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Valve Studies: Hydrogen Fluoride Monitoring of UF₆ Cylinder Valves

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TABLE OF CONTENTS

LIST OF FIGURES	v
LIST OF TABLES	vii
ACRONYMS AND ABBREVIATIONS	ix
EXECUTIVE SUMMARY	xi
1. INTRODUCTION AND BACKGROUND	1
2. EQUIPMENT	5
3. TEST PROGRAM	7
3.1 BENCH-TOP TESTS OF PORTABLE TOXIC GAS DETECTORS	7
3.1.1 Response and Recovery Tests	7
3.1.2 Sensitivity Tests	8
3.2 ENVIRONMENTAL TESTING OF CHEMSTRIPS	8
3.3 FIELD TESTING OF CHEMSTRIPS	10
3.4 FIELD TESTING OF PORTABLE TOXIC GAS DETECTOR	12
4. TEST PROGRAM RESULTS	15
4.1 BENCH-TOP TESTS OF PORTABLE TOXIC GAS DETECTOR ..	15
4.1.1 Response and Recovery Tests	15
4.1.2 Sensitivity Tests	15
4.2 ENVIRONMENTAL TESTING OF CHEMSTRIPS	16
4.3 FIELD TESTING OF CHEMSTRIPS	17
4.4 FIELD TESTING OF PORTABLE TOXIC GAS DETECTOR	21
5. FIELD APPLICATION	23
5.1 STANDARD OPERATING PROCEDURE	23
5.1.1 Identification of Cylinder Valves with Evidence of Contamination	25
5.1.2 Photographing Cylinder Valves	26
5.1.3 Initial HP Survey	26
5.1.4 Preparation of HF Sensor Packets	27
5.1.5 Installation and Removal of HF Monitors	29
5.1.6 Inspection of HF Monitors	30
5.1.7 Decontamination Service	31
5.1.8 Second Health Physics Survey	32
5.1.9 Quantitative Measurement of HF Leak	32

5.1.10 End of Monitoring Process	33
5.2 IMPLEMENTATION OF THE HF MONITORING PROCEDURE	34
6. CONCLUSIONS AND RECOMMENDATIONS	35
6.1 EQUIPMENT	35
6.2 FIELD APPLICATION	35
6.3 RECOMMENDATIONS	37
7. REFERENCES	39

LIST OF FIGURES

1. Diagram of UF ₆ cylinder and location of cylinder valve	2
2. Cross-section of cylinder valve	3
3. Chemstrip packet with HF-exposed test strip used in environmental tests	9
4. Chemstrip card and "bagged" cylinder valve used in field tests	11
5. Cylinder valve with black bucket as used in field monitoring	11
6. Photograph of cylinder valve showing deposit indicative of active leak	13
7. TLD-1 portable toxic gas detector being field tested	13
8. Chemstrip packet after 4 weeks in sun	17
9. Purple test chemstrip shows positive exposure to HF	20
10. Flow chart indicating the action steps of the HF monitoring procedure for field application and data collection	24
11. Schematic of the TLD-1 toxic gas detector	28
12. Example of a complete HF sensor packet	29
13. Photograph of suspect cylinder valve that tested negative for leakage	36
14. Revised flow chart indicating action steps of the HF monitoring procedure as developed for monitoring multiple cylinder valves	38

LIST OF TABLES

1. Results of sensitivity testing of solvents and acids with HF	16
2. Results of field testing of Chemstrips	19
3. Results of Chemstrip lifetime laboratory testing	20

ACRONYMS AND ABBREVIATIONS

CRADA	Cooperative Research and Development Agreement
DOE	U.S. Department of Energy
HF	Hydrogen Fluoride
HP	Health Physics
NIOSH	National Institute for Occupational Safety and Health
NMCA	Nuclear Materials Control and Accountability
OSHA	Occupational Safety and Health Administration
ppm	parts per million
RCO	Radiological Control Organization
UCLIM	UF ₆ Cylinder Location, Inspection, and Measurement (Program)
UF ₆	uranium hexafluoride

EXECUTIVE SUMMARY

Portable toxic gas detectors, the MDA Scientific, Inc., TLD-1 and the Sensidyne Model SS2000, were tested as possible candidates for monitoring uranium hexafluoride (UF₆) cylinder valves for leaks. These monitors were tested for their ability to detect hydrogen fluoride (HF), which is produced by the reaction of moist air and (leaking) UF₆:

Hydrogen fluoride (HF) monitors and detectors can serve as an early warning system for valve leaks, limiting the loss and dispersion of cylinder contents as well as valve damage and the subsequent need for costly maintenance to effect valve replacements. The use of HF provides a four-to-one molar advantage over UF₆ or UO₂F₂ and is not compromised by the high radiological background or by conflicting visual inspection methods. The TLD-1 detector was tested in the field to determine any environmental conditions that may hinder accuracy of readings.

The possibility of using reactive strips from the Chemcassette Detector System ("Chemstrips" from the MDA Scientific, Inc., TLD-1 monitor) without the monitor was evaluated. Chemstrips can be used to qualitatively detect HF gas leakage if used under strict guidelines. The use of Chemstrips without the TLD-1 monitor allows long-term monitoring of multiple cylinders at a reduced cost since the TLD-1 monitor is not needed to make a qualitative determination.

The MDA Scientific, Inc., (TLD-1) toxic gas detector proved more useable in the desired field applications than the Sensidyne (SS2000) portable toxic gas monitor. The TLD-1 Chemcassette Detection System, used independently of the TLD-1, provides an economical means of long-term monitoring and allows detection of leaks with minimum interaction with the cylinder valves.

The UF₆ Cylinder Location, Inspection, and Measurement (UCLIM) Program visually identified 753 cylinders in E and K Yards at the K-25 Site (Oak Ridge, Tennessee) as having evidence of contamination on their valves. Of these, 437 were HF monitored with the Chemstrip packets and 102 were radiologically monitored. HF monitoring indicated that only 1.6% (7/437) of the valves were actively leaking. Radiological monitoring documented only 7.8% (8/102) of the valves with levels above the 1000 dpm/100 cm² limit, and

only one of these was an actively leaking valve. Hence, neither visual nor radiological inspection is a clear indication of a valve's current status.

