3D printing lifts the lid on black box instruments

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Introduction

The use of additive manufacturing (3D printing) in educational contexts has grown exponentially in the past several years [1–3]. Within the educational community, 3D printing has two primary forms: usable laboratory equipment/experiments [4–11], and in-class demonstrations/models, including orbitals [12, 13], surfaces [14, 15], molecular models [16–18], biological molecules [19–21], and data visualization [22, 23]. While numerous laboratory examples have demonstrated the analytical use of 3D printing in the educational laboratory, little work has been demonstrated in the literature for analytical educational aids. A recent survey of over 450 papers describing 3D printing in biological education found only 13 studies that assessed benefits to students [24]. Of the four that measured changes in students' conceptual understanding, all saw improvements when students used printed models. Of the 8 studies that surveyed student satisfaction, all reported positive student perceptions. These gains are consistent with object-based learning (OBL) which hypothesizes that tactile interaction with objects facilitates student learning—allowing them to develop their understanding and better visualization concepts [25, 26].

For the modern chemist, a thorough understanding of chemical instruments is paramount towards understanding

the data obtained by instruments in daily use. In addition, proper selection of instrumental parameters for a specific experimental process can only be achieved if the underlying principles of separation, detection, and/or physical processes of the instruments are fully understood. Modern gas chromatography serves as an excellent example in this regard, where the selection of injection type (split, splitless, cold on-column, etc.), type of column, and type of detector(s) each play a role in the overall analysis, and the meaning of the data obtained from an experiment. Combining this with secondary information-rich detectors, such as mass spectrometry, and the complexity increases substantially.

However, the teaching of these concepts in the analytical classroom relies upon either images or direct access to instrumentation. Textbook images are 2-dimensional and static. Several manufacturers and educators have developed videos demonstrating the inner workings of chemical instrumentation, some of which have been combined into video playlists for ease of access [27]. A curated list of videos is included in the [Supplementary Information](#). These videos provide excellent supplemental resources for student learning, but fail to provide students the hands-on experience to personally gauge the size and complexity of each component. In addition, since instruments are 3-dimensional, but often sealed to protect the instrument components and operators, it is impossible for students to see the working components. Instruments may be disassembled for students to see the inner workings, but most departments are loathed to allow students to take apart >\$100k instrumentation. As a consequence, chemical instrumentation remains a black box for many students who have only “seen” the inside through crude diagrams or photographs. In this work, 3D printing is applied to resolve this comprehension gap by removing the black box from instrumentation and providing a series of models and teaching tools for educators to use in the instrument/analytical classroom and labs.

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Materials and methods

3D design

All 3D designs were created in-house using OnShape CAD system [28] (Boston, MA). Models were based upon physical examples taken directly from instruments or were created as teaching examples based upon instrumental diagrams found in *Quantitative Chemical Analysis*, 10th ed. [29].

3D printing

Models have been printed at four separate institutions using four different 3D printers to confirm cross-platform applicability of the 3D designs. All printers used were fused deposition modeling (FDM) style printers which heat plastic to melting, and apply successive layers of plastic to build a model up. Printers confirmed to work include the inexpensive (~200 USD) Ender 3 Pro (Comgrow 3D Technology Co, Limited; South Tsim Sha Tsui Kowloon, Hong Kong) [30] and Prusa MK2/S (Prusa3D; Prague, Czech Republic) [31] and the more advanced Lulzbot Taz 5 (LulzBot; Loveland, CO) [32] and Ultimaker 3 (Ultimaker; Framingham, MA) [33], which are more expensive (5000 USD) but offer dual print head extrusion and automatic system optimization. All printers utilized Cura 4.9 [34] slicing software to translate 3D models (.stl files) into code readable by the printers. Prior to printing, all models were rotated in the Cura software to place the largest flat surface available on the build plate to avoid the need for excessive support structures. Several models require multiple parts to be printed and/or the addition of non-3D printed hardware for full functionality. Detailed directions for assembly and a bill of materials for each model are provided in the [Supplementary Information](#).

