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EMISSION SPECTROCHEMICAL ANALYSIS IN THE ATOMIC ENERGY INDUSTRY

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Introduction

One major research project at the Oak Ridge National Laboratory is the isolation, purification, and study of the properties of all isotopes of all elements. Considering the fact that about 320 isotopes occur naturally and some 700 others have been produced artificially, the task is monumental. And its goals are most practical—to find unique materials for new and unusual applications. Already, research, industry, and medicine have benefited enormously from the program through the use of these new "raw materials". Strong gamma emitters like cobalt-60 and cesium-137 are now regularly used to treat cancer and to take "X-ray" photographs of metal structures with portable on-the-spot equipment. Iodine-131, which is absorbed preferentially by the thyroid gland, is used both to diagnose and treat thyroid disturbances. Carbon-14, phosphorus-32, and potassium-40 are employed in the synthesis of organic compounds which, in turn, have become indispensable tools in the biological sciences. The labeled molecules can be traced as they perform their various functions.

The atomic energy industry is its own best customer for many of these elements and nuclides. Hafnium-free zirconium, a rarity in pre-atomic days, has become an important structural material in reactors. Gadolinium-157 is an excellent neutron absorber and may be used to control the rate of fission in a reactor. Man-made plutonium is the heart of the atomic bomb; uranium-235 the nuclear fuel of most reactors; polonium an alpha emitter widely used to discharge static electricity; heavy water the moderator in several reactors.

The emission spectrograph is one of the principal analytical tools used to determine the tremendous number of isotopes, elements, and combinations used in the atomic energy industry. It is used to prevent mix-ups and check conformance to specifications, especially where purity is concerned. Almost everywhere purity requirements are stringent. Structural materials must be free of neutron absorbing impurities—often in the parts-per-million range. A trace of hafnium in the zirconium fuel elements of a reactor will capture enough neutrons emit-

ted by the fission of the uranium-235 fuel to depress or halt the chain reaction. Traces of impurities in the coolant water become radioactive creating a disposal problem. The spectrograph is indispensable for the analytical work involved.

Reactor History

Very early in the atomic age, isotope separation presented a major challenge to emission spectroscopy. After the Chicago reactor proved it was possible to control fission, the necessity of isolating the uranium-235 isotope became apparent. Competing crash programs were swiftly organized, first on a laboratory scale and, when they showed promise, on a pilot plant and even production scale. Concurrently, analytical methods had to be developed to determine what separation, if any, was being achieved. Radioactive measurements proved inadequate because they were not specific: there was always the possibility that alpha-emitting substances other than uranium-235 were being separated. Mass spectrometry, while specific for elements or isotopes, could not distinguish between isobars—nuclides of identical mass. Emission spectroscopy did prove successful in differentiating uranium-235 from all other isotopic and elemental species when it was discovered that with a fairly large spectrograph the uranium-235 4244.12A and uranium-238 4244.37A lines could be resolved.

U-235 U-238
4244.122 4244.371



Spectra in the sixth order taken with a large commercial spectrograph of normal uranium; 50:50 uranium-235 and uranium-238; highly enriched uranium-235; iron. The isotope lines are clearly resolved for analysis.

Today, the spectrographic assay of uranium isotopes is commonplace. One technique is to use the 2nd or higher order of a 3-meter (or larger) spectrograph with a fairly narrow (20-micron) slit. The actual sample—which may be gaseous uranium hexafluoride, uranium metal, alloys, oxides or scrap—is converted to uranium oxide, mixed with graphite, and arced. A typical procedure employs a 1:5 oxide to graphite mixture and a sustaining arc of 8 amperes. The electrode is a narrow one (1/8" d.) with a 1/4" deep crater. Over the entire range of uranium-235 (0.7-93%), the assay may be conducted with an accuracy of 3-4%. Even this is not good enough, however, for establishing the price of uranium compounds which is based on their enrichment so primary assays are conducted by the mass spectrometer. The emission spectrograph is needed both to check on the primary assay and to make sure that, somewhere along the line, dilution has not occurred.

After a uranium-bearing ore is located, the spectrograph guides the concentration and purification of the uranium. Samples at various production stages are converted to the oxide and analyzed by the carrier distillation technique devised in the early 1940's by Scribner and Mullin. As originally developed, the method called for the powdered sample to be mixed with gallium oxide in the ratio 98:2. One hundred milligrams of this blend are burned in a 10-12 amp. dc arc for about 45 seconds in a deep crater electrode. The gallium oxide, volatilizing at a low temperature, simultaneously sweeps the impurity elements into the arc column (the refractory uranium oxide is not excited). Trace concentrations of 30-odd elements are determined in this fashion. Over the years, the principal change made has been in the carrier. Frequently, silver chloride, sodium chloride, and various fluorides are used. At Numec, J. E. Scott prefers silver chloride for boron and cadmium, recording the results on Ilford Q-2 plates. Cadmium and boron—both very strong neutron absorbers—are detected in the ppm range by this technique. (To extend the sensitivity for boron even further a copper fluoride carrier is often used.) A second burning, exposed on SA#1 plates, is made for the bulk of the elements and a third, with sodium chloride as the carrier, for silver and tungsten.

Reactor Technology

At Combustion Engineering, approximately 5000 spectrographic analyses are conducted on reactor components before a reactor is released, striking evidence of the need for the spectrograph in the atomic industry. As has already been indicated, purity requirements are exceptionally severe everywhere. It is necessary to exclude not only neutron absorbing nuclides but those which, inside the reactor, would be transmuted to neutron absorbers. Further, those isotopes which become radioactive with long half-lives are undesirable in expendable parts of the reactor such as fuel elements.

