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# Hands-On Model of the Principle of Isotope Dilution Analysis for Use in an Interactive Teaching and Learning Classroom Exercise

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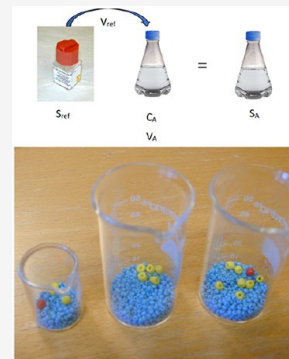
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**ABSTRACT:** A simple hands-on model for illustrating the concept of isotope dilution analysis (IDA) has been devised. The model consists of two sets of beads of different sizes, with one set representing atoms of the analyte and the other set representing solvent water molecules. Phase separation is mimicked by sieving the beads, and the results are detected according to the color of the analyte beads. In this paper, the following three IDA methods are illustrated using the model: (1) direct IDA, (2) substoichiometric IDA, and (3) IDA-assisted neutron activation analysis. The model was demonstrated for a small group of graduate students with previous knowledge of nuclear chemistry, and the response from an inquiry held after the demonstration was good. It is suggested that the model can be used in a dry chemistry laboratory exercise to demonstrate the methodological differences between the different IDA methods, without the need for costly radioisotopes or irradiation facilities. It can also be used as a tool for engaging students in meta-modeling activities.



**KEYWORDS:** Upper-Division Undergraduate, Analytical Chemistry, Hands-On Learning/Manipulatives, Nuclear/Radiochemistry, Isotopes

## INTRODUCTION

The concept of models in science is of fundamental importance. By scientific models, the human brain has explored the world, and to have the brain understand natural science without models would probably be very difficult.

Scientific models serve many purposes: often they are used for the conceptualization of an observed process, which usually increases the abstraction level. However, models can also be used for the concretization or illustration of an abstract concept.

The use of physically recognizable forms in scientific models is likely to help in the understanding of abstract concepts. This has been proven to be the case not only for pedagogical purposes but also for the advancement of scientific research.

Two classic examples in chemistry are Kekulé imagining the C<sub>6</sub> chain biting its own tail, which led him to the structural formula for the benzene ring, and Watson and Crick who built a physical model of the DNA molecule that was found to match all the spectroscopic data.

From this reasoning, one can conclude that scientific models serve double purposes that are mutually dependent on each other, and by this interdependency, these two purposes cross-fertilize each other, providing more refinement of both aspects of the model and ultimately leading to model advancement.

The generalization and predictive power of abstract models invites the user to explore their usefulness. “What happens if I apply the model on *this*?” is perhaps the fundamental question of the scientific process. Science has progressed by building ever more refined models of nature, and it is essential that students,

by investigating the use of models in both their abstract and concrete forms, can grasp the scientific process as an ever-on-going model-building project.

This kind of meta-modeling knowledge—that is, general knowledge about the scope and limitations of models and how models can be used for different purposes—is, however, a subject that is seldom taught in classrooms.<sup>1</sup>

The importance of the use of models in scientific teaching has been emphasized by Harrison and Treagust,<sup>2</sup> wherein they argue that teachers should be aware of both the similarities and the differences between the models that they use in their teaching. Especially, the shared and nonshared attributes of analogies used in models should be discussed in the classroom. Otherwise, there is a risk that students’ interpretations of a model are likely to diverge from the one that the teacher intended.<sup>3</sup>

The skill levels in meta-modeling acquired by students and other users of scientific models have been investigated by Grosslight et al.,<sup>4</sup> and they suggest a three-level classification system, as follows:

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- At **Level 1** understanding, the users perceive models as replicas of reality, although, of course, on a different scale. At this level of understanding, the definition of a model is usually strictly confined to physical objects only and abstract models are not perceived as models at all.
- At **Level 2** understanding, the users realize that several models can coexist, that they are not exact representations, and that they can highlight different aspects of reality by having different abstraction levels. At this level of meta-modeling knowledge, the models are perceived as different forms of communication devices between the user and reality.
- At **Level 3** understanding, the users are aware that models are tools for understanding reality and that these tools can be manipulated to give further insights. Additionally, it is usually realized at this level of understanding that models are not static but are perpetually re-evaluated and refined.

In a recent study by Lazenby et al.,<sup>5</sup> the meta-modeling knowledge of undergraduate students was evaluated by giving the students the task of giving two examples of scientific models. It was found that the examples of models presented were usually of the low abstraction level according to the Harrison and Treagust<sup>2</sup> proposed typology of models used in teaching (Table 1).

**Table 1. Model Typology<sup>a</sup> with Examples Taken from Chemistry**

Abstraction Level	Model Type	Works on/with	Examples from Chemistry
Low	Scale	Size	Ball-and-stick molecules
	Analogical	Similarities	Harmonic oscillator and chemical bonding
	Symbolic	Representation	Element symbols
	Mathematical	Quantification	Equations
	Theoretical	Concepts	Electron shells
	Maps, diagrams, tables	Systemized information	Periodic table
	Concept-process	Generalizations	Chemical equilibrium
High	Computational	Calculation algorithms	Molecular dynamics

<sup>a</sup>Based on Harrison and Treagust<sup>2</sup> and expanded with the adaption made by Lazenby et al.<sup>5</sup>

From this, it seems that the students' perception of a scientific model was mainly as a visualization tool and that more abstract models, such as equations, were not considered models.

Another exercise given to the students in the study<sup>5</sup> was to evaluate six common chemistry models used in teaching: ball-and-stick molecules, Lewis structures, and more abstract conceptual models, such as chemical equilibrium. The model evaluation was according to White et al.,<sup>1</sup> who defined the characteristics of a good scientific model by five evaluation criteria:

- **Accuracy:** It should accurately represent reality within its scope.
- **Coherence:** It should be coherent with other models.
- **Generality:** It should cover all phenomena within its scope.
- **Parsimony:** It should be as simple as possible, without a loss of accuracy.
- **Usefulness:** It should have an application for understanding or predicting.

It was found that the six example models were generally ranked highly by the students for their usefulness for visualization or for predictive power. The models were not ranked highly for anything else. From both exercises, the authors concluded that the students' meta-modeling knowledge was limited and that students should be more directly engaged in meta-modeling activities during lectures, for example, in how to evaluate models and how to modify models for other purposes.<sup>5</sup> These findings confirm results from previous studies.<sup>3,4</sup>

Especially the field of chemistry, which deals with large assemblies of nano- to micro-sized objects not observable by the naked eye and their interactions, seems to benefit from the use of models, and examples are easily found in all the categories listed in Table 1.

Teaching with models puts a special demand on the teacher, who must adapt the use of models in teaching to the appropriate level of the students' ability for conceptual thinking.<sup>2</sup> In this process, the *Focus–Action–Reflection* (FAR) scheme, developed by Treagust et al.<sup>6</sup> can serve as a guide for the teacher.

In the *Focus* phase, the teacher must consider whether the model he/she intends to use is appropriate for the students' level of knowledge. In the *Action* phase, the teacher discusses the model with the students, especially focusing on the shared and nonshared attributes of the analogies of the model. Finally, in the *Reflection* phase, the teacher must do a self-evaluation of the use of the model and conclude whether its use was of any help for the students. This should lead to a modification of the pedagogical approach.<sup>6</sup>

There are many examples in the literature depicting how simple visualization models can be introduced in existing curricula. Such models can serve the purposes of giving the students a better understanding of an abstract process and also engaging them in meta-modeling activities.

