BASF Mercury Sorbent HX™

William Hizny¹, Fabien Rioult PhD¹, Xiaolin Yang PhD¹

¹BASF Corporation, 25 Middlesex/Essex Turnpike, P.O. Box 770, Iselin, NJ 08830

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ABSTRACT

BASF’s Mercury Sorbent HX™ is a brominated mineral sorbent enabling a low carbon solution for mercury emissions compliance. Utilities operating coal-fired electricity generating units are challenged to control mercury emissions to meet state and federal regulatory requirements, such as MATS (Mercury and Air Toxics Standards). For many of these utilities, adoption of dedicated mercury control technologies, such as powdered activated carbon injection, may threaten valued revenue streams. For example, Class C fly ash contaminated by moderate levels of activated carbon may become unsalable into ready-mix concrete applications, such as a partial replacement for Portland cement. Mercury Sorbent HX™ offers an innovative, cost-effective solution to meet these challenges of regulatory compliance and preserved fly ash value. Results from the latest full scale field demonstrations of Mercury Sorbent HX™ are shared in which the technology satisfied impending MATS performance requirements for controlled mercury emissions. The value differentiation of Mercury Sorbent HX™ over other sorbent technologies in preserved fly ash salable properties is reviewed via both economic modeling and by concrete air characterization testing conducted by BASF Construction Chemicals.

INTRODUCTION

In February 2012, the United States Environmental Protection Agency (US EPA) published the Mercury and Air Toxics Standards (MATS) rule to control emissions of heavy metals and acid gases classified as toxic air pollutants from sources that include coal-fired electricity generating units. MATS established numerical emissions limits for mercury, particulate matter (as a surrogate for toxic non-mercury metals), and HCl (a surrogate for all toxic acid gases)¹. With respect to mercury emissions from coal-fired units designed to burn coal with a Gross Calorific Value (GCV) ≥ 8,300 Btu/lb (19,306 kJ/kg), MATS requires existing units to meet either a heat input-based emission limit of 1.2 lb/TBtu (0.516 mg/GJ) or an electrical output-based emission limit of 1.3 x 10⁻² lb/GWh (0.0059 kg/GWh).²

Existing coal-fired electricity generating units not equipped with both an SCR (selective catalytic reduction system) for NOₓ control and a wet-FGD (flue gas desulfurization scrubber) for SO₂ control, and thus unable to claim the co-benefit of mercury control offered by these technologies used in series, are most likely to adopt a dedicated mercury control technology such as mercury sorbent injection. Powdered activated
carbon in conjunction with supplemental halogen chemistry supplied either on the carbon sorbent itself or via chemical addition to the coal has been shown to be an effective means to control mercury emissions. However, powdered activated carbon may not be a preferred solution for those electricity generating units whose fly ash is sold into ready-mix concrete applications as a partial replacement for Portland cement. Powdered activated carbon is known to absorb air entraining admixtures used in concrete. The loss of entrained air and the deterioration of the air void system—larger air voids spaced farther apart—reduce the concrete’s freeze-thaw durability and ultimately may make the fly ash containing the powdered activated carbon unwanted and thus unsalable. The loss of fly ash sales coupled with the incurred cost of fly ash disposal may pose a significant economic challenge to coal-fired utilities. For these units, a mercury sorbent technology that can both satisfy the MATS mercury requirement and be economically cost-effective by preserving fly ash salability may be a preferred solution.

**Mercury Sorbent HX™**

To meet the challenge of reliable mercury emissions control and preserved fly ash salability, BASF has developed Mercury Sorbent HX™, a brominated mineral sorbent enabling a low carbon solution for mercury emissions compliance.

Mercury Sorbent HX™ is a physical mixture of a brominated mineral with an activated carbon. The bromine, which is preferentially coated on the periphery surface of a macroporous mineral, acts as an oxidizing agent for mercury. As a functional surface, the mineral extends the oxidizing agent throughout the flow facilitating interactions between vapor phase elemental mercury and both activated carbon and the native fly ash. Activated carbon acts as a mineral particle flow enhancement agent and the primary mercury capture site. The nominal roles of each component in Mercury Sorbent HX™ may not be mutually exclusive.