All 3D models were printed in polylactic acid (PLA) plastic, an inexpensive, simple to use, and structurally robust 3D printing filament available for 25–50 USD per kilogram of material (at the time of writing). As a biodegradable, renewably sourced plastic, PLA also has a distinct advantage over petroleum-based plastics from the perspective of green chemistry. As most models use significantly less than 50 g of material, a single kilogram spool may be expected to last through numerous models, and the inexpensive nature of the filament lends the capability of retaining multiple colors of filament to provide for visual teaching aids. Print settings were optimized according to Table 1 (these settings were chosen to produce consistent, high-quality models; higher speed or lower resolution settings may be used as desired but will require optimization prior to printing). Printing and assembly instructions and STL files for all models (stereolithography; a file type which all 3D printers may utilize natively) are provided in the [Supplementary Information](#).

Table 1 Confirmed print settings for optimal 3D printing using FDM style 3D printers

Setting name	Value
Layer height	0.1 mm
Wall thickness	1.0 mm
Top/bottom thickness	1.0 mm
Infill density	35%
Infill pattern	Grid
Print speed	60 mm/s
Enable retraction	Yes
Z hop when retracted	Yes
Enable print cooling	Yes
Generate support	Yes
Support placement	Everywhere
Support overhang angle	60°
Enable prime blob	Yes
Build plate adhesion type	Raft
For dual extrusion models	
Enable prime tower	Yes
Build plate adhesion extruder	User choice
Support extruder	User choice

Student surveys

A brief, optional, survey was provided to students who participated in Analytical Chemistry (CH335), an upper division chemistry majors offering which covers basics of solutions chemistry and instrumentation in a 1-semester sequence at Whitworth University. The models were utilized during the instrumental portion of this course. A copy of the survey tool is provided in [Supplemental Information](#). The survey was administered online, via Qualtrics, and was provided to all students who participated in the lecture course. Of the 22 students enrolled, 10 chose to respond.

3D models

Student learning objectives

All models demonstrated were developed with the purpose of addressing specific gaps in comprehension of the workings of the instrumentation during in-class discussion. Specific learning objectives for each model are given in the [Supplemental Information](#) document “Learning Objectives.” This document provides instructors with a framework for the use of the models, simulations, and videos as provided herein. The overarching learning objective of using these models, simulations, and videos is to allow students to develop hands-on experience with the 3-dimensional nature of chemical instrumentation that is critical to the proper function and maintenance of this equipment.

Spectrophotometry

Instrumental models were developed across the Analytical/Instrumental Chemistry curriculum, including models related to spectroscopy, mass spectrometry, chromatography, and sample preparation. While the field of instrumental chemistry is broad, models were chosen to represent the most common analytical instruments encountered by students in the typical instrumental analysis curriculum, as well as those which were available in disassembled form for directly modeling. Figure 1 combines the models created for the field of spectroscopy. Figure 1a demonstrates a monochromator model developed as an in-class teaching tool. This model includes an optional color graphic which may be printed and cut out to place on the bottom of the monochromator to demonstrate the typical light path through a reflective grating monochromator. If desired, inexpensive 1-inch round mirrors may be coupled with a simple LED light source (see Supplementary Information for construction and wiring instructions) and a 1/2-inch reflective grating to provide partial functionality of this model. Included in the attached files is an adapter that allows the user to use a small piece of a CD or DVD in place of the diffraction grating [35, 36]. While the optical characteristics of such a grating are not ideal, these do provide some separation in wavelengths.

When constructed, the grating may be rotated in place to demonstrate sweeping the diffraction-separated wavelengths across the exit slit. Two versions of this model are provided.

One uses a dual-print head capable 3D printer to print white plastic over the exit slit. This makes the diffraction pattern easier to observe. However, if a dual-head printer is not available, a single-color model is provided which may be printed in all white (or all black) to serve the same purpose. In the pictured model, a piece of white cardstock is used to display the diffraction pattern. This model has not been optically optimized for use with concave mirrors to allow beam collimation, but does serve to demonstrate the basic functionality of a monochromator. While the literature provides numerous examples of diffraction grating tools and examples [37–40], these often focus only on the action or physics of the grating and not on its application to spectroscopic measurements. Thus, the function of a monochromator may also be reinforced in class through a computer simulation of a diffraction grating (Fig. 2a). This was programmed in LabVIEW; both an executable file and the editable VI version of this software are available in Supplementary Information. Within this demonstration software, the instructor may alter the incident angle of the white light, as well as the details of diffraction (grooves/mm and diffraction order) for a reflective grating.

