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Comprehensive Forensic Analyses of Debris from the Fatal Explosion of a "Cold Fusion" Electrochemical Cell

ABSTRACT: Selected components of explosion debris from the SRI International incident of January 2, 1992 were subjected to forensic analyses to elucidate potential causes of, or contributing factors to, the explosion. Interrogation of the debris encompassed nuclear, chemical, physical, and materials investigations.

Nuclear studies for the determination of tritium and neutron-activation products in stainless steel and brass were conducted. No evidence for signature species indicative of orthodox nuclear events was detected. The inorganic and particulate analyses were likewise negative with respect to residues of unexpected chemical species. Such target compounds included conventional explosives, accelerants, propellants, or any exceptional industrial chemicals.

Materials characterization identified the type of stainless steel used in the manufacture of the electrolytic cell as one relatively high in Mo concentration, probably type 316. Metallurgical analyses of the cell vessel wall and its detached base provided no evidence of corrosion or hydrogen embrittlement, leaving only ductile failure of the weld as contributing to the incident. The weld was found to have missed the center-line of the step joint, and the average penetration of the weld was measured to be 54%.

The GC-MS analyses of trace organic components in the explosion debris provided a most interesting result. Although no evidence of organic explosives, oxidizers, or other unusual compounds was detected, the presence of an organic oil in the interior of the electrochemical cell was established. It is likely that the source of this oil was lubricating fluid from machining the metal cell components. If residues of hydrocarbon oils are present during "cold fusion" experiments, the potential exists for an explosive reaction in the enriched oxygen atmosphere within the pressurized headspace of an electrolytic cell. It is possible that the oxidation of organic oil contributed to the total energy inventory, and perhaps the initiation, of the subject explosion.

KEYWORDS: forensic science, fatal explosion, "cold fusion," hydrocarbon oil, analyses by nuclear, physical, chemical, isotopic, materials, and engineering techniques

In April 1992, Lawrence Livermore National Laboratory (LLNL) began a dialogue with California's Division of Occupational Safety and Health (Cal-OSHA). As part of an incident investigation, Cal-OSHA desired the analytical interrogation of debris material confiscated by the Menlo Park Fire Department after the January 2, 1992 explosion in an electrochemistry laboratory at

SRI International (SRI). The explosion resulted in the death of scientist Andrew Riley, and gained some notoriety [1,2] due to its association with experimental work in the controversial field of "cold fusion" research. (Throughout this report, the term "cold fusion" is used as a convenient descriptor for electrolysis experiments designed to study the generation of excess heat by currently unknown mechanisms; whether conventional nuclear fusion might be a contributing factor to this phenomenon is problematical at the present time. A number of books describing the history, debate, and sociology of the "cold fusion" sensation are available [3–6]).

LLNL agreed to serve as an objective, third-party analytical laboratory for forensic analyses of the SRI debris. The Laboratory has a commitment to service in the public interest, and, in addition, as a major nuclear facility within the U.S. Department of Energy, had an inherent curiosity in the novelty of "cold fusion" since its announcement in 1989 [7]. Past applications of forensic science at LLNL have been predominantly in the realms of national security, defense, and intelligence operations.

On April 16, 1992, Cal-OSHA transferred custody of the explosion debris to LLNL. The evidence was handled by several people and organizations before receipt by LLNL, and the detailed history of these examinations is largely unknown to us. This fact may or may not be influential in the results of the forensic analyses, but present thinking is that it is but a minor effect in the overall analytic scenario. Some of the interesting debris remained in SRI's possession after the incident. These included the electrochemical palladium (Pd) electrodes as well as platinum-coated alumina (Al$_2$O$_3$) catalytic spheres. SRI conducted their own independent investigation of the incident [8–11].

For several weeks, the only analysis performed on the debris was nondestructive gamma-ray spectroscopy of selected metallic components. After signing a Cal-OSHA Confidentiality Agreement in May 1992, however, LLNL began more conventional forensic analyses of the SRI debris in the attempt to elucidate underlying mechanisms or contributing factors to the explosion. An initial, detailed inspection of the explosion residue in custody was undertaken. The components of the debris were weighed and photographed at that time. Selected photographs from this layout are presented as Figs. 1–5.

The more interesting pieces of debris (the main, stainless-steel cell vessel, the detached cell base, and the ripped Teflon liner) were then sampled for ensuing inorganic, organic, and physical analyses at the LLNL Forensic Science Center. Finally, interrogation of various debris components for structural integrity and materials characterization was performed by LLNL radiography, metallurgical, and engineering personnel. The results of most of the analyses performed at LLNL are presented here.

FIG. 1—*Main electrolysis "cold fusion" cell with detached base (inner surface shown) and ripped Teflon insert. The main cell impacted the head of the victim and caused the fatality.*

FIG. 2—*Main cell, detached base, Teflon insert, and pieces of brass heat-exchanger fins. The cell and the fins were analyzed separately for evidence of residual radioactive species.*

Nuclear Analysis

In the short history of "cold fusion" experimentation, perhaps the most contentious claims from various laboratories involve an association of excess heat generation with the presence or absence of signature species indicative of known nuclear processes. These nuclear signatures have included the production of neutrons, tritium, ^{4}He, and prompt gamma (γ) rays. Of these, the generation of neutrons as fusion by-products appears to be gaining some acceptance among the skeptics of "cold fusion." For example, one report claimed that on the order of 50 laboratories around the world have detected the presence of neutron emission in their

