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iSpec: A Web-Based Activity for Spectroscopy Teaching

Thomas Vosegaard

Danish Center for Ultrahigh-Field NMR Spectroscopy, Interdisciplinary Nanoscience Center and Department of Chemistry, Aarhus University, Aarhus, Denmark.

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Manuscript type: Article (5000 words)

ABSTRACT

10 Student's skills in structure elucidation of organic molecules are developed by training them to understand advanced spectroscopic measurements and elucidate structures of small organic molecules from mass spectrometry (MS), infrared (IR), ultraviolet (UV), and ^1H and ^{13}C nuclear magnetic resonance (NMR) spectroscopic data. The present work puts specific focus on developing student skills to ensure that the students (*i*) are aware of the resources involved in obtaining the spectral data and (*ii*) obtain the skills necessary to handle spectra which have not been processed by experts, to better reflect the use of spectroscopic techniques in their later work life. This is achieved through the web-based spectroscopy activity iSpec, in which the basic idea is that every action costs resources. The cost of a resource is given in the form of resource points (RP), such that different experimental data can be "purchased" for a number of RP.

KEYWORDS

25 Second-Year Undergraduate Chemistry, Analytical Chemistry, Organic Chemistry, Web-based learning, Problem Solving/Decision Making, IR Spectroscopy, Mass Spectrometry, NMR Spectroscopy, UV-Vis Spectroscopy

GRAPHICAL ABSTRACT

INTRODUCTION

Training of student's skills in spectroscopy is essential for their future work in several areas of chemistry, and since the early days of spectroscopic techniques, this has been the case.¹ For example, our second-year class in spectroscopy serves as an indispensable tool for later classes in organic synthesis, analytical chemistry, and materials science. During the class, students learn to understand advanced spectroscopic measurements and elucidate structures of small organic molecules from mass spectrometry (MS), infrared (IR), ultraviolet (UV), and ¹H and ¹³C nuclear magnetic resonance (NMR) spectroscopic data.

The student skills are developed through learning the basic concepts of MS, IR, UV, and NMR and by interpretation of such spectra of small organic molecules, not only in

our curriculum, but as a general theme in every chemistry degree program. A typical curriculum of an undergraduate spectroscopy course has a strong emphasis on interpretation of experimental spectra.²⁻⁶ When interpreting spectra, students are normally instructed to extract *all* information from *all* spectra to ensure that they make conclusions that are supported by all the spectral information. In addition, some activities focus on only one spectroscopic technique⁷ and specific challenges in interpretation of such spectra.⁸ There are numerous examples of computer-based versions of spectroscopy problems,^{9,10} and teachers make use of spectroscopy databases like SDBS¹¹ to get a large and relevant set of exercises.¹² Finally, it is worth noting that many courses include lab exercises, where students do their own experiments.¹³ In our experience, this is a very good activity for student learning, but it is obviously challenging in terms of resources and capacity; We have chosen to put this in a more advanced course that is not part of the mandatory chemistry curriculum and hence with fewer students.¹⁴ We manage to do the experimental part with 50-60 students, who in groups of two acquire their own NMR, IR, MS, and UV spectra, but we do not have capacity to extend this to the mandatory course that is attended by ca 100 students.

Focusing on teaching forms that are scalable for many students, i.e. without lab work, we use the above approach where student practice comes through analysis of a large number of spectra. The exam tests the student's ability to extract all information from all spectra, and we generally see in the exams that the students indeed get the desired skills. However, the current setup has a few deficits that we would like to address. (i) The students are not encouraged to think of the resources involved in obtaining the spectral data; often it is sufficient to record only a subset of the spectra to save time. (ii) The students get handouts of the spectra, on which some interpretation has already been done (e.g. by plotting expansions of relevant spectral regions and

performing postprocessing), and hence they already get some help this way. Students need to master both points in their later independent work in the laboratory, for example in organic synthesis, where the analytical tools are used routinely to verify steps in a synthesis. For such a task, the aim is to verify a given structure by a minimum effort, implying that often a ^1H NMR spectrum is sufficient, in which case it would be a waste of time and resources to acquire IR, UV, and MS spectra. In other cases, it could be sufficient with an IR spectrum, e.g. to verify the presence of a carboxylic acid.

