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Design Features of the Laboratory-Scale Radiochemical Immobilization System

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May 1980

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RADIOCHEMICAL IMMOBILIZATION SYSTEM

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INTRODUCTION

Under the High-Level Waste Immobilization Program, the Pacific Northwest Laboratory (PNL), operated by Battelle Memorial Institute for the Department of Energy, is studying various ways to solidify high-level nuclear wastes. A variety of waste forms and processes are being investigated, with the most highly developed process being spray calcination coupled with in-can melting. This report describes a laboratory-scale system that was designed for the purpose of investigating the effects of different operating conditions and waste compositions on the product and on the effluents generated. In the past, studies in the areas of effluent control and product evaluation have been conducted using engineering-scale and full-scale systems. However, engineering- and full-scale systems are inflexible and very expensive to operate.

To provide the necessary data in a cost-effective manner, a remote laboratory-scale radiochemical immobilization system was designed and built. It is termed "laboratory-scale" because of its nominal 1 L/h feed rate as compared to well over 300 L/h for "full-scale" equipment at PNL. The equipment currently consists of a feed system, a spray calciner, an in-can melter, and an effluent control system. It is operated in a shielded radiochemical hot cell using radioactive high-level liquid waste (HLLW) to answer questions on the deposition of radiochemicals during actual waste processing. The effluent control system can be modified in order to test different effluent systems, one of which has been proposed by the Savannah River Laboratories (SRL) for use in the Savannah River Plant vitrification system. The laboratory-scale system can also be used to test alternative immobilization processes, since spray calcination is a common processing step in many alternative waste form flowsheets. Thus, only the addition of a specific forming step such as pelletizing or sintering is necessary. Liquid and solidified waste handling and effluent treatment would require no changes.

This report presents the overall design of the remote laboratory-scale radiochemical immobilization system and briefly summarizes the functions and relationships of its components.

BASIC OBJECTIVES AND DESIGN CRITERIA

The objectives in designing the system were to:

- evaluate effluents as a function of calciner and melter operating conditions and waste compositions with actual radioactive wastes
- test and improve effluent decontamination systems and evaluate a system similar to the proposed SRL effluent control system
- produce high-level solidified waste forms for evaluation
- provide a waste solidification system that can be routinely operated at minimum cost with a minimum amount of radioactive materials consumed
- establish a system for radioactive testing of alternative waste forms.

These objectives led to the following basic design criteria:

- The equipment must operate at a very low feed rate.
- The system must be flexible.
- The system must easily accommodate frequent sampling.
- The equipment must be of relatively low cost.

Tests with HLLW performed in the engineering-scale system are limited to a small number of waste compositions and operating conditions. The laboratory-scale system is functionally similar to the engineering-scale system, but operates at less than one-tenth the feed rate. The system was designed on the basis of a 1 L/h feed rate to a spray calciner. This figure was selected as the smallest flow rate for which an air-atomized spray nozzle identical to those used in larger spray calciners was available. It was also thought to be the smallest feed rate for which pumps, flowmeters and other mechanical devices would be readily available.

The low feed rate provides the necessary effluent and product data from a much larger range of operating conditions and waste compositions. High-level waste procurement, a major cost in other programs, can be satisfied by using wastes remaining at the end of engineering-scale operations (tank heels, high-level condensates, etc.). Many different commercial and defense waste compositions can be processed by spiking a cold chemical blend with this material. In

addition, the low feed rate operations can be of extended duration, allowing for the investigation of long-term operating effects. Finally, the low feed rate will result in relatively low costs due to the low volume of secondary wastes generated and the fact that operations can be handled by a small staff.

Extensive operations with actual radioactive wastes in the laboratory-scale system will be used to predict the results of operating full-scale, radioactive equipment. Results of operations with simulated wastes in a laboratory-scale spray calciner compared favorably to data collected during full-scale operations. The similarity between the two sets of data indicates that scaling factors will not be a major consideration in the interpretation and usage of the results obtained from these laboratory-scale operations.

The laboratory-scale system was also designed to be flexible in order to test different operating modes and configurations. For example, the feed system allows denitration in any one of three areas: the calciner, the feed evaporator or the batch feed tank. The effluent control system allows for the "plugging in" of various types of decontamination equipment for testing. In particular, the SRL-proposed off-gas system can be tested. In the calciner, the filters can be changed easily, which allows for the testing of a variety of filter types and configurations.