Using XPS (X-Ray Photon Spectroscopy), a consistent mass balance of the surface bromine-to-mineral ratio before and after the activated carbon inter-mix step in the production of Mercury Sorbent HX™ supports the hypothesis that the bromine resides on the mineral component of the sorbent and does not migrate to the activated carbon. By remaining uncoated, the activated carbon may make more efficient use of its pore structure to capture mercury, thus minimizing its relative content in the sorbent.

**EXPERIMENTAL METHOD**

**Full Scale Field Demonstration for Mercury Control**

A full scale field demonstration of Mercury Sorbent HX™ was conducted to assess its ability to capture mercury at levels consistent with MATS compliance. This demonstration was part of a larger test program conducted by an anonymous US utility to assess its MATS compliance options.
The test site had a full power load rating in excess of 800 MW and was fueled by Powder River Basin (PRB) coal. Although a coal assay was not performed, it was reasonable to assume that the coal was not atypical from the commonly understood properties of PRB coal with respect to low sulfur and low halogen content. The unit was equipped with a cold-side electrostatic precipitator (ESP) having a specific collection area in excess of 500 square feet per 1000 actual cubic feet per minute (98.5 square meter per actual cubic meter per second) exhaust gas flow. The utility currently sells its fly ash through a marketer into the ready-mix concrete industry. Retention of these sales while being MATS compliant for mercury emissions was of significant concern to the utility.

The test program commissioned by the utility took advantage of the unit’s symmetry by designating one side as the “test” side in which mercury sorbent was injected and the other side as the “control” side so as to capture real-time fluctuations in the uncontrolled mercury emission. A schematic of the unit illustrating this arrangement is presented in Figure 1.

![Figure 1. Field demonstration site schematic](image)

Mercury sorbents undergoing tests were injected upstream of the air preheater on the “test” side using a proven, portable sorbent injection system fed by supersacks suspended over and sealed to a conical hopper. A volumetric screw feeder metered sorbent from the hopper into an eductor, which was driven by a blower drawing ambient air into the system as a means of pneumatically conveying the sorbent through temporary delivery lines and into the duct. Load cells integrated into the injection system superstructure supplied a feedback signal tracking the net loss of sorbent over time and trimming the sorbent injection rate to a fixed set-point. Mercury Sorbent HX™ was fed through this same system as were the powdered activated carbons evaluated by the utility.
A Continuous Emissions Monitoring System (CEMS) for vapor phase mercury was installed downstream of the ESP on both the “test” and “control” sides. Thermo Scientific Mercury Freedom System™ CEMS were used. Modified EPA Method 30B (sorbent trap) sampling was conducted at selected times to confirm the validity of the CEMS readings.

The testing firm conducted mercury sorbent injection at preplanned rates of 0 lb/hr (0 kg/hr) baseline, 200 lb/hr (90.7 kg/hr), and 400 lb/hr (181.4 kg/hr) to thereby characterize the potential performance of the material. The corresponding average sorbent injection concentrations were 0 lb/MMacf (0 mg/acm), 2.1 lb/MMacf (33.6 mg/acm), and 5.7 lb/MMacf (91.3 mg/acm) respectively. The plant load was reduced from 100% to 30% (part load) during the latter half of the second sorbent injection rate, thus producing a lower exhaust flow and skewing the average second sorbent injection concentration to a value greater than twice that of the previous injection concentration. Data was presented at the 400 lb/hr (181.4 kg/hr) injection rate both before and after the plant dropped load, yielding two different sorbent injection concentrations.

Test Program for Concrete Properties

Because the demonstration of Mercury Sorbent HX™ was of short duration – one day – the fly ash collected in the hoppers during the test was deemed by the testing firm as not likely to be representative of that expected during extended and continuous use of the sorbent.

