

TREATMENT METHODS FOR RADIOACTIVE MIXED WASTES  
IN COMMERCIAL LOW-LEVEL WASTES - TECHNICAL CONSIDERATIONS\*

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ABSTRACT

Treatment options for the management of three generic categories of radioactive mixed waste in commercial low-level wastes (LLW) have been identified and evaluated. These wastes were characterized as part of a BNL study in which LLW generators were surveyed for information on potential chemical hazards in their wastes. The general treatment options available for mixed wastes are destruction, immobilization, and reclamation. Solidification, absorption, incineration, acid digestion, wet-air oxidation, distillation, liquid-liquid solvent extraction, and specific chemical destruction techniques have been considered for organic liquid wastes. Containment, segregation, decontamination, and solidification or containment of residues, have been considered for lead metal wastes which have themselves been contaminated and are not used for purposes of waste disposal shielding, packaging, or containment. For chromium-containing wastes, solidification, incineration, wet-air oxidation, acid digestion, and containment have been considered. For each of these wastes, the management option evaluation has included an assessment of testing appropriate to determine the effect of the option on both the radiological and potential chemical hazards present.

INTRODUCTION

Background

The disposal of low-level radioactive waste (LLW) is regulated by the U.S. Nuclear Regulatory Commission (NRC) under 10 CFR Part 61.<sup>(1)</sup> Concerns have emerged regarding the applicability of U.S. Environmental Protection Agency (EPA) regulations and permit requirements, under the Resource Conservation and Recovery Act (RCRA), to the potentially hazardous chemical content of some LLW, and the appropriate methods for managing such wastes. In order to establish a data base on their quantities and characteristics, Brookhaven National Laboratory (BNL) conducted a limited survey of reactor and non-reactor waste generators.<sup>(2)</sup> Based on information obtained in this survey, low-level wastes which are potentially hazardous under EPA regulations (hereafter referred to as "mixed wastes") have been identified. A subsequent study<sup>(3)</sup> included a technical evaluation of treatment options for managing these potential mixed wastes, and forms the basis for this paper. A companion paper, "Management of Radioactive Mixed Wastes in Commercial Low-Level Wastes," (presented in Session IV of "Waste Management '86") emphasizes regulatory concerns, presenting an outline of the pertinent NRC and EPA similarities and differences, an overview of potential inconsistencies or data gaps, and several unresolved issues and possible solutions.

BNL Survey Results

The mixed wastes identified in the BNL survey included organic liquid, lead, and chromium-containing wastes. Details of their characteristics have been given in the BNL survey report<sup>(2)</sup> and also are being presented in a Waste Management '86 paper by B. S. Bowerman et al. entitled, "Identification of Radioactive Mixed Wastes in Commercial Low-Level Wastes."

Inclusion of annual generation total values for LLW and for EPA-hazardous wastes will aid in putting the mixed waste amount in perspective. The radioactive organic liquid category of LLW is the largest mixed waste category (2.3% by volume of total LLW) identified in the BNL survey.<sup>(2)</sup> Total annual commercial LLW generation represents =0.04% (by weight) of the annual EPA-hazardous waste total.<sup>(4)</sup> This means mixed waste comprises a few parts in a million of total EPA-hazardous waste.

TREATMENT OPTIONS

The treatment options for management of mixed waste can be categorized as destruction, immobilization, and recovery and reclamation. For lead metal and chromium-containing wastes, destruction by itself is not a possible management option. The discussion that follows will first consider destructive methods as applied to organic wastes. Following that, immobilization and recovery methods will be given as applied to all of the potential mixed wastes.

Destructive Methods

The destructive processes of incineration, acid digestion, wet-air oxidation and specific chemical destruction techniques are applicable essentially only to organic materials. They have an important advantage over other types of treatment in that they either chemically destroy the components of concern to EPA (e.g., scintillation wastes), or accomplish relatively large volume reduction.

Incineration is a process of high-temperature oxidation (thermal decomposition/combustion) whose products include CO<sub>2</sub>, H<sub>2</sub>O, SO<sub>x</sub>, NO<sub>x</sub> and hydrogen halides for sulfur-, nitrogen- and halogen-containing wastes, and metal oxides for inorganic components of the waste. It is the only general process applicable to all organic liquid wastes, and is suitable also for organic ion-exchange resin waste. The wastes must be well characterized so that their expected chemical behavior, interactions with other wastes, and heat of

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combustion are known. Capital cost and cost of set-up, installation, and testing can be high; however, incineration should be cost-effective for large volume, low radioactivity, known composition wastes. The process results in destruction of the original waste chemical hazard and a consequent substantial waste volume reduction. Gases and, occasionally, aerosols are emitted and must be monitored for radioactivity. The incinerator ash residues will normally present no chemical hazard unless, for example, they contain an EPA-listed heavy metal.

