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ATOMIC SPECTROSCOPY

VOLUME 3

CHAKRABARTI

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VOLUME 3

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Volume 3

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ANALYSIS OF NUCLEAR ENERGY MATERIALS

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Analysis of Nuclear Energy Materials

G. Baudin

INTRODUCTION

Nuclear energy is produced by the fission of an atomic nucleus of a fuel element under the impact of a neutron. This fission is accompanied by the production of neutrons which, in their turn, split other nuclei, giving rise to a chain reaction. The latter is only sustained if the neutrons are not absorbed elsewhere, e.g. by other elements. In actual fact, many elements with high neutron capture cross-sections absorb neutrons. Their presence, even at very low levels (10^{-5} to $10^{-7}\%$) is undesirable in nuclear fuels or elsewhere where they may affect the process.

The need to check this nuclear purity emerged very early in the history of nuclear science. In his introduction to his book on the Analytical Chemistry of the Manhattan Project, Rodden began by saying, 'the necessity for high chemical purity of many of the materials used for the production of atomic energy was recognized in the early stages of the project'.

This need to determine the combined presence of many impurities and to check very low levels of concentration, partly explains why the rapid development of emission spectroscopic methods in the fifties occurred simultaneously with that of nuclear energy. Nor is it surprising that many original methods (carrier distillation, copper spark, porous cup, etc.) were developed at centres for nuclear research.

Spectroscopic methods still account for a major part of nuclear testing today. The methods generally used are optical emission, because of the large number of elements to be determined, atomic absorption (chiefly flameless methods) because of the ability of analysing microsamples, and X-ray fluorescence, which still remains one of the major methods for determining traces of uranium.

The importance of nuclear energy research during the sixties was reflected by a plethora of publications. In recent years, with the major decisions already having been taken and brought to the industrial stage, research efforts have been reduced considerably and some laboratories have even been forced to convert to other fields of application with the result that publications on nuclear analysis have become a rarity.

As far as can be ascertained, only two publications have appeared in the past three years concerning the determination of fissile elements in rocks using the inductively-coupled plasma (ICP) together with a dozen or so publications on the analysis of the oxides of uranium (U_3O_8) and zirconium (ZrO_2), and of the metals, zirconium, uranium and plutonium. None offers any truly new ideas. This statement does not take into account certain publications dealing with the analysis of water and steels, which are of interest for nuclear applications.

This relative reduction of interest in spectrochemical methods in the nuclear field is due to the fact that they are poorly adapted to the new centres of interest. They are not sensitive enough for pollution measurements, especially for determining uranium and plutonium, in comparison to radioactivity measurement methods. They are also not accurate enough with respect to the overall balance, because the reproducibility required is at the level of 0.1% relative.

EMISSION SPECTROSCOPY

1 TYPES OF MATERIAL AND CONCENTRATION RANGES COVERED

Purity requirements apply to all components of a reactor core, viz. fuels (uranium, plutonium, thorium, etc.) cladding materials (magnesium, zirconium, stainless steel), moderators (graphite, heavy and light water), and coolants, particularly those used in the primary circuits which penetrate into the core (water, sodium, organic liquids, molten salts, etc.).

All these ingredients are combined together to form reactors of a certain type, for example, uranium-magnesium-graphite-carbon dioxide for graphite-gas reactors, uranium-zirconium-water for some pressurized water reactors, and (uranium-plutonium)-stainless steel-sodium for fast neutron 'breeder' reactors.

The types of impurity and maximum allowable concentrations for any given material depend on the type of reactor in which it is used. Table 1 provides an example for permissible maximum impurities in uranium and plutonium oxides.

Since the essential component is the fuel element, the most stringent checks are applied to it throughout all stages of its service life. Consider the uranium cycle. At the outset, it is present in the form of ores of different origins, and an attempt is made to determine its concentration accurately. Concentrates are produced by chemical processing of this ore. Generally these concentrates are magnesium and ammonium uranates. The uranates may be converted to fluorides from which the metals are extracted by calciothermy or magnesiothermy. All uranium-bearing compounds are checked together with the materials employed, in order to determine the concentration of metals such as calcium. The fuel is then clad with metals and alloys to which specific nuclear limitations are applied in addition to others imposed for metallurgical reasons. Hence very low boron, cobalt and nitrogen contents are required for a stainless steel used to clad a fuel element.