1. INTRODUCTION AND BACKGROUND

Uranium hexafluoride (UF_6) cylinder valves have, like the cylinders, been in use and/or storage for periods ranging from 15 to 44 years.¹⁻⁵ Visual inspection of the cylinders has shown that the extent of corrosion and the overall cylinder condition varies widely throughout the storage yards. (A diagram of the type of cylinders studied in this report is shown in Figure 1.) One area of concern is the integrity of the cylinder valves. Visual inspection has found deposits which have been identified as radioactive material on or near the valves.^{1,5} These deposits suggest leakage of UF_6 and may indicate valve degradation; however, these deposits may simply be residual material from cylinder filling operations. See Figure 2 for a cross-section of the cylinder valve.

As UF_6 interacts with atmospheric moisture, hydrogen fluoride (HF) gas is produced; therefore, HF-sensitive monitors were chosen to identify and track leakage of UF_6 cylinder valves. Identification of leakage and characterization of its development and progress are important to storage management, since the objectives of storage management are to maintain containment integrity and to minimize or prevent the dispersion of uranium to the environment.

While valve leaks have been identified, it has not been established how or why they occur; whether they are self-stopping, cyclic, or continuing; or how effective corrective measures such as retorquing might be in stopping leaks. Some leaking valves show evidence of corrosive damage from HF attack on both aluminum-bronze and Monel components.

The use of radiological inspection methods to detect actively leaking valves is limited due to the background and interferant conditions. The use of meters for in situ radiological contamination measurement is inadequate because of the abnormally high background in the cylinder yards. Smear samples, taken by physically wiping an object to collect transferable surface contamination, are taken to a lab for measurement and can only determine if the deposit material is radiologically active. The HP methods have less accurate results for detecting leaks than HF monitoring.

The use of visual inspection methods does not necessarily find all valves that leak and can result in false positives as well as false negatives. Visual inspection is ultimately subjective and varies from one inspector to the next. A positive inspection could be the result of contamination or corrosion material found on an actively leaking valve, a valve that previously leaked but later sealed itself, or residual material from filling

operations. Conversely, material produced by a leak may be washed away by rain or wind erosion, and a negative inspection may result.

The presence of HF can be used to definitively identify an actively leaking valve, providing the basis for a system to detect leaking valves at the earliest possible moment.

This work consists of the evaluation of HF monitoring devices to indicate if and when suspect valves are leaking. The project includes testing and adapting HF detection devices to identify and preclude conditions that could produce false positive results and developing a procedure for monitoring and testing valves.

Figure 1. Diagram of UF₆ cylinder and location of cylinder valve.

Figure 2. Cross-section of cylinder valve.

2. EQUIPMENT

The equipment evaluated for monitoring HF leaks includes two portable toxic gas detectors and the indicator paper from one of these detectors.

The Sensidyne Model SS2000* portable toxic gas monitor (SS2000) is a complete, self-contained, single-channel monitoring system that includes the sensor, readout, alarm circuitry, and rechargeable battery. A quantity of air is manually pumped through a sensor fixture to a sensor electrode. An analog meter indicates the concentration of the toxic gas as seen by the sensor on a linear scale from 0 to 10 parts per million (ppm). The alarm produces both audible and visual signals and is factory set at 10% full scale; however, it is operator adjustable. This system was originally considered, but proved to be less functional than the other (MDA Scientific) portable HF monitor used.

The MDA Scientific, Inc., TLD-1** toxic gas detector (TLD-1) is a line- or battery-powered instrument. The rechargeable battery has an 8-hour lifetime. The TLD-1 uses the Chemcassette Detection System, which is a colorimetric-based sensor and uses a special substrate that has been impregnated with proprietary chemical formulations that produce a dry chemical reaction when exposed to a specific gas or family of gases. The manufacturer recommends that the Chemcassettes be stored in their protective packaging to prevent exposure to air or light, and that they be stored in a freezer to maintain optimum sensitivity. The manufacturer also suggests that the Chemcassettes be replaced 2 weeks after the package is opened, and they assign an expiration date which represents the date after which it should not be used. There is usually a 2.5-3.5-month lifetime upon receipt of a Chemcassette.

The TLD-1 system operates by pumping a known quantity of air through the Chemcassette tape. If HF is present, a stain proportional to the concentration of gas develops; the higher the concentration, the darker the stain. The density of the stain is measured by the monitor's photo-optical system, compared to a preprogrammed standard, and translated into a gas concentration value. This value is expressed in parts per million (ppm).

*Sensidyne, Inc., 16333 Bay Vista Drive, Clearwater, Florida 34620

**MDA Scientific, Inc., 405 Barclay Blvd., P.O. Box 1405, Lincolnshire, Illinois 60069

The TLD-1 gives results on the digital readout from 0.0 to 9+ ppm. An audible alarm sounds at 6.0 ppm. Exposure limits recommended by the National Institute for Occupational Safety and Health (NIOSH) and the Occupational Safety and Health Administration (OSHA) are time-weighted averages and have been set at 3 ppm. NIOSH has set a ceiling exposure limit at 6 ppm and OSHA has set a short-term exposure limit at 6 ppm. A detachable strip chart recorder uses pressure-sensitive paper as a permanent record. The adaptation of the Chemcassette Detector System permits long-time analysis, which is the focus of the present work.

3. TEST PROGRAM

Although commercial HF detectors are available, the concentration of HF present outside of the valve leak site at a single point in time may be below the detection limits of these detectors and the precise quantity of HF present at the valve is not critical. However, it is necessary to determine that there is HF present at the valve. Hence, sampling protocols had to be developed which are either more sensitive or allow sample collection over a longer period of time.

The TLD-1 toxic gas detector and the Chemcassette Detection System were tested for accuracy and alternative ways of detecting HF in the field by (1) conducting bench top tests to determine time of response/recovery and the sensitivity of the Chemcassette Detector System, (2) monitoring how environmental conditions may hinder accuracy of readings, (3) testing a long-term field monitoring system of the Chemcassette Detection System (Chemstrip), and (4) field testing the TLD-1 toxic gas detector.

