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W. B. McDonald
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## ABSTRACT

This report describes the design and use of a sampling system which permits the removal of operating-fluid samples from a forced-circulation corrosion-testing loop while the loop is in operation. The device is mounted over the one free surface of the fluid and utilizes a dip tube which can be inserted into the liquid and removed without contaminating the inert cover gas of the system.

## INTRODUCTION

A sampling device has been developed for use in corrosion-testing systems in which reactor construction materials are exposed to various molten-salt mixtures. The test system consists of a model LFB centrifugal pump, two resistance-heated pipe sections, a cooling coil, and a drain tank.[1] There is an inert gas cover over the liquid surface in the sump-type pump. These systems, of which there are 15, may operate for a year or longer at high temperatures (1100°F and above).

The sampling device described here was developed in order to obtain samples during operation. The molten-salt samples extracted are analyzed for corrosion products to determine, for example, the rate at which chromium is leached from the container walls.[2] Removal of samples is accomplished without contaminating the inert gas atmosphere in the pump tank.

## EQUIPMENT

The sampling device consists of two subassemblies, one permanently mounted on the loop and the other removable. A section view of the entire assembly mounted on the upper flange of a model LFB centrigual pump is shown in Fig. 1.

The permanently mounted subassembly, consisting of the ball-plug valve and adapters, is attached to the pump flange during fabrication of the loop and remains a part of the loop during the entire test period. The ball-plug valve is fabricated of stainless steel and contains Teflon seals throughout. The adapter between the pump flange and the valve is finned to keep the valve seals and gaskets relatively cool during operation. The pipe nipple above the valve is kept capped during normal operation of the loop to prevent entry of foreign matter into the upper compartment.

The removable head subassembly consists of a modified Wilson seal with Teflon gaskets and a sample dip tube. The Wilson seal maintains a tight seal around the outer surface while the dip tube is being inserted or withdrawn. The sample dip tube consists of a $\frac{1}{4}$-in.-OD copper tube with a short section of $\frac{3}{8}$-in.-OD copper tubing brazed on the lower end. A porous, sintered, copper disk is brazed on the lower end of the $\frac{3}{8}$-in.-OD section in order to retain, until it freezes, molten salt that is drawn up into the tube.

The auxiliary equipment required for the operation of the sampler includes a vacuum source, a regulated inert gas source, and the associated valves and gages. The sample dip tube is connected by flexible polyethylene tubing to a manifold incorporating means for either pulling a vacuum on the system or pressurizing it with inert gas. The sampling device is shown in Fig. 2 mounted on the pump flange with the dip tube in position for insertion. In the background may be seen the connecting tubing and the manifold. The mask is worn by the technician to prevent inhalation of toxic vapors of the beryllium-bearing salt contained in the system.

[1] J. L. Crowley, *MSR Quar. Prog. Rep. June 30, 1958*, ORNL-2551, p 33.

[2] J. H. DeVan and J. R. DiStefano, *MSR Quar. Prog. Rep. June 30, 1958*, ORNL-2551, p 58.

