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REMOVAL OF AN ACID FUME SYSTEM CONTAMINATED WITH PERCHLORATES LOCATED WITHIN A HOT CELL

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ABSTRACT

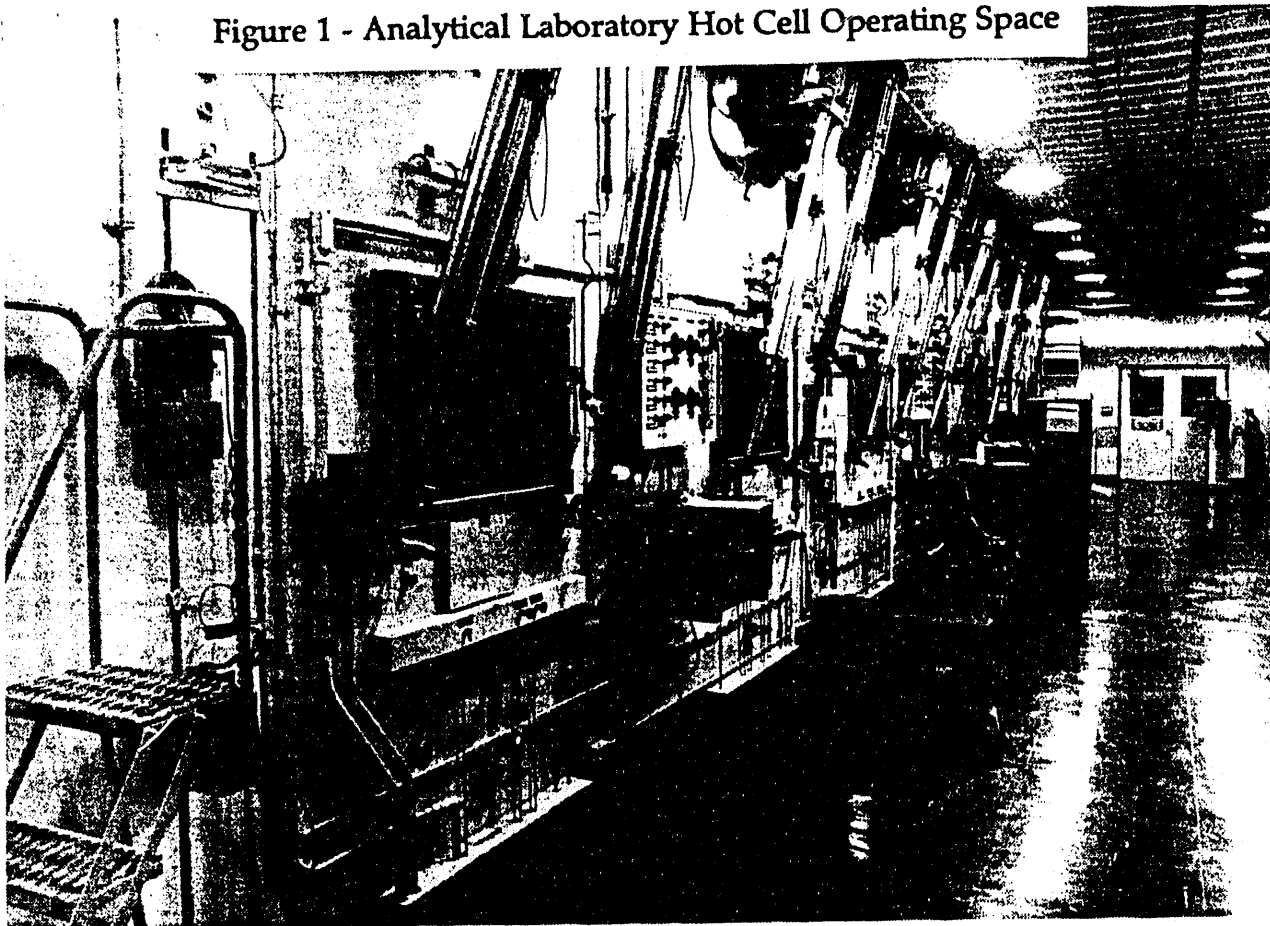
An acid scrubbing system located within the confines of a highly radioactive hot cell at Argonne National Laboratory-West (ANL-W) was remotely removed. The acid scrubbing system was routinely used for the dissolution of irradiated reactor fuel samples and structural materials. Perchloric acid was one of the acids used in the dissolution process and remained in the system with its inherent risks. Personnel could not enter the hot cell to perform the dismantling of the acid scrubbing system due to the high radiation field and the explosion potential associated with the perchlorates. A robot was designed and built at ANL-W and used to dismantle the system without the need for personnel entry into the hot cell. The robot was also used for size reduction of removed components and loading of the removed components into waste containers.

