

Treatment and conditioning of radioactive organic liquids

*Technical manual for the management of
low and intermediate level wastes
generated at small nuclear research centres
and by radioisotope users in medicine, research and industry*



INTERNATIONAL ATOMIC ENERGY AGENCY

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FOREWORD

The International Atomic Energy Agency (IAEA) has published Technical Reports Series and Safety Series documents on radioactive waste management over nearly three decades. These documents have served Member States presenting basic reference material and comprehensive surveys of the 'state-of-the-art' technologies applied to radioactive waste management.

The need for assistance in specific waste management problems facing many countries has been demonstrated in IAEA activities including technical assistance projects and Waste Management Advisory Programme (WAMAP) missions. Technical Reports Series and Safety Series documents usually reflect:

- technological solutions based on experience and resources normally available in countries managing nuclear fuel cycle wastes;
- volumes and activities of radioactive wastes of orders of magnitude greater than those generated in countries without nuclear power.

A new series of technical documents is being undertaken especially to fully meet the needs of Member States for straightforward and low cost solutions to waste management problems. These documents will:

- give guidance on making maximum practicable use of indigenous resources;
- provide step-by-step procedures for effective application of technology;
- recommend technological procedures which can be integrated into an overall national waste management programme.

The series entitled 'Technical Manuals for the Management of Low and Intermediate Level Wastes Generated at Small Nuclear Research Centres and by Radioisotope Users in Medicine, Research and Industry' will serve as reference material to experts on technical assistance missions and provide 'direct know-how' for technical staff in Member States. Currently, the following manuals have been identified:

- Minimization and Segregation of Radioactive Wastes
- Storage of Radioactive Wastes
- Handling, Conditioning and Disposal of Spent Sealed Sources
- Handling and Treatment of Radioactive Aqueous Wastes
- Treatment and Conditioning of Radioactive Solid Wastes
- Treatment and Conditioning of Carcasses and Biological Material
- Treatment and Conditioning of Radioactive Organic Liquids

- Treatment and Conditioning of Spent Ion Exchange Resins from Research Reactors, Precipitation Sludges and Other Radioactive Concentrates
- Design of a Centralized Waste Processing and Storage Facility.

The order of preparation of the manuals is based on priority needs of Member States and it is recognized that additional areas of technical need may be identified as this programme is implemented. In this regard the programme is flexible, should other manuals or modifications prove necessary.

The objective of this manual is to provide essential guidance to Member States without a nuclear power programme on selection, design and operation of cost-effective treatment and conditioning processes for radioactive organic liquids arising as effluents from institutions or small research centres.

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EDITORIAL NOTE

In preparing this material for the press, staff of the International Atomic Energy Agency have mounted and paginated the original manuscripts and given some attention to presentation.

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CONTENTS

1. INTRODUCTION	7
2. WASTE ARISING AND CHARACTERISTICS	7
2.1. Liquid organic waste types	7
2.1.1. Oils	7
2.1.2. Scintillation liquids	7
2.1.3. Solvent extraction liquids	8
2.1.4. Miscellaneous solvents	8
2.2. Waste volumes arising	8
3. WASTE MANAGEMENT STRATEGIES	10
3.1. Strategic considerations	10
3.2. Pre-processing considerations	10
3.3. Preliminary waste management steps	11
4. TREATMENT AND IMMOBILIZATION PROCESSES	13
4.1. Treatment options	13
4.1.1. Incineration	13
4.1.2. Wet oxidation process	18
4.1.3. Silver II electrochemical oxidation process	19
4.1.4. Acid digestion	20
4.1.5. Phase separation by adduct formation	21
4.1.6. Alkaline hydrolysis	21
4.1.7. Distillation	23
4.1.8. Absorption	24
4.1.9. Cementation	26
4.1.10. Absorption and cementation	28
4.2. Process selection	29
4.2.1. General	29
4.2.2. Process combination guidelines	29
5. QUALITY ASSURANCE	33
5.1. Quality assurance requirements	33
5.2. System requirements	33
6. INTERIM STORAGE OF CONDITIONED WASTES	33
7. PROCESS SAFETY	35
8. CONCLUSIONS	36
REFERENCES	37

1. INTRODUCTION

Liquid organic radioactive wastes are generated from the use of radioisotopes in nuclear research centres and in medical and industrial applications. The volume of these wastes is small by comparison with aqueous radioactive wastes, for example; nevertheless, a strategy for the effective management of these wastes is necessary in order to ensure their safe handling, processing, storage and disposal. Aqueous radioactive wastes may be discharged to the environment after the radioactivity has decayed or been removed. By contrast, organic radioactive wastes require management steps that not only take account of their radioactivity, but also of their chemical content. This is because both the radioactivity and the organic chemical nature can have detrimental effects on health and the environment.

Liquid radioactive wastes from these applications typically include vacuum pump oil, lubricating oil and hydraulic fluids, scintillation cocktails from analytical laboratories, solvents from solvent extraction research and uranium refining, and miscellaneous organic solvents.

The report describes the factors which should be considered in the development of appropriate strategies for managing this class of wastes from generation to final disposal. Waste sources and characterization, treatment and conditioning processes, packaging, interim storage and the required quality assurance are all discussed. The report is intended to provide guidance to developing Member States which do not have nuclear power generation. A range of processes and procedures is presented, though emphasis is given to simple, easy-to-operate processes requiring less sophisticated and relatively inexpensive equipment.

2. WASTE ARISING AND CHARACTERISTICS

The volume of radioactive organic liquid wastes produced from medical, industrial and research applications is small compared to other classes of radioactive waste. Generally, solvent and oil wastes are kept separate during collection. However, frequently contaminants such as small quantities of water and sludges may be present. Often, miscellaneous solvent wastes are mixed. This section of the report describes typical waste types, and the quantities likely to arise in developing countries [1].

2.1. Liquid organic waste types

2.1.1. Oils

Radioactive oil wastes consist of lubricating oils, hydraulic fluids and vacuum pump oils. These wastes contain only relatively small quantities of $\beta\gamma$ -emitting radionuclides. All of these wastes arise from activities in nuclear research centres. In addition, vacuum pump oils contaminated with ^3H arise from medical and industrial uses of gloveboxes for analytical and fabrication duties.

2.1.2. Scintillation liquids

Scintillation liquids result from radiochemical analysis and consist of a three component mixture of a solute, a solvent, and the sample under investigation. Non-aqueous solvents such as steroids, lipids; and non-polar solvents such as toluene, xylene, and hexane are the most

commonly used materials. Scintillation liquids are used most commonly for measuring ^3H and ^{14}C , and less widely for ^{125}I , ^{32}P and ^{35}S . The level of radioactivity for this type of waste is typically about 350 MBq/m^3 .

2.1.3. Solvent extraction liquids

The most commonly used extraction solvent is tri-butyl phosphate (TBP). For the extraction process the TBP is diluted, usually with a light saturated hydrocarbon, often dodecane or a mixture of paraffins. Industrial arisings of this waste come from uranium extraction operations associated with the production of yellow cake from extracted ores. The contaminants are U and Th.

Research centre activities associated with nuclear fuel reprocessing research produce solvents contaminated with Pu and fission products, in addition to U.

Sometimes other organic compounds are used for the extraction of heavy metals, including tri- and tertiary amino-compounds, though the volumes are usually very small in comparison to TBP.

2.1.4. Miscellaneous solvents

A variety of organic decontamination liquids arises from miscellaneous operations. These include toluene, carbon tetrachloride, acetone, alcohols and trichloroethane. Aqueous solutions of organic acids, such as citric acid, picolinic acid, ethylene-diamine tetra-acetic acid (EDTA) are also commonly used in the decontamination of equipment.

Dry cleaning produces small quantities of perchloroethylene and Freon 112 wastes. The gross $\beta\gamma$ activity of this waste is usually less than about 200 MBq/m^3 .

2.2. Waste volumes arising [2]

Table I shows typical volumes of the different organic liquid wastes produced per annum in the various groups of Member States. The countries belonging to Groups A,B, and C generate relatively small volumes of wastes compared to countries in Groups D and E where the quantity may be two or three orders of magnitude higher. Member States have been classified into five groups [3] on the basis of the type and quantity of radioactive waste produced and the types of facilities used, as follows:

- Group A Member States which use radioisotopes at a few hospital locations with the waste containing only short lived radioisotopes. Only one institute uses long lived radioisotopes.
- Group B Member States which have multi-use of radioisotopes in hospitals and other institutions, and need a central collection and processing system.
- Group C Member States which have multi-use of radioisotopes and a nuclear research centre which is capable of indigenous production of several radioisotopes.
- Group D Member States with the features of Group C but, in addition, are constructing and/or operating nuclear power plants.

TABLE I. TYPES AND VOLUMES OF CONTAMINATED ORGANIC LIQUIDS
IN DIFFERENT COUNTRIES

Member States in Organic Liquids	Group A	Group B	Group C
Oil, lubricants m ³ /a A in MBq/m ³ Radioisotopes	- - -	0.005 30 H-3, C-14	0.1 300 Co-58, Co-60, Cs-137 H-3, C-14
Scintillation liquids m ³ /a MBq/m ³ Radionuclide	0.01 30 H-3, C-14	0.01-0.02 30-50 H-3, C-14	0.05 300 H-3, C-14, I-125, P-32, S-35
TBP-containing solvents m ³ /a MBq/m ³ Radionuclide	0.01 30 U, Th,+ daughters	0.01 30 U, Th,+ daughters	0.05-0.2 < 10 000 U, Th,+ daughters
Extraction agents (TTA, TOPO) m ³ /a MBq/m ³ Radionuclide	- - -	- - -	0.01 < 30 U, Th,+ daughters
Other organics m ³ /a MBq/m ³ Radionuclide	- - -	- - -	0.05 300 H-3, C-14, Co-58, Co-60, Cs-134, Cs-137

- Group E Member States that are involved in most or all of the steps of the nuclear fuel cycle.

This report is concerned only with those Member States belonging to Groups A, B and C.

3. WASTE MANAGEMENT STRATEGIES

3.1. Strategic considerations

Radioactive organic liquid wastes from medical, industrial and research centre users of radioisotopes form a relatively small volume in comparison to other radioactive wastes, as noted in Sections 1 and 2. Nevertheless, it is important that effective waste management practices are established for these wastes. A number of processing options is applicable to each of the different waste types covered in this report. However, several additional factors have to be taken into account in arriving at the selected strategy:

- Care should be taken to minimize environmental risks both in terms of the natural environment and harm to the public by selection of the best practical environmental option (BPEO concept).
- It is a good waste management practice to ensure that radiation exposure to the workforce is as low as reasonably achievable (ALARA).
- Where it is not yet possible to undertake all of the management the steps from waste arising to final disposal it is advisable that steps which are taken avoid foreclosing later options, since these may require reconditioning the waste with consequent cost and radiation dose penalties.
- As many organic wastes are incompatible with the natural environment the "dilute and disperse" option open for some aqueous and gaseous wastes is not appropriate for the wastes covered in this report. Accordingly, strategies incorporating confinement and concentration of the waste are preferred.
- The preferred processes should be the cheapest and simplest to procure and operate that still enable compliance with the foregoing criteria.

