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Case Studies

A Mysterious Yellow-White Paste: Evaluation of an Unanticipated Contaminant

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Introduction

Industrial hygiene is defined as the "science and art devoted to the anticipation, recognition, evaluation, and control of those environmental factors or stresses arising in or from the workplace that may cause sickness, impaired health and well-being, or significant discomfort and inefficiency among workers or citizens of the community."(1) Categories of environmental agents that may be present in chemical processing operations include raw materials, catalysts, auxiliary materials, products, by-products, additives, lab chemicals, biological agents, and in some situations construction materials.(2) This evaluation involves an adulterant or unanticipated contaminant that appeared in a stable, long-term, continuous process operating under controlled conditions. The purpose of this study was to evaluate potential exposures to arsenic contaminants that had inadvertently been introduced into the process. This evaluation reaffirms the importance of the anticipation and recognition aspects of industrial hygiene and provides a contemporary example. The importance of periodically assessing the appropriateness of assumptions regarding a process that has not changed is identified. The outcome of such an evaluation may provide new insight regarding potential occupational health hazards, requires reevaluation of the suitability of control measures currently in place, and may introduce some additional considerations or concerns that need to be addressed due to the acquisition of new hazard information.

Background

The process evaluated in this study involves the enrichment of uranium for use in commercial and military nuclear reactor fuel rods.(3) Uranium enrichment by gaseous diffusion is an iterative physical process which depends upon the separation effect arising from the phenomenon of molecular effusion to separate the uranium isotope ^{235}U from the more abundant ^{238}U isotope.(4) The diffusion product contains ^{235}U at a level of 2 to 5 percent, above the naturally occurring level of 0.7 percent ^{235}U. A gaseous diffusion plant may have thousands of separative stages through which uranium hexafluoride (or process gas) passes to achieve the desired level of uranium enrichment. Enriched uranium (or product) is withdrawn from the top end of the process, where the material has completed passage through numerous separative stages. Depleted material (or tails) may be withdrawn at various locations from the separative stages.(5) This is a dedicated process, and narrowly defined operating conditions are continuously maintained. The facility had been in operation for about 40 years. The raw (or feed) material used was uranium hexafluoride (UF_6).

Contaminants associated with the process include fluorine, chlorine trifluoride, chlorofluorocarbons, and uranium radiological decay products. Fluoride-containing compounds are formed if process gas combines with atmospheric moisture. Chlorofluorocarbons infiltrate into the process from heat exchange equipment. Contaminants present in the feed material may also enter the process. The process gas is incompatible with organic materials.

Contaminant Discovery

Process maintenance workers removing blocked instrument lines from process equipment observed deposits dissimilar in appearance to those normally encountered. Obstruction of instrument lines generally occurred from the influx of moist air and its reaction with UF_6, resulting in the formation of a solid, yellowish deposit of uranyl fluoride (UO_2F_2).(6,7) These obstructions in process equipment lines are removed by treating the equipment with a mixture of chlorine trifluoride and fluorine (ClF_3-F_2) to remove all traces of moisture, organic material, or UO_XF_Y. The UO_XF_Y plug is converted to UF_6. The equipment is off stream (blocked off from the process and purged of process gas) when these treatments are performed.(8)

The various contaminants present in the process tend to concentrate at different locations within the process equipment. This facility consists of several thousand interconnected separative stages (referred to as a cascade). The light gases that have entered the process through time (e.g., chlorofluorocarbons, acidic gases such as fluorine and chlorine trifluoride, etc.) concentrate at locations near the top end of the process equipment. In October 1993, workers removing blocked instrument lines from off-line equipment near the top end of the process (where lighter contaminants were generally found to collect) noticed a difference in the appearance of the deposits obstructing the line compared to the UO_2F_2 deposits normally observed. A viscous, pale yellow-white material with the appearance of butter or taffy was blocking the line. A second deposit was reported to have a light green color. Both deposits released a smoke that varied in color from green to white, depending upon the deposit exposed to atmospheric moisture. The hygroscopic reaction product was reported to be an arsenic oxide (e.g., arsenic pentoxide or As_2O_5).(3)