An example of a recent model devised specifically for use in chemistry teaching is the work by Mulchandani et al.,<sup>7</sup> which describes a model for demonstrating the concept of the specific surface area and its effect on the adsorption capacity. The model consists of adhesive blocks of similar volumes but different areas that should represent different adsorbents. The students can use these adsorbents to collect pompoms, which represent pollutants. The blocks are then "desorbed", the weights of collected pollutants are recorded, and Langmuir adsorption isotherms can be derived for each type of adsorbent.<sup>7</sup>

The work presented herein has a similar approach. A model is presented for the principle of isotopic dilution analysis (IDA). Its purposes are to provide a tool for the visualization of the principle and for generating input data for the mathematical model. In the specific case of teaching activities, the subject of IDA is part of a lecture about the use of radioactive tracers. The lecture is held as part of a course in general nuclear chemistry for both undergraduate and graduate students.

The method used here for modeling IDA is a physical representation of atoms or molecules together with a means by which to separate them in order to simulate the chemical separation that is always a required step in IDA. The resulting data generated by the model should be readily accessed to give results congruent with the mathematical model of IDA.

Nuclear chemistry and related topics of radioactivity can be difficult to teach without access to radioactive material, detectors, and proper laboratories. However, the use of physical models in teaching activities can make the topic more tangible for the students.<sup>8</sup>

The model, which is presented here in its initial form, is devised as an IDA demonstration tool for use during a lecture but can possibly also be given as a dry chemistry laboratory exercise, in which the students can work in a smaller group with supervision.

In this work, the model is used for the illustration of three common applications of the IDA principle: (1) the direct IDA analysis, (2) the improved substoichiometric IDA analysis, and (3) IDA-assisted neutron activation analysis. These three analytical methods are also the examples taken up by the textbook<sup>9</sup> that was used in a course of general nuclear chemistry.

Several more variants of IDA exist,<sup>10</sup> and the visualization model proposed in this work can possibly also be adapted to provide more examples of IDA-based methods.

## THEORY OF IDA

The fundamental scientific work that would subsequently lead to the widespread use of IDA as an analytical tool in chemistry, biology, and medicine was made by Hevesy and co-workers for studying the solubilities of inorganic salts. At this time, the method that would later become IDA was known as the indicator method, and it used a radioisotope as a tracer for the main element.<sup>11</sup>

When Hevesy worked on a method for the determination of radioisotope elements in the earth's crust, he found that the then-available analytical methods for the lead contents in meteorites and rocks were not sufficiently exact and he then developed the IDA method of analysis. To a solution of rock sample was added a small amount of <sup>210</sup>Pb. The lead was then deposited as a peroxide on a Pt anode. They found that the specific radioactivity on the anode was diluted from the original by the natural lead present in the sample. By correcting for this, the amount of nonradioactive lead could be determined, and a new method of analytical chemistry was invented.<sup>12,13</sup>

Later, Hevesy's interest was directed toward medical applications of the IDA method. In 1944, Hevesy received the Nobel prize in Chemistry for 1943 for his discoveries of radioanalytical methods and essentially founding the field of nuclear medicine.<sup>13</sup>

The method would subsequently find its widest application in the analysis of blood for any substance that (1) can be radiolabeled, (2) will generate antibodies by the immune system to be bound in an antigen–antibody interaction, and thus (3) can be isolated for radioactivity measurement. For this method of radioimmunoassay (RIA), Rosalyn Yalow received the Nobel prize in Medicine for 1977.

Traditionally, short-lived radioisotopes of elements to be analyzed have been used for IDA because they are usually easy to detect. With the advent of mass-spectrometry, long-lived radioisotopes or nonradioactive isotopes can be detected and they have thereby also found their use in IDA.

The proportional relationship of the radioactivity  $A$  (Bq) and the number of radioactive atoms  $N$  is

$$A = \lambda \cdot N = \lambda \cdot m \cdot N_A \quad (1)$$

with the proportional factor of the decay constant  $\lambda$  ( $s^{-1}$ ), which means that radioactivity and mass  $m$  (mol) are readily interchangeable quantities. Here, radioactivity will be used for the deduction of equations, according to the classical IDA methods.

## Direct IDA

In its simplest form, IDA is a method for analyzing the amount  $m_A$  of an unknown analyte "A", where the analyte is usually an element dissolved in an aqueous solution. This is accomplished with the help of the addition of an amount  $m_{\text{ref}}$  of reference of the same element but with a distinct isotopic composition.

The traditional methodology is to add a radioactive isotope mixture to a natural (and nonradioactive) isotope mixture. The radioactive isotope will have a certain specific radioactivity  $S_{\text{ref}}$  (Bq/mol), according to the definition of specific radioactivity

$$S_{\text{ref}} = A/m_{\text{ref}} \quad (2)$$

Usually,  $m_{\text{ref}}$  is the sum of all isotopes present in the reference, rather than only the radioactive isotope. In direct IDA, it is assumed that the  $S_{\text{ref}}$  value is a known quantity.

Here, the mass unit  $m$  is most conveniently expressed in moles. The specific radioactivity of a sample can be determined by measuring its radioactivity and weight, utilizing the atomic or molecular mass of the compound. For commercially available radioisotopes, the specific radioactivity is stated in a certificate.

When mixing a radioisotope of an element "A" of mass  $m_{\text{ref}}$  (mol) with a nonradioactive isotope mixture of the same element with an unknown mass  $m_A$ , the resulting mass-balance equation can be solved for the unknown specific activity  $S_A$  of the mixture

$$S_A = \frac{S_{\text{ref}} \cdot m_{\text{ref}}}{m_A + m_{\text{ref}}} \quad (3)$$

$S_A$  will always be less than  $S_{\text{ref}}$ , hence the method designation of isotope dilution.

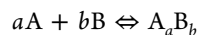
The next step in the procedure is to react the analyte element A with a reactant B to accomplish a phase transition to solid, which makes it possible to isolate and weigh a certain amount of the product AB. Another way to accomplish this is by electroplating.

If the product AB can be isolated, weighed for its mass  $m_{\text{AB}}$ , and measured for its count rate  $R_{\text{AB}}$  (cps), the measured specific radioactivity of the product  $S_{\text{AB}}$  is defined by

$$S_{\text{AB}} = \frac{R_{\text{AB}}}{\Psi \cdot m_{\text{AB}}} \quad (4)$$

Note that the detector efficiency  $\Psi$ , the weighed mass of product AB, and the molecular weight of AB must be known in order to calculate  $S_{\text{AB}}$  in the unit of becquerel per mole.

Since  $a$  mol of A will react with  $b$  mol of B to form the compound  $A_aB_b$  according to



then  $m_{\text{AB}}$  in eq 4 can be replaced with  $m_A/a$ .

By appreciating the fact that all the measured radioactivity in compound AB will come from A only, one will get

$$S_{\text{AB}} = a \cdot S_A \quad (5)$$

The specific radioactivity of the element A,  $S_A$ , is the same as the unknown specific radioactivity in eq 3, since it was prepared in the initial isotope dilution step. Therefore,

$$S_{\text{AB}} = \frac{a \cdot S_{\text{ref}} \cdot m_{\text{ref}}}{m_A + m_{\text{ref}}} \quad (6)$$

By substituting eq 4, one gets

$$\frac{R_{AB}}{\Psi \cdot m_{AB}} = \frac{a \cdot S_{\text{ref}} \cdot m_{\text{ref}}}{m_A + m_{\text{ref}}} \quad (7)$$

In eq 7, all the variables on the right side except  $m_A$  are known, and if  $S_{AB}$  can be determined experimentally according to left-hand side, the value of  $m_A$  can be calculated.

