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SIXTH

**HOT LABORATORIES
AND EQUIPMENT
CONFERENCE**

**International Amphitheatre
Chicago, Illinois**

MARCH 19-21, 1958

**UNITED STATES ATOMIC ENERGY COMMISSION
Technical Information Service Extension, Oak Ridge, Tenn.**

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Frank Ring, Jr., Compiler

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**OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee**

Introduction

Panel members representing extensive Hot Laboratory operating experience in several types of laboratories throughout the country were assembled in an attempt to bring out the valuable comments to questions concerning design and operations initiated from the floor by people interested in this field of activity. The panel members are identified as follows:

- R. C. Westphal, Panel Chairman. Supervisory Engineer in charge of the Hot Laboratory Operations, Bettis Plant, Westinghouse Electric Corporation
- E. S. Schwartz. Supervisor of the Hot Cell Facility of the Solid State Division, Oak Ridge National Laboratory
- D. D. LaRocque. Supervisor of the Radiation Materials Laboratory of the Knolls Atomic Power Laboratory, General Electric Company
- W. B. Doe. Remote Control Engineering Division, Argonne National Laboratory
- L. D. Turner. Supervisor, Radiometallurgy Laboratory of the Hanford Laboratories, General Electric Company
- G. J. Deily. Hot Laboratory Operations of the Research and Development Division, Savannah River Laboratory, E. I. du Pont de Nemours & Co.
- R. R. Fouse. Supervisory Engineer, Hot Laboratory Operations, Bettis Plant, Westinghouse Electric Corporation

Contents

Introduction	11
Minutes of Round Table Discussion Presented at the Sixth Hot Laboratories and Equipment Conference in Chicago	1
A Corrosion Test Facility for Irradiated Samples A. L. Maharam	23
The Design of the Idaho Expanded Core Facility C. E. Langlois	27
The Hot Laboratory Facility at Knolls Atomic Power Laboratory. D. D. LaRocque	45
Hot Laboratory Problems in Isolating Gram Quantities of Transplutonium Elements. C. H. Youngquist and P. R. Fields	49
Hot Laboratory Operations at Argonne National Laboratory. William B. Doe	62
Operational Aspects of Hot Laboratories G. J. Deily	65
Hot Cell Operations, Solid State Division, Oak Ridge National Laboratory E. S. Schwartz	69
New Multi-Cell Facility in Idaho D. C. Durrill and R. D. Dwigans	73
Density Measurement Problems in Hot Cells K. Stratton	81
Bettis Hot Laboratory Operations R. R. Fouse	96
Some Safety Recommendations Pertinent to Hot Laboratories L. G. Stang, Jr.	100
Hot Laboratory Panel Discussion at the Sixth Hot Laboratories and Equipment Conference—1958 Nuclear Congress, March 16–21, 1958 L. D. Turner	113
A Conducting Plastic for Metallographic Specimen Mounts D. C. Durrill	117

Modifications to Master Slaves at Bettis Hot Laboratory	123
E. H. Stearns	
A Continuously Recording Micrometer-Profilometer for Hot Laboratory Applications	130
K. Stratton	
Remote Metallographic Equipment and Practices	135
F. M. Cain and F. O. Bingman	
Coloration of Shielding Window Glasses	155
K. R. Ferguson and R. L. Reed	
Shielding Window Design for the EBR-II Process Building	160
K. R. Ferguson and L. M. Safranski	

Minutes of Round Table Discussion Presented at the Sixth Hot Laboratories and Equipment Conference in Chicago

Mr. Westphal

There is a question from the floor.

Mr. Ed Slater - Picker X-Ray

I want to ask Mr. Dowling about this 500 cfm which you felt is one half of what you would like. What was the volume of the cell? In other words what was the ratio between the air flow and the length of the sides of the cell?

Mr. Dowling

Well, I think that these are fairly typical figures. Our cells are approximately 6 x 12 in floor area and exhausting 500 cu/ft/min from this volume means that you have a gentle draft or breeze downward through the cells. It is by no means a capture velocity.

Mr. Harry Nelson - Atomic Energy of Canada

I am primarily interested in operating a chemical facility and I would like to ask Mr. Fouse who I believe operates the chemical facility in their hot laboratories whether he knows of a blower that will stand up to nitric acid. I am thinking of something in the order of 2 or 3 cfm.

Mr. Fouse

I do not know of any blower, at least not by experience, that will stand up under nitric acid. I think that enough equipment is being made with the new plastics that you could probably find one.

Mr. Walter Shuler - Frankfurt, Germany

I wonder if you could give a few comments on the relative merits or disadvantages of the various types of doors—sliding doors, hinge doors, vertically split or horizontally split doors, etc.

Mr. Westphal

There certainly are a wide variety of such types of access doors into cells. Usually, the decision is based on a compromise between cost and utility or convenience. George, I believe you have the guillotine type.

Mr. Deily

Yes, we do and we also investigated all the different kinds of doors in the design phase of our new facility so I have pretty definite opinions about this. The choice depends on the individual's circumstances. The cheapest door is probably a plug type door which could be made of concrete cast in a steel box and mounted on rails. This presents some problems in the loading area or the back side of the caves. You have rails in the floor and when you have the door open you have this big thing sitting in your way. The bank vault type may be easy to operate but when it swings open it uses up some of your valuable floor space behind the cell and generally gets in your way. We use the horizontally by-parting doors. The advantage is that they are inside the wall and out of the way and in our opinion offsets the higher cost. You can operate them easily. We have had no maintenance problems but I would say these are by no means a panacea. There is only one

other type of door that I can think of and that is one mounted on an overhead rail. These are pretty good doors, in my opinion, but the only trouble is that if you have a row of cells you have no place to roll them all.

Mr. Westphal

Mr. Doe here has some rolling doors. Let's see what he has to say about them.

Mr. Doe

Well, we have the plug type that rolls straight back out of the cell on overhead rails and the by-parting doors that roll horizontally with overhead rails also. We prefer the overhead rails because they are relatively free of contamination problems. Rails in the floor would interfere with the free rolling of equipment, pots, and casks into the cells. We have almost completed a cave with two cells in a row where the doors are all on one rail; you can open either one of these cells completely by pushing the doors on the other cell along but never opening it. Perhaps this method could be used where there are more than two cells in a row.

Mr. Westphal

Mr. LaRocque, you have an unusual type of door in your cell.

Mr. LaRocque

Yes, our two older cells have the horizontal moving doors. In the newer cells, the doors go down into the floor. There are four doors for the two cells. They are somewhat more expensive than the other types of doors so far mentioned but they do eliminate the space problem and also the hazard of a guillotine type door which is aptly named.

Mr. Schwartz

We have two types of doors. On our small master-slave cells we use

the plug type which roll back on rails. These, as mentioned before are the cheapest from the standpoint of money, however, they are expensive from the standpoint of space and then, of course, there is also the problem of rolling equipment across a floor that has rails in it. On our larger cells we have hinge doors. One disadvantage with this has been that the access to the cell is above the floor level of the loading area. Heavy equipment is put into the cell by removing one of the roof slabs.

Mr. Turner

My comment is that we don't have any doors as such. We have six demountable sections. Our entire cell can be dismantled down to a flat table top. Our cells are opened whenever major maintenance is required or about once every year or two years depending upon the change in program. The end sections of the cell are just a T-slot where the wall will nest in and form a part of the shielding and we just pull it up, move it over and put it on a rack and that's it. It seems to boil down to buy the best door that you can afford. This is not always clear cut because it is a compromise on first, cost, capital equipment vs maybe building costs that have to be added to the cell area and finally operating costs of your facility.

Mr. Steele - Livermore

At Livermore we have had very good success with our bank type doors, and it cost us \$235/ton which I think is pretty cheap. We were very careful on the prints--not to state that these were safe-type doors because you immediately pay a premium for this. The door is just supported on a simple thrust bearing at the top and a roller bearing at the bottom. We are also cursed with the guillotine type which we had to buy when money wasn't so available. We actuate these by picking them up with the service

crane over the back cell service aisle. Of course, the crane is always busy when you want to open the door but for the swinging doors we have had no trouble at all.

Mr. Westphal

Of course, in your area, if I remember, you had a large building structure to start out with and building costs for working space didn't enter into the problem.

Mr. Steele - Livermore

That's correct.

Mr. Fouse

All of our cells have guillotine type doors and the new cells also have guillotine type barriers. By partially opening the door a small aperture is provided for transferring equipment just at the work table height. It has cut down on our transfer time, and it has saved us a lot of burn up. Of course, they are expensive, but we have had very little trouble with them.

Mr. Steele - Livermore

The question I raise about doors that go into the floor is that you are building up contamination in spite of the fact that you might have a wiping seal against the door. Sometime someone has to go down there and decontaminate.

Mr. Blomgren - Argonne

Would the panel discuss the relative merits between sodium and mercury lighting and also if there is any tie in between the glass and zinc bromide windows and lighting?

Mr. Westphal

That's a big order. Let's take up the first part. I think we can get an answer to that rather quickly. The question is: What type of

lighting is preferable vapor, mercury, fluorescent, or what have you.

George Deily.

Mr. Deily

I think I can sum this up very briefly. Both types are satisfactory. I would say that the sodium light has a harsher light and some people might find it disagreeable but this can be gotten used to with a few weeks of training. For a given amount of light intensity within the cells, I suspect there is more heat output with its concurrent effect on the ventilation. If you require a high optical resolution then the monochromatic sodium light is probably required to provide this. If you do not have to worry about this, then the mercury light is a little better, that is your color transmittance is a little truer and you can distinguish different colors within the cells which is a great help in normal manipulation. We have some of each and we have gone to the mercury vapor lights in our laboratory. Its advantages are lower heat emission, better color rendition and the smaller power consumption.

Mr. Schwartz

I can say very briefly that we had been using sodium lights in our cell, and we are converting to mercury vapor lights mainly for the following reasons: One, greater illumination and the second, the mercury lights are so much easier to replace when they burn out.

Mr. Westphal

In general, the life of the mercury lights is better and they are less subject to intermittent operation.

Mr. Schwartz

That's true. Also the sodium lights usually take from 20 to 30 minutes to warm up and sometimes this may be a factor.

Mr. Westphal

In general, sodium vapor lights are becoming more difficult to obtain from a supplier—they are going out of general use and this is a consideration for the future certainly.

Mr. Doe

I agree that the mercury lights are cheaper and take less space, but I don't agree with George that the sodium lights are harsher. I think that mercury lights, particularly when you have yellow glass in the window, give a sickly green color. I prefer the looks of the sodium but I don't think this is important. To me the real important factor here is when working very close to the inside of the window—say within a foot or two, the mercury light does not give you as good a vision as the sodium light does because the mercury is not nearly as monochromatic. You get color fringing due to the dispersion of the window. I think that the sodium lights, if you can afford them, are worth the extra expense.

Mr. Westphal

The second phase of the question was concerned with the use of glass vs bromide windows? Correct? Let's start at the other end this time. Lou.

Mr. Turner

Well, I have to pass this question, since we only use 6.2 g/cc density lead glass for viewing.

Mr. Westphal

Mr. LaRocque uses both types.

Mr. LaRocque

We use a combination of zinc bromide and lead glass. In our newer cells, we have two slabs of four inch thick lead glass and approximately 27 inches of zinc bromide. There has been a paper presented by Bob Stearns

on the difficulties we have encountered with one window becoming cloudy out of a total of seventeen. This was primarily due to the breakdown of the system 31 paint which was not properly applied by a general contractor. The one cell where we have just the solid slab of lead glass is only a nine-inch thick steel cell. As far as the economics at the time we purchased these, which was in 1953-54, the combination of the zinc bromide and lead glass was the cheapest and as I stated we have had only one go bad on us in about a little over five years time. Also I would like to add this concerning the discussion of sodium vs mercury lighting, we just went ahead and used regular fluorescent lights and we are satisfied. We know that when looking at the edge of something you occasionally get some color but it does not bother us very much. I think there is probably too much concern put on these color fringes.

Mr. Stratton - Westinghouse Bettis

I agree with Don LaRocque's point of view here from the standpoint of one that has to spend a large part of the day staring through windows—I have heard about these color fringes, etc. but I have never seen them. We just converted from sodiums to mercury. There is a kind of—as Mr. Doe points out—an icky green but this really turns out to be rather restful in a sense. I had only one other comment that I would like to make in regard to the lighting proposition. We have found that it is extremely valuable to think a little bit about where we put the lights inside the cell. We are now going to placement of lights down very low—down to your table tops of slightly above it.

Mr. Glen - ORNL

In our new cells we use mercury fluorescents and also the choice between zinc bromide and lead glass is based on the type of material we

are going to use to make the shielding walls out of. If we have space enough to use a cheap lime stone concrete we use zinc bromide. If we are kind of tight for space we use baryties or some other kind of high density concrete and lead glass.

Mr. Westphal

I think those comments concerning the type of shielding windows were very much to the point.

Mr. Boudreaux - Savannah River

We have both sodium vapor and mercury vapor lamps in the cells but for the past two years in the operation of the chemistry facility we have used nothing but daylight fluorescent tubes and we found that they have been quite satisfactory. The main thing that we liked about them is the fact that we were able to determine colors to a very fine degree. We have used both front lighting in a chemistry box and a diffused light through the back side. It is a little hard on the eyes at times but very good for determining just how much liquid you have in a vessel after the thing has become so brown that you can't see into it any more.

Mr. Doe

I think that there is one other thing that should be considered here. We have measured the resolution that you achieve using the two types of lights. Some say "maybe we can't see as well but we don't think we are losing very much." With a 3 foot thick zinc bromide window and the test chart a few feet in front of the window using mercury lights you lose about 20 per cent of your resolution. White lights were not even considered because they were so bad. Now why worry about this? I think in many cases people build cave walls thin by using expensive high density material so that they can see as well as possible. You have paid a lot of money to get a

high density wall and window to get up close but now if you use white light you throw this advantage away.

Mr. Westphal

One thing on lighting---there is a rather complicated physiological effect on the human being aside from the various types of illumination. It is just as important to have the level of illumination controlled and the proper orientation of the light source for good vision.

Mr. Zabowski - Wright Air Development Center

I would like to get the panel's opinion about television in this whole business of vision.

Mr. Westphal

Is there any one here on the panel that has a television installation? LaRocque?

Mr. LaRocque

The only application that we have made of the closed circuit television at KAPL has been on the Tukon micro-hardness tester where we used a camera to bring the optical path out of the cell in the cheapest way we could. We actually operate the Tukon micro-hardness tester faster with the T.V. system than without it.

Mr. Turner

I might make one comment here Don that we do use one in each of our reactors for looking into areas that are some distance away but yours is the only application that I know of in hot labs. My personal feeling is that anything you put between you---the shielding is bad enough to start with---and the actual operation is another maintenance chore.

Mr. Westphal

Well, we have spent a good deal of time discussing lighting and windows. There's a new question.

Mr. Simon - Argonne

In view of the remarks on cost of construction--what are the possibilities of going underground with caves and what are the opinions of the panel on other cost reduction means?

Mr. Westphal

In some areas excavation is as expensive as building above ground level depending on the rock structure underneath. In other cases, it is fairly easy to excavate. Is there any one present who knows of a facility where underground shielding is used for operation? It is used very commonly for such things as sample storage and the like where no manipulation of samples is involved.

Mr. Davis - Atomics International

Our present hot cells are underground located beneath the reactor building and connect through the reactor floor by way of plugs so that a fuel element can be lowered into the cells. One end of one cell and half of the back of the same cell are essentially earth shielded with ordinary concrete walls. We don't think that we saved very much money in this because one still had to get around one's hot cell, maneuver around it, see into it and usually work on one side and load and unload on the other side. We had to blast a hole in rock and this is quite a job, so it isn't a fair cost comparison. But, in general, I would say that unless you have a rather unique cell loading problem, such as we had, there would be little point in putting the cell underground.

Mr. Westphal

The point is that it is rather hard to work around a hole in the ground.

Mr. Sullivan - Vallecitos

Are we open for general questioning?

Mr. Westphal

Yes, we are.

Mr. Sullivan - Vallecitos

Several years back we had some interesting figures on cell utilization. Now, this can be defined as the amount of time a cell does useful work which in turn may be defined as gathering data on machining operations, etc. I would like, perhaps, for each of the members of the panel to comment on what these figures are now that we have progressed to a certain degree in our technology.

Mr. Westphal

It looks like you are under pressure to get some work out too. This was one of the general subjects that we had hoped to get to in this afternoon's discussion and haven't reached this point and time is getting late.

Mr. Deily

This is basically an economic question. I wonder how many of us in our technical labs, our chemical labs, etc. keep track of how many hours the facility is being used and how many hours it sits vacant. I doubt whether the utilization of an ordinary research lab exceeds 10 or 20 per cent but caves cost much more money, therefore, we try to make as much use out of them as we can. Our experience at SRL has been that for a cave that is used for miscellaneous experiments, that is you are putting things in and taking them out frequently, we have obtained utilization figures of approximately in the order of 50 per cent. We think that with sufficient engineering, sufficient manpower, sufficient scheduling, we could push this up to between 70 and 80 per cent, but we don't think we could go up much higher. However, if you have an experiment that runs more or less continuously,

that is, you put the equipment in and leave it there until it wears out or falls apart, and you have enough work to keep it busy, you probably could get between 80 and 90 per cent utilization. We have done this with our chemistry facility on occasions.

Mr. Westphal

That would parallel the experience at Bettis, I am quite sure.

Mr. Fouse

I would like to add one thing. It isn't actually how long a cell is being used, but how much work is being done in a cell that really counts. Suppose you tie up a cell and work in it for 24 hours on sort of a slip-shod basis. It would be better to take a little more time in setting up and get the same amount of work done in 8 hours. So really the amount of time that a cell is in use is a poor measure of the efficiency of a hot lab.

Mr. LaRocque

Too often in hot lab operations people use these terms--cell utilization, useful cell hour, etc. I think that at the present state of the art, and I still say art, I think that we are doing about as well as can be expected with the changing nature of the work since hot labs first started say five or six years ago. The kinds of things that we were doing, as Lou Turner pointed out, was skimming the cream. Now, they want more kinds of things done to the experiment. That might be summed up by saying less volume but a greater variety of things being done. When we speak of cell utilization, we are trying to measure what is fundamentally a research and development operation by production standards and by no stretch of the imagination could this be appropriate. I think that at the present time we are not wise enough to define what effective cell utilization is and if we do agree to an arbitrary definition, I say that we are not yet wise enough to know how to measure it.

Mr. Vank

I would like to comment on this question. We have kept records for the past two years on our one cell which is used for a terrifically large variety of applications ranging from metallurgical, some chemical and just about anything the scientists dream up. We have averaged 2-1/2 months of active operation for every quarter. The rest of the time is spent in decontamination, changing equipment or making a few minor repairs.

Mr. Westphal

Mr. Doe has some remarks to make.

Mr. Doe

I think there is more to this than just the cell efficiency. You should really consider the overall operation of the laboratory, particularly the efficiency with which you use the manpower. If you have enough manpower, you can put in the effort to carefully protect every single piece of equipment. When you are cleaning the cells you can send in a large crew and tear out the lining in the cell or whatever--in other words use a lot of manpower to minimize the down time, but you are not using your manpower very efficiently because they will be idle while the experiment is in progress. I think there must be a balance somewhere between the amount of time that your cell is utilized and the amount of time that your manpower is being used.

Mr. Westphal

I think that it goes even one step further than that. What's the object? If you can set up equipment that can handle 50 items one day and be off the next day changing equipment as required, but with another method you can do only 10 items a day on a continuous basis, then you are far better off by the first method which averages 25 items per day, although by some

standards this is only 50 per cent cell utilization. So the problem is very complicated. There isn't a clear cut answer to every situation.

Mr. Turner

Ray, can I make a few points? I brought some data along expecting a question like this. For calendar year 1957, we had an average of 23-1/2 employees in our organization--with that we got 14,400 manhours of laboratory examination time. This figures out a little under 30 per cent. The remaining time was spent in building operation, tours, clean up, equipment design and maintenance, training new employees in absences, and 6000 manhours in supervising and clerical work. Don't forget the clerical end of our business. Accountability for one thing. For our total we spend \$348,900. This boils down to actual laboratory time of \$24.20 per laboratory hour.

Mr. Schwartz

I would like to add that our experience at ORNL has given similar data.

Mr. Westphal

Another question from the floor?

Mr. Gordon - Aero-Jet

In looking at costs from a private concern's viewpoint, how long do you feel a cell should last or when do you have to finally bulldoze it under?

Mr. Westphal

It would depend largely on the type of operation involved. As long as you have a means of decontaminating it to be used for other functions or continuing functions. Basically, it should last indefinitely.

Mr. Fouse

Probably one thing that will obsolete a cell as fast as anything is the development of new equipment, because it is not adaptable to your old

cells. In our west cell block for instance we had to modify it to take master-slaves. Eventually we are going to come up with equipment that we can't modify that cell block for. Then the cell block will probably eventually become obsolete. Actually, now the cell has been running for seven years and the cell block is operating as well as ever.

Mr. Westohal

The matter of obsolescence with new and better ways of doing things is probably far more important and even here it is a matter of economics in the end whether you modify the existing structure to accommodate this or whether you just give up and fill it up with concrete and start over again. Looking over past experience there are cells now approaching ten year life that are still functioning. Some have been modified to accommodate the increased knowledge in the art.

Mr. LaRocque

I think basically we are paying somewhat of a premium now to provide flexibility in design of these various hot labs so they don't become obsolete. For example, at KAPL if we had not provided in the original design the flexibility of these large removable square plugs and various holes that we could pull out and replace with direct view windows and slaves, the first cell blocks would have been obsolete completely. If you provide flexibility, I don't see any reason why the cells couldn't last for 25 or 30 years. I doubt very much, based on talking to the various hot lab people, that there has ever been a contaminated cell that has not been cleaned up regardless of how hot it was.

Mr. Turner

I would like to add one quick word. Ray, actually we find the engineers today are very unsatisfied in waiting three months to look at a

piece. Instead of waiting the normal decay period they want to look at it tomorrow and so we are getting hotter and hotter material all the time. I don't think you can over design a cell. I think that some of the fellows that I talked to today on the floor have the same experience. You always swear that they have over designed it and you'll never get to that level. You always exceed it. If you have it you'll have experiments to fit into it.

Mr. Westphal

Are there any further questions from the floor?

Not Identified

I have a question relating to lighting and windows. To what extent do you screen your operator for visual sharpness due to the variability in the human eye? Can you improve yourself a lot by being real selective in your choice of operators or do you have any selection?

Mr. Westphal

I know that at Bettis the technicians are screened only by the standard medical inspection that takes place for any other employee. They have a certain standard of vision or it has to be corrected by use of lenses. I don't know about the rest of the people on the panel. Mr. Schwartz?

Mr. Schwartz

At ORNL we follow in the same standard as they do at Westinghouse. The only screening that is done is by the Laboratory Health Division on their medical examinations for all incoming employees. Incidentally, this does not include a test in depth perception.

Mr. Doe

At Argonne we require that they have stereo vision. I have heard that not over 90 percent of the people have true stereo vision. If this is true—one out of ten can't be used very well as a cave operator. I also

think they should be screened for at least normal if not better than normal acuity because we do think that vision is important. However, we don't do this--we take the usual Laboratory screening for normal acuity but we do require the stereo vision.

Mr. Westphal

I think these points are desirable--whether you can include them in your own employment program may be another thing. Mr. Turner?

Mr. Turner

Our primary requirements are the intelligence and the mechanical ability. Of course, we don't want any one blind either. I think that outside the normal examination that most companies give new employees we look for mechanical ability and intelligence first in our engineering assistant caliber people.

Not Identified

When a fellow comes into Vallecitos, during the interview period we actually hand him a pair of slaves and put a little bottle and cork inside just to see if he can coordinate his hands by putting this cork anywhere near the bottle. A very brief screening will knock out perhaps 80 per cent. We have been very successful this way. Some people just can't even come close. They do not know what to do with their hands or their eyes.

Mr. Deily

I'll endorse Mr. Turner's remarks. The first thing you want in a man is the will to work. After that if a man would have a physical defect that would disqualify him this would become obvious very soon and then you could transfer him to some suitable work. I think that visual acuity is one of the lesser or secondary characteristics that you wouldn't consider in the light of the primary ones unless it were a very serious one and was

disqualifying completely. I'm speaking now of men who have ambition and intelligence. An able worker can overcome some of the visual defects that you would encounter and still be a very valuable man to the group.

Mr. LaRocque

I think that the agreement is that people in hot lab operation should be given some sort of special tests but how stringent these tests will be depends upon the need and demand for these people. If you need just one or two people you can be quite stringent. If you need a lot of people you will be much less selective.

Mr. Westphal

It is important to have the best class of help that you can get in an operation like this. There are some particular personal characteristics and traits that are valuable over and above what is required for normal mechanical operations or laboratory operations and these have to do with a certain degree of patience, that is they do not allow themselves to be easily frustrated.

Mr. Stratton - Westinghouse Bettis

I would like to ask the panel if there is any one here who knows of a situation where there is an Industrial Hygiene or Health Physics Group that is an integral part of the hot laboratory organization and is directly responsible to the hot lab supervisor and if there is not, if they have any comments on the desirability of such a setup?

Mr. LaRocque

At KAPL they are separate and I think that it should be this way because even though we require our technicians to be skilled on the use of radiation detection instruments, contamination control, etc. we still need someone who is not being pressed to get the work out. You need an

audit—you need someone who has a good understanding of the operations and yet is not concerned whether this hardness test gets done today, tomorrow or the next week. I feel he should be under separate management so that effectively the hot lab management would not be wearing two hats. There aren't too many people that can wear two hats very effectively.

Mr. Westphal

Does any one have a different situation?

Mr. Stratton - Westinghouse Bettis

I would like to say that I wear two hats. It has worked quite satisfactorily. It is my personal philosophy that if you have a separation you get in the situation where the health group can say: I don't think you ought to do that and the technician will say fine and as soon as the health man turns his back the technician goes ahead regardless and walks out of the hot cell spreading contamination for fifty feet. If you have a man that has the authority to say you cannot do that and he is backed up by management, well then I maintain that you have more operating time because you have less spills. We have found from experience that by having more or less semi-police control over the operation—we get more work done because we have less down time.

Mr. Westphal

From my own knowledge it is rather unusual to have both activities under the same management for the location. Maybe under the next level of management but generally not on the same level. Any other comments along this line?

Mr. Fouse

Isn't there enough work for one man to do the job and enough for

the second man to watch the radiation? I fail to see why it would be necessary just for a police protection to have a second man watching over your shoulder. We don't do it when it comes to other items of safety. We have a safety engineer perhaps but we rely on the individual's own integrity not to cut off his finger on the machines for example. This is just the same sort of thing in my opinion. Does any one else have an opinion on this?

Mr. Westphal

Often times radiation control is a little less obvious than when you stick your finger under a stamping press. That is why it takes more forethought and more individual thinking on part of another person perhaps to have to follow up the man who is actually performing the job.

Mr. Cunniff - Livermore

The question was asked if there was a situation where people concerned with safety, report to one person and the answer is yes—they do it at Livermore, Pratt and Whitney. But I think that if you didn't know you couldn't tell the difference between them and any other health physics group.

