AECL HOT-CELL FACILITIES AND POST-IRRADIATION EXAMINATION SERVICES

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ABSTRACT

This paper presents an overview of the post-irradiation examination (PIE) services available at AECL’s hot-cell facilities (HCF). The HCFs are used primarily to provide PIE support for operating CANDU power reactors in Canada and abroad, and for the examination of experimental fuel bundles and core components irradiated in research reactors at the Chalk River Laboratories (CRL) and off-shore. A variety of examinations and analyses are performed ranging from non-destructive visual and dimensional inspections to detailed optical and scanning electron microscopic examinations. Several hot cells are dedicated to mechanical property testing of structural materials and to determine the fitness-for-service of reactor core components. Facility upgrades and the development of innovative examination techniques continue to improve AECL’s PIE capabilities.

INTRODUCTION

AECL has a long history of providing PIE services to CANDU reactors in Canada and abroad. During the era of fuel development for the first CANDU power reactors the HCFs were used primarily for the examination of experimental fuel bundles and components irradiated in the research reactors at the CRL and the Whiteshell Laboratories (WL). As CANDU reactors have matured, this role has broadened to include comprehensive examination and analysis services on all types of irradiated fuels and components requiring remote handling in support of reactor surveillance and plant-life extension (PLEX) activities. AECL has recently expanded its PIE services business by contracting to do hot-cell work for vendors of commercial light-water reactor (LWR) fuel in the United States, and examination of irradiated materials for customers in Europe and the UK.

The wide variety of PIE services offered at AECL places a heavy demand on its HCFs and equipment. Recent technological innovations and facility upgrades, which have significantly improved AECL’s examination and analysis capabilities, are described.
HOT-CELL FACILITIES

The shielded facilities for PIE at AECL comprise the Universal Cells and Fuels and Materials Hot Cell Facility (FMHCF) at the CRL, and the Shielded Facilities (SF) at the WL. All these facilities are operated and administered by the Radioactive Materials Services Branch.

There are three Universal Cells, housed in Building 234 at the CRL site. UC-1 is used primarily for isotope production and development. UC-2 is used for spark machining flaws in reactor pressure tubes and burst testing them, and for receiving the interproject shipping flasks. UC-3 is used for welding and machining pressure and calandria tubes, assembly and disassembly of loop fuel strings, fuel bundle/element visual examination, profilometry and other measurements. It can accommodate fuel assemblies up to 3.6 m long and 15 cm in diameter. Examinations of full length LWR fuel rods are performed in this cell.

The FMHCF, known more commonly as the “metallurgical cells” is located on the basement floor of building 375. A major refurbishment of this facility, completed in 1987, was presented at the 2nd International Conference on CANDU fuel [1]. It is used primarily for the destructive examination of irradiated fuels and materials. A variety of operations are performed, from visual examination and fuel element leak testing to gamma scanning and optical microscopy. Three specialized cells, MTC-1, -2 and -3, located in this facility, are dedicated to the mechanical testing and examination of non-fissile materials.

The Shielded Facilities (SF) at the WL site include the HCF and the Immobilized Fuel Test Facility (IFTF). The maximum dimensions of fuel assemblies or reactor components that can be received and handled directly in the HCF are 8 m in length by 15 cm in diameter. The HCF provides a full range of PIE services for CANDU customers. The IFTF provides space and facilities for a wide range of experiments using radioactive materials in support of the Canadian Nuclear Fuel Waste Management and CANDU reactor safety research programs.

Some general specifications and functions for all the hot-cells are given in Table 1.

AUXILIARY FACILITIES

The spent fuel (rod) bays associated with the NRU reactor serve a multi-purpose function in delivering PIE services. The bays can accommodate the unloading of large flasks (NLI-1/2, LWT), that cannot be unloaded directly into the hot cells. Fuel and fuel channel components are stored in the bays for an interim period while awaiting examination in the hot cells and some PIE tasks, such as visual inspection and leak testing of fuel bundles, can be performed. Visual examinations are performed with a portable field-model Questar telescope and remote TV camera. Areas of interest are photographed by attaching a Hasselblad camera with a Polaroid magazine or a 35-mm camera to the telescope.