To assess the impact of Mercury Sorbent HX™ on the unit’s fly ash if used in concrete applications, a controlled lab test program was conducted by BASF Construction Chemicals, a leading supplier of chemical systems and formulations for customers from the construction industry. A “baseline” fly ash sample was obtained during operation of the unit exclusive of the mercury technology test program. The baseline fly ash sample was used to produce three concrete mixes: the first mix incorporated the fly ash “as is” at a 20% replacement rate for the Portland cement constituent, the second mix used the fly ash pre-mixed with Mercury Sorbent HX™ at 1%-weight of sorbent in the fly ash, and the third mix used the fly ash pre-mixed with Norit Darco® Hg-LH powdered activated carbon mercury sorbent also at 1%-weight of sorbent in the fly ash, see Table 1. A 1%-weight of mercury sorbent in the fly ash was estimated to be consistent with a sorbent injection concentration of about 3.5 lb/MMacf (56.1 mg/acm). Since the Darco® Hg-LH sorbent is recognized by its manufacturer as not being “concrete compatible”, its inclusion in the test program was to serve as a known reference.
The air entraining admixture used in the test program was BASF MB AE 90, which is recommended by the manufacturer for use in concrete exposed to cyclic freezing and thawing. The air entraining admixture was dosed to achieve an initial air content of 5% - 7% in the concrete, while water was adjusted to achieve an initial slump of 4 – 6 inches (102 – 152 mm). Consistent with expectations, the Norit Darco® Hg-LH product required significantly more air entraining admixture in its concrete mix relative to the other samples.

The concrete samples were evaluated for fresh concrete properties—slump, concrete air, and unit weight—upon their initial mix and after 60 minutes of slow agitation in the lab mixer. Specimens for compressive strength testing were cast at the end of the mixing cycle and evaluated for compressive strengths after 7, 28, and 90 days of curing in 100% humidity.

The technical literature\textsuperscript{4,5} has shown that the predominant impact of powdered activated carbon mercury sorbents on concrete is the absorption of air entraining admixtures used to entrain air in concrete and thereby improve its freeze-thaw durability. This absorption is not limited to the initial exposure of the mercury sorbent to the concrete, but rather may continue to occur during prolonged mixing of the concrete, such as when a delivery truck traverses the distance from the ready-mix concrete supplier to the intended job site. To characterize the concretes and predict their freeze-thaw durability, BASF Construction Chemicals withdrew 2 samples from each of the concrete mixes, one upon their initial mix at 20 rpm in a lab mixer and one after an additional 60 minutes of mixing at a slow agitation rate (3 rpm). The alteration of the air void system in the concrete from the initial structure through the end of the mixing cycle was determined using petrographic analysis per ASTM C 457.

<table>
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<tr>
<th>Parameter</th>
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<th>Sample 3</th>
<th>Sample 4</th>
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<td></td>
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<td>451 (267.6)</td>
<td>455 (269.9)</td>
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<td>Class C</td>
<td>Class C</td>
</tr>
<tr>
<td>Mercury Sorbent Mixed into Ash</td>
<td>-</td>
<td>-</td>
<td>BASF Mercury Sorbent HX(\textsuperscript{TM}) Norit Darco® Hg-LH</td>
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<td>Sorbent Content in Ash</td>
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<td>1%</td>
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<tr>
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<td>113 (67.0)</td>
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<td>1,357 (805.1)</td>
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<td>1,755 (1,041.2)</td>
<td>1,771 (1,050.7)</td>
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<td>Water</td>
<td>lb/yd(^3) (kg/m(^3))</td>
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<td>254 (150.7)</td>
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<td>Admixture Dose</td>
<td>oz/cwt (mL/100kg)</td>
<td>1.60 (102)</td>
<td>2.20 (141)</td>
<td>4.50 (288)</td>
</tr>
</tbody>
</table>

The air entraining admixture used in the test program was BASF MB AE 90, which is recommended by the manufacturer for use in concrete exposed to cyclic freezing and thawing. The air entraining admixture was dosed to achieve an initial air content of 5% - 7% in the concrete, while water was adjusted to achieve an initial slump of 4 – 6 inches (102 – 152 mm). Consistent with expectations, the Norit Darco® Hg-LH product required significantly more air entraining admixture in its concrete mix relative to the other samples.
RESULTS & DISCUSSION

Mercury Capture Performance

The mercury capture performance of Mercury Sorbent HX™ is summarized in Figure 2.

