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Nuclear Science and Technology Division

# MEASUREMENTS OF MERCURY RELEASED FROM SOLIDIFIED/STABILIZED WASTE FORMS–FY 2002

C. H. Mattus

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Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6285
managed by
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## ACRONYMS

| | |
|---|---|
| ALTER | Accelerated Life Testing and Environmental Research Corporation |
| ATG | Allied Technology Group |
| BNL | Brookhaven National Laboratory |
| CAA | Clean Air Act |
| DOE | U.S. Department of Energy |
| DTC | dithiocarbamate |
| EPA | U.S. Environmental Protection Agency |
| IMERC | incineration of mercury waste |
| LDPE | low-density polyethylene |
| LDR | land disposal restrictions |
| MLLW | mixed low level waste |
| MWFA | Mixed Waste Focus Area |
| NFS | Nuclear Fuel Services |
| ORNL | Oak Ridge National Laboratory |
| ppm | parts per million |
| RCRA | Resource Conservation and Recovery Act |
| RMERC | retorting or roasting mercury waste |
| SPSS | sulfur polymer stabilization/solidification |
| TCLP | Toxicity Characteristic Leaching Procedure |
| TLV | threshold limiting value |

## EXECUTIVE SUMMARY

This report covers work performed during FY 2002 in support of treatment demonstrations conducted for the U.S. Department of Energy (DOE) Mixed Waste Focus Area (MWFA) Mercury Working Group. To comply with the requirements of the Resource Conservation and Recovery Act, as implemented by the U.S. Environmental Protection Agency (EPA), DOE must use one of the following procedures for mixed low-level radioactive wastes containing mercury at levels above 260 ppm: a retorting/roasting treatment or (if the wastes also contain organics) an incineration treatment. The recovered radioactively contaminated mercury must then be treated by an amalgamation process prior to disposal. The DOE MWFA Mercury Working Group is working with EPA to determine whether some alternative processes could be used to treat these types of waste directly, thereby avoiding a costly recovery step for DOE.

In previous years, demonstrations were performed in which commercial vendors applied their technologies for the treatment of radiologically contaminated elemental mercury as well as radiologically contaminated and mercury-contaminated waste soils from Brookhaven National Laboratory. The test results for mercury release in the headspace were reported in two reports, *Measurements of Mercury Released from Amalgams and Sulfide Compounds* (ORNL/TM-13728) and *Measurements of Mercury Released from Solidified/Stabilized Waste Forms* (ORNL/TM-2001/17). The current work did not use a real waste; a surrogate sludge had been prepared and used in the testing in an effort to understand the consequences of mercury speciation on mercury release.

Two vendors applied their processes to treat elemental mercury, and samples were tested for the release of mercury in the headspace. Both processes significantly improved the release of mercury at 20EC, one (sulfur polymer cement) by a factor of about 10 and the other (macroencapsulation) by a factor of about 20. At 60EC, the same factor of 10 for the sulfur polymer cement was found, but the macroencapsulation behaved worse than at lower temperature, and the reduction factor when compared with that of untreated elemental mercury was close to 2 only.

The release of mercury above the headspace of the untreated and treated MER04 surrogate sludge was studied as a function of temperature. Two temperatures were selected: room temperature (~20EC) and 60EC. Measurements were performed at three time intervals — 1, 3, and 7 days — to ensure that equilibrium between the solid and gas phases was reached. The concentration of mercury in the headspace was found to be almost null in both the raw and treated sludge., Because no analysis to measure the total concentration of mercury in the sludge tested was performed it is suspected that heterogeneity of the mercury compounds within the sludge at the time of sampling may be causing these results. Therefore, no conclusion can be drawn regarding ability of the various processes to treat specific species of mercury.

# 1. BACKGROUND

Significant quantities of radioactive mercury waste [mixed low-level waste (MLLW)] are currently stored at the U.S. Department of Energy (DOE) facilities. Disposal of MLLW must meet the U.S. Environmental Protection Agency (EPA) Land Disposal Restrictions (LDR) under the Resource Conservation and Recovery Act (RCRA), as set forth in 40 CFR 268.40. Amalgamation is the standard treatment used to meet the EPA LDRs.

For radioactively contaminated wastes containing mercury at levels above 260 ppm, one of two treatment standards precedes the amalgamation step. If the waste does not contain organic constituents, retorting or roasting (RMERC) in a thermal processing unit is the treatment standard to follow; if the waste contains organics also, then incineration (IMERC) is the approved treatment standard. The recovered radioactive mercury then undergoes an amalgamation step before final disposal can occur. In an effort to reduce the costs associated with this two-step treatment, the DOE Mixed Waste Focus Area (MFWA) and Mercury Working Group are working together with the EPA to determine whether some form of direct treatment would meet the goal of the EPA Toxicity Characteristic Leaching Procedure (TCLP) extract concentration limit of 0.025 mg/L mercury (maximum) while reducing the cost for final disposal of these wastes.

This effort has been an ongoing project for several years. Various aspects of the complex chemistry of mercury have been explored within the following demonstration campaigns.

- MER01 was a demonstration of the amalgamation processes of two different vendors for the treatment of radiologically contaminated elemental mercury wastes. The measurement of mercury in the headspace of the samples was performed, and the results are found in ORNL/TM-13728 (Mattus 1999). Other reports cover this demonstration (MWFA 1999a, 1999b)

- MER02 was a demonstration of the stabilization process for treatment of radiologically contaminated wastes in which the mercury concentration was < 260 ppm. Two vendors treated an ion exchange process stream and an other one a sludge and laboratory residues. Other reports cover this demonstration (MWFA 1999c, 1999d, 1999e; ATG 1998)

- MER03 was a demonstration of stabilization processes for the treatment of radiologically contaminated mercury waste. The waste was a soil from Brookhaven National Laboratory (BNL) that was contaminated with mercury at levels around 4500 mg/kg and that contained $^{241}$Am or $^{152}$Eu. Two reports were issued from this campaign characterizing the treatment processes and the product they generated. The first one, *Use of a New Leaching Test Framework for Evaluating Alternative Processes for Mercury Contaminated Mixed Waste*, issued by Vanderbilt University (Sanchez et al. 2001, 2002 a, 2002b), covers the leaching performances of the treated wastes. The second one, *Measurements of Mercury Released from Solidified/Stabilized Waste Forms* (Mattus 2001) reports the results of the measurement of mercury in the headspace of treated and nontreated samples. Other reports cover this demonstration (ATG 2000).

- MER04 is the current effort, a study for the treatment of a mercury-contaminated surrogate sludge. Some reports were issued to document this demonstration. (NFS 2001, ATG 2001, Adams 2001, SAIC 2002, Morris et al. 2002).

A summary of the participants for each campaign as well as the processes they used is provided in Table 1.

**Table 1. Summary table for DOE–sponsored MER demonstrations**

| Vendor | MER01 | MER02 | MER03 | MER04 | Elemental Hg | Treatment process |
|---|---|---|---|---|---|---|
| ADA Technologies | X | | | X | | Sulfur process to produce a stable mercury sulfide product then addition of binders and last addition of coating agents to form a waste form |
| Allied Technology Group (ATG) | | X | X | X | | Chemical stabilization process: Dithiocarbamate (DTC) formulation (MER02); DTC and liquid sulfide reagents (MER03); sodium hydrogen sulfide (NaSH) formulation (MER04) |
| Brookhaven National Laboratory (BNL) | | | X | X | X | Sulfur polymer stabilization/solidification (SPSS) |
| GTS Duratek | | X | | | | Cement-based grout |
| Nuclear Fuel Services (NFS) | X | X | X | X | X | Proprietary DeHg™ process; different equipment in MER01 demonstration |
| SepraDyne-Raduce | | | X | | | High-vacuum rotary kiln thermal desorption |

Concerns have arisen about the release of mercury vapors from amalgamated or stabilized wastes. Much work has been done to stabilize/amalgamate the mercury, and success was declared when the leaching results were found to be satisfactory. However, no measurement of the headspace of the waste form was performed, and the possibility for volatilization of the mercury was overlooked. In the work performed in FY 1999 for the Mercury Working Group, the author of this report measured significant amounts of mercury vapor released by some amalgams prepared by commercial vendors (Mattus 1999). Hamilton and Bowers (1997) have studied the release of mercury vapors from waste solidified/stabilized in a Portland cement matrix. Their findings corroborate the author's: the

concentration of mercury in the vapors increased with temperature and time when oxide or elemental mercury species were solidified in Portland cement. The mercury was released quickly, and the headspace above the samples became saturated within a few hours. However, when mercury was stabilized with sulfide, no release of mercury was measured.

## 2. INTRODUCTION

One of the primary performance requirements specified in the MWFA technology development requirements document, *Mercury Amalgamation* (MWFA 1997), is related to vapor emissions: "The process must not release mercury vapors into the environment above the limits established by the applicable air permit [in accordance with Clean Air Act (CAA) requirements]. In addition, the process should not expose operators to mercury vapors above the established Threshold Limiting Value (TLV) of 0.05 mg/m$^3$. .. Using the TLV as a basis, the final waste form must have a vapor pressure of less than $10^{16}$ torr at 140EF."

"Vapor pressure" is defined as the pressure at which a liquid or solid is in equilibrium with its vapor at a given temperature (Considine and Considine 1984). This property depends only upon the temperature and the composition of the material being considered. For a typical liquid, a constant and reproducible vapor pressure exists and varies only with the temperature (i.e., it increases as the temperature rises).

The modified test procedure used in this study was very similar to the static headspace analysis method used by Kriger and Turner (1994). In this technique, the mercury vapor pressure was allowed to reach equilibrium in a static headspace. A commercial mercury vapor analyzer was then used to measure the mercury concentration (mass/volume) in the headspace. This instrument was used successfully in the work performed during previous years (Mattus 1999, 2001) and was also used by other scientists for similar work (Hamilton and Bowers 1997, Kriger and Turner 1994).

