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Microscopical Methods for Vapor Analysis

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KEYWORDS

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ABSTRACT

The microscope is not the first instrument that comes to mind when one thinks of analyzing a substance in the vapor phase. Solid particles are commonly placed onto microscope slides, dispersed, crushed, immersed in an appropriate mounting medium, topped with a coverglass, and thus prepared for microscopical analysis. Liquids can be placed onto a slide and analyzed as well, but how can gases be examined microscopically? This paper addresses the idea of using microchemical methods to detect vapors generated by chemical reactions conducted on microscope slides. The identification of vapors can aid in determining the chemical composition of the original sample.

INTRODUCTION

Many chemical reactions generate vapors and the detection of these gaseous products can help an analyst identify a substance from which vapors originate. In conducting chemical reactions on microscope slides, methods can be developed and microtechniques can be used to identify vapors formed through such reactions. Sometimes vapors are released at standard temperature and pressure and can be analyzed directly, but more commonly, the analyst will need to induce the release of a vapor by heating the sample or conducting a chemical reaction. Several microscopical techniques for inducing the evolution of gases will be discussed, and a variety of methods for detecting vapors will be presented. This article includes specific examples of vapor analysis.

MATERIALS AND METHODS

When an analyst has little or no information about a sample, some important clues can reveal themselves when the sample is being prepared for analysis. By making careful observations, one may notice that the specimen is hygroscopic or that it crushes easily when pressing on the coverglass. The hardness, texture, color, luster, or opaqueness may be revealed. It may dissolve or react with the mounting medium. These are all important characteristics that are indicative of the sample's chemistry.

Likewise, the sample in question may exhibit a particular odor. If so, the sample is obviously emitting a vapor. If one detects an odor of vinegar, acetic acid is present; if an odor of rotten eggs is noted, then hydrogen sulfide is present; if a burning sulfur odor, then sulfur dioxide; if a garlic odor, then arsine or phosphine; if an almond odor, then cyanide, etc. If the odor is part of the analyst's organoleptic repertoire, a chemical identification of the vapor may be made immediately; if not, then microchemical vapor analysis is an option.

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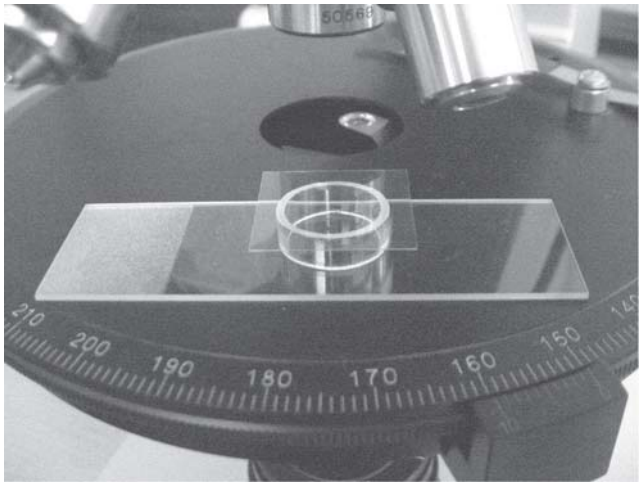

Figure 1. Vapor chamber on a microscope stage.


Figure 2. Chemically induced effervescence.

One of the best ways to capture a vapor and subsequently perform a chemical test is to use a vapor chamber (Figure 1). This is a very simple, inexpensive device constructed by simply placing a glass ring on a microscope slide and then placing a coverglass on top of the ring. Most chemical microscopists are familiar with this technique which is often used for solubility testing, but it is also ideal for testing unidentified vapors. (Solubility testing is performed by placing a solid particle on the underside of the coverglass and sequentially placing drops of various solvents at the base of the ring. Vapors of each solvent fill the chamber and if soluble, the edges of the particle in question begin to round off or dissolve.) A sample placed within the chamber may emit a vapor spontaneously or vapor formation may be while performing microchemical tests.

Gases can be formed by using reagents that react with the substance in question to generate a vapor that is subsequently detected and identified. For example, if one places sodium hydroxide on a sample containing the polyatomic ion ammonium, hydrogen ions from the ammonium react with the hydroxide ions from the sodium hydroxide, and the result is ammonia gas is generated and can be detected using litmus paper or Nessler's reagent. By treating nitrates and nitrites with potassium hydrogen sulfate or dilute sulfuric acid, nitrogen dioxide, a reddish brown gas, is liberated. Potassium hydrogen sulfate can also be used to liberate carbon monoxide from oxalates. When treated with acid, the more reactive metals will generate hydrogen gas. Sulfuric acid reacts with fluoride compounds to form hydrofluoric acid. In the later case, the vapor may etch the coverglass covering the chamber. If effervescence is observed after applying a reagent, gases are obviously being produced (Figure 2). To diminish the rate at which the bubbles of the liberated gas dissipate, allowing more time for observation, a specimen may be covered with a viscous liquid through which a chemical reagent is applied.

