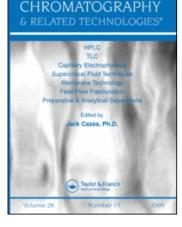
This article was downloaded by: On: 24 January 2011 Access details: Access Details: Free Access Publisher Taylor & Francis Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK

Journal of Liquid Chromatography & Related Technologies

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t713597273



LIQUID

Robots in Radiation Environments

Tony J. Beugelsdijk^a; Dan W. Knobeloch^a ^a Los Alamos National Laboratory Los Alamos, New Mexico

To cite this Article Beugelsdijk, Tony J. and Knobeloch, Dan W.(1986) 'Robots in Radiation Environments', Journal of Liquid Chromatography & Related Technologies, 9: 14, 3093 — 3131 To link to this Article: DOI: 10.1080/01483918608074170 URL: http://dx.doi.org/10.1080/01483918608074170

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Tony J. Beugelsdijk and Dan W. Knobeloch Los Alamos National Laboratory Los Alamos, New Mexico 87545

Abstract

Los Alamos National Laboratory has historically been involved with routine handling of a wide variety of hazardous materials. The majority of activities involve nuclear materials in projects ranging from medicinal uses to weapons technology development.

The robotics development program at the Laboratory is primarily focused on identifying and automating operations that subject personnel to ionizing radiation, are repetitive, and are subject to human error. Development of automated technology for these operations will help meet national nuclear materials processing demands through the transfer of expertise gained at Los Alamos to other Department of Energy facilities.

Robotic equipment that is compatible with glove box environments analytical instrument and in particular LC apparatus does not exist commercially, hence there is a need to design and/or modify systems that can tolerate environments encountered in Special Nuclear Material processing operations. Because of the interaction of atomic fission fragments with the

3093

almost unlimited array of solid materials, developing equipment for this environment involves a wide variety of factors. This report provides a brief review of general considerations for material compatability in radiation environments.

Several different types of robotic applications are currently under development at Los Alamos National Laboratory. Some of the special considerations both in materials compatability and constraints operational are summarized in the individual project descriptions.

Introduction

Hazardous material handling has developed into its own discipline in relation to the broad range of basic research, technology development, and practical applications. In staff and technical capabilities, Los Alamos is one of the largest multidisciplinary, multiprogram national laboratories in the United States (1,2).

About 85% of Los Alamo's total effort is nuclear related. When discussing hazardous material handling at this Laboratory, the topics of major concern are nuclear materials and the equipment required for their safe handling in day to day operations. The Center for Nonlinear Studies, Center for Material Sciences, and Institute of Geophysics and Planetary Physics are gaining recognition for work involving multinational cooperation programs (3).

In support of the demands for reduced exposure to all forms of hazardous materials, improving safety, and developing expertise with new technologies, the Laboratory initiated the formation of a robotics and automation section. This effort is

coordinated through the Materials Science and Technology (MST) Division. The scope of the work in the robotics section also encompasses the development of automated equipment in all areas of research currently being done at the Laboratory. Hazardous material handling activities in the MST robotics section is not limited to nuclear material processing and includes analytical procedures.

Automation Incentives at Los Alamos National Laboratory

The MST Division supports material research and chemical processing research principally focused on advanced methods for plutonium separation, recovery, Automation was immediately suggested and production. because it would reduce the number of labor intensive tasks and also reduce radiation exposure and improve Automation can also allow implementation of safety. additional new processes to handle material throughput and highly active isotopes at lower cost. Thus, MST Division manages that robotics section to develop robotic and automatic systems that will be used primarily in the plutonium facility (4).

Peaceful and safe of radiation isotopes has led to the development of many of the synthesis methods for incorporation them into natural products, drugs, and other compounds (5).

Work with radioactive materials and machines is a common activity throughout the Department of Environment(DOE) complex. The history of operating in such an environment has taught personnel to treat such materials and machines with more respect.

Basic radiation protection policies outlined by the DOE state that radiation exposures be maintained "as low as reasonably achievable", or ALARA. All operations are to be conducted "in a manner to assure that the radiation exposures to individuals and population groups are limited to the lowest level technically and economically feasible".

total of 89,526 DOE and DOE A contractor employees were monitored for whole-body ionizing radiation exposures in 1984 (6). The distribution of the annual whole-body exposures in each of 11 facility categories are summarized in Figure 1. Los Alamos National Laboratory reported 1055 person-rem with 38% received by the Plutonium Facility personnel. This data provides additional justification for the robotic effort at Los Alamos.

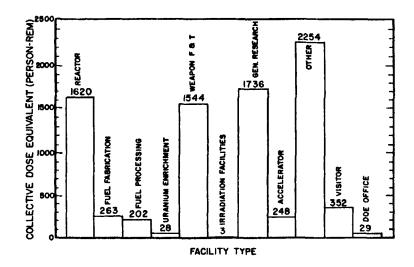


Figure 1. Contribution of each facility type of the total collective dose equivalent, 1984.

FUNDAMENTAL CONSIDERATIONS IN IRRADIATION OF MATERIALS USED IN ROBOTIC SYSTEMS (7).

A. General Irradiation Effects in Crystalline and Non-Crystalline Materials with a View to Their Use in Robot Construction

Atomic fission produces many kinds of radiation or nuclear particles, but only two of these, neutrons and gamma photons, are able to penetrate more than a few centimeters of solid material. Either one of these energetic nuclear particles can interact with matter by collision with individual atoms or electrons.