Mr. Schwartz

I would like to say that I agree with Don. I am more in favor of the, shall we say, disinterested or objective observer. The way that we operate at Oak Ridge is with the separate Health Physics Division although supervision of our group is still responsible for the safety of its employees. The Health Division does not have any real authority per se, only the power of recommendation.

Mr. LaRocque

This is pretty much the setup at KAPL but the Health Physics peo-

ple are permanently assigned to the hot lab. However, the responsibility still remains with the technician for the control of his own radiation time. We don't request the Health Physics people to run an audit on how much radiation the man has received from day to day. They do provide us with a monthly report on the film badge and dosimeters that the people wear in the hot lab. The Health Physics people, even though they don't have responsibility or authority of position, they certainly must have the authority of knowledge. They should know more about Health Physics aspects of the particular work than the hot lab operators.

Mr. Cunniff - Livermore

To substantiate our work philosophy out there, the contamination control group is basically a health and safety group but they go one step further in that they are more on an engineering level. The usual health and safety group it seems to me comes in and says that you need guards around this machine and you need safety glasses here. What we try to do is educate the health safety man to become a back door engineer and he looks on it as an engineering problem but he also has the health and safety aspects of it so that it is an integrated part in that he tries to stop some of the problems before they get in. You might say that we have a round table discussion and then his worry is the health and safety aspect of it.

Mr. Westphal

The time is approaching 5:30. The interest shown here indicates that this sort of a program is worthwhile. However, we certainly haven't milked the subject dry, but we must now adjourn.

A Corrosion Test Facility for Irradiated Samples

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Abstract

An important characteristic of nuclear reactor elements is their corrosion resistance at elevated temperatures. This paper describes a test facility, located at the Bettis Plant Hot Laboratory, in which corrosion tests are performed on radioactive materials. The design of the equipment is such that a minimum use of hot cell time is required. It consists of individually shielded, portable autoclaves and associated apparatus. The facility has proved to be effective and requires little maintenance.

Introduction

One of the requirements of materials used in nuclear reactors is good resistance to corrosion at elevated temperature and pressure. The corrosion resistance of these materials is evaluated at the Bettis Hot Laboratory by weighing a sample before and after extended immersion at reactor temperature and pressure in an autoclave.

Corrosion resistance tests at the Westinghouse Hot Laboratory were previously conducted in autoclaves located in a high level cell. While this setup furnished valuable information, the operating efficiency was quite low and a backlog of samples accumulated. One of the major disadvantages of this setup was the large stresses placed on electrical manipulators when used to tighten the autoclave head bolts, which resulted in breakdown of the manipulator and resultant delay in testing. Another disadvantage was the inability to repair one autoclave until the adjoining autoclaves were shut down and their radioactive samples removed. It was also difficult to detect or repair leaks in the autoclave piping.

Description of Equipment

The new corrosion resistance test facility now operating at the Bettis Hot Laboratory has been designed to overcome the difficulties of the "in-cell" facility. A photograph of the autoclave enclosure with two of the cubicle doors open is shown in Figure 1. (Negative No. 21377) The complete installation consists of six autoclaves, a control panel, and instrument panel, an autoclave

enclosure, an exhaust system, a sample box, and make-up pump, interconnecting piping and gages. The autoclaves are rated at 650° F and 2500 PSIG and have a capacity of one gallon. Except for bottom piping connections and a reduction in

Fig. 1—Autoclave cabinet.

the number of head bolts from twelve to eight, the pressure vessel is of standard construction. A steel collar clamped to the autoclave, is keyed to the shield to prevent rotation of the pressure vessel when tightening the head bolts. Strip heaters are strapped to the sides of the vessel and an 1/2" thick insulation can is welded to the steel collar to reduce the heat flux to the shield. Gas bleed-off and liquid drain lines are socket welded to the bottom of the pressure vessel and thermocouple leads are located in a tube which extends five inches up into the pressure vessel.

The pressure vessel is shielded by a six inch thick cylinder of lead with detachable top and bottom shields. All the lead is completely jacketed by one-half inch steel plate to prevent loss of shielding by melting in the event of overheating. The piping is passed through tubes in the shield at a "non-streaming angle" and connected to valves mounted on the outside of the shield. The heater power and thermocouple leads are routed through separate curved passages through the shield and terminated at Amphenol receptacles. The autoclave may, therefore, be disconnected from its associated equipment in the cubicle; i.e. pressure gage, pressure cut-out switch, rupture disc, and drain manifold to permit transfer to a high level cell. Six 1/8" diameter passages near the bottom of the side shield and in the top shield allow natural convection cooling of the lead shield.

The autoclave cabinet is made of 1/4" plate, suitably reinforced with channels. Its function is to support the autoclaves and piping and to prevent the spread of air-borne contamination from the autoclaves in the event of leakage. The cabinet is maintained at approximately one inch of water negative pressure by a 1000 CFM fan which exhausts through a filter to the atmosphere.

All the piping in the cabinet is Autoclave Engineering Co. high pressure tubing. The liquid drain lines from the six pressure vessels join together in a manifold and discharge into a surge tank which is protected by a 600 PSIG relief valve. Similarly, the gas bleed-off lines discharge into the surge tank. The gas bleed-off lines also are connected to an overpressure switch and a pressure gage. A rupture disc with a manual-by-pass valve is connected directly to the gas bleed-off valve.

One of the shielded autoclaves is modified to permit the extraction of a water sample from the pressure vessel while a test is in progress. The liquid drain line has been routed through a heat exchanger to a glove box. The operator discards sufficient water to clear the piping and then collects a quantity sufficient for analysis. Pre-heated, degassed water is then pumped back through the liquid drain line to the pressure vessel. A differential pressure gage is connected between the liquid drain and the gas bleed-off lines to indicate the water level inside the pressure vessel. The water sampling system provides an indication when the sample has failed. Therefore, a sample need not be tested to completion of the scheduled run, but may be removed to release an autoclave for other testing.

Operation

Loading, unloading and viewing of samples is accomplished by disconnecting three piping connections and two amphenol connectors at the autoclave, then lifting the autoclave (pressure vessel and shield) out of the cubicle with a fork truck and placing them on a transport dolly. (See Figure 2) The technician loosens the head

Fig 2—View of operator loading autoclave into its cubicle

bolts with lead filled wrenches, built into the shield lid, and pushes the dolly into the cell where electric manipulators are used to remove the shield lid and the head of the pressure vessel. The specimen may then be removed for examination. Reversal of the above procedure returns the loaded autoclave to its cubicle and a new test may begin. Time of utilization of a cell is approximately one hour and slight personnel radiation exposure is received. When samples of low activity are to be examined, the autoclave is placed in an

air lock adjacent to the cell door. After the shield lid and pressure vessel head are removed, the sample is removed with tongs and placed in the cell for examination. Cell utilization is further reduced by this method.

Summary

The present corrosion resistance testing facility utilizing the shielded pressure vessels and other innovations provides the following advantages:

1. Direct manual tightening of the head bolts to insure proper sealing.
2. Breakage and excessive wear of the electric manipulators due to autoclave loading and unloading has been eliminated.
3. Utilization of cell space and interference with other cell work has been minimized.
4. Autoclave loading and unloading time has been decreased.
5. Small leaks from the autoclave and its associated piping are readily detected.
6. Inspection and maintenance of the autoclaves is facilitated.

The present system increases the testing capacity, and reduces the personnel exposure encountered in the radioactive materials corrosion resistance study program at the Bettis Hot Laboratory.

The Design of the Idaho Expanded Core Facility

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It has been recognized for some time that there is much to be learned from a core after it has been taken out of operation. A plant is under construction at NRF, Idaho which will be equipped to cut up, and analyze cores as well as to prepare the fuel-bearing material for recovering of fuel by a chemical processing plant. This article gives a brief description of the special features of design of that facility and the reasons for them.

INTRODUCTION

The Expanded Core Facility (ECF), the first full-scale facility of its kind, is presently under construction at the Idaho Naval Reactor Facility of the National Reactor Test Station, Arco, Idaho. Bettis Plant is responsible for the design and is also responsible for the construction and ultimate operation of the facility for the Atomic Energy Commission. Construction is scheduled for completion about April 15, 1958 and operations are expected to commence May 1, 1958. This article briefly describes the facility, highlighting its special design features.

The physical plant of ECF is essentially a large building about 342 ft x 193 ft encompassing, among other features, 245 sq ft of water pits 20 to 30 ft deep, megacurie hot cells of various sizes, radiochemistry laboratories, a decontamination facility, a "hot" shop, and other supporting services (see Figs. 1, 2, and 3). Inside this building, expended reactor cores will be received by rail in heavily shielded containers. They will be unloaded from their containers, inspected and dismantled, underwater. Sample specimens will be taken from them for physical, chemical, radiochemical, and metallurgical testing in the hot cells and laboratories. The remaining fuel-bearing material, relieved of excess structural material, will be loaded into shielded containers and shipped to a chemical processing plant for reclamation of useable fuel.

1. RECEIVING AND SHIPPING AREA
2. HOT CELL LOADING AREA
3. DECONTAMINATION ROOM
4. WATER PITS
5. HOT CELLS
6. TRANSFER TUNNEL

7. HOT CELL OPERATING GALLERY
8. RADIOCHEMISTRY LABORATORIES
9. INDUSTRIAL HYGIENE OFFICE
10. CLEAN MAINTENANCE SHOPS
11. 20 TON CRANES
12. 125 TON CRANE

13. COUNTING ROOM
14. ASSEMBLY AND LAYDOWN AREA
15. DARK ROOM
16. LUNCH ROOM
17. OFFICES

1 1/2" CONDUIT DOWN TO REMOTE (□) PROBE LOCATED 15 FT FROM CATION EXCHANGER TANK EL. 23'-6" CONDUIT FROM THIS PROBE TO REMOTE (□) PROBE LOCATED 15 FT FROM MICRON FILTER EL. 14'-6"

2. 1" CONDUIT TO REMOTE (□) PROBE LOCATED IN EQUIPMENT ROOM NO. 1 AT ELEVATION 70' 17" ABOVE THE HOT CELL ELEVATOR
3. REMOTE (□) PROBE ON HOT CELL WALL 6'-0" ABOVE FLOOR

Fig. 1—First floor plan, ECF physical plant.

LOCATION

Early in 1955, the need for a facility to process the expended reactor cores of power-producing type reactors was recognized, and Bettis Plant was commissioned to design, build, and operate such a facility. The develop-

1. SHIPPING AND RECEIVING AREA	15. PIPE TRENCH
2. PASSAGE NO. 2	16. SAW PIT
3. PASSAGE NO. 3	17. TRANSFER CANAL
4. STORAGE	18. STORAGE PIT
5. INSTRUMENT MAINTENANCE	19. TUNNEL
6. 20-TON CRANE	20. CONTAMINATED TOOLS
7. 125-TON CRANE	21. OPERATING GALLERY
8. SWITCHGEAR	22. EQUIPMENT ROOM NO. 1
9. CONTAMINATED SHOP	23. MEZZANINE
10. MAINTENANCE SHOPS	24. HOT CELLS
11. ASSEMBLY AND MONITORING	25. HOT CELL LOADING
12. CHLORINATOR ROOM	26. HOT CORRIDOR
13. COOLING TOWER	27. MASS SPECTROMETER LAB.
14. WATER PITS	

Fig. 2—Traverse sections, ECF physical plant.

mental nature of present cores and cores of the near future dictated that the facility have as its mission the procurement of information from expended cores, as well as the routine preparation of expended fuel-bearing material for a chemical reprocessing plant.

Fig. 3—Expendable core facility process flow chart.

The Idaho Naval Reactor Facility was selected as the site for the Expanded Core Facility. In addition to being readily available, this site has the advantages of possessing administrative facilities and utilities provided for the Naval Reactor Facility as well as proximity to a chemical processing plant and ready access to a newly constructed railroad spur.

The location within the NRF places the facility downwind and downstream (groundwater) of existing facilities.

Its precise location within the NRF Site was determined after a limited number of core borings were taken to determine the location and depth of subsurface rock. Previous geological work in connection with the development of the site indicated the probable presence of a subsurface valley in the lava beds, and a boring plan was designed to verify this. Excavations for the 30-ft deep pits did not disclose any appreciable amounts of rock.

PROCESS FLOW AND GENERAL PLAN

The designers of ECF were fortunate in having the benefit of the experience of the Naval Reactor Facility and Bettis Plant in the examination, dismantling, and sampling of one prototype reactor core. This experience, together with advice and experiences gathered from other AEC installations such as Idaho Chemical Processing Plant, Materials Testing Reactor, Oak Ridge National Laboratory, Hanford, and Savannah River, was utilized in the development of the process flow for the facility and in the design of the special equipment required.

From the beginning of the design, all concerned recognized the need for emphasis on flexibility and ease of expansion. Economy was also an important consideration.

In collaboration with Arthur G. McKee & Company, the Architect-Engineer, an economical plan was developed. It gives the desired flexibility, lends itself to expansion, accommodates the process flow (Fig. 3) at the desired level of operation, permits adequate contamination control (particularly in the water pits and hot cells), and makes optimum use of weight handling facilities such as the 125-ton core-unloading crane.

WATER PITS

Layout

The size, orientation, and configuration of the water pits was an important consideration in the development of the plan. It was considered essential that water pits be used because they provide the simplest, most economical solution to the problem of unloading very heavy, highly radioactive reactor cores which are generating heat at relatively high rates. Economy dictated limitations on heavy concrete perimeter walls. On the other hand, troublesome and expensive liquid radioactive waste problem made it necessary to limit the volume of water which might become contaminated by escaping fission products. Separate pits were considered because they would afford maximum contamination control and permit access on three or four sides. They were rejected in favor of the indicated plan (see

Fig. 1) because the latter requires less concrete (and therefore costs less) and, through the use of the gates and diaphragms, permits highly flexible work areas. At the same time, sources of contamination can be confined to relatively small volumes of water. Moreover, a single underwater manipulator can serve all of the underwater work areas.

Special Features

Other special features were required of the water pits by virtue of their process requirements. Among the more important of these were:

- (1) a means of purifying and clarifying the water to remove mixed fission products, contaminated metal chips and machinings, and turbidity;
- (2) a strong, smooth, protective coating for the concrete possessing excellent decontamination properties, high resistance to radiation damage, high impact strength, and good ductility;
- (3) a means of cooling selected portions of the water volume to remove excessive decay heat;
- (4) sufficient water depth to provide necessary shielding for operators during the performance of various operations on the expended fuel components;
- (5) underwater viewing system to permit remote accomplishment of fine machine tool operations as well as inspections;
- (6) underwater handling equipment;
- (7) underwater machine tools required for the destructive disassembly of cores and the removal of non-fuel bearing materials from them prior to shipping the fuel-bearing material to the chemical reprocessing plant.

The above requirements are met in the plant now being built. A cation demineralizer system is provided for the removal of fission products. A diatomaceous earth filter supplemented by strategically located cup strainers and mechanical filters are provided for clarification purposes. In addition, provision is made for the non-routine injection of chlorine into the water to facilitate algae control if required.

A thermo plastic, polyester resin-type covering was found, Evershield Liquid Tile (Ref. 1), which seemed to possess all of the required properties for the concrete covering. This is specified for the walls and floors of the pits and for the floor of the building in areas subject to impact, wear, and radioactive contamination. A lightweight glass fabric is applied between the prime and finish coats of Liquid Tile where high impact resistance and resistance to abrasion are desired. Amercoat 33 is specified for the removable gates and diaphragms.

Provision is made to circulate any isolable volume of water in the pits through a 500-kw heat exchanger unit. A cooling tower on the roof

will serve the heat exchanger and the design is arranged to permit the addition of another heat exchanger and a second cooling tower if required later.

One 12-ft² pit is designed to give a 30-ft water depth to permit the safe underwater removal of long, irradiated components from their shipping containers. The water depth in the remaining areas of the pits is 20 ft.

Equipment

The entire water pit area is served by a General Mills underwater mechanical manipulator supported by a bridge having a 36-ft span. This manipulator has a straight-arm capacity of 750 lb and will be supplemented by various hand tools which can be used alone or in conjunction with the 125/25-ton bridge crane which also serves the area.

A closed circuit underwater television camera and monitor is provided for underwater viewing. Necessary lighting is provided by 1000-watt portable diving lights. The camera is tripod-mounted and can be located anywhere in the water pits.

The machine tool equipment for the water pits presently consists of a specially designed underwater hack saw which is capable of sawing 85-in. long components with diameters as large as 40 in. and a specially designed underwater milling machine which is capable of precision milling operations on components up to 85-in. long with a 7-in. diameter. Also provided are special underwater racks with boron-stainless steel inserts, designed to provide storage of fuel-bearing components in a fixed geometrical pattern assuring that a critical configuration is not accidentally achieved. Other equipment for the water pits will be procured as necessary to meet the increasing work load, to allow for the expected changes in core design, and make provisions for obtaining any new types of information desired.

The water pit area is connected to the hot cells by means of a water-filled canal, 6-ft wide and 20-ft deep. Material from the water pits is transported to Hot Cell 1 by means of a small, cable-operated car which moves along the bottom of the canal until it reaches a remote-operated fork lift located just beneath the bottom entrance door to hot cell No. 1. The fork lift elevates the radioactive material to the level of the hot cell floor where it can be removed by the hot cell handling equipment.

HOT CELLS

Plan

The hot cells are designed around hypothetical process requirements. These requirements are based upon work previously performed on an actual core and are considered to be representative of the type of work which will be required by all developmental core designs. The process requirements are graphically indicated in Fig. 3. The arrangement of the water pits, the hot cells, and the radiochemistry laboratories, where the processes take place, is shown in Figs. 4 and 5.

Figure 4 suggests a production-line type of operation to the extent that the equipment and sequence of operations will be quite similar for

Fig. 4—Cutaway view of water pits, hot cells, and transfer tunnel.

Fig. 5—High level radiochemistry laboratory.

each type of core. The design of the hot cells is based on this philosophy of operation in order to obtain optimum production at a minimum cost. Accordingly, the dimensions, shielding, viewing windows, ventilation, equipment, etc. are designed to serve the particular purpose for which the hot cell is intended. This does not mean that the hot cell cannot be used for other purposes. There are many modifications possible with a minimum of effort. It does mean that the cells, taken individually, do not have the degree of versatility usually desired in research operations. The hot cell bank as a whole, however, is highly versatile.

Special Features

In the interest of economical construction, all cells, with the exception of the metallographic cell extension, are constructed of reinforced concrete, and a uniform wall thickness is maintained throughout. A 3-ft thickness is specified, partly because much of the available hot cell equipment is designed around that wall thickness, and partly because necessary shielding can be obtained without resorting to concretes of excessively high density. The 3-ft thickness also permits viewing windows to be kept thin enough to afford good perspective viewing and clear enough to avoid excessive loss of light transmittance.

Other features which the hot cells have in common are:

- (1) All cells are 6-ft deep to permit efficient operation and easy removal of conventional master-slaves;
- (2) Stainless steel floors and liquid tile-coated walls and roofs facilitate decontamination;
- (3) Removable precast block partitions between cells (except between 2 and 3), to allow a temporary increase in cell length if necessary;
- (4) Concrete plug-type doors to permit easy access and for mounting special experimental setups;
- (5) Removable precast concrete roof slabs to provide an access opening as large as the cell cross-section.
- (6) Mercury vapor operating lights and fluorescent maintenance lights;
- (7) A utility service trench located at the base of the cells in the operating gallery and a similar service duct located above the plug doors in the hot cell loading area, each carrying air, water, steam, and both single and three phase electric power, all provided with strategically located cutoff valves or switches and quick-disconnect couplings or outlets;
- (8) A high-suction vacuum cleaning outlet for cleaning and contamination control purposes;
- (9) A floor drain connected to the high level radioactive waste disposal system;

- (10) A ventilation system exhausting to atmosphere through a 100 ft high stack, with rough and fine filtration between the hot cell and the stack;
- (11) High density glass windows, non-browning quality glass designed to have a shielding value equal to that of the concrete wall in which they are mounted;
- (12) All served by one of the two 20-ton cranes.

The hot cells are interconnected by means of a chain-driven conveyor system located along the rear wall of the hot cells and with the conveyor pan operating at a level above the tops of the plug doors. The openings through the hot cell partitions are covered by stainless steel sliding gates arranged to permit passage of a piece of material as large as 12 in. x 12 in.

Functional Design

A glance at the process flow chart (Fig. 3) will show the reasons behind the variations in some of the design features of the hot cells. For example, Hot Cell 1 is designed to handle large fuel-bearing sections up to 9 ft in length. It is 16-ft long and 15-ft high inside. Also, it is served by two large viewing windows and equipped with two General Mills mechanical manipulators. Exterior walls are constructed of ferro-phosphorus aggregate concrete having a density of 290 lb/cu ft. This limits the maximum radiation dose rate in the operation gallery to less than 2.5 mr/hr even when a large, highly irradiated fuel unit is inside. At a level of 8 ft above the working floor, the concrete is changed to conventional density of 150 lb/cu ft. Since the operations within this cell are comparatively "clean", the ventilation air is exhausted through coarse and fine (dry) filters.

Hot Cell 2, on the other hand, is to be engaged in machining operations on smaller elements. It is, therefore, shorter in length, but has the same interior height permitting it to be served by the two General Mills manipulators which serve Hot Cell 1. Its shielding is the same as Hot Cell 1, but since its machining operations may be very contaminating, ventilation from the cell passes through a Peabody wet scrubber in addition to the coarse and fine dry filters.

The operations in the remaining cells are on small specimens, hence the cells are all 5 ft long and only 11 ft high inside. These cells require no heavy mechanical manipulator and are equipped with master-slaves only. They each have only one viewing window and the shielding is reduced to that afforded by magnetite concrete having a density of 190 lb/cu ft. Only dry filters are provided in the ventilation systems from these cells, except in the case of the chemical cells. In the latter cells, the operations require that a Peabody wet scrubber be added to the ventilation system.

Cell 9 serves as a sample preparation cell for the steel metallographic cell extension. The two are connected by a shielded conveyor belt system which is designed to carry the prepared specimen into the steel cell for metallographic examination. The steel metallographic cell is specially

designed for the operations intended and, because of the very small sample size, shielding is reduced to the equivalent of 6 in. of steel. Model 4 manipulators (Central Research Laboratories) are used in this cell.

Improved Equipment

The equipment for the hot cells is the minimum required for initial operations in accordance with the process flow (Fig. 3). It is similar in design to that used for like operations in other hot cell laboratories. However, efforts have been made to improve the equipment based on the experience of other users. Principal items of hot cell equipment are listed in Table I. Simplicity, ease of maintenance and decontamination, and contamination control are requisites for the hot cell equipment.

RADIOCHEMISTRY LABORATORIES

The radiochemistry laboratories are served primarily by the chemistry hot cells. Aliquot samples are transferred from the hot cells to the hot cell loading area by way of a transfer mechanism built into the hot cell plug door. The sample, in a shielded container, will be transferred to the high level radiochemistry laboratory by means of a special manually-operated dolly.

The high level laboratory with its principal items of equipment are shown in Fig. 5. The equipment is essentially conventional with the exception of the two gamma boxes. These are special designs arranged to utilize two ANL, Model 7 manipulators and to provide 5 in. of lead shielding.

There are four low level radiochemistry laboratories, each approximately 20 ft x 11 ft. These laboratories are equipped in a conventional manner and supplied with customary services. A heavily shielded counting room serves the laboratories and a mass spectrometer facility supplements them. A separate ventilation system equipped with coarse filters at the source, a Peabody wet scrubber, and fine filters, serves the laboratory area. This system discharges into the stack which also serves the hot cell ventilation system.

SUPPORT FACILITIES

The principal facilities supporting the water pits, hot cells, and radiochemistry laboratories are:

- (1) The decontamination shop;
- (2) The "hot" maintenance shop, which is a machine shop designed for working on equipment and material which cannot be decontaminated to normal tolerance levels;
- (3) The "cold" maintenance shop which is a conventional maintenance machine shop;
- (4) The instrument maintenance shop;

TABLE I
Major Pieces of Hot Cell Equipment

39

Item	Manufacturer	Description, Function and Capacity	Location	Quantity
Stereoviewer	Bausch & Lomb Optical Co.	For stereovision and stereophotography Magnification range from 3/4 to 3 1/4 X	Any Hot Cell	1
GM Manipulators Model L3	General Mills, Inc.	All electric, operates cell equipment; handles core components; load capacity 750 lb.	Hot Cell 1 and 2	2
Specimen Viewing Table	Challenge Machine Co.	Chrome plated, 96 in. x 18 in., used for measurements and viewing	Hot Cell 1	1
Water Channel Micrometer	Bettis Design	Measures variations in water passages between fuel elements	Hot Cell 1	1
Punch	Famco Machine Co.	Five-ton capacity; variety of punching operations	Hot Cell 2	1
Power Hack	Keller Hack Saw Co.	General utility, cuts all types of fuel elements - Jefferson Model 601	Hot Cell 2	1
Precision Shear	O'Neill Irwin Mfg.	Rough shaping of tensile test specimens, shearing width 12 in. capacity 16-gauge sheet steel	Hot Cell 2	1
Test Specimen Shaper	Sieburg Industries	Finished machining of rectangular tensile and bond specimens	Hot Cell 2	1
Fuel element cutoff machine	Irwin Tool & Die	Initial preparation of metallographic specimens	Hot Cell 2	1

TABLE I (Continued)

Item	Manufacturer	Description, Function and Capacity	Location	Quantity
Model 8 Masterslaves	Central Research Laboratory	General purpose remote handlers; load capacity is about 17 lbs. in any direction	Hot Cells 3, 4, 5, 6, 7, 9	6 pr.
Autoclaves and Controls	Autoclave Engineers, Inc	Used for corrosion testing of irradiated fuel, operates at 650° F water at working pressure of 3000 psi	Hot Cell 4	2
Vacuum pump	Welch Scientific Co	Used in chemical operations; fission gas determination and preparation of aliquots	Hot Cell 3	1
40 Centrifuge	Fisher Scientific Co	Used in chemical operations; fission gas determination and preparation of aliquots	Hot Cell 3	1
Remote Izod tester	Baldwin-Lime- Hamilton Corp with Bettis Alt.	Determines toughness of specimen. Special vice heats or cools impact specimen for special tests	Hot Cell 6	1
Tensile Tester	Instron Engineering Corp	Per forms tensile, elongation and bond strength tests; load capacity 10,000 lbs	Hot Cell 5	1
200 gm balance	American Balance Co	Weighing	Hot Cell 7	1
1-kg balance	Mettler Co	Gross weighing and gross density deter- mination	Hot Cell 7	1
Micrometer, re- mote-operated	Bettis Design	Physical measurements	Any Hot Cell	1

TABLE I (Continued)

Item	Manufacturer	Description, Function and Capacity	Location	Quantity
Profile recorder	Bettis Design	Studies surface and over-all distortions of samples, handles samples up to 3" wide 10" long, and 1/4" thick of any shape	Hot Cell 7	1
Lapping Machine	Crane Packing Co	Prepares specimen before polishing	Hot Cell 9	1
41 Polishing Wheels	Bettis Design	Prepare metallographic specimens for microscopic examination. Can handle four metallographic samples at one time	Hot Cell 9	3
Cleaning Machine	Bettis Design	Ultrasonic; removes oil, abrasives, and stains before microexamination	Hot Cell 9	1
Mounting Machine	Bettis Design	Standard jig-type; mounts specimen before polishing	Hot Cell 9	1
Cathodic etcher and MG set		Produces etches on specimens for macro and microscopic studies. Power unit can supply 5000 volts at 30 ma.	Metallographic Extension	1

- (5) A photographic darkroom and a "cold" metallographic laboratory which are used in the control and development of metallographic procedures;
- (6) Administrative facilities.

