SYNROC PROCESS FOR URANIUM RICH WASTE FROM ⁹⁹MO PRODUCTION

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ABSTRACT

The Australian Nuclear Science and Technology Organisation (ANSTO) irradiates Uranium (U) targets to produce ⁹⁹Mo, the precursor to radiopharmaceutical ^{99m}Tc. A process to convert the U-rich dissolver waste, from the U-targets, into a durable synroc type ceramic has been demonstrated at laboratory scale by ANSTO and the Idaho National Engineering and Environmental Laboratory (INEEL). Synroc is a particular kind of "synthetic rock," developed in 1978 by the late Professor Ted Ringwood of the Australian National University. It is an advanced ceramic comprising geochemical stable natural titanate minerals, which have immobilized uranium and thorium for millions of years. These mineral structures can incorporate and immobilize nearly all of the elements present in high-level radioactive waste (HLW). A joint effort between ANSTO and the INEEL was conducted to evaluate 1) a fluidized-bed to calcine the U-bearing liquid waste surrogate solutions mixed with a synroc precursor and 2) conversion of the calcine into synroc via hot isostatic pressing (HIPing).

The feasibility study was completed using a lanthanide-rich non-radioactive surrogate of the U-rich waste (a nitrate based solution), combined with a titanate-rich precursor suspension. Having prepared 20-L batches of feed solutions for the calcination trials, they were calcined in a H_2/N_2 reducing atmosphere at 575-620°C with an electrically heated 82 mm diameter fluidized-bed reactor. The feed was spray dried and calcined on a starting bed of 200-400 μ m Synroc B precursor particles. A series of calcination tests were performed, achieving >90% bed turnover to the desired synroc precursor.

Non-volatile (Sr, Ba, Ca, K, Ce, La, Nd, Pr, Al, and Ni) elements in the surrogate HLW feed were effectively converted to an oxide calcine product, with minimal generation of fines in the bed. Bed particulate growth was well behaved with the production of homogeneous spherical particles suitable for bed fluidization and feed spray coating. Fines generation was reduced to approximately 10% of the oxide products produced by the liquid feed. The fines were efficiently collected by a cyclone separator and sintered metal blowback filter (>99% solids disengagement). The fines and bed were collected for physical and chemical analysis. Residual nitrate levels in the calcined bed were less than 0.5 wt%, which is a prerequisite for HIPing. The partitioning efficiency of potentially volatile radioactive Ru and Cs in the calcine product was greater than 99.98%.

The synroc precursor calcine was mixed with 2 wt% of powdered titanium metal in a steel can. It is then evacuated, sealed, and hot isostatic pressed (HIPed) at 1250°C. The HIPing process produced a chemically durable, homogeneous (at the 50 micron scale) ceramic having nearly 100% theoretical density, and a waste loading of approximately 35 wt% was achieved. The physical and chemical characteristics of the HIPed sample were evaluated. Scanning Electron Microscopy (SEM) reveal a homogeneous crystalline synroc was produced. Waste form durability is predictably superior to those of alternative vitrified waste forms.

INTRODUCTION

The Australian Nuclear Science and Technology Organisation (ANSTO) irradiates Uranium (U) targets to produce ⁹⁹Mo, the precursor to radiopharmaceutical ^{99m}Tc. ANSTO produces ⁹⁹Mo as a commercially available radioisotope. Uranium waste and fission products are produced after the dissolved uranium targets are separated from ⁹⁹Mo. ANSTO has two viable waste options for disposal: 1) reclaim U or 2) immobilize U in synroc. The latter option is presently under consideration and is the focus of this paper. A joint-effort between ANSTO and the INEEL was conducted to evaluate: 1) a fluidized bed to calcine a surrogate U bearing liquid solution into a synroc precursor, 2) conversion of the calcine into synroc via hot isostatic pressing (HIPing).

Synroc is a geochemical stable titanate-based ceramic developed for immobilization of high level waste (HLW). The concept of Synroc ("<u>Syn</u>thetic <u>roc</u>k") was initially proposed by the late Professor Ted Ringwood of the Australian National University after studying naturally occurring titanate minerals which have immobilized uranium and thorium for millions of years. These mineral structures can also incorporate and immobilize nearly all of the elements present in high-level radioactive waste (HLW). Over the past 25 years, ANSTO has developed synroc chemistries and process technologies to produce a family of synroc waste forms that can be tailored to specific radioactive waste compositions. ANSTO has oriented its synroc research to military HLW wastes, and in particular to the clean-up problems faced by US Department of Energy (DOE) at their Savannah River Site (SRS), INEEL, and Hanford sites. For example, Synroc D was developed to immobilize the Al-Fe hydroxide sludges at the SRS.

