

# Integrated Instructions and Solvent Polarity Indicators: Reducing the Complexity of First-Time Distillation

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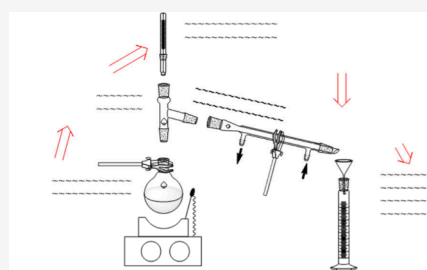
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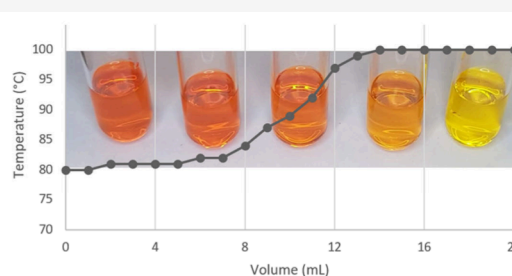
 Metrics & More

 Article Recommendations

 Supporting Information



Integrate instructions into diagram  
Work without splitting attention



Solvatochromic dye  
Visualizes distillate

**ABSTRACT:** Practical work carries a high cognitive load, particularly when unfamiliar or complex equipment is first introduced. “Integrated Instructions” have been previously used to reduce cognitive load in secondary education practical work by placing instructions within diagrams, reducing the need to integrate disparate sources of information. Here, we use this approach in an undergraduate-level distillation experiment to focus attention, reduce cognitive load, and make space for light “elements of inquiry” learning. The use of a solvatochromic dye as a polarity indicator also adds an unusual visual aspect to distillation, allowing students to easily estimate or verify the composition of fractions.

**KEYWORDS:** *First-year Undergraduate/General, Laboratory Instruction, Hands-on Learning/Manipulatives, Separation Science*

The undergraduate chemistry laboratory is a complex learning environment.<sup>1</sup> Students must manage multiple demands on their attention, some of which are caused by the innate challenge of the work itself (intrinsic cognitive load) and some caused by factors relating to how information is presented (extraneous cognitive load).<sup>2</sup> When the total cognitive load exceeds a student’s working capacity, they can fall back to less-effective practices, diminishing the cognitive benefits of the lab.<sup>3,4</sup>

A common strategy to reduce overall cognitive load is to reduce the extraneous portion by carefully designing learning resources. One such strategy in UK secondary education is the “integrated instructions” approach, where instructions are integrated into a schematic of equipment or a workflow.<sup>5–8</sup> Student attention is focused in one place, avoiding the extraneous cognitive load required to integrate multiple sources of information (the split-attention effect).<sup>9</sup> This integration may itself be a valuable skill and part of the requirement of operating as an expert chemist, but if a given experiment exceeds the working capacity of a learner, then they may fail to learn anything at all.<sup>10</sup> In particular, the recent work of Paterson provides a roadmap for introducing “integrated instructions” into existing practicals, by laying out some principles for adoption such as using clear numbering,

progressing in a logical order around a diagram, or using pictograms to reinforce operations (for example, an eye symbol whenever observations are made).<sup>6,7,11</sup>

## OUR CONTEXT AND DESIGN RATIONALE

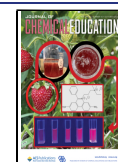
At the University of Strathclyde, approximately 120 students are enrolled annually into the first year of our chemistry program and undertake a mandatory laboratory course. In Scotland, higher education typically begins a year earlier than the rest of the UK, so this year sits at an equivalent academic level to the final year of schools outwith Scotland, or a university foundation year. The lab runs for a single three-hour session each week for 16 total weeks, split across two semesters and covering all areas of chemistry. Each session is preceded by a low-weighted prelab quiz (supported by videos and simulations) and assessed by a combination of lab reports