After unloading a length of pressure tube, the initial visual examinations, photography and gamma-field measurements are made in the rod bays. Sections of pressure tube can also be
transferred to special racks for ultrasonic and eddy-current inspections. Components that are too
large to be transferred to the hot cells in one piece are sectioned using an abrasive cut-off wheel.

POST-IRRADIATION EXAMINATION SERVICES

Visual Inspection - Bundles and Elements

Hot cells at each of the facilities are equipped with periscopes and stereo microscopes for
viewing and inspecting bundles and elements. The periscopes in the Universal Cells can be
interfaced with a CCTV camera for video monitoring and recording, a Polaroid camera, or a 35-
mm camera. Video monitoring is very useful for viewing large objects and for close-up viewing
of machining operations. More detailed examination is performed with stereo microscopes that
have a magnification range of 3X to 12X and provide large depth-of-field viewing. A remotely
operated wall-mounted stage is used to manipulate items for viewing, and a rotating device can
be set on the stage for full circumferential inspection of cylindrical objects.

Dimensional Measurements - Bundles and Elements

Measurement of dimensional changes after irradiation is important to monitor the axial and
diametral strains that result from pellet-clad interaction and internal gas pressure. Diametral
expansions at the mid-pellet and pellet interface positions are measured regularly on discharged
fuel elements to ensure that sheath strains are within acceptable limits. Changes in element
diameter and bow can also affect the thermalhydraulic performance of the bundle.

Length changes in both bundles and individual elements are measured. The bundle length
measurement apparatus consists of a base plate with motor-driven rollers and a yoke fitted with
an anvil and displacement transducer that contacts the outer face of the end plate. Length
measurements on individual elements are performed with an apparatus comprising a base plate
with two adjustable V-blocks, with brackets at each end to hold an anvil and a dial gauge. Thermocouples in contact with the sheath measure sheath temperature so that length
measurements can be normalized to 20°C.

Both the CRL and WL profilometers are constructed from a modified 1.2-m-long South Bend Lathe. A linear variable differential transducer (LVDT), attached to the fuel carriage,
measures the change in diameter along the length of the fuel element. The original single
transducer system was replaced with a two-transducer system at CRL in 1980 (L.N. Herbert,
unpublished data) to provide simultaneous measurement of element diameter and bow, and to
simplify hardware setup for different types of elements. The addition of chisel-tip probes
minimized the effect of fuel element bow and sag on diameter measurement. The SF at WL still
use the single transducer system that allows measurement of bundle diameter.

The profilometer at CRL was recently upgraded to provide computer control and digital
recording of the data (J. Montin, D. O'Brien and R. Moeller, unpublished data). The reversing
motor used to move the transducers, and the manual mechanism used to rotate the fuel element were replaced with computer-controlled stepping motors. Custom software was written for the stepping motors to control the movement of the transducers and the rotation of the fuel element. The transducers were replaced and new collets were fabricated to hold the elements and provide calibration steps. The upgraded profilometer can measure diameters to within 0.010 mm, with a precision of 0.004 mm (2σ), and it meets the accuracy standard of ±0.1% for CANDU fuel-element diameters.

**Corrosion Film Thickness Using FTIR Interferometry**

Corrosion film thickness measurement is an integral part of the PIE of zirconium alloy components removed from nuclear reactors. Under normal operating conditions, corrosion and the associated hydriding of zirconium alloys are important factors affecting the integrity of reactor core components. Interferometry is a non-destructive and non-contacting technique that can be used for the accurate estimation of oxide film thickness from the spacing of interference peaks in the mid-infrared. AECL has developed the application of Infrared Reflection-Absorption Spectroscopy (IRAS) to measure corrosion film thickness on irradiated zirconium alloy components [2]. Corrosion films on fuel elements discharged from CANDU reactors have been examined by IRAS in the CRL HCF [3]. These films are normally thin and IRAS offers a non-destructive and non-contacting technique with good sensitivity for the investigation of the effects of cladding microstructure, coolant chemistry and surface deposits on corrosion.

When compared with the usual procedure of metallographic examination, the rapidity and non-destructive nature of the spectral measurement eliminates the various steps in preparing cross sections and saves valuable hot-cell time. IRAS demonstrated this advantage during the Pickering and Bruce re-tubing projects by measuring oxide film thickness on several hundred pressure tube samples in a very cost-effective manner.