Decontamination Shop (Figure 6)

Of these support facilities, the decontamination shop is worthy of special comment. Essentially it is a three-part area covering about 1750 sq ft. A "gross" decontamination area is located in the high bay area of the building and separated from it by a 10-ft high concrete block wall. There is no ceiling over this area, permitting it to be served by the 125-ton bridge crane. The floor and walls of this area are covered with Liquid Tile and special down-draft ventilation is provided. The area contains two large stainless steel tanks for soaking and electrolytic stripping operations, a degreaser, and a ventilation outlet for a portable hood. Steam, water, air, and electric power are provided.

Smaller equipment and parts are decontaminated in a low bay area adjacent to the gross decontamination and disassembly area. This low bay area, with a 14-ft ceiling, has a floor area of about 1200 sq ft and is equipped with sinks and work benches only. Another 1200 sq ft of floor space opens off this low bay area. Served by the 20-ton bridge crane of the "cold" maintenance shop, it is available for final decontamination, assembly and monitoring.

SYSTEMS AND UTILITIES

The process and process supporting facilities described above obviously require both conventional and unconventional systems or utilities to serve them. The conventional systems -- electrical, water, plumbing, sanitary sewage, steam and laboratory services -- presented no unusual problems. Some of the less conventional systems -- for example, the Safety Radiation Monitoring and the Ventilation System -- were treated in what is now becoming more or less standard where radioactive materials are handled. Some of the outstanding features of the ventilation system were mentioned previously in connection with the description of the hot cells and radiochemistry laboratories. In general, the guiding principles for ventilation were:

1. Maintenance of a pressure gradient from areas of low contamination to areas of high contamination;
2. Adequate filtration of air as near the source of contamination as practicable.
3. Positive air flow at high velocity (125 fpm) across openings into highly contaminated areas like hot cells and radiochemistry hoods.
4. Duplicate equipment and emergency power sources to insure high reliability of ventilation for highly radioactive areas such as hot cells.

Fig. 6—Receiving area and decontamination shop.

The remaining unconventional systems; i.e., the Pit and Canal Water System and the Radioactive Waste System presented a number of problems. Most of them are familiar ones to individuals working in the field, but a few of them can be profitably considered here without getting involved in the design details of the systems.

The principal components of these systems are located in an equipment pit at the west end of the canal. This permits the maximum use of gravity drainage with its obvious advantages. As previously mentioned, a cation demineralizer system is used to decontaminate the pit and canal water. A thin-film centrifugal Kontro-Artisan type evaporator is used to concentrate the highly radioactive and corrosive wastes from the laboratories, decontamination shop and hot cells before it is discharged to a buried permanent storage tank located just outside the building. Other major components in the equipment pit include the tanks, pumps and equipment associated with the evaporator system and three sets of retention tanks for three classes of radioactive wastes categorized according to their expected levels of radioactivity. Concrete shielding walls are provided around high activity equipment. Valves located behind these walls are operated with extension stems through the walls.

The corrosive nature of the demineralized pit water as well as its probable radioactivity content, and the presence of flourides and decontamination chemicals in the high level radioactive waste lines required careful choice of material to serve the purpose with minimum cost. In general, where lines and fittings are accessible, corrosion resistance is sacrificed to some extent in the interest of lower first cost. Inaccessible components and piping and those likely to become highly contaminated are usually made of the most corrosion resistant material available for the service intended. These general policies explain the variety of corrosion resisting materials used in the systems. Process piping and fittings are made of saran-lined carbon steel, type 316 stainless steel, and Carpenter 20. Tanks are carbon steel, type 316 stainless steel, carbon steel lined with Heresite 403, carbon steel clad with Carpenter 20, carbon steel clad with type 304 stainless, and concrete with a protective coating such as Amercoat 33 or Liquid Tile. Inconel and type 316 stainless steel are used to meet the corrosion problems in the radioactive waste evaporator system. Type 304 stainless steel is utilized for the exhaust ductwork of the radiochemistry laboratory and the chemistry hot cells' ventilation systems.

REFERENCES

1. Clayton, N. J., "Effects of Radiation and Contamination on a Polyester Resin Protective Coating", Bettis Plant Report WAPD-CTA(EC)-305, February 12, 1957.

The Hot Laboratory Facility at Knolls Atomic Power Laboratory

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I. The Hot Lab as a Part of KAPL

Figure 1 gives a summary picture of how the Hot Lab fits into the over-all KAPL Project-type organization. In general, it will be noted that we are a part of the Technical Operation and specifically part of the Irradiations Laboratory.

The present functions of the Hot Lab are as follows:

- (a) Post-irradiation testing.
- (b) Engineering assistance for the design and development of Hot Lab facilities, equipment and test apparatus.
- (c) Hot Lab decontamination and maintenance.

II. Facilities

Figure 2 shows the physical lay-out of the Hot Lab. This consists of eight cells, four of which are the back to back type, each having a radiation lock and three work stations or modules.

The other four cells are somewhat special purpose cells as follows:

- 1. Cell #5 - Remote metallography.
- 2. Cell #6 - High pressure and temperature loop for post-irradiation corrosion testing.

Fig. 1—Radioactive materials laboratory, KAPL.

9 TECHNICIANS

Fig. 2—Physical lay-out of the hot laboratory.

3. Cell #7 - Chemistry.

4. Cell #8 - Annealing or Heat Treating.

The four large cells are serviced by General Mills manipulators and plug-in type Master-slaves, while the others use the Model 4 or 7 master-slaves.

The supporting facilities are as follows:

- (a) Office space
- (b) Dark room
- (c) Storage cell
- (d) Water pit
- (e) Small machine shop
- (f) Engineering mock-up area
- (g) In-floor sample storage and manipulator repair area
- (h) Crafts work area
- (i) Cask storage
- (j) Decontamination area
- (k) Instrument storage
- (l) Ventilation equipment (not shown on figure). This equipment is located on mezzanines over the Hot Lab office and over the manipulator repair area, instrument storage and crafts work area.

III. Funding

All funds for operating the Hot Lab are supplied by the customers using the lab.

Hot Laboratory Problems in Isolating Gram Quantities of Transplutonium Elements

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This paper shows how the experience gained in processing small research quantities of irradiated plutonium will be applied to the processing of irradiated plutonium fuel assemblies.

The material being processed is about 300 times greater than the earlier research samples and will present problems in methods and equipment for processing. In addition, the proportionately higher neutron emission from the transplutonium elements will require the use of neutron caves as a substitute for glove boxes.

In the spring of 1951, plans were made at Argonne for the irradiation of plutonium in the Materials Testing Reactor, then under construction, to take advantage of the high flux which would be available close to the core. This flux was expected to be about 10^{14} n/cm²/sec.

Plutonium irradiation samples in the form of aluminum clad plutonium-aluminum alloys were fabricated by the Argonne Metallurgy Division¹ from alloys prepared by Los Alamos Scientific Laboratory. Each sample was a 1.25 inch outside diameter by 2 inch long cylinder, as shown in Figure 1. Because of its size and shape the sample was likened to a dinner napkin ring, and the expression "napkin ring" has been used ever since.

The napkin rings were made with 5 per cent alloys containing about 170 milligrams of plutonium each and with 10 per cent alloys containing about 350 milligrams of plutonium. The total weight of the finished ring was about 20 grams.

AS-108

Fig. 1—Napkin ring.

Fig. 2—Preparing to transfer napkin ring into cave.

The irradiation of the napkin rings was begun at the Materials Testing Reactor in September of 1952. The first napkin ring was brought back to Argonne after almost a year's irradiation, and processed in August of 1953. In January of 1954, a second sample was processed and in May of 1954, a third napkin ring was processed.

Results of these irradiations were reported at the 1955 Geneva Conference.²

The actual processing of the napkin ring in a cave has involved a preliminary cleaning followed by measurement of weight and density by the Metallurgy Division in order to determine whether or not any physical change has occurred. Radiation readings taken at the same time have been about 25 roentgens per hour at a distance of six feet or more.

The napkin ring has then been moved from the Metallurgy Cave, in a shielded shipping container, to the Chemistry Cave. The shipping container is fitted with a flanged tube for loading the napkin ring into the cave, as shown in Figure 2. A ramrod action is used to push the napkin ring out of the shipping container through the tube, into the cave.

The ring is then dissolved in a sodium hydroxide solution in a closed glass vessel. The temperature of the reacting solution is controlled by a water bath that is heated or cooled by steam or cold water flowing through an immersed coil of copper tubing.

The dissolution leaves the heavy elements and most of the fission products in a suspension from which they are removed by centrifuging. The precipitate is dissolved in concentrated HCl and the solution is put through a Dowex-1 resin anion column to remove the plutonium isotopes, and some of the fission products. These fission products are mainly elements which occur in the fifth period of the periodic chart of the atoms. The effluent is then dried, redissolved in dilute HCl and put through a Dowex-50 cation resin column. The second column holds the rare earth fission products with the alkali metals and alkali earths going through before the heavy elements. The effluent from the resin column is assayed by removing 1 lambda (1 cubic millimeter) samples from the cave with the aid of self-filling pipets. These samples are removed through a radiation lock and taken to a hood where counting plates are prepared.

The desired fraction containing the alpha active heavy elements, stripped of most of the fission products is removed from the cave in a sealed container and taken to gloved boxes for separation into individual transplutonium elements.

The transplutonium elements which have been studied in the past several years have been produced by the irradiation of napkin rings in the Materials Test Reactor in a neutron flux of about 3×10^{14} for periods up to six years.

Under the neutron bombardment about 90 per cent of the Pu^{239} undergoes fission. The remaining 10 per cent captures the neutrons and is converted to heavier elements, some of which are also fissionable.

The neutron capture by an element increases its mass number by one. Higher masses of the element are built up in this manner until a beta-emitting isotope is produced. The beta decay results in nuclei whose mass remains the same, but whose positive charge is increased by one, thereby resulting in an isotope of the next higher atomic number.

Neutron capture and beta decay have thus built up the elements of atomic number from 95 through 100. The amount of the heavy elements formed from the original plutonium is governed by the amount of plutonium originally put in the reactor, the reactor flux, and the time in the reactor. The amount and composition of the heavy elements produced as a function of time in a reactor is shown in Figure 3.

Fig. 3—Production of heavier nuclides when Pu^{239} is irradiated at a flux of 3×10^4 neutrons/cm² for long time periods. The case treated represents one in which an individual Pu^{239} sample is placed in a reactor and the composition calculated for various alternative times of withdrawal.

Table I shows similar information based on an initial quantity of 100 grams Pu^{239} . The numerical values are rounded off to simplify the information. From the table, about 2 grams of curium-244 will be produced after irradiation in a flux of 3×10^{14} for about 2-1/2 years.

A program for irradiating large quantities of plutonium to make sufficient quantities of heavy elements for research chemistry is now under way.

In December of 1956, a snim rod containing 77 grams of plutonium was loaded into the Materials Test Reactor. This is the first of what will probably be a series of such irradiations. The fuel assembly will

TABLE I

Amount of Transplutonium Element Formed in Grams
from an Initial 100 Grams of Pu²³⁹

(Months in Reactor: Flux = 3×10^{14})

	0	3	6	9	12	15	18	21	24	27	30
Pu ²³⁹	100	8	1	.1	--	--					
Pu ²⁴⁰	--	13	5	2	.1	--	--	--			
Pu ²⁴¹	--	5	2	1	.2	.1	--	--			
Pu ²⁴²	--	3	5.7	6.4	6.4	6	5.7	5.4	5	4.7	4.3
Am ²⁴³	--	.06	.3	.6	.8	1	1.1	1.1	1.2	1.2	1.1
Cm ²⁴⁴	--	--	--	.2	.4	.6	.9	1.2	1.4	1.7	2
Cm ²⁴⁵	--	--	--	--	--	--	--	.011	.014	.016	.019
Cf ²⁵⁰	--	--	--	--	--	--	--	--	--	--	--
Cf ²⁵²	--	--	--	--	--	--	--	--	--	--	--

be brought back to Argonne and processed to remove the heavy elements in early 1959.

The scale up from 350 milligram samples to fuel assemblies in the 100 gram category will require some changes in processing techniques since it involves problems that are several magnitudes greater than those of the small samples heretofore processed.

Consideration is given here to the processing of a fuel assembly which initially contains 100 grams of plutonium 239, a more convenient reference value than the 77 grams in the first actual assembly. After irradiation, the upper and lower sections will be cut off leaving a trimmed fuel assembly about 28 inches long and 2.84 inches x 3.07 inches in cross section, as shown in Figure 4. After two years irradiation in a flux of 3×10^{14} , it should have the following composition as received:

1100 Aluminum	5 Kilograms
Heavy Elements	
Pu ²⁴² (Approx. 98% of total Pu)	5 grams
Am ²⁴³	1.2 grams
Cm ²⁴⁴	1.4 grams
Cm ²⁴⁵	.014 grams
Cf ²⁵⁰	0.4 micrograms
Cf ²⁵²	1.2 micrograms

Fig. 4—Plutonium section, Cd-Pu shim control rod.

If it is assumed that most of the plutonium was completely fissioned during the first month of irradiation, then the long-lived fission products would be as follows after two years of cooling:

Long-lived radioactive fission products

Krypton	85	.025 grams (11 curies)
Strontium	90	0.80 grams (225 curies)
Ruthenium	106	0.40 grams (2700 curies)
Cesium	137	2.94 grams (470 curies)
Cerium	144	0.36 grams (2320 curies)
Promethium	147	0.80 grams (750 curies)

Stable Inert Gases

Krypton	.36 grams
Xenon	10.8 grams

However, this distribution is modified by the fission products themselves being irradiated for two years and being converted to other isotopes. In addition, some fission products which are of little consequence in themselves, may become converted to significant radiation sources as a result of being irradiated for two years.

Gamma shielding for such conditions cannot therefore be predicated on the isotope distribution as determined from fission yield curves. Ruehle has worked out a useful series of curves and data for determining the gamma dose for an irradiated fission source.³

For the fuel assembly considered here, the isotopes governing the shielding problem are listed together with the gamma dose from each individual isotope after two days of cooling:

	mr/hr thru 8" lead	mr/hr thru 11" lead
Ru 106	33	---
Cs 137	8	---
Ce 144	73	1.1
Eu 156	1178	16.5
	<u>1292</u>	<u>17.6</u>

Note that europium which did not even appear as a significant fission product, dominates the shielding problem after two years of irradiation. The amount of stable xenon will not be any greater than that indicated, since the isotopic arrangement is such that more xenon will be converted to a radioactive form that will decay away than the new xenon formed.

A cask for shipment of the fuel assembly would probably have a cavity 6 inches in diameter and 30 inches long, with 12 inches of lead shielding. Such a cask will weigh about 8 tons.

After the fuel assembly is received and loaded into the cask, it will be dissolved in a sodium hydroxide solution. The addition of sodium nitrate will result in the formation of ammonia gas instead of explosive hydrogen. The reaction between the aluminum and the sodium hydroxide and sodium nitrate can be represented by the following equations:

Each of the above equations will represent the reaction of some portion of the aluminum. Blanco⁴ states that the evolution of gas is at a minimum when conditions approximate Equation 4. He also states that the evolution of hydrogen approaches a minimum of 3 milliliter/gram of aluminum dissolved if the concentration of NaNO_3 in the dissolver solution does not fall below 100 grams/liter.

On this basis, plus previous experience with napkin ring samples, a 5000 gram fuel assembly would be dissolved in a solution consisting of

12.5 kilograms NaOH
25 kilograms NaNO_3
62.5 liters H_2O

The gases evolved would be as follows:

H_2 - 15 liters
 NH_3 - 625 liters
 Kr^{85} - 6.6 milliliters
Stable Kr - 95 milliliters
Stable Xe - 1800 milliliters

The fuel assembly will be dissolved in a closed vessel that is slightly negative with respect to air pressure. This vessel will be a stainless steel tube 8 to 12 inches inside diameter and 30 to 40 inches high. It will have coils brazed around the outside for heating or cooling this content to control the rate of chemical reaction. The slurry resulting from the dissolution will be drained out from the bottom of the dissolver to a peristaltic pump which will discharge the slurry into a group of about six glass settling columns.

Gases given off from the dissolver will be drawn through a reflux condenser toward chilled evacuated steel bottles filled with charcoal for gas adsorption. A cartesian manostat in the line permits a flow from the dissolver to the evacuated bottle while maintaining the original pressure in the dissolver. This system has been described by M. D. Thaxter.⁵

Since the off gas volume is large, and composed principally of ammonia, the gas will be passed through a system for the removal of the ammonia. This may be accomplished by bubbling the gas through an acid solution to neutralize the ammonia, or by passing it through a cold trap when the ammonia might be condensed out. A material being given consideration is a dry resin in the form of small pellets which will hold the ammonia. This material which may be on the order of a cubic foot or less in volume for the quantity of ammonia to be absorbed, would lend itself very well to subsequent waste disposal.

After the ammonia is removed, only about 17 liters of gas remain. Since the activated charcoal can absorb seven or eight times its nominal volume of air at room temperature and 40 to 45 times its volume at -77°C , only a few one liter evacuated steel bottles, filled with activated coconut charcoal will be necessary to adsorb the remaining hydrogen and inert gases.

After the slurry has been transferred to the glass settling columns, which will be about 6 inches inside diameter by 4 feet high, additional water is added to the dissolver as a rinse and then pumped into the settling columns. The total volume of rinse water used will be about 60 liters, which will cut the salt concentration in half. The contents of the columns will be stirred by bubbling air or steam through them. The resulting total volume of liquid will then be 125 liters, or about 21 liters in each column. This will be allowed to stand for a period of about six hours to permit partial settling out of the precipitate. It is expected that about 75 per cent of the liquid can then be decanted, leaving about 30 liters to be centrifuged. This will be done in a large centrifuge having four 250-milliliter cups. Centrifuging will then proceed at a rate of about 1 liter every 20 minutes, or 3 liters per hour. Ten to twelve hours should complete the centrifuging.

The moist cake should comprise about 9 liters of volume. It will be dissolved in about 12 liters of concentrated HCl (about 12 molar), and the solution adjusted to 8.5 molar.

The HCl solution is then added to a number of anion columns containing a resin for adsorbing the Pu^{242} . These columns may be about four inches inside diameter, and about four to six such columns will be used simultaneously. A scale up of previous experience suggests that the flow rate of these columns should be about 500 milliliters per hour. The eluate from these columns will then be evaporated to dryness.

The evaporation will be conducted in closed vessels with the water vapor and HCl being pumped off through an aqueous solution to neutralize the HCl and also through a chilled tube to condense out the water vapor. The air which is drawn through the system will pass through high efficiency filters which will be located as close to the source of activity as conveniently possible.

After being evaporated to dryness, the resulting solid will be redissolved in about 3 liters of 1 molar HCl.

This solution will still contain about 6000 curies of beta activity and about 240 curies of alpha activity. It will then be put on several cation columns where the fission product activity will be held and concentrated. The large amount of radioactivity at this stage causes a considerable heat generation from the energy of the emitted alpha and beta particles. From studies made by Way-Wigner⁶, it is expected that about 35 watts of heat will be generated by the beta activity. If it is assumed that all alpha particles from 200 curies of activity are emitted with an energy of 6 Mev, then this emission will result in 7.1 watts of heat being given off.

The total heat generated in the column will then be in the order of 42 watts. The first bubble formation starts at about 40°C when the dissolved gases are driven out of solution, and the rate of formation increases with increased temperature.

Another limiting condition on the concentration of activity in the column is the ionization of water due to the intense alpha bombardment.

Work by Hart and Gordon⁷ indicates that this may be about 1 cc of gas per minute for 200 curies of 6 Mev energy absorbed.

Smaller scale work with resin columns has involved separations in which 3 to 4 milligrams of curium have been put through a column of 2 millimeters inside diameter that had a gas pressure on it of one atmosphere to force the liquid feed through at a more rapid rate.

By using the same technique, and cutting the concentration to the equivalent of 2 milligrams in a 2 millimeter column, the 1.4 grams of curium should require a column cross section of 700 times greater than the 2 millimeter column. Two columns, 1.5 inches inside diameter will give a very adequate flow area. By pressurizing the column, the liquid feed is moved through rapidly and the minute gas bubbles formed do not have a chance to agglomerate and form large bubbles that disturb the resin bed. The high flow rate will also carry off the heat generated.

The flow rate can be expected to be at least 12.5 milliliters per minutes for each column. The temperature rise of the eluate due to radioactive heating may be about 20°C.

Earlier work with the napkin ring⁸ has shown that we can expect spontaneous fission to occur in both the curium-244 and californium-252 at a rate of one fission giving 3 neutrons for each 10^6 alpha decays for the curium, and one fission giving 4 neutrons for each 32 alpha decays for the californium.

The curium fission will emit about 1.2×10^7 n_f/sec/gm, and the californium fission will emit about 2.5×10^6 n_f/sec/microgram.

For the quantities of heavy elements present, this would result in a total neutron emission of 1.9×10^7 n_f/sec. This neutron emission rate, if measured at a distance of 10 cm, gives a flux of 1.5×10^4 n_f/cm²/sec. At a distance of 50 cm, this flux is reduced to 600 n_f/cm²/sec. For neutron shielding alone, the cave should attenuate this flux by at least a factor of ten. To reduce a fast neutron flux by a factor of ten requires 5.5 inches of steel or 8.7 inches of 200 lb. per cubic foot magnetite concrete. There is, however, some variation in figures on neutron attenuation. It should be emphasized that after the gamma emitters have been separated from the heavy elements, heavy shielding is still required to protect the scientist from the neutron flux.

Since the interest in curium and californium will continue to increase as more of the elements are found, it will be wise to design future caves as neutron shields as well as gamma shields, and in addition, provide for good containment of the neutron emitter which is also an alpha emitter.

In order to handle future fuel assemblies and other irradiations of similar magnitude, the Argonne Chemistry Division has proposed the construction of some new caves. This system of caves is shown in Figure 5. The heavy walled caves, shown in detail in Figure 6, are for megacurie levels of activity, and the lighter walled caves shown in Figure 7 are for kilocurie levels.

Entrance to any of the working cells can be through one of the transfer cells. Experience with chemical processing and subsequent decontamination has shown that a good transfer cell or radiation lock

Fig. 5—Proposed chemistry hot laboratory.

will permit almost continuous entrance and exit from the hot working area with no shielding complications or interruption in the work.

Air from this transfer lock will also be sampled and counted for airborne activity before opening the outer door. Surface contamination and activity levels of items being removed will be checked with survey instruments whose probes will be inside the cell and handled remotely. The multiplicity of caves will be necessary when, because of neutron emission, the simple gloved box is replaced by a cave. Plans are already underway to produce hundreds of grams of curium by irradiating kilograms of plutonium. The program will eventually yield about 100 milligrams of Cf^{252} . In processing such quantities, we will have to work with about 2000 curies of curium alpha activity with approximately 10^9n/sec and approximately $3 \times 10^{11}\text{n/sec}$ from the Cf^{252} .

The proposed caves are designed to contain such quantities safely. Initial processing, such as the separation of heavy metals from the aluminum carrier and the fission products will take place in the heavy walled caves. Continued work with curium and californium will then take place within the neutron shielding afforded by the lighter walled caves.

References

- (1) Shuck, A. B. The Manufacture of Aluminum Clad Aluminum-Plutonium Alloy Irradiation Specimens. ANL-5043; April 1952.

Fig. 6—High level cave.

Fig. 7—Low level cave.

- (2) Bentley, W. C., et al. The Formation of Higher Isotopes and Higher Elements by Reactor Irradiation of Pu²³⁹. Paper No. 809, International Conference of the Peaceful Uses of Atomic Energy; Geneva, Switzerland; 1955.
- (3) Ruehle, W. G., Jr. The Determination of Fission Product Gamma Doses. GEAP-0974, General Electric Atomic Power Equipment Department; San Jose, California; February 1957.
- (4) Blanco, R. E. Dissolution and Feed Adjustment. Symposium on the Reprocessing of Irradiated Fuels; TID-7534, Brussels; May 1957.
- (5) Thaxter, M. D., Cantelow, H. P. and Burk, C. Off Gas Treatment in Berkeley Enclosures. Proceeding of the Fifth Hot Laboratories and Equipment Conference; Philadelphia, March 1957.
- (6) Way, K. and Wigner, E. P. Rate of Decay of Fission Products. In Radiochemical Studies: The Fission Products, Book 1, p. 436, National Nuclear Energy Series, Division IV, Vol. 9, McGraw-Hill Book Co., New York, 1957.
- (7) Hart, E. J. and Gordon, S. Gas Evolution for Dosimetry of High Gammas, Neutron Fluxes. Nucleonics XII, April 1954, pp. 40-43.
- (8) Studier, M. H. and Huizenga, J. R. Correlation of Spontaneous Fission Half-lives. Physical Review 96: 545 (1954).

Hot Laboratory Operations at Argonne National Laboratory

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The physical and metallurgical testing hot laboratory at Argonne is a general purpose facility available for use by all of the divisions of the Laboratory. It is designed for non-chemical or "dry" examination and testing of highly radioactive materials. Other high level cells for more specialized work either exist or are being constructed within the separate divisions of the Laboratory. These are for handling cyclotron targets, for use in connection with reactor operations, and for chemistry and chemical engineering work.

The general purpose facility is operated by a hot laboratory group which is part of the Remote Control Engineering Division⁽¹⁾. It is responsible for the scheduling of cave time and furnishes assistance to users from other divisions in experiment planning and cave operation. In the group are two staff men, three technicians for actual cave operations, and two shop men for equipment maintenance and for immediate fabrication of specialized equipment.

In addition to the Remote Control Engineering Division responsibilities, three of the essential functions are performed by groups from other divisions of the Laboratory. Radiation monitoring and air sampling are carried out by two technicians of the Industrial Hygiene and Safety Division who are assigned to the building. This has purposely been made the responsibility of another division because it was believed that self auditing of radiation safety would not be entirely effective. Low level, high level, and liquid wastes are removed from the building by waste disposal groups. Equipment which has become so badly contaminated that normal cleaning procedures are not effective may be transferred to the Reclamation Department for complete cleaning and overhaul. This department will also supply decontamination crews for work in the hot laboratory if major decontamination of the caves is required.

The building is divided into a contamination-suspect area for the "hot" operation and a "cold" area for offices, laboratories, photographic dark rooms, and a machine shop. (See illustration). The suspect area contains a three cell cave with a capacity of 100 curies which is used continuously by the Metallurgy Division for metallographic work⁽²⁾. Equipment is installed for electrical discharge cutting, sample preparation, microhardness determinations, and magnified examination and photography. Under construction is a small cell of similar capacity to house an X-ray diffractometer.

Physics and Metallurgy Hot Laboratory, Building 301, Argonne National Laboratory

For the more general type of work where the samples may be larger and therefore more radioactive are two 10,000 curie cells. Construction is essentially complete on a two cell 100,000 curie cave to be used for the same purpose⁽³⁾. The capacity of these cells can later be increased to one million curies by the addition of more shielding at the windows. A wide variety of remotely controlled cutting and testing equipment is available for use in the general purpose caves.

