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Using Flavor Chemistry To Design and Synthesize Artificial Scents and Flavors

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Supporting Information

ABSTRACT: In this project for the organic chemistry laboratory, we bring together and expand upon two classic undergraduate organic chemistry experiments to create a flavor chemistry unit in which students design and develop a novel smelling product. The scents are derived from a synthetic angle, utilizing an expanded ester synthesis, and from a natural products angle by the isolation of fragrance molecules from natural materials using classic extraction techniques. Students are given an overview of flavor chemistry, and each group designs a novel product: a scented soap or candle. The project culminates in an oral presentation and a written report that is submitted in a journal style format for grading.

KEYWORDS: *Second-Year Undergraduate, High School/Introductory Chemistry, Organic Chemistry, Hands-On Learning/Manipulatives, Esters, Public Understanding/Outreach, Communication/Writing, Consumer Chemistry*

■ INTRODUCTION

The design of an artificial flavor or scent consists of two principal components, the molecules responsible for the scent itself and the diluent or medium in which the flavor is expressed. The diluent needs to keep the principal scent molecules in solution, inhibit chemical reactions, regulate the strength, act as a preservative, and determine the way the flavor or scent appears when it goes to market (liquid, paste, powder, etc.)

The design of the scent component is also complex. Most artificial and natural scents will have more than one molecule. For example the orange contains over two hundred molecules that give it a characteristic flavor and smell,[1] but an artificial orange drink contains only a few of those molecules. When developing a simple artificial scent or flavor, there are three components to the flavor or scent aspect: the character item, which is essential for the flavor or smell; the contributory item, which helps enhance the main flavor or scent; and the differential item, which gives the final combination a unique experience.[2] For example, a characteristic cherry flavor can be changed slightly to a black cherry flavor by adding acetophenone, which contributes to the black cherry experience.[3] In contrast, the differential item may bear little resemblance to the flavor molecule, both structurally and in scent/flavor. As an example, adding vanillin to a fruit flavor creates a "sweeter" experience while mint can create a "greener" experience. Often the differential item has little resemblance to the main flavor or scent.

Each of these compounds, the character (main scent), the contributory, and the differential, has distinct potency and volatility. They can also be combined in different ratios giving a different experience. Food product labels list the ingredients in order of decreasing amounts, but the exact ratio is not given. In addition, some components are listed as simply natural flavors. The exact molecules and ratios are trade secrets, and process flavors that are included in the human diet at low levels are generally recognized as safe to the consumer.[4]

Ultimately, some type of sensory assessment is essential for documentation of qualitative flavor characteristics of flavor and aroma chemicals.[4] In industry, a range of concentrations of a selected flavor compound in a defined medium (water, air, wax, etc.) are presented to a sensory panel, and each panelist indicates whether or not the compound can be detected. The experience of taste and that of smell are intricately linked. The act of chewing releases molecules that are received by special receptors in the roof of the nasal cavity,[5] indicating that there is a tremendous amount of overlap between aroma and taste.

This project for the undergraduate organic laboratory was inspired by the classic ester synthesis contained in most undergraduate Organic Chemistry Laboratory manuals in which students combine acetic acid and isopentyl alcohol in a Fischer esterification to synthesize isopentyl acetate or banana oil.[6] This same experiment can be expanded to a combinatorial approach in which students combine different carboxylic acids and alcohols to make a library of different smelling esters.[7,8] It provides a nice introduction to design and synthesis and one that we have incorporated into this series of experiments. In particular, we have explored how branching of either the

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alcohol or carboxylic acid can alter the final scent of the product ester.

Another classic experiment in most organic laboratory manuals is some type of essential oil isolation, which can include limonene from oranges, eugenol from cloves, or one of the terpines from pine needles.[9−11] We chose to focus on the orange oil extraction in order to work with a single theme of natural products. We expanded this experiment to include other not commonly isolated citrus oils. In part this was done to provide an array of scents to choose from, but also for their antimicrobial uses in product preservation.[12] The classic ester synthesis and essential oil extraction experiments are combined by students using their fragrances as additives in a consumer product.[13−15] Students thus apply organic chemistry to manufactured goods, encountering concepts related to scent composition, stability, and concentration.

EXPERIMENTAL SECTION

This series of experiments is conducted during the second semester of organic chemistry. Students work in groups of two. To save time, the first part (week one) is carried out at the end of a regularly scheduled laboratory.

