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Gravimetric Determination of the Nonvolatile Content of Paint

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This activity provides the student with an introduction to gravimetric procedures and at the same time introduces the basic components of paints. A small amount of paint is weighed into one of two aluminum foil weighing dishes. Nesting the second dish in the first covers the paint sample, making it possible to weigh the sample without weight loss due to evaporation of volatile components, which begins to occur as soon as the paint is exposed to the atmosphere. The paint sample is then spread into a film by pressing the nested (second) dish against the first. The two dishes are separated and the paint films on them are dried in a forced-air oven. After 30 minutes of drying, the dishes are reweighed. The mass data is used to calculate the percent nonvolatile matter in the paint sample.

Safety, Handling, and Disposal

It is your responsibility to specifically follow your institution's standard operating procedures (SOPs) and all local, state, and national guidelines on safe handling and storage of all chemicals and equipment you may use in this activity. This includes determining and using the appropriate personal protective equipment (e.g., goggles, gloves, apron). If you are at any time unsure about an SOP or other regulation, check with your instructor.

Work in a fume hood or well-ventilated area. Avoid breathing vapors from oil-based paints. When working with paint, avoid contact with skin. Some paints are flammable; keep paints away from flame and excessive heat.

Materials

Per determination

- 2 round aluminum foil weighing dishes about 1.8 cm (3/4 inch) deep, with diameter short enough to allow placement in the foil tray (see Procedure, step 1)
- rectangular piece of aluminum foil about 13 cm x 30 cm (5 inches x 12 inches)
- analytical balance sensitive to 0.0001 g
- forced-air oven thermostatically controlled at 105–107°C
- about 0.5 g paint
- wooden tongue depressor
- small piece of paper

Procedure

1. Use the rectangular piece of aluminum foil to make a small tray by first folding the piece in half to form a double layer and then folding up the edges. The dimensions of the tray must be such that it fits on the sample pan of the balance.

2. Place the two aluminum foil dishes in the tray and determine the combined mass of the three items. Record this as the mass of dishes and tray (mass A in step 9).

3. Make sure that the paint has been thoroughly mixed prior to use. Weigh a 0.5 ± 0.1 g paint sample into one of the aluminum dishes (This is only a crude weighing for approximate sample size; the accurate mass measurement is made in step 4.) Immediately nest the second dish in the dish containing the paint sample, thereby trapping the sample between the two dishes.
One convenient method of transferring the paint from its container to the aluminum dish is to use a wooden tongue depressor. First dip the end of the stick into the paint, remove it, and hold it horizontally over the paint container to allow excess paint to drip off. When the paint has stopped dripping, move the stick so that the paint-covered end is over the aluminum dish. Tilt the stick slightly so that paint slowly drips into the dish. When the specified amount has been added, return the stick to a horizontal position to stop the flow of paint. It may be advisable to practice the maneuvers of the paint transfer over the paint container.

4. Promptly determine the combined mass of the tray, nested dishes, and wet paint sample. Record the mass measurement (mass B in step 9).

5. Remove the nested dishes containing the paint sample from the tray. Place a small piece of paper in the inner dish to prevent the transfer of oil from fingers. Spread the paint sample into a film by using finger pressure in the inner dish and rotating the outer dish around the inner one. Remove the paper. Take care to avoid losing any of the paint from the dishes during the spreading. If any part of the paint sample is lost, a new sample must be started beginning again with step 1.

6. Holding the nested dishes over the tray to catch any drips, separate the dishes from one another. Place the dishes in the tray so that each has the surface wetted with paint facing up (the outer dish will be right-side-up and the inner dish will be upside down).

7. Place the tray containing the two paint-covered dishes in an oven pre-heated to and maintained at 105–107°C (225°F) for 30 minutes.

8. Remove the tray and dishes from the oven and allow them to cool at room temperature for 5 minutes. Determine the combined mass of the tray and dishes with dried paint sample (mass C in step 9).

9. Calculate the fraction nonvolatile matter by weight as a percent:

$$\text{percent nonvolatile matter} = \frac{\text{mass of the dried paint residue}}{\text{mass of the wet paint sample}} \times 100$$

where mass of the dried paint residue (the nonvolatile matter) = C – A

and mass of the wet paint sample = B – A

Discussion

Consider a more detailed version of the equation in step 9 above:

$$\text{fraction of nonvolatile matter} = \frac{\text{mass of nonvolatile residue}}{\text{mass nonvolatile + mass volatile liquid}}$$

Discuss how variations in procedures and handling might influence the numerator or denominator of the fraction and what effect this would have on the calculated fraction of nonvolatile matter. As an example, consider what would happen if the second dish was not nested quickly into the dish containing paint in step 3. Some of the volatile liquids would evaporate before the mass is measured. This evaporation would cause the contribution from the liquid to the measured mass of wet sample to be lower than the true value. Thus, the denominator of the fraction would be smaller than it should be and the calculated fraction would be too high.

Explanation

The determination of the nonvolatile content of paint as done in this activity illustrates a gravimetric method, one based on the measurement of mass. This particular gravimetric determination is based on volatilization. The volatile components of the paint are eliminated during heating. The mass of the resulting dry paint is compared to the mass of the wet paint sample to calculate the fraction of nonvolatile matter.

The volatile components of paint are mainly the solvents or diluents used to thin the paint. In water-based latex paints, this volatile component is water. In oil-based paints, the volatile components may be any of many organic compounds including aliphatic hydrocarbons, terpenes, aromatic hydrocarbons, alcohols, esters, ketones, or ether-alcohols.

The dry paint film left after heating constitutes the nonvolatile component. It contains the colored pigment as well as extender pigments and the film-former or binder, which not only binds the pigment particles together but also holds them to the surface to be covered.

Since the measurement of mass is the only data obtained in a gravimetric method, procedures and techniques of sample handling that would yield the incorrect mass would be sources of error. If the paint sample is not completely dried before the final mass measurement, the calculated percent nonvolatile will be too high. If some of the paint sample is lost due to handling or dripping after the initial weighing, the calculated results will be too low. The moisture and oil from fingerprints on the container may have significant mass and therefore influence the results. The degree to which fingerprints might influence the results is hard to predict, since fingerprints consist of varying amounts of added matter, some of which might be partially removed by the heating process. Even complete cooling of the dried sample before making the mass measurement is important; although mass is not temperature-dependent, the convection currents present when a heated object is on the balance pan make it difficult to accurately measure the mass. In general, gravimetric procedures require clean and efficient sample handling.

In the paint industry, this relatively quick method of determining nonvolatile content of paint finds use in quality control as well as in evaluation of competitive materials. It would also be part of a determination of volatile organic compounds (VOC). The method is most useful for VOC

determination when the coating is known not to contain water. Nonvolatile determinations are important information in the purchase of paint under specifications. Paint specifications almost invariably include minimum nonvolatile content.

References

Southern Society for Paint Technology. "A Rapid Method for the Determination of Nonvolatile Content for Liquid Coatings," *Journal of Paint Technology*, 1968, 40 (517), 93–98.

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