FIG. 3—*Side-view of the electrolysis cell. Note bulge below the threaded metal. Internal pressure estimated at 300 atm distorted the 0.2-cm thick, stainless-steel vessel from its initial configuration of a right-circular cylinder.*
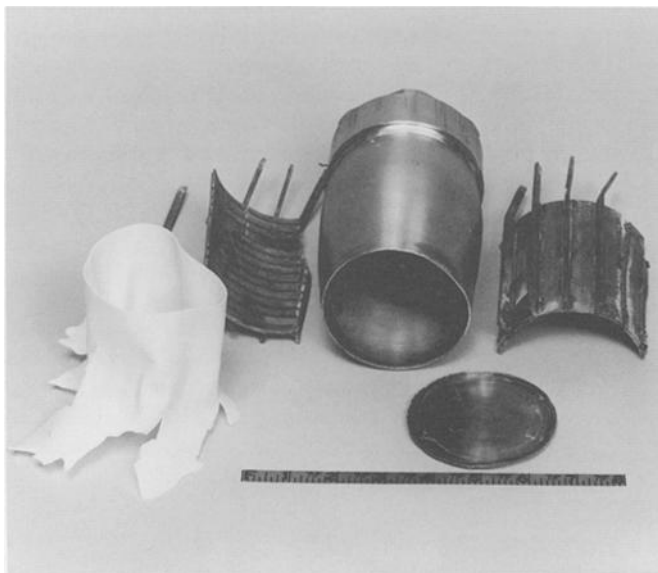
electrochemical experiments [12]. If a burst of neutrons was associated with the SRI explosion, it is possible that detectable activation products were produced in the surrounding materials. With a sufficiently high neutron fluence, radioactivity induced in the near environment might be detected and discriminated from background radiation by nondestructive γ-ray spectrometry.

Extensive studies along these lines were conducted at LLNL, and the question of induced radioactivity in the debris was initially the foremost topic of interest to the general public. Nevertheless, one referee of this paper thinks that the "nuclear-forensic" aspect of this investigation is too unfamiliar to, and thus inappropriate for, the readership of this journal. We have therefore reluctantly excised this section of the manuscript and submitted it elsewhere [13].

Chemical and Physical Sampling Methods

The pieces of debris sampled for chemical and physical analyses were the stainless-steel (SS) cell vessel (with threaded cap intact),
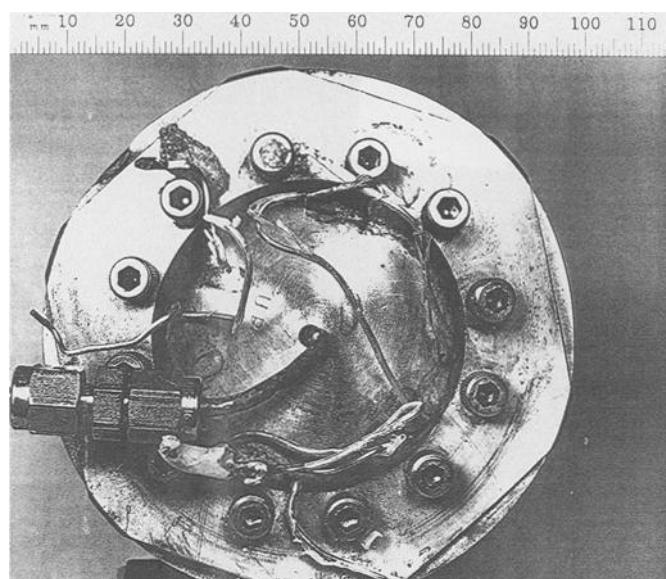
FIG. 4—*Top-view of the cell, showing a hex-head bolt assembly and the Swagelok-fitted gas delivery tube (which had been capped off prior to the explosion). Tissue from the victim is visible around the bolts at 11–12 o'clock.*
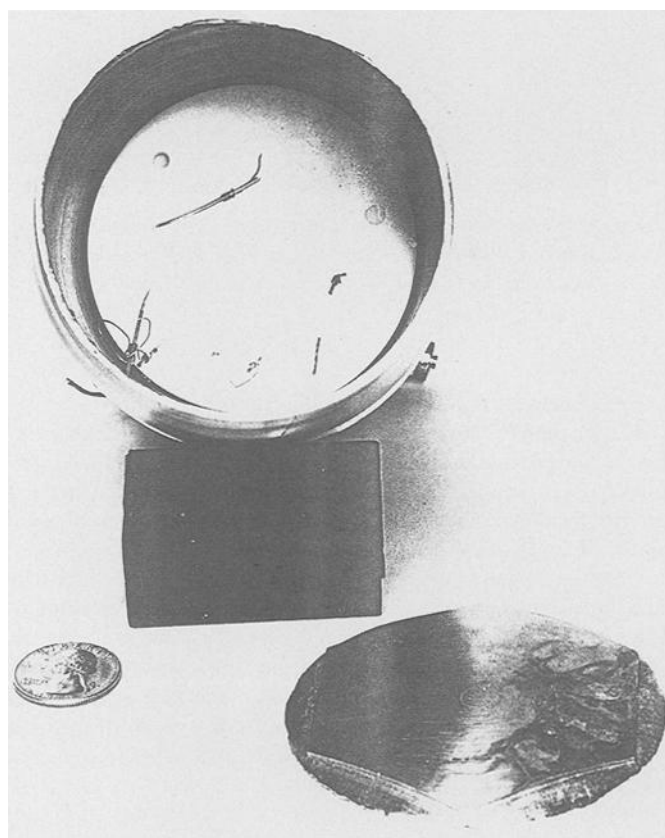

FIG. 5—*View into the interior of the main cell from the opening left by the detached base (also shown, external surface). The Teflon end-piece is clearly visible, as are severed electrical wire leads.*

the detached SS cell base, and the ripped Teflon liner (see Fig. 1). Small, particulate residues were removed from the interior of the cell vessel with scalpel and forceps. These particles could be visually grouped into white, black, and brownish components. As is visible in Figs. 1 and 2, a very small quantity of white residue was caked on the interior surface of the detached cell base. This material had to be pared off with a scalpel, and it appeared more homogeneous and fragile than the particles from the interior of the main cell vessel. A photomicrographic record of these specimens was obtained and archived.

