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Determination of Mercury in Some Medicines by Flame Atomic Absorption Spectrometry Using Cold Vapour

Ki-Won Cha† and Sang-Gun Ha
Dept. of Chermistry, Inha University, Incheon 402-751, Korea
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Introduction

Mercury is a very poisonous element and used as medicine and steilizer. So, food, soil and our environment are widely contaminated with mercury. It is important to determine the trace amount of mercury.

Among the analytical method of the trace amount of mercury are ultraviolet spectrophotometry[1], gas chromatography[2] and atomic absorption spectrometry[3~7]. Mercury is a volatile element, so it is important to dissolve the mercury sample. There are two dissolving methods. One of them is V_2O_5 catalyst method and other is distillation method.

In this paper the mercury percentages of the medicine samples were determined by flame atomic absorption spectrophotometer and the mercury amounts dissolved in acidic solution like stomach acid solution were determined by flameless atomic absorption spectrophotometer.

Experiments

Reagents and Apparatus : All reagents used are extra pure reagent for trace amount determination of mercury and mercury Standard solution for atormic absorption method was prepared dissolving $HgCl_2$ in 1:1 hydrochloric acid solution. Water is demineralized water. Instrumentation Laboratory Model 257 is used as atomic absorption spectrophotometer with accessory of cold vaper cell analysis. Medicine samples were the sample collected in the medicine store.

Procedure of dissolving and determining mercury sample : Weight out 80 mg sample and transfer it to the flask A of *Fig.* 1. Connects the cooling water tube to condencer C and add 30m*l* of 1% sulfuric acid to absorber E. Through the D tube add 20m*l* of 1:1 sulfuric acid and heat the sample for 2 hours at 250°C.

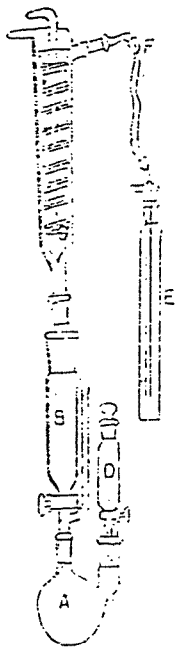

Fig. 1. Apparatus of sample decomposition

After the heating disconnecte the absorber E in order to resist the back current of the dilute sulfuric acid in absorber and cold the apparatus. Then reconstitute the absorber E and through the D tube, add 15m*l* of 3:1 mixture solution of nitric and perchloric acid. Heat again for 30 minute and get white fume. If the content of flask was vaporized almost, stop the heating and cold the flask. Here back current of dilute sulfuric acid in absorber is permited. Combine the flask solution and distilled solution in absorber E into 100m*l* mass flask and then dilute to the mark with water. Neublize this sample solution and $HgCl_2$ standard solution into air-acetylene flame alternately and measure the absorbance of those samples at 259.7nm using mercury hollow cathode. Correct the average values for background absorbance. Determine the mercury in the medicine by comparition with the standard curve.

Procedure of dissolving mercury sample in the acidic solution like stomach solution : Weigh out the medicine sample of the dose amount of medicine and powder it fine particle in the mortar and transfer to 1L mass flask. Add 2g sodium chloride and dilute to mark with 0.1M hydrochloric acid and stand up in 37°C water bath for 1 hour. We thought the above conditions as a similar stomach solution. Filter the solution and transfer 5m*l* of the filtrate to the cold vaper cell of *Fig.* 2 and add 0.5m*l* of 5% stannous chloride solution and stir the solution for 2 minute and pass nitrogen gas to vaporize mercury atom into light path. Measure the absorbance of mercury at 253.7nm.

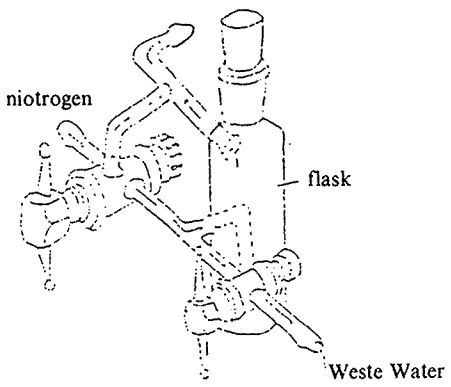

Fig. 2. Cold cell vapor analyzer

Results and Discussion

Calibration curve of mercury : Calibration curve of mercury was prepared as following. Transfer 2m*l*, 5m*l* and 10m*l* of mercury standard solution to sample decomposition apparatus and treat as descrived in the procedure of dissolving and determining mercury sample and measure the absorbance. The result is *Fig.* 3. As shown in this *Fig.* the relationship of absorbance and mercury concentration has linearity in the range of 0-100ppm. By using this standard curve, mercury percentage in medicine sample were obtained. This results are *Table* 1. As shown in *Table* 1 mercury percentage

