STEAM REFORMING TECHNOLOGY FOR DENITRATION AND IMMOBILIZATION OF DOE TANK WASTES

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ABSTRACT

THOR Treatment Technologies, LLC is a joint venture formed in June 2002 by Studsvik, Inc. (Studsvik) and Westinghouse Government Environmental Services Company LLC (WGES) to further develop, market, and deploy Studsvik's patented THORSM non-incineration, steam reforming waste treatment technology. This paper provides an overview of the THORSM steam reforming process as applied to the denitration and conversion of Department of Energy (DOE) tank wastes to an immobilized mineral form. Results of demonstration test programs on nonradioactive surrogates for Hanford Low Activity Waste (LAW) and Idaho National Engineering and Environmental Laboratory (INEEL) Sodium Bearing Waste (SBW) will be discussed. Results of the Hanford disposal performance evaluations will be presented. Modular design and deployment options will be outlined for DOE applications.

Using the THORSM steam reforming technology to treat nitrate containing tank wastes could significantly benefit the DOE by reducing capital and life-cycle costs, reducing processing and programmatic risks, and positioning the DOE to meet or exceed its stakeholder commitments for tank closure. Specifically, use of the THORSM technology can facilitate processing of up to 75% of Hanford tank wastes without the use of vitrification, yielding substantial life-cycle cost savings.

INTRODUCTION

Tank wastes are composed of a wide range of radioactive salt cakes and supernatant solutions that contain high concentrations of sodium, aluminum, nitrates, nitrites, nitric acid or hydroxides, and sulfates with significant concentrations of chlorides, fluorides, heavy metals, radionuclides, and most other inorganic elements as well. The THOR SM steam reforming process has been proven to efficiently immobilize the radionuclides, sodium, sulfate, chlorides, fluorides and nonvolatile heavy metals into a stable, water insoluble mineral matrix.

In the THORSM process, waste feed, superheated steam, and co-reactants are introduced into a steam reformer vessel where liquids are evaporated, organics are destroyed, and reactive chemicals in the waste feed are fully converted to a stable waste product that incorporates essentially all of the radionuclides. The fluidized bed design of the steam reformer provides a large surface area for the waste to fully and efficiently react. Carbon and iron-based co-reactants (reductants) are used to convert nitrates and nitrites directly to nitrogen gas in the reformer. Clay or other inorganic co-reactants are added to the waste feed or bed to convert the radionuclides,

alkali metals (sodium and potassium), sulfate, chloride, fluoride, phosphates, and non-volatile heavy metals into an immobilized mineral product. The process operates safely at near ambient pressure and at moderate temperatures (600–750˚C).

The steam reforming process destroys RCRA and TSCA organic constituents. Toxic organics are converted to carbon dioxide and water vapor in the steam reformer by a combination of steam reforming and oxidizing reactions.

The off-gases are cooled and filtered so that the primary emissions released to the atmosphere from the process are carbon dioxide and water vapor.

The THORSM steam reforming process chemistry is summarized as follows:

- Nitrates, nitrites and nitric acid are directly reduced to nitrogen gas with less than 300 ppm of NOx present in the off-gas from the fluid bed;
- Sodium, potassium and aluminum in the waste feed are converted to a stable, water insoluble mineral product, e.g. mostly Nepheline $Na₂O-Al₂O₃-2SiO₂$ (Na-Al-Si/NAS), Nosean, and Sodalite, that each have a Na₂O loading of greater than 19% by weight;
- Sulfates, chlorides, and fluorides in the waste feed are bound as a part of the NAS mineral structure so that less than 5% is volatized to the off-gas. Phosphates are also bound into a mineral structure;
- Radionuclides in the waste feed are retained (>99.9%) in the solid, mineralized product, with the exception of tritium, carbon¹⁴, and iodine that are largely volatized;
- The mineralized product will pass the Toxicity Characteristic Leaching Procedure (TCLP) test Uniform Treatment Standard (UTS) limits for heavy metals, and has less than 10 ppm residual nitrates and nitrites; and
- An optional sodium carbonate, Na_2CO_3 , product can be produced that can be directly vitrified in a joule-heated melter due to its low melting point $(*845^{\circ}C)$.