To get a better insight into the technology of a reactor, let's discuss each of its components. Although there are many different types, each contains:

- a neutron source, fuel, a moderator, means for heat transfer, means for controlling the fission rate, shielding, and structural components.

The function of the neutron source is to provide, at the time of start-up of the reactor, the neutrons which act as atomic bullets and start the fission of the uranium-235 fuel. When "criticality" is reached, i.e., the point where enough neutrons are being produced by the fission reaction to sustain itself, the source may be withdrawn. In a sub-critical reactor, the extra supply of neutrons furnished by the source is required to sustain the fission. One convenient source is a can of radioactive polonium mixed with beryllium powder. The former, a pow-

erful alpha emitter, knocks out neutrons from the beryllium nuclei.

The fuel elements ordinarily contain uranium alloys or compounds at various isotopic enrichments of uranium-235. Because of the hot, corrosive environment in which they operate, the fissionable fuel is usually encased in one of several materials. Pure aluminum is one choice although, depending on the application, stainless steel and Zircaloy-2 (an alloy containing zirconium and tin), are also used. Experimental homogeneous reactors have been built in which the fuel is in the form of a circulating uranium salt solution. At Brookhaven, a novel approach in its Liquid Metal Fuel Reactor Experiment is the use of a solution of enriched uranium in molten bismuth.

In reactors used for production of power, several moderator materials are being used or are under test. Heavy water, beryllium or its oxide, graphite, and organic liquids are typical. Coolants may be heavy water, organic liquids, helium, nitrogen, carbon dioxide, molten sodium or lithium. The latter two are particularly attractive because of their high thermal conductivity but are notoriously corrosive. Periodic spectrographic examination of the metal is made to determine if corrosion is taking place and, if so, to what degree. There is a constant search for alloys which will withstand the coolants and for coolants which will not attack the existing alloys. Niobium metal, niobium-zirconium, and nickel-molybdenum alloys are a few of the weird ones encountered by the spectrographer in the atomic industry.

Frequently in nuclear reactors, the same material serves both as the moderator and the coolant. In low power reactors, where water is used, it also serves as a shield. Thus, the swimming pool reactor, as its name implies, uses a deep pool of de-ionized water. As a moderator the water serves to slow down neutrons (slow or "thermal" neutrons are required to sustain fission). As a coolant, the water is recirculated after it passes through a heat exchanger. The 17-foot thickness of water supplements the shielding effect of the concrete walls of the pool.

When water is used as a moderator its purity must be maintained and checked—again, a spectrographic job. As elsewhere in reactor components, impurities become radioactive. Accordingly, the concrete walls are painted to prevent leaching and the water is constantly treated by ion exchange resins to remove impurities. Radiation has a tendency to dissociate water and this may be inhibited by the addition of copper ions.

Of the many materials in use for controlling the fission rate in a reactor, cadmium and boron-steel are most common. Gadolinium, having a tremendous cross section of about 40,000 barns has certain specialized uses. It is interesting to note that the various isotopes of cadmium and gadolinium vary greatly in their capture cross section. Thus, gadolinium-157 has a value of 160,000 barns compared to 0.4 barns for gadolinium-160. Similarly, the cadmium-113 isotope, comprising but 12% of the natural metal, does most of the absorption of neutrons being converted to cadmium-114 in the process. Proof of the latter was obtained by Dempster (A. J. Dempster, *Phys. Rev.*, 71, 829, 1947) using mass spectrographic techniques. After exposure to slow neutrons, the surface of the cadmium was compared with the interior using a mass spectrometer. The 113-mass line disappeared altogether from the surface while the 114-mass line was enhanced. Whether similar experiments have been performed using the emission spectrograph is not known. Hafnium is another important control rod material in military reactors. It occurs naturally with zirconium and has chemical properties very similar to zirconium. Because of its scarcity and high cost, its use in commercial atomic reactors is limited.

Fission fragments, the products of the split uranium-235 atom, have a disturbing effect on the control problem. As they build up they act like control rods themselves, slowing down the reaction continuously. To offset their influence, "a burnable poison", such as boron-10, is often used. This is initially a high cross-section material which, upon irradiation, is transmuted to low cross-section nuclides of lithium and helium. The trick is to add just enough boron to the fuel to compensate for the increasing build-up of other poisons, another important application of the emission spectrograph.

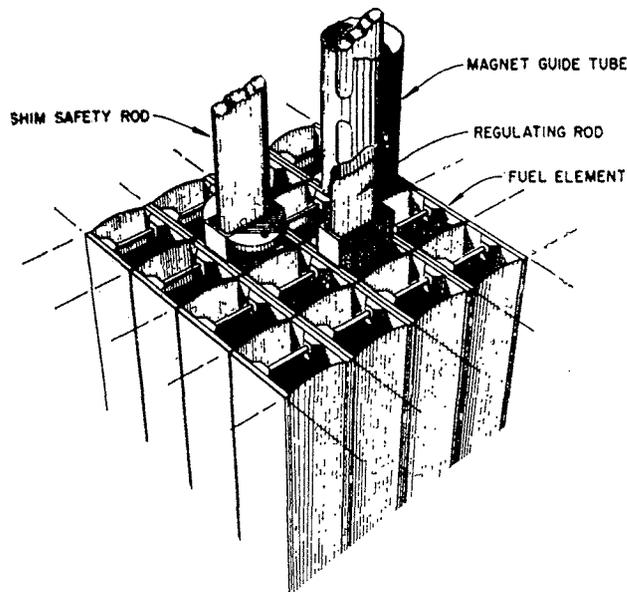
Shielding personnel from radiation is achieved with concrete in most land-based reactors. Alpha and beta radiation, with little penetrating power, is absorbed in a few centimeters of most materials. High energy gamma and neutron radiation require thick layers of heavy materials. Lead may be used but is expensive and a poor structural material. Concrete, a far better structural material, has the added advantage of being able to slow down neutrons which are then more easily captured. Special "heavy" concretes containing aggregates such as barium sulfate and iron ore are used in eight-foot thicknesses for such purposes.