Manipulation within all of the caves is by Argonne Model 8 manipulators. In addition, cranes are provided in the general purpose cells for heavy duty lifting.

The particular group of people doing the work in the cells depends upon the nature of the work. For example, routine operations may be performed entirely by Remote Control Engineering Division personnel and the raw data supplied to the user. When the work is not of a routine nature, the scientist is usually present throughout most of the operation to observe and direct the course of the experiment. On the other hand, divisions which have an essentially continuous hot cell program, maintain a group of technicians in the building who are trained in hot laboratory procedures. Their work is carried forward with a minimum amount of assistance required from Remote Control. In any case, the basic philosophy of operation is to insure that the scientist can remain in close contact with his work.

REFERENCES

1. Doe, W. B.: Hot Laboratory Procedures at Argonne, Proceedings of the Fifth Hot Laboratories and Equipment Conference, Vol. III, Pergamon Press, 1957, pp. 51-55.
2. Rylander, E. W. and R. A. Blomgren: Operating Procedures of a Hot Lab for Solid-State Tests, Nucleonics, Vol. 12, No. 11, pp. 98-100, November 1954.
3. Doe, W. B.: Megacurie Cave for Metallurgical Testing, Sixth Hot Laboratories and Equipment Conference, Chicago, March 1958.

Operational Aspects of Hot Laboratories

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I. The High Level Caves at the Savannah River Laboratory

The kilocurie cave facility at the Savannah River Laboratory consists of three caves in a single cell block, as shown in Figure 1. The facility is similar in many respects to other caves throughout the country.

Each cell is approximately 6 ft. x 12 ft. in floor area and 17 ft. high. The walls are three feet thick and provide shielding for about 5000 curies of 1 Mev gamma radioactivity. There are two windows in the operating face of each cell with a slot in the wall over the windows for master-slave manipulators. Two heavy-duty electric rectilinear manipulators and a one-ton bridge crane are mounted on continuous rails that run the entire length of the cell block. They can be operated in any cell, as required. The manipulators and experimental apparatus are controlled from the "Operating Area" which is maintained in a contamination-free condition.

Personnel enter the cells and equipment is installed only from the rear or "Loading Area". There is an access door and the cell covers can be removed. The Loading Area is a high bay area served by a 10 ton bridge crane.

The Loading Area is generally maintained in a contamination-free condition. However, radioactive materials are handled here and contamination does occur at times. The Loading Area is entered via a "Change Room" where appropriate protective clothing is available. All persons and equipment must be monitored for contamination before leaving the Loading Area.

The cave facility is operated by the "High Level Caves Group", which is a part of the Laboratory Services Division of the Savannah River Laboratory. The relationship of this group to the over-all laboratory organization is shown in Figure 2. The Laboratory contains

Fig. 1 —High level caves, Savannah River Laboratory.

Fig. 2—Organization chart, Savannah River Laboratory.

seven research divisions and three service divisions. Some of the services provided by the service divisions are listed in Figure 2.

The High Level Caves Group undertakes a study program when requested to do so by one of the research divisions. The requestor must supply the irradiated specimens and a detailed description of the work required. The High Level Caves Group takes over from there, performs the experiment, and returns the data to the requestor for evaluation. (Portions of the work are sub-contracted to the other service groups in the laboratory wherever possible.) The following services are available, as needed:

1. Design and fabrication of equipment to carry out the work.
2. Installation of equipment in a cell.
3. Shipment of specimens to the cave facility.
4. Actual operation of the experiment, "obtaining the data".
The requestor is free to observe the work if he wishes, but all operation of equipment and manipulators is done by the High Level Caves Group.
5. Decontamination of cells and equipment.
6. Waste disposal.
7. Disposal of specimens.

This cave facility has been in operation for about four years.

Hot Cell Operations, Solid State Division

Oak Ridge National Laboratory

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Oak Ridge, Tennessee

At the present time the hot cell facility at the ORNL Solid State Division is operated entirely on a service basis, with all operating costs distributed among the various users. Much of the work performed in the cells is for projects originating within the Solid State Division, but a great deal of supporting work is also carried out for other reactor research and development efforts, both at ORNL and at other sites.

Because of the diversified nature of the demands placed upon them the cells must be quite versatile. The physical layout consists of eight cells, seven of which are in a tandem arrangement, with the eighth being an annex to cell 2, the remote metallographic cell. This is shown in Fig. 1. The shielding is 32-in. barytes concrete on the main cell block with the metallographic cell annex having 6-in. laminated steel.

The first three cells are served by two General Mills manipulators while the others use the Model 4 master-slave type. In addition, Model 8 master-slaves are being installed in the large cells. Other permanently installed equipment includes a remote lathe and milling machine in cell 1, remote metallographic equipment (consisting of cut-off machine, mounting press, lapping and polishing machines, ultrasonic cleaning station, diamond abrasive dispenser, Tukon hardness tester, stereo unit, and metallograph)

in cells 2 and 2A, and a through-the-wall periscope in cell 3. An integral in-cell vacuum cleaning system is under construction.

Fig. 1—Floor plan, Solid State Division hot cell facility.

The Hot Lab Section is divided nominally into two groups: Hot Cell Operations and Remote Metallography. This is largely a theoretical division, however, since in actual practice the personnel of one group frequently are utilized in the other. At the present time the full-time staff consists of eight men: four technicians and four professional people. It is possible to operate with a staff of this size because many of the groups or projects requesting cell work frequently provide their own personnel for experimental operations as well as for installation and testing of equipment and decontamination. A rough organizational outline is shown in Fig. 2.

The Hot Lab Section does not have an operating budget, per se. Instead, as indicated previously, all operating costs are distributed among the various users, with the amount charged being determined by the proportion of the total time used by the particular customer. For example, if one customer uses 100 hours of the group's effort in a particular month and all customers together use 500 hours, then the charge for that customer will be $100 \div 500$ or 20% of the total operating cost for that month.

Fig. 2—Organization chart for hot laboratory section.

It is not intended that this presentation be concerned with the various problems encountered in our operations, but rather to give only a brief description of our facility and organization as a background for further discussion.

New Multi-Cell Facility in Idaho

D. C. Durrill and R. D. Dwigans

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Idaho Test Station
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Construction of a new facility comprising four small hot cells is nearly complete at the Aircraft Nuclear Propulsion test station operated by the General Electric Company in Idaho. Special features include split-level operating areas, concrete-filled vault-type doors for personnel and equipment entry, a remotely-operated underfloor dolly system and a remote vacuum cleaning and wash down system.

The new hot cell facility, consisting of four hot cells and miscellaneous work areas at the Idaho Test Station, was designed and built to satisfy the need for space in which reactor fuel and mechanical components could be studied, sectioned and tested to the fullest extent to determine their post-operations condition. Study of the various operations to be carried out led to dividing the cell working space needed into four equal parts; the type and degree of contamination to be expected being slightly different in each. The inside dimensions of each cell are 6 feet x 8 feet x 12 feet high.

The basic design criteria were:

1. Each cell was to be shielded against mixed-spectrum radiation totaling 1×10^7 R/hour.
2. A 5-ton monorail crane was to be installed to unload shipping casks from tracks, thence to the new cells or to the ITS storage pool. Emptying of the casks into the cells was to be done through means of an underfloor dolly.
3. The building housing the cells was also to provide space for:
 - a. Control gallery.
 - b. Personnel change room.
 - c. "Warm" Set-up area for equipment check-out.
 - d. Hot tool storage and decontamination room.
 - e. Equipment room (blowers, compressors, motor-generators, etc.)

4. A separate crane of 30 tons capacity was to be built entering the pool building directly from a truck unloading area, for use in unloading heavy casks underwater.
5. A shielded intercell transfer drawer system was to be provided so that samples could be moved from cell to cell. A second transfer drawer system for tools or small parts was to be provided from a set-up area to the interior of each cell.
6. A concrete-filled vault-type hinged door was to be used for equipment and personnel entry into each cell.
7. Provision was to be made for installation of a false floor immediately below the window level of each cell. The false floor height and area were to be such that model 8 master-slave manipulators could reach all points on its surface.
8. Shield windows were to be of such design that operators could view the ceilings, floors, and both side walls to within one foot of the front walls.
9. Bridge cranes of 1000-lb. capacity were to be installed in both end cells. Rails were to be installed in the ceilings of the remaining two cells for future installation of similar cranes. A 1000-lb. manually-operated bridge crane was to be installed over the control area to carry manipulators, periscopes, etc. during installation and removal.
10. Unistruts were to be cast into walls and ceilings to provide means of mounting remote-handling equipment without drilling into walls.
11. A central vacuum cleaning system was to be installed with a pre-filtered outlet in each cell. For further decontamination, a wash-down system of pipes and spray-heads in each cell supplied by a central jet pump was to be provided. The jet pump was to provide means for introducing decontamination agents into the wash-down solution.
12. Lighting in cell interiors was to be of the mercury-arc type and at a level of 300-foot candles 30 inches above the floor of each cell.
13. Intercommunication was to be provided from the interior to the exterior of each cell, between the various areas external to the cells and from the HCA to the other remote-handling areas in the adjoining building.
14. The interior of each cell was to be painted with a covering resistant to hot water, chemicals and wear.
15. The exhaust ventilation system was to maintain each cell at a negative 0.25 inches of water with respect to atmospheric pressure. An auxiliary air exhaust system in each cell was to become automatically operative when the personnel access door was open to maintain 200 cfm inflow to the cell.

16. Services to each cell as well as other work areas were to include:

- a. Industrial hot and cold water.
- b. Demineralized water.
- c. 110 psig compressed air.
- d. 110 psig instrument quality compressed air.
- e. 480-volt 3-phase 60-cycle AC.
- f. 115-volt 1-phase 60-cycle AC.
- g. 115-volt 1-phase 400-cycle AC.
- h. 28-volt DC.
- i. Vacuum cleaning system sized for a minimum vacuum of 60 inches water gage at each outlet.
- j. High vacuum roughing system sized with sufficient capacity to evacuate 6 cubic feet from an atmospheric pressure of 12.3 psia to a vacuum of 24 inches of mercury (measured at 5,000 feet elevation) 3 times in 5 minutes.

17. Access holes for periscopes, control wiring, tubing, etc. were to be provided in each cell as follows:

- a. 10 to 12 inch stepped holes.
- b. 1/2, 3/4, 1 and 2 inch "Zsa" tubes.
- c. 4 to 5 inch stepped holes.

An artist's conception of the area is shown in Figure 1.

Fig. 1—Artist's conception, Idaho multi-cell facility.

Figure 2 shows the over-all floor plan.

The first problem to be solved in approaching the final cell design was to decide upon the concrete density to be used in the shield walls. Calculations showed that the equivalent of 3 feet of magnetite concrete (with a density of 220 lbs/ft³) were required, and that an all-glass window made up of non-browning, protected lead and high density lead glass could

be constructed of equal thickness and shielding properties. Higher density concrete would permit a corresponding reduction in wall thickness but the windows would then project beyond the surface plane of the wall on at least one side. Use of normal density concrete would result in a thicker wall with high density inserts around the windows and save little cost. After balancing operating requirements and design simplicity against cost, the three-foot thickness of magnetite concrete was used. Two feet of magnetite concrete were used in the walls between cells, reducing the maximum radiation contribution from one cell to another to 100 mr/hr, and two feet of normal density concrete were used for the ceilings.

Fig. 2—Over-all floor plan, Idaho multi-cell facility.

A 5-ton monorail crane was installed in one straight run from the truck unloading area outside the building along the back of the cells and ending over the storage pool under the 30-ton crane. A hatchway back of Cell No. 1, immediately under the monorail, leads to the under floor tunnel and dolly system that will carry casks under the cell for unloading.

The change room is located along the only passageway from the contaminated to the clean area. Hand-wash fountains and a pass-through shower are available for personnel decontamination.

The hot tool area is provided with a double sink for decontamination of small tools and equipment needing maintenance or alterations.

The set-up area is sufficiently large to enable personnel to test run all new equipment before it is installed. It is anticipated that tool benches will be added there for maintenance work.

Floor drains are provided in all areas; all but the two in the control area lead to hot catch tanks.

Figure 3 shows the floor plan of Cell No. 4 (also typical of Cells 2 and 3). The intercell transfer drawer is operated from the control area by a hand wheel. The future-window liner is filled with dry ferro-phosphorus aggregate which can be easily removed for installation of a shielding window when required. The 12-1/2 ton shielding doors hung on two hinges provide a suitable way of entering the cells without the loss of any floor space when they are in the closed position.

The exhaust air filters in the cells are held to the ductwork by electro-magnets. Filters may be remotely changed with manipulators in a matter of minutes.

All of the cells, with the exception of No. 1, are provided with false floor supports and hot drains. After equipment is installed, the false floor will be fitted around it to bring the working level up to just below the viewing windows.

Figures 3 and 4 show plan and elevation sections of the final shield window design used in all cells. The glass slabs, all of which are oil-laminated, are made up as follows:

- Hot Side - One inch 2.7 density cover sheet of non-browning glass.
- Next - Nine inch 2.7 density slab of non-browning glass.
- Next - Nine inch 3.3 density slab of non-browning lead glass.
- Next - Nine inch 3.3 density slab of non-browning lead glass.
- Next - Seven inch 6.2 density high density lead glass.
- Cold Side - One inch 2.5 density cover sheet of lime plate glass.

The large steps at the window top are required to afford operators a clear view of the crane movements with respect to the mercury lights and manipulators.

Fig. 3—Floor plan of Cell No. 4.

Figure 4 shows a section through Cell No. 1. The cask on the dolly is so arranged that a manipulator can reach through the cell floor hatch and pick up radioactive materials. The control area in front of Cell No. 1 was designed to be 3' - 0" lower than that for the other three cells so the window could be

installed just above the cell floor. The master-slave manipulators used for other operations may also be used to load and unload casks. The control area width was set at a minimum of 13' - 6" to allow installation and removal of manipulators without difficulty.

The vacuum cleaning system has a simple latch on top of the filter housing so that it is possible to change the filter bags remotely with manipulators when required. The vacuum cleaning system consists of a vacuum producer and primary and secondary separators in the mechanical equipment room. Second stage decontamination will be effected by a central wash-down system supplied by a steam-operated jet pump which will feed hot water, with or without detergent additives, into spray in the ceiling of each cell.

One-half ton bridge cranes with bridge and trolley speeds of 9" per minute are provided in Cells 1 and 4. Each cell has four recessed incandescent lights in the ceilings in addition to three swivel-type mercury-arc spot lights, rated at 400 watts each, located around the viewing windows. Each cell has unistruts imbedded in the walls and ceiling to which equipment or shelving may be attached.

Fig. 4—Section through Cell No. 1.

Figure 5 shows a section through Cell No. 4 (also typical for Cells 2 and 3 except for the future window). This figure shows the height of the false floor with respect to the viewing windows. A drain will be built into each false floor extending to the existing drain below.

Three 400-watt mercury arc lights, of the type shown, were installed in each cell under the original contract and provisions were made for installation of two more if needed. The bulbs are of the flood type, with built-in reflector coating, and are supplied from ballast transformers.

Figure 6 shows a typical elevation of the hot cells from the control area side. Included in the services for the cells are plant and instrument air, hot and cold industrial water and demineralized water. Electrical services

consisting of 480-volt 3-phase 60-cycle AC, 115-volt 1-phase 60-cycle AC, 115-volt 1-phase 400-cycle AC and 28-volt DC are available on the face of the cells as well as at different points throughout the facility. The hot cells are provided with several spare holes, pipe sleeves and conduits.

The intercom set shown is typical of the four that connect the interior and exterior of each cell and are used both as a means of communication during installation and check-out of equipment and as sound monitors during operations. A second intercom (similar to a telephone in Figure 6) connects the control and set-up areas to the remainder of the existing building.

Fig. 5 — section through Cell No. 4.

The total floor area in this facility is approximately 4650 square feet, of which 182 are useful hot cell floor space. Although this appears to reflect an expensive ratio, it is believed that the space provided for hot tool and equipment storage, testing and maintenance work are very nearly as valuable to this type of operation as cell space itself.

The low bid for construction of the facility was \$469,100. An additional sum of approximately \$5,000 was spent for changes and additions after construction started, bringing the total to \$474,100. A sum of \$32,000 was spent for General Electric furnished equipment, bringing the construction total to \$506,100. This is approximately \$108.80 per square foot. It should be noted that these figures do not include Architect-Engineer fees. Several cost breakdowns that may be of interest follow:

1. Shield windows approximately \$15,000 each.
2. Shield doors approximately \$4,000 each.

3. Magnetite concrete approximately \$250/yard (in place).
4. Normal concrete approximately \$85/yard (in place).
5. Mercury lights approximately \$60 each including ballast.
6. Manipulators with electrically-powered side motion approximately \$4,950 each.
7. Intercell transfer drawers approximately \$525 each.
8. Remote-controlled underfloor dolly system approximately \$1,275.
9. Lead plugs to fill spare holes approximately \$2,550.

Fig. 6—Typical elevation of the hot cells from the control area side.

Density Measurement Problems in Hot Cells*

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Abstract

Problems associated with the precise determination of densities in hot cells are considered in connection with pycnometric and loss of weight in liquid methods. The latter technique involves problems of vibration and temperature control, radiation resistance of balance components, drafts and disturbances due to cell ventilation, access for maintenance, and ease of operation by master slave manipulators. The immersion of radioactive specimens in a liquid requires further consideration of radiation stability and radioinduced heating of the liquid, thermal instabilities, precise temperature measurement, and appropriate sling design. Means are presented for reducing or correcting these sources of error. A special hot cell design is described which eliminates many of these errors.

Introduction

An important factor in the development of improved reactor fuel and structural materials is an understanding of mechanisms producing radiation-induced volume changes. Experimental data obtained for the purpose of examining such effects on many types of material and under varying reactor conditions are increasing at a rapid rate, and more sophisticated techniques are being required from hot laboratory facilities to detect and measure these phenomena. One of the more sensitive and convenient methods for obtaining such information is by measurement of specimen density. (a)

Extensive information is available in the literature (b) on classical

*This paper may also be identified as Report WAPD-T-670.

- (a) The density of a gross sample obtained by hydrostatic, pycnometric, or flotation techniques provides no useful information on localized volume changes within the sample itself. Such local changes can be obtained by direct measurement with a micrometer or profilometer, and data at the submacroscopic level can be obtained by X-ray-diffraction techniques.
- (b) For example: P. Hidnert and E. L. Peffer, Density of Solids and Liquids, NBS Circular 487.
J. Reilly and W. N. Rae, Physics-Chemical Methods, Vol. 1, D. VanNostrand Co., Inc., 1953.
A wealth of specialized applications and methods of interest are found in Chemical Abstracts and Nuclear Science Abstracts.

methods and techniques for the determination of density. This discussion is limited to a consideration of the so-called hydrostatic (loss-of-weight-in liquid) and pycnometric methods, emphasizing techniques that are particularly

Fig. 1—Weld pycnometers and test specimens.

affected by the radioactive nature of the specimen, and by conditions of the hot laboratory environment. It should be mentioned that important work is now being done at many laboratories in applying flotation methods to radioactive materials, particularly porous, irregularly shaped samples such as UO_2 .

This discussion is guided by the following general objectives: (1) accuracies in the order of 0.01 to 0.001 gr/cc are desired for densities

in the range of 8 to 10 gr/cc; (2) methods employed must be rapid and within the skills of the operating staff; (3) techniques must be conveniently adaptable to hot laboratory conditions requiring remote manipulation and lowest possible personnel exposure.

Pycnometric Method

The pycnometric approach to density determination is attractive from a number of standpoints. The apparent simplicity of the operations requiring remote manipulation, the elimination of "wet weights" and associated attention to exacting experimental technique required in hydrostatic weighing, and the elimination of error due to non-uniform heating of the liquid by the radioactive specimen, are special advantages of this method. An effort was made, therefore, to evaluate this system from the standpoint of obtainable accuracy and adaptability to hot cell manipulation.

Initially, an out-of-cell evaluation was made by comparing the results obtained from special non-radioactive samples with the values determined by calibration against an NBS standard. The samples were precision ground to dimensions of 1" x 1/4" x 1/8", all dimensions $\pm .001$ ". The volume of each sample was then $.0312 \pm .0004$ in³. Two Weld pycnometers of 10 ml. capacity were used. (Fig. 1) The density obtained for sample #1 by NBS calibration was $6.544 \pm .001$ gr/cc at 25°C. The average of 13 determinations, corrected for air buoyancy and adjusted to 25°C was 6.529 gr/cc with $\sigma = 0.051$. Results from the other samples generally yielded similar results. The comparatively poor precision obtained from the pycnometric determination is attributed to (1) uncertainties in the uniformity of water temperature; (2) varying water meniscus at the overflow plug; (3) difficulty in drying the pycnometer while maintaining constant volume, particularly on the surfaces within the evaporation cap but outside of the overflow plug.

An evaluation of the in-cell handling convenience of the method revealed several difficulties associated with pycnometer design. The meniscus was particularly difficult to control, even after the addition of wetting agent. Unfortunately, no special equipment was available to maintain water temperature, while in-cell ambient temperatures varied from 18 to 24°C. Since direct temperature measurement of the water in the pycnometer is difficult, considerable doubt existed concerning the water density at the time of observation. Conventional pycnometer designs do not readily accommodate specimens of widely varying size and geometry, a serious disadvantage from the standpoint of efficient cell utilization. The precision and accuracy of results obtained on the same test specimens in cell was found to be particularly sensitive to the ability and techniques of different operators.

It is felt that most of the sources of experimental error and operational disadvantages mentioned can be eliminated by a more suitable pycnometer design. Such a design is presently underway at this laboratory.

Hydrostatic Method

The most widely used method for the determination of density is that due to Archimedes. Apparent or relative densities are obtained directly, and these may be corrected for air buoyancy and adjusted to a standard temperature (usually 25°C) if desired. The balance may be placed directly in the hot cell, or a special facility may be designed to place the balance outside of a shielded enclosure in which the specimen is suspended by a suitable wire.

Balances

A wide choice of balance designs are presently available on the American market. Two-pan equal arm balances with chain-type fractional weights may

be readily adapted to in-cell applications where wide temperature extremes are especially troublesome. For general uses, these are not as rapid or convenient as the single-pan substitution type balances where no weight manipulation of any kind is required. Torsion balances are sometimes used for special applications, and electric balances are now being offered which seem particularly applicable in the micro-range.

This discussion will be concerned primarily with the single-pan substitution design. (Fig. 2) Most of the considerations given to this design are also applicable to the equal-arm two-pan design.

(A) Radiation Effects

The elimination of weight handling is usually accomplished by the use of an optical system which should be made of non-browning glass. Some balances are now being supplied with non-browning optics as standard equipment. No undesirable effects have yet been experienced in this laboratory from irradiation of knife edges, bearing surfaces, or binder materials. Synthetic sapphire tends to brown after extended irradiation, but one set of knife edges tested showed a tendency to increase in hardness. It has generally been found convenient to replace plate glass surrounding the weighing compartment with plexiglass. This material is somewhat more resistant to the many types of in-cell impacts that can occur, and is somewhat more resistant to radiation browning.

(B) Temperature Effects and Drafts

Most hot cells are ventilated with fairly high-velocity unconditioned air which produces undesirable temperature variations and drafts. Usually the temperature-time gradient is not unmanageable if frequent checks of balance zero are made. However, under severe diurnal and seasonal conditions, zero drifts of 2 to 5 mg/hr have been experienced at this laboratory. Drafts can prove to be particularly troublesome with balances using air-damped beams that are not properly adjusted. Under severe conditions when adjustment of the damping vent is not sufficient, isolation boxes must be employed. Magnetically damped beams are not sensitive to draft disturbances, and this consideration may be important in the selection of balance design.

Drifting of the balance from the presence of thermally hot radioactive specimens on the weighing pan is a particularly difficult problem when the sample is large and has experienced high burnup. A canopy type baffle in the weighing compartment, carefully designed to eliminate rubbing against pan or suspension wire can be effective in reducing this effect. In extreme cases it may be necessary to wait for decay of the sample and lowered surface temperatures.

Direct heating of the balance by cell lighting or other sources may be reduced by refinishing with a suitable reflective surface.

(C) Vibration

No single method for the isolation or absorption of vibration can be suggested that will be effective for all situations. The frequencies and amplitudes present should be analyzed (c) and mounting designed whose natural frequency is widely different from the source frequency. (Appendix B) A balance table designed for frequencies in this laboratory due to cell

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- (c) Many industrial concerns are equipped to perform an analysis of frequencies present and to suggest methods for isolating the sources. Survey equipment is also available at moderate cost, for example, Consolidated Electrodynamics Corp. Model 1-128.

ventilating equipment is shown in Fig. 3. This arrangement is universally used rather than a table fixed to a main building member, for purposes of convenience when the table must be moved. A 24" x 32" piece of 1" steel plate is placed on 4 number 15 rubber stoppers. A suitable number of concrete blocks are placed on this plate to bring the table to a convenient height. Another 1" steel plate is placed on top of the concrete block. A 4" steel slab "floats" on the upper 1" plate with special isolation rubber pads. (d) This arrangement has eliminated vibration due to 1950 rpm electric motors with about 96% efficiency.

Techniques for the Determination of Wet Weights

A container fabricated of plexiglass has been found convenient for the liquid in which the specimen is immersed. This container should be as large as possible to minimize rubbing between the sample and container wall, and also to provide the largest possible volume of liquid. The sample is suspended from the beam by a wire and sling of corrosion resistant material such as platinum, nickel, molybdenum, or nichrome, depending on the immersing liquid used. It is sometimes desirable to oxidize the portion of the wire penetrating the liquid surface in order to reduce spurious surface tension effects. The application of lamp black in a thin solution of binder is also effective. Whereas the wet weight of the sample is obtained by subtracting the wet weight of the sling alone from that of the sample and sling, surface tension effects are evident when the magnitude of the force is not constant at the time of each weighing. The sample may be attached to the suspending wire in a sling, cradle, cup or other means convenient to the sample geometry. In general, it is desirable to use a sling design that will not trap air bubbles, and which is as light as possible. A convenient method for remote manipulation consists of an alligator-clip arrangement which is modified according to the previous considerations. This method provides a positive grip on the sample, is very easy to load remotely, and has been used with very favorable results on all types of sample sizes and geometries.

The liquid container may be provided with an elevator to lift the liquid up into the sample after it has been suspended from the beam hook. This technique was employed for some time, but later was abandoned when the proficiency of the operating staff during the loading operation obviated its need. Without the elevator it is simpler to convert the balance from a dry to wet weight configuration remotely. The addition of plastic mirrors to the floor and rear wall of the weighing compartment assists in making this conversion.

Liquids

The choice of immersing liquid is determined by the chemical reactivity with the material under study, stability under intense γ fields, the availability of precise temperature-density data, and ease of handling. Widely used liquids fulfilling these requirements are water, carbon tetrachloride, toluene, 1-octanol, bromobenzene, and ethylene dibromide. The latter two chemicals offer attractive possibilities for increased accuracies due to their high densities, but little information has been reported on their stability under γ irradiation. Where chemical reactions are not a factor, water is the most commonly used liquid. It is extremely important that freshly distilled water be used in determinations where accuracies greater than 0.01 gr/cc are desired. The tendency for air bubbles to form on the surface of the specimen, aggravated by the production of hydrogen and oxygen from radiolytic decomposition, produces serious errors, particularly with specimens of poor surface quality or irregular shape. The addition of wetting agent to the water will help to minimize this effect. The improved wetting of water with the addition of

(d) MB Manufacturing Co. Isomode Pad. This material is loaded to 50 lbs/in².

