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# Open digestion of some plant and fungus materials for mercury analysis using different temperatures and sample sizes

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**Abstract**

Dry samples of lichen, moss and mushroom were treated by open wet digestion at different temperatures and different sample sizes and analyzed for mercury. No temperature (+80°C, +90°C, +105°C) dependent differences were observed for the moss and mushroom analyses. For lichen the variations were larger. In this material the digestion was more efficient at +120°C and +140°C than at lower temperatures. For this matrix the best results were obtained when using smaller samples at higher temperatures. The sample weight seemed to be even more important for the digestion efficiency.

*Keywords:* Lichen; Moss; Mushroom; Mercury; Open digestion

## 1. Introduction

The general purpose for pretreatment of biological samples for elemental analysis is a complete sample mineralization without analyte losses. Incomplete digestion may be due to low temperature, insufficient amount of acid (in relation to sample size), insuffient digestion time and/or inefficient mixture of acids and oxidizing agents. On the other hand, it is possible to have a loss of mercury at high temperatures (Semu et al., 1985). Attempts to achieve complete wet digestion have often included the use of heating in closed vessels at elevated pressure (May and Stoeppler, 1984). However, the traditional wet digestion method in open vessels is widely used because it is cheaper and easier to use (Haas and Krivan, 1984; Delves, 1992).

The highest recoveries of mercury from fish material have been found at +100°C to +120°C; at lower temperatures the recovery is poorer and at 140°C there are losses, which are presumed to be due to volatilization (Sadiq and Zaidi, 1983; Sadiq et al., 1991). However, Rasmussen et al. (1991) obtained good precision and accuracy with plant samples (trees, mosses, lichens and mushrooms) after an open digestion at +250°C.

The digestion of plant material is often more difficult than for animal tissues. Lichens are rather

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Table 1
Mercury concentrations (ng/g dry wt.) of moss (*Sphagnum fuscum*) after digestion at different temperatures

| Replicate | +80°C | +90°C | +105°C |
|---|---|---|---|
| A | 97 | 89 | 98 |
| B | 80 | 79 | 82 |
| C | 87 | 99 | 75 |
| D | 86 | 93 | 91 |
| E | 89 | 96 | 88 |
| F | 88 | 85 | 99 |
| Mean | 88 | 90 | 89 |
| S.D. | 5.0 | 6.7 | 8.5 |
| R.S.D. % | 5.7 | 7.5 | 9.5 |
| Min. | 80 | 79 | 75 |
| Max. | 97 | 99 | 99 |

R.S.D., relative standard deviation.

special biological samples consisting of algae and fungi. The epiphytic lichen *Hypogymnia physodes* has proved to be an effective species for biomonitoring of mercury and other metals. The concentrations even in unpolluted areas are high enough to be easily detected by commonly used analytical methods. However, this species is rather difficult to digest and this may be one reason for the differences observed between laboratories in intercalibration studies (Quevauviller et al., 1993).

Table 2
Mercury concentrations ($\mu$g/g dry wt.) of mushroom (*Agaricus sp.*) after digestion at different temperatures

| Replicate | +80°C | +90°C | +105°C | +120°C |
|---|---|---|---|---|
| A | 12 | 8.3 | 9.3 | 9.5 |
| B | 9.1 | 9.9 | 9.6 | 9.5 |
| C | 10 | 11 | 10 | 9.2 |
| D | 10 | 9.8 | 10 | 9.3 |
| E | 10 | 11 | 10 | 9.7 |
| F | 8.3 | 9.3 | 8.7 | 10 |
| G | 9.6 | 9.0 | 10 | 9.9 |
| H | 8.5 | 9.4 | 9.2 | 8.5 |
| I | 9.2 | 8.6 | 9.5 | |
| J | 11 | 9.8 | 9.2 | |
| K | 9.2 | 10 | 9.3 | |
| L | 9.4 | 10 | 10 | |
| Mean | 9.7 | 9.7 | 9.6 | 9.5 |
| S.D. | 0.92 | 0.78 | 0.50 | 0.51 |
| R.S.D. % | 9.5 | 8.1 | 5.1 | 5.4 |
| Min. | 8.3 | 8.3 | 8.7 | 8.5 |
| Max. | 12 | 11 | 10 | 10 |

R.S.D., relative standard deviation.