3.1 BENCH-TOP TESTS OF PORTABLE TOXIC GAS DETECTORS

Response/recovery and sensitivity tests were performed by using a capped, 1-L Nalgene test bottle and securing the detector's intake tube through a hole drilled in the cap. A hole for ventilation was drilled near the bottom of the bottle, another hole was drilled into the cap for injection of the tested vapors.

Vapors were collected (5 cc) in a syringe from the headspace of the test liquid (at room temperature), taking care not to collect any liquid. The syringe was then inserted through the hole in the cap of the Nalgene bottle and the vapors were injected. The concentration of tested liquids or vapors was not determined.

HF vapors were collected as described above and injected into the test bottle. The approximate concentration of HF in the test bottle was 3 ppm.

3.1.1 Response and Recovery Tests

Tests to determine the response and recovery time of the HF monitors were conducted by collecting 5 cc of HF vapors and injecting them into the test bottle. The monitors were then alternately tested with HF vapors and room air by inserting the intake tube into the test bottle containing HF vapors

and timing the response or positive reaction, then removing the intake tube from the test bottle into room air and timing the recovery to a negative response. Results are given in Section 4.1.1.

3.1.2 Sensitivity Tests

Tests were conducted using the TLD-1 to determine if any common solvents or acids that might be used in decontaminating the valves could result in false positive readings. Included in the test were solvents (acetone, freon, isopropanol, and dichlorodifluoromethane) and acids (acetic, hydrochloric, nitric, and sulfuric).

First, 10 cc of the liquid substance to be tested was poured into the test bottle, providing a high concentration of vapors. Vapors present in the headspace were sampled to see if they could be falsely identified as HF vapors. If the liquid's vapors tested negative, 5 cc of HF vapors were injected into the bottle and the subsequent mixture tested. The testing of this mixture was used to ensure that the additional component did not interfere with the detection of HF.

If the liquid's vapors tested positive, a lower concentration of material was tested by collecting a vapor sample of 5 cc from the headspace of the test liquid and injecting it into another test bottle.

Dichlorodifluoromethane (a gas) was tested by placing the gas nozzle in the neck of the test bottle and spraying for approximately 2-3 seconds. The bottle was then capped and the low concentration vapor was tested. In addition, 5 cc of HF vapor was injected and the subsequent mixture was tested. Results are given in Section 4.1.2.

3.2 ENVIRONMENTAL TESTING OF CHEMSTRIPS

A study of the Chemstrips was performed to determine if environmental conditions would affect results. Indoor and outdoor test factors of varying degrees of light were examined. Strips were exposed from the period of October 6, 1992, through April 1, 1993.

The study utilized Chemstrips that were old (out-of-date), current (in date at beginning of study), and new (in date a few months later than the current). Plastic zipper bags containing a test Chemstrip and a separately-bagged control Chemstrip were assembled (Section 5.1.4). Four packets (one old, one new, and two current) were assembled for each environment tested.

The test Chemstrip in one of the current packets was exposed to HF to provide the chemical reaction of a positive test for HF and to allow observation of the environmental effects on this positive test (Figure 3). Thus, each packet contained three unexposed test strips and one exposed test strip (along with the sealed control strip).

These packets were gathered on a regular schedule for 5 months. A digital camera, a computer, and digital imaging software were used to collect and analyze color images of each packet.

Each digital image includes the test packet and reference color strips of red, yellow, blue, and green. The images, therefore, contain semiquantitative data that provides for computer analysis of the color shifts due to environmental differences and aging of the test packets relative to the control colors of the color strips.

Images were archived to create a visual record of the changes in Chemstrip appearance while aging under different environmental conditions. Results are given in Section 4.2.

Figure 3. Chemstrip packet with HF-exposed test strip used in environmental tests.

3.3 FIELD TESTING OF CHEMSTRIPS

A version of the packet that was used in the environmental testing was designed for use in field testing the Chemstrips for monitoring the cylinder valves. This packet includes a Teflon* bag which is gathered around the base of the cylinder valve to contain the monitoring card with two Chemstrips (Figure 4). Two opaque bags covered the valve, the entire packet, and the valve cover to protect the Chemstrips from exposure to sunlight. For the field tests, only the opaque bags had to be removed to visually inspect the Chemstrips.

As a result of field testing the Chemstrips and in consideration of mass monitoring of cylinder valves, the two opaque plastic bags that enclosed the entire assembly, including the valve covers, were replaced by opaque black plastic buckets (Figure 5). These buckets were modified by inserting three heavy duty magnets around the upper edge, enabling the technician to inspect each valve test by simply removing and replacing the bucket over the Teflon bag containing the valve and the Chemstrip test packet.

Six cylinder valves were monitored. Of the six valves chosen for monitoring, four cylinder valves (119406, 118326, 119101 and 119199) had deposits that were indicative of leaking (Figure 6). The last two cylinder valves (116774 and 115347) were not suspected of leaking.

This test began March 1, 1993, and continued through September 30, 1993. The Chemstrip packets were checked after 1 week. If the initial reaction was positive, the packet was replaced with a new test packet and monitored for a total of three test periods. Results are given in Section 4.3.

If the initial reaction was negative, the packets were checked every 2 weeks. If the Chemstrip packets continued to have a negative HF reaction for a period ranging from 7 to 21 weeks, the test packets were replaced with new Chemstrip packets. The negative Chemstrip packets removed from the cylinders were returned to the lab where the control and test Chemstrips were then tested by preparing a syringe of HF vapor (Section 3.1) and injecting the vapor directly onto the Chemstrips to see if they were still reactive to HF. These lab tests were performed to determine the field lifetime of the Chemstrips. See Section 4.3 for the results of this test.

*Teflon is a registered trademark of the E.I. DuPont deNemours Company.

Figure 4. Chemstrip card and "bagged" cylinder valve used in field tests.

Figure 5. Cylinder valve with black bucket as used in field monitoring.