The site laboratory analyses indicated that the original deposits may have been chlorylarsenic hexafluoride ($ClO_2 \cdot AsF_6$) or arsenic tetrachloride–arsenic hexafluoride ($[AsCl_4^+] \cdot [AsF_6^-]$). X-ray diffraction analyses of residual solids following hydrolysis of the deposit samples taken from the instrument lines reportedly produced similar diffraction patterns. Crystalline material was identified as hydrogen arsenate ($H_5As_3O_{10}$) and copper arsenate ($Cu_2As_2O_7$).(3) Accumulation of arsenic deposits in the copper instrument lines was attributed to the copper acting as a getter for arsenic compounds. Contact gettering occurs when residual gas is absorbed by a metallic deposit. Traditionally this process has been used commercially in evacuated systems such as electron tubes.(9)

The plant process engineers determined that the supplier of feed UF_6 shipped material containing significant amounts of arsenic as an impurity to the site during the 1980s. The mechanism by which arsenic was introduced into the feed material was through the use of arsenic-contaminated HF and F_2 used in UF_6 production from uranium octoxide (U_3O_8). This contaminant came from fluoride-bearing ore which had not had the arsenic completely removed before generating the HF and F_2 used in the uranium fluorination process. The supplier had reportedly improved operations to effectively reduce the arsenic contamination of feed UF_6 by 1990.(10)

Methods

The principal concern arising from the discovery of a previously unrecognized and unanticipated contaminant in the process equipment was whether this represented an additional workplace health hazard, and if it was adequately controlled by existing exposure controls. The National Institute for Occupational Safety and Health (NIOSH) received a confidential request through the diffusion plant unions to conduct a health hazard evaluation. The request focused upon tasks and locations where work on process equipment was performed. The workers considered at greatest potential risk of exposure were operations and maintenance workers opening system equipment.

The NIOSH industrial hygiene survey evaluated potential airborne concentrations of arsenic-containing compounds encountered by workers performing maintenance on process equipment. Previous industrial hygiene sampling conducted at the facility for arsenic found predominantly nondetectable concentrations with an occasional airborne arsenic concentration up to 6 $\mu g/m^3$ of air. Arsenic pentafluoride (AsF_5) had been identified in the cascade near the top end of the process, in the segment of process equipment where the light gases accumulate. The possibilities of multiple arsenic-containing compounds, potentially low airborne concentrations, numerous sampling limitations, and an interest in total arsenic concentrations were considered in developing the survey protocol.

NIOSH Method 7901 was used to obtain personal and area air samples for total arsenic. The method utilizes a sodium carbonate (Na_2CO_3)-impregnated filter and back-up pad to collect both particulate arsenic compounds and arsenic trioxide vapor.(11) The company had also been using this method. NIOSH Method 6001 for arsine was also used in series behind the filter for area air samples.(11) Concerns that a gaseous form of arsenic (AsF_5) may also be present prompted the inclusion of this second, sorbent tube method for a limited number of area samples. Personal samples were collected at an average flow rate of 3.6 L/min, and area samples with the sorbent tube in line (NIOSH Method 6001) used a flow rate of 2 L/min. All equipment upon which work was performed by workers and which necessitated opening the system had been purged and tested to ensure that process gas had been removed before the system was opened.

All samples were collected for the period of time encompassing the actual task and varying periods of time before and after the work was performed. This was required for sample equipment setup and to allow for a health physics survey prior to the release of sampling equipment from the radiological contamination zone. Access restrictions associated with the contamination zones precluded access to sampling equipment once it was placed inside the area or taken in on the worker as a personal air sampler. Several sample cassettes required special handling, shipping, and analytical procedures due to the presence of technetium (^{99}Tc) contamination on the filters.

All workers entering the radiological areas to perform maintenance work during the NIOSH survey were monitored for airborne arsenic concentrations. Personal samples were obtained outside of the respirators. This ranged from one to five workers per location, depending upon the number of workers assigned to the task. Area samples were placed as close as practical to the work area without interfering with the work or workers' mobility. Job titles evaluated included maintenance mechanic, welder, instrument mechanic, chemical operator, converter mechanic, and security guard. Tasks for which airborne arsenic concentrations were evaluated were alumina trap changeout, positioning and welding of back-up strips onto a converter removed from the cascade, changeout of P-nut valves, and removal of an evacuation booster station valve.