Acid digestion involves dehydrogenation and/or dehydration of organic compounds by concentrated  $H_2SO_4$  at elevated temperatures ( $\approx 250^\circ C$ ) followed by oxidation of the resulting carbon to CO and  $CO_2$  with  $HNO_3$  or  $H_2O_2$ . The severity of these conditions requires special materials of construction for the equipment. The resulting high cost, coupled with the hazardous nature of the reagents, tends to make the process unattractive. It has been applied successfully to ion exchange resin wastes both in the U.S. and other countries, but as far as is known no generators or burial site operators in the U.S. presently use the process to treat LLW. Results of work with liquid organic waste streams<sup>(5)</sup> indicate that considerable development would be required to make the process suitable for essentially complete destruction of volatile organic liquids.

Wet-air oxidation, used for decades in treating a wide variety of aqueous industrial wastes containing dissolved or suspended organics, is a combustion process which converts the organics (ideally) to  $CO_2$  and  $H_2O$ . The aqueous waste is heated to  $200-250^\circ C$  and contacted with compressed air or oxygen at sufficient pressure (600-700 psi) to maintain the water in the liquid phase. While applicable to organic ion-exchange resin waste, wet-air oxidation would be a feasible treatment for only a limited and highly selective group of organic liquid wastes. Since the destructive oxidation reaction occurs almost entirely in the condensed phase, the process is not applicable to volatile liquids which are insoluble in water, such as benzene, toluene and paraffinic hydrocarbons. A recently developed catalyzed process has permitted destruction of a number of compounds previously found difficult or impossible to treat adequately, such as acetic acid and PCBs. The lower temperatures used in the catalyzed system might permit treatment of certain relatively volatile wastes, but this would have to be demonstrated for each particular waste.

Specific laboratory destruction techniques will presumably not be applicable to large volume wastes. However, certain wastes may be suited to this treatment, particularly small quantities of known composition or wastes that are produced on a non-routine basis. The latter have been classed in the BNL study as miscellaneous wastes and include off-specification or outdated radioisotope-labeled reagent chemicals and side-product wastes from specific reactions used in production of radioisotope-labeled compounds. A number of detailed procedures are outlined in Reference 6 for chemical destruction of a wide variety of organic, inorganic, and organo-metallic compounds. Following application of the destruction methods outlined, and given that no potentially hazardous material (e.g., heavy metal) is in the (radioactive) residue, it may be disposed of according to the guidance of 10 CFR Part 61.

#### Immobilization Methods

Sorption of liquids by diatomaceous earth, vermiculite, clay products, and natural/artificial

fiber products is considered solidification by members of the chemically hazardous waste-handling community. This is not the case for those involved with LLW, since solidification is differentiated from absorption in 10 CFR Part 61; Section 61.56 (a)(2) states that liquid waste must be either solidified, or packaged in sufficient absorbent material to absorb twice the volume of the liquid. Immobilization by solidification, sorption or containment in a high integrity container (HIC) are applicable to the liquid organic mixed wastes identified, as well as to residues resulting from application of destructive or reclamation processes.

#### Solidification

Solidification, according to the meaning of 10 CFR Part 61, refers to fixation or immobilization of either liquids or dispersible solids in a solid monolithic waste form. The solidification agent or binder most commonly used for LLW is Portland cement. Other agents are bitumen, thermosetting polymers such as vinyl ester-styrene (VES), and inorganic cements other than Portland. The latter include the proprietary materials, Delaware Custom Material (DCM) and Envirostone. Glass is not only the choice for immobilization of HLW in the U.S. but has also been investigated as a waste form for LLW.

With proper care, direct solidification of organic liquid wastes should be possible with Portland and other inorganic cements. However, waste loading would be low, leading to a relatively large volume increase of the waste. Waste oil, an organic liquid waste, has now been proposed by EPA as a hazardous waste.<sup>(7)</sup> Portland cement and DCM have been used to make structurally acceptable waste forms with simulated oil waste over a reasonable range of oil concentrations.<sup>(8)</sup> Up to 35 weight percent oil was incorporated in Portland cement concretes and roughly 20 weight percent in DCM, with or without the use of sorbent. Other work with Portland cement<sup>(9)</sup> and a different oil indicated much lower amounts of oil could be held, so that waste form formulations should be carefully tested for each type of oil being disposed of.