Following removal of the particulate samples, the SS vessel, the detached base, and the ripped Teflon liner were each divided into four quadrants for chemical sampling of the specimens' interiors. One quadrant was wiped for organic residues with absorbent paper saturated with 3:1 methylene chloride/isopropanol solvent, another quadrant was wiped with paper and 0.05 M HNO_3 for inorganic residues, and a third quadrant was wiped with paper and 0.05 M NaOH, also for trace inorganic species. The fourth quadrant was left undisturbed for possible future interrogation by another laboratory, if desired. For the purposes of the chemical sampling, the interior of the SS cell vessel was separated into two distinct components: 1) the SS wall; and 2) a Teflon end-piece still intact in the cell cap, with holes and protruding wires visible upon viewing from the base of the open cell (see Figure 5).

The debris parts sampled for organic and inorganic chemical analyses were thus the interior portions of: 1) the Teflon end-piece in the cell cap; 2) the SS cell vessel walls; 3) the detached SS base; and 4) the ripped Teflon liner. Those numbers were also those assigned for sample identification purposes in the chemical analyses (that is, SRI-2 indicates a sample from the SS cell vessel walls, etc.). Appropriate blank specimens for all wipe procedures were generated at the time of the sampling.

Inorganic Chemistry Analyses and Results

The purpose of these analyses was to search for any unusual elemental or inorganic species that might be present in the debris. Although not expected from what was known about this incident, we routinely interrogate diverse samples for residual evidence of propellant or conventional explosive signatures. These tests are very sensitive and were conducted in the present investigation for completeness of the overall analysis.

Sample Pretreatments

The wipes from the four debris components and the blank were chemically manipulated to convert the samples to forms suitable for instrumental inorganic analyses. Each acid specimen was transferred quantitatively to an Erlenmeyer flask, and the absorbent paper was wet-ashed with concentrated HNO_3 and $HClO_4$. The residue was then dissolved in 4 M HNO_3 and centrifuged. The supernate from the centrifugation was transferred to a 50 mL class "A" volumetric flask and diluted to the mark with 4 M HNO_3. Aliquots from these acid samples were taken for elemental analysis by means of inductively-coupled plasma mass spectrometry (ICP-MS).

The basic wipe samples were processed differently. The absorbent paper was leached with a large excess of 0.05 M NaOH and sonicated for approximately 10 minutes. The leachate was decanted off, and this procedure was repeated twice. The three leachates were then combined and centrifuged. The supernate from the centrifugation was transferred to a 50-mL class "A" volumetric flask and diluted to the mark with 0.05 M NaOH. Aliquots from these

basic samples were taken for tritium analysis and for the inorganic analysis of a variety of anionic species by ion chromatography.

Tritium Analyses

The formation of tritium (^{3}H or T) in "cold fusion" cells has been taken as evidence of nuclear processes in the past [14]. However, the deuterated or "heavy" water (D_2O) used in electrochemistry experiments around the world has frequently been contaminated with minute quantities of DTO. The detection of T in "cold fusion" work has consequently become unpersuasive as an indication of nuclear events [15]. Nevertheless, we routinely perform high-sensitivity T measurements, and the SRI debris was also interrogated for this nuclide.

A 5.0 mL aliquot of each of the basic-wipe samples and the blank was analyzed for T by liquid scintillation counting (LSC). The aqueous fraction was combined with 10 mL of Ultima-Gold XR reagent to form a LSC cocktail, and the samples were then counted for 250 minutes each on a Beckman 9800 liquid scintillation counter.

None of the debris samples returned a counting rate above the LSC limit of sensitivity, nor did the process blank. The calculated LSC detection limit for T activity in these samples was 1.7 pCi/mL. Thus, no evidence for the presence of T was detected in the explosion debris that was examined by LLNL.

ICP-MS Analyses

Aliquots of each of the acid-wipe samples and the process blank were diluted by a factor of 10 and assayed via ICP-MS. The instrument was a VG PlasmaQuad Model PQ1 utilizing an argon plasma source, quadrupole mass spectrometer, and an electron multiplier detector. Spectra were acquired over the entire periodic table of naturally-occurring elements, between masses 5 and 240, with sensitivities on the order of nanograms of analyte per mL of original solution (μg/mL or ppb). The chemical elements which were difficult or impossible to analyze by this technique were Na, Mg, Si, P, S, K, Ca, Fe, and Se.

Concentration data for 21 elements (Li, B, Al, Ti, Mn, Ni, Cu, Zn, Sr, Zr, Mo, Pd, Ag, Cd, Sn, Ba, W, Pt, Pb, Bi, & U) were measured from sampling the explosion debris. The estimated concentrations of these analytes detected by ICP-MS were determined by comparison to a 50 ppb praseodymium internal standard added to each sample, with additional adjustments made to reflect differences in instrument sensitivity in different mass regions. The resultant values were thus considered semi-quantitative at best. However, due to the somewhat subjective nature of the wipe sampling, as well as to the fact that no normalization for differing sample surface areas was attempted, the ICP-MS data could ultimately be considered as qualitative only. The actual concentration measurements, which were provided to Cal-OSHA in a table in an initial, confidential report, were subsequently claimed to be proprietary information by SRI. As LLNL had signed the agreement with Cal-OSHA prohibiting the disclosure of collateral trade-secret discoveries, the detailed ICP-MS data table cannot be presented here.

Definite conclusions were possible from these ICP-MS results. Relative to the blank, all of the debris samples were enriched in traces of Li, Ag, and Pt. Selected debris specimens also indicated the presence of trace Mo, Pd, and Cd. In addition, the wipe of the SS cell vessel wall showed evidence of elevated Al and Fe. [Fe was not incorporated in the data table because analysis of the principle mass 56 peak is hindered by interference from the ArO$^+$ molecular ion originating in the plasma torch; secondary Fe mass

peaks were observed in the sample from the SS cell vessel, however.]