As shown in Figure 2, several operating conditions were discerned in the data. Baseline operation, with no sorbent injection, occurred for about the first 3 hours of the test program. Mercury Sorbent HX™ was injected at 200 lb/hr (90.7 kg/hr), a 2.1 lb/MMacf (33.6 mg/acm) concentration, for the next two hours. The sorbent injection rate was increased to 400 lb/hr (181.4 kg/hr) during the last stage of the trial. This rate yielded a sorbent injection concentration of 4.2 lb/MMacf (67.3 mg/acm) during full load operation but an average sorbent injection concentration of 7.6 lb/MMacf (121.7 mg/acm) as the plant reduced and achieved a part load condition.

The test of Mercury Sorbent HX™ was appended onto an ongoing test program evaluating dedicated mercury control technologies. The baseline data set reflects this with a slightly upward recovery trend in the “test side” Hg CEMS signal towards the “control side” Hg CEMS signal. Based on their experience and expertise, the testing firm deemed the “test side” to have recovered sufficiently to commence the injection of Mercury Sorbent HX™. Modified EPA Method 30B (sorbent trap) sampling on the “test side” and “control side” confirmed the accuracy of the CEMS during baseline testing.
Mercury Sorbent HX™ showed a dramatic and immediate reduction of vapor-phase mercury emissions upon injection at 200 lb/hr (90.7 kg/hr), a 2.1 lb/MMacf (33.6 mg/acm) sorbent concentration. As sorbent injection continued, the controlled mercury emissions trended downwards and attained an hourly averaged value of 1.4 lb/TBtu (0.602 mg/GJ). The equivalent percent reduction in hourly-averaged mercury emissions, which is defined as the percent reduction between the “control side” and “test side” signals, at this time was about 81%. It is reasonable to expect that a slightly higher injection rate or a slightly prolonged injection period at the same injection rate would have demonstrated the MATS compliance limit of 1.2 lb/TBtu (0.516 mg/GJ).

After two hours, the injection rate of Mercury Sorbent HX™ was increased to 400 lb/hr (181.4 kg/hr), a 4.2 lb/MMacf (67.3 mg/acm) sorbent injection concentration during plant full load operation. The predetermined utility test plan to which Mercury Sorbent HX™ was joined evaluated mercury sorbent performance in a step-wise parametric fashion rather than to target and hold a compliant 1.2 lb/TBtu (0.516 mg/GJ) mercury emissions rate. The unit responded to the increase in Mercury Sorbent HX™ injection and attained a controlled, hourly-average mercury emission rate significantly below the MATS mercury compliance limit of 1.2 lb/TBtu (0.516 mg/GJ), with 0.5 lb/TBtu (0.215 mg/GJ) being measured after 90 minutes. The equivalent hourly average percent reduction in mercury emissions at this time was about 91%. A Modified EPA Method 30B (sorbent trap) sampling of the “test side” confirmed the CEMS measurement of superior performance.

The plant reduced load by about 70% during the last stages of the demonstration. Although the sorbent injection rate was held constant at 400 lb/hr (181.4 kg/hr), the sorbent injection concentration increased to an average value of about 7.6 lb/MMacf (121.7 mg/acm) due to the reduced exhaust gas flow at part load. Mercury Sorbent HX™ performance responded with a controlled, hourly average mercury emission rate of about 0.25 lb/TBtu (0.108 mg/GJ), corresponding to an emission reduction of about 94%.

To facilitate a comparison, the Mercury Sorbent HX™ trial data was overlaid onto a plot published in the technical literature by ADA-ES summarizing published industry trends for brominated powdered activated carbon mercury sorbents. Data from a different, previous trial of Mercury Sorbent HX™ on a PRB coal-fired application equipped with a fabric filter (FF) also was included for reference purposes.