## 3. EQUIPMENT DESIGN — MERCURY VAPOR ANALYZER

The instrument used for measurement of the vapor pressure of mercury was a Jerome 431-X gold-film mercury vapor analyzer from Arizona Instruments (Phoenix, Ariz.). The range of detection is 0.000 to 0.999 mg/m$^3$ mercury. The sensitivity of the instrument is 0.003 mg/m$^3$, well below the threshold limiting value (TLV) of 0.05 mg/m$^3$. Air sampling is performed with the aid of an internal pump. The amount of air sampled and analyzed each time is 87.5 mL. The air flows through a guard column packed with soda lime for removing moisture and acid gases. The resulting dry vapor is deposited onto a gold film, which forms an amalgam with mercury, thus increasing the electrical resistance of the film. This instrument is stable and selective for mercury and, unlike ultraviolet analyzers, is not prone to interferences from contaminants such as water vapor or hydrocarbons. When the sensor approaches its saturation limit, the instrument provides a warning. Regeneration of

the sensor takes about 10 min, but the instrument should not be used for about 30 min after regeneration of the sensor to allow the metal to cool down to room temperature.

## 4. ELEMENTAL MERCURY

### 4.1 WASTE AND TREATMENT DESCRIPTIONS

Two vendors provided amalgamated samples for evaluation. BNL sent a monolithic sample of elemental mercury treated by its process. The waste form has a waste loading of 33 wt % of elemental mercury. NFS sent four containers of amalgamated elemental mercury. This material looked like dark grey sand; some white particulates were present in two of the four samples. Table 2 contains information pertaining to the treatment of the samples of elemental mercury treated by the two vendors.

**Table 2. Summary of technologies used for treatment of elemental mercury**

| | BNL | NFS |
|---|---|---|
| Process | Formation of mercury sulfide followed by thermoplastic encapsulation using sulfur polymer cement stabilization/ solidification process | Amalgamation and stabilization process with precipitation of stable salt. |
| Waste loading (dry basis, wt %) | 33 | 20.1 |
| Final form of treated waste | Monolithic | Soil-like |

### 4.2 EXPERIMENTAL PROCEDURE

The objective of this set of experiments was to study the effect of temperature on the mercury vapor released from the various waste forms and untreated sludges. Measurements were made at two temperatures, ambient (~20–22EC) and 60EC. These results were then compared with those for pure elemental mercury.

The literature provides the mercury vapor pressure above pure mercury as a function of temperature. The expected gas space concentration of mercury at each temperature can be calculated from the mercury partial pressure by using the ideal gas law [see Eq. (1)].

$$\frac{w}{v} = \frac{pM}{RT} \qquad (1)$$

where

$p$ = vapor pressure of the sample (Pa),
$w$ = mass of vaporized material (g),

$M$ = molecular weight of mercury ($g \cdot mol^{-1}$),
$R$ = gas constant (8.31 Pa @ $m^3$ @ $mol^{-1}$ @ $K^{-1}$),
$T$ = temperature (K),
$v$ = volume analyzed ($m^3$).

The sample preparation was done in glass sample vials with Teflon septa. Samples held at 60EC were placed into a water bath to avoid temperature fluctuation during sampling. The temperature of the water was read from a digital readout placed on the equipment.

Approximately 15 mL of treated material, equivalent to a tablespoonful, was introduced into each vial. The release of mercury in the headspace is independent of the amount of sample present. All the samples were prepared in triplicate. For each temperature, as a quality assurance/quality control check, a blank (empty vial) and a sample containing pure mercury metal were also run in triplicate. The samples were measured at selected time intervals.

Volumes of 0.2 to 10 mL of the headspace sample diluted to a total of 87.5 mL by room air were found to be appropriate for use in this set of experiments. The total concentration had to be recalculated to take into account the dilution made during sampling. Even though equilibrium between the sample and the air above is reached rapidly (Hamilton and Bowers 1997), measurements were made at 1, 3, and 7 days to confirm that the data obtained were representative of an equilibrium condition. Each vial was sampled and analyzed three times, and then the results were averaged. The standard deviation on the nine measurements made for each sample was used for calculating the error on the average concentration. The plots in Figs. 1 and 2 represent the domain of error associated with the results.

### 4.3 MEASUREMENTS OF MERCURY RELEASED AT 20EC

The samples were maintained at room temperature (~20EC) for this set of experiments. The data obtained at 1, 3, and 7 days are summarized in Tables A-1 and illustrated in Fig. 1.

### 4.4 MEASUREMENTS OF MERCURY RELEASED AT 60EC

The data showed more fluctuation for this series of tests. Opening the water bath probably caused the temperature of the samples to drop; furthermore, the smaller volume of air sampled introduced a larger error in the measurement of mercury concentration. The data obtained are compiled in Table A-2 and are plotted in Fig. 2.

### 4.5 DISCUSSION OF RESULTS

#### 4.5.1 QA/QC Samples

At ambient temperature, which was measured by thermocouples to be ~20EC, the measurements were found to be in the range of 15.5 to 18.5 $mg/m^3$ for the pure mercury samples. The theoretical

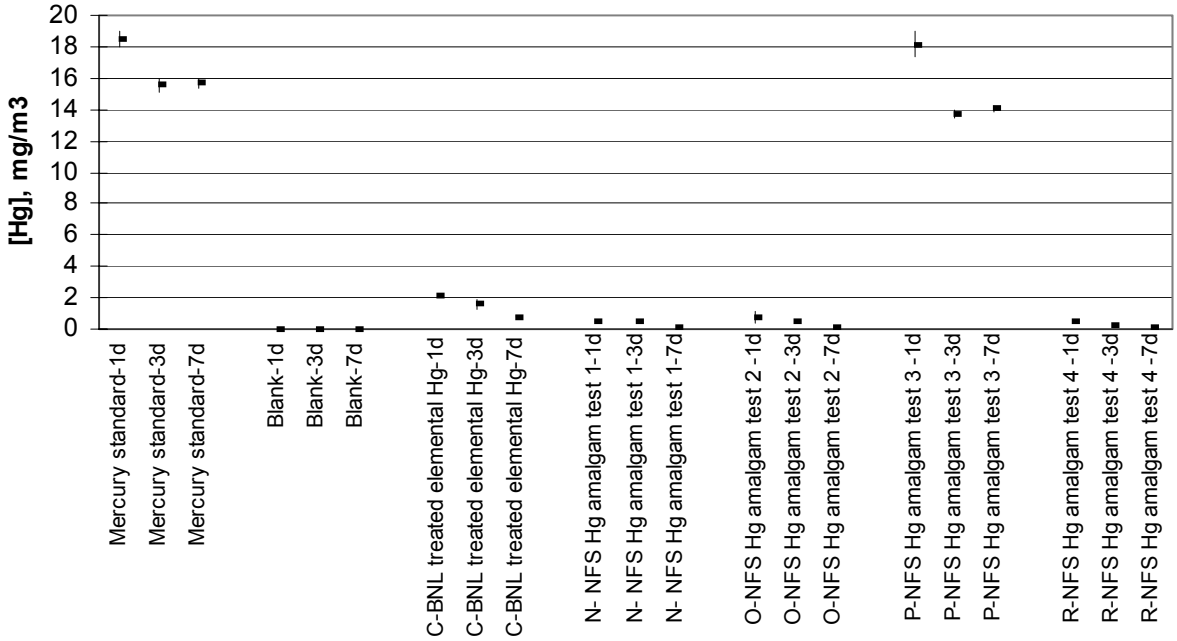


**Fig. 1 Mercury concentration in headspace of treated elemental mercury samples maintained at 20EC.**

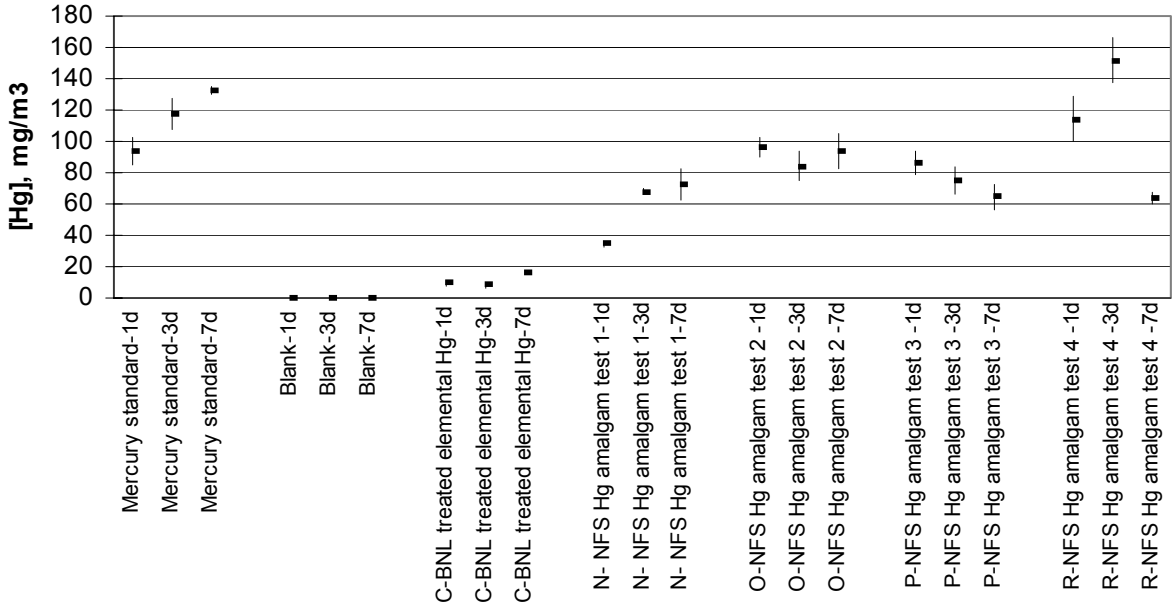


**Fig. 2 Mercury concentration in headspace of treated elemental mercury maintained at 60EC.**

values are 15.54 and 19.91 at 22 and 25EC, respectively. At 60EC, the mercury concentration was found to be between 94 at day 1 and 132 mg/m$^3$ at day 7. The theoretical value is 240 mg/m$^3$ for that temperature. This difference was probably due to the smaller volume of headspace gas analyzed; only 0.2 mL was used so that the sensor would not become saturated too rapidly. It was also noticed that the temperature of the bath dropped when the cover was removed during the measurements. Only one of the replicates for the blank at 60EC was found to be slightly above zero, showing that the instrument was responding correctly for the low end of the concentration range.