Fig. 2—Typical balance modified for remote operation. Standard controls on weight selectors, zeroing prism, and pan release are replaced with more suitable remote knobs. Glass windows are replaced with Plexiglas. Handle on balance cover facilitates remote beam taring when required.

Fig. 3—Balance isolation stand.

dioctyl sodium sulfosuccinate (e) may be inferred from measurements made of the tension required to pull a constant diameter loop of wire free from a water surface as a function of aerosol concentration. (Fig. 4) The addition of aerosol beyond .1% produces no appreciable increase in wetting. It can be shown that the density of water containing .1% aerosol is not significantly altered from that given in standard density tables. (See Appendix A)

A serious source of error inherent with radioactive specimens is that due to heating of the liquid in contact with a thermally hot surface produced by decay energy. (Appendix C) The magnitude of this error is dependent on specimen composition, irradiation history, and time since discharge. The magnitude of this energy release can be estimated from available data on fission product decay. (f) This information may be used with known experimental conditions to determine a correction factor for this effect. (g) For specimens of low activity it is possible to reduce heating errors by using water as the immersing liquid and cooling it, in conjunction with thorough mixing, to 4°C, where density is relatively insensitive to temperature variations. It is presently assumed that direct heating of the water by gamma absorption is negligible compared to other errors discussed previously.

Special Density Cell

A large number of the problems discussed can be directly attributed to the operation of balances in the unfavorable hot cell environment. Other laboratories have recognized the difficulties associated with such an arrangement, and special purpose cells, such as that described by Mallett (h), have been proposed. A somewhat similar arrangement is shown in Fig. 5. The balance is located on top of a shielded enclosure and the entire unit is located in a room where ambient temperature and humidity is accurately controlled and drafts are eliminated. The balance is not subjected to radiation, is available for direct maintenance when necessary, and need not be modified for remote manipulation. The room is comparatively free of vibration, but the balance is set on a steel slab which "floats" on the top of the shielded enclosure. Specimens are transferred into the cell in small pigs which are lifted into the cell by a winch arrangement shown. The enclosure is normally ventilated by a household vacuum cleaner which can be equipped with a filter suitable for trapping airborne contamination. The comparatively simple operation of transferring a specimen from the transfer pig to the sling can be accomplished by a simple manipulator. The floor of the enclosure is sloped in such a way that a dropped specimen will tend to fall or roll away from inaccessible corners towards the center of the base. The liquid container is larger than that which can be accommodated in a balance weighing compartment, and is equipped with an electric stirrer and thermometer. The container can be filled or drained by gravity feed through tygon tubing to the outside. A small portable control panel is provided for control of the winch, electric stirrer, lighting, and a motor driven jack used for lifting the transfer cask to the range of the winch. For determinations where high accuracy is required, an equal arm balance is employed with the unknown suspended from one arm and an accurately known unirradiated control is suspended from the other. Only small differences are measured which usually require balancing by the chain alone. For other applications, a substitution type balance may be used with equal facility.

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- (e) Sold in 10% water solutions as Laboratory Aerosol by Fisher Scientific Corp
 - (f) C. D. Coryell and N. Sugarman, Div. IV, Vol. 9, Book 1, Radiochemical Studies: The Fission Products
 - (g) General methods for determining these corrections are the subject of a report in preparation.
 - (h) G. R. Mallett, HW-34079 (Rev.), Dec. 10, 1954, (UNCLASSIFIED)

Fig. 4—Wetting and surface tension of various metals in aerosol-water solution.

Fig. 5—Controlled-environment density cell.

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A P P E N D I X A

The apparent density for hydrostatic weighing is given by

$$\rho = \frac{W_a \rho_l}{W_a - W_l} \quad (1)$$

where ρ_l = density of immersing liquid

W_a = weight of sample in air

W_l = weight of sample in liquid

For equal arm balances, neglecting errors due to non-symmetrical geometry from the liquid bath, it can be shown that the true density, ρ_t , corrected for air buoyancy is given by

$$\rho_t = \rho \left[1 - \frac{\rho_a}{\rho_l} \frac{W_l}{W_a} \right] \quad (2)$$

where ρ = apparent density

ρ_a = air density at a given temperature and barometric pressure.

Since ρ_a/ρ_l is in the order of 10^{-3} for the more common liquids, it is possible to substitute $W_a \cong \rho V$ and $W_l \cong V(\rho - \rho_l)$ in equation (2), provided accuracies greater than .002 gr/cc are not required.

In this case

$$\rho_t = \rho \left[1 - \frac{\rho_a}{\rho_l} \left(1 - \frac{\rho_l}{\rho} \right) \right] \quad (3)$$

For unequal arm balances, the above equations hold true provided a suitable correction factor for the asymmetrical geometry is added. This correction factor is independent of the weights on the pan(s), and can be supplied by the particular manufacturer, or can be determined experimentally from known standards. (i)

(i) To correct W_a for air buoyancy, $W_{at} = W_a + K$. For the Mettler Model B5,

$$K = d \left[\frac{1}{d_2} - \frac{1}{d_1} \right] + 0.0130$$

where $d = \rho_a$, the air density at given T and P

d_1 = density of balance weights

d_2 = density of material weighed

Adjustment of the density from room temperature to any other temperature may be made in the usual way, knowing the volume coefficient of the material.

The addition of 0.1% dioctyl sodium sulfosuccinate to the water produces a negligible density change. A 10% water solution of this wetting agent has a specific gravity of 1.0024 at room temperature. The addition of 1 cc of this solution to 100 cc of water (.1% concentration) at 23°C yields approximately 101 cc of solution weighing 100.7558 gr., or a density of .997582 gr/cc. The change in density due to "aerosol" addition is therefore 0.000044 gr/cc at this temperature.

Differentiating equation (1) with respect to the weighed quantities only gives the absolute uncertainty in ρ

$$d\rho_e = \rho_e \frac{W_a dW_e - W_e dW_a}{(W_a - W_e)^2} \quad (4)$$

For radioactive samples, in general, $dW_e \neq dW_a$ because of heating of the liquid in contact with a thermally hot specimen surface. This effect should be taken into account, as well as thermal convection in the liquid due to possible non-isotropic thermal (density) distributions, when estimating dW_e . (See Appendix C) The convection effect is difficult to estimate because of the many parameters involved (g), however, it should be pointed out that with specimens of moderate activity, the magnitude may be minimized to a negligible degree by stirring or otherwise maintaining a uniform temperature distribution before and after each weighing.

For specimens of low activity, where the heating effect is not significant, or in border-line cases where it may be reduced to insignificance by proper experimental technique, $dW_a = dW_e = dW$ and equation (4) reduces to

$$d\rho = \frac{\rho}{W_a} dW \quad (5)$$

where, following previous notation, ρ is the density of the specimen, and dW is the total estimated error in weighing under the experimental conditions.

Differentiating equation (1) with respect to the density of the liquid only gives

$$d\rho = \frac{W_a}{W_a - W_e} d\rho_e \quad (6)$$

For $d\rho_e \leq .0001$ gr/cc at 20°C, the temperature of water, or the error in its measurement must not exceed $\pm 0.5^\circ\text{C}$, whereas at 4°C , an error of $\pm 3.5^\circ\text{C}$ will yield a value of ρ_e that does not change in the fourth decimal place. This wider latitude exists because the cubic coefficient for water contains significant terms in t^3 (for $d\rho_e \leq .0001$ gr/cc) with a maximum at about 4°C . This property of water may be significant when high accuracies are required for radioactive specimens that would otherwise produce unmeasurable density gradients near the water-surface interface. (See Appendix C)

A P P E N D I X B

The disturbing frequency should be measured at the location where it is desired to isolate the balance. If a vibration pickup is not available, it is sufficiently accurate to assume that the disturbing frequency is equal to that generated by the source. The disturbing frequency of a rotating machine, for example, is equal to the rpm of the motor. For most purposes, an isolation efficiency of 96% or a transmissibility of 4% is desired where balances are read to 0.1 mg. The transmissibility of an isolation mount is defined as

$$T(\%) = \frac{100}{1 - \left(\frac{\nu_d}{\nu_m}\right)^2} \quad (7)$$

where ν_d = disturbing frequency of source

ν_m = natural frequency of the isolation mount and balance

Equation (7) shows that for 4% transmissibility, ν_d/ν_m should be approximately 5. The natural frequency required for any type of isolation device is thus established directly from the disturbing frequency and the efficiency desired. In cases where the natural frequency of a particular design may not be easily calculated, it may be determined by direct measurement of the static deflection of the mount, D , by

$$\nu_m = \frac{188}{\sqrt{D}} \quad (8)$$

where D is in inches and

ν_m is in cycles per minute

Isolating systems using a rubber-in-shear design are generally more effective than those employing rubber-in-compression, however, the latter method has substantially greater resistance to shock or additional loads. This factor was considered important in selecting the design shown in Fig. 3 for in-cell use.

The weight loading of the special rubber isolating pads determines the static deflection and thus the natural frequency (Equation 8) of the design. This relationship can be established by the manufacturer of the particular material.

A P P E N D I X C

For a given time t after the slow neutron fission of U^{235} , the rate of γ energy release per fission can be given in the form

$$\Gamma(t) = A_t t^{B_t} \quad \text{Mev/sec/fission} \quad (9)$$

where $\Gamma(t)$ is a piece-wise continuous function with values of A_t and B_t defined for closed time intervals from 0 to $t \rightarrow \infty$ sec. A continuous curve form for equation (9) is

$$\Gamma(t) = \sum_{j=1}^n A_j e^{-\lambda_j t} \quad (10)$$

Integration of equations (9) and (10) for $0 \leq t \leq \infty$ are in good agreement. (f)

If K is the average number of fissions/cm³-sec during a reactor operating time T_0 , then

$$K = 3.1 \times 10^{10} \left(\frac{P}{V} \right) \quad (11)$$

where P = power in watts of specimen

V = active volume of specimen in cm³

The rate of energy release at a time t after fission is

$$\Gamma(t) K dt \quad \text{Mev/cm}^3\text{-sec} \quad (12)$$

and the rate of γ energy release is

$$E_\gamma(T_0, T_S) = K \int_{T_S}^{T_0+T_S} 1.6 t^{-1.2} dt \quad (13)$$

where T_S is the time after shutdown.

For $10 \leq T_S \leq \infty$, and from equation (9)

$$E_\gamma(T_0, T_S) = K \int_{T_S}^{T_0+T_S} 1.6 t^{-1.2} dt \quad (14)$$

where the values of A_t and B_t have been fit from the decay curve. (f)

Integrating equation (14)

$$E_\gamma(T_0, T_S) = K \left[-\frac{1.6}{.21} t^{-.21} \right]_{T_S}^{T_0+T_S}$$

or

$$E_{\gamma}(T_0, T_s) = -7.62 K \left[\frac{1}{t^{.21}} \right]_{T_s}^{T_0+T_s} \quad (15)$$

For $T_0 \rightarrow \infty$

$$E_{\gamma}(\infty, T_s) = \frac{7.62 K}{T_s^{.21}} \quad (16)$$

The rate of γ energy release as a function of T_0 and T_s is shown in Fig. 6.

It has been shown (j) that β energy release is equal in magnitude to E_{γ} , therefore the total energy release is

$$E_T = E_{\gamma} + E_{\beta} = 2E_{\gamma} \quad (17)$$

Energy from delayed neutrons is significant only during the first few seconds after shutdown.

Thus the total energy being released from a radioactive specimen may be estimated after determining the power produced by the specimen during T_0 . This information is frequently available from the operating records of the in-pile experiment, or may be deduced from radiochemical analysis. Estimates of various specimens determined in this laboratory give values ranging from 25 to 150 watts/cm² of specimen surface.

(j) K. Way and E. P. Wigner, Phys. Rev. 73, 1318 (1948)

Fig. 6—Gamma energy release from slow neutron fission of U^{235} as a function of time in reactor and time since shutdown.

Bettis Hot Laboratory Operations

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The physical design of the Bettis Hot Lab has been discussed at several earlier meetings; however, in order to indicate some of our operating procedures, I will briefly review the layout of the lab. Figure 1 - Basically it consists of eight high level cells arranged in two blocks as shown. The five cells in the west cell block are each 11 feet long by 6 feet deep. Each cell is an individual operating cubicle with access being provided by movable barriers in the upper part of the cell. The three cells in the east cell block are each 6 feet long by 5 feet deep, however, floor to ceiling movable barriers allow operating them as one cell over 20 feet long. Other design features such as heavier shielding and a full width movable barrier at one end of the block provide much better operating characteristics in our newer cells. We operate the lab on a clean control area and a contaminated loading area basis with the two areas being separated by a change room.

Support facilities for the high level cells consist of an intermediate level metallographic cell, an Argonne type junior cave, six shielded autoclaves, a conveyor type dry storage unit, a water pit, a decontamination room, a hot waste storage pit, a machine shop and a tool room all located in the contaminated area. Associated with the high level hot lab is an intermediate level radiochemistry lab with access through the loading area and a transfer port from the west cell block. Located in the clean area are a dark room and a slave repair area.

The laboratory operates as a service organization on a two-shift basis. The organization is as follows: Figure 2 - Overall responsibility for all the hot lab functions lies with the hot lab supervisor. Under him, a foreman directs operations on the first shift and a supervisory engineer on the second shift. In addition, reporting directly to the hot lab supervisor are three operating engineers each of whom coordinates and directs the hot laboratory work of one or more of the Bettis projects. Also reporting directly to the supervisor are two engineers responsible for the design and construction of new facilities and equipment plus one engineer responsible for maintenance. Indirectly under hot lab supervision are two production clerks who assist in planning and scheduling hot lab operations, two industrial hygiene technicians who survey contamination and activity levels in the laboratory, and the technician in charge of the metallography cell. The hot lab technicians on each shift are divided into two groups

Fig. 1—Floor plan of the Bettis Hot Laboratory.

Fig. 2—Bettis Hot Laboratory organization chart.

designated as support activities and cell operations. Support activities include such items as operation of the autoclaves, the dry storage facility, accountability, general maintenance, etc. Cell operations include setup, operation and removal of tests as well as maintenance of the cells and the cell equipment. Each group is headed by a group leader. Due to varying personnel exposures, there is usually some interchange between the groups and each technician is required to have a variety of skills.

Work is initiated in the hot laboratory by means of a standard form work request issued by one of the Bettis projects. The work request is reviewed by the hot lab supervisor who may suggest changes in order to facilitate handling in the lab. After the work request has been approved, it is processed by the production clerks who include it in their schedules and forward copies to the proper operating engineer as well as the foreman. Each week the production clerks make up a tentative schedule based on outstanding work requests. The schedule is reviewed by the hot lab supervisor and engineers and revised where necessary. It is then issued to the projects, so that their engineers can personally follow the tests if they so desire. This procedure provides for operation of the hot lab by specially trained technicians in a systematic manner and yet allows for ample control of testing procedures by project engineers.

Some Safety Recommendations*

Pertinent to Hot Laboratories

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INTRODUCTION

Since Brookhaven National Laboratory was formed in 1947, the Laboratory, the Nuclear Engineering Department, the Hot Laboratory Division, and other groups at the Laboratory have had continuing safety programs. Nevertheless in spite of this there occurred on May 15, 1957, in the "Hot Area" of the Hot Laboratory, an accident which attracted wide-spread attention. Because of the presence of a large number of press reporters who were covering an Inter-American Symposium at the time, the incident received far more publicity than it deserved.

The purpose of this paper is to point out and discuss the recommendations, some of which were rather novel, which grew out of a study of this accident. This paper will describe the occurrence, particularly with respect to the behavior of people during and following it. The paper will not deal with the direct technical causes nor with the damage to the plant itself, except to point out very briefly for background purposes the large number of safety devices employed in this plant and the high degree of safety consciousness and numerous precautions which went into the design and operation of the equipment in question.

INCIDENT OF MAY, 1957

On Wednesday, May 15, 1957, at approximately 1:45 P. M. a series of about five explosions in rapid sequence occurred in a dissolving vessel in which uranium was being dissolved in bromine trifluoride in one of a series of pilot plant runs being conducted by the "Volatility Project" as part of the study of the non-aqueous dissolution and re-purification of uranium metal. In the so called fluoride volatility process the metal is fluorinated by an interhalogen mixture consisting principally of bromine trifluoride with lesser amounts of bromine pentafluoride, uranium hexafluoride and bromine. The uranium is fluorinated to the hexavalent fluoride state and as such is separated from the fission products by distillation. This particular plant was a continuous one rather than a batchwise one and had provisions for adding new uranium metal

*This paper may also be identified as Report BNL-3641.

while the dissolving step was proceeding. The plant had operated very satisfactorily during the eight dissolution runs which preceded the one in question. Although the plant was later to have been used with irradiated uranium containing fission products and ultimately with uranium containing plutonium, all previous runs and the present run involved only unirradiated uranium.

The plant was fully instrumented. Temperature and pressures at a large number of strategic points throughout the system were indicated and recorded at a control panel which also indicated or recorded other operating variables such as current and voltage to pump motors, liquid flow rates, liquid levels, high pressure, high temperature, position of critical valves, etc. In addition there was a system of two 3000-gallon tanks located outside of the building and connected to various parts of the plant by rupture discs, the purpose of which system was to accommodate unpredicted rapid surges in pressure.

In addition to all of these safety precautions there was designed, installed, and operating at the time a rather unique "slug feeler", the purpose of which was to detect the height of the uppermost piece of uranium metal in the dissolver so that by comparing this height with the measured liquid level in the dissolver it could be determined how much liquid was covering the metal and whether or not any metal was exposed above the surface of the liquid. This was considered necessary for it was known from the outset that, whereas the reaction between uranium metal and liquid bromine trifluoride could be smoothly controlled, the reaction between uranium metal and gaseous bromine trifluoride is often uncontrollably rapid due to the poorer heat transfer in the gas phase with the result that the exothermic heat of reaction is not dissipated sufficiently rapidly. This slug feeler consisted of a series of closely spaced concentric wire rings attached to the lower end of a rod which could be run down to touch the top of the metal, the position of the rod then being read from outside the vessel. Although it cannot be proven, it appears quite likely that this safety device may have caused the explosion by hooking onto a piece of partially dissolved uranium and then withdrawing it into the vapor phase when the rod was retracted after the last reading just prior to the accident.

With the exception of some valves which were installed with bolted flanges, all joints were Heliarc welded and all welds were radiographed.

The point to be emphasized here is the very high degree of safety consciousness which went into the design, construction, and operation of the plant from the very beginning up through the time of the accident. The design, construction, and operation of the plant were reviewed from a safety standpoint several times both by a committee and by a consultant.

The plant was located in the northwest corner of the "Semi-Works Area" of the Hot Laboratory, see Figure 1.

On the afternoon of May 15, 1957, the ninth dissolving run was proceeding in a routine manner until approximately 1:45 P. M. - one or two minutes after the slug height reading was taken - when a series of about five explosions occurred in this plant. The events inside the dissolver occurred so rapidly that no visible or audible warning of an abnormal process condition was sensed by any of the instruments. Although the sound in the Hot Area was sharp and intense, in the remainder of the building it was heard as a muffled sound best described as "trash cans rolling down stairs" or "jets penetrating the sound barrier".

Fig. 1 — Plan of BNL Hot Laboratory.

The most universal initial response with people in the "Cold Area" was that of mild curiosity. This response, however, rapidly gave way to general confusion and in some cases consternation when it became obvious that the sound was due to explosions which released bromine trifluoride from the Volatility Project pilot plant.

The most common first reaction was to get out of the building and away from the Hot Area. However, there were many whose first thought was of the safety of those involved in the accident and who, therefore, reacted by attempting to enter the Hot Area or otherwise caring for those who had already come out of the Hot Area. In spite of the confusion many individuals reacted in an intelligent and coordinated manner.

At the time of the explosion there were nine people in the Hot Area and forty-eight people in the Cold Area. Of those in the Hot Area there were four in the Semi-Works Area, one near Hot Cell No. 3, one in the Gamma Irradiation Facility, one in Semi-Hot Lab No. 5, and two in Semi-Hot Lab No. 4. No other parts of the Hot Area were occupied at the time. Of those in the Cold Area there were seven in the Machine Shop and two in the Monitor Station. Two "local emergency wardens" were in the building at the time on the ground floor while three others together with the local emergency supervisor and the deputy local emergency supervisor were all out of the building.

Time and space do not permit summarizing the actions of everyone during and following the accident but it will be of interest to note the exit paths of those in the Hot Area. Only one man was injured sufficiently to require hospital confinement. (He recovered completely after being hospitalized for one month as a result of burns from the liquid bromine trifluoride). At the time of the accident this man headed east past the pilot plant control panels, turned right, (south) went straight out door H-105, ran along the ground level and up the stairs at the Gas Cylinder Storage Room, ran into the building through door C-241 but apparently turned around and came out this same door almost immediately. By this time he had his shirt and undershirt ripped off with the assistance of several others, and at this point he got into a private car which was parked just back of the Process Development Lab and was driven to the Clinic. It may be noted here that in following this path he passed by two safety showers in the Hot Area, past the outside of the Monitor Station which has a safety shower in it, and was thought by others to be heading for the showers in the Men's Locker Room at the east end of the building, although he presumably changed his mind upon reaching door C-241.

Two others ran east past the hot cells ahead of the first man, turned left, then right, and went into the Monitor Station from where one of them went to the showers in the Men's Locker Room. A fourth took the same route as the first but upon reaching the Gas Cylinder Storage Room he turned in the house fire alarm which is located just west of door C-242 and then helped the first man in removing his shirts. (Note here that in following this path, the fourth man passed directly by a fire alarm box located just inside door H-107.)

The fifth man (who was knocked down by the explosion, as were also two of the previous men) crawled on his hands and knees to door H-103, ran around the northwest corner of the building presumably making use of a ladder to get around a fence, ran east along the front of the building into the Lobby and up to the cold corridor, west to the Monitor Station where he reported that everyone had escaped, then

back east along the cold corridor to the Men's Locker Room where he took a shower.

The man in the Gamma Irradiation Facility ran east through door H-125, turned right and out door H-105, turned left and ran east to the Gas Cylinder Storage Room. The three men who were nearly trapped in Semi-Hot Labs 4 and 5 managed to exit apparently without difficulty, through the Alpha Facility by forcing open door H-152. From the Alpha Facility two of them went out through door H-107 and ran east while the third ran through the Monitor Station and then together with another man attempted to re-enter the Hot Area through door C-227 in order to rescue the first man; however, this re-entry was unsuccessful since the Hot Area very rapidly became completely full of dense amber fumes of bromine and bromine trifluoride - in fact so dense that the overhead lights could not be seen and the area appeared to have suffered a power failure. As exterior doors were opened these fumes rolled out and were swept towards the Cold Area and the parking lot by a brisk southwest wind.

It apparently was determined within a matter of minutes that everyone had escaped, although it took some several minutes before this word was finally passed all around, and during this period there was considerable concern over the whereabouts of the first man. Apparently while he was being taken to the Clinic a doctor who had been summoned from the Clinic was already on his way to the Hot Lab; those who escorted this doctor knew that a man had been injured but did not know that he had been taken to the Clinic until this point was cleared up by inquiring via a two-way car radio used by the Site Police.

The firemen arrived very quickly followed very shortly by a number of supervisors. The first step taken was to open as many exterior doors as possible in order to allow the wind to blow the fumes out. This was not only desirable in itself but was necessary in order to check and make absolutely certain that no one was left inside. Using Chemox masks the firemen succeeded in opening the large roll-up door (H-158) by entering the building through the adjacent personnel door (H-103). An attempt was made to turn on the two auxiliary roof fans in order to clear the Hot Area more rapidly of fumes but it was not possible to do so until about fifteen minutes after the explosion because of the fact that both areas in which the two parallel sets of manual switches are located were filled with dense fumes.

As soon as the area began to clear somewhat of fumes the overhead lights started to become visible and it became apparent that there was a small fire burning at the instrument panel board where some of the BrF_3 liquid had splashed. The firemen extinguished this in about five minutes using CO_2 extinguishers from the fire truck.

Shortly after the explosion the electric power to the pilot plant was cut off using a master switch in the Electrical Equipment Room on the ground floor but it was decided that it would serve no useful purpose and might simply be a nuisance if other power were shut off, particularly the lighting circuits. Therefore the remaining power was intentionally left on. As soon as the Hot Area had started to clear and the fire had been extinguished, the Cold Area was checked and doors and windows were opened to allow cross ventilation to sweep out fumes which had backed in from the Hot Area.

A discussion of the physical damage resulting from the accident is not pertinent to this paper except to indicate an order of magnitude. Damage to the building itself and to general equipment located therein,

exclusive of the Volatility Project pilot plant itself, ran to about \$8,000 worth of replacement parts and supplies and somewhere between 4,000 and 4,500 man-hours involved in general cleanup and repair of instruments, and so on. About \$850 worth of damage occurred to nine personal automobiles parked near the scene of the accident. Some temporary (but no permanent) damage was noted on several dozen trees several hundred feet from the accident in the downwind path of the fumes.

The point of the foregoing description is to illustrate generally how even the best designed plant complete with numerous safety devices can turn a peaceful spring afternoon into a state of confusion and to illustrate in particular the reasons for the recommendations which will now be discussed below.

DISCUSSION AND RECOMMENDATIONS

The Plant Itself

It is strongly felt that in the case of this particular plant no one could have foreseen the possible hazards or taken precautionary steps to prevent them in a better manner or to any greater degree than was taken by the people involved with the design, construction, and operation of this plant. The meticulous attention paid to details both large and small is a matter of record. It is felt that the fact that the accident occurred simply proves that it is impossible to foresee every last possible hazard in this kind of an operation. This being the case it follows that in the future no one will be able to guarantee that all possible hazards will have been considered and sufficient precautions taken to prevent them. Therefore, it also follows that experiments of the type carried on in this plant involving explosion hazard accompanied by release of highly corrosive chemicals and/or contamination should be completely isolated from all other work either by suitably designed pressure-tight buildings or by distance or both. Accordingly this pilot plant has been dismantled and removed from the Hot Lab. If reassembled it will be housed in its own building.

Safety Showers

Obviously an adequate number of safety showers suitably located inside a building is a safety requirement which has long been well recognized. This accident did not point up any lack of such interior showers, at least not in the part of the building which was directly affected by the accident, although it did call attention to the fact that additional showers are desirable in the Cold Area for reasons mentioned below. However, the accident did reveal certain other deficiencies.

In looking at the behavior of people immediately following the accident certain patterns of behavior stand out clearly. In an accident of this sort in which dangerous fumes are being released the overwhelming tendency is to run as far away from the scene as possible and as fast as possible first and then to take a shower. This has two implications: With regard to interior showers these should be located all over the building and not just in laboratory areas. The reason for this is that by the time the first wave of extreme excitement has subsided the individual may find himself some distance from the scene of the accident in an area having no safety shower. Moreover, in getting away from the scene of the accident rapidly the person may choose a path leading outside the building and it may not be possible or desirable to re-enter the building. This implies

that some safety showers should be installed directly outside of certain exterior doors even though the nature of the area is such that normally no experimental work would be carried on outside. It is now felt that in a real emergency such an exterior shower would probably find use even in the winter time.