As shown in Figure 3, the performance of Mercury Sorbent HX™ compared favorably to the industry trends, being in the top half of the given range for brominated powdered activated carbon on PRB fired applications equipped with an ESP (black dashed line region) and within the industry trends for PRB fired applications equipped with a fabric filter (red dashed line region). It should be noted that both the ESP and fabric filter trials of Mercury Sorbent HX™ focused on meeting a pre-defined regulatory mercury emission limit on a lb/TBtu or lb/GWhr basis rather than demonstrating a maximum achievable mercury reduction for the sorbent.
Figure 3. Comparison of Mercury Sorbent HX™ with industry trends
Concrete Performance – Fresh Properties

Figure 4 presents the relative change in slump, concrete air, and unit weight after initial mixing and after 60 minutes agitation. The trends among the samples were self-consistent. The loss of concrete air after 60 minutes agitation manifested itself in an increase in density and a loss of workability (loss of slump). The Norit Darco® Hg-LH mix showed a slightly, relatively greater loss per these metrics.

![Graphs showing changes in slump, concrete air, and unit weight](image)

**Figure 4. Concrete mixes - fresh concrete properties**
Concrete Performance – Compressive Strength

As shown in Figure 5, all samples showed an expected increase in compressive strength with time. For a given sample set in time, relatively greater strengths among the samples was due to relatively lower air content in the concrete. All samples exceeded a compressive strength of 4,000 psi (27,579 kPa) at 28 days, which is a generally accepted, minimum performance requirement in the construction industry, although compressive strength specifications are site and application specific.

Figure 5. Concrete mixes – compressive strength
Concrete Performance – Petrographic Analysis

An air entraining admixture is added to concrete in order to entrain sufficient air to satisfy freeze-thaw durability requirements. As concrete cures, excess water that does not participate in the hydration reaction may remain trapped in the material or, at the immediate surface of the concrete, may evaporate and thus form a network of pores accessible to water intrusion from the ambient environment. Water expands its volume upon freezing and thus produces internal expansive stresses within the concrete that may be sufficient to promote cracking and wear. An air-void matrix that is sufficient in volume, preferentially sized towards smaller voids, and well-dispersed throughout the concrete mix relieves and mitigates these stresses. In lieu of actual freeze-thaw cycling testing per ASTM C 666, the durability of concrete made with fly ash-sorbent mixtures may be estimated by petrographic techniques such as those specified in ASTM C 457.

Each concrete mix in the test program yielded two cured samples for petrographic analysis. One sample was withdrawn from the initial mix of the concrete, cured, and submitted for analysis. Another sample was withdrawn after 60 minutes of slow agitation, cured, and submitted for analysis also. The 60 minutes interval was selected to nominally simulate the mixing experienced by concrete in transit via a concrete truck from the ready-mix cement plant dispatch to the end use job site.

As per ASTM C 457, three metrics were tracked for each concrete sample submitted for analysis. Concrete air volume measured the total volume of air voids divided by the total volume of concrete. A typically minimum acceptable value of 3% was adopted to discern satisfactory performance, as this value is the nominal criteria to distinguish between entrained and entrapped air. Entrapped air does not protect concrete from freeze/thaw distress. Specific surface area measured the total air void surface area divided by the total air void volume. A typically minimum acceptable value of 600 in\(^2\)/in\(^3\) (23.6 mm\(^2\)/mm\(^3\)) was adopted to discern satisfactory performance. Smaller air voids will have higher specific surface areas. Spacing factor measured the average distance between air voids. A typically maximum acceptable value of 0.008 inches (0.20 mm) was adopted to discern satisfactory performance. A well dispersed air void system will have a smaller spacing factor. It should be noted that specific applications, such as high performance concrete, may require specific values for concrete air, specific surface area, and spacing factor that differ from these generally accepted values.
Figure 6 shows these three metrics for each of the three concrete mixes’ two samples—initial mixing and after 60 minutes agitation—compared to the satisfactory performance threshold values, which are indicated by the horizontal line on each plot. The arrow on each plot represents the preferred direction towards more generally satisfactory values.