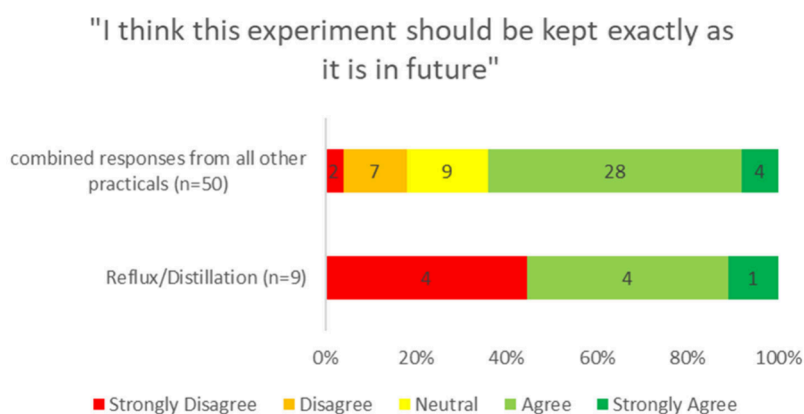
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**Figure 1.** Graduate Teaching Assistant responses to an end-of-module survey asking them to rate each of the experiments they had just taught. One question asked them to rate their agreement with the statement "I think this experiment should be kept exactly as it is in future" on a standard 5-point Likert scale.

and on-the-day worksheets. Sessions are delivered on a rota, so content is only weakly aligned with lectures. Students attend laboratories in groups of the same 12 students all year, with a consistent graduate teaching assistant.

Distillation is one of the most complex techniques that students encounter, with the most potential for misadventure and the least familiar equipment. Distillation was previously presented as a rushed part of an existing reflux/distillation experiment, where students needed to build a set of scaffolding to support glassware at two separate points, maintaining an airtight connection between flask, column, still-head, and condenser. Despite video and diagrammatic guidance, we often encountered equipment that had been set up incorrectly leading to breakages, escaping vapor, and the risk of heat burns or spillages of hot liquid. Instructor practices and materials necessarily focused on equipment and safety, and success was often reliant on direct intervention from instructors. Distillation products were colorless liquids, which required complex, slow, hands-off analysis to identify and left the student with minimal opportunity to explore, inquire, or investigate. The traditional experiment was entirely expository (recipe-based) in nature, with no opportunity for inquiry or experimentation, and the only learning objective was to "gain practice in distillation".

At the end of the 2017/18 academic year, as part of routine quality checks, graduate teaching assistants were surveyed about their perceptions of individual experiments and almost half of respondents strongly disagreed with the suggestion that the existing distillation sequence should remain unchanged (Figure 1).

This existing experiment sat within a first year lab module that otherwise contained numerous "elements of inquiry" experiments, a design strategy that gives students light experiences of inquiry-style lab learning very early in their higher education journey.<sup>12,13</sup> Inquiry-style lab learning is a popular instructional strategy that has students use inductive investigation to build research skills.<sup>14</sup> Inquiry can be introduced at various levels and intensities, from structured or guided inquiry all the way to full inquiry.<sup>15,16</sup> Our "elements of inquiry" approach introduces first year students to light aspects of experimental design and parameter choice early on, with the main aim of normalizing nonexpository approaches from day one.<sup>3</sup>

