Example Analysis Using Both IR and H-1 NMR

Transcript

Instructor: Brett McCollum

00:00:00:00 - 00:00:23:31
Instructor: All right. Now we're ready for one of the more challenging problems, almost at that limiting level that you're going to be experiencing in the course, where we're going to combine information from nuclear magnetic resonance with IR spectroscopy and solve an unknown compound. This one has a lot of peaks. There's a lot of information that we're going to have to deal with. Let's get started.

00:00:23:31 - 00:00:58:54
Instructor: First thing we recognize is what information is being provided to us. We have our chemical formula, and we also have been told some of the IR peaks. Alternatively, we could have been given the IR spectra and need to extract that information out of it, but we can handle it this way. Now that we have our chemical formula to start us off, we're going to determine the degree of unsaturation. We have five carbon atoms, 11 hydrogens, and we also have a halogen, the bromine.

00:00:58:54 - 00:01:38:37
Instructor: We have ten plus two is 12 subtracting 12 gives us a degree of unsaturation of zero, that there are no double bonds and no rings in our unknown compound. Then let's look at our IR spectrum. We see that we have peaks in the region around 2,950 to 2,850, but that's where we expect any signals associated with alkanes to show up. Given that it's an organic molecule, that's not surprising. The other peak that we've noted is a signal around 1,050.

00:01:38:37 - 00:02:22:07
Instructor: This tends to correspond to a CO bond that is stretching. Well, we have an oxygen present so that matches, but combining the fact that we don't see any signal in the region around 1,700, meaning there's no carbonial group. That also matches our degree of unsaturation, so we're confident, it's going to be an oxygen that's either in the middle of the chain, an ether, or an oxygen that's at the end of the chain with a hydrogen attached, and alcohol. Those are the only two choices that we have, so we're going to investigate. Now we look here and look at that NMR spectrum.

00:02:22:07 - 00:02:51:08
Instructor: Do you see an alcohol present? Remember, if an alcohol is there, we're going to see it showing up as a single it with integration of one somewhere between 0.5 and 5 PPM. We have integrations given to us, and the only signals that have an integration of one hydrogen are showing up has multiples. There isn't an alcohol present.

00:02:51:08 - 00:03:35:74
Instructor: That gives us a really strong signal that we have an ether that we're trying to assemble. All right. Let's build our table and extract the remaining information that we can from our spectrum. 66 00:03:26,000 --> 00:03:27,340 Okay. I've put in my chemical shift values based on the spectrum, and they might be off from the values that you have by about 0.1.

00:03:35:74 - 00:03:56:64
Instructor: We're looking for an estimate here do as well as you can. You try and get it as close to what you believe it is. But if you're off by just a little bit by about 0.1, you're still going to be able to make that assignment effectively to determine your unknown compound. Now let's determine our integrations, those have been given to us.

00:04:01:88 - 00:04:31:76
Instructor: Count up the number of protons represented by our integrations. We have three, three, three, that's nine plus two more, that's 11, which matches our chemical formula. All of our protons are showing up here, and there is no mirror plane in the molecule that would cause any of these to actually the entire set to be doubled. Now we can try and determine our multiplicity. Let's look at our spectrum.

00:04:31:76 - 00:04:54:77
Instructor: A is a doublet. B is a doublet. C is a singlet. D and E are multiplets. If we look carefully, they appear to be quintets.

00:04:54:77 - 00:05:18:68
Instructor: Now, there might always be additional peaks that are just too small to detect. You have to be careful about making the assumption, but they do appear to be that. We'll make a little note for ourselves. We have our data table. We suspect that this is an ether.

00:05:18:68 - 00:05:50:26
Instructor: We've found all of the protons present in the molecule. We have five different types of chemical environments for our protons, all of which are on a carbon, and there are five carbons present. We've likely got a good way to detect all of our carbons, all of our hydrogens, the ether is where that oxygen is going to be, and then there should be a bromine somewhere. Let's start fitting our pieces together. So, why don't we start off with signal A?