PROCESS TECHNOLOGY OVERVIEW

Na-Al-Si is the preferred final waste product from steam reforming for applications that require waste immobilization without vitrification or grouting. For sites that require burial of only immobilized monolithic solids, the monolithic Na-Al-Si form can be produced. For wastes that must be vitrified, the preferred product from steam reforming is sodium carbonate, which can be readily vitrified in joule-heated melters. The sodium carbonate product can be provided in the dry granule/powder form or as an aqueous solution and/or slurry for feed to a vitrification melter or to a grout facility. The granular/powder forms of Na-Al-Si and sodium carbonate for transuranic (TRU) wastes are suitable for direct disposal at the Waste Isolation Pilot Plant (WIPP).

The following sections of this technical overview provide discussions of process chemistry in the steam reformer, reduction of nitrates to nitrogen, conversion of alkali metals to stable mineral form, neutralization of acid gases, removal of solids carryover from the reformer, destruction of RCRA and TSCA organics, reduction and immobilization of hazardous metals, and finally, secondary process waste generation, emissions, and discharges. A simplified flow diagram of the steam reforming process is provided in Fig. 1. A more detailed process flow diagram is provided in Fig. 2.

Fig. 1 Simplified flow diagram of THORSM fluidized bed steam Reforming Process

Fig. 2 Process flow diagram of the THORSM fluidized bed steam reforming process.

PROCESS CHEMISTRY IN THE STEAM REFORMER

In the first stage steam reformer (DMR), the granular/particle bed material is fluidized with near ambient pressure superheated steam. The tank waste is injected directly into the fluidized bed just above the fluid gas (steam) distributors by means of a slurry pump. Solid co-reactants, such as granular carbon, clay, and iron oxide catalyst are also pneumatically transferred into the fluid bed. The DMR fluid bed is operated in strongly reducing conditions to facilitate high reduction of nitrates and nitrites to nitrogen gas. A second stage reformer (CRR) is operated under oxidizing condition by injection of oxygen into the middle zone of the CRR fluidized bed. Process gases from the DMR pass through the CRR oxidizing zone which converts residual carbon reductants and organics into carbon dioxide and water vapor. Several chemical and physical reactions take place in the steam reformers:

- All liquids are evaporated in the DMR.
- Sodium, potassium, and aluminum in the waste feed are converted in the DMR into a stable mineralized product, Na-Al-Si, which contains essentially all the radionuclides and inorganic elements in the waste feed stream.
- Nitrates and nitrites in the waste feed are converted into nitrogen gas in the DMR by the carbon and metal reductant solids and CO and H_2 gases in the bed.
- Organics are initially converted into light volatile hydrocarbons such as methane, carbon monoxide, hydrogen, carbon dioxide, and water in the DMR bed. In the CRR bed, oxygen is injected to fully oxidize the gaseous constituents. The off-gas stream from the dual stage reformers consists of water vapor, carbon dioxide, nitrogen, fine particles of solid product, and trace quantities of acid gases not converted to a mineralized form in the reformers.
- Certain hazardous metals are reduced to a non-hazardous valence state (e.g., Cr(VI) is reduced to Cr(III) and are chemically bound in the solid product. Other hazardous metals, such as lead, are also chemically bound in the solid Na-Al-Si product in the DMR.

The DMR steam reformer is operated at near ambient pressure at 600° C to 750° C and can be electrically heated (small units) or operated in an auto-thermal mode, whereby the energy needs are supplied by the incoming superheated steam and by the oxidation of organics from the waste and carbon reductants. For production scale units, auto-thermal steam reforming is the preferred mode of operation. The CRR reformer operates under vacuum at 800° C to 1000° C.

NITRATE REDUCTION TO NITROGEN GAS

Substantial research has been performed by many organizations over the past 50 years to develop an efficient nitrate destruction process. Studsvik developed the $THOR^{SM}$ steam reforming technology to provide unparalleled performance with the following demonstrated results:

- Residual nitrate content in the reformed solid waste product is $\langle 10 \text{ ppm} \, ^{[1,2]} \rangle$,
- Direct conversion of >98% of nitrates and nitrites in the waste feed to nitrogen gas $[1,2]$, and
- Total NOx in off-gas from the reformers is <300 ppm **[1,2]**.