### 4.5.2 Elemental Mercury Treated Samples

The waste form made by BNL released little mercury in the headspace at either temperature: below 2 mg/m$^3$ at 20EC and below 20 mg/m$^3$ at 60EC.

NFS provided four containers of treated amalgamated mercury, and all four were tested. At 20EC, three of the four samples had concentrations of mercury in the headspace below 1 mg/m$^3$, but one test had results close to that of the pure mercury standard. Heterogeneity may be the reason for this abnormally high result if there was some unreacted mercury present in the treated material. The results obtained at 60EC were higher than those obtained from the BNL samples; results varied from 35 to 150 mg/m$^3$. The four samples from NFS at 60EC did not have outliers as seen at 20EC.

Both processes decrease the concentration of mercury in the headspace, but none is apparently able to treat the mercury so that no vapor pressure is present. This finding may be explained by an incomplete reaction that leaves a minute amount of unreacted material that is enough to liberate mercury into the headspace.

## 5. MER04 SURROGATE SLUDGE

### 5.1 WASTE AND TREATMENT DESCRIPTION

The MER04 sludge surrogate was designed by ALTER (The Accelerated Life Testing and Environmental Research Corporation, Dillsboro, Ind.) and contained some “difficult to treat” species of mercury with an overall mercury concentration of 0.5 percent. The sludge composition is found in Table 3. It must be stated that this composition was the prepared one, and that the samples that were sent for mercury vapor measurement at ORNL were not tested for total mercury content, thus not verified to actually contain this amount of mercury.

The different vendors involved in the demonstration sent samples of the raw MER04 sludge as well as the treated samples. BNL raw sludge is a mixture of a solid phase (~2/3 of the total sludge) that has a clear brown color and a dark brown greenish liquid phase. The treated samples from BNL were dark grey cylinder monoliths 2 × 2 in. in diameter and height.

**Table 3. Characterization of the MER04 surrogate sludge**

| Sludge constituent | Weight percent | Mercury concentration, mg/kg |
|---|---|---|
| Phenyl mercury acetate | 0.08 | 500 |
| Mercury nitrate | 0.17 | 1000 |
| Elemental mercury | 0.15 | 1500 |
| Mercury oxide | 0.11 | 1000 |
| Mercury chloride | 0.14 | 1000 |
| Diatomaceous earth | 20 | 0 |
| Aluminum hydroxide | 10 | 0 |
| Ferric chloride | 10 | 0 |
| Sodium chloride | 10 | 0 |
| Water | 49.35 | 0 |
| Total | 100 | 5000 |

Allied Technology Group, Hayward, Calif. (ATG), sent two bottles of raw MER04 sludge corresponding to two different batches ("1" and "2"). They were similar to the sludge from BNL but contained less liquid phase (~1/4 or 1/5 of the total sludge volume). Four samples of the treated sludge were received for testing at ORNL. These samples were monoliths cast in 500-mL plastic bottles. The monoliths were hard to break and were grey on the outside. However, a fresh fracture revealed the black color of the inside of the material.

Nuclear Fuel Services, Erwin, Tenn. (NFS) sent two bottles of raw MER04 sludge. This sludge was slightly different from the two others. It had a darker brown color and very little or no liquid phase. The treated sludge looked like a clay-like soil with a greenish brown color. The stabilized samples did not have mechanical strength. A second treated sample was received, which was drier than the first one and looked like agglomeration of soil in pellets. A summary of the different treatments is presented in Table 4.

**Table 4. Summary of technologies used for treatment of the MER04 surrogate sludge**

| | BNL | NFS | ATG |
|---|---|---|---|
| Process | Formation of mercury sulfide followed by thermoplastic encapsulation using sulfur polymer cement stabilization/ solidification process | DeHg® amalgamation and stabilization process of elemental Hg followed by precipitation of stable salt | Formation of mercuric sulfide followed by cement-containing proprietary stabilization agents |
| Waste loading (dry basis) (wt %) | 30 | batch 1: 44.9<br>Batch 2: 47.0 | 25.4 |
| Volume or weight increase | 233 wt % on dry basis<br>36 vol % | batch 1: 123 wt %<br>batch 2: 113 wt % | 25 vol. %<br>294 wt % |
| Final form of treated waste | Monolithic | Soil-like | Monolithic |

## 5.2 EXPERIMENTAL PROCEDURE

The sample preparation was done in 160-mL low density polyethylene (LPDE) sample vials for the samples maintained at room temperature and in glass sample vials with Teflon septa for the samples maintained at 60EC. Samples held at 60EC were placed into a water bath to avoid temperature fluctuation during sampling. The temperature of the water was read from a digital readout placed on the equipment.

Approximately 15 mL of material, equivalent to a tablespoonful, was introduced into each vial. The raw sludges were too liquid to be measured effectively. A layer of water covering the solid phase prevents the release of mercury in the headspace. Therefore, a sample preparation step was performed by drying the samples in an oven at 66EC to remove the excess water. No analysis for total mercury was performed on the sludge prior and after the drying stage, therefore, it is not possible to evaluate the amount of mercury lost during the two days of drying. The treated samples did not present that problem, so they did not undergo such a treatment step. All the samples were prepared in triplicate.

Volumes of 0.2 to 10 mL of the headspace sample diluted to a total of 87.5 mL by room air were found to be appropriate for use in this set of experiments. Measurements were made at 1, 3, and 7 days.

Each vial was sampled and analyzed three times, and then the results were averaged. The standard deviation on the nine measurements made for each sample was used for calculating the error on the average concentration. The plots in Figs. 3 through 8 represent the domain of error associated with the results. The results obtained for the pure mercury standard are not presented on these plots because of a scaling problem that would not differentiate the results obtained for the MER04 samples.

## 5.3 MEASUREMENTS OF MERCURY RELEASE AT 20EC

The samples were maintained at room temperature (~20 to 21.5EC) for this set of experiments. The data obtained at 1, 3, and 7 days are summarized in Tables A-1 to A-4, presented in Appendix A. Figures 3 through 5 plot the average values obtained for the MER04 sludge.

## 5.4 MEASUREMENTS OF MERCURY RELEASE AT 60EC

The data showed more fluctuation for this series of tests. Opening the water bath probably caused the temperature of the samples to drop; furthermore, the smaller volume of air sampled introduced a larger error in the measurement of mercury concentration. The data obtained are compiled in Tables A-5 through A-8 presented in Appendix A and are illustrated in Figs. 6 through 8.

