Petition to Delist Tritiated Mixed Waste Treatment Residues

June 30, 1999

Prepared for

Delisting Program Coordinator
Waste Management Division
State Programs and Compliance Branch
U.S. Environmental Protection Agency, Region IX
75 Hawthorne Street
San Francisco, CA 94105
Attention: Cheryl Nelson, Senior Regulatory Advisor

Prepared by

Lawrence Berkeley National Laboratory
University of California
Berkeley, California 94720

SECTION 1: DELISTING ADMINISTRATIVE INFORMATION

1. Name of petitioner

a. Name of individual or firm submitting petition:

University of California -- E.O. Lawrence Berkeley National Laboratory (LBNL)

b. Mailing address of individual or firm:

Street / P.O. Box: One Cyclotron Road, Mailstop 85B

City: Berkeley

State: California Zip Code: 94720

2. People to contact for additional information pertaining to this petition

Name	Title	Telephone No.	Mailing Address
Robin A. Wendt	LBNL Waste Management Group (WMG) Leader	(510) 486-6012	See 1(b)
Dr. Philip G. Williams	Co-Principal Investigator and Facility Manager for National Tritium Labeling Facility at LBNL	(510) 486-7336	One Cyclotron Road, Mailstop 75-123, Berkeley, California 94720
Dr. Li-Yang Chang	LBNL Generator Assistance Technical Liaison	(510) 486-4843	See 1(b)
Nancy E. Rothermich	LBNL WMG Compliance Team Leader	(510) 486-4644	See 1(b)

3. Facility responsible for generating petitioned waste

a. Facility Name: National Tritium Labeling Facility (NTLF) at LBNL

b. Location of facility:

Street / P.O. Box: One Cyclotron Road, Mailstop 75-123

City: Berkeley

State: California Zip Code: 94720

c. USEPA ID number: CA 4890008986

4. Location of petitioned waste

a. Name of facility: Hazardous Waste Handling Facility (HWHF) and National Tritium Labeling Facility, LBNL

b. Location of facility:

Street / P.O. Box: See 1(b) and 3(b) above

City: Berkeley

State: California Zip Code: 94720

c. USEPA ID number: CA 4890008986

5. Describe the proposed delisting action

Pursuant to 40 CFR Section 260.22(b), Petitioner Lawrence Berkeley National Laboratory requests that the Environmental Protection Agency (EPA) delist certain "derived-from" F-002, F-003, and F-005 treatment residues. The treatment residues subject to this petition are the products of catalytic oxidation of organic tritiated wastes that, as generated, were or will be D001 high-TOC ignitable; contained or will contain one or more F002, F003, or F005 constituents; and at times were or will be D-coded for organics such as chloroform (D022). The petitioned treatment residues consist of (1) the oxidized and condensed liquid catalytic oxidation product (tritiated water or HTO) and (2) oxidation process bubbler water (HTO) stabilized on silica gel.

These treatment residues (HTO and HTO on silica gel) contain no detectable levels of the constituents that caused the EPA Administrator to designate the "as-generated" wastes from which they are derived as F002, F003, and F005-listed; exhibit no RCRA characteristics; and contain no RCRA-regulated constituents above universal treatment standards (UTS; 40 CFR 268.48). In addition, based on process knowledge, the treatment residues contain no other 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern.

In essence, these residues consist solely of moderate to high-activity tritiated water. Nevertheless, because these treatment residues are still regulated under RCRA as F-listed wastes based on the "derived-from" rule (40 CFR Section 261.3(c)(2)(i)), there are currently no feasible disposal options for them. No RCRA-permitted mixed waste disposal sites have radioactive materials licenses that will allow acceptance of tritium activity levels as high as those in the treatment residues. In addition, low-level radioactive waste disposal sites licensed to accept such activity levels lack the RCRA permit required to accept the treatment residues, so long as they remain F-listed. Pursuant to 40 CFR Section 260.22(b), in order to open options for subsequent management of these treatment residues, LBNL is submitting this petition to exclude them from 40 CFR Section 261.3(c).

In conjunction with this delisting petition, LBNL seeks EPA's concurrence with our conclusion that our catalytic oxidation technology is within the federal regulatory definition of "combustion" (40 CFR 268.42, Table 1) for the purpose of meeting the technology-based standard for D001 high-TOC ignitable wastes. Alternatively, we seek approval of our catalytic oxidation technology as an alternative treatment method in lieu of combustion, pursuant to 40 CFR 268.42 (b) (see Attachment A).

As the EPA and the Nuclear Regulatory Commission have acknowledged in regulatory guidance documents and in final and proposed rulemakings, dual regulation of RCRA mixed waste under the Atomic Energy Act and the Resource Conservation and Recovery Act (RCRA) presents a number of difficulties. In the particular case of LBNL's tritiated mixed waste, the radioactive component of the "as-generated" waste contains moderate to high levels of tritium. The hazardous component includes F-listed spent solvents, is designated as high-TOC ignitable D001 waste, and at times is D-coded for constituents such as chloroform. Under RCRA, prior to disposal, treatment to meet concentration-based land disposal restrictions (LDRs) for the F-listed and D-coded components must be performed. Also, the D001 high-TOC designation of the waste means that the technology-specific LDR of combustion must be met. However, available offsite commercial or DOE options for performing this RCRA-required treatment are not desirable in LBNL's view, based on environmental consequences associated with the high tritium activity in the waste and on expense. (See Item 6).

In order to explore an alternative to incineration that would be environmentally and fiscally preferable, LBNL initiated a tritiated mixed waste treatability study at its National Tritium Labeling Facility (NTLF) in 1996. The study's primary purpose was to evaluate the effectiveness and efficiency of treating tritiated mixed waste using catalytic chemical oxidation (CCO) technology in conjunction with solvent vacuum extraction technology. The CCO technology involves high-temperature decomposition of organic chemicals in the presence of a catalyst. This technology has been demonstrated successfully for organic chemical treatment in remediation projects, where it has been shown that the process can accommodate both organic and aqueous mixtures and achieve a decomposition and removal efficiency (DRE) in excess of 99.999%.

A summary of the strategy developed for NTLF mixed wastes in connection with the treatability study is as follows:

(1) Identify the tritiated mixed waste streams suitable for study.
(2) Evaluate the hazardous and radiological characteristics of the identified waste streams.
(3) Evaluate the effectiveness and efficiency of the CCO and solvent vacuum extraction technologies.
(4) Evaluate the characteristics of the oxidized and condensed liquid product.
(5) Identify regulatory constraints.
(6) Obtain delisting approval for residues of waste that originally contained F-listed components, and obtain concurrence with our conclusion that the LBNL CCO technology is within the regulatory definition of "combustion" (40 CFR 268.42, Table 1) or is an acceptable alternative treatment methodology for D001 high TOC waste.
(7) Dispose of the delisted CCO treatment residues at a licensed low-level waste disposal site, or send them off site for tritium recycling.

LBNL has been exploring two similar CCO systems built for comparative studies of treatment of simulated nonradioactive surrogate samples (the CCO-1 system) and radioactive tritiated samples (the CCO-2 system). Attachment B of this petition discusses these CCO processes in detail.

Treatment through CCO was expected to generate nonhazardous products, principally carbon dioxide and tritiated water (HTO). The NTLF treatability study has in fact demonstrated that the CCO technology is capable of destroying common organic solvents and can achieve a measure of performance equivalent to that achieved by other combustion methods specifically identified in 40 CFR Section 268.42, Table 1. It has also demonstrated that the residues of the CCO treatment should be exempted from regulation as listed "derived-from" wastes. As stated earlier, they contain none of the constituents that were the basis for the EPA Administrator's F-listing of the "as-generated" wastes from which they were derived, exhibit no RCRA characteristics, contain no underlying hazardous constituents above UTSs, and (based on process knowledge) contain no other 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern.

6. Provide a statement of your interest in the proposed action

The United States biomedical R&D community faces extremely limited treatment and disposal options for many of the mixed wastes generated by their research activities. These options are particularly limited for tritiated mixed waste that has both moderate to high tritium content and RCRA-regulated hazardous components. The treatment and disposal problems for mixed wastes exist on a relatively large scale in the pharmaceutical industry, where isotopes such as tritium and carbon-14 are used regularly in research and drug development projects, and process solvents are contaminated. On a smaller scale, academic institutions conducting life science and biomedical research invariably produce mixed wastes that face similar treatment and disposal challenges. LBNL believes it is essential to develop processes for ensuring that mixed wastes, especially those similar to the mixed wastes generated by LBNL's National Tritium Labeling Facility, may be treated and disposed in a manner that minimizes impacts on the environment.

We believe we have achieved this goal with the CCO process, which is the subject of the NTLF treatability study. We believe our CCO process meets the federal definition of combustion or, alternatively, has been demonstrated to be an appropriate alternative methodology, as set forth in detail in Attachment A. The CCO methodology achieves a DRE equivalent to that of other combustion technologies specifically identified in 40 CFR Part 268.42, Table 1, and uses engineering controls to avoid emission of tritium. As well, the CCO treatment residues that are the subject of this petition are appropriate candidates for delisting. They contain no detectable levels of the constituents that caused the EPA Administrator to designate the wastes from which the treatment residues are derived as F002, F003, or F005-listed; exhibit no RCRA characteristics; and contain no hazardous constituents above UTSs (40 CFR 268.48) or other 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern. Once approvals of our CCO technology and of our petition to delist the treatment residues are obtained, disposal at a low-level radioactive waste landfill or recycling of tritium will be possible.

The following sections provide additional pertinent information.

<u>No feasible commercial options are currently available.</u>

To our knowledge, no feasible commercial option that allows for desirable environmental or fiscal consequences is currently available for the treatment, disposal, or recycling of LBNL tritiated mixed waste containing moderate to high levels of tritium (e.g. > 1 curie). The only possible commercial option for treatment of such tritiated mixed waste, incineration, would release tritium to the environment without engineering controls designed to minimize tritium air emissions; it is also extremely expensive. Although a number of technical solutions other than incineration exist for the destruction of the hazardous component of tritiated mixed waste, to our knowledge none of them provides a final disposal solution, since further regulatory action (such as requested by this petition) is required before disposal. Furthermore, there will be little incentive for commercial vendors to invest in and make available alternative technical solutions to managing mixed wastes such as LBNL's until the full path from treatment to final disposal has been demonstrated.

It is important to note that, from a regulatory standpoint, disposal of the ash resulting from incineration would be easier than disposal of the treatment residue from catalytic oxidation. The incinerator ash would not contain the moderate to high tritium activity in the CCO treatment residue, since during incineration the tritium in the waste would have been emitted to the atmosphere. Accordingly, disposal in a mixed waste landfill would be possible, obviating the need for a delisting and approval of our CCO system as an acceptable combustion methodology or alternative. In LBNL’s view, however, while the incineration option may be simpler from a regulatory standpoint, it is not a preferred environmental approach, specifically because the tritium would be released to the environment without engineering controls during the incineration process.

Incineration at a DOE facility is not a desirable solution.

DOE has built various advanced incineration devices to address mixed waste (e.g., the Waste Experimental Reduction Facility at the Idaho National Engineering and Environmental Laboratory). However, the radioactive isotope in tritiated mixed waste would be released to the environment with the incineration products (as tritiated water) without engineering controls, rather than retained in the ash. Therefore, incineration at a DOE facility is not a preferred approach.

Disposal of NTLF tritiated mixed waste oxidized through catalytic oxidation is not possible without a delisting.

Based on the lack of feasible commercial or DOE treatment and disposal options, LBNL conducted a treatability study on use of catalytic oxidation to meet land disposal requirements. That study has shown catalytic oxidation to be successful in solving the problem of eliminating or reducing hazardous organic constituents through use of a technology other than incineration. However, disposal or recycling of NTLF tritiated mixed waste treatment residues will not be possible without a delisting.

No regulatory relief is forthcoming.

Although the proposed Hazardous Waste Identification Rule (HWIR) and several other initiatives, such as the EPA proposed Mixed Waste Storage and Disposal Rule and the EPA proposed Low Activity Mixed Waste Disposal Rule or the Advanced Notice of Proposed Rulemaking (ANPR) on storing mixed low-level radioactive waste, offer hope for resolution of some aspects of this situation, final adoption of those proposed initiatives and implementation in states such as California is uncertain.

Site Treatment Plan commitments by the Department of Energy

As a step toward providing leadership, Congress enacted the Federal Facility Compliance Act of 1992 (FFCAct). The FFCAct in part required DOE facilities to develop Site Treatment Plans (STPs) that commit to schedules for identifying unique solutions to the problem of meeting EPA land disposal requirements for mixed waste. The LBNL tritiated mixed waste stream LB-W125 (inventory waste) discussed in this petition is covered by the LBNL Site Treatment Plan, which is the subject of a Consent Order between DOE and the State of California.