In the method, it is not necessary to separate the entire amount of AB, but the amount must, of course, be possible to weigh on a balance.

### Substoichiometric IDA

One drawback of the direct IDA method is that the stoichiometric factor  $a$  must be known. Additionally, another problem is that, if the reference solution cannot be measured with the same geometry as the separated sample, knowledge of  $\Psi$  is necessary.

To overcome these drawbacks, an improved IDA method was developed on the basis of two samples—the one to be analyzed and one standard sample that will undergo the same process as the unknown sample. The method is usually referred to as the substoichiometric IDA method.

In this case, there are two mass balances to consider. In addition to eq 3 for the unknown sample, there will also be a mass balance for the standard sample

$$S_{\text{std}} = \frac{S_{\text{ref}} \cdot m_{\text{ref, std}}}{m_{\text{std}} + m_{\text{ref, std}}} \quad (8)$$

Normally, it is not necessary to add an inactive element  $m_{\text{std}}$  to the standard sample, since the radioisotope reference solution is usually made with inactive, so-called carrier isotope, from the beginning. Then, eq 8 will simply be reduced to  $S_{\text{std}} = S_{\text{ref}}$  with the consequence that the standard and reference solutions are the same.

A carrier isotope is necessary to make radioisotope solutions behave chemically similarly to the unknown sample. If no carrier isotopes are present, it is likely that the very small amount of radioisotope will be lost to sorption onto container walls.

The next step is to react both the unknown and standard samples with reactant B and to isolate the product AB from both samples. Equation 4 is still valid for the unknown sample. The corresponding equation for the standard sample is

$$S_{AB, \text{std}} = \frac{R_{AB, \text{std}}}{\Psi \cdot m_{AB, \text{std}}} \quad (9)$$

By the same reasoning, the equation corresponding to eq 6 will be

$$S_{AB, \text{std}} = \frac{a \cdot S_{\text{ref}} \cdot m_{\text{ref, std}}}{m_{\text{std}} + m_{\text{ref, std}}} \quad (10)$$

By substituting eq 9, one gets

$$\frac{R_{AB, \text{std}}}{\Psi \cdot m_{AB, \text{std}}} = \frac{a \cdot S_{\text{ref}} \cdot m_{\text{ref, std}}}{m_{\text{std}} + m_{\text{ref, std}}} \quad (11)$$

The advantage is that the additional eq 11 can be used to eliminate  $S_{\text{ref}}$  in the mass balance for the unknown sample in eq 7. Doing this will yield

$$\frac{R_{AB} \cdot m_{AB, \text{std}}}{R_{AB, \text{std}} \cdot m_{AB}} = \frac{m_{\text{ref}} \cdot (m_{\text{std}} + m_{\text{ref, std}})}{m_{\text{ref, std}} \cdot (m_A + m_{\text{ref}})} \quad (12)$$

Compared with the direct IDA equation, eq 7, again, the experimentally determined values are on the left side, and the

known quantities, except  $m_A$ , are on the right side. The equation can be solved for  $m_A$ .

The advantage of eq 12 over eq 7 is that the relative measurement eliminates (1) the stoichiometric factor  $a$ , (2) the detector efficiency  $\Psi$ , and (3) the molecular weight (if using moles) of the separated compound from the evaluation.

Furthermore, if the separated amounts in the two samples can be assumed to be the same, and if the amounts of added radioisotope reference solution to unknown and standard solutions can also be assumed to be identical, one will get the simplified equation

$$\frac{R_{AB}}{R_{AB, \text{ref}}} = \frac{(m_{\text{std}} + m_{\text{ref}})}{(m_A + m_{\text{ref}})} \quad (13)$$

Further simplifications can be introduced if carrier isotope is already present in the reference solution, which means that the reference solution can be used as a standard solution.

Under this condition, the corresponding equations to eqs 12 and 13 will be, respectively,

$$\frac{R_{AB} \cdot m_{AB, \text{std}}}{R_{AB, \text{std}} \cdot m_{AB}} = \frac{m_{\text{ref}}}{(m_A + m_{\text{ref}})} \quad (14)$$

$$\frac{R_{AB}}{R_{AB, \text{ref}}} = \frac{m_{\text{ref}}}{(m_A + m_{\text{ref}})} \quad (15)$$

The substoichiometric IDA method also allows for alternative phase separation methods, for example, solvent extraction, where the separated compound AB can be dissolved in organic solvent and does not have to be isolated in pure form for the weighing and radioactivity measurement.

Some applications of the substoichiometric method in laboratory exercises for students have been suggested.<sup>14,15</sup>

### IDA-Assisted NAA

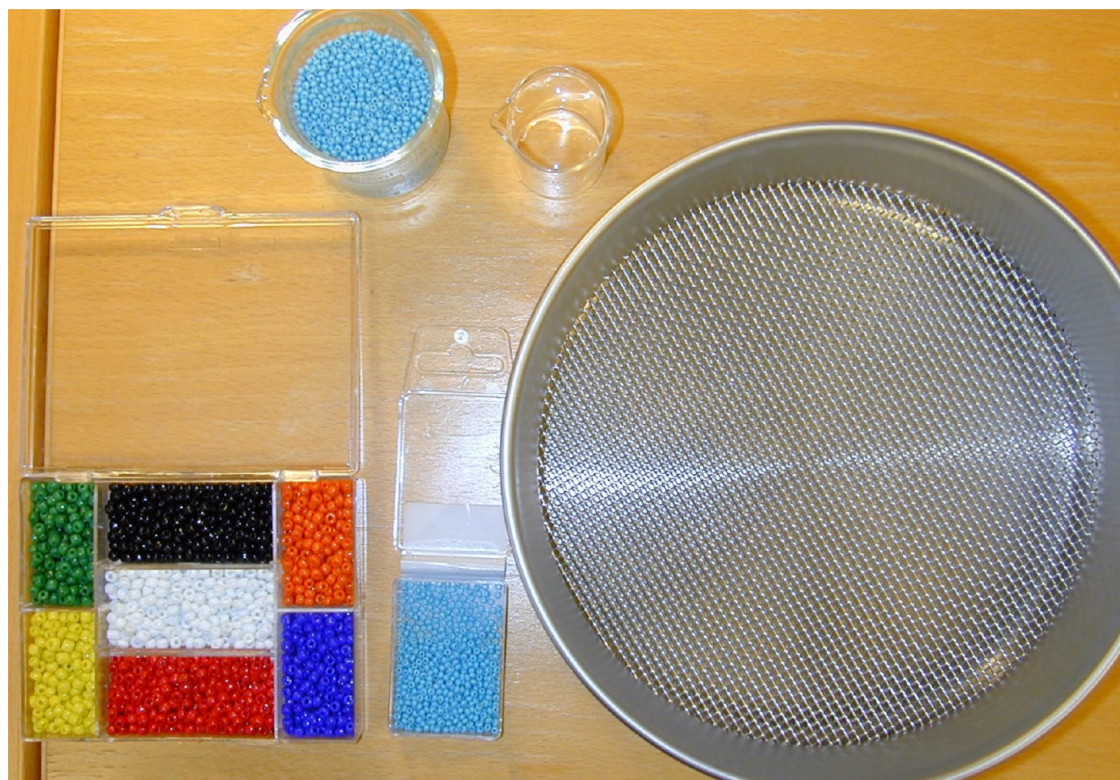
The final example of how IDA can be used as an analytical tool is in neutron activation analysis (NAA). This is also a radio-analytical method, but in this case, the radioactivity is induced in the sample by bombardment with neutrons that will collide and react with a few atoms of the sample, creating extremely small amounts of short-lived daughter radioisotopes, which are not necessarily of the same element as the mother isotope.

