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An Experiment for Introductory Analytical Chemistry

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The most frequently used method of moisture analysis today is the coulometric Karl Fischer (KF) titration technique (*1*). Redox chemistry forms the basis of this method. Moisture reacts stoichiometrically with iodine and sulfur dioxide to form iodide and SO_3:

$$I_2 + SO_2 + H_2O = 2HI + SO_3 \quad (1)$$

The current required to electrolytically generate iodine at the anode

$$2I^- - 2e^- = I_2 \quad (2)$$

is measured and stoichiometrically related to the amount of moisture introduced. The technique is highly accurate and precise, and extremely sensitive. Determinations of low levels of moisture (ca. 50 ppm) can be accomplished in only minutes.

The KF technique is widely used by practicing analysts and continues to be actively investigated (*2–4*). A survey of attendees at the 1999 Eastern Analytical Symposium revealed that 20% use a KF instrument in their work. Moisture has been quantitatively correlated with a wide range of useful properties in an even wider array of diverse materials including foodstuffs, petrochemicals, pharmaceuticals, and cosmetics (*5, 6*). For example, in foodstuffs, water content has been shown to be a useful indicator of a product's freshness.

Owing to its widespread utility, the technique represents an important application of redox chemistry and electrochemistry and is frequently discussed in introductory analytical textbooks in sections about these topics (*7–9*). Thus we were surprised to find that no experiments based on this important technique have been reported for the analytical teaching laboratory in either this *Journal* or *The Chemical Educator*.

There are several likely reasons for this obvious omission. Most likely the omission is due to the specificity of the KF instrument for moisture analysis coupled with the fact that the KF coulometer is not frequently found in undergraduate analytical chemistry teaching laboratories. However, this is most unfortunate, since the KF instrument is relatively inexpensive (ca. $6000), simple to operate, robust toward student use, and microprocessor driven (there is no software to master), and its scientific principles are easily understood. This makes it ideal for introducing students to modern analytical instrumentation and methodology. Furthermore, accurate and precise determinations can be accomplished in a relatively short time (several minutes). Thus one instrument (and one analytical balance) can readily accommodate even a large class with more than 80 students divided among five laboratory sections, when students work as teams with a staggered lab schedule.

In view of the void in the educational literature and the widespread utility and advantages of the technique, we developed and implemented a quantitative analysis lab experiment that uses the KF coulometer to examine the moisture content of various commercial hand and body lotions. It provides an interesting illustration of the theory and practice behind the KF coulometric method of moisture analysis and has proven extremely popular, educational, and instructionally effective with students.

We use real-life samples for analysis in this experiment. As Anne Sherren pointed out several years ago in this *Journal* (*10*), use of real-life samples interests students, helps them to relate personally, and produces greater student satisfaction. While we will illustrate a lab experiment built around the analysis of lotions, many variations on this exercise involving other interesting samples (identified by students), including analgesics, paint, and beverages, can also be envisioned. This is a real strength of using the coulometric KF method and instrument in the analytical teaching laboratory.

We decided to analyze hand and body lotions for several reasons. First, the analysis of moisture in lotions allowed us to help students to develop good critical thinking skills. Most students readily admitted that they had never really thought about how lotions work. After the experiment most students realized that all lotions have a similarly high moisture content and they began to think about the role of the other listed ingredients (many relatively hydrophobic) present in lotions. Most importantly, college men and women both seemed to find the moisture analysis of lotions interesting.

Experimental Procedure

An important part of this experiment is the prelab assignment students are asked to complete one week in advance of the actual laboratory experiment. Students are asked to examine several lotions and to list the major ingredients (see Table 1). They are then asked to consider the solubility properties of these ingredients and identify an appropriate solvent for dissolution of a lotion sample. To help students think about how a lotion works, we ask them to hypothesize which lotion they believe would be the best moisturizer and to provide the rationale for their selection. We also use this exercise as an opportunity to get students to think about solvent compatibility with the analysis and with the materials such as the Acrodisc filter units (0.45-μm pore size, 25-mm diameter) they will use to filter their lotion samples during the experiment.

In the analytical laboratory, students, working in self-identified teams of four, select a lotion sample from a series of available lotions. The viscous nature of lotion precludes direct sample introduction into the KF coulometer. Thus, the lotion sample must be dissolved in some solvent, filtered, and then introduced into the KF instrument for moisture analysis. Consequently, student teams begin the experiment by actively investigating their lotion's solubility in a series of several commonly used organic solvents. For practicality (class size >80 students) as well as safety considerations, the students investigate four common solvents: dimethyl sulfoxide (DMSO, 67-68-5), dimethyl formamide (DMF, 68-12-2), dichloromethane (or methylene chloride, CH_2Cl_2, 75-09-2), and methanol (CH_3OH, 67-56-1). They must select a solvent that both dissolves the lotion sample and is compatible with the KF method. For most lotion samples, methanol, which is frequently used as the solvent for the KF method, fills the bill.