The purpose of this study was to investigate the effects of temperature and sample weight on the digestion efficiency in mercury analyses of lichen and some other dry biological materials.

## 2. Material and methods

Samples of three different matrices were used: moss (*Sphagnum fuscum*), epiphytic lichen (*Hypogymnia physodes*) and mushroom (*Agaricus sp.*). The samples were collected by hand in southern Finland, placed in paper bags, dried at +40°C and homogenized using an electric homogenizer. The dry weight of the samples used for digestion varied between 0.2 and 0.5 g.

The samples were digested by using conc. $H_2SO_4$ and $HNO_3$ (4:1) in 100-ml pyrex glass tubes and heated in an aluminium block with temperature control (accuracy $\pm$3°C). The mercury was oxidized by $KMnO_4$. The excess oxidant was reduced by $OH \cdot NH_3HCl$ and the mercury finally liberated by $SnCl_2$. All chemicals used were of pro analysi purity. The amount of mercury was measured by CVAAS (cold vapour atomic absorption spectrometry) using a Bacharach MAS 50B analyzer. The result obtained by using this method after digestion at +85°C for reference sample BCR 62 (Olive leaves) was 0.31 $\pm$ 0.03 (S.D.) $\mu$g Hg/g ($n$ = 4) while the certified value was 0.28 $\pm$ 0.02 $\mu$g/g (95% C.I.; n = 10) (C.I. Confidence interval). The detection limit using this method is ~5 ng/g. The analyzer was calibrated using 1 $\mu$g of $HgCl_2$ in 100 ml of distilled water.

The sample sizes and temperatures used were:

| | |
|---|---|
| Moss (~ 0.5 g) | +80°C, +90°C, +105°C |
| Mushroom (0.1–0.4 g) | +80°C, +90°C, +105°C, +120°C |
| Lichen (0.2–0.3 and 0.4–0.5 g) | +80°C, +90°C, +105°C, +120°C, +140°C |

The results are expressed as geometric means, ranges, standard deviations (S.D.) and relative standard deviations (R.S.D. % = S.D./mean × 100). Two results differing more than 15% from

Table 3
Mercury concentrations (ng/g dry wt.) of lichen (*Hypogymnia physodes*) after digestion at different temperatures; sample weight 0.4–0.5 g

| Replicate | +80°C | +90°C | +105°C | +120°C | +140°C |
|---|---|---|---|---|---|
| A | 160 | 90[c] | 170 | 160 | 170 |
| B | 170 | 150 | 120 | 140 | 160 |
| C | 130 | 130 | 140 | 180 | 180 |
| D | 140 | 130 | 160 | 170 | 190 |
| E | 150 | 140 | 74[c] | 160 | 170 |
| F | 140 | 110 | 150 | 150 | 170 |
| G | 100 | 140 | 130 | 150 | 180 |
| H | 140 | 150 | 150 | 140 | 170 |
| Mean | 140[a] | 140[a] | 140[a] | 160[b] | 170[b] |
| S.D. | 19 | 16 | 23 | 11 | 8.6 |
| R.S.D. % | 14 | 11 | 16 | 7.2 | 4.9 |
| Min. | 100 | 110 | 120 | 140 | 160 |
| Max. | 170 | 150 | 170 | 180 | 190 |

R.S.D., relative standard deviation.
[a,b]Means belonging to different groups according to Tukey's test (rejection level = 0.05).
[c]Sample excluded from statistical calculations.

the mean ± S.D. were considered erraneous and excluded from statistical calculations. The differences between treatments of lichen samples was tested using the Tukey's test.

## 3. Results and discussion

The mercury concentrations were low in mosses (Table 1) and high in mushrooms (Table 2). For these samples no significant differences between digestion temperatures were detected. The relative standard deviations varied between 5 and 10%. Obviously temperatures in the range of +80°C to +120°C do not affect the digestion efficiency of moss and mushroom.

