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MIXED WASTE INTEGRATED PROGRAM

**A TECHNOLOGY ASSESSMENT FOR MERCURY-CONTAINING
MIXED WASTES**

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EXECUTIVE SUMMARY

The treatment of mixed wastes must meet U.S. Environmental Protection Agency (EPA) standards for chemically hazardous species and also must provide adequate control of the radioactive species. The U.S. Department of Energy (DOE) Office of Technology Development established the Mixed Waste Integrated Program (MWIP) to develop mixed-waste treatment technology in support of the Mixed Low-Level Waste Program. Many DOE mixed-waste streams contain mercury. This report is an assessment of current state-of-the-art technologies for mercury separations from solids, liquids, and gases. A total of 19 technologies were assessed. This project is funded through the Chemical-Physical Technology Support Group of the MWIP.

Incineration is the only available technology for solids and liquids that are made up primarily of combustible constituents. Examples of DOE combustible solid mixed wastes are rags and wipes, and examples of liquid combustible wastes are various cutting and lubricating oils. EPA has selected a best-demonstrated available technology (BDAT) of incineration for a DOE hydraulic oil contaminated with mercury and tritium.

Mercury can be separated from noncombustible solids and aqueous sludges by acid leaching, thermal treatment, contact with chlorine or iodine to form soluble halide complexes, or contact with sulfide solutions to form soluble mercury polysulfide. Acid leaching is the BDAT for K071 nonwastewaters. Contact with KI/I_2 solution to form soluble mercury iodide complexes is the subject of a recent General Electric Company (GE) patent. Leaching tests with metallic mercury and eight mercury compounds from a synthetic soil matrix gave very promising results.

Nine technologies were found for mercury separation from aqueous liquids. Sulfide precipitation is the BDAT for K071 wastewaters. A number of promising ion-exchange resins were identified. Both strongly basic and weakly basic anion-exchange resins can be used to remove halide complexes, but they cannot be used selectively. The strongly basic anion resins can be used over a wide pH range, while the weakly basic resins can be used for neutral and acid solutions and regenerated with alkaline solutions. Resin containing the iminodiacetic group (e.g., Ionac SR-5) pick up cationic mercury selectively from calcium and magnesium; however, cobalt and copper are also picked up. A thiol resin, which is very selective for mercury in the cationic form, has been used at the Savannah River Plant. Thiol resins are available commercially. Regeneration of thiol resins is difficult because of the insolubility of mercuric sulfide.

Chelating resins containing the isothiuronium group were found effective for both inorganic mercury and dimethyl mercury from solutions containing large amounts of divalent cations. A vendor survey is required to determine if a suitable chelating resin is commercially available.

A technology with unique potential advantages for aqueous liquids is the 3-M Corporation-(3M-IBC) membrane technology. A highly porous Teflon membrane contains small (5 to 10 μ) silica particles with crown ethers attached to their surfaces. Flow rates can be 10 times larger than through ion-exchange beds. The crown ethers can be highly specific for cationic and methyl mercury. This technology is in commercial use for some contaminants, but it is only in the development stage for mercury.

For the removal of traces of mercury from gases, two available technologies were found: Mersorb[®] pellets and the Boliden-Norzinc process. Beds of Mersorb pellets offer the potential for a much simplified flowsheet with no process control complications. The

Boliden-Norzinc process utilizes a mercurous chloride/mercuric chloride reactor and a scrubber. The flowsheet is relatively complex, but the process yields a mercurous chloride product.

Recommendations

Thermal treatment technologies will play a major role in mixed-waste treatment. Incineration and retorting/roasting are commercially available technologies, but a demonstration of complete process flowsheets is needed. For mercury removal from noncombustible solids or aqueous sludges, the KI/I_2 -leaching technology that is being developed by GE appears applicable to a large number of mercury compounds. DOE should investigate this technology for DOE mixed wastes.

For mercury removal from aqueous liquids, several ion-exchange resins are promising. A test program should be set up to evaluate commercially available basic anion exchange; thiol and chelating exchangers for selectivity, capacity, difficulty of regeneration; and finally process economics. Borohydride reduction should be evaluated for applications where selectivity is not important and a wide range of pH may be encountered. The 3M-IBC membrane technology holds promise for providing compact, high-throughput, very selective separations and should be pursued at the engineering development scale. The evaluation and development of these technologies must include pretreatment needs (e.g., suspended solids removal and pH adjustment for ion exchange) and specificity requirements.

Mercury removal from gases can be accomplished by using Mersorb beds or by the Boliden-Norzinc process, both available commercially. Design studies should be made for several DOE applications to determine process economics, and a demonstration should be carried out for the process that appears more favorable.

1. INTRODUCTION

Mixed waste is waste containing both chemically hazardous and radioactive chemicals. The treatment of mixed wastes must meet U.S. Environmental Protection Agency (EPA) standards for the chemically hazardous species and also must provide adequate control of the radioactive species. The U.S. Department of Energy (DOE) Office of Waste Management established the Mixed Low-Level Waste Program to identify and implement the optimal treatment, storage and disposal options for mixed waste. The DOE Office of Technology Development established the Mixed Waste Integrated Program (MWIP) to develop mixed-waste treatment technology in support of the Mixed Low-Level Waste Program.

DOE has classified individual mixed-waste streams into broad categories, with mercury-containing wastes as one of these categories. To help MWIP select priorities for technology development activities, an assessment of current state-of-the-art technologies for mercury separations has been carried out. Technologies were researched in the open literature, patent literature, and government reports. Assessments were made using the information found there.

MWIP technology areas are illustrated by the flowsheet in Fig. 1. Mercury-containing mixed wastes can occur in a number of physical forms such as aqueous and organic liquids and combustible and noncombustible solids. Wastes that are primarily organic material would go to the "waste destruction and stabilization" operation, and the subsequent gas cleanup operation would produce condenser-scrubber liquid and gas streams requiring mercury separation. Inorganic solids might be leached to transfer the mercury to a liquid phase, with subsequent removal of mercury from the liquid. More detailed flowsheets are presented in a Bechtel National, Inc., design study.¹

In the search for applicable technologies, careful attention was paid to EPA best-demonstrated available technologies (BDATs). The source of this information was *Final BDAT Background Document for Mercury-Containing Wastes D009, K106, P065, P092 and U151*.² K106 waste is a wastewater treatment sludge from the mercury cell process for chlorine production, the second largest user of mercury in the United States. P065 is mercury fulminate. P092 is phenylmercuric acetate, which is used as a bactericide in latex paint formulations. U151 is mercury. D009 wastes exhibit the characteristic of toxicity as measured by the toxicity characteristic leaching procedure (TCLP).