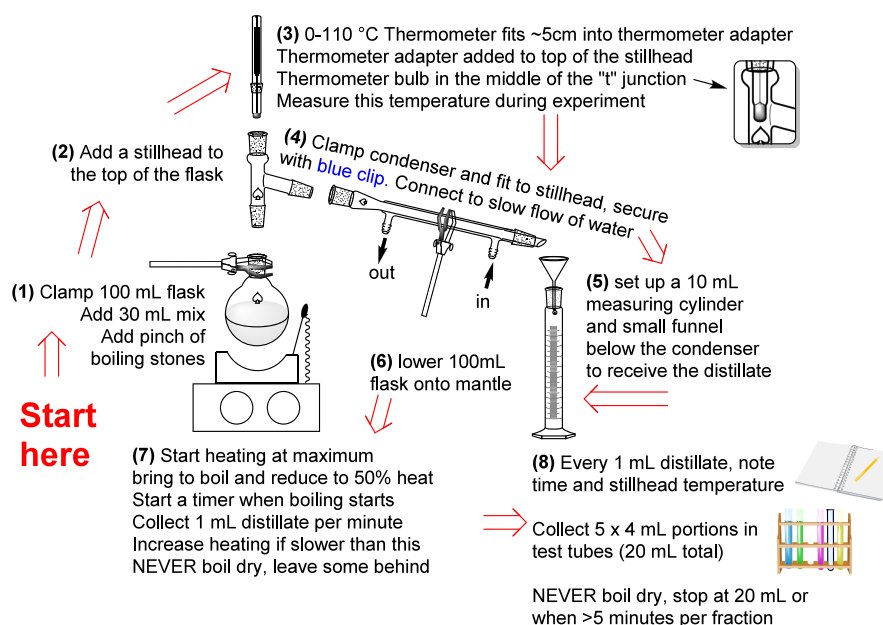
Typically, full inquiry laboratories often occur toward the middle or end of a degree, but there is a growing body of work that implements guided inquiry into first year laboratories, albeit usually as a multiday or multiweek sequence.<sup>17</sup> Of particular note is the work of Stewart et. al, who used guided inquiry to teach experimental design in a single-day first year lab sequence.<sup>18,19</sup> Inspired by these precedents and our prior work, we therefore sought to modify the existing distillation experiment, using the "integrated instructions" approach to clear extraneous cognitive load and make space for some "elements of inquiry" learning. Though lightweight by even introductory inquiry standards, these "elements" normalize nonexpository approaches and build toward future structured- or guided-inquiry sequences.

## THE EXPERIMENT

### Setup and Distillation Using Integrated Instructions

Prior to the lab, students now complete a prelab quiz built around best practices of supportive information, including safety questions and a short video of apparatus being assembled.<sup>20</sup> The video covers aims (separating a mixture by distillation), which are briefly reiterated during a teaching assistant welcome briefing in the lab itself. Students now work in pairs to follow a revised lab manual which includes a single main diagram with embedded instructions (the "integrated instructions" approach, Figure 2). Students work through the instructions around the diagram in order, constructing a typical distillation setup beginning with a flask clamped in a heat source, adding a still head and thermometer, and finally a condenser and some kind of receiving apparatus. Recognizing the potential for cognitive overload, this specific section of the experiment (construction and initial operation) was purely expository, allowing students to focus on equipment.

In a later section of the experiment, students were assigned a fractionating column (entirely open, or packed with glass wool) and asked to install it between the flask and still head before repeating the distillation. This required partially disassembling and reassembling equipment, and students were not given explicit separate instructions. This raised the cognitive load, but at a point where equipment handling is the sole operation under consideration and one of the objectives of the lab. In both cases, students were asked to record the temperature after each 1 mL of distillate, and collect a fraction every 4 mL. These collected fractions were analyzed for



**Figure 2.** Integrated Instructions diagram for assembling and conducting distillation. No other written instructions are given, though students watched a video of equipment being assembled as part of a required prelab activity.

ethanol/water composition using a “solvent polarity indicator”, a solution of solvatochromic dye. The specific learning objectives are now to “set up and conduct a short-path and long-path distillations at ambient pressure” and “use a calibration curve to semi-quantitatively analyze samples by color”. Although no calibration curve is plotted, the same base operations feed into a future analytical lab.

### Fraction Analysis Using Solvatochromic Dyes

A solvatochromic dye is a molecule that exhibits a large range of color in solution, dependent on the polarity of its solvent. The pH indicator bromophenol blue is solvatochromic and has previously been used to indicate the composition of a distillation column.<sup>21</sup> We did not choose this dye as it is also fully dependent on pH, though it could be used instead in cases of availability or color blindness. We instead used Brooker’s Merocyanine, which has been previously reported for educational applications as varied as photochemistry, polarity observation, and partition coefficient estimation.<sup>22–24</sup> Brooker’s Merocyanine is pale yellow in aqueous solution, reddish-purple in 95+% ethanol, and a range of intermediary colors at other compositions (Figure 3, top). It is commercially available, and can also be synthesized in a procedure straightforward enough to have been previously reported in this journal as a hands-on student activity.<sup>25</sup> A dilute solution of the dye in 40% aqueous ethanol will keep for several months in a dropping bottle and can be used directly by students as a consistent, reliable, black-box “solvent polarity indicator”.