The current hot-cell system is limited to accurate measurement of thicknesses less than 25 μm. In this way it complements Eddy Current Testing (ECT) that can reliably measure film thicknesses of over 100 μm, but which is less sensitive for film thicknesses less than 10 μm.

**Gamma Scanning**

Axial gamma scanning of full-length CANDU fuel elements is performed routinely at both sites. Movement of the fuel element past a lead collimator is controlled automatically; the fuel element can either be moved continuously at different speeds or stepped incrementally. The element can also be rotated continuously or stepwise to allow any circumferential position to be scanned. The scanning, detection and measurement of the emitted gamma energies is controlled by a PC, which along with the amplifiers and multi-channel analyzer serves as a single integrated package for gamma spectrometry. Gross gamma energies and the distribution of individual isotopes can be measured to yield information on axial flux shape, end-flux peaking, pellet cracks and inter-pellet gaps.
Fission Gas Collection and Analysis

Element Puncture and Gas Volume Measurement. Fission gases released from the fuel during irradiation lead to an increase in internal element pressure. This is particularly important at extended burnups, since failures caused by stress corrosion cracking may occur at high internal gas pressure. High internal gas pressure could also lead to reduced thermal conductance across the fuel-to-sheath gap, resulting in higher fuel temperatures. The normal method for determining fission gas volume is to puncture the element, collect the gas in a standard volume and measure the pressure. A puncture tool and stainless steel gas collection apparatus, with processor controlled pneumatic valves (FCV1000) was designed and fabricated at CRL [4] for this purpose. The FCV1000 features automated gas collection and void volume measurement, capability for puncturing fuel elements with internal gas pressures up to 6.9 MPa, and an isolation system to detect water-logged fuel elements.

The FCV1000 puncture and gas collection system has significantly reduced the time required for puncturing, collecting gas and measuring void volume, which has resulted in much more effective use of valuable hot-cell time. It has an accuracy of 2.5% and a precision of ±1% for void volume measurement and an accuracy of 3% and a precision of ±2% for total gas volume measurement.

Fission-Gas Analysis. A substantial amount of gas (1 L) is typically collected during puncturing for mass spectrometer analysis to provide a quantitative measurement of each gas component and its isotopic composition. This is required to determine the actual volume of fission product gases released from the fuel, as distinct from any fill gases that may be present, such as He and Ar. Traces of H₂, O₂, and CO₂ could indicate the ingress of water through a pinhole defect in the sheathing. At CRL a VG 601 magnetic-sector mass spectrometer is used with a customized gas introduction system. The mass spectrometer response is calibrated by the analysis of prepared known mixtures. A range of mixtures is analyzed and the results are fitted to establish a relationship between maximum signal intensity and partial pressure of each component. Fission-gas analysis at WL is carried out on a VG MM8-80 mass spectrometer with fixed exit slits using the VG Process Soft and Spectrascan software. Calibration is carried out in two stages. First, a gas mixture containing Xe, Kr, CO₂ and H₂ is used to calibrate the mass scale. Second the relative sensitivities of the gas components are determined from known mixtures.

At CRL, the accuracy and precision is at the 2% (1σ) level for Xe, Kr and He and within 30% for the trace components, except O₂. At WL, the accuracy and precision are in the 2-10% (1 σ) range for Xe, Kr and He, and within 50% for the trace components, except Ar. The WL analysis is within requirements for the important components, He, Xe, Kr and CO₂. From the analysis of the gas composition, the total Xe and Kr released to the element's free volume is calculated. This is compared with the total inventory of fission gas produced during irradiation, calculated using a computer modeling code, such as ELESIM [5], to yield the fraction of gas released.
Microstructural Characterization

Optical Metallography and Ceramography. Optical light microscopy is extensively used to examine microstructural features in ceramic fuels and Zircaloy sheathing. Fuel channel components, such as pressure tubes and rolled joints, are examined for hydride distribution and flaws [6]. Both the CRL and the WL hot cells are well equipped for remote optical metallographic and ceramographic examination of reactor core materials. Sample preparation cells are equipped with high-speed cutting wheels and belt grinders for trimming and removal of large quantities of material. Final grinding and polishing is done with commercially available equipment that has been specially adapted for remote work. The light microscope laboratory at CRL comprises a suite of self-contained blister cells, constructed of 125-mm-thick lead walls, which accommodate a low-power microscope, two high-power microscopes, and a sample storage block. A Leitz MM5RT and a Reichert Telatom, modified for remote operation by their respective manufacturers, are used for bright field, dark field, and polarized light examinations. The Leitz is equipped for micro-hardness testing. Similar capability is available at WL, where a Leitz MM5RT is used for micro- and macro-optical examination. It is also equipped with a micro-hardness tester.