Once students identify an appropriate solvent, they are asked to weigh an 0.4-g lotion sample, dissolve it in 2 mL of methanol, and using the appropriate Acrodisc filter unit, filter the lotion sample for analysis. They also prepare a blank by filtering a 2-mL sample of methanol. Students then analyze their blank and samples for moisture content using the KF unit, a Mitsubishi CA-06 Karl Fisher coulometer (Cosa Instruments), in triplicate. The experiment from start to finish requires 1–1.5 hours.

Hazards

There are two hazards in the experiment as described. Students may or may not understand the health and flammability hazards associated with the KF reagents (anode and cathode solutions) and the organic solvents used. Dichloromethane is a carcinogen and dimethylformamide is listed in at least one compilation of carcinogens. In addition, if the KF unit used requires injection of samples, students will need instruction concerning the proper handling and disposal of disposable syringes and syringe needles. See supplemental material.[W]

Results and Discussion

Table 1 provides representative moisture analysis data for three lotions that we have successfully used in this experiment. Accuracy is strongly dependent on the care with which students prepare their samples. Overall, students usually obtain good precision (0.4–1.5%).

Table 1. Principal Ingredients and Representative Results for Moisture Content of Selected Lotions

Lotion	Principal Ingredients	Moisture Content ± SD (%)
Lubriderm Skin Therapy Moisturizing Lotion	Water Mineral oil Petrolatum Lanolin Sorbitol Stearic acid Lanolin alcohol Cetyl alcohol Glyceryl stearate/ PEG-100 stearate	76.03 ± 1.22
Vaseline Intensive Care Aloe & Naturals Lotion	Water Glycerin Stearic acid *Aloe barbadensis* gel Glycol stearate Sunflower seed oil Sweet almond oil Soya sterol Lecithin Tocopheryl acetate	83.72 ± 0.44
Nivea Body Original Lotion (dry skin)	Triple purified water Mineral oil Glycerin Isopropyl palmitate Glyceryl stearate SE Cetearyl alcohol Tocopheryl acetate Lanolin alcohol Isopropyl myristate Simethicone	80.88 ± 1.55

We have experienced few real difficulties in executing the KF experiment as described above. Perhaps the most significant difficulty was presented by a teaching assistant who asked students to rinse their sample vials with acetone not realizing that the acetone is an interferant that requires the use of special KF reagents. In large laboratory sections, the high moisture content of most lotions may necessitate the analysis of small amounts of lotion to keep the analysis times for individual samples short. The analysis of 0.1-mL injections of samples consisting of 0.40 g of lotion dissolved in 1.6 g of methanol can be accomplished in 5 to 10 minutes.

Student response to the KF experiment has been most gratifying in terms of both student satisfaction and impact on student learning. Students identified this experiment as the best experiment overall, the most useful experiment, and the experiment from which they learned the most. They offered the same wide range of explanations in each case. These included "best instrument", "saw practical applications for instrument", and "I understood it". We believe that in large part it was the consumer product analysis aspect of the KF experiment to which students strongly related. Seventy-six percent of the students responding indicated they enjoy lab experiments more when the experiments involve the analysis of consumer products. The KF experiment is the only consumer analysis experiment in our current lab curriculum.

There have been a few criticisms of the KF experiment. Surprisingly, the major criticism voiced by students was that the moisture determination, which usually was accomplished in 5 to 10 minutes, "took forever". This was most intriguing, in view of the students' prior experience with a traditional, noninstrumental acid–base titration (typically the longest lab experiment)! At any rate, students' desires for a more speedy analysis clearly did not adversely affect their satisfaction or learning.

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[W]Supplemental Material

Prelab questions, detailed instructions for the experimental procedure, a discussion of hazards, and notes for TAs are available in this issue of *JCE Online*.

Literature Cited

1. Scholz, E. *Karl Fischer Titration: Determination of Water. Chemical Laboratory Practice*; Springer: New York, 1984.
2. Dantan, N.; Frenzel, W.; Kuppers, S. *Talanta* **2000**, *52*, 101–109.
3. Nordmark, U.; Cedergren, A. *Anal. Chem.* **2000**, *72*, 172–179.
4. Margolis, S. A. *Anal. Chem.* **1998**, *70*, 4264–4270.
5. DeSandro, J.; Kertis, M. E. *Am. Lab.* **1986**, *18*, 106–113.
6. Masa, C. G. *R & D* **1998**, *40*, 40–46.
7. Mendham, J.; Denney, R. C.; Barnes, J. D.; Thomas, M. *Vogel's Textbook of Quantitative Chemical Analysis*, 6th ed.; Pearson Education: Edinburgh Gate, 2000.
8. Harris, D. C. *Quantitative Chemical Analysis*, 3rd ed.; Freeman: New York, 1991.
9. Skoog, D. A.; West, D. M.; Holler, F. J.; Crouch, S. R. *Analytical Chemistry: An Introduction*, 7th ed.; Saunders: Fort Worth, TX, 2000.
10. Sherren, A. T. *J. Chem. Educ.* **1991**, *68*, 598–599.