EPA classifies hazardous wastes as either wastewaters or nonwastewaters. Wastewaters are wastes that contain less than 1 wt % suspended solids and less than 1 wt % total organic carbon. Wastes not meeting this definition are nonwastewaters. For all wastewater forms of K106, P065, P092, and U151, EPA promulgated a treatment standard of 0.030 mg/L based on the performance of sulfide precipitation treatment of K071 wastewaters. K071 is a brine purification mud from the mercury-cell process for chlorine production. For D009 wastewaters, EPA adopted the characteristic level of 0.2 mg/L.

For nonwastewater forms of D009, K106, P065, P092, and U151, EPA established two subcategories: high mercury, over 260 mg/kg, and low mercury. For nonwastewater forms of K106 and U151 in the high-mercury subcategory, EPA promulgated a treatment standard of retorting/roasting. Residues from this process can be land-disposed provided they meet a leaching standard of 0.20 mg/L as measured by TCLP. For nonwastewater forms of K106 and U151 that are not residues from retorting/roasting and are in the

MIXED WASTE INTEGRATED PROGRAM TECHNOLOGY AREAS

Fig. 1. MWIP technology areas.

low-mercury subcategory, EPA promulgates a treatment standard of 0.025 mg/L based on the BDAT for K071 nonwastewaters of acid leaching followed by sulfide precipitation.

The treatment standard for D009 high-mercury nonwastewaters is expressed as either retorting, roasting, or incineration because these wastes may contain inorganic mercury constituents or organomercury compounds. Incinerator nonwastewater residues in the high-mercury category must be retorted or roasted prior to disposal. Nonwastewater residues from retorting or roasting are eligible for land disposal provided they meet the standard of 0.20 mg/L. For D009 low-mercury nonwastewaters, EPA has promulgated a treatment standard of 0.20 mg/L.

Information provided to EPA by DOE indicated that the BDAT technologies and standards for nonradioactive wastes were not applicable to two particular D009 mixed wastes. One is elemental mercury contaminated with tritium. The BDAT EPA promulgated for this waste is amalgamation with zinc, and the treatment standard is amalgamation with zinc as a method of treatment. The second is hydraulic oil contaminated with mercury and tritium. Incineration is the BDAT for this waste, with nonwastewater incineration residues required to comply with a TCLP mercury standard of 0.20 mg/L and with incineration scrubber waters required to meet the 0.20-mg/L total concentration mercury standard.

2. INVENTORIES FROM SAMPLE DOE SITES

To provide a realistic picture of the varieties of mercury-containing mixed wastes that must be treated, inventories for four major sites were obtained from the Hazardous Waste Remedial Actions Program (HAZWRAP).³ The items obtained from HAZWRAP were condensed by listing for each waste item only (a) EPA codes, (b) whether remote or contact-handled, and (c) volumes. These are tabulated in Appendix A. The HAZWRAP inventories contained very little information on mercury-containing mixed wastes at the Y-12 site. These wastes will be a significant source. Most K-25 site entries did not include volumes. Volumes were summed by grouping entries into five categories: (1) aqueous liquids and sludges; (2) combustible liquids; (3) noncombustible, nonaqueous liquids; (4) combustible solids; and (5) noncombustible solids. These volumes are presented in Table 1. The volume of aqueous liquids and sludges is the largest, but this category includes first-cycle fuel-reprocessing wastes, which are intensely radioactive. These wastes will probably be vitrified for permanent storage without mercury separation. This category also contains low-level evaporator service tank sludges and mercury-contaminated heavy water. Combustible liquids are typically cutting oils, lubricating oils, and solvents. An example of a noncombustible, nonaqueous liquid is metallic mercury, which is contaminated with various radioactive species, including tritium. Combustible solids are mostly rags and wipes. Noncombustible solids are highly variable; they include such items as gold traps, lead bricks, and fluorescent light bulbs.

Table 1. Inventory of mercury-containing mixed wastes at sample DOE sites

Site	Aqueous liquids and sludges (M ³)	Liquids		Solids	
		Combustible liquids (m ³)	Noncombustible, nonaqueous liquids (m ³)	Combustible solids (m ³)	Noncombustible solids (m ³)
Savannah River	119,650	12.7	0.18	7240	213
Rocky Flats	0.21	110			21.8
Richland	213,870	0.68	1.0	4.43	105.8
Oak Ridge	1,793	19.0	1.1		681
K-25 ^a	?	?		?	76.2+?

^aFormerly, the Oak Ridge Gaseous Diffusion Plant, Oak Ridge, Tennessee.

3. TREATMENT TECHNOLOGIES

A total of 19 treatment technologies were found. A summary list of these is presented in Table 2. Technology evaluation sheets for each of these are given in Appendix B. The sheets briefly describe the respective technology and then place it in one of three status classes: (1) accepted, if it is fully developed and commercially available; (2) demonstration, if the technology has been developed on an engineering scale but is not yet commercially available; and (3) predemonstration, if the technology is at the conceptual to bench-scale level of development. The evaluation sheets also list (1) the science and technology work needed to bring the technology to the accepted level for DOE wastes (2) documentation, or (3) literature references to the technology.

The treatment technologies for solids were designed to separate mercury from solids into liquid or gas from which the mercury can then be concentrated into a small volume by the technologies listed under "aqueous liquids" and "gases." (See Appendix B.) The mercury must be separated from radionuclides at some point. This process might be achieved either when the mercury is separated from the solid phase or when the mercury is subsequently removed from the liquid or gas phase. The only available technology suitable for combustible solids is incineration. Noncombustible solids might be acid-leached or thermally treated at temperatures up to 600°C, depending on the composition of the solids and mercury speciation. Some relevant properties of mercury and its compounds are presented in Table 3, which shows that most mercury compounds volatilize or decompose below this temperature.

