

## **EPA'S FINAL DELISTING RULE FOR TRITIATED MIXED WASTE AND DETERMINATION OF EQUIVALENT TREATMENT METHOD FOR A CCO PROCESS**

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### **ABSTRACT**

The United States biomedical R&D community faces 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 contains RCRA-regulated hazardous components. Lawrence Berkeley National Laboratory (Berkeley Lab or LBNL) believes it is essential to develop processes for ensuring that mixed wastes may be treated and disposed of in a manner that minimizes impacts on the environment.

In 1996 Berkeley Lab decided to conduct a treatability study on tritiated mixed waste, using catalytic chemical oxidation (CCO) technology. Along with selection of a suitable treatment technology, it was necessary to formulate a reasonable management and disposal route. This included ensuring the appropriate legal status of this waste by obtaining a Determination of Equivalent Treatment (DET) for the oxidation approach, and by Delisting the oxidized products. Following these actions, products may be disposed as low-level radioactive waste (LLRW) at a permitted facility.

In June 1999, LBNL submitted a "Petition to Delist Tritiated Mixed Waste Treatment Residues", including a DET petition, to the Environmental Protection Agency (EPA) Region IX. After 4 years careful analysis and public education, the EPA granted both LBNL's petitions for its tritiated mixed waste on August 7, 2003. In the final rule, EPA concluded that the petitioned waste is no longer hazardous and that the CCO treatment is equivalent to combustion.

### **BACKGROUND**

United States pharmaceutical and biomedical research institutes regularly use tritium and carbon-14 in research and development projects, and generate mixed waste with Curies (Ci) of tritium activity, or mCi of carbon-14. In LBNL's case, the hazardous component of this waste stream included F-listed spent solvents, was designated as high total organic carbon (TOC) ignitable D001 waste, and some batches contained chlorinated hydrocarbons (code D022) [1].

As the EPA and the Nuclear Regulatory Commission (NRC) have acknowledged, dual regulation of mixed waste under the Atomic Energy Act and the Resource Conservation and Recovery Act (RCRA) presents a number of difficulties for storage, treatment, and disposal. Commercial options for treatment and disposal (incineration) of tritiated mixed waste have undesirable environmental (tritium release) and fiscal consequences (e.g., \$15 to \$30/mCi). As an example, the estimated cost for disposal of the tritiated mixed waste inventory at Berkeley Lab by a permitted treatment facility would be more than \$35 million.

### **LBNL'S APPROACH**

Table I lists the hazardous solvents normally used in the LBNL tritium labeling process that have EPA RCRA waste codes [2]. When faced with unsatisfactory long-term storage and disposal options for tritiated mixed waste, the LBNL decided to conduct a mixed waste treatability study (following the California Code of Regulations Chapter 22, section 66261.4) [3] using a catalytic chemical oxidation

(CCO) technology. This triggered administrative requirements. Treatability studies require, for example, notification to the regulatory agency, an EPA identification number for the facility conducting the study, a record of annual waste shipping and treatment quantities, reporting (the annual report must be submitted to the regulatory agency no later than March 15 of each year), and record-keeping requirements for all of the studies (for three years). The primary goal of using the CCO technology was to ensure that the mixed waste treatment residues would meet land disposal requirements (LDR) [2] and universal treatment standards (UTS) [4].

Table I Solvents common in tritium labeling and their RCRA codes

Code	Solvent	Universal Treatment Standard (UTS) for Nonwastewater
F002	methylene chloride	30 mg/kg
F003	acetone	160 mg/kg
	ethyl acetate	33 mg/kg
	methanol	0.75 mg/L TCLP
F005	toluene	10 mg/kg
	pyridine	16 mg/kg
	benzene	10 mg/kg
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, ether, ethyl acetate, hexane, isopropanol, methanol, methyl acetate, methylene chloride, pyridine, tetrahydrofuran, tetramethylethylene diamine, toluene, triethylamine	CMBST, POLYM, or RORGS
D022	chloroform (> 6 mg/L TCLP)	6 mg/kg