Table 1. Maximum permitted concentrations for metal impurities in uranium and plutonium oxides ($\mu\text{g/g}$)

Specification	Ceramic grade UO_2	Ceramic grade PuO_2	Mixed oxide fuel pellets	Insulator pellets
Al	500	250	500	500
B	10	10	20	20
Be	20	20	20	20
Ca	100	500	250	250
Cd	20	20	20	20
Co	10	20	20	10
Cr	200	200	250	250
Fe	400	350	500	500
K	200	200	200	200
Li	10	10	10	10
Mg	25	100	25	25
Na	500	300	500	500
Ni	400	300	500	500
Ta	400	400	400	400
W	100	100	100	100
V	400	200	500	500
Cu, Si, Ti, Zn,	800	800	800	800
Ag, Mn, Mo, Pb, Sn	200	200	200	200
Dy, Eu, Gd, Sm	100	100	100	100

The fuel burns in the reactor, giving rise to many new elements created by fission processes, so-called fission products, e.g. transuranium elements, rare earths, noble metals, molybdenum, strontium, cesium, etc. Their concentrations and proportions vary with the irradiation rate, the fuel composition and the type of reactor. In a final stage of the cycle, the irradiated fuel is sent to re-

processing plants for the recovery of unburnt fuel, certain transuranium elements (chiefly plutonium) and finally, for separation of the fission products.

Spectrographic inspection in these two latter stages becomes far more intricate owing to the radioactivity present, and requires the use of special installations such as glove boxes or lead wall shielding for sample handling. As is evident, the materials requiring testing are numerous; some of them are specific to nuclear techniques and will be dealt with in detail. Others, which have many uses, will be considered from the nuclear aspect only. The reader is referred to the remaining sections for greater detail.

2 URANIUM

Natural uranium or uranium enriched with the isotope ^{235}U is the most widely used fuel in the form of the low alloy metal (zirconium-uranium, zirconium-uranium-niobium, aluminium-uranium), UO_2 oxide, mixed oxides $\text{UO}_2\text{-PuO}_2$ and ceramics (UC, UN, etc.). In most cases, testing may be reduced to that of the uranium metal.

The first problem encountered by the spectroscopist is due to the highly complex spectrum of uranium which consists of thousands of lines of almost equal intensity. With a spectrograph of average dispersion, the spectrum appears continuous and it is impossible to detect the presence of lines of nearly all the elements requiring trace determination. It is therefore essential to eliminate the uranium spectrum. Three techniques are available for doing this, viz. carrier distillation of impurities, physical separation of impurities by thermal evaporation with condensation on a support and chemical separation before spectrographic analysis.

As long as the impurity level is not too low, it is possible by using a high dispersion spectrograph, to perform the analysis by the direct excitation of the uranium.

2.1 Carrier Distillation

A very elegant procedure for separating impurities was suggested by Scribner and Mullin (1). It consists of placing in the bottom of the crater of a graphite electrode (Fig. 1), 100 mg of a mixture containing 98% uranium oxide (U_3O_8) and 2% gallium oxide (Ga_2O_3). By using a specially shaped tool (Fig. 2), a chimney is created in the centre of the oxide charge for removal of the gases and vapours. The graphite electrode, placed on a 1/8 inch diameter pedestal serves as the anode, and the counter-electrode, also 1/8 inch in diameter, is separated from it by a gap of 4 mm. The distillation and excitation is achieved

using a 13 A DC arc. Under these conditions the uranium is not excited and the spectrum obtained results exclusively from the impurities and the gallium. Chromium at a concentration of 1% is added to the carrier and is used as an internal standard (2). By using this method, the authors determined 33 impurities in a single exposure. This novel method has, since its origin, undergone several modifications, some slight, others far more extensive, particularly with respect to the selection of different carriers.