The above referenced high conversion of nitrates to nitrogen requires that several specific features and reactions be incorporated into a single step thermal process: 1) fluidized bed material with high surface area and high heat transfer capability, 2) high energy generation to evaporate and superheat the water in the typical aqueous waste feed, 3) strongly reducing condition that is provided by the carbonaceous and metal-based reductants, 4) superheated steam that generates carbon monoxide and hydrogen inside the bed from the reaction of steam with the carbon reductant(s), and 5) co-reactant(s) that convert the alkali metals (Na and K) into higher melting point mineral compounds to prevent formation of low melting point eutectic salts and unwanted bulk agglomerations in the fluid bed.

The fluidized bed material can include ceramic media and/or reformed product granules/powders. The incoming waste feed coats the fluidized particles and is instantly dried. The large active surface of dried nitrates readily reacts with the hot carbon reductant particles, carbon monoxide and hydrogen gases, and the reduced metal and metal oxides particles in the fluidized bed.

Experience has shown that thermal denitration without the presence of a reductant will result in reduction of residual nitrates in the solid product of only up to 90%, i.e. 90% of the incoming nitrates are thermally volatized leaving approximately 10% of incoming nitrates in the final solid waste product. The typical total NOx content, without use of a reductant, will range from 20,000 ppm to over 50,000 ppm total NOx in the process off-gas. The addition of carbonaceous reductants to nitrate wastes in a thermal denitration process has shown substantial improvements with up to 98% nitrate reduction in the solid product and a corresponding reduction of total NOx in the process off-gas to $3,000$ ppm to $10,000$ ppm $\left[1,2\right]$. A further order of magnitude improvement in nitrate conversion is possible by use of both carbonaceous and metal-based reductants in the THORSM DMR fluid bed. The presence of low-cost metal oxides, e.g. iron oxide particles, in the fluid bed results in the unexcelled performance of the THORSM process for effective reduction of NOx in the off-gas and only trace levels of residual nitrates in the solid reformed product, as summarized above.

ALKALI METAL CONVERSION TO MINERAL FORM

The THORSM steam reforming process has the unique capability to convert alkali metals, Na and K, as well as other inorganic components into stable minerals in the fluid bed. The presence of alkali metals in fluid beds has historically proven to be a serious operational problem as sodium and potassium form a variety of low melting point salts with sulfate, chloride, silica, and borate materials found in typical tank wastes. These low melting point salts combine to form eutectic mixtures that generally melt at much lower temperatures than the pure compounds. The presence of eutectic compounds in a fluid bed usually results in the formation of bulk agglomerations as the melted salts tend to bind the fluidized media particles into larger and larger pieces.

Studsvik has performed intensive work in developing and proving that the addition of selected inorganic co-reactants to a reducing fluid bed can prevent the formation of low melting point eutectic compounds. Certain inorganic co-reactants preferentially substitute for the typical low melting point anions of the Na and K salts. The preferred co-reactants include clay (an aluminosilicate), and Ca and Al compounds that combine with alkali metals to form synthetic naturally occurring minerals. The typical minerals formed for addition of clay as the co-reactant as listed below (Note - only the main elemental constituents are shown for simplicity).

- Nepheline, Na-Al-Si
- Nosean, Na-Al-Si-SO₄

The generation of water-insoluble products is preferred, however, most sodium and potassium compounds are water-soluble, e.g. sodium carbonate, sodium aluminate, etc. The water-soluble sodium compounds will generally require further stabilization (grouting, solidification, vitrification, etc.) prior to disposal to prevent water dissolution of the buried product into the ground water. Therefore, the generation of water-insoluble sodium products is very desirable. For this reason, the preferred products are the water-insoluble species such as Nosean and Nepheline. The Nepheline, Nosean, and related sodium-alumina-silicate (Sodalite) compounds form a crystalline, cage-like structure that has the ability to substitute and bind large atoms (such as Cesium, Technetium, and other radionuclides and heavy metals) within the crystalline structure to produce a highly leach-resistant product ^[1,2,4,5,6]. The NAS compounds have demonstrated TCLP leaching that is substantially lower than the Land Disposal Restrictions (LDR) Universal Treatment Standard (UTS) limits for heavy metals. Typical aluminosilicate compounds are shown in Fig. 3.