Processes leaching or dissolving the mercury species away from a solid matrix must contend with peculiar mercury chemistry. Only a few mercurous compounds can be formed. The halides comprise most of these. The fluoride is unstable when combined with water, and the other halides are highly insoluble. Mercurous nitrate, which is very soluble, is known only as the dihydrate. When dried, it becomes explosive. Most mercuric salts are only moderately to very slightly soluble in water. An unusual characteristic is that they are bonded covalently rather than ionically, and they become only slightly dissociated in solution. The mercuric halides form anionic soluble complexes, which are soluble in excess of the corresponding halide, HgX_4^{--} .

Aqueous sludges are listed with noncombustible solids in Table 2 because the same set of possible technologies is applicable to both. EPA established roasting and retorting as the BDAT for high-mercury nonwastewaters except for radioactive mixed wastes, for which BDATs have not yet been proposed. Thermal treatment appears likely to be a strong candidate for many mixed wastes because many physical forms can be handled in the same process equipment. Acid leaching of the solids to dissolve mercury species may be a useful technology provided the nonradioactive constituents and the nonchemically hazardous constituents are not also dissolved. Acid leaching is the BDAT for K071 nonwastewaters. A commercial process for K106 nonwastewaters using a sulfuric acid-sodium hypochlorite leach is the Innochem process.

The use of solutions containing "active" chlorine or "elemental" chlorine to produce soluble mercury chloride anionic complexes is established in two patents.^{4,5} A patent application has been filed by the General Electric Company (GE) for a process using a KI/I_2 solution to oxidize and form soluble mercury iodide complexes.⁶ Tests on a synthetic soil matrix dosed with metallic mercury and several mercury compounds including

oxides, chlorides, sulfide, phosphate, nitrate, and methyl mercury chloride gave excellent separation.

Table 2. Technologies for mercury separation

Waste	Technology
Combustible solids	Combustion and mercury separation from gas
Noncombustible solids and aqueous sludges	Acid leach and mercury separation from leach solution
	Thermal treatment and mercury separation from gas
	Chlorination to HgCl_2 or HgCl_4 and mercury separation from liquid
	Contact with aqueous sulfide solution to form soluble mercury polysulfide
Organic liquids	Combustion and removal from gas
Aqueous liquids	Activated carbon impregnated with sulfur
	Borohydride reduction and filtration of mercury
	Sulfide precipitation and filtration of mercuric sulfide
	Ion exchange
	Extraction with liquid organic amines
	Crown ethers in membranes or beads
	Chelating resins (e.g., isothiuronium group)
Gases	Sorption on biomedica
	Cementation (amalgamation)
	Activated carbon impregnated with sulfur (Mersorb)
	Polysulfide resin (Phillips Petroleum)
	Boliden-Norzinc process (chlorine reaction)

Table 3. Selected properties of mercury and some mercury compounds^a

Compound	$\Delta G_{\text{formation}}$ (kcal/mol)	Solubility (g/L)	Boiling or sublimation temperature °C
Hg	0	6×10^{-5}	356.9
HgO	-13.9	0.052	d ^b 476
Hg ₂ O	-12.8	Insoluble	d 100
HgS	-8.8	1×10^{-5}	sub ^c 583
Hg(C ₂ H ₃ O ₂) ₂	-139.2	250	d
HgBr ₂	-9.7 (aq)	5	322
Hg ₂ Br ₂	18	7×10^{-8}	sub ^c 345
HgCl ₂	-23.2(aq)	6.7	304
Hg ₂ Cl ₂	14	4×10^{-4}	383.7
Hg ₂ (NO ₃) ₂ •2H ₂ O	-13	Very soluble	expl ^d
Hg ₂ I ₂	23	2×10^{-7}	sub 140 d 310
HgI ₂	-	0.06	350
Hg ₂ SO ₄	-149	0.4	d
Hg ₂ F ₂	-	d	Sub 240
HgF ₂	-	d	647

^aCompiled from various handbooks.^bd = decomposes.^csub = sublimes.^dexpl = explodes.

For aqueous liquids, several sorbents and ion-exchange technologies can be considered. Commercially available sorbent technologies are the activated carbon pellets impregnated with sulfur (Mersorb™) and ion-exchange resins containing thiol groups such as Ionac® SR-4. If commercial suppliers of chelating resins are available, this technology would be available in the short term. Mersorb™ can be used over a fairly broad pH range, from about 10.5 down to a pH of 3 to 4. It will sorb other metals that are disposed to form sulfides. If nontoxic metals are present in the liquid waste in high concentrations, the use of Mersorb™ may be expensive. The isothiuronium resin degrades at an alkaline pH but the resin can be used at an acidic pH down to 1. The thiol resin can be used for alkaline solutions, provided the mercury is in cationic form.

Mercury can be removed from aqueous liquids by borohydride reduction to the metallic form. This technology has a history of practice for gold and silver recovery. A commercial reagent is available. Borohydride reduction was developed by Merck & Company, Inc., to remove mercury from an organomercury-containing liquid waste.⁷ The method is not specific to mercury; consequently, group VIII metals including

ruthenium and above would also be removed: These include rhodium, palladium, osmium, iridium and platinum. If ruthenium is present in the liquid to be treated, borohydride reduction is not suitable. An effort is underway by the Chlorine Institute to make borohydride reduction the BDAT for mercury cell wastewaters.

A technology with unique potential advantages for treatment of aqueous liquid streams is the 3M membrane combined with IBC sequestering agents. This technology is currently under investigation through Battelle Pacific Northwest Laboratories for cesium and strontium removal. IBC Advanced Technologies has developed a system of making highly selective, nonion exchange, organic ligands chemically bonded to solid supports such as silica particles. The 3M Corporation has developed methods for incorporating these particles into matrixes resulting in Teflon™ membranes that are highly porous to afford very high flow rates. This technology is promising for applications in which highly selective removal of mercury may be required from an aqueous stream.