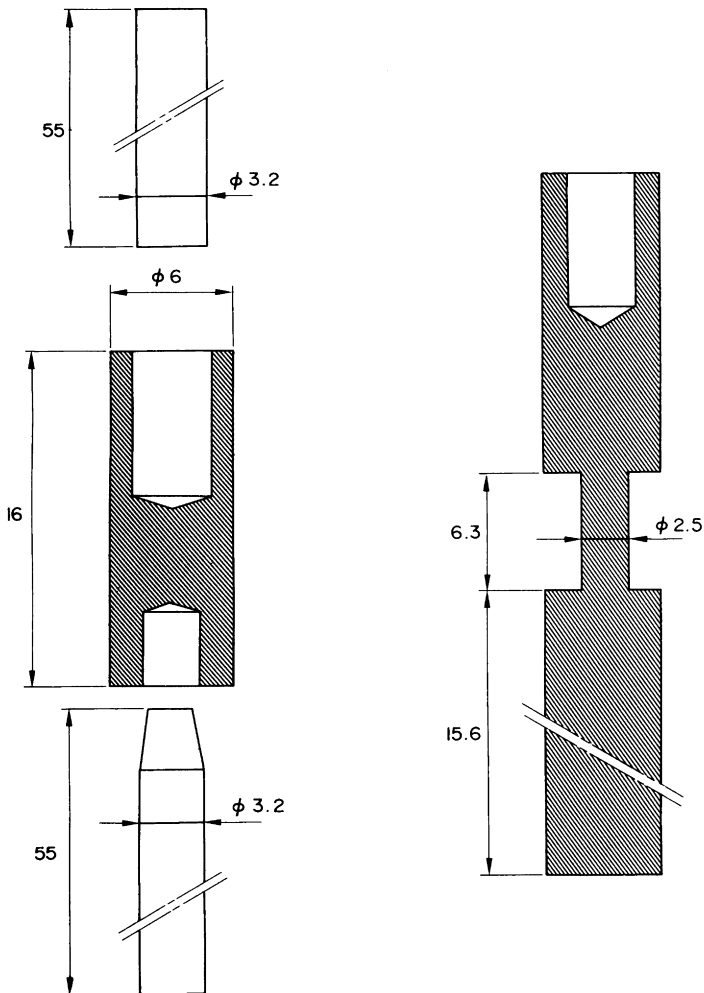


Fig. 1. Electrodes for carrier distillation.

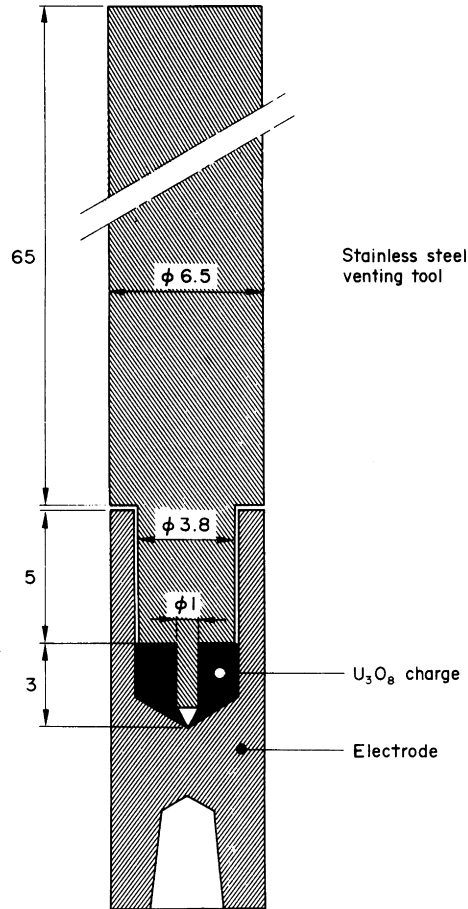


Fig. 2. Stainless steel venting tool.

2.1.1. Gallium Oxide. In arcing an electrode containing the sample as described above, three periods are distinguishable in the burn-off process in the arc, firstly, a short period lasting about 10 seconds, called the 'hissing period', secondly, a period of great arc stability or 'silent period' which is related to the amount of gallium oxide added to the charge (about 30 seconds for 2% Ga_2O_3 and perhaps 60 seconds for 6% Ga_2O_3 ; the duration also varies with the size and packing of the particles and it is during this period that the impurity emission is recorded) and finally, a period of combustion of the charge during which impurity emissions continue, but where the uranium emission becomes significant.