The proprietary Envirostone process has been investigated as a method for solidifying both vacuum pump oil and organic solvents without added sorbent.<sup>(9)</sup> Acceptably stable forms could be prepared using the process, although the waste loading had to be kept relatively low, as with Portland cement and DCM. On the basis of the investigations carried out with oil,<sup>(8-10)</sup> as well as the organic solvent work with the inorganic cement Envirostone,<sup>(9)</sup> it appears probable that solvents can be incorporated in Portland cement and DCM either with or without sorbents.

Solidification of organic liquid waste will likely require a certain amount of development work for specific waste mixtures. It also has the disadvantage of increasing rather than reducing the volume of waste for ultimate disposal. On the other hand, residues from destruction of organic liquid wastes (e.g., incinerator ash) can be solidified at relatively high waste loadings in any of the NRC-approved solidification agents, including bitumen.

A glass furnace process developed at Mound Laboratories combines destruction and immobilization in a single step.<sup>(11)</sup> In it, organic LLW is incinerated over molten glass so that solid residue containing the radioactivity (and any chemically hazardous constituents) falls into the glass pool and is solidified in a high quality glass waste form when the

melt is cooled. The process can be applied to organic liquids as well as to solid organics such as ion-exchange resins. This process might be economical for treatment of the small fraction of organic liquid waste which contained isotopes needing immobilization in a high quality waste form. Solidification in glass as a separate step, one of the treatment methods used to immobilize residues from the acid digestion process,<sup>(12)</sup> while leading to a high quality waste form, would probably be inappropriately expensive.

### Sorption

Immobilization of oils by sorption has been documented as it pertains to radioactive wastes by Lin and MacKenzie,<sup>(8)</sup> Seidenberger and Barnard,<sup>(14)</sup> Liskowitz et al.,<sup>(15)</sup> and many others<sup>(3)</sup> have considered the use of sorbents for cleanup of hazardous organic materials. Lin and MacKenzie performed comparative tests on a number of materials to determine typical liquid:sorbent ratios and also to clarify sorbent retention abilities under simulated transportation conditions. They found there was no release of oil from any of the sorbents except very small amounts from Instant-Dri, and possibly vermiculite. In all cases, the sorption measured in the transportation tests was within 1% of that measured in the static tests.

Seidenberger and Barnard described the use of clay sorbents and Solusorb for cleanup of spilled organic solvents. The flash points of several solvents (ethyl alcohol, methyl iso-butyl ketone, and toluene) were substantially increased when sorbed on Solusorb, but not on clay sorbent. Additionally, they provide data on straight and sorbed solvent (toluene) evaporation rates for paper toweling, pure solvent, clay absorbent and Solusorb. In all cases except the toluene on Solusorb, losses of at least 20% by weight of a 10:1 volume ratio of sorbent:solvent were observed within an hour. The toluene on Solusorb lost no weight in the hour time frame.

A number of patents have been awarded for the development of sorbents designed specifically for selective uptake of oils, organic liquids and/or liquid hazardous chemicals. Several of these have properties which make them useful for preferentially sorbing organic or hazardous chemicals over water (Appendix E of Reference 3).

Retention of liquids by sorption is dependent on the strength of the interaction between sorbent material and the sorbed liquid. In the sorbing process, chemical affinity between sorbent and sorbed liquid, and pore- or vacancy-filling mechanisms would be expected to occur. Should the energy of initial sorption not be exceeded at some future time, it is thermodynamically unlikely for the sorption process to spontaneously reverse itself. Removal of sorbed organics could be caused by thermal gradients, pressure, agitation or vibration, and leaching by "pure" water and by water containing acquired components. For the removal to occur, the conditions must be different from those under which the sorption process originally took place. Future conditions to which sorbed organic liquid wastes might be exposed would include the burial environment and possibly the storage environment. Conditions during transportation of sorbed wastes would involve vibrational agitation. Such processes have been simulated in sorbent tests with oils<sup>(8)</sup>, and the majority of sorbents tested retained sorbed oil. Retention of sorbed organic liquids has been demonstrated under rather high pressure conditions (e.g., under a body of water but forced

release of organics through application of high pressure has been effected for sorbents whose retention mode was principally a pore-filling one.<sup>(3)</sup>