Figure 1b demonstrates the functionality of a Michelson interferometer. A printed sheet placed on the bottom of the model provides the wavelength demonstration with positionally appropriate wavelength interference markings for a movable piece labeled as mirror 2 (orange in the image). This is a non-functional model which demonstrates the stationary and

Fig. 1 Spectrophotometric models developed for 3D printing. **a** Monochromator semi-functional model allows in-class demonstration of diffraction and selection of a wavelength through rotating the diffraction grating. A color printout (available in [Supplemental Information](#)) was cut to fit into the monochromator to provide the light path image on the bottom of the monochromator. In **a**, white cardstock was placed in front of the exit slit to provide contrast for viewing the diffraction pattern. **b** Michelson interferometer semi-functional demonstration aid showing a movable and stationary mirror interfering along a set of wavelength measurements. Similar to **a**, a color page printout illustrates the light path within this model. **c** ICP nebulizer designed as a cut-away model, and **d** ICP torch designed as a cutaway model. The nebulizer and torch models may be connected using Keck clips

Fig. 2 **a** LabVIEW program demonstrating diffraction angle and color depending on incident angle, diffraction order, and number of grooves on a reflective grating (assuming 1-in² surface area illuminated). **b, c** Fourier transform demonstration software allowing the generation of a frequency-domain signal from a convoluted time-domain (b) and the generation of a time-domain signal from peaks with variable widths (c)

movable mirrors which provide a changeable interference pattern in a typical interferometer. The process of interference to create a time-dependent signal and deconvolution of a time-domain signal to the frequency domain through a Fourier transform (fast Fourier transform, FFT) may be reinforced through a computer simulation of the Fourier transform (Fig. 2b, c). This software provides two modes of operation, selected through tabs at the top of the screen: In (b) Sine Wave, individual frequencies may be input in the array on the

righthand side of the screen. When *Recalculate* is pressed, the time-domain and frequency-domain signals will refresh to demonstrate multiple, overlapping frequencies within a single signal and the process of deconvolution via FFT. In (c) Peaks, the reverse process is demonstrated, where Gaussian peaks may be created (given specific times, heights, and standard deviations) and the system performs a reverse FFT to demonstrate the fading signal inherent in a time-domain signal of broad peaks. The combination of these modes allows the

instructor to work through the process of Fourier transform in its application to spectroscopic or NMR-based measurements.

Figures 1c and d represent an inductively coupled plasma (ICP) nebulizer and torch, respectively. These models were designed in cutaway halves, which may be clipped together with printed plastic clips in order to demonstrate an entire part or the inner workings of the respective piece. In addition, the ICP torch may be connected to the nebulizer using a printed Keck clip to demonstrate the gas/sample flow patterns within this instrumentation.

Mass spectrometry

Due to the high vacuum requirements of mass spectrometers (MS), the mass analyzers within are rarely seen by undergraduate students. Instead, the workings of these instruments are commonly relegated to 2-dimensional diagrams or descriptions that fail to lend the true nature of the ion optics and dynamics that result in mass separation. Figure 3 demonstrates 3D printed models of several common mass analyzers. Figure 3 a and b demonstrate versions of the quadrupole mass analyzer (a—quadrupole ion trap, b—quadrupole). The quadrupole mass analyzers (a/b) are shown in three colors to delineate the polarity of the poles in a single snapshot of time and allow an instructor to detail the oscillating RF field that results in mass stability/instability within this space. For the quadrupole (3b), an optional (and removable) model of a single-tube secondary electron multiplier may be attached to the second blue holder to demonstrate ion detection in this analyzer (pictured in [Supplemental Information](#)). In class, this model was used in conjunction with a SIMION 8.1 [41] simulation of a quadrupole

demonstrating both stable and unstable ion trajectories within the quadrupole space. A recording of this simulation is available in [Supplementary Information](#). The quadrupole ion trap (QIT) (a) is presented in two different versions. The design on the right in Fig. 3a is fit in three pieces that are loosely assembled so that students or instructors may take the model apart and observe the full ring electrode and trapping region. The left-hand model is intended to be glued permanently to provide a cutaway view of the trapping region of this mass analyzer to demonstrate the direction of ion travel during the ejection and detection stages of mass analysis and MS/MS capabilities.

