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Peak Race: An In-Class Game Introducing Chromatography Concepts and Terms in Art Conservation

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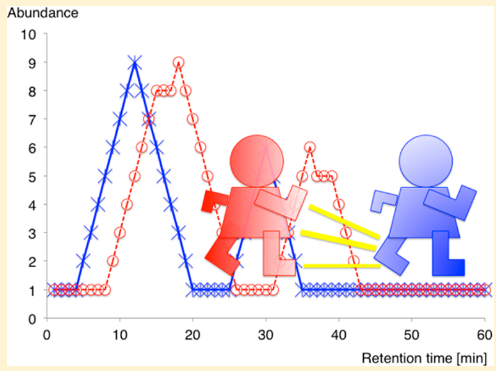
Supporting Information

ABSTRACT: Chromatography is an indisputably useful analytical technique in the Cultural Heritage arena, and as such it is part of the science Art Conservation curriculum at the Winterthur/University of Delaware Program in Art Conservation (WUDPAC). Future conservators must understand that development of a chromatogram is the result of different interactions with sample components, mobile and stationary phases, that result in observable peaks at specific retention times. Art and archeology analyses are special in that samples are usually scarce and contain aged and/or degraded complex mixture components. Broadening and overlapping of peaks can be two consequences of the variables, and oftentimes the logics behind such important outcomes can be challenging for students. Here we present an innovative teaching method in the form of a game to help in their understanding of a chromatogram development, why different compounds have specific retention times, and how peaks get broader and/or overlap.



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INTRODUCTION

The Winterthur/University of Delaware Program in Art Conservation (WUDPAC) relies heavily on science. During their second year, students enrolled in the program conduct a technical study on a historical, archeological, or artistic artifact using both spectroscopic and chromatographic techniques. The main goal of the technical study is that future conservators learn the techniques so they can decide which one(s) is (are) more suitable for a particular conservation-related problem: identify possible degradation causes, constituent and previous restoration materials, etc.

The one-year project is divided into two semesters: The first semester of science class includes several spectroscopies (X-ray fluorescence and diffraction; Fourier transform infrared, Raman, and ultraviolet−visible) as well as scanning electron microscopy; the second semester is entirely dedicated to chromatographic techniques (gas, thin layer, liquid chromatography).

Since most of the students in the Program will be conservation and not scientific professionals (such as Conservation Scientists), this practical exercise aims to help them in understanding the techniques to facilitate future conversations, collaborations, and projects with scientists and other conservators.

Although students have some chemistry background and many of them have even worked with conservation scientists or majored in Chemistry, for many the science curriculum can be challenging. Given a limited classroom time, instructors should make sure the basics are covered while considering that the application in conservation is the most important factor. Overall, in terms of the separation science component of the Program, it is desirable that after graduation students

- Have gained familiarity with chromatography concepts and applications.
- Understand why they, as users but not strictly interpreters of these analytical techniques, should know basic chromatography.
- Reason what can affect the result of a chromatogram.
- Make appropriate decisions about suitable and useful analyses for samples.
- Extrapolate what they know of a sample or an artifact to consciously choose an analytical technique.

It is worth highlighting that conservation is an inherently multidisciplinary field, and as such, it is of utmost importance to provide conservation students with useful information for their future careers and collaborations.

Teaching and learning chromatography can be especially laborious for students with minimal chemistry background. As a result, a number of novel and even witty approaches to chromatography have been published. Nagel[1] used "a trip to the mall" to (ref 1, p 93)

> *Illustrate the fundamentals of separations without the need for chemicals or any chemistry background.*

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Brozec[2] imagined a mixture that included wood pieces and denser objects such as pebbles. In this later case the "chromatography setup" is a stream (ref 2, p 83):

Flowing water is then the moving phase, and the bottom of the stream is the stationary phase.

Separation science in classrooms tends to be taught on a "case study" basis, and most of the time there are very few opportunities to actively engage the class on the development of a chromatogram. As a consequence many still struggle to understand why peaks overlap or broaden. Activities such as the "Virtual Laboratory",[3] the Gel Permeation Chromatography Simulator from JCE WebWare,[4] the "Separation Anxiety" game,[5] and the "Checkerboard Chromatography"[6] are indeed excellent ways to provide a dynamic and clear explanation to challenging chromatography-related aspects. Here we present a novel educational idea in the form of a game where students compete to develop chromatograms with sharp peaks. This game illustrates important concepts such as retention time, mobile and stationary phases, band broadening, and resolution. These, perhaps, are the concepts that for conservators are most important, but can be applicable to any audience that is starting to gain familiarity with chromatography.

