Determination of Total Mercury in cosmetic consumers product, utilizing Direct Combustion in compliance with ISO 23674.

INTRODUCTION

Mercury and harmful substances have been found in cosmetics, often without proper labelling. Some creams claim to remove blemishes and lighten the skin using mercury's low cost and preservative properties. Because of these properties, mercury in its toxic form, methylmercury, can be added later to already manufactured products.

Despite its uses in cosmetic applications, mercury is highly toxic for human health, even at low concentration.

Long-term exposure can cause damage to the kidneys, liver and nervous system, leading to personality changes and loss of vision.

Due to its high toxicity, FDA and other regulatory bodies banned mercury in cosmetics products [1].

The International Organisation for Standardisation (ISO) has developed a method for detecting trace amounts of mercury in cosmetic [2]. This method involves the direct analysis of cosmetic samples without the need for sample preparation. It allows the direct quantification of the total amount mercury in both solid and liquid cosmetic samples. This has led to the introduction of ISO 23674, which focuses on direct mercury analysis techniques.

In this application report we demonstrate how Milestone DMA-80 *evo*[3] responds to the requirements of the ISO 23674.

| EXPERIMENTAL

INSTRUMENT

- Milestone DMA-80 *evo*
- Configuration Tricell Double-Beam

Figure 1 Milestone's DMA-80 evo Direct Mercury Analyzer

The configuration used was in fully compliance with ISO 23674 requirements.

METHOD

The DMA-80 *evo*'s method used for establishing the procedure by ISO and used in our internal study was as follows:

Drying Step	30s
	200°C
	300s
Decomposition Step	➢ 2 min at 250 °C ➢ 1,5 min from 250 to 650 °C ➢ 1,5 min at 650 °C
Purge Step	60s
Amalgam Step	12s
	850-900°C

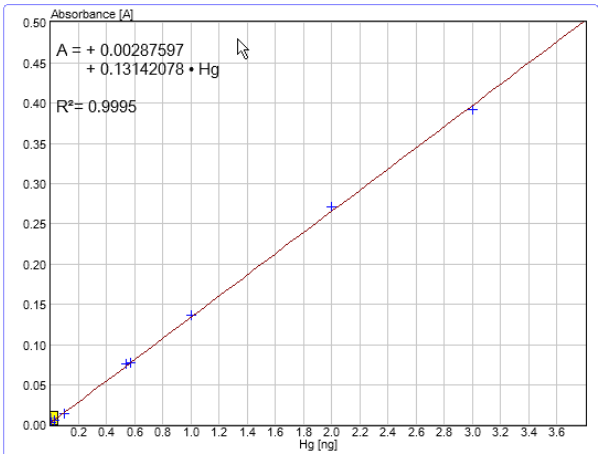
Table 1 Analysis Operating Parameters condition

CALIBRATION

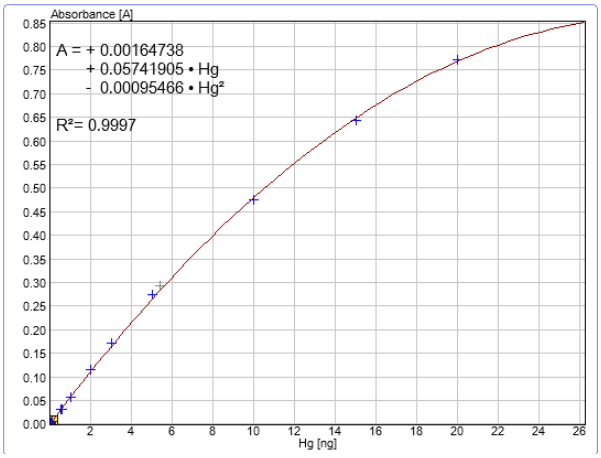
Liquid calibration standards can be prepared by using a commercially available solution of 1000 mg/L Hg, which is preserved in 5% HNO_3.

Working standards from 1 to 1000 ppb were prepared and preserved in 2% HCl, then stored in amber glass vials. Calibration curves were created by injecting increasing volumes of standards (from 1ppb to 1000ppb) directly into the quartz sample boats, thus covering the range of 0-500 ng of mercury. As the technique is matrix independent, calibration standards can be either liquid or solid.

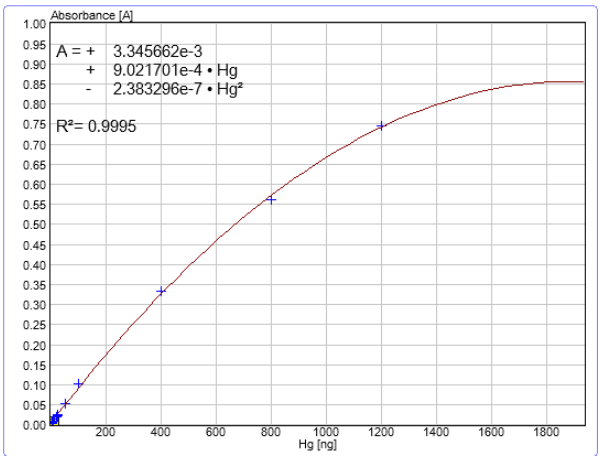
Cell 0 calibration curve:



Cell 1 calibration curve:



Cell 2 calibration curve:



| RESULTS AND DISCUSSION

ANALYTICAL PROCEDURE

This application report validates DMA 80-*evo*'s compliance with ISO 23674 through a recovery study. Samples were homogenized and introduced into quartz boats for analysis using the DMA-80. The focus is on testing the ISO 23674 method at lower concentrations to achieve a relative standard deviation within the range specified by the International Standards Organization.

