This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.

Glass Apparatus Development Lab

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Background

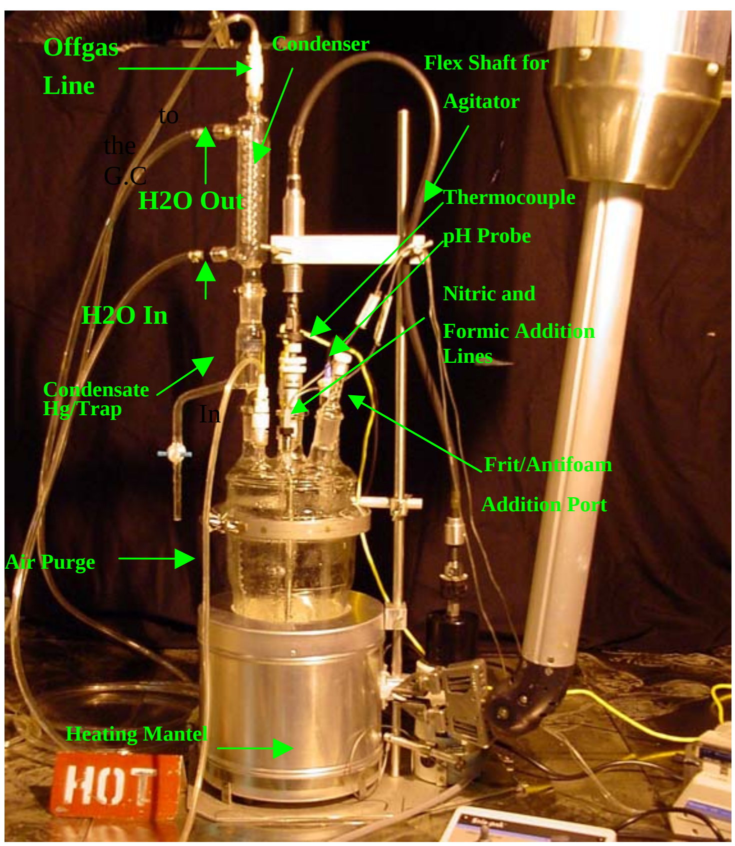
The Defense Waste Processing Facility (DWPF), at the Savannah River Site (SRS), is processing and immobilizing the radioactive high level waste sludge slurry at SRS into a durable borosilicate glass for final geological disposal. Each time a new batch of radioactive sludge is to be processed by the DWPF, the process flow sheet is to be tested and demonstrated to ensure an acceptable melter feed and glass can be made. These demonstrations are completed in the Shielded Cells Facility in the Savannah River National Laboratory at SRS.

System Description

The latest SRAT/SME vessel design used for the DWPF melter feed qualification testing in the SRNL Shielded Cells Facility, consist of a glass reaction vessel approximately 13 inches (33cm) in height and 6 inches (15 cm) wide. The SRAT/SME vessel has a capacity of approximately 2 liters, and the top of the SRAT/SME vessel has a series of ports and openings. These ports and openings are for the installation of equipment (i.e. pH probe, thermocouple, agitator, etc.) and process lines (acid addition, air purge, etc.). The condenser, mercury/condensate trap, and cold trap connected to the SRAT/SME vessel are also made out of glass.

To supply heat to the SRAT/SME vessel, a heating mantle is used. Also, a laboratory chiller unit is used to supply the chilled water for the condenser. Figure 1 is a picture taken in the Mockup Cells of the system prior to installation in the Shielded Cells.

Picture of the SRAT/SME Vessel in the Mockup Cells of the Shielded Cells Facility (Figure 1)



Information on the DWPF SRAT and SME CYCLES

Producing an acceptable melter feed from radioactive sludge slurry is demonstrated by performing two separate DWPF processing cycles. The first cycle is the Sludge Receipt and Adjustment Tank (SRAT) cycle and the second cycle is the Slurry Mix Evaporator (SME) cycle. Approximately 1 L of radioactive sludge is used to demonstrate both the SRAT and SME cycles.