To maximize the data obtained from operating the laboratory-scale system, samplers were installed on all of the liquid storage tanks. In addition, sample ports were installed downstream of every component in the effluent control system for obtaining gas samples. The liquid samplers can be operated at any time without creating upset conditions. However, to minimize system upsets only one gas sample can be taken at a time.

FLWSHEETS AND LAYOUTS

The general flowsheet for the laboratory-scale system is shown in Figure 1. Dilute HLLW is transferred into the hot cell and stored in a process tank (TK-D1). This dilute solution is pumped to the evaporator for concentration. The evaporator bottoms are drained to another process tank (TK-D2) to be stored as feed for the spray calciner (CA-D1). Evaporator overheads are condensed in a condenser (C-D1) and the condensate is stored in a third process tank (TK-D3). Calciner feed is pumped from TK-D2 through an air-atomized spray nozzle into the calciner where it is "dried" to a calcine. This calcine falls by gravity into a canister in the in-can melter where it is mixed with glass frit and melted to a homogeneous glass product.

An air-operated jet maintains the entire system under vacuum so effluents generated during waste processing are removed through the effluent treatment system before being vented to the building off-gas system. Primary effluent flow is from the calciner through three sintered-metal filters in the calciner filter cone to the venturi scrubber. The effluents then flow through the fission-product adsorbers (TK-D4 and TK-D5), the NO_x destructor (B-D1), the iodine adsorber (TK-D7), a high-efficiency particulate air (HEPA) filter and the packed scrubber (TK-D6). Scrub solution is recycled to TK-D6 after being cooled in Heat Exchanger No. 3. Two seal pots (TK-D8 and TK-D9) provide pressure release for the system. (The SRL off-gas system consists primarily of the addition of a cyclone after the venturi and the rearrangement of the order of the other components.)

Figure 2 shows the layout of the system inside the hot cell. The entire cell is 13 ft wide x 22 ft long x 12 ft high, and the remote, laboratory-scale immobilization system is located in approximately 1/2 of the cell. (Relative elevations can be determined from the flowsheets.) As can be seen from the layout, the system was constructed in three modules: the feed system, the spray calciner/in-can melter and the effluent control system. All of the modules are intertied with remotely connectable pipe jumpers as shown in Figure 3, which is a photograph of the assembled system before installation in the cell. Lines to and from pumps, flow indicators, valves and other mechanical items

FIGURE 2. Hot Cell Layout

FIGURE 3. Remote Laboratory-Scale Radiochemical Immobilization System

are connected to a "manipulator face." This face is a set of remotable pipe nozzles that are easily accessible by the manipulators. The mechanical items are connected here which allows easy access for repair or change. Other mechanical items that cannot be placed on the face, such as tank agitators, are accessible via the hot cell crane. Figures 4 and 5 show the feed and effluent control modules in greater detail. Figures 6, 7 and 8 show as-built pictures of the feed, spray calciner and effluent control modules.

FIGURE 4. Feed Module

FIGURE 5. Effluent Control Module

FIGURE 6. Assembled Feed System Module

FIGURE 7. Assembled Spray Calciner/In-Can Melter Module

FIGURE 8. Assembled Effluent Control Module

Connections between the system and building services are made with remotely connectable pipe jumpers between the system and block connectors on the west wall of the cell. These service connections include a supply of 100-lb air, a raw water inlet for cooling coils (noncontaminated services), a raw water outlet via the building's radioactive liquid waste sewer system (detects contamination and diverts contaminated water to storage instead of to the building sewer), a line to the building's high-level vaults, two lines from another cell where high-level waste is stored and can be jetted to TK-D1, and a connection to the building's off-gas system.

Effluents leaving the hot cell are processed through a condenser, a heater and a series of filters before being vented to the building stack. Effluent monitors are located in the stacks to detect alpha, beta and gamma releases. Operations will be shut off if effluents in the stack exceed the building limits. Liquid wastes are jetted to the building's high-level waste vault tanks. Solid wastes (manipulator boots, broken tools, irreparable equipment, etc.) are removed from the cell through an air lock for disposal via burial boxes with other solid wastes. Ruthenium and iodine are adsorbed in the effluent system onto solid beds. When these beds are loaded, they are removed and stored for later studies on incorporating these spent beds into solidification products as part of the immobilization process.