For lichen the variation between replicate determinations was clearly higher: R.S.D. 5–16% (Tables 3 and 4). The big variation in *Hypogymnia*

Table 4
Mercury concentrations (ng/g dry wt.) of lichen (*Hypogymnia physodes*) after digestion at different temperatures; sample weight 0.2–0.3 g

| Replicate | +80°C | +90°C | +105°C | +120°C | +140°C |
|---|---|---|---|---|---|
| A | 220 | 180 | 180 | 210 | 240 |
| B | 150 | 170 | 200 | 190 | 200 |
| C | 200 | 220 | 190 | 210 | 210 |
| D | 170 | 170 | 170 | 180 | 200 |
| E | 190 | 190 | 200 | 200 | 160 |
| F | 180 | 170 | 170 | 180 | 180 |
| G | 170 | 160 | 160 | 190 | 180 |
| H | 180 | 180 | 170 | 190 | 180 |
| Mean | 180[a] | 180[a] | 180[a] | 190[a] | 180[a] |
| S.D. | 20 | 17 | 14 | 13 | 23 |
| R.S.D. % | 11 | 9.7 | 7.7 | 6.8 | 12 |
| Min. | 150 | 160 | 120 | 180 | 160 |
| Max. | 220 | 220 | 170 | 210 | 240 |

R.S.D., relative sandard deviation.
[a]According to Tukey's test (rejection level = 0.05) there was no difference between the means.

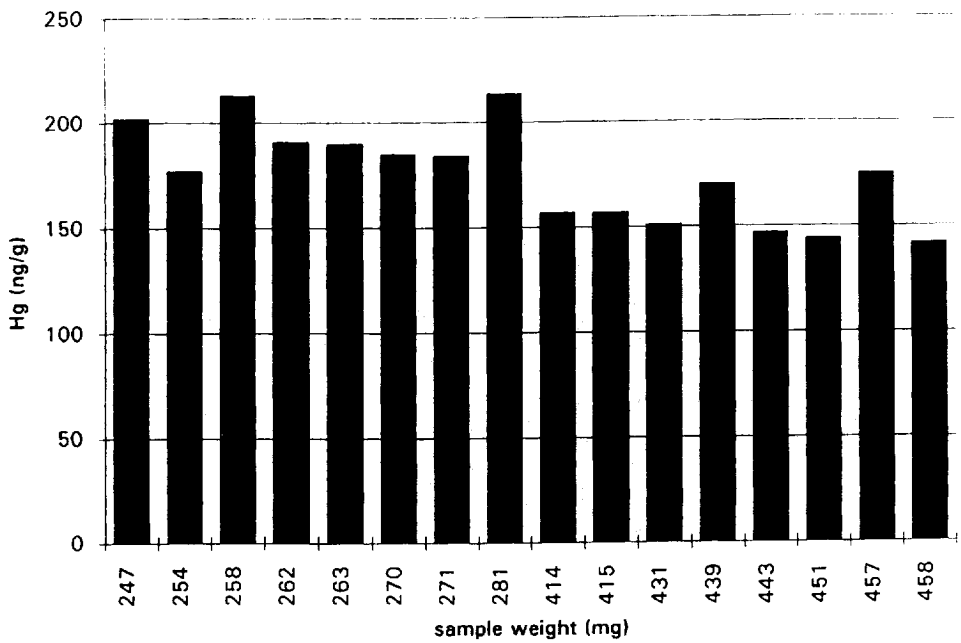


Fig. 1. Obtained mercury concentrations of *Hypogymnia physodes* after digestion of different sample weights at +120°C.

once again indicates that lichen is a difficult matrix for chemical analysis. There was a tendency for higher results at +120°C and +140°C. The best results were obtained after digestion of small samples (0.2–0.3 g) at +120°C or +140°C. For lichen, the sample weight seemed to be more important than the digestion temperature (Fig. 1).

## 4. Conclusions

For moss and mushroom samples analysed in this investigation, all temperatures in the range +80°C to +120°C seem to give the same digestion efficiency. For lichens, more effective digestion is needed and both digestion temperature and sample weight are important, the best results being achieved with smaller weights and/or higher temperatures.

The digestion temperature and the sample weight are only two of several factors affecting the digestion efficiency; it was not our intention to test the total accuracy of this method. A better efficiency could possibly also be reached by using larger aliquots of acid, additional oxidizers or using closed systems.

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