■ THE GAME

Materials

For the game, dice and software to create graphs in real time (Excel, Origin, etc.) are needed. As instructors, we have to prepare chromatograph-looking mock-up graphs as well as questions for the topic we wish to review or evaluate in advance (see examples in the Supporting Information). The instructor arbitrarily assigns both x ("time") and y ("abundance") values.

Rules

There will be at least two teams. Each team will eventually develop a component of an "imaginary mixture". The two chromatograms are on the same baseline; hence they are read as sample components.

Each team has to roll the dice. The number they get is the number of "mobile phase points" or minutes that they get to move (x-axis, see Figures 1 and 2), but only if they answer a question correctly. In the case of our students, we asked questions related to spectroscopy, conservation, art, and art history such as "What technique can identify unequivocally the different phases of titanium white?" or "When was indigo first synthesized?" (see examples in the Supporting Information).

If the team does not respond or provides an incorrect answer, the other team has the opportunity to answer and by doing so "steal" the opponent team's points if they answer correctly. After having the chance to steal the opponent's points, they roll the dice again and get a question themselves.

This dynamic continues until the end of the chromatogram or the end of the "run", for as long as the "chromatogram" is "programmed" (in Figures 1and 2, it is 60 min) before the "next injection" (another game). The winner is the one with the sharpest peaks.

All figures in this paper illustrate a game with a 60 min run. This does not necessarily imply that the game is played in 60 min. Instead, each extra x value or minute that passes in the graph can be related to the difficulty of the question (the more time passes, the more difficult the questions). The instructor arbitrarily assigns abundance.

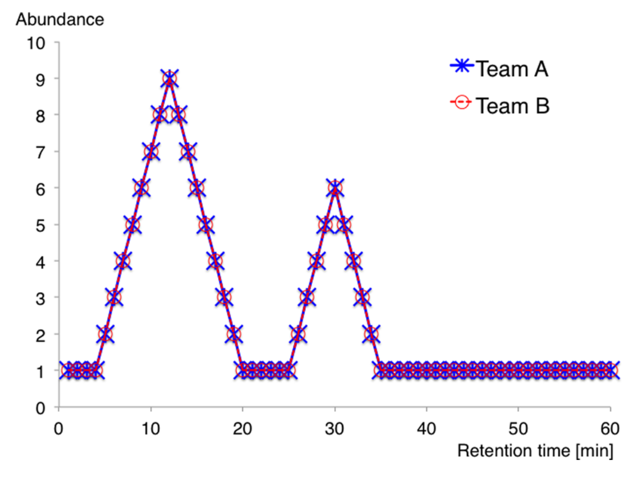

Figure 1. Ideal chromatogram. Notice that two teams (blue crosses and red circles) are generating the same two peaks (same sample components, possible overlapping of peaks).

Outcomes during and after the Game

When moving (getting "mobile phase points") after providing the correct answer, the team that moves eventually sees at least a peak appear (see Figure 2). When a team gets an answer wrong they still have to move but at the same signal height at which they were before on the x-axis, hence displaying longer retention times (Figure 2a) or broadening the peaks (Figure 2b).

To illustrate the retention times and how a peak can broaden, each team has up to 180 s for answering a question, but after 60 s, the instructor will add the same previous value to the graph every 10 s so that the band broadens up to "2 min" or two x-values (see Figure 2b). These additional and repetitive values will delay using the mock-up graph that instructors prepared beforehand, e.g., if they were at the 15th value (coordinate 15 min on the x-axis with an abundance of 8 on the y-axis) of the mock-up graph and they respond in 90 s, they will have three additional (15 min, 8 abundance) values, hence 3 min of delay and a broader peak by 3 min.

If a team does not respond correctly for more than one turn, the generated graph will look like Team B's in Figure 2b. A chromatogram like this can allow for discussion of resolution and peak broadening. If both teams respond to all answers right from the beginning, they can have a perfect chromatogram like Teams A's in Figure 2b. If something like this happens, it is an opportunity to discuss peak overlap or comparison to standards and substances with known retention times. It is encouraged that instructors have graphs where peaks are overlapped, perfectly and partially resolved, with different retention times and questions that are both easy and difficult to respond to so they expand the discussion possibilities (see sample questions for our Art Conservation second year students in the Supporting Information).