In the above accident it is understandable that the safety showers in the Hot Area were not used because the Hot Area was rapidly filling up with dense fumes and the overwhelming and justifiable tendency was to get out of that area as rapidly as possible. However, having once gotten out of the Hot Area some of the personnel did run past one or more safety showers on their way to another shower, the location of which was more familiar to them. This calls for increased measures to acquaint everyone with the location of every safety shower in the building, not just the one in his own regular working area.

Following the accident the Hot Laboratory has tried successfully a number of different things: First, the wattage on some of the green lights marking the safety showers has been increased so as to make the light considerably brighter. Second, on other green lights flashers have been installed, and it has been found that a flashing 10-watt bulb is more noticeable than a steady 60-watt bulb. The value of these flashers is thought to lie not so much in calling attention to the location of the shower during an emergency but rather to call attention to the shower under normal day-to-day working conditions. Judging from the reactions and comments from a number of our employees these flashing lights have served to impress upon them the location of the safety showers in a way far greater than could be accomplished by mere signs, safety meetings, or other suggestions. Even when not looking directly at the shower the flashing lights are very noticable out of the corner of one's eye and one either turns the head to see the cause of the flash and perceives that it is a safety shower or else, having done so previously, he once again realizes that there is a safety shower at that particular location.

In addition to the flashing green lights we have also installed at a number of safety showers a bright, white, flood light. Although this is not as effective in normal day-to-day work in calling attention to the location of the safety shower as is the flashing green light, nevertheless it is thought that during an emergency, particularly one involving a failure of the normal power, these flood lights will help to make the locations of the safety showers stand out more clearly.

As a corollary it goes almost without saying that the flashing green lights and the white, flood lights should both be on the emergency power supply so that in the event of failure of normal lights these emergency lights would remain on.

We have also successfully undertaken another means of acquainting people with the locations of safety showers. Prior to the accident the safety showers had been tested once a week by one or two of our personnel in charge of maintenance-type work. Recently we have instituted a system whereby everyone in the building including scientists, technicians, supervisors, secretaries, janitors, etc. takes his turn and tests these showers daily. This is done in pairs, one pair a day, according to a schedule set up in advance. A person goes around on the first day with a member of the team from the preceding day; the following day that person goes around with a new man, and so on. A check-off sheet is provided wherein the individuals note the performance of each shower and any other safety comments

pertinent to the area around the showers. The total time required is about fifteen to twenty minutes per day and with about sixty people working in the building this means that each person checks the showers on a pair of successive days occurring once every three months. Employee cooperation has been better than expected and it is felt that this procedure does truly educate everyone as to the location of the safety showers.

Although it was not a factor in this accident we have adopted the standard policy that every safety shower shall have next to it or under it an eyewash fountain installed in such a way that either the fountain or the shower can be used independently of the other or both can be operated at the same time.

The absence of safety showers in certain locations of the Cold Area of the Hot Lab was in no way a factor during the above accident. However, in reviewing the safety shower situation as a result of this accident it was realized that safety showers should be added in at least two locations in the Cold Area because of the fact that over the past several years areas which had not previously been laboratory space have since been remodeled into laboratory space and therefore the concentration of safety showers per unit area should be increased here accordingly.

Before leaving the subject of safety showers we should stress emphatically the necessity of educating as many people as possible - not just laboratory workers - with the advisability of dousing a victim immediately with large volumes of water in all cases involving accidental contact with corrosive or hazardous chemicals.

The above discussion can be summarized in the following recommendations:

- (1) Safety showers should be provided outside as well as inside a laboratory building.
- (2) Some safety showers should be provided in all areas of the building with perhaps the greater concentration being in the laboratory areas.
- (3) Safety showers should be marked with a bright, green, flashing light and preferably also with a bright, white, flood light.
- (4) All these lights together with any other emergency lighting should be on an emergency power circuit, such that these lights remain on at all times regardless of power failure or intentional shut off of normal power.
- (5) Eye-wash fountains should be installed at every safety shower.
- (6) Everyone in the building should participate in a regular scheduled safety shower check.

Door Closers and Locks

Without going into minute details unnecessarily it may be said that as a result of this accident we have come to the conclusion that every door, whether interior or exterior, should be readily openable from both

sides. For instance, in an effort to control traffic all exterior doors of the Hot Lab with the exception of the main lobby entrance were openable from the inside by means of emergency crash bars but were openable from the outside by means of keys only (with the exception of five doors which couldn't be opened from the outside at all). As noted above, in an emergency of the type described here the first reaction is to get out of the building. However, the second reaction is to get back in for purposes of rescue, fire control, and other emergency procedures. With exterior doors arranged as described it would be necessary to find someone with a master key in order to re-enter the building. Fortunately such persons were available and no difficulty was experienced in returning to the building, but it is not hard to imagine other situations in which a key for re-entry would not be readily available. As a practical solution we are installing outside and adjacent to each exterior door a key in a box with a glass front which can be easily broken in an emergency.

It is equally true that every interior door must be readily openable from both sides. For instance, when door H-152 was originally installed it was intended to be an emergency exit from the Alpha Facility into the Semi-Hot Lab corridor. However, it was later realized that in case of a serious emergency this door might also have to serve as an emergency exit from the Semi-Hot Lab corridor back through the Alpha Facility. This door had been provided with a crash bar on the Alpha Facility side only and nothing on the Semi-Hot Lab corridor side. When it was later realized that the door may have to serve as an emergency exit in both directions a hole was drilled in the door, a cable run through and fastened to the panic bar on the Alpha Facility side terminating with an ordinary round brass door knob on the Semi-Hot Lab side. However, the instinctive response to such a knob is to twist it whereas in this case it should have been pulled; accordingly this knob has been changed to a loop type handle and a sign has been installed over it reading "In Case of an Emergency Pull Handle".

For traffic control purposes door C-226 between the Machine Shop (in the Cold Area) and the Hot Area had previously been arranged so that it was always unlocked from the Machine Shop side but always locked from the hot side; this permitted personnel entry from the shop into the Hot Area via this door but required that personnel pass through the Monitor Station in returning to the Cold Area from the Hot Area. It has since been realized that an emergency might occur which would require rapid passage from the Hot Area directly into the Machine Shop through this door. Since it is desired to lock the Machine Shop at night the only practical solution to this situation is to remove the lock originally installed on this door, to provide a crash bar on the hot side and to adopt the following procedure: Each night a readily breakable mechanical seal is padlocked to the hot side of this door and removed next morning. This permits emergency access in both directions at all times, and normal access from the Cold to the Hot Area during the day; personnel are instructed not to use the crash bar going from the Hot Area to the Cold Area except in cases of emergency.

It may be noted that part of the uranium contamination and damage from corrosion which occurred in the Cold Area resulted from the fact that the automatic door closer on door C-228 (the door between the Monitor Station and the cold corridor) was not functioning properly at the time of the accident. This door is normally held wide open by a spring device and the ventilation produces a flow of air from the Cold Area to the Hot Area through this door. However, when the accident occurred and the exterior doors were opened a west wind blew fumes from the Hot Area to the Cold Area. If the door closer had been working properly it would have held the door closed once the door had been shut

and would have prevented this backup of fumes. Nevertheless, it had been found to be defective and had been repaired twice in the two days prior to the accident but was still not working properly. As a result the door was held in a half-open position. In this particular case the result was more of a nuisance than anything else although under different circumstances the results might have been serious. This does illustrate, however, the necessity of good maintenance at all times inasmuch as a simple thing like a defective door closer can contribute to the spread of contamination and damage in the event of a sudden emergency.

The following recommendations summarize this part of the discussion:

- (1) All doors whether interior or exterior should be readily openable from both sides.
- (2) The means by which these doors are opened in an emergency should be clearly obvious.

Ventilation

With regard to the ventilation not too much can be said here which will be pertinent to other installations and other situations although a few points may be noted. The accident created a highly abnormal situation with respect to ventilation--one for which no ventilation system would be adequate. As mentioned before, the fumes of bromine trifluoride which were released were so dense that they blotted out the light from the overhead lights plunging the Hot Area into total darkness within a few minutes, giving the appearance of a power failure. In order to enable rescue workers to search and guarantee that no one had been left inside (and to permit firefighters to extinguish the fire) it was necessary to clear the area of the fumes with all possible speed. For this reason all exterior doors were opened as rapidly as possible permitting the brisk west wind to blow out most of the fumes. As mentioned before some of these fumes were carried into the Cold Area through one of the interior doors which failed to close properly. Some fumes were also carried from the Hot to the Cold Area through two one-foot-square openings in the wall separating the two areas; these openings are associated with the pressure differential sensing devices which are used to control fans which maintain the air pressure in the Hot Area lower than that in the Cold Area. These openings are now being provided with flap dampers so that if for any reason the flow does reverse in spite of the fans the dampers will close and prevent flow from the Hot Area into the Cold Area. However, some fumes also entered the Cold Area through the normal ventilation supply, the intakes of which are located downstream of the accident.

Two other factors contributed in a minor way to our harassment. First, the automatic pressure differential sensing devices were not properly adjusted and it is believed that the auxiliary roof fans did not turn on as soon as they should have. Second, there were two separate sets of manual switches, located some distance from each other, the purpose of which was to over-ride the automatic system and cause the auxiliary fans to operate manually. Although both sets of switches were separated just so as to be able to get at one set if the other set could not be reached, nevertheless both sets were in the Hot Area and therefore neither could be reached until about 15 minutes after the accident. However, because of the overwhelming nature of the situation it is quite doubtful that these roof fans could have helped to any measurable degree and the question of their proper functioning in this case is therefore largely academic. Nevertheless, a third set of manual switches has been installed under glass just outside of exterior door H-107.

Emergency Plan

Since January, 1953 the Hot Laboratory has had its own local emergency plan operating in conjunction with the over-all emergency plan of the Laboratory. These plans are currently being studied for possible improvement. One innovation which has been made successfully is that of maintaining someone "in-charge-of-quarters" at all times in the Hot Lab between the normal working hours of 8:30 A. M. and 5:00 P. M. This system works as follows:

In the main office of the Hot Laboratory building is a large board divided into six horizontal lines and three vertical columns. The arrangement is such that the names of six men can be shown on the board, one name on each line, in such a way that each name can be slid horizontally into one of the three columns. The center column is labelled "Local Emergency Supervisor" and the other two columns are labelled "In" and "Out," respectively. Beginning at the top, the names include the name of the person who is normally the local emergency supervisor, the deputy local emergency supervisor, and the four wardens. At the beginning of each day the name of the top man is moved to the center column showing that he is in charge of any emergency which should arise at that time and the names of the other five men are shown in either the "In" or "Out" column depending on whether they are in or out of the building. When the man shown at the top has to leave the building, even if for only a few minutes, his name is moved to the "Out" column and whoever is next on the list in the "In" column is notified that he is responsible during any emergency which should arise, his name being moved to the center. If and when the second man has to leave the building the chain of command moves on down to the next man in the same manner. When one of these men returns to the building the chain of command does not pass back up to him immediately but remains with whoever was in charge while he was out; the purpose of this provision is to prevent the chain of command from bouncing back and forth too frequently during a day. If five of the men are out of the building and the sixth man wants to leave, it is up to him to find a suitable person to leave in charge of the building and to notify that person personally before he leaves; the name of this seventh man would be added temporarily to the bottom of the board in the center column but in such a case the chain of command passes back again to whichever of the six men shown permanently on the board returns to the building first. The purpose of this latter provision is to restore the emergency supervision to those most familiar with it; so far there has been no occasion when all six of the permanent emergency staff have been out of the building at the same time. An important provision of this plan is that, while during a non-emergency situation a man may relieve himself of emergency supervisory responsibility by telephoning the next man below him who is present in the building, once an emergency has begun whoever is in charge at the time remains in charge until relieved in person by someone higher on the permanent emergency staff. The purpose of this provision is to insure that during an emergency two people located in different parts of the building are not, unknown to each other, trying to take charge.

Other features of the emergency plan which are still being studied include the following: (1) The most effective way of reporting an emergency (i.e. whether to dial a particular telephone number located in the building or to pull a fire alarm); (2) the use of the "buddy system" as a means of ascertaining the whereabouts of everyone during and following an emergency; (3) the advisability of some sort of fire drill; and (4) the authority to keep professional fire fighters out of an area in which the local emergency supervisor feels that it is detrimental or unnecessary for them to enter.

Protective Clothing and Eye Protection

For several years the policy concerning eye protection was that "safety glasses are to be worn by everyone performing a hazardous operation or by persons in the vicinity of such an operation". Examples of hazardous operations were quoted. However, it was found that that rule leaves too much to the judgement and discretion of the individual and, as a result, the rule has been changed to require everyone, including transient visitors, to wear safety glasses at all times in the Hot Laboratory except in the offices and in the cold corridor.

At our request a private manufacturer of safety equipment is developing a new one-piece plastic suit covering the entire body from head to toe including the hands and feet. This suit will be a coverall type with full length zipper. It will contain its own compressed air bottle with a ten-minute supply and a quick disconnect fitting for rapidly attaching a new bottle or for connection to the house compressed air system where more extended use is required. Several of these will be hung around the building in plain sight in strategic areas ready to step into at a moment's notice. The suits will be slightly pressurized and should provide protection from contamination as well as from most chemicals.

Miscellaneous Recommendations

Following are some miscellaneous safety suggestions which have recently been put into effect, some of which, however, have no connection with the accident mentioned above:

(1) It is assumed that every laboratory building comparable in size to the Hot Laboratory has a public address system which is used routinely for a variety of non-emergency uses. Inasmuch as such a system becomes very valuable in an emergency it is important that it be connected so as to operate from the emergency power supply as well as from the normal power supply so that it can continue to function at all times during all kinds of emergencies.

(2) We have been re-indoctrinating people from time to time in the use of emergency equipment such as Chemox masks, Scott Air-Packs, etc. Without such re-indoctrination people tend to forget how some of these things are used.

(3) Exterior electric outlets are being added so as to facilitate operating portable pumps for emergency air sampling and monitoring.

(4) During a recent test it was found that there were areas in the building in which the house fire gongs could not be heard. Additional fire gongs are being added so that they will be audible in all parts of the building.

(5) About 40% of the occupants of the Hot Laboratory building are organizationally outside of the Hot Laboratory Division, which therefore has little, if any, direct administrative control over such people. The accounting of personnel after an emergency can be no better than one's knowledge of whom to expect in the building prior to an emergency. Therefore all personnel presently working in the Hot Laboratory building have been instructed to advise the Hot Laboratory office whenever they are aware of a new person starting to work in the building. The purpose of this is to permit us to issue to this employee (who may either be new to Brookhaven or just new to the Hot Laboratory) various safety memos, a copy of the building rules, the Brookhaven safety manual, safety shoes,

and safety glasses, not to mention a locker, driver's license, mailbox, etc. as necessary. This procedure of notification is believed to be more foolproof than one which would rely on a new employee's supervisor notifying the Hot Laboratory office.

(6) In an effort to make safety posters and other important signs stand out more clearly a committee was formed to inspect the building with a view towards eliminating all unnecessary or unimportant signs.

Hot Laboratory Panel Discussion at the Sixth Hot Laboratories and Equipment Conference 1958 Nuclear Congress, March 16-21, 1958

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The Radiometallurgy Laboratory at the Hanford Atomic Products Operation has been in operation since July 1953 and some portion of the structure and operating philosophy has been discussed at the 5 previous hot laboratory meetings. This short report will provide a basis for panel discussion.

The building that was designed to satisfy the specific problems posed by Hanford Technology is shown in Figure 1 (combination exterior, interior and line drawing of floor plan). The work area is 30' x 173' (5190 sq. ft.), serviced by a 15 ton traveling crane, with four permanent type cells with interior space of 4' x 6' x 4' shielded uniformly on all sides with meehanite cast iron of 7.0 g/cc, one single purpose cell for room temperature tensile testing and three temporary, lead brick shielded, cells for high temperature tensile testing, X-ray diffraction analyses and for sampling operations with an abrasive cut-off wheel. The remaining part of the building includes the necessary supporting functions such as offices, locker room, decontamination room, dark room, machine shop, sample and equipment storage space, air-conditioning and a full basement for storage, mock-up and air control

Radioactive samples are received in lead filled, stainless lined casks, placed in a water filled basin for identification and transferred to laboratory type through-hole casks for handling to and from the shielded cells. Inside the strip coated cells, the samples are viewed and photographed through 6.2 g/cc lead glass viewers and then the required mechanical and physical property tests are made. There is a 30 minute long film which shows some of these operations in detail and is available upon request. After testing, the samples are either dis-

Fig. 1(a)—Radiometallurgy building, interior.

Fig. 1(b)—Radiometallurgy building, exterior.

Fig. 1(c)—Radiometallurgy building, floor plan.

Fig. 2—Laboratory organization chart.

carded to waste or saved by storing in the dry storage vault (coke machine). The standard remote equipment is usually used repeatedly until it is necessary to provide maintenance to the equipment. Short term experimentation is rare but can be easily arranged, by careful utilization of the removable plugs.

The organization of the laboratory reflects the nature of the HAPO organization and provides on a two shift basis complete flexibility to support operational requirements or to obtain data in support of Research and Development Programs (Figure 2). The engineers are assigned work in their specialties and are requested to provide summary and conclusion type reports.

Each question or study asked of our laboratory requires from one hundred to several thousand manhours of laboratory work to provide adequate answers. To satisfy all these requests is impossible and once a month, all interested organizations meet to indicate, in their opinion, what kind of laboratory work will most benefit HAPO in the coming month. We attempt to satisfy the conclusions of this meeting, but the inescapable fact is - that more work comes into a hot laboratory in a year than there is manpower, facilities and equipment to provide all the details that are so necessary to the establishment of adequate fuel element designs.

A Conducting Plastic for Metallographic Specimen Mounts

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It is difficult to prepare good surfaces for metallographic studies on some alloys without electrolytic etching. Copper-filled mounting powders available for such use require excessively high mounting pressures. This paper describes a method of loading Bakelite with graphite to obtain conducting mounts with electrical resistances of from one to several thousand ohms. Resistances may be closely controlled by proportioning the mix appropriately.

The necessity for use of electrolytic etching procedures on various alloys used today led to the development of a means for producing electrically conductive mounts. The method is applicable for use in hot cells where such mounts must be produced by remote-handling techniques.

A commercially available copper-plastic molding material was first investigated but its use was discarded because of the high molding and mold-removing pressures required. Such pressures were found substantially to increase the maintenance time on the remote press used. Since Bakelite molding powders require lower pressures, experiments were carried out in which graphite additives were mixed with the Bakelite in various proportions, molded, and electrical resistance measurements taken. Several commercially available powder and flake forms of graphite were investigated for such use; 2XX flake^a was finally selected since it provided both the electrical properties required and an excellent lubrication action on the press mold and cylinder.

a. "Superflake" from the Superior Graphite Company, Chicago, Illinois.

The graphite was mixed with both black and green Bakelite molding powders^b in several different proportions by weight per cent and trial mounts prepared. A constant pressure spring^c was molded into the front face and a pre-numbered washer into the back face of each mount, both for identification and for magnetic remote-handling. A sketch of a typical mount is shown in Figure 1. After cooling, the electrical

Figure 1

resistance of each mount was measured between the washer and spring. A thickness measurement was taken on each mount and the resistance values corrected to a consistent unit. (Total thickness of spring plus washer = 0.315 inches.) All mounts were one and one-quarter inches in diameter, and were molded at 6000 psi. The data taken are shown in Tables 1 and 2.

The resulting data from Tables 1 and 2 are shown in Figure 2 on a semi-logarithmic plot.

All weight proportions were made on a laboratory analytical balance weighed to one milligram accuracy. Thickness measurements were taken with a micrometer to 0.001 inches and resistance measurements with a laboratory wheatstone bridge of 2% accuracy. The variations in resistivity within each mixture are most likely due to non-homogeneities resulting from variations in the degree of mixing. Tests of plastic hardness with a Brinell machine showed no pattern of variation with graphite content.

No good reason was found for the differences in resistance values between the two types of Bakelite, but it was noted that the green

b. "1380 AB Black Bakelite" and "1382 AB Green Bakelite" mounting powders, Buehler Ltd, Evanston, Illinois.

c. "Neg'ator", Hunter Spring Company, Lansdale, Pennsylvania.

TABLE I

Electrical Resistances of Graphite and Black Bakelite Mounts

Mount No.	% Graphite	Total Resistance	Total Thickness In Inches	Plastic Thickness In Inches	Specific Resistance
0A	0	> 10 ⁸ ohms	0.927	0.612	> 10 ⁸ ohms/in.
0B	0	> 10 ⁸ "	0.905	0.590	> 10 ⁸ " "
0C	0	> 10 ⁸ "	0.936	0.621	> 10 ⁸ " "
1A	1	95.9 "	0.946	0.631	152 " "
1B	1	79.9 "	0.907	0.592	135 " "
1C	1	68.4 "	0.905	0.590	116 " "
2A	2	17.9 "	0.913	0.598	30.0 " "
2B	2	24.8 "	0.936	0.621	40.0 " "
2C	2	21.2 "	0.957	0.642	33.0 " "
3A	3	6.72"	0.926	0.611	11.0 " "
3B	3	6.10"	0.913	0.598	10.2 " "
3C	3	4.87"	0.902	0.587	8.30" "
4A	4	2.48"	0.952	0.637	3.89" "
4B	4	1.62"	0.966	0.651	2.49" "
4C	4	2.26"	0.999	0.684	3.30" "
5A	5	0.749 ohms	0.891	0.576	1.30" "
5B	5	0.641 "	0.898	0.583	1.10" "
5C	5	0.734 "	0.927	0.612	1.20" "
6A	6	0.228 "	0.947	0.632	0.361" "
6B	6	0.255 "	0.953	0.638	0.400" "
6C	6	0.326 "	0.966	0.651	0.501" "
7A	7	0.105 "	0.934	0.619	0.179" "
7B	7	0.134 "	0.925	0.610	0.220" "
7C	7	0.114 "	0.913	0.598	0.191" "

TABLE II

Electrical Resistance of Graphite and Green Bakelite Mounts

Mount No.	% Graphite	Total Resistance	Total Thickness In Inches	Plastic Thickness In Inches	Specific Resistance
OA	0	$> 10^8$ ohms	0.810	0.495	$> 10^8$ ohms/inch
OB	0	$> 10^8$ "	0.817	0.502	$> 10^8$ " "
OC	0	$> 10^8$ "	0.936	0.621	$> 10^8$ " "
1A	1	$> 10^8$ "	0.849	0.534	$> 10^8$ " "
1B	1	$> 10^8$ "	0.917	0.602	$> 10^8$ " "
1C	1	$> 10^8$ "	0.913	0.598	$> 10^8$ " "
2A	2	250,000 "	0.916	0.601	416,000 ohms "
2B	2	800,000 "	0.938	0.623	1,284,000 " "
2C	2	410,000 "	0.926	0.611	671,000 " "
2.3A	2.3	1,160 "	0.764	0.449	2,584 " "
2.3B	2.3	2,750 "	0.890	0.471	5,840 " "
2.3C	2.3	3,400 "	0.800	0.485	7,010 " "
2.6A	2.6	481 "	0.791	0.476	1,011 " "
2.6B	2.6	578 "	0.836	0.521	1,110 " "
2.6C	2.6	273 "	0.845	0.530	515 " "
3A	3	41 "	0.852	0.537	76.4 " "
3B	3	150 "	0.870	0.555	270 " "
3C	3	59 "	0.966	0.651	90.6 " "
3.5A	3.5	15.1 "	0.865	0.550	27.5 " "
3.5B	3.5	11.3 "	0.850	0.535	21.1 " "
3.5C	3.5	11.2 "	0.855	0.540	20.7 " "
4A	4	6.66 "	0.887	0.572	11.6 " "
4B	4	7.05 "	1.01	0.695	10.1 " "
4C	4	8.7 "	1.00	0.685	12.8 " "
5A	5	2.82 "	0.896	0.581	4.85 " "
5B	5	1.96 "	0.890	0.576	3.40 " "

TABLE II (Continued)

Mount No.	% Graphite	Total Resistance	Total Thickness In Inches	Plastic Thickness In Inches	Specific Resistance
5C	5	2.63 "	0.907	0.592	4.44 " "
6A	6	0.987 ohms	0.899	0.584	1.69 ohms/inch
6B	6	0.975 "	0.906	0.591	1.65 " "
6C	6	0.692 "	0.878	0.563	1.23 " "
7A	7	0.284 "	0.866	0.551	0.515 " "
7B	7	0.435 "	0.873	0.558	0.780 " "
7C	7	0.381 "	0.875	0.560	0.680 " "

granules were larger in size. Rechecks of size and resistivity of the mounts over a 6 months period yielded data indicating physical growth and increasing resistance of those made from black Bakelite, while those made of green Bakelite appear to have fairly constant characteristics; insufficient time has elapsed to make a quantitative survey of these variations. In general, it was found that the surface quality, dimensional stability, and erosion characteristics during subsequent lapping and polishing operations exhibited by the green Bakelite mixture were superior to those of the black. Standard electroetching procedures worked equally well with either type providing the resistance was approximately 2 ohms or less.

Figure 2

Modifications to Master Slaves at Bettis Hot Laboratory

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Abstract

Several modifications have been made to both Argonne Model 4 and Model 8 Master-Slave Manipulators to meet the operating conditions at Bettis. Most of these modifications were made to increase the capacity or prevent failures on the original units.

For the last two years the need for master slaves at the Bettis Hot Lab has been steadily increasing. This has necessitated utilizing each slave for nearly sixteen hours per day. Because of this a maintenance program was started to keep the "down time" of each slave to an absolute minimum.

During the early months of this program, it became apparent that both the Argonne Model 4 and Model 8 Slaves had certain components with high repetitive maintenance. These components are the general purpose tongs, the counterbalance tapes and the handle sub-assembly.

The first Model 8's that Bettis purchased were equipped with the remotely interchangeable tongs sold as standard equipment by the manufacturers. These tongs used a small radius pulley in the actuating linkage which caused rapid fatigue of the 1/32" cable being used. This resulted in premature cable breakage.

In addition to the cable breakage, the interchangeable tong assembly would come loose from the slave. This not only wastes time but endangers both equipment and experimental samples.

At that time there were two Model 4's in operation at Bettis with MSM-6B-1000 tongs. These were of the non-removable type and did not use

small radius pulleys. These tongs would be ideal for Bettis Hot Lab use if there were some way to remotely install new rubber faces on the jaws of the tongs.

The original design for a removable jaw face was copied after one in use by Phillips Petroleum at the MTR Site in Idaho. Their basic idea was used, but modified to meet Bettis' requirements. The jaws were scaled so that the modified units with faces intact were the same size as the original MSM-6B-1000 type. A removal jig was developed to remove the faces remotely when the rubber came off the jaw faces. The jaw faces were relatively cheap so several new ones can be readily kept in stock. (Figure 1)

This proved satisfactory for a period of time, but under the heavier loads to which the slaves were being subjected, the 1/32" cable proved to be too weak. These tongs did not have the small radius pulleys so a 1/16" x 7 x 19 stainless steel cable could be used in place of the smaller one. Another factor which proved to be troublesome was the method of fastening the cable to the link. This caused two difficulties: the cable pulling loose from the link and the cable jumping out of its groove thus damaging the cable. To remedy this, the link to which the cable is fastened was altered and a new method of fastening the cable was developed. A "Nicopress" hand squeezer was purchased to use in conjunction with the T-46-e2 plain ball ends used to hold the 1/16" cable in the link. (Figure 2) This tool also made it possible to use the Kearney A-1 (6-5) swaging machine, instead of the more expensive Model 7-1, on the cable turn buckle ends.