### High Integrity Containers

High integrity containers (HICs) may be used for immobilization of solidified or sorbed organic wastes, incinerator ash, and ion-exchange resin waste containing chromate. It may also be feasible to dispose of lead wastes in a high density polyethylene high integrity container, assuming that the package will remain unbreached as a result of typical burial site processes like deformation, biodegradation and irradiation. Packaging of lead wastes in a HIC should be performed in such a way that the structural strength of the container would not be exceeded. The disposal of lead in a HIC should prevent the release of lead to the environment for the lifetime of the container.<sup>(3)</sup> Such a disposal option may be consistent with EPA objectives, given a period of performance for lined trenches of 30 years, whereas the lifetime of a HIC is expected to be at least 300 years. Solid lead wastes (e.g., slag from melting, and mechanically removed surface lead) and LLW requiring lead for shielding are candidates for disposal in a HIC.

### Recovery/Reclamation and Segregation/Separation

Recovery, reclamation, and segregation/separation may be feasible management options for many wastes; they are likely to involve changes in procedures or processes and will necessitate characterization of materials with respect to expected chemical behavior, lability of radioactive species, etc. They are also quite dependent on economic considerations.

Distillation may be used to separate organics or aqueous and organic mixtures. It is particularly suited for solvent recovery. Depending on the nature of the radioactive contamination, distillation may result in concentrated labeled chemical fractions. Should these be hazardous, they may be subjected to specific chemical destruction techniques. Should the radioactive fraction(s) be of no concern with regard to chemical hazard, their packaging for disposal can be performed according to 10 CFR Part 61.

Liquid-liquid solvent extraction may be used as a segregation or separation method, but it is not a final treatment for potential chemical hazards. It may not compete economically with sorption for treatment of large quantities of very dilute wastes. Residues from either distillation or solvent extraction must be assessed for hazard and, if necessary, further treated (solidified, sorbed, destroyed, etc.) prior to disposal.

### Application to Lead Wastes

The lead waste identified in the BNL survey can be divided into two main categories: (1) lead that itself is generally non-contaminated and forms an integral part of a waste package by reason of the need for shielding of discarded materials (generally sealed sources of a variety of radioisotopes), and (2) lead that has become radioactive through surface contamination. The former category is necessary for minimization of the immediate radiation exposure hazard. The second category represents a waste with a potential for segregation for decontamination and re-use, or for immobilization by containerization.

Decontamination of lead waste should be considered as a means of waste volume reduction and also as a recovery method. Decontamination by electrolysis

has been studied for lead and a variety of other metals, including copper, aluminum, and stainless steel.<sup>(16)</sup> Melting of contaminated lead and removal of the contamination fraction followed by re-solidification into useful forms, such as bricks or sheets, has been considered for investigation at EG&G Idaho.<sup>(17)</sup> Methods are available to decontaminate lead metal for unrestricted use. Although this will reduce the volume of lead waste to be disposed of, the resulting decontamination wastes may be hazardous because of lead. Liquid decontamination wastes can be processed for disposal by solidification methods discussed earlier, although cement waste forms may not provide adequate immobilization.

#### Chromium Wastes

Process wastes from light water reactors (LWRs) which use chromates as corrosion inhibitors are potential mixed wastes. These process wastes can be in the form of organic ion-exchange resins or evaporator bottoms. Immobilization by solidification with the standard binders is the most logical treatment for evaporator bottoms, but ion-exchange resins can be immobilized by solidification or containment in a HIC, or treated destructively and the residue solidified.

Solidification of evaporator bottoms in inorganic cement or thermosetting polymer results in a moderate waste volume increase. With bitumen as the solidification agent a modest volume decrease is even possible. For ion-exchange resins in cement, the volume increase is particularly large since stable waste forms can be made only with relatively small proportions of waste. Solidification with bitumen and thermosetting polymers results in a considerably smaller volume increase. For both evaporator bottoms and ion-exchange resins, encapsulation by bitumen and thermosetting polymers provides better fixation of chromate than does cement solidification. In fact, cement waste forms may not retain chromate to a significant extent on contact with water.<sup>(18)</sup>

Incineration, wet-air oxidation and acid digestion are all effective when applied to ion-exchange resin waste. However, treatment of the residue from wet-air oxidation and acid digestion is not as straightforward as for the incineration process. It should be noted that residues from the destruction processes would contain both chromium and radioactivity and would thus still be potential mixed wastes. Residues from incineration and wet-air oxidation would likely contain chromium in the trivalent state, which would mean they might no longer be mixed wastes. However, residues from acid digestion would likely still contain chromate.