NAA is a method for analyzing trace elements in difficult matrixes and is very sensitive, but in addition to the trouble of finding a suitable neutron source (usually a particle accelerator or a nuclear power reactor), the method also relies on a number of experimental variables such as the neutron flux and reaction probabilities (cross sections) that can be difficult to assess.

Therefore, NAA is usually employed with an additional standard sample with a known content of the analyte that will undergo the same irradiation process. Again, by employing relative analyses, experimental variables that can be difficult to assess can be omitted from the evaluation.

On the contrary, this introduces the problem of attaining identical measurement geometries for the unknown and standard samples. Solid samples from irradiation are therefore usually dissolved.

For the dissolution to work properly for such minute amounts of radioactive material, a carrier isotope is usually added to the irradiated samples and, after a certain time period to allow for mixing and isotope exchange, a reactant that will facilitate a phase transfer is added to the samples. This is the IDA-assisted



**Figure 1.** Model “kit” consists of two sets of beads of different sizes, one sieve with collector tray, and several glass beakers.

step in the NAA method. Here, one can note that a dilution of the nonradioactive isotope takes place.

Modifying the mass balances for the IDA-assisted NAA will give the following two equations, with one corresponding to the unknown sample and one corresponding to the standard sample

$$S_A = \frac{A_A^*}{m_A + m_{\text{ref}}} \quad (16)$$

$$S_{\text{std}} = \frac{A_{\text{std}}^*}{m_{\text{std}} + m_{\text{ref, std}}} \quad (17)$$

The induced activity is denoted by the asterisk symbol. The induced activity  $A^*$  will be proportional to the amount of analyte  $m$  in moles present in the samples by the direct NAA relation:

$$A^* = \frac{\phi \cdot \sigma \cdot m \cdot N_A}{\lambda} \cdot (1 - e^{-\lambda \cdot t_{\text{irr}}}) \cdot e^{-t_{\text{cool}}} \quad (18)$$

Here,  $\phi$  (neutrons/s) is the neutron flux,  $\sigma$  ( $\text{m}^2$ ) is the reaction cross-section,  $\lambda$  ( $\text{s}^{-1}$ ) is the decay constant of the induced radioisotope,  $N_A$  (atoms/mol) is Avogadro's number, and  $t_{\text{irr}}$  and  $t_{\text{cool}}$  are the irradiation and cooling times, respectively. Since all the experimental variables except  $m$  are equal for both samples, one can assume that

$$S_A = \frac{k \cdot m_A}{m_A + m_{\text{ref}}} \quad (19)$$

$$S_{\text{std}} = \frac{k \cdot m_{\text{std}}}{m_{\text{std}} + m_{\text{ref, std}}} \quad (20)$$

These are the mass balances for the isotope dilution step, which have now been modified with the initial NAA conditions collected together as  $k$ . The next step is to add reactant B again

and to separate the product AB for the radioactivity measurement. This will give the same equations as shown above for the substoichiometric method—eq 4 and eq 9 for the measured specific activities  $S_{AB}$  and  $S_{AB, \text{std}}$ , respectively.

The equations corresponding to eq 7 and eq 11 will then be

$$\frac{R_{AB}}{\Psi \cdot m_{AB}} = \frac{a \cdot k \cdot m_A}{m_A + m_{\text{ref}}} \quad (21)$$

$$\frac{R_{AB, \text{std}}}{\Psi \cdot m_{AB, \text{std}}} = \frac{a \cdot k \cdot m_{\text{std}}}{m_{\text{std}} + m_{\text{ref, std}}} \quad (22)$$

By employing the substitution of  $k$  in eq 21 with eq 22, this gives

$$\frac{R_{AB} \cdot m_{AB, \text{std}}}{R_{AB, \text{std}} \cdot m_{AB}} = \frac{m_A \cdot (m_{\text{std}} + m_{\text{ref, std}})}{m_{\text{std}} \cdot (m_A + m_{\text{ref}})} \quad (23)$$

This is the full equation for IDA-assisted NAA, which can be solved explicitly for the unknown  $m_A$ . As usual, some simplifications can be assumed to be valid. Normally, one assumes that the amount of separated compound AB is equal in both the unknown and standard samples, giving  $m_{AB} = m_{AB, \text{std}}$  and thereby eliminating the need for any sample weighing.

The final eqs 7, 12, and 23, for the respective analysis, may seem difficult to relate to what actually happens in the methods. In order to visualize this, a model with easily detectable analyte and radioactive molecules was devised.

## METHODS

For the modeling of molecules, two sets of beads of different diameters are used, with one set of smaller light blue beads (<2 mm) simulating water molecules and one set of larger beads (>2 mm) of different colors simulating analytes. The chemical

separation is simulated by a physical separation using a 2 mm sieve. The set also includes small (25 and 100 mL) glass beakers. For transferring the beads from one vessel to another, the use of a funnel is recommended. The model “kit” is presented in Figure 1.

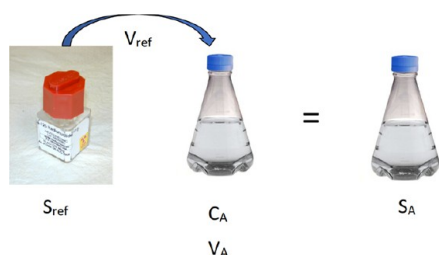
Here, it will be obvious that a nonshared attribute of the model and reality is the separation method. This is performed by size (of the beads) instead of by a chemical process of transferring the analyte to another phase, which is usually carried out by precipitation to a solid phase. This phase transfer is one of the key elements in IDA.

However, for the benefit of being able to visualize the process, this approximation may be acceptable. Additionally, with the model, the detection of the chemical species separated is, of course, performed by eyesight and not by some analytical instrument.

It should be noted that the smaller beads, simulating water molecules, are regarded as volumes, while the larger beads, simulating analyte molecules, are regarded as “dissolved” mass.

### Modeling the Principle of Direct IDA

The initial step in direct IDA is to “spike” the solution of unknown concentration of analyte A,  $C_A$ , and of volume,  $V_A$ , with a small volume,  $V_{ref}$ , of the radioisotope with specific radioactivity,  $S_{ref}$ ; the process is illustrated in Figure 2.



**Figure 2.** First step of direct IDA: “spiking” an unknown solution of analyte A with a radioactive isotope of A.

The addition of the radioactive reference solution to the unknown solution results in isotope dilution, which in turn means that the specific radioactivity of the unknown solution  $S_A$  will always be lower than that of  $S_{ref}$ . The volume increase due to the addition is of no importance.

The second step is to induce a chemical reaction that transfers an exact amount of the element to be analyzed to another phase. Usually, in the reaction, a solid phase is precipitated, which can easily be separated from the remaining solution.

