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July 31, 1964

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## AN OPERATING TRANSCURIUM ELEMENT RADIOCHEMISTRY FACILITY\*

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## ABSTRACT

The Livermore neutron cave facility has been modified to accept extended-reach master-slave manipulators. Two additional process boxes that are fully interchangeable with the original enclosure have been constructed to accommodate specialized equipment necessary to remotely perform transcurium element chemistry. Some of the items included in these enclosures are: high geometry fast-neutron detectors for the determination of spontaneous fission content of process solutions, electro-mechanical devices to count and collect a predetermined number of drops of ion-exchange eluate per tube, built-in console-operated chemical process system, and an ultramicro remote pipetting system for the radioanalysis of very alpha active solutions.

## INTRODUCTION

The basic configuration of the Livermore neutron cell was reported at the 1961 Chicago meeting.<sup>(1)</sup> This unit, which is a water-shielded cave, can be equipped with several different containment enclosures. The original cell box is outfitted with electrically driven polar manipulators. The biological shield has recently been modified to accommodate a special version of extended reach-master-slave manipulators as well; these units are used to perform chemical separations in the high alpha enclosures. The most recently constructed boxes include console-operated process systems to reduce the amount of manual manipulation required of the operator. These

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\*This work was performed under the auspices of the U. S. Atomic Energy Commission.

systems, which are described elsewhere, <sup>(2)</sup> have been in use at Livermore for some time.

One of the major difficulties in the isolation of transcurium elements is the necessity of working with very small volumes of solution. For example, the ion-exchange columns used to fractionate the purified actinide fractions frequently are less than 10 inches high and 1/4 of an inch in diameter. Other severe requirements are that equipment used to perform this work must be capable of operation in high concentrations of hydrochloric acid fumes, be resistant to organic solvents, and operate routinely in severe radiation fields. Still other requirements are that the equipment must be simple to operate, and have minimum service requirements.

For these reasons, the major portion of the equipment is designed to be easily replaced and to function with the aid of master-slave manipulators.

Manipulator System. It is essential to be able to reach as much within the enclosure with the slave fingers as possible so as to reduce the amount of shielded space required for performance of chemical operations. At the same time, the contamination levels encountered in these operations demand air tight seals throughout the entire system.

Fig. 1 indicates the scheme utilized to accomplish the above requirements. The manipulator through-tubes, which are filled with maximum possible polyethylene shielding, penetrate the biological shield 88 1/2 inches above floor level. This places the master hand controls at about 3 feet elevation - or about 6 inches above the control console. (This cell is designed for sit down operation). The hand position can be varied for operator comfort by use of the X-Y-Z electrical extension controls. [The switch for operation of the X-Y controls is located near the viewing window, the Z control is on the master hand.]

The containment box is constructed with two large bag ports mounted at a low angle to allow the manipulator extension tubes to enter the booting horizontally. As the slave end enters the boot the arm is swung downward by use of the electrical Y control (the Z motion is kept fully retracted). This process is continued until the extension tube is in the vertical position and the master end of the manipulator is in its proper horizontal location. Fig. 2 illustrates the method of insertion into the manipulator boot. Note that insertion into the boots can be performed either by moving the manipulators with respect to the wall, with the box in normal position, or by moving the box with respect to the manipulators which are left in a stationary position in the wall. Both methods can be performed with ease. The former method is used when extracting manipulators for repair during a process operation; the latter used when cell boxes are exchanged between operations.

Manipulator Booting. The booting is fabricated from vinyl sheeting and utilizes a method of folding to obtain the bellows configuration rather than by welding. The method of fabrication is described elsewhere.<sup>(3)</sup> As is shown in Fig. 1, the boot contains two separate bellows sections; the first accommodates the power extension section and the second "the manual extension tube." The upper bellows is much larger in diameter as it must clear the azimuth rotation pulleys and extension cables; the lower is only large enough to clear the manual extension tube. The upper bellows rides over, and is supported by, a mylar telescoping sleeve. The lower end of this bellows joins an aluminum disc that connects to the manipulator body above the manual extension. This connection is formed by three snap-action teeth that engage a mating flange attached to the power extension tube below the pulleys. This coupling can easily be made or broken remotely by the use of only one manipulator.