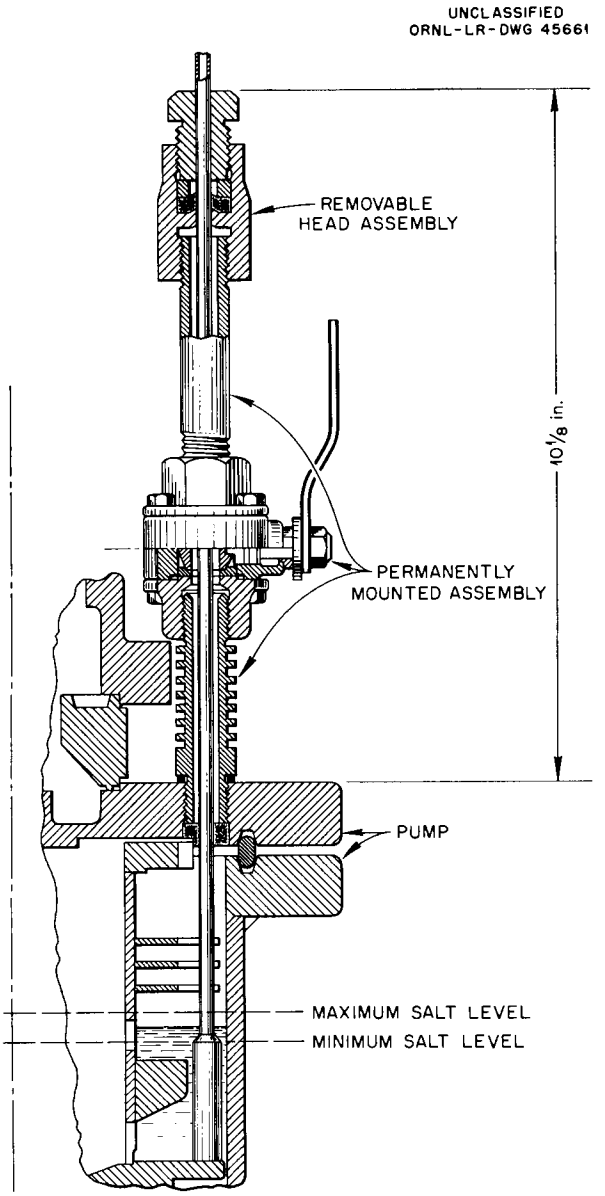


**Fig. 1. Sectional View of Molten-Salt Sampling Device Mounted on a Model LFB Centrifugal Pump.**

## OPERATION

The removal of samples from the molten-salt system requires some advance preparation, although the actual sampling may be accomplished in a matter of minutes. The sample dip tubes, made of copper, are hydrogen-fired on the internal surface to remove scale and surface contamination prior to insertion into the removable head subassembly containing the seal. The same number of subassemblies are prepared as samples are desired, and the sampling operation is ready to begin.

The following procedure is used to ensure proper control and to prevent the possibility of contamination of the system during removal of samples.

1. With the ball-plug valve closed, the protective cover is removed, and the dip tube and removable head subassembly is attached to the permanently mounted subassembly.

2. The necessary connections are made from the dip tube to the gas and vacuum supply manifold. The volume above the ball-plug valve is then evacuated through the dip tube and checked for leak tightness by shutting off the vacuum source and noting the pressure rise, if any, on the compound pressure gage.

3. As soon as a tight system above the ball-plug valve is evident, the space is purged by repetitive evacuation and pressurizing with inert gas from the manifold.

4. When the volume above the ball-plug valve has been sufficiently purged, inert gas is admitted at a pressure equal to that maintained in the pump. Thus when the ball-plug valve is opened, the system is not disturbed.

5. With the pressures above and below the ball-plug valve equalized, the valve is opened and the sample dip tube is inserted into the molten salt in the pump.

6. The sample dip tube is allowed to remain in the molten salt a short time to permit it to be heated to the operating temperature (1100°F or higher). The inert gas supply is then cut off at the manifold and a vacuum is applied to draw a molten-salt sample through the sintered copper filter into the dip tube.

7. The sample dip tube is then raised far enough to clear the ball-plug valve, which is then closed. The sintered copper filter retains the sample until it has solidified in the tube.

8. The removable head and dip tube subassembly is removed from the permanently mounted subassembly and replaced with another dip tube subassembly, if a second sample is desired, or by the protective cover.

9. The sample tube is then extracted from the removable head for chemical analysis and another sample tube is inserted for a future sample.

Drawing the hot molten salt up the sample tube in contact with the Teflon gaskets does not seem to adversely affect the quality of the seal obtained, and many samples may be taken before it is necessary to replace the gaskets.

The normal high-low liquid level tolerance in the model LFB centrifugal pump makes approximately