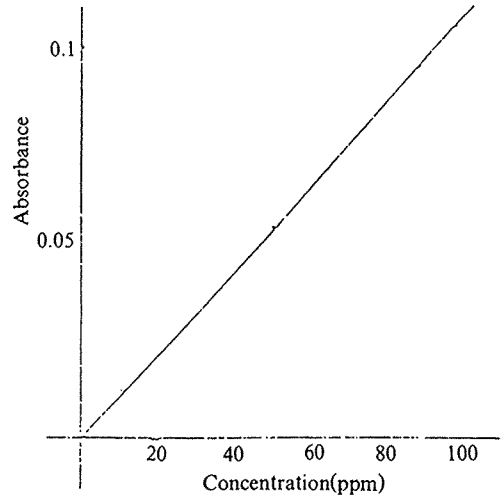

Fig. 3. Standard curve of mercury by flame mechod

Table 1. Analytical data of mercury in medicine samples

Sample \ Run No	1(%)	2(%)	3(%)	av(%)
Pill	3.13	3.16	3.25	3.18
Vermillion	5.11	5.68	5.61	5.46
Chung Sim Whan	1.56	1.59	1.52	1.56

of Pill sample is 3.18% and Chung Sim Whan has 1. 50%. This results have a good reproducibility in analytical error range but the average analytical data of Vermillion sample is 5.46% and three analytical values have poor reproducibility. This factor was interpreted as error in the diluting the sample solution.

In order to determine the mercury dissoved in acidic solution like stomach solution, calibration curve was prepared using flameless absorption spectrophotometry as directed for the procedure of dissolving mercury sample in the acidic solution like stomach solution. The result is *Fig.* 4. when *Fig.* 3 was Compared with *Fig* 4 the sensitivity of flameless absorption method has 1000 fold greater than that of flame absorption method. This means that the atomization procedure of flameless absorption method is as good 1000 fold as the flame atomization method. As shown in the *Fig.* 4 the relationship of absorption and mercury concentration has a linerity in the range of 0~1.0 ppm. This method was applied to determine mercury sample less than 0.1 ppm mercury solution.

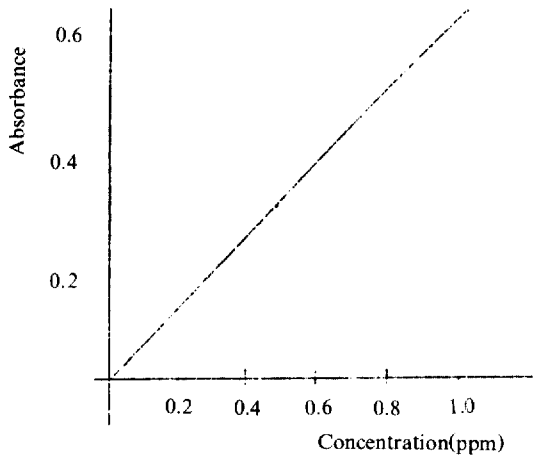

Fig. 4. Standard curve of mercury by flameless method

The analytical data of mercury in medicine dissolved in acidic solution like stomach solution was summarized in *Table* 2. In this table the amount taken is the dose amount of medicine. 2.4g Pill was dissolved in 1L acidic solution like stomach solution and the mercury content of this solution is 1.92 ppm while the mercury percentage is 3.18%.

Table 2. Analytical data of mercury in medicine dissolved in acidic solution like stomach solution.

Sample	Amount taken(g)	Data(ppm)	RSD
Pill	2.40	1.92	1.5
Vermillion	1.10	0.01	6
Chung Sim Whan	3.80	0.01	6
Mush-So Hap Won	1.20	0.01	6

Normal person takes 0.02mg mercury from the food everyday and 0.012mg mercury is driven out through urine[8]. In the medicine sample dissolved 0.01ppm mercury we will take 0.01mg mercury from the taking the medicine and this amount is less than mercury amount taken from food but in the Pill medicine sample 1. 92mg mercury will be dissolved and this amount is as much 100 fold as 0.02mg taken from food. If we eats this medicine the poison effect of mercury will be appeared.

Conclusion

1. Mercury percentages of medicine sample were determined using flame atomic absorption method after decomposing the sample in the decomposition apparatus.

2. Mercury amount dissolved in acidic solution like stomach solution was analyzed by flameless atomic absorption method after dissolving the sample in 1L solution of 0.1M HCl and 2g sodium chloride.

3. The sensitivity of flameless atomic absorption method is 1000 fold greater than that of flame atomic absorption method.

References

1. Japan analytical chemistry society, "Principles of pollution materials", Konglip(1972).
2. Reberf 1., *Nature*, **242**, 13(1973).
3. Frank D., *J. AOAC*, **56**, 378(1973).
4. Ernest A. E., *J. AOAC*, **49**, 792(1966).
5. Kolthoff I. A. and Stenger V. A. "Volumetric analysis" Vol. 11, Interscience Publisher(1947).
6. J. B. Willis, *Anal. Chem.*, **34**, 614(1962).
7. Lindstrom, *Anal. Chem.*, **31**, 461(1959).
8. Thomas A. and Gonzaless H. O., "Legal medicine pathology and toxicology", Applefon century crofts(1973).