EQUIPMENT DESCRIPTIONS

The laboratory-scale system was designed to fit the dimensions of the hot cell, to allow for maximum flexibility of operation, and to have equipment that is remotely replaceable. The equipment was constructed as three modular units: the feed system, the spray calciner/in-can melter and the effluent control system.

FEED SYSTEM

The feed system provides a flow of HLLW to the calciner at a nominal rate of 1 L/h with a maximum rate of 3 L/h. High-level waste is first transferred into the cell and stored in a 150-L tank. It is then pumped to an evaporator where it is evaporated to a predetermined specific gravity. The concentrated HLLW is stored in a 20-L tank to be used as feed for the spray calciner. The vapors from the evaporator are processed through condenser CD-1. The condensate from CD-1 is stored in another 150-L tank. The entire feed system is operated under 10 to 20 in. of H_2O vacuum to ensure that any leakage is into the system. Any gases present in any of these vessels are processed through the effluent control system. The feed system module can be operated continuously or as a batch operation.

Process Tanks

Each of the process tanks is made from 304L stainless steel pipe and has stainless steel cooling coils. Each tank has connections for process input, process outlet, chemical addition, liquid sampling and gas outlet. Each is also connected to a transfer header, through which process fluids can be transferred between any of the tanks. An instrument system is provided for measuring the specific gravity, weight factor, pressure and temperature in each tank. Figure 9 shows Tank 1; Tanks 2 and 3 are of a similar design. Tanks 1 and 3 have 1/3-hp Mixmor, Inc. mixers to keep the fluids agitated and to keep solids in solution. Tank 2 is equipped with a Tekmar model SD-45N homogenizer. This unit emulsifies and homogenizes concentrated feeds with a wide range of viscosities. Since these concentrated solutions have a large quantity of solids present (up to 25 wt%), the homogenizer must keep the solids in suspension

and break down larger particles. This is essential to prevent plugging of the calciner feed lines and the calciner nozzle.

Evaporator

The evaporator (see Figure 10) was designed for boiling dilute waste at a nominal rate of 8 L/h with a maximum rate of 16 L/h. This unit is made from 6-in. 304L stainless steel pipe and has a volume of 20 L. Waste is boiled to a predetermined specific gravity by steam circulating through 4.8 ft² of coil

FIGURE 10. Feed Evaporator

in the evaporator. The evaporator is insulated the length of the steam coils. When the waste has been concentrated to a set value, steam flow is shut off while an air purge is started to the coils and the valve is opened to drain the concentrated waste to Tank 2. Instruments record the specific gravity, weight factor, temperature and pressure in the evaporator. Flow to the evaporator (and from Tank 2 to the calciner) is measured by Ramapo Mark X[®] flowmeters. These flowmeters are miniature, variable-area flow sensors. The evaporator flowmeter has a range of 10 to 500 cm³/min. The calciner flowmeter has a range of 3 to 100 cm³/min and has an inverted modification to allow for the solids present in the concentrated waste. Flow to the evaporator and to the calciner is controlled by adjusting the rotational speed of the pumps on Tanks 1 and 2.

Condenser

The condenser CD-1 (see Figure 11) is a shell and tube heat exchanger. The process fluid flows through the tubes, and cooling water flows through the shell. It is made from 6-in. stainless steel pipe and 1/2-in. stainless steel tubes. There are 8.5 ft² of heat transfer surface area and the unit has an estimated shellsize capacity of 15 L. It has an overall heat transfer coefficient of 50 BTU/h ft²°F to condense 20 L of water/h.