High-resolution alpha- and beta-gamma autoradiography techniques are also available for the qualitative examination of irradiated fuels. Alpha-autoradiography is useful for showing the distribution and homogeneity of plutonium-rich zones in MOX fuel.

Scanning Electron Microscopy (SEM) and X-ray Microanalysis. AECL's HCFs have the capability for topographical and compositional analysis of active samples in scanning electron microscopes. The instruments at both CRL and WL are housed in steel and lead enclosures, which provide a radiological barrier for the operator and the main console electronics. In both cases the instruments sit above the hot cells, and active samples are transferred to the microscopes by a small elevator, eliminating the need to remove highly radioactive samples from the hot cells and keeping personnel exposure to a minimum.

Both microscopes are equipped with energy dispersive and wavelength dispersive X-ray spectrometers (EDS and WDS) for elemental analysis, permitting the instrument to function as an electron microprobe (EPMA). The X-ray microanalytical capability is useful for studying fission-product distribution and migration in irradiated fuels, as well as fission products deposited on the inner sheath surface. The examination of fracture surfaces in the SEM (fractography) has proven invaluable for determining the root cause failure mechanism in fuel bundles and pressure tubes.

Transmission Electron Microscopy (TEM). Examination by Transmission Electron Microscopy can yield information on radiation damage and deformation at the atomic level in fuel, sheathing and other reactor structural components. Of special interest is the effect of precipitates (hydrides) on the formation and movement of dislocations and point defects in Zircaloy materials, and the precipitation of fission gases and solid fission products in fuel.
Zircaloy specimens are normally prepared for TEM by cutting slices of material about 0.25 mm thick in a hot cell using a slow-speed diamond saw. Further thinning to about 0.1 mm is performed by swabbing with a chemical polish. At this point the specimens can be removed from the hot cell, and final thinning is performed in a fume-hood using a Metalthin twin-jet polishing apparatus.

Preparation of irradiated ceramic fuel is somewhat more difficult. The method employed at CRL, originally developed at the Transuranium Institute, is to crush small samples into fine particulates and sandwich them between two carbon-coated, copper mesh screens. The edges of these particulates are usually thin enough for TEM examination.

**Micro-Sectioning**

Examining the distribution of fission products, and the effects of densification and swelling in a fuel cross section, is enhanced by having the capability for extracting small samples at precise locations. AECL's hot cells provide the required precision and remote control for cutting small samples from highly active material by using a computerized numerical control (CNC) milling cutter. The CNC machine tool is equipped with processor-controlled, three-dimensional motion that uses a diamond wheel to cut samples as small as 1 mm x 1 mm x 2 mm across the diameter of a fuel pellet. Measurement by various physical and chemical techniques on several of these samples can provide a radial profile of density, O/M ratio and retained fission products. An unsaturated polyester resin is used to impregnate and stabilize irradiated ceramic fuel during the cutting operation. The resin can withstand the radiation damage and is easily and completely dissolved from the individual samples.

**Burnup Analysis by HPLC**

The determination of burnup by high-performance liquid chromatography (HPLC) uses La-139 as a fission monitor. The need for isotopic analysis is avoided because La-139 is monoisotopic in fission. The fuels are dissolved and small aliquots of the fuel solution, quantitatively diluted with the HPLC mobile phase, are injected into a reversed phase column. Solution standards prepared to simulate fuel compositions are used to calibrate the HPLC signals. The number of fissions, and hence, the burnup are determined from the measured U and La concentrations and the appropriate fission yield, corrected for neutron capture in La-139. The effective fission yield is obtained from an irradiation simulation generated by one of the established reactor physics codes. The method has been successfully applied to a variety of fuels, including UO₂ and UAl [7].