Because of the almost unlimited array of solid materials, one convenient classification is according to morphology. Crystalline materials, metal, and ceramics as a class are relatively insensitive to nuclear radiation. Noncrystalline materials, and organic materials in particular, are severely damaged by radiation doses thousands of times lower than necessary to affect metal and ceramics detectably.

Organic materials consist primarily of carbon and hydrogen bound together by chemical bonds that are relatively easy to break with the addition of Both gamma rays and neutrons can cause energy. molecular changes which will greatly affect the properties of the material. Fast neutrons can react in several ways. In each collision, incident fast neutrons give up approximately one-half of their energy to the target hydrogen atoms. These recoiling atoms can become ionized, and the slow neutrons resulting from the collisions can react with other nuclei, releasing strong gamma rays. The gamma rays from these capture reactions may cause the formation of new radical groups or free radicals and

ionization. Organic materials are, as a result, inferior to metals with respect to radiation stability.

Factors affecting radiation damage include the amount. and rate of radiation and the type, composition and the volume of the material subjected to radiation. Damage is proportional to the amount of radiation absorbed. Because of the differences in the type and energy of radiation, comparisons of damage to crystalline materials are difficult to make. However, with organic materials, these differences in radiation sources are not so important and it can be assumed that that total energy absorbed determines the amount of damage, whether this energy comes from neutrons, gammas, or both.

Commerically available robotic equipment, as a rule is constructed with metallic compounds capable of withstanding the level of radiation encountered in open hoods, glove boxes and hot cells. The following discussion is provided to give a basic understanding about applying robotic and automation to radiation environments and covers a wide variety of concerns about nuclear fission fragments interacting with matter.

B. Metal Components

The predominant radiation effects in metals result from the presence of either interstitials and vacancies or displacement spikes. Vacancies or displacement spikes are caused by the primary knock-on atom dissipating its energy by actually melting its immediate environment for a short time. This has the effect or arranging most of the atoms in the melted region. The average displacement spike produced by fast neutrons in copper is estimated to be 75A in diameter, a volume which contains 2×10^4 atoms. The following discussion is devoted to the specific property changes associated with these two effects.

1. Electrical Resistivity

Changes in electrical resistivity are perhaps the simplest to interpret from an atomistic viewpoint and one of the most widely studied. Strong correlations exist that allow one to make accurate predictions about the effects of radiation on other properties.

Radiation-induced resistivity increases are of the introduction primarily a result of interstitials and vacancies. These lattice defects introduce local distortions in a crystal which cause the conduction electrons to be scattered more frequently that in an undistorted crystal. This resistivity increase is in many respects similar to the increases that are due to impurities. Both are independent of temperature and for small concentrations are proportional to the number of defects (or impurities) present. Most of the resistivity increases that are introduced bv irradiation will anneal out relatively low at temperatures.

Many useful engineering studies of radiation-induced resistivity changes have been made and a useful rule-of-thumb has emerged. If a proposed metal has an observed resistivity increase when irradiated at temperatures expected in practice, one should make measurements of other properties in which a change would be detrimental. Conversely, if there are no resistivity changes due to irradiation, it is probably safe to assume that there will be no

other property changes.

2. Mechanical Properties

indentation hardness of most metals The is increased as а result of irradiation and is frequently measured in both fundamental studies and engineering test. The radiation-induced hardness increase is of about the same magnitude as that which can be produced by severe cold work; like cold work effects, this hardness tends to saturate after very large amounts of radiation. Yield stress and tensile strength also increase with irradiation and are often accompanied by a decrease in elongation. Here, also, the amount of change in the tensile properties is comparable with that observed as a result of cold work. The radiation-induced mechanical property changes often relax at about the same temperatures as required for cold work effects. With the exception of uranium and certain alloys, radiation-induced changes in the mechanical properties of metals are not great enough to be of much concern from an engineering viewpoint. In our experience, standard robotic equipment rarely fails due to mechanical stress or strain failures caused by radiation-induced materials changes. However, design of custom automatic equipment sometimes requires concern about changes in mechanical properties if the item is part of a structural component or excessive radiation is anticipated.

3. Chemical Properties

Often the choice of metals for radiation environments is not governed by resistivity or mechanical property concerns as much as chemical resistance to the environment. Processes that occur in glove box trains run from parts handling to

complex chemical operations. Chemical mists generated in these latter processes are often corrosive to the metal components. Any corrosion might result in ultimate structural failure of the part or contamination of the process by corrosion products. This latter consideration is of primary concern where high purity materials are being handled or processed. Gas purging of the interior of a robot is an effective technique in eliminating these mists from the small spaces created when the arm moves about in the work envelope.

C. Organic (Polymeric) Components

General Effects of Radiation on Polymeric Materials

Much of the work on the radiation resistance of polymeric components has been carried out under Air Force contracts in studies of various materials deployed on jet fighters and space craft. As such. of the available literature has not been much traditionally referenced by workers in robotic fields. However, much of this work is directly applicable robotic systems in radiation to environments.

Polymeric materials exhibit several types of changes upon irradiation. While most changes depend only on the total dose, some are does-rate dependent because of the influence of dose-rate on free-radical concentration. Some changes are of high enough order to serve as a measure of radiation dose.

Radiation-induced changes have their origin in the rupture of covalent bonds in organic molecules. Effects are small in simple organic compounds, but are much more pronounced in polymers. Among radiation-induced changes in rubber and plastics are

those in appearance, chemical state, physical state, and mechanical properties. Appearance changes with temporary and permanent color effects and bubbling. Chemical changes include double bond formation, dehydrochlorination. crosslinking, oxidation degradation, polymerization, depolymerization, and gas evolution. The new chemical bonds formed by radiation are irreversible and cannot be removed by post-irradiation heating. Physical changes include effects on viscosity, solubility, conductivity, free-radical fluorescence. spectra, and crystallinity. Changes in crystallinity are indicated by measurement of density, heat of fusion, X-ray diffraction, and other properties. Mechanical properties that change are tensile strength, elastic modulus, hardness, elongation, flexibility, etc.