As this game continues, it is possible to create a relationship between the time teams get to respond and the actual retention time. For instance, we could think of difficult questions as those having higher retention times (they get more difficult questions as we move to higher retention times). These diverse retention times can allow discussions regarding the influence of analyte functional groups and the polarity of a column, for example. Students can also see that not answering quickly broadens peaks and can even lead to overlap, but if we help them with hints ("modify conditions"), it is possible to improve

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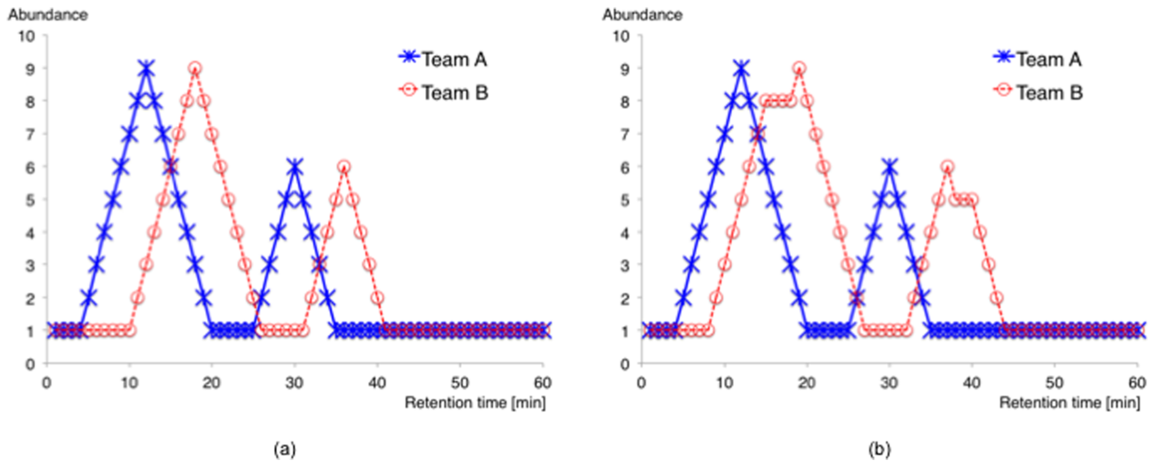

Figure 2. Development of "real time" chromatograms during class. (a) Team A (blue, crosses) is ahead of team B (red, circles, with a lower retention time) by 6 min ("mobile phase points") because team B did not answer a question. (b) Team A took a long time to answer two questions (at abundance 8 and 5), therefore broadening both peaks.

resolution. This translates to discussions on the use of different solvent mixtures, columns, flow, and the various parameters that can be adjusted to allow better resolution. Furthermore, the different chromatograms developed during this in-class game can be used as a springboard for introducing terminologies that are normally used in separation science (i.e., mobile phase, diffusion, affinity, etc.). One advantage of this game over other methods for introducing chromatography and related terminology is that the chromatogram is obtained in what can be considered "real time".

Additional Considerations

This is a simple and practical way to illustrate the basics of chromatography while simultaneously reviewing material from previous courses. However, this pedagogical approach only aims at an introductory level, and by no means will it emulate perfectly real case studies, so it is advised that these are also used. After completing the game, or perhaps as side discussions during the game, it is highly recommended that instructors discuss the chromatographic principles that influenced each peak, i.e., retention times and shapes. Instructors can decide whether or not to use examples from literature. If they have chromatography equipment available, perhaps they can have the same two mixture components separated with different solvent mixtures, different columns, at different rates, etc. so students can see what happens in real case scenarios and relate the game to these results.

■ CONCLUSIONS

We used this game during the first day of teaching the second semester of Analytical Techniques (Separation Methods) in the WUDPAC Program. With this game we illustrated the basics of chromatography and introduced separation science to our second year students. As we moved forward into the course, studying different chromatographic techniques and case studies, the class expressed how the game helped them to recall how the basic principles worked. It also aided in understanding why it is difficult to get good separations (if possible at all) and why in conservation science we can afford long runs. This is among the areas where we can compromise the speed, because we mostly care about good separation/resolution and sensitivity.

We successfully developed a pedagogical approach that served both as a review of the students' prior course (Spectroscopic Methods) and as an introduction to the separation science semester ahead. This helped them to gain familiarity with the chromatographic principles and separation science that are necessary for conservation practice. This exercise resulted in a fun and engaging way to understand why compounds have different retention times and what can be inferred by interpreting a chromatogram. This "peak developing race" group exercise also allowed them to suggest or request modifications to the settings of a run, for instance: if two peaks are not well resolved they can easily infer that more time, temperature/solvent gradients, or lower rates could provide a higher quality separation (terms introduced during the course).

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00326.

"Peak Race" example and sample questions for Art Conservation students (PDF, DOCX)
"Peak Race" graph (PDF, TIF)

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Notes

The authors declare no competing financial interest.

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