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# A Pilot Survey of Mercury in Drugs, Cosmetics and Household Products Using Reliable Analytical Methods

**Lian Liang[1*], John Gilkeson[2], Ed Swain[3], Elizabeth Bennett[1], Maya Li[1], Mei Deng[1], Patrick Pang[1]**

[1]Cebam Analytical, Inc., Bothell, USA; [2]Minnesota Pollution Control Agency, Resource Management and Assistance Division, St. Paul, USA; [3]Minnesota Pollution Control Agency, Environmental Assessment and Outcomes Division, St. Paul, USA.
Email: [*]liang@cebam.net

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## ABSTRACT

The concentration of mercury (Hg) was accurately determined in more than 228 drugs, cosmetics and household products manufactured in a variety of countries. Some drugs were found to contain up to 4424 ppb Hg, and some skin creams contained up to 2769 ppm Hg. Hg in skin creams was found to be almost 100% elemental Hg ($Hg^0$), a volatile species of Hg. $Hg^0$ can enter the human body through inhalation and skin absorption, potentially resulting in the serious consequence of mercury poisoning. The mercury can also volatilize, contaminating the surrounding air. Other people, for example, infants and children, who are close to or contacting the skin of the person using the cosmetics, can also absorb the mercury. Total mercury (THg) was determined by combustion/trap/CVAFS. Methyl mercury (MeHg) and inorganic mercury ($Hg^{2+}$) were determined by the ethylation based method. The emission of $Hg^0$ was determined by evaporation/trap/CVAFS. All analyses were performed in accordance with explicit quality assurance and quality control protocols and procedures.

**Keywords:** Mercury Speciation; Drugs; Cosmetics; Households; Reliable Methods

## 1. Introduction

As a well-recognized toxic metal [1], Hg has drawn attention from scientists all over the world to investigate its effects on the environment and human health. However, the research works focused on its biogeochemical cycling in which water, soil, air, biota were directly involved [2, 3]. Compared to cycling in environmental media, little attention has been paid to drugs and cosmetics. Because drugs are directly taken into human gastrointestinal system, and cosmetics are directly put on human skin, these products can affect human health more directly and immediately than Hg in media from the surrounding environment [4]. To protect human health and environment, accurately monitoring Hg in these products is necessary, and this research indicates that there is much work to be done.

Hg has a long history of use in many cultures as a readily available chemical that seems to have net benefits as a preservative or active ingredient in personal care products, and pharmaceuticals. However, these uses are now seen to be unnecessary, counterproductive, or better accomplished by substituting modern chemical agents that have fewer negative effects, are biodegradable, and do not have significantly greater cost [5]. Due to the persistence and toxicity of Hg and its compounds, government agencies should consider banning its use in human personal care and pharmaceutical products. Government agencies have repeatedly warned the public about the dangers [6-10]. However, the situation apparently has not been improved in recent years.

Some drug and biologic products are still being manufactured with Hg as a preservative, fungicide, or antiseptic, even though effective biodegradable non-Hg alternatives are available. Some high level Hg-containing anti-aging and skin lightening creams, lotions, and soaps are being produced by manufacturers from developing countries, and the availability of these products seems to be increasing in recent years [4]. These products rarely identify Hg as an ingredient and are distributed worldwide through various channels of commerce, marketed primarily to women. Since these products "work well",

[*]Corresponding author.

some women favor the products and it is difficult to persuade people not to use them. Most people who use these products don't know that they contain high levels of Hg, and that the Hg in these products can cause damage to the kidneys and nervous system and even interfere with the development of the brain in the unborn and in young children. Children can be exposed just by touching a parent or even a countertop contaminated with residual product. Some women even know how dangerous the products could be, and they still use them after weighing the "benefits" of "beauty" against the dangers.

Since most of these products are oil containing substances, analysis of these products for Hg and its compounds using traditional wet chemistry methods has been difficult as both analytes and media are volatile. This might account for the fact that governments issued warnings about some dangerous products but did not identify the mercury concentrations found in them. Even the manufacturers may not know exactly how much Hg is in their products. This can make it difficult for governments to establish necessary regulations for limiting and banning the use of Hg in these products. Any amount of mercury that is intentionally added is too much mercury. US FDA has established a limit of 1 ppm as the threshold for an "adulterated product."