Only three technologies were found for mercury separation from gases. These do not include condensing-scrubbing, which would be used to cool gases from incineration or thermal treatment and absorb soluble species. The solutions resulting from the condensing-scrubbing operation would be treated for mercury removal by the methods listed under "aqueous liquids." Gases leaving a condenser or scrubber at temperatures below 100°C would carry extremely small amounts of mercury species other than metallic mercury. For example, an Antoine extrapolation of available vapor pressure data for mercuric chloride (corrosive sublimate) down to 100°C gives a value around 10^{-5} mm Hg. This converts to about 0.1 µpm for one of the most volatile of the mercury salts.

The Mersorb technology listed for treatment of aqueous liquids is also used for gases. To prevent oxidation of the sulfur in the pellets, temperatures are limited to 100°C or below in oxygen-containing gases. The gas humidity in the pellet beds must be maintained below 90% to prevent condensation of water in the pellet voids. Vendors' literature gives a rule-of-thumb minimum contact time of 10 s.

Two other technologies listed for gases are a patented polysulfide resin and the Boliden-Norzinc process. The resin was patented in 1986, and no information was found indicating whether the technology was commercialized. The commercial availability of this resin and its performance relative to Mersorb are not known. The Boliden-Norzinc process is the base-line process for the mixed waste treatment project.

REFERENCES

1. *Mixed Waste Treatment Project Process Systems and Facilities: Design Study and Cost Estimates*, Bechtel Corporation, September 1992.
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3. L. Wachter, Martin Marietta Energy Systems, Inc., Oak Ridge, Tenn., personal communication.
4. E. Wygasch and G. Weiss, U.S. patent 3,755,110; August 28, 1973; assigned to Badische Anilin-& Soda-Fabrik AG, Germany.
5. G. A. Carlson, U.S. patent 3,785,942; January 15, 1974; assigned to PPG Industries, Inc.
6. D. F. Foust, "Extraction of Mercury and Mercury Compounds from Contaminated Material and Solutions;" U.S. patent application 07/963,225; October 15, 1992; assigned to General Electric Company.
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Appendix A

INVENTORIES FROM SAMPLE DOE SITES

Savannah River Site

Aqueous Liquids/Sludges:

SRS 1	221-F Canyon High Level Liquid Waste EPA codes D002 D005 D007 D008 D009 D011 Remote-handled HLW 54,719 m ³
SRS 2	221-H Canyon High Level Liquid Waste EPA codes D002 D005 D007 D008 D009 D011 Remote-handled HLW 64,825 m ³
SRS 3	Mercury-Contaminated Heavy Water EPA codes D009 Contact-handled LLW 8.95 m ³
SRS 4	SRL High Activity Waste EPA codes D002 D007 D008 D009 D018 Remote-handled LLW 71.1 m ³
SRS 5	SRL Low Activity Waste EPA codes D002 D007 D008 D009 D018 Remote-handled LLW 25.6 m ³

Combustible Liquids:

SRS 6	Tritiated Oil from Tritium Prod Facility EPA code D009 Contact-handled LLW 12.7 m ³
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Non-Combustible, Nonaqueous Liquids:

SRS 7	Tritiated Mercury EPA code D009 Contact-handled LLW 0.182 m ³
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Combustible Solids:

SRS 8	Solvent Contaminated Applicators (LLW) EPA codes D004 through D043 except D0016, F001 F002 F003 F005 LLW 2.5 m ³
SRS 9	Solvent TRU Waste EPA codes D001-D004 D006-D009 D011 D018 D019 D022-D027F001-F003 F005 P012 P015 P048 P113 P120 U002 U032 U052 U080 U122 U134 U144 U151 U154 U161 U209 U211 U220 U226 U239 Contact-handled TRU 3583 m ³
SRS 10	Solvent Waste <100 nCi/gm TRU Rad EPA codes D001-D004 D006-D009 D011 D018 D019 D022-D026 F001-F003 F005 P012 P015 P048 P113 P120 U002 U032 U052 U080 U133 U134 U144 U151 U154 U161 U209 U211 U220 U226 U239 Contact-handled TRU 3629 m ³
SRS 11	Thirds TRU Waste EPA codes D001-D004 D006-D009 D011 D018 D019 D022-D026 P012 P015 P048 P113 P120 U002 U032 U052 U080 U133 U134 U144 U151 U154 U161 U209 U211 U220 U226 U239 Contact-handled TRU 20.6 m ³
SRS 12	Thirds Waste <100 nCi/gm TRU Rad EPA codes D001-D004 D006-D009 D011 D019 D022-D026 P012 P015 P048 P113 P120 U002 U032 U052 U080 U133 U134 U144 U151 U154 U161 U209 U211 U220 U226 U239 Contact-handled LLW 8.1 m ³
SRS 13	Toxic Cleanup Material EPA codes D004-D011 Contact-handled LLW 0.2 m ³

Non-Combustible Solids:

SRS 14	Gold Traps EPA code D009 Contact-handled LLW 1.00 m ³
SRS 15	ITP Filters EPA codes D009 D018 Remote-handled LLW (anticipated)
SRS 16	LLW Lead EPA code D008 Remote-handled LLW 212 m ³

Rocky Flats Plant

Aqueous Liquids/Sludges:

RFP 1	Wastewater Containing Sodium Carbonate EPA codes D002 D009 F001 F005 Nonradioactive 0.21 m ³	RFP 1141
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Combustible Liquids:

RFP 2	FBI Oil EPA codes D001 D007 D009 D019 D022 F001-F003 F005 LLW 109.7 m ³	RFP 1013
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Non-Combustible, Nonaqueous Liquids:

none

Combustible Solids:

none

Non-Combustible Solids:

RFP 3	Fluidized Bed Incinerator Ash (TRU) EPA codes D004-D009 D0101? D011 F001-F005 TRU 1.033 m ³	RFP 1080
RFP 4	Fluorescent Light Bulbs EPA code D009 Nonradioactive 12.48 m ³	RFP 1081
RFP 5	Glass/LLW Mixed EPA codes D008 D009 Contact-handled LLW 6.21 m ³	RFP 1085
RFP 6	Ground Glass/LLW Mixed EPA codes D008 D009 LLW 1.91 m ³	RFP 1090
RFP 7	Ground Glass/TRU Mixed EPA codes D008 D009 TRU 0.208 m ³	RFP 1091