BACKGROUND

The Analytical Laboratory (AL) located at Argonne National Laboratory-West (ANL-W) contains six interconnected hot cells used for examining spent fuel as part of ongoing reactor research programs. Figure 1 shows the hot cell operating corridor.

Each hot cell is 183 cm (72 in) wide by 168 cm (66 in) deep by 376 cm high (148 in), with a working tray located 96 cm (38 in) above the floor. The hot cell walls are made from high density barite concrete and are 61 cm (24 in) thick. A shielding door, 91 cm (36 in) wide by 203 cm (80 in) high, located in the rear of the hot cell is the only access into the hot cell. As shown in Figure 1, each hot cell has a leaded glass window to view the internal area of the hot cell.

Figure 1 - Analytical Laboratory Hot Cell Operating Space



The cells had become highly contaminated over the years. An acid scrubbing system existed within the confines hot cell #2. The acid scrubbing system was routinely used for the dissolution of fuel samples and structural materials; a vital and necessary step in many of the analysis processes. The acid scrubbing system consisted of a hood, scrubber, demister, HEPA filter and a blower. With the exception of the hood, the entire system was located underneath the hot cell working tray. An isometric sketch of the acid scrubbing system is shown in Figure 2.

The acid scrubbing system was highly radioactive (16 R/hr beta/gamma at approximately 1 m (39 in) and 6 R/hr gamma at approximately 1 m (39 in) from the HEPA filter). The major radiation sources were the HEPA filter and the demister.

The hot cell #2 acid scrubbing system was used from the mid-1960's until 1988.

Consequently, the system was badly deteriorated and considered unreliable. Further, in the late 1960's and early 1970's the system was used to fume perchloric as well as other acids (HCL, HNO₃, HF, H₂SO₄, and H₃PO₄). The maximum amount of perchloric acid theoretically released into the system was calculated to be 1.8 to 2.3 kg (4 to 5 lb). Although no perchloric acid was used in the system since the early 1970's, perchlorate compounds remained with their inherent explosive risks.

The surface of the metal hood which channels the flow of acid fumes down to the acid scrubber below the #2 hot cell working tray was swiped with damp cloth smears in six locations for the analyses of perchlorates and radioactivity including plutonium. Figure 3 shows the location of smear locations.