National leadership

The NTLF treatability study and this petition for a delisting are important initiatives designed to help resolve a national mixed waste problem faced by the DOE, other research organizations, and the pharmaceutical industry. With the treatability study and this petition, LBNL is

(1) developing an alternative to incineration,
(2) addressing related regulatory issues, and
(3) identifying an appropriate disposal or recycling route for the treated waste.

As noted, the treatment residues currently are regulated as hazardous waste based on the “derived-from” rule, even though they meet land disposal requirements, exhibit no RCRA characteristics, have no detectable amounts of the constituents that caused the EPA Administrator to list the “as-generated” waste from which the residues are derived, and based on process knowledge, contain no 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern. Delisting will allow the treatment residues to be disposed in a low-level waste landfill or sent for tritium recycling.

Other benefits associated with the NTLF treatability study and approval of this petition include the following:

(a) reduction of the amount of mixed waste in storage at LBNL;
(b) encouragement of waste minimization and tritium recovery;
(c) rejuvenation of scientific and manufacturing sectors that have become moribund as a result of unresolved mixed waste problems; and
(d) demonstration that a commercial opportunity exists for treatment of tritiated mixed waste through a nonincineration technology. This will be cost-effective and result in net environmental benefits by reducing tritium emissions.

7. Provide a statement of the need and justification for the proposed action

Existing regulations do not fully consider either (a) the regulatory burden relative to the small chemical scale of tritiated mixed wastes, or (b) the environmental consequences of approved treatment technologies when applied to tritiated mixed wastes.

As a consequence of constrained mixed waste management options, in some instances research institutions have banned the use of tritium in research. Even in institutions where tritium is still used, the regulatory challenges associated with tritiated mixed waste have had a profoundly restrictive effect on biomedical, chemical, agricultural, and pharmaceutical research in the United States. All of these research disciplines use radioactive tracers as fundamental tools for improving knowledge of chemical properties, drug discovery, pharmaceutical transport, and a host of other essential research needs. These constraints exist in spite of the scale of the mixed waste generation, which usually only entails a small number of gallons per year at any one institution.

Some advanced incineration devices could treat NTLF mixed waste. However, the radioactive isotope in tritiated mixed waste would be released to the environment with the incineration products (as tritiated water) without engineering controls, rather than be retained in the ash. We believe that from an environmental perspective, the incineration approach is undesirable, especially in comparison with the CCO approach. The CCO technology includes engineering controls designed to capture tritium and retain it in the waste and treatment residue, so that it can be managed subsequently in a manner that prevents release to the environment.

Catalytic chemical oxidation has been shown to achieve EPA requirements for destruction of regulated organic compounds in a manner that prevents the emissions of tritium to the environment that would result from incineration. Regulatory approval of the CCO technology and delisting of the treatment residues (HTO and HTO on silica gel) will allow subsequent management of the treatment residues through disposal at a low-level radioactive waste landfill or offsite tritium recycling.

This combination of technology application and administrative action will resolve a tritiated mixed waste treatment and disposal problem in a manner that will result in a net benefit to the environment. In addition, demonstration of this complete solution to the tritiated mixed waste problem will encourage growth of commercial capacity for the CCO approach.

8. Signed Certification Statement

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this demonstration and all attached documents, and that, based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

Signed by Authorized Representative,

Typed Name	David McGraw
Title	Division Director Environment, Health and Safety Division E.O. Lawrence Berkeley National Laboratory University of California

SECTION 2. DELISTING WASTE AND WASTE MANAGEMENT INFORMATION

BASIS FOR THE WASTE LISTING

1. The most appropriate descriptions for the petitioned waste

The petitioned wastes are treatment residues derived from the oxidation of F002, F003, and F005 listed mixed wastes.

These treatment residues consist of (1) the oxidized and condensed liquid product (HTO) and (2) oxidation process bubbler water (HTO) stabilized on silica gel derived from catalytic oxidation of organic solvent mixed waste mixture in LBNL's CCO system.

Before oxidation, each "as-generated" mixed waste mixture was or will be high-TOC D001 ignitable; contained or will contain water and one or more regulated F002, F003, F005 constituents; and at times was or will be D-coded for organics. Examples of hazardous constituents, as identified in 52 samples prior to oxidation, are provided in the following table (Table 2-1).

Table 2-1. Chemical constituents identified in multiple mixed waste samples prior to oxidation

EPA Hazardous Waste Code	Hazardous Waste Description
F002	methylene chloride
F003	acetone, ethyl acetate, methanol
F005	benzene, pyridine, toluene
D001	high-TOC (>10%) non-wastewater mixture that might contain one or more of the following chemicals: acetic acid, acetic anhydride, acetone, acetonitrile, benzene, bromonitromethane, chloroform, cyclohexylamine, dimethylformamide, dioxane, ethanol, ethyl acetate, hexane, isopropanol, methanol, methyl acetate, methylene chloride, pyridine, tetrahydrofuran, tetramethylethylene diamine, toluene, triethylamine
D022	chloroform

During the treatability study to date, some of these chemicals were identified infrequently in the mixed waste samples (see Item 5a in Section 5). Over the period of generation, the following chemicals have also been used for labeling experiments: butane, cyanogen bromide, cyclohexene, dibromomethane, dimethylacetamide, dimethylsulfoxide, heptafluorobutyric anhydride, methylmorpholine, pentane, propanolamine, quinoline, thionyl chloride, triethylborane, and trifluoroacetic acid. These chemicals are used rarely and in low quantity in the labeling experiments and they were not identified in the waste samples used in the treatability study (see Table 5-1 in Section 5).

Following the oxidation process, the petitioned treatment residues generated from the CCO system contain no detectable amounts of regulated F-listed constituents, exhibit no RCRA characteristics, and meet LDR concentration levels for underlying hazardous constituents. In addition, based on process knowledge, the treatment residues contain no other 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern. As stated above, these treatment residues consist of the oxidized and condensed product (HTO) and bubbler water (HTO) stabilized on silica gel.

Common name of petitioned waste: N / A

Solid waste(s) common name(s): N / A

Because the NTLF tritiated mixed waste (liquid) was characterized as high-TOC ignitable liquid (RCRA D001) ("high TOC ignitable Characteristic Liquids Subcategory based on CFR 261.21 (a)(1) -- Greater than or equal to 10% total organic carbon"), the applicable treatment standard is "RORGS, POLYM, or CMBST" (40 CFR 268.40). The "CMBST" standard is defined as "high temperature organic destruction technologies, such as combustion in incinerators, boilers, or industrial furnaces operated in accordance with applicable requirements of 40 CFR Part 264, Subpart O, or 40 CFR Part 265, Subpart O, or 40 CFR Part 266, Subpart H, and in other units operated in accordance with applicable technical operating requirements ... " (40 CFR 268.42). Because the CCO is a high-temperature organic destruction technology, we believe it qualifies as a "CMBST" treatment standard technology. We request EPA's concurrence with our assessment. Alternatively, in conjunction with this delisting petition, we request approval of catalytic oxidation as an alternative treatment method in lieu of combustion (See Attachment A).

2. The following describes the physical forms of the petitioned waste.

Liquid: the oxidized and condensed product (tritiated water or HTO) generated from the CCO system.
Solid: bubbler water (HTO) stabilized on silica gel.

3. The petitioned waste is neither sludge nor a mixture of solid and liquid.

HISTORY OF WASTE GENERATION

4. The following describes the generation of the petitioned waste.

There are three sources of the "as-generated" tritium-containing mixed waste from which the petitioned treatment residues are derived. General descriptions of the "as-generated" waste source and the associated petitioned treatment residues are provided below.

1) Inventory tritiated mixed wastes absorbed on silica gel generated between January 1993 and February 1996 (LBNL Mixed Waste Site Treatment Plan ID Number LB-W125).

During the treatability study, the liquid mixed waste was transferred from silica gel, using a solvent vacuum-extraction system, directly to a sample-collection container (flask). The organic solvent mixture was then oxidized in the CCO system. The oxidized liquid product was collected in the condensers and cold trap as tritiated water (HTO). The oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel are the petitioned waste.

2) Liquid tritiated mixed wastes generated from tritium labeling experiments and purification processes after February 1996 to date.

This “as-generated” liquid mixed waste was first transferred from containers used in tritiation reaction experiments and purification processes through a vacuum line directly to a sample-collection container (flask). The organic solvent mixture was then oxidized in the CCO system. The oxidized and condensed liquid product was condensed in the condenser and cold trap as tritiated water (HTO). The oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel are the petitioned waste.

3) Liquid tritiated mixed wastes that will be generated from future tritium labeling experiments and purification processes. In the future, these wastes will be oxidized in the CCO system. The oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel will be the petitioned waste.

VOLUME OF PETITIONED WASTE

5. Is the petition for a waste of fixed quantity?

No. It is a combination of a fixed quantity of LBNL’s inventory wastes (STP LB-W125) and wastes generated since February 1996 and to be generated in the future on a routine basis.

a. Petitioned waste generated prior to date is a fixed quantity.

	Quantity	**Sources**
Estimated volume (HTO)	16 liters (liquid)	oxidized and condensed product
Estimated volume (HTO) (Estimated volume—solid)	20 liters (liquid) (100 gal solid)	bubbler water (stabilized on silica gel)

Describe the method of volume estimation: Direct measurement

In order to meet the acceptance criteria of offsite disposal facilities, the oxidized and condensed liquid product might be repackaged in the future for disposal as low-level radioactive waste. An example of this repackaging would be stabilization by absorbtion on absorbents.

b. Petitioned wastes to be generated on a routine or continuous basis.

	Average Liters/yr	**Maximum Liters/yr**
Oxidized and condensed product	3	10
Bubbler water (stabilized on silica gel)	3 (15 gal solid)	10 (50 gal solid)

Describe the method of volume estimation: Process knowledge

In order to meet the acceptance criteria of offsite disposal facilities, the oxidized and condensed liquid product might be repackaged in the future for disposal as low-level radioactive waste. An example of this repackaging would be stabilization by absorbtion on absorbents.

HISTORY OF WASTE MANAGEMENT

6. As appropriate, describe the present, past, and proposed waste management methods for the petitioned waste.

a. Present waste management methods, and offsite facility or facilities used (name, address, and waste management method):

The petitioned treatment residues (the oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel) generated from the CCO system are currently stored at the permitted Hazardous Waste Handling Facility (HWHF) at LBNL pending shipment off-site for disposal as low-level radioactive waste (or for recycling of tritium) following receipt of EPA and authorized state delisting and alternative treatment methodology approvals.

The “as-generated” tritiated liquid mixed wastes from which the treatment residues are derived are accumulated at the mixed waste satellite accumulation areas (MW-SAAs) at the NTLF for up to a year. They are then either transferred to the HWHF for storage or used as treatability study samples. A treatability study using the CCO systems is being conducted at the NTLF. Treatability study samples have been obtained from both the HWHF (inventory wastes) and the MW-SAA (as-generated wastes) at the NTLF.

b. Past waste management methods, if different from present, and offsite facility or facilities used (name, address, and waste management method):

Same as above.

c. Proposed waste management methods if Delisting Petition is granted, and offsite facility or facilities to be used (name, address, and waste management method):

Once our requests for a delisting and for approval of CCO technology are granted, the petitioned waste will either be shipped as low-level radioactive waste to a DOE radioactive waste disposal site (such as the Hanford site at U.S. Department of Energy, 825 Jadwin Avenue, P.O. Box 550, Richland, WA

99352), to other DOE disposal facilities, or to a commercial radioactive waste disposal site, or shipped to a recycling facility for tritium recycling.

Other treatment residues (such as silica gel remaining following separation of tritiated solvent mixture through vacuum extraction prior to catalytic oxidation) and debris (such as broken glass bottles and rubber bungs) will also be stored at the HWHF and subject to future technical and regulatory analysis.

SECTION 3: PROCESS AND WASTE MANAGEMENT INFORMATION

GENERAL OPERATIONS AT THE GENERATING FACILITY

1. Descriptions of the NTLF business areas and operations

The petitioned waste streams described in this Delisting Petition are generated by the National Tritium Labeling Facility (NTLF) at Lawrence Berkeley National Laboratory (LBNL). LBNL is a noncommercial research organization with an SIC code of 8733.

The NTLF is a designated Department of Energy (DOE) and National Institutes of Health (NIH) National User Facility engaged in tritium labeling research and development. It offers the United States and international biomedical research community a fully equipped laboratory for the synthesis and analysis of tritium-labeled compounds. Located at LBNL, the NTLF was formally established in 1982. Its scientific function is funded by NIH through the Biomedical Technology Area (BTA) of the National Center for Research Resources (NCRR). The NTLF is managed by the University of California (UC) at LBNL, under contract with the Department of Energy.