Results

Samples were collected at three different locations within one of the process buildings and at one location in the converter maintenance facility. All area and personal exposure samples obtained during the placement and welding of back-up strips onto a converter were below detectable levels. All area and personal exposure samples for workers replacing P-nut valves were also below detectable levels. Area and personal exposures during the alumina trap changeout of top purge traps ranged from below detectable levels up to 0.3 $\mu g/m^3$ of total arsenic.

Potential personal exposures in excess of all relevant airborne arsenic exposure criteria were documented during the removal of a valve on the evacuation booster station located at the top end of the process. Personal exposure levels to arsenic for the four workers performing the evacuation booster valve removal ranged from 1 to 109 $\mu g/m^3$ of arsenic. The average exposure for these four workers was 37 $\mu g/m^3$, with a standard deviation of 50 $\mu g/m^3$. The chemical operator conducting the decontamination after removal of the valve had the highest exposure. The overhead crane operator's arsenic exposure was below detectable levels. All values presented in this paragraph represent time-weighted averages (TWAs) over the actual sampling period. Eight-hour TWAs calculated for all samples ranged from below detectable levels up to 22 $\mu g/m^3$ of arsenic. The two area samples located in the immediate proximity of the removal process demonstrated airborne arsenic concentrations of 0.7 $\mu g/m^3$. Area samples were not considered representative of the workers' personal samples. Tables 1 and 2 present the personal and area sampling results for airborne arsenic. The workers wore task- and work area-specific personal protective equipment.

The American Conference of Governmental Industrial Hygienists 8-hour TWA threshold limit value and the Occupational Safety and Health Administration (OSHA) 8-hour TWA permissible exposure limit (PEL) for arsenic are 10

TABLE 1. Range of Airborne Arsenic Concentrations Encountered by Job Title, Calculated as Both Sample Time-Weighted and 8-Hour Time-Weighted Averages

Task	Job Title (No. Sampled)	Minimum TWA* (μg/m^3)	Maximum TWA* Sample (μg/m^3)	Maximum TWA* 8-Hour (μg/m^3)
Alumina trap changeout	Chemical operator (n = 3)	ND	Trace	Trace
	Security guard (n = 1)	—	ND	—
Converter maintenance	Converter mechanic (n = 1)	—	ND	—
P-nut valve replacement	Instrument mechanic (n = 3)	—	ND	—
Booster valve removal	Maintenance mechanic (n = 3)	ND	5	3
	Welder (n = 1)		33	22
	Chemical operator (n = 1)		109	8

*Trace concentrations were calculated to be less than 0.1 μg/m^3 of total arsenic, and nondetectables (ND) less than 0.5 to 0.1 μg/m^3 depending upon sample duration. Sample times for the 13 samples ranged from 35 to 322 minutes, with a mean value of 156 minutes and a median of 127 minutes. TWA values are calculated two ways: the 8-hour TWA, which assumes zero exposure during the unsampled time period, and the sample TWA, which is calculated over the sample duration alone. All airborne contaminant values are in micrograms per cubic meter of air.

μg/m^3.(12,13) The OSHA arsenic standard also has an action level of 5 μg/m^3.(13) The NIOSH recommended exposure limit (REL) for arsenic is 2 μg/m^3 as a 15-minute ceiling value. Due to the carcinogenicity of arsenic, NIOSH recommends that exposure levels be maintained at the lowest feasible levels.(14)

One should note that all workers performing tasks inside of the radiological contamination areas wore personal protective clothing, including positive pressure supplied-air respiratory protection, anticontamination coveralls, gloves, and shoe covers. Table 3 lists the types of personal protective equipment in use. All work was performed inside of delimited radiation contamination zones. Sample cassettes were located outside of the workers' protective clothing at collar level.

Discussion

A review of the available arsenic sampling results collected by the site industrial hygiene department indicated that for a number of activities where potential arsenic exposures may occur, the levels were low (below 5 μg/m^3) or nondetectable. The NIOSH sampling data support the facility's findings that potential arsenic exposures occur with activities involved in opening uranium enrichment process equipment. All workers evaluated for personal exposure to arsenic compounds, with the exception of one individual, wore supplied-air respiratory protection. One worker overseeing the P-nut valve replacement was outside of the radiation contamination zone, and thus his work did not place him in the immediate vicinity of the work. The personal protective equipment in use was intended to protect workers from radiological contamination, process gas, its reaction products upon combining with air, and HF.