We also sought to ensure the appropriate legal status of this waste by applying for a Determination of Equivalent Treatment [5] for the oxidation approach, and Delisting the treatment residues [6]. A summary of the strategy developed for LBNL's tritiated mixed wastes is as follows:

- 1) Evaluate the hazardous and radiological characteristics of the identified waste streams.
- 2) Evaluate the effectiveness and efficiency of the CCO and solvent extraction technologies.
- 3) Characterization of the treatment residues (oxidized and condensed liquid product) with an in-house GC/MS and using a commercial analytical laboratory.
- 4) Obtain delisting approval from USEPA (40 CFR260.22(b)) [6] 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 I) [7].
- 5) Dispose of the delisted treatment residues at a licensed low-level radioactive waste disposal site, or send the residues off-site for tritium recovery.

LBNL planned to use this combination of technology application (CCO) and administrative action (Delisting Petition and Determination of Equivalent Treatment) to resolve our tritiated mixed waste disposal problem, with a net benefit to the environment.

## CCO DESIGN

LBNL's CCO system consists of (a) a preheater, (b) an oxidation cell with two spark sources, (c) a packed-bed tubular reactor filled with platinum-coated alumina catalyst, and (d) an oxidation product (tritiated water) recovery and emission reduction system which consisted of two condensers, a dry-ice cold trap, three water bubblers in series, and a silica gel filter. A set of general operating conditions for the CCO system was developed: (a) preheater set at 300 °C, (b) oxidation cell controlled near or above 500 °C, (c) catalytic bed controlled at 500 °C or above, and (d) sample flow rate set between 1 and 2 mL/min based on the composition of the sample.

Since it is impossible to have commercial analytical work done on radioactive samples with moderate activities (up to 180 Ci), two separate CCO systems, CCO-1 and CCO-2, were built. The CCO-1 system was used only for non-radioactive, simulated waste (surrogate) tests. The CCO-2 system was used for the tritiated mixed waste samples. While the surrogate products from the CCO-1 were subjected to both external analytical tests by a commercial analytical laboratory and in-house GC/MS analyses, the radioactive products from the CCO-2 were only analyzed using the in-house GC/MS system.

## ANALYTICAL METHODOLOGY

The in-house GC/MS analysis procedure used for each oxidation test included the following steps:

- 1) Using the in-house GC/MS, we analyzed the mixed waste or surrogate sample prior to oxidation (liquid sample was directly injected into the GC/MS).
- 2) After oxidation, we collected the oxidized and condensed liquid product (water or tritiated water) from each batch of mixed waste or nonradioactive surrogate sample from the condenser and cold trap of the CCO system.
- 3) Using the in-house GC/MS, we analyzed the control blanks (i.e., tap water) and the oxidized and condensed liquid product (water or tritiated water) from the pre-run of nonradioactive isopropanol for each batch process. This provided quality assurance on the GC/MS analysis, and on the oxidation efficiency of the CCO.
- 4) Using the in-house GC/MS, we analyzed the oxidized and condensed liquid product of each batch of mixed waste or nonradioactive surrogate sample (we also analyzed the bubbler water occasionally as a QA check).
- 5) The GC/MS performance was checked for every mixed waste batch with a commercial 1-ppm standard containing up to fifteen of the target solvents in the mixed waste samples.
- 6) In step (4), the in-house GC quantitation limits were also assessed for the direct injection analysis approach. The GC quantitation limits for chloroform and methylene chloride ranged from 0.25 to 0.5 mg/liter. For the following organic chemicals, the GC quantitation limits are about 0.10 mg/liter (100 ppb): acetone, acetonitrile, benzene, dioxane, dimethylformamide, ethanol, ethyl acetate, isopropanol, methanol, pyridine, tetrahydrofuran, triethylamine, and toluene.