Fig. 3 Steam reformer chemistry converts sodium, potassium, sulfates, chlorides, fluorides, and radionuclides into sodium-aluminosilicates (simplified formulas shown)^[1].

DESTRUCTION OF RCRA AND TSCA ORGANICS

The THORSM steam reforming process destroys organics in the wastes including any RCRA and TSCA organic constituents. Specifically, the process destroys RCRA organic waste codes D018 through D043 and F001 through F005 and TSCA-listed PCBs. The THORSM process is a nonincineration thermal treatment system.

The organics are initially volatized and steam reformed in the DMR into carbon monoxide, hydrogen and a small quantity of light hydrocarbons, such as methane and ethylene **[1]**. The light hydrocarbons and the carbon monoxide and hydrogen gases generated from the steam reformation of carbon and organic materials are further oxidized in the CRR to carbon dioxide and water vapor by addition of oxygen to the middle zone of the CRR bed.

The formation of dioxin and furan compounds has been studied by the DOE for a steam reforming process that has similar organic destruction capability as the THOR SM process. The results of the extensive organic destruction demonstration program (DOE Contract No. DE-AR21-95MC32091 with Thermo-Chem, 1995) confirm that dioxins and furans are not generated in the steam reformer and typical off-gas systems. Indeed, any dioxins or furans in the waste feed can also be destroyed in the reformer systems.

NEUTRALIZATION OF ACID GASES AND REMOVAL OF SOLIDS CARRYOVER FROM OFF-GAS

Testing on Hanford Low Activity Waste (LAW) waste feed surrogates has shown that over 94% of the sulfur compounds, fluorides and chlorides in the waste feed react in the DMR with the clay co-reactant and become an integral part of the final NAS waste product crystalline structure. A small amount of the S, Cl, and F in the incoming waste feed forms volatile acid gases, which pass through the steam reformer and are largely absorbed on the Ca-rich bed in the CRR **[1,2]**.

A filter removes and collects fine particulates that may carryover from the DMR and CRR reformers for packaging with the solid product from the reformers. The vast majority of the solid product comprises granular NAS minerals removed from the bottom drain on the DMR.

IMMOBILIZATION OF HAZARDOUS METALS

The strong reducing environment in the steam reformer converts certain hazardous heavy metals to non-hazardous valence states. For example, Cr(VI) is reduced to Cr(III) and is incorporated into the NAS product. In addition, virtually all of the lead that enters the reformer is chemically bound up in the NAS product as well. The NAS product has been shown to pass the TCLP test easily meeting the LDR UTS limits for leachability by a factor of 10 to 100 for Cr, Pb and Ni **[1]**.

Additional work is needed to verify the performance of the THOR SM process on other heavy metals. It is expected that other heavy metals will be retained in the solid mineralized product with the exception of mercury. If mercury is present in the waste feed, a mercury removal capability will need to be provided in the $THOR^{5M}$ off-gas system as mercury will volatize to the gas phase during thermal treatment. It is expected that a commercially available granular activated carbon media can be placed downstream of the HEPA filter to remove essentially all volatile mercury from the off-gas stream.

SECONDARY WASTE GENERATION, EMISSIONS, AND DISCHARGES

A significant benefit of the THORSM steam reforming process is that the facility produces zeroliquid releases. A production scale $THOR^{SM}$ steam reforming plant has been constructed by Studsvik in Erwin, Tennessee (SPF) for processing Low Level Radioactive Waste. The Erwin steam reforming facility has been commercially operational at full-scale for over four years with no release of liquids. All water is released as water vapor out the monitored ventilation stack.

All organics are processed through the reformer process system and are converted to carbon dioxide and water vapor with Destruction and Removal Efficiency exceeding 99.99%.

The emissions of SOx, HCl, HF, NOx and other regulated pollutants are estimated in Table I. The emissions data presented in Table I represent actual readings from the Erwin steam reforming facility Continuous Emissions Monitoring System (CEMS) and correlations based on CEMS data from previous bench-scale and pilot-scale demonstration programs performed by Studsvik, Bechtel National Inc, and Idaho National Engineering and Environmental Laboratory.