Figure 3 shows the shape of the emission curves as a function of time (burn-off) for five elements, representing three groups exhibiting the same behaviour, viz. Group I - boron, cadmium and tin, Group II - iron, cobalt, manganese, silicon and aluminium and Group III - calcium and magnesium. The burn-off characteristics are distributed in accordance with the volatility of the elements alone if they vaporize at the temperature of the charge, estimated at 1300 to 1400°C for 10 A and 1600°C for 12 A. These temperatures vary no more than 100 degrees from one point to another. The emission of the first group goes through a clear peak after 20 seconds, followed by the second group after 40 seconds. The third group exhibits a lower peak with very slow decay.

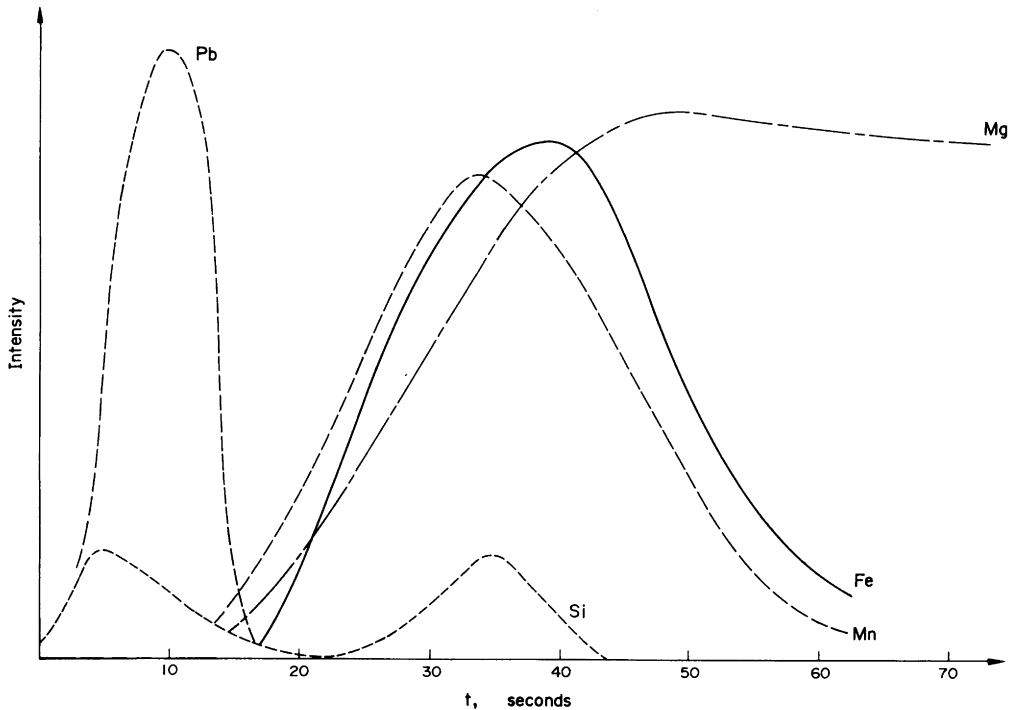


Fig. 3. Typical distillation curves.

2.1.2 Modification of the Charge. The uranium oxide U_3O_8 is converted in the first few seconds to the oxide UO_2 , which is liable to reoxidize on exposure to air at the end of arcing to give the oxide U_4O_9 . The oxide UO_2 is sintered into a solid bead. The small amount of powder present at the end of the test after 45 seconds appears to

be due to reoxidation to U_3O_8 .

If sintering is too rapid, the large amount of gas released (oxygen, gallium vapour) does not escape and the charge is ejected. This is known as 'popping'. The degree and kinetics of sintering are related to the particle size and carrier content. The gallium oxide content decreases steadily in the charge, reaching about 0.2% after an interval which varies directly with the initial carrier concentration.

2.1.3 The Role of Gallium Oxide. Gallium oxide performs a dual role. It enhances the excitation of the impurities in the plasma and it facilitates their liberation from the charge. By lowering the plasma temperature and increasing the electron pressure the gallium also limits the degree of ionization in the arc (3) and increases the residence time of elements in the plasma (4), thus promoting excitation (5, 6). This increase in the atomic population is especially high at the centre of the gas column (7). The vaporization kinetics and time intensities are only slightly affected if the gallium is added as the oxide or element, but it must be introduced at the same time as the impurities into the arc.