3.4 FIELD TESTING OF PORTABLE TOXIC GAS DETECTOR

The TLD-1 was taken to the cylinder yard to test the same cylinder valves as those used in field testing of the Chemstrips, Section 3.3. For field testing, the black bags were removed from the valve and a small hole was cut in the Teflon bag to insert the monitor's intake tube (Figure 7). Each cylinder valve was monitored for several minutes to observe any fluctuation of measurement readings. Also, monitoring was done at different times of the day to observe differences caused by daily temperature fluctuations. Results are given in Section 4.4.

Figure 6. Photograph of cylinder valve showing deposit indicative of active leak.

Figure 7. TLCD-1 portable toxic gas detector being field tested.

4. TEST PROGRAM RESULTS

4.1 BENCH-TOP TESTS OF PORTABLE TOXIC GAS DETECTOR

4.1.1 Response and Recovery Tests

The response and recovery time of the SS2000 is operator-dependent because the pumping is performed manually. It could take several minutes for the portable toxic gas monitor to respond to or recover from exposure to HF vapor. Therefore, though it may be acceptable for detection of suspect spot leakage, it is not preferred for long-term, unattended detection.

Preliminary testing of the TLD-1 showed that the system's pump could not draw a vacuum. It is necessary, therefore, to assure that when using the TLD-1 in the field, there is sufficient ventilation around the valve for sample collection. Performance of the test described in Section 3.1.1 on the TLD-1 confirmed the response and recovery time of 30 seconds as stated in the MDA Scientific, Inc., *Guide to Operation*.

4.1.2 Sensitivity Tests

Results show that the TLD-1 is resistant to most laboratory solvents; however, acids other than HF may result in a false positive. Table 1 displays the results of the sensitivity testing of the TLD-1 along with the listing of those solvents or acids tested.

The solvents tested (acetone, freon, isopropanol, methanol and dichlorodifluoromethane) did not interfere with the detection of HF. Sulfuric acid did not interfere with the detection of HF. However acetic (high concentration only), hydrochloric, and nitric acids did result in a false positive reaction for the detection of HF.

These results suggest that solvent use does not interfere with HF detection, whereas other acids may interfere. Caution should be used when using other chemicals (during decontamination, instrument maintenance, etc.) around the valve when HF monitoring is expected to be performed within a short period of time (i.e., less than 10 minutes). Adequate vapor dispersion and complete material removal should be ensured to minimize false readings.

**Table 1. Results of sensitivity testing of solvents
and acids with HF**

Solvent/ Acid	High concentration pure vapor	Vapor + HF	Low concentration pure vapor
Acetone	*	+**	†
Isopropanol	-	+	
Methanol	-	+	
Freon	-	+	
Dichlorodifluoromethane	†	+	-
Acetic Acid	+	†	-
Hydrochloric Acid	+		+
Nitric Acid	+		+
Sulfuric Acid	-	+	

* Negative test results for detecting HF vapor.

** Positive test results for detecting HF vapor.

† Those solvents that reacted negatively at a high concentration were not tested at a low concentration. The acids that reacted positively at a high concentration were not tested for a reaction when mixed with HF acid vapors. Dichlorodifluoromethane is a gas and, therefore, was not tested as a liquid or at high concentration.

4.2 ENVIRONMENTAL TESTING OF CHEMSTRIPS

Environmental conditions were shown to greatly influence the usefulness of Chemstrips for detecting HF gas. Color measurements were made from the digitized images. (Unfortunately, this data was not useful in determining exposure amounts.) This data allowed a tracking of color shift over time. As the Chemstrips aged, they tended to become less saturated, the HF spots tended to fade, losing saturation also. Other effects were specific to certain exposure conditions and environments.

Chemstrip packets that were in total darkness throughout the test period were not greatly affected. These strips did change color slightly, fading to a less saturated yellowish color.

Chemstrip packets that were exposed to varying degrees of light had more deleterious results. The most pronounced effect occurred in the sunlight exposed strips. These strips developed dark spots and the original yellow color completely faded away (Figure 8). In these samples, the HF spot completely disappeared.

Analysis of the test results showed that Chemstrip material could be used after the manufacturer's specified dates as long as the strips were not exposed to sunlight (direct or reflected).

4.3 FIELD TESTING OF CHEMSTRIPS

The field monitoring system tested here will provide an economical means of accomplishing long-term valve monitoring as well as detecting suspect spot leakage. The system facilitates leak detection without removing the valve bag or relying on the presence of residual uranium compounds. This system is complementary to the uranium characterization study, which will assist in identifying technologies that can be used to identify the presence of uranium compounds.

Figure 8. Chemstrip packet after 4 weeks in sun.

The Chemstrip packets were attached to the cylinder valves and inspected for results during the first week of placement. If the packet was negative, it was inspected approximately every 2 weeks thereafter. (Occasionally, field monitoring activities were limited or delayed by weather and other yard activities.)

Six cylinder valves were monitored using the Chemstrip packets. Four cylinder valves (119406, 118326, 119101 and 119199) had a deposit that was indicative of leaking. Two cylinder valves (116774 and 115347) were not suspected of leaking.

Two of the cylinder valves (119406 and 118326) had a deposit that was indicative of leaking and were monitored three consecutive times, resulting in positive tests for exposure to HF. These results are consistent with those observed when field testing the portable toxic gas monitor (Section 4.4).

Two of the cylinder valves (119101 and 119199) had a deposit that was indicative of leaking and were monitored for two consecutive test periods (a total of 192 days); both were negative for exposure to HF.

The last two cylinder valves (116774 and 115347) were not suspected of leaking and also resulted in negative tests for HF exposure two consecutive times for periods ranging from 58 to 134 days. See Table 2.

An actual cylinder valve HF exposure reaction is illustrated by the image in Figure 9. The whole test strip reacts to turn from the original yellow color to purple. All field-tested Chemstrip packets were protected from exposure to sunlight by using an opaque covering.

The Chemstrip packets that had a negative reaction for exposure to HF through periods ranging from 7 to 21 weeks (50 to 142 days) were returned to the lab where the control and test Chemstrips were then tested to determine if they were still reactive to HF vapor. A reaction was positive if a stain appeared within 30 seconds of being exposed to HF vapor. Table 3 gives the preliminary results of this lab testing and shows that the test Chemstrip still responds reliably for approximately 2 months.