Technically the most attractive option is probably the glass furnace method described earlier.<sup>(11)</sup> It is effective in fixation of both chromium and radioactive constituents, but has the possible disadvantage that some volatilization of radioactive Cs might occur during the process, producing a small amount of a secondary radwaste stream.

#### Miscellaneous Wastes

On the basis of the BNL survey,<sup>(2)</sup> it must be assumed that many generators will produce small amounts of waste which may contain some hazardous constituents (i.e., compounds listed in 40 CFR Part 261, Appendix VIII). Responses to the survey indicated that the listed constituents are likely to be present in trace amounts or relatively low concentrations,<sup>(2)</sup> and to be in either the organic liquid

waste or in general LSA trash (paper, contaminated rubber gloves, lab clothing, glassware, etc.).

Generators of these wastes will have the same options as apply to liquid organic wastes. For generators that produce small amounts of hazardous or acutely hazardous constituents, a useful option could be chemical destruction on a laboratory scale. It has been pointed out in "Prudent Practices for Disposal of Chemicals from Laboratories"<sup>(6)</sup> that laboratory destruction of the hazardous characteristic of a chemical compound, including destruction of Appendix VIII constituents, is part of an experiment, not treatment in the regulatory sense. Thus, if applied as a management option, it can prevent potential hazardous waste from ever becoming actual hazardous waste (although it may still be low-level radioactive waste). The options discussed for lead (immobilization and recovery or reclamation) will in general be applicable to other inorganic wastes.

#### Effects of Treatment Options

Destruction, immobilization, and recovery or reclamation of organic liquid wastes (or any other waste) result in a product or residue, or both. For example, acid digestion of organic liquids yields CO, CO<sub>2</sub>, and H<sub>2</sub>O products and leaves the acid and oxidizing agent residue, while immobilization by sorption yields the sorbent-sorbed-liquid matrix. The effectiveness of the treatment options may be determined through testing of the products and residues.

The effectiveness of destruction methods is reflected in the absence from either emissions or residue of the original chemicals of concern. Thus, the products and residues should be analyzed for hazardous materials of concern. Immobilization methods should render the potentially chemically hazardous species isolated from the environment. Testing to determine the extent of immobilization of both organic and inorganic wastes should include leach testing by a method such as the EP toxicity test (or TCLP test under EPA's new land disposal restrictions), since EPA regulations stipulate that the hazardous nature of these species is dependent on their concentration when leached from a waste material.

Recovery and reclamation both involve residue and recovered species. Effectiveness of these methods should be reflected in the purity of the recovered or reclaimed fraction. The residues should be expected to retain the undesired materials, and the further management of residues (e.g., subjection to incineration, immobilization or specific chemical destruction techniques) should involve, as a minimum, their analysis for potentially hazardous species (or species which might cause the waste to exhibit a hazardous characteristic).

#### CONCLUSIONS

Several treatment options have been considered for their applicability to mixed wastes. For organic liquid low-level wastes, recycling or reclamation of the hazardous components may be feasible. For those cases in which disposal is required or chosen, this waste can be either incorporated into a suitable waste form or, more effectively, treated destructively. Incineration is the most widely applicable destruction method for organic liquid wastes. Processing may yield residues which can be secondary wastes and may require another treatment step. Solidification and containment in a HIC are satisfactory options for treating the secondary waste streams.

Lead wastes are of concern for their potential EP toxicity. They may be divided into two groups: those which are an integral part of a waste package and serve as shielding to minimize the radiation from contained wastes (usually discarded sources), and those which are themselves contaminated. The latter type of lead has been emphasized in this report but the HIC management option would also be applicable to the former wastes. Destruction of lead is not an option; decontamination and immobilization appear to be the principal viable management options for these wastes.

Process wastes from LWRs which use chromates as corrosion inhibitors are mixed wastes because of EP toxicity considerations. They can occur as organic ion-exchange resins or as evaporator bottoms. Incineration, wet-air oxidation, and acid digestion are applicable to ion-exchange resins, though incineration has an advantage due to ease of residue treatment. Ion-exchange resins can also be solidified satisfactorily. Solidification in cement results in a large waste volume increase, while the volume increase is only moderate for bitumen and thermosetting polymers. Evaporator bottoms are appropriately treated by solidification, which results in only a moderate volume increase, or even, with bitumen, a moderate decrease.

Based on the findings of this study, it appears that application of a management option with the purpose of addressing EPA concerns can, at the same time, address stabilization and volume reduction concerns of NRC.

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