00:05:50:26 - 00:06:19:92
Instructor: This is going to have an integration of three. I interpret that as a CH3 group. It should have one more bond to complete the octet of that carbon and given that its

multiplicity is a doublet, how many neighboring protons will it have? Just one neighboring proton. Signal B is similar.

00:06:22:96 - 00:06:56:64
Instructor: Signal C is a CH3 group showing up as a singlet, meaning that whatever it's attached to, there are no neighboring protons. Signal D has an integration of one. It's a CH group, and we believe it's a quintet. It's definitely a multiplet, but we think it's a quintet. That would mean that there are four neighbors.

00:06:56:64 - 00:07:33:44
Instructor: We already see some CH groups that have a CH three next to them. They need one more neighbor. So, CH3 group with three and one making four neighboring protons, but we do need one more bond to satisfy the octet of that carbon. D or E is in a similar environment. And there we have the pieces of our molecule.

00:07:33:48 - 00:08:10:08
Instructor: We suspect that we have a CH3 group attached to a CH. That CH will then be attached to another CH, which would be attached to a CH3. Let's draw that portion of it. CH3 CH, to CH to CH3, and we have two bonds left to deal with. What else is remaining in our molecule?

00:08:10:08 - 00:08:54:50
Instructor: We have signal C, which is a CH3 group that for some reason, is not coupling to anything else. Based on our chemical formula, we have both an oxygen and a bromine remaining. This could be a bromine here, and then an oxygen with the remaining CH3 group attached to it. Let's check if this matches our chemical shifts. This group right here, which would be signal C, we know that when you have a CH3 group next to an oxygen, where should that show up?

00:08:54:50 - 00:09:23:55
Instructor: Go check your spectroscopy reference table. You've looked at your spectroscopy reference table, and you see that a CH3 next to an oxygen, should be somewhere between 3.3 and 3.6 with that lower end of the range representing the CH3, rather than a CH2 or a CH. We expect this CH3 to be around 3.3 PPM.

00:09:23:55 - 00:09:55:90
Instructor: It showing up at 3.4, that matches our signal right here, signal C. Now, we've still got four other signals, A and B, D and E that we want to try and match up. We recognize that what they're attached to is going to influence the amount of deshielding they experience. What is causing the deshielding is both the oxygen and the bromine?

00:09:55:90 - 00:10:31:22
Instructor: The things that are near the bromine are going to be the same pair, and the things that are near the oxygen are going to be the same pair. In other words, this group and this group will be either A and D or B and E. Then the other group, here and here, will be the

other pair. Let's see if we can figure out what causes more deshielding. Is it being near an oxygen or being near a bromine?

00:10:31:22 - 00:11:06:52
Instructor: Look at your spectroscopy reference table and see if you can figure out which group causes more deshielding. When you looked at your table, you saw that for a CH group, the high end of the range, we expect a CH next to oxygen to be deshielded around 3.6. While a CH next to bromine would be deshielded around 3.8 PPM.

00:11:06:52 - 00:11:58:74
Instructor: In other words, this pair of the CH and the CH3 should be more deshielded than this pair of CH and CH3. That tells us that B and E are going to be the pair close to the bromine. Since B is the CH3 group over here, that means that E is the CH, and the other pair A and D, A is the CH3 group, and D is the CH group. There we have the ether component, which is signal C, and we've been able to figure out all of the components of our molecule. Let's just draw one more time.

00:11:58:74 - 00:12:37:38
Instructor: We have a CH3 to a CH, to a CH to a CH3. We have a bromine here, and we have a methoxide over here. We've assigned each of the sets of equivalent protons within our molecule as being group B, group E, group A, group D and group C over here. There we go. This was a challenging problem.

00:12:37:38 - 00:12:59:66
Instructor: Don't worry if we need to rewatch this video a few times to get comfortable with the process that we used to solve it. Before you work through the next video, look at the spectra inside your book and see if you can solve it, then watch the video and see how you did. Good luck.