For quality assurance purposes, more than 10 sets of oxidized surrogate sample were collected from the CCO-1 system and analyzed by a commercial analytical laboratory. The EPA test methods 8015-modified (for industrial solvents), 8260 (for volatile organic), and 8270 (for semi-volatile organic) were performed by the analytical laboratory.

## **OXIDATION PERFORMANCE AND RESULTS**

Using the optimum operating conditions for each sample, analysis of the oxidation product yields a destruction and removal efficiency (DRE) of organic constituents exceeding 99.999%. This high oxidation efficiency was also supported by absence of CO and hydrocarbons (HC) in the exhaust gas above the one mg/liter detection limit on oxidation of any waste or surrogate samples. However, achieving this DRE requires careful attention and fine tuning for the oxidation process (see Table II). Although none of the target organic compounds was detected in the condensed liquid product (HTO) above a one mg/liter level using the in-house GC/MS, the variability in results and operating conditions indicated the effects of the unique characteristics of each sample on the oxidation and emission reduction processes.

Table II Examples of catalytic oxidation results of mixed waste and surrogate samples

Date and ID	Sample Composition before Oxidation	Concentration in Oxidized Water (mg/L)	Operating Conditions
(02/18/00) R021130	ACN 1.8%, DMF 26.5%, EtOH 1.8%, IPA 24.6%, MeOH 10.3%, water 35%  Vol. 285 ml; 81 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999% (CO and HC < 1 ppm)	CCO-2; 1.5 - 1.8 mL/min; 85% sample + 15% IPA to 80% sample + 20% IPA Oxid. cell 483–538 °C
(03/16- 17/00) R021144	ACN 38%, DMF 12%, EtOAc 0.6%,  EtOH 0.2%, ether 0.5%, MeOH 4.2%, toluene 0.5%, THF 14%, water 30%  Vol. 375 ml; 58.2 Ci	<1.0 (in-house GC/MS)  (all components) DRE > 99.999% (CO and HC < 1 ppm)	CCO-2; 1.5 - 1.8 mL/min;  85% sample + 15% IPA to 80% sample + 20% IPA Oxid. cell 460–521 °C (experienced water bubbler complications)
(06/30/00) R026730	ACN 24%, acetone 1%, EtOAc 6.5%, EtOH 4%, IPA 24%, MeOH 3.5%, THF 1%, water 36%  Vol: 420 ml; 43.2 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999% (CO and HC < 1 ppm)	CCO-2; 1.1–1.5 mL/min; 75% sample + 25% IPA to 80% sample + 20% IPA; Oxid. cell 486 – 525 °C (experienced water bubbler complications)
(09/07/00) R026749	ACN 23%, DMF 10%, EtOAc 5%, IPA 13%, MeOH 2%, THF 7%, water 40%  Vol. 415 ml; 61.3 Ci	<1.0 (in-house GC/MS) (all components) DRE > 99.999% (CO and HC < 1 ppm)	CCO-2; 1.5–2.0 mL/min; 75% sample + 25% IPA to 80% sample + 20% IPA; Oxid. cell 497–527 °C (experienced oxygen flow complications)
Non-rad. Surrogate (8/97)	ACN 7.4%, Chloroform 3.2%, EtOH 3.5%, IPA 21.7%, MeOH 34.7%, THF 2.2%, Toluene 2.2%, Water 25.1% Volume: 250 mL	<1.0 (all components) DRE > 99.999% Verified by a commercial laboratory	CCO-1; 1.4–1.75 mL/min; 80% sample + 20% IPA to 95% sample + 5% IPA Oxidation cell 495–508°C Acidic liquid neutralized.
Non-rad. Surrogate (7/97)	ACN 7.8%, DMF 8.2%, EtOH 18.7%, EtOAc 1.5%, IPA 7.8%, MeOH 21%, THF 7.8%, Water 25.7% Volume: 260 mL	<1.0 (all components) DRE > 99.999% Verified by a commercial laboratory	CCO-1; 1.2–1.4 mL/min; 70% sample + 30% IPA Oxidation cell 480–520 °C Acidic liquid neutralized
Non-rad. surrogate (01/98)	IPA 26%, EtOH 2%, H <sub>2</sub> O 31%, MeOH 2%, ACN 32%, EtOAc 2%  THF 5%.  Volume: 200 mL	<1.0 (all components) DRE > 99.999%  Verified by a commercial laboratory	CCO-1; 1.0 - 1.5 mL/min;  100% sample  Oxid. Cell 490 - 523 °C  acidic liquid neutralized