The goal of the present activity is two-fold; ensuring that the students (i) develop skills in prioritising which experiments are needed to shed light on a given problem and (ii) learn to do basic data processing such that they can manage data with no prior interpretation by experts. The first goal is addressed by asking the students to select which spectra they want, forcing them to take into consideration how much effort is required to obtain the experimental data. For example, acquisition of a ^{13}C NMR spectrum may take so long time that the spectrometer's queue system automatically postpones such an experiment to the night queue, where longer experiments are typically performed to keep the daytime free for shorter analyses such as ^1H NMR spectra. Under such circumstances, it is disadvantageous to use ^{13}C NMR if an immediate answer is required. Point (ii) above addresses the issue that when students are presented with exercises on paper, the spectra are typically prepared with peak labels, integrals, relevant expansions, etc, which is not the case when they work by themselves in the laboratory. The goal here is to provide the students with the raw spectra and tools to do the basic interpretation on their own. While point (ii) above has been addressed to some extent by other web-based activities^{9,10} or in lab work, the first point has, to my knowledge, not been addressed in detail as part of the undergraduate spectroscopy teaching. In our curriculum, this activity is implemented in a mandatory

second-year undergraduate chemistry spectroscopy course. The students are introduced to spectroscopy in the first-year organic chemistry course. The present activity builds on these competences, as the focus on resources is on a higher learning level.

THE ACTIVITY

To emphasize the use of resources in the process of determination of structures from spectroscopic data, we have implemented a web-based spectroscopy activity, iSpec.¹⁵ The basic idea of the activity is that every action costs resources, which in the activity is associated with resource points (RPs). Each exercise comes with a number of RPs that may be spent on “purchasing” the different spectra. A ^1H NMR spectrum typically costs 10 RP while a ^{13}C NMR spectrum costs 20 RP reflecting the longer experiment time of the latter experiment. MS spectra also cost 20 RP as the process of obtaining them takes time, while IR spectra cost 10 RP since they are generally very easy to obtain. We have chosen that UV spectra are available for 10 RP, although this may be slightly low given that it is often necessary to make time-consuming dilution series. On the other hand, these are experiments that may be done on relatively simple instruments, compensating for the time spent. Additional resources are the time used to solve a given exercise and obtaining the molecular mass with high precision from electrospray ionization (ESI) MS at the cost of 40 RP.

The activity is performed in a web browser¹⁵ on either a computer or a portable device and starts with two windows (see Fig. 1). The window on the left (titled “General”) lists available experiments for the exercise and gives other information. The molecule-drawing window (titled “Molecule”) is for drawing the molecular structure in question. The exercise is solved when the correct molecular structure is drawn in the molecule drawing window. An exercise may be configured with a certain intro-text that sets the

scene for the given exercise, e.g. with a text explaining how the compound was achieved
or other relevant information that may help guiding the students.

Figure 1. Overview window for the iSpec exercise titled Training 1. The available spectra and their “price” (resource points, RPs) are shown in the General window. The Molecule window is used to draw the molecular structure.

When “purchasing” a spectrum, an interactive window that displays the data is opened, as illustrated for UV, IR, MS, and various NMR spectra in Fig. 2. The spectrum windows provide the possibility to zoom and peak-pick interactively. For NMR spectra, it is also possible to integrate peaks and change between ppm and Hz scales using the jsNMR tool described elsewhere.¹⁶ Figure 3 shows an example, where the student has zoomed in on a region of a 1D ^1H NMR spectrum and performed integration and peak picking of selected peaks.