In the normal operation of "cold fusion" electrochemical cells, materials incorporating Li, Pd, and Pt are expected. Fe, of course, is an unremarkable component of SS, and Mo is a minor component of this same material (ICP-MS evidence for Mo was limited to the metal debris specimens). The source of the Ag in the SRI debris was likely a silvered glass Dewar known to be surrounding the cell in its experimental configuration [11]. This Dewar was shattered by the explosion.

Ion Chromatography Analyses

Suitable aliquots of each of the basic-wipe samples and the blank were taken for an ion-chromatographic investigation of the possible presence of unusual inorganic anions. The ions investigated were F$^-$, Cl$^-$, Br$^-$, NO$_3^-$, NO$_2^-$, HPO$_4^=$, and SO$_4^=$, and the instrument employed was a Dionex Model 2110i Ion Chromatograph with conductivity detection. The analytical column was an anion separation column Model AS4A, which was used in tandem with a conductivity suppressor column and 25 mN H_2SO_4 regenerant. Reagent eluants used for the separations were 2.5 mM $Na_2B_4O_7$ for F$^-$ and Cl$^-$, and 1.7 mM $NaHCO_3$ − 1.8 mM $NaCO_3$ buffer for the rest of the anions.

Although F$^-$ was not detected in the blank, it was measured in all of the debris samples except SRI-3, the detached cell base. The highest measured F$^-$ concentration was 16 μg/mL(ppm) in the wipe solution from the cell interior (SRI-2), but the comments regarding the subjective nature of the sampling and the unequal sample surface areas mentioned in the ICP-MS discussion above also hold for these ion chromatography results. The ubiquity of fluoride on the debris specimens is not surprising in light of the large quantity of Teflon present in the interior of the electrochemical cell.

Nitrate (NO$_3^-$) was slightly elevated in the SRI debris relative to the blank, but not by significant amounts (the maximum measured nitrate concentration was approximately 0.6 ppm). Nitrite (NO$_2^-$) results were below the limit-of-detection for that analyte (∼0.1 ppm) in all samples. The absence of nitrate and nitrite in the explosion debris is noteworthy because they are inorganic residues often present in the aftermath of certain conventional explosions.

Conclusions from Inorganic Analyses

Inorganic chemistry results for tritium were negative, and atomic analyses by ICP-MS did not indicate the presence of any unusual elements that would not be expected in a normal experimental configuration of a "cold fusion" electrochemical cell. Inorganic speciation analyses by ion chromatography likewise proved negative for signatures of conventional explosives. Thus, the inorganic analytical results afforded no indication of a nuclear involvement, nor did they provide evidence for the presence of any unusual compounds suggestive of explosives, accelerants, propellants, or other exceptional industrial chemicals. However, in the absence of prior knowledge of the details of this incident, the sensitive detection in the debris of trace amounts of Pt, Pd, Li, and enriched D/H (see next Section) would have led to the identification of the apparatus as a "cold fusion" experiment with a high degree of probability.

Particulate Analyses

The quantity of solid material recovered from the interior walls of the electrochemical cell and the detached base was minuscule

and was estimated to weigh perhaps a total of a few hundred micrograms. Such small samples precluded most conventional non-destructive analytical techniques for the chemical study of solid samples (for example, quantitative X-ray fluorescence spectroscopy). However, LLNL is capable of performing sensitive analyses of individual particulate samples as small as a few microns in length. These analyses provide information on elemental concentrations, oxidation states, and isotopic compositions of an individual particle specimen.

Debris particles recovered from the stainless-steel cell vessel were analyzed separately from those scraped from the detached cell base. The techniques used were ion microprobe mass spectrometry (IMMS), scanning electron-probe microanalysis (SEM/EDX), and electron spectroscopy for chemical analysis (ESCA).

Electron and Ion Microprobe Analyses

Distinct white, black, and brownish particles were selected from the sampling of the cell interior for analyses by IMMS and SEM/EDX. The more homogenous white particles from the cell base were analyzed as a single population by these same instrumental techniques. A total of seven particulate specimens were taken for these studies, and their composite weight was estimated at ~200 μg.

The detailed results of the IMMS and SEM/EDX analyses, including a number of photomicrographs, are available elsewhere [16]. The black and brown particulates from the cell interior were identified as consisting fundamentally of an Al_2O_3 matrix with 10-μm subhedral inclusions of Pt interspersed throughout. The white particles from both the cell interior and the detached cell base were characterized as LiOH + LiOD.

Although isotopic analysis of D/H in these latter specimens revealed enrichment factors of 80–180 relative to normal samples, an initial puzzle was that the most enriched sample analyzed still contained 40 times more protons than deuterium atoms. For electrochemical experiments at high concentrations of LiOD in reasonably pure D_2O, one might expect very much higher D/H ratios in the residual LiOD than those actually measured.

A plausible explanation for this finding is isotopic exchange between LiOD and H_2O in normal atmospheric water vapor over the five months since the initial explosion. LiOH-LiOD is known to readily absorb water from the air, and no attempt after the incident was made to isolate the small and finely-divided quantity of residual LiOD from subtle environmental interactions. The loss of deuterium enrichment through free exchange with atmospheric H_2O of normal isotopic composition is consistent with what is presently known about exchange equilibria in the H/D isotopic system [17].

ESCA Analyses

The remainder of the particulate specimens were taken for ESCA analysis. They were adhered to small pieces of carbon tape and mounted on posts in order to concentrate them into as small an area as possible. However, the amounts of sample available from both the cell-interior and detached-base locations were so small as to present a serious challenge for composition determinations by the ESCA technique.

The interrogation of both specimens over a suitable surface area was performed with a Perkin-Elmer 5400 ESCA system via X-ray photoelectron spectroscopy. An extremely long (~14 hours), broad scan was taken to identify the elemental composition of each sample with maximum sensitivity. This initial scan was then followed by high-resolution scans to measure atomic concentrations and effect oxidation-state analyses. Speciation determinations by ESCA are accomplished by means of regression analysis at appropriate regions in the photoelectron spectrum.