Figure 3 c and d demonstrate modern, high-resolution mass analyzers, the Orbitrap (d) and Fourier transform ion cyclotron resonance (FTICR) (c). The Orbitrap model was designed to allow a student or instructor to observe the unique center-electrode geometry of this mass analyzer while describing the ion resonance that is created using these electric fields, as well as the method of detection through Fourier transform on the resonant axial frequencies [42]. The FTICR, being a simple mass analyzer when the magnet is excluded, was designed as a small box, with colors and surface-etched indicators of magnetic field direction and emitter vs. detector plates. This box may be glued to provide a handheld object for discussion on the method of detection in this analyzer. The non-destructive nature of this analyzer is reinforced by the entrance and exit holes in the end plates of the analyzer model. The use of these models was combined with SIMION to demonstrate the ion motion within these mass analyzers through inject and detection sequences. Video recordings of these simulations are provided in [Supplemental Information](#).

Fig. 3 Mass spectrometry 3D models. **a** Quadrupole ion trap in two forms: cutaway and whole. **b** Quadrupole mass analyzer with detachable secondary electron multiplier (excluded here for clarity of image; shown in [Supplemental Information](#)). **c** FTICR mass analyzer model (magnet not included), showing the detector/RF plates as well as injection hole (blue). **d** Orbitrap mass analyzer model cutaway for demonstration purposes

Chromatography

As the fundamentals of a chromatographic separation are molecular in nature, the 3D models prepared for chromatography focus on the ancillary parts of a chromatograph, especially sample introduction and detection. Figure 4 demonstrates semi-functional models prepared for chromatography, including an HPLC injector model (a) and a GC solid-phase microextraction (SPME) fiber model (b). The diagrammatic model of an HPLC injector was designed in 3 parts, the main stator body (blue in image), the inner rotor (orange), and a 3D printed nut to hold them together. When assembled, this model provides a facsimile of the function of an HPLC injector by allowing the user to turn the inner rotor with the exterior handle (similar to manual HPLC injectors). This alters which ports in the stator (labeled on the outer housing) are connected, allowing an instructor to talk through the process of quickly switching between “LOAD” and “INJECT” modes of operation while the system is at operating pressure. Figure 4b represents an oversized model of a solid-phase microextraction (SPME) holder and fiber. Due to the limitations in printing size for a round object, this model is much larger than the actual devices used. However, it creates the same functionality of fiber retraction for sampling/

injection and was designed as an educational tool, similar to the HPLC injector. For teaching and demonstration purposes, this model was used to show how/when the fiber is retracted in sample and injection steps of the SPME process. While some misunderstanding of size is expected with the use of this model, the oversized nature was actually beneficial when used in a large lecture format as it was easy for students to see the operation as a demonstration. These skills are then easily translated to a traditional SPME fiber where conceptual errors in size were quickly debunked.

Figure 5 demonstrates models of gas chromatography (GC) components to be used as teaching tools in the classroom. The injection port (a) was modeled after an Agilent 7890 injection port. This model can be fully disassembled to allow an instructor to talk through each part (pictured is the main body, left; 3D printed liner, center; and cap with space for septum, right). The cap was printed in red to demonstrate that this part is generally extremely hot in a “real” instrument, with the heat-dissipating fins included as a talking point. In addition, the printed injector port was designed to scale, so it will accept actual injection port liners and septa. In this way, the model may be used in class or lab to allow students to “inject” an air sample through the syringe in order to gauge the pressure and technique

Fig. 4 HPLC/GC sample injection models. **a** Teaching demonstration model of an HPLC injection port. The orange handle can move to demonstrate the connections between the various ports on the injection port and allows an educator to talk through the functioning of this device. **b** Oversized SPME injector with inner fiber (white, lower) that may be pulled back into the barrel of the fiber holder during injection. An internal spring provides resistance in this motion

Fig. 5 GC instrumentation. **a** GC injection port modeled from Agilent 7890 gas chromatograph. Injector liner (black) may be printed (pictured) or commercial injection port liners will fit to demonstrate the use of these devices. **b** ECD model for GC detection of halogenated compounds. Green inset indicates radioactive foil. **c** FID model for teaching purposes. Upper yellow cylinder is the collector electrode, while lower yellow cone is the jet nozzle. Blue bead is removable to simulate the Rb bead used to switch from FID to NPD mode in this type of detector

required to pierce the septum in an actual GC experiment. Figure 5 b and c are both GC detectors, with (b) representing an electron capture detector (ECD) and (c) demonstrating a flame ionization/nitrogen phosphorous detector (FID/NPD). The ECD model was printed with dual-print heads to demonstrate the inner radioactive foil (green) and was designed as a cutaway model. The FID/NPD is a cutaway model printed in multiple parts that must be assembled by the user. However, the inner blue bead as pictured is a model of position of the Rb bead used to alter the flame chemistry for NPD detection. This bead is actually a small electronic capacitor heat welded into the yellow holder intended to demonstrate the function/positioning of the alkali bead in an NPD detector.