Figure 2. Typical spectra available for a given exercise displayed in the interactive windows of iSpec. Basic visualization and processing features (e.g. zooming, peak-picking, peak integration) are available. The type of spectrum is given in the title bar of each spectrum.

Figure 3. User-processed ^1H NMR spectrum zoomed to a region of interest, with peak picking and integration of selected peaks.

Figure 4. Utility windows providing extra insight or help to solve an exercise. (a) Calculation of chemical shifts for benzene rings by selecting substituents from a dropdown list. (b) Simulation of spectra for weakly coupled ¹H spins. (c) Listing of possible molecular formulas based on exact mass with a given precision. The two examples show the possibilities when the mass is determined with two decimals (top) and four decimals (bottom) precision. To help filtering out unlikely formulas, the number of double-bond equivalents (DBE) are shown. Note that the windows shown in this figure come from different exercises and hence do not refer to the same molecule. For the calculation of benzene chemical shifts, the columns H_{ortho}, H_{meta}, ... represent the value for the position (ortho, meta, ...) relative to the functional group. For example, the H_{ortho} contribution (-0.14 ppm) of the ethyl group is added to H5 and H1.

In addition to obtaining spectra for structure elucidation, iSpec offers a number of tools to help the students understanding the spectra. These tools are illustrated in Fig. 4. For example, a tool for calculation of ¹H and ¹³C chemical shifts in substituted benzene rings is available (Fig. 4a). Additionally, students may use the tool to predict the ¹H coupling patterns caused by *J* couplings (Fig. 4b) or use the formula calculator to obtain suggestions for the chemical formula of the molecule based on the exact molar mass. For small organic molecules, knowing the mass with two digits precision will typically provide numerous possible solutions, while the molar mass with four digits precision will only return a single formula, as illustrated in Fig. 4c.

When the student purchases a spectrum, the activity timer starts. Since the time spent on an exercise is also considered a resource, some of the exercises are configured such that time spent on the exercise costs 1 RP/min. This feature can be configured separately for each exercise.

Once the spectra are analysed, the student submits the correct structure by drawing it in the molecule window (see Fig. 1), created by the JSME JavaScript tool.¹⁷ Internally, the structure is converted to the simplified molecular-input line-entry system (SMILES)¹⁸ format, which is compared to the correct solution. Submission of a wrong answer will be penalised by withdrawal of 5 RP, to prevent students from doing too many unqualified guesses. Submitting the correct solution ends the activity, and the student is rewarded 40 RP for his/her achievement which, together with the remaining RP are converted into experience points (XP). The motivation for doing this is that students get more experienced the more exercises they solve. The experience points are accumulated for all exercises and potentially from course to course. This way, students can compete on who gets most XP during a class.

Hints

Hint 1
In the IR spectrum, there are two peaks at 1820 and 1750 cm⁻¹. What could be the origin of these two peaks?

Carboxylic acid

CO₂

Carboxylic acid anhydride

Carboxylic acid chloride

Show correct
Submit answer

Questions

Question 2 (10 XP)
Determine the chemical shift for C2

☐ Alkane ☒ Alkene ☐ Benzene

Shift		136.0
-COOR	1	6.3
-OR	1	29.0
-Et	2	-9.7
-CH ₂ OH	2	-8.4
Steric	Cis pairs	-1.1
Steric	Cis pairs	-1.1
Steric	Geminal pair C1	-4.8
Steric	Geminal pair C2	2.5

Show correct
Submit answer

Figure 5. Example of a hint (left) that may guide the student to the correct solution to an exercise, in this case through a guiding multiple-choice question. The answer is given by clicking the correct box, here highlighted with a yellow frame. Example of a question (right) allows probing the student knowledge on a specific topic related to the exercise, here in the form of a ¹³C NMR chemical shift calculation. When starting, the table is empty. A new contribution is added by the "Add row" button. Note that the present table is larger than the window, so the user should scroll to see the bottom rows of the table.