The elements identified in the residue from the detached cell base by ESCA were Si, O, Li, and Mg, with the Si present as the R-Si-O functional group (102-eV binding energy). The ESCA sample from the interior of the SS cell was a mixture of black and white particles. The elements present at highest concentration in this sample were C, O, Li, Al, and F, and all were measured at levels greater than 2 atomic percent (Li was present at 13 atomic percent). Aluminum was characterized as Al_2O_3 and Si again as R-Si-O. Approximately 10% of the assayed carbon was identified as $-CF_2-$ (the Teflon polymer chain). The Li binding energy of 55.5 eV was tentatively ascribed to LiF; however, LiOD could not be ruled out as the actual chemical form of the Li because the ESCA spectrometer was not calibrated for that particular species.

None of the ESCA results were surprising when considered in light of the usual reagents and materials expected in the normal operation of a "cold fusion" electrolytic cell. Considering the meager quantity of analyte and the more heterogeneous nature of the ESCA analytical samples, consistency of the ESCA results with the IMMS and SEM/EDX analyses was quite satisfactory.

Materials Characterization and Structural Analyses

Nondestructive XRF Analyses

One of the unanswered questions at SRI during their analysis of this incident was the specific type of stainless steel (SS) used in the fabrication of this particular electrochemical cell. According to one of their scientists, the most likely choices were type 304 SS or type 316 SS. To answer this question, we performed nondestructive X-ray fluorescence (XRF) spectroscopy on strategic portions of the SS cell vessel and the detached base. As type 316 has a significantly higher concentration of Mo in its composition than type 304, XRF analyses of Mo concentration were considered decisive for materials discrimination.

We used a Kevex Analyst Model 770 energy-dispersive XRF spectrometer with secondary-target fluorescence. The secondary target utilized for analyses of the cell debris was Ag, so that the effective elemental upper limit for the X-ray spectra was Rh. The SRI samples were analyzed at instrument settings of 50 keV and 0.2 mA, and peak identification and integration were performed with proprietary Kevex software for spectral deconvolution. Quantitative analysis was effected by comparison with NBS (now NIST) primary SS standards.

XRF analyses of one type-304 NIST primary SS standard (SRM 1152) and two type-316 NIST primary SS standards (SRM 1155 and SRM 1185) were used for system calibration. The NIST-certified values for the weight-percent ratio of Cr to Mo [W(Cr/Mo)] in these standards were 50.5, 7.8, and 8.5, respectively. Thus, W(Cr/Mo) in type 304 SS is approximately a factor of six greater than W(Cr/Mo) in type 316 SS, and this measurement can serve as a basis for characterization.

Qualitative XRF results for the elements present at detectable levels in the SRI debris identified Cr, Mn, Fe, Co, Ni, Nb, and Mo in all samples. Determinations of W(Cr/Mo) in these specimens resulted in the following values: 8.4 for the detached base, 8.6 for the body of the cell vessel, 6.5 for the threaded cell cap at an undistinguished location, and 6.3 for the threaded cell cap at the location of a black smudge mark. The XRF data from this last

position also indicated the presence of Cu and Zn in roughly the correct proportions for metallic brass.

Provided that the only choices were type 304 or type 316 SS, the XRF measurements indicated that the electrochemical cell was manufactured from type 316 SS. However, this analysis did not preclude the possibility of other Mo-rich stainless steels, such as types 316L or 317. The XRF data also suggested that perhaps the cell impacted a brass object at the location of the black mark on the cell cap following the explosion. Alternatively, the brass component may reflect imbedded material from the heat-exchanger fins that surrounded the cell vessel at the time of the explosion.

High-Energy X-Ray Radiography

Before the complete disassembly of the threaded-cap structure from the SS vessel wall, the interiors of the electrochemistry cell and a gas fill/vent tube were imaged by means of a Varian industrial radiography instrument. This machine is an electron linear accelerator with continuous energy variability up to 9 MeV, and high-energy X-radiation is produced as bremsstrahlung in a tungsten target [18].

Two X-ray energies were employed for the interrogation of the SRI experimental apparatus. A lower energy (200-keV) beam was used for higher resolution within the gas tube, while high-energy (4-MeV) X-rays were used for better detail inside the thicker SS walls. A detailed radiography report can be found in Ref 16, while a representative radiograph is shown in Fig. 6. Unfortunately, positive prints such as Fig. 6 do not possess the clarity of the original X-ray negatives.

Some information important to the investigation was obtained from the X-ray radiographs. Primary was the fact that there were no obstructions in the damaged gas tube from the top of the cell cap through the Swagelok fitting. This greatly diminished the possibility of an accidently-plugged vent as a possible contributor to the incident. Additionally, the radiography also showed that the Teflon end-piece within the cell cap was skewed within the cap enclosure. This deformity could have arisen from the significant increase in pressure in the cell prior to the explosion, or it could have been dislodged by the explosion itself or by post-explosion impact(s) of the cell.

Post-Explosion Cell Dimensions

Accurate measurements of the dimensions of the stainless steel cell vessel and the detached cell base were made. These measurements included the thickness of the cell wall at various strategic locations: approximately 0.2 cm. The cell was initially a right-circular cylinder of diameter 8.7 cm; the post-explosion diameter of maximum deformation was determined to be 9.4 cm, an expansion of 8%. The details of this work, and appropriate sketches, are given in Ref 16.

Metallurgical Analyses

The steel cell vessel and detached base were sectioned for metallurgical analyses via SEM and metallography. Both the base metal and the weld material were found to be free of significant impurities or contamination. The weld at maximum penetration missed the center-line of the step joint by 0.5 to 1.3 mm, as measured at three different debris locations, and the weld penetration was incomplete. Four separate measurements of weld penetrations gave an average value of $(54 \pm 8)\%$.

