9A-2 Electrothermal Atomization

- Enhanced sensitivity due to
  - entire sample is atomized in a short period,
  - the average residence time of the atoms in the optical path is a second or more.
- A few microliters of sample are first evaporated at a low temperature and then ashed at a somewhat higher temperature in an electrically heated graphite tube or in a graphite cup.
- Then the current is rapidly increased to several hundred amperes, which caused the temperature to soar to perhaps 2000°C to 3000°C; atomization of the sample occurs in a period of a few milliseconds to seconds.
- The absorption or fluorescence of the atomic vapor is then measured in the region immediately above the heated surface.

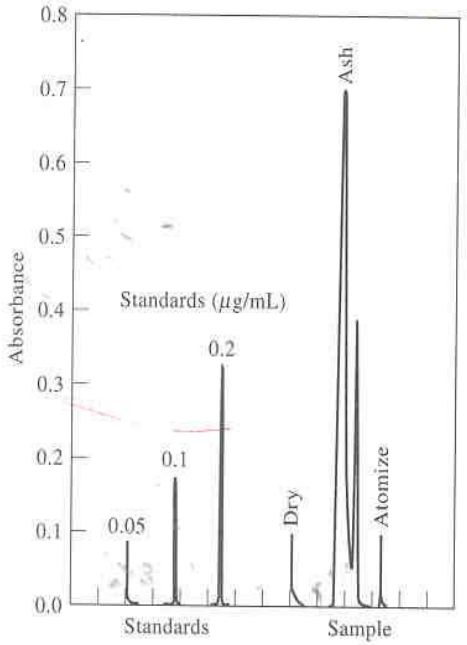
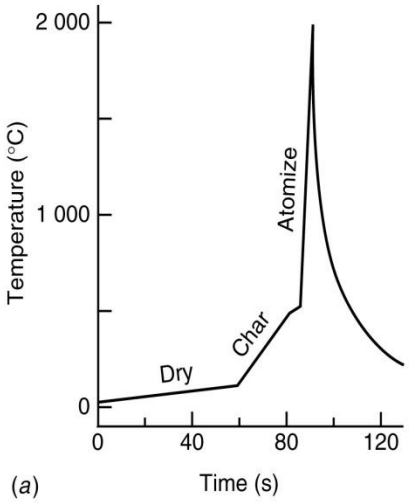


Figure 9-7 Typical output from a spectrophotometer equipped with an electrothermal atomizer. The sample was 2 μL of canned orange juice. The times for drying and ashing are 20 and 60 s, respectively. *(Courtesy of Varian Instrument Division, Palo Alto, CA.)*