If the student runs out of RP for an exercise (e.g. after too much time or too many wrong guesses), the exercise changes to a so-called training mode, where all spectra are unlocked such that the student has better possibility to find the correct structure. Additionally, a few hints may be offered to guide the student towards the correct answer. In training mode, the award for answering is lowered to 20 RP, as some help has been provided. Figure 5 shows one example of a hint. The series of hints should lead to the right solution. As hints are unlocked when the student runs out of RP, they are provided at no cost. Hints offer additional learning for the students facing problems in determining the correct structure and should not be considered part of the “competition” in solving the problems using as few resources as possible.

Once the correct solution is submitted, iSpec may offer additional questions to verify the student’s insight and understanding. The questions are optional, but the motivation for answering is that it is possible to gain further XP by answering correct. Figure 5 shows an example of a question, where the students should demonstrate their ability to do chemical shift calculations using normal empirical formula, in this case with an implementation of the Grant-Paul algorithm.^{19,20} Other examples of questions may be multiple-choice questions related to other spectra. The idea behind the extra questions is to ensure that students reflect on all types of spectra, not only the popular ones they purchase to solve the structure.

RESULTS AND DISCUSSION

We have used the activity in the second-year chemistry course on spectroscopy⁵ with participation of 91 students. The activity was a supplement to normal exercises and lectures; it was not a mandatory part of the course, but students were strongly encouraged to use it. The major reason for not making it mandatory the first time was that the specific coupling between the resources spent and the data obtained has not

been a direct part of the learning objectives and exam problems of the course. The exercises were released gradually over the course with six training exercises at the beginning followed by two exercises per week during the seven-week course.

Figure 6. Statistics on the student activity during the course. (a) Student activity measured by the percentage of the students starting (yellow) and finishing (orange) the different exercises (91 students participated, no exercises were mandatory). (b) Distribution of spectrum “purchases” of the different kinds of spectra. Mass/2 refers to the molar mass ($\cdot M^+$) with two-digit precision. ESI/4 refers to the ESI-determined mass (MH^+) with four-digit precision.

Since the student activity (“purchase” of spectra, guessing the structure, answering questions) is logged by the iSpec server, we can provide statistics on the usage. This insight can help adjusting of the teaching and work load during the course. Figure 6a shows the percentage of students who have started and finished each exercise at the end of the class. Midway in the class, the students were confronted with the fact that the percentage was decaying during the class. They gave two useful answers: (i) They were too busy with other classes to engage in optional activities, so they suggested to make some of the iSpec activities mandatory and (ii) they planned to use iSpec as part of the preparation for the exam in the class. Accordingly, we have considered that next year, one or two of the iSpec exercises may be made mandatory and must be solved in order to qualify for the exam. When discussing the activity with the students at the

lectures, they have generally been very enthusiastic about iSpec, but it has not been possible to obtain any statistics on the student satisfaction or their view on the learning outcome.

The normal written exam in our spectroscopy course does not include exercises of this type, but we plan to modify the exam accordingly to include iSpec-type exercises. As it may be difficult to assess and grade the iSpec exercises based on the student's selection of spectra, we have not found a good way to integrate this into the exam. However, the fact that iSpec offers the possibility to give the students spectra that have not been subjected to prior analysis by the teachers (e.g. peak picking, phasing, etc), is still relevant and which we will exploit in the future exam problems. For example, we plan to provide all experimental spectra for the next exam through the iSpec/jsNMR visualisation interface, this way requiring the students to demonstrate their skills in processing experimental data.

In the present implementation, there is no control of whether students solve the exercises individually or they collaborate/exchange information in groups. When the library of exercises grows, this may be implemented by randomising the exercises given to different students. However, as the exercises are aligned with the lectures, it will require a very large database of compounds to achieve this. The alignment means that, for example, when students have learned about the isotope patterns of $^{79/81}\text{Br}$ or $^{35/37}\text{Cl}$, we use problems with Br- or Cl-containing for the exercises that week. This is challenging for randomisation.