Some reactors are designed mainly for research, a typical one being the Bulk Shielding Reactor (Swimming Pool) at Oak Ridge. Designed for low energy levels, its operating temperature is low and aluminum is used to clad the fuel rods which consist of a 93% enriched uranium-aluminum alloy. In addition to protecting the fuel from corrosion by the surrounding water, the aluminum case retains the fission products preventing their escape into the water. In this type of reactor, water serves the multiple purpose of moderator, coolant, and shield. In a newer, experimental Oak Ridge Organic Moderated Reactor, diphenyl is the moderator-coolant.

Although aluminum's low neutron capture cross sections make it desirable as a structural part of a reactor core, its use is limited by its weakness and its inability to withstand the corrosive environment and high temperature of a high energy reactor. Stainless steel, though more wasteful of neutrons, is often specified because of its strength and corrosion resistance. Still another choice is Zircaloy-2, which is exceptionally corrosion resistant, a weak neutron absorber, and capable of withstanding high temperatures.

Because of its high temperature properties, Zircaloy-2 is preferred in many power reactors. The permissible level for each of the trace elements depends on its neutron cross section or its effect on corrosion resistance and mechanical properties. These specifications for Zircaloy-2 lend themselves beautifully to spectrographic analysis (see Table 1). The

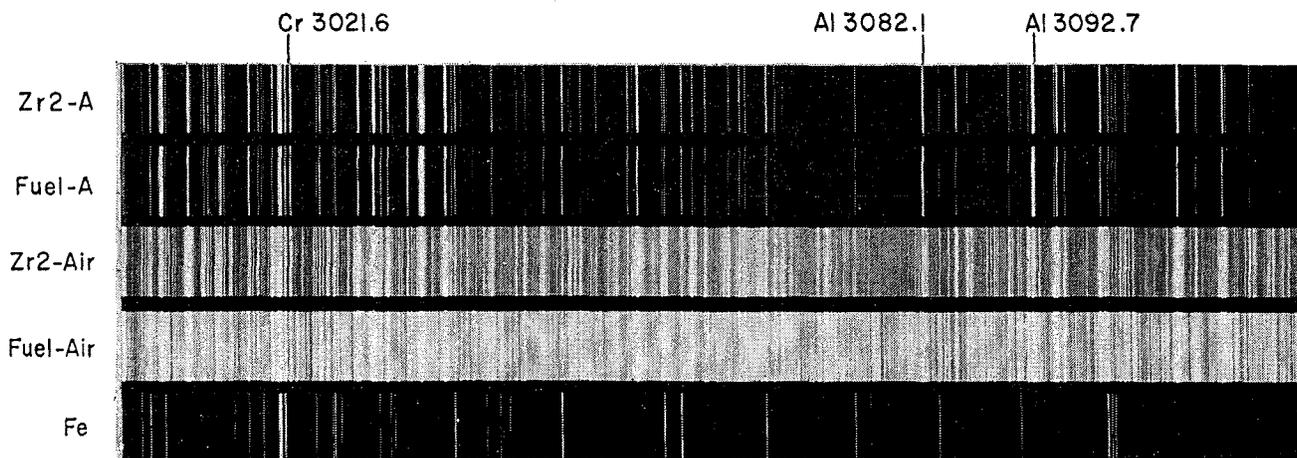
starting point of a typical method for trace analysis is to convert the alloy to the oxide by heating chips or turnings in a muffle. At Combustion Engineering it is mixed 9:1 with sodium fluoride, a 25 mg sample being excited in a 12 amp dc arc. The arcing is done in an argon atmosphere making use of a stainless steel shell which surrounds the lower, undercut electrode. With this technique, Co, Al, Ti, B, Mn, Si, Mg, Cu, and Mo are detected down to 10 ppm or less. An argon flow of 7 CFH is used. Where standards are available, a point-to-plane



Assembly inside the Bulk Shielding Reactor. Adjustment of the shim safety rod, consisting of boron carbide powder in a lead-weighted aluminum can, controls the reactor rate. The stainless steel regulator rod provides a fine adjustment.

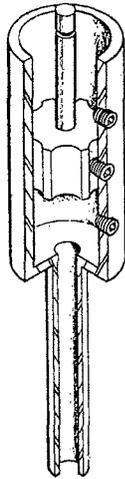
TABLE I
Specifications for Zircaloy-2

Constituents	Maximum Impurities			
	Al	Mg	Mn	U
Sn	1.20-1.70%	75 ppm	20 ppm	
Fe	.07-.200%	0.5 ppm	50 ppm	
Cr	.05-.15%	0.5 ppm	80 ppm	
Ni	.03-.08%	270 ppm	120 ppm	
Zr	Remainder	20 ppm	20 ppm	
		50 ppm	50 ppm	
		200 ppm	100 ppm	
		25 ppm	3.5 ppm	
		130 ppm	0.025 ppm	



Illustrating the effects of an argon atmosphere on cleaning up the spectra of Zircaloy and a Zircaloy-uranium fuel.

method may be used for most impurities. The naval reactors built at Combustion are typical of those making extensive use of Zircaloy-2.