Several reactions occur with irradiation, but the dominant reactions and the rates at which they proceed depend upon the chemical structure of the material. For many plastics and rubbers, the effect is essentially a curing process characterized by an increased in hardness, a decrease in solubility, and sometimes, initially, by an increase in strength. A moderate amount of radiation may be beneficial to these materials, but ultimately, in a radiation field, they lose tensile, shear, and impact strengths and elasticity, and finally become brittle. Gas is often evolved during irradiation. Other degradation symptoms include softening and becoming sticky or eventually disintegrating into a powdery substance. Irradiation also make organic materials more susceptible to oxidation.

Materials in which the curing or crosslinking effect predominates include polyethylene, polystyrene, silicone, natural rubber, neoprene, styrene-butadiene rubber, and Buna-N rubber. Teflon, Kel-F, Lucite, cellulose plastics, butyl rubber, and Thiokol are predominately affected by chain scission and, with consequent softening of the material.

Certain materials, called antirads, are known to inhibit radiation damage. These include aromatic structures, certain antioxidants, and mineral fillers. Inorganic fillers usually increase radiation resistance. Laminates of glass cloth have more structural stability than the resins alone, but fillers such as cloth and paper reduce radiation resistance. Use of these antirads is limited in most automation applications due to the inherent difficulties in shaping, machining, or cutting the actual material.

Sisman and Bopp (8) have determined the effects of polymeric structure on the radiation stability of plastics and have ranked the structural groups in the order of their stability. They found that the benzene group attached to the main chain but not part of the chain backbone provides the greatest radiation polymers, while the presence stability to of quaternary carbon atoms leads to radiation is believed that instability. Therefore, it polystyrene has high radiation resistance because the benzene ring absorbs energy due to resonance within the ring structure.

Certain organic groups are known to be more stable than others. Appendix I summarizes general rules for formulation of radiation-resistance species (9). Given these considerations, we can understand the ordering of the classes of polymeric material presented in Figure 2 and 3. Figure 2 illustrates radiation stability of a series of thermosetting resins while Figure 3 presents corresponding data for thermoplastic resins.

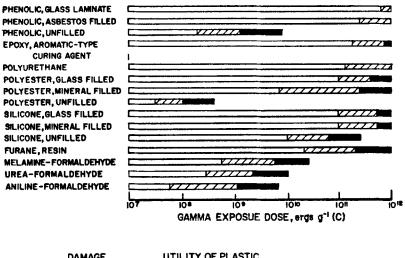




Figure 2. Relative radiation stability of thermoplastic resins.

Note in Figure 3 the relative stability ordering is in agreement with the rules listed above. Also of consequence, not only for use in robot construction, for its of but also use in the automation radiochemical procedures is the position of teflon. Aside from poor gamma radiation resistance, its efficiency in (a,n) reactions results in a source of energetic neutrons. Despite its superior chemical resistance, therefore, teflon is a poor choice for radiation environments.

Life Expectancy of Polymeric Materials

There are a great many variables affecting the service life of a material under irradiation.

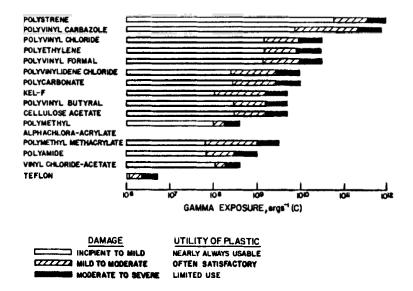


Figure 3. Relative radiation resistance of thermosetting resins.

The cure, filler, and other compounding ingredients each has some effect, and the environment to which the material is subjected will also determine, to some extent, its service life. A fairly extensive tabulation of life expectancies has been made (10). specific applications utilizing polymeric For compounds in automation devices information on service life tends to be based on experience. At the plutonium facility, for example, each glove box train offers a micro-environment wherein radioactivity levels and chemical corrosion can vary. Sweeping generalization on life expectancy are largely non-transferrable as a result.

Effect of Dose Rate On Polymers

Dose rate seems to have an important effect on the degradation mechanism of polymeric materials. In the case of irradiations carried out in air, the degradation process may be regarded as being due to the diffusion of oxygen into the specimens, which yields peroxidation and hydroperoxidation of the radicals formed by the irradiation. The degree to which these reactions affect the physical properties of the material is related to the diffusion rate of oxygen in the matrix and on the geometry of the For electron irradiation the free radical specimen. production rate may be greater than the oxygen diffusion rate causing crosslinking reactions to predominate. In the case of gamma and neutron irradiations, free radical production is sufficiently low to allow the diffusion of oxygen to influence the degradative process thereby causing chain shortening and inhibition of crosslinking. For experimental studies at high dose rates, the time scale of irradiation should be sufficiently long to allow secondary processes to proceed to their full extent before the results can be extrapolated to materials under working conditions.

Radiation Effects on Specific Polymeric Components

a. Coatings

In general for robotics and automation, coatings are to be avoided whenever possible. Bare metal finishes of various textures are preferable. However in some case, e.g. chemically corrosive environments, coatings may be necessary.