A total mercury (THg) analytical method based on combustion/trap/CVAFS has been developed and published [11-13]. Using the method, samples including liquids, solids and gases were introduced into the system without the need for sample preparation. In the system, mercury compounds were decomposed at 800˚C into elemental Hg ($Hg^0$) which was then carried to a gold trap and collected onto the trap by amalgamation. $Hg^0$ collected on the trap was finally measured by CVAFS. In the past years, the method detection limit has been frequently determined and found to be around 0.1 ng/g for liquids and solids. The method has been successfully applied for determination of Hg in crude oils and related products, and the performance such as the accuracy and validity of the technique has been detailed in our previous publications [14-17]. In this pilot survey, this technique was used for determination of THg in the products, and ensured high quality results for the study.

## 2. Experimental

Except where specifically addressed below, equipment, materials, and methods used for this work are the same as those described in our previous publications. A BR(III) Hg analyzer (Brooks Rand, Seattle) was used for measurement of elemental Hg ($Hg^0$). A sensitive balance (Mettler, AT261, d = 0.01 mg) was used for weighing sample aliquots.

### 2.1. Determination of THg by Combustion/Trap/CVAFS Method

#### 2.1.1. The Setup of the System

The combustion/trap/CVAFS system has been described in our previous publications [13]. To suit various products, a combustion column with an 8 mm inside diameter was used which is larger than that (6 mm) used in our previous system. The working conditions are the same, *i.e.*, 800˚C for combustion, 20˚C to 800˚C for sample introduction, and the flow rate of carry air is 250 to 300 mL/min.

#### 2.1.2. Sample Introduction

Samples, liquid or liquid-like, were drawn and injected into the combustion column using syringes (Hamilton, Nevada, USA), while solid or solid-like samples were weighed into quartz glass boats using the sensitive balance, then the boats were inserted into the column for analysis (**Figure 1**).

#### 2.1.3. Calibration of Results and Quality Control

Standards, 10 ng/mL to 1000 ng/mL of methyl Hg (MeHg) as Hg prepared in toluene, were used for calibration depending on Hg concentration levels of samples. A 100 ppm Hg standard (Canostand™, USA) was diluted with toluene and used as the lab control sample (LCS) for liquid samples, while certified reference materials such as NIST1575a (Pine leaves), IAEA 142 (fish tissue), Dorm-2 (fish tissue), and NIST 2709 (soil) were used for solid samples. All samples were analyzed in duplicate, and related percent differences (RPD) between duplicate analyses were <10%, with most <5%. High Hg level samples were analyzed in multiple replicates. Recoveries of LCS were mostly between 95% and 105%.

#### 2.1.4. Analysis of High Hg Concentration Samples

Considering the representative of sample aliquots and the

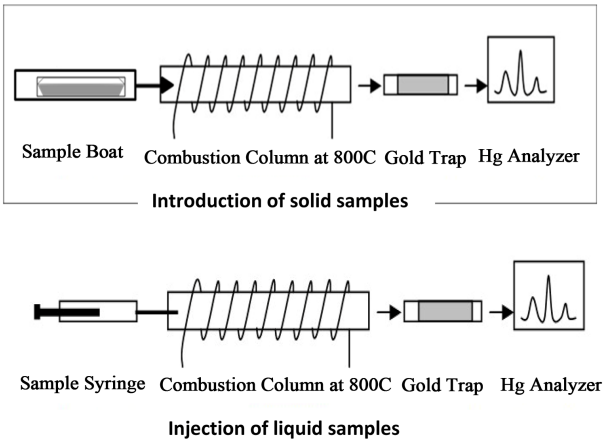


**Figure 1. Sample introduction.**

reliability of weights, generally the masses of the sample aliquots should not be less than 0.2 mg. Thus it is impossible to get high level samples analyzed by reducing the mass of the sample aliquots. For samples with Hg concentrations higher than 200 ppm, the $Hg^0$ loaded on gold traps was leached with concentrated $HNO_3$ in loosely capped vials at 75˚C in a water bath for 30 min. The leaching solutions were then analyzed for Hg by $SnCl_2$ reduction/amalgamation/CVAFS [18]. To reduce the cost for analysis, iodated charcoal (IC) traps (Cebam, Bothell, UAS, or equivalent) were used instead of gold traps for $Hg^0$ collection for high Hg level samples. The $Hg^0$ loaded on ICT was leached with concentrated $HNO_3$ in loosely capped vials at 80˚C in a water bath for 3 hours followed by $SnCl_2$ reduction/amalgamation/CVAFS [18] for analyzing Hg.