Combustion Engineering's device for bathing Zircaloy samples in argon.

Precautions must be taken with all marine reactors to make sure they "fail safe" in the event of destruction, for widespread contamination of the water would result if the fuel elements or radioactive waste products became exposed. To ward off such a disaster the fuel elements are fabricated from an alloy of enriched uranium and Zircaloy-2 clad in Zircaloy-2. Such a reactor would safely settle to the ocean floor.

Possible destruction is also an important consideration in the Army's Package Power Reactor (APPR) designed for the production of electricity in remote areas. Its fuel elements consist of enriched uranium dioxide sintered in a stainless steel matrix and clad in a stainless steel jacket. If destroyed, rain water could not leach out the fuel and so contamination would be kept local.

The stainless steel specified in the APPR is type 348, niobium stabilized 18-8. It differs from the more conventional 347 in that low tantalum niobium is employed. Ordinarily, the specifications for 347 call for a combined concentration of niobium plus tantalum, the two metals being difficult to separate. Because tantalum has a high neutron capture cross section, however, it is restricted to 0.1% in type 348. At this concentration a direct point-to-plane spark spectrographic determination may be used. When the value is lower, it is necessary to separate the niobium-tantalum from the stainless steel chemically, convert to the mixed oxides, and arc the resulting powder.

The application of the spectrograph to stainless steel identification is a "natural". Spectrographic tests are essential because of the wide variety of such steels, their incompatibility, and the fact that the different types appear identical so can be mixed up quite readily. Type 347 or 348 resists corrosion in reactors, may be welded with the same material, and has unusually good fatigue strength. If a sheet of unstabilized stainless steel were welded in inadvertently or if a single wrong welding rod were used, it would immediately be the focal point of a severe corrosion attack and, in time, a dangerous leak might develop. The spectrographer is constantly on the alert to warn of such impending trouble. During assembly, every batch of steel—sheet, welding rod, structural members, etc.—is tested for conformance to 348 specifications. After

start-up of the reactor, the water coolant is examined periodically to determine what is being picked up. If a build-up of a particular element occurs, it may spell corrosion. The spectrograph is thus an essential member of the preventive-maintenance team.

Niobium, the stabilizer in 348 stainless, promises to be a useful structural material in its own right. The same general problems of purity apply to niobium and, like zirconium, a great many elements must be kept in the ppm range. At the Wah Chang Smelting and Refining Co., a principal supplier of the metal, 20 elements are sought ranging in limits from about 1 to 1000 ppm. John Gillespie of Wah Chang prepares samples by converting them to the oxide and mixing them 1:1 with a carrier. The latter, in turn, consists of 1:1 strontium chloride and graphite. Samples are burned in a 15 amp dc arc using undercut electrodes and in a single burning all of the elements with the exception of tungsten are determined. For tungsten, as in the work at Numec on uranium oxides, sodium chloride is the preferred carrier. The niobium oxide is mixed in the ratio 30:70 with sodium chloride for the tungsten determination.

The main difference in the procedures adopted by others for niobium analysis is in the carrier. L. S. Brooks of duPont uses lanthanum oxide in a 1:1 ratio with the sample oxide. This carrier has the advantage of having a fair number of spectral lines for internal standardization at different wave lengths. In the duPont procedure, 20-odd elements are determined using a 20-amp dc arc. At Pratt & Whitney, CANEL Operations, D. E. Fornwalt prefers a 1:3 lithium carbonate graphite mixture as the carrier which is mixed in a 1:1 ratio with the sample oxide. A 20-amp dc arc is again used but with a Stallwood jet to stabilize the arc. Mr. Fornwalt has stated that the addition of the Stallwood jet has permitted the complete analysis of each sample in a single burning. Triplicate burnings had previously been required in an open arc both to obtain the desired precision and to obtain suitable registry of lines of all of the elements.

The spectrographer is often called on to analyze a very tiny sample—perhaps a milligram or two of a corrosion deposit or scrapings from the needle on a valve. So prevalent are these tiny samples that in the laboratory of J. E. Norris at Oak Ridge, most methods have been developed for 1 mg samples. A comprehensive analysis covering major as well as trace elements on a 1 mg sample of Zircaloy-2 aluminum or stainless steel is routine. The sample in the form of a dried salt is weighed using a torsion microbalance then mixed with 9 milligrams of graphite powder. Analyses are conducted on both prism and grating spectrographs.

Another important reason for working with tiny samples at Oak Ridge is the program mentioned in the beginning of this article, that of isolating and purifying each of the nuclides. In this research effort, samples are necessarily minute and may be the result of many tedious hours of preparation. The first step in the isolation process is to place the sample in a calutron, i.e., a mass spectrometer designed to separate rather than analyze samples. In the calutron the sample is vaporized, ionized, and by means of a strong magnetic field, isobars are separated. The individual ions impinge on a graphite collector plate from which they are later removed with appropriate solvents. In this fashion, microgram quantities of a sample may be collected to be identified by the spectrographer. Since the sample will have a known mass number but may consist of one or more elements, the spectrographer's task is at first qualitative: to tell the chemist what elements are present so he can devise means for separating them chemically. Later,

as the chemist attempts separation techniques, the spectrographer must refine his method to a more quantitative one. Particular guidance is required here because the chemist may be working with elements never before separated. Called for are ingenuity and skill rather than an analytical cook book.