Organic coatings are generally based on the following materials: phenolics, furanes, alkyds,

silicone alkyds, vinyls, nitrocellulose, neoprene, styrene-butadiene, or epoxy. Additives such as pigments, plasticizers, and other coating ingredients, as well as the type of surface on which Highly pigmented coatings are generally the coating. radiation resistant than similar more coatings containing lesser amounts of pigments. Coating systems, especially lacquers, employing wash primers become brittle, and poor adhesion between the coating system and the substrate takes place (11, 12).Degradation occurs at lower doses for coatings applied to steel as opposed to coatings applied to other substances. Applied coatings, irradiated while they are wet, do not have the stability observed for coatings which have been dried first. Aluminum and stainless steel, two of the most commonly used metals in radiation environments, generally do not require a coating.

b. Electrical Insulators

most insulators, permanent changes For in electrical properties with irradiation are minor, and life of the insulation the depends upon its resistance to mechanical damage. Most polymers used for insulators harden and eventually become brittle in a radiation field. This results in peeling and especially when flexed. Inorganic chipping, insulators such as ceramics, glass, and mica, and organic-inorganic combinations, such as mica and glass used with silicone or phenolic varnishes, can successfully be in high-temperature used and high-radiation environments. Most of the plastics can be used in medium radiation intensities if their temperature limits are not exceeded. Teflon. however, should not be used due to its poor radiation resistance. Often electrical insulators used in robotic systems can be enclosed in housings which protect them from radiation and chemical corrosion as well as mechanical abrasion.

Elastomers

In general. elastomers are less radiation resistant than other plastic materials. Among the more resistant elastomers are polyuethrane, natural, and adduct rubbers in latex form. Most elastomers increase in hardness when irradiated. The silicon and fluorine containing polymers are the most satisfactory for use above 300°F. Filler loaded elastomers are more radiation resistant than is pure gum stock: carbon black appears to be the best compound's filler for improving a radiation Curing conditions are also important; resistance. advantages in having a slightly undercured compound is indicated. Additional stability can also be attained through use of antirads. These are materials added specifically to absorb radiation and dispel the energy via a non-degradative mechanism. However, antirads are specific in that some are more effective with one type of polymer than another.

the Based on change of its over-all is properties, natural rubber the better among radiation resistant elastomers. Irradiation of natural rubber induces crosslinking. The elastic properties decrease and the hardness of the compound increases giving results similar to those obtained by overvulcanization. On prolonged irradiation, natural rubber acquires a rigidity comparable to that of glass. Polyurethane rubber, a synthetic rubber, is equal to or better than natural rubber with respect to radiation resistance. The presence of benzene-ring structures in the diisocyanate coupling improve polyurethane's agents stability. The aromatic composition and structure the of

polyurethanes promote "endlinking", that is, the chain ends recombine to form crosslinks. Thus physical properties do not deteriorate.

Silicone rubbers see wide use in radiation environments. Damage will vary with the type and amount of irradiation, the composition of material, time of cure, the volume of the sample exposed, and environmental factors. Radiation attacks silicones directly and indirectly by ionizing the molecules, which indirectly leads to the formation of free radicals, ethylenic unsaturation, and molecular rearrangement. As a result, both crosslinking and chain scission occur simultaneously, but not to the same extent. Radiation damage to silicone rubber is less severe in an inert atmosphere than in air.

Plastics and Resins

The physical properties of plastics generally degrade before electrical properties are seriously affected. Electrical resistance gradually decreases with time during irradiation, recovering after removal from the radiation field. Silicone resins, in general, are much more resistant to radiation than silicone elastomers. The major dielectric properties of solventless silicone resin are not damaged by gamma-radiation doses as high as 1,000 megarads.

Laminates

This class of materials consist primarily of glass fabrics laminated with a thermosetting resin. The principle organic binders generally used for these laminates are silicones, phenolics, polyesters, heat-resistant polyesters, epoxy, and heat-resistant epoxy resins. The style of weave used in the glass fabric does not noticeably affect the durability of the finished laminate. None of these materials show radiation-induced anv significant change in electrical properties. Because of nearly constant dielectric constant and loss tangent, the selection of a laminate is determined mainly by structural stability under conditions of heat, stress, and radiation. The main use of laminates in robotic systems is in printed circuit boards rather than structural components and these can generally be removed out of the glove box or effectively shielded in a gas-purged enclosure.

O-rings and Seals

O-rings made of Viton A (a copolymer of hexafluoropropylene and vinylidene fluoride) appears to be a good all round choice for O-ring material. Despite the fact that its radiation resistance is not as great as desired, Viton has good high temperature characteristics and reasonable service life. Principal factors causing seal failure are permanent set and shrinkage after prolonged exposure to high temperatures and pressures. A relatively high squeeze on the O-ring is necessary for sealing over a wide range of temperatures to overcome the effect of permanent set of the O-ring and the difference between the thermal expansion of the seal and that of the sealing land.

OPERATIONAL CONSIDERATIONS

1. Electronics Requirements

A primary concern with robotic and automation electronic control circuitry in radiation environments is the service life of the components employed. Issues concerning the stability of the circuit board laminates have already been addressed

and stem largely from a position of mechanical integrity rather than radiation effects. A common problem is contact radiation from *alpha emitters* that *will eventually destroy semiconductor devices*. The best protection against alpha radiation is to gas purge a shrouding containing these components.

