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Brief 71-10359

NASA TECH BRIEF

Lewis Research Center

Simplified Procedure for Emission Spectrochemical Analysis

A simplified single procedure for rapid quantitative spectrochemical analysis can handle virtually any metallic sample, regardless of size or composition.

Emission spectroscopy, a well established technique in chemical analysis, is especially suited to high volume routine analyses of a single sample type; i.e., samples of the same physical form and of relatively narrow composition ranges. The technique becomes progressively more limited in applications with samples of varying sizes, compositions, and physical structures, and ultimately becomes impractical for the quantitative analysis of few-of-a-kind samples. The major limitation in the spectrochemical analysis of non-routine samples is the necessity to develop detailed procedures, such as sample preparation, spectra excitation, quantitative calibrations, and data reduction, for each different sample type. The time and cost required to quantify the procedures effectively cancels the potential advantages of the spectrochemical technique for analyzing only a few samples.

In the new technique, a relatively simple procedure involving dissolution of samples is used to prepare all samples for analysis, and all samples are analyzed using a single set of experimental conditions. In many cases, no comparison standards are necessary because the procedure effectively reduces so-called matrix effects. (When standards are necessary, they can be easily prepared by blending stock solutions of the various metal constituents.) This single procedure can be used to analyze a wide variety of materials and compositions, without compromising analysis accuracy and without requiring numerous separate and more complex procedures.

Because the identical procedural steps are used for all dissolved samples, the procedure has been automated to achieve a high sample rate. With the use of this automated spectrometer, the per sample analysis time is about four minutes after sample dissolution. Normally, triplicate excitations are made of each sample solution. The system will perform automatic excitations of a series of samples and will record the analysis data from a maximum of 22 metal constituents. Computer data processing is used to compute either absolute microgram amounts of metals or percentage compositions. The computer program also provides automatic correction of spectral line interferences.

The instrument system and procedures have been successfully applied to the quantitative analysis of a wide variety of sample types, including high temperature and refractory alloys, extracted phases from these alloys, oxide vapor deposits on platinum and glass substrates, 10-microgram microspheres of condensed metal aerosols, metal constituents concentrated at fracture surfaces of alloys, industrial hygiene and pollution samples, and trace metals in biological samples.

This development was made possible by several key refinements in the use of a direct current arc which is used to excite the atomic spectra. The elimination of arc wander and the control of light intensity or arc current, together with the operation of the arc under closely controlled conditions in argon atmospheres, resulted in the required detection limits and precision for the successful application of this procedure.

Notes:

1. Control of the arc current light intensity is described in NASA Tech Brief 67-10404, Control Apparatus for Spectral Energy Source, which may

(continued overleaf)

be obtained from the Technology Utilization Division, NASA, Code KT, Washington, D. C. 20546.

2. The following documentation may be obtained from:

   National Technical Information Service
   Springfield, Virginia 22151
   Single document price $3.00
   (or microfiche $0.95)

   Reference:
   NASA-TN-D-5532 (N70-10367), Quantitative Direct Current Arc Analysis of Random Compositions of Microgram Residues in Silver Chloride Common-Matrix

3. Technical questions may be directed to:

   Technology Utilization Officer
   Lewis Research Center
   21000 Brookpark Road
   Cleveland, Ohio 44135
   Reference: B71-10359

Patent status:

No patent action is contemplated by NASA.

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