Although an actively leaking valve may be determined to be leaking during 1 month of monitoring, further testing should be conducted to determine whether a valve is leaking intermittently due to seasonal/daily temperature fluctuations, or if the leak could be self-sealing.

Table 2. Results of field testing of Chemstrips

Cylinder- Test	Day											
	2	5	9	15	50	58	77	87	105	115	134	142
119406-1			+									
119406-2	+											
119406-3		+										
118326-1	+											
118326-2		+										
118326-3				+								
119101-1								-				
119101-2									-			
119199-1												-
119199-2					-							
116774-1										-		
116774-2							-					
115347-1						-						
115347-2											-	

* Chemstrip packets were inspected within the first week of placement and approximately every 2 weeks thereafter. The result for testing the cylinder valves for leakage is given at the time of removal of the Chemstrip packet, and denotes the length of time monitored. At this point, the Chemstrip packet was removed from the cylinder valve and replaced with a new Chemstrip packet. After two consecutive tests, monitoring was discontinued. Replaced negative Chemstrip packets were returned to the laboratory and tested for sensitivity to HF vapor. This is part of the field lifetime test (Table 3).

Figure 9. Purple test chemstrip shows positive exposure to HF.

Table 3. Results of Chemstrip lifetime laboratory testing

Cylinder number- test number	Time in field (days)	Control Chemstrip reaction	Test Chemstrip reaction
119101-1	87	+	+
119101-2	105	-	-
119199-1	142	-	-
119199-2	50	+	+
116774-1	115	+	-
116774-2	77	+	-
115347-1	58	+	+
115347-2	134	-	-

4.4 FIELD TESTING OF PORTABLE TOXIC GAS DETECTOR

Cylinder valves 119406 and 118326, which had tested positive for the presence of HF when field testing the Chemstrip packets, and cylinder valves 119101, 119199, 116774, and 115347, which had tested negative (Section 4.3), were used to field test the TLD-1.

Cylinder valve 119406 was field tested three times in late March. The first two tests were performed midafternoon, and the positive test results ranged from a concentration of 1.3 to 2.1 ppm. The third portable toxic gas detector field test of valve 119406 was performed midmorning and ranged from 0.1 to 1.1 ppm.

The portable toxic gas detector was field tested on cylinder valve 118326 mid-morning on April 13. The intake tube was inserted through the hole in the Teflon bag and a concentration of 9+ ppm was detected. The Teflon bag was removed from the valve because some of the deposit had fallen off and collected in the old bag and could have caused some interference during the test. A new Teflon bag was placed over the valve and a small hole cut for insertion of the monitor's intake tube. HF was again detected at a concentration level of 9+ ppm.

The remaining four cylinder valves were each monitored twice and the results were negative. The results from portable toxic gas detector testing in the field is consistent with the findings of the Chemstrip packet field test (Section 4.3).

5. FIELD APPLICATION

Based on the results of the test program discussed in Sections 3 and 4, a standard operating procedure was developed for use in the field. Section 5.1 describes this procedure, and Section 5.2 describes the implementation of that procedure in Cylinder Yards K-1066-E and K-1066-K from June 1994 through September 1995.

This procedure was written for the preliminary purpose of collecting data to discover whether leaks are caused by valve degradation, residual material from cylinder filling operations, seasonal changes, or whether the leaks are continuous, cyclic, or self-stopping. Therefore, some steps may be above and beyond the need to discover whether a valve is leaking.

5.1 STANDARD OPERATING PROCEDURE

A procedure was drafted using the findings of the test program discussed in Sections 3 and 4 and is described in detail in Sections 5.1.1-10. This procedure was applied to HF monitoring of cylinder valves in K-1066-E (E Yard) and K-1066-K (K Yard) cylinder yards. A flow chart indicating the action steps of this procedure is shown in Figure 10.

Cylinder valves are inspected visually for evidence of contamination. This can be difficult since the extent and nature of the contamination or suspected contamination can vary greatly. Visual disparity has been found to range from large, multicolored, three-dimensional deposits to thin films of discoloration. No direct correlation between visual appearance and current leaking activity has been found. If there is no evidence of contamination, the cylinder is placed in the HF monitoring database for reinspection in 2 years.

If there is evidence of contamination, the valve is photographed and a Health Physics (HP) smear sample for transferable radioactive surface contamination is taken by the Radiological Control Organization (RCO). If the smear results meet the free release criteria, the cylinder valve is decontaminated, photographed, and reinspected in 4 weeks, at which point the process begins again.

Figure 10. Flow chart indicating the action steps of the HF monitoring procedure for field application and data collection.

If the results of the smear for transferable surface contamination are greater than the free release criteria, an HF sensor packet is attached and the HF monitoring process begins. The HF sensor packet is inspected every 2 weeks and, if negative, replaced every 6 weeks. If the monitoring results remain consistently negative for 1 year, the cylinder valve is decontaminated, photographed, and placed in the database for reinspection in 2 years, at which point the process begins again.

If the monitor reacts positively, the cylinder valve is decontaminated and smeared for transferable surface contamination until the results meet the free release criteria. The valve is then photographed and an HF sensor packet is attached and inspected after 2 weeks. If negative, the HF sensor packet is inspected every 2 weeks and, if negative, replaced every 6 weeks. If the monitoring results remain consistently negative for 1 year, the cylinder valve will be placed in the database for reinspection in 2 years, at which point the process begins again. (The initial positive result is interpreted as being due to preexisting surface contamination.)

If monitoring results are again positive, the HF sensor packet is replaced and the monitoring process is continued on a weekly basis. After three positive results, the TLD-1 portable toxic gas monitor is used to acquire quantitative results. This cylinder valve is then declared to be leaking and Facility Operations Division is notified. The cylinder is then placed on the list for remedial action.

This procedure was written for the preliminary purpose of collecting data to discover whether leaks are caused by valve degradation, residual material from cylinder filling operations, seasonal changes, and whether the leaks are continuous, cyclic, or self-stopping. Therefore, some steps may be above and beyond the need to discover whether a valve is leaking.

