

# MERCURY POLLUTION

## Integration and Synthesis

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## Spatial Distribution Patterns of Mercury in an East-Central Minnesota Landscape

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**ABSTRACT:** We investigated total mercury (Hg) distribution in relation to forested slope position and to vegetation cover type, ranging from abandoned agricultural field to mature forest, at Cedar Creek Natural History Area in east-central Minnesota. Mean concentration of Hg was 94 ng g<sup>-1</sup> in the organic layer at the soil surface, 20 ng g<sup>-1</sup> in the 0 to 10 cm soil layer, and 8 ng g<sup>-1</sup> in the 10 to 50 cm soil layer. Distribution of Hg across the landscape was significantly related to cover type, with more Hg present in surface soils under forests than in fields. Mercury concentration and mass were related to levels of soil organic matter, and were therefore also related to the time when fields had been abandoned from agriculture and the resulting period of organic matter accumulation. Surface soils in forests are enriched in Hg relative to nitrogen compared to those in fields; processes that affect the variation of Hg with soil organic matter differ in those systems. The Hg concentration in the 10 to 50 cm soil layer was assumed to be a measure of background levels in the uniform soil parent material, and net increase of Hg on the landscape was calculated. Lowest net increase was in fields, 0.3 mg m<sup>-2</sup>, and highest was in forests, 2.4 mg m<sup>-2</sup>; net increase in transitional vegetation was also low (0.7 mg m<sup>-2</sup>). These quantities are comparable to those we have measured in forest soils across the Great Lakes States. Because terrestrial landscapes are the receptors for the majority of Hg deposited from the atmosphere, the accumulation and transport of Hg in those landscapes merits further study.

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## I. INTRODUCTION

Mercury (Hg) has been recognized as a potential health threat in relation to consumption of fish in the Great Lakes states and the adjacent Province of Ontario. This recognition has resulted in significant research on both deposition of Hg from the atmosphere<sup>1</sup> and its levels and behavior in aquatic systems.<sup>2-4</sup> Although terrestrial systems occupy a much greater proportion of the landscape in affected regions, levels and behavior of Hg in terrestrial systems have not been intensively investigated. Both accumulation and translocation of Hg in terrestrial systems and movement to aquatic systems are important processes that are poorly understood.

In previous work, we found a gradient in Hg content in soils along a geographic gradient across the Great Lakes states.<sup>5</sup> Increasing Hg levels were associated with an increase in acidic deposition across the gradient. Sampling in that study was restricted to forest stands in nearly level, well-drained sites. In the present study, our interest shifted to a local scale to determine the influence of land use, vegetation structure, and topography on Hg content of soils. We chose an area of very uniform soil parent material so that Hg differences due to intrinsic soil properties would be minimized. Our hypothesis was that levels of Hg in soil would be related to organic matter accumulation and vegetation structure, both of which are influenced by land-use history. Specifically, we hypothesized: (1) more Hg in forests than in fields because of greater organic matter in forest soils and greater interception of atmospherically deposited Hg by forest canopies, and (2) more Hg in fields that were abandoned from agriculture relatively long ago compared to those more recently abandoned because of more time for both Hg deposition and concomitant Hg and soil organic matter accumulation.

## II. METHODS

### A. LOCATION

The study was conducted at Cedar Creek Natural History Area (CCNHA), Minnesota. This 2300-ha area, a National Science Foundation Long Term Ecological Research Site, is located in the Anoka Sand Plain in east central Minnesota (45° 25'N, 93° 10'W). The area is characterized by gently rolling topography, a high groundwater table, organic soils in wetlands, and sandy (>90% sand) mineral soils on uplands.<sup>6</sup> Upland soils at CCNHA, and at the study sites, are mapped as Alfic Udipsamments (Zimmerman series) and Typic Udipsamments (Sartell series).<sup>6</sup> Annual precipitation averages 66 cm.<sup>6</sup> Climate interacts with the sandy soils to create typically dry sites.

Approximately half of the upland area has been cultivated, but most agricultural fields have been abandoned over the last 60 years, resulting in a series of "old fields" that are undergoing secondary succession.<sup>7</sup> The remaining area is currently closed canopy forests dominated by northern red oak (*Quercus rubra* L.), northern pin oak (*Quercus ellipsoidalis* E.J. Hill), and bur oak (*Quercus macrocarpa* Michx.), but other species are also present, including red pine (*Pinus resinosa* Ait.), white pine (*Pinus strobus* L.), basswood (*Tilia americana* L.), sugar maple (*Acer saccharum* Marsh.), red maple (*Acer rubrum* L.), and paper birch (*Betula papyrifera* Marsh.).