2. Descriptions of processes conducted at the NTLF

The role of the NTLF is to conduct research and to supply educational and tritium labeling support for biomedical researchers in the United States and other countries. It provides the environment and professional assistance for biomedical researchers and pharmaceutical companies to produce very high-specific-activity tritiated compounds with high radiochemical purity. Tritiation reactions are based on standard hydrogen isotope exchange and synthetic labeling techniques. In general, synthetic approaches are preferred because higher specific activity and specifically labeled products are obtained. The most often used synthetic processes are hydrogenation, tritiodehalogenation, methylation, reduction by hydrides, hydrolysis of Grignard or lithiated compounds, and reactions with T_2O and other reagents.

In addition to the development of labeling methodologies, the staff of the NTLF are involved in many diverse collaborative research projects, including the preparation of high-specific-activity metal-tritide reagents, tritium labeling of carbohydrates and nucleosides, general synthesis of tritiated alkenes via the Shapiro reaction, tritiodesilylation reactions, and synthesis of high-specific-activity N-(tritioacetoxy)-succinimide.

The NTLF occupies a small portion (four laboratories, occupying 1600 sq. ft.) of a multipurpose building (Building 75) at LBNL. The facility has a clear perimeter, and access is well-controlled. Two adjacent trailers provide office and storage space. The NTLF has a permanent staff of four full-time employees, and hosts as many as 60 external visitors to LBNL each year.

3. Description of the radioactive and mixed wastes generated from NTLF

The description of low-level radioactive and mixed wastes generated from tritium labeling experiments and purification processes is presented in Attachment C.

4. Description of the NTLF's processes, waste treatment areas, and waste management units

The physical plant of the NTLF (vacuum lines, gloved containment boxes, etc.) is designed to provide a high level of protection for workers, the public, and the environment, while maintaining the adaptability and flexibility of research equipment. In Attachment C, Figures C-1 and C-2 are the waste generation process diagrams, Figure C-3 shows the floor plan of the NTLF, and Figures C-4 to C-7 are photos of the laboratory of the NTLF.

Building 75, Room 103 contains the tritium storage (15,000 Ci maximum), desorption, and re-adsorption equipment. Components are connected via nuclear-grade stainless-steel tubing and valves in a closed system. The system is designed to operate under a vacuum at all times. Tritium desorption processes are conducted as needed for specific experiments by manual placement of a special heater around the primary uranium storage bed, which is a stainless steel capsule containing depleted uranium.

Approximately 100 tritium desorption operations are conducted per year, typically no more frequently than once per day. Significant quantities of tritium (up to 200 Ci) are present in the system in gaseous form for approximately 20 minutes during a desorption operation. Between operations, the tritium is chemically bound within the primary or capture reaction vessels (uranium beds). Each of these beds is a stainless steel cylinder 5.6 cm in length by 3.8 cm in diameter (slightly larger than a 35-mm film canister).

Building 75, Room 106 is used for scintillation counting and nuclear magnetic resonance (NMR) analyses. It contains less than 5 Ci of tritium at any time, and negligible quantities (in milliliter or mL per year) of toxic or flammable chemicals.

Rooms 102 and 107 are chemical laboratories used for chemical synthesis, general research, and labeling. Typically, Room 102 does not contain radioisotopes. During most procedures, Room 107 contains less than 5 Ci of tritium, and under no circumstances would it contain more than 25 Ci of tritium. Rooms 102 and 107 contain a wide variety of toxic and flammable chemicals in quantities representative of a research laboratory (typically less than 500 mL or 500 g of any substance, with the exception of certain common flammable solvents).

The treatment process used in the NTLF treatability study consists of the following systems: (1) the silica gel solvent vacuum extraction system (see Figure 1) for the inventory mixed waste samples, and (2) two catalytic chemical oxidation (CCO) systems. The CCO-1 system (see Figure 2; for simulated nonradioactive surrogate tests) is located in Room 102, and the solvent vacuum extraction system and the CCO-2 system (see Figure 3; for mixed waste tests) are located in Room 103. Attachment B is a detailed description of these two CCO systems.

The tritiated mixed wastes, as generated, are accumulated at the mixed waste satellite accumulation areas (MW-SAAs) at the NTLF (in Room 103) for up to one year. The low-level radioactive wastes, as generated, are accumulated in both Rooms 103 and 107 (radioactive material areas) at the NTLF.

Figure 1

Figure 2

Figure 3

5. Descriptions of the regulatory status of all onsite waste treatment, storage, and disposal units

In 1996, LBNL initiated tritiated mixed waste treatability study activities. A letter and three annual treatability study reports (for years 1996, 1997, and 1998) have been submitted to the California Department of Toxic Substances Control since June 1996 (see Attachment D). The study was designed to evaluate the effectiveness and efficiency of treating tritiated mixed waste using Catalytic Chemical Oxidation (CCO) technology. Treatability study samples and NTLF activities and equipment devoted to and in support of the NTLF treatability study are exempt from hazardous waste regulation, subject to the conditions specified at Title 22 California Code of Regulations (CCR), Section 66261.4 (e) and (f).

After oxidation of waste samples at the NTLF, the treatment residues (i.e., the oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel) are stored at the permitted Hazardous Waste Handling Facility (HWHF) at LBNL pending shipment off site for disposal as low-level radioactive waste (or for tritium recycling) following approval of LBNL delisting and alternative treatment methodology requests.

As noted above, the “as-generated” tritiated liquid mixed wastes from which the petitioned treatment residues are derived are accumulated at the mixed waste satellite accumulation area (MW-SAA) in accordance with Title 22 CCR Section 66262.34(e). They are then either transferred to the HWHF for storage or used as oxidation samples for treatability studies. Other treatability study treatment residues (such as silica gel remaining following separation of tritiated solvent mixture through vacuum extraction prior to catalytic oxidation) and debris (such as broken glass bottles and rubber bungs) are also stored at the HWHF and subject to future technical and regulatory analysis.

The HWHF has a hazardous waste facility permit (EPA ID number CA4890008986) issued by the California Environmental Protection Agency’s Department of Toxic Substances Control pursuant to California Health and Safety Code Section 25200. Attachment E includes a table listing all LBNL permits, including hazardous waste and other permits issued under federal, state, and local environmental statutes, and the list of hazardous waste streams stored at the HWHF.

CONTRIBUTING RESEARCH PROCESSES

6–9. Descriptions and schematics of all the contributing research processes at NTLF

Attachment C summarizes the tritiation reaction protocol at the NTLF, integral processes, and associated waste streams (also see Item 2 of this section). As described in Attachment C, the waste generating processes, except the chromatography process contributing to the mixed waste, are performed in a set of gloved containment boxes in Building 75, Room 103. The various pieces of apparatus involved in the processes are hard-plumbed to facilitate efficient and safe transfer of material and to prevent release of tritium. (See photos and diagrams in Attachment C.)

The mixed waste samples oxidized or to be oxidized by the CCO-2 system were or will be generated from NTLF’s tritiation reaction experiments and purification processes. The petitioned waste was or will be generated from the CCO-2 system used in the treatability study.

10. Descriptions of operating cycles for all contributing processes at NTLF

Operations related to tritium labeling, including tritiation reactions, product purification [such as high-pressure liquid chromatography (HPLC)], and filtration steps, are executed routinely at the NTLF (also see Attachment C).

11–12. Assessment of extent of past and future process variability and discussion of potential for waste variability due to research process variability

Each tritiation reaction uses different organic compounds. Therefore, the tritiated mixed wastes generated from many tritium labeling experiments might have different chemical compositions. However, the waste variabilities of purification and filtration processes are very low because solvent constituents used for these processes are consistent (see Attachment C).

The following is a list of chemicals used in the tritium labeling and purification experiments and identified in 52 mixed waste samples oxidized in the treatability study (also see description of Item 5a in Section 5):

acetic acid, acetic anhydride, acetone, acetonitrile, benzene, bromonitromethane, chloroform, cyclohexylamine, dichloromethane (methylene chloride), dimethylformamide, dioxane, ethanol, ethyl acetate, hexane, isopropanol, methanol, methyl acetate, pyridine, tetrahydrofuran, tetramethylethylenediamine, toluene, triethylamine, and water.

Because the variability of LBNL's oxidation process (CCO system) is very low as described in Attachment B, the compositions of the petitioned waste (i.e., the oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel) are consistent (see Tables 5-1 and 5-4).

13. Does a waste-treatment process contribute to the petitioned waste?

Yes. The petitioned waste is generated from the catalytic oxidation of mixed waste using the CCO-2 system.

CONTRIBUTING WASTE TREATMENT PROCESSES

14–18. Descriptions and schematics of all the contributing treatment processes

The mixed waste oxidation process used in the NTLF treatability study consists of two systems: (1) the silica gel solvent vacuum extraction system, and (2) the catalytic chemical oxidation system.

(1) The silica gel solvent vacuum extraction system is designed for inventory mixed waste samples (STP LB-W125, consisting of spent solvent mixture absorbed onto silica gel). The silica gel solvent vacuum extraction system (see Figure 1) is a container equipped with a heating device and connected to a vacuum system. As shown in Figure 1, a glass bottle containing the inventory waste is located and sealed in the container. The bottle is crushed while under vacuum by gradually screwing down the bellows. The diamond tip on the valve breaks the glass bottle.

A vacuum line is used to extract the solvent mixture out of the silica gel. Under high vacuum and approximately 200°C heating conditions, the volatile solvent mixture is completely evaporated and transferred to a sample collection container (a flask) immersed in liquid nitrogen. This process usually takes 2 to 8 hours to complete. The residues of this vacuum extraction process are silica gel and broken glass bottles, which are not included in the petitioned waste.

(2) The as-generated solvent mixture (i.e., mixture not absorbed on silica gel and transferred through a vacuum line into a sample collection container), or the collected solvent mixture from step (1) is oxidized in the CCO-2 system at the NTLF. During 1996 and early 1997, NTLF and EH&S Division staff focused their efforts on designing and optimizing the operation of the CCO system. The CCO system involves high-temperature organic chemical decomposition in the presence of platinum-coated alumina pellets as catalysts. Two separate CCO systems (CCO-1 and CCO-2) were designed and constructed by the LBNL scientists. The CCO-1 (see Figure 2) and CCO-2 (see Figure 3) systems are designed for treating simulated nonradioactive surrogate mixtures and tritiated mixed waste samples, respectively.

Attachment B describes the CCO system and its oxidation efficiency. In general, each CCO system consists of

- sample pumps (at 1 to 2 mL / min waste flow rate),
- a sample preheater,
- a heated oxygen source (at 4 liter / min flow rate),
- an oxidation cell,
- a packed-bed tubular reactor filled with platinum / alumina pellets, and
- an emission-reduction device consisting of condensers, cold trap, water bubblers, and silica gel.

Liquid samples are pumped through the preheater. The preheater temperature is maintained at near 300 °C. The temperatures of both the oxidation cell and catalytic reactor are controlled near or above 500 °C.

The oxidized and condensed liquid product (tritiated water) is collected using multiple reflux condensers and a dry-ice cold trap in series. The trapping efficiency for tritiated water is greater than 98%. After these water traps, the gas stream goes through multiple water bubblers and silica gel before exiting to the NTLF stack.

The oxidized and condensed liquid product (tritiated water or HTO) was analyzed using a HP 6890 gas chromatograph (GC) equipped with both a flame ionization detector (FID) and a HP5973 mass selective detector (MSD). The efficiency of solvent oxidation was also monitored continuously by measuring carbon monoxide and residual hydrocarbon in the exhaust gas.

19. Descriptions of operating cycles for all contributing processes

In the treatability study conducted to date, 52 mixed waste samples were oxidized using the CCO-2 system. Table 5-1 (in Section 5) lists these 52 oxidation events. On average, LBNL has oxidized two to three mixed waste samples per month. The two CCO systems were always operated in the batch mode.

20. Assessment of extent that all contributing treatment processes, operations, process materials, or generated wastes have varied in the past or may vary in the future.

The variability of the mixed waste oxidation process was, is, and will be very low. (See Attachment B for the CCO system design, operation procedure, and oxidation efficiency.) Examples of the operating records of the CCO system are included in Attachments I and J. The composition of mixed waste samples may differ each time because the solvents used for each tritium labeling experiment and purification process might vary. Table 5-1 lists the composition of 52 mixed waste samples. Table 5-4 (also in Section 5) lists the composition of several representative mixed waste samples.

21. Describe how the composition and generation rate of the petitioned waste may periodically vary due to any aspect of treatment process variability.

The compositions of the treatability study mixed waste samples vary depending on the solvents used for each tritium labeling experiment and purification process (see Attachment C). The variability of LBNL's treatment process (CCO system) is very low, as described in Attachment B. Thus, the compositions of the petitioned waste (i.e., the oxidized and condensed liquid product (HTO) and bubbler water (HTO) stabilized on silica gel) are consistent (see Tables 5-1 and 5-4).