Brooker’s Merocyanine will remain yellow in acidic water/ethanol mixtures, rarely caused by residual surfactant in poorly rinsed test-tubes. Unlike bromophenol blue, the merocyanine dye remains fully polarity-sensitive at neutral or basic pH, so the dispensing bottle can be formulated with alkali already included, although this reduces its solution shelf life to about one month (See SI for full synthesis, formulation, and stability testing information). Alternatively, a few drops of dilute bench sodium hydroxide solution can be added to any problematic fractions, although students will likely not spot when this is necessary.

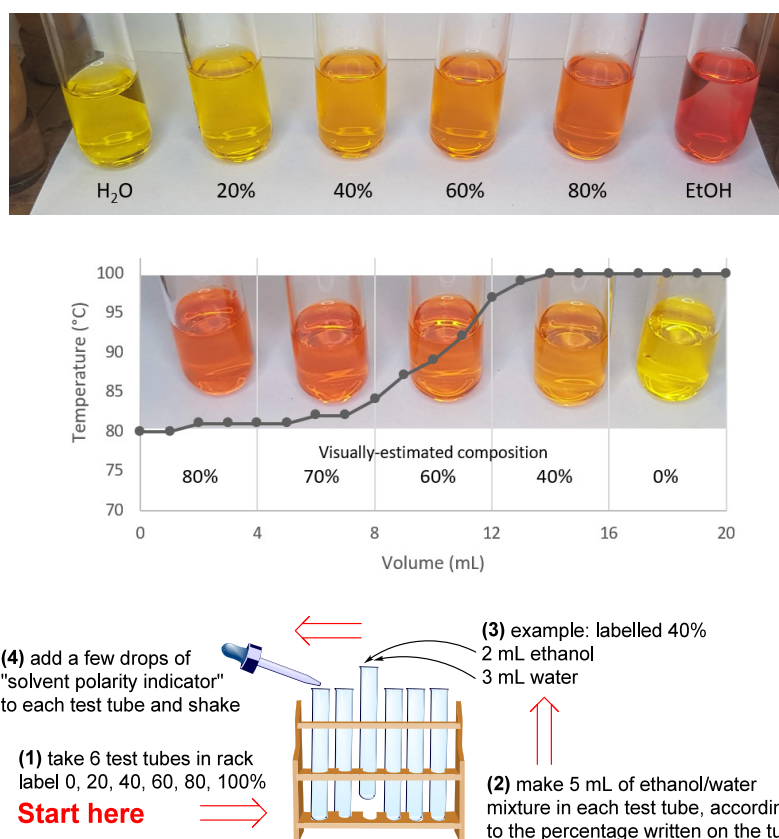
The purpose of using a solvatochromic dye is to add a visual interpretation aspect to distillation, conferring similar educational benefits as the use of indicators in titrations and avoiding the need for more complex analytical techniques to confirm fraction identity.<sup>26,27</sup> The immediate visual feedback also allows the introduction of an “element of inquiry” for fraction analysis. After conducting the first short-path distillation, students are asked to prepare a series of standards of 0 to 100% ethanol in water, adding a few drops of “solvent polarity indicator” to each. These standards can then be used as an immediate visual reference for estimating the composition of collected fractions, with little guidance given (Figure 3, top).

### Assessment and Attainment

10% of the total grade was awarded by a prelab quiz, used as a way to incentivize effective preparation. The prelab quiz ran online and gave instant feedback, with an option to reattempt any failed questions without penalty. As such, prelab grades were always very high and analysis did not yield useful results.

For the first three years of operation, the remaining grade came from a postlab lab report, which was then converted to an in-lab worksheet with the same assessment criteria. Worksheet assessment has been used as part of a normal lab for two subsequent years so far (See SI for full worksheet). A direct comparison of academic attainment with the previous reflux/distillation sequence was not possible, owing to a change in learning objectives and policies limiting data retention. However, grades for both lab report and worksheet assessment were higher than the whole module average, and stayed consistently high over three monitored years (Figure 4).