### B. FIELD

Transects, 30 to 50 m in length, were established from the summit to base of three forested slopes. Slopes were approximately 15% and aspect varied among the transects. Five positions were chosen along each transect: summit, shoulder, backslope, footslope, and toeslope (nomenclature follows Ruhe and Walker<sup>8</sup>). Sites were chosen to have relatively constant vegetation along transects. Additional transects were established from seven abandoned agricultural fields and one (never-cultivated) prairie into adjacent mature forests. Transects were placed at fairly level sites to minimize the influence of topography on soil properties.

Along each transect, five positions were chosen, typically two in the field, one on the field-forest transition, and two in the forest. Known dates since abandonment of fields from agriculture ranged from 3 to 63 years. One field had been abandoned more than 65 years before sampling, and the prairie site had never been cultivated.

At each position along the transects, five sampling points were established perpendicular to and within 5 m of the transect. At each sampling point, the organic material (O<sub>i</sub> + O<sub>e</sub> horizons) on the mineral soil surface was collected from a 15.5 × 15.5 cm square. No collections of this layer were made from the prairie-to-forest transect because a prescribed burn shortly before sampling had consumed most of that layer. A core (3.75 cm diameter) of mineral soil was taken to a depth of 50 cm at each sampling point. Slopes were sampled in August 1989 and field-to-forest transects in June 1990.

### C. LABORATORY

Samples of organic horizons were oven-dried (70° C). Mineral soil samples were air-dried and roots were removed with forceps. For each transect position, samples representing the organic layer, the upper 10 cm of mineral soil, and the 10 to 50 cm layer were composited from samples from the five sampling points. A subsample of each composite was ground and analyzed for Hg. Samples were digested at 145° C with sequential additions of concentrated HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, and HCl followed by cold digestion with KMnO<sub>4</sub> and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>. Mercury was determined by cold vapor atomic absorption spectrophotometry in conjunction with a gold trap.<sup>9</sup>

A subsample of material was also oven-dried at 105° C and soil organic matter (SOM) was determined by loss-on-ignition at 450° C.<sup>10</sup> Another composite of the upper 10 cm of mineral soil from each sampling point was also ground, oven-dried, and total C and N concentrations were determined with a Carlo Erba CNS analyzer.

### D. NUMERICAL

Measured concentrations of Hg were converted to mass per unit area based on the mass of the sampled organic layer, measured mass of the 0 to 10 cm soil layer determined from the core samples, and estimated mass of the 10 to 50 cm layer based on a bulk density-SOM relationship.<sup>11</sup>

Analyses of variance were used to determine significance of differences between variables by transect position, with individual transects treated as blocks.<sup>12</sup> Analyses of variance were also used to determine significance of differences with vegetative cover (field, forest, or transition), irrespective of transect. Separation of means was by Bayes least significant difference (BLSD).<sup>13</sup> Linear regression analyses were by SYSTAT.<sup>14</sup>

## III. RESULTS AND DISCUSSION

### A. ANALYTICAL PRECISION

Laboratory blanks averaged  $-0.78 \text{ ng g}^{-1}$  ( $n = 16$ ), Hg recovery in spiked samples averaged 106% ( $n = 15$ ), and duplicates had a coefficient of variation of 18% ( $n = 63$  pairs). Analyses of National Institute of Standards and Technology (NIST) reference materials were near the upper limits of their confidence intervals (Estuarine Sediment, SRM 1646, mean =  $78 \text{ ng g}^{-1}$ ,  $n = 9$ , NIST =  $63 \pm 12 \text{ ng g}^{-1}$ ; Citrus Leaves, SRM 1572, mean =  $97 \text{ ng g}^{-1}$ ,  $n = 6$ , NIST =  $80 \pm 20 \text{ ng g}^{-2}$ ).

Two samples of the 0 to 10 cm horizon, collected at toeslope positions on the forested slopes, were treated as outliers in the analysis. Their Hg concentrations were more than 14 standard deviations from the remainder of the population of samples from that layer. They were excluded from the analyses except in the calculation of net increase of mass of Hg.