Booting Supports. The boots are supported by a gimbal-type device made such that the outer ring rotates freely about the vertical axis. In effect, this is a large ball bearing that is suspended externally by a universal joint of the type

commonly used to support gyroscopes or compasses. The manipulator extends through the hollow center of the device and the upper bellows section of the boot attaches to the inner ball bearing race. This device allows the boot to turn freely around the manipulator, with the transition piece between the enclosure port and the bellows absorbing the circular motion by a wrapping action. This action does not interfere with the freedom of motion of the manipulator.

Fig. 1 illustrates the method of support for the manipulator boots. The floating gimbal is suspended by twin 1/16 inch diameter cables that are controlled from the cave face. To remove the manipulator from the boot, these cables are lowered thereby allowing the full weight of the boot to be taken by the snap-lock flange. This weight disconnects the flange and the boot slumps down until the transition section is fully extended. The manipulator is then withdrawn as indicated in Fig. 2.

Fraction Collector Device. Fig. 3 is a photograph of the remote end of the electromechanical device that is used to count individual liquid drops and to collect them in glass tubes.

The drops from the glass tip of the ion-exchange column pass through a detector head which contains a small light bulb oriented at  $120^{\circ}$  to a solid state photo-diode. As the drop falls, the light reflected from the inner surface of the drop passes across the photo-diode; the output pulse is amplified and used to drive a mechanical scaler. The scaler can be preset to trip at any number of events from 1 to 400, and advances the turntable when its preset value is reached. The scaler is then automatically reset. The control circuit also actuates a second set of two registers that indicate: (1) the total number of drops that have fallen and (2) the total number of drops that have been collected in the tube under the detector. (The second register is reset to zero each time the tube is changed). <sup>(4)</sup>

Either of the two turntables mounted on the "lazy susan" can be used with either detector head. The main table, which supports the two smaller tables, can be remotely positioned in any one of 6 positions that line tubes up under the column tips.



Fast Neutron Detector. Each box contains a corrosion-proof tube that extends through the floor of the cell box into a water filled shield. (See Fig. 1.) Surrounding the tube at its lower extremity is a multi-ring fission detector. Each of the oppositely polarized rings is electroplated with approximately  $500 \mu \text{ gm/cm}^2$  of very highly enriched  $\text{U}^{238}$ . This isotope fissions only when the incident neutron is greater than 700 keV energy. As the average energy of neutrons from spontaneous fission is about 1 MeV, only those neutrons will cause fission that have not encountered an elastic collision with the surrounding hydrogen atoms in the shield. Although the cross-section for fast fission is low, this built in discrimination allows the detection of primary spontaneous-fission neutrons with high reliability. This in-cell counter has about a  $1 \times 10^{-3}$  percent overall efficiency for the detection of fast neutrons with a negligible background count rate. The useful range of the detector corresponds to 1/10 microgram  $\text{Cf}^{252}$  at a lower limit with the upper limit somewhat above 100 milligrams. The latter value is equivalent to about  $3 \times 10^{11}$  n/sec., the design limit for the neutron cell itself. No data has been obtained in the upper detection range as the largest  $\text{Cf}^{252}$  source available at the present time is somewhat less than 100 micrograms.

Ultramicro Remote Pipettor. In order to determine the concentrations of the highly active solutions, a servodrive pipettor has been installed in the enclosure. This unit is used with replaceable micropipetts and has been used successfully to measure one lambda volumes of solution (one-thousandth of a milliliter). This device has a reproducibility of a two percent on this size of sample; larger volumes have correspondingly increased accuracy. This unit is servodriven through a series of internal hydraulic pistons which allow an accurate scale reduction of driving fluid volume to pipetted volume. The final driving piston is a very accurately machined platinum shaft that displaces the same volume of hexane as the volume of the process sample being measured out. More recent versions of this unit have been constructed

by use of a differentially-threaded screw that is servo-motor driven in place of the more complex hydraulic linkage. Absolute read out has been included by use of an electrical data loop. As this unit is still in its last stages of development it will be reported on more thoroughly in future papers.

Special Purpose Equipment. One device that has been used successfully during recent operations is a light-sensitive photo-diode mounted below the ion-exchange columns. This device monitors the light given off by active solutions passing through the detector. The luminescence associated with highly active solutions varies the resistance of the photo-diode and allows a strip recorder to plot the shape of the elution curve as a function of time. The plots made in this manner closely represent those made by radioassay. The device is not sensitive to cell lighting, and can be used without turning off the normal cell illumination. It appears to be more sensitive to alpha active species than for beta emitters, and should prove to be a useful radiochemical tool. More work will be done on the development of this device in the near future.