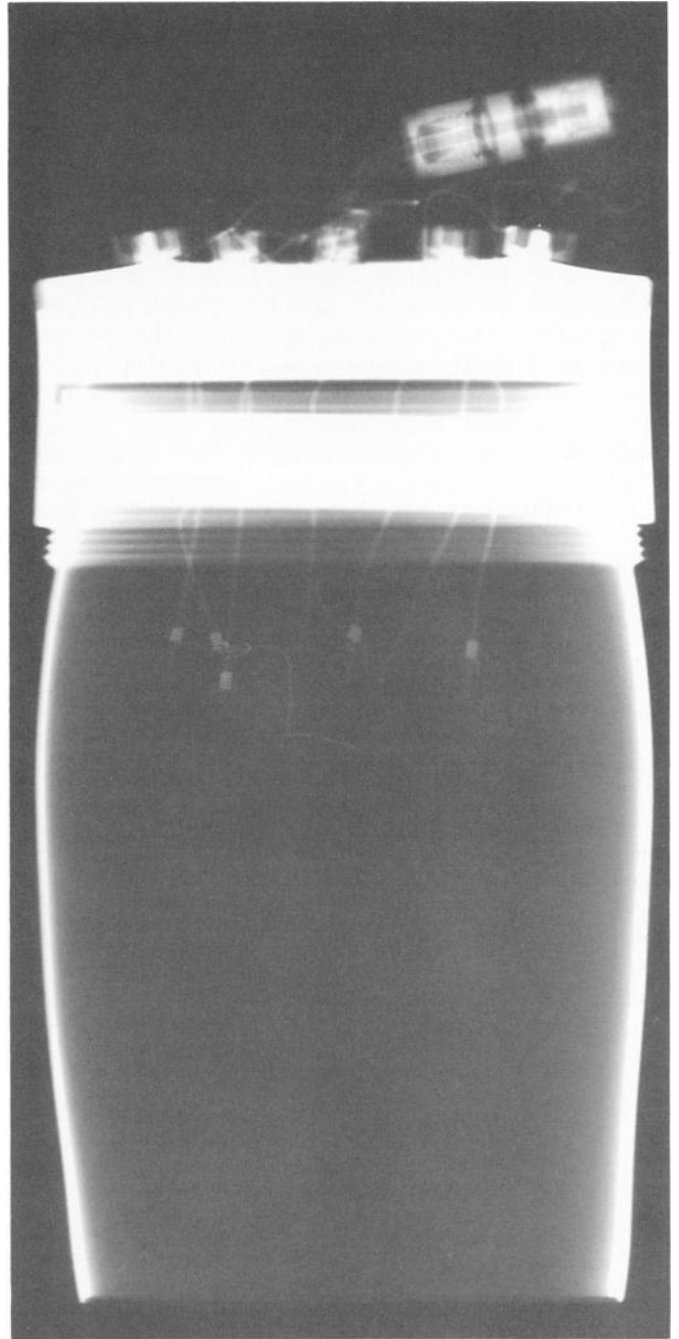No evidence of corrosion or hydrogen embrittlement was

FIG. 6—X-ray radiograph (4-MeV image) of the interior of the electrolysis cell and gas delivery tube.

observed. Interrogation of the debris by SEM revealed dimple-rupture and wavy-glide features, thus indicating mechanisms of ductile failure from overload of the weld and parent-metal intersection at the cell base. The region of initiation of the vessel failure was identified.

The details of the complete metallurgical report are given in Ref 16. Also found there are appropriate photomicrographs at magnifications from 32–100× and SEM micrographs at magnifications of approximately 500 and 1000×.

The explosion thus resulted from an extraordinary increase in internal pressure that caused the welded base to "unzip" from the main cell structure (the SRI pressure estimate via computational

techniques and various assumptions was 300 atm [*8–11*]). The consequent pressure release propelled the 1.7-kg cell through the laboratory with appreciable momentum and resulted in the death of the victim by direct impact.

Organic Chemistry Analyses and Results

The purpose of these analyses was to search for any unusual organic species that might be present in the debris. Although not expected from what was known about this incident, we routinely interrogate diverse samples for residual evidence of propellants or conventional explosive signatures. Like the inorganic analyses, these methods are also exceedingly sensitive and were implemented here for analytic thoroughness.

In fact, however, the possibility of organic reaction mechanisms in "cold fusion" experiments was quite unanticipated. Orthodox discussion of this controversial and unorthodox field provides no hint of considerations of organic chemistry. As these analyses provided the most interesting result of our studies, though, the case is made for completeness and unbiased scrutiny during any forensic examination.

In order to determine what trace organic compounds may have been present on the interior of the SRI explosion debris, selected components were extracted with pre-cleaned wipes saturated with organic solvent. The organic species were then removed, concentrated, and analyzed by computer-guided gas chromatography-mass spectrometry (GC-MS).

Sample Pretreatment

The portions of the SRI debris specimens that were described in the Sampling Methods section of this paper were extracted for possible trace organic species via the following protocols.

1. Absorbent Paper for Wipe Samples
   Cellulose papers (Whatman No. 4, ash-free) were folded and clamped with forceps. The paper was immersed in a clean solvent system of methylene chloride/isopropanol (3:1, Fisher Scientific, Optima Solvents) and extracted three times with fresh, clean solvent to remove most of the background organic contaminants in the paper. The final rinse with the extraction solvent was analyzed as a blank sample. The papers were then suitable for sampling the interiors of the SRI debris specimens. A separate, pre-cleaned paper was used to extract the trace organic species from each debris component of interest.
2. Organic Extraction
   The selected area of each debris specimen was wiped with one pre-cleaned paper. Following each wipe, the extraction paper was soaked in a 10-mL vial containing 5 mL of methylene chloride/isopropanol solvent and squeezed semi-dry. This process was then repeated at a new location on the specimen surface. The final wipe was again soaked in the same 5 mL of solvent, squeezed dry, and the paper archived for later analysis.
3. Concentration of Organic Extract
   The 5-mL solvent volume from each of the extractions was evaporated under a stream of ultrapure nitrogen to a 10-μL concentrate. From this concentrated sample, an aliquot of 1.0 μL was then analyzed by GC-MS.