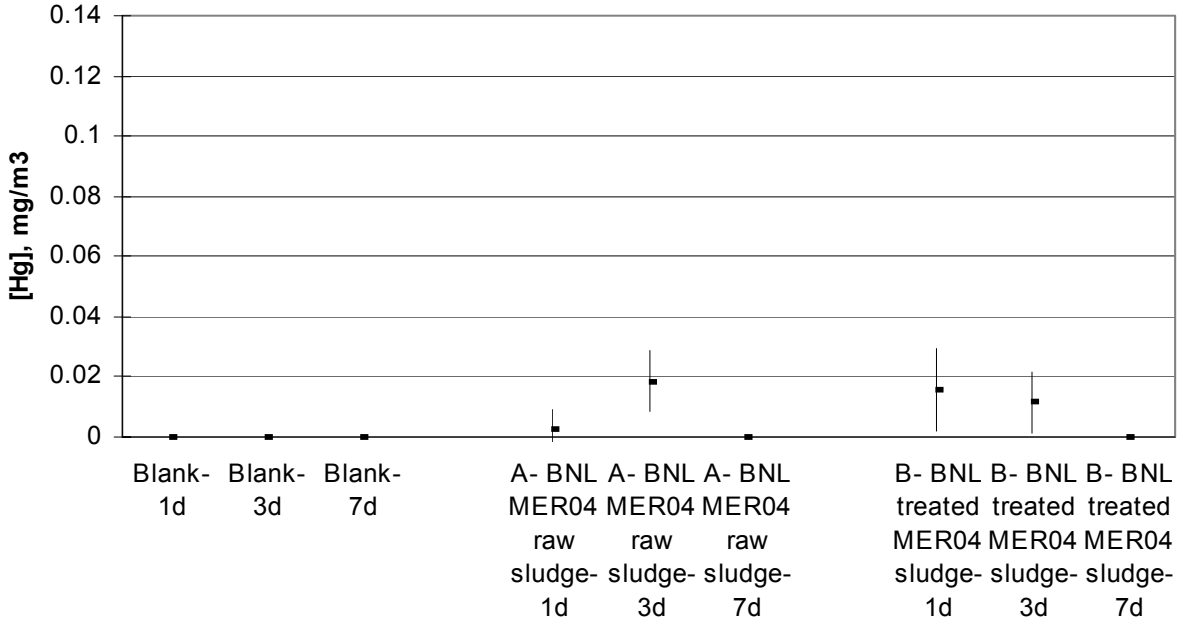


**Fig. 3 Mercury concentration in headspace of MER04 samples from BNL maintained at 20EC.**

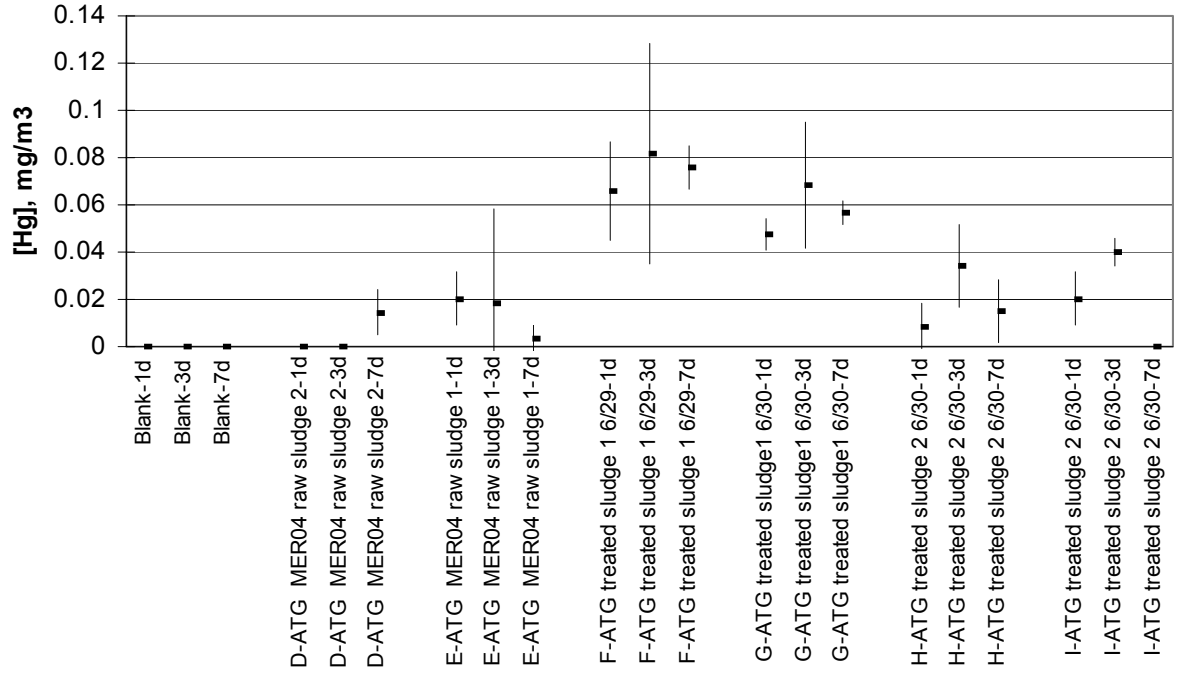


**Fig. 4 Mercury concentration in headspace of MRE04 samples from ATG maintained at 20EC.**

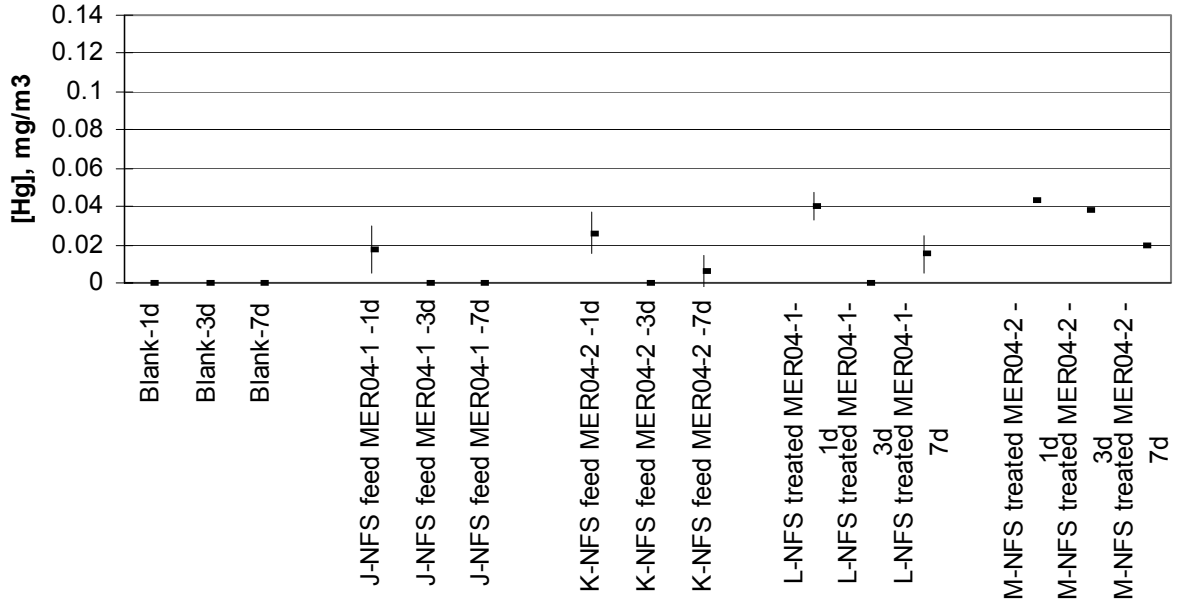


**Fig. 5 Mercury concentration in headspace of MER04 samples from NFS maintained at 20EC.**

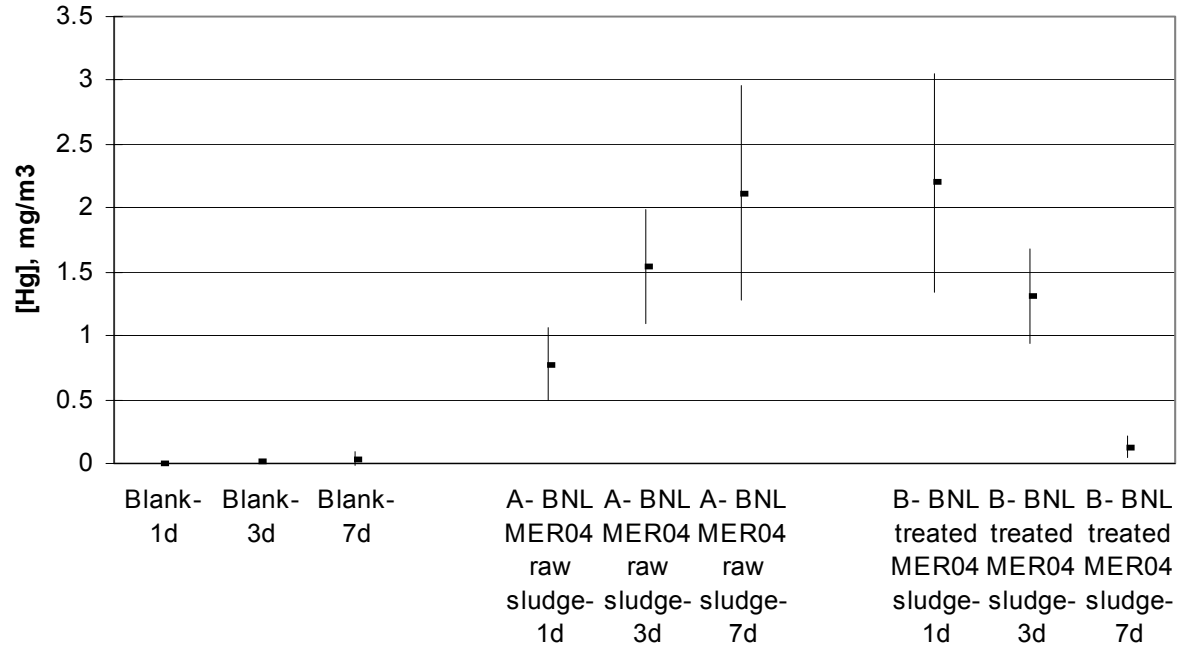


**Fig. 6 Mercury concentration in headspace of MER04 samples from BNL maintained at 60EC.**

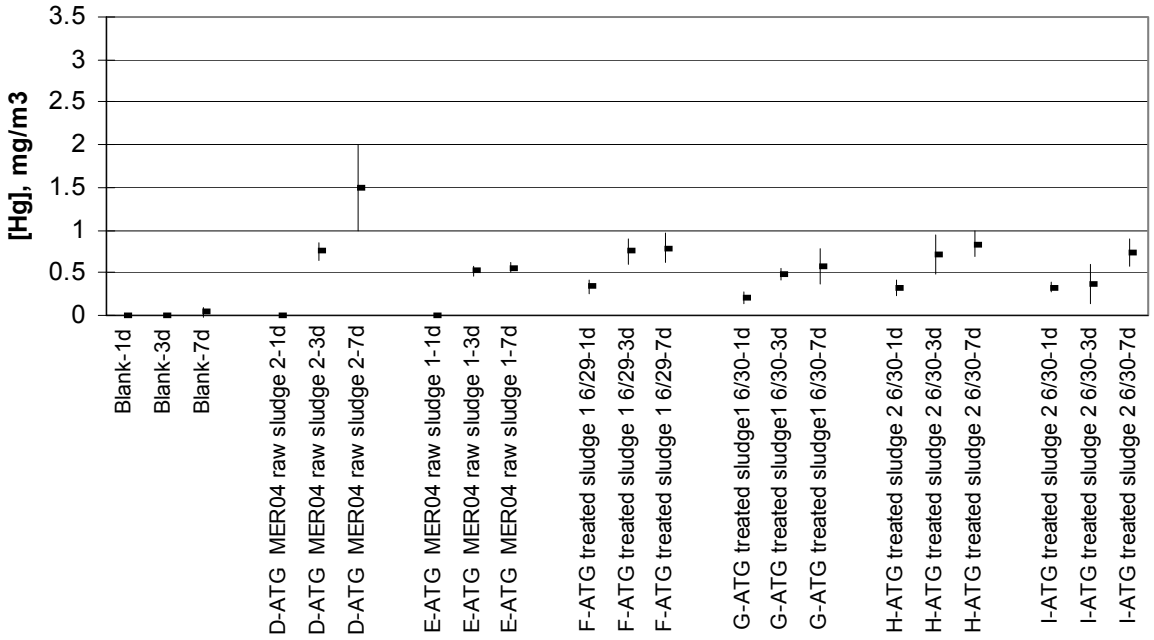


**Fig. 7 Mercury concentration in headspace of MER04 samples from ATG maintained at 60EC.**

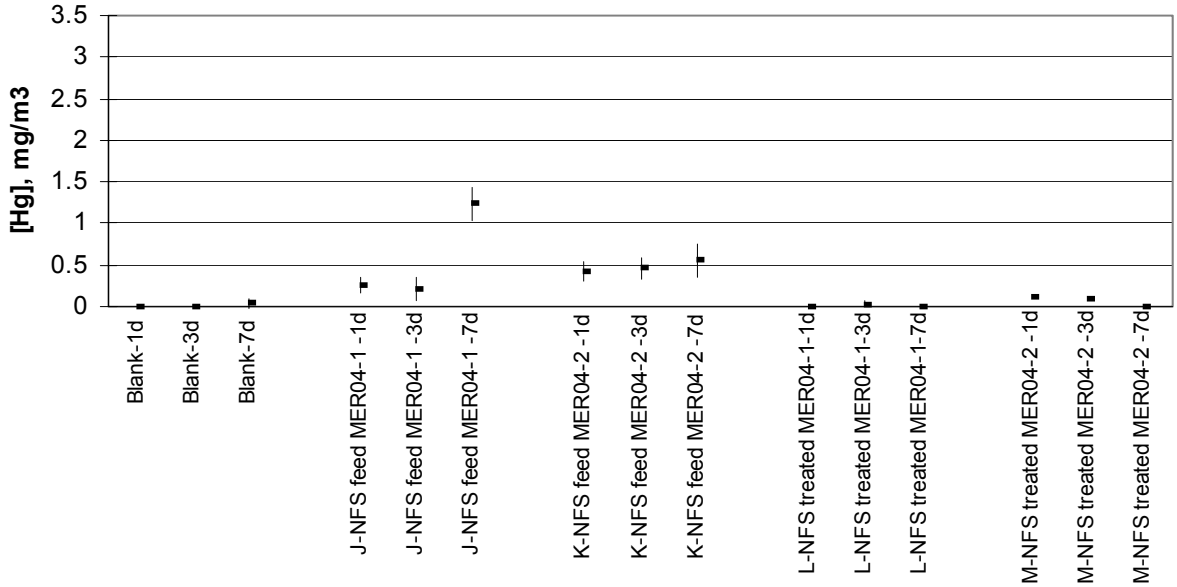


**Fig. 8 Mercury concentration in headspace of MER04 samples from NFS maintained at 60EC.**

## 5.5 DISCUSSION OF RESULTS

### 5.5.1 QA/QC Samples

At ambient temperature, which was measured by thermocouples to be ~20 to 21.5E, the measurements for the mercury standard were found to be in the range of 15.5 to 18.5 mg/m$^3$. The theoretical values are 15.54 and 19.91 at 22 and 25EC, respectively. At 60EC, the mercury concentration was found to be between 94 at day and 132 mg/m$^3$ at 7 days. The theoretical value is 240 mg/m$^3$ for that temperature. This difference was probably due to the smaller volume of headspace gas analyzed; only 0.2 mL was used so that the sensor would not become saturated too rapidly. It was also noticed that the temperature of the bath dropped when the cover was removed during the measurements. Only one of the replicates at 60EC was found to be slightly above zero, showing that the instrument was responding correctly for the low end of the concentration range.

### 5.5.2 MER04 Surrogate Sludge

Very surprisingly, the results obtained at both temperatures for the raw sludge from all vendors showed that almost no mercury was present in the headspace of the samples, even though elemental and ionic mercury were present in the synthetic sludge. The raw sludge samples had been dried at 66EC for two days prior to testing to eliminate the excess water that would have prevented the mercury vapor from exiting the material. Again, the raw surrogate sludge was not analyzed for total mercury prior of after drying; therefore, it is not possible to evaluate the amount of mercury lost during the drying preparation stage as well as knowing how much mercury was effectively present in the surrogate.

The heterogeneity of the material could explain this result. Mercuric nitrate is very soluble in water and the solubility of mercuric chloride is 6.9 g/100cc, so these two species should dissolve during the sludge preparation and be homogeneously dispersed. However, elemental mercury is heavy and insoluble, and mercuric oxide as HgO is not very soluble; its solubility is only 0.0053g/100 cc. These two species will not dissolve in the sludge and will have a tendency to settle to the bottom of the container in which the sludge is prepared. Even with mixing of the tank of sludge, when the subsampling is taking place, it is difficult to aliquot a representative sample containing the elemental and oxide mercury forms. When a subsample was taken for testing, it may not have contained all the mercury species introduced during the preparation of the sludge. That problem may have biased the results obtained. It would have been valuable to control the amount of mercury present in the raw sludges prepared by each vendor when they took aliquots for testing of the mercury vapor.

The results obtained for the treated MER04 samples are not significantly different from those obtained for the raw sludge at either temperature.