The rate of tritiated mixed waste generation from multiple tritium labeling experiments is estimated at 1 to 2 liters per year. The rate of tritiated mixed waste generation from labeled product purification processes is estimated at up to 80 liters per year.

In the treatability study, the rate of mixed waste sample oxidation in the future should be approximately one sample a month. The rate of petitioned waste generation is estimated at 3 to 10 liters per year.

22. Has the petitioned waste been managed in a land-based unit?

No.

WASTE MANAGEMENT OPERATIONS

23. Descriptions of waste management practices

After the tritiated mixed wastes have been oxidized at the NTLF, the waste treatment residues and debris are accumulated temporarily at the NTLF's MW-SAA and then transferred to LBNL's permitted HWHF pending further regulatory actions or analyses that will allow final offsite disposal to occur. Figure 4 shows the treatability study mixed waste flow path.

As stated earlier, the HWHF has a hazardous waste facility permit issued by the California Environmental Protection Agency's Department of Toxic Substances Control.

Figure 4

24. Descriptions and schematics of the waste units

After undergoing the treatability study at the NTLF, the oxidized and condensed liquid product (HTO), bubbler water (HTO) stabilized on silica gel, solid treatment residues (silica gel remaining following separation of tritiated solvent mixture through vacuum extraction prior to catalytic oxidation), and debris (broken glass bottles and rubber bungs) generated from the CCO system are stored in mixed waste storage units (MW4, MW5, and MW6) in the permitted HWHF at LBNL.

The mixed waste storage units (MW4, MW5, and MW6) are rooms located on the first floor of the HWHF. MW4 and MW5 have interior dimensions of 20 x 11 x 8 feet, approximate floor surface areas of 300 ft^2, maximum storage capacities of 1,320 gallons and 2,200 gallons, respectively, and available secondary containment capacities of 240 gallons each. MW6 has an interior dimension of 40 x 25 x 8, an approximate floor surface area of 1,000 ft^2, a maximum storage capacity of 1,100 gallons, and an available secondary containment capacity of 240 gallons. They all have a concrete floor coated with a chemical-resistant epoxy coating, a foam fire protection system, and a ventilation system.

Attachment F shows a schematic of the HWHF and the mixed waste storage units including MW4, MW5, and MW6.

PROCESS MATERIALS

25. List of all materials used in the operations that contribute to the petitioned waste

Section 2.1 describes (a) constituents identified in 52 tritiated mixed waste samples oxidized in our treatability study conducted to date (see Table 2-1), and (b) other organic chemicals occasionally used in tritium labeling experiments and purification processes and unidentified in these mixed waste samples used in the treatability study.

Attachment B and Figure 3 describe the materials used in the CCO system. Attachment C describes (a) chemicals used in tritiation reaction experiments and purification processes, and (b) radioactive and mixed wastes generated from the tritium labeling experiments and purification processes at the National Tritium Labeling Facility.

26. Material safety data sheets (MSDSs) for all nonelemental and trade name materials used in contributing processes

The MSDSs of 19 organic chemicals used in tritiation reactions and identified in the 52 mixed waste samples, of 2 catalysts (palladium on barium sulfate and palladium oxide) used in tritiation reactions, and of 1 catalyst (platinum on alumina) used in the CCO systems are included in Attachment G.

27. Sources of oil and grease in contributing processes

None.

SPECIAL INFORMATION

Since LBNL is not requesting an upfront exclusion for a waste that is not currently generated but will be in the future, this section is not applicable.

SECTION 4: DELISTING ANALYTICAL PLAN DEVELOPMENT

1. The complete list of the constituents and parameters of concern identified for the petitioned waste based on appropriate waste analyses

The petitioned waste is certain treatment residues generated from the oxidation of one or more listed mixed wastes using the CCO system. These treatment residues, which consist of the oxidized and condensed liquid (HTO) and bubbler water (HTO) stabilized on silica gel, do not contain any detectable levels of the constituents that caused the EPA Administrator to designate the “as-generated” wastes from which they are derived as F002, F003, and F005-listed. In addition, these treatment residues exhibit no RCRA characteristics, and contain no RCRA-regulated constituents above universal treatment standards or any other 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern.

However, prior to oxidation, each mixed waste mixture contained or will contain water and one or more regulated F002, F003, F005 constituents; was or will be D-coded for organics; and was or will be a D001 high-TOC ignitable waste. The following table lists the regulated solvents identified in 52 tritiated mixed waste samples (see Table 5-1) using the in-house GC / MS, and oxidized in the treatability study.

EPA Hazardous Waste Code	Hazardous Waste Description
F002	methylene chloride
F003	acetone, ethyl acetate, methanol
F005	benzene, pyridine, toluene
D001	high-TOC (>10%) non-wastewater mixture that might contain one or more of the following chemicals: acetic acid, acetic anhydride, acetone, acetonitrile, benzene, bromonitromethane, chloroform, cyclohexylamine, dimethylformamide, dioxane, ethanol, ethyl acetate, hexane, isopropanol, methanol, methyl acetate, methylene chloride, pyridine, tetrahydrofuran, tetramethylethylene diamine, toluene, triethylamine
D022	chloroform

Attachment I presents four analyses of oxidized nonradioactive surrogate test samples verified by an independent commercial laboratory (BC Laboratories) and two analyses of oxidized nonradioactive surrogate test samples from the in-house GC / MS; Attachment J presents seven analyses of the oxidized tritiated mixed waste samples from the in-house GC / MS. The analytical results of GC / MS analyses demonstrate that all of the listed chemicals in the table have been decomposed with a DRE >> 99.999%.

2-3. Not relevant.

4. **Explanation of why the petitioned waste does not exhibit any hazardous waste characteristic for which analysis was not conducted.**

The petitioned treatment residues exhibit no RCRA hazardous characteristics of ignitability, corrosivity, reactivity, and toxicity.

Ignitability. The analysis of ignitable characteristic was not conducted for the petitioned waste (i.e., treatment residues). NTLF's in-house GC / MS analytical results indicate no F003 or D001 constituents remain in the oxidized and condensed liquid product or bubbler water collected from the CCO systems.

Reactivity. The analysis of reactivity was not conducted for the petitioned waste (i.e., treatment residues) because both the in-house GC / MS analysis (for the oxidized and condensed tritiated sample) and the commercial laboratory analysis (for the oxidized nonradioactive surrogate sample) and process knowledge indicate that the treatment residues are, themselves, essentially tritiated water (HTO) or pure water. As such, the treatment residues would not exhibit any characteristic of reactivity described at 40 CFR 261.23. For example, the treatment residues are stable and will not react with water, will not form potentially explosive mixtures with water, or generate toxic gases, vapors, or fumes when mixed with water.

The petitioned waste is not a cyanide- or sulfide-bearing waste, based on process knowledge that (a) no cyanide or sulfide compounds are used in tritiation reaction experiments and purification processes, and (b) the solvent mixtures in mixed waste samples were completely decomposed (DRE > 99.999%) through the CCO system and converted to tritiated water (or water), carbon dioxide, or trace amounts of nitric acid or HCl, depending on the composition of mixed waste (or simulated nonradioactive surrogate) sample used in the experiments.

TCLP. TCLP analysis of regulated inorganic and organic chemicals was not conducted because the oxidized and condensed liquid product and bubbler water (HTO) were 100% liquid and contained no solids (i.e., << 0.5% dry solids). For purposes of the TCLP test, the residue is, itself, considered the extract, and direct analysis is appropriate for comparison to TCLP limits.

Toxicity. SW-846 analysis of toxicity characteristic was not conducted for the petitioned waste because

(1) in-house GC / MS analytical results demonstrate nondetectable levels of all organic chemicals that were identified in the 52 mixed waste samples treated through LBNL's CCO-2 system;

(2) based on process knowledge, there are no other 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX organic or inorganic constituents, other than barium, used in the tritiation labeling / purification experiments and catalytic chemical oxidation processes, and thus they cannot appear in the petitioned waste; and

(3) although one of catalysts, palladium (10%) on barium sulfate, was used in tritiation reactions, it could not be carried into the mixed waste samples and processed through the CCO system because (a) the catalysts used in each tritiation reaction are separated from the labeled products and

solvents by filtration, and (b) none of the catalysts, including palladium on barium sulfate, are volatile chemicals and thus they cannot be vaporized through the CCO system and appear in the petitioned waste.

To demonstrate that our in-house GC/MS approach produces analytical results that are consistent with those from SW-846 analysis, LBNL staff sent oxidized and condensed liquid product samples from simulated surrogate oxidation tests using the CCO-1 system to BC Laboratories (see Table 5-2 and Attachment I). The analytical results from BC laboratories (SW-846 methods of modified 8015, 8260, and/or 8270) were consistent with those from the in-house GC/MS approach.

It should also be noted that, to our knowledge, commercial environmental analytical laboratories have radioactive material license limits that would restrict the amount of petitioned waste that could be sent for analysis to such a degree that the dilution required to conduct the analysis would render the results unreliable. To our knowledge, there are currently no commercial environmental laboratories available to LBNL that could accept larger sample sizes with relatively high radioactivity.

We also believe that even if such an analytical laboratory were available, analysis of petitioned waste samples would violate the As Low As Reasonably Achievable (ALARA) principle that applies to radioactive materials handling especially considering the reliable information we already possess on the waste. Decontamination of such a laboratory would be required following analysis. In addition, the level of radioactivity could require the workers conducting the analysis to undergo bioassay to assure there was no personnel exposure.

Corrosivity. At the end of the treatability study for a tritiated mixed waste sample, the oxidized and condensed liquid product (HTO) was collected inside a glass bottle. Many tritiated mixed waste samples contained acetonitrile, and some contained chloroform (see Table 5-1 in Section 5), so the oxidized and condensed liquid product was generally acidic (pH normally in the range of 2 to 3). LBNL staff measured the pH of each batch of liquid product and then neutralized it (pH between 5 and 9) when necessary.

Bubbler Water Analysis. On the basis of the analysis of the oxidized and condensed liquid product (HTO) collected from the condenser and cold trap of the CCO-2 system, LBNL believes that the bubbler water (HTO) does not contain any detectable F-listed and D-coded chemical constituents. Therefore, sampling of bubbler water has not been conducted for the CCO-2 system.

To support this conclusion, bubbler water samples collected from the CCO-1 system for two simulated nonradioactive surrogate oxidation tests were analyzed using in-house GC/MS. The analytical results showed that the bubbler water samples contained no detectable levels of chemical constituents originally contained in these two simulated surrogate samples (see Table 5-3 in Section 5) before oxidation or any 40 CFR Part 261 Appendix VIII or Part 264 Appendix IX organic constituents. The analytical results of these two samples are included in Attachment I.

SECTION 5: DELISTING SAMPLING AND ANALYSIS INFORMATION

1. LBNL discussed its sampling and analysis plan with EPA Region IX at a number of meetings prior to petition preparation. LBNL also submitted a draft data package to EPA Region IX for review in December 1998. The package included LBNL's in-house GC/MS analytical results of three oxidized and condensed liquid products (HTO) generated from tritiated mixed waste oxidation tests and a laboratory report (prepared by BC Laboratories) of the oxidized and condensed liquid product (water) generated from a simulated nonradioactive surrogate oxidation. Comments and suggestions from EPA Region IX have been incorporated.

WASTE SAMPLING INFORMATION

2. All sampling-related activities were performed by LBNL staff.

   Dr. Chit Than, the NTLF chemist, conducts all of the in-house sampling and analysis for the oxidized and condensed liquid products. Dr. Philip Williams, the co-principal investigator of NTLF and a chemist, supervises the entire treatability study and reviewed the data package for each batch of oxidized and condensed liquid product. Dr. Li-Yang Chang, a Waste Management engineer in the EH&S Division of LBNL, serves as technical support staff and an independent reviewer of all of data packages. Resumes of all individuals who participated and/or designed the sampling plan and sample collection and analysis are in Attachment H.

SAMPLING STRATEGY

The objectives of LBNL's sampling strategy are to

(1) characterize each tritiated mixed waste sample prior to oxidation,

(2) characterize each batch of oxidized and condensed liquid product (mixed waste treatment residue) collected from the CCO-2 system following the oxidation of mixed waste samples,

(3) assure the oxidation performance of the CCO-2 system by oxidation of simulated nonradioactive surrogate samples in the CCO-1 system, as appropriate, and as a part of LBNL's quality control (QC) procedure,

(4) as an additional QC step, validate the results identified in (3) by sending the oxidized and condensed liquid product samples (surrogate treatment residue) collected from the CCO-1 system to an independent commercial laboratory, and

(5) characterize bubbler water samples after oxidation of simulated nonradioactive surrogate samples in the CCO-1 system, as appropriate, and as a part of LBNL's quality control (QC) procedure.