The first step of the SRAT cycle is to heat the sludge to 93°C. After reaching this temperature, nitric and formic acids are added to the sludge slurry to react with carbonates and hydroxides in the sludge to adjust its rheology and to reduce Hg ions in the sludge. At the completion of the acid additions, the sludge is heated to boiling to steam strip mercury and remove water by obtaining the proper weight percent solids. During this cycle, the pH is monitored and a 5 % He/air mixture continuously purged the SRAT/SME vessel to measure offgas composition. The offgas exiting the vessel is monitored by a gas chromatograph for H_2, He, CO_2, and N_2O. The product of this cycle is then analyzed for chemical composition, weight percent solids and density as a precursor for starting the SME cycle.

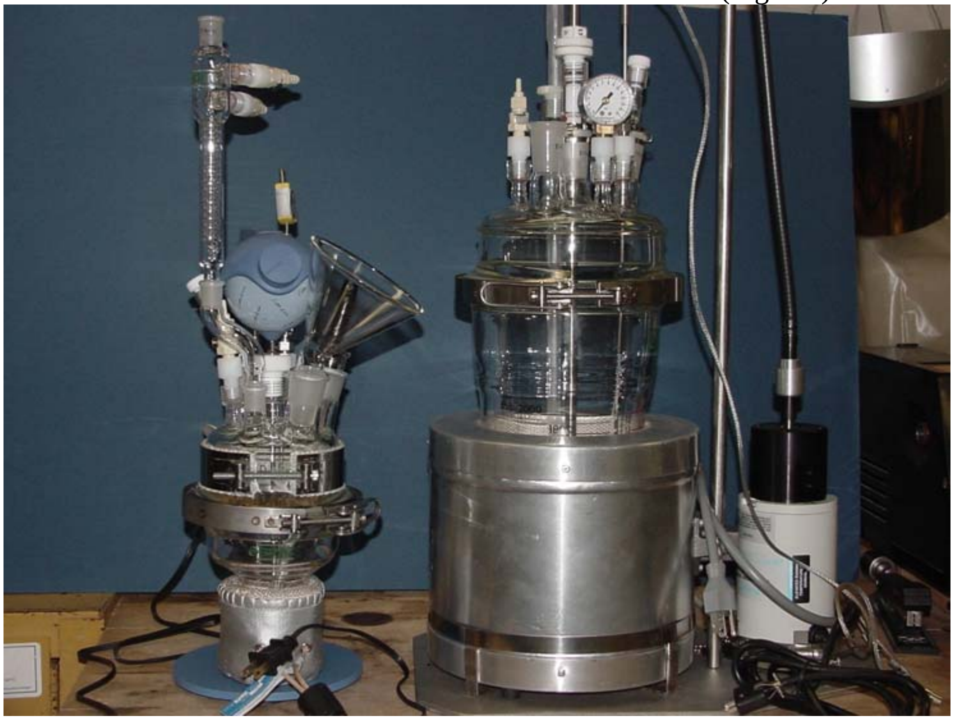
Prior to the start of the SME cycle, the chemical compositions of acidified sludge and glass forming frit are entered into the DWPF process control program. This program contains algorithms that predicted certain properties that the melt and final product must meet in order to make a durable glass that can be processed through a melter. The program prescribes the proper amount of frit to be added and predicts the composition of the final product. After the calculations demonstrate that the product meets the acceptable properties, the acidified sludge is heated to 90°C to start the SME cycle. Upon reaching 90°C, the prescribed amount of glass forming frit is added as a dry powder to the acidified sludge. Dilute formic acid water additions followed the frit addition to simulate the frit slurry addition method in the DWPF. After the additions are complete, the acidified sludge/frit mixture is heated to 100°C to remove water to obtain the correct weight percent solids. The SRAT/SME vessel is also continuously purged during this cycle by a 5 % He/air mixture. The offgas exiting the vessel is monitored by the gas chromatograph for H_2, He, CO_2, and N_2O.

