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Commentary

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My Dear Buret, Your Time Has Indeed Come!

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ABSTRACT: The authors present a case for mass-based metering of titrant volume during titrations. The mass based approach offers improved precision and is compatible with electronic data acquisition at lower cost when compared to use of burets.

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We estimate that each year, thousands of burets are manufactured and sold at a cost of $150−250 (USD) each. The education sector is believed to account for a significant fraction of buret sales, with enrollment growth and breakage and replacement driving purchasing decisions. Funds can be saved if alternative approaches to metering titrant are employed without sacrificing performance.

PROBLEMS WITH BURETS IN THE TEACHING LABORATORY

Typical 50 mL Class A burets have a tolerance of ±0.05 mL or 0.1% relative uncertainty at best. However, when an undergraduate student randomly chose five burets from our university's quantitative analysis laboratory and performed a gravimetric calibration of each, none of the burets were found to perform within tolerance. For delivery of ca. 25.00 mL of water, a relative error/uncertainty of 0.37−1.04% was found for N = 5 trials (data set mean was 0.78%). In addition to being expensive, glass burets are fragile and sharp glass edges pose a safety hazard in the laboratory.

ONE POTENTIAL SOLUTION

A recent article by McMills et al.[1] extends upon the previous arguments of S. J. Hawkes[2] and R. W. Ramette[3] in favor of using gravimetric measurement of titrant delivered during titrations. In this approach, a plastic wash bottle or dropper is used to add titrant to the reaction flask. Gravimetry is used to determine the quantity of titrant added. The titrant concentration is expressed in m/m terms. Ramette's work has introduced the concentration unit "*molamity*" as moles of solute per kilogram of solution. This allows simple conversion to moles of titrant through multiplying by the dispensed solution's mass.

The major technical advantage of using the mass standard is believed to be lower relative uncertainty and better precision. Very inexpensive ($5−15), compact, pocket balances are now available with two decimal digits of precision. The tolerance of these balances is ±0.01 g and this corresponds to a 5-fold improvement in precision over burets for a solution with density of 1.0 g/mL. Also of note is this improved precision is provided by a device that costs only a small fraction of a glass buret—under $15, versus $150!

To assess whether this improvement in precision would be realized in a laboratory setting, we have titrated a 5.0 mL vinegar sample using both the mass-based and volume-based approaches. We found that the percent relative standard deviation of the vinegar concentration determined for N = 6 replicate analyses was 0.60% and 1.10% for the mass-based and volume-based titrations, respectively. This finding is in agreement with the work of McMills et al.,[1] who also found the mass-based titration yielded improved precision for titration reactions. We also note this experiment used a 5.0 mL volumetric pipet for dispensing the vinegar; further increases in performance may be obtained by using a mass standard for dispensing the sample.

To investigate further, we have performed entirely gravimetric titrations. In these experiments, samples of primary standard potassium hydrogen phthalate (KHP) were weighed and added to titration flasks. After solvent and indicator were added to the solid KHP sample, a wash bottle containing aqueous sodium hydroxide titrant was weighed and initial mass was recorded. The KHP samples were then titrated to the end point, and the final mass of the wash bottle was recorded. The difference in mass between the two weighings represents the mass of titrant dispensed. The number of moles of KHP (and therefore NaOH) can be determined from the mass of solid used for each experiment. Consequently, this experiment performs a mass-based standardization of the aqueous sodium hydroxide, and offers a simple route to experimentally measure the *molamity* of the solution. Five replicate titrations allowed standardization of the sodium hydroxide solution with a precision of 0.15% relative standard deviation (RSD).

The standard solution of sodium hydroxide was then used to titrate N = 8 samples of vinegar. For these experiments, roughly 5 g of vinegar was dispensed into the titration flask for analysis. The vinegar was diluted with a small amount of water (∼20 mL) and indicator was added. The wash bottle containing titrant was weighed before the titration and again after reaching the end point. The measured *molamity* and measured mass dispensed were used to determine the moles of titrant consumed. We found that the samples could be titrated such

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that the indicated acetic acid concentration in the vinegar differed by only 0.4% RSD. This data pool contains two trials in which the sample was visibly overtitrated, and the RSD was <0.3% if excluding these datums. Indeed, use of gravimetry for metering both titrant and sample produced improved precision for the analysis.

In addition to substantially reducing cost, the mass-based electronic balance approach is also compatible with electronic acquisition of data and data automation that has become quite common. Indeed, several attempts to electronically monitor titrant volume have appeared in this *Journal*.[4,5] Inexpensive pocket balances convert observed mass into the electronic domain and, therefore, could be adapted for digital signal acquisition. Direct readout from the balance eliminates parallax error and simplifies data collection. An additional benefit of using the wash bottle to dispense the titrant is eliminating the need to refill the buret after each experimental trial. For our experiments, the entire titration (including weighing sample) could comfortably be completed in under 10 min per trial.

Possible experimental errors for the mass-based approach include evaporation of solvent from the wash bottle or deposition of fingerprints onto the bottle between weighings. However, experiments we conducted have cast doubt on the significance of these errors. A study of the solvent evaporation rate (water) resulted in the finding that approximately 0.25 mg/min evaporated from an open Erlenmeyer flask at room temperature in our laboratory. If a titration requires 10−20 min, this mass loss would likely not be recorded on a balance with ±0.01 g precision (mass lost is within the balance tolerance). In addition, the mass of a fingerprint residue is believed to be ≪1 mg, which is far too small to measure with a balance capable of ±0.01 g precision.

■ CONCLUSIONS

It appears that the emergence of inexpensive balances have indeed brought glass burets one step closer to being removed from laboratory service and placed within museums, as R.W. Ramette has suggested in his 2004 work.[3] We encourage textbook authors to adopt the "*molamity*" concept and the creation of laboratory exercises that can be broadly disseminated to introduce mass-based titrations to the next generation of quantitative chemists.

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Notes

The authors declare no competing financial interest.

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