The two CCO systems used in LBNL's treatability study are always operated in the batch mode. Each batch of simulated nonradioactive surrogate or mixed waste sample is sampled and analyzed before and after oxidation. Thus, LBNL staff sampled 100% of the waste samples and their oxidized and condensed liquid products.

Before the oxidation of each tritiated mixed waste or simulated surrogate sample, LBNL staff thoroughly mixed the sample collected in the sample container. Whenever necessary, a mixed waste sample is also homogenized in the sample container by adding suitable amount of isopropanol. Therefore, each tritiated mixed waste sample was essentially uniform and homogeneous prior to characterization and oxidation.

The oxidized and condensed liquid products generated from the CCO systems were either water (from the CCO-1 system) or tritiated water (from the CCO-2 system). Before each sample was taken, LBNL staff thoroughly mixed the oxidized and condensed liquid product collected in the glass container; therefore, the liquid was essentially uniform and homogeneous.

3a.-d. Description of the sampling strategy LBNL followed to ensure that the samples were representative.

LBNL's sampling is performed in conjunction with its treatability study. Described below are the treatability study procedures and the sampling strategy.

(A) Treatability Study Procedure. LBNL's treatability study procedure includes the following steps:

(1) Transfer of the waste to the sample-collection container of the CCO system.

(2) Characterization of the waste sample and preparation of simulated nonradioactive surrogates.

(3) Oxidation of the simulated nonradioactive surrogate or the tritiated mixed waste.

(4) Characterization of the oxidized product (water or tritiated water).

These four steps involve many substeps, in particular:

(1) Transfer of the tritiated mixed waste sample (organic solvents and water), using the solvent vacuum extraction system for the inventory waste sample (STP LB-W125 waste: solvents absorbed on silica gel) or using a vacuum line for the as-generated liquid mixed waste, from a waste storage bottle or container to the sample collection container.

(2) Characterization of the mixed waste sample on the basis of process knowledge, using GC/MS for organic components and a liquid scintillation counter for radioactivity.

(3) As part of QC procedures, preparation of simulated nonradioactive surrogates based on the results of step (2), and optimization of the operating conditions by oxidation of nonradioactive surrogates using the CCO-1 system.

(4) GC/MS analysis of the condensed liquid product of oxidation of simulated nonradioactive surrogate sample, and, as a part of QC procedures, sending the liquid product to an independent commercial environmental laboratory for analysis verification. (Four sets of analytical results are presented in Attachment I.)

(5) Inspection of the CCO system during oxidization of nonradioactive pure isopropanol; analysis of the condensed liquid product of isopropanol oxidation by in-house GC/MS as a part of QC procedures.

(6) Oxidation of the tritiated mixed waste sample using the CCO-2 system with the optimized operating conditions obtained from step (3) or from previous oxidation experiments, and analysis of the oxidized and condensed liquid product with GC/MS. (Seven sets of analytical results are presented in the Attachment J.)

(7) Counting the radioactivity in the oxidized and condensed liquid product with a liquid scintillation counter.

(8) Cleaning the CCO-2 system by oxidizing pure and nonradioactive isopropanol in the system.

(9) Counting the radioactivity of each batch of condensed liquid product generated from the oxidation of isopropanol and of the bubbler water touched by the effluent gas (tritium trap).

(10) For emission-abatement purposes, capturing the released tritiated water from the CCO-2 system located in a glove box with silica gel located at the glove box exit. The silica gel is then used to absorb the bubbler water used in each oxidation experiment.

At the end of the treatability study for a tritiated mixed waste sample, the oxidized and condensed liquid product (HTO) is collected inside a glass bottle and neutralized if it is acidic. Because most tritiated mixed waste samples contain acetonitrile and some of them might also contain chloroform (see Table 5-1), the oxidized and condensed liquid products could be acidic (pH is normally in the range of 2 to 3). LBNL staff measure the pH of each batch of liquid product and then neutralize it (pH is normally between 5 and 9) when necessary. The glass bottle is then placed in a can, filled with absorbents, sealed, and returned to the HWHF. The oxidized and condensed liquid product awaits future disposal (or tritium recovery).

The bubbler water (see Figure 3) is absorbed onto silica gel, which was also used in the treatability study (see step (10) above), stored in a can, sealed, and shipped to the HWHF for storage.

(B) Sampling Procedures. The following describes the sampling locations, techniques, and devices for the CCO-1 and CCO-2 systems.

(i) Mixed waste oxidation sampling and analysis. The mixed waste oxidation process consists of two systems: the solvent vacuum extraction system and the CCO-2 system. These two systems are housed in two adjacent glove boxes in Building 75, Room 103. There are two sample collection points in the glove boxes: one for the solvent vacuum extraction system (see Figure 1), and another for the CCO-2 system (see Figure 3).

Before oxidation, the waste sample is mixed thoroughly, and a representative waste sample is collected from the sample collection container (see Figure 1) using a clean syringe or pipette. The sample is then injected into a small glass vial or container. Because the mixed waste sample normally contains a high organic solvent content, LBNL staff dilute the sample with de-ionized or tap

Table 5-1. Sampling and Catalytic Oxidation Record of the Petitioned Waste

	Sample ID	Date Sample Taken	Original Waste Composition	Inventory or New Waste	Oxidized Liq. Sample Composition	Est. Vol. (mL)	Physical Description
1	R016225	13 Mar 97	ACN, EtOAc, EtOH, IPA, MeOH, THF, water	New	HTO	250	Clear water & one phase
2	R016243.01 R016243.02 R016243.03	12 May 97 20 May 97 21 May 97	ACN, DMF, EtOAc, EtOH, IPA, MeOH, THF, water	New New New	HTO	90 180 220	Clear water & one phase
3	R016318.01 R016318.02	24 Jul 97 25 Jul 97	ACN, DMF, EtOAc, EtOH, IPA, MeOH, THF, TMEDA, toluene, water	New New	HTO	180 120	Clear water & one phase
4	R016323	13 Aug 97	ACN, chloroform, EtOH, IPA, MeOH, THF, toluene, water	R016202 R016206 (inventory)	HTO	170	Clear water & one phase
5	R016337	03 Oct 97	ACN, DMF, dioxane, EtOAc, EtOH, IPA, MeOH, THF, water	New	HTO	550	Clear water & one phase
6	R018926	15 Dec 97	acetic acid, ACN benzene, IPA, MeOH, THF, water triethylamine	New	HTO	400	Clear water & one phase
7	R018930	29 Jan 98	ACN, DMF, EtOAc, EtOH, IPA, MeOH, THF, water TMEDA	R014473.06 R014473.07 (inventory) + new	HTO	300	Clear water & one phase
8	R018934	11 Feb 98	ACN, DMF, dioxane, EtOAc, EtOH, hexane, IPA, MeOH, THF, water	New	HTO	320	Clear water & one phase
9	R018937	27 Feb 98	ACN, DMF, EtOAc, EtOH, IPA, MeOH, THF, water TMEDA	R014473.03 (inventory)	HTO	210	Clear water & one phase

Table 5-1. (continued)

	Sample ID	**Date Sample Taken**	**Original Waste Composition**	**Inventory or New Waste**	**Oxidized Liq. Sample Composition**	Est. Vol. (mL)	**Physical Description**
10	R018938	04 Mar 98	ACN, benzene, chloroform, DMF, EtOH, IPA, MeOH, THF, water	New	HTO	200	Clear water & one phase
11	R018941	09 Mar 98	EtOAc, EtOH, IPA, MeOH, THF	New	HTO	150	Clear water & one phase
12	R018931	16 Mar 98	ACN, IPA, MeOH, cyclohexylamine, THF, water	New	HTO	255	Clear water & one phase
13	R018373	27 Mar 98	ACN, benzene, EtOAc, EtOH, IPA, methylene chloride, methyl acetate, MeOH,THF, toluene, water	New	HTO	250	Clear water & one phase
14	R018377	30 Mar 98	ACN, EtOAC, EtOH, IPA, MeOH, THF, water	New	HTO	260	Clear water & one phase
15	R018382	31 Mar 98	acetone, IPA, MeOH, THF, water	New	HTO	235	Clear water & one phase
16	R018384	02 Apr 98	acetone, benzene, IPA, MeOH, THF, water	New	HTO	310	Clear water & one phase
17	R019156	07 Apr 98	ACN,dioxane EtOAC, EtOH, IPA, MeOH, THF, toluene, water	New	HTO	225	Clear water & one phase
18	R019152.01	09 Apr 98	EtOAC, EtOH, IPA, MeOH, water	New	HTO	325	Clear water & one phase
19	R019154	24 Apr 98	ACN, EtOAC, EtOH, IPA, MeOH, THF, water	New	HTO	200	Clear water & one phase

Table 5-1. (continued)

	Sample ID	**Sample Taken Date**	**Original Waste Composition**	**Inventory or New Waste**	**Oxidized Liq. Sample Composition**	Est. Vol. (mL)	**Physical Description**
20	R019163	04 May 98	ACN, dioxane, DMF, EtOAc, EtOH, IPA, MeOH, THF, water	New	HTO	365	Clear water & one phase
21	R019166	26 May 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R018379 R018380 R016322 R016321 R016320 (inventory) + new	HTO	290	Clear water & one phase
22	R019167	28 May 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	New	HTO	270	Clear water & one phase
23	R019168	02 Jun 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R014473.05 R016324 (inventory) + new	HTO	315	Brownish & one phase
24	R019170	09 Jun 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R016208 R016209 (inventory) + new	HTO	350	Clear water & one phase
25	R019171	12 Jun 98	ACN, benzene, EtOH, IPA, MeOH, THF, water toluene	R014473.02 R014473.08 (inventory) + new	HTO	270	Clear water & one phase
26	R019176	16 Jun 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R014473.11 R014473.14 (inventory) + new	HTO	260	Clear water & one phase
27	R019178	24 Jun 98	ACN, chloroform, EtOH, IPA, MeOH, water	R014473.04 R014473.09 + New	HTO	410	Clear water & one phase
28	R019186	25 Jun 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	New	HTO	375	Clear water & one phase

Table 5-1. (continued)

	Sample ID	Date Sample Taken	Original Waste Composition	Inventory or New Waste	Oxidized Liq. Sample Composition	Est. Vol. (mL)	Physical Description
29	R019188	30 Jun 98	ACN, benzene, EtOAC, EtOH, IPA, DMF, MeOH, THF, water	New	HTO	300	Clear water & one phase
30	R019189	02 Jul 98	ACN, benzene, EtOAC, EtOH, methylene chloride, IPA, MeOH, THF, toluene, water	New	HTO	305	Clear water & one phase
31	R019191	14 Jul 98	ACN, EtOH , IPA, MeOH, THF, water	R014473.10 R014473.17 (inventory)	HTO	255	Clear water & one phase
32	R019193	16 Jul 98	ACN, benzene, EtOAC, EtOH, IPA, DMF, MeOH, THF, toluene, water	New	HTO	370	Clear water & one phase
33	R019194	23 Jul 98	ACN, benzene, chloroform, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R014473.15 R014473.24 R014473.25 R016224.07 (inventory) + new	HTO	300	Clear water & one phase
34	R019197	04 Aug 98	ACN, benzene, EtOH, IPA, MeOH, methylacetate, THF, toluene, water	R016224.06 R014473.28 R014473.22 (inventory) + new	HTO	325	Clear water & one phase
35	R018327	06 Aug 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R014473.18 (inventory) + new	HTO	395	Clear water & one phase
36	R018328	11 Aug 98	ACN, benzene, EtOAC, EtOH, IPA, DMF, MeOH, THF, toluene, water	New	HTO	375	Clear water & one phase
37	R018329	13 Aug 98	ACN, benzene, EtOH, IPA, DMF, MeOH, THF, water	R014473.31 R014473.23 (inventory)	HTO	310	Clear water & one phase

Table 5-1. (continued)