The tongs could be improved further by providing a method to change the jaw faces with another slave. This would make possible the use of specialized jaw faces for handling different shaped objects. These tongs will fit all the commercially available Argonne manipulators by changing only three guide pulleys in the wrist.

Tape breakage was observed to be the next largest maintenance problem. The tapes that broke the greatest percentage of the time were those on the counterbalance assembly on the Model 8's and the "Z" motion tapes on the Model 4's. (Figure 3) Both of these cases were caused by the tapes not having the ability to absorb the shock caused by stopping the downward "Z" motion quickly, such as hammering a cover on a can. Also in both cases there were two tapes being opposed by the four wrist tapes. Doubling the two tapes did not remedy this situation due to the difficulty in obtaining equal tension in each tape. By changing these tapes to 1/16" x 7 x 19 stainless steel cable the problem was eliminated completely.

Model 4's originally used two tapes opposing each other in the "Z" motion. A single cable was substituted which ran the complete distance and then clamped to the point where the original two tapes were fastened together; this gave equal tension in each cable and improved the balance of the slave. (Figure 4) The cables had the necessary resilience to prevent this breakage, yet at the same time did not produce a "mushy" feeling in the slave.

During early operations at Bettis it became apparent that an ambidextrous finger grip would be preferable due to the numerous times

0 1 2 3
INCHES

Fig. 1—Remotely removable jaw faces; jig jaw at extreme left is one with nonremovable face.

0 1 2 3
INCHES

Fig. 2—Left: original tong link with $\frac{1}{32}$ -in. cable. Right: new link showing $\frac{1}{16}$ -in. cable and method of fastening.

that a slave would be moved from one side of a window to the other. Several ambidextrous finger grips were fabricated and tried until one was found that the majority of the operating technicians preferred. (Figure 5) All of the slave were then equipped with new linkages and finger grips of this type supplied by an outside vendor to our design.

Fig. 3—Model 4—“Z” motion schematic.

After these grips had been in operation for about a month, a majority of the technicians preferred them in all cases over the individual right or left hand grips.

The final change was made on the cable linkage in the grip. The problem here was essentially the same as in the tongs: $1/32$ " diameter cable, small radius pulley and the tendency for the cable to jump out

Fig. 4—Model 8—"Z" motion schematic.

of the pulley groove. The solution to the problem in this case was to substitute a bell crank linkage and use 1/16" cable. (Figure 6) As in the case of the tong end, a "Micropress" sleeve holds one end of the cable and a T-46-e2 plain ball holds the other end. The cable rubs against the edge of the hole in the yoke, but total friction in the unit is actually lower than with a pulley. In this case, the only alteration necessary was to deepen the groove in the yoke pulleys.

Fig. 5—Ambidextrous finger grips.

It has been found desirable to rebuild each slave unit once every six months. This includes a complete strip down of the unit, then cleaning and inspecting each part. The bearings are cleaned in an ultrasonic cleaner and then relubricated with the lubricant recommended by the manufacturer.

A dolly was developed (Figure 7) to help in the transfer of the units and provide a variable height working platform for the slaves when they are undergoing repairs. This dolly is also used for slave removal and installation in the cells.

Fig. 6—Bell crank assembly on hand when tong is in the closed position.

Fig. 7—Dollies in different positions for slave maintenance.

A Continuously Recording Micrometer-Profilometer for Hot Laboratory Applications

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Abstract

An instrument is described which provides a continuous record of thickness change or surface profile for irradiated plate and rod specimens. Two linear displacement transducers are oriented in an opposed position and the samples are moved between roller actuators at a preselected speed, providing suitable recorder magnification. Total dimension changes obtained in this manner are independent of sample warpage or bowing. Disconnecting one transducer provides a surface contour with high resolution of surface defects. Samples 6" high, 8" wide, and 16" long are accommodated. Routine accuracies of .5 to .1 mils can be obtained under normal hot cell operating conditions.

Introduction

Current interest in the development of reactor fuel alloys with good dimensional stability under conditions of high burnup and high temperature has created a need for more extensive and reliable dimensional analyses of irradiated specimens. Length, width, and thickness measurements are of primary importance in detecting volume changes, and more recently, valuable information has been obtained by correlating the variation in surface profile with regions of high neutron flux, local boiling, high temperature gradients, etc. Several types of remotely-operated micrometers have been developed to provide these data, but many of these techniques are laborious and time consuming when extensive dimensional surveys are required, and are not sufficiently versatile to provide a continuous record of surface profile. An apparatus has been developed at the Bettis Hot Laboratory which combines both of these functions

in a mechanically simple, inexpensive system specifically designed for the hot cell environment, and which can be operated conveniently with master-slave manipulators. All adjustments (zero set, calibration, etc.) are accomplished without requiring access to the cell. Conversion from one specimen size or geometry to another may also be accomplished remotely.

Principle of Operation

The fundamental principle of the micrometer-profilometer is based upon the precision presently available from the linear variable differential

Fig. 1—Cross section of typical linear variable differential transformer (LVDT).

transformer (LVDT) as a displacement-voltage transducer. These transducers are available (a) in a wide range of designs where voltage is always linearly proportional to the displacement of a core moving within a primary and two coaxial secondary coils. A typical LVDT is illustrated in Figures 1 and 2. Output voltage as a function of primary excitation frequency and load resistance is shown in Figure 3. A linearity check of a $\frac{1}{2}$ ".100" LVDT was performed by moving the core with a micrometer reading to ".0001". In general, the linear output voltage extended well beyond the manufacturer's specified linear range. With special attention given to line voltage stabilization and a rigid, vibration-free mounting for the LVDT, reproducible readings to ".0001" could be obtained. The LVDT used in this application does not contain "potting" or encapsulating resins, and has exhibited good resistance to gamma deterioration. The micrometer-profilometer has been operated for extended periods of time in the presence of 10,000 curie sources without detectable variation in accuracy or sensitivity. The coils are insulated by a vacuum-impregnated varnish, providing satisfactory stability under high humidity conditions. The primary is operated at a frequency such that the reactive impedance is substantially greater than the temperature-sensitive d-c coil resistance, and adequate temperature stability has been experienced under normal hot cell operating conditions. Where temperature extremes are a factor, the LVDT is available with coils wound of manganin wire. The LVDT is sensitive to magnetic materials nearby, and a magnetically shielded unit (a) should be used when such conditions are anticipated.

Fig. 2—LVDT output voltage and phase as a function of core position.

Fig. 3—Linear voltage output of typical LVDT into various resistance loads for primary excitation of 60 and 400 CPS.

General Description

The micrometer-profilometer consists of three basic units shown in Figure 4. From left to right these are the amplifier, the remote measuring unit, and the recorder.

Fig. 4—Left to right: displacement indicator, remote unit recorder.

The remote unit is shown in greater detail in Figure 5, and consists of a rigid, adjustable mounting for two transducers and a motor-driven reversible table for holding the specimen. The two transducers are

Fig. 5—Enlarged view of remote unit.

mounted in a vertically opposed position. Each LVDT core is spring-loaded against a $3/4$ " stainless steel roller actuator which is itself hinged in such a way to allow a total displacement range of .200". The upper transducer is clearly seen in Figure 5, mounted to a rigid adjustable support, providing accommodation for specimen size and geometry. The lower transducer is permanently mounted below the moving specimen table,

and only the roller actuator is movable, thus providing maximum protection against contamination in hard-to-clean regions of the unit. The specimen is clamped into position by a vise which is attached to the movable portion of the table. The table can be moved through a 12" travel by a dynamically-braked reversible motor (b) which is hinged so that three separate gears may be selected to drive the table. These gears provide table speeds of 1-1/2, 2 and 3 inches per minute. When used as a micro-meter, the output of each transducer is electrically integrated in a junction box, shown on the lower left of the remote unit. (Figure 5) This box contains a connector for feeding the resultant signal to the amplifier, a three-position switch for in-cell control of table motion, and another connector for paralleling the table motion control with an out-of-cell control station. When used as a profilometer, the output of the upper LVDT is fed directly to the amplifier, by-passing the second LVDT and the integrating circuit.

The signal from one or both transducers is fed by a shielded cable from the remote junction box through a cell access port to the amplifier. (c) This unit contains all necessary calibration controls, a stabilized a-c voltage supply for the transducer primaries, an amplifier for the output signal, and a direct-reading displacement meter. The meter may be paralleled with any properly matched recorder. The operation of the amplifier is sensitive to varying line supply voltage, and it is usually necessary to provide a constant voltage power supply if variations of ± 15 volts occur in the lines.

- (a) Available from the Daytronic Corporation, 216 S. Main Street, Dayton 2, Ohio. The units used in this equipment are Daytronic Model 100 transducers, without magnetic shielding, and with a linear voltage-displacement range of .060".
- (b) Bodine Electric Company, 2254 West Ohio Street, Chicago, Illinois, Model KYC-23RB, 60 rpm.
- (c) Daytronic Model 300.

Calibration

Initial adjustment for sample size and geometry is accomplished by clamping the support arm of the upper LVDT in a position that will assure operation of the transducer within its linear range. The zero point is set electrically by an appropriate control on the amplifier. A mechanical zero shift is also available by turning a knob on the transducer itself. Gauge blocks of known thickness are inserted between the roller actuators, and the proper deflection can then be set on the displacement indicator by an electrical calibration adjustment.

Acknowledgement

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Remote Metallographic Equipment and Practices

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Abstract

This paper is a review of equipment and practices used in the remote metallographic facility at the Bettis Site of the Westinghouse Atomic Power Division. The modifications of various pieces of equipment are outlined in addition to their operation and advantages. Deviations from normal operations or special techniques are discussed for specific operations. There are sections on the construction of the basic cell, the supporting equipment such as the liquid waste disposal system, design and operation of a remote sectioning machine, mounting techniques, grinding and polishing procedures, etching equipment and techniques, transfer and storage facilities, photography, hardness testing and operating techniques for specific materials as related to the problems encountered in processing radioactive materials.

Introduction

Early in 1955 when it became evident that the existing Bettis Plan hot laboratory facilities were inadequate to handle anticipated demands, an expansion program was initiated. In conjunction with this expansion a low level radiation cell was designed to house the complete facilities for a remote metallographic operation. This cell is a separate installation located in the loading area of the hot laboratory.

1. Steel Cell

A photograph of the low level radiation cell used for metallography is shown in Figure 1. The design is a modification of the remote metallographic cell at Oak Ridge National Laboratories designed by M. J. Feldman. It is constructed of steel in the form of a box with

outside dimensions of 11' x 6' x 4'. It weighs approximately 30 tons and has a usable floor space of 50 sq. ft. The 6" thick walls and floors contain six 1" plates welded together affording a total attenuation factor of 3 decades for a 1 mev gamma source. The lead glass windows (density 6.2 gms/cc) for viewing have equivalent shielding.

Fig. 1—A view of the steel cell showing the B&L Research Metallograph and the cell control panel.

The roof slabs are 2" thick consisting of two 1" thick plates, and are readily removed with the laboratory crane. In order to minimize the spread of contamination and also to protect the optical equipment from abrasive and chemical fumes, the cell was divided into two sections by a 1" thick steel barrier. This barrier which separates the optical equipment from the processing equipment can be removed with the laboratory crane.

The recessed end of the cell accommodating the Metallograph (Figure 1) consists of a 1/2" thick steel plate backed up with 4" of lead. This construction affords slightly greater shielding than 6" of steel, and was easier to fabricate into the desired form. The cell is supported at a height of 36" from the floor by 6 legs of 6" Schedule 80 pipe. Such an arrangement allows easy accessibility for maintenance on the supporting equipment located beneath the cell.

In order to best take advantage of the shielding four 1" diameter holes were drilled through the floor of the cell to accommodate the shafts of automatic polishers, instead of removing a large section for each unit such as the 12" x 24" hole necessary for the Tukon Hardness

Tester. In addition, four 6" diameter and two 4" diameter holes were cut through the floor for special services and equipment.

2. Supporting Equipment and Services

A panel that extends the full length of the cell houses all the controls for remote equipment and laboratory services. Remote service is supplied by three Model 4 master-slave manipulators manufactured by Central Research Laboratories. Two of the slaves are located in the mechanical section and one in the optical section. The coverage of the cell by the slaves is illustrated in Figure 2. All house services (air, water, electricity, etc.) are piped into the cell through access plugs located on the bottom of the cell. There are junction boxes and pipe couplings located on each side of the access plug to facilitate easy maintenance or removal. Contaminated liquids resulting from metallographic operations are collected in the storage tanks of the special waste disposal system of the hot laboratories. This system, referred to as the special hot drain, consists of four shielded evaporators and supplementary equipment that dispose of radioactive liquids at the rate of two gallons per hour. A 500 cfm exhaust system with separate ducts to each section maintains airborne contamination at a minimum. Manual service is obtained through a series of portholes in the base of the cell, a 24" x 28" door at the end of the cell, the roof slabs of the cell and the 12" diameter motorized elevator.

3. Metallographic Equipment

Sectioning

The 1114 AB abrasive cutoff machine was made suitable for remote sample cutting by redesigning the recirculating coolant system and the specimen clamp. A small centrifugal pump was placed in the drain line of the coolant system to force the liquid through a line filter into a storage tank. The filter (95% efficient when filtering particles 10 microns in diameter and larger) is contained in a 3" lead disposal cask mounted on wheels, and is connected to the recirculating system with short hose connections; hence, it can be easily disconnected and moved in the cask to another area for disposal. It was observed that as the filter became loaded that the pressure drop across it increased; therefore, a pressure gage was installed on the inlet side of the filter to indicate its condition. The filter is changed when the pressure exceeds 15 psi or the radiation level approaches the tolerance for a 3" lead disposal cask. The coolant is disposed of by a series of valves that route the liquid into the special hot drain (Figure 3). As a means of reducing airborne contamination during cutoff operations, the facility was designed so that the entire exhaust capacity of the cell could be centered at the cutoff wheel by means of blast gates. On occasion, air counts up to 1.5 Roentgen/hr. were recorded in the exhaust system of the cell, while other stations in the vicinity of the cell registered less than 600 disintegrations per minute; 1.36 Roentgens/hr. at a meter is equivalent to 2.22×10^{12} disintegrations per minute with a cobalt-60 source.⁽¹⁾ Although this indicates that the airborne activity is being contained within the cell, portable air monitors are moved into the area for safety reasons when the cutoff wheel is in operation.

Fig. 2—Sketch showing coverage of Model 4 master-slave manipulator.

Fig. 3—Modified recirculating coolant system for the cutoff machine.

When working with samples which must be examined on a given plane, precision cutting adjacent to the plane desired reduces subsequent grinding time. Such sectioning was made possible by designing a removable sample vise which afforded the operator maximum visibility during sample alignment (Figure 4). This vise handles specimens of dimensions up to

Fig. 4—Removable sample vise for the cutoff machine.

1" x 1" x 4" and has an inter-changeable face to permit processing of round samples (Figure 4). It is held in position on the cutoff wheel by two locating pins and a small air cylinder. The specimen assembled in the vise is moved against the cutoff wheel by the master-slave manipulator (Figure 5). Completion of a cut is indicated by a decrease in the amperage drawn by the cutoff motor as indicated by an ammeter placed in the motor circuit.

Mounting

The #1330 Buehler Speed Press was modified for remote sample mounting (Figure 6). The size of the unit was reduced by replacing the outer shell with 1/8" aluminum sheet and relocating the preheater tubes. The preheater tubes were rotated to the vertical position and relocated on the front panel of the mounting press. All three tubes were modified to accept only 1-1/4" diameter bakelite premolds. The screw-type locking mechanism on the top of the die was replaced with a small hydraulic ram. This top ram was connected to a hand pump located on the control panel by a flexible hydraulic hose. The hydraulic pump action was removed from the press and relocated on the control panel. All hydraulic lines were equipped with quick disconnect couplings for easy removal. Only minor changes were made in the electrical circuit. The electrical controls were located in the control panel with leads extended into the cell where the specimen heaters were connected. An additional set of electrical leads were built into the press so that either the split or circular type heating elements could be used.

Past experience has shown that Bakelite is more resistant to high specimen activities than other common mounting materials. As a result, Bakelite is used primarily as a mounting medium.

Fig. 5—Moving sample vise of cutoff machine with master-slave manipulator.

Fig. 6—Modified mounting press.

Kelon liquid plastic is used as a supplementary mounting material. Suitable molds and a small drying oven comprise the necessary equipment for this mounting medium. Glass or plastic tubes, cut into right circular section 1" long by 1-1/4" I.D. serve as ideal molds when one end is sealed to a flat glass plate by a thin layer of vacuum grease. This produces a mold that will contain the liquid until it hardens and also aids in the removal of the specimen from the glass tube.

It is necessary to take special precautions when mounting this specimen in either of the above mentioned materials. Constant force springs (Figure 7) are used to hold thin samples upright. A fixture

Fig. 7—Applicator and negator springs.

consisting of both a stationary and movable post was built to apply these springs to samples (Figure 7). The movable post is connected through a cam to a knob; as the knob is rotated, the posts are spread, opening the spring to accept the sample. When the knob is returned to the original position, the spring clamps the sides of the sample giving it the stability necessary for mounting. The specimen with the spring attached is easily removed from the jig for further processing.

Grinding and Polishing

Of the several available commercial polishing units the "Jarret Precision Automatic Polisher" appeared to be the most appropriate to adapt for remote metallography (Figure 8). The low speed and availability of a variety of laps such as iron, lead and stone make the unit sufficiently versatile for both grinding and polishing operations. It is also possible

Fig. 8—Part of automatic polisher located above cell floor.

to cement various grades of grinding paper to the lapping plates to supplement the numerous grades of grinding compounds used with the lead and cast iron laps. The modifications for cell operation were rather simple. The manual brake was replaced with an AC solenoid brake. The polishing lap and specimen holder drive mechanism were placed inside the cell. The motor and transmission were mounted beneath the floor of the cell and the drive shaft was extended through a one inch hole in the cell floor. An idler pulley added between the polishing wheel and drive shaft tightens the belt drive (Figure 9). The other features of the machine were retained including the specimen holder and accessories. The drains from each machine were connected to a manifold that empties into the special hot drain. There is a separator in the special hot drain that removes most of the abrasives before the water reaches the evaporators. This separator is located under the cell and is readily accessible for cleaning. At the present time two of the four units are used as polishers, one as a grinder and one as a silk lap for processing powder compacts. In the future it is planned to supplement these units with a vibratory polishing machine. The application of the vibratory polishing machine to specific samples has not yet been established, although there are indications that the machine can produce satisfactory surfaces.⁽²⁾

Cleaning

Ultrasonic cleaning has been previously applied to irradiated metallographic specimens.⁽³⁾ The generator presently used in this remote operation is not entirely suited for efficient cleaning. The high frequency, one megacycle, of this unit does not give the required degree of cavitation; however, the unit is being used until a low frequency generator can be obtained. Several types of solvents such as trichloroethylene, perchloroethylene and alcohol have been tested, but it was

Fig. 9—Modified portion of automatic polisher located above cell floor directly beneath assembly shown in Fig. 8.

found that water performs as well as any of these. The cleaning water is disposed of in the special hot drain.

Etching

A particular difficulty in metallographic preparation is the etching of samples for microscopic examination. The methods used in the present installation are chemical, electro-chemical, and cathodic vacuum etching. The activity of irradiated samples seems to determine to some degree the strength of the etching solution required. Past experience indicates that some materials such as U-Nb alloys that are subjected to high irradiation exposure with resulting high activity are extremely difficult to etch; two to three months after their irradiation, certain materials are found to respond normally to standard strength etchants. The exact reason for the behavior of irradiated samples in chemical etchants, is not known at this time. High beta and gamma fields generated at the metal-solution interface may cause this interference. This is evidenced by the fact that while most inorganic acids tend to be stable under irradiation, water and most organic acids dissociate.⁽⁴⁾ Another possible source of interference is that the radiation damage of the sample causes the unusual behavior of the etchant; i.e. transmutation or other irradiation-induced phenomena may alter the etching characteristics of the alloy in a given etchant.

Chemical etchants are used in the conventional manner, either by dipping or swabbing, although at times pouring the etchant over the sample surface produces the desired results. Such flow seems to aid the etching by continually keeping fresh solution on the sample surface.

Electrolytic etching is done using two types of voltage supply. The most common type is a dry cell battery with the leads extended into the cell. The desired electrolyte is placed in a dish containing a suit-

able cathode and the specimen is connected to the anode which consists of a pair of stainless steel tongs. The same incell arrangement is used for a rectified-a-c-voltage supply that is used for electrolytic polishing and etching. The "Disa Electropol" can also be used for either polishing or etching. This unit is simple to modify for remote operation as the power supply and timing controls are separate from the electrolytic cell.

The samples that do not respond satisfactorily to the chemical or electrochemical etchants are then subjected to cathodic vacuum etching. Cathodic vacuum etching has proven very useful in the metallographic examination of dissimilar metal couples.⁽⁵⁾ The unit used for remote etching is unique in that only the components enclosed in the bell jar are inside the cell. The design was patterned after a previous cathodic vacuum etcher⁽⁶⁾ with only a few modifications necessary for this installation.

Viewing (Mechanical Side)

Preliminary viewing of a prepared surface is done in the mechanical section of the cell. A conventional monocular bench microscope was modified for this operation (Figure 6). The image is projected through the wall of the steel cell by means of an optical extender system. The system consists of two parts, a cover glass to replace the eyepiece on the monocular microscope tube and an extender tube 15 inches long. The tube contains a prism in one end to project the image from the microscope to the eyepiece at the other end outside the cell. The system also contains a reducing lens which eliminates the expected increase in magnification due to tube length. In order to maintain the extender tube in a level position, an eccentric bushing was mounted in the access plug for fine alignment. Controls for the coarse and fine focus and the two stage motions of the microscope were extended through the cell face. The control knobs were located in a removable access plug and were connected to the microscope by means of flexible cables. The coarse focus control on the stage of the microscope was extended four inches and a T-handle installed. This permits the quick positioning of the sample under the objective by employing the master-slave manipulators for rough adjustment. Illumination is supplied by a Bausch and Lomb tri-vert illuminator. The leads between light bulb and transformer were extended through the bottom of the steel cell. The quadruple nose piece with a full compliment of objectives (6.5 x, 13 x, 20 x, and 37 x) can be rotated into position by the master-slave manipulator. Since the microscope is utilized only for preliminary examination the iris and field diaphragms are preset and are not adjustable remotely. Due to the position of the microscope in the cell, an additional viewing window was required. This window is 4 inches square and is located in such a position that the stage and objectives are in full view.

Transfer and Storage Equipment

Two methods of transferring samples into the cell are available. Samples that can be placed in small containers and handled with 6 ft. tongs are introduced into the cell either through portholes located in the front of the cell or by an elevator through the floor of the cell. Highly radioactive samples can be introduced into the cell by removing

the roof slabs and lowering a sample cask into the cell where the samples are removed with the master-slave manipulators. Transferring samples from one section of the cell to the other is done by a small four-wheeled cart that is run on a rack and pinion operated from the cell face.

The storage facility for mounted metallographic samples is a 4 inch lead cask that has been welded to the bottom of the cell (Figure 10). The cavity of this cask is accessible through the floor plug that can be removed. The samples are placed in a rack (Figure 10) and lowered into the cask for storage, thus reducing the radiation damage to the viewing windows and other optical equipment located in the cell. The rack is made of a light weight material and is easily handled by the master-slave manipulator.

Photography

The new cell has facilities for both macro- and micro-photography. The macro-photography is done on a modified Vickers Metallograph. The carbon arc unit was replaced with a "Zircon" lamp that is mounted on a small laboratory scissors jack. Consequently, the lamp elevation can be adjusted by the master-slave manipulator. The coarse focus control was extended through the cell face. The macro-image can be projected through a port in the bottom of the cell onto a front surface reflector that transmits the image through a tube to a ground glass located in the control panel. This unit has a minimum tube length identical to the maximum tube length of the regular unit. The illumination must be adjusted prior to the introduction of radioactive samples into the optical section of the cell. This is done by removing two access plugs and adjusting the position of the components by hand until a satisfactory image is obtained on the ground glass. The master-slave manipulator is used to position the sample on the stage of the Vickers macro-camera.

The micro-photography is done on a modified B & L Research Metallograph (Figure 11). Shielding patterned after that of Oak Ridge and equivalent to that used in the body of the cell was placed around the stage and vertical illuminator of the metallograph. A door was located in the shielding so that the operator has access to the iris and field diaphragms, the objective, and the water cell. The water cell and condensing lens are an integral part of the shielding on the lamp house side of the metallograph. The stage controls are extended through the cell wall by means of flexible cables and right angle drives. A third motion was added to the stage of the Metallograph so that the specimen could be rotated. The cable controls of the stage are connected to a gear box that is mounted on the table of the Metallograph and then are extended to the rear support of the optical bed. This allows the stage controls to be operated conveniently regardless of the bellows extension. A 1/8 inch copper sheet was made to fill the area not covered by the stage to prevent samples from accidentally being dropped into the well formed by the shielding that surrounds the Metallograph. The prism used to divert the image for visual examination of a sample is controlled by a push-pull lever in the front shielding panel. Visual examination is done through an extender system that is connected to the monocular viewing tube of the microscope through the shielding panel.

Fig. 10—Cross section of storage cask and sample rack in steel cell.

Fig. 11—B&L Metallograph installation.

The remote and fine focus controls are standard equipment on the Metallograph. The coarse focus control was coupled to a knob on the face of the shielding by a link chain for easy focusing when the visual examination system is being used. The carbon arc illuminator and camera attachment are readily accessible to the operator.

Hardness

A remote Tukon Micro-Hardness Tester built by the Wilson Mechanical Instrument Division of American Chain and Cable Co., Inc. serves as a remote hardness tester (Figure 12). It is located in the optical section

(a)

(b)

Fig. 12—Remote hardness tester. (a) Arrangement of hardness tester components outside the cell. (b) hardness tester inside the cell.

of the cell and the major part of the instrument extends through a 24 inch x 12 inch hole in the cell floor. A lead brick chimney is constructed around the section that extends below the cell floor to protect the operator from radiation. A 4 inch square window is positioned in the back wall for viewing the stage of the Tukon. This unit is designed for remote operation by the manufacturer; hence, few modifications were necessary. The image is projected to the filar eyepiece through an 18 inch extender system similar to that previously described for the bench microscope. This extender differs in that the prism is located on the microscope rather than the extender tube. The focusing controls are located in an access port below the extender system and are connected to the microscope by flexible cable. A supplementary control was added to the unit to move the stage from the indenter to the microscope. This motion incorporates a rack and pinion connected through a speed reducer (for necessary torque) to the control knob by flexible cable.