Gamma and neutron radiation causes extensive and unpredictable damage to semiconductor devices and the packaging they come in. Much work has been done to harden such devices. However, it seems impractical present to replace all circuitry with these at hardened components. Where feasible, circuitry can shielded with hydrogen-rich materials such as Kel-F; however the best approach is to physically the robotic control circuitry remove from the radiation environment. Signal and power lines can be brought through glove box walls using service panels. Conductors used inside hot boxes should be covered with a stainless steel braid for mechanical abrasion resistance. proper shielding of the conductors inside helps to prevent spurious signals from being generated by exposure to radiation.

2. Failsafe Modes

Engineering designs must incorporate failsafe operational modes in the event of а power Many robotic systems incorporate interruption. incremental encoding on joint motions. This generally results in a return to a home position upon power up so that all counters can be initialized to a known position. The disadvantage of this feature is a reinitialization sequence from an unpredictable position upon restoration of power to a system. Hence, reinitialization sequences must be very well characterized or avoided altogether upon power up after an outage. A possible solution to this problem

is the use of absolute joint encoders. The robot position can then be determined upon power up and appropriate action decision trees can be determined. gripper devices and other end-of-arm tooling must also have a failsafe mode. For example, the use of mechanical springs to apply gripping force to an object with the use of power only to release objects is indicated. Uninterruptable power supplied can be critical non-failsafe used. especially if and operations are required.

3. Maintainability

Given that there is a limited reach into glove boxes through lead-lined gloves and that manual dexterity is severely compromised, the maintenance of mechanical components within glove boxes is at best difficult. Mechanical designers must be cognizant of these limitations and incorporate features such as interchangeable subassemblies. easilv weight reduction "friendly" fasteners, etc. No critical parts of the robot work envelope should be outside the reach of the gloveports should a mishap occur. In the economy of robot this regard, motion ís secondary consideration to accessibility. Shrouding of the arm and base should be such as to be easily Flexibility bellows are often used decontaminated. to cover mechanically exposed and intricate parts, however, they must be easily replaceable.

REMOTE HANDLING AND ROBOTIC ACTIVITIES AT LOS ALAMOS

Robotic systems development at Los Alamos National Laboratory is driven by several factors. The need to reduce radiation exposure to personnel and accessibility to the need to reduce nuclear materials. Remote control and/or automating the involve the handling operations that of these

materials lessens the possibility of unwanted diversion. Furthermore, using robotic systems to handle transfers and certain glove box operations improves the reliability of the nuclear materials accountability process since the human element of record keeping can be minimized.

Robotic equipment that is compatible with glove box environments does not exist commerically, hence there is a need to modify and/or design systems that can tolerate hot cell environments in Special Nuclear Materials (SNW) production operations.

The robotics development program has four major objectives. The first is to focus on specific areas in the nuclear materials production facility. This is accomplished by identifying operations that subject personnel to ionizing radiation, are repetitive, subject to human errors, and which can be performed with readily available equipment without significant modification.

The second objective is to remedy weaknesses of 'off-the-shelf' robotic and automatic equipment with respect to operation in glove boxes.

The third objective is to tie the robotics development areas together with the new nuclear materials process development activities currently being researched within the production facility.

A fourth and longer range objective will be to build an expert system and artificial intelligence base to complement the robotics development activities.

Robotics tends to be an interdisciplinary field with most of the applications coming from end users. The expertise in robotics, on the other hand, tends to be concentrated in a relatively few fields. effectively communicate across traditional discipline boundaries, a staff with a variety of technical experience training and world has been real assembled. The training includes electrical engineering, computer science, mechanical engineering, and nuclear and analytical chemistry. Experience within the group covers nuclear materials handling and processing, a knowledge of glove box operations, material compatibility, and knowledge of the nuclear materials safeguards and accountability systems and requirements. The support skills drawn mechanical upon include engineering design, fabrication, and electronics. The applications given below do not include LC analyses but many of the procedures could be .related directly to sample preparation and sample processing in LC. It is likely that such procedures will, in due course, be for the LC analysis of radioactive employed materials.

1. Direct Oxide Reduction

Plutonium metal is produced from plutonium oxide by the direct oxide reduction (DOR) process. The procedure calls for introducing three reagents: PuO2, calcium metal, powdered calcium chloride, and a pressed calcium chloride cylinder into a magnesia crucible inside of a glove box. The reagents and the CaCl₂ cylinder are placed in the crucible which is then inserted into a stainless steel can and into a reduction furnace. At approximately 800°C, the reduction reaction starts and stirring of the molten After the exothermic reaction is mixture begins. complete, the components are disassembled and the plutonium metal button is broken away from the bottom of the crucible. The product metal and salt residues are then transferred out of the glove box.

The robotic automation of this process calls for four phases which have counterparts in LC analyses:

- 1. Measure reagents and assemble components
- 2. Control furnace cycle and mixture stirring.
- 3. Disassemble components.
- 4. Separate product and residues, and assay.

An evaluation of the DOR process indicated the need for a precise, medium speed, gantry robot with a wide range of sensory inputs and outputs. The IBM 7565 robot has been selected as the system to implement the automation and to demonstrate the feasibility of the project. A complete set of standard DOR components has been obtained and installed in the robot's workspace. Each of the above phases will be initially implemented with as little impact on the components as possible. This will be accomplished, for example, by using stainless steel instead of the brittle magnesium oxide crucible Lead oxide will be used in cold for the mock-up. simulation since its pyrochemical and physical properties approximate those of plutonium oxide. fume hood is being constructed around the robot to contain lead vapors.

