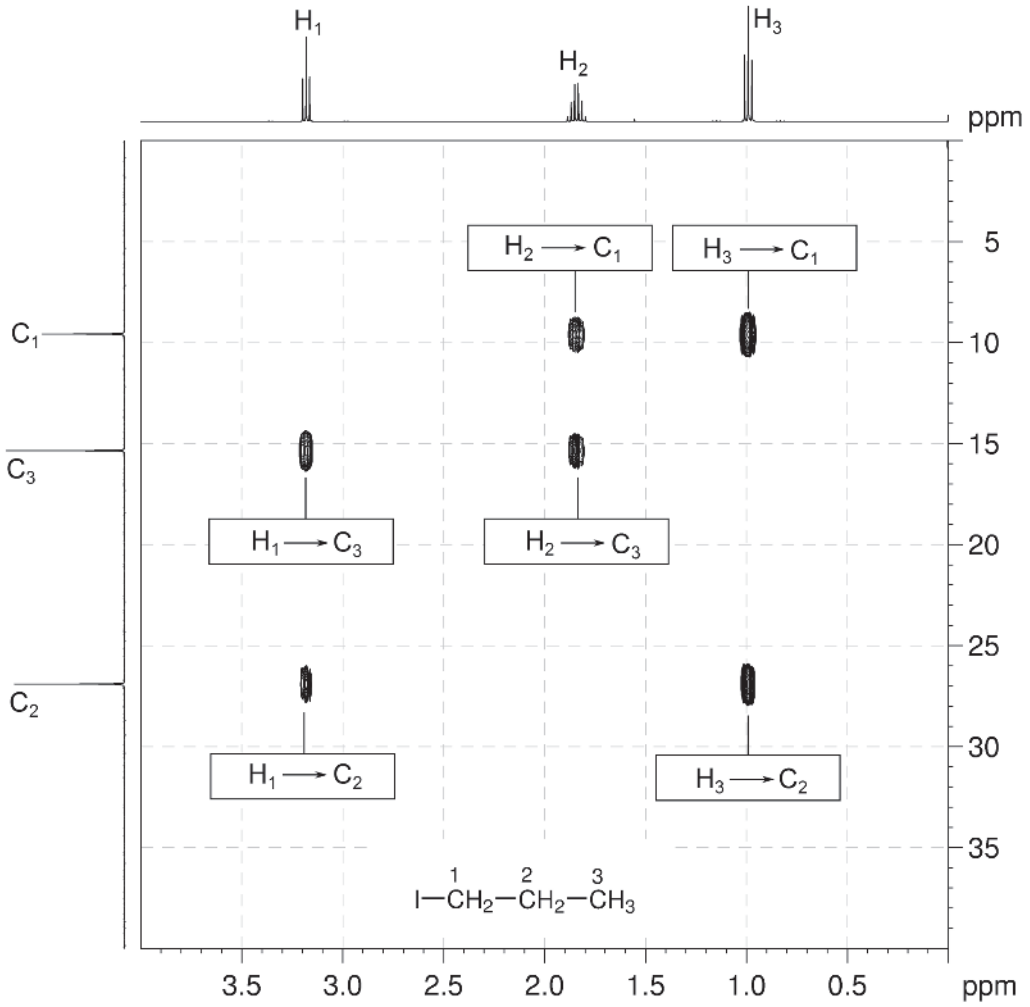
^{1}H–^{13}C me-HSQC spectrum of 1-iodopropane ($CDCl_3$, 400 MHz)



3. In HMBC spectra, remember that, for alkyl systems, both two- and three-bond C–H coupling can give rise to strong cross-peaks.

4. H_1 correlates to C_2 and C_3. H_2 correlates to C_1 and C_3. H_3 correlates to C_1 and C_2.

^{1}H–^{13}C HMBC spectrum of 1-iodopropane ($CDCl_3$, 400 MHz)



5. The INADEQUATE spectrum shows one-bond ^{13}C–^{13}C connectivity. There are correlations between C_1 and C_2, and C_2 and C_3.

INADEQUATE spectrum of 1-iodopropane ($CDCl_3$, 150 MHz)