3.2. Pre-processing considerations

Because individual producers of these wastes may not have interest or expertise in waste management, and as the treatment of small quantities of waste may not be cost effective, it may be appropriate to consider having a central waste management facility where the necessary expertise, infrastructure and quality assurance capability can be built up. Accordingly, this concept envisages that the wastes from a variety of producers would be transported to the central facility for subsequent management. In many countries the national nuclear research organization is the central agency with responsibility for radioactive waste management, with regulation provided by an appropriate Government department.

This concept enables the responsibilities of the waste producers and the receiving agency to be assigned as follows:

- The waste receiving agency defines the information and quality assurance requirements for effective waste management. This is likely to include physical, chemical and radiological characteristics, and quantity of the waste.
- The producer would be responsible for characterizing each waste in accordance with the agency requirements.
- The waste management agency would assume complete responsibility for subsequent management of the wastes including any necessary verification of the producer's data.
- The agency would be responsible for establishing the costs associated with management of the wastes and recovery of these charges.

3.3. Preliminary waste management steps

Radioactive organic liquid waste generated by the producer will require collection and storage until a sufficient quantity has accumulated to justify transport to the radioactive waste management agency. The following steps should be taken to ensure safety during this phase and to avoid later difficulties at the processing stage. Safety aspects of the storage of radioactive wastes are described in the IAEA Safety Series [4,5].

During waste collection, segregation of the different waste types should be practised. For example, wastes containing short lived radioisotopes should not be mixed with wastes containing isotopes with long half-lives, liquid scintillation solvents should be kept separate from other solvents, and oils and solvents should not be mixed. Suitable records should be maintained identifying the type of waste in each container, the radioisotopes present, and their activity level. It is preferable that the wastes be collected and stored in containers suitable for transport, such as steel drums of an appropriate size for the waste quantities generated. Glass and plastic containers should be avoided because of their susceptibility to shattering and puncturing. Because of the chemically active and flammable nature of organic liquid wastes, their collection and storage area should be isolated from the producer's and other activities and should have adequate fire protection and ventilation. In extreme cases, inert gas, CO₂, or nitrogen blanketing systems may be considered [6].

After receipt of the wastes at the radioactive waste management agency, some verification of the producer's characterization data will be necessary. After this verification the wastes can be segregated into those containing only short lived radioisotopes and those containing isotopes with long half-lives. The wastes containing short lived radioisotopes can be subjected to interim storage to allow decay to a substantially non-radioactive state. At this point, the wastes can be released for reuse where possible, or alternatively for disposal as a chemical waste only. Again, because of the toxic and fire hazards of these wastes, the storage area should be separate from processing areas and should be ventilated and protected against fire as discussed previously. The wastes containing long lived radioisotopes should be segregated on the basis of chemical type and radiological hazard. Subsequent arisings of similar wastes can be consolidated until a sufficient quantity of each is available for cost effective conditioning.

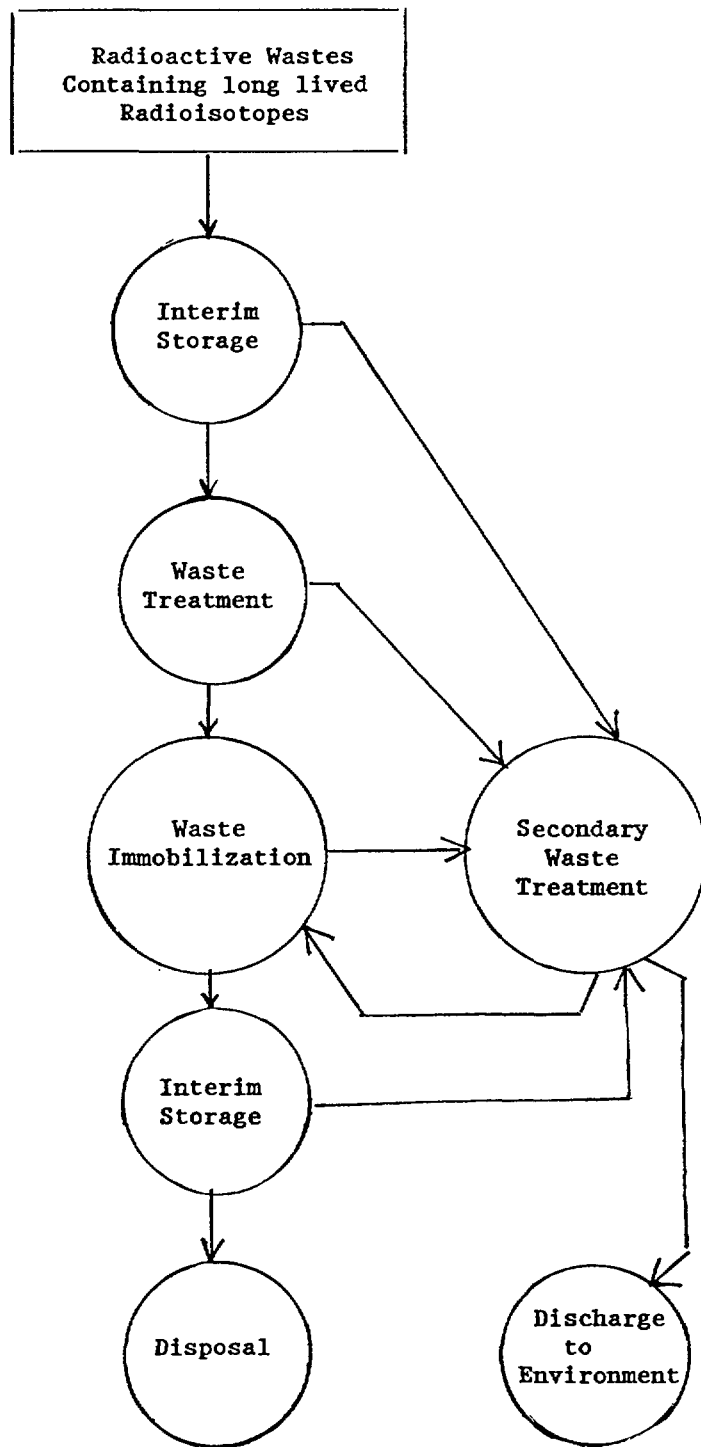


FIG. 1. Block diagram for the management of radioactive wastes containing long lived radioisotopes.

The steps in the waste management strategy following segregation and consolidation to final disposal are shown in Figure 1. The waste is stored pending treatment both to accumulate enough waste for cost effective treatment and for selection of the appropriate conditioning process.

The waste management strategy may require treatment of the wastes such as by distillation to separate the radioactive and non-active components or by incineration to destroy the organic material. The treated waste may then need to be immobilized to prevent radioactivity from escaping into the environment.

Following immobilization a complete waste management strategy would include both storage and disposal. However, in developing Member States the quantities of waste for disposal may not be sufficient to justify construction of a waste repository. Consequently, the conditioned waste may have to be stored for an extended period of many years.

4. TREATMENT AND IMMOBILIZATION PROCESSES

Several techniques for conditioning organic liquid radioactive wastes have been developed and evaluated. In some cases these have been implemented in different countries. Section 4.1. describes existing techniques which could be applied to the different wastes, and Section 4.2. provides guidance on the selection of appropriate processes for particular wastes.

4.1. Treatment options

4.1.1. Incineration

Incineration is an attractive technique for treating organic liquids because they are readily combustible, and high volume reduction factors can be achieved. In principle, the products of complete combustion are the oxides of the elemental constituents, and carbon dioxide and water are, thus, always formed. Other oxides will also be formed, depending on the composition of the waste. For example, P_2O_5 and NO_2 are formed from the combustion of TBP and amines, respectively, and these acid gases can produce corrosive conditions in combination with water. Sulphur and chlorine also produce corrosive combustion products (not the oxide in the case of chlorine, but HCl), which if present in substantial proportions in the waste can require special means to limit the effects of corrosion.

An incineration system consists of the steps shown in Figure 2. An incineration system includes the following components:

- a waste feed preparation and delivery system,
- a combustion chamber,
- an off-gas treatment system, including induced draught fan and a stack,
- ash removal system, possibly interfacing with an ash immobilization system,
- and appropriate control and monitoring instrumentation.

The principal objectives of incineration are to achieve the complete combustion of the waste to inorganic products, appropriate off-gas cleaning, and radiological protection. A radioactive waste incineration system must be radiologically safe by providing containment of the

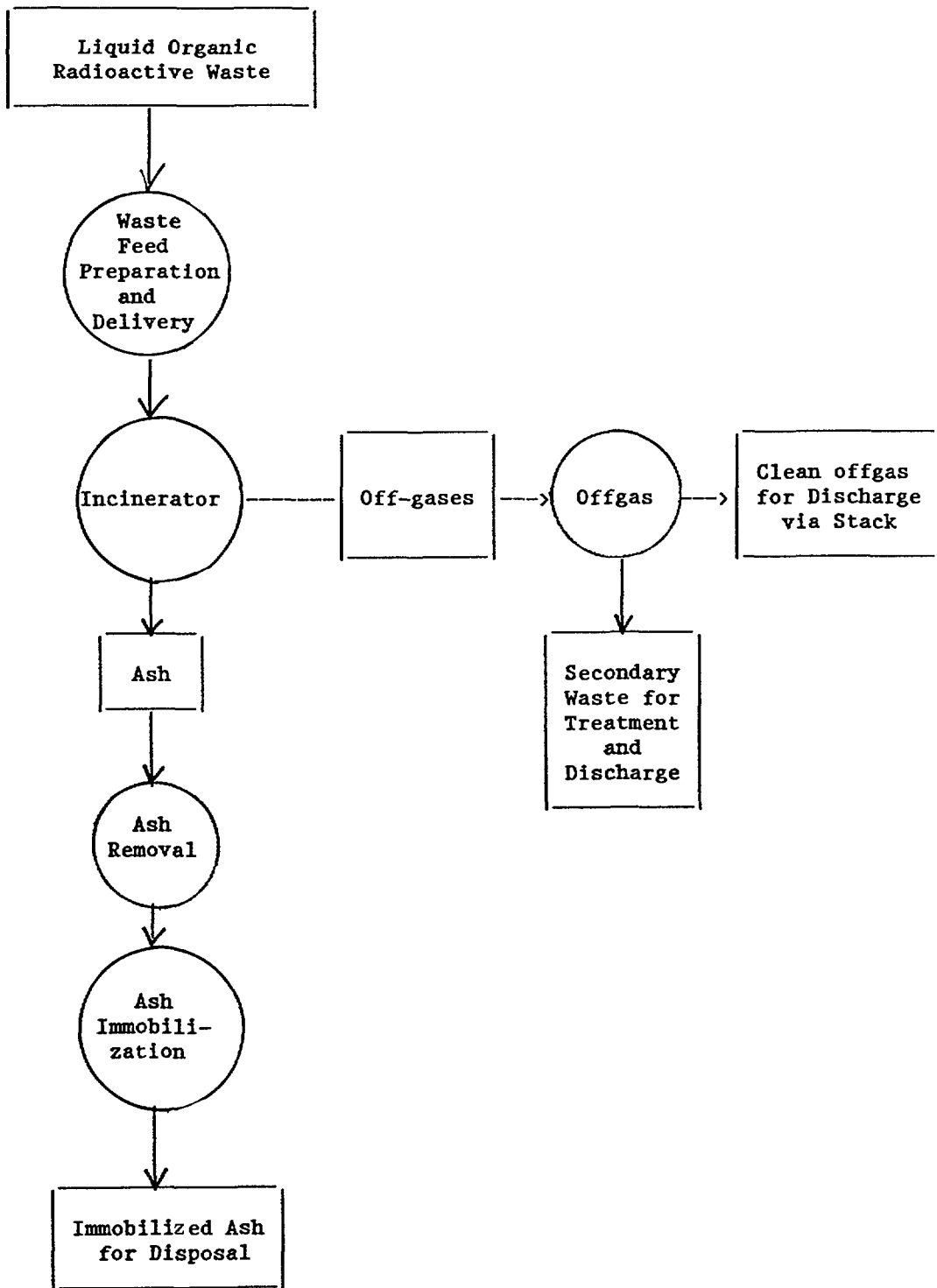


FIG. 2. Block diagram for organic liquid radioactive waste incineration.

radioactive species throughout the process, and the incinerator itself must additionally provide containment of the volatile organic wastes and so avoid the formation of unconfined explosive mixtures of gases and vapours.