Sample preparation

The final category of instrumentation is those used to prepare samples for analysis in an analytical environment. Figure 6 demonstrates a semi-functional shatterbox used to finely mill and homogenize solid or particulate samples prior to further sample preparation or analysis. This model was printed in three parts: an outer housing (blue), and inner ring (red), an inner puck (blue). This is a dynamic model where a student shaking the model can observe

the semi-chaotic motion that allows reproducible homogenization of solid samples. A design was created for a Soxhlet extractor model (Supplementary Information), but the authors recommend the functional Soxhlet extractor 3D printed and demonstrated by David Cocovi-Solberg and Manuel Miró [43]. Their model may

Fig. 6 Shatterbox interactive model

Fig. 7 Student survey results following use of 3D models in an upper division analytical chemistry course. **a** Likert scale responses ($n = 8$) of student perceptions of learning outcomes. Error bars indicate 1 standard deviation from the mean. **b** Evaluation of individual 3D printed models from same group of students using the scale: did not use (0), very unhelpful (1), unhelpful (2), neutral/no opinion (3), helpful (4), and very helpful (5). Those models included in the survey but not used in the course are not included in this figure. Error bars indicate 1 standard deviation from the mean

be used practically or as a classroom demonstration similar to those described herein.

Student response

Students ($n = 22$) in an upper division analytical chemistry course were surveyed at the end of the course following use of 3D models during lecture. Figure 7 summarizes the responses received ($n = 8$). Not all models presented herein were available during this lecture course, so only those utilized are presented in Fig. 7. Figure 7a shows student perceived positive benefits to their understanding and ability to visualize the instruments, and favored greater utilization of 3D printed instrument components. The favorable student outcomes and satisfaction are consistent with the positive results for 3D printed models used in anatomy, dentistry, and biochemistry [24].

The same group of students were also asked to select the appropriate response for each 3D printed model: did not use (0), very unhelpful (1), unhelpful (2), neutral/no opinion (3), helpful (4), and very helpful (5). Figure 7b shows that students found the monochromator and quadrupole mass analyzer most helpful models. The high rating of the quadrupole shows that even simple models can be effective pedagogical tools.

Indeed, model simplicity is a guiding principle of the *Seeing and Touching Structure Concepts* program used to teach civil engineers structural concepts [44]. The ICP torch and spray chamber were the lowest rated models. However, colloquial comments throughout the course demonstrated student enthusiasm for all of the models that were utilized, resulting in numerous “ah hah!” moments throughout the course for various students.

Conclusions

Presented are a series of 3D models of analytical instruments for use in the analytical/instrumental chemistry curriculum at the undergraduate level. These models were prepared based upon actual instrumentation or diagrams of instruments, and were designed to provide instructors with hands-on tools to demonstrate the inner functionality of instrumentation beyond slide or board-based diagrams. Limited student survey results demonstrated efficacy of the models (in conjunction with traditional lecture material) in improving student knowledge in analytical instrumentation, and feedback from students was overwhelmingly positive in both the written surveys and in-person conversations. The models were designed for printing on inexpensive FDM printers

which allow for multiple color schemes and rapid production of scale-model, accurate 3D diagrams. However, some instructors noted that the resolution was too poor to truly see accurate representations in the models. This can be mitigated through the use of other printing methods (such as stereolithography), at the sacrifice of cost per item printed. Not considering the cost of the printer itself, models were printed at a materials cost of \$2 or less for each model. All 3D designs are provided in Supplemental Information and .stl files and provided to the community for free use.

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Author contribution All authors contributed to the study's conception and design. Models were designed by Levi Garza and Michael Jones, models were printed and tested by Caley Craven, the first draft of the manuscript was prepared by Eric Davis with contributions from Charles Lucy, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Data availability The datasets (models) generated during the current study are available in the Supplemental Information.

Code availability The code generated during this study is available in the Supplemental Information.

Declarations

Research involving human participants Student surveys were approved through Azusa Pacific University IRB panel as exempt.

Informed consent All surveys performed included an informed consent statement and acknowledgment thereof.

Source of biological material No biological materials were used in this study.

Statement on animal welfare No animal subjects were utilized in this study.

Conflict of interest The authors declare no competing interests.

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