If vapors are not naturally emitted from the sample or chemically induced, they can sometimes be generated by applying heat to the sample. Upon heating, water vapor frequently condenses on the coverglass to form water droplets. This may be an indication that the original sample was a hydrated (solvated) compound, and a color change may accompany this decomposition. Another consequence of heating is sublimation. Many organic substances, including drugs and paint pigments, as well as a few inorganic compounds such as arsenous oxide and mercuric sulfide, will sublime. The vapor then condenses on the coverglass forming tiny crystals. This can be used as a separation technique by isolating a pure substance and allowing it to recrystallize. The resulting crystals are pure, well-formed, and very suitable for optical characterization (Figure 3).

A vapor filling the chamber may exhibit color (Figure 4). For example, iodine vapor is violet, chlorine gas appears yellow or yellow-green, and bromine is reddish-brown, but if the vapor is colorless, a variety of other detection methods can be used. Any vapor emitted from the sample is trapped in the chamber and may then react with: a small crystal suspended beneath the coverglass via static attraction with a drop of liquid suspended beneath the coverglass via surface

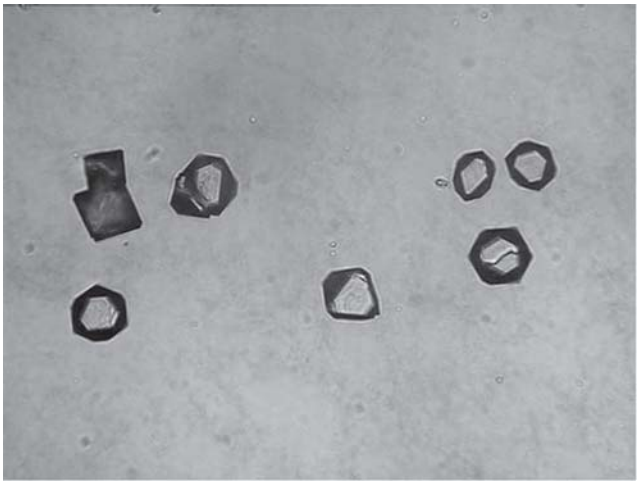

Figure 3. Thermally induced emission of vapor forming a sublimate (As_2O_3) on coverglass.

Figure 4. Iodine vapor condensing on coverglass to form shiny diamond-shaped crystals.

tension or with a piece of moistened test paper stuck to the bottom of the coverglass. By using various techniques, practically any chemical reaction involving a gas can be conducted.

A hanging reagent drop can be used to detect the presence of specific vapors. Carbon dioxide vapor can be detected by reacting with a hanging drop of aqueous barium hydroxide due to the formation of barium carbonate, resulting in a cloudy or milky liquid. A hanging drop of diphenylamine will react with any oxidizing vapor, resulting in a deep blue coloration. Many such color reactions can be devised by adapting general spot tests to a micro scale and performing them in a vapor chamber.

With respect to test papers being placed beneath the coverglass in a vapor chamber, litmus paper can be used to determine the acidity or basicity of a vapor. Lead acetate paper can be used to detect hydrogen sulfide or hydrogen selenide. Potassium iodide-starch paper turns blue in the presence of chlorine gas, bromine gas, and nitrogen dioxide. As with these examples, numerous tests can be conducted by impregnating filter paper with any chemical of choice, allowing the vapor in question to react with the test paper, and noting any color change.

Another vapor detection technique is to place a glowing splint above the sample in place of the coverglass. If hydrogen gas is being generated, a popping sound will be heard, and if oxygen is being formed, the splint will burn. On the other hand, if the splint is exposed to carbon dioxide, sulfur dioxide, or nitrogen gas, the glow will be extinguished. All vapor tests should be conducted using a microscope dedicated to microchemical testing, and the objective lens should be protected by a coverglass or some other transparent, chemically resistant material.

CONCLUSIONS

In summary, vapors that are either naturally emitted, chemically formed, or thermally induced can be tested using a variety of microscopical techniques and a number of detection methods. A good understanding of chemistry and chemical principles is helpful in developing these methods and is necessary to accurately interpret the results, but through careful observations and with a little practice, the analyst can use the simple and inexpensive techniques presented here to widen his or her scope of analysis to include vapor testing.

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