![Figure 6](image-url)

Figure 6. Concrete mixes – petrographic metrics

The concrete sample prepared with fly ash intermixed with Norit Darco® Hg-LH demonstrated satisfactory performance per all three petrographic metrics for the initial mix sample only. The sample after 60 minutes agitation showed significant loss of concrete air, a predominance of larger air voids (i.e. smaller specific surface area), and a greater spacing between the air voids. This concrete would not be expected to demonstrate acceptable freeze-thaw durability.

The concrete sample prepared with fly ash only—no intermixed mercury sorbent—demonstrated satisfactory performance per all three petrographic metrics for both samples—initial mix and after 60 minutes agitation. This was consistent with expectations, as the utility currently sells its fly ash into the ready-mix concrete market.

The concrete sample prepared with fly ash intermixed with Mercury Sorbent HX™ also demonstrated satisfactory performance per all three petrographic metrics for both samples—initial mix and after 60 minutes agitation. The retention of satisfactory concrete properties by fly ash intermixed with Mercury Sorbent HX™ demonstrates the minimal impact the sorbent has on the underlying concrete properties of the fly ash.
Economics of Preserved Fly Ash Salability

The value of fly ash as a cementitious material sold into the ready-mix concrete industry is determined by local construction demand. Likewise, the expense associated with landfilling fly ash is locally driven, depending upon such things as available landfill space and permitting requirements. Thus, no one analysis could adequately cover all coal-fired power plants and their economic operating scenarios. However, a hypothetical example illustrating the benefits of retained salable properties for fly ash may serve to illuminate its relative economic importance.

In May 2007, the U.S. Department of Energy / National Energy Technology Laboratory released an updated economic analysis of activated carbon injection for mercury emissions control. The analysis identified two main drivers of O&M (Operations and Maintenance) costs: the cost of the mercury sorbent material consumed and, where applicable, the costs associated with lost sales revenue and incurred disposal expenses for fly ash entrained with sorbent that precluded the continued sale of the fly ash into applications such as ready-mix concrete. To illustrate the relative magnitude of the second driver, a hypothetical case is presented.

Presuming an intermix ratio of 1%-weight mercury sorbent into fly ash, one ton, 2,000 lb (907 kg), of fly ash would be expected to contain 20 lb (9.07 kg) (1.0%-weight) of a mercury sorbent. Assuming the economic values used in the DOE/NELT analysis of $18/ton fly ash lost sales revenue and $17/ton fly ash incurred disposal expenses, the total negative economic impact of fly ash rendered unsalable by a mercury sorbent is $35/ton of fly ash, or $1.75/lb ($3.86/kg) mercury sorbent entrained in the ash at the stated rate. In this hypothetical case, assuming a nominal halogenated powdered activated carbon market price of $1.25/lb ($2.76/kg), the loss of fly ash sales would more than double the O&M costs of mercury emissions compliance for a utility. Thus, preserved fly ash salability is a significant criterion for a utility deciding on a mercury control technology.
SUMMARY

Electric utilities operating coal-fired power generating units are challenged to control mercury emissions to meet state and federal regulatory requirements, such as MATS (Mercury and Air Toxics Standards) while preserving their economic viability, including the revenue contribution from fly ash sold into concrete applications as a partial replacement for Portland cement.

Mercury Sorbent HX™ demonstrated on a PRB coal-fired electricity generating unit its ability to achieve the MATS limit for mercury, 1.2 lb/TBtu (0.516 mg/GJ), at an expected sorbent injection concentration slightly greater than 2.1 lb/MMacf (33.6 mg/acm). Mercury Sorbent HX™ also demonstrated vapor phase mercury reduction rates that were consistent with the industry trends for brominated powdered activated carbon.

Concretes prepared using the PRB coal-fired electricity generating unit fly ash, both with and without intermixed Mercury Sorbent HX™, satisfied generally accepted industry metrics per petrographic analysis (ASTM C 457) for estimating freeze-thaw durability and thus demonstrated an example of Mercury Sorbent HX™ preserving fly ash salability.
ACKNOWLEDGEMENTS

BASF would like to acknowledge the anonymous utility that graciously extended an invitation to join their test program and demonstrate Mercury Sorbent HX™.

REFERENCES