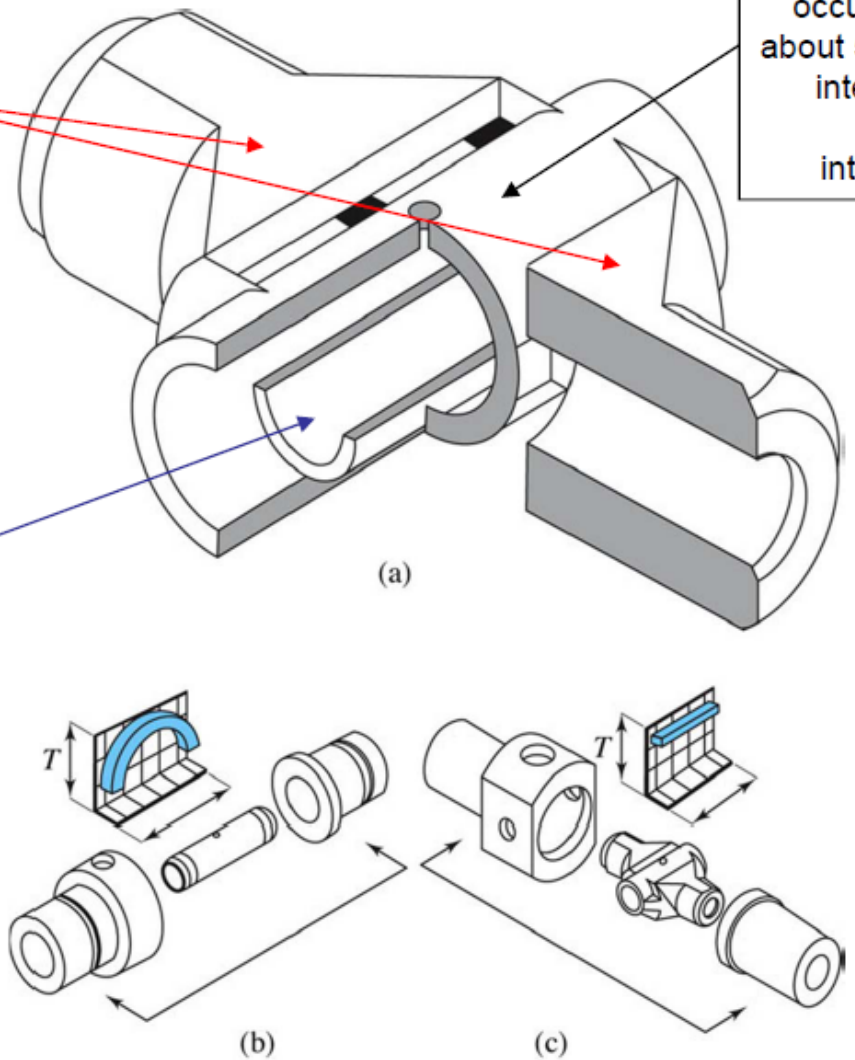


Commercial electrothermal atomizer.

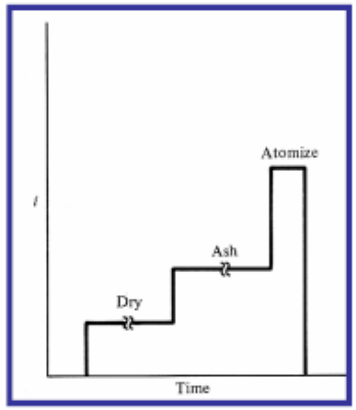
Cylindrical graphite electrical contacts. These contacts are held in a water-cooled metal housing.

Cylindrical graphite tube where atomization occurs. Dimensions: about 5cm long and 1cm internal diameter. This tube is interchangeable.

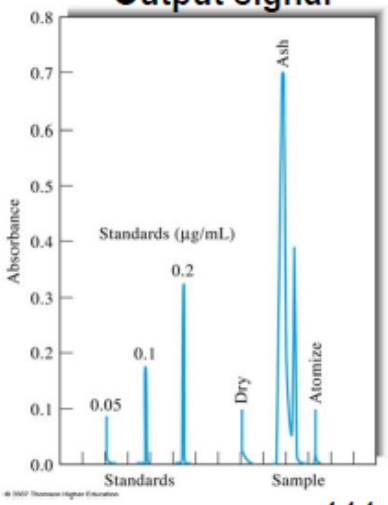
L'vov platform. Made of graphite, sample is evaporated and ashed on this platform. Temperature on the platform does not change as fast as it changes in the walls of the furnace. Atomization occurs in an environment where temperature does not change so fast, which improves reproducibility of measurements. Facilitates furnace cleaning, which reduces memory effects.



Longitudinal (b) and transversal (c) furnace heating. Transversal mode is preferred because it provides a uniform temperature profile along the entire length of the tube and optical path.



Output signal



Pyrolytic coating of graphite tubes

-reducing the natural porosity of the graphite tube minimizes some sample matrix effects and poor reproducibility associated with graphite furnace atomization.

-During atomization, part of the analyte and matrix apparently diffuse into the surface of the tube, which slows the atomization process, thus giving smaller analyte signals.

-To overcome this effect, most graphite surfaces are coated with a thin layer of pyrolytic carbon, which seals the pores of the graphite tube.

Performance Characteristics of Electrothermal Atomizers:

- Electrothermal atomizers offer the advantage of unusually high sensitivity for small volumes of sample. Typically, sample volumes between 0.5 and 10 μL are used;
- Absolute detection limits lie in the range of 10^{-10} to 10^{-13} g of analyte.
- The relative precision of electrothermal methods is generally in the range of 5% to 10% compared with the 1% or better that can be expected for flame or plasma atomization.
- Because of the heating-cooling cycles, furnace methods are slow; typically requiring several minutes per element.
- A final disadvantage is that the analytical range is low, being usually less than two orders of magnitude.