The expression for  $C_A$  is deduced from eq 7 by solving this for  $m_A$  and replacing mass with concentration times volume to get

$$C_A = \left( \frac{a \cdot S_{ref} \cdot m_{AB}}{R_{AB}} - 1 \right) \cdot \frac{C_{ref} \cdot V_{ref}}{V_A} \quad (24)$$

To calculate  $C_A$ , everything, except  $m_{AB}$  and  $R_{AB}$ , is known from the start. The final step of direct IDA is therefore to (1) weigh the separated compound and calculate the mass in moles and (2) to measure the radioactivity of the separated compound.

The use of the model for the visualization of the direct IDA process will now be described.

Two “solutions” with, for example, volumes of  $V_A = 80$  mL and  $V_{ref} = 10$  mL are prepared with the smaller beads. For the element to be analyzed, some beads of the larger type of a different color are selected. In this case, 4 “moles” of yellow

beads are selected as the unknown A, so  $C_A$  in this case is 50 M. Note that, if the high value of the concentration is found to be disturbing to a chemist, one can denote that each bead represents a millimole or micromole instead. For obvious pedagogical reasons, this number should not be revealed in advance of the exercise.

To show the effect of the stoichiometric factor  $a$ , a stoichiometric amount of four B colored white ( $a = 1$ ) is also added.

For the 10 mL reference solution, we utilize one yellow bead and one red bead, where the former represents nonradioactive carrier isotopes and the red bead represents the radioactive isotope. One can then calculate  $S_{ref} = 0.5$  Bq/mol and  $C_{ref} = 200$  M. Again, we add white beads of B in the appropriate stoichiometric amount, which is 2 beads.

The initial solutions are shown in Figure 3. In the exercise, there should be thorough mixing of the beads; otherwise, the separation step will not be necessary to deduce the results.

The next step is to add the reference solution to the unknown solution, and then, separation is accomplished by sieving the resulting mixed solution. The “analytical” result is shown in Figure 4.

From this, one can calculate that, in the separated compound AB, there is one radioactive red bead A per 6 AB (white-yellow or white-red pair of beads) in total, and consequently,  $S_{AB} = 0.167$  Bq/mol. When “detecting” the radioactivity, it is assumed that the efficiency of detection is 100%.

Calculating  $C_A$  by eq 24 gives

$$C_A = \left( \frac{1 \cdot 0.5 \text{ Bq/mol} \cdot 6 \text{ mol}}{1 \text{ Bq}} - 1 \right) \cdot \frac{200 \text{ M} \cdot 10 \text{ mL}}{80 \text{ mL}} = 50 \text{ M}$$

If the stoichiometric factor  $a = 2$ , one should decrease the number of white beads to half in the preparation step in order to get 3 mol of  $A_2B$ s in total after separation, giving  $S_{AB} = 0.333$  Bq/mol.

### Modeling the Principle of Substoichiometric IDA

In the Theory of IDA section, it was pointed out that the direct IDA method has some drawbacks. To overcome these drawbacks of direct IDA, there is an improved method in which one also performs a separation step with a standard solution, which is called substoichiometric IDA.

The procedure is the same as that for direct IDA, apart from the fact that there now are two solutions to analyze—the unknown solution and the standard solution.

Here, we will take advantage of having a radioactive reference solution with an added carrier, which is also normally the case, and we can use the reference solution also as a standard solution (see the Theory of IDA section).

From this simplification, eq 14 can be used instead of eq 12. The evaluation of  $C_A$  for substoichiometric IDA can be deduced from eq 14 by solving this for  $m_A$  and replacing the mass with concentration times volume to get

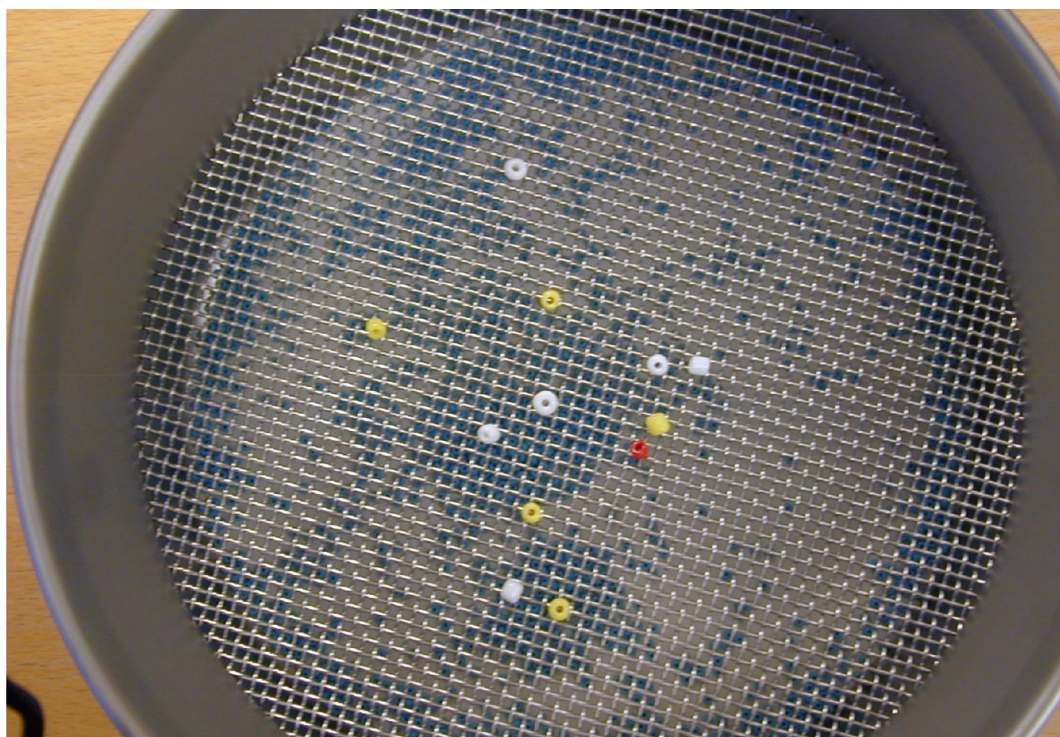
$$C_A = \left( \frac{R_{AB, \text{std}} \cdot m_{AB}}{R_{AB} \cdot m_{AB, \text{std}}} - 1 \right) \cdot \frac{C_{ref} \cdot V_{ref}}{V_A} \quad (25)$$

Here,  $R_{AB, \text{std}}$  and  $R_{AB}$  are the count rates (cps) in the separated standard sample and unknown sample, respectively.  $m_{AB, \text{std}}$  and  $m_{AB}$  are the corresponding separated amounts (mol).

The advantage of eq 25 over eq 24 is the elimination of the stoichiometric factor  $a$  and the detector efficiency needed to



**Figure 3.** Initial “solutions” are prepared for direct IDA. Left, unknown solution; right, reference solution. Color key: yellow = analyte “A”, red = radioactive “A”, white = reagent “B”.



**Figure 4.** After mixing the “solutions” with each other, the “water phase” beads are separated, which leaves the analyte beads in the sieve for “detection”.

determine  $S_{AB}$ . The use of the model for the visualization of the substoichiometric IDA method will be described next.

This time, one reference solution and one standard solution is needed—the former is added to the unknown solution and the latter is used directly in the separation step. Both solutions will be identical. In the more general case (see the [Theory of IDA](#)

section), four solutions are used—two references, with each to be added to one unknown solution and one standard sample solution, respectively.