A variety of incinerator types and concepts has been tried throughout the world. In general, incinerators for low level radioactive wastes have proved more successful than for intermediate level radioactive wastes, since they tend to be less demanding to construct, operate and maintain [1,7]. Incineration is often practised in dual-purpose solid and liquid radioactive waste incinerators, but these tend to be less successful than dedicated liquid incinerators which have been optimized for the purpose. Dual-purpose incinerators are not described in this report, though reference to these may be found in the Ref. [1] and in Section 4.2.

Solid waste incinerators can be classified as excess-air or starved-air (pyrolysis) types, but incinerators for liquid wastes are almost exclusively of the excess-air type. The high calorific value of organic wastes and the high demand for oxygen can lead to vigorous and turbulent burning conditions requiring filtration to prevent the escape of the radioactive ash into the environment.

A pilot plant incinerator at Cadarache, France [8] was reported in 1985 to have been in operation for 5000 h since 1981 for the destruction of waste solvents including chlorinated solvents, oils, scintillation liquids and TBP which arise from nuclear research centres and reprocessing plants, and from institutes and hospitals. During this period 130 m³ of solvent were incinerated with a volume reduction factor of 30-300. Radioactive contamination of the solvents was limited to 37 MBq/m³ and 3.7 GBq/m³ for α - and $\beta\gamma$ -activity, respectively. The system is illustrated in Figure 3. Incineration is carried out at 900°C in a horizontal refractory-lined chamber, with temperature control by the addition of air. Waste solvent and fuel are fed via pumps at controlled rates. The combustion gases are cooled from 900°C to 600°C by water spray addition, followed by dilution air to reduce the temperature to 220°C. The gases can then be filtered in a fibre prefilter and a HEPA filter, followed by caustic scrubbing to neutralise acid gas products of combustion. Chlorine, phosphorus and fluorine concentrations in the feedstock are limited to 20%, 0.1% and 50 ppm, respectively. However, it is possible to burn 30% TBP/diluent when sodium carbonate is added to neutralise phosphoric acids. 95% of the active ash is trapped by the filters, and the 5% balance remains in the incinerator, there being no special ash off-take.

KfK Karlsruhe, Germany has a dedicated incinerator for organic liquid wastes which burns a feedstock of the following composition: 40% oil, 34% solvent, 10% scintillation liquid, and 16% water. By the end of 1983, 360 m³ of radioactive liquid had been incinerated containing 18.5 GBq/m³ and 2 TBq/m³ for α - and $\beta\gamma$ -activity, respectively [9-11]. The incinerator system is illustrated in Figure 4 and is of similar configuration to the Cadarache system described above except that liquid scrubbing precedes gas filtration.

A simple cyclone-type incinerator has been developed at Mound Laboratory, USA [12,13] which consists of a mild steel drum as the main incinerator vessel fitted to a closure in place of the normal drum lid. This closure contains a number of penetrations for feed and off-gas pipework, and an ignitor. This simple concept, shown in Figure 5, allows the ash to accumulate in the drum, so that the drum and its contents may be removed when appropriate for disposal, and a new drum fitted for a subsequent campaign. Off-gases are treated in a wet scrubbing system with recirculating scrub liquor.

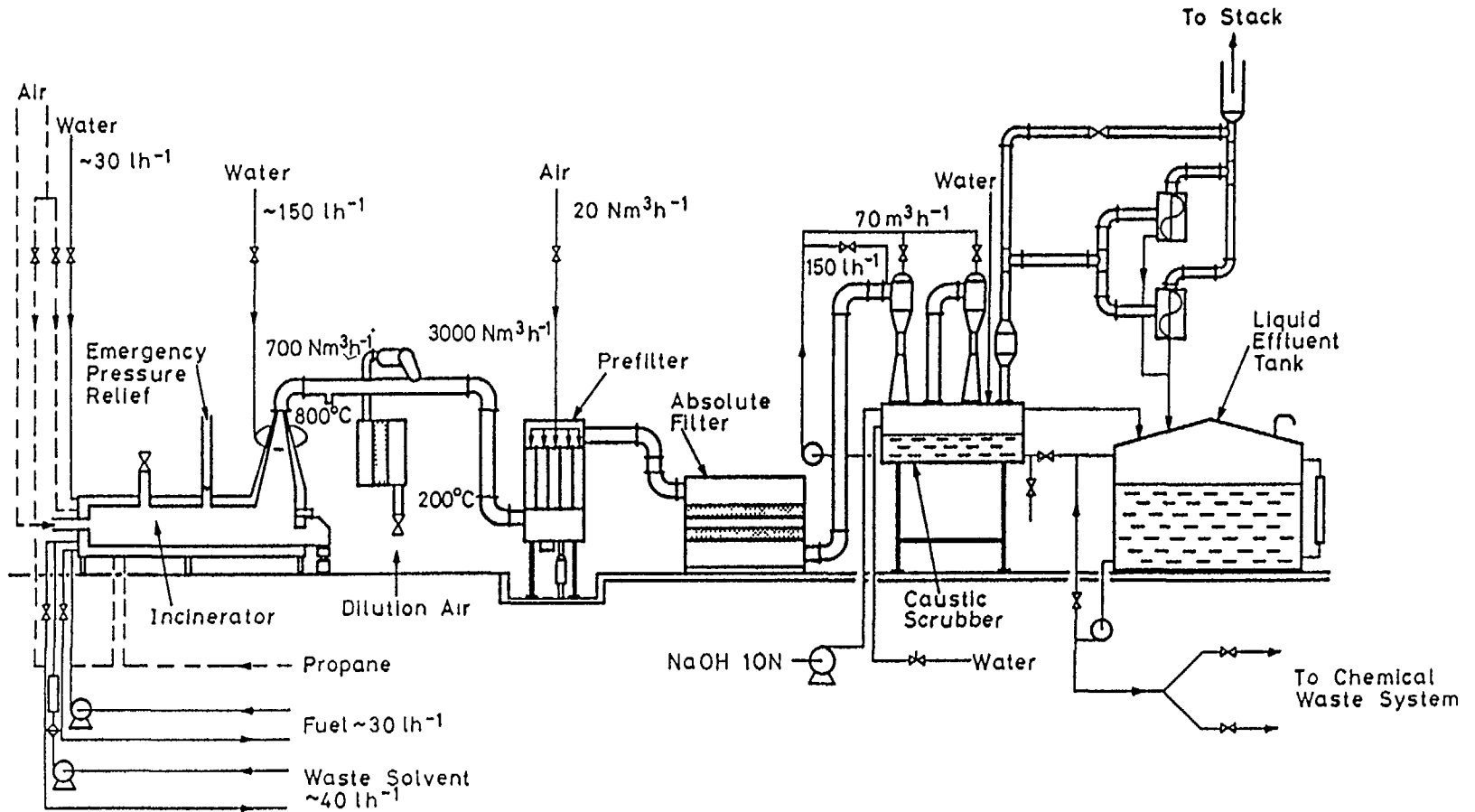


FIG 3 Plant diagram of low level solvent incinerator, Cadarache (France)

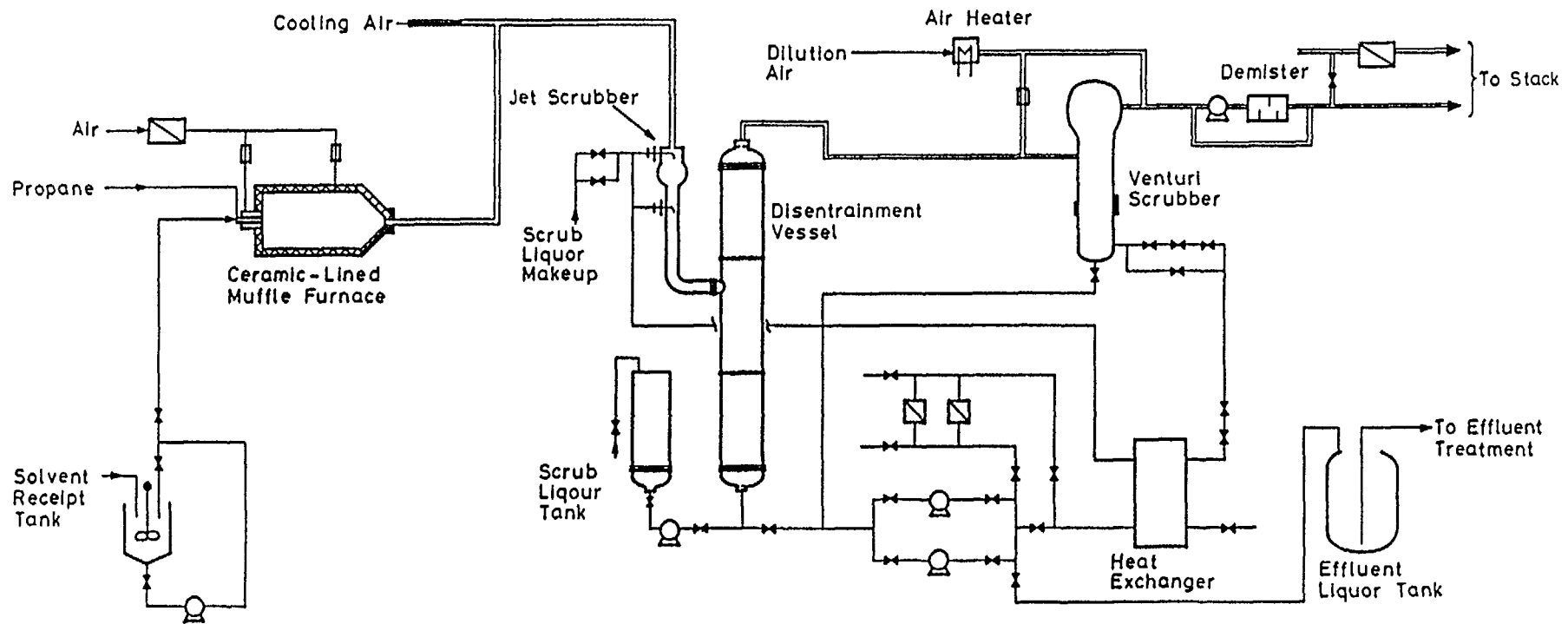


FIG. 4. Plant diagram of low active liquid waste incinerator, Karlsruhe (Germany).