Feed Makeup

A colloidal suspension of titanate additives and surrogate wastes was developed by ANSTO for demonstrating the immobilization of the dissolved U targets in ANSTO's ⁹⁹Mo target waste solution. The Synroc G formulation consisted of Ti oxides, Ca nitrates, Al nitrates, Ni nitrates, and Ba hydroxides (the additives) mixed with a lanthanide-bearing radioactive surrogate of Ce, La, Nd, Pr, Sr, Ru, and Cs nitrates. Elements from the lanthanoid series (Ce, La, Nd, and Pr) were used as surrogates for the uranium and other actinides present in the ⁹⁹Mo target waste solution. The surrogate HLW feed was formulated to test the following molecular characteristics: 1) immobilizing Ce nitrates (surrogate for U nitrates) and toxic metals (Ni and Ba), and 2) volatility of Cs and Ru. The Synroc G formulation, including lanthanoid fission and toxic compounds, was dissolved in nitric acid and collectively used as feed for the fluidized bed demonstration.

The surrogate HLW feed mixture was a mildly acidic, colloidal, solution containing small micro-sized dissolved anatase particles of titanium. The surrogate HLW feed solution was made up as 20L batches. The surrogate HLW feed simulated a 25% (by weight) solids loading, producing a theoretical yield of 250 grams solid oxide product per liter of surrogate HLW feed solution calcined. The average product generation rate was typically around 280 grams oxide per liter of surrogate HLW feed solution calcined. This was attributed to incomplete calcination/denitration of the surrogate HLW feed, but may also indicate higher oxidation states of the solid products, or possibly (but unlikely) hydration of the fines in the cyclone and sintered metal filter.

ANSTO Research and Development

It may be interesting to note that this test activity is not the first trial for producing a synroc precursor in a fluidized-bed reactor. In 1983, testing at moderately high operating temperatures (500-600°C), with systematic variation of feed rates, fluidizing gas rates, and redox conditions, was completed to demonstrate synroc production of uranium-rich wastes (Acherman *et. al.*, 1983). ANSTO has also

recently completed a series of fluidized-bed calcination tests using sol-gel feeds and various methods of heat deposition and feed encapsulation by micro spheres (Sizgek *et. al.*, 2002, 1998).

The process of consolidating calcined synroc waste forms has been an area intensely studied by ANSTO over many years and various options tested include: Sintering, Melting, Hot Uniaxial Pressing and HIPing. These have been tested both at laboratory and production scale, including having built a Synroc Demonstration Plant (SDP) and associated facilities to demonstrate the processes. All are feasible processes for the production of ceramic and glass–ceramic radioactive waste forms, offering relative advantages and disadvantages depending on the type of waste and the volume to be processed. The most appropriate technology for the waste considered here is HIPing, which has also been shown to be viable method by other laboratories such as Argonne National Laboratories-West (ANL-W), Idaho.

HIP technology was invented in 1950's at Battelle Memorial Institute as means for diffusion bonding nuclear fuel and cladding material. The basic technology consists of a pressure vessel containing an electrically heated furnace. Components to be processed are placed in the furnace and a pressure is applied via the compression of an inert gas such as argon. The gas pressure is sufficient so that it behaves essentially as a fluid and an isostatic pressure is exerted on the component. HIPing is an efficient consolidation method and has the added benefit for radioactive waste forms, in that the waste form constituents are sealed in a stainless steel canister. Therefore having the advantage that there is zero radioactive emissions from process.

INEEL Research and Development

The INEEL has built and operated fluidized bed reactors to solidify dissolved HLW solutions for over 50 years. Various scales of fluidized-bed pilot plants and test beds were set up to investigate uranium nitrate and waste solution solidification chemistry, particle mechanics, and off gas emissions control. A small fluidized-bed denitrator/calciner facility was built in the mid 1970's, and operated up until the early 1990's, for evaporating and denitrating uranyl nitrate solutions derived from fuel reprocessing activities at the INEEL. The uranyl denitrator was comprised of a critically-safe, 130-mm internal diameter, fluidized-bed vessel that was capable of processing uranyl solutions having a nominal concentration of 350 g/L U at a rate of 4-6 L/hr.