In addition to working with minute samples, the spectrographer may require higher resolution than normal in order to identify individual isotopes. A 21-foot focal length Paschen-mount "walk-in" spectrograph-spectrometer, one of the most versatile instruments in the world, was built for this purpose at Oak Ridge. Anywhere along the optical track ordinary 4"x10" plates may be inserted. Alternatively, photomultipliers may be positioned to record both lines and backgrounds. Background corrections are made electronically for both the unknown and internal standard lines so the instrument records true intensity ratios. Accurate trace-element determinations as well as determinations of major elements in very small samples are thus simplified. On this instrument a notable piece of work was reported in 1955 by J. R. McNally and associates (*J.O.S.A.* 45, 202, G. K. Werner, D. D. Smith, S. J. Owenshine, O. B. Rudolph, and J. R. McNally) determining lithium-6/lithium-7 ratios. Using a 30,000 lpi grating, they were able to apply the instrument to two lines in the 6707A doublet where the isotope shift amounted to but 0.16Å. A hollow cathode source was used in order to obtain fine lines, and assays were conducted over a range of 0.2 to 98.3% lithium-6 with an average deviation of but 1.3%.

Another example of the way high resolution is almost taken for granted at Oak Ridge is the determination of cesium-137 using a grating flame photometer built at Oak Ridge National Laboratory. According to Cyrus Feldman, it was developed to check the concentration of "dead" (non-radioactive) cesium in samples. The instrument is now used routinely and specifically for the determination of total cesium in cesium-137 preparations because of the growing application of this isotope in radio-therapy and radiography.

Health and Safety

Throughout this article, the stress on personnel protection is evident. The body, which over eons has built up protective devices against heat and cold, uncounted bacteria, viruses, and poisons, has simply not learned how to cope with a new danger—radiation. Accordingly, quite small doses can be harmful. To prevent the spread of radioactive materials, wastes must be disposed of safely and over the long periods associated with the half-lives of many isotopes.

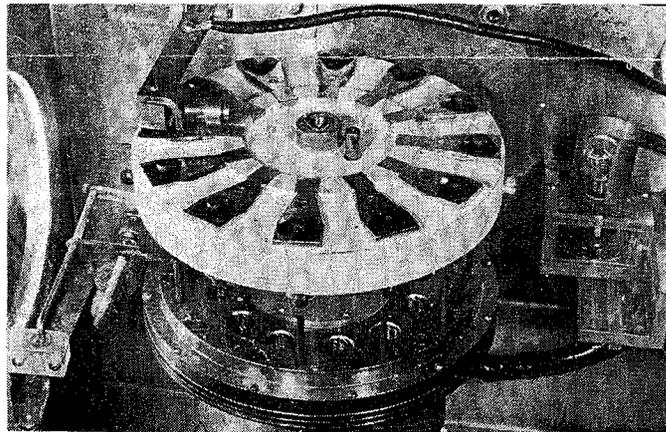
One obvious precaution is to have two separate sewage systems, one for normal waste and the other for radioactive products. This is done at Oak Ridge and elsewhere. Another is to separate the radioactive materials into fractions having comparable half-lives. After "cooling", the short-lived components can be disposed of anywhere without danger. Meanwhile, the bulk of the longer-lived components is reduced simplifying their disposal correspondingly.

Before such separations can be accomplished, it is necessary to analyze the waste material and so ascertain how much of what elements and isotopes are present. Used for cooling low-power experimental reactors, air is one such waste product. It contains carbon, nitrogen, oxygen, and argon which, upon irradiation, emit gamma rays and neutrons. Fortunately, all of these have very short half-lives, argon-41 having the longest (110 minutes) and disposal of the air from a high chimney is quite safe. (When weather conditions indicate the possibility of down drafts, automatic monitoring stations in the vicinity of the reactor give warnings.) There is the possibility too of

dust in the air becoming radioactive so, before discharge, the air is cleaned with filters and electrostatic precipitators.

Water used for cooling presents not much more of a problem than air. After a sufficient "cooling off" period, it is discharged at a controlled rate into large rivers. As emphasized earlier, however, the cooling water must be freed of impurities beforehand. The disposal of liquid wastes from spent fuel elements creates a much more serious problem on which considerable research is being done. Especially where isotopes have half-lives of long duration—many years in some instances—it is necessary to store the waste effluents to assure safety for long periods. In addition to the analysis of the wastes to guide separation into fractions of equivalent half-lives, the spectrograph is used in studies to establish such factors as the leaching rate of materials in which it is proposed to store the wastes. Among many techniques, bonding the wastes as ceramics or storing them in special concrete have been tried but are costly. Another approach is to bury the wastes deep in abandoned mines and tests are being conducted along these lines, too. The spectrograph is valuable in all of these long-range experiments in identifying traces of escaped isotopes.

Despite the precautions taken in atomic technology, some radioactive materials have escaped into the soils and atmospheres of the earth, mostly through nuclear weapons testing. Here spectrographic research is being conducted to find answers to many questions. Where do nuclides such as strontium-90 deposit in the body and to what extent? What foods have the largest concentration of radioactive isotopes? Can strontium-90 be "fixed" in the soil preventing its uptake by plants and, therefore, animals? Will the trace-element balance of the body be changed by small increases in radioactivity?



Brookhaven National Laboratory's arc stand for handling radioactive samples. Twelve electrodes are loaded and burned successively into arcing position inside a sealed chamber.