60% of the grade was awarded for data quality, graphing, reporting of solvent standards, and general lab good practice, with the remaining 30% from three assessed questions that required students to interpret their results and apply a measure of problem-solving. This was a low weighting, but in line with other “elements of inquiry” experiments. The average grade was lower for the problem-solving mark, averaging 69% (sd: 24%) versus 94% (sd: 9%) for the conventional mark. The



**Figure 3.** Top: A student-prepared gradient showing a gradient of 0 to 100% ethanol in water, with solvatochromic dye added. Middle: student fractions collected with a less efficient short-path distillation setup, showing partial purification of ethanol overlaid onto the corresponding temperature–volume curve. Percentages are the v/v ethanol/water composition, estimated visually. Bottom: the “integrated instructions” diagram for preparing the standards.

difference was statistically significant (Wilcoxon paired test,  $p < 0.05$ , see SI for details).

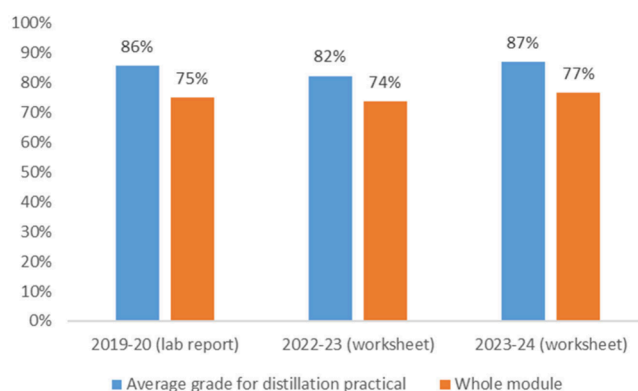
In the problem-solving questions, students were first asked to determine the overall composition of their initial unknown mixture, using their temperature–volume curves and the color change in the composition of their fractions. Relatively little guidance was given, though students were steered toward identifying the volume of pure ethanol distillate recovered.

Second, students were asked to comment on the separation “efficiency”, being expected to identify that a sharper temperature or solvent polarity color transition indicates a more complete separation of ethanol and water.

Finally, students were asked to speculate as to why a less efficient separation might be better (as expected, fractionating column use invariably gave a more efficient separation but took longer). The location of the experiment in the curriculum, before any theoretical teaching on distillation, meant that we were looking for light inductive reasoning, derived from observations only. This question had no model answer, requiring only a demonstration of some form of reasoning based on observation. Most students correctly recognized that separation efficiency might not matter as much as throughput for some applications, though this may be due in part to embedded cultural knowledge of the malt whisky industry.

### Evaluation of Design Aims

The switch from a take-home lab report to an in-lab worksheet meant that students had to tackle problem-solving questions during the lab session itself. Since no decline in relative grade



**Figure 4.** Total grade awarded to this experiment versus the entire lab module, as assessed by a take-home lab report (one year) and in-lab worksheets (two years). The experiment was assessed as a lab report in 2017–18 and 2018–19 but grades are not available due to institutional policy. Teaching in 2020–21 and 2021–22 were dramatically disrupted due to COVID-19 and data is not comparable.

was observed coincident with this switch of format (Figure 4), this may imply that the experiment had ample space to support inquiry-type thinking and deductive reasoning during experimentation, and any loss of working time from having to complete assessment in-lab was balanced by access to peers or tutors. These implications were backed up by informal staff and graduate teaching assistant observations, noting that students frequently discussed their interpretations of solvent color, and relation to fraction composition or vapor temper-

atures. Students would frequently refer back to their own colored solvent fractions as standards, demonstrating the usefulness of a solvent polarity indicator.

When first implemented, and on an ongoing basis, students were monitored for indications of fallback behaviors caused by excessive cognitive load (such as delaying or avoiding answering theoretical questions) and only low levels were seen, in line with other experiments in the same lab course.<sup>3,4</sup> These behaviors were observed frequently in the previous activity this one had been developed to replace. Notably, students now set up and modify their distillation apparatus with only low levels of tutor support, and low instances of breakages or spillages. These were the main issues we sought to address by adopting integrated instructions diagrams.

