CHAPTER V

DISCUSSION

The quantitative recovery of mercury through the proposed separation procedure was primarily checked using mercury-203 as a tracer. At least three determinations for each separation were performed. The results of these tests indicated the complete recovery of the tracer, as shown in Table 4.7 and 4.8. After these preliminary investigations the developed method was checked for reliability by analyzing some already certified Standard Reference Materials, having various varieties of sample matrices. The results were in good agreement with the certified values. (Table 4-11). The limit