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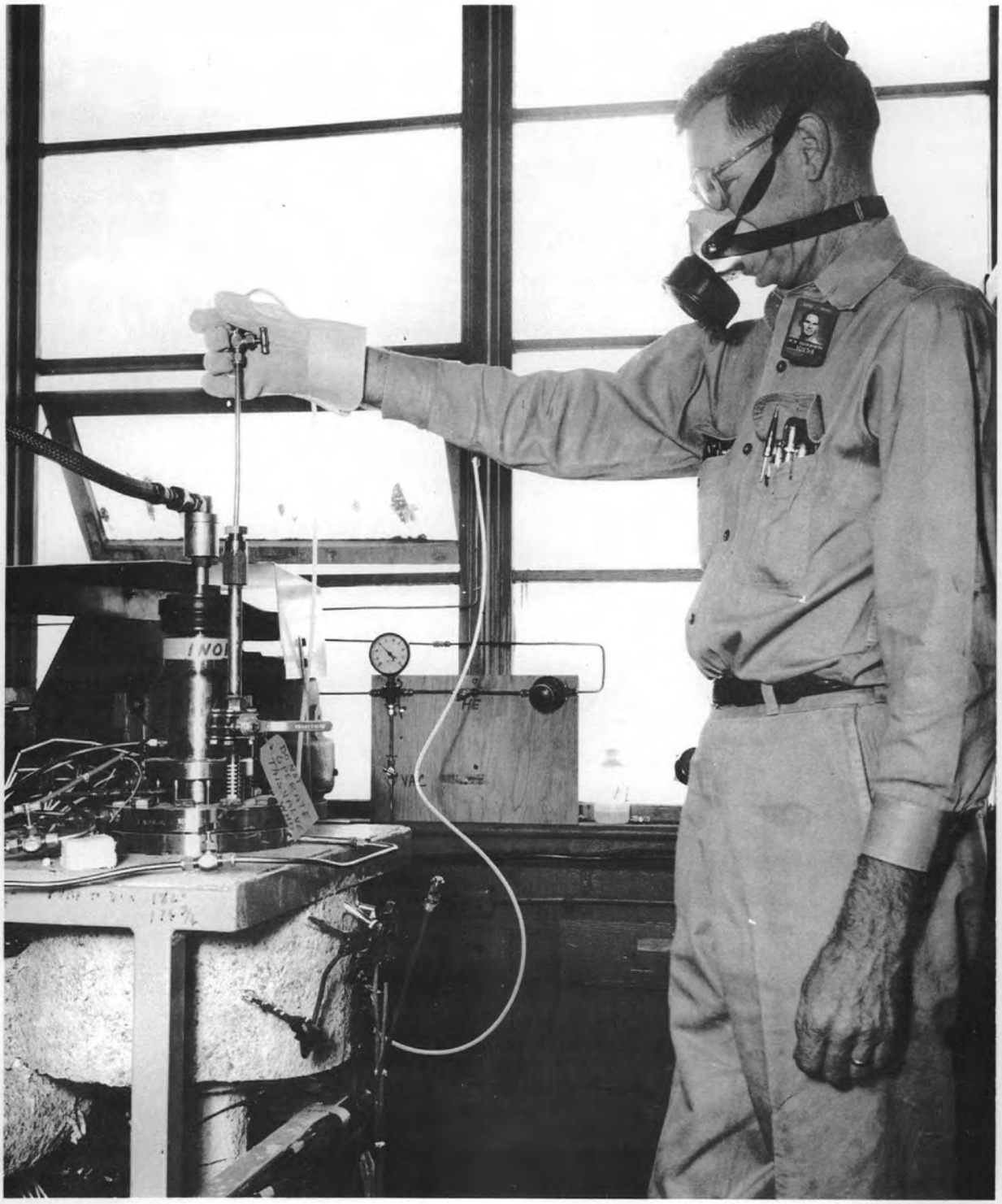

Fig. 2. Sample Tube Being Inserted into the Liquid of an Operating Pump in a Corrosion Test Loop.

9 in.$^3$ of molten salt available for sampling purposes. On the loops equipped with the sampling device, however, the normal tolerance is doubled so that approximately 18 in.$^3$ of molten salt is available for sampling. An average of 0.75 in.$^3$ of molten salt is removed from the system with each sample, and thus 24 samples are available from one filling.

## RESULTS AND DISCUSSION

At the time this report was prepared more than 130 samples had been successfully removed from six pumps equipped with the sampling device. The success of this type of sampling device in preventing contamination of the pump-tank atmosphere depends a great deal upon the proficiency of the operator in obtaining the proper seals and tight connections and in properly purging the volume above the ball-plug valve before the system is opened. For this reason only experienced technicians are allowed to operate the sampling device.

This type of device could possibly be used as a means for enriching the fuel of a molten-salt reactor. In this case the enriched fuel would be lowered into the reactor system and allowed to dissolve. Tests are being conducted to determine the feasibility of this operation.

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