Richland Site

Aqueous Liquids/Sludges:

RS 1	DST Complex Concentrate EPA codes D001-D011 F003 F005 Remote-handled LLW 11,061 m ³
RS 2	DST Dilute Complexed Waste EPA codes D002 D004-D011 F003 F005 Remote-handled LLW 3410 m ³
RS 3	DST Double Shell Slurry EPA codes D001-D011 F003 F005 Remote-handled LLW 7880 m ³

RS 4	DST Double Shell Slurry Feed EPA codes D002 D004-D011 F003 F005 Remote-handled LLW 15,754 m ³
RS 5	DST Miscellaneous LLW EPA codes D002 D004-D011 F003 F005 Remote-handled LLW 36,261 m ³
RS 6	Single Shell Tank Wastes EPA codes D002 D005-D011 F003 HLW 139,500 m ³

Combustible Liquids:

RS 7	Appendix IV Labpacks (CA,Hg) EPA codes D001 D002 D006 D008 D009 Contact-handled LLW 0.68 m ³
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Non-Combustible, Nonaqueous Liquids:

RS 8	Purex Storage Tunnels Mercury Waste EPA code D009 LLW 0.01 m ³
RS 9	Radioactive Elemental Mercury (CA) EPA code D009 Contact-handled LLW 1.05 m ³

Combustible Solids:

RS 10	TC Metal Org. Solid Debris, TRU (Hg, PCB) EPA Codes D006 D008 D009 Contact-handled TRU 3.17 m ³
RS 11	TC Metal Organic Solid Debris (Hg) EPA Code D009 Contact-handled LLW 0.63 m ³

RS 12 TC Metal Organic Solid Debris, TRU (Hg)
 EPA Codes D005-D009
 Contact-handled TRU
 0.63 m³

Non-Combustible Solids:

RS 13 Lead Acid Batteries, TRU (Hg)
 EPA Codes D002 D005-D009
 Contact-handled TRU
 0.42 m³

RS 14 Mercury Sludges/Dry Part. (Hg>260 mg/kg)
 EPA Codes D002 D009
 Contact-handled LLW
 0.02 m³

RS 15 PNL Hot Cell Analytical Waste
 EPA codes D005-D011 E003 F005
 LLW
 ?

RS 16 Radioactive Lead Solids, TRU (Hg)
 EPA codes D006 D008 D009
 Contact-handled TRU
 0.21 m³

RS 17 Solvent TC Metal Sludges/Dry Part (Hg)
 EPA Codes D001 D009 F003
 Contact-handled LLW
 0.21 m³

RS 18 Solvent/TC Metal Soils (Hg)
 EPA Codes D006-D009 D011 F001-F003 F005
 Contact-handled LLW
 0.21 m³

RS 19 TC Metal Inorg. Glass Solid Debris (Hg)
 EPA Codes D005 D006 D009
 Contact-handled LLW
 4.11 m³

RS 20 TC Metal Inorg. Solid Debris-TRU (Hg,PCB)
 EPA Codes D006 D008 D009
 Contact-handled TRU
 66.18 m³

RS 21	TC Metal Inorganic Solid Debris (Hg) EPA Codes D002 D003 D006 D009 Contact-handled LLW 1.16 m ³
RS 22	TC Metal Inorganic Solid Debris (Hg, PCB) EPA Codes D006 D009 Contact-handled LLW 9.61 m ³
RS 23	TC Metal Inorganic Solid Debris, TRU(Hg) EPA Codes D005-D009 Contact-handled TRU 7.98 m ³
RS 24	TC Metal Sludges/Dry Particulate (CA, Hg) EPA Code D009 Contact-handled LLW 0.21 m ³
RS 25	TC Metal Sludges/Dry Particulate (Hg) EPA Codes D001 D002 D005-D011 Contact-handled LLW 15.51 m ³

Oak Ridge National Laboratory

Aqueous Liquids/Sludges:

ORNL 1	Evaporator Service Tank Sludges EPA Codes D002 D006-D009 Remote-handled TRU 119 m ³
ORNL 2	Inactive Waste Storage Tank Contents-A EPA Codes D002 D007-D009 Contact-handled LLW 1088 m ³
ORNL 3	Inactive Waste Storage Tank Contents-C EPA Codes D006-D009 LLW 62.4 m ³

ORNL 4	Inactive Waste Storage Tank Contents-D EPA Codes D006-D009 TRU 110 m ³
ORNL 5	LLLW Collection Tanks Sludge EPA Codes D002 D006-D009 LLW ?
ORNL 6	Melton Valley Storage Tank Sludges EPA Codes D002 D006-D009 Remote-handled TRU 414 m ³

Combustible Liquids:

ORNL 7	Flammable, EP Toxic Liquids EPA Codes D001 D009-D011 Contact-handled LLW 0.02 m ³
ORNL 8	Solvent Waste EPA Codes D009 F001-F005 Contact-handled LLW 19 m ³

Non-Combustible, Nonaqueous Liquids:

ORNL 9	Mercury (D009) EPA Code D009 Contact-handled LLW 0.4381 m ³
ORNL 10	Mercury (U151) EPA Codes D009 U151 Contact-handled LLW 0.6705 m ³

Combustible Solids:

none

Non-Combustible Solids:

ORNL 11	CH-TRU EPA Codes D008 D009 Contact-handled TRU ?
ORNL 12	EP Toxic Contaminated Solids EPA Codes D004 D006-D011 Contact-handled LLW 0.86 m ³
ORNL 13	TRU Waste EPA Codes D008 D009 Remote-handled TRU 680 m ³

Y-12 Plant

Only one item was listed for the Y-12 Plant, and it has been shipped to the K-25 Site for long-term storage. Hence it is included in this report in the K-25 inventory as K-25 11.