**Hydrogen and Deuterium Analysis**

Excess hydrogen and deuterium in Zr-4 fuel bundle components and Zr-2.5 wt % Nb alloy pressure tubes precipitate as hydrides/deuterides and may lead to embrittlement problems.
Although it is rare, defects can occur in fuel sheathing from hydride accumulation, and hydrogen/deuterium pickup is regularly monitored as part of CANDU fuel surveillance. A high concentration of hydride platelets that have their inplane dimensions in the crack growth direction (radial hydrides) can lead to a reduction of fracture toughness in Zr-2.5 wt % Nb pressure tube material. Accurate measurement of the initial hydrogen concentration and the accumulated deuterium concentration during irradiation is required to monitor H/D levels and determine if these core components are fit for service.

**Hot Vacuum Extraction Mass Spectrometry.** AECL uses a method known as hot vacuum extraction mass spectrometry (HVEMS) to measure hydrogen concentrations in zirconium alloys (L.W. Green and G.A. Bickel, unpublished data). In this method, the sample is heated to a high temperature in vacuum to extract the gas that is transferred with the use of a vacuum pump to a mass spectrometer inlet. The HVEMS has high sensitivity and is designed to prevent fractionation effects between the hydrogen in the metal and gas phases. The mass spectrometer allows determination of both hydrogen and deuterium in the same sample. The HVEMS system can accommodate small samples of fuel sheathing or end-plate, and scrape and pellet samples from pressure tubes. The accuracy and precision meet CANDU customer requirements of ±5% (2σ) for concentrations above 10 μg/g, and ±0.5 μg/g for concentrations below 10 μg/g.

**Differential Scanning Calorimetry (DSC).** The excess hydrogen and deuterium concentration permissible in zirconium alloy components at reactor operating temperatures is defined by the terminal solid solubility (TSS) of hydrogen in zirconium. The enthalpy change associated with the dissolution or precipitation of the hydride/deuteride can be accurately detected with DSC. By using HVEMS to determine the corresponding total H/D concentration in the sample, a correlation can be established between H/D concentration and the TSS transition temperature. TSS transition temperatures have been measured by DSC for concentrations as low as 10 μg/g, and DSC is now routinely used on pressure tube scrape samples and other zirconium alloy core components to estimate H/D concentrations.

**Ion Beam Techniques**

Ion beam techniques provide a method for the chemical characterization of irradiated materials. The sample surface is bombarded with energetic particles that slowly erode it away by a process termed sputtering. In Secondary Ion Mass Spectrometry (SIMS), analysis of the sputtered material is performed with an energy filter and a mass spectrometer. For the examination of Zircaloy components, SIMS is particularly useful because it can detect hydrogen and its isotopes over a wide range of concentrations. Composition versus depth profiles are easily obtained as the material is sputtered away, providing information on corrosion and hydrogen pickup in fuel sheathing and pressure tubes. The SIMS is also a versatile tool for determining ppm levels of fission products and other elements in ceramic fuels. At AECL's HCFs, SIMS is complemented by other surface analysis techniques, such as Scanning Auger Microscopy (SAM) and X-ray Photoelectron Spectroscopy (XPS), to provide a broad range of quantitative analyses.
Measurement of Mechanical Properties

The function of the Mechanical Test Cells at both laboratories is to investigate the mechanisms of deformation and failure in CANDU reactor structural components. Consequently, they are well equipped with electro-mechanical and servo-hydraulic machines for the mechanical testing of irradiated materials. Furnaces and ovens are available for testing at elevated temperatures up to 400°C.

**Tensile, Fracture Toughness and Fatigue Testing.** Tensile and fracture toughness tests can be performed using standard ASTM procedures. Spark machining is used to fabricate specimens from sections of pressure tube. Zircaloy fuel sheathing is normally defuelled and tested using custom-designed tubing grips. The hot cells also have facilities for performing both S/N and fatigue crack growth rate tests.

**Delayed Hydride Cracking (DHC).** Considerable effort goes into the investigation of delayed hydride cracking of zirconium alloys, and the hot cells have extensive computer-controlled testing rigs for measuring threshold stresses for the initiation of cracks by hydrogen induced cracking mechanisms in irradiated material. Crack velocities are measured using acoustic emission techniques under computer control.

**Burst Testing.** There are a number of facilities for performing burst tests under either rising or static pressure on tubes up to 100 mm diameter. In the case of constant load tests the pressure and temperature can be programmed to follow reactor histories. Zircaloy sheathing is tested with the centre core of fuel drilled out to accommodate the hydraulic fluid.