Figure 2 - Isometric Sketch of the Acid Scrubbing System

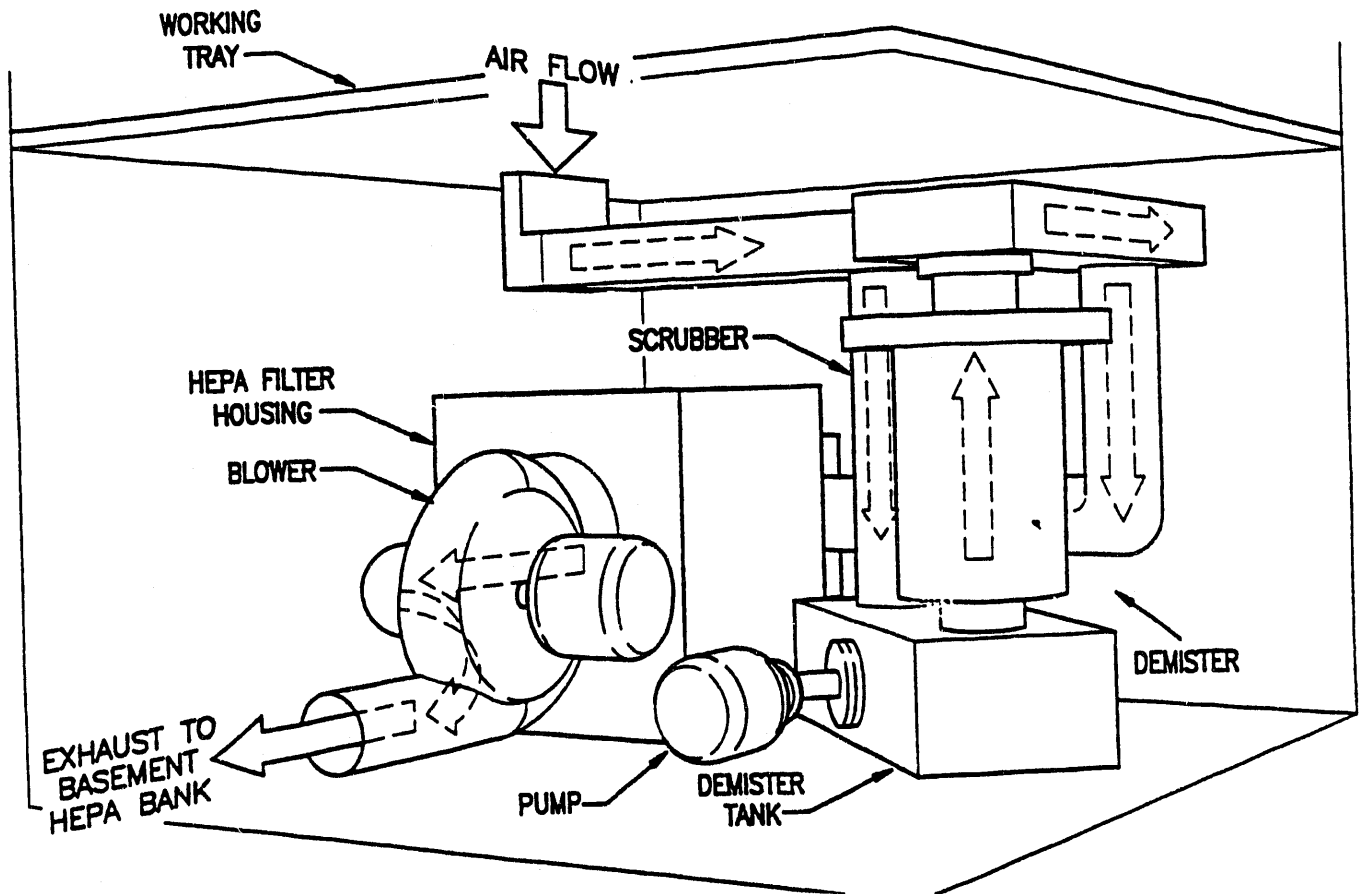
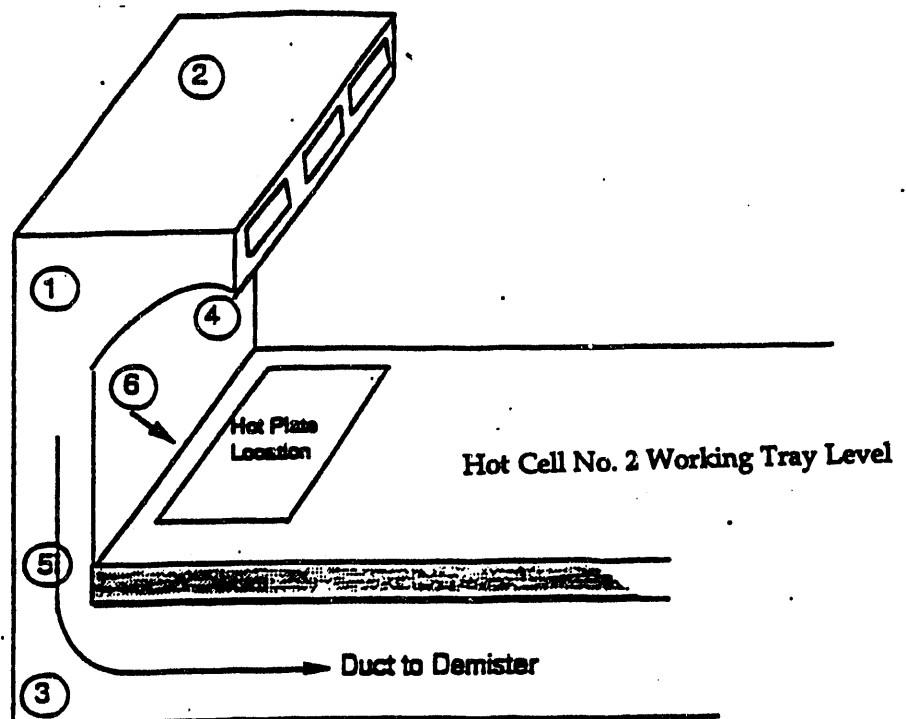