	Sample ID	**Date Sample Taken**	**Original Waste Composition**	**Inventory or New Waste**	**Oxidized Liq. Sample Composition**	Est. Vol. (mL)	**Physical Description**
38	R018338	18 Aug 98	ACN, IPA, MeOH, water	R014473.30 R014473.26 (inventory)	HTO	310	Clear water & one phase
39	R018340	20 Aug 98	ACN, benzene, EtOAC, EtOH, IPA, MeOH, THF, toluene, water	R014473.29 R016210 (inventory)	HTO	305	Clear water & one phase
40	R018343	24 Aug 98	ACN, benzene, EtOH, IPA, MeOH, pyridine, THF, toluene, water	R014473.27 R016301 (inventory)	HTO	310	Clear water & one phase
41	R019660	28 Aug 98	ACN, benzene, EtOH, IPA, MeOH, THF, water	R016214 R016215 R016201 (inventory)	HTO	390	Clear water & one phase
42	R019661	03 Sep 98	ACN, EtOAc, EtOH, IPA, MeOH, THF, methyl acetate, water	R016212 R016213 R016207 (inventory)	HTO	340	Clear water & one phase
43	R019662	11 Sep 98	ACN, IPA, MeOH, THF, water, bromonitromethane	R016216 R014473.01 (inventory)	HTO	260	Clear water & one phase
44	R019684	14 Oct 98	ACN, chloroform. EtOAc, EtOH, IPA, methyl acetate, MeOH, THF, water	R016211 R014473.19 (inventory)	HTO	250	Clear water & one phase
45	R019689.01 R019689.02	16 Oct 98 20 Oct 98	acetic anhydride, ACN, benzene, EtOH, IPA, MeOH, THF, toluene, water	New New	HTO	325 250	Clear water & one phase
46	R019688	23 Oct 98	ACN, benzene, EtOH, IPA, MeOH, THF, water	R016211 R016205 R014473.21 R016224.05 (inventory)	HTO	250	Clear water & one phase

Table 5-1. (continued)

	Sample ID	**Date Sample Taken**	**Original Waste Composition**	**Inventory or New Waste**	**Oxidized Liq. Sample Composition**	Est. Vol. (mL)	**Physical Description**
47	R019700	05 Nov 98	dioxane, EtOAc, EtOH, IPA, MeOH, THF, water	New	HTO	230	Clear water & one phase
48	R020062	17 Nov 98	benzene, EtOH, IPA, MeOH, THF water	R016224.04 R016224.02 (inventory)	HTO	230	Clear water & one phase
49	R020069	25 Nov 98	IPA, MeOH, THF, water	R016224.01 R016224.03 (inventory)	HTO	300	Clear water & one phase
50	R020090	22 Dec 98	ACN, benzene, EtOAC, EtOH, IPA, DMF, MeOH, THF, toluene, water	New	HTO	320	Clear water & one phase
51	R020624.01 R020624.02 (1st batch)	10 Mar 99	ACN, benzene, EtOAC, EtOH, IPA, DMF, hexane, MeOH, THF, toluene, water	New	HTO	290 215	Clear water & one phase
52	R020625 (2nd batch)	10 Mar 99	ACN, benzene, EtOAC, EtOH, IPA, hexane, MeOH, THF, toluene, water	New	HTO	140	Clear water & one phase

The abbreviations of several chemicals used in Tables 5-1 and 5-2 are listed below:

ACN = acetonitrile, $CHCl_3$ = chloroform, DMF = dimethylformamide,
EtOAc = ethylacetate, EtOH = ethanol, IPA = isopropanol, MeOH = methanol,
THF = tetrahydrofuran, TMEDA = tetramethylethylenediamine

water before any GC/MS analysis. A clean pipette is always used to take 2 µl of this sample from the small glass vial and transfer it to a small GC/MS autosampler glass vial containing 1 mL of deionized or tap water (i.e., 500x dilution). An in-house GC/MS is used for organic-compound analysis. The liquid sample (tritiated water) is injected directly into the GC/MS (see (iii) below).

After oxidation, the oxidized and condensed liquid product is collected from the condenser and cold trap of the CCO-2 system (see Figure 3) into a glass bottle (250 or 500 mL). LBNL staff then mix the product in the bottle and transfer a sample with a clean syringe or pipette (i.e., a grab sample) to a small GC/MS autosampler glass vial for analysis. LBNL staff then analyze the liquid samples using an in-house GC/MS for organic compounds. The liquid sample (tritiated water) is directly injected into the GC/MS.

Approximately 2 µl of sample are also collected with a clean pipette for radioactivity analysis using an in-house liquid scintillation counter.

On the basis of the analysis of the oxidized and condensed liquid product collected from the condenser and cold trap [and analysis of two bubbler water samples of the CCO-1 system described in (ii)], LBNL believes that the bubbler water (HTO) and silica gel through which the exhaust gas passes after sampling and before being collected contain no detectable F-listed and D-coded chemical constituents. Therefore, sampling of bubbler water on silica gel is not necessary.

(ii) Simulated nonradioactive surrogate oxidation sampling and analysis. LBNL staff also conduct surrogate oxidation tests using the CCO-1 system with various simulated nonradioactive solvent mixtures. The CCO-1 system is located in Building 75, Room 102 (the NTLF nonradioactive laboratory).

In-house GC/MS analysis. Before oxidation, the nonradioactive surrogate mixture is prepared based on the composition of tritiated mixed waste and contained in a clean glass bottle (See Treatability Study Procedure described above). The surrogate mixture is mixed well in the bottle. Then a surrogate sample is withdrawn with a clean syringe or pipette and transferred to a small glass vial. Because the sample normally contains a high organic solvent content, LBNL staff dilute the sample with de-ionized or tap water before GC/MS analysis. They use a clean pipette to take 1– 2 µl of this sample from the small glass vial and transfer it to an autosampler glass vial containing 1 mL of deionized or tap water (i.e., 500x to 1,000x dilution). The liquid sample is injected directly into the GC/MS.

After sample oxidation, the oxidized and condensed liquid product is collected from the cold trap of the CCO-1 system (see Figure 2) into a small GC/MS autosampler glass vial for analysis. LBNL staff then analyze the liquid oxidation product sample using an in-house GC/MS for the organic compounds. The liquid sample (water) is directly injected into the GC/MS (see (iii) below).

Bubbler water samples of the CCO-1 system were also sampled and analyzed using in-house GC/MS, as appropriate, and as a part of quality control (QC) procedures. The analytical results indicate that the chemical constituents in the simulated surrogate samples prior to oxidations were not detected in the bubbler water after oxidations (see Attachment I).

Analysis by an Independent Commercial Laboratory. LBNL staff also send oxidized and condensed nonradioactive surrogate liquid samples to an independent commercial analytical laboratory (BC Laboratories) for result verification as a QC step. Samples (approximately 40 mL each) are collected in clear glass containers provided by BC Laboratories. The glass containers are then stored in a refrigerator pending pickup. BC Laboratories normally picks up these glass containers on the same day or within three days and ship them back to the laboratory in a cooler. BC Laboratories and its subcontractor used EPA SW-846 standard methods 8015 (modified), 8260, and/or 8270 for the requested analyses.

(iii) GC/MS analysis procedure following oxidation. Following oxidation, the in-house GC/MS analysis procedure includes the following steps:

(1) Collect the oxidized and condensed liquid product (water or tritiated water) of each batch of mixed waste or nonradioactive surrogate sample in a glass container from the condenser and cold trap of the CCO system.

(2) Analyze the control blanks, such as tap water, and the condensed liquid product (water or tritiated water) of oxidation of nonradioactive isopropanol per batch using the in-house GC/MS as a QC step.

(3) Analyze the oxidized and condensed liquid product (water or tritiated water) of each batch of mixed waste or nonradioactive surrogate sample using the in-house GC/MS.

(4) Check the GC/MS frequently (every batch) with a commercial 1 ppm standard containing up to 12 or 13 solvents. The composition of the standard is developed on the basis of those most commonly identified constituents in mixed waste samples (see descriptions in 5a of this section).

(5) As appropriate, also conduct GC/MS analyses of spiked oxidized and condensed liquid product generated from the CCO-2 system as an additional QC step (see Attachment J).

3e. Description of the deviations of the original sampling plan and strategy and the impact of these deviations

LBNL's sampling plan and strategy have not been changed during the treatability study conducted to date except to add the spiking of the oxidized and condensed liquid product step described above as a QC step of the GC/MS analysis procedure (step (5) above). This step further assures LBNL staff that the functions of GC/MS are adequate and consistent.

3f. Discussion of the representativeness of samples for the petitioned waste

Each batch of mixed waste sample was thoroughly mixed and homogenized before sampling and oxidation. After oxidation, each oxidized and condensed liquid product (HTO) was collected for in-house GC/MS analysis. The procedure is described in Item 3a-d. Thus, LBNL staff sampled 100% of the waste samples and their oxidized and condensed liquid products.

In addition, the condensed liquid product (water) of each oxidized nonradioactive simulated surrogates was collected from the CCO-1 system and sent to an independent commercial laboratory (BC Laboratories) for analysis (15 samples total). Before each liquid product sample was taken, LBNL staff thoroughly mixed the oxidized and condensed liquid product collected in the glass container; therefore, the liquid product was essentially uniform and homogeneous.

Two bubbler water samples of the CCO-1 system were sampled and analyzed using in-house GC/MS following the procedure described in Item 3a – d. The analytical results indicate that none of chemical constituents originally detected in the simulated surrogate samples prior to oxidation were detected in the bubbler water after oxidation (see Attachment I).

Four sets of BC Laboratories' analytical reports and two sets of in-house GC/MS analytical results for the nonradioactive surrogate samples (Attachment I) and seven sets of in-house GC chromatograms for the tritiated mixed waste samples (Attachment J) are included in this petition.

The 16 chemical constituents identified in the 6 nonradioactive simulated surrogate samples prior to oxidation using the CCO-1 system are

- acetic acid
- acetonitrile
- benzene
- chloroform
- dimethylformamide
- dioxane
- ethanol
- ethyl acetate
- hexane
- isopropanol
- methanol
- pyridine
- tetrahydrofuran
- toluene
- triethylamine
- water

The 16 chemical constituents identified in the 7 liquid mixed waste samples prior to oxidation using the CCO-2 system are

- acetone
- acetic acid
- acetonitrile
- benzene
- chloroform
- dimethylformamide
- dioxane
- ethanol

- ethyl acetate
- isopropanol
- methanol
- pyridine
- tetrahydrofuran
- toluene
- triethylamine
- water

The analytical results from both the in-house GC/MS and BC Laboratories indicate that the organic chemical constituents in the oxidized and condensed liquid products and bubbler water (water or tritiated water) are all in the nondetected range or below UTSs.

LBNL believes that the analytical results for the liquid treatment residues yielded through oxidation of the 13 mixed waste and simulated nonradioactive surrogate samples included in Attachments I and J are representative of all liquid oxidation products generated and to be generated through LBNL's catalytic oxidation system. This belief is based on a review of the analytical results for the 52 mixed waste and 17 surrogate samples oxidized at LBNL to date and their oxidized and condensed liquid products. The 13 mixed waste and surrogate samples included in Attachments I and J contained 17 of the 23 total chemical constituents detected in the 52 total mixed waste samples that were oxidized. All of the samples that have been oxidized at LBNL to date contained, following oxidation, no detectable levels of the F002, F003, and F005 constituents for which they were listed, exhibited no hazardous characteristics, met UTSs, and, through process knowledge, contained no other Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern.

On the basis of the analyses of the oxidized and condensed liquid product collected from the condenser and cold trap of the CCO-2 system and the in-house GC/MS analytical results of two bubbler water samples of the CCO-1 system (see Attachment I), LBNL also believes that the bubbler water (HTO) stabilized on silica gel contains no detectable F-listed or D-coded chemical constituents, exhibits no hazardous characteristics, meets UTSs, and contains no other Part 261 Appendix VIII or Part 264 Appendix IX constituents at levels of concern. Based on the same information, LBNL also believes that the in-house GC/MS analytical results of the two bubbler water samples from the CCO-1 can represent all of the bubbler water samples generated and to be generated from multiple batches of waste sample oxidation using the CCO-2 system.

4. Fifty-two (52) tritiated water samples from the tritiated mixed waste tests were collected and analyzed by in-house GC/MS. Fifteen (15) water samples from the simulated nonradioactive surrogate oxidation tests were collected by LBNL staff and analyzed by BC laboratories. Several other water samples from the simulated nonradioactive surrogate oxidation tests were also collected and analyzed using in-house GC/MS.

5a. Tables 5-1 and 5-2 list the mixed waste and simulated nonradioactive surrogate samples, respectively, oxidized during the treatability study. LBNL staff have sampled 100% of the mixed waste and simulated nonradioactive surrogate samples and their oxidized and condensed liquid products.

(i) Table 5-1 provides the sample identification number, the sampling date, the original waste composition, the oxidized liquid sample composition, and the physical description of each oxidized tritiated liquid sample. All oxidized and condensed liquid product (tritiated water or HTO) samples were collected directly from the condenser and cold-trap of the CCO-2 system (see Figure 3). Each individual sample was collected in a clean glass bottle (nonpreserved). A grab sample was then taken from the glass bottle for NTLF's in-house GC/MS analysis (as described in 3a - d).