The site's industrial hygiene results were subject to the same difficulties experienced by the NIOSH investigators. Work tasks of short duration and the limited sampling periods for contaminant collection may result in nondetectable values higher than the NIOSH REL. The NIOSH sample results demonstrated two exposures in excess of any previously observed on site. These samples occurred at or near the segment at the top end of the cascade process referred to as the "arsenic bubble." The uranium enrichment process, as noted previously, is a physical separation process and appears to result in the concentration of different process impurities at various locations within the process equipment. Samples for arsenic contaminants within the process have identified arsenic concentrations approaching 4000 parts per million in "on stream" cells (several hundred milligrams/cubic meter of arsenic). (Note that a cell is an assembly of stages, each stage being the smallest complete separatory unit within the process.)

A modification of the sampling train used to evaluate airborne arsenic was incorporated into the area sampling conducted during this survey. An initial question was whether a filter sample for arsenic would capture all of the arsenic present. A standard charcoal tube was placed in line behind the air sampling

TABLE 2. Range of Area Airborne Arsenic Concentrations by Location as Both Sample Time-Weighted and an 8-Hour Time-Weighted Averages

Task	Location (No. Sampled)	Minimum TWA* Sample (μg/m^3)	Minimum TWA* 8-Hour (μg/m^3)	Maximum TWA* Sample (μg/m^3)	Maximum TWA* 8-Hour (μg/m^3)
Alumina trap changeout	Process building X-326 (2)	0.2	0.1	0.3	0.1
Converter maintenance	X-700 building (1)	—	—	ND	—
P-nut valve replacement	Process building X-326 (2)	—	—	ND	—
Booster valve removal	Process building X-326 (2)	0.7	0.5	0.7	0.5

*Trace concentrations were calculated to be less than 0.1 μg/m^3 of total arsenic, and nondetectables (ND) less than 0.5 to 0.1 μg/m^3 depending upon sample duration. Sample times for the 13 samples ranged from 35 to 322 minutes, with a mean value of 156 minutes and a median of 127 minutes. TWA values are calculated two ways: the 8-hour TWA, which assumes zero exposure during the unsampled time period, and the sample TWA, which is calculated over the sample duration alone. All airborne contaminant values are in micrograms per cubic meter of air.

TABLE 3. Personal Protective Equipment Used by Workers

Respiratory Protection	Protective Clothing
Process equipment tasks (inside the radiation contamination zones):	
Supplied-air respirators with full facepiece or hood; alternate is self-contained breathing unit	Nonwelding tasks: cloth coveralls (referred to as Anti-C's), polyethylene-coated Tyvek®-QC coveralls (for HF, technetium), double bib rad safe hood, booties and shoe covers, chloroprene gloves (taped) Welding/cutting tasks: as above, with possible substitution of regular Tyvek coveralls and omission of rad safe hood, welder's hood, and lenses over full-face respirator facepiece, cloth hood under welder's hood, booties, rubber gloves, light leather overgloves
Work on equipment wet-decontaminated or exposed to atmospheric moisture; no evidence of gaseous or high levels of particulate arsenic compounds:	
Full-face gas mask with high efficiency particulate air/acid gas canister	Anti-C's, rubber gloves, booties

cassette in an effort to determine whether gaseous arsenic compounds may also be captured. Although the number of these samples was limited, all of the sorbent tubes were nondetectable for arsenic. The arsenic values for these two part samples, if above detectable levels, were due exclusively to the contaminant collected on the filter.

This evaluation did not involve assessing worker exposures during work conducted on equipment or equipment lines that contained visible deposits of arsenic-containing materials. The NIOSH data apply primarily to the tasks evaluated on the process equipment and an example of work performed on a purged, cleaned, and isolated converter removed from the cascade building. The company had located tasks and work locations that presented greater potential for excessive arsenic exposures based upon their body of previously collected exposure information.