Figure 3 - Hood Smear Locations



Significant levels of perchlorates and radioactivity including plutonium were found. The results are listed in Table 1:

Table 1 - Analyses of Smears from Fume Hood

<u>Smear ID</u>	<u>Radiation Reading mr/hr @ 76mm (3")</u>		<u>Perchlorate Content</u>	
	<u>β,γ</u>	<u>γ</u>	<u>mg/smear</u>	<u>mg of Pu/smear</u>
1	2000	400	1.5	
2	12000	300	.62	
3	7000	1200	13.5	0.76
4	700	200	<.3	
5	7000	1400	9.8	0.76
6	3000	600	8.3	0.26

A solution of water was placed in the transition box just upstream of the HEPA filter. This solution along with smear #6 was analyzed for the presence of several ions. Results are presented in Table 2:

Table 2 - Ion Concentration

<u>Anion</u>	<u>Smear #6</u> <u>mg/smear</u>	<u>Solution from transition box*</u> <u>mg/ml</u>
ClO_4^-	8.3	8.36
Cl^-	2.8	.77
NO_3^-	8.3	4.42
$\text{SO}_4^{=}$	9.1	2.27
F^-	2.3	<0.05

*The pH of the solution was measured as approximately 4.0.

Smears were also taken from the working tray and significant levels of perchlorates were found.

The confirmed presence of perchlorates in the hood, the attached exhaust system, and external to the acid scrubbing system was sufficient evidence to recognize that a safety hazard did exist.

Two consultants with expertise involving perchlorates were hired to address how the system should be dismantled. Each consultant spent time at ANL-W to obtain an appreciation and understanding of this project.

The consultants addressed the topic of whether the perchlorate containing material present in the system would actually have the potential of being explosive. The consultants stated that the most explosive sensitive and dangerous group of perchlorates are organic perchlorates.

The probability of the presence of dangerously explosive organic perchlorates was determined to be very low. However, the presence of inorganic perchlorates mixed with organic materials and sensitive heavy metal perchlorates could not be determined. The sensitivity of inorganic perchlorates to explosion or ignition increases significantly if perchlorates are intimately mixed with organic or other oxidizable materials such as powdered metals.

Organic materials present in the acid scrubbing system included a neoprene gasket, a Teflon gasket in one tank and a small amount of epoxy resin used as a sealant in the HEPA filter. The neoprene gasket was visibly cracked due to the high radiation fields present in hot cell #2. It was assumed

that some perchlorates could penetrate into neoprene cracks. This would result in an increased sensitivity to ignition of the gasket material. However, the sensitivity of inorganic perchlorates - organic material mixtures is vastly reduced in a hydrated state. Even a small amount of water is sufficient to hydrate most of the perchlorates with a diminishing effect on explosive sensitivity and sensitivity to ignition.