K-25 Site

Aqueous Liquids/Sludges:

K-25 1	Corrosive Liquids (Bulk), Waste ID 142 EPA Codes D002 D007-D011 Contact-handled LLW ?
K-25 2	Electroless Nickel Solution, Waste ID 8 EPA Codes D002 D007 D009 F006 Contact-handled LLW 0.8 m ³
K-25 3	Laboratory Bases, Waste ID 71 EPA Codes D002 D007 D009 D011 Contact-handled LLW ?
K-25 4	TSCA Feed Materials, Waste ID 159 EPA Codes D008 D009 F001-F003 Contact-handled LLW

Combustible Liquids:

- K-25 5 Flammable Liquids (Bulk), Waste ID 138
EPA Codes D001 D004 D006-D011 D018-D043 F001-F003 U002
Contact-handled LLW
?
- K-25 6 Hazardous Waste Liquid (Bulk), WID 60
EPA Codes D004 D006-D010 D012 D018-D043 F001-F003 U131
U226
Contact-handled LLW
?
- K-25 7 Laboratory Acids, Waste ID 70
EPA Codes D001 D002 D006-D010
Contact-handled LLW
?
- K-25 8 Laboratory Organics, Waste ID 72
EPA Codes D001 D002 D004 D006-D010 D018-D043 F001-F003
F005
Contact-handled LLW
?
- K-25 9 Waste Oils, Waste ID 42
EPA Codes D001 D004 D006-D011 D018--D043 F001-F003
Contact-handled LLW
?

Non-Combustible/Nonaqueous Liquids:

none

Combustible Solids:

- K-25 10 Oily Rags and Solids, Waste ID 145
EPA Codes D004 D006-D010 D018-D043
Contact-handled LLW
?

Non-Combustible Solids:

K-25 11	Mercury Contaminated Waste EPA Code D009 Contact-handled LLW 75.2 m ³
K-25 12	Batteries, Waste ID 161 EPA Codes D002 D003 D006-D009 Contact-handled LLW ?
K-25 13	Hazardous Waste Solid Bulk, Waste ID 122 EPA Codes D004-D011 D018-D043 F001-F003 F005 F006 Contact-handled LLW ?

Appendix B

TECHNOLOGY ASSESSMENT SHEETS

MWIP MERCURY CONTROL

MWIP MERCURY CONTROL

Waste Stream Type: Combustible solids.

Technology Description: Combustion, with mercury recovery from gases. The gases would be cooled to below 100°C to reduce any mercury salts to extremely low concentrations and then treated by technologies listed under "Gases."

Technology Status: Accepted. A recent journal publication listed 44 full-scale thermal treatment remediation projects completed or in progress by 17 contractors (see documentation).

Science and Technology Needs: Better characterization of DOE wastes.

Documentation: J. J. Cudahy and W. L. Troxler, "1991 Thermal Treatment Remediation Industry Contractor Survey," *J. Air Waste Manag. Assoc.*, 42, 844-849 (June 1992); E. T. Oppelt, "Incineration of Hazardous Waste. A Critical Review," *JAPCA*, 37, 558-586 (May 1987).

MWIP MERCURY CONTROL

Waste Stream Type: Noncombustible solids and aqueous sludge.

Technology Description: Acid leach followed by mercury separation from leach solution. The Innochem Engineering, Ltd., process for K106 waste is similar; it uses sulfuric acid-sodium hypochlorite leach followed by iron cementation to recover the mercury from the leach solution.

Technology Status: Accepted. This is the BDAT for K071 nonwastewaters.

Science and Technology Needs: Better characterization of DOE mercury-containing mixed wastes to determine extent of dissolution of other constituents of solid waste.

Documentation: J. R. Berlow and J. Keenan, *Onsite Engineering Report of Treatment Technology Performance and Operation for Vulcan Materials Company*, U.S. Environmental Protection Agency, January 26, 1988; U.S. Environmental Protection Agency, Office of Solid Waste, *Best Demonstrated Available Technology (BDAT) Background Document for K071*. U.S. Environmental Protection Agency, Washington, D.C., 1988; M. Rockandel, "Non-thermal Processing of K106 Mud," in R. J. Turner and J. Labiosa, "Arsenic and Mercury: Workshop on Removal, Recovery, Treatment and Disposal," EPA/600/R-92/105, U.S. Environmental Protection Agency, August 1992.

MWIP MERCURY CONTROL

Waste Stream Type: Noncombustible solids and aqueous sludge.

Technology Description: Thermal treatment with mercury recovery from gases. The gases would be cooled to 100°C to reduce any mercury salts to extremely low concentrations, and then treated by technologies listed under "Gases."

Technology Status: Accepted. This is the BDAT for high mercury nonwastewaters.

Science and Technology Needs: Better characterization of DOE mercury-containing mixed wastes to determine extent of vaporization of other constituents of solid waste.

Documentation: L. Rosengrant and J. Labiosa, *Final BDAT Background Document for Mercury-Containing Wastes*, EPA/530-SW-90-059Q, U.S. Environmental Protection Agency, May 1990. PB90-234170.

MWIP MERCURY CONTROL

Waste Stream Type: Noncombustible solids and aqueous sludge.

Technology Description: Chlorination, followed by separation of soluble mercury chloride complexes from solution by methods listed under "aqueous liquids."

Technology Status: Demonstration. This technology is the subject of two patents, but it is not known whether full-scale processes were developed. One patent was developed for application to brine filter sludge in chloralkali plants (K071 waste). The other patent was developed for "electrolytic cell caustic filter solids," which may also be K071 waste.

Science and Technology Needs: Thorough characterization of candidate DOE sludges would be required to determine if chlorination would be compatible with other sludge constituents. For example, if ruthenium is present, chlorination would also create soluble ruthenium complexes.

Documentation: E. Wygasch and G. Weiss, U.S. patent 3,755,110; August 28, 1973; assigned to Badische Anilin- & Soda-Fabrik AG, Germany; G. A. Carlson, U.S. patent 3,785,942; Jan. 15, 1974; assigned to PPG Industries, Inc.

MWIP MERCURY CONTROL

Waste Stream Type: Noncombustible solids and aqueous sludge.

Technology Description: Contacting the solids with an aqueous sulfide solution to form soluble mercury polysulfide complexes. The solution must be strongly alkaline. The mercury-containing solids are first treated with a hypochlorite oxidizing agent.

Technology Status: Demonstration. This technology is the subject of one patent. No documentation was found indicating scale-up to commercial operation. It was developed for application to electrolytic cell solids, comprising magnesium hydroxide, ferric hydroxide, and calcium carbonate.