However, in this case, we assume that the standard and reference samples are identical, i.e.,  $S_{ref} = S_{std}$  and then, only three solutions are needed for the analysis.



**Figure 5.** Initial “solutions” for substoichiometric IDA. Top, unknown solution; bottom, the two identical reference solutions. Color key: yellow = analyte “A”, red = radioactive “A”, white = reagent “B”.

The initial unknown solution is, for example, composed of 6 yellow beads in 70 mL volume, giving a  $C_A = 85.7$  M. The two reference solutions have the same compositions used in the example with direct IDA. Again, white beads are added according to the stoichiometric factor  $a = 1$ .

The solutions are shown in Figure 5.

The next step is to add one reference solution to the unknown solution, and then, a separation is accomplished by sieving the mixed solution. Then, the standard solution is also separated by sieving. The respective results are shown in Figure 6a,b.

For the unknown solution, the sieve separates 16 beads altogether, among which one is radioactive. Since the stoichiometric factor  $a = 1$ ,  $m_{AB} = 8$ . In the standard solution, 4 beads are separated, of which one is radioactive, and  $m_{AB,ref} = 2$ . The stoichiometric factor is now irrelevant; if  $a = 2$ , for example,  $m_{AB} = 4$  and  $m_{AB,ref} = 1$  but the ratio  $m_{AB}/m_{AB,ref}$  is constant.

Inserting these numbers into eq 25 gives

$$C_A = \left( \frac{\left(\frac{1}{2}\right) \text{ cps/mol}}{\left(\frac{1}{8}\right) \text{ cps/mol}} - 1 \right) \cdot \frac{200 \text{ M} \cdot 10 \text{ mL}}{70 \text{ mL}} = 85.7 \text{ M}$$

### Modeling the Principle of IDA-Assisted Neutron Activation Analyses (NAA)

In NAA, the analyte is detected by inducing radioactivity in the sample. It was pointed out in the Theory of IDA section that the analysis is greatly facilitated by having a standard sample with a known concentration to measure the activation yield.

A digestion of the samples is usually required to achieve the same measurement geometry. A carrier isotope is added, and then, the analyte is transferred to another phase by the IDA method.

The radioactivity induced by NAA is very small in relation to that of the natural isotopes in the samples. Additionally, the IDA step dilutes the samples with natural isotopes and not with radioisotopes.

The equation for evaluating the unknown concentration  $C_A$  with IDA-assisted NAA is deduced from eq 23, first by replacing the mass with concentration times volume to get

$$C_A = \frac{R_{AB} \cdot m_{AB, \text{std}} \left( \frac{C_{\text{ref}} \cdot V_{\text{ref}} + C_A \cdot V_A}{C_{\text{std}} \cdot V_{\text{std}} + C_{\text{ref, std}} \cdot V_{\text{ref, std}}} \right) \cdot C_{\text{std}} \cdot V_{\text{std}}}{R_{AB, \text{std}} \cdot m_{AB}} \quad (26)$$

However, in this form, eq 26 does not give  $C_A$  explicitly. The explicit solution for  $C_A$  is

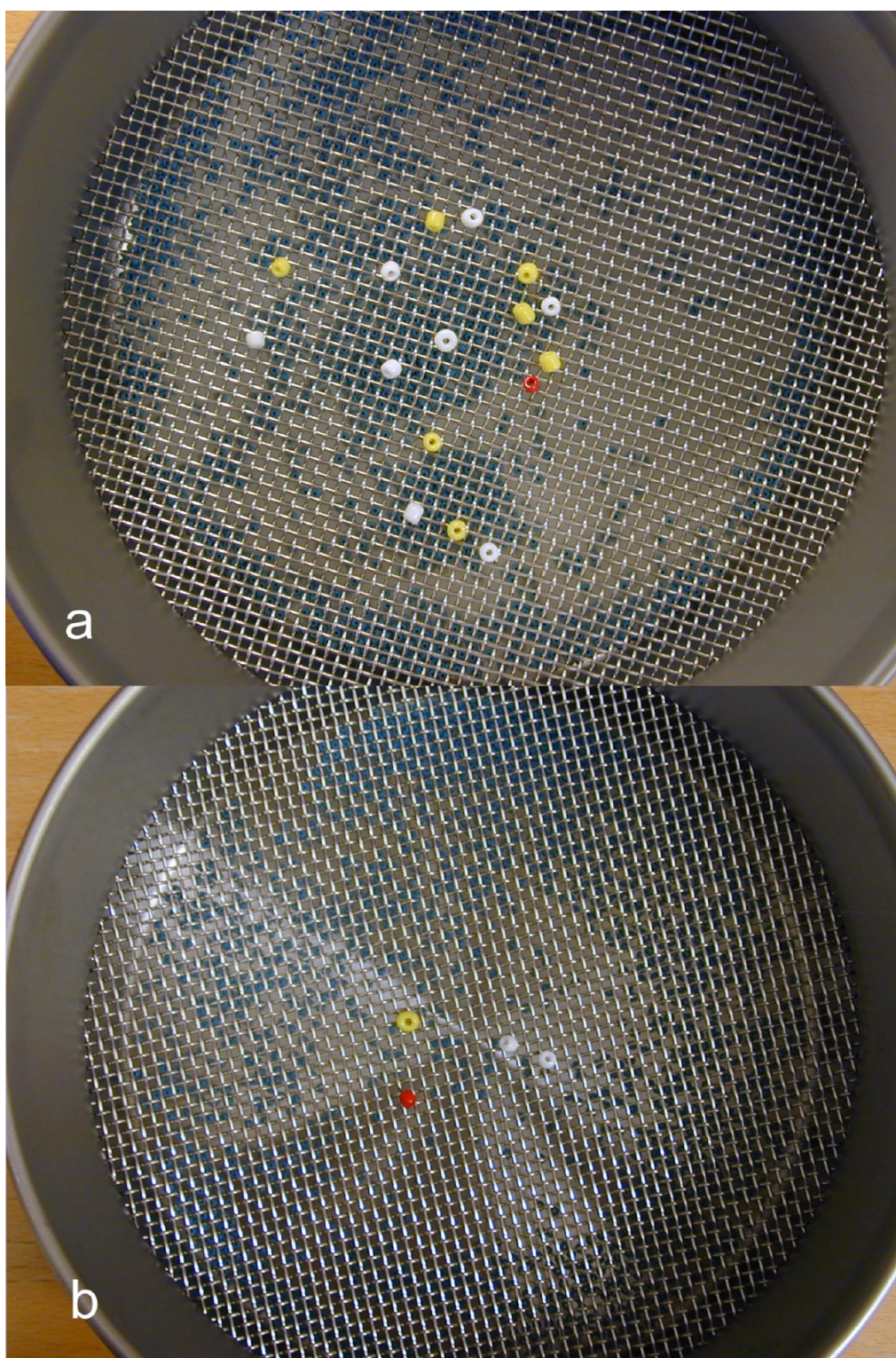
$$C_A = \frac{C_{\text{ref}} \cdot V_{\text{ref}} \cdot R_{AB} \cdot m_{AB, \text{std}}}{V_A \cdot R_{AB, \text{std}} \cdot m_{AB} \cdot \left( 1 - \frac{R_{AB} \cdot m_{AB, \text{std}}}{R_{AB, \text{std}} \cdot m_{AB}} + \frac{C_{\text{ref, std}} \cdot V_{\text{ref, std}}}{C_{\text{std}} \cdot V_{\text{std}}} \right)} \quad (27)$$

For this example, it is suggested that more advanced students in nuclear chemistry try to figure out for themselves how IDA-assisted NAA can be modeled with the use of the same model demonstrated for them for the direct and the substoichiometric IDA methods.