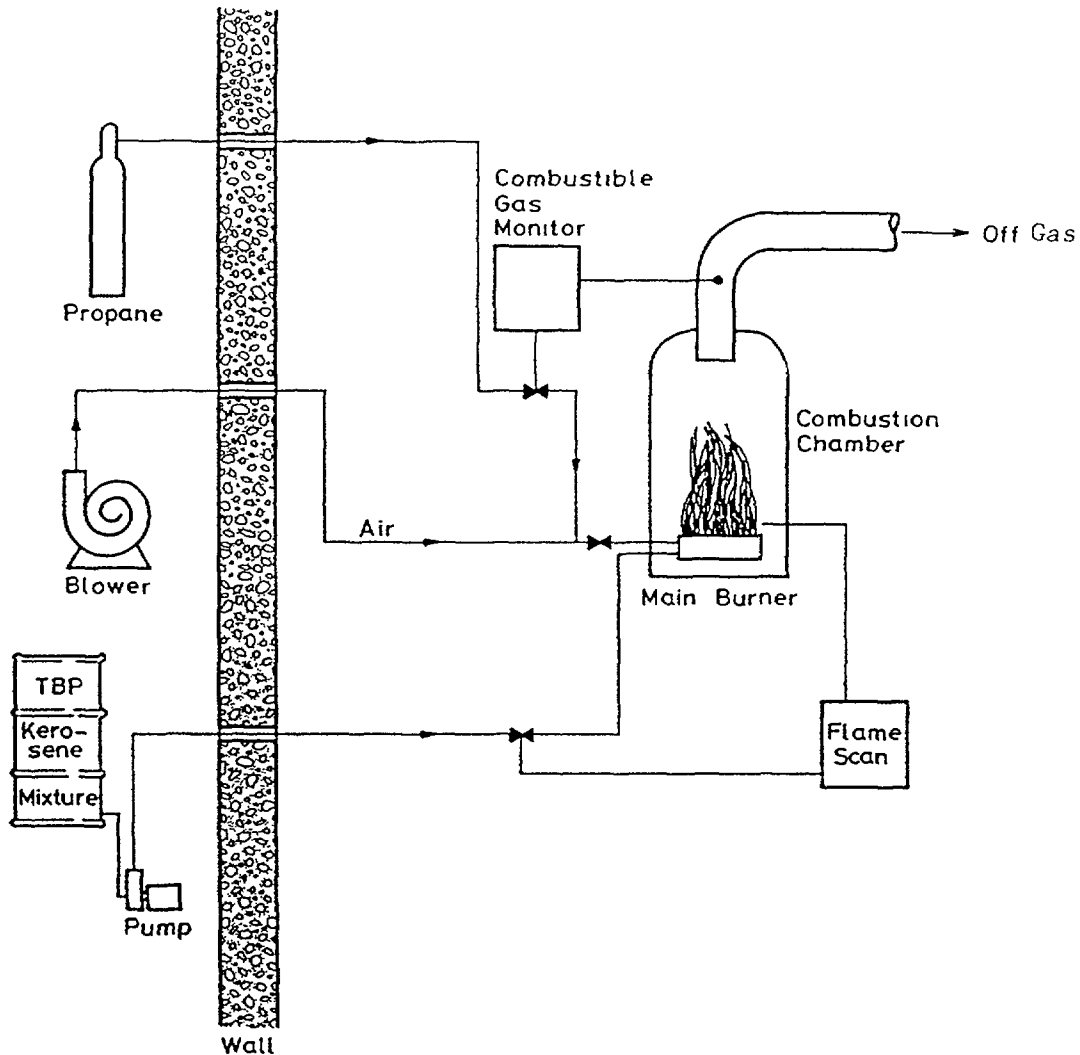


FIG. 5. Liquid feed system to cyclone incinerator, Mound (USA).

4.1.2. Wet oxidation process [14-16]

Wet oxidation is a technique for breaking down organic materials to carbon dioxide and water in a process which is analogous to incineration. The organic waste is reacted with hydrogen peroxide in the presence of a catalyst at 100°C with excess water distilled or evaporated to leave a concentrated inorganic waste which contains the radioactivity. The main advantages of the process are low temperatures and an aqueous waste which is easy to treat. Good contact of the organic wastes with the aqueous phase is obtained. Early applications have been for polar materials including TBP and organic acid decontamination reagents, but more recently development has extended to the treatment of non-polar materials. Early reports during the development stage of the process indicated the need for high temperatures and pressures, implying the use of sophisticated process control and skilled process operators. However, more recent developments have enabled operation at modest temperature and atmospheric pressure, thereby greatly simplifying process requirements. A plant has been constructed at Winfrith, UK, for treating up to 200 kg batches of waste. The process is also suitable for toxic chemical wastes, since the stringent procedures required to prevent the release of radioactivity are ideally suited to the containment of toxic materials. Figure 6 shows a block diagram for the wet oxidation process.

4.1.3. Silver II electrochemical oxidation process [17]

This process offers an alternative oxidation method to incineration that has some parallels with the wet oxidation process described in Section 4.1.2. Both processes operate at much lower temperatures than incineration and do not demand the off-gas cleanup challenge posed by incineration. Both wet oxidation and the electrochemical oxidation process feature the use of strong oxidising agents. Whereas the wet oxidation process featured the use of hydrogen peroxide, the electrochemical oxidation process uses Ag(II) in a solution of silver nitrate and nitric acid, by placing the solution in the anode compartment of an electrochemical cell and passing a current.

The anode and cathode cells are separated by a semi-permeable membrane which allows passage of the ions which carry the current through the cell but prevents gross mixing of the contents of the compartments. Aqueous Ag(II) is reduced to Ag(I) and this is regenerated electrochemically. The

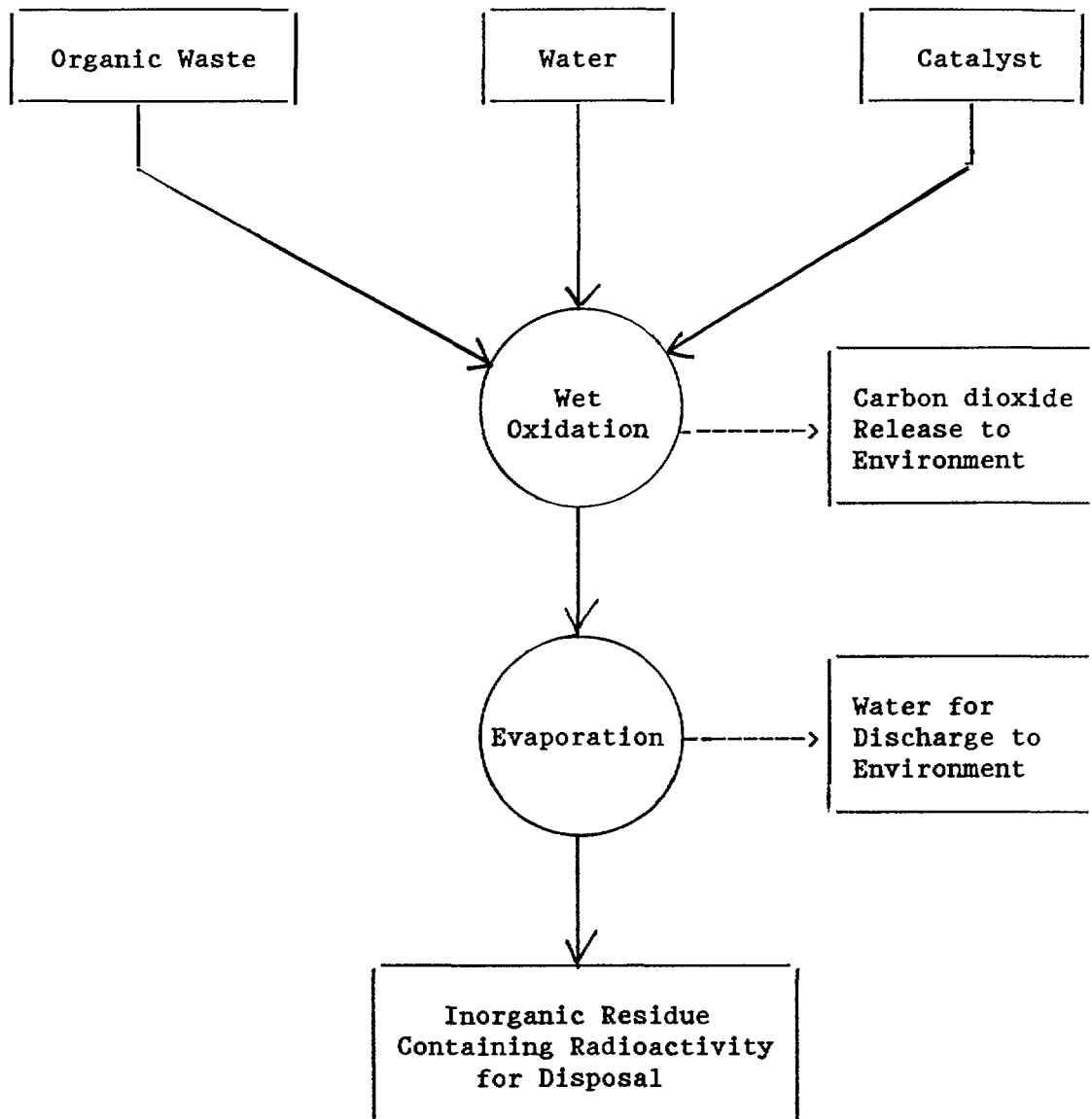


FIG. 6. Block diagram showing the principle of the wet oxidation process.