Need for Mini-SRAT Reaction Vessel for Rheology Studies

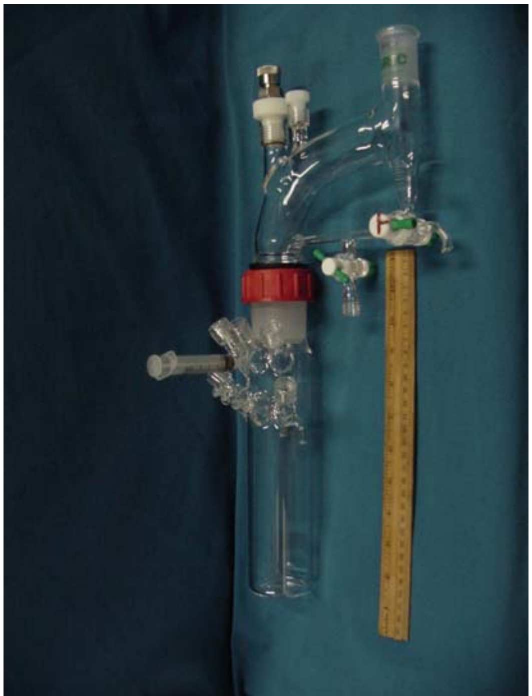
The Glass Apparatus Development Lab (GADL) was asked to design and manufacture four each Mini-SRAT vessels to be used for rheolgy studies supporting research at the Savannah River National Laboratory (SRNL). One of the problems associated with the vessel's design was the small volume of sample that was offered for testing. The customer requested the vessel to hold approximately 250 to 300ml, with a workable mixing volume of 150ml and measuring no taller than 3.5 to 4 inches. This was considerable smaller than the original SRAT which measured 13 inches tall and approximately 6 inches wide with a capacity of 2-liters. The slurry was to be heated using a controlled heating source and all instrumentation that was used on the larger SRAT vessel were to be incorporated into the new design.

The bottom section of the Mini-SRAT was made using glass tubing measuring 57mm in diameter. This would allow the use of a standard heating mantle, normally used for heating beakers with the same diameter. A standard minitrol was used to control the temperature of the vessel by adjusting the amount of power that supplied to the unit. A 100mm Duran flat ground, o-ring grooved flange was used to mate with the top section, similar in every detail except for its physical size. By designing the vessel to use a standard heating mantle, time and money was not wasted on design and special ordering a mantle for the unique vessel. The vessel top was made using the same orientation as with the larger vessel, with a few modifications. The alignment and placement of the connectors were even more critical, due to the limited space on the vessel top. Special off-set adapters were made to accommodate the installation of equipment as before (i.e. condenser, pH probe, stirrer, thermocouple, pressure relief valve, process lines, acid addition, air purge, etc.). Figure 2

Picture of the SRAT/SME Vessel and Modified Mini-SRAT (Figure2)