4. Procedures and Special Applications

Bakelite premolds are used as the basic mounting material. A small amount of Precisionite or red bakelite is used to obtain a hard surface for polishing. Samples are identified on the back face by a tag made of aluminum or stainless steel. This tag is stamped with the sample number and is imbedded in the bakelite when the specimen is mounted. The mold is lubricated with Dow Corning stopcock grease before each sample is mounted to prevent the mount from sticking. This lubricant serves the same purpose when samples are mounted in liquid plastic.

Kelon liquid plastic has a setting time of two to four hours, but past experience has indicated that a time lapse of eight to ten hours between casting and drying results in a better mount. The mounts are cured for two hours at 150°F before they are ejected from the molds. For plastic mounts, a poultry band is used for sample identification. The sample number is stamped on the wide section of the band and the

Fig. 13—Specimen holder for automatic polisher.

narrow section is bent in such a manner that the numbered section appears at the back surface of the mount. Liquid plastic is used primarily when the pressure and temperature involved in bakelite mounting would be harmful to the sample or when the material is porous or fragile. Fragile materials are mounted in liquid plastic prior to the cutting operation in order to get a section satisfactory for processing. Liquid Plastic can be forced into the open pores of a porous sample by a hydrostatic pressure of approximately 150 psig. The penetration of the plastic into the open pores is very beneficial in the preparation of irradiated sintered powders as it holds the powder together while the sample is being processed.

The mounted samples are placed in the standard specimen holder of the automatic polisher for processing (Figure 13). The specimen holder

has openings for twelve samples. When fewer than four samples are processed dummy blocks are inserted to balance the holder. The grinding operation is performed on a #600 grit silicon carbide stone lap with running water as a coolant. The water washes the grinding residue from the lap and reduces the amount of airborne particles. Average grinding removes sample material at the rate of 10 mils per hour and is continued until all the surfaces are ground to the same plane. The grinding rate can be increased considerably by adding 600 grit silicon carbide abrasive in water to the stone lap.

The specimen holder is removed from the grinder with the manipulators and is cleaned thoroughly with running water. This first cleaning is supplemented by ultrasonic cleaning.

The specimen holder is then transferred to one of the polishing units for final lapping. The gamal covered polishing wheel contains a glass disc between the metal lap and the cloth. A mixture of Linde B (0.1 micron sapphire) and water is applied to the lap from a polyethylene shaker bottle with a manipulator. The glass plate provides a flat surface for polishing and prevents the acids used for attack polishing from corroding the metal polishing lap. A dilute HNO_3 -HF solution serves as the acid for attack polishing.⁽⁷⁾ The combination of metal dissolution by the acid and abrading by the Linde B results in a polished surface relatively free of disturbed metal and suitable for final etching. By varying the amount of acid on the lap it is also possible to satisfactorily etch samples in this manner. The time required to polish one sample holder containing a dozen samples is between one and two hours.

Sintered UO_2 compacts are handled by a similar procedure except that the final polishing is done on a silk covered lap, with diamond compound as an abrasive and mineral oil as a lubricant. In most cases, the compact surfaces resulting from stone lap grinding can be satisfactorily polished with a 0-1 or 0-2 micron diamond compound.

Samples requiring special preparation for X-ray diffraction are handled by general metallographic procedures with but few exceptions. Bakelite raises the background intensity of the spectrometer patterns reducing the resolution power; hence, Kelon liquid plastic is used as it has little effect on background intensity. Since a cold worked sample surface is deleterious to X-ray patterns, samples to be X-rayed were either electro-polished or etched deeply after mechanical polishing to remove the possible disturbed metal surface.

On occasion it is necessary to obtain samples of inclusions for X-ray analysis. This is done by first dislodging the particle with the diamond indenter of the Tukon hardness tester and then lifting the particle from the surface with a piece of Scotch tape attached to a rubber stopper. The particle is then monitored for activity, enclosed in a bottle, and removed from the area for further processing.

Mica-filled bakelite is generally a suitable mounting medium for good edge preservation of metallographic samples. However, when preparing samples containing very thin oxide films for examination at 500x or higher, it is necessary to use small metal clamps. The clamps must be strong enough to resist bending but small enough to fit inside the mold

of the mounting press. A 0.020" thick strip of annealed copper is placed between the sample and the clamp. The clamp is then placed in a special vise which is closed pressing the copper tightly against the oxide surface. After the bolts are tightened, the clamp is mounted in a 1-1/4" diameter bakelite mold. An exploded view of the components for performing this operation remotely is shown in Figure 14. In a particular application for fuel plates the clamps are made of the same material as the cladding thus eliminating any anodic protection that a dissimilar metal clamp may afford, the sample being etched.

Fig. 14—Assembly vise and metal clamp components for oxide film preservation.

Metallographic Results

Typical metallographic results of irradiated samples prepared and photographed remotely are shown in Figures 15 through 22 inclusive with descriptive captions. Detailed sample histories and compositions were not mentioned for reasons of document classification.

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(a)

(b)

Fig. 15—Hydride formation at surface of Zircaloy tube exposure, approximately 1×10^{20} NVT. (a) Attach polished and etched in a solution of 40% HNO_3 —50% H_2O_2 —10% HF. Magnification 250 $\times$. (b) Same preparation as (a). Magnification 500 $\times$.

Uranium alloy
Zircaloy cladding

Fig. 16—Longitudinal section at the interface of an irradiated metal couple, etched by acid polishing with dilute HNO_3 —HF, 54 at.% fissioned of total uranium.

Fig. 17—Two-phase structure uranium alloy revealed by acid polishing with dilute HNO_3 —HF, 20 at.% burnup of total uranium.

(a)

(b)

Fig. 18—Irradiated uranium alloy etched in an aqueous solution of 4 g CrO_3 , 10% HNO_3 and 2% HF . (a) Alpha lamella in a gamma matrix irradiated to 0.001 at.% burnup of total fuel alloy. Magnification 500 $\times$. Fuel partially transformed prior to irradiation. (b) Gamma quenched irradiated to 0.48 at.% burnup of total fuel alloy. Magnification 500 $\times$.

Fig. 19—Partially transformed structure of a uranium alloy, etched in an aqueous solution of 4 g CrO_3 , 10% HNO_3 and 2% HF irradiated to 0.03 at.% burnup of alloy. Magnification 1500 $\times$.

Fig. 20—Oxide film preservation on an irradiated Zircaloy sample, as polished. Magnification 1000 $\times$. Exposure, 2.6×10^{20} NVT.

Fig. 21 —Particle preservation in a silver matrix—irradiated sample as polished. Magnification 250 $\times$. Exposure, 18.9×10^{20} unperturbed NVT.

(a)

(b)

Fig. 22 —Sintered irradiated UO_2 compacts. (a) Structure developed by attach polishing with dilute HNO_3 — HF solution. Irradiated to 0.352 at.% burnup of total uranium. Magnification 100 $\times$. (b) Etchant 10% H_2SO_4 in H_2O_2 irradiated to 0.344 at.% burnup of total uranium.

References

1. Los Alamos Physics' Handbook.
2. Krill, F. M., "Vibrating Polishing of Metallographic Samples," Metals Progress, July 1956.
3. Feldman, M. J., "Advantages of Ultrasonic Cleaning," Nucleonics, March, 1954.
4. Allen, A. O., "Decomposition of Water and Aqueous Solutions Under Mixed Fast Neutron and Gamma Radiation." AECU-1413, 1951.
5. Padden and Cain, "Cathodic Vacuum Etching", Metals Progress, July 1954.
6. Padden Roll, "Remote Cathodic Vacuum Etching", to be published.
7. Woods, H. W., "Attack Method of Preparing Tungsten", Metals Progress, February 1947, p.261.

Coloration of Shielding Window Glasses

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Abstract

Gamma irradiation tests on commercially produced shielding glasses with sp. gr. of 2.7 and 3.3 have been extended to 10^{10} r. Data on the rate of fading after exposure and the rate of re-coloring with re-exposure are included. Data are presented on the effect of the intensity of irradiation and the temperature during irradiation on the total coloration produced by a given exposure. Results of tests on experimentally melted glasses with improved resistance to coloration are included.

Introduction

Shielding windows have been used extensively for the remote viewing of research and process operations involving intensely radioactive materials.¹ The development of special glasses, for use in windows, which have increased resistance to coloration induced by gamma radiation have been under investigation since 1949.² The coloration of glass by exposure to gamma radiation is effected primarily by the energy absorbed per unit volume. However, other factors such as the intensity of the radiation and the physical environment of the glass during irradiation effect the coloration.

Test Procedures

The source of fission product gamma rays used in most of these tests was MTR spent fuel elements which had cooled from 30 to 120 days. The radiation intensity incident at the sample is in terms of the absorbed dose in water as measured by ferrous sulphate dosimetry. (One roentgen in water was taken as equal to 93 ergs/gm rather than the presently

accepted value of 97 ergs/gm). Some of the irradiations were made with a 350 curie radio-cobalt source. All other test procedures were similar to those previously reported;² with the exception that the surfaces of the 2.7 as well as the 3.3 sp. gr. glass samples were aluminum oxide polished after a long exposure.

Test Results

The coloration of representative samples of commercial shielding glasses after exposure to 10^9 r was reported a year ago. At that time, there was an indication that the rate of coloration might increase sharply after an exposure of 10^9 r. The irradiation of these same glass samples has been continued to an accumulative exposure of 10^{10} r with the result that no abrupt change in the rate of coloration has been observed.

Fig. 1—Coloration of protected silicate glass by exposure to a fission source.

The range of the relative transmittance as a function of exposure for representative samples of 3.3 sp. gr. glass produced by the Pittsburgh Plate Glass Company and the Corning Glass Works is shown in Fig. 1. As the exposure increases, it can be seen that there is a proportionately greater increase in color that fades rapidly at room temperature than for color that persists for 100 hours.

The relative transmittance of a 2.7 sp. gr. protected silicate glass produced by the Pittsburgh Plate Glass Company is shown in Fig. 2. In contrast to the 3.3 sp. gr. glass, the coloration that persists 100 hours after exposure more than doubles as the exposure is increased from 10^9 to 10^{10} r. The two glasses should be compared for a thickness which absorbs the same amount of energy. At 0.7 mev one inch of 3.3 sp. gr. glass is equal to 1.34 inches of 2.7 sp. gr. glass. In addition, the 2.7 glass has about a one per cent lower internal luminous transmittance

Fig. 2—Coloration of protected silicate glass by exposure to a fission source.

than the 3.3 glass. On the basis of total light transmittance, the 2.7 glass is about equal to the best of the 3.3 glass after an exposure of 10^8 r. For exposures greater than about 5×10^8 r, the 2.7 glass has a total light transmittance less than the lowest of 3.3 glasses.

Fig. 3—Fading of protected silicate glass after exposure.

Figure 3 shows the fading curves for the 3.3 and 2.7 sp. gr. glasses after an exposure of 8.8×10^9 r. The change in light transmittance is nearly proportional to the log of the fading time between 10 and 1000 minutes and the curves tend to flatten above and below these times.

It was found previously² that most of the color which faded in 100 hours was restored by about 10^7 r exposure. For purposes of comparison identical tests were run on samples of 2.7 and 3.3 sp. gr. glass. Samples of each glass were exposed to 10^7 , 10^8 and 4×10^9 r. After fading for one week, the glasses were re-exposed to 10^6 , 10^7 and 10^8 r. It was found for both glasses that about one-half of the original coloration was restored by the 10^6 r exposure and that essentially all of the color produced by the original exposure was restored by the 10^7 r exposure.

To determine if this phenomenon of more rapid coloration on re-exposure persists for a long period of time, a sample of 3.3 glass which had received an exposure of 1.4×10^8 r was faded for nearly one year. It was then re-exposed with an identical control that had not been previously exposed. The two samples received an exposure of 10^6 r to a Cobalt 60 source at an intensity of 4×10^4 r/hr. The control had a relative transmittance after exposure of 99 per cent while the re-exposed sample had a relative transmittance of 98 per cent.

Only a preliminary investigation of the effect of radiation intensity upon the coloration has been carried out. For exposure to a fission products source an increase in the intensity from 4×10^5 r/hr. to 1.3×10^6 r/hr. resulted in a decrease in the relative transmittance of 1.0 to 1.5 per cent per inch in the exposure range from 10^7 to 10^8 r.

The effect of the cerium content in experimental 3.3 sp. gr. glasses melted in our laboratory was investigated. The lead silicate glass contained from 10 to 20 weight per cent of alkali oxide. The cerium was added to this glass in the form of CeO_2 in the range of 1 to 10 weight per cent. The coloration as a function of cerium content is shown in Fig. 4. It can be seen from this curve that for a minimum attenuation at a given exposure there is an optimum cerium content. Under these conditions of exposure, the optimum content for 10^6 r is 1.5 to 2.0 per cent of CeO_2 . For the conditions of the 10^8 r exposure, it is about 2.2 to 2.6 per cent. When similar glasses are melted commercially the cerium content may need to be adjusted slightly from these values to achieve the minimum attenuation for similar conditions of exposure.

Preliminary tests have been carried out to determine the effect of the glass temperature on the coloration of a 3.3 sp. gr. glass containing about 3 per cent of CeO_2 . Four identical samples each maintained at different temperatures were irradiated with a Cobalt 60 source to 10^8 r. There was no significant change in light transmittance which could be attributed to the temperature over a range of 30 to 125 degrees centigrade. Hence, it appears that this glass could be used at high radiation intensities where gamma heating would maintain the glass at an elevated temperature.

Conclusion

Further investigation has shown that present commercial 2.7 and 3.3 sp. gr. glasses cannot be used satisfactorily in windows receiving an incident exposure in excess of 5×10^8 to 1×10^9 r. However, experimentally melted 3.3 glasses with increased cerium content show promise of being used in windows receiving an incident exposure up to 10^{10} r.

Fig. 4—Coloration of experimental 3.3 sp. gr. glasses. The attenuation per inch for each cerium content was reduced by a factor of 1.02 to approximate the reduction in attenuation which will occur when a similar glass is melted commercially. The 10⁶ r exposure was at an intensity of 5×10^4 r/hr with Co⁶⁰. The 10⁸ r exposure was at an intensity of 1×10^6 r/hr with a fission product source. All attenuations are for 10 min after exposure.

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References

1. Ferguson, K. R.: Windows for Remote Viewing, Fourth Annual Symposium on Hot Laboratories and Equipment, Washington, D.C. Office of Technical Services, Dept. of Commerce, Washington, D.C. TID-5280(suppl. 1), pp. 73-83, January 1956.
2. Ferguson, K. R.: Coloration of Shielding Window Glasses, Proceedings of the Fifth Hot Laboratories and Equipment Conference, Vol. III, pp. 218-21, Pergamon Press, 1957.

Shielding Window Design for the EBR-II Process Building

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Abstract

The shielding window design for the facility for processing and refabrication of fuel elements for the Experimental Breeder Reactor No. II is described. The window is designed for a gamma radiation intensity up to 10^6 r/hr. for eight hours per day. Steel shutters will shield the window when it is not in use. The design reasons for the window configuration and the choice of materials are discussed. Operation of the plant in an inert atmosphere requires a gas tight seal at the window aperture. The design details of the seal and test data on its performance are included. Also, data are included on the computed light transmittance of the window before and after a 10^{10} r exposure.

Introduction

The large shielded cell required to carry out the pyrometallurgical processing and fuel element fabrication to be used in the closed fuel cycle for the EBR-II fast breeder reactor has been described.¹ All general operations in this cell will be viewed through shielding windows. Inspection and other operations requiring magnified or close vision will use specially designed optical periscopes. Service operations on the manipulator equipment requiring a view from the top of the cell will use a wide angle optical viewer.

From the plan view of the cell (Fig. 1) it can be seen that the eight windows in the wall of the central control room allow nearly the

entire operating area of the argon-filled cell to be viewed. By moving the eyes from one side of the window to the other, the included angle between the limits of vision is a little over 90°. The eight windows in the outer circular shielding wall will afford viewing for all the operations in the various process steps. These windows also offer a view of the small sectors of the operating area not directly visible from the central observation room. The window liners installed in the outer circular shielding wall between the existing windows will be used either for additional windows where required by the process operations

Fig. 1 —Plan view, EBR-II process building windows.

or for the installation of the optical periscope. The operating area in the argon-filled cell is 16 ft. deep and the outer circumference is 195 ft. with 5-ft-thick barytes concrete walls. The air cell is 15 ft. wide by 47 ft. long and contains nine windows on about 10-ft. centers.

An elevation view of the operating area in the argon-filled cell is shown in Fig. 2. Again, it can be seen that most of the operating volume is viewed from the central control room and that a good view of the process equipment is obtained from the windows in the outer shielding wall. With the eyes at a fixed position one foot from the window, the included angle of view is about 35 degrees. By moving the eyes from the top of the window to the bottom of the window, the included angle between the limits of vision is a little over 90 degrees. In the outer shielding wall the tapered inner section is offset downward to give a better view of the floor. The same window configuration is used for the inner shielding wall except that it is inverted to give a larger angle of view upward. The floor of the central observation room is 3 ft. below the floor of the outer operating annulus. The center line of the inner windows is at about 66 inches above the floor rather than 54 inches for the outer windows to afford a more natural view of the overhead manipulator equipment while the operator is in a standing position. In elevation the entire operating volume is viewed either through the inner or outer windows up to the level of the manipulator-crane bridges.

Fig. 2—Elevation view, EBR-II process building windows.

Fig. 3—Shielding window. All 3.3 sp. gr. glass except for the cover plates on the liquid-filled section and the gas containment plate. The edges of the steel framed castings may be insulated to reduce thermal stress.

Description of the Window

A planned view of the window is shown in Fig. 3. The window is of the all-glass type and consists of essentially two major sections. These are separated by a glass plate which forms the gas seal and contamination barrier. The outer section is about 40 inches thick and is oil laminated. It consists of about 9-in-thick 3.3 specific gravity glass castings; the outer casting is about 33 inches high by 36 inches wide and the inner casting is about 24 high by 30 inches wide. The inner section of the window will consist of a number of 3.3 specific gravity glass castings held in two frames. The larger casting is 36 by 36 inches. The frame holding this casting is hinged to swing into the cell for ease in cleaning the inner glass surfaces and to facilitate possible replacement of this section. The other frame will require a special remotely operated fixture in the event it ever needs to be removed for replacement or for cleaning of the glass surfaces.

The "hour-glass" shaped window has been selected because it affords a larger over-all angle of view for a given amount of glass than the more conventional tapered window. This configuration also gives a minimum size for the glass plate which forms the gas seal for the window. This primary sealing member is located about 18 inches from the cell interior where the radiation intensity is reduced by a factor of one thousand. The total dose at the gaskets will be 10^7 r for a maximum operating intensity of 10^6 r/hr. and for a total operating period of 10^4 hours. For this lifetime dose a number of elastomer materials can be used. One of the more radiation resistant materials which has been tested is an "O" ring made from polyethylene tubing. The "O" ring is formed by thermal butt welding. Quantitative leakage tests have been made on tubing of 5/8, 1/2 and 3/8 in. O.D. with a 1/16 inch wall. All leakage rates were less than 10^{-5} cu. f/min. per linear foot of gasket for a pressure differential of 5 inches of water. The window openings require about 200 linear feet of gasket which is estimated to give 20 per cent of the design leakage of the cell. The seal will consist of two concentric "O" rings to facilitate leak testing. An additional seal would be formed at the observer's side of the window between the oil-filled window section and the window liner by means of a flat gasket. This seal would be readily repairable, and in the event of leakage at the inner seal, the volume between the two seals could be slightly pressurized with argon gas. The dust seals between the inner window sections and the liner may be either a replaceable polyethylene tubing seal or a copper-asbestos seal.

The void between the oil-filled window section and the liner, required for access to the inner gas seal, will be filled with a material such as steel shot. This allows flexibility and independence in the design of the liner.

A 6-in.-thick steel shield or shutter will be moved in front of the window when it is not being used routinely to protect the glass and the seals from radiation damage. In the case of one-shift operation, this will lengthen the life of the windows by at least a factor of three. In addition, at such times when the oil-filled section of the window is required to be removed for service, or when the inner gas seal requires repair, the movable shield plus the inner glass sections of the window will give an attenuation of about 10^5 .

The radiation induced coloration of the shielding glass limits the useful lifetime of the window. A lifetime of 10^4 hours gives an incident exposure of 10^{10} r for a design intensity of 10^6 r/hr. Tests on commercial 3.3 sp. gr. glasses have shown that they cannot be used at this large exposure.² Commercial glass samples tested have had a light attenuation at 550 millimicrons of 1.13 to 1.16 per inch after 10^8 r and 1.20 to 1.29 per inch after 10^{10} r. It has been found with glasses melted experimentally in our laboratory that if the cerium content is increased from the present commercial range of 1.3 to 1.8 weight per cent of CeO_2 to about 2.5 to 3.0 per cent, that the light attenuation for 10^{10} r is reduced to 1.12 per inch.³ This value is extrapolated from measurements in the range of 10^8 to 10^9 r.

The light attenuation for experimental glasses melted in our laboratory as a function of cerium content and exposure is shown in Fig. 5. For a minimum attenuation at a given exposure there is an optimum cerium content. It appears that considerable decrease in the total attenuation for a window can be achieved by increasing the cerium content of the glass in steps as the thickness of the window increases. About one per cent of CeO_2 is a good choice for the glass for the observer's side of the window up to a point where it will receive 10^6 to 10^7 r in 10^4 hours; since this amount of cerium introduces very little attenuation before exposure and since the attenuation after a 10^6 r exposure increases very rapidly with lower concentrations. A glass to be used in the window where it will be exposed to 10^7 to 10^8 r in 10^4 hours should have a cerium content of about 2.2 per cent which is above the content of the present commercial glasses. For an exposure range from 10^8 to 10^{10} r, the cerium content needs to be increased to nearly 3 per cent. Further increase in the cerium content for this exposure range has not decreased the attenuation in the experimental glasses we have melted and tested. In commercial glasses the cerium content may need to be adjusted slightly from these values to achieve the minimum attenuation.

The accumulative light attenuation as a function of window thickness before and after an incident exposure of 10^{10} r is shown in Fig. 6. The radiation intensity incident at various depths into the window was computed for spent fuel from a fast reactor after 135 days of operation and 15 days cooling.⁴ The radiation was assumed to be a parallel beam and the energy absorbed at a given point in the window was estimated from the radiation spectrum at that point. The light attenuation per inch for the commercial glass before exposure was taken to be 1.03. For the experimental glasses before exposure, a value of 1.02 was used for the No. 1 glass containing 1 per cent of cerium; 1.05 for the No. 2 glass with 2.2 per cent of cerium; and 1.07 for the No. 3 glass with 3 per cent of cerium. If the No. 1 experimental glass is used for the oil-filled section of the window instead of present commercial glass, there is a saving in light attenuation of about 50 per cent both before and after 10^4 hours of exposure. For the glass in the first steel frame of the dry section, the average attenuation per inch for 9 inches of the No. 2 experimental glass after exposure is 1.07; for the 10 inches of the No. 3 experimental glass in the hinged frame, the attenuation is 1.11 after exposure. The attenuation for the total window thickness is 8.4

Fig. 4—Temperature profile in a window induced by gamma heating. Thermal conductivity is $0.002 \text{ cal-cm/sec-cm}^2\text{-}^\circ\text{C}$. One dimensional heat flow in a continuous medium was assumed, and the peak temperature was taken at a depth of three relaxation lengths for each gamma energy group.

Fig. 5—Coloration of experimental 3.3 sp. gr. glasses. The attenuation per inch for each cerium content was reduced by a factor of 1.02 to approximate the reduction in attenuation which will occur when a similar glass is melted commercially. The 10^6 r exposure was at an intensity of $5 \times 10^4 \text{ r/hr}$ with Co^{60} . The 10^8 r exposure was at an intensity of $1 \times 10^6 \text{ r/hr}$ with a fission product source. All attenuations are for 10 min after exposure.

Fig. 6—Accumulative light attenuation as a function of window thickness before and after an incident exposure of 10^{10} r. The radiation intensity incident at each point in the window is given for spent fuel from a fast reactor with the gamma energy spectrum shown in Fig. 4. The computation which allowed for "broad-beam" geometry gave a nearly constant 10th-value layer through the window of 6.5 in.

before exposure and increases by a factor of about 2.3 to 19 after exposure. For a design having ten glass-to-air surfaces, these values must be increased by a factor of 1.7 to allow for surface reflections.

Another radiation effect besides coloration is cracking of the glass induced by gamma heating. An approximate representation of the temperature profile through the window is shown in Fig. 4. This profile is based on the assumption of one dimensional heat flow in a continuous glass medium. The analysis used has been described previously.⁵ The approximate 50 degrees centigrade rise in the first 10 inches of the window is more than can be safely allowed in a single glass casting. Several ways of reducing the thermal stress are under consideration. One way, of course, is to use several castings in this front section at the sacrifice of increased surface reflection. Another way would be to cool the window by forcing filtered gas from the cell between the two large inner castings. Still another possible solution is to use a thin cover plate separated from the large casting by the proper insulating gas space so that the front surface of the casting will be hotter than any point in its interior. In this case, the large temperature rise will occur across the gas space rather than in the glass. Electrical analog studies and physical measurements using electrical heaters laminated between layers of glass are planned by our laboratory to determine the temperature profile in three dimensions for the inner sections of the

window both for transient and static conditions. Preliminary studies employing infra-red heating have been carried out by the Pittsburgh Plate Glass Company.

Another radiation effect which can result in breakage of the inner section of the window is dielectric discharge.⁶ One such incident has occurred in a shielding window. A thick glass casting can be thought of as a large capacitor which is charged by secondary electrons from the Compton effect. The glass breaks when this charge is released in some manner such as a mechanical blow to the surface. Radiation intensity as low as 10^3 r/hr may be sufficient to keep a 8-in-thk. casting charged.⁷ However, a thin plate, such as one inch thick, may be essentially immune to breakage. This effect is being studied by Dr. Vaughn Culler of the Corning Glass Works. The one inch thick plate shown at the inside of the window in Fig. 3 will serve, then, to protect the large glass casting from breakage resulting from dielectric discharge, mechanical impact, or transient thermal stresses.

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References

1. Bernstein, G. J., J. E. A. Graae, M. Levenson and J. H. Schraidt: Design for a Remotely Operated Facility for High Temperature Processing of Spent Reactor Fuel, Sixth Hot Laboratories and Equipment Conference, Chicago, March, 1958.
2. Ferguson, K. R.: Coloration of Shielding Window Glasses, Proceedings of the Fifth Hot Laboratories and Equipment Conference, Vol. III, pp. 218-21, Pergamon Press, 1957.
3. Ferguson, K. R. and R. L. Reed: Coloration of Shielding Window Glasses, Sixth Hot Laboratories and Equipment Conference, Chicago, March, 1958.
4. Burris, Leslie Jr. and Ira G. Dillon: Estimation of Fission Product Spectra in Discharged Fuel from Fast Reactors, ANL-5742, July, 1957.
5. Ferguson, K. R. and W. B. Doe: The Performance of Shielding Windows at High Radiation Intensity, The Third Information Meeting on Hot Laboratories, Reprints of the "Official Use Only" and "Unclassified" papers from Secret report BNL-302, 12-54-200, May, 1954.
6. Gross, Bernhard: Irradiation Effects in Borosilicate Glass, Physics Review 107, pp. 368-73, July, 1957.
7. Culler, Vaughn, private communication, Corning Glass Works, Corning, New York.