Week One

Students design a product for a scented soap or candle. They are instructed to draw from the 3 components of an artificial scent: A principal scent or character item, a contributory item, and a differential item. Table 1 represents some ideas, but students are encouraged to go beyond the suggested scents. Each group designs its ester synthesis from a list of available carboxylic acids and alcohols (Figure 1) and extraction from a citrus fruit (orange, lemon, lime, or grapefruit). The differential item is provided or borrowed from another group. Students are given a brief lecture on the contents contained in the introduction of this paper and provided with specific instructions in their lab manual (see Supporting Information). They are encouraged to look on the Internet for ideas. We also make available a copy of a supplemental textbook.[4]

Table 1. Examples of Molecules Used as Character, Contributory, or Differential Flavor or Scent Items

Character Item (Essential Flavor or Scent)	Contributory Item (Enhances Flavor or Scent)	Differential Item (Makes Flavor or Scent Distinct)
Isopentyl ethanoate (banana)	(*R*)-(+)-Limonene (orange)	Vanillin: "sweet" (vanilla)
Heptyl benzoate (peach)	Lemon oil (multiple molecules)	Methyl benzoate: "green" (spearmint)
Ethyl butanoate (pineapple)	Octyl ethanoate (oranges)	Myristicin: "nutty, sweet" (nutmeg)

Week Two

Students perform a Fischer esterification as previously described[6] (Figure 1).

Week Three

Students isolate an essential oil from a citrus fruit using steam-distillation and extraction as previously described.[10] Each group obtains an IR spectrum and ^{1}H NMR spectrum of their ester and natural product to be included in their laboratory report.

Week Four

Students prepare their product scent. Students begin with a suggested ratio of 4:2:1 (in drops: principal:contributory:differential) and repeat until they achieve the exact scent experience they desire. Once they have determined the ratio, they can scale up the mixture to a final volume of 3 or 4 mL. Soap or candle wax is heated in the microwave oven until it melts and allowed to cool to 85−95 °C. If the soap is too hot, the scent molecules may evaporate and be lost. The scent mixture is added, and a few drops of food coloring (optional: from the grocery store) may also be added. The mixture is poured into a cast obtained from a craft store. Students may use their own soap made in a prior experiment.[16,17] Alternatively, they can use soap or candle wax from a kit. The latter is recommended when working with high school students.

HAZARDS

The experiments described in this procedure consist of a variety of chemicals, and each group prior to starting laboratory work should always consult MSDS data sheets for their individual experiment and wear proper safety attire. In particular, ethyl acetate, dichloromethane (also a carcinogen), and the hexanes are volatile and highly flammable. Carboxylic acids can be irritating to the upper respiratory system. If the students make their own soap, they should note that sodium hydroxide is corrosive. Some students may be sensitive to concentrated scents, and all products should be treated with the same precautions that are used when smelling anything in the organic lab. When it is necessary to smell a chemical in the lab, use proper smelling techniques (wafting) as demonstrated in the "Starting with Safety" video made available by the ACS.[18] Final scent molecules and synthesized soaps may not be pure, so students are instructed **not** to take their soaps home.

RESULTS AND DISCUSSION

The novel product design project ties in nicely with several experiments that we have used for many years in the organic chemistry laboratory curriculum. Natural product isolation reinforces the skills of extraction and separation. The ester synthesis emphasizes design and synthesis of organic compounds. Both experiments reinforce confirmation of structure by spectroscopy. The product design project is a nice way to finish out organic chemistry, emphasizing the applications of synthesis and purification and also encouraging original ideas and scientific writing skills.

The project culminates in a written report that is presented for grading in a journal style format. The project is fun, and student enthusiasm and engagement are very high, with more than 75% of the students receiving a grade of B+ or above on the grading rubric,[19] thus reinforcing scientific writing skills. The final products are presented at a college-wide symposium in an oral poster format, with groups of 4 students presenting a single poster. This was a good introduction to scientific meetings, and one poster took third place in the college-wide judging. Table 2 contains samples of the best flavor combinations and ratios. Students were encouraged to smell and rate each other's products.

Several of our science education majors have taken ideas from this experience for inspiration in their student teaching assignments and volunteer activities. During the summer months, we placed two ACS Project SEED students[20] on this project to test some extractions and flavor combinations. The Project SEED students are new to the laboratory, so we adapted the project to the high school level by using commercially available extracts and soaps from a craft kit.