2. Milliwatt Generator Calibration Line

 238 PuO₂ heat sources are routinely produced at Los Alamos. These sources generate heat by the alpha decay of 238 PuO₂ which is converted to electricity by the use of a thermopile. The electricity generated is used to power remote systems such as satellites, and Arctic and subsea installations. The thermal calibration of these heat sources is now carried out manually and is very routine in nature. A typical

Downloaded At: 15:37 24 January 2011

measurement currently takes approximately two hours. A robotic system, based on the Zymark robot, is being developed to perform this operation automatically. The robot has been interfaced to a Hewlett-Packard 9825 desktop computer that operates the two calorimeters and maintains the data base for the results. In addition, it controls the Zymark robot through a custom interface whenever sample change out is required.

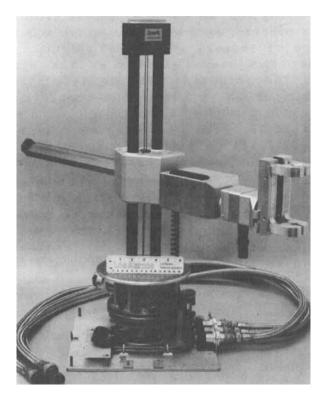


Figure 4. Radiation-resistant modifications to the Zymark laboratory robotic arm showing the remote electronics and the all metal construction.

Because the robot will be operating in a radiation environment, several modifications were made to the robot arm. These include all aluminum parts, a remote electronic control box, and dry lubricant bearings. Figure 4 shows some of these modifications are consistent with the special considerations for robot construction and materials compatibility mentioned above.

3. Isotope Detector Fabrication

In this process, which takes place in controlled atmosphere glove box, highly radioactive oxides are subdivided, weighed, and placed into a capsule which is then welded shut. Operational parameters include a controlled atmosphere and non-routine welds. A special fixture eliminates the need for the robot to perform the welding function. The robot selected for this application is Precision Robot Inc., Model PRI-1000. The clean room compatible robot is designed for small parts work (up to ten pounds). The y-axis of the robot can be extended up to twenty feed allowing the five axis arm to produce accurate linear motions within its work envelope. This work envelope allows the peripheral equipment used for oxide handling to be arrayed so that other manual operations can be performed in the The payload capacity is suitable same glove box. for using commercially available tool changing equipment. The clean-room construction allows for complete containment of the robot arm and track polvurethane bellows within а to prevent contamination and easy clean up of the system.

4. Automatic Bagout System

Much of the waste generated inside of glove box trains is extracted in one gallon cans. Removal of

these cans from the glove box is called a "bagout". This operation involves extracting a can from the glove box through a special port into a plastic bag without breaching the atmosphere of the box. This is a relatively high exposure task which requires suiting up in protective clothing and the wearing of a respirator. As such it is a very tedious task and one of the less desirable activities associated with nuclear materials processing.

An automatic bagout system is currently under development using a six-axis robot manufactured by American Robot Co. As the can is extracted by the a plastic bag collapses around it. robot, A gathering and clipping mechanism necks down the bag between the extracted can and the glove box port. Four metal clips are applied at this point, isolating the waste can from the glove box. A pneumatically driven knife severs the bag between the two inner clips with two clips each sealing the newly formed ends of the bag.

The will American robot be working in conjunction with a robot of our own design which will be compatible with this environment inside the glove This second robot will be feeding cans from box. inside of the box to the bagout port so that they can be grabbed by the American robot. A cold glove box is in place and has been fitted with a port designed for this application. A fail-safe gripper has been developed and is functioning on the robot arm (see Figure 5). The bag gathering and sealing mechanism has been fabricated and is in the process of being The integrated into the system. complete configuration will also include remote TV cameras so that each state of the operation can be watched by remotely-sited personnel. This extra precaution is taken because of the hazards associated with

breaching the glove box atmosphere.

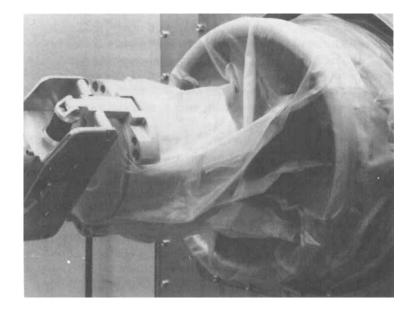


Figure 5. The failsafe custom gripper removing a waste can in a simulated bagout operation.

Although some time saving will be realized through robotic automation, the reduction in radiation exposure and elimination of an unpleasant task are overriding motivators for this project. An additional benefit will be the ability to do continuous bagouts in contrast to the current batch mode of operation.

5. Environmental Gamma Spectrometers

The Health, Safety, and Environment Division is in charge of monitoring the environment around Los Alamos County to insure that radioactive contamination is not being released due to the many activities of the Laboratory. To discharge this role, the division operates a gamma spectroscopy laboratory where environmental samples (i.e., soils, rocks, grasses, etc.) are counted for activity. Due to the extremely low activity of these samples, long counting periods (10,000 seconds) are commonly used. Hence, in an eight hour day, no more than three samples can be measured on any of the six counters in the laboratory. To make more efficient use of the counting equipment and to use the evening and weekend hours more productively, a robotic system is being developed to automatically change samples in each counter at the conclusion of each counting period.

The robot chosen for this application is the PRI Model 2000. This system was specified with a y-axis Reach in the x-and z- dimensions is 22.5 or 17 feet. The long y-axis makes this an ideal robot inches. for servicing these counters when arranged in a The payload (10 lbs.) and the manner. linear reproducibility (+/~ 0.004") are also well-suited to In use, the counters will signal the this task. robot to change samples, and when a new sample has been introduced, the robot will signal the counter to start a new counting period. Data transfer to a mainframe computer will occur for the previous sample during a sample changeout. No modifications were required for this robot due to the extremely low levels of radioactivity encountered with these environmental samples.