Fluidized bed processing accomplishes slurry-drying, calcination, and oxidation-reduction reactions between waste salts and ceramic formers in a single processing unit. Fluidized-bed processes have several advantages over typical spray drying, wiped-film, rotary-kiln, and fixed or moving bed thermal treatment processes. To prevent harmful exposure to radiation, high-level radioactive waste processes typically require a closed system that can be remotely operated. The process must be robust, remotely maintainable, and efficient. A fluidized-bed reactor is conducive to calcining radioactive waste, especially when: 1) the waste is a liquid or fine suspension that can be spray-fed onto the bed, 2) solid bed material can be added through a lock hopper, by a screw conveyor line, or by a pressurized solids injector, 3) a relatively high bed temperature (400-750°C, depending of process chemistry) can be maintained using inbed combustion or external electrical resistance heaters, 4) temporal conditions (temperature, time, and composition) are suitable to drive key physical and chemical reactions to completion, 5) the bed particle size distribution can be held approximately constant to maintain adequate bed fluidization and mixing characteristics, 6) the particulate bed is non-agglomerating, 7) cleanup of high volumes of off gas (i.e., bed fluidizing gas requirements) is not a deterrent, 8) the bed particles and fines generated that are carried out with the fluidizing gas can be efficiently captured and recycled to the bed or collected with the bed product, 9) the bed and fines product can be remotely collected and containerized or transported to a storage bin, and 10) the off gas can be cleaned to meet applicable emission requirements.

Objectives

The primary objective of the INEEL laboratory scale trials was to demonstrate that the surrogate HLW feed can be reliably fed to a small electrically heated fluidized bed process and successfully converted into a uniform and remotely manageable synroc precursor that is immediately suitable for HIPing. This objective was accomplished by operating a fluidized-bed test unit at the INEEL. Variation of key operating conditions was intended to elucidate the effects of: 1) bed temperature, 2) feed flow rate (*i.e.*, bed processing capacity), 3) nozzle atomizing gas ratio (NAR)^a (*i.e.*, feed spray characterization), 4) fluidizing gas velocity, 5) waste loading, and 6) fluidizing gas composition.

Performance objectives of the trials included: 1) maintain a non-agglomerating stable bed that exhibits acceptable fluidization and mixing, is uniform in temperature, and reaches a steady mean particle diameter, 2) maximize the product (bed particles) to fines particles ratio (P/F), 3) achieve a bed turnover of 90% (containing less than 10 wt% of the Synroc B starting bed), 4) produce a product that contains less than 1 wt% nitrate/nitrite salt and that exhibits a uniform macro-crystalline or amorphous chemical matrix that can be converted into a ceramic synroc via HIPing, 5) minimize volatile cesium and ruthenium species carryover into the off gas system, and 6) produce a remotely transportable granular solid and fines that can be easily conveyed, gravity fed, or pneumatically transferred to a container.

Test Equipment

The INEEL 3.25-inch (82-mm) bench-scale fluidized bed test facility consisted of the following system components: 1) stainless steel hood enclosure, 2) fluidized bed calciner vessel, 3) solid feed addition line, 4) product collection pot, 5) liquid surrogate HLW feed system, 6) compressed gas delivery system, 7) electrical clamshell heating units and a temperature control system, 8) fines collection system, including a cyclone and a sintered metal filter, 9) off gas condenser with condensate collection unit, 10) off gas ducting with sampling ports and 11) temperature, pressure, and flow instrumentation.

Synroc B was used as the starting bed material, which consisted of the Zr, Ti, Al, Ba, and Ca oxide species. A reduced gas mixture, predetermined by ANSTO, and a compressed air environment was tested as alternative fluidizing/atomizing gas reactants throughout the subsequent tests. The fluidizing gas was delivered through a distribution system using a 3 tuyere bubble cap plate. Surrogate HLW was feed (using a Masterflex pump) to the calciner at rate that ranged from 0.5 - 1.5 L/hr with a mean of 1L/hr over 53 hours of feed addition. Approximately 1400 mL of starting bed was charged to the reactor, providing an initial fluidized bed height of 380 to 450 mm (15-18 inch). The system is designed for dynamic addition of solids during a run whenever attrition depletes the bed at a faster rate than bed growth. The product collection port was used extensively during the ANSTO trials as bed growth exceeded attrition losses.