## 6. CONCLUSIONS

Two vendors treated elemental mercury, and the measurements of the mercury in the headspace of the treated waste form was improved at 20EC. For each process a reduction factor of mercury concentration of about 10 or 20 was found. However, at 60EC, the sulfur polymer cement waste form showed a better reduction factor (~10) than the macroencapsulation process from NFS, which exhibited a reduction factor of only ~2. It is noteworthy that even though these processes improved the release of mercury in the headspace of the waste form, that concentration was still higher than the TLV value of 0.05 $mg/m^3$.

The testing of various species of mercury was not conclusive for the release of mercury vapor in the headspace of the samples. Neither untreated nor treated synthetic MER04 sludge showed the presence of mercury. It is suspected that for the raw sludge, the heterogeneity of the salts repartitioned in the sludge during the sampling and that the repartitioning was the cause of these results. Elemental mercury and mercury oxide are not soluble in water; therefore these species are difficult to mix homogeneously in a large batch, and a subsample is likely to miss them.

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# APPENDIX A

**Table A-1. Summary data for the MER04 and amalgamated elemental mercury samples from BNL maintained at 20EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] mg/m$^3$ | 3 days–01/10/02 | | | [Hg] mg/m$^3$ | 7 days–01/14/02 | | | [Hg] mg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Blank–Blk1 | 1d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| Blank–Blk 2 | 3d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| Blank–Blk3 | 7d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| Average | | | | | **0.00** | | | | **0.00** | | | | **0.00** |
| Error | | | | | **0.00** | | | | **0.00** | | | | **0.00** |
| Mercury standard–Hg1 | 1d : 1 | 0.228 | 0.218 | 0.212 | 19.192 | 0.187 | 0.186 | 0.18 | 16.129 | 0.178 | 0.18 | 0.186 | 15.867 |
| Mercury standard–Hg2 | 3d : 1 | 0.208 | 0.21 | 0.206 | 18.200 | 0.173 | 0.179 | 0.174 | 15.342 | 0.182 | 0.181 | 0.167 | 15.458 |
| Mercury standard–Hg3 | 7d : 1 | 0.212 | 0.204 | 0.202 | 18.025 | 0.177 | 0.174 | 0.168 | 15.138 | 0.181 | 0.181 | 0.178 | 15.750 |
| Average | | | | | **18.5** | | | | **15.5** | | | | **15.7** |
| Error | | | | | **0.5** | | | | **0.4** | | | | **0.4** |
| A1–MER04 raw sludge from BNL | 1d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0.003 | 0.003 | 0.018 | 0 | 0 | 0 | 0.000 |
| A2–MER04 raw sludge from BNL | 3d : 10 | 0 | 0 | 0.003 | 0.009 | 0 | 0.003 | 0 | 0.009 | 0 | 0 | 0 | 0.000 |
| A3–MER04 raw sludge from BNL | 7d : 10 | 0 | 0 | 0 | 0.000 | 0.003 | 0.003 | 0.004 | 0.029 | 0 | 0 | 0 | 0.000 |
| Average | | | | | **0.00** | | | | **0.02** | | | | **0.00** |
| Error | | | | | **0.01** | | | | **0.01** | | | | **0.00** |
| B1–treated MER04 sludge from BNL | 1d : 10 | 0.004 | 0 | 0.003 | 0.020 | 0.003 | 0 | 0.003 | 0.018 | 0 | 0 | 0 | 0.000 |
| B2–treated MER04 sludge from BNL | 3d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0.003 | 0.009 | 0 | 0 | 0 | 0.000 |
| B3–treated MER04 sludge from BNL | 7d : 10 | 0 | 0.004 | 0.005 | 0.026 | 0 | 0 | 0.003 | 0.009 | 0 | 0 | 0 | 0.000 |
| Average | | | | | **0.02** | | | | **0.01** | | | | **0.00** |
| Error | | | | | **0.01** | | | | **0.01** | | | | **0.00** |
| C1–treated elemental Hg from BNL | 1d : 10 | 0.263 | 0.265 | 0.253 | 2.278 | 0.175 | 0.175 | 0.168 | 1.511 | 0.096 | 0.11 | 0.107 | 0.913 |
| C2–treated elemental Hg from BNL | 3d : 10 | 0.21 | 0.216 | 0.204 | 1.838 | 0.138 | 0.152 | 0.147 | 1.275 | 0.076 | 0.082 | 0.074 | 0.677 |
| C3–treated elemental Hg from BNL | 7d : 10 | 0.258 | 0.262 | 0.249 | 2.243 | 0.181 | 0.255 | 0.273 | 2.068 | 0.08 | 0.097 | 0.083 | 0.758 |
| Average | | | | | **2.1** | | | | **1.6** | | | | **0.78** |
| Error | | | | | **0.2** | | | | **0.3** | | | | **0.09** |

