Student Preparation of Standard Solutions

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It is frequently the case that those responsible for undergraduate teaching in classical and instrumental analysis laboratories make the lives of their students easier by having most, or even all, of the stock standard solutions prepared in advance of a class. Reasons for this include:

- The time-saving factor which takes account of the 3- to 4-h (a) period for completion of a typical experiment.
- (b) The knowledge that the students are all dealing with the same stock standard solutions and that any errors in analysis must be due solely to poor technique.
- (c) The presumption that all the students are aware of how to prepare molar, parts per million, and parts per billion solutions.

While the first two factors certainly deserve serious consideration, the presumption that second- or third-year undergraduate students must know or remember how to prepare their own standard solutions should be evaluated on an individual or at least a class basis. Appraisal of recently published surveys in this Journal tends to substantiate this view (1, 2). A check on presumed knowledge is particularly important for those students taking analytical chemistry courses since the preparation of accurate standard solutions is of such critical importance. For most universities and colleges running instrumental analysis courses, emphasis is placed on the theory and, to a lesser extent, the use of the instruments themselves. Preparations that precede the determination, and which the majority of students dismiss as irrelevant, are most often ignored by course designers also. An analogy may be drawn, however, between the use of log tables or slide rules and simple to use multifunction pocket calculators. How many of today's generation of analytical chemistry students are capable of effectively using the older methods of computation has been addressed previously in this Journal (3). Indeed, this condition has been recognized in a more general sense as evidenced by several recent papers also in this Journal (4-7). For teachers of analytical chemistry, knowledge of these concerns should also lead us to question how many students are competent at calculation and preparation in simple analysis.

In the author's experience, students are more likely to calculate correctly the weight of a solid compound than a liquid substance for preparation of a molar solution. Errors usually relate to the absence of factors for specific gravity or percent assay of the reagent from the calculation. With regard to the preparation of part-per-million solutions, the concept appears to be confusing to a lot of students and the more expressive synonyms micrograms per milliliter and milligrams per liter are not generally recognized as such. A similar observation could be made of part-per-billion concentrations and synonyms nanograms per milliliter and micrograms per liter.

Setting problems relating to work of such a practical nature outside the laboratory is self-defeating if an objective assessment of class aptitude for preparative work is sought. Two simple exercises that have been well tested here at Pittsburgh may be used in other establishments to determine individual or class understanding of these important principles:

(a) In the text of an appropriate experiment, include the requirement that the student should prepare his/her own solution of 0.1M HCl and, if thought necessary at this level, to perform a titration in order to standardize the acid. Reporting of all calculations pertaining to this experiment should be mandatory. Failure to include factors for percent assay and specific gravity may be pointed out and hopefully lead the student to adopt a more careful and studied approach to the preparation of similar solutions in the future.

(b) Design and/or implement an experiment that includes preparation of a standard solution from a salt containing two or more atoms or molecules of the species of interest. Prior to the start of one of our instrumental analysis laboratory experiments here at Pittsburgh, students are told to prepare a milligrams-perliter stock standard solution of quinine from quinine sulfate, $[(C_{20}H_{24}N_2O_2)_2 \cdot H_2SO_4 \cdot 2H_2O]$. This solution is used in the quantitative determination of quinine in tonic water. The typical class size is 40, and usually the majority of students fail to take into account the presence of two molecules of quinine in the dihydrate salt and most base their calculations on the formula weight of quinine sulfate rather than the amount of quinine contained in the salt. A similar procedure and end result is usually noted for an atomic absorption analysis using ammonium molybdate(VI) tetrahydrate [(NH₄)₆Mo₇O₂₄ · 4H₂O] for preparation of a stock standard solution of molybdenum.

An approach that the author has also found useful at the start of a term is to provide a class of students with a choice of several starting materials from which they have to choose the one they consider to be the most suitable for a given exercise. For instance, in a recently implemented experiment, successive classes of students were asked to prepare a 1000-mg/L Na⁺ solution and were given the choice of an unopened bottle of sodium chloride, a bottle of sodium sulfate open to the atmosphere, a solution containing approximately 5000 mg/L Na⁺, and a lump of sodium metal. That some students chose hygroscopic sodium sulfate or an approximately known concentration of sodium in unknown media with which to prepare working standard solutions is perhaps understandable, but attempts by others to secure the lump of sodium for aqueous standard solution preparation is more disturbing. Similar strategies may be employed prior to the start of most analytical chemistry laboratory courses and-at the very least-may serve as efficient exercises in making students more aware of the differences between primary and secondary standards. If the students show that they are capable of preparing their own stock solutions, then they should be rewarded in subsequent classes with the provision of already made stock standard solutions. Implementation of such a program would undoubtedly result in some grumbling and dissent among those students used to experiments from less demanding designers, but hopefully the educative value of such a strategy would be of subsequent benefit.

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