Once the uranium has been converted to UO_2 , the crystalline system is reorganized and certain volatile oxide impurities are liberated, while others are trapped in the new lattice. They in turn are liberated because of the formation of a liquid eutectic UO_2/Ga_2O_3 , formed in the neighbourhood of the Ga_2O_3 grains. This eutectic moves to the surface with gallium vaporization, leaving at the end a sintered oxide containing a low concentration of impurities. The effect of impurity retention by the crystal lattice has been confirmed by showing that if volatilization occurs with the oxide UO_2 (produced by the reduction of U_3O_8 with hydrogen), followed by the destruction of the lattice, the emission intensity is 8 to 10 times greater (8). Marinkovic thus confirmed the important role played by impurity inclusions in the crystal lattice of UO_2 (9). It should be noted that among oxygen-containing compounds, apart from gallium, only silica, lead chromate and indium oxide, have been used. Silica is particularly well suited for determining cadmium. Finally, to enhance the reduction of certain oxides, carbon may be mixed with the gallium (10).

2.1.4 Halide Carriers. Certain elements which form refractory oxides such as zirconium, hafnium and the rare earths, are impossible to determine when using gallium oxide as a carrier. Others such as aluminium are relatively insensitive. Consequently, very early in the development of the method this gave rise to the idea of using halide carriers which could promote the formation of volatile compounds.

In 1947 Harrison and Kent suggested the use of silver

chloride as a carrier (11). This resulted in more intense background radiation, requiring the use of a high dispersion spectrograph which delayed the extensive use of the method until the sixties. With the use of sodium fluoride however, the background is much weaker in the ultra-violet spectral region and this carrier has been used since 1952 to determine aluminium (12).

Since then many halide carriers have been employed, either pure, intermixed, or mixed with gallium oxide for the determination of certain very low level impurities (B, Al, etc.), or more frequently for a group of about 40 elements. Table 2 summarizes most of the carriers employed.

Table 2. Halide salts used as carriers

Carriers	References	Carriers	References
AgCl	15, 32, 33, 10	AgCl-SrF ₂	15, 16, 26, 30, 31, 14
NaCl	17, 18	AgCl-LiF	15, 16
RbCl	20	AgCl-AgF	8, 29
NaF	16, 13, 18, 35	AgF-NaF	27, 13
BaCl ₂	19	Ga ₂ O ₃ -SrF ₂	28, 34
NH ₄ F	21, 22, 23	Ga ₂ O ₃ -AgCl	29
PbCl ₂	24	NaCl-NaF	39
LiF	25	Sb ₂ O ₃ -BaCl ₂	37
Teflon	38		

2.1.5 The Role of the Halide Carrier. The halide compound reacts with the impurity oxides to form halides, which are generally more volatile, thus rapidly liberating the elements into the arc (30). By virtue of its metallic constituent the carrier stabilizes the discharge and enhances the spectrum.

The calculation of reaction energies ΔG at the charge temperature, around 1400°C (40), provides confirmation of the possibility of the reaction between halides and oxides (41, 42). Hence, with silver salts, fluorination is possible for all elements. The formation of the volatile chloride would in most cases be impossible if the metal chloride carrier (e.g. silver chloride) acted alone. The presence of carbon, however, which takes part in the reaction makes it possible. This also applies to the formation of bromides.

Reactions with iodine are generally impossible because the iodine decomposes at the charge temperature.

For lithium and sodium salts in the carrier charge, several different reactions may take place including the direct reactions with other element oxides. The solubility of

the oxide in the molten carrier at the charge temperature also has an effect on the evaporation properties. The formation of polyborates and aluminates, the reactivity of which with fluoride ions is different from that of the oxide (40), is possible.

Once these different compounds have been formed, their volatility must be explained. In effect, the sensitivity of an element is fairly closely linked to this volatility, e.g. nickel fluoride is formed very easily, but owing to its low vapour pressure (1400°C) it distills slowly into the arc. Consequently, the detection limit for nickel converted to the fluoride is inferior to that obtained when gallium oxide is used in the charge.

