

9442.1991(02)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

FEB 22 1991

Mr. Herschel Cutler  
Institute of Scrap Recycling Industries, Inc.  
1627 K Street, NW  
Washington, DC 20006-1704

Dear Mr. Cutler:

I appreciate your providing us with the documentation of your analyses of the fluff materials. We are returning the documentation, per your request. Please note that in order to make a copy for our records we were obliged to remove the binding. I trust this will not be too great an inconvenience.

The comparison of the results between our contractor (MRI) and your contractor (BCM) indicated a difference in some of the results, as you had previously pointed out. BCM's EP Tox results were approximately 3 - 30 fold lower than those from MRI for lead and approximately 2 - 10 fold lower for cadmium. The TCLP results were generally comparable, although BCM did have two significantly lower lead values and one lower cadmium value.

The attached report describes the evaluation we conducted of the analytical documentation from both BCM and MRI. I would like to highlight those areas we thought were most noteworthy.

Since pH is one of the key factors effecting the leachability of metals into an aqueous medium, we have carefully reviewed the data provided by both laboratories with special attention to the pH data.

For the EP Tox procedure, it is necessary to adjust the pH to 5.0 +/- 0.2 with an acetic acid solution (see attachment for details on procedure). MRI provided full documentation of the pH adjustment step, including the pH values before and after each adjustment. BCM did not provide documentation of the pH values after adjustment for some of the samples. Therefore, it was difficult to fully evaluate the impact of this step in a comparative manner. Data from BCM

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indicates that the pH before each adjustment was much higher than 5.0 +/- 0.2, and this may indicate that overall, the pH was higher in the samples leached by BCM than MRI. That may be one reason that the concentrations of lead derived from the EP Tox test as performed by BCM were significantly lower than MRI'S. Also, during the on site review, a discussion with BCM personnel indicated that, due to the large quantity of acid required and the time involved, a decision was made to lower the pH to only 5.2. This is the upper limit of the allowable pH range, and this factor may also help explain the lower levels of lead found in the samples analyzed by BCM. (Details provided in attached report.)

When using the TCLP procedure, a selection must be made between two different extraction fluids. This selection is made based on the pH of the material. In two cases MRI used extraction fluid #2, whereas BCM used extraction fluid #1 in all cases. For both of those samples where MRI used extraction fluid #2, MRI reported a higher concentration of lead than did BCM. The use of extraction fluid #2 for these samples had no apparent effect on the concentration of cadmium. MRI provided data to support the use of extraction fluid #2, while BCM provided no data to indicate which fluid should be used. (See attached report.)

Relatively minor analytical anomalies were noted in some of the data submitted by both laboratories. It is the opinion of the reviewer that the anomalies would not make a major contribution to the differences observed. However, these are also included in the attached report.

We conclude that the data provided does not support the contention that the TCLP is a more aggressive test than the EP Tox. We are aware that laboratories do have more difficulty in using the EP Tox, and we feel that this study emphasizes that observation. Data from MRI show that the results are roughly comparable between the EP Tox and the TCLP. We do not believe that the documentation provided by BCM is adequate to support the proposal that the two leaching procedures are not comparable.

We appreciate your participation in this comparison study. We feel it has provided further clarification of our reason for replacing the EP Tox with the TCLP. If you have any questions on this evaluation,, please contact Alec McBride on 382-4761.

Sincerely,

David Bussard  
Director  
Characterization and Assessment Division

cc: Alec McBride  
Jeanne Hankins  
Gail Hansen  
Steve Cochran  
John Scalera, OTS  
Dan Reinhart, OTS

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## REVIEW OF DATA ON FLUFF SAMPLES

This review covers all the data provided by both BCM and MRI. The data was reviewed for internal consistency, calculation errors, compliance with the method, and quality control measures.

### EP TOX

#### pH DETERMINATION

After addition of the waste and the distilled water into the extractor, the pH must be measured and then adjusted if it is greater than 5.2. The pH must be monitored during the course of the 24 hour extraction period according to the following schedule:

adjust the pH of the solution in 15-, 30-, or 60-minute intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5 pH units.

continue the adjustment for at least 6 hours.

if, at the end of the 24 hour extraction period, the pH of the solution is not below 5.2, and the maximum amount of acid has not been added, the pH should be adjusted to  $5.0 \pm 0.2$ , or until the maximum allocated amount of acid is utilized, and the extraction continued for an additional 4 hours, during which the pH should be adjusted at 1 hour intervals.

Data forms provided by MRI allowed for simple and easy determination of the pH adjustment process. The following information was included: time and interval, pH before and after adjustment, volume of acid added and cumulative amount, post 24 hour adjustment, and final volume of water added.

Data provided by BCM was difficult to comprehend. only in some cases was the time and/or interval indicated. In those cases where the pH was provided, it was apparently the pH before adjustment with the acetic acid solution. It was not possible to determine what the pH was at the end of each adjustment or at the end of the 24 hour period and whether the leaching period was extended an additional 4 hours if the pH was greater than 5.2. It was also unclear whether

there was any addition of water at the end of the leaching period.