Formic Acid Feed System

Provisions have been made for denitrating nitric acid waste solutions with formic acid. Denitration of nuclear wastes reduces the nitric concentration to 0.5M and reduces the volatility of ruthenium during processing of the waste. Different methods for adding the formic acid can be tested. For example, the formic acid can be added to Tank 2 for batch denitration of concentrated waste. It can also be added directly to the evaporator or to the calciner. The rate of formic acid flow to these units is a function of the flow rate of the waste solution and is controlled by adjusting the rotational speed of the pump. Because the nitric acid/formic acid mixture is very reactive, the quantity of formic acid and the rate at which it is added must be carefully controlled. A

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(a) Tube Bundle

(b) Top View

(c) Front View

FIGURE 11. Condenser

flow alarm on the formic acid feed system shuts off the pump if the rate at which formic acid is added exceeds a set value. Batch denitration is also temperature dependent so the pump automatically shuts off if the temperature varies from an acceptable range. A premeasured amount of formic acid from the chemical storage tank outside the cell is added to a small, closed stainless steel tank inside the hot cell. The amount of acid stored inside the cell is only that needed to react with the waste. The tank in which it is stored is vented to prevent an accumulation of fumes.

SPRAY CALCINER/IN-CAN MELTER

The spray calciner and in-can melter are the components in the solidification system that convert the liquid HLLW to a vitreous product. The spray calciner and in-can melter are operated in a batch mode to allow product removal. The in-can melter is easily replaceable for testing alternative waste form processes.

Spray Calciner

The spray calciner is shown in Figure 12. It calcines liquid waste at a nominal rate of 1 L/h or a maximum rate of 3 L/h, at a nominal temperature of 800°C. The calciner spray chamber is a 5-in.-ID cylinder made of Inconel® 601. The chamber is surrounded with five zones of half-section, insulated heaters. The furnace assembly is surrounded by a stainless steel shell. Ten thermocouples are welded to the exterior wall of the spray chamber. One thermocouple in each of the five calciner zones provides a signal which controls power input to the heaters in that zone. The other five thermocouples (one in each zone) record the temperature history.

The waste is sprayed into the calciner through an air-atomized nozzle. The nozzle is an internal-mix unit with a spray orifice of 0.02 in. The nozzle has an air-operated cleanout needle mounted on top of it to remove any solids plugging the orifice. A manipulator rack is located on the side of the

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(a) Top View

(b) Front View

FIGURE 12. Spray Calciner

calciner with lines for the feed, feed recycle, nozzle air and formic acid. The Ramapo flowmeter and a cartridge filter are on the feed line. An air-operated vibrator is mounted midway down the side of the calciner. It is operated at intervals to remove calcine adhering to the furnace walls.

Glass frit is added to the calciner via the frit addition system. Pulses of air open the jet on the bottom of the frit feed hopper, and a batch of frit falls into the calciner cone. The duration and/or frequency of these pulses depends on the amount of frit to be added. The glass frit and the calcine fall through the stainless steel calciner cone into the melter. The system is operated under 10 to 20 in. of H_2O vacuum. Any gases generated by calcining are removed through the calciner filter chamber into the effluent control system. The filter chamber contains three stainless steel sintered-metal filters that trap dust particles entrained in the gases. These filters are automatically pulsed with air to remove the dust particles whenever the pressure drop across these filters exceeds a set value. A seal pot filled with liquid and connected to the filter chamber would, if necessary, relieve excess pressure from within the calciner. The calciner, filter chamber and cone are mounted on a stainless steel support frame, which permits easy movement of the in-can melter under the calciner cone.

In-Can Melter

The in-can melter is a Thermcraft, Inc. tube furnace with a 7-in.-ID, 24-in.-long heated tube. The furnace has two heating zones, each controlled by feedback from a thermocouple. Four additional thermocouples (two per zone) record the temperature history. The furnace has a temperature range of $0^{\circ}C$ to $1200^{\circ}C$ with a maximum total wattage of 10.9 kW. The melter rests on a cart, which can be rolled back and forth under the calciner.

When the canister is placed inside the melter, the melter is positioned under the cone and the canister is attached securely to the bottom of the cone. The canister is secured to the calciner via the canister lifting mechanism. The canister neck rests on the canister support flange. Two air cylinders raise the canister to the cone and hold it in place during operations. A section of 6-in. Inconel pipe modified with cross bars across the pipe diameter

are inside the tube furnace to serve as a support for the canister; thus, the canister neck always extends above the furnace and the manipulators can grasp it. The calcine/frit mixture falls into the canister and is melted to a molten glass product. When the canister has cooled, it is detached from the calciner by turning the air supply to the cylinders off. The cart is then rolled away from the calciner and the canister is removed from the melter.