The use of the model for the visualization of the IDA-assisted NAA process will now be described.

Initially, two solutions are prepared—the unknown solution and the standard solution. For example, we selected  $C_A = 150$  M solution with  $V_A = 20$  mL and  $C_{\text{std}} = 300$  M with  $V_{\text{std}} = 20$  mL. In addition, two portions of the nonradioactive carrier isotope solutions are needed, for which we selected  $C_{\text{ref}} = C_{\text{ref, std}} = 400$  M and  $V_{\text{ref}} = V_{\text{ref, std}} = 10$  mL.

For the model to work, the following points need to be figured out by the students: (1) The radioactive red beads should be



**Figure 6.** Substoichiometric IDA: (a) Results after separation and detection of the unknown solution and (b) results after separation and detection of the standard solution.

proportional to the yellow analyte beads, since the radioactivity is induced and not added to the samples. (2) The white beads are added as usual to simulate a separated compound AB with  $a = 1$ , but here, the students must realize that the induced radioactivity of the red beads is is not compensated for by the white beads, because the induced radioactivity is insignificant compared to

that of the analyte. The prepared solutions should look as shown in Figure 7.

The two carrier solutions are then added to the unknown and standard samples, respectively, and the separation by sieving is performed. The results are shown in Figure 8a,b.

Now, the data can be collected. First, we note the number of red beads so that  $R_{AB} = 1$  cps and  $R_{AB, std} = 2$  cps. The amounts of



**Figure 7.** Preparation of the model for IDA-assisted NAA. Clockwise from top left: the unknown sample, the standard sample, and the two identical carrier (reference) solutions. Color key: yellow = analyte “A”, red = radioactive “A”, white = reagent “B”.

analytes separated are  $m_{AB} = 7$  mol and  $m_{AB, \text{std}} = 10$  mol, respectively. The radioactive red beads should not be included in the masses. Using eq 27, we obtain

$$C_A = \frac{400 \text{ M} \cdot 10 \text{ mL} \cdot 1 \text{ cps} \cdot 10 \text{ mol}}{20 \text{ mL} \cdot 2 \text{ cps} \cdot 7 \text{ mol} \cdot \left(1 - \frac{1 \text{ cps} \cdot 10 \text{ mol}}{2 \text{ cps} \cdot 7 \text{ mol}} + \frac{400 \text{ M} \cdot 10 \text{ mL}}{300 \text{ M} \cdot 20 \text{ mL}}\right)} = 150 \text{ M}$$

## RESULTS OF A STUDENT SURVEY

The model was demonstrated for a small group of seven students of mixed undergraduate and graduate (MSc chemical engineering) levels.

First, the students were briefly refreshed about the IDA concept. All the students had already taken a course in general nuclear chemistry, in which IDA is one of the topics. The course includes practical laboratory exercises with radioactivity, however, for the moment not IDA.

All three demonstration examples, direct IDA, substoichiometric IDA, and IDA-assisted NAA, were presented on the blackboard in the conventional teaching style.

The model was then presented, along with the shared and nonshared attributes of the model and reality. Especially, it was pointed out that the separation was performed according to the sizes of the beads and not by a chemical reaction. Additionally, the limited visual resolution (“pixels”) of the model was pointed out, as it is difficult to have enough analyte beads (especially due to the smaller number of “radioactive” beads) to account for different separation amounts, for which beads would be split into fractions to get the correct results.

The first two of these cases were demonstrated using the model, and after this, the IDA-assisted NAA example was given. For the latter, the students were “activated” with giving them the task of figuring out how to set up the model to demonstrate the

method with correct outcome. Here, the concentration of the unknown solution was given in advance, and the student task was to figure out how the model should be set up to give the correct answer. In total, the demonstration and student task took approximately 45 min.

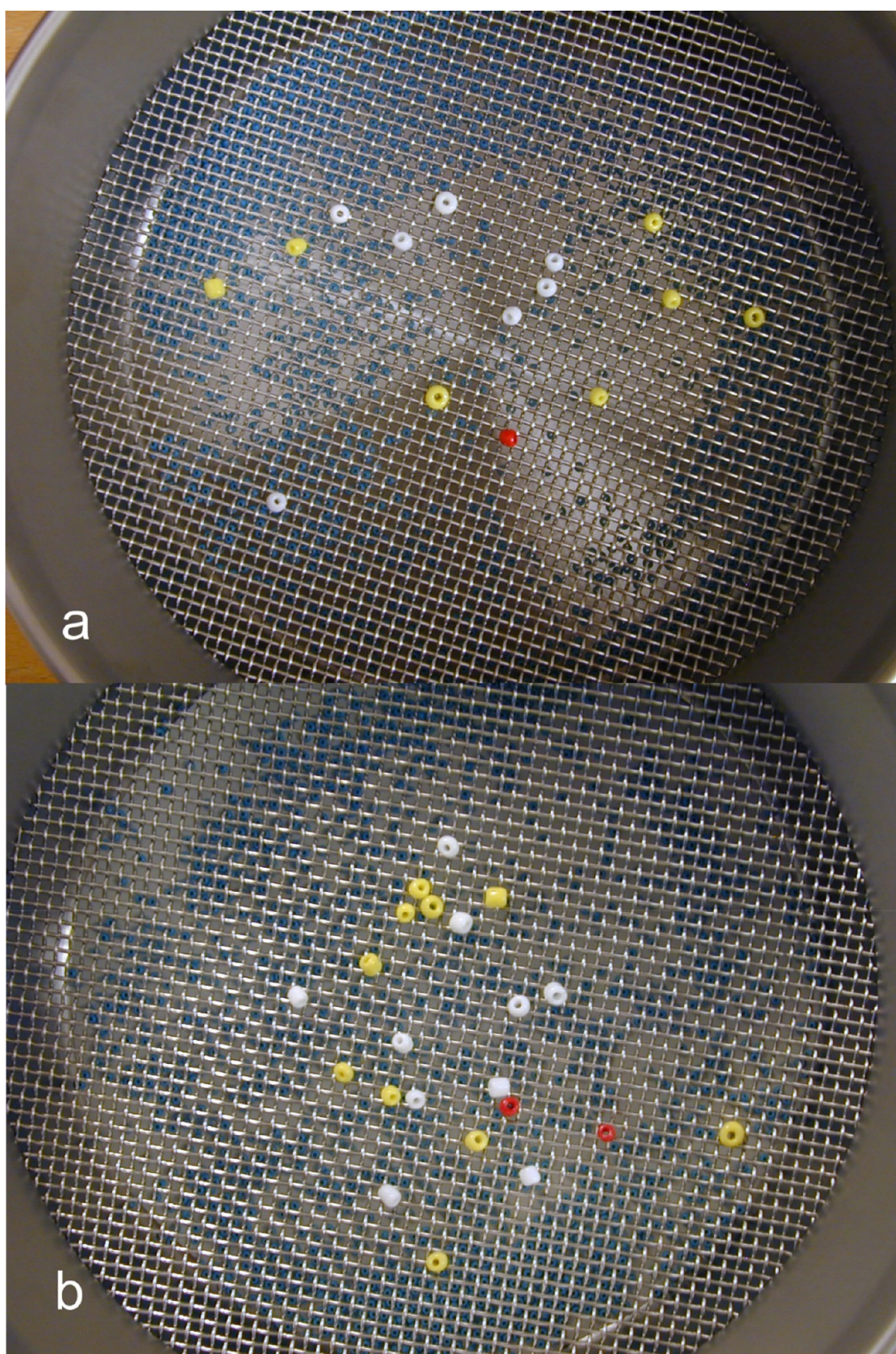
After these exercises, a written enquiry was handed out, in which the students were supposed to rate the model as a pedagogical tool. The given scale included 1 (= bad), 2 (= mediocre), 3 (= average), 4 (= good), and 5 (= excellent). The parameters intended for the students to judge were the same as those suggested for the quality assessment of a scientific model: accuracy, coherence, generality, parsimony, and usefulness.<sup>1</sup>

If the student had no opinion on a question, it was suggested that they leave the answer field blank. The responses are shown in Figure 9.