The routine required personal protective equipment (clothing and respiratory protection) used to prevent exposure to radiological contamination, the process gas (UF_6), and HF also offers by default substantial protection against arsenic compounds present in the process as a contaminant. Respiratory protection specified in the OSHA arsenic standard permits the use of supplied-air respirators with full facepiece hoods, helmets, or suits operated in the positive pressure mode for arsenic concentrations up to 20,000 $\mu g/m^3$.(13) The workers should be adequately protected from arsenic hazards when utilizing the protective equipment required in the radiological contamination zones. Supplied-air respiratory protection and protective clothing should be worn when working on arsenic-contaminated equipment in the absence of radiological contamination until the potential for arsenic exposures under these conditions is more fully evaluated. This requirement could be relaxed if the equipment is known to be free of arsenic-containing residues or levels are such that alternative personal protective equipment is judged to provide adequate protection. The OSHA arsenic standard permits, as an alternative to supplied-air respiratory protection, the use of full facepiece air-purifying respirators equipped with high efficiency filters for inorganic arsenic concentrations without significant vapor pressure below 500 $\mu g/m^3$ total arsenic. Arsenic compounds with significant vapor pressure below airborne concentrations of 500 $\mu g/m^3$ total arsenic may use front- or back-mounted gas-masked equipped with high efficiency filters and acid gas canisters.(13) Although NIOSH does not currently approve any air-purifying respirators for protection against arsenic exposures, except for the limited application of emergency escape purposes, NIOSH has identified air-purifying respiratory protection which may be suitable for protecting against airborne arsenic-containing compounds under defined conditions.(15,16)

The protective clothing required for use against other hazards also appeared to meet the OSHA requirements for full-body covering, gloves, shoes or coverlets, and face shields. The use of disposable protective clothing during the processes monitored by NIOSH addressed protective clothing provisions of the OSHA arsenic standard.(13)

The discovery of arsenic as a contaminant in the process prompted the site to begin regular testing of incoming feed material for this agent. The observations of workers familiar with the process and cognizant of deposits normally encountered in production equipment noted the presence and reaction of a previously unidentified material in the system. This prompted an investigation and the identification of a contaminant for which there was no readily plausible source in the process. Process, industrial hygiene, and laboratory personnel evaluating the new contaminant defined the origin of the contaminant and assessed the potential for exposures and the adequacy of worker protection. A stable, dedicated, long-duration process presented an unanticipated challenge. A toxic contaminant surfaced within the primary production process and remained undetected until sufficient quantities were deposited within the equipment lines, becoming noticeable to the workers.

Recommendations

Exposure monitoring and exposure control for arsenic at the facility should aim at reducing and maintaining arsenic exposures at the lowest level feasible. The OSHA PEL and action level may be used as guides in addressing exposures, although measurable airborne arsenic concentrations below a target level of 5 $\mu g/m^3$ do not denote the absence of an occupational health hazard. This is because of the concerns regarding the carcinogenicity of arsenic compounds. The amount and type of monitoring for arsenic exposures should focus efforts on characterizing high level or ill-defined arsenic exposures. Monitoring on a less intense basis should be continued for tasks previously evaluated that demonstrate negligible arsenic exposures to ensure that they remain unchanged. Exposure monitoring conducted to evaluate airborne arsenic contaminants should strive to evaluate levels below the most restrictive evaluation criteria.

The workers appeared to be adequately protected from arsenic hazards when utilizing the protective equipment required in radiological contamination zones; however, supplied-air respiratory protection and protective clothing should also be worn when working on arsenic-contaminated equipment in the absence of radiological contamination. This level of respiratory protection may be reduced, or at least need not address arsenic exposures, if the workpiece is known to be free of arsenic-containing deposits. NIOSH does not currently approve any air-purifying respirators for protection against arsenic exposures, except for the limited application of emergency escape purposes.(16) OSHA allows for the use of air-purifying respirators in the arsenic standard.(13) NIOSH has identified air-purifying respirators which may be suitable for use under defined conditions.(15)

The reuse of personal protective clothing that may have become contaminated with arsenic-containing dusts should be avoided. This did not appear to be a problem for the tasks evaluated by NIOSH because of the extensive use of disposable clothing. This could become a potential problem in instances where tasks involving arsenic-contaminated materials are being worked on, but are not subject to the constraints (and accompanying disposable protective clothing requirements) associated with handling radiologically contaminated equipment.

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