Gas Chromatography-Mass Spectrometry Analyses

The analysis of each of the samples used computer-guided GC-MS techniques for the separation and identification of all organic compounds extracted by the wipe sampling. Gas chromatography was performed on a Varian model 3400 instrument fitted with a 30 m × 0.25 mm i.d. DB-1 column with 0.25 μm film thickness (J&W Scientific). Injections were done in the splitless mode with a purge delay of 0.6 min and a purge vent of 100 mL/min. The injector and transfer-line temperatures were 250°C, and the column was programmed from 60–300°C at 8°C/min with a 10 min plateau at 300°C. A Finnigan ion-trap mass spectrometer (ITMS) was used for the analysis and identification of all compounds eluted during chromatography. The ITMS was operated in the electron-ionization mode, full scan, with automatic gain control.

The chromatograms depicted in Figs. 7 and 8 show the relative concentrations of various eluted compounds (y-axis) as a function of chromatography retention-time (x-axis). Typically, the larger and/or more polar the analyte compound, the longer the retention-time observed.

The results of the GC-MS analyses are summarized below. All of the samples tested positive for some residual organic matter. The identification of distinct species was accomplished with our
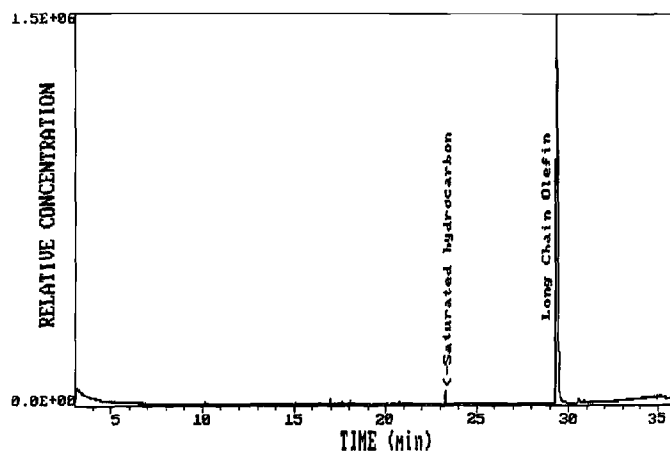

FIG. 7—*GC-MS chromatographic profile of the procedural blank specimen for organic analyses.*
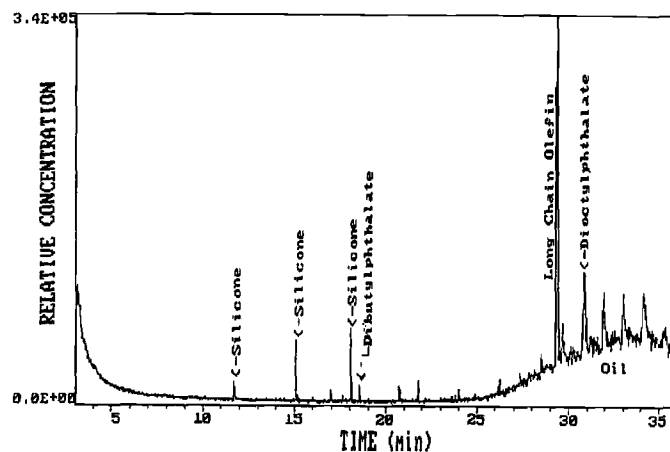

FIG. 8—*GC-MS chromatographic profile of the organic wipe of the interior of the main stainless steel cell.*

analysis software, *M Code* [*19*], along with a computerized library search and comparisons with known compounds in a NIST database [*20*].

1. Procedural Blank

    See Fig. 7. The elution of extraction solvent during a GC-MS separation typically occurred over an interval of approximately 3 to 7 minutes after sample injection. The slight rise in the chromatogram baseline from 31–35 minutes is attributed to column bleed.

    The total ionization plot of the concentrated sample obtained from the procedural blank (solvent saturation of Whatman No. 4 paper) revealed a long-chain olefinic compound eluting at 29.2 minutes after the start of the GC-MS run as the major component. This impurity was likewise observed in the majority of the wipe samples. A minor hydrocarbon peak was also seen at 23.2 minutes. As a result of these GC-MS results, the procedural blank was judged acceptable and a valid point of comparison for subsequent interpretations of debris-specimen spectra.

2. Ripped Teflon Liner Wipe Sample (SRI-4)

    The interior of the Teflon liner revealed a series of silicones, phthalates, and short-chain hydrocarbons. Dioctylphthalate was the major compound isolated from the surface of the Teflon. The other compounds identified were tetramethylsiloxanes and other methyl-substituted siloxanes.

3. Detached-Base Wipe Sample (SRI-3)

    The detached stainless steel base was found to be coated with similar silicones and plasticizers. Dioctylphthalate was again the major compound on the surface of this debris specimen. No other major components were seen.

4. Teflon End-Piece Wipe Sample (SRI-1)

    The wipe of the solid Teflon piece in the cap of the reaction cell revealed the background olefinic impurity, as well as silicones and dibutylphthalate. No other unusual species were seen.

5. SS Cell Vessel Wall Wipe Sample (SRI-2)

    The surface area sampled with this wipe was estimated to be approximately 80 cm^2. This sampling of the metal walls of the electrolysis cell produced a chromatogram which identified silicones, dibutylphthalate, dioctylphthalate, and a series of compounds which were indicative of organic oil.