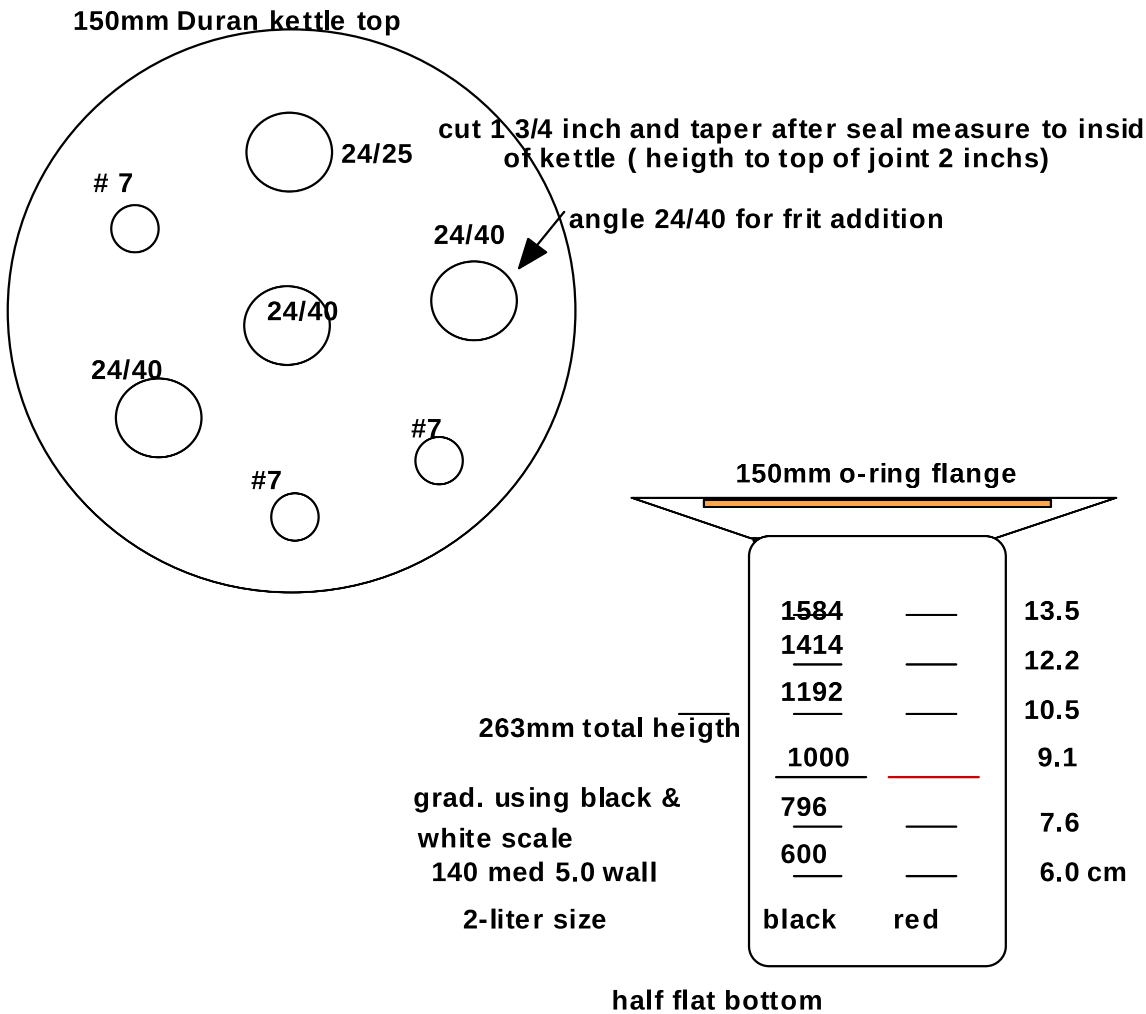


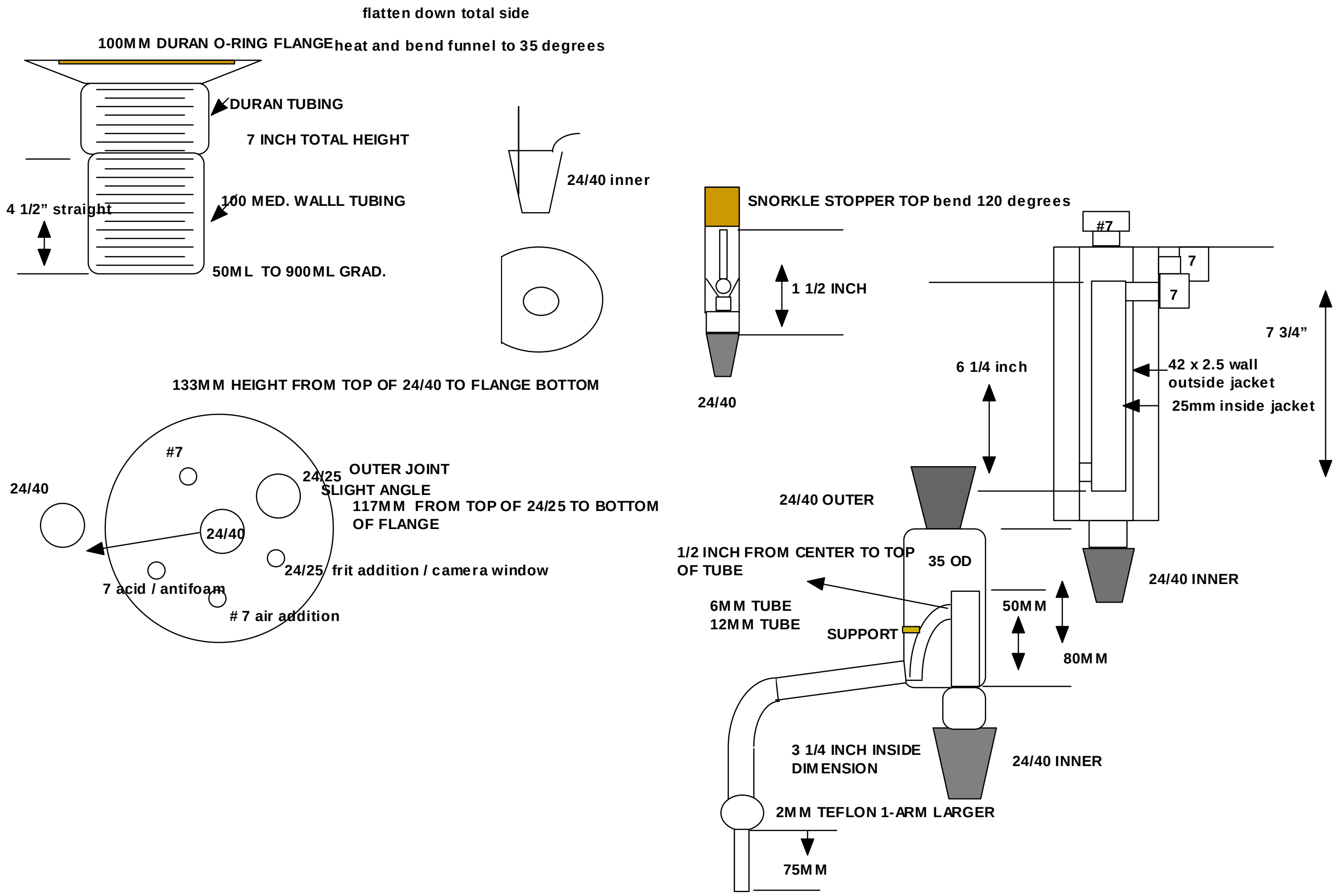
The design of the Mini-SRAT by the GDL and testing preformed by the SRNL Shielded Cells had a positive impact on the feed qualifications and scheduling of the immobilizing of high level waste sludge slurry. The quick design of the vessels enabled fast turn-around of results needed to satisfy concerns at the Defense Waste Processing Facility. These studies were important to maintain the current schedule which is directly associated hard dollars and bonus awards for filling of canisters that met or exceeds DOE’S expectations.




Lab-Scale
SRAT / SME

Description Or Sketch





flatten down total side
100MM DURAN O-RING FLANGE
heat and bend funnel to 35 degrees
DURAN TUBING
7 INCH TOTAL HEIGHT
4 1/2" straight
100 MED. WALLL TUBING
50ML TO 900ML GRAD.
24/40 inner
SNORKLE STOPPER TOP bend 120 degrees
#7
7
7
1 1/2 INCH
7 3/4"
6 1/4 inch
42 x 2.5 wall
outside jacket
25mm inside jacket
24/40
133MM HEIGHT FROM TOP OF 24/40 TO FLANGE BOTTOM
#7
24/25
OUTER JOINT
SLIGHT ANGLE
117MM FROM TOP OF 24/25 TO BOTTOM
OF FLANGE
24/40
24/40
24/25 frit addition / camera window
7 acid / antifoam
7 air addition
24/40 OUTER
1/2 INCH FROM CENTER TO TOP
OF TUBE
35 OD
24/40 INNER
6MM TUBE
12MM TUBE
SUPPORT
50MM
80MM
3 1/4 INCH INSIDE
DIMENSION
24/40 INNER
2MM TEFLON 1-ARM LARGER
75MM