Canister

The canister (see Figure 13), made of Inconel or stainless steel, fits securely against the bottom of the calciner cone. It takes approximately 8 to 10 h of operation to fill one canister. A larger canister (5 in. in ID x 18 in. long) can also be used. An Inconel "C" or "E" seal gasket is used to seal the canister to the cone. Other canister sizes can be adapted for use with the laboratory-scale system by providing them with a flange that mates with the calciner cone.

FIGURE 13. Canister

EFFLUENT CONTROL SYSTEM

One of the objectives in designing the laboratory-scale system was to be able to sample and evaluate effluents and to test methods for decontaminating them. Effluents are treated in a system consisting of a calciner filter chamber, a venturi scrubber, volatile fission product adsorbers, a nitrogen oxide reducer, an iodine absorber, a HEPA filter and a packed scrubber. In addition, a mockup of a proposed SRL flowsheet off-gas system will be tested. In this mockup, mercury control methods can be tested.

Venturi Scrubber

The venturi scrubber (see Figure 14) cools and partially condenses the off gases from the calciner. Entrained particulates are scrubbed from the gas stream, and water vapors are condensed. The venturi scrubber cools 130 g-moles of noncondensable and 70 g-moles of condensable gases per hour from 350°C to 100°C using 3 gph of scrub solution. The flow rate of scrub solution is measured by a flowmeter and controlled by adjusting the rotational speed of the pump. The gas/scrub solution is separated in a condensate tank (CD-2; see Figure 15). The scrub solution is then cooled and stored in CD-2 to be recycled to the scrubber. The venturi scrubber is made of 304L stainless steel with a 0.275-in.-ID throat. Tank CD-2 is made from 10-in. stainless steel pipe, has cooling coils with a surface area of 6.2 ft² and has a capacity of 20 L. Instruments record the weight factor and specific gravity of the scrub solution and the temperature of the gas leaving CD-2.

Volatile Fission-Product Adsorbers

Two volatile fission-product adsorbers remove essentially all of the volatilized ruthenium by passing the gas through beds of iron oxide. The two adsorbers can be operated in series or in parallel. An 1850-W, in-line heater preheats the gas to 600°C. The adsorbers are made from 6-in. stainless steel pipe. They each have a 6-in. bed depth of iron oxide and operate with a gas velocity of 0.4 ft/s and a bed loading of 136 g/ft³. The adsorbers are heated by two 1070-W heaters to maintain bed temperatures of 200°C. A thermocouple in each of the adsorber beds provides a signal that controls the

FIGURE 14. Venturi Scrubber

power to the heaters. The pressure drop across the beds is recorded and the beds are changed when the drop exceeds a set value or when a different bed material is to be tested. The beds can be changed remotely. The adsorbers are shown in Figure 16.

(a) Top View

(b) Front View

FIGURE 15. Venturi Scrubber Condensate Tank

FISSION PRODUCT ADSORBER

FIGURE 16. Volatile Fission Product Adsorbers

Nitrogen Oxide Reducer

Nitrogen oxides are destroyed by a reaction with ammonia gas at 400°C to 500°C over a bed of synthetic hydrogen mordenite pellets. At this temperature and with a certain ratio of NH_3 to NO_x , the nitrogen oxides are reduced to nitrogen and water. For this process, the gas is preheated to 500°C in an in-line heater before it enters the ammonia burner (see Figure 17). The gas then mixes with the ammonia at the top of the bed. The reaction is completed as the gas stream passes through the bed. The bed is 8 to 10 in. deep, the gas

(a) Top View

NITROGEN OXIDE REDUCER

(b) Front View

FIGURE 17. Nitrogen Oxide Reducer

velocity is 1.65 ft/s, and the ratio of NH_3 to NO_x is 1.3 to 1. The ammonia burner is heated by two 825-W heaters to maintain the required temperature of 400°C to 500°C . Power to these heaters is controlled by a signal from a thermocouple located in the middle of the bed. The temperature at the top of the bed is recorded. If the temperature is not within the required range, an alarm is tripped and the flow of ammonia is shut off. The pressure drop across the burner is also recorded.