Acknowledgments

It took a lot of cooperation from many individuals to assemble the information for this article. We hope that in thanking the following we have not omitted anyone. If we have, thanks to you, too. Harold Mullin, AEC, New Brunswick, N. J., Joseph Forrest, Brookhaven National Laboratories, Donald Fornwalt, CANEL Operations, Middletown, Conn., Allen Powell, Richard Gale, Combustion Engineering Co., Windsor, Conn., LeRoy Brooks, DuPont, Wilmington, Del., Daniel Turissini, General Chemical, Morristown, N. J., Louis Owen, Goodyear Atomic, Portsmouth, Ohio, Charles Pepper, National Lead Co., Cincinnati, Ohio, James Scott, Numec, Apollo, Pa., Cyrus Feldman, Rand McNally, John Norris, Union Carbide Nuclear Co., Oak Ridge, Tenn., Isabel Tipton, University of Tennessee, Knoxville, Tenn., John Gillespie, Wah Chang, Glen Cove, N. Y.

TURNTABLE SAMPLE HOLDER FOR X-RAY SPECTROSCOPY

A factor influencing the precision of x-ray spectroscopic analysis is orientation of the sample with respect to the optics. Not only are there effects due to the crystallinity, segregation, and grain structure of the material being analyzed but, in surfacing it with a sander or lathe, additional orientation changes are introduced. The result is that there is a strong dependence of the x-ray intensity on the rotational position of the sample.

One obvious solution is to rotate the sample as it is irradiated and, as a matter of fact, provisions for doing this have been made in several of the newer x-ray spectrometers. Based on a design by K. H. Storcks, T. C. Loomis and C. R. Geith of The Bell Telephone Laboratories, the Spex Turntable Sample Holder is made to fit only the General Electric XRD-3 and XRD-5 instruments in which it serves as an exact replacement for the original sample drawer. To use the Turntable, simply plug it into a 115 vac main.

In operation, the sample, which may be as much as 5/16" thick, is placed on the turntable by swiveling it to the open position. On the other side of the "see-saw" is the motor which serves to load the front face of the sample against a ball-

bearing race. This constrains the axis of rotation of the sample to one perpendicular to its front surface. A rubber connector between the drive shaft and the ball race compensates for any off-parallelism of the back surface of the sample.

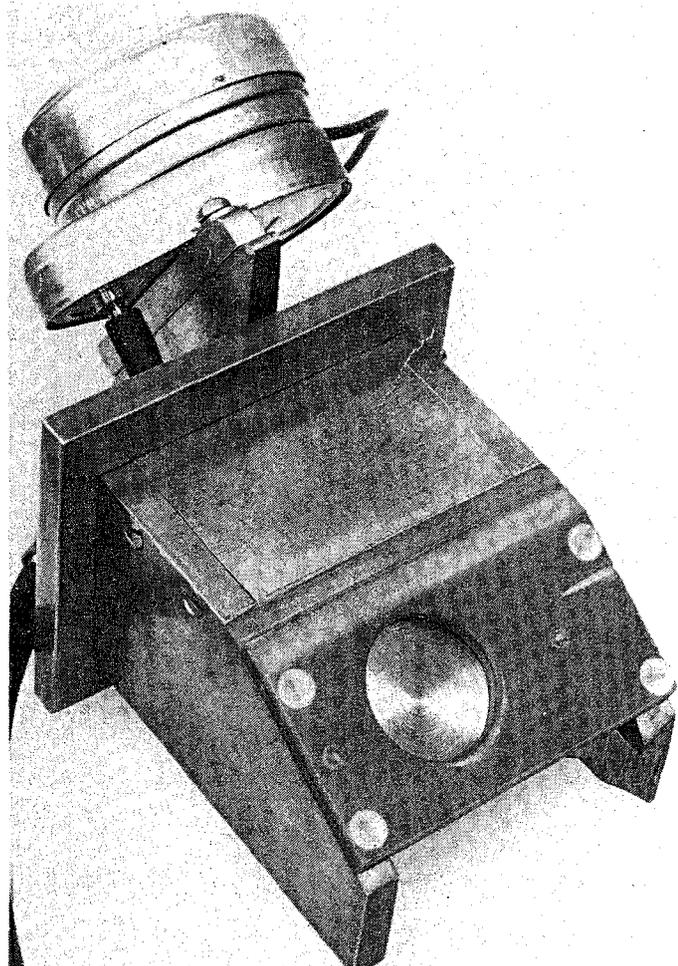
For powder specimens, a separate removable plastic holder is provided. It can be loaded in the usual fashion and then pinned in position.

To demonstrate the usefulness of the Turntable, three high-constituent alloys were analyzed 10 times each by: 1) rotating the specimen; 2) having the specimen remain stationary; 3) "jogging" the specimen, i.e., having it remain stationary but rotating it at random between determinations.

Summarized in the table below, results indicate that there is a real difference between the counts obtained when the sample is stationary as compared with when it is either rotated or jogged. But while the rotating and jogged samples gave about the same number of counts, the precision of the rotating specimen was substantially better. Both pieces of evidence are indicative of orientation and/or segregation effects which are minimized when the sample is rotated.