Table 1 Results of analysis of variance of differences in Hg at five stations along three transects from summit to toeslope in forests.

Layer	Units	Mean	MSE <sup>√a</sup>	F <sup>b</sup>	d.f. <sup>c</sup>	Prob.
Organic	ng g <sup>-1</sup>	142.92	28.32	2.14	4,8	0.167
	mg m <sup>-2</sup>	0.33	0.23	1.08	4,8	0.426
0-10 cm	ng g <sup>-1</sup>	36.25	6.66	14.71	4,6	0.003
	mg m <sup>-2</sup>	3.41	0.51	8.69	4,6	0.011
10-50 cm	ng g <sup>-1</sup>	11.31	4.44	1.24	4,8	0.367
	mg m <sup>-2</sup>	7.47	2.90	1.24	4,8	0.368

<sup>a</sup> Square root of mean square error.

<sup>b</sup> F-ratio.

<sup>c</sup> Degrees of freedom.

Table 2 Results of analysis of variance of difference in Hg at five stations along eight transects from abandoned agricultural fields into forests.

Layer	Units	Mean	MSE <sup>√a</sup>	F <sup>b</sup>	d.f. <sup>c</sup>	Prob.
Organic	ng g <sup>-1</sup>	76.09	20.67	19.36	4,24	0.0001
	mg m <sup>-2</sup>	0.12	0.06	16.05	4,24	0.0001
0-10 cm	ng g <sup>-1</sup>	14.37	3.45	16.40	4,28	0.0001
	mg m <sup>-2</sup>	1.74	0.39	12.16	4,28	0.0001
10-50 cm	ng g <sup>-1</sup>	7.72	3.14	0.78	4,28	0.546
	mg m <sup>-2</sup>	5.11	2.08	0.78	4,28	0.548

<sup>a</sup> Square root of mean square error.

<sup>b</sup> F-ratio.

<sup>c</sup> Degrees of freedom.

## B. FORESTED SLOPE TRANSECTS

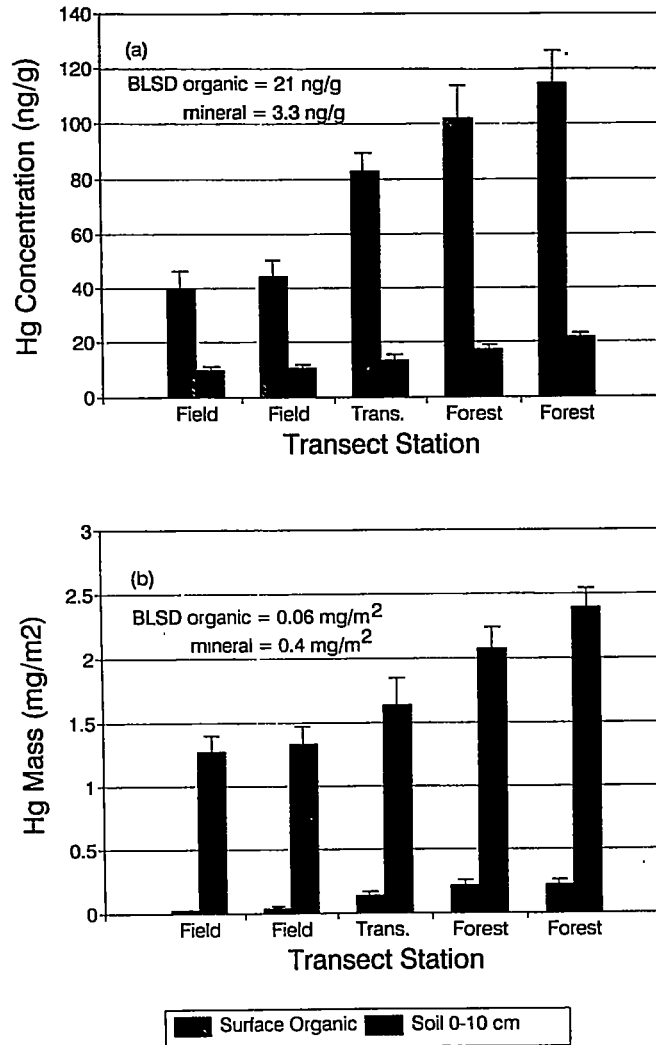
Differences in Hg on the forested slopes were significant in the 0 to 10 cm soil layer, but not in either the organic layer or the deeper soil layer (Table 1). In the 0 to 10 cm layer, maximum concentrations (83 ng g<sup>-1</sup>) and mass (5.4 mg m<sup>-2</sup>) of Hg were found at toeslope positions, but higher levels were also found on the shoulder of the slope (44 ng g<sup>-1</sup>; 4.4 mg m<sup>-2</sup>), with lowest levels at the summit and backslope (about 29 ng g<sup>-1</sup>; 2.9 mg m<sup>-2</sup>). Increased Hg in toeslope positions may result from translocation of organic-Hg complexes by interflow or runoff.<sup>15,16</sup> We have no explanation for increased levels in shoulder positions. The lack of a unidirectional change in Hg from summit to toeslope is consistent with irregular patterns of many other soil properties on slopes in this landscape.<sup>17</sup>