Samples of the bed particles mixed with the fines material were HIPed to examine phase development elemental distribution and final consolidation characteristics. The samples were sealed in stainless steel cans and the HIPing was carried out in an AIP6-30H unit under the conditions of 1250°C/ 100MPa for 2 hours.

Powders produced were characterized using a JEOL JSM6400 scanning electron microscope (SEM) that was operated at 15 keV and equipped with a Noran Voyager energy-dispersive spectroscopy system (EDS).

ANALYSES/DISCUSSION

Two external mixing, siphoning nozzles provided by Spraying Systems were used in the fluidized bed demonstration: 1) a 40/100^b S.S. liquid cap with a 120 S.S. gas cap and 2) a 28/50 S.S. liquid cap with a 70 S.S. gas cap. The beginning fluidized bed tests were performed with a 40/100 liquid cap with a 120 gas cap, which in retrospect was too large for a flow rate of 1 L/hr or less and resulted in more occurrences of nozzle beards and cones. After abandoning the 40/100 liquid cap with a 120 gas cap for the smaller 28/50 liquid cap with a 70 gas cap and conducting spray pattern tests using the surrogate HLW feed, an upper and lower acceptable NAR for the smaller nozzle was determined to be between 700 and 200. Lower NARs resulted in large droplets, which quenched single particles and resulted in bed agglomeration. Conversely, extreme NARs resulted in increased fines generation by the following two mechanisms: 1) flash evaporation, and 2) particle attrition and particle grinding. An optimum NAR of 600 was determined to properly atomize the surrogate HLW feed and minimize undesirable processing conditions.

Solid Analyses

The product to fines ratio (P/F) is a measure of the mass ratio of the product and fines generated during a test and is a function of NAR and fluidizing velocity. The overall test results produced a P/F of 9.5 (less than 10 wt% fines), which was indicative of consistent bed growth with minimal particle attrition and minimal spray drying of the atomized surrogate HLW feed. Test results proved that a low NAR resulted in bed agglomeration and nozzle cones formation (or nozzle bearding). In cases with NAR levels above 600 (and especially above 900), fines generation by spray drying was unacceptable (>20 wt%). A fines generation of 10 wt% or less is desirable, provided the fines can be denitrated, recycled to the reactor, or either dissolved or suspended and recycled back to the feed solution.

Prior to the start of the fluidized bed trials, the Synroc B starting bed material was fluidized in an unheated solid fluidization column in order to establish an empirically optimum fluidization velocity. This was especially important for the irregular shaped Synroc B particle material. The minimum fluidization velocity was found to be 0.15 m/s for Synroc B starting bed. Next, the fluidizing rate was increased until vertical column mixing was observed, a condition that is desired in order to achieve good bed mixing and uniform deposition of the liquid spray on the bed. The preferred fluidizing velocity was 1.5-2.5 times the minimum for fluidization.

The trials utilized the sintered Synroc B as the starting bed (provided by ANSTO), which has an initial mass mean particle diameter (MMPD), harmonic mean particle diameter (HMPD), and bulk density of 0.377 mm, 0.304 mm, and 1.71 g/cm³, respectively. Figure 1 illustrates the particle dynamics for fluidized bed trials. MMPD signifies the particle diameter at which 50 wt% of the particles has a smaller diameter, while HMPD is interpreted as the particle diameter having the average external surface area. The HMPD is used when calculating pressure drop through the bed. The HMPD and MMPD are also used as a good indicator to determine whether agglomeration of the bed was occurring.



Fig. 1. Plot of HMPD, MMPD, and bulk density during waste calcination trials.

It can be assumed from Figure 1 that an optimum MMPD, HMPD, and bulk density was achieved without significant agglomeration after 53 hours of subsequent operation.