**Residual Stress.** AECL has also developed techniques for the measurement of residual stresses using strain gauge techniques, specifically for rolled joints between zirconium alloy pressure tubes and stainless steel end fittings, but they could also be applied to other radioactive tubular components.

For all these testing techniques accurate machining of specimens is required, and in-cell machining techniques using lathes, milling and spark machines have been developed.

Measurement of Physical Properties

During irradiation the fuel and sheathing both undergo significant radiation damage that can alter their physical properties from the unirradiated state. In addition, fuel accumulates high levels of gaseous and solid fission products that can have an effect on properties such as gas diffusivity, thermal conductivity and O/M ratio. There is an increasing requirement for accurate values of physical properties to improve the predictive capabilities of fuel modeling codes for extended burnup and high-temperature transient scenarios. AECL’s hot-cell facilities are responding by developing techniques to measure physical properties on irradiated materials.
Microdensity Measurement. Dimensional changes can occur in the fuel as a result of densification and/or fission product swelling. These temperature-dependent, off-setting effects can vary across the pellet diameter, and this variation can only be measured on very small samples with a sensitive microbalance. A shielded mini-cell with a high precision electronic balance, accurate to ±0.125 μg, is used at CRL to measure the densities of samples cut from precise radial locations with the CNC saw.

Oxygen/Metal (O/M) Ratio. The O/M ratio, which signifies whether there is an excess of oxygen vacancies or interstitials, has an influence on diffusion rates in oxide fuels and can affect physical properties such as fission-gas diffusivity and thermal conductivity. A coulometric titration apparatus is available at CRL (P.G. Lucuta, R.A. Verrall and L.E. Bahen, unpublished data) to measure O/M ratios on small samples of irradiated fuel. The equipment is designed to control the oxygen potential of a stream of gas flowing over the heated sample, and measures the amount of oxygen the sample either absorbs or releases to that gas atmosphere. Deviations from stoichiometry, as low as 0.0003, have been measured (P.G. Lucuta, R.A. Verrall and L.E. Bahen, unpublished data).

Thermal Diffusivity. The technique of “flash diffusivity” is a practical means of measuring thermal diffusivity at high temperatures (>1000°) in irradiated materials. Thermal diffusivity is measured by flashing the front surface of the sample with a pulse of heat and monitoring the temperature rise on the back surface as a function of time. The thermal conductivity of the sample can be calculated from the thermal diffusivity, provided values of specific heat and density are known at the desired temperature. AECL is experimenting with the application of an accelerator to flash the front face of the sample with an electron beam. The sample can be heated to temperature with the beam prior to flashing the front face. This offers an advantage over more conventional methods (laser flash), as no separate heating furnace is required.

SUMMARY

The full range of AECL’s HCFs is utilized to provide PIE on fuel and fuel channel components, and to respond to urgent requests at CANDU nuclear stations. Post-irradiation examination has played a significant role in successfully resolving problems with fuel bundle assemblies and fuel channel components. Timely resolution has saved Canadian nuclear utilities millions of dollars in lost production.

As part of AECL’s goal to provide state-of-the-art PIE services, the HCFs are increasing the application of electron and ion beam techniques to characterize irradiated materials for advanced CANDU products. Commercial contract work with off-shore utilities and fuel vendors contributes to AECL’s revenue generation and provides additional stimulus for upgrading our technical expertise. There is a positive feedback from meeting the demands of external customers, and the implementation of quality assurance ensures that the facilities and services described in this paper are continually improved.
REFERENCES


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<td>13</td>
<td>5.6 x 3.0 x 6.2</td>
<td>0.7 (concrete)</td>
<td>1</td>
<td>250 x 200</td>
<td>receiving, general purpose</td>
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<td>14</td>
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<td>0.1 (lead)</td>
<td>1</td>
<td>25 x 35</td>
<td>waste management</td>
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<td>15 and 16</td>
<td>1.5 x 1.5 x 1.2</td>
<td>0.1 (lead)</td>
<td>2</td>
<td>25 x 35</td>
<td>waste management</td>
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<td>18</td>
<td>3.3 x 1.1 x 1.2</td>
<td>0.1 (lead)</td>
<td>1</td>
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