Alloy	Element Determined	Average Counts			% Average Deviation		
		Rotating	Stationary	Jog	Rotating	Stationary	Jog
46 Ni, 44 Fe	Fe	24,724	23,742	24,734	.19	.16	.40
46 Ni, 44 Fe	Ni	21,196	21,016	21,181	.17	.10	.31
Nisiloy (55 Ni, 8 Fe, 26 Si)	Fe	17,631	18,984	17,839	.20	.19	.42
7.8 Cr, 24 Cu, Rem. Ni	Cr	7,992	7,958	7,993	.55	.20	.53

3520 Turntable Sample Holder for X-ray Spectroscopy, fits General Electric XRD-3 and XRD-5 instruments. As described in text complete with 30 rpm synchronous motor (115 vac) and separate holder for powder specimens **\$200.00**



CARBON STEEL SPECTROGRAPHIC STANDARDS

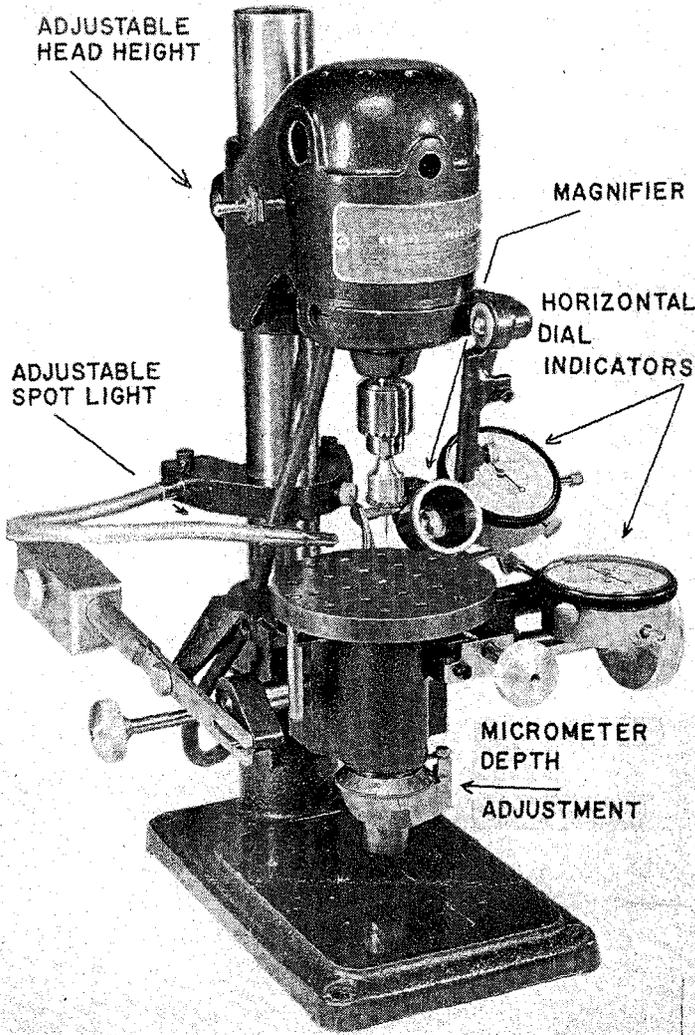
A new set of carbon steel standard discs has just been announced by the British Bureau of Analysed Samples. Standardized for carbon, silicon, sulfur, phosphorus, manganese and arsenic, the discs will be welcomed in routine laboratories as well as those where occasional steels must be analyzed. Composition is certified as follows:

SS No.	C %	Si %	S %	P %	Mn %	As %
31	0.47-	0.55-	0.029	0.043	0.36	0.11-
32	0.071	0.063	0.010	0.019	1.14	0.003
33	0.63-	0.26-	0.089	0.067	0.62	0.070
34	0.15-	0.36-	0.012	0.004	1.47	0.001
35	0.26-	0.54-	0.054	0.036	0.72	0.024

BSS31-35 Carbon Steel Standards, set of 5, 1/2" thick x 1-3/4" dia. **Per Set of 5 \$80.00**

BXS31-35 Carbon Steel Standards, set of 5, 1/2" thick x 1-3/8" dia. (These are cut down for use with Philips x-ray spectrometers). **Per set of 5 80.00**

MICRO-DRILL



Metallurgists are increasingly concerned with micro-segregation in alloys, especially those under development. Information on the composition of the segregates is vital in obtaining superior alloys. The most elegant way of studying the microstructure of materials is with the electron probe—in principle, a scaled down X-ray spectrometer. The high cost of such a unit, however, usually precludes its use. Another method of study is to traverse the specimen with a narrow spark and examine the light with an emission spectrograph. But the most direct and simplest technique is to drill out the segregate, maneuver it into a tiny graphite electrode and examine its arc spectrum. Bryan and Neveu (*Metal Progress*, Dec. 1953) described such a technique in which the sample, polished and etched as for metallographic examination, is used. After the segregate is drilled, the chips are collected with a drop of collodion and placed on top of a flat, high-purity graphite rod which is then arced at low current for a few seconds. Bryan and Neveu claim that with this technique any chip that is visible will produce the spectrum of its major elements. Drillings may be made with a drill as small as 0.002" dia. to a depth of as little as 0.003".

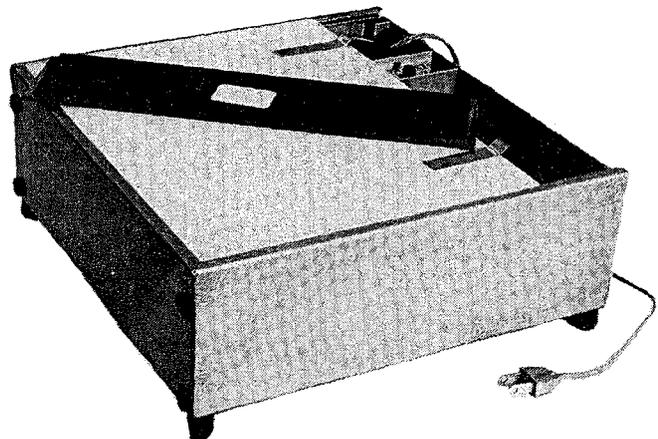
The Dumore Micro-Drill is ideal for the purpose. As pictured, it has a number of convenience features for locating and drilling holes. After a sample is clamped to the base, it is positioned by turning two knobs at right angles to each other. A 10x magnifier, gooseneck light, and calibrated dial indicators aid in locating the exact spot to be drilled. Speed is controlled through an auto-transformer up to 17,000 rpm. Depth of drilling is controlled by means of a micrometer advance of the specimen table. This feature is especially handy for spectrochemical analysis enabling comparison of two or more specimens of the same mass. Simplicity of design plus adequate instructions covering the instrument permit the spectrographer to drill holes in most materials after just a few minutes of practice.