**Table A-2. Summary data for the amalgamated elemental mercury from NFS maintained at 20EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] mg/m$^3$ | 3 days–01/10/02 | | | [Hg] mg/m$^3$ | 7 days–01/14/02 | | | [Hg] mg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N1–NFS Hg amalgam test 1 | 1d : 10 | 0.063 | 0.063 | 0.061 | 0.545 | 0.063 | 0.065 | 0.059 | 0.545 | 0.019 | 0.018 | 0.018 | 0.160 |
| N2–NFS Hg amalgam test 1 | 3d : 10 | 0.057 | 0.057 | 0.057 | 0.499 | 0.049 | 0.047 | 0.044 | 0.408 | 0.015 | 0.016 | 0.016 | 0.137 |
| N3–NFS Hg amalgam test 1 | 7d : 10 | 0.07 | 0.065 | 0.06 | 0.569 | 0.053 | 0.049 | 0.048 | 0.438 | 0.017 | 0.017 | 0.016 | 0.146 |
| Average | | | | | **0.54** | | | | **0.46** | | | | **0.15** |
| Error | | | | | **0.03** | | | | **0.05** | | | | **0.01** |
| O1–NFS Hg amalgam test 2 | 1d : 10 | 0.147 | 0.161 | 0.153 | 1.345 | 0.084 | 0.074 | 0.07 | 0.665 | 0.017 | 0.017 | 0.017 | 0.149 |
| O2–NFS Hg amalgam test 2 | 3d : 10 | 0.052 | 0.052 | 0.051 | 0.452 | 0.041 | 0.039 | 0.039 | 0.347 | 0.012 | 0.011 | 0.012 | 0.102 |
| O3–NFS Hg amalgam test 2 | 7d : 10 | 0.012 | 0.125 | 0.121 | 0.436 | 0.052 | 0.05 | 0.046 | 0.432 | 0.013 | 0.015 | 0.014 | 0.123 |
| Average | | | | | **0.74** | | | | **0.48** | | | | **0.12** |
| Error | | | | | **0.39** | | | | **0.11** | | | | **0.02** |
| P1–NFS Hg amalgam test 3 | 1d : 1 | 0.202 | 0.224 | 0.219 | 18.813 | 0.152 | 0.156 | 0.158 | 13.592 | 0.161 | 0.164 | 0.159 | 14.117 |
| P2–NFS Hg amalgam test 3 | 3d : 1 | 0.195 | 0.218 | 0.211 | 18.200 | 0.157 | 0.159 | 0.148 | 13.533 | 0.163 | 0.163 | 0.154 | 14.000 |
| P3–NFS Hg amalgam test 3 | 7d : 1 | 0.186 | 0.21 | 0.202 | 17.442 | 0.16 | 0.161 | 0.161 | 14.058 | 0.16 | 0.162 | 0.158 | 14.000 |
| Average | | | | | **18.2** | | | | **13.7** | | | | **14.0** |
| Error | | | | | **0.8** | | | | **0.3** | | | | **0.2** |
| R1–NFS Hg amalgam test 4 | 1d : 10 | 0.046 | 0.048 | 0.047 | 0.411 | 0.022 | 0.022 | 0.022 | 0.193 | 0.01 | 0.013 | 0.011 | 0.099 |
| R2–NFS Hg amalgam test 4 | 3d : 10 | 0.053 | 0.05 | 0.044 | 0.429 | 0.028 | 0.027 | 0.026 | 0.236 | 0.006 | 0.013 | 0.012 | 0.090 |
| R3–NFS Hg amalgam test 4 | 7d : 10 | 0.074 | 0.068 | 0.068 | 0.613 | 0.042 | 0.039 | 0.036 | 0.341 | 0.022 | 0.018 | 0.015 | 0.160 |
| Average | | | | | **0.48** | | | | **0.26** | | | | **0.12** |
| Error | | | | | **0.07** | | | | **0.05** | | | | **0.03** |

**Table A-3. Summary data for the MER04 and amalgamated elemental mercury samples from BNL maintained at 60EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] mg/m$^3$ | 3 days–01/10/02 | | | [Hg] mg/m$^3$ | 7 days–01/14/02 | | | [Hg] mg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Blank–Blk1 | 1d : 3 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0.006 | 0.058 |
| Blank–Blk 2 | 3d : 3 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| Blank–Blk3 | 7d : 3 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0.003 | 0.029 | 0 | 0 | 0.005 | 0.049 |
| Average | | | | | **0.00** | | | | **0.01** | | | | **0.04** |
| Error | | | | | **0.00** | | | | **0.02** | | | | **0.05** |
| Mercury standard–Hg1 | 1d : 0.2 | 0.181 | 0.227 | 0.198 | 88.375 | 0.265 | 0.28 | 0.235 | 113.750 | 0.295 | 0.305 | 0.294 | 130.375 |
| Mercury standard–Hg2 | 3d : 0.2 | 0.226 | 0.258 | 0.248 | 106.750 | 0.308 | 0.307 | 0.296 | 132.854 | 0.304 | 0.315 | 0.316 | 136.354 |
| Mercury standard–Hg3 | 7d : 0.2 | 0.194 | 0.178 | 0.218 | 86.042 | 0.252 | 0.23 | 0.244 | 105.875 | 0.301 | 0.305 | 0.291 | 130.813 |
| Average | | | | | **94** | | | | **117** | | | | **132** |
| Error | | | | | **9** | | | | **0.45** | | | | **3** |
| A1–MER04 raw sludge from BNL | 1d : 3 | 0.017 | 0.017 | 0.015 | 0.476 | 0.036 | 0.042 | 0.028 | 1.031 | 0.052 | 0.045 | 0.037 | 1.303 |
| A2–MER04 raw sludge from BNL | 3d : 3 | 0.054 | 0.041 | 0.033 | 1.244 | 0.097 | 0.072 | 0.055 | 2.178 | 0.163 | 0.11 | 0.071 | 3.344 |
| A3–MER04 raw sludge from BNL | 7d : 3 | 0.026 | 0.021 | 0.016 | 0.613 | 0.059 | 0.043 | 0.042 | 1.400 | 0.069 | 0.053 | 0.055 | 1.721 |
| Average | | | | | **0.78** | | | | **1.31** | | | | **2.12** |
| Error | | | | | **0.29** | | | | **0.38** | | | | **0.85** |
| B1–treated MER04 sludge from BNL | 1d : 3 | 0.019 | 0.028 | 0.032 | 0.768 | 0.023 | 0.029 | 0.041 | 0.904 | 0 | 0 | 0 | 0.000 |
| B2–treated MER04 sludge from BNL | 3d : 1 | 0.311 | 0.079 | 0.096 | 2.382 | 0.04 | 0.034 | 0.038 | 1.089 | 0.005 | 0.006 | 0.005 | 0.156 |
| B3–treated MER04 sludge from BNL | 7d : 1 | 0.439 | 0.118 | 0.123 | 3.442 | 0.055 | 0.069 | 0.075 | 1.935 | 0.005 | 0.01 | 0.01 | 0.243 |
| Average | | | | | **9.5** | | | | **1.31** | | | | **0.13** |
| Error | | | | | **2.0** | | | | **0.38** | | | | **0.08** |
| C1–treated elemental Hg from BNL | 1d : 10 | 0.199 | 0.214 | 0.229 | 6.242 | 0.057 | 0.062 | 0.073 | 5.600 | 0.185 | 0.192 | 0.221 | 17.442 |
| C2–treated elemental Hg from BNL | 3d : 10 | 0.311 | 0.365 | 0.378 | 10.247 | 0.095 | 0.1 | 0.111 | 8.925 | 0.199 | 0.199 | 0.201 | 17.471 |
| C3–treated elemental Hg from BNL | 7d : 10 | 0.439 | 0.378 | 0.426 | 12.085 | 0.121 | 0.121 | 0.132 | 10.908 | 0.167 | 0.165 | 0.15 | 14.058 |
| Average | | | | | **9.5** | | | | **8.5** | | | | **16.3** |
| Error | | | | | **2.0** | | | | **1.7** | | | | **1.4** |