Science and Technology Needs: Thorough characterization of candidate DOE sludges and solids would be required to determine whether the sulfide solution would be compatible with the mercury-containing sludge/solid constituents.

Documentation: W. E. Dean and W. E. Makris, U.S. patent 3,836,441; Sept. 17, 1974; assigned to PPG Industries, Inc.

MWIP MERCURY CONTROL

Waste Stream Type: Noncombustible solids and Aqueous sludge.

Technology Description: Contact with KI/I_2 solution to form soluble mercury iodide complexes.

Technology Status: Demonstration. A patent has been filed by GE describing this separation method followed by an electrolytic membrane process for iodine recovery and recycle.

Science and Technology Needs: Thorough characterization of DOE wastes would be required to determine if other waste constituents would be compatible with iodine chemistry.

Documentation: D. F. Foust, "Extraction of Mercury and Mercury Compounds from Contaminated Material and Solutions," U.S. patent application 07/963,225, Oct. 15, 1992; assigned to General Electric Company.

MWIP MERCURY CONTROL

Waste Stream Type: Organic liquid.

Technology Description: Incineration, with mercury recovery from gases.

Technology Status: Accepted. EPA promulgated incineration as the BDAT for hydraulic oil contaminated with mercury and tritium.

Science and Technology Needs: Better characterization of DOE mercury-containing organic liquids to determine those that might contain constituents making incineration unsuitable.

Documentation: L. Rosengrant and J. Labiosa, *Final BDAT Background Document for Mercury-Containing Wastes*, EPA/530-SW-90-059Q, May 1990. PB90-234170.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Adsorption/reaction of mercury on beads of activated carbon impregnated with sulfur. Contacting is accomplished typically by passing the liquid through columns filled with the beads. Limited to solution pH above 3 or 4 to avoid hydrogen sulfide production.

Technology Status: Accepted. The material is available as "Mersorb," from Nucon International, Inc. A few bench-scale tests were reported in the *ORNL Chemistry Division Annual Report* of 1985. Distribution coefficients of 10^5 to 10^6 L/kg were measured. Vendors' literature gives a rule-of-thumb design throughput of 1 gpm/ft³ of sorbent.

Science and Technology Needs: Engineering development tests are required with actual wastewaters and secondary wastewaters to determine kinetic and equilibrium parameters for design and analysis work.

Documentation: *Oak Ridge National Laboratory Chemistry Division Annual Progress Report for Period Ending January 31, 1985*, ORNL-6152, Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory, Oak Ridge, Tenn., May 1985; Nucon International, Inc., literature of vendors.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Borohydride reduction and filtration. Direct reduction of Group VIII and 1B metals (ruthenium and above) with borohydride occurs across a broad pH range. A preoxidation step with hypochlorite may be required to break mercury complexes. Borohydride reduces Hg^{++} to metal, which is removed by filtration or adsorption. If ruthenium is present, some of it will accompany the mercury.

Technology Status: Demonstration. Currently used commercially to recover silver from spent photographic film and gold from jewelry scrap. Morton International sells a sodium borohydride solution under the VenMet trademark and is working with the Chlorine Institute to develop a process for mercury recovery from K106 wastewaters. The intent is to replace the current BDAT with borohydride reduction. The chlorine-caustic soda industry is the second-largest user (after the battery industry) of mercury.

Science/Technology Needs: Better characterization of DOE wastes to determine mercury speciation and competing ions. For secondary applications, speciation will be known. Mercury metal may be produced in colloidal form, and engineering development may be required to identify optimal means of recovery.

Documentation: R. S. Stennick et al., pp. 325-327 in *Proc. 46th 1991 Industrial Waste Conference*.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Precipitation of mercuric sulfide. The extremely low solubility of mercuric sulfide in neutral to acidic solutions is the basis for this technology as the BDAT for K071 wastewaters. Mercuric sulfide is soluble in strongly alkaline sulfide solutions.

Technology Status: Accepted. This is the BDAT for K071 wastewaters.

Science and Technology Needs: Better characterization of DOE mercury-containing mixed wastes to determine if sulfide precipitation will separate mercury from other constituents of the waste.

Documentation: L. Rosengrant and J. Labiosa, *Final BDAT Background Document for Mercury-Containing Wastes*, EPA/530-SW-90-059Q, U.S. Environmental Protection Agency, May 1990. PB90-234170.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Ion exchange. Resin containing the iminodiacetic acid group will pick up cationic mercury selectively from calcium and magnesium, but copper and cobalt are also picked up. Mercury in the form of anionic complexes (e.g., HgCl_3^- or HgCl_4^-) can be picked up by anion exchange resins but not selectively. The Savannah River Plant has used a thiol resin, Duolite GT-73, which is very selective for mercury in the cationic form. The resin was effective in both dilute waste streams and those containing concentrated salt. Regeneration was not recommended. Another thiol resin available commercially is Ionac SR-4.

Technology Status: Accepted. A vast variety of ion-exchange separations are carried out commercially.

Science and Technology Needs: Better characterization of DOE wastes to identify waste stream compositions that are amenable to ion-exchange separations.

Documentation: F. X. McGarvey, "Mercury Removal with Ionac Ion Exchange Resins," in *Arsenic and Mercury: Workshop on Removal, Recovery, Treatment and Disposal*, ed. R. J. Turner and J. Labiosa, EPA/600/R-92/105, August 1992. J. Bibler, R. Wallace, and M. Ebra, "Mercury Removal from SRP Radioactive Waste Streams Using Ion Exchange," p. 471 in *Waste Management, High Level Waste*, Vol. 2, 1986.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Extraction with liquid organic amines. In bench-scale tests, quaternary amines (e.g. Aliquat 336-S and Adogen-464) extracted mercury from both alkaline and acidic brine solutions.

Technology Status: Demonstrated. This technology is the subject of one patent. No documentation was found that indicates a scale-up for process operations. The patent does not indicate the area of potential application.

Science and Technology Needs: Further research is required to learn more about limiting conditions and selectivity of the process.