2.1.6 Choice of the Carrier. The choice of the halogen used in the carrier thus represents a compromise between the oxide-halogen chemical reactivity of the carrier and the volatility of the halide produced by the reaction. If a single element is to be determined, the carriers may be divided roughly into four groups (43, 44).

Fluoride carriers are preferable for determining the elements beryllium, titanium, zirconium, mercury, niobium, tantalum, zinc, boron, aluminium, gallium, silicon and lead, the oxides of which react well with fluorine, giving very volatile salts. Moreover, for some of these, the fluorides eliminate the possibility of carbide formation.

Chloride carriers are excellent for determining chromium, molybdenum, tungsten, manganese, iron, cobalt, nickel, copper, magnesium and possibly calcium and strontium. The results are improved if the chloride is mixed with 1 to 2% of graphite powder, which enhances chloride formation.

There is little difference between gallium chloride and oxide for the elements zinc, cadmium, indium, thallium, germanium, tin, lead and arsenic, the oxides of which are already highly volatile. This also applies to the elements ruthenium, rhodium, iridium, palladium, platinum and gold, which do not react with halides, but which are very slightly volatile.

In practice about twenty elements need to be determined. Therefore, in order to state which carrier is superior to another, the analytical requirements must be known. In most cases, the tendency is to use combined fluoride-chloride carriers, although these are not very favourable for elements, the oxides of which react rapidly with fluorine to form relatively non-volatile fluorides, e.g. cobalt, manganese, chromium and nickel. When using combined carriers, the choice of cations must be taken into account. Sodium reduces the background more than does lithium or silver, but lowers the arc ionization potential more than do these two elements (39). Among the combined carriers used is the mixture silver chloride-strontium fluoride, which is simple to prepare as the ingredients are not

hygroscopic, are free from impurities and are easy to mix. It is nevertheless necessary to carry out the grinding and mixing steps sheltered from natural light. This carrier has been adopted in the ASTM Standard Method C.696-74 (45). It consists of 16.4 mole % SrF_2 in silver chloride. A mass of 50 mg of the combined carrier is mixed with 450 mg of the U_3O_8 sample.

2.1.7 Preparation of Samples. Since analyses are concerned with samples of the oxide U_3O_8 , it is necessary to bring the uranium to this state of oxidation.

Metal: The oxide is obtained by combustion of the metal in air, in a platinum dish for turnings or in a tantalum cup for powder. It should be noted that the reaction is highly exothermic and may result in the destruction of the container. The sample is maintained at a temperature of between 600 and 1000°C for 30 to 45 minutes in a furnace in an air atmosphere, most authors specifying 900°C for 30 minutes. This is followed by manual or mechanical grinding and then mixing with the selected carrier. Because the distillation of the impurities is never complete, as it is related to the physical properties of the powder, it is important to obtain a uniform particle size distribution and constant powder density.

About 100 mg are then weighed and packed into the electrode crater. Good reproducibility is only obtained if the packing is identical for all samples, thus requiring the operator to be careful and skillful. To eliminate this problem, Heres described an automatic packing system which ensures that the pressure applied to the charge is always identical (46) (Fig. 4). Tolk preferred to make a pellet under pressure (0.6 t) and to place it in a special electrode fitted with a ventilation hole (47).

Salts and solutions: The two types of salt chiefly used in nuclear energy are uranates and fluorides, together with nitric acid solutions. With respect to the latter, simple evaporation followed by calcination suffices to produce an oxide without element loss.

With fluorides, it is important to eliminate the fluoride ion, which gives rise to heavy losses during calcination. For the salt UF_4 , this is achieved by chemical attack with slow evaporation. Various solutions have been recommended for this purpose, e.g. nitric acid-hydrogen peroxide, ammonia-hydrogen peroxide, ammonium carbonate-hydrogen peroxide (48), sulphuric acid-hydrogen peroxide (49), hydrofluoric-nitric acid (50), ammonium sulphate (51) and ammonium carboxalate (52).

For gaseous uranium hexafluoride UF_6 , the product is first hydrolysed by bubbling through water cooled to 0°C. The uranyl fluoride produced is treated as UF_4 . Note that since losses are liable to occur for boron and silica, it suffices to evaporate the solution to dryness at a temper=