Análisis de Mercurio en productos cosméticos mediante el Analizador Directo Modelo Hydra II-C

Introduction

The toxicity of some mercury compounds as well as their ability to be absorbed through the skin has been long understood. Interestingly, mercury has been used as a skin bleaching agent and as a preservative in cosmetics for many years. Regulatory guidelines regard a cosmetic's mercury concentration as safe at less than 1ppm for products used around the mouth, or less than 65 ppm for products used around the eye[1].

The ***Hydra-C Direct Mercury Analyzer*** provides fast, simple and convenient analyses of these materials without sample pretreatment or production of hazardous chemical waste. The entire analysis takes only 5 to 10 minutes and virtually eliminates the need for the often cumbersome chemical digestion step required by other approaches. The Hydra-C employs EPA Method 7473 which has been approved for both laboratory and field analysis.

Instrumentation

The Hydra-C (shown to the right) is fully automated for unattended operation, comes complete with a 70-position autosampler and has onthe-fly loading capability for virtually unlimited sampler capacity. Hydra-C operates from a single 110/220V, 50/60 Hz power supply and oxygen supplied at 15-20 psig. All instrument operating parameters (e.g. furnace temperatures, gas flows, autosampler control) and process stages are computer controlled for ease-of-use.

Hydra-C Mercury Analyzer

Principle of Operation

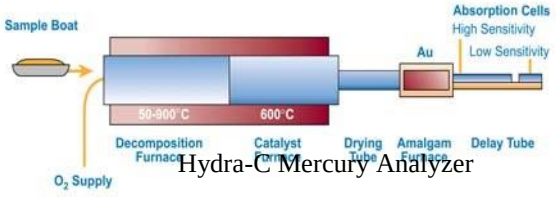


Figure 1. Hydra-C Principle of Operation

Hydra-C operates on the principle of thermal decomposition to liberate elemental mercury from solid or liquid samples. Figure 1 shows a schematic diagram of Hydra-C's principal of operation. First, a weighed sample is deposited into a sample boat and introduced into the decomposition furnace. After the furnace is closed, an oxidant (typically oxygen or compressed air) begins to flow over the sample and the furnace temperature is ramped in two stages; first to dry the sample, then to decompose it.

1. Source 21 CFR, Part 700

The analytical process typically involves combusting (thermal decomposition) the sample at high temperatures with oxygen; although, for some applications gentle heating of the sample in the air is adequate to release the mercury. During the combustion step the evolved gases are carried through a heated catalyst to produce free mercury while removing halogens, nitrogen oxides, and sulfur oxides. The remaining combustion products including elemental mercury (Hg) are swept first through a dryer and then through a gold amalgamation trap where all elemental mercury is captured. Following the decomposition step, the amalgamation trap is heated and the free mercury is carried into an atomic absorption spectrometer. The mercury level is reported using a wide dynamic range detection system that operates from 0.005 ng (its detection limit) to its upper limit of 1000 ng. For applications requiring significantly higher detection capability an optional high range detection system is available which can be used to analyze samples containing up to 20,000 ng of Hg.

Experimental

Samples of face powder, lipstick, face cream and nail polish were obtained from international sources. The samples were analyzed in triplicate using the Hydra-C.

Table I shows the instrument parameters employed for the samples analyzed using the Hydra-C. For this analysis nickel boats were used for all samples.

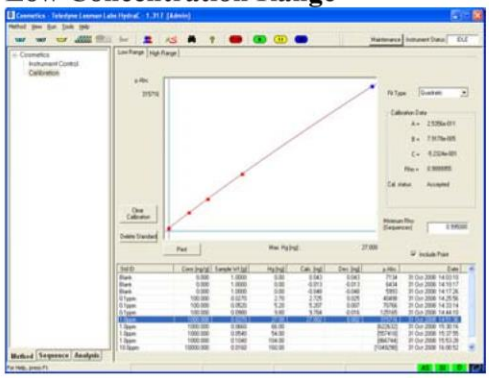
Table I: System Parameters

Parameter	Setting
Dry	300□C for 70 sec.
Decomposition	800□C for 350 sec.
Catalyst	600□C
Catalyst Wait Period	60 sec.
Gold Trap	600□C for 30 sec.
Measurement	100 sec.
Oxidant Gas Flow	350 ml/min (O_2)

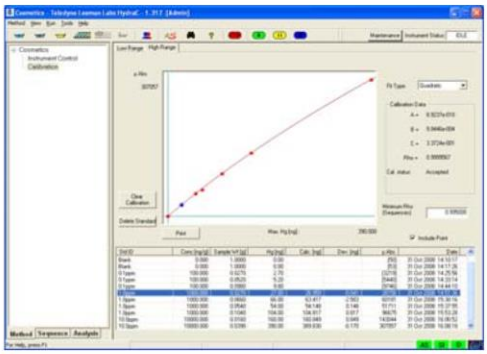
Calibration

Calibration was completed using aqueous standards prepared in 1.0% HNO_3 from Plasma-Pure 100 ppm stock Hg solution. Working standards were blank, 0.1, 1.0 and 10.0 ppm. Calibration plots are displayed as microabsorbance of Hg versus mass of mercury. The low calibration covers the 0-25 ng range and the high calibration covers the 25-400 ng range.

Low Concentration Range



High Concentration Range



Comparison with Cold Vapor Technique

For comparison with reference cold vapor techniques, each sample was analyzed in triplicate by cold vapor atomic fluorescence using the ***Teledyne Leeman Labs Hydra AF***. With the cold vapor technique the samples do require chemical pretreatment and we employed an ASTM digestion method. In this case, 8 ml of aqua regia was added and the samples were heated for 1 hour at 80°C. After cooling 40 ml deionized water and then 5ml potassium permanganate were added. The excess permanganate was then reduced using 1ml hydroxylamine sulfate/sodium chloride and brought to a final volume of 100ml prior to analysis.

Results

The results for both analyses are presented in Table 2.

Table 2. Sample results for both analysis methods in parts per billion (ng Hg/g)

Sample Name	Hydra-C analysis results	Replicate Standard Deviation	Digestion / Hydra AF results	Replicate Standard Deviation
US face powder	2.60	0.27	2.84	0.37
International face powder	4.68	1.03	4.23	0.25
International lipstick	1.13	0.35	1.44	0.84
International face cream	0.58	0.23	0.55	0.02
US nail polish	0.36	0.14	0.29	0.26
International nail polish	1.90	0.13	1.44	0.13

Conclusions

Hydra-C gives good results for the variety of cosmetic samples tested. All the samples had results well below the levels considered to be safe for use by the US FDA.. The results are comparable to those obtained using the digestion/cold vapor atomic fluorescence technique without the need for sample preparation.