## C. FIELD-TO-FOREST TRANSECTS

Both concentration and mass of Hg in the organic layer and in the surface mineral soil varied significantly with location along the field-to-forest transects (Table 2). Both measures of Hg were lowest in the field portion of the transects, and increased through the transition into the forest (Figure 1). Neither the concentration nor the mass of Hg in the deeper soil layer (10 to 50 cm) varied significantly along the transect (Table 2). Either the deposition of Hg or its retention appears to differ with land use history and vegetation structure.

## D. EXPLANATORY VARIABLES

Soil organic matter is variable in the CCNHA landscape due to land-use history. Forest and savannah soils contain greater amounts of SOM in the surface mineral layer than fields;<sup>18</sup>



**Figure 1** Mercury concentration (a) and mass (b) in soils at five positions along eight transects from abandoned agricultural fields (and a native prairie) into adjacent oak and pine forests at Cedar Creek Natural History Area, Minnesota. Standard error indicated. Mercury concentration of the 10 to 50 cm soil layer did not differ significantly among stations (mean = 7.7 ng g<sup>-1</sup>, pooled SE = 1.1 ng g<sup>-1</sup>).

consistent with the reduction in SOM upon cultivation.<sup>19</sup> Detailed sampling of a range of fields of differing periods since abandonment indicates that many soil chemical and microbial properties, including organic matter, increase exponentially, approaching an asymptote about 70 years after abandonment.<sup>18</sup> Concentration of Hg and SOM are closely related in the surface mineral soil layer ( $r^2 = 0.84$ ,  $n = 53$ ,  $prob. = 0.000$ ). In fact, when all mineral soil samples from fields, forests, and transition zones are included in a regression, a strong relationship between Hg (ng g<sup>-1</sup>) and SOM (%) persists,

$$Hg = 0.74 + 6.6 \text{ SOM}, (r^2 = 0.82, Sy.x = 4.84, n = 108, prob. = 0.000) \quad [1]$$

The relationship is much poorer in the organic layer ( $r^2 = 0.02$ ). These results suggest regulation of Hg retention in the mineral soil by decomposed, humified SOM.

Mercury concentrations decreased with depth (Table 1 and Table 2) for two reasons; (1) atmospheric deposition of Hg is to the soil surface, and hence Hg concentrations at the surface should be higher than in the subsurface, and (2) SOM decreases with depth so that closely associated Hg also decreases. Conversely, the mass of Hg per unit area apparently increases with depth (Table 1 and Table 2) because the mass of the three soil horizons that we sampled increases from the surface downward as both their thickness and density increase. The lesser concentration, multiplied by the greater mass of soil, yields a greater mass of Hg per unit area with depth.

Mercury concentrations of the organic and the mineral surface soil layers of the field samples from the field-to-forest transects are weakly but positively correlated with time since agricultural abandonment (when known) ( $r^2 = 0.67$  for the organic layer and 0.38 for the surface soil layer,  $n = 12$ ). Such trends are lacking in the deep soil layer ( $r^2 = 0.05$ ). Agricultural manipulation of soil decreases SOM; likely decreasing Hg content. Increasing time since abandonment may provide a longer period of accumulation of both Hg from atmospheric deposition and SOM from plant growth.