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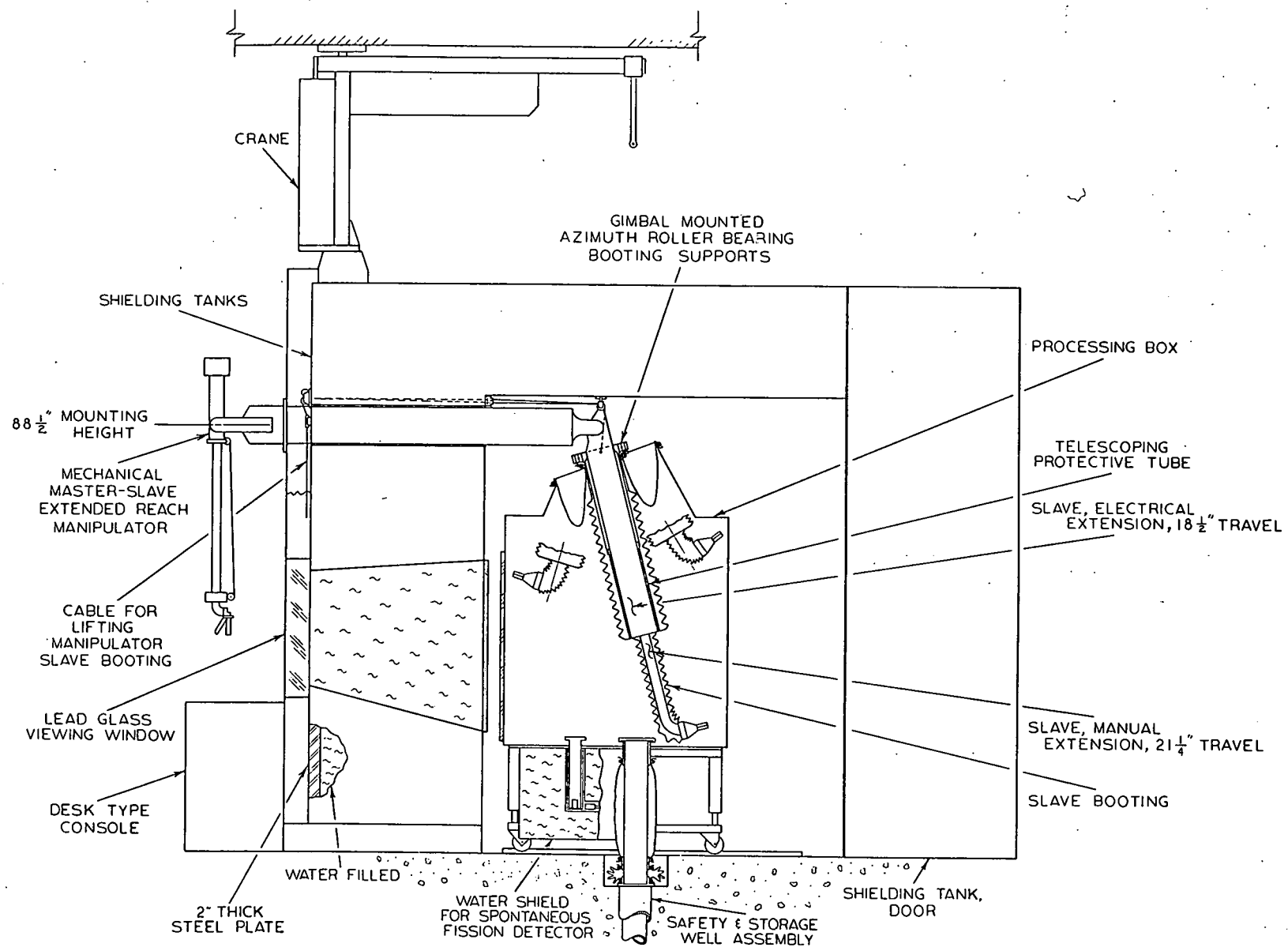
(1) M. S. Coops and C. L. Hanson, "Livermore Alpha-Gamma-Neutron Chemistry Cell," Proceedings of the Ninth Conference on Hot Laboratories and Equipment, November 7-9, 1961, pp. 12-21.

(2) M. S. Coops, C. L. Hanson, R. W. Hawk, and V. E. Scribner, "Remotely-Operated Trans-Plutonium Chemical Process Cells," UCRL-7845, In Press.