**Table A-4. Summary data for the amalgamated mercury from NFS maintained at 60EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] $mg/m^3$ | 3 days–01/10/02 | | | [Hg] $mg/m^3$ | 7 days–01/14/02 | | | [Hg] $mg/m^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N1–NFS Hg amalgam test 1 | 1d : 0.2 | 0.088 | 0.081 | 0.081 | 36.4 | 0.162 | 0.156 | 0.158 | 69.4 | 0.199 | 0.185 | 0.201 | 85.3 |
| N2–NFS Hg amalgam test 1 | 3d : 0.2 | 0.083 | 0.081 | 0.076 | 35.0 | 0.163 | 0.155 | 0.155 | 68.9 | 0.131 | 0.128 | 0.118 | 54.9 |
| N3–NFS Hg amalgam test 1 | 7d : 0.2 | 0.081 | 0.069 | 0.072 | 32.3 | 0.148 | 0.154 | 0.143 | 64.8 | 0.161 | 0.178 | 0.183 | 76.1 |
| Average | | | | | **35** | | | | **68** | | | | **72** |
| Error | | | | | **2** | | | | **2** | | | | **10** |
| O1–NFS Hg amalgam test 2 | 1d : 0.2 | 0.249 | 0.24 | 0.239 | 106.1 | 0.223 | 0.232 | 0.232 | 100.1 | 0.243 | 0.267 | 0.261 | 112.4 |
| O2–NFS Hg amalgam test 2 | 3d : 0.2 | 0.218 | 0.197 | 0.234 | 94.6 | 0.161 | 0.164 | 0.171 | 72.3 | 0.21 | 0.183 | 0.21 | 87.9 |
| O3–NFS Hg amalgam test 2 | 7d : 0.2 | 0.211 | 0.203 | 0.199 | 89.3 | 0.184 | 0.179 | 0.181 | 79.3 | 0.189 | 0.179 | 0.189 | 81.2 |
| Average | | | | | **97** | | | | **84** | | | | **94** |
| Error | | | | | **6** | | | | **9** | | | | **11** |
| P1–NFS Hg amalgam test 3 | 1d : 0.2 | 0.208 | 0.218 | 0.223 | 94.64 | 0.185 | 0.202 | 0.204 | 86.1 | 0.167 | 0.169 | 0.18 | 75.2 |
| P2–NFS Hg amalgam test 3 | 3d : 0.2 | 0.214 | 0.214 | 0.194 | 89.68 | 0.179 | 0.184 | 0.183 | 79.6 | 0.151 | 0.164 | 0.153 | 68.2 |
| P3–NFS Hg amalgam test 3 | 7d : 0.2 | 0.178 | 0.153 | 0.171 | 73.20 | 0.139 | 0.133 | 0.132 | 58.9 | 0.113 | 0.116 | 0.116 | 50.3 |
| Average | | | | | **86** | | | | **75** | | | | **65** |
| Error | | | | | **6** | | | | **9** | | | | **8** |
| R1–NFS Hg amalgam test 4 | 1d : 0.2 | 0.348 | 0.284 | 0.258 | 129.8 | 0.357 | 0.418 | 0.381 | 168.5 | 0.161 | 0.156 | 0.154 | 68.6 |
| R2–NFS Hg amalgam test 4 | 3d : 0.2 | 0.269 | 0.265 | 0.282 | 119.0 | 0.378 | 0.358 | 0.345 | 157.6 | 0.151 | 0.147 | 0.132 | 62.7 |
| R3–NFS Hg amalgam test 4 | 7d : 0.2 | 0.199 | 0.242 | 0.198 | 93.2 | 0.304 | 0.302 | 0.278 | 128.9 | 0.133 | 0.129 | 0.147 | 59.6 |
| Average | | | | | **114** | | | | **152** | | | | **64** |
| Error | | | | | **15** | | | | **14** | | | | **4** |

**Table A-5. Summary data for the MER04 samples from ATG maintained at 20EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] mg/m$^3$ | 3 days–01/10/02 | | | [Hg] mg/m$^3$ | 7 days–01/14/02 | | | [Hg] mg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| D1–ATG MER04 raw sludge batch 2 | 1d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0.003 | 0 | 0.003 | 0.018 |
| D2–ATG MER04 raw sludge batch 2 | 3d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0.003 | 0.003 | 0.018 |
| D3–ATG MER04 raw sludge batch 2 | 7d : 10 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0.003 | 0.009 |
| Average | | | | | **0.00** | | | | **0.00** | | | | **0.01** |
| Error | | | | | **0.00** | | | | **0.00** | | | | **0.01** |
| E1–ATG MER04 raw sludge batch 1 | 1d : 10 | 0 | 0 | 0.003 | 0.009 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0.003 | 0.009 |
| E2–ATG MER04 raw sludge batch 1 | 3d : 10 | 0 | 0.004 | 0.004 | 0.023 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| E3–ATG MER04 raw sludge batch 1 | 7d : 10 | 0.003 | 0.003 | 0.004 | 0.029 | 0 | 0 | 0.019 | 0.055 | 0 | 0 | 0 | 0.000 |
| Average | | | | | **0.02** | | | | **0.02** | | | | **0.003** |
| Error | | | | | **0.01** | | | | **0.04** | | | | **0.006** |
| F1–ATG treated sludge batch 1 6/29 | 1d : 10 | 0.003 | 0.007 | 0.006 | 0.046 | 0 | 0 | 0.017 | 0.050 | 0.007 | 0.008 | 0.008 | 0.067 |
| F2–ATG treated sludge batch 1 6/29 | 3d : 10 | 0.004 | 0.007 | 0.008 | 0.055 | 0 | 0.011 | 0.018 | 0.085 | 0.008 | 0.008 | 0.008 | 0.070 |
| F3–ATG treated sludge batch 1 6/29 | 7d : 10 | 0.008 | 0.012 | 0.013 | 0.096 | 0.011 | 0.013 | 0.014 | 0.111 | 0.009 | 0.011 | 0.011 | 0.090 |
| Average | | | | | **0.07** | | | | **0.08** | | | | **0.08** |
| Error | | | | | **0.02** | | | | **0.05** | | | | **0.01** |
| G1–ATG treated sludge batch 1 6/30 | 1d : 10 | 0.006 | 0.004 | 0.004 | 0.041 | 0.007 | 0.004 | 0 | 0.032 | 0.008 | 0.007 | 0.006 | 0.061 |
| G2–ATG treated sludge batch 1 6/30 | 3d : 10 | 0.006 | 0.005 | 0.005 | 0.047 | 0.007 | 0.006 | 0.011 | 0.070 | 0.006 | 0.006 | 0.006 | 0.053 |
| G3–ATG treated sludge batch 1 6/30 | 7d : 10 | 0.007 | 0.006 | 0.006 | 0.055 | 0.013 | 0.01 | 0.012 | 0.102 | 0.007 | 0.007 | 0.006 | 0.058 |
| Average | | | | | **0.05** | | | | **0.07** | | | | **0.06** |
| Error | | | | | **0.01** | | | | **0.03** | | | | **0.01** |
| H1–ATG treated sludge batch 2 6/30 | 1d : 10 | 0 | 0 | 0 | 0.000 | 0.009 | 0.004 | 0.005 | 0.053 | 0.004 | 0.005 | 0.003 | 0.035 |
| H2–ATG treated sludge batch 2 6/30 | 3d : 10 | 0 | 0 | 0.003 | 0.009 | 0 | 0.004 | 0 | 0.012 | 0 | 0 | 0 | 0.000 |
| H3–ATG treated sludge batch 2 6/30 | 7d : 10 | 0.003 | 0.003 | 0 | 0.018 | 0.005 | 0.004 | 0.004 | 0.038 | 0 | 0.003 | 0 | 0.009 |
| Average | | | | | **0.01** | | | | **0.03** | | | | **0.02** |
| Error | | | | | **0.01** | | | | **0.02** | | | | **0.01** |
| I1–ATG treated sludge batch 2 6/30 | 1d : 10 | 0.003 | 0.004 | 0 | 0.020 | 0.005 | 0.004 | 0.003 | 0.035 | 0 | 0 | 0 | 0.000 |
| I2–ATG treated sludge batch 2 6/30 | 3d : 10 | 0.003 | 0 | 0.004 | 0.020 | 0.004 | 0.006 | 0.005 | 0.044 | 0 | 0 | 0 | 0.000 |
| I3–ATG treated sludge batch 2 6/30 | 7d : 10 | 0 | 0.003 | 0.004 | 0.020 | 0.005 | 0.005 | 0.004 | 0.041 | 0 | 0 | 0 | 0.000 |
| Average | | | | | **0.02** | | | | **0.04** | | | | **0.00** |
| Error | | | | | **0.01** | | | | **0.01** | | | | **0.00** |

**Table A-6. Summary data for the MER04 samples from NFS maintained at 20EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] $mg/m^3$ | 3 days–01/10/02 | | | [Hg] $mg/m^3$ | 7 days–01/14/02 | | | [Hg] $mg/m^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| J1–NFS feed MER04-1 | 1d : 10 | 0 | 0 | 0.003 | 0.009 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| J2–NFS feed MER04-1 | 3d : 10 | 0 | 0.004 | 0.004 | 0.023 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| J3–NFS feed MER04-1 | 7d : 10 | 0.004 | 0.003 | 0 | 0.020 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| Average | | | | | **0.02** | | | | **0.00** | | | | **0.00** |
| Error | | | | | **0.01** | | | | **0.00** | | | | **0.00** |
| K1–NFS feed MER04-2 | 1d : 10 | 0.004 | 0 | 0 | 0.012 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| K2–NFS feed MER04-2 | 3d : 10 | 0.004 | 0.004 | 0.003 | 0.032 | 0 | 0 | 0 | 0.000 | 0 | 0 | 0 | 0.000 |
| K3–NFS feed MER04-2 | 7d : 10 | 0.004 | 0.004 | 0.004 | 0.035 | 0 | 0 | 0 | 0.000 | 0 | 0.003 | 0.003 | 0.018 |
| Average | | | | | **0.03** | | | | **0.00** | | | | **0.006** |
| Error | | | | | **0.01** | | | | **0.00** | | | | **0.008** |
| L1–NFS treated MER04-1 | 1d : 10 | 0.006 | 0.003 | 0.004 | 0.038 | 0 | 0 | 0 | 0.000 | 0 | 0.003 | 0.003 | 0.018 |
| L2–NFS treated MER04-1 | 3d : 10 | 0.004 | 0.006 | 0.004 | 0.041 | 0 | 0 | 0 | 0.000 | 0 | 0.003 | 0.003 | 0.018 |
| L3–NFS treated MER04-1 | 7d : 10 | 0.006 | 0.004 | 0.004 | 0.041 | 0 | 0 | 0 | 0.00 | 0 | 0 | 0.003 | 0.009 |
| Average | | | | | **0.04** | | | | **0.00** | | | | **0.01** |
| Error | | | | | **0.01** | | | | **0.00** | | | | **0.01** |
| M1–NFS treated MER04-2 | 1d : 10 | 0.004 | 0.003 | 0.004 | 0.032 | 0.003 | 0 | 0.003 | 0.018 | 0 | 0.003 | 0.003 | 0.018 |
| M2–NFS treated MER04-2 | 3d : 10 | 0.006 | 0.004 | 0.007 | 0.050 | 0.003 | 0 | 0.009 | 0.035 | 0.003 | 0.003 | 0.003 | 0.026 |
| M3–NFS treated MER04-2 | 7d : 10 | 0.004 | 0.004 | 0.009 | 0.050 | 0.006 | 0.009 | 0.006 | 0.061 | 0.003 | 0.003 | 0 | 0.018 |
| Average | | | | | **0.04** | | | | **0.04** | | | | **0.02** |
| Error | | | | | **0.01** | | | | **0.02** | | | | **0.01** |

