Uranium Isotopes Quantitatively Determined by Modified Method of Atomic Absorption Spectrophotometry

**The problem:**
To determine the quantities of uranium isotopes present in a sample by the use of atomic absorption spectrophotometry. In atomic absorption spectrophotometry, the atoms must be chemically unbound, and in their minimum energy state. This condition is usually achieved by vaporizing the sample in a flame; however, refractory elements such as uranium are not easily dissociated by the flame.

**The solution:**
The use of hollow-cathode discharge tubes to effect dissociation of the uranium atoms. By this technique, a large population of ground state uranium atoms can be produced to absorb the incident radiation that is slightly different for the two major isotopes of uranium. This technique can also be applied for the elemental analysis of uranium.

**How it's done:**
There are two methods by which this technique may be used in the quantitative determination of uranium isotopes. In Method I, the absorption tube contains a relatively pure sample of the isotope to be determined; in Method II, it contains the actual sample to be analyzed.

**Method I**
Two emission tubes are used. The sample for analysis is placed in one emission tube, and relatively pure samples of the isotope to be analyzed are placed in both the other emission tube and in the absorption tube. The incident emitted light from excitation of the sample in emission tube No. 2 is focused on the hole of the cathode electrode of the absorption tube by two parallel lenses. A mirror is placed in the path of light between emission tube No. 2 and the absorption tube. This mirror can be rotated 45° to block the light path from the emission tube No. 2, and reflect the incident light from emission tube No. 1 toward the absorption tube.

A 60-cycle mechanical chopper in front of the absorption tube modulates the incident light. The two parallel lenses focus the transmitted light onto a 25μ entrance slit of a Jarrell–Ash Model 82-00 Monochromator equipped with a 1P21 photomultiplier tube.

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As an example of the operation of the system, assume that pure uranium-238 is used in the absorption tube and in one emission tube, and the other emission tube contains an unknown sample composed of both U-238 and U-235 isotopes. The concentration of U-238 in the unknown sample can be determined directly since the U-238 concentration is a linear function of transmittance. The U-235 can thus be determined by difference, if, as is usual, the total U-234 and U-236 concentration is 1% or less.

Method II

In this procedure, the unknown sample to be analyzed is placed in the absorption tube. The emission tubes contain U-235 and U-238, respectively. Results of analysis are then obtained from an established analytical curve. The actual operation is analogous to Method I.

Notes:

1. In Method I, more freedom of sample choice is possible when the unknown sample is used in the emission source, since the proportion of incident light absorbed is independent of emission intensity. Thus, metal chips, oxides and compounds can be used as samples.

2. Method II lends itself to the analysis of uranium rods used in nuclear reactors. To prepare a sample for the absorption source, a minimum amount of work is required by just drilling a hole through a section of a rod to obtain a cylindrical insert for the absorption tube.


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