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Spring 2006

Spectroscopic Analysis of 4B, ethyl-2-methyl-4-pentenoate

This paper analyzes spectroscopic data for unknown compound 4B. Our proposed structure, ethyl-2-methyl-4-pentenoate, is shown in Figure 1. When I refer to carbons or hydrogen groups that are labeled in this figure, I use italics (e.g., *C4*). The following paragraphs describe the correlation between our spectroscopic data and our proposed structure.

MS: The M^+ peak was at 142, the molecular mass of our compound. There were also significant peaks at 127 (M-15), 97 (M-45), and 69 (M- 73), corresponding to loss of CH_3, O-CH_2-CH_3, and O=C-O-CH_2-CH_3, respectively. The peak at 41 (M-101) represented loss of everything except the end H_2C=CH-CH_2 group.

IR: The three peaks that lie just below 3000 cm^{-1}—at 2980, 2938, and 2909 cm^{-1}—indicated the presence of several sp^3 C-H stretches. The peak at 3080 cm^{-1} represented an sp^2 C-H stretch. The IR spectrum also showed C=O, C=C, and C-O stretches at 1736, 1641, and 1183 cm^{-1}, respectively.

^{13}C NMR: Our broadband-decoupled spectrum showed eight distinct types of carbon. The only signal above 165 ppm was our C=O (*C1*), at 176 ppm; because this carbon had no hydrogens, it didn't appear in either DEPT spectrum. Between 100 and 165 ppm were sp^2 carbons: the DEPT-90 showed that the carbon at 136 ppm was CH (corresponding to *C4*), while the DEPT-135 revealed that the carbon at 117 ppm was CH_2 (*C5*). The rest of the signals were in the range of sp^3 or sp carbons. The CH_2 carbon at 60 ppm was the most shifted, so it must have been the carbon attached to the oxygen (*C6*). *C2* and *C3* were slightly less shifted, at 39 and 38 ppm, respectively. The distant CH_3 carbons, *C7* and *C8*, were at 17 and 14 ppm, although we couldn't tell which was which.

^{1}H NMR: The two groups of signals above 4.5 ppm corresponded to the double-bond hydrogens, *H4* and *H5*. The group at 5.7 ppm integrated to one hydrogen (*H4*); the multiplet splitting pattern was consistent with the fact that *H4* was surrounded by two groups of two hydrogens each. *H5* at 5.0 ppm integrated to two hydrogens and showed the doublet-of-doublets pattern that one would expect when two distinct hydrogens are split by an adjacent hydrogen. The remaining hydrogens were those attached to sp^3-hybridized carbons. The most shifted of these was *H6*, the hydrogen group next to electronegative oxygen. Falling at 4.1 ppm, it integrated to two hydrogens and showed the expected quartet pattern. The CH_3 hydrogens, *H7* and *H8*, were around 1.2 ppm. Unlike the carbons to which they were attached, we could tell these two groups apart, because of splitting patterns. *H8*, split by only one hydrogen, was the doublet; *H7* was split by two hydrogens and showed the triplet pattern. The remaining hydrogens were *H2*, *H3.1*, and *H3.2*. As *H3.1* and *H3.2* were diasterotopes, they must have been the two adjoined splitting patterns that fell around 2.5 ppm. The doublet-of-doublets patterns are consistent with the fact that each diastereotopic hydrogen was also split by two other, unique hydrogens. Finally, the single hydrogen at 2.2 ppm corresponds to *H2*; the splitting pattern resembles a sextet, which we expect from the fact that *H2* is surrounded by five other hydrogens.