Note: ACN = acetonitrile, CO = carbon monoxide, DMF = dimethyl formamide, EtOAc = ethyl acetate, EtOH = ethanol, HC = hydrocarbons, IPA = isopropanol, MeOH = methanol, THF = tetrahydrofuran, TMEDA = tetramethylethylene diamine

As our CCO process design, operating conditions, and oxidation results (DRE >> 99.999%) demonstrated [8 - 11], the CCO technology achieves a measure of performance equivalent to that of other combustion methods specified in 40 CFR 268.42, Table I [7]. Thus, we believe the CCO process meets the definition of combustion, and it is appropriate for the D001 or F003 codes of the treatment residues to be deleted.

The analyses performed by a commercial laboratory using the EPA test methods demonstrated that the underlying hazardous constituents or possible byproducts included in EPA Methods 8260, 8270, and

8015 were not produced at the detection levels during our CCO treatment. More than 120 analytes were checked by these EPA test methods. As examples, a list of chemicals with the EPA test methods used for each compound is presented in Table III. These 23 chemicals are included in 40 CFR 261 Appendix VIII [12] and they were not detected in our liquid products.

Table III Examples of underlying hazardous constituents that are not detected in the liquid product produced by the CCO process

Chemicals	EPA Test Method*	Concentration in Product
Aldrin	8270	nondetect (ND)
benzidine	8270	ND
butyl benzyl phthalate	8270	ND
di-chlorobenzene (1,2-, 1,3-, and 1,4-)	8260	ND
1,2,4,5-tetrachlorobenzene	8260	ND
hexachlorobutadiene	8260	ND
1,2,4-trichlorobenzene	8260	ND
hexa-chlorobenzene	8270	ND
1,2-dichloroethane	8015, 8260	ND
1,1-dichloroethene	8260	ND
1,2-dichloroethene	8015, 8260	ND
trichloroethene	8015, 8260	ND
tetrachloroethene	8015, 8260	ND
vinyl chloride	8260	ND
carbon tetrachloride	8015, 8260	ND
2,4-dichloro-phenol	8270	ND
penta-chlorophenol	8270	ND
trichloro-phenol (2,4,5- and 2,4,6-)	8270	ND
diethyl-phthalate	8270	ND
dimethyl-phthalate	8270	ND
dinitro-toluene (2,4- and 2,6-)	8270	ND
nitro-benzene	8270	ND
4-nitro-phenol	8270	ND

\* For the eleven compounds tested by Method 8260, the detection limits are 0.5 ppb ( $\mu\text{g/L}$ ). By contrast, for the 12 compounds tested by Method 8270, the detection limits vary from 20 to 200 ppb (depending on the volume of sample analyzed), and for the 5 compounds tested by Method 8015, the detection limits vary from 100 to 200 ppb.

Our results also indicated that for samples containing chloroform or methylene chloride (7 out of 70, low concentrations) prior to oxidation, there were no chlorinated compounds (in the  $\mu\text{g/liter}$  range) detected in the oxidized liquid product using our GC/MS and using the EPA test Methods 8015-modified, 8260, and 8270 (analyzed by a commercial laboratory).

We did not observe the formation of soot or particulate materials in our CCO process (except in three cases out of 70 tests; mainly due to the oxygen deficiency of the process for a few seconds).

We believe that:

- 1) The presence of steam enhanced the conversion of CO and other hydrocarbons to  $\text{CO}_2$  and water,
- 2) The preheater of our CCO equipment completely vaporized the samples, and the sample injection port provided excellent mixing conditions for the vapor and oxygen; thus, the oxidation efficiency was greatly enhanced, and
- 3) The Pt catalyst further enhanced the conversion of CO or other hydrocarbons to  $\text{CO}_2$  and water.