DEMONSTRATION PROGRAM EXPERIENCE

Studsvik developed the THORSM steam reforming process over a period of several years. Extensive bench-scale and pilot-scale test programs were implemented to qualify and optimize the steam reforming process and to make the technology economical, reliable and robust with unsurpassed safety. Six separate demonstration programs have been or are currently in progress specifically for processing nitrate wastes.

Studsvik performed the initial proof-of-process denitration test program concurrent with the qualification test program performed for the Studsvik Processing Facility (SPF). The test was performed using the Studsvik 6" fluid bed system at the Hazen Research facility located in Golden, CO. The test was performed on 5.2 M NaNO₃ with sugar, carbon, and iron oxide reductants. The fluid bed operated throughout the entire demonstration run with no in-bed agglomerations, with NOx in the reformer off-gas consistently below 500 ppm. For over half of the test program the NOx levels in the reformer off-gas were <100 ppm. A low volume sodium carbonate product was obtained.

A second test program was performed by Lockheed Martin Idaho Technologies Company (LMITCO) using acidic INEEL sodium bearing waste (SBW) surrogate. The demonstration investigated several changes in operating parameters and use of additives in a small, 3", lab-scale fluid bed system installed at SAIC's STAR facility in Idaho Falls, ID. The final process runs demonstrated high conversion of nitrates to nitrogen with <500 ppm NOx in the reformer offgas.

Studsvik performed a third demonstration under contract with Bechtel National Inc to demonstrate that the THORSM process could produce a leach resistant mineral product. The bench-scale demonstration of the THORSM process used Hanford Low Activity Tank Waste (LAW) surrogate. The demonstration utilized Studsvik's 6" fluid bed system located at Hazen's facility. The demonstration was performed in November and December 2001. The test results were very positive, with successful operation of the process to generate consistent NAS and sodium carbonate products. The off-gas from the process over a several day period confirmed the high nitrate conversion to nitrogen gas with the same results as in the initial Studsvik test. Extensive tests and evaluations performed at Pacific Northwest National Laboratory indicated that the long-term leach resistance of the steam-reformed NAS product surpassed the performance of most boro-silicate glasses. The results of this demonstration are documented by five reports [1,2,4,5,6].

The fourth test program was performed by Bechtel BWXT Idaho, LLC (BBWI) to demonstrate the THOR SM steam reforming process using a new 6" fluid bed reforming system installed at SAIC's STAR facility in Idaho Falls, Idaho. The purpose of the demonstration was to produce a sodium carbonate product while maintaining NOx emissions <1000ppm while processing INEEL Sodium Bearing Waste (SBW) surrogate. The test program was performed in January, 2003 using an acidic nitric acid-sodium nitrate solution as SBW feed. A sodium carbonate product was produced with >98% nitrate conversion to nitrogen and <10ppm nitrates in the solid product **[2]**.

The fifth test program was performed by BBWI to demonstrate the THORSM steam reforming process using the 6" fluid bed reforming system installed at SAIC's STAR facility. The purpose of the demonstration was to produce a sodium carbonate product and destroy tetraphenylborate (TPB) and benzene while processing SRS Tank 48H tank waste surrogate. The test program was performed in September 2003 using a basic sodium nitrate solution with TPB as waste feed. The test program produced a sodium carbonate and sodium silicate products with high nitrate conversion to nitrogen. TPB and benzene were destroyed **[3]**.

The sixth test program was performed by BBWI to demonstrate the THORSM steam reforming process using the 6" fluid bed reforming system installed at SAIC's STAR facility. The purpose of the test was to produce sodium carbonate and NAS products while optimizing the use of two reductants, sugar and carbon. Carbon reduction parametric tests were performed. The test program was performed in November 2003 using an acidic-sodium nitrate solution as SBW surrogate. A high density sodium carbonate product was produced with high conversion of nitric acid and nitrates to nitrogen gas. A second test run was performed to produce a water-insoluble NAS product. The addition rate of co-reactants was adjusted to produce a NAS product even with the high aluminum content of the SBW surrogate.