**Table A-7. Summary data for the MER04 samples from ATG maintained at 60EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] mg/m$^3$ | 3 days–01/10/02 | | | [Hg] mg/m$^3$ | 7 days–01/14/02 | | | [Hg] mg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| D1–ATG MER04 raw sludge batch 2 | 1d : 3 | 0 | 0 | 0 | 0.000 | 0.035 | 0.03 | 0.029 | 0.914 | 0.083 | 0.077 | 0.077 | 2.304 |
| D2–ATG MER04 raw sludge batch 2 | 3d : 3 | 0 | 0 | 0 | 0.000 | 0.025 | 0.022 | 0.022 | 0.671 | 0.067 | 0.033 | 0.034 | 1.303 |
| D3–ATG MER04 raw sludge batch 2 | 7d : 3 | 0 | 0 | 0 | 0.000 | 0.023 | 0.021 | 0.024 | 0.661 | 0.032 | 0.031 | 0.028 | 0.885 |
| Average | | | | | **0.000** | | | | **0.75** | | | | **1.50** |
| Error | | | | | **0.000** | | | | **0.10** | | | | **0.50** |
| E1–ATG MER04 raw sludge batch 1 | 1d : 3 | 0 | 0 | 0 | 0.000 | 0.021 | 0.02 | 0.022 | 0.613 | 0.023 | 0.024 | 0.02 | 0.651 |
| E2–ATG MER04 raw sludge batch 1 | 3d : 3 | 0 | 0 | 0.003 | 0.029 | 0.018 | 0.015 | 0.015 | 0.467 | 0.018 | 0.019 | 0.018 | 0.535 |
| E3–ATG MER04 raw sludge batch 1 | 7d : 3 | 0 | 0 | 0 | 0.000 | 0.019 | 0.017 | 0.013 | 0.476 | 0.018 | 0.017 | 0.017 | 0.506 |
| Average | | | | | **0.01** | | | | **0.52** | | | | **0.56** |
| Error | | | | | **0.02** | | | | **0.06** | | | | **0.05** |
| F1–ATG treated sludge batch 1 6/29 | 1d : 3 | 0.005 | 0.007 | 0.01 | 0.214 | 0.032 | 0.029 | 0.027 | 0.856 | 0.026 | 0.034 | 0.025 | 0.914 |
| F2–ATG treated sludge batch 1 6/29 | 3d : 3 | 0.016 | 0.015 | 0.013 | 0.428 | 0.019 | 0.016 | 0.016 | 0.496 | 0.02 | 0.015 | 0.014 | 0.457 |
| F3–ATG treated sludge batch 1 6/29 | 7d : 3 | 0.012 | 0.014 | 0.013 | 0.379 | 0.032 | 0.034 | 0.028 | 0.914 | 0.036 | 0.015 | 0.009 | 0.360 |
| Average | | | | | **0.34** | | | | **0.75** | | | | **0.58** |
| Error | | | | | **0.08** | | | | **0.07** | | | | **0.20** |
| G1–ATG treated sludge batch 1 6/30 | 1d : 3 | 0.009 | 0.007 | 0.007 | 0.224 | 0.022 | 0.019 | 0.019 | 0.583 | 0.035 | 0.034 | 0.025 | 0.914 |
| G2–ATG treated sludge batch 1 6/30 | 3d : 3 | 0.009 | 0.013 | 0.007 | 0.282 | 0.014 | 0.012 | 0.014 | 0.389 | 0.018 | 0.015 | 0.014 | 0.457 |
| G3–ATG treated sludge batch 1 6/30 | 7d : 3 | 0.005 | 0.005 | 0.003 | 0.126 | 0.017 | 0.015 | 0.015 | 0.457 | 0.013 | 0.015 | 0.009 | 0.360 |
| Average | | | | | **0.21** | | | | **0.48** | | | | **0.58** |
| Error | | | | | **0.06** | | | | **0.07** | | | | **0.16** |
| H1–ATG treated sludge batch 2 6/30 | 1d : 3 | 0.012 | 0.013 | 0.015 | 0.389 | 0.022 | 0.026 | 0.025 | 0.710 | 0.032 | 0.039 | 0.034 | 1.021 |
| H2–ATG treated sludge batch 2 6/30 | 3d : 3 | 0 | 0.008 | 0.012 | 0.194 | 0.015 | 0.013 | 0.01 | 0.369 | 0.022 | 0.02 | 0.017 | 0.574 |
| H3–ATG treated sludge batch 2 6/30 | 7d : 3 | 0.015 | 0.014 | 0.011 | 0.389 | 0.038 | 0.039 | 0.034 | 1.079 | 0.031 | 0.033 | 0.031 | 0.924 |
| Average | | | | | **0.32** | | | | **0.72** | | | | **0.84** |
| Error | | | | | **0.10** | | | | **0.23** | | | | **0.16** |
| I1–ATG treated sludge batch 2 6/30 | 1d : 3 | 0.014 | 0.014 | 0.014 | 0.408 | 0.022 | 0.024 | 0 | 0.447 | 0.023 | 0.02 | 0.021 | 0.622 |
| I2–ATG treated sludge batch 2 6/30 | 3d : 3 | 0.013 | 0.011 | 0.009 | 0.321 | 0.022 | 0.004 | 0 | 0.253 | 0.017 | 0.019 | 0.024 | 0.583 |
| I3–ATG treated sludge batch 2 6/30 | 7d : 3 | 0.01 | 0.008 | 0.008 | 0.253 | 0.017 | 0 | 0.023 | 0.389 | 0.034 | 0.034 | 0.035 | 1.001 |
| Average | | | | | **0.22** | | | | **0.36** | | | | **0.74** |
| Error | | | | | **0.06** | | | | **0.23** | | | | **0.15** |

**Table A-8. Summary data for the amalgamated mercury from NFS maintained at 60EC**

| Sample name | Sample size (mL) | 1 day–01/08/02 readings | | | [Hg] mg/m$^3$ | 3 days–01/10/02 | | | [Hg] mg/m$^3$ | 7 days–01/14/02 | | | [Hg] mg/m$^3$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N1–NFS Hg amalgam test 1 | 1d : 0.2 | 0.088 | 0.081 | 0.081 | 36.4 | 0.162 | 0.156 | 0.158 | 69.4 | 0.199 | 0.185 | 0.201 | 85.3 |
| N2–NFS Hg amalgam test 1 | 3d : 0.2 | 0.083 | 0.081 | 0.076 | 35.0 | 0.163 | 0.155 | 0.155 | 68.9 | 0.131 | 0.128 | 0.118 | 54.9 |
| N3–NFS Hg amalgam test 1 | 7d : 0.2 | 0.081 | 0.069 | 0.072 | 32.3 | 0.148 | 0.154 | 0.143 | 64.8 | 0.161 | 0.178 | 0.183 | 76.1 |
| Average | | | | | **35** | | | | **68** | | | | **72** |
| Error | | | | | **2** | | | | **2** | | | | **10** |
| O1–NFS Hg amalgam test 2 | 1d : 0.2 | 0.249 | 0.24 | 0.239 | 106.1 | 0.223 | 0.232 | 0.232 | 100.1 | 0.243 | 0.267 | 0.261 | 112.4 |
| O2–NFS Hg amalgam test 2 | 3d : 0.2 | 0.218 | 0.197 | 0.234 | 94.6 | 0.161 | 0.164 | 0.171 | 72.3 | 0.21 | 0.183 | 0.21 | 87.9 |
| O3–NFS Hg amalgam test 2 | 7d : 0.2 | 0.211 | 0.203 | 0.199 | 89.3 | 0.184 | 0.179 | 0.181 | 79.3 | 0.189 | 0.179 | 0.189 | 81.2 |
| Average | | | | | **97** | | | | **84** | | | | **94** |
| Error | | | | | **6** | | | | **9** | | | | **11** |
| P1–NFS Hg amalgam test 3 | 1d : 0.2 | 0.208 | 0.218 | 0.223 | 94.64 | 0.185 | 0.202 | 0.204 | 86.1 | 0.167 | 0.169 | 0.18 | 75.2 |
| P2–NFS Hg amalgam test 3 | 3d : 0.2 | 0.214 | 0.214 | 0.194 | 89.68 | 0.179 | 0.184 | 0.183 | 79.6 | 0.151 | 0.164 | 0.153 | 68.2 |
| P3–NFS Hg amalgam test 3 | 7d : 0.2 | 0.178 | 0.153 | 0.171 | 73.20 | 0.139 | 0.133 | 0.132 | 58.9 | 0.113 | 0.116 | 0.116 | 50.3 |
| Average | | | | | **86** | | | | **75** | | | | **65** |
| Error | | | | | **6** | | | | **9** | | | | **8** |
| R1–NFS Hg amalgam test 4 | 1d : 0.2 | 0.348 | 0.284 | 0.258 | 129.8 | 0.357 | 0.418 | 0.381 | 168.5 | 0.161 | 0.156 | 0.154 | 68.6 |
| R2–NFS Hg amalgam test 4 | 3d : 0.2 | 0.269 | 0.265 | 0.282 | 119.0 | 0.378 | 0.358 | 0.345 | 157.6 | 0.151 | 0.147 | 0.132 | 62.7 |
| R3–NFS Hg amalgam test 4 | 7d : 0.2 | 0.199 | 0.242 | 0.198 | 93.2 | 0.304 | 0.302 | 0.278 | 128.9 | 0.133 | 0.129 | 0.147 | 59.6 |
| Average | | | | | **114** | | | | **152** | | | | **64** |
| Error | | | | | **15** | | | | **14** | | | | **4** |

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