(3) T. C. Parsons, L. F. Deckard and P. W. Howe, "An Improved Socking Technique for Master-slaves," UCRL-9658, October 29, 1962, Unpublished.

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- Figure 1. Line diagram illustrating relation of containment box to cell and manipulators, including manipulator slave booting method.
- Figure 2. Diagram showing method of insertion (or removal) of manipulator into booting, indicating relative position of slave to box during insertion sequence.
- Figure 3. Photograph of remote portion of drop counter and fraction collector device.



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Figure 1

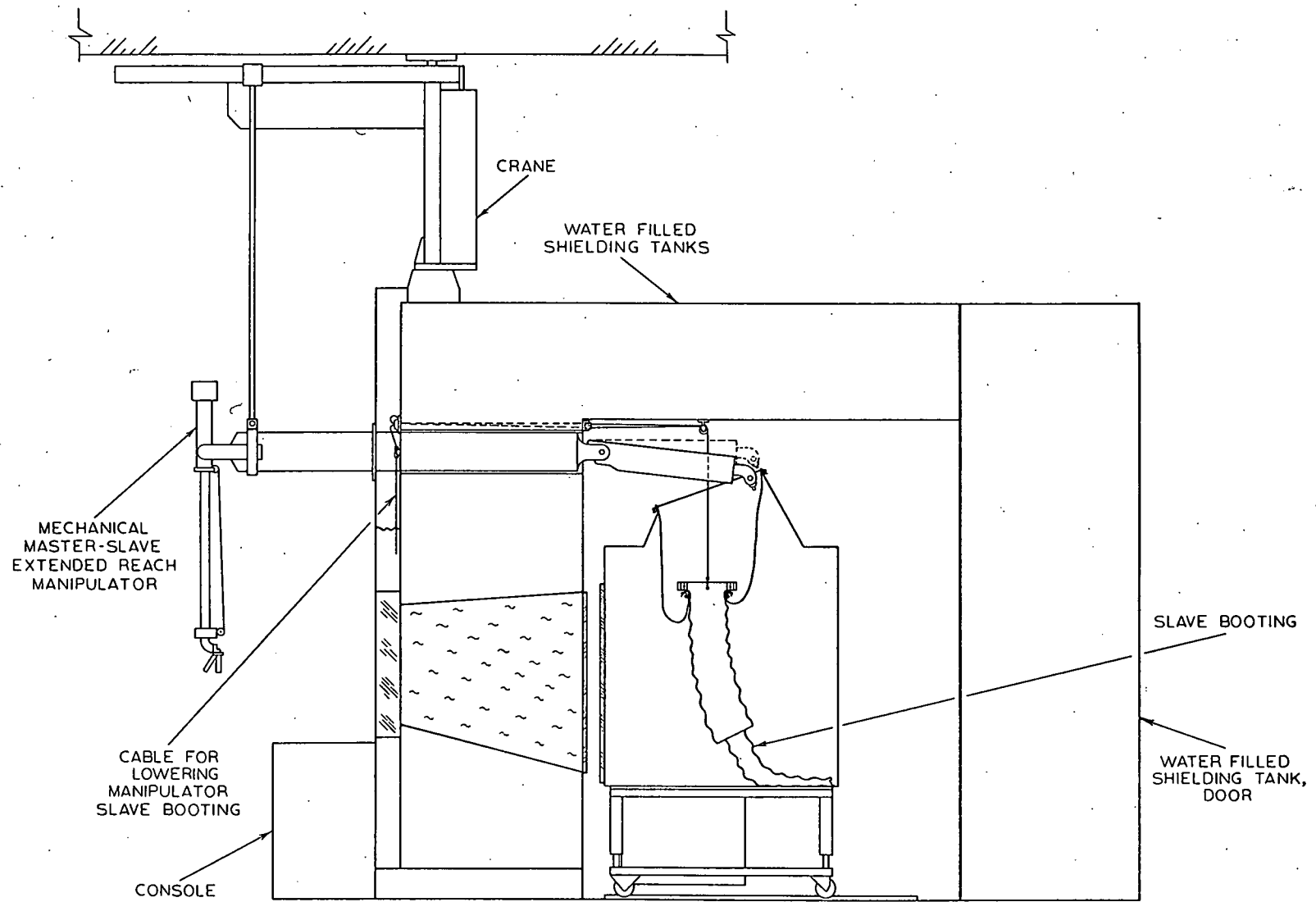


Figure 2



FIGURE 3