We also found that the condensers, cold trap, water bubblers, and silica gel filter confined more than 99.9% of tritium in the waste samples. Thus, this process exceeds the DRE standards for incineration (99.99%), while emission of tritium is greatly reduced to << 0.1% of that from incineration. The emission reduction devices also minimized the emission of acidic gas (condensed as hydrochloric or nitric acid in the liquid product) along with the tritium. This level of performance clearly meets the goals of LBNL's pollution prevention program. Examples of the CCO operating conditions and oxidation results are presented in Table II.

Based on more than 80 CCO test results of mixed waste and non-radioactive surrogate samples, we have demonstrated that:

- a) The organic constituents in the treatment residues were much less than 1 mg/L, thus the F002 and F005 codes of the residues derived from the original listed solvents should be delisted, and
- b) The D001 and F003 codes of the liquid products of oxidation should be deleted because the performance of CCO system exceeded the performance standard for incineration, and
- c) The treatment residue is water or tritiated water only, exhibits no RCRA characteristics (after neutralization), and contains no RCRA-regulated underlying hazardous constituents above UTS (40 CFR 268.48) [4] and no 40 CFR 261 Appendix VIII constituents [12].

With these results, we believe it is appropriate for the treatment residues from mixed waste oxidation to be disposed as low-level radioactive waste at a permitted facility, or for tritium in the liquid products to be recovered.

In June 1999, LBNL submitted a "Petition to Delist Tritiated Mixed Waste Treatment Residues" to the EPA Region IX (<http://www.lbl.gov/LBL-Programs/tritium/delisting/index.html>). We included 6 sets of analytical data from non-radioactive surrogate samples and 7 sets of analytical data from tritiated waste samples in the petition.

## **RCRA RULES AND DELISTING PETITION**

Under RCRA, prior to disposal, the treatment residues must not exhibit RCRA characteristics, must meet the concentration-based LDR treatment standards for the F-listed and D-coded components, and the UTS for the underlying hazardous constituents. Also, the D001 high-TOC designation of the waste means that the technology-specific LDR of combustion must be met. Thus, after the treatability study, a delisting petition for the federal codes that the waste originally had and a petition for the determination of equivalent treatment method for the CCO process to EPA was necessary for the management and disposal of our treatment residues.

The EPA's delisting petition process includes the following steps:

- 1) Conduct pre-petition scoping meetings and the petitioner submits the petition to the regional EPA office.
- 2) The regional EPA office acknowledges the receipt of the petition.
- 3) Within 7 days of receipt of the letter from the EPA, the petitioner submits to the local newspaper a public notice and a copy of the notice must be sent to the regional EPA office within 2 days.
- 4) The regional EPA office conducts a completeness review and a technical review (comments or notices of deficiency will be sent to the petitioner if necessary).

- 5) The petitioner responds to the comments.
- 6) The regional EPA office recommends approval or denial of the petition.
- 7) EPA publishes the decision on the Federal Register for a 45-day public comment period (EPA may also conduct public hearings, if requested).
- 8) EPA responses to comments are included in the final decision.
- 9) EPA publishes the final notice or rule in the Federal Register, with a letter sent to the petitioner.

In order for the petitioned waste to be excluded from RCRA by EPA, the petitioner must demonstrate that the petitioned waste (the treatment residues from oxidation, in this case):

- 1) Does not meet the criteria for which it was listed (F002, F003, and F005),
- 2) Does not exhibit any of the hazardous waste characteristics (i.e., ignitability, corrosivity, reactivity, and toxicity), and
- 3) Does not exhibit any additional factors or constituents that may cause the petitioned waste to be a hazardous waste (such as the underlying hazardous constituents).

