UKAS TESTING 1011

NIRAS
Analytical Services

NIRAS

Report

Report Reference: L080402-Second Report

Customer: IRAS Ltd

Leaching of Mercury from Insulation Material, Dust and Other Debris.

Issue date: 27 October 2008

The applicability of UKAS accreditation to this report is detailed in section 3.1

Prepared by: K G Bradshaw	**Keith Bradshaw Laboratory Technical Manager**
Checked and approved for issue by: J. D. Turner	JOE TURNER SENIOR SCIENTIST

NIRAS is a trading name of AMEC Nuclear UK Limited
The Renaissance Centre, 601 Faraday Street, Birchwood Park, Birchwood, Warrington, Cheshire WA3 6GN

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1 Administrative Details

1.1 Laboratory Job Details

1.1.1 Laboratory Job Number

L080402

1.1.2 Quotation/Tender Reference

X2424 (04 April 2008)

1.1.3 Testing Laboratory

Chemical testing subcontracted to:

Scientific Analysis Laboratories Ltd (SAL Ltd)
Hadfield House,
Hadfield street
Old Trafford
M16 9FE

1.1.4 Laboratory Contact

Keith Bradshaw (Tel: 01925 675643 or 675366, e-mail: keith.bradshaw@amec.com).

1.1.5 Sample Receipt Date

02 May 2008

1.2 Customer Details

1.2.1 Customer Name and Address

Andrew Frith
IRAS Ltd
Bold Business Centre
Bold Lane
St Helens
WA9 4TX

1.2.2 Order Numbers

PO 2008014 (1) and PO 2008014 (2)

2 Scope of Work

Previous work has shown that low temperature leaching in 20% (v/v) nitric acid overnight was not sufficiently effective in leaching mercury from the material. This report concerns the investigation of overnight leaching with 50% (v/v) nitric acid, at room temperature and at 60℃.

Two samples were selected for the trials, one of cotton-based insulation material and the other of dust from a HEPA vacuum cleaner.

3 Quality Assurance

3.1 Quality System and Applicability of UKAS Accreditation

SAL Ltd are UKAS accredited for mercury in leachates.

Any comments, opinions and interpretations expressed in this report are outside the scope of UKAS accreditation.

4 Tests and Experimental Methods

4.1 Sample Preparation and Pretreatment

The samples were used in their as-received condition, mixing thoroughly before sub-sampling.

4.2 Mercury Leaching Investigation

The solids were weighed into glass beakers. Nitric acid solution (50% of concentrated acid by volume, or approximately 8 M concentration) was added to cover the solids. This filled the beakers up to approximately the 200 mL mark. The beakers were covered with clock-glasses and left at room temperature overnight.

The following day, the solids and leachates were separated by filtration through glass-fibre filters (Whatman GFC). The solids were washed with water four times and the washings collected with the leachates. The total amount of each solution was found.

The solids were returned to the beakers, and fresh acid added as before. The beakers were covered and the solution maintained at 60℃ overnig ht.

The following day, the beakers were allowed to cool and filtration was done as before. The solids were washed free of acid and dried to constant weight at 30℃. The amounts of solids (post-leaching) and leachates were recorded.

The solids and sub-samples of the leachates were forwarded to SAL Ltd for mercury analysis.

4.3 Metals by ICP-OES (SAL Ltd)

The sample is digested with nitric/hydrochloric acid. The sample is filtered and then made up to volume. An extract is then applied to ICP instrument, which determines the presence and amount, by the emission spectra from each metal. The intensity of the emission is measured against calibrated standards for qualification.

5 Results

5.1 Observations

Little reaction was observed at room temperature between the MRV6 solid and the acid solution. With the MR6 cotton-based insulation material, there was significant brown fume generation at room temperature. When the temperature was raised both samples were seen to evolve brown fumes to some degree, with MR6 evolving the most.

5.2 Masses of Solids

The solids were weighed (dry) before and after leaching, to find any mass loss during the process. These results are given in Table 1, along with the mass change on leaching.

Table 1 Mass change of solids with leaching

NIRAS reference	Client Reference	Description	Mass prior to leaching (g)	Mass post-leaching (g)	% mass change
L080402-5(1)	MR6	Cotton-based insulation	20.56	9.62	-53.2
L080402-9(1)	MRV6	Dust HEPA vac	21.23	11.51	-45.8

(1) Redesignated as NIRAS references L080402-44 and 45 after leaching and drying

There is a significant apparent mass loss during the process. This can be explained by nitric acid oxidising organic matter to gaseous products, which are lost to atmosphere, and also by solubilisation of material which is subsequently collected in the aqueous leachates. It is not known from the results which mechanism is most important, in fact this could vary with the organic matter content of the material.

5.3 Mercury Determinations

5.3.1 Solids

The solids were dried to constant weight at 30℃ and analysed for mercury. The results are given in Table 2

Table 2 Mercury in solids before and after leaching

NIRAS reference	Client Reference	Mercury prior to leaching (SAL Ltd) (mg kg^{-1})	Mercury prior to leaching (STL Ltd) (mg kg^{-1})	Mercury post-leaching (SAL Ltd) (mg kg^{-1})	Total mercury post-leaching (mg)
L080402-5	MR6	10000	84000	10000	96
L080402-9	MRV6	11000	8800	3000	35

5.3.2 Leachates

Table 3 Mercury in leachates

NIRAS reference	Description	Leachate mass (g)	Mercury (mg L^{-1})	Total mercury (mg)
L080402-40	MR6 room temp leach	749.51	4000	2998
L080402-41	MRV6 room temp leach	933.85	730	682
L080402-42	MR6 60℃ 2 nd leach	982.90	120	118
L080402-43	MRV6 60℃ 2 nd leach	548.07	110	60

5.4 Leaching Efficiency

It is apparent that there is a discrepancy in the mercury analytical results. SAL Ltd report exactly the same concentration for the before-leaching and post-leaching MR6 solid (10000 mg kg^{-1}), despite there being significant concentrations of mercury in the leachates. This could be explained if the solids are heterogeneous.

This being the case, it is preferable to calculate the leaching efficiency relative to the total amount of mercury in solid and both leachates. This has been done and the results are presented in Table 4.

Table 4 Calculated leaching efficiencies

Client Reference	Total Hg (mg)	% Hg in first room temp. leach	% Hg in second 60°C leach	% Hg unleached	Total %
MR6	3212	93.3	3.7	3.0	100.0
MRV6	777	87.8	7.8	4.4	100.0

The first leaching with 50% (v/v) nitric acid at room temperature removes the bulk (approximately 90%) of the mercury. The second leach at 60℃ removes comparatively little, and there is a small fraction of the mercury left in the solid phase after the process.

6 Conclusions

- Leaching with 50% (v/v) (approximately 8 M) nitric acid is significantly more effective at removing mercury from these materials than 20% (v/v) nitric acid (approximately 3 M), as was used in the previous report (*L080402*).
- Approximately 90% of mercury is leached in 50% (v/v) nitric acid at room temperature overnight in both sample types.
- A second leach at 60℃ removes a few percent furth er mercury.
- Significant noxious fume generation takes place, due to oxidation of organic matter by nitric acid during the process. This can occur at room temperature, and is accelerated by heating.
- The mass of material remaining post-leaching is significantly less than the starting mass.