6. Nevada Test Site Solution Dispensing Station

Core samples taken from 'drill backs' after test firings at the Nevada Test Site are analyzed by the Isotope and Nuclear Chemistry Division. Preparation of these samples is done in a glove box train and consists of washing, selection of subsamples, and size reduction. After removal to a fume hood, the powders are then dissolved with various acids. Each preparation is placed in a cleaned commercial acid reagent bottle. Four to six bottles result from each test shot. Aliquots are taken from each bottle by the different analytical chemistry sections for isotopic various determinations. The sizes and numbers of these aliquots are determined in advance and entered into a ledger for each test shot. Analyst this ledger before withdrawing refer to their aliquots.

A robotic system has been developed to automate the dispensing operation, a procedure, in fact, often used in LC sample preparations. Several factors motivated this project including reducing radiation exposure, eliminating the incidence of spills, and more error-free record keeping. Moreover, since the aliquots are now weighed instead of being dispensed by volume, more precise analytical determinations are possible. A unique design requirement for this project included the development of a solution dispensing station that would to itself be contamined by the radioactive solution being transferred. Such a pump station was built using peristaltic pumps and three-way pinch valves.

A dedicated personal computer is interfaced to the robot controller so that all pertinent test shot information (i.e., number of assays, number of aliquots, destination of each aliquot, etc.) can be entered in an interactive session by the operator. Extensive error-checking was also incorporated so that the correct number of aliquots, the correct container size, etc., are verified before the dispensing operation is begun. Due to the low levels of radioactivity for this part of the operation, no modifications were made to the Zymark robot used in this project. A corrosion resistant version of their standard robot was used because of the hydrochloric acid fumes present. The system has been installed and has seen service for several test shots.

7. Acid Dissolution of Plutonium Metal

Plutonium metal and oxide samples are generated in the Plutonium Facility and are submitted to the Analytical Chemistry Group for analysis. Pieces of metal and oxide are subdivided from the original These subsamples are cleaned and weighed samples. distributed for containers which are then into various compositional analyses. Samples sizes and recorded destinations are for accountability Many of the sections performing these purposes. analyses dissolve their samples in acid. These solutions or aliquots thereof are then used in the actual analytical procedures.

Much time can be saved by performing one dissolution on a larger sample and then dispensing aliquots to the individual sections. This procedure is currently being implemented manually.

automate this dissolution/dispensing Τo procedure, a robotic system is currently being developed using a commerically available robot. Extensive modification of this system is complete and includes remoting all electronics and replacing all plastic components with metal. Other components of the robotic system include a barcode reader, a barcode label printer/applicator system, and a dedicated personal computer for supervisory control of the workcell, data entry, and interaction with a computer data base for accountability purposes. The entire system will be placed inside a standard 5'x6'

glove box. Components which will need to be accessed will be located near glove ports. As mentioned before, a secondary consideration in workcell layout is the economy of robot motion.

8. Beta Test of the Perkin-Elmer Robot System

In additional to developing robotic systems for nuclear applications, the Robotics and Automation Section also serves in a consulting capacity to recommend robotic equipment appropriate to various automation problems. During the past three years, we have developed an extensive technological base of commericial robotic hardware and software. An ongoing activity within the section is the evaluation of robotic systems and components.

There are several successful laboratory robotic implementations at Los Alamos National Laboratory based on the Zymark Corporation Zymate[™] system. Α Beta-test site agreement with Perkin-Elmer Corporation was entered to evaluate their Masterlab 9000™ robotic for system use in radiation Having both systems has provided the environments. opportunity to make critical comparison between the This assessment is currently in progress and two. will be the subject of a future publication.

9. Sample Preparation for the Radiochemical Analysis of Plutonium and Americium.

A Zymark Corporation robotic system has been assembled and programmed to prepare samples for plutonium and americium analysis by radioactive counting. The system can perform two procedures: (1) a series of simple dilutions and, (2) a liquid-liquid extraction of plutonium from americium. To perform these procedures, the robotic system executes eleven unit operations such as weighing, pipetting, mixing, centrifugation, calculation, etc. Approximately 150 programs requiring 64 kilobytes of computer memory control the system. The entire robotic system is enclosed within a plexiglass hood for control of contamination (see Figure 6).

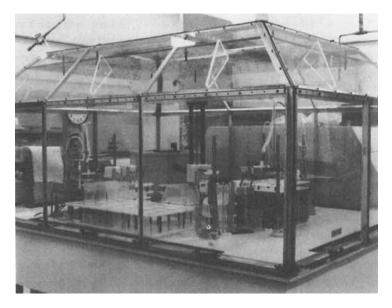


Figure 6. Downdraft hood enclosure and robotic workcell for the radiochemical plutonium and americium sample preparation system.

The robot is currently being tested with high-purity plutonium metal samples and has produced results that agree to within 5% of the values obtained for plutonium spectrophotometric measurements and weight titrations. Throughput of the system is four samples per hour. The system has been preparing actual radioactive samples since February of 1985. No modifications to the robot were deemed necessary for this application.

10. Monitor (13).

Prior to the organization of a robotics group at Los Alamos, the Meson Physics Division developed the Monitor system. Monitor (see Figure 7) is a remote handling teleoperated system that was developed to repair, replace, and maintain the main experimental beam line at LAMPF.