Documentation: F. L. Moore, U.S. patent 3,785,803, Jan. 15, 1974; assigned to U.S. Atomic Energy Commission; F. L. Moore, "Solvent Extraction of Mercury from Brine Solutions with High-Molecular-Weight Amines," *Sci.*, 6, 525 (June 1972).

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Crown ethers attached to silica particles embedded in a coherent fibrous matrix membrane. 3M proprietary process under the Empore trademark. Crown ethers can remove Hg^{++} and methyl mercury according to 3M. Use of 5- to 10- μm size particles gives extremely high surface area in a controlled porosity membrane. Membrane matrix is PTFE (teflon). The Empore membrane has the potential to accomplish better separations than packed columns in a much more compact apparatus. As with all membrane processes, feed solution must be clean.

Technology Status: Predemonstration. Development is at the bench-scale level for mercury separation applications. Battelle PNL and IBC Advanced Technologies, Inc working with 3M to develop an Empore membrane for strontium and cesium removal. EPA has allowed five test methods involving Empore extraction disks with seven more pending (all for organic compounds).

Science and Technology Needs: Mercury separation applications just recently targeted by 3M. Development might be accelerated through a CRADA.

Documentation: Vendor's literature.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Chelating resins. Processes using these resins would be similar to conventional ion-exchange operations, except chelating resins either cannot be regenerated or are regenerated only with some difficulty. A resin containing an isothiuronium group was studied in 1971 for removal of both Hg^{2+} and CH_3Hg^- . The selectivity in the presence of dissolved iron, nickel, copper and zinc at concentrations up to 10 g/L was complete. A second account of a chelate resin application was found in a U.S. patent assigned to a Japanese company.

Technology Status: Demonstration. The isothiuronium resin may have been used commercially for gold and platinum recovery, but it is no longer available from the original manufacturer (Ionac Chemical Company). No documentation was found indicating scale-up of the Japanese process.

Science and Technology Needs: A vendor survey is required to determine if chelating resins are available for mercury recovery operations. Uptake tests may be required. Better characterization of DOE wastes is needed to determine mercury speciation and competing ions.

Documentation: S. L. Law, "Methyl Mercury and Inorganic Mercury Collection by a Selective Chelating Resin," *Sci.*, **174**, p. 285 (1971). K. Motani, et al., U.S. patent 3,790,535; February 5, 1974; assigned to Tokuyama Soda KK, Japan. F. McGarvey and E. Hauser, "Removal of Mercury from Industrial Wastes as Well as from Potable Water," in *Proc. Sixth International Conference on Chemistry for the Protection of the Environment*, September 15-18, 1987, Torino, Italy.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Adsorption on biomedial. A patented immobilized algal biomass sorbent material is available commercially in bead form for packed-column operations.

Technology Status: Predemonstration. Material is available from Biorecovery Systems, Inc. Uptake tests were done on Y-12 site groundwaters, giving a relatively low equilibrium loading of 0.0001 mol/kg of sorbent.

Science and Technology Needs: Uptake experiments are required to provide data for pilot-scale tests. Better characterization of DOE wastewaters is needed to determine mercury speciation and competing ions.

Documentation: B. Volesky, Ed., *Biosorbents and Biosorption Recovery of Heavy Metals*, CRC Press, 1990.

MWIP MERCURY CONTROL

Waste Stream Type: Aqueous liquid.

Technology Description: Cementation. More active metals (e.g., aluminum, copper, iron, zinc) will reduce mercury to metallic form as they are dissolved. In the Innochem process, ground steel scrap is used in a rotary contactor.

Technology Status: Accepted. This technology has been used in the mining industry for many years.

Science and Technology Needs: Mercury reduced and removed from solution is replaced by the more active metal. Potential applications of this technology must be compatible with increased concentrations of active metal ions. Tests are required to determine whether EPA concentration standards can be met.

Documentation: R. Perry, *Mercury Recovery from Contaminated Wastewater and Sludges*, Report 660/2-74-086, U.S. Environmental Protection Agency, December 1974; P. F. Waltrich, "Apparatus for the Removal of Mercury from Effluent Streams," *Chem. Abstr.* 78, 235 (1973); R. F. Gerow and S. B. Soule; U.S. patent 3,802,910, April 9, 1974; assigned to GAF Corporation.

MWIP MERCURY CONTROL

Waste Stream Type: Gas.

Technology Description: Adsorption and reaction of mercury in beads of activated carbon impregnated with sulfur. Contacting is done typically by passing gases through columns packed with the beads. Temperature limited to 100°C in oxygen-containing environment. Relative humidity must be below 90% to prevent condensation in pores.

Technology Status: Accepted. The sorbent is commercially available as Mersorb, from Nucon International, Inc. The Vendors' literature gives recommended minimum contact time of 10 seconds.

Science and Technology Needs: Specification of thermal treatment parameters.

Documentation: Vendors' literature.

MWIP MERCURY CONTROL

Waste Stream Type: Gas.

Technology Description: Polysulfide resin. This is the subject of a U.S. patent. The resin is produced by contacting sodium polysulfide with a strongly basic anion-exchange resin, resulting in a sorbent having about 8–10% sulfur content. The patent claims the sorbent is useful in removing mercury from gases at low concentration levels typified by natural gas.

Technology Status: Demonstration. No documentation has been found indicating scale-up to commercial operation.

Science and Technology Needs: A commercial source of the resin is not known. Uptake tests to determine kinetic and equilibrium parameters might be required.

Documentation: R. L. Horton, U.S. patent 4,591,490, May 27, 1986; assigned to Phillips Petroleum Company.

MWIP MERCURY CONTROL

Waste Stream Type: Gas.

Technology Description: Boliden-Norzinc process. Mercury-containing gases are scrubbed with a solution of mercuric chloride that reacts to form mercurous chloride, which has a very low solubility. Some mercurous chloride is purged from the system, equivalent to the mercury scrubbed from the gases. The remainder is reacted with chlorine gas to produce mercuric chloride solution.

Technology Status: Accepted. This process was selected by Bechtel for the Mixed Waste Treatment Project.

Science and Technology Needs: Concentrations of other constituents of the gases which might react with mercurous/mercuric chloride.

Documentation: *Mixed Waste Treatment Project Process Systems and Facilities: Design Study and Cost Estimates for LLNL, September 1992.*

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