    The chromatographic signature of the oil was indicated by the rise in baseline starting at a retention-time of 25 minutes and continuing on to 35 minutes after injection (see Fig. 8). Examination of the mass-spectral data over this time period revealed a continuum of hydrocarbon compounds, similar to that of saturated oil residues. Fragment ions at mass-to-charge ratios (m/z) of 57, 71, 85, 99, . . . were thus indicative of hydrocarbons ($\Delta m = 14$, the $-CH_2-$group). The presence of these species, along with the characteristic chromatographic profile at elevated temperature (corresponding to longer retention time), is consistent with the presence of oil.

Conclusions from Organic Analyses

All of the wipe samples of the SRI debris contained trace amounts of organic contaminants. Compounds such as silicones and phthalates were identified in these samples, and such species are very common in the normal environment. The plasticizers are

almost always associated with commercial plastics. The bags in which the specimens were stored could have been a likely source of these phthalates, as might reagent bottles used to initially contain reactant materials utilized in "cold fusion" experimentation.

The source of the silicones could have been the cell's internal o-ring or perhaps a silicone grease used to lubricate the o-ring prior to sealing the reaction vessel. Like plasticizers, silicones are found on nearly all industrial materials and are utilized for a variety of purposes, such as antifoaming agents and lubricants.

Mass-spectral data and GC-profile information strongly suggest the presence of organic oil on the walls of the metal reaction vessel. Similar hydrocarbon oil was not detected on any of the other debris specimens that were wiped. The GC-MS profile of the oil present on the cell vessel wall was indicative of a heavy oil (estimated $\sim C_{20}$-C_{34} branched hydrocarbons), and not a kerosene or diesel-type fuel typified by high and medium volatility, respectively. The instrumental limit-of-detection for this oil was assessed to be on the order of a few nanograms for the wipe area of $\sim$80 cm^2. However, analytical experience in the processing of similar oil samples allowed an estimate of hundreds of μg of oil in the subject debris wipe. In considering this information, it should be appreciated that the wipe sampling interrogated only a fraction of the interior surface of the steel cell and that sampling occurred after an explosion of considerable force.

Supplementary Remarks

The presence of silicones, phthalates, and short-chain hydrocarbons in the debris is not unusual. Nearly all materials either contain these compounds, or are coated with such lubricants and plasticizers; appropriate cleaning of ordinary items with suitable, ultraclean solvents in order to remove all traces of organic residues is rarely performed. No organic explosives, oxidizers, or other unusual compounds associated with more conventional explosions were detected in the SRI debris samples.

The oil identified in the interior of the electrolytic cell may have originated from the fabrication of the metal container. Much hydrocarbon oil is often used during machining to cut threads, drill holes, turn on a lathe, and fabricate most metal parts.

We cannot absolutely guarantee that the detected oil was a component of the "cold fusion" reaction mixture at the time of the explosion. The debris was handled by several organizations after the incident in a fashion largely unknown to us. It is possible that the oil was introduced as an external contaminant during the 3.5 months between the explosion and receipt of the evidence at LLNL. However, none of the other sampled specimens tested positive for this analyte, and the interior of the steel cell was more protected from potential environmental influences than the rest of the debris. Moreover, the GC-MS analyses were also very sensitive to the presence of palmitic acid, stearic acid, and cholesterol. The former two compounds are signature species for fingerprint residues and common human handling, while the latter is a general indicator of biologic contamination. None of these particular indicator compounds were detected in any of the GC-MS analyses.

Organic oil in the interior of an electrochemical "cold fusion" cell may be quite important from a safety viewpoint. The reaction mechanism of these experiments involves the electrolytic decomposition of D_2O to form overpressures of gaseous D_2 and O_2, with some sorption of D_2 into the matrix of a Pd electrode. Consequently, the cell headspace becomes enriched in pressurized O_2, and hydrocarbons are known to be an excellent fuel for combustion with

oxygen. Indeed, some bipropellant rocket fuels use only oxygen and a hydrocarbon source (kerosene) as the propulsion system.

A well-known problem in the manufacture of rocket engines, as well as in the high-pressure gas industry, is the instability generated when oxygen is exposed to organic residues. Pressurized oxygen can react violently with trace amounts of grease, oil, or other readily oxidizable material, and these substances ignite more readily and burn more rapidly than under normal conditions. "*If such contamination exists, explosive oxidation may occur when contact is made with the compressed oxygen*" [21]. Reports of anomalous and energetic incidents with cylinders of oxygen persist to the present time [22].

If oil residue is present in the interior of a "cold fusion" reaction cell when overpressures of oxygen are generated during electrolysis, there is a distinct possibility that an exothermic, explosive reaction could take place. The utilization of organic residues (including silicones and phthalates) as a combustion source is quite credible. It is possible that the oxidation of hydrocarbon oil contributed to the total energy inventory, and perhaps the initiation, of the subject explosion.

An important protocol in the implementation of high-pressure gases is that any components to be used in an oxygen atmosphere must be scrupulously cleaned to avoid explosions and catastrophic overpressures caused by heat from the combustion of organic residues. The American Society for Testing and Materials (ASTM) has set standards for clean-room conditions and decontamination procedures for any components used in oxygen containment. All equipment utilized for oxygen service, whether the oxygen is a component of a mixture or present as a pure gas, must be specifically designed to avoid organic constituents and cleaned to remove any traces of oils, greases, or other hydrocarbon materials. Mechanical and chemical procedures for cleaning components and systems to be used for oxygen containment have been clearly outlined [23]. A direct quote from this reference reads, "*Caution: Never permit oil, grease, or other combustible substances to come in contact with cylinders, valves, regulators, gases, hose, and fittings used for oxidizing gases, such as oxygen and nitrous oxide, which may combine with these substances with explosive violence.*"

In conclusion, it would be prudent to establish effective cleaning methods for hydrocarbon impurities likely to be present on apparatus utilized in the interior of "cold fusion" electrochemical cells. Such measures should be relatively unobtrusive in the design and practice of these experiments. The Special Order imposed by Cal-OSHA on SRI prior to their resumption of "cold fusion" experimentation after the explosion mandated just such cleaning procedures.

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