Alternatively, since it is difficult to assess the student's ability to prioritise which spectra to obtain (there is no *correct* solution, but a number of reasonable choices can be made), iSpec can also be configured to run without resource points, in which case it allows students to access all experimental data through the spectrum viewers. This provides a good environment for working with raw data, but does not allow to prioritise

data. We typically configure the first introductory exercises in this mode for the students to familiarize themselves with the interface and data processing.

In addition to reporting the overall participation in the different exercises, Figure 6a also reports on the number of students who manage to solve each exercise (orange bars in Fig. 6a). Comparing the number of students initiating the exercise (yellow bars in Fig. 6a) with those finishing, we observe that out of the total of 1062 exercises initiated, the students managed to solve the exercises in 943 cases, corresponding to 89% success. Digging deeper into the numbers, we can see that 75% of the successful answers were solved without hints, implying that in 67% of all exercises, the students managed to solve the exercise without help. For comparison, 60% of the students came up with the correct molecule at last year's written exam. Despite these promising numbers, we do not yet have sufficiently solid data to conclude whether the better overall result obtained with iSpec is due to improved learning or is just due to fluctuations.

We have analysed the distribution of spectrum "purchases" chosen by the students to solve the 18 exercises (see Fig. 6b). It is noteworthy that the students indeed select which spectra to obtain instead of just unlocking all spectra as seen from the large fluctuations in the percentage of spectrum purchases for the different types of spectra, in line with the philosophy of the activity. Overall, the distribution is not surprising; for small organic molecules, ^1H and ^{13}C NMR generally provide a superior level of detail than the other methods. It was somewhat surprising to observe that entry ESI/4, which refers to the ESI-determined mass with four digit precision, was very low. Especially considering that this knowledge will almost always provide the correct chemical formula of the molecule, this was surprising. When discussing this with the students, they told that they did not use this, since they suspected there was a bug in iSpec, as they never got any useful information from the high-precision mass. It turned out that iSpec was correct but that most students had missed the point that ESI provides a measure of the

protonated molecule,²¹ so they needed to compensate the ESI mass by the mass of H⁺ before they could use the formula calculator (see Fig. 4c).

The fact that we can monitor the student's actions during the problem solving allows us to adjust the teaching during the course. Based on the above example with the misunderstanding of the ESI MS interpretation, we promptly revisited the ESI principles in the class to avoid further confusion. One can envisage other adjustments in the future, for example, we could develop more exercises where UV spectra are more essential, if we find that the percentage of UV-spectrum purchases drops too low.

To monitor the student's participation and actions when performing the activity, we are developing a teacher back-end for iSpec, see Supporting Information. Currently this is under development, as we establish more experience from a larger user base that will provide a clearer picture of which information is relevant to extract from the database.

Each exercise may be accompanied by a short description of the problem in question. Such a description could be: "An acid-catalysed reaction between a carboxylic acid and an alcohol yielded the product investigated in this exercise", which may guide the students to consider the expected outcome of such a reaction. However, even if an exercise does not have such a description setting the scene, the priority of which spectra to obtain is still relevant; The students typically "purchase" the ¹H NMR spectrum, and based on their conclusions from this spectrum, decide on what further information is needed.

CONCLUSION

The web-based activity iSpec for practising the use of spectroscopic techniques in elucidation of the chemical structure of small organic molecules is presented. The two-fold aim of the activity is to develop the student's awareness of and ability to prioritise resources (spectra) to answer a given question and to work with data that has not been processed by experts. This is achieved by the present activity that will also stimulate

their interest through competition. The web-interface of iSpec allows executing the activity on any platform (computer, tablet or phone) and furthermore the backend database provides insight into the student learning and work load, both of which allow to make adjustments of the teaching during the class.

ASSOCIATED CONTENT

Supporting Information

A short description of the teacher back-end and link to the user manual of iSpec are given as Supporting Information.