Increased Hg concentrations and contents in forest vs. field sites may be due to several processes. The loss of organic matter with cultivation<sup>19</sup> may lead to leaching losses of Hg in these clay-poor soils. The higher concentrations of Hg in the organic layer of forests may be due to greater Hg deposition. Fitzgerald et al.<sup>2</sup> suggest that about one third of the total deposition to sites in central Wisconsin may occur as dry deposition, which would be highest on forest sites because of greater surface area of the canopy and greater interaction with air masses. Stomatal uptake of Hg<sup>0</sup> has also been shown to occur in forests,<sup>20</sup> leading to eventual incorporation in leaf litter. Harvest of agricultural crops, including hay, reduces Hg accumulation by this process.

### E. ANTHROPIC INFLUENCES

The data can be used to provide an estimate of the net amount of Hg that has been accumulated or lost from soils at CCNHA. Mercury in the soil samples with lowest SOM, those from the deep layer of the field to forest transects, did not differ with transect station (Table 2). The concentration of Hg in those samples was considered to be the background concentration for the soil parent material at CCNHA ( $7.7 \text{ ng g}^{-1}$ ,  $\text{SE} = 0.5 \text{ ng g}^{-1}$ ). This value is exactly the same as that predicted from a regional equation for background levels of Hg in soil based on silt-plus-clay content.<sup>5</sup> If that background concentration is subtracted from that of the surface mineral soil layer, the difference represents net Hg loss or enrichment in the surface. The result, when converted to mass and added to the mass of Hg in the organic layer, can be compared over the landscape.

If the samples are considered to simply represent three classes of vegetative cover—field, transition, and forest—then fields have the lowest net increase in Hg mass ( $0.30 \text{ mg m}^{-2}$ ) compared to the transition cover ( $0.74 \text{ mg m}^{-2}$ ) and the forest ( $2.38 \text{ mg m}^{-2}$ ;  $F = 21.35$ ;  $\text{d.f.} = 2,52$ ;  $\text{prob.} = 0.000$ ;  $\text{MSE}'' = 1.08 \text{ mg m}^{-2}$ ;  $\text{BLSD} = 0.73 \text{ mg m}^{-2}$ ). These net increases are the same magnitude as those we have reported for forested sites in a transect across the Great Lakes states, ranging from  $<0.5 \text{ mg m}^{-2}$  in forests in northwestern Minnesota to  $2.5 \text{ mg m}^{-2}$  in eastern Michigan.<sup>5</sup>

Although this difference in Hg increase can be strictly attributed to differences in SOM accumulation among the three cover types (field =  $2.71 \text{ kg m}^{-2}$  to 10 cm, transition =  $3.80 \text{ kg m}^{-2}$ , forest =  $8.44 \text{ kg m}^{-2}$ ), that is not a complete explanation. The stoichiometry of the soil organic matter in relation to Hg differs among the three cover types. The molar ratio of Hg to N, both constituents of SOM, is significantly higher in the surface mineral soil layer of the forest ( $1.49 \text{ } \mu\text{mole mole}^{-1}$ ) than in fields ( $0.95 \text{ } \mu\text{mole mole}^{-1}$ ), or in transition areas ( $1.07 \text{ } \mu\text{mole mole}^{-1}$ ) ( $F = 6.50$ ;  $\text{d.f.} = 2,52$ ;  $\text{prob.} = 0.003$ ,  $\text{MSE}'' = 0.51 \text{ } \mu\text{mole mole}^{-1}$ ;



B LSD = 0.37  $\mu\text{mole mole}^{-1}$ ). These differences imply that different processes are influencing the retention of Hg in those three systems.

#### IV. CONCLUSIONS

Historical land use has differentiated the upland landscape at CCNHA into fields and closed canopy forests. Although the upland soils have very uniform parent material, the fields contain lower amounts of SOM because of prior cultivation. As we hypothesized, these factors have influenced the distribution of Hg in soils across the landscape. The higher Hg concentrations in the organic layer and surface mineral soil in the forests, and the higher Hg/N ratios in that surface, may be due to higher Hg deposition related to the structure of the forest canopy. Higher Hg concentrations in the surface mineral layer of forests parallel higher levels of SOM, and suggest that Hg retention is controlled by interaction with decomposed, humified organic matter. Leaching losses of Hg associated with loss of SOM due to cultivation of these dry, sandy soils before abandonment is also likely. Controls on Hg deposition and retention in terrestrial ecosystems merit further investigation.

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