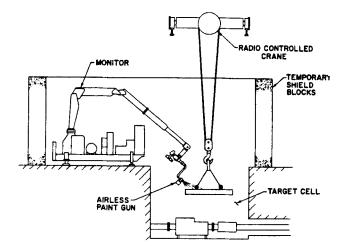


Figure 7. The Monitor arm being deployed in a paint spraying operation inside the LAMPF facility.

It has been operational since 1976, and helps assure the continual operation of the LAMPF. The Monitor system is based on using distance for shielding, electric master-slave servomanipulators and a video monitor system for viewing. It consists of these primary components: A slave unit is placed at the worksite and consists of a one-tone hydraulic crane that is used to place the electric master-slave servomanipulators at the specific work area. It also has multiple closed-circuit television cameras for proper viewing of the work area. It provides air, hydraulic, and electrical power connections to run the various tools as well.

A control station is housed in a trailer that provides a quiet, isolated location in which to do the demanding job of remote handling. It provides the masters for the electric master-slave manipulators, hydraulic crane controls, camera controls, tool power controls, as well as numerous television monitors and other operating status indicators.

A 30-ton radio-controlled building crane for handling objects beyond the capability of the manipulators.

The original system, developed in 1976 has been expanded to two identical systems capable of working within the main experimental hall at LAMPF. A third system, which is just becoming operational has a portable power generator, thus making it possible to operate in any area accessible by truck.

Since 1976, the Monitor systems have developed so that the operating personnel are capable of performing many of the tasks done by a high-level technician. Examples of these skills are:

Welding - arc and MIG for stuctures;

Brazing/soldering --silver and soft;

Installation and removal of threaded connections, tube fittings, and vacuum systems;

Drilling, tapping, and sawing;

Sandblasting, grinding, polishing, and painting;

Flame cutting of steel to 8 inches thick.

CONCLUSIONS

We have attempted to address some of the issues of implementing robotics in radiation environments. Examples of the application of these principles are taken from past and current projects. Although the specific use of the radiation resistant robot to LC analyses is not addressed, the unique procedures and operations are common to those used in sample preparation, derivatization and other processes common to LC analysis

REFERENCES

 B.J. O'Keefe, Nuclear Hostages, Boston, MA.: Houghton Mifflin Company, 1983.

2. L. Badash, J.O. Hirschfelder, and H.P. Broida, ed. Reminiscences of Los Alamos, 1943-1945, vol. 5 Holland/Boston: D. Reidel Publishing Company, 1980.

3. Los Alamos National Laboratory, Research Highlights 1985. Los Alamos, NM.: Los Alamos National Laboratory Printing Office, LALP 1986, 85. 4. Los Alamos National Laboratory. Material Science and Technology 1985 Review. Los Alamos, NM: Information Services Division, LALP 85-11, 1986.

5. Los Alamos National Laboratory. Isotope and Nuclear Chemistry Division Annual Report. Springfield, VA: National Technical Information Service, LA-10366-PR, 1985.

6. Pacific Northwest Laboratory. Seventeenth Annual Report On Radiation Exposures For DOE and DOE Contractor Employees - 1984. Richland Washington: Department of Energy, 1985.

7. C.R. Tipton Jr., ed. Reactor Handbook, Materials vol. 1 New York: Interscience Publishers, Inc., 1980.

8. O. Sisman and C.D. Bopp, Physical Properties of Irradiated Plastics, Oak Ridge, TN: Oak Ridge National Laboratory - 1951, pg. 928.

9. A.J. Mione, Materials in a Nuclear Environment, Talk given at the 25th Annual Meeting of the Aeronautical Science, New York City, January 29, 1957.

10. R.W. King, N.J. Broadway and S. Palinchak, The Effect of Nuclear Radiation on Elastomeric and Plastic Components and Materials, Report No. 21, Columbus, OH: Radiation Effects Information Center, Battelle Memorial Institute, 1961.

11. J. Ohr, Studies of Organic Coatings Exposed to Gamma Radiation, Philadelphia, Pa.: Naval Material Center., NAMC-AML-AE-1089, Partial Report, 1959.

12. J. C. Bresee, C.D. Watson and J.S. Watson, Gamma Radiation Damage Studies of Organic Protective Coatings and Gaskets, Oak Ridge, TN: Oak Ridge National Laboratory, ORNL-2174, W-7405-Eng-26, AEC, 1956.

13. D.L. Grisham and J.E. Lambert, Monitor, The Prelude to Robotics, Los Alamos, NJ: Los Alamos National Laboratory, 1985.

APPENDIX

Rules of Thumb for Radiation Resistant Polymers

1. Aromatics are more stable than aliphatics because of the resonance energy and greater bond strength of the aromatics. The greater the resonance energy, the greater the stability. The order of decreasing stability is anthracene, naphthalene, benzene, and aliphatics.

2. Substituted aromatics are more resistant than non-substituted aromatics. A side group acts as a point of entry for energy to enter the ring and be dissipated. The order of decreasing stability is ortho, para, and meta substituted groups.

3. Basic compounds are more stable than acid compounds because the bonds of the COOH group are relatively weak.

4. The order of decreasing stability is alkanes, alcohols, and ketones. ethers. esters, These the relative relations may due to be electronegativity and relative bond strengths.

5. Saturated aliphatic structures are more stable than unsaturated aliphatic structures. Compounds with terminal unsaturated are less stable than compounds with interior unsaturated.

6. Small molecules are more stable than large molecules. Smaller volume per molecule means less absorption per molecule.

7. Non-branched chains are more stable than branched chains. There is a greater possibility of crosslinking in branch chains.

8. Trans-isomers are more stable than cis-isomers and equatorial groups are more stable than axial groups. Steric hindrance of axial groups promotes instability.