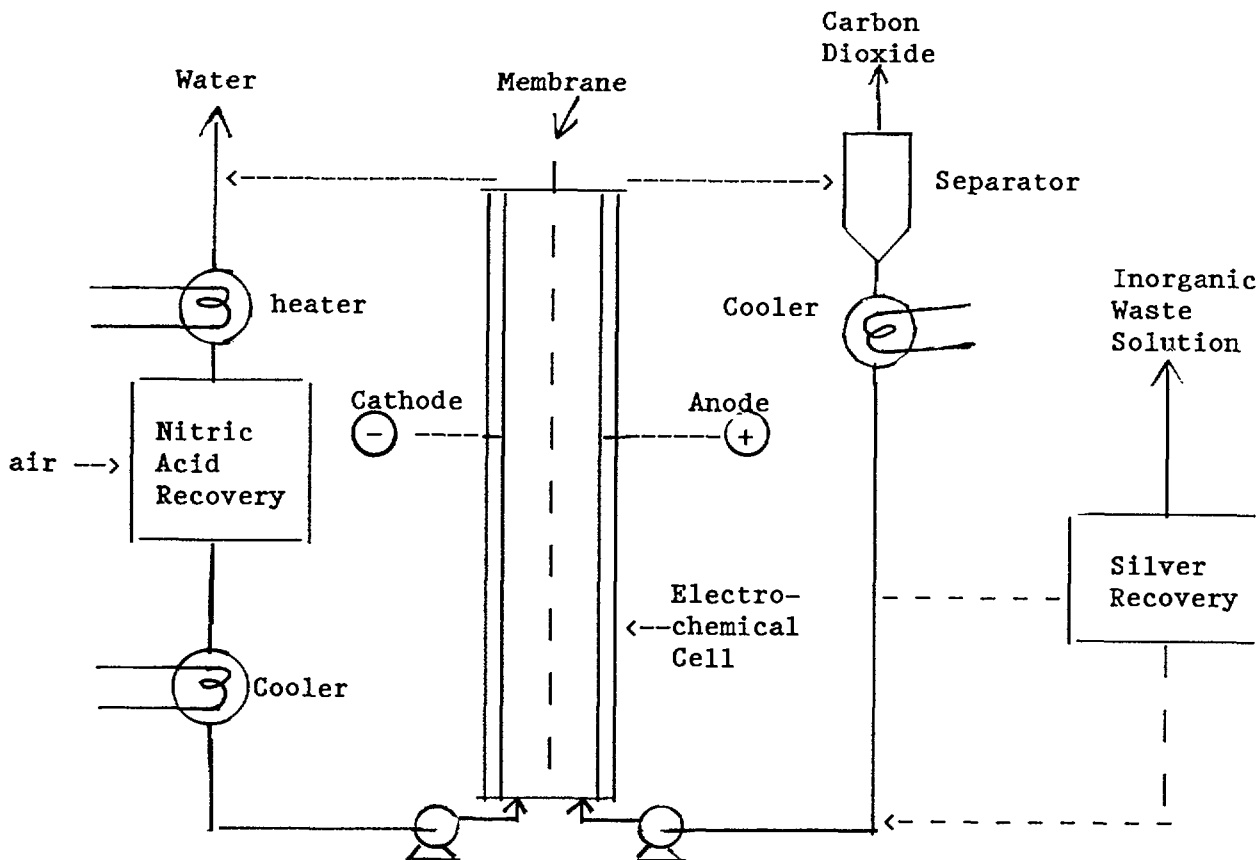


FIG. 7. Schematic diagram of Ag II electrochemical oxidation process.

oxidising or reactive species formed during the reaction of the Ag(II) react with the organic waste oxidising it to carbon dioxide, water and organic species arising from any halogens, nitrogen, phosphorous and sulphur present in the waste. Figure 7 shows a simplified scheme of the process. Essentially the same operating conditions can be used for a variety of waste types, there is no volatilization of low molecular weight species which are formed as intermediates during the destruction process, and the process can be readily started and stopped by switching the current on or off.

4.1.4. Acid digestion [18]

This process has been under development in a number of countries, though only Germany and the USA have any significant experience in the operation of an industrial scale plant. The process requires the use of mixed nitric and sulphuric acids at $\sim 250^{\circ}\text{C}$, with consequent need for expensive corrosion resistant materials of construction. Extensive off-gas scrubbing is required since sulphur dioxide and nitrogen dioxide are generated during the process. Some organic liquids such as hexone and TBP can be treated effectively, though others, such as paraffins only partly digest, with the balance being lost from the reaction vessel by distillation. Trichloroethane and toluene do not digest well unless they are well dispersed by atomization.

4.1.5. Phase separation by adduct formation [19-22]

Phase separation by adduct formation has been investigated as a pretreatment prior to destruction or disposal of TBP and diluent. This is done by contacting contaminated TBP/diluent with concentrated phosphoric acid at room temperature in conventional solvent extraction equipment, such as mixer-settlers or pulsed columns. The TBP is solubilized quantitatively in the phosphoric acid and forms a TBP-acid polar adduct, whose formula is quoted as ranging between $3\text{TBP}\cdot\text{H}_3\text{PO}_4\cdot 6\text{H}_2\text{O}$ and $\text{TBP}\cdot 4\text{H}_3\text{PO}_4$. Almost all the radioactivity and the degradation products are transferred with the TBP into the adduct phase, which can be burned, or possibly recycled. The proposed treatment for the adduct is either to split it to recover the phosphoric acid and dispose of the TBP and associated radioactivity, or to pyrolyse it, thereby recovering phosphoric acid from both the initial phosphoric acid and the TBP, and converting the degradation products to combustible hydrocarbons.

Processes used in Germany feature a preliminary wash of contaminated TBP/diluent in sodium carbonate or hydroxide solution. 300 m^3 of 2-30% TBP/dodecane containing 400 GBq/m^3 - 4 TBq/m^3 from the WAK plant have been treated in this way, removing 95% of the radioactivity before treatment with concentrated phosphoric acid to form the adduct. The dodecane phase contains only 37 kBq/m^3 . The adduct is split by dilution with water into an aqueous phase containing 4% of the original activity and a TBP phase containing 1% of the original radioactivity. The aqueous effluents from washing and adduct splitting were routed to the medium active liquid waste treatment plant and the TBP incorporated into PVC.

In another version of the process it was proposed to recycle or burn the diluent and recycle the TBP or incorporate it with PVC chips to form a solid mass for disposal.

In Belgium, the Eurowatt process has been developed to a pilot scale plant in which 100 L of spent 16% TBP/diluent were treated at 1 L/h. A flowsheet for the Eurowatt process is shown in Figure 8. The adduct was formed by contacting with 15M phosphoric acid in an organic:aqueous ratio of 50:1 in a minimum of 10 contact stages to effect complete separation of the diluent. The molar ratio of phosphoric acid to TBP was maintained ideally at 1:2.5. If its value falls to unity the TBP can form a third phase.

The TBP-phosphoric acid adduct was split by contacting with water and dilute sodium hydroxide in a series of pulsed columns. Subsequent work featured adduct formation and phase separation using inactive 30% TBP/diluent processed at 25 L/h in mixer-settlers, followed by pyrolysis and incorporation of the phosphatic residue into glass by a vitrification process. Active trials of this later work have not been reported and the unit has now been decommissioned.

4.1.6. Alkaline hydrolysis [23-25]

The alkaline hydrolysis process was investigated in Germany as a chemical treatment to destroy the TBP in a TBP/diluent mixture leaving the diluent suitable for reuse. The process required prewashing the TBP/diluent with sodium carbonate. This removed uranium which would otherwise have caused precipitation and emulsification during the hydrolysis. Hydrolysis was conducted by heating a mixture of TBP/diluent with 50% sodium hydroxide solution to 125 - 130°C for 7 h. In this way a few hundred litres of active waste solvent from the Wiederaufarbeitungsanlage

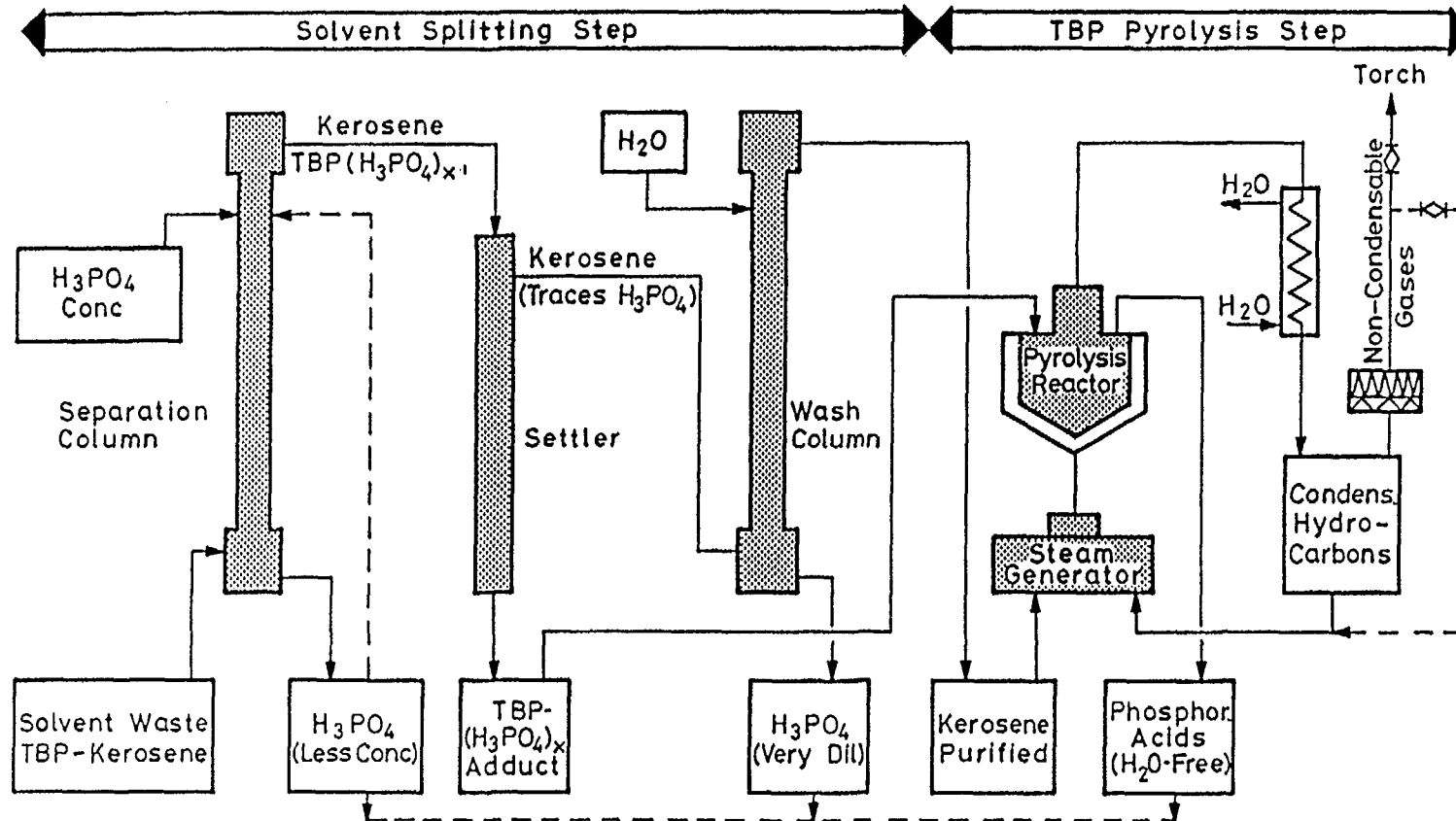


FIG. 8. Simplified flow sheet of the Eurowatt process (three phase system).

Karlsruhe Betriebsgesellschaft mbH, Eggenstein Leopoldshafen (WAK), were treated in 50 L batches and the diluent recycled, being free of nitro- and nitrate-compounds. Incineration of condensed diluent and butanol from the hydrolysis was also demonstrated. The process was proposed for the treatment of 30% TBP/diluent, and phase separation was proposed for the treatment of solutions containing 2-20% TBP/diluent. The suggested disposal route for the lower phase from the hydrolysis process containing sodium dibutyl phosphate (DBP), butanol, methanol and water was evaporation of the water followed by incorporation of the residue into bitumen.