7300 Micro-Drill, complete with 115v, 1/16 hp, 50-60 cy., ac motor; speed control box with dial settings up to 17,000 rpm; two-way table travel with dial indicators; 10x Hastings triplet magnifier; goose-neck spot light; micrometer depth control, magnetic chuck kit for holding drills down to 0.001" dia. . . . Each \$625.00

7301 Drills set of 12 in assorted sizes 3.50

GLOW-BOX

Here's a viewing box that should be evaluated in terms of its versatility and usefulness rather than its low cost. The 11" x 9" viewing surface, of 1/4" thick Plexiglas, is illuminated with a high-intensity, rapid-start, cool, circular fluorescent lamp. The overall dimensions of the box (11-3/4" x 11-3/4" x 4" high) permit it to be stored and used in a desk drawer where it is instantly available yet out of the way when not wanted. Retractable legs permit the box to be flush-mounted or tilted for better viewing. The dimensions of the viewing surface are sufficient for examining two 4" x 10" plates or an 8-1/2" x 11" film. Other suggested uses for the Glow-Box are: 1) illuminating samples as they are being titrated; 2) color comparison; 3) tracing; 4) comparing graphs on different sheets of paper.



3710 Glow-Box, viewing box as described above; weight 7 lb., complete with 22-watt, circular fluorescent lamp, 115 vac, carrying handle; finished in baked hammertone gray enamel. Each \$34.50

30-TON HYDRAULIC PRESS

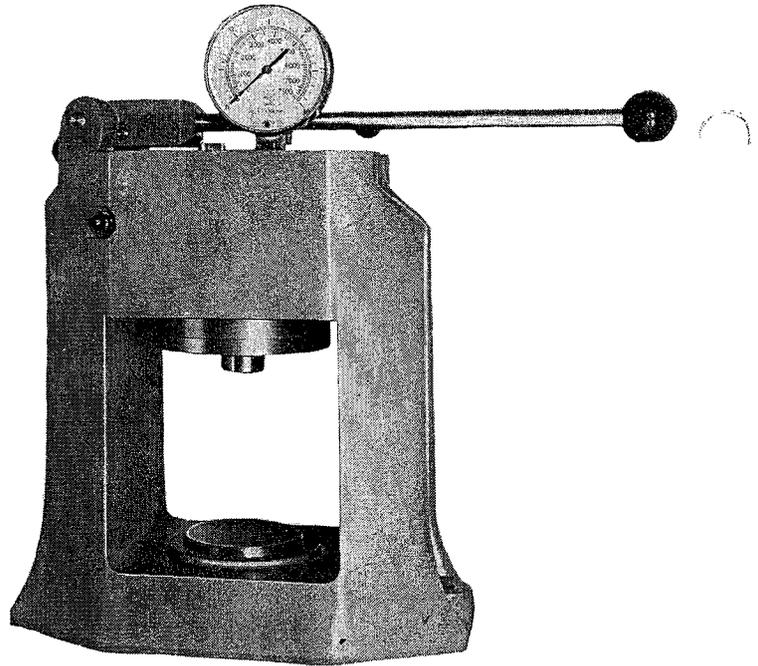
The Research & Industrial Instruments hydraulic press has been designed for pressing a wide range of pellets, tablets or discs used throughout industry. With a total capacity of 67,000 pounds, the press uses but a fraction of its strength for preparing 1/2" dia. KBr discs. Its total capacity is also sufficient to apply a pressure of 40,000 p.s.i. on a 1-1/4" diameter die—the maximum suggested for preparing X-ray pellets. (See "Some Practical Aspects of X-ray Spectrography", M. C. Lambert, *The Norelco Reporter*, Vol. VI, No. 2, 1959, p. 47.)

Built as a virtually maintenance-free machine, the press is cast in one piece from high-strength, non-deforming Meehanite. Sanded smooth, the casting is finished in grey hammertone stoving enamel while all the exposed machine parts have a polished chrome-plate surface or are in stainless steel. A component vulnerable in many presses is the high-pressure oil seal which, in this instrument, actually consists of six high-duty gaskets, one behind the next. The failure of all of them would be necessary for an oil leak to occur.

When used for repetitive work, the die is centered on the stationary platen with the help of a number of concentric rings. The main valve is then closed by turning a handle through 180° and the pressure brought up by pumping the long handle. When the required pressure is reached, the valve is opened momentarily causing the upper moving platen to spring back just far enough to remove the die. This reduces the pumping time for subsequent samples.

Specifications

Pressure	Maximum—30 tons (67,200 pounds)
Platen Stroke	Maximum—1 inch
Vertical Opening	Maximum—5-3/8 inches



Dimensions	15" wide x 9" deep x 16" high
Weight	Net—210 pounds Crated—265 pounds

RH30-1	30-ton Hydraulic Press as described, complete with hydraulic oil	\$525.00
3621	1/2" diameter die, non-evacuatable	138.00
3622	1-1/4" diameter die, non-evacuatable	210.00

SPEX

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