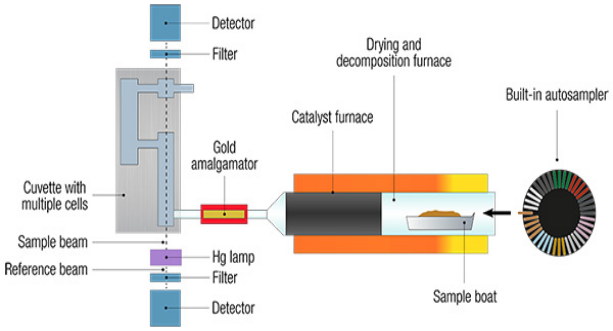


Figure 2 An Internal Schematic of Milestone's DMA-80 evo.

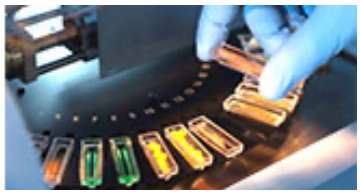
Samples are thermally decomposed in the oxygen/air flow of the instrument at temperatures between 650 °C to 900 °C. Combustion gases pass through a catalyst tube set at ~615 °C. The result of this step is the conversion of all mercury in its atomic form (Hg^0), while interferences are flushed out. Mercury atomic vapours are then enriched on a gold amalgamator and thermically released at 800°-900°C. Finally, the atomic vapours pass through a UV measuring cuvette system at the wavelength of 253.7 nm. The whole process takes less than 6 minutes per sample.

TESTS AND RESULTS

Foundation, Body Lotion and Shampoo samples underwent analysis in our internal lab, using the ISO 23674 method and adding 0.1mL of a 100ppb (10ng Hg) standard as a recovery control spike. At first, these samples were analysed to measure the mercury content in the commercial products.

Sample ID	S. weight (g)	Measured Hg (ng)	Mean Hg content (n=3) (ng)	Std Dev. (ng)	RSD%
Foundation	0.1014	0.3016	0.294	0.007	2.4
	0.1024	0.2912			
	0.1014	0.2881			
Body Lotion	0.1094	0.3271	0.338	0.010	2.9
	0.1109	0.3450			
	0.1098	0.3436			
Shampoo	0.1014	0.1146	0.110	0.002	1.9
	0.1018	0.1112			
	0.1009	0.1107			

Table 2: Summary of tests done on samples as is.



The Table 2 reports an average content of Hg of 0.294 ng for foundation, 0.338 ng for body Lotion and 0.110 ng for shampoo. Spiked samples were then analysed (Table 3).

Sample ID	S. weight (g)	Expected Hg (sample + 10ng spike) (ng)	Measured Hg (ng)	Recovery (%)	Measured Hg (n=3) (ng)	Std. Dev (n=3) (ng)	RSD% (%)
Foundation	0.1003	10.2895	10.4281	101.2	10.386	0.226	2.18
	0.1019	10.2941	10.1417	98.5			
	0.1009	10.2912	10.5886	102.9			
Body Lotion	0.1001	10.3080	10.3639	100.4	10.348	0.016	0.16
	0.1004	10.3089	10.331	99.9			
	0.0999	10.3074	10.3489	100.1			
Shampoo	0.1077	11.2144	10.2565	101.4	10.137	0.104	1.02
	0.1027	11.2094	10.0833	99.7			
	0.1042	11.2109	10.0703	99.6			

Table 3: Data for direct determination of mercury in spiked cosmetics sample by means of direct mercury analyzer DMA-80 by ISO 23674; Spiked standard of 10ng Hg

ISO enabled to validate the analytical methodology described in this standard for the ranges 0.15 mg/kg (ppm) to 1 mg/kg (ppm) (second ring test) and 0.5 mg/kg (ppm) to 2.5 mg/kg (ppm) (first ring test) with an acceptance limit of ±30 %.
Our study highlighted how it's possible to achieve even better results on cosmetics samples, respectively with an RSD% of 2.18%, 0.16% and 1.02% on spiked samples, adhering to ISO requirements and using the method detailed in the standard.

CONCLUSION

The results shown in Table 3 indicate that direct mercury analysis is an effective technique for the determination of mercury in cosmetics and similar products. Additionally, the results demonstrate the DMA-80 *evo*'s capability to analyse samples with exceptionally low levels of mercury while maintaining superior precision. Our experiments yielded RSD% safely adhere to the ISO standard boundary of a maximum of 20% RSD%. These findings increase our confidence in the instrument's accuracy and precision. The DMA-80 *evo* doesn't require sample preparation, resulting in lower detection levels than traditional techniques. This further emphasize its proficiency when dealing with cosmetic samples, notoriously of varying forms.

REFERENCES

- [1]: https://www.fda.gov/cosmetics/laws-regulations/prohibited-restricted-ingredients
- [2]: https://www.iso.org/standard/76615.html
- [3] Mercury Analysis http://www.milestonesrl.com
- [4] https://www.milestonesrl.com/download/newsletter/Technology%20report%20-%20Auto-blank%20feature%20-%20DMA-80%20evo.pdf

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