To date, 52 tritiated mixed waste samples were processed through the CCO-2 system. Approximately 16 liters of oxidized and condensed liquid product (HTO) were collected. In these 52 samples, many different combinations of the following 23 chemicals were identified and oxidized:

- acetic acid
- acetic anhydride
- acetone
- acetonitrile (ACN)
- benzene
- bromonitromethane
- chloroform
- cyclohexylamine
- dichloromethane (methylene chloride)
- dimethylformamide (DMF)
- dioxane
- ethanol (EtOH)
- ethyl acetate (EtOAc)
- hexane
- isopropanol (IPA)
- methanol (MeOH)
- methyl acetate
- pyridine
- tetrahydrofuran (THF)
- tetramethylethylenediamine (TMEDA)
- toluene
- triethylamine
- water

In the 52 total samples, some chemical constituents, such as acetic acid, acetic anhydride, bromonitromethane, cyclohexylamine, pyridine, and triethylamine, were identified only once; acetone, hexane, and methylene chloride (or dichloromethane) were identified twice; and methyl acetate and tetramethyl ethylenediamine were identified three times.

Table 5-2. Catalytic Oxidation of Simulated Nonradioactive Surrogate Samples (QC Samples) Analyzed by BC Laboratories

	Sample ID	Date Sample Taken	SW-846 Test Method	Original Constituents for Analysis	Oxidized Liq. Sample Composition	Description
1	NTLF062797	27 June 97	8260 / 8015	ACN, EtOAc, hexane, MeOH, THF, toluene	water	Clear water & one phase
2	NTLF071197	10 Jul 97 11 Jul 97	8260 / 8015 8260 / 8015	ACN, DMF, EtOH, EtOAc, IPA, MeOH, THF	water water	Clear water & one phase
3	NTLF071897	18 Jul 97	8260 / 8015	ACN, EtOAc, hexane, MeOH, THF, toluene	water	Clear water & one phase
4	NTLF072897	28 Jul 97	8260 / 8015	ACN, benzene, DMF, EtOAc, hexane, IPA, MeOH, THF, toluene,	water	Clear water & one phase
5	NTLF080797	06 Aug 97	8260 / 8015	ACN, EtOAc, hexane, IPA, MeOH, THF	water	Clear water & one phase
6	NTLF081297	12 Aug 97	8260 / 8015	ACN, $CHCl_3$, EtOH, IPA, MeOH, THF, toluene	water	Clear water & one phase
7	NTLF082797	27 Aug 97	8260 / 8015	ACN, benzene, $CHCl_3$, EtOH, IPA, MeOH, THF, toluene	water	Clear water & one phase
8	NTLF090397	03 Sep 97	8260 / 8015	ACN, benzene, $CHCl_3$, EtOH, IPA, MeOH, THF, toluene	water	Clear water & one phase
9	NTLF090997	09 Sep 97	8260 / 8015	ACN, benzene, $CHCl_3$, DMF, dioxane, EtOH, EtOAc, IPA, MeOH, THF,	water	Clear water & one phase

Table 5-2. (continued)

	Sample ID	Date Sample Taken	SW-846 Test Method	Original Constituents for Analysis	Oxidized Liq. Sample Composition	Description
10	NTLF091597	15 Sep 97	8260 / 8015 8270	ACN, acetic acid, benzene, DMF, EtOH, EtOAc, IPA, MeOH, THF, pyridine	water	Clear water & one phase
11	NTLF102497	24 Oct 97	8260 / 8015 8270	ACN, acetic acid, benzene, MeOH, THF, triethylamine	water	Clear water & one phase
12	NTLF011598	15 Jan 98	8260 / 8015	ACN, EtOH, EtOAc, IPA, MeOH,THF	water	Clear water & one phase
13	NTLF052198	21 May 98	8260 / 8015	EtOH, benzene, $CHCl_3$, EtOAc, MeOH, IPA, THF, toluene	water	Clear water & one phase
14	NTLF120498	04 Dec 98	8260 / 8015	ACN, benzene, EtOH, EtOAc, IPA, MeOH, THF, toluene	water	Clear water & one phase
15	NTLF121198	11 Dec 98	8620 / 8015 8270	ACN, benzene, DMF, dioxane, EtOH, EtOAc, IPA, MeOH, pyridine, THF, toluene	water	Clear water & one phase
16*	NTLF031699	16 Mar 99	In-house GC / MS*	ACN, benzene, DMF, Dioxane, EtOH, EtOAc, IPA, MeOH, pyridine, THF	water bubbler water	Clear water & one phase Clear water & one phase
17*	NTLF051499	14 May 99	In-house GC / MS*	ACN, benzene, DMF, Dioxane, EtOH, EtOAc, IPA, MeOH, pyridine, THF	water bubbler water	Clear water & one phase Clear water & one phase

* These two samples are included in Attachment I and were not analyzed by BC Laboratories.

The most commonly identified constituents in these 52 waste samples are the following 12 chemicals:

- acetonitrile
- benzene
- chloroform
- dimethylformamide
- dioxane
- ethanol
- ethyl acetate
- isopropanol
- methanol
- tetrahydrofuran
- toluene
- water

(ii) Table 5-2 provides the sample identification number, the sampling date, test method, constituents for analysis, oxidized surrogate sample composition, and physical description of each oxidized and condensed nonradioactive surrogate liquid sample. All of the oxidized and condensed liquid product (water) samples were drained directly from the cold trap of the CCO-1 system (see Figure 2). Each individual sample was collected in a clean glass container. A grab sample was then taken from the glass container for NTLF's in-house GC/MS analysis (as described in Item 3 a-d). The glass containers of these 15 samples were then shipped to an independent commercial laboratory for analysis.

5b. Description of sample collection method and the point of collection

Each oxidized liquid sample was collected directly from the condenser and cold trap of either the CCO-1 or the CCO-2 system into glass containers (described in detail in Item 3a - d; also see Figures 2 and 3). Two bubbler water samples were collected from the water bubbler container (flask) of the CCO-1 system into glass containers (see Figure 2).

5c. Description of the sampling location and the specific sampling point

The sampling location and the specific sampling point are described in Item 3a-d and are shown in Figures 2 and 3 for the oxidized and condensed liquid products of nonradioactive simulated surrogate and tritiated mixed waste samples, respectively.

5d. None of the samples were composite samples.

5e. The physical description of each oxidized and condensed liquid product is listed in Tables 5-1 (for tritiated water) and 5-2 (for nonradioactive water).

5f. None of the samples were composite samples.

5g. Description of handling and preparation techniques used for each sample

Before oxidation, the mixed waste sample was thoroughly mixed and a representative waste sample was collected from the sample collection container (flask) of the solvent vacuum extraction system (see Figure 1) using a clean syringe (as described in 3a-d).

All of the oxidized and condensed liquid products (tritiated water) were drained directly from the condenser and cold trap of the CCO-2 system (see Figure 3) into a nonpreserved glass bottle. A grab sample was then taken from each glass bottle using a clean syringe or pipette and analyzed using the in-house GC / MS (as described in Item 3 a-d).

Prior to oxidation, the simulated nonradioactive surrogate sample was prepared and thoroughly mixed in a clean glass bottle. A representative surrogate sample was collected from the bottle using a clean pipette for the in-house GC / MS analysis.

All of the nonradioactive oxidized and condensed liquid products (water) were drained directly from the cold trap of the CCO-1 system (see Figure 2). Each individual sample was collected in a clean glass container. A grab sample was then taken from the glass container using a clean syringe or pipette for the in-house GC / MS analysis (as described in Item 3 a-d). The glass containers of the 15 oxidized simulated surrogate samples were then shipped to an independent commercial laboratory for analysis as appropriate.

6. Describe the weather conditions during sampling (if conducted outdoors).

   Sampling was always performed indoors.

7. Describe any facility activities separate from sampling that occurred at the same time and that might have affected sample representativeness.

   The sample representativeness was not affected by any other research activities. Waste sample extraction, oxidation, and sampling were always performed in gloved containment boxes physically separated from any other research activities conducted in Room 103 of the NTLF. The nonradioactive surrogate sample oxidation and sampling were performed on a bench in a separate room (Room 102) of the NTLF. Other research activities were performed either on other benches or in hoods in Room 102.

8. LBNL staff always use clean sampling devices for waste, surrogate, and product samples.

9. The chain-of-custody procedures specified in EPA SW-846 were followed when LBNL staff sent the oxidized and condensed nonradioactive surrogate liquid samples to an independent commercial analytical laboratory (BC Laboratories). The BC Laboratories' analysis reports include the chain-of-custody sheets. (Four set of laboratory reports are included in Attachment I.)

11. The samples were collected to represent the mixed waste, surrogate sample, and oxidized products and were not from any "hot spots" of the petitioned waste.

16. The petitioned waste was generated only at the NTLF. The samples were not collected to characterize any wastes generated by a multiple waste treatment facility.

WASTE ANALYSIS INFORMATION

21. The petitioned waste sample (tritiated water) analyses were conducted by LBNL staff. Their resumes are included in Attachment H. LBNL has also sent the oxidized nonradioactive surrogate test samples (water) to an independent commercial laboratory (BC Laboratories) for verification. The laboratory's address and phone number are listed below.

Name BC Laboratories

Street 4100 Atlas Court

City Bakersfield State CA 93308

Telephone (661) 327-4911

22. Attachment I shows analytical results from BC Laboratories for oxidized and condensed liquid products from four simulated surrogate oxidation tests and from in-house GC/MS for oxidized and condensed liquid products and bubbler water of two simulated surrogate oxidation tests. Attachment J shows analytical results from in-house GC/MS for seven oxidized and condensed liquid products (tritiated water or HTO). Other analytical results are available on request.

23. Information of each sample and each analysis

a-b. Sample identification numbers and types of sample are presented in Tables 5-1 and 5-2 for the oxidized and condensed tritiated liquid products (HTO) and nonradioactive liquid product (water), respectively.

c-k. Nonradioactive surrogate oxidation sampling and analysis. LBNL staff conducted simulated nonradioactive surrogate oxidation tests using the CCO-1 system with various solvent mixtures. Fifteen oxidized and condensed liquid products from the CCO-1 system (see Table 5-2) were analyzed by BC Laboratories. Four data sets from this laboratory and two data sets from in-house GC/MS are included in Attachment I. These data are summarized in Table 5-3.

The dates of sample receipt by BC Laboratories, the sample preparation method, the name of the person conducting the analysis, the test methods (i.e., SW-846, Methods modified 8015, 8260, and/or 8270), the test results, the specific constituents for which the analysis was conducted, and the quantitation limits of some organic chemicals are all included in Attachment I. Also see Table 5-2.

Mixed waste oxidation sampling and analysis. LBNL staff conducted tritiated mixed waste sample oxidation tests (see Table 5-1) using the CCO-2 system. An in-house GC/MS was used for organic compound analysis. Seven data sets from NTLF are included in Attachment J and are summarized in Table 5-4. The experimental data sheets for each test, the test results (GC chromatograms), and the specific constituents for which the analysis was conducted are included in Attachment J. Also see Table 5-1.

The sampling and analysis was conducted by Dr. Chit Than, a NTLF chemist. The date of analysis of each sample is shown on the chromatogram. The oxidized and condensed liquid product (tritiated water) was collected from the condenser and cold trap of the CCO-2 into a small glass container for analysis. The liquid sample

Table 5-3. Examples of Analytical Results for the Oxidized Nonradioactive Surrogates (QC Samples)

Date & ID	Sample Composition before Oxidation (% estimated)	Concentrations in Oxidized Water, mg/L	Operation Conditions
07/28/97 NTLF072897	ACN 30%, DMF 2%, EtOH 4%, EtOAc 5%, hexane <1%, IPA 20%, MeOH 12%, THF 8%, toluene <1%, H_2O 18%	Nondetected (every component) DRE > 99.999% Verified by BC Lab.	CCO-1; 1.2–1.3 mL/min; 95% sample + 5% IPA; Oxid. cell 496–529 °C
10/24/97 NTLF102497	ACN 14.5%, acetic acid 1%, benzene 1%, MeOH 4.5%, THF 0.1%, triethylamine 3%, H_2O 75.9%	Nondetected (every component) DRE > 99.99% Verified by BC Lab.	CCO-1; 1.0–1.5 mL/min; 20% sample + 80% IPA to 55% sample + 45% IPA; Oxid. cell 462–489 °C
05/21/98 NTLF052198	benzene 4%, chloroform 2%, EtOH 10%, EtOAc 5%, IPA 25%, MeOH 20%, THF 5%, toluene 5%, H_2O 24%,	Nondetected (every component) DRE > 99.999% Verified by BC Lab.	CCO-1; 1–1.3 mL/min; 70% sample + 30% IPA to 95% sample + 5% IPA; Oxid. cell 503–519 °C
12/10-11/98 NTLF121198	ACN 14.4%, benzene 0.6%, DMF 2.7%, dioxane 0.7%, EtOH 0.3%, EtOAc 0.3%, IPA 14.4%, MeOH 5.5%, pyridine 2.8%, THF 2.8%, toluene 0.3%, H_2O 55.2%	Nondetected (every component) DRE > 99.999% Verified by BC Lab.	CCO-1; 1–1.5mL/min; 30% sample + 70% IPA to 50% sample + 50% IPA; Oxid. cell 478–508 °C
03/16/99* NTLF031699	ACN 24.8%, benzene 0.7%, DMF 6.3%, dioxane 1.4%, EtOH 1.3%, EtOAc 1.4%, H_2O 20%, IPA 24.8%, MeOH 3.3%, pyridine 10.2%, THF 5.8%,	Nondetected (every component) DRE > 99.999% In-house GC/MS	CCO-1; 1.1–1.4mL/min; 40% sample + 60% IPA to 30% sample + 70% IPA; Oxid. cell 502–514 °C
05/14/99* NTLF051499	ACN 24.5%, benzene 0.3%, DMF 4.0%, dioxane 1.5%, EtOH 1.4%, EtOAc 1.4%, IPA 27.2%, MeOH 3.4%, pyridine 10.6%, THF 5.8%, H_2O 20%,	Nondetected (every component) DRE > 99.999% In-house GC/MS	CCO-1; 0.9–1.5mL/min; 30% sample + 70% IPA to 50% sample + 50% IPA; Oxid. cell 488–493 °C

The abbreviations of several chemicals used in Tables 5-3 and 5-4 are listed below:
ACN = acetonitrile, MeOH = methanol, EtOH = ethanol, IPA = isopropanol,
THF = tetrahydrofuran, EtOAc = ethylacetate, DMF = dimethylformamide,
TMEDA = tetramethyl ethylene diamine, HC = hydrocarbon, CO = carbon monoxide
* Samples were analyzed using in-house GC/MS only.