The bed was maintained between 575 to 620°C, which adequately evaporated the water and nitric acid in the surrogate HLW feed. The evaporated surrogate HLW feed (Synroc G) left behind solid-phase, calcined, metal compounds that formed additional bed material. Product was interactively recovered through bed and fines collection pots to maintain a constant bed height. As a result, the starting bed for subsequent tests was obtained by screening the bed of the previous run, until eventually; the starting bed for the latter tests was principally Synroc G calcine. A final bed turnover of 90% (containing less than 10 wt% of the Synroc B starting bed) was calculated assuming no bed attrition of the Synroc B starting bed.

Residual nitrate levels in the bed were less than 0.5 wt%, which is a prerequisite for HIPing. The granular product was then sent to ANSTO for chemical, solid analyses and HIPing treatment, which converted the particles via HIPing into its final ceramic waste form.

A high magnification image of the bed material surface revealed micron-size particles on the bed particle surfaces in Figure 2. This was indicative of fine feed droplets, which undergo flash evaporation and film boiling on the exterior of the hot particles. Film boiling is believed to become significant above 600°C, the characteristic heat and mass capacity of the bed particles. Film boiling on individual particles was also higher at low feed flow rates, since the mass of liquid deposited on a single particle was relatively low and did not quench the particle temperature.



Fig. 2. SEM secondary electron image of the bed material surface.

The backscattered electron image of bed particles in Figure 3 showed uniform deposition of the feed and homogeneous particle growth of surrogate HLW feed on the Synroc B seed particles. The bright (white) areas were Synroc G and the black areas were Synroc B. Bed agglomeration was also absent in all the SEMs as observed in the following figure.



Fig. 3. SEM backscattered image of the bed material.

The SEM image, Figure 4, of a final bed particle produced at the end of the fluidized bed runs, showed a 3-dimensional spherical character. Such particle geometry was very amenable to stable dense-bed fluidization.



Fig. 4. SEM secondary electron image of a final bed particle produced at the end of 53 hours of operation.

X-ray mapping was carried out on a sample of final bed material to examine the distribution of the major elements within the particles. The elements mapped were Ti, Al, Ni, and La. As seen in Figure 5, they were well distributed through out the particle.

An efficient cyclone separator and sintered metal filter (2 μ m pore) achieved greater than 99% fines collection, as indicated by the specific gravity measurements of the condensate being near unity. The fines lost from the bed exhibited 1-2 wt% residual nitrates, indicative of inadequate operating conditions (time and temperature) in the fines collection equipment. The cyclone gas outlet temperature was approximately 410°C, while the fines filter outlet gas temperature was approximately 325°C. The cyclone and filter fines vessels would require further heating to completely calcine the particles.

Fines generation is attributed to two mechanisms -1) spray drying (light porous particles in the SEM graphs, principally Synroc G) and attrition of bed particles (dark fines with straight edges, principally Synroc B) as observed with the cyclone fines in Figure 6. Approximately 50% of the cyclone fines were generated by spray drying and 50% by attrition. The figure also revealed larger fines with "dusting" or adhesion of smaller particles to the large fine particles surfaces.



e) Ni X-ray map Fig. 5. X-ray maps for a bed sphere comprised of Synroc G.



Fig. 6. SEM Backscattered electron image of cyclone fines.

The majority of the filter fines are sub micron and were attributed mainly to spray drying, indicating a majority of Synroc G compounds. The fines collected by the fines filters were extremely small (<10 μ m diameter, with an average less than 1 μ m). The SEM image in Figure 7 indicated that fines cluster due to electrostatic attraction forces. This phenomenon has the advantage of making the fine ensembles behave with single-particle mechanics. The fines appeared to be hollow micro spheres produced by spray drying of the atomized feed. The fines generated in the process remained in an "entrainable" non-caking state with no indication of particle agglomeration.



Fig. 7. SEM Backscattered electron image of filter fines.

SEM images indicate dense particle morphology that can be achieved with the surrogate HLW feed. SEM backscattered electron images of the cyclone fines and filter fines particles reveal that a large fraction of the fines (white, Synroc G fines) generated by the process are small (0-100 μ m) and appear to be porous. The fines analyses reveal a bi-modal distribution of fines, with a mean at approximately 0.8 μ m and 15 μ m for the cyclone fines and 0.6 μ m and 6 μ m for the filter fines.

Chemical Analyses

Both an oxidizing and reducing atmosphere were tested separately to fluidize the bed particles during the trials. NO_x reduction in the bed was highly efficient at both reducing and oxidizing gas conditions. However, the colorless off gas, as well as qualitative measures using Draeger tubes tube (1-10 PPM scale), revealed that the concentrations of NO_x were low.