### 2.2. Speciation of Hg by Ethylation Based Method

Samples were extracted with alkaline solutions at 80˚C for 3 hrs in closed Teflon vials. Hg in aqueous phase extracts was then speciated using the ethylation based method described in our previous publication [19-21]. The published method is for simultaneous determination of methyl and inorganic Hg (MeHg and $Hg^{2+}$), but here $Hg^0$ was also collected onto a gold trap that was connected downstream of a Tenax trap (**Figure 2**). Thus, ethylated products of MeHg and $Hg^{2+}$ were collected onto the Tenax trap, while $Hg^0$ passed through the Tenax trap and was captured on the gold trap. MeHg and $Hg^{2+}$ on the Tenax trap were separated and quantified by GC and CVAFS detection as described by Liang, *et al*. [19, 20]. $Hg^0$ on the gold trap was measured by CVAFS against standards generated by $Sn^{2+}$ reduction [18,22]. Here, the measurement of $Hg^0$ should only be used for qualitative assessment of the species' presence because some amounts of $Hg^0$ may evaporate into the headspace of the vial during alkaline extraction at 80˚C for 3 hrs, and then escape to the air when the vial was opened.

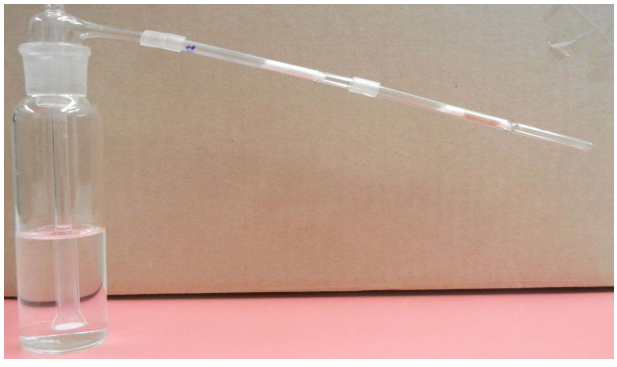

**Figure 2. Experimental set up for speciation of Hg using ethylation based method.**

### 2.3. Emission of $Hg^0$ from Skin Creams

Appropriate aliquots of cream samples were weighed on small pieces of tissue paper, and then the tissues were placed in glass vials. The vial has an outlet on the cap (**Figure 3**). A gold trap was connected to the outlet of the vial for collecting emitted $Hg^0$. The trap collected emitted Hg for 12 hrs at 20˚C, and then the trap was replaced with another clean gold trap for collection of another 12 hrs. This cycle can be repeated until $Hg^0$ is emitted completely. The time length of the periods and how many periods should be taken depended on Hg concentration, mass of aliquot, and the experimental purpose. The traps loaded with $Hg^0$ were measured for $Hg^0$ by CVAFS [18]. Results were calibrated by $Hg^0$ standards generated by $Sn^{2+}$ reduction and collected on gold traps [18]. If the purpose was to measure the total amount of $Hg^0$ emitted from an aliquot, and the estimated amount of $Hg^0$ was out of the linearity range of EPA 1631 method, an IC trap was used instead of the gold trap as described above (2.1.4) for collection of emitted $Hg^0$ for long enough until $Hg^0$ was emitted completely. The $Hg^0$ loaded on IC traps was determined using the procedure of 2.1.4.

### 2.4. Collection of Products

Products were obtained by donations from manufacturers and individual consumers, and purchased from shops. In total, 228 samples of various products manufactured in several countries including USA, France, Germany, and China (including Hong Kong) were collected and analyzed. Some of products are shown in **Figure 4**.

## 3. Results and Discussion

### 3.1. Results of THg

All samples were first analyzed for THg using combustion/trap/CVAFS method. Samples found to contain Hg >200 ppm were re-analyzed using the procedure described in the paragraph 2.1.4. Results are listed in **Table 1**. These formulated cosmetic products are homogeneous and therefore well suited to this analytical method. This ensured high quality results for samples analyzed using the method.