The decision was made to remove the existing system and replace it with a state-of-the-art system as part of an upcoming hot cell refurbishment project. The combination of the explosion potential of the perchlorates and the high radiation/contamination of the hot cell made this task orders of magnitude more difficult from an environment, safety, and health standpoint. The perchlorates present in the acid scrubbing system were not suspected to be intimately mixed with oxidizable materials or organics but nevertheless a conservative approach to the dismantling effort was employed.

In addition to the consultants' recommendations, literature on perchloric acid systems was obtained and the guidance on dismantling perchlorate contaminated systems was considered during the preparation of the removal procedure.

CRITICALITY CONCERNS

Chemical analysis of the water added to the transition box, located directly upstream of the HEPA filter housing (See Figure 2), showed that the water contained 2 grams of uranium. The identification of uranium in the transition box confirmed that uranium did exist in the system. Due to the presence

of perchlorates in the system, the scrubber, demister and the demister tank were to be filled with water and let soak for a minimum of 24 hours prior to the initiation of the dismantling effort. The fact that uranium exists within the system combined with the use of water to flood the system components, required that the potential for criticality be addressed.

This was reviewed by ANL-W's Office of Nuclear Safety and it was concluded that the situation would not present a criticality concern. However, to be conservative, borated water was used in lieu of normal water in the soaking of the scrubber, demister, and demister tank. The solution was borated to a level of .12 weight percent boron. This solution allowed concentrations as high as 72 g/liter U-235, significantly higher than anything this system could encounter.

ACID SCRUBBING SYSTEM REMOVAL

The task at hand was to remove the acid scrubbing system, including the hood and working tray, from hot cell #2 without having personnel exposed to the high radiation field and the explosion potential associated with the perchlorates. An Exposure Limiting Robotic Apparatus (ELRA), designed and built at ANL-W, overcame these obstacles and was used to disassemble the system. Reference 1 gives details on the features associated with the ELRA. Figure 4 shows a photograph of the ELRA.

The dismantling effort was performed on weekends and on backshift when occupancy of the area was at a minimum. A removal procedure was prepared and approved by

upper level management prior to any dismantling activities beginning.