COMMERCIAL RADIOACTIVE WASTE PROCESSING EXPERIENCE

The Studsvik Processing Facility (SPF) located in Erwin, Tennessee has been in commercial operation processing low-level radioactive waste (LLW) for over four and one-half years. The SPF has the capability to safely and efficiently receive and process a wide variety of solid and liquid LLW streams including: ion exchange resins (IER), charcoal, graphite, sludge, oils, solvents, and cleaning solutions with contact radiation levels of up to 400 R/hr. The licensed and heavily shielded SPF can receive and process liquid and solid LLWs with high water and/or organic content.

The SPF employs the THORSM process. The THORSM technology is suitable for processing hazardous, mixed, and dry active (DAW) LLW with appropriate licensing and waste feed modifications. The final reformed residue product comprises a non-dispersible, granular solid suitable for long-term storage or direct burial in a qualified container.

Operations have demonstrated consistent, safe, reliable, robust operating characteristics with volume reductions up to 15:1 and weight reductions up to 10:1 when processing depleted, mixed bed, ion exchange resins, with over 99.8% of all radionuclides in the waste feed incorporated in the final solid residue product. Over 110,000 cuft of LLW ion exchange resin, oils, aqueous liquids, granular carbon, paper and plastics have been successfully processed for commercial nuclear power plant customers since commencement of commercial operation in July, 1999. A description of the SPF is available on the internet – search for "Studsvik Processing Facility" or visit the Studsvik website.

DEPARTMENT OF ENERGY DEPLOYMENT INITIATIVES

Over the past three years the US Department of Energy (DOE) has expressed interest in the THOR SM process for denitration and stabilization of the large quantities of high nitrate tank wastes. Two DOE procurements are underway that could provide the opportunity to implement the steam reforming process at the Hanford and SRS sites. Both these procurements are based upon the deployment of modular steam reforming systems. Figure 4 illustrates the modular system designed for deployment at the SRS site for processing Tank 48H waste. The modular system includes all process vessels shown in Fig. 2 individually arranged in vertical modules that can be shipped over-the-road from the fabrication and testing shop to the site. A modular steel shield wall (not shown in Fig. 4) is installed around the modules to provide effective shielding. Both the shield structure and the modular process systems are designed to meet all seismic and spill protection requirements. A separate ventilation module houses the HEPA filters and ventilation blowers. A separate additives skid provides the clay, carbon and iron oxide catalyst to the DMR in proportion to the waste feed injection rate. Typical utility services for a full-scale system includes: low-pressure steam, instrument air, water for direct spray cooling of the CRR off-gas, electricity for the blowers and fluid gas superheater, nitrogen for purges, and oxygen.

Fig. 4 Modular Steam Reforming System for Savannah River Site Tank 48H Application

CONCLUSION

In summary, the THOR SM process has many salient features as listed below:

- Waste feed can include solids, liquids, slurries, and gases
- Acidic and basic tank wastes can be processed without neutralization
- Nitrates, nitrites, and nitric acid are destroyed with $\langle 10 \rangle$ ppm residual nitrates in solid product, < 300 ppm NOx in reformer off-gas
- S, Cl, F, and P are immobilized in a stable mineral form, with no secondary scrubber solution waste stream and <10 ppm SOx, HCl, or HF in off-gas at stack
- Alkali metals, radionuclides, and non-volatile heavy metals are immobilized in stable, water insoluble, Na-Al-Si/NAS mineral form.
- Greater than 99.9% of radionuclides are incorporated into the solid product. Tritium, carbon 14 , and iodine are generally partitioned to the off-gas.
- Na-Al-Si product meets LDR UTS limits for heavy metal leachability (TCLP test)
- Final solid product can be sodium-alumina-silicate or sodium carbonate in granular/powdered or monolithic form
- Systems operate at near atmospheric pressure or under vacuum (20-40 inch W.C.)
- Safe passive shutdown can be accomplished in ≤ 15 seconds
- Reformer contains very low inventory of unreacted waste
- Vessels can be drained of contents and decontaminated by acid or water washes to allow contact maintenance on most components
- Facility is zero-liquid release as water vapor is discharged up the stack
- Wastes with high water content, high organic contents, and high sulfate content are efficiently processed
- The THORSM process is covered by issued and pending patents, see US Patent No. 6,280,694 and 6,084,147.

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FOOTNOTES

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