THg was found to be lower than 10 ppb in most prod-


**Figure 3. Experimental set up for emission of $Hg^0$ from skin creams.**

ucts analyzed, which is similar to the concentrations found in most crude oils processed in the United States [15]. In about 6% of samples, such as eye cosmetics and some drugs, THg ranged from 10 ppb to 50 ppb. These levels generally indicate that mercury has not been intentionally added to these products. Three skin cream samples were found to contain THg from 300 to 3000 ppm and two drug samples from 1 to 4.5 ppm (**Figure 5**).

**Figure 4. Some of products collected.**

## 3.2. Results of Hg Speciation in High Level Hg Skin Creams and Drugs

Only samples with high THg concentrations were analyzed for speciation. Five samples, three skin creams and two western/eastern mixed drugs were analyzed for species, MeHg, $Hg^{2+}$ and $Hg^0$, using the procedure in 2.2, and results are listed in **Table 2**.

For the three cream samples manufactured for the use of skin whitening/shining and anti-speckle, $Hg^{2+}$ was found to be about 2% to 3% of THg. The $Hg^{2+}$ is likely the fraction oxidized from $Hg^0$ during manufacturing, storage, or daily opening by consumers. These three creams had been opened many times by the consumers prior sending to the lab for analysis. Significant amounts of $Hg^0$ were found in three creams, indicating $Hg^0$ was the dominant fraction but this species could not be quan-


**Figure 5. High concentration Hg containing skin creams (left) and drugs (right).**

**Table 1. THg concentration ranges of various products collected and analyzed in this work.**

| Product Category | # of samples | THg concentration range, ppm | Regulation Limit (RL), ppm | Times of exceed RL |
|---|---|---|---|---|
| Body lotion | 16 | 0.0019 - 0.0054 | | |
| Liquid soap | 15 | 0.0004 - 0.0011 | | |
| Solid soap | 22 | 0.0008 - 0.0024 | | |
| Cosmetic Lotion | 23 | 0.0020 - **2.769**[*] | 1 (USFDA) | 2.769 |
| Medicinal Lotion | 26 | 0.0004 - 0.0064 | | |
| Skin Cosmetics | 19 | 0.0020 - 0.0150 | | |
| Eye Cosmetics | 24 | 0.0020 - 0.0494 | | |
| Lipsticks | 22 | 0.0026 - 0.0147 | | |
| Perfume | 8 | 0.0024 - 0.0028 | | |
| Hair Color | 7 | 0.0056 - 0.0097 | | |
| Dental Care Iodine | 1 | 0.0212 | | |
| Western drugs | 14 | 0.0012 - 0.0037 | No regulation limits established yet | |
| Western/eastern mixture medicine | 31 | 0.0020 - **4.424**[*] | No regulation limits established yet | |
| Total | 228 | | | |

Note, [*]: US FDA limit of 1 ppm applies to all products regulated under the US Food, Drug, and Cosmetic Act, except for the Act's allowance of 65 ppm Hg for eye-area cosmetics. This includes all but the last three product categories in this table. However, the 1 ppm limit does not apply to the last two product categories in the table. US FDA has published a list of product ingredients, including many Hg compounds, that have been approved in the past, but which are not allowed in new products "…since there are inadequate data to establish general recognition of the safety and effectiveness of these ingredients for the specified uses…" (23).

**Table 2. Results of Hg speciation in high level Hg drugs and skin creams.**

| Sample name | Hg, Mean ± SD, ppm | | | |
|---|---|---|---|---|
| | THg | $Hg^{2+}$ | MeHg | $Hg^0$ |
| Cream-A | 2124 ± 81 (n = 5) | 46.73 ± 2.29 (n = 3) | Not detectable | Significant amounts |
| Cream-B | 339.8 ± 12.2 (n = 4) | 8.840 ± 0.504 (n = 3) | Not detectable | Significant amounts |
| Cream-C | 2769 ± 21 (n = 3) | 80.31 ± 5.78 (n = 3) | Not detectable | Significant amounts |
| Drug-A | 1.235 ± 0.032 (n = 3) | 1.217 ± 0.075 (n = 3) | Not detectable | Not detectable |
| Drug-B | 4.424 ± 0.102 (n = 3) | 4.498 ± 0.103 (n = 3) | Not detectable | Not detectable |

tified here for the reasons described above (see paragraph 2.2.). It seems that these creams were likely manufactured by simply mixing $Hg^0$ in creams. MeHg was not detectable in any products analyzed. In addition to MeHg, an ethyl Hg containing compound (thimerosal) has been using as vaccine preservative, but no vaccine products were collected, thus ethyl Hg was not measured in this work.