AUTHOR INFORMATION

Corresponding Author

*E-mail: tv@chem.au.dk

ORCID

Thomas Vosegaard: 0000-0001-5414-4550

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REFERENCES

- (1) Martin, J. C. NMR spectroscopy as an analytical tool in organic chemistry. *J. Chem. Educ.* **1961**, *38* (6), 286.
- (2) UC Irvine, USA. Chem 203: Organic Spectroscopy http://ocw.uci.edu/courses/chem_203_organic_spectroscopy.html (accessed Oct 22, 2017).
- (3) Master Organic Chemistry. Organic chemistry resources. <http://www.masterorganicchemistry.com> (accessed Oct 22, 2017).
- (4) University of Cambridge, United Kingdom. Course Guides | Teaching. <http://www.ch.cam.ac.uk/teaching/course-guides> (accessed Oct 22, 2017).
- (5) Aarhus University, Denmark. Course description. <http://kursuskatalog.au.dk/en/course/65763> (accessed Oct 22, 2017).
- (6) Uppsala University, Sweden. Syllabus for Spectroscopic Methods for Organic Chemistry. <http://www.uu.se/en/admissions/master/selma/kursplan/?kKod=1KB465> (accessed Oct 22, 2017).
- (7) Fleischer, J. M. Organic Spectroscopy-A Capstone Experience. *J. Chem. Educ.* **2002**, *79* (10), 1247.
- (8) Angawi, R. F. Using a Problem Solving-Cooperative Learning Approach To Improve Students' Skills for Interpreting ¹H NMR Spectra of Unknown Compounds in an Organic Spectroscopy Course. *J. Chem. Educ.* **2014**, *91* (6), 823–829.
- (9) Merlic, C. A.; Fam, B. C.; Miller, M. M. WebSpectra: Online NMR and IR Spectra for Students. *J. Chem. Educ.* **2001**, *78* (1), 118.
- (10) University of Calgary, Canada. Interactive Spectroscopy Problems <http://www.chem.ucalgary.ca/courses/351/WebContent/spectroscopy/index.html> (accessed Oct 22, 2017).
- (11) National Institute of Advanced Industrial Science and Technology, Japan. Spectral Database for Organic Compounds (SDBS). <http://sdb.sdb.aist.go.jp> (accessed Oct 22, 2017).
- (12) Kandel, M.; Tonge, P. J. Personalized Combined Organic Spectroscopy Problems—Online and in the Lab. *J. Chem. Educ.* **2001**, *78* (9), 1208.
- (13) Shine, T. D.; Glagovich, N. M. Organic Spectroscopy Laboratory: Utilizing IR and NMR in the Identification of an Unknown Substance. *J. Chem. Educ.* **2005**, *82* (9), 1382.
- (14) Aarhus University, Denmark. Spectroscopy in Organic Chemistry. <http://kursuskatalog.au.dk/en/course/65764> (accessed Oct 22, 2017).
- (15) Thomas Vosegaard, iSpec overview, <http://www.ispec.dk/> (accessed Oct 22, 2017).
- (16) Vosegaard, T. jsNMR: an embedded platform-independent NMR spectrum viewer: jsNMR spectrum viewer. *Magn. Reson. Chem.* **2015**, *53* (4), 285–290.
- (17) Bienfait, B.; Ertl, P. JSME: a free molecule editor in JavaScript. *J. Cheminformatics* **2013**, *5* (1), 24.
- (18) Weininger, D. SMILES, a chemical language and information system. 1. Introduction to methodology and encoding rules. *J. Chem. Inf. Comput. Sci.* **1988**, *28* (1), 31–36.
- (19) Grant, D. M.; Paul, E. G. Carbon-13 Magnetic Resonance. II. Chemical Shift Data for the Alkanes. *J. Am. Chem. Soc.* **1964**, *86* (15), 2984–2990.
- (20) Williams, D. H.; Fleming, I. *Spectroscopic Methods in Organic Chemistry*; McGraw Hill Education, New York, 2007.
- (21) Cody, R. B. In *Applied Electrospray Mass Spectrometry*; Pramanik, B. N., Ganguly, A. K., Gross, M. L., Series Eds., Practical Spectroscopy, Marcel Dekker, New York, pp 1–104.

