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NASA TECH BRIEF



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Standards for Electron Probe Microanalysis of Silicates Prepared by Convenient Method

The problem:

The effective use of the electron probe for microanalysis of various silicates is often made difficult or impossible by the lack of adequate standards approximating the composition of the silicate samples to be analyzed.

The solution:

Standard compositions, suitable for electron probe microanalysis, prepared by a method involving coprecipitation of specified salts with colloidal silica to form a gel, which is then decomposed by heating into a mixture of powdered oxides that is compressed into thin pellets.

How it's done:

Standardized aqueous solutions of the desired salts (e.g., aluminum nitrate and ferric nitrate) are mixed with dilute nitric acid and a commercial mildly alkaline aqueous sol of silica to form a gel. This gel, which is an aggregate of the salts and silica particles, is rapidly desiccated by spraying it onto platinum foil whose undersurface is heated with a gas burner. The desiccated powder on the platinum foil is then converted into an intimate mixture of oxides by heating it in a furnace at approximately 900°C for two hours. The resulting powdered oxide mixture is scraped from the platinum foil and formed into a dense, smooth surfaced, translucent pellet using a pair of tungsten carbide anvils with 0.25-inch-diameter polished faces under a pressure in the range of 30 to 50 kilobars. The pellet is then coated with carbon and mounted on a microprobe specimen holder for use as a standard.

Notes:

1. The pellets are not perfectly homogeneous to the

microprobe and thus require special analytical techniques for their use. A suitable technique is to integrate X-ray counts from the pellet while it is slowly moved under the electron beam to average out local variations in composition.

2. Only portions of the prepared solutions need be converted to gels and desiccated at one time. The remaining solutions can be stored and later modified in composition as required.
3. The pellets can be used as standards for microprobe analysis of multicomponent silicate systems by successive approximations. In this method a rough approximation of the silicate sample composition is made and a suitable standard prepared. Comparison of the sample with this standard will indicate the composition of a second standard that more closely approximates the sample composition. This process is repeated until the desired precision is obtained.
4. These easily prepared standards are useful when more homogeneous, stable crystalline or glassy standards are not available.
5. Inquiries concerning this innovation may be directed to:

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Reference: B66-10234

Patent status:

No patent action is contemplated by NASA.

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