# Adding Gas Chromatography−Mass Spectrometry Data to a Melting Point and Thin-Layer Chromatography Laboratory

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# Adding Gas Chromatography−Mass Spectrometry Data to a Melting Point and Thin-Layer Chromatography Laboratory

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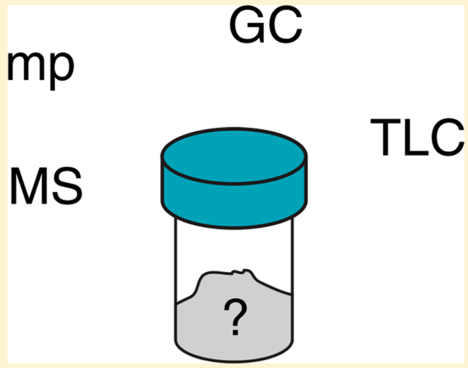
*Supporting Information*

**ABSTRACT:** The addition of gas chromatography−mass spectrometry (GC−MS) data interpretation to a thin-layer chromatography (TLC) and melting point (mp) laboratory for an introductory organic course is described. Students are given a sample containing one unknown and a list of 24 unknown compounds, organized in groups by similar mp ranges. Students identify the unknown through mp and TLC. Students are then given GC−MS data containing three unknowns and are asked to determine GC peak corresponding to their unknown using the MS spectra. Upon completion of the laboratory, students have gained an understanding of how many components are present given GC−MS data and how to identify each component from the molecular ion. They also become familiar with the physical characteristics of melting point, retention factor, and retention time. These skills are utilized in other experiments throughout the academic year




**KEYWORDS:** *Second-Year Undergraduate, Laboratory Instruction, Organic Chemistry, Hands-On Learning/Manipulatives, Problem Solving/Decision Making, Gas Chromatography, Mass Spectrometry, Physical Properties, Thin Layer Chromatography*

Melting point (mp) and thin-layer chromatography (TLC) are often utilized in organic experiments and are covered early in a laboratory curriculum to introduce students to the concept of using these properties for compound identification. There are several experiments in the literature designed as an introduction to mp,[1,2] TLC,[3−5] or both of these techniques.[6] Gas or liquid chromatography can be covered at the same time as melting point as both of these techniques rely on physical properties of compounds. Gas chromatography (GC) relies on similar physical characteristics (polarity and boiling point).

Spectroscopic identification of organic compounds is usually deferred until late first semester of organic chemistry or more frequently to second semester. Mass spectrometry (MS), although not a spectroscopic technique, is often covered with spectroscopic methods for compound identification both in lecture and the laboratory.[7] In the literature, there are examples of structure identification using combined techniques in an upper-level laboratory.[8−11] However, many undergraduate curricula include this in the second semester of organic chemistry.

Organic chemists rely on MS to confirm the molecular weight of a compound, determine the presence of certain atoms in a molecule (Br, Cl, N, etc.), and occasionally, to determine the most stable fragment ion (the base peak). Literature experiments that utilize MS often focus on full spectroscopic analysis,[7,9,12] identification of compounds by fragments,[13] or library searches for the best match.[14] These concepts require a familiarity with organic structures typically not achieved until later in the curriculum.

Early introduction of GC−MS analysis as an analytical tool allowed the use of this method throughout the academic year. The goals of the addition of GC−MS to this experiment were to have students determine the number of components by GC, identify each component by MS, and utilize GC−MS as a means of verifying product mixtures in future experiments.

## ■ HAZARDS

Gloves and appropriate eyewear are required. In general, the chemicals used may be harmful if inhaled, cause respiratory tract irritation, be harmful if absorbed through the skin, cause skin irritation, cause eye irritation, and be harmful if swallowed. See Supporting Information for specific hazards.

## ■ THE EXPERIMENT

Students were given a sample containing one unknown and a list of 24 unknown compounds, organized in groups by similar mp ranges (see Table 1 in the Supporting Information). Students determined the mp of their unknown to narrow the list to compounds with similar melting points. TLC analysis was performed with the unknown along with standards of the three similar compounds and the identity of their unknown was determined similar to the procedure outlined by Levine.[6]

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The GC−MS data for the mixtures of the eight groups of three melting point-related compounds was posted online according to group number. Students identified the appropriate group by the mp of their unknown and retrieved the GC−MS data during the laboratory period. Each student identified their unknown compound in the mixture by locating the molecular ion peak (molecular weight) (see the Supporting Information). The retention time for all three compounds was recorded and compared to the retention factor, $R_f$, determined by TLC.

At the end of the first semester (before a detailed discussion of mass spectrometry), a laboratory quiz was given showing the GC−MS of an incomplete reaction. The quiz results indicated that 65−70% of students have gained some understanding of GC and determined that the reaction was incomplete. Many (85%) students identified the starting material and product by their molecular ions in the mass spectra, determined the relative amounts of each compound (65%), and correctly utilized all the data presented in the GC−MS spectra (40%).

## ■ CONCLUSIONS

The introduction of GC−MS analysis to a physical characteristic experiment allowed the use of this instrumental method throughout two semesters of an organic chemistry laboratory course. GC−MS analysis was used in four additional experiments in the first semester and three experiments in the second semester. This has been successfully implemented in two academic years to over 400 undergraduate students. By the end of the first semester of organic chemistry, many students were simply able to match the correct molecular ion to their desired molecular weight, but this provided a strong foundation for the detailed discussion of MS the following semester.

The analysis of mixtures by GC−MS is a common modern method for determining purity and identification of constituents. In addition to the traditional methods of mp and TLC, GC−MS analysis allowed students to have a more complete understanding of the composition of a mixture and the confidence to support their answer with data analysis. By incorporating GC−MS analysis of reaction mixtures throughout the semester, students gained an understanding of how this technique is utilized by synthetic chemists.

## ■ ASSOCIATED CONTENT

### Supporting Information

Student experiment; instructor information; MS data for the unknowns. This material is available via the Internet at http://pubs.acs.org.

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### Notes

The authors declare no competing financial interest.

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