5.1.1 Identification of Cylinder Valves with Evidence of Contamination

A visible indication of potential valve leakage is green, white, or yellow deposits found on or near the valve. These deposits often appear on the valve stem end of the packing nut, the port cap, encircling the valve stem end, or encompassing the entire cylinder valve.

Candidate valves should be identified visually by looking for green, white, or yellow deposits. Visual disparity has been found to range from large, multicolored, three-dimensional deposits to thin films of discoloration. If no

discoloration or deposit is seen, the HF monitoring database should be updated with the cylinder valve inspection date and scheduled for reinspection in 2 years (Section 5.1.10).

If evidence of contamination is seen, the HF monitoring database should be updated and the cylinder valve photographed (Section 5.1.2).

5.1.2 Photographing Cylinder Valves

Cylinder valves should be photographed to document as clearly as possible the stains, discolorations, or deposits present on the valve. A marker board should be placed beneath the valve to identify the yard, cylinder, row, position number, and date of photograph.

Photography occurs after initial visual inspection determines that the valve shows evidence of contamination. The date of photography should be recorded in the HF monitoring database, and an HP smear should be requested for transferable surface contamination (Section 5.1.3).

After a valve for which the initial HP smear meets the free release criteria has been decontaminated, it is again photographed to record the cleaning, then scheduled for reinspection in 4 weeks. The date of photography should be recorded in the HF monitoring database.

5.1.3 Initial HP Survey

HP surveys using alpha or beta-gamma detectors do not provide useful results because of the high background levels near the UF₆ cylinders. However, testing for radiological surface contamination may provide some insight into past performance or operation and identify valves with a higher risk of future leaking than nonradiologically contaminated valves. HP surveys can be part of a risk ranking system to ensure that the manpower and resources are properly focused and utilized on valve management and replacement activities. Thus, HP surveys are an important part of the procedure.

Radiation Control personnel will survey requested valves for transferable surface contamination. Smears will be taken near the valve

containing the deposit and analyzed and recorded following Section 5.4 of HPD-OP-2.05, *Basic Contamination Survey and Documentation Techniques*.

The data reported on the Equipment-Radioactive Contamination Survey form (UCN-11635) will be used to determine which valves will be monitored for HF leakage. The values recorded for "Maximum Reading Alpha" and "Maximum Reading Beta-Gamma" will serve as the criteria to identify valves that have removable radioactive surface deposits.

Those valves for which the HP smears meet the free release criteria specified in U.S. Department of Energy (DOE) Order 5620 and Standard Practice Procedure (SPP) 804 for both alpha and beta-gamma (1000 dpm/100 cm²) will be considered "nonleaking." These valves will be decontaminated (Section 5.1.7), photographed (Section 5.1.2), and scheduled for reinspection in 4 weeks (in case decontamination opens a plugged leak). The HF monitoring database should be updated to record which valves have been analyzed for transferable radioactive surface contamination (predecontamination) and the values of the analyses.

Any valve that fails to meet the free release criteria specified in DOE Order 5620 and SPP 804 for alpha or beta-gamma will be considered "potentially leaking" and will enter the HF monitoring process. The HF monitoring database should be updated to record which valves have been analyzed for transferable radioactive surface contamination (predecontamination) and the values of the analyses.

5.1.4 Preparation of HF Sensor Packets

HF sensor instrumentation consists of (1) HF sensor packets using Chemstrips from the as-supplied Chemcassette Detection System and (2) the TLD-1 toxic gas detector for checking the strip functionality and for quantitative measurements.

The TLD-1 and the Chemcassette should be checked according to *TLD-1 Toxic Gas Detector Guide to Operation*. Operators should read this manual prior to use. A schematic of the TLD-1 is provided in Figure 11.

Before installation the Chemcassette should be checked for functionality and the package opening date written on the outside of the package. Unopened Chemcassettes that have been frozen should be discarded

2 months past their expiration date. Chemcassettes that have been opened and then stored in the freezer should be discarded 2 months after the opening date. This step of the process should be noted in the record logbook.

Each Chemcassette is approximately 60 feet long as supplied. The desired number of 2.5-inch Chemstrips should be cut, but the Chemstrips should not be handled with bare hands. Each valve to be monitored will need two Chemstrips—a test Chemstrip and a control Chemstrip. An example of a complete HF sensor packet is provided in Figure 12.

Figure 11. Schematic of the TLD-1 toxic gas detector. (Source: MDA Scientific, Inc., *TLD-1 Toxic Gas Detector Guide to Operation*, MDA Scientific, Inc., Lincolnshire, Illinois.)

Figure 12. Example of a complete HF sensor packet.

The HF sensor packet consists of card stock with a test and a control Chemstrip attached. Cut card stock into pieces of approximately 3.5 x 4.25 inches. The control Chemstrip should be sealed inside a small polyethylene bag (e.g. a 3 x 4-inch zipper bag) to prevent exposure to HF. Both the bagged control Chemstrip and the test Chemstrip should be taped to the card.

Information to be noted on each packet includes: yard, cylinder number, row number, position number, and date of instrumentation (the date the sensor packet was attached to the cylinder valve).

5.1.5 Installation and Removal of HF Monitors

HF monitoring hardware installed around each valve consists of the HF sensor packet (Section 5.1.4), a Teflon bag (chemically resistant to HF vapor) secured with a plastic tie-wrap, and an opaque plastic cover (a round 6-quart pail attached to the cylinder head with magnets is suitable).

The HF sensor packet should be placed inside the Teflon bag. The bag should be arranged over the cylinder valve with the sensor packet easily visible to the operator. The sensor packet should be facing the valve with about 0.5 inch distance between surfaces to allow air circulation and visual inspection. The Teflon bag should be secured around the base of the cylinder valve using a plastic tie-wrap.

The entire assembly needs to be covered with an opaque cover to protect the Chemstrips from exposure to sunlight, which deteriorates the sensors.