During early work on the hydrolysis process, it was thought that TBP was converted to inorganic phosphate. However, subsequent investigation at Harwell showed that the principal reaction product was DBP with only very small quantities of monobutyl phosphate (MBP) and inorganic phosphate. During development at Harwell both reflux and distillation modes of the hydrolysis process were investigated. Optimum conditions in the reflux mode required treatment for 7 to 8 hours at 125°C with 50% aqueous sodium hydroxide in the molar ratio NaOH:TBP = 1.5:1. The products of the hydrolysis were in two or three liquid phases. Washing the three phase liquid mixture with water produced two liquid phases and one white solid phase containing the sodium dibutyl phosphate.

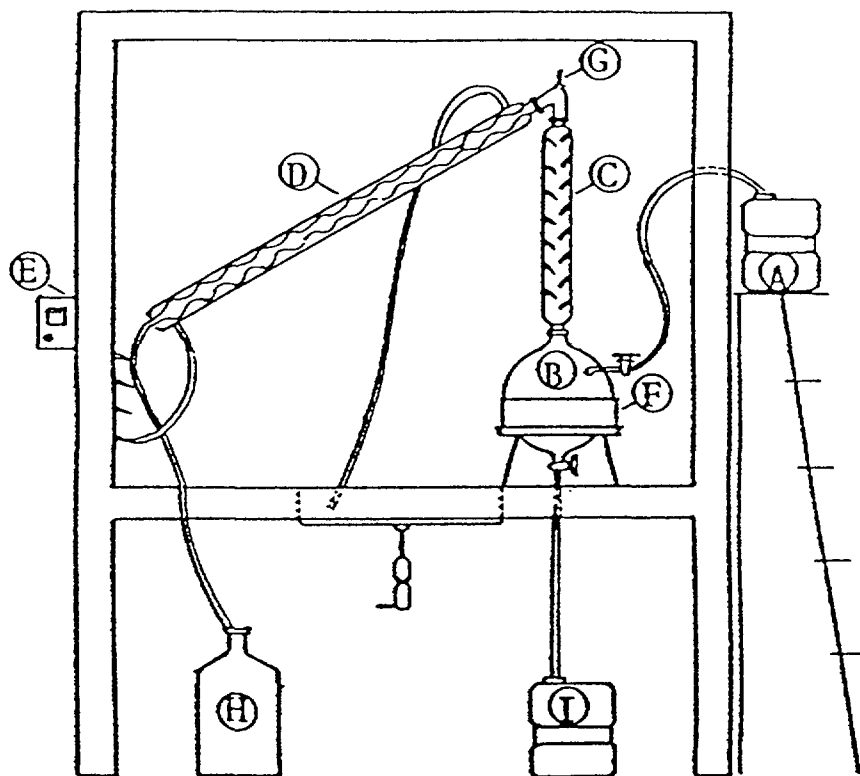
The alkaline hydrolysis process is suitable for the treatment of solutions containing between 6% and 30% TBP in diluent. For lower concentrations of TBP the addition of 0.2 volumes of 1.5M sodium DBP to the TBP/diluent is recommended in order to improve contact of the reactants and thus enhance the otherwise slow rate of reaction.

4.1.7. Distillation

Simple distillation may be used for the pretreatment of scintillation fluids and miscellaneous solvent wastes. Substantial volume reduction is possible as the activity is generally concentrated in the residue. The recovered organic solvent could be used as a technical grade solvent or as a fuel for an incinerator.

The distillation of scintillation fluid has been practised in the USA and Brazil [26]. At IPEN, Sao Paulo, a 20 L/day unit shown in Figure 9 was operated. At 85°C the azeotropic mixture begins to distill and when the water is exhausted the temperature is raised to 110°C to distill off the balance of the organic solvent. The water/solvent distillate separates into two phases with any activity carry over remaining in the aqueous phase. The organic solvent can be separated from the aqueous phase for reuse elsewhere. A waste volume reduction of 40%, a process yield of 80% and a 10 L batch operation time of 50 minutes are claimed.

In the USA, a type of steam distillation has been used to recover liquid scintillation counter solvents contained in plastic vials [27]. In this case the vials do not need to be emptied for processing. The process uses a steam sterilizer autoclave with a volume of 360 L. Disposable aluminium trays are placed in the bottom of the autoclave to collect the melted plastic vials. Each charge could accommodate approximately 400 vials. The autoclave is pressurised to about 500 kPa with steam for 1 hour. The steam is released slowly into the coils of a cooling can to condense the water and solvent vapour which is collected in a separatory funnel. The condensate consists of approximately 1 part water and 3 parts organic solvent. After separation into the two phases, the water phase is usually dilute enough in activity to allow discharge. The organic solvent can again be used as a technical grade solvent or as a fuel. The melted plastic vials can be incinerated or treated as a solid waste.



- A. Storage and feeding flask
- B. Distilling flask
- C. Reflow column
- D. Condenser
- E. Temperature control and indicator
- F. Heater
- G. Thermocouple
- H. Separation flask
- I. Waste storage flask

FIG. 9. Distillation apparatus for scintillation solutions.

Vials containing liquid scintillation counter fluids may be uncapped by hand to release the solvent. Alternatively, they could be crushed in quantity using equipment similar to that shown in Figure 10. In either case fire and respiratory protection must be provided because of the flammable and toxic nature of the solvent [28].

Distillation can be practised with conventional readily available equipment and space requirements for the equipment are small. The process is simple and well known, and operators require only limited training. The process is cost effective in that valuable solvent can be recovered for some form of reuse. The active residue could be either immobilized or destroyed if incineration facilities are available.

4.1.8. Absorption

The treatment of organic liquid radioactive wastes with absorbents is a simple way of converting the liquid to a solid form. As long as there is an excess of absorbent there is no need even for mixing; the liquid waste can be added to the absorbent in a suitable container and eventually all



- | | |
|---|---|
| 1 Latched safety door, cannot be opened when basket is in filling position | 6 Adjustable legs, raise or lower Vyleater to accommodate various sizes of waste containers up to 55 Gal |
| 2 Track, guides travel of basket | 7 Leveling pads, to accommodate uneven floors |
| 3 Basket, protective-coated counter balanced holds 400 to 1 000 vials depending on size | 8 Indicator, position of adjustable crusher which can accommodate vials of various sizes |
| 4 Drain hose | 9 Exhaust vent, discharges fumes from inside Vyleater |
| 5 Cover with viewing window, fits 55 Gal drums
Discharge sleeve guides crushed material into drum | 10 Screen access door, permits quick easy cleaning or replacement of screens |

FIG 10 Crusher for glass or plastic vials

TABLE II. PERFORMANCE OF ABSORBENTS FOR ORGANIC LIQUIDS

Product	Absorbency waste/sorbent (ratio by volume)	Organic waste by volume (%)	Volume increase (%)
Natural fibre	0.9	47	111
Synthetic fibre	0.8	44	125
Clays	0.6	33	167
Diatomaceous earth	0.65	40	154
Vermiculite	0.35	26	286
Imbiber beads	4.0	80	25

the liquid will be taken up. This technique has been routinely used for the solidification of radioactive turbine and pump oil [29]. The following main categories of absorbent are commonly used:-

- natural fibre (sawdust, cotton)
- synthetic fibre (polypropylene)
- vermiculite (mica)
- clays
- diatomaceous earth
- imbibers beads (alkyl styrene polymer).

The use of absorbents converts the liquid waste into a form which can vary from loose dry particles to a jelly-like solid. The waste forms have no special integrity and are only restrained from dispersing by the container. As shown in Table II, the absorption efficiency of the different absorbents can vary by a factor of 2 to 3, and the waste volume increase can be up to almost 300%.

The suitability of absorption alone for the solidification of organic liquid wastes is only moderate; the process efficiency can be adversely affected by the presence of water or other ionic contaminants, and variations in waste viscosity can cause significant reductions in the quantity of liquid absorbed. Finally, the waste form is readily dispersible in air or water if the product container is breached.

This process is probably the simplest technique for conversion of organic liquids to a solid form and is practised extensively in many areas. The use of excess absorbent can even eliminate any need for mixing as it will soak up all free liquid with time.

4.1.9. Cementation

Cement by itself has limited efficiency for the solidification of organic liquid wastes. Only about 12 vol% of oil can be incorporated directly into cement and still retain a waste form that is dry and monolithic. However, significant increases in waste loadings can be obtained when emulsified and multiphased (oil/water/solvent) wastes are used. Mixing of the components need only require an in-drum mixer or drum roller. A flowsheet for liquid waste solidification with cement is given in Figure 11.

Cementation has been used extensively in the USA to solidify waste turbine oil, pump oils and TBP/dodecane solvents [30].

A typical composition for radioactive oil solidification in a 200 L waste form would be [1]:

- 165 kg Portland Cement
- 17 kg lime
- 72 L oil
- 62 L emulsifier
- 14 L water
- 7 L silicate accelerator

The cement and lime are dry mixed thoroughly in the waste container. The oil and emulsifier are mixed in a separate container, water is added, and the components are mixed to form an oil-in-water emulsion. The emulsion is added to the cement/lime powder and the whole stirred until the batch is homogeneous. The silicate cure accelerator is added and stirring