Iodine Adsorber

Iodine is removed by passing the gas stream through a silver zeolite bed. The iodine adsorber (TK-D7; see Figure 18) has a bed depth of 2 in., operates

FIGURE 18. Iodine Adsorber

with a gas velocity of 0.67 ft/s at 150°C, and has a silver loading of 85 mg/cm³. The bed temperature and pressure drop across the bed are recorded. After the adsorber, the gas is heated to 150°C to vaporize any water before passing through a HEPA filter.

Packed Scrubber

The final decontamination step in the effluent control system is scrubbing the gas in a packed column. The packed scrubber (TK-D6) is a 4-ft-high bed of 1/2-in. stainless steel raschig rings. It scrubs 4 ft/min of gas at 150°C with 1.65 gpm of scrubber solution. Entrained solution is removed by a demister pad at the top of the column. The scrub solution is pumped through a heat exchanger (CD-3; see Figure 19), where it is cooled before it is recycled to

FIGURE 19. Packed Scrubber Heat Exchanger

the scrubber. The scrubber (see Figure 20) is made from 6-in. stainless steel pipe. Instruments record the bed temperature, the weight factor of the scrub solution and the pressure drop across the packed column.

FIGURE 20. Packed Scrubber

Savannah River Laboratory Off-Gas System

The SRL off-gas system involves passing the off gases through a venturi scrubber, a cyclone, a mercury scrubber, volatile fission-product adsorbers, iodine adsorbers and a filter. To simulate this system, the laboratory-scale off-gas system can be piped to 1) include a cyclone, 2) use the packed scrubber as a mercury scrubber, and 3) by-pass the NO_x reducer. The process fluids from the venturi, cyclone and mercury scrubber can be stored and recycled from CD-2. The cyclone (see Figure 21) was designed for a gas flow rate of $6.75 \text{ ft}^3/\text{min}$ at 350°C . The dimensions of the cyclone are those recommended by the

FIGURE 21. Cyclone

Stairman design for a high-efficiency cyclone. With the recommended dimensions, the cyclone has an 88% efficiency for removing particulates from the gas stream. When the gas leaves the cyclone it flows to the packed scrubber where mercury is removed, and then passes through the volatile fission product adsorbers. For the SRL off-gas system, the adsorbers can be operated in series with a bed of 6 to 12 mesh silica gel particles. The gas flow rate is 0.4 ft/s and the operating temperature is 80°C to 90°C. The ruthenium is removed at 4.8×10^{-3} g Ru/cm³ of silica gel bed. The gas then flows directly to the iodine adsorber and the HEPA filter.

SAMPLING SYSTEM

Liquid and gas samples will be a major source of data. Liquid samples are taken from each of the tanks and from the venturi and packed scrubbers. Gas samples are taken after the gas passes through each stage in the effluent control system.

Liquid Samples

Each of the tanks has a liquid sampler (see Figure 22). Solution is jetted from the bottom of the tank, through a sample collection bottle and back to the tank. When the bottle has been filled with a representative sample, the jet is shut off and the bottle is disconnected and capped. The venturi and packed scrubbers have sample ports on the discharge side of their pumps for pulling a sample by opening the valve on the port.

Gas Samples

The method of gas sampling is shown in Figure 23. The gas is drawn by an air jet through a polypropylene filter which removes submicrometer dissolute particulates from the gas. The gas then bubbles through a scrubber to trap volatile metals. The gas flow rate through the sampler is measured by the Ramapo flowmeter and is controlled by an automatic control valve.

FIGURE 22. Liquid Sampler

(a) Front View

(b) Side View

FIGURE 23. Gas Sampling System

INSTRUMENTATION AND CONTROL

Since this is a fully remote, research-oriented system, special consideration was given to the instruments used. The majority of the operational data are remotely recorded on strip chart and multipoint recorders. The signals that are recorded come from a variety of sources, including thermocouples for temperatures and differential pressure cells for pressures. An automatic process controller will be used to program the intervals between and duration of air to the sintered-metal filters, the vibrator and the frit addition jet. The system is set up and the order of processing is selected with manual ball valves operated by master-slave manipulators.

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