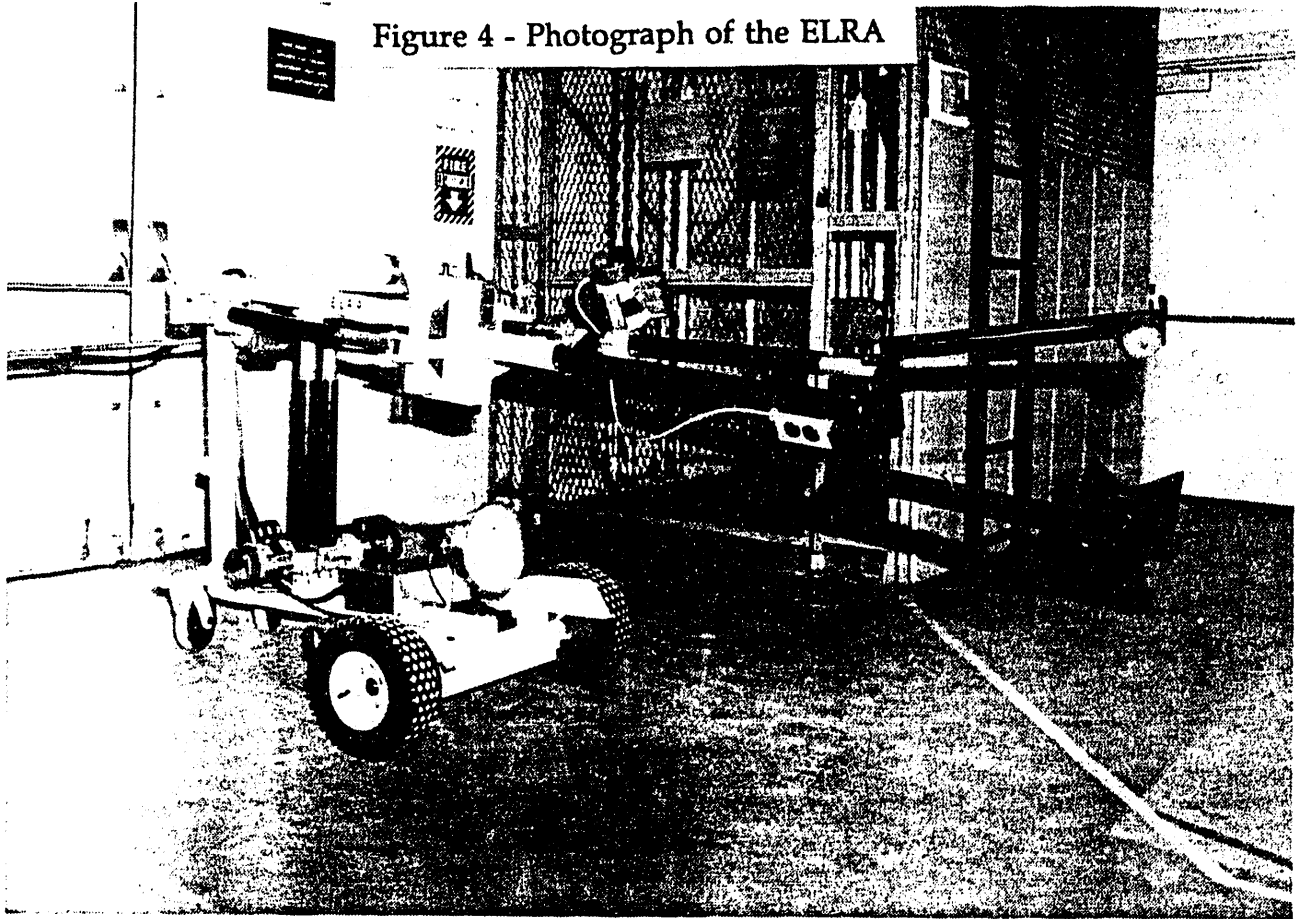
Initially, the scrubber, demister, and demister tank were filled with borated water. This U-shaped arrangement had a capacity of approximately 79.5 liters (21 gallons). These components were filled with water to place any perchlorates in solution and render them insensitive to explosion. The water was left to soak for 24 hours and was then removed with the aid of a peristaltic pump. The remainder of the system was not filled with water due to (1) holes within components, and (2) the relative height of the system with respect to the filter system located in the basement (see Figure 2).

The first components to be removed were the hood followed by the working tray. Once the working tray was removed, the operators of the master-slave manipulators and the ELRA would have a view of the acid scrubbing system. Analyses performed on smears taken from the hood internals and the underside of the working tray confirmed that perchlorates were present.

Spraying down the hood internals and the underside the tray with water could not guarantee that the surfaces would be thoroughly wetted. The alternative to spraying water was to use saturated steam. An electric steam generator delivering steam at approximately 103 kPa (15 psi) was employed. A 15.25 m (50 ft) steam hose carried the steam from the generator to the hot cells.

The ELRA's first job was to assist in the steaming of the hood and tray. The removal procedure mandated steaming each area for a minimum of 15 minutes. The ELRA entered the rear of hot cell #2 through a 1 m (40")

Figure 4 - Photograph of the ELRA



shielding door opening and with the aid of the master-slave manipulators, the nozzle of the steam hose was pointed towards the hood interior and the underside of the tray. The steaming of the hood internals and the underside of the tray was performed just prior to the cutting of that area. This philosophy, regarding the steaming of the components, was utilized for the removal of the remainder of the acid scrubbing system.

The hood was removed with the aid of a reciprocating saw. The ELRA held the saw during all cutting operations and water was directed onto the saw blade, during cutting operations, with the assistance of the master-slave manipulators. The water source came from within the hot cell. The saw blades had to be changed frequently and this operation was performed remotely with the master-slave manipulators.