Table 5-4. Examples of Catalytic Oxidation of Tritiated Mixed Waste Samples

Date & ID	Sample Composition before Oxidation (% estimated)	Concentration in Oxidized Water (mg/L)	Operation Conditions
(10/03/97) ***new waste*** **R016337**	ACN 16.8%, DMF 18.4%, dioxane 0.8%, EtOH 4.5%, EtOAc 3%, IPA 0.3%, MeOH 15.1%, THF 15.6%, H_2O 25.5% Vol: 550 mL; 20.4 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.99%	CCO-2; 1.0–1.2 mL/min; 40% sample + 60% IPA to 75% sample + 25% IPA; Oxid. cell 472–511 ^oC
(12/15/97) ***new waste*** **R018926**	ACN 14.1%, acetic acid 0.7%, benzene 1%, MeOH 4%, THF 0.1%, triethylamine 3.1%, H_2O 77%, + IPA (added later) vol. 400 mL; 11.89 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999%	CCO-2; 1.2–1.9 mL/min; 60% sample + 40% IPA to 90% sample + 10% IPA; Oxid. cell 470–520 ^oC
(03/31/98) ***new waste*** **R018382**	Acetone 2.7%, MeOH 33.2%, IPA 40.4%, THF 3.7%, water 20%, Vol. 235 mL; 3.5 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999%	CCO-2; 1.2–1.6 mL/min; 60% sample + 40% IPA to 100% sample; Oxid. cell 506–524 ^oC
(05/26/98) ***inventory + new waste*** **R019166**	ACN 5.4%, benzene 6.1%, EtOH 16.2%, EtOAc 1.1%, IPA 39%, MeOH 3.9%, THF 1.2%, toluene 2.2%, H_2O 25% Vol: 290 mL; 14.8 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999%	CCO-2; 1.0–1.2 mL/min; 40% sample + 60% IPA to 75% sample + 25% IPA; Oxid. cell 472–511 ^oC
(06/24/98) ***inventory waste*** **R019178**	ACN 0.2%, chloroform 8.1% EtOH 0.1%, IPA 47.5%, MeOH 8.1%, H2O 36%, Vol. 410 mL; 21.8 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999%	CCO-2; 1.0–1.2 mL/min; 60% sample + 40% IPA to 80% sample + 20% IPA; Oxid. cell 467–531^oC
(08/24/98) ***inventory waste*** **R018343**	ACN 0.2%, benzene 21.6%, EtOH 0.9%, IPA 45.9%, MeOH 10.9%, pyridine 0.2%, THF 0.1%, toluene 0.2%, H2O 20% Vol. 310 mL; 30.07 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999%	CCO-2; 1.2–1.5 mL/min; 90% sample + 10% IPA to 95% sample + 5% IPA; Oxid. cell 496–514 ^oC
(11/25/98) ***inventory waste*** **R020069**	MeOH 20%, IPA 30% THF 5%, H_2O 45%, Vol: 300 mL, 9.5 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999%	CCO-2; 1.2–1.9 mL/min; 80% sample + 20% IPA to 95% sample + 5% IPA; Oxid. cell 490–538 ^oC

(tritiated water) was then taken from the container and directly injected into the GC/MS (see Item 3 a-d).

Quantitation Limits for the in-house GC. The in-house GC quantitation limit (direct liquid injection) for chloroform is in the range of 0.5 mg/liter. For the following most commonly identified organic chemicals, the in-house GC quantitation limits (direct liquid injection) are in the range of 0.10 mg/liter (see attachment J):

- acetonitrile
- benzene
- dioxane
- ethanol
- ethyl acetate
- isopropanol
- methanol
- pyridine
- tetrahydrofuran
- toluene

24. The oxidized and condensed tritiated liquid products (HTO) and two bubbler water samples were analyzed by an in-house HP 6890 gas chromatograph (GC) equipped with both a flame ionization detector (FID) and a HP5973 mass selective detector (MSD). See also Item 26.

25. All information is included.

26. Independent Commercial Laboratory's Quality Control. The four sets of quality control analysis data for the oxidized and condensed nonradioactive surrogate liquid products (see Table 5-3) prepared by BC Laboratories are included in Attachment I.

After receiving the BC Laboratories' analysis reports of each sample (see Table 5-2), Dr. Li-Yang Chang, a Waste Management specialist in the EH&S Division of LBNL, reviewed the data package. This data review task was also supported by the Data Validation Specialist (Nahid Mahani) of the Waste Management Group of LBNL. An example of a complete laboratory analysis report and a Laboratory Documentation Requirements for Data Validation report for one oxidized and condensed nonradioactive surrogate liquid product prepared by the BC Laboratories is included in Attachment I. LBNL's data validation is also included.

LBNL's in-house GC/MS Quality Control. LBNL's in-house GC/MS quality control analysis of the oxidized and condensed tritiated and nonradioactive surrogate liquid products consists of the following elements:

(i) The NTLF chemist (Dr. Chit Than) checks GC/MS with standards (1 ppm range) regularly (every batch of samples). The reproducibility of the 1-ppm standard is also checked for some samples. The standard contains several different combinations of the following organic chemicals: acetonitrile, benzene, chloroform, dioxane, dimethylformamide, ethanol, ethyl acetate, isopropanol, methanol, pyridine, tetrahydrofuran, triethylamine, or toluene. The composition of the standard was developed on the basis of those most commonly identified constituents in mixed waste samples. (See descriptions in 5a of this section.)

(ii) Dr. Than also runs control blanks, such as tap water, and analysis of the oxidized and condensed liquid product (water) generated from the isopropanol oxidation test, every time before any analysis of the oxidized and condensed liquid product generated from the oxidation of mixed waste sample to ensure absence of lab contamination.

(iii) When necessary, Dr. Than also spikes some oxidized tritiated liquid samples (tritiated water) with a known amount of standards, such as 2.5 ppm of deuterium-labeled acetonitrile and toluene.

(iv) The data package is then independently reviewed by Dr. Li-Yang Chang for each batch of samples.

The data package in Attachment J includes the in-house GC / MS quality control analysis data.

27. There is no correction based on QC results.

28. Any inconsistencies or deviations found in the reported analytical results?

Four analytical reports from BC Laboratories showed ≤ 10 ppb chloroform in four oxidized nonradioactive surrogate products (sample numbers NTLF091597, NTLF102497, NTLF120498, and NTLF121198). However, in these four nonradioactive surrogate solvent mixtures, LBNL personnel did not use any chloroform (see Table 5-2). These liquid products might have been cross-contaminated when the simulated surrogate mixtures were oxidized in the CCO-1 or during the sampling and analysis (because several other surrogate mixtures oxidized previously contained chloroform). Since the chloroform concentrations in these four oxidized products were very low (8.3, 2.4, 10, and 7.7 ppb, respectively) and, below UTS, no corrections were necessary.

Several other minor deficiencies of BC Laboratories' reports have also been identified by LBNL's data validation specialist, Nahid Mahani. For example, the practical quantitation or reporting limits were not accurate for a few chemicals (naphathalene and 1,2,4-trichlorobenzene) for the analysis of samples NTLF072897-A and NTLF 072897-F; the CCV recoveries were low for a few chemicals for the analysis of sample NTLF121198; methylene chloride was detected in the method blank for the analysis of sample NTLF120498; and some laboratory control sample, surrogate, and matrix spike recoveries were low for the analysis of sample NTLF102497 (A, B, and C). However, those deficiencies did not have any influence on the analytical results. Thus, those reports were accepted. LBNL notified BC Laboratories of those deficiencies, and no corrections were necessary. Examples of LBNL's data validation documents are presented in Attachment I.

29. No calculations were involved in the analysis.

SECTION 6: GROUNDWATER MONITORING INFORMATION

Since the petitioned waste is not managed in a land-based waste management unit, this section is not applicable.

ATTACHMENTS

ATTACHMENT A	Demonstration of Combustion Technology / Petition for Approval of Alternative Treatment Method
ATTACHMENT B	Description of LBNL's Catalytic Chemical Oxidation systems
ATTACHMENT C	Descriptions of the radioactive and mixed wastes generated from NTLF's labeling and purification experiments; Pictures of NTLF rooms and glove boxes.
ATTACHMENT D.	Treatability study annual reports and notice letter to DTSC.
ATTACHMENT E.	List of LBNL's Permits
ATTACHMENT F.	Schematics of HWHF units for mixed waste storage.
ATTACHMENT G.	Material Safety Data Sheets of major chemical constituents used in tritiation reactions and identified in multiple mixed waste samples.
ATTACHMENT H.	Resumes of LBNL sampling and analysis staff
ATTACHMENT I.	In-house GC / MS data packages for two samples and BC Laboratories analytical reports for four simulated nonradioactive surrogates.
ATTACHMENT J.	NTLF GC / MS results of seven mixed waste samples and their oxidized and condensed liquid products.

ATTACHMENT A. Demonstration of Combustion Technology/Petition for Approval of Alternative Treatment Method

ATTACHMENT B. Description of LBNL's Catalytic Chemical Oxidation systems

ATTACHMENT C. Descriptions of the radioactive and mixed wastes generated from NTLF’s labeling and purification experiments; Pictures of NTLF rooms and glove boxes.

ATTACHMENT D. Treatability study annual reports and notice letter to DTSC.

ATTACHMENT E. List of LBNL’s Permits

ATTACHMENT F. Schematics of HWHF units for mixed waste storage.

ATTACHMENT G. Material Safety Data Sheets of major chemical constituents used in tritiation reaction and identified in multiple mixed waste samples.

ATTACHMENT H. Resumes of LBNL sampling and analysis staffs.

ATTACHMENT I. BC Laboratories analytical reports for four simulated non-radioactive surrogates.

Attachment I – Table

BC Lab's report of Sample NTLF072897

BC Lab's report of Sample NTLF102497

BC Lab's report of Sample NTLF052198

BC Lab's report of Sample NTLF121198

In-house GC / MS Data package of Sample NTLF031699

In-house GC / MS Data package of Sample NTLF051499

ATTACHMENT J. NTLF GC / MS results of seven mixed waste samples and their oxidized and condensed liquid products.

Attachment J – Table

In-house GC / MS Data package of Sample R016337

In-house GC / MS Data package of Sample R018926

In-house GC / MS Data package of Sample R018382

In-house GC / MS Data package of Sample R019166

In-house GC / MS Data package of Sample R019178

In-house GC / MS Data package of Sample R018343

In-house GC / MS Data package of Sample R020069

In-house GC / MS Data package of Quantitation Limits

Quantitation Limits for NTLF's in-house GC

The in-house GC quantitation limit (direct liquid injection) for Chloroform is in the range of 0.5 mg / liter.

For the following most commonly identified organic chemicals, the in-house GC quantitation limits (direct liquid injection) are in the range of 0.10 mg / liter:

- Acetonitrile (ACN)
- Benzene
- Dioxane
- Ethanol (EtOH)
- Ethyl acetate (EtOAc)
- Isopropanol (IPA)
- Methanol (MeOH)
- Pyridine
- Tetrahydrofuran (THF)
- Toluene