#### **EQUIVALENT TREATMENT METHOD**

Because most of our tritiated mixed waste was also characterized as D001 high-TOC ignitable liquid, which has more than 10% total organic compounds, the applicable treatment standard is "RORGS, POLYM, or CMBST" [7]. Thus, in conjunction with the delisting petition, we also sought EPA's concurrence with our conclusion that the catalytic oxidation technology we used to treat the D001 high-TOC non-wastewater, D022, F-002, F-003, and F-005 waste samples from which the treatment residues are derived is within the federal regulatory definition of combustion (CMBST). Alternatively, we sought a determination of equivalent treatment method [5] for the catalytic oxidation technology.

Following EPA's delisting petition and treatment standard guidance [2, 5 - 7], we developed our study strategy, sampling and analysis methodology, and CCO operating procedures as previously described. In our treatability study, we conducted multiple nonradioactive simulated surrogate oxidation tests using the CCO-1 process and mixed-waste sample oxidation tests using the CCO-2 process.

Since the first studies in March 1997, two sources of tritium-containing mixed waste have been tested:

- 1) Liquid tritiated mixed waste generated from tritiation reactions, and
- 2) Tritiated mixed waste absorbed on silica gel, as legacy waste generated from the process described in (1) above.

Up to the conclusion of this work in June 2002, 70 mixed waste samples containing 2,300 Ci of tritium activity were oxidized, products condensed and collected from the CCO process, giving approximately 23 liters of liquid product (tritiated water).

#### **EPA'S FINAL EXCLUSION.**

After 3 years extensive evaluation and public outreach, the EPA proposed to grant LBNL's two petitions on July 31, 2002. The proposed rule was opened for public comments until February 6, 2003. During



that time, the EPA Region IX also held a public hearing on January 23, 2003. Overall, 192 comments were received by EPA. Among them, 172 were supportive, 6 neutral, and 14 non-supportive. On August 7, 2003, EPA granted LBNL's two petitions and published the final rule (Federal Register, Vol 68, No. 152, pp.46951-57) [13].

In this final rule, EPA determined that the CCO technology is equivalent to combustion (one of the required LDR treatment standards for organic wastes such as D001) because:

- 1) The DRE of the organic solvents exceeds 99.999%,
- 2) The CCO system does not emit HCl vapor and particulate matter, and
- 3) The CCO system was operated in compliance with local, state, and federal regulations.

EPA granted the delisting petition because the oxidized mixed waste residues:

- a) Do not exhibit any RCRA hazardous characteristics,
- b) Have met the applicable LDR technology-based standard (combustion) for D001 waste, and
- c) Have met the LDR numeric standards for F002, F003, and F005 wastes.

#### **POTENTIAL BENEFITS OF THE FINAL RULE AND LBNL'S APPROACH**

In this rulemaking, the U.S. EPA granted LBNL site-specific regulatory relief so that small volumes of treated mixed waste are excluded from RCRA, and the residues must now be managed as low-level radioactive waste.

Even though this is a site-specific exclusion, the success of this rulemaking represents a much broader gain for the US research community. The final exclusion and determination serves as a precedent for generators of tritiated mixed waste throughout the USA. The success of LBNL's petitions also demonstrates working with the EPA to achieve our mutual pollution prevention goals and to gain regulatory relief on mixed waste issues.

Our experience with the Treatability Study and Delisting Petition suggests that this is a viable management option for a small quantity of tritiated mixed waste. Correct application of the treatability study process and appropriate characterization of the treatment residues paved the way for these materials to exit RCRA Subtitle C regulation. The benefits of our approach are:

- a) Development of an alternative to incineration (i.e. CCO),
- b) Elimination of tritiated mixed waste storage and disposal problems,
- c) Encouragement of tritium recovery and pollution prevention,
- d) Demonstration of an appropriate disposal or recycling route for the treated waste,
- e) Avoidance of multi-million dollar TSD costs for commercial treatment, and
- f) Demonstration of a route for small volume mixed waste generators to reduce their regulatory burden and cost liability.

**REFERENCES**

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