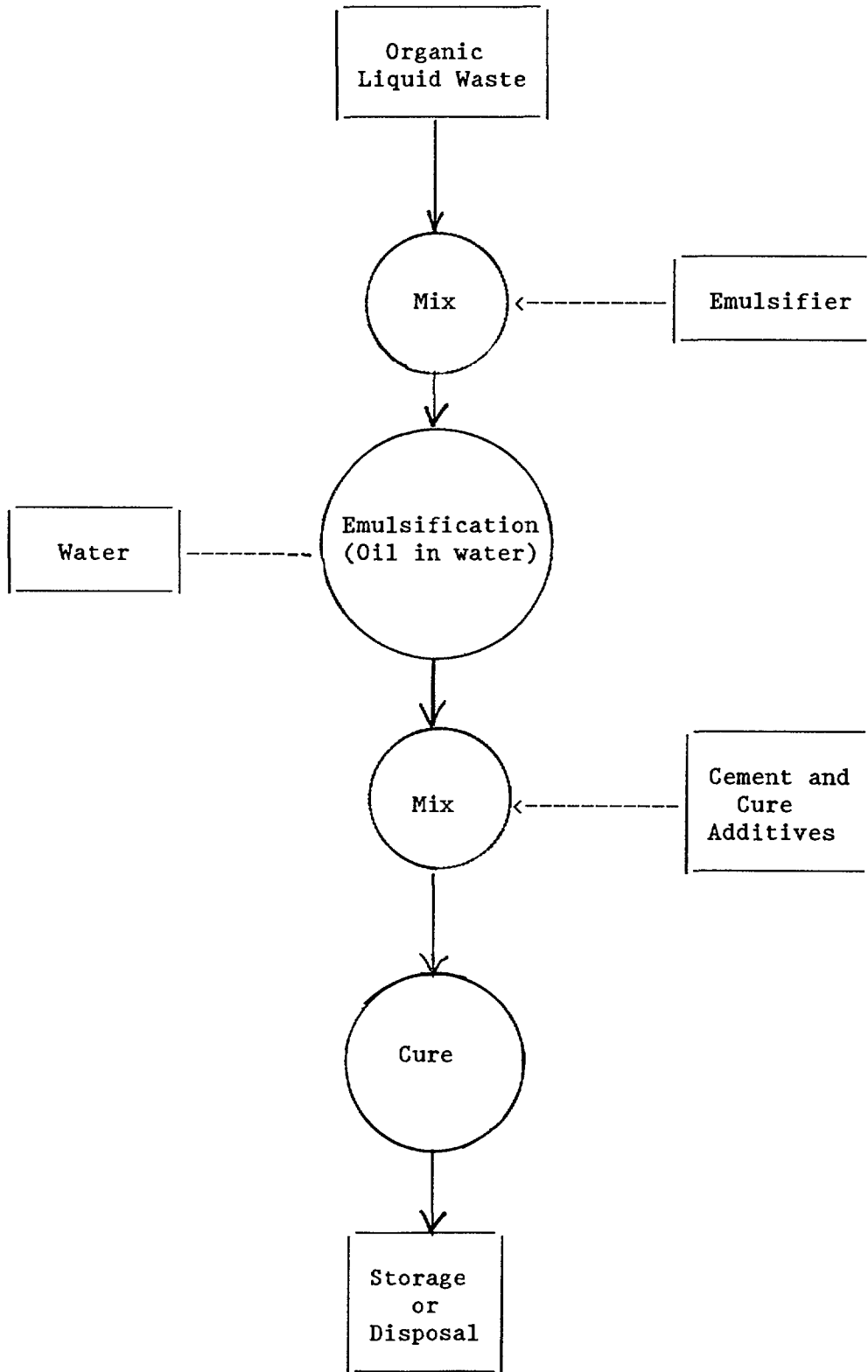


FIG. 11. Flow sheet for organic liquid waste solidification with cement.

is continued for a short time to ensure uniform dispersion of the accelerator. The mixer can then be removed and the container closed and stored for product cure to take place. Typically, adequate cure for transport to the interim storage/disposal site will require 4 to 28 days.

Although cementation is a simple process requiring only rudimentary equipment, it is important to note that the degree of cure and resultant waste form integrity can be adversely affected by minor compositional changes in the waste feed. Thus, it is essential that small sample solidification tests be done on each batch of waste before full scale solidification. This will determine whether formulation modification is necessary to obtain the correct desired waste form.

4.1.10. Absorption and cementation [31]

Instead of emulsifying the organic liquid waste before introducing it to the cement powder, this approach converts the organic liquid to a dry particle form which is then mixed with the cement powder and water to produce the final waste form. The advantages of this technique are that the procedure is more tolerant of batch to batch waste variability, although not completely so, and higher waste loadings of up to 56% vol organic liquid can be attained. A disadvantage is the need to convert the organic liquid waste to a dry solid before adding it to the solidification matrix.

Some typical compositions of solidified waste forms using this technique are given in Table III.

TABLE III. OIL WASTE SOLIDIFICATION WITH ABSORBENTS AND CEMENT

Absorbent	Cement (g)	Oil (g)	Absorbent (g)	Water (g)	Liquid Waste Content (%vol)
Clay	200	32.2	71	71	15.6
Vermiculite	200	84	24	120	21.8
Natural fibre	200	321	11	70	56.0
Diatomaceous earth	200	372	265	160	38.75
Synthetic fibre	200	295	34	165	44.5

If the organic liquid waste is contaminated with water it may be possible to reduce the amount of added water given in Table III by a similar amount to that contained in the waste feed. However, it is important that the mix should contain enough water to hydrate the cement adequately, otherwise the solidified waste form may not have sufficient integrity.

This technique, as for simple cementation, is a low cost process requiring minimal operator skills and cheap readily obtained equipment. Even the increase in volume of waste resulting from this process may not be a disadvantage where only small volumes of organic liquid waste are expected to arise.

4.2. Process selection

4.2.1. General

This section of the report attempts to provide guidance to Member States in Groups A, B and C in the selection of appropriate processes for treatment of radioactive organic liquid wastes. It is assumed that the differences in volumes, types, and radioisotopes content of wastes for the Member State groups will present different requirements which may influence an individual state's preference for a given process.

Typical volumes and characteristics of organic liquid wastes and the importance of this characterization have been emphasised in Section 2 of this document. This information has to be taken into account in the selection of the waste treatment process. However, a special characteristic of scintillation fluid and the miscellaneous solvent streams is the potential for solvent recovery and reuse. This feature provides a cost benefit and should not be overlooked in the selection process.

A major factor in process selection is the available resources. With regard to personnel it is considered necessary that supervisors have appropriate training and skills in radioactive waste management practices to ensure process operations meet the appropriate safety, regulatory, and quality assurance standards. Plant operators require adequate skills to operate the process equipment correctly and it may be appropriate to select processes which have parallels in the indigenous non-nuclear industries.

Since aqueous and solid radioactive waste volumes greatly exceed those of organic liquid wastes, it is likely that priority will have to be given to their treatment. In the majority of cases, processes and equipment selected for the treatment of aqueous and solid wastes can be adapted to the processing of organic liquid wastes and combined processing could be cost effective. For example, cementation can be used for the direct solidification of organic liquid wastes as well as the solidification of aqueous and solid wastes. Incineration or wet oxidation treatment of organic liquid wastes produce inorganic residues which thereafter can be treated with other inorganic wastes.

Where dedicated equipment for the destruction of organic liquid wastes is desired, equipment cost, versatility for the treatment of a number of organic liquid wastes, equipment availability, reliability in operation, and ease of maintenance are all factors to be considered in the selection process.

4.2.2. Process combination guidelines

Often, substantial advantages can be accrued by selecting a combination of two or more processes, rather than a single process, for the treatment of organic liquid wastes. For example, the multiple process approach may allow resource recovery, convert the organic material into an inert inorganic waste, provide volume reduction, or allow processing in equipment designed for solid wastes. Figures 12, 13 and 14, suggest some possible process combinations for treating scintillation liquids and miscellaneous solvents, oils, and solvent extraction wastes, respectively. Some of the advantages and disadvantages of each combination are also presented.

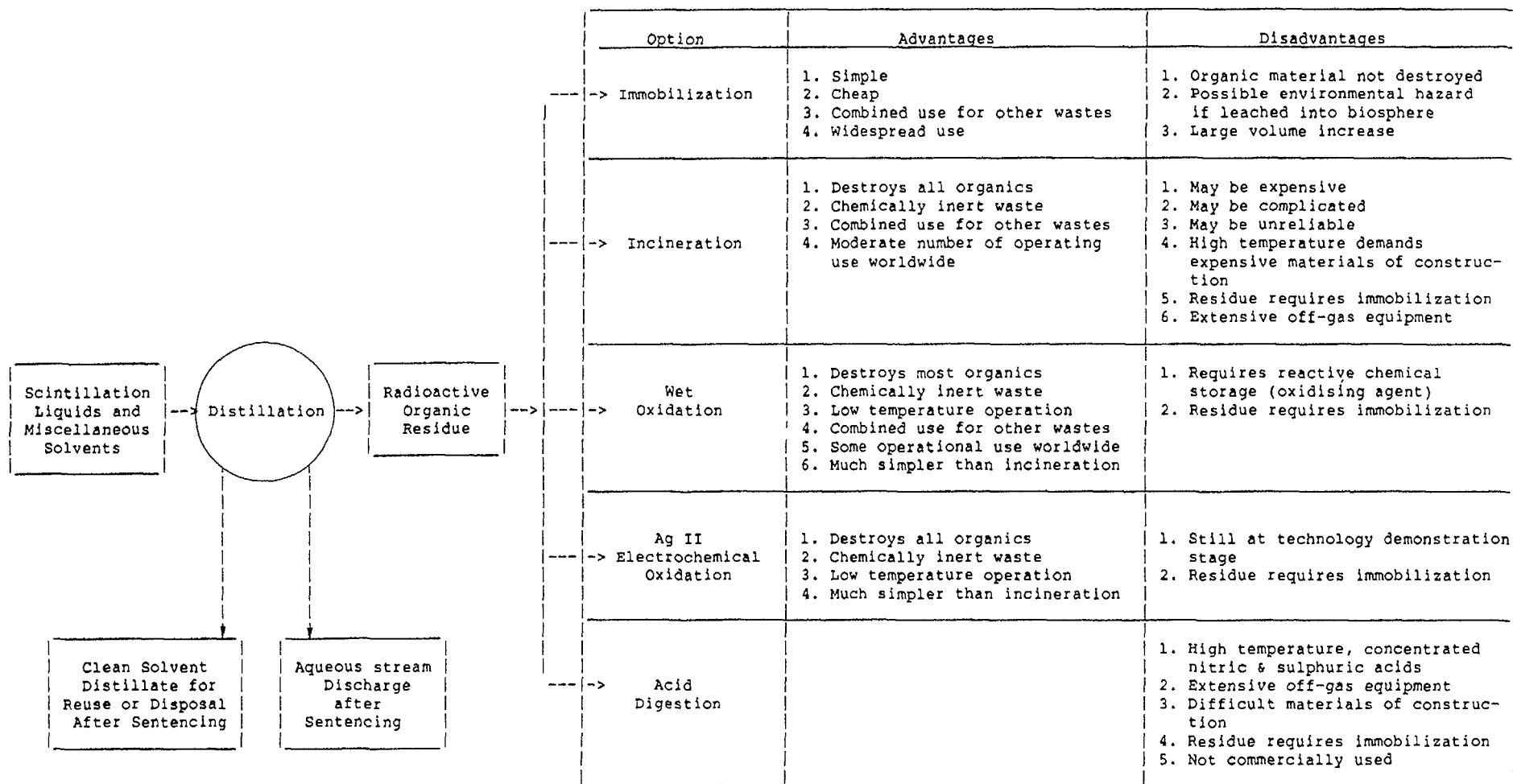


FIG. 12. Combination of process options for treating scintillation liquids and miscellaneous solvents.