The next task was removal of the working tray. The working tray was made from 5 mm (.19") stainless steel and supported by an angle iron frame. A hole saw was employed to cut approximately 127 mm (5") diameter holes in the working tray. A nibbler was then used to cut up the stainless steel sheet. The master-slave manipulators pushed and guided the nibbler through the stainless steel sheet while the nibbler was supported by the ELRA and a makeshift chainfall. The angle iron support structure was removed with the reciprocating saw. Water was always sprayed onto the saw blade, hole saw and nibbler punch during cutting operations. Removal of the working tray presented the operators of the ELRA and the master-slave manipulators with a view of the acid scrubbing system.

The approach taken in removing the remainder of the system was to first spray down the internals of the component to be removed with steam. Then with the ELRA holding the reciprocating saw and the master-slave manipulators spraying water on the area being cut, the components were physically cut and removed. The removal procedure mandated that all cuts into the system be made at no more than 610 mm (24") from an accessible opening. This ensured that the internals of the component being cut had been wetted down with steam.

Two video cameras were used in the disassembly effort. One camera was located on the back wall of the operating galley behind the hot cells. This camera was used mainly to view the position of the robot. The other camera was designed and built at ANL-W for insertion into a 76.2 mm (3") penetration located on the front of the hot cell wall. This camera penetrated the 610 mm (24") thick concrete wall and was able to

view the area below the working tray. Capabilities existed for indexing the camera lens through various angles to view areas under the working tray level. The camera was initially installed prior to beginning any dismantling activities so a removal procedure could be developed. These cameras proved to be instrumental in assisting the operators during the removal effort. Both monitors were located adjacent to the the leaded glass window so the operators of the ELRA and the master-slave manipulators could view them.

Visibility of the hot cell #2 working area was hindered due to (1) large amounts of stains and imperfections on the leaded glass window, and (2) low illumination of the working tray area due to the inefficient lighting system within the cell. Additional lights were brought into the cell and directed on the components that were being removed.

Bolts and other fasteners connecting the acid scrubbing system together were never loosened during the dismantling effort. Friction or shock during the loosening of a fastener could have generated a localized reaction between the inorganic perchlorates and organic material which could have resulted in an explosion or fire. Submerging the jointed parts in water and loosening the fasteners was considered, but this could not be attained due to the configuration of the acid scrubbing system.

The removed components were transferred into an adjacent cell with the aid of the ELRA or through the 610 mm (24 in) high by 305 mm (12 in) wide transfer port common between the cells. The cut up components were size reduced with the aid of the nibbler, reciprocating saw, master-slave manipulators

and the ELRA. The sized pieces were washed in a series of three tubs to ensure that all perchlorates were in solution. Samples of the wash water were taken after each 30 minute soak and a determination was made as to whether all of the perchlorates had been placed in solution. Also, the washing process served as a method for decontaminating the removed components. The pieces of metal were then disposed of. Reference 3 presents an overview on the removal of waste from the Analytical Laboratory Hot Cells.

The ELRA was used for other tasks such loading of the size reduced components into waste containers.

The ELRA became highly contaminated during the acid fuming system removal and was subsequently disposed of as radioactive waste.

AVOIDING HIGH RADIATION EXPOSURE

Radiation dosimetry was placed on the ELRA throughout the removal effort. The ELRA received 911 Rem non-penetrating radiation and 141 Rem penetrating radiation during the four week removal effort. ANL-W administrative control limits on penetrating dose radiation exposure to personnel over a four week period is .5 Rem.

CONCLUSIONS

An acid scrubbing system that was located within the confines of a small hot cell which was highly radioactive and contaminated with potentially explosive perchlorates was successfully removed remotely with the aid of an inexpensive robot and master-slave manipulators. The use of the ELRA eliminated personnel exposure to high radiation and the need for personnel to enter

the hot cell. The current DOE culture mandates that environmental, safety, and health (ES&H) concerns are given the utmost priority in accomplishing all work to provide for a safe, effective and efficient workplace. This has been successfully implemented through the application of the ELRA. This project also proved that a robotic device can be built for minimal costs and function very similar to expensive robotic equipment.

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