At the end of the inspection and monitoring period (Section 5.1.6), the opaque cover should be removed, the tie-wrap cut with a pair of snips, and the Teflon bag and sensor packet pulled off the cylinder valve, being careful not to dislodge any material from the cylinder or valve. The tie-wrap and sensor packet must be properly disposed of. The Teflon bag should be inspected for holes or tears and replaced if necessary. A new sensor packet is then inserted into the Teflon bag and reattached to the cylinder, after which the opaque cover is replaced. Repeat process every 6 weeks unless the monitor gives a positive response (Sections 5.1.6 and 5.1.9) or has been cumulatively, continuously negative for 1 year (Section 5.1.10).

5.1.6 Inspection of HF Monitors

After installation of the HF monitors, visual inspection of the HF sensor must be performed to identify leaking valves. A color change of the test Chemstrip from yellow to purple or brown, with no change in color to the control Chemstrip, is indicative of exposure to HF. A color change to either Chemstrip due to deterioration from exposure to sunlight ranges from very faded yellow to spotty beige. Figures 8 and 9 provide examples of both situations. Visual inspection of the control Chemstrips will be used to identify deterioration of the sensor strips. If the control Chemstrip has deteriorated, the HF sensor packet should be replaced immediately.

Visual inspection of the HF sensor packet should be performed every 2 weeks. If inspection results are negative, inspections should continue on a biweekly basis for 1 year or until a positive inspection is found, replacing the HF sensor packets every 6 weeks to ensure reliable results. The HF monitoring database should be updated to record the date, results, and replacement of inspected valve sensor packets.

If the inspection is positive for the first time, the valve should be decontaminated (Section 5.1.7), HP smeared (Section 5.1.8), and photographed (Section 5.1.2). The HF monitoring database should be updated to record the decontamination, HP smear, and photography dates and results.

Following the decontamination, smear, and photography, a new HF sensor packet will be attached and inspected after 1 week. If postdecontamination inspection results are negative, inspections should continue on a biweekly basis for 1 year or until a positive inspection occurs, replacing the HF sensor packets every 6 weeks to ensure reliable results. The HF monitoring database should be updated to record the date, results, and replacement of inspected valve sensor packets. Section 5.1.10 defines the action to be taken after 1 year of negative monitoring.

If the postdecontamination monitoring results are positive, refer to Sections 5.1.9 and 5.1.10 after a valve triggers a positive response in three HF packets. Although two positive HF monitoring tests indicate a high probability that a valve is actively leaking, a third test ensures that valve replacement operations are not done without justifiable cause. The variations in the HF response can also help provide some insight into the extent of leakage and what type of HF release (i.e., large or small) might be expected. The HF monitoring database should be updated to record the date, results, and replacement of inspected valve sensor packets.

5.1.7 Decontamination Service

Valves will be cleaned following Standard Operating Procedure SO 2801, *Radiological Field Decontamination Procedure* until all evidence of contamination has been removed.

As noted in Section 5.1.3, those valves that meet the free release criteria specified in DOE Order 5620 and SPP 804 in both "Maximum Reading Alpha" and "Maximum Reading Beta-Gamma" categories during the initial Radiation Control survey will be considered "nonleaking." These valves will be cleaned and reinspected after 4 weeks for visual evidence of redevelopment of contamination. If visual indicators stay negative, the valve will go into the long-term monitoring schedule (Section 5.1.10). These valves will not be monitored for HF leakage but will be scheduled for visual inspection in 4 years in accordance with UCLIM-scheduled inspections. The HF monitoring database should be updated to record which valves have been cleaned.

If visual indications are positive after the 4-week period, the valve will be reintroduced to the start of the inspection and monitoring cycle.

As noted in Section 5.1.6, if a valve reacts positively to HF for the first time in the HF monitoring process, it will be cleaned by decontamination operators following Standard Operating Procedure SO 2801, *Radiological Field Decontamination Procedure* until all evidence of contamination has been removed and the valve meets the free release criteria specified in DOE Order 5620 and SPP 804 in both "Maximum Reading Alpha" and "Maximum Reading Beta-Gamma" categories during a second Radiation Control survey. The valve is then photographed and the HF monitoring process continued.

The decontamination, HP smear, and photography should all be performed and documented within a 24-hour period, the HF monitoring database should be updated appropriately.

5.1.8 Second Health Physics Survey

Cylinder valves that have undergone the first HP survey, started the HF monitoring process, and resulted in a positive response for HF, require simultaneous decontamination and a second HP survey to confirm that transferable radioactive contamination levels are within the "clean area" limits specified in SPP 804. The HF monitoring database will be updated to record which valves have been analyzed for transferable radiocontaminants (postdecontamination).

It is important that the second HP survey closely follow the decontamination procedure so that any ensuing HF monitoring is not interpreted as resulting from preexisting surface contamination. It is recommended that both procedures be performed on the same day.

5.1.9 Quantitative Measurement of HF Leak

If it appears that a valve is leaking continuously, based on three positive readings, the TLD-1 detector will be used to quantify the HF. Quantitative measurements should be taken at various times of the day.

The TLD-1 should be checked according to *TLD-1 Toxic Gas Detector Guide to Operation*. Operators should read this manual prior to use. A schematic of the TLD-1 is provided in Figure 11.

A 6-inch length of Teflon tubing should be attached to the TLD-1 intake. The Teflon bag and sensor packet should be removed carefully as described in Section 5.1.4. The TLD-1 should be operated using a rechargeable battery.

For operator safety, the operator should use the detector to monitor the air at a distance of about 2 feet from the valve for 30 seconds. Next, the air should be monitored at progressively shorter distances (1 foot, 6 inches, etc.) from the valve for 30-second time periods. Quantitative measurements should be taken while monitoring the surface of the valve at a distance from the valve surface to the intake tube of approximately 0.25 inches. This will determine whether there is an active leak of HF.

The TLD-1 responds with a digital readout on the monitor and graphically if the printer is attached. The HF monitoring database should be updated by recording the results from the TLD-1.

The Teflon bag should be reattached and vented by cutting off one corner (preferably bottom oriented).

Custody of the leaking valve must be relinquished to UF₆ Cylinder Operations for remedial action.

5.1.10 End of Monitoring Process

Valves that have been recorded in the HF monitoring database as not leaking will be scheduled for reinspection again in 2 years.