Option	Advantages	Disadvantages
<div style="border: 1px solid black; display: inline-block; padding: 2px;">Oils</div> ---> Absorption ---> Immobilization	<ol style="list-style-type: none"> 1. Simple 2. Cheap 3. Combined use for other wastes 4. Widespread use 5. Absorption pretreatment preferred, because of higher waste loading 	<ol style="list-style-type: none"> 1. Organic material not destroyed 2. Possible environmental hazard if leached into biosphere 3. Large volume increase
---> Absorption ---> Incineration	<ol style="list-style-type: none"> 1. Destroys all organics 2. Chemically inert waste 3. Combined use with solid waste incinerator 4. Moderate number of operating units worldwide 	<ol style="list-style-type: none"> 1. May be expensive 2. May be complicated 3. May be unreliable 4. High temperature demands expensive materials for construction 5. Residue requires immobilization 6. Extensive off-gas equipment
---> Incineration	<ol style="list-style-type: none"> 1. Destroys all organics 2. Chemically inert waste 3. Simple incinerator possible 4. Moderate number of operating units worldwide 	<ol style="list-style-type: none"> 1. May be expensive 2. May be complicated 3. May be unreliable 4. High temperature demands expensive material for construction 5. Residue requires immobilization 6. Extensive off-gas treatment 7. Requires dedicated incinerator
---> Wet Oxidation	<ol style="list-style-type: none"> 1. Destroys most organics 2. Chemically inert waste 3. Low temperature operation 4. Combined use for other wastes 5. Some operational use worldwide 6. Much simpler than incineration 	<ol style="list-style-type: none"> 1. Requires reactive chemical storage (oxidising agent) 2. Residue requires immobilization

FIG. 13. Combination of process options for treating oils.

Option	Advantages	Disadvantages
-> Immobilization	<ol style="list-style-type: none"> 1. Simple 2. Cheap 3. Combined use for other wastes 	<ol style="list-style-type: none"> 1. Organic material not destroyed 2. Possible environmental hazard if leached into biosphere
-> Distillation ---> Incineration	<ol style="list-style-type: none"> 1. Destroys all organics 2. Chemically inert waste 3. Combined use with other wastes 4. Distillation provides decontamination of incinerator feed 	<ol style="list-style-type: none"> 1. May be expensive 2. May be complicated 3. May be unreliable 4. High temperature demands expensive materials for construction 5. High corrosion rate 6. Extensive off-gas treatment 7. Distillation provides radioactive aqueous stream requiring immobilization
-> Alkaline Hydrolysis ---> Incineration	<ol style="list-style-type: none"> 1. Destroys all organics if alkaline hydrolysis supplemented with acid hydrolysis, otherwise organic is water-soluble 2. Substantially inactive incineration 	<ol style="list-style-type: none"> 1. Not developed for other organic liquid treatment 2. Phosphoric acid corrosion if acid hydrolysis step included. 3. Residue requires immobilization
-> Ag II Electronic Oxidation	<ol style="list-style-type: none"> 1. Destroys all organics 2. Chemically inert waste 3. Low temperature operation 4. Much simpler than incineration 	<ol style="list-style-type: none"> 1. Still at technology demonstration stage 2. Residue requires immobilization
-> Wet Oxidation ---> Incineration	<ol style="list-style-type: none"> 1. Chemically inert waste 2. Low temperature operation 3. Combined use for other wastes 4. Some operational use worldwide 	<ol style="list-style-type: none"> 1. Requires reactive chemical storage (Oxidising agent) 2. Residue requires immobilization 3. Incinerator maybe required for diluent burning
-> Adduct Formation ---> Incineration	<ol style="list-style-type: none"> 1. Low temperature operation 2. Pilot plant demonstrated 	<ol style="list-style-type: none"> 1. Phosphoric acid corrosion of materials 2. Extensive equipment 3. Incinerator required for diluent burning 4. Residue requires immobilization

Solvent
Extraction
Liquids

FIG. 14. Combination of process options for treating solvent extraction liquids.

5. QUALITY ASSURANCE

5.1. Quality assurance requirements

A comprehensive quality assurance programme for the management of organic liquid wastes should be prepared and operated. Guidance on the development and implementation of such a programme may be found in the Code on the Safety of Nuclear Power Plants: Quality Assurance (IAEA Safety Series No. 50-C-QA) Rev.1 and other Safety Series Reports [4, 5].

5.2. System requirements

Quality assurance requirements should be established for all phases of the waste management process to ensure that each waste is correctly processed and the final waste form has the required properties. This requires quality assurance parameters to be specified. Performance data must be documented for each parameter in order to demonstrate compliance. The quality assurance programme should cover the following aspects:

- waste characterization,
- waste management process specification,
- processing conditions,
- product specification,
- transport,
- storage, and
- disposal.

6. INTERIM STORAGE OF CONDITIONED WASTES

After the waste has been immobilized, the waste packages are normally placed in an interim storage facility for a period which may last for many years. The main reason for interim storage of the wastes is because a repository site is not immediately available. Also, interim storage can be used to take advantage of any activity decay with time, thereby facilitating later handling, transport and disposal of the wastes. Until repositories are available, the national interim storage facilities for conditioned waste can be developed in several different ways.

A simple way, especially for Member States not operating a complete nuclear fuel cycle, is the use of a large transportable container normally used as a shipping container (Figure 15). The container could be set up at a suitable place, i.e. at a centralized collection site, in a small nuclear research centre, nuclear power plant or a guarded area under government control. Depending on the size, between 40 and 70 drums could be stored within a container serving as a barrier against unauthorized contact with the waste. Later on when a repository is available, the container, including waste drums, can be transported directly without additional reloading steps.

Another approach for interim storage of conditioned waste, especially for Member States having a small nuclear research centre, is the erection of a simple hall on the ground surface with a steel construction and corrugated transit sheets covering the walls and the roof (Figure 16). The storage hall should be built above groundwater level and not be reached by a potential flood or groundwater. Where this is not possible, the building must be constructed with appropriate protective systems to prevent the inleakage of groundwater. The capacity for the waste storage facility should be designed for a period of 10 years.

The possibility of capacity extension should be provided for in the design of the facility.

To prevent radiation exposure to on-site personnel, it is recommended that the interim storage facility should be constructed away from waste treatment plants or other buildings.

At the end of the interim storage period, the waste containers must be capable of being identified, retrieved and transported to the final disposal site. The final disposal of conditioned wastes is not covered in this report as the subject has to be considered under the special situation of the respective Member States.

The storage of radioactive wastes is considered in detail in a related document [6].



FIG. 15. Large scale container used in the Nuclear Research Centre Karlsruhe for interim storage of radioactive wastes.

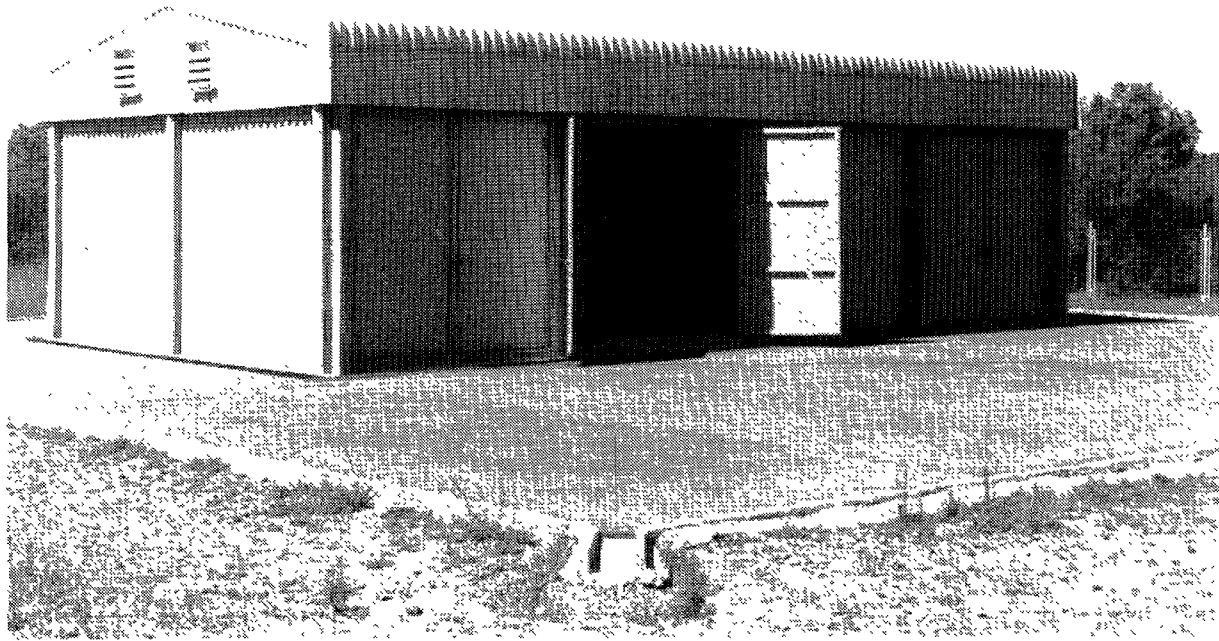


FIG. 16. Simple hall for interim storage of conditioned radioactive wastes in Chile.

7. PROCESS SAFETY

This Section covers the safety aspects which need to be considered in the handling and processing of organic liquid radioactive wastes. The special hazards associated with these wastes are: flammability, toxicity and explosion, as well as radioactivity. These dictate that the receiving and initial handling area has to provide radioactive containment, have suitable fire protection systems, and adequate ventilation. In addition, because the waste contains organic liquids it is necessary to have a suitable collection sump in the event of spillage.

Personnel protection will need to meet ICRP radiation recommendations. In addition, suitable protective clothing, rubber gloves, face mask and visors should also be provided. All the previously outlined requirements are necessary in the waste processing area.

In the development of a radioactive waste management centre, Member States should review the process options before selecting a preferred process for inclusion in the facility. At the conceptual design stage it is necessary to include safety analyses.

It will be necessary to assess the radiation exposure of the plant operators on the site, non-radiation workers at the facility and neighbouring public. For the process workers in particular, staffing levels and job functions will need to have dose budgets estimated.

Hazard and operability (HAZOP) studies on the plant and process at the conceptual design stage will reveal any aspects which may require revisions to the design or operating procedures.

The assessment of accident scenarios includes the estimation of the frequency and consequences of possible accidents related to the plant and the materials which it handles. As a result risk assessment analysis is possible.

Radioactive liquid and gaseous discharges should be as low as reasonably achievable (ALARA) and meet regulatory and environmental restrictions.

Details of the safety assessment methodology will be given in a planned technical document on the design of a centralized waste processing and storage facility.

8. CONCLUSIONS

This report provides guidance to Member States belonging to Groups A, B and C who have small volumes of organic liquid radioactive wastes arising from medical and industrial applications, and the operation of nuclear research centres.

1. The wastes must be adequately characterized; chemically, physically, and radiologically as a precursor to waste management.
2. In identifying treatment options, the best practical environmental options (BPEO) should be selected, with radiation doses and discharges as low as reasonably achievable (ALARA).
3. The waste management strategy should include all the steps from waste arising to final disposal.
4. It may not be possible in the short term to undertake all of the waste management steps however, those steps which are undertaken should not foreclose options for the later steps.
5. With liquid organic waste, the process option should aim to confine and concentrate the wastes.
6. The preferred process options will be those that are cheapest to procure and operate, and which satisfy the aforementioned conclusions.
7. The small volumes of organic liquid wastes suggest that coprocessing with other larger waste streams is likely.
8. It is considered important that a central agency should have the responsibility for managing the waste.
9. Several possible process options have been identified for treating organic liquid radioactive wastes. Guidelines are provided for the selection and combination of process options.
10. The need for quality assurance throughout waste management is emphasized.
11. It is suggested that cheap simple structures will be adequate for interim storage prior to disposal.

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