## DISCUSSION AND REFLECTION

According to the survey performed immediately after demonstrating the IDA model for a small group of seven students, the model received the evaluation shown in Figure 9. The model accuracy was rated mostly “good” (average score: 3.9), coherence was slightly above “average” (average score: 3.4), generality was rated “good” (average score: 4.4), and parsimony was rated “good” (average score: 4.1). The rating of the usefulness was perhaps the most interesting, since it seems to have divided the students in two categories—it was rated either as “average” or as “excellent” (average score: 4.3).

The statistical foundation for the enquiry is obviously too small to give any definite answer to how students in nuclear chemistry would rate the model. However, from this evaluation, the model seems to give an initial positive impression on the students and shows that further use of the model in similar demonstrations can potentially be useful for student learning.

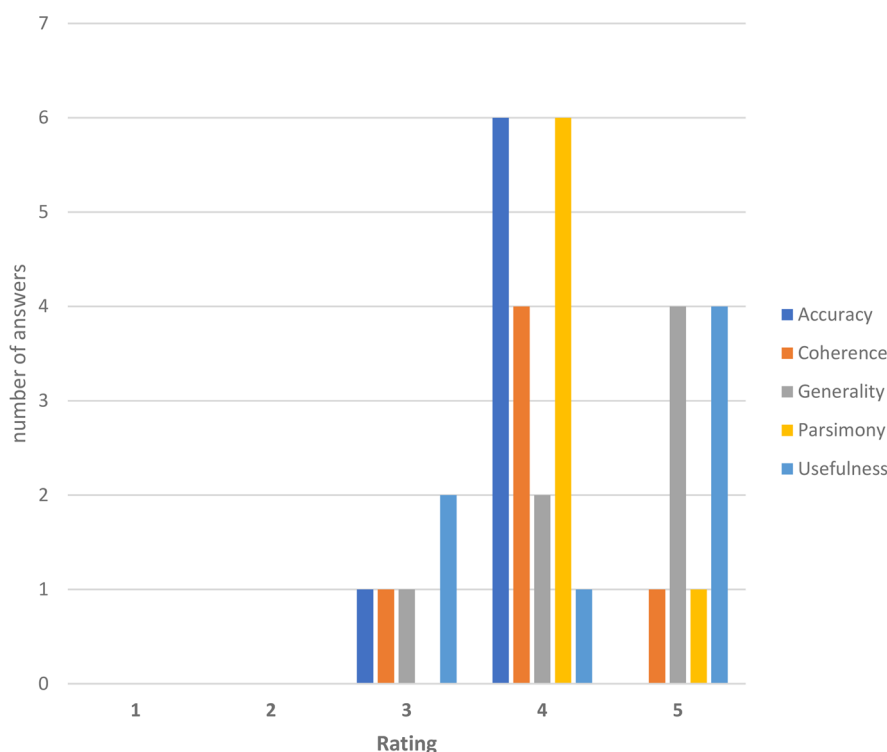


**Figure 8.** IDA-assisted NAA: (a) Results of the separation and detection of the unknown sample and (b) results of the separation and detection of the standard sample.

An alternative to the current approach with a teacher-led demonstration of the model is to give one model set each to smaller groups of students, where they have to analyze unknown solutions with the different methods and hand in the analysis results. However, it is essential that the teacher have first given an introduction lecture to the IDA methods, otherwise the

usefulness of the model for student learning will probably be very limited.

In the reflective portion, a question from a student that came up was “If we choose to separate a smaller amount than what is there, how can the system “know” how to separate a representative sample?”



**Figure 9.** Responses from seven students rating the IDA model: 1 = bad, 2 = mediocre, 3 = average, 4 = good, and 5 = excellent.

This is good example of the important question, as emphasized by Harrison and Treagust<sup>2</sup> of how the model compares to reality. The shared and nonshared attributes of analogies usually reveal the fundamental weaknesses of models since models always simplify.

Since the model uses a comparatively low number of discrete particles, the automatic statistical selection performed in a real system is, in this case, difficult to illustrate since it will require an impracticable large number of analytical beads.

In the examples shown above, 100% separation efficiency has always been ensured by selecting the number of B molecules (white beads) as being equal to the number of A molecules (red plus yellow beads, except in NAA: only yellow). This would, of course, not be possible in real world, since the number of A is unknown, at least in the sample for analysis.

If, instead, a smaller amount than that available is separated, the measured radioactivity will be a fraction of the total available—the amount separated divided by the total amount times the total radioactivity. Due to the large number of atoms in real systems, the separated amount will always be large enough for a statistical selection that will automatically give the correct answer. The model, on the contrary, requires the user to compensate for this deficit in the model since each singular bead is essentially a representation of many of atoms.

If the calculation example given above for substoichiometric IDA is modified to separate equal amounts, for example, 2 AB molecules, the initial amount of B should also be 2 in the unknown solution. Then, the corresponding measured radioactivity is (to be deduced by the user) to be  $(1 \cdot 2/8) = 0.25$  cps (a quarter of a bead!) since the separation efficiency will be  $2/8$  in the unknown sample (but  $2/2$  in the standard sample). The analysis results will be the same.

In reality, the radioactive yield will follow the separation efficiency automatically, but when separation efficiency is <100%, the model requires the user to work out the statistics

of the radioactive yield themselves. This is a nonshared attribute between the model and reality that should be discussed when the model is demonstrated and may also be an excellent opportunity for students to engage in a meta-modeling activity.

Also, the model does not incorporate radioactive decay. In real analyses, this is usually only an issue in NAA, where the induced radioactivity can be very short-lived (typical half-life of minutes). The use of a standard sample will automatically correct the analysis results for the radioactive decay; the problem can instead be to find anything left to measure.

## CONCLUSIONS

A simple hands-on model for the principle of IDA analysis has been devised. The model is intended both to be a visualization tool and to generate input data for the mathematical equations of IDA analysis. The model is specifically adept at demonstrating the differences in approach between different IDA methods, which can be advantageous for student learning.

The model has been used as a demonstration tool for a small group of graduate students (MSc in chemical engineering), and the initial feedback was generally positive.

The largest potential of the model is perhaps for performing a dry experimental exercise to illustrate the different concepts of IDA analysis. This can show students what happens in each method without necessarily performing the actual wet chemistry experiments. For teaching situations in which actual experiments are not possible or are too expensive, conducting this demonstration might be an option.

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## Notes

The author declares no competing financial interest.

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