The condensate contained higher amounts of nitrate and nitrite ions when the system was operated in an oxidizing rather than reducing mode. The condensate catch exhibited a specific gravity averaging near 1.0 g/ml, indicative of minimal fines dissolution. The condensate collected was also moderately acidic (pH = 0.3-2.0 depending on oxidizing versus reducing gas conditions). A single shell-and-tube condenser cooled the off gas while collecting 90% of the water in the feed. The remaining water vapor was vented with the gas to the atmosphere through the building exhaust.

Potentially volatile Cs and Ru both exhibited a decontamination factor of >99.98 wt% (concentration in the condensate versus the concentration in the liquid feed) under either oxidizing or reducing gas conditions. This result (<100 wt%) may have been due to condensation of volatile Ru and Cs compounds in the fines filter cake. The condensate sample exhibited negligible solubility of either Cs or Ru compounds. Both Ru and Cs were expected to exhibit relatively high volatility under oxidizing gas and reducing conditions and at a test temperature of 615°C.

HIPed Product

The SEM examination of the HIPed samples showed a dense ceramic, with little or no porosity associated with the Synroc G. The majority of the porosity found was associated with the initial starting bed material, sintered Synroc B, and the Ti metal addition. Sintered Synroc B was chosen as the seed material, apart from its fluidizing characteristics, because it is compositionally different and would aid in distinguishing it from the Synroc G. This can be seen in Figure 8 in which (a) shows a Synroc B seed at the center of the particle and (b) showing a particle built up on a new seed of Synroc G, confirming that as the calcination process progressed, particle production was attributed to new seed material being created in the fluidized bed calciner.

It was also found that by recovering the fines (cyclone and filter fines), heat treating them to remove residual nitrates and then adding them to the synroc calcine that is to be HIPed; the packing density improved by 20%. There are multiple benefits for the waste immobilization process in doing this as it increases the throughput of the HIPing process and reduces the amount secondary waste produced by the calcination process.



Fig. 8. Microstructure of HIPed calcine (a) Initial product growth on Synroc B starting bed and (b) as calciner run time increased more particle production on seed material generated insitu.

CONCLUSION

Calcination of the surrogate HLW feed was feasible at test temperatures ranging from 575-620°C. No appreciable agglomeration occurred that could be attributed to molten or liquidous phases, except when the feed was not properly atomized and large droplets impinged on the bed. Less than 1.5 wt% residual nitrates remained in the cyclone and filter fines while nitrate levels in the bed were less than 0.5 wt%. Particle diameters and bulk particle density reached optimum levels after 53 hours of subsequent operation. The series of tests eventually provided a composite bed containing >90 wt% Synroc G and a P/F of 9.5. Both reducing and oxidizing gas conditions resulted in efficient NO_x reduction in the bed and decontamination factors of >99.98 wt% for volatile Cs and Ru compounds. The following conditions which proved highly successful were: 1) a bed temperature of 600°C, 2) a fluidizing gas velocity of 0.32 m/s, 3) a surrogate HLW feed rate of 1.0L/hr, and 4) a NAR of 600.

The synroc ceramic processing successfully demonstrated the feasibility of producing a homogeneous, dense waste form product from a surrogate U-rich dissolver waste. Fluidized bed technology conducted on a laboratory scale calcined a surrogate HLW feed into a synroc precursor. HIPing converted the calcined synroc precursor into a synroc ceramic. The combined technologies demonstrated, through significant reductions in waste volume, and immobilization of the surrogate radioactive constituents, that a final synroc ceramic waste form could be produced. The synroc ceramic production done here provided valuable data and operating experience to design, fabricate, and operate equipment that produces synroc ceramic products on a scale for the actual U-rich waste, of the same quality as produced on the laboratory scale.

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FOOTNOTES

- ^a Defined as the volumetric flow rate of gas in the air-side (annular orifice) divided by the volumetric flow rate of liquid feed through the center orifice of an external mix nozzle.
- ^b Cap dimensions are in 1000th inch, measured as inside orifice diameter and outside orifice diameter of the liquid cap and inner diameter of the orifice for the gas cap. S.S. is 304 Stainless Steel.