ispec.pastis.dk

iSpec exercise: Training 1

✓

▶

General

Thomas Vosegaard

tv@chem.au.dk

XP: 6121

Logout

Exercise

Training 1 (100 RP)

New mode

Resource points: 100

Elapsed time: 0:00

Available spectra

UV (10 RP)

IR (10 RP)

MS (20 RP)

¹H NMR (10 RP)

¹³C NMR (20 RP)

Help / Questions

No help or questions

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JSME Molecular Editor by Peter Ertl and Bruno Bienfait ▶

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Benzene chemical shifts

Calculation of chemical shifts in substituted benzene rings

$$\delta(^1\text{H}) = 7.27 + \sum H_i$$
$$\delta(^{13}\text{C}) = 128.5 + \sum C_i$$

	¹ H	¹³ C	H _{ortho}	H _{meta}	H _{para}	C ₁	C _{ortho}	C _{meta}	C _{para}
1 H	7.27	128.1	0.00	0.00	0.00	0.0	0.0	0.0	0.0
2 H	7.83	128.6	0.00	0.00	0.00	0.0	0.0	0.0	0.0
3 COCH ₃	—	135.0	0.62	0.14	0.21	9.3	0.2	0.2	4.2
4 H	7.83	128.6	0.00	0.00	0.00	0.0	0.0	0.0	0.0
5 H	7.27	128.1	0.00	0.00	0.00	0.0	0.0	0.0	0.0
6 CH ₂ CH ₃	—	148.4	-0.14	-0.06	-0.17	15.7	-0.6	-0.1	-2.8

b

J couplings

Insert J-coupling values and multiplicities and see the resulting NMR spectrum.

Version 1.2.0, Thomas Vosegaard

Add coupling Calculate Linewidth: 0.1

none dou tri qua qui sex sep oct non

J₁ = 17 ☐ none ☒ dou ☐ tri ☐ qua ☐ qui ☐ sex ☐ sep ☐ oct ☐ non

J₂ = 10 ☐ none ☒ dou ☐ tri ☐ qua ☐ qui ☐ sex ☐ sep ☐ oct ☐ non

J₃ = 7 ☐ none ☐ dou ☒ tri ☐ qua ☐ qui ☐ sex ☐ sep ☐ oct ☐ non

c

Formula calculator

Mass: 60.06 Execute

Formula	M/z	ΔM	DBE
H ₁₂ OS	60.06	-0.00	-5
H ₉ O ₂ F	60.06	0.00	-4
H ₁₁ NCI	60.06	0.00	-4.5
C ₃ H ₈ O	60.06	0.00	0
CH ₆ N ₃	60.06	0.00	0.5
C ₂ H ₈ N ₂	60.07	-0.01	0

Formula calculator

Mass: 60.0575 Execute

Formula	M/z	ΔM	DBE
C ₃ H ₈ O	60.0575	-0.0000	0

Hints

Hint 1

In the IR spectrum, there are two peaks at 1820 and 1750 cm⁻¹. What could be the origin of these two peaks?

Carboxylic acid

CO₂

Carboxylic acid anhydride

Carboxylic acid chloride

Show correct

Submit answer

Questions

Question 2

(10 XP)

Determine the chemical shift for C2

☐ Alkane ☒ Alkene ☐ Benzene

Shift	Add row	136.0
-COOR	1	6.3
-OR	1	29.0
-Et	2	-9.7
-CH ₂ OH	2	-8.4
Steric	Cis pairs	-1.1
Steric	Cis pairs	-1.1
Steric	Geminal pair C1	-4.8
Steric	Geminal pair C2	2.5

Show correct

Submit answer

a

b