It was noted that the volume of acid added by BCM was approximately two fold greater than that added by MRI, which could indicate a difference in concentration of the acetic acid reagent. Otherwise, one would expect the BCM results to show greater leaching. The incremental volumes themselves frequently differed by approximately a factor of 10, most notably at the initiation of the leaching procedure. MRI in most cases adjusted the pH very close to 5.0, with two excursions below 4.8 (duplicates of sample 5,5 were adjusted to a pH of 4.71 and 4.45 initially). Since BCM did not provide pH data after the addition of acid, it is not possible to determine whether the adjustment procedure affected the amount of leaching. Conversations between BCM personnel and the on site observers from EPA indicated that the pH adjustment was stopped at a pH of 5.2 due to the large volume of acid added and the extended length of time for adjusting the pH. The pH recordings (presumably before adjustment) which were documented by BCM were much higher than 5.2. This might mean that the amount of lead or cadmium extracted could be lower because of a higher overall average pH during extraction.

In summary, there are several apparent inconsistencies. MRI added smaller increments of acid, a smaller total volume of acid, but, in those cases where it was possible to make a comparison with BCM, MRI samples had a lower pH at the end of the 24 hour leaching period. It should be noted, however, that in no case did data from MRI or BCM show that the pH was less than 5.2 at the end of 24 hours. Therefore MRI continued the leaching procedure for an additional 4 hours as per the method, which may have increased the amount of lead and cadmium leached from the fluff. It was not possible to determine if BCM had an additional 4 hour extraction period.

## TCLP

### Extraction Fluid Selection

In order to determine the appropriate extraction fluid for the TCLP, one must use the following procedure:

Weigh 5 g of the solid phase of the waste (particle size 1 mm diameter) into a 500 mL beaker or-flask.

Add 96.5 mL of reagent water to the beaker, cover with a watch glass and stir for 5 minutes.

Measure and record the pH. If the pH is less than 5.0 use extraction fluid #1.

If the pH is greater than 5.0, add 3.0 mL 1.0 N HCl, cover with a watch glass, heat to 50°C for 10 min.; let cool to room temperature and record the pH.

If the pH is now less than 5.0, use extraction fluid #1. If the pH remains greater than 5.0, use extraction fluid #2.

MRI provided information which detailed the pH values at each step. Based on the pH, two of the samples were extracted with fluid #2: samples 3,12 and 5,5. BCM used extraction fluid #1 for all samples, and did not provide any documentation of pH checks. The lead analyses of these two samples revealed significant differences in concentration, but the cadmium results were not significantly different.

Sample 6,4 had a higher concentration of lead and a lower concentration of cadmium when analyzed by BCM as compared to MRI. No differences in procedure were noted that might have accounted for this inconsistency.

In summary, the differences noted in the TCLP results for lead analyses in samples 3,12 and 5,5 would appear to be due to the use of different extraction fluids. Further information is needed to determine whether the correct extraction fluid was selected by BCM. The data does not indicate any variation in method for the differences noted in sample 6,4.

#### ANALYSIS BY FLAME ATOMIC ABSORPTION SPECTROMETRY

MRI documented instrument drift in several instances when using flame atomic absorption spectrometry (FAAS). Logbooks from MRI describe the techniques used to compensate for the drift, including 1) manually establishing the baseline from the strip chart recorder and 2) subtracting the blank from the preceding 10 samples. The second practice is not recommended as it would tend to inappropriately lower the concentration in the samples analyzed

first. Strip chart recordings would be needed to verify concentrations under the first condition.

Examination of the BCM data also indicated possible instrument draft. Several QC samples had to be reanalyzed before they met QC limits. Also, several analytical runs were missing from the package. No explanation was given for these anomalies. Other observations include the following: In the initial calibration for lead, BCM did not use the 25 mg/L sample to establish the calibration curve. During the cadmium analysis, the auto-zero function was initiated after calibration. MRI had a high spike recovery (approximately 10% higher than expected) for cadmium in sample 6,4. Reanalysis confirmed the high recovery.

In summary, the data from MRI may be biased low for cadmium in EP Tox, based on the technique for compensating for baseline drift. Cadmium results for the TCLP leachate of sample 6,4 by MRI may be biased high. However, the effects of these biases on the results would be relatively minor, probably no more than +/- 10%. Further information would be needed to understand what effect the anomalies found in the BCM data would have on the bias of any results.

#### GRAVIMETRIC DATA

Both the EP Tox and the TCLP require the use of a 100 g sample which must be weighed out before commencing the procedure. BCM provided data on the weight determination of seven samples. Several errors were identified (6 of 7). Although these errors would have only a nominal effect on the overall accuracy of the results it indicates a potential weakness in the quality assurance system. The following data compares the results calculated by BCM and the EPA reviewer:

SAMPLE NO	BCM	EPA
2,5	100.23	99.40
2,5	100.31	99.73
2,4	100.35	100.35
2,4	100.85	100.70
2,12	100.38	100.08
2,12	100.16	99.55

3,5      100.32      100.73

Results for all other weight determinations were not provided by  
BCM nor MRI.