UF₆ Cylinder Operations will be notified for remedial action of cylinder valves that have been recorded in the HF database as leaking, based on the criteria outlined above (Sections 5.1.6 and 5.1.9).

5.2 IMPLEMENTATION OF THE HF MONITORING PROCEDURE

UF₆ Cylinder Operations inspect annually all cylinders that have been previously determined to be in poor condition. All cylinders that are in good condition and are stored under good circumstances are inspected quadrennially. The defect categories of interest to the HF Monitoring Program are

- evidence of contamination on valve,
- missing or cracked packing nut, and
- missing or cracked port cap.

E Yard contains 154 cylinders identified as having contamination on valves, 35 valves with the packing nut missing or cracked, and 7 valves with the port cap missing or cracked. During the course of the project, an additional 10 cylinder valves were identified by project personnel as showing evidence of contamination. All of the valves listed above (206 total) were included in the HF monitoring program.

K Yard contains 516 cylinders identified as having contamination on valves, 6 valves with the packing nut missing or cracked, and 25 valves with the port cap missing or cracked. All of the valves with cracked or missing packing nuts or port caps plus 200 of the 516 valves with contamination (a total of 231 cylinder valves) were included in the HF monitoring program.

The implementation of the HF monitoring project began in E Yard in March 1993 with the six valves that were part of the test program described in Sections 3 and 4. By February 1995, 206 cylinder valves in E Yard were instrumented and were being regularly checked and changed. The E Yard HF monitoring process continued through October 1995. The length of time a valve in E Yard was monitored ranges from 8 to 26 months.

HF monitoring in K Yard began in March 1995 with the instrumentation of the 31 cylinder valves with cracked or missing port caps or packing nuts. Fifty additional cylinder valves showing evidence of contamination were instrumented each month through July 1995 for a total of 231 cylinder valves being monitored. The K Yard HF monitoring process continued through September 1995. The length of time a valve in K Yard was monitored ranges from 2 to 6 months.

6. CONCLUSIONS AND RECOMMENDATIONS

6.1 EQUIPMENT

For the purposes of this project, the MDA Scientific, Inc., TLD-1 toxic gas detector proved most useable in the field as opposed to the Sensidyne SS2000, which requires manual pumping.

Chemstrips were proven effective qualitative indicators of HF leakage but require proper maintenance and handling to ensure reliable results. It is possible to economically monitor or screen multiple cylinders with the mounted Chemstrip technique.

6.2 FIELD APPLICATION

Valves were visually, radiologically, and HF monitored in E and K Yards at the Oak Ridge K-25 Site.

A total of 437 valves (of the total of 753 identified as possible leakers in E and K Yards) were monitored with the HF chemstrip packets. Zero valves in K Yard and seven valves in E Yard were found to be leaking. All of these valves had visual signs of leakage, but only 1.6% (7/437) of the valves were found to be actively leaking HF. Hence, visual inspection is not a clear indication of current leaking status. See Figure 13 for a photograph of a cylinder valve (Cylinder #117531) that shows evidence of contamination but for which HF monitoring did not confirm a leak.

A total of 102 valves were HP analyzed for removable contamination prior to decontamination. Only eight (7.8%) valves of the 102 were found to be above the 1000 dpm/100 cm² limit. The measurements ranged from 0dpm/100 cm² to 3046.5 dpm/100 cm².

Of the seven actively leaking valves, only one had radiological limits over the free release criteria (1000 dpm/100 cm²). The measurements ranged from 0 dpm/100 cm² to 1831 dpm/100 cm². Hence, HP results do not provide a reliable basis for determining the current leaking status of the valve.

Figure 13. Photograph of suspect cylinder valve that tested negative for leakage. (Photo shows deposit indicative of possible leak.)

PAI Corporation of Oak Ridge, Tennessee, created a PC-based relational database to manage all of the valve data. This relational database currently has entries for the 437 valves that have been HF monitored. An additional 316 valves that have been visually identified as potential leakers by UCLIM have also been entered in the HF database. It is planned that this database will be able to interface directly with the other UF_6 cylinder databases [UCLIM and Nuclear Materials Control and Accountability (NMCA)] to ensure that the optimum use of the data can be obtained.

Based on the results of the bench-top testing of the portable toxic gas monitors, the environmental and field testing of the Chemstrips, and the number of definitely leaking valves discovered via visual, radiological, and HF monitoring, neither visual nor radiological testing are reliable guages of the current leaking status of the valve. The mounted Chemstrip technique is an effective qualitative indicator of HF leakage and makes it possible to economically monitor and screen multiple cylinders.

The flow chart indicating the action steps of the HF monitoring procedure as proposed for monitoring multiple cylinder valves has been revised and is shown in Figure 14. For the purpose of monitoring multiple cylinders all photography will be omitted, monitoring will discontinue if negative after 6 weeks, and a cylinder valve will be turned over the Facility Operations for remedial action after only two positive reactions. The procedure will require, however, that all cylinder valves be HF monitored once even when there is no evidence of contamination observed on the valve.

Based on the results of this project, this technology is applicable and will be revised as needed for use in the detection of leaking cylinder plugs and possibly the detection of leaking patched, breached cylinders.

6.3 RECOMMENDATIONS

Recommended follow-up actions include

- (1) final approval of standard operating procedure SMTLS-OP-205, *HF Monitoring of UF₆ Cylinder Valves*;
- (2) mass screening of cylinder valves for HF leakage by the mounted Chemstrip method;
- (3) follow-up quantification of leaking cylinder valves with the TLD-1;
- (4) explorations of the possibility of establishing a Cooperative Research and Development Agreement (CRADA) with MDA Scientific to develop a more rugged test strip;
- (5) examination and characterization of valves that have been confirmed as leaking, old valves that have been in use for several years and are not leaking, or valves that look like they should be leaking but are not;
- (6) revision and approval of a standard operating procedure, *HF Monitoring of UF₆ Cylinder Plugs*;
- (7) cylinder valve inspection on a 2-year basis.

This information could serve to identify faulty valves on a case-by-case basis before a breach and environmental insult occurs.

Figure 14. Revised flow chart indicating action steps of the HF monitoring procedure as developed for monitoring multiple cylinder valves.

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