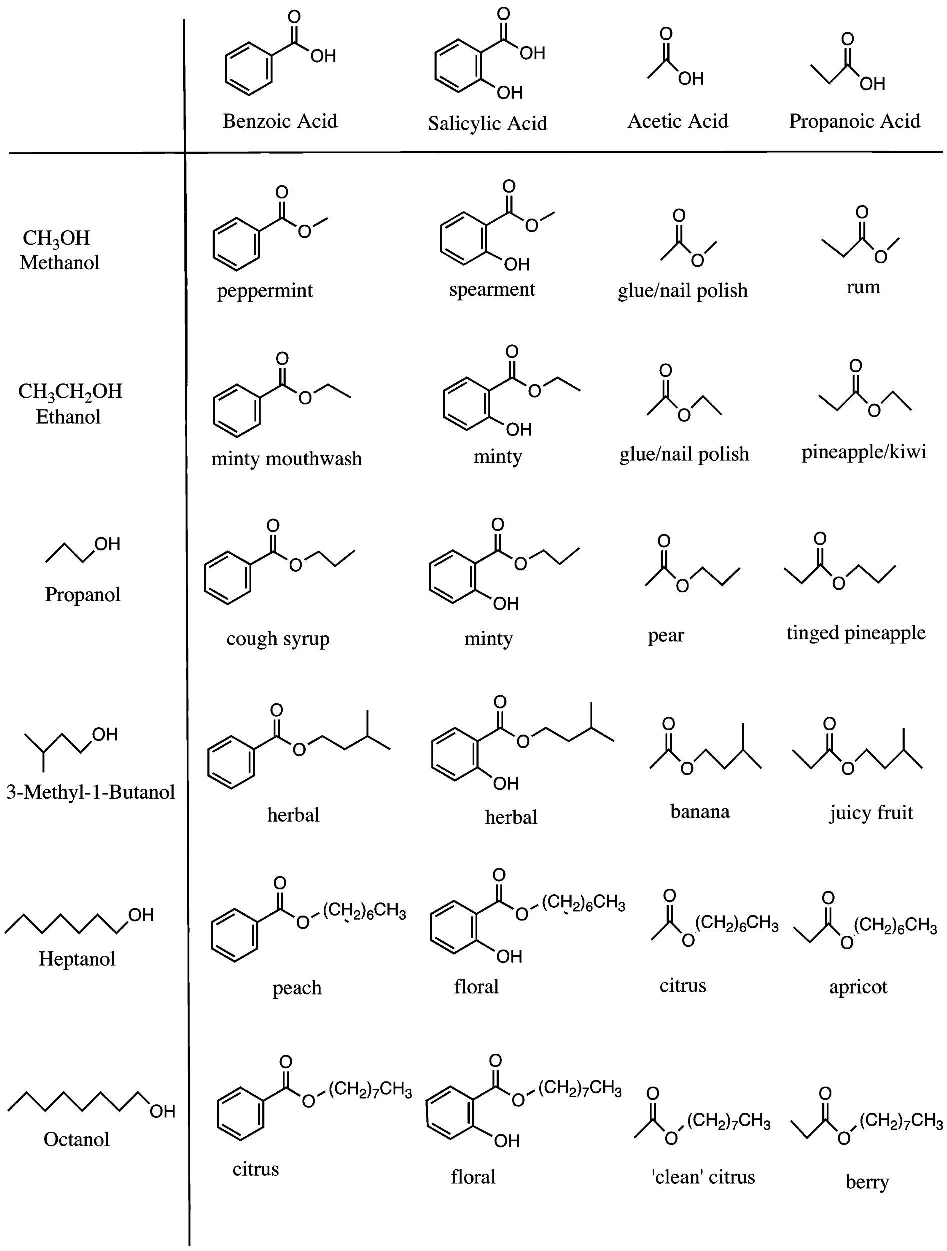

Figure 1. A combinatorial approach to ester synthesis using available alcohols and carboxylic acids. Additional information can be found in the Supporting Information.

Table 2. Sample Flavor Combinations and Ratios

Combinations of Flavors[a]	Ratios	Flavor or Scent Experience
Banana:lemon:coconut	2:2:1	Lemon poppy muffin
Banana:lemon:nutmeg	2:2:1	Crisp, juicy gum
Lemon:mint:coconut	2:2:1	Fresh, sweet with hint of lemon
Orange:peach:nutmeg	1:2:1	Crisp, sweet orange

[a]The first component is the character item (obtained by either synthesis or extraction), the second component is the contributory item (obtained by either synthesis or extraction), and the third component is the differential item (borrowed from another group or purchased). In the examples given, banana is the ester isopentyl ethanoate; mint is the ester methyl benzoate; and peach is the ester heptyl benzoate. Orange and lemon are extracts containing predominantly (+)-limonene along with other compounds. Nutmeg is an extract containing myristicin. Coconut is a store-bought extract.

One of our project SEED students presented his project and placed first at a regional science fair. This project has thus proved versatile and engaging for a wide range of students.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures; materials distributed to students; grading rubrics. 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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