As part of ongoing quality control and informal action research loops, student, tutor, technician and staff peer feedback was regularly collected and analyzed, along with informal lab supervisor observations, and used to iteratively improve the experiment particularly in the first few years of implementation (examples of iterative improvements will be discussed in limitations and lessons from experience). As of the current iteration, staff and tutors unanimously agreed that the experiment should be kept in its current form or with very minor tweaks. This stands in contrast to the previous version of this experiment, with a positive tutor evaluation of < 60% (Figure 1)

## HAZARDS

Lab coats, safety glasses, and appropriate attire are required as with any lab. Students are also required to pass a mandatory safety quiz at the start of the academic year, and as part of the preparation for each lab. This requires students to read and interpret a simplified combined risk assessment and COSHH form, then answer graded questions.

The distillation itself followed routine local institutional safety procedures, detailed in the [Supporting Information](#).

Solvatochromic dye is used as a very dilute solution in ethanol, kept in a pipet dispensing bottle. Risks associated with the dye are minimized by the small amount required, and students are required to wear gloves when dispensing or disposing of the dye. All solvents or fractions should be disposed of in a non-halogenated solvent waste stream.

Synthesis of Brooker's Merocyanine involves the use of an alkylating agent, and should only be conducted by an expert chemist with full local appropriate safety assessment. An alternative, safer benzylic variant has been recently reported as an undergraduate experiment.<sup>28</sup>

## LIMITATIONS AND LESSONS FROM EXPERIENCE

We found that the high heat flux required to distill water on a reasonable time scale meant that we could not use standard electric hotplates, employing instead older-style heating mantles capable of greater output. Appropriate use of metal foil also helped retain heat and accelerate distillation. Nonetheless, the distillation of water was still so (relatively) slow that we began advising students to end data collection as soon as 2–3 mL of distillate was collected at the higher temperature. Any future adoption in another context would need to screen for appropriately powerful heat sources first.

We also found that acidic soap residues in test-tubes sometimes impaired the function of Brooker's Merocyanine, so we began to add alkali to our formulation after the first year

(see [SI](#) for full stability study information). The integrated instructions diagram received minor updates in the first few years of use, mainly as a result of GTA and lab head feedback on points of particular student difficulty.

Teaching assistants are crucial to the success of any teaching lab, but even those who studied under nonexpository methods themselves may require additional support in delivering an inquiry-based lab.<sup>29</sup> Training on the nature and benefit of inquiry learning was therefore built into the induction process and supporting documents for our teaching assistant program.

Evaluation was limited, since no direct comparison data was available and local ethical guidelines discourage running a parallel conventionally written procedure. Attainment data likely only reflects the relative difficulty of different topics within the lab, but indicates that the experiment meets typical academic quality control standards.

We found that the process of adopting an existing experiment to use "integrated instructions" was straightforward, and supported by literature guidelines.<sup>6,7</sup> However, there were some additional considerations to applying the approach to university-level work which would be of interest to anyone wishing to do the same. Some first drafts of diagrams were quite text-heavy, so a careful balance should be struck between essential facts and volume of text. Adapting longer schemes of work sometimes exceeded a single page in size, which would reintroduce the split-attention effect if not carefully segmented. For example, our solvent standard preparation was a separate, smaller integrated-instructions diagram on a separate page of the lab manual. Anyone looking to implement integrated instructions diagrams should be careful to map out or monitor for places where cross-referencing or lookup still happen, and iteratively improve on designs. In our case, instructions were given on reusable laminated cards but student results were written on a separate worksheet, potentially retaining some unavoidable split-attention effect.

## CONCLUSIONS

"Integrated instructions" provide an elegant solution for lab experiments where students are cognitively overloaded trying to follow a conventional procedure that requires flipping between multiple sources of information. Solvatochromic dyes also provide an immediate visual cue for determining the composition of simple binary mixtures of solvents, such as distillation fractions. Both of these approaches together significantly improved an existing distillation experiment.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.4c00466>.

Student Worksheet ([PDF](#), [DOCX](#))

Lab manual, tutor and technician instructions, details of stability testing ([PDF](#), [DOCX](#))

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## Notes

The authors declare no competing financial interest.

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