Most products with elevated Hg have no ingredient labels and the manufacturer is not identified on the label. Generally these products were not traded legally in public markets. Consumers often purchased them "under the table" or through personal relationships. Government officials have been warning consumers not to use skin creams, beauty and antiseptic soaps, or lotions that might contain Hg. However, the use of these products has been increasing in developing countries, especially in Asian countries. The products are generally used by women with darker skin, for example, women in or from Africa, Middle East, Asia, and Central and South America. A USFDA web page shows similar products, but no exact Hg contents in these products were reported [7].

For the two drugs, the $Hg^{2+}$ was found to be identical to THg, and no MeHg/ $Hg^0$ were found. The two drugs were Western/eastern mixture medicine manufactured to alleviate the symptoms of colds and allergies, such as nasal congestion.

### 3.3. Results of Analysis of $Hg^0$ Emission from Skin Creams

An aliquot of 5.03 mg of cream A was taken for the $Hg^0$ emission experiment using the setup in **Figure 3**. $Hg^0$ was collected for 30 min on a gold trap, then a new trap was placed for another 30 min, and then a third trap was placed. The 3 traps were measured for $Hg^0$ and about 34 ng of Hg was found in each trap, with no significant difference in Hg amount, indicating $Hg^0$ emitted steadily over the time under the experimental conditions (**Figure 3**). According to this rate, $Hg^0$ contained in this aliquot needs about a week to be completely emitted. To simplify the analysis, an IC trap was then used to collect remaining $Hg^0$ for a week. The trap was then analyzed for Hg using the procedure described in 2.1.4. The sum of $Hg^0$ collected onto 3 gold traps and the IC trap was calculated to be about 11 μg, corresponding to 2187 ppm of $Hg^0$ in this sample, close to the amount of THg found by combustion/trap/CVAFS method in this sample (2124 ppm). The similarity of the two concentrations indicates that $Hg^0$ was emitted completely from the aliquot, and also confirmed the conclusion (**Table 2**) that Hg in this cream is almost 100% $Hg^0$. For cream B and C samples, only IC traps were used for collection of $Hg^0$ emitted from the sample aliquots, and the amounts of $Hg^0$ were also found to be close to THg determined by combustion/trap/CVAFS method in these samples, thus also confirming that Hg in these two creams is almost 100% $Hg^0$. Some studies conducted to date have found calomel ($Hg_2Cl_2$) in skin lightening creams [4]. In creams analyzed in this work, the presence of calomel could be ruled out since the $Hg^0$ concentrations were found to be equivalent to THg concentrations in these products.

According to the emission rate (5.03 mg cream emitting 34 ng of $Hg^0$ to air at 20˚C in a 30 min period), if a person applies 1 g of cream A on their face, then about 160 μg of $Hg^0$ would be emitted to air over 12 hrs. Meanwhile some of $Hg^0$ would be absorbed through the skin and further into the blood stream, posing a risk to human health. It is worth noting that the 160 μg of $Hg^0$ emission was estimated at 20˚C without air flowing, thus the actual amount at human face temperature with air flow and open environment should be higher than 160 μg.

## 4. Conclusion

Some drugs and cosmetics are found to contain high levels of Hg that are potentially toxic to consumers. These high Hg level products are manufactured in developing countries, but spread in the world through different legitimate and gray market/black market channels, making it difficult to regulate import and sale of the products. Consumers need to know that Hg has been added and is potentially toxic, and regulations need to be established to govern manufacturers, perhaps to ban the use of Hg in products.

Manufacturers and government regulatory agencies may need to test cosmetic and pharmaceutical products for Hg concentrations. Robust and cost-effective methods such as the methods used in this work should be employed to ensure high quality results. USFDA regulations already ban import and sale of these products but they get into the United States anyway. What mechanisms to ban worldwide manufacture and distribution would be effective? How can we effectively educate women about the danger of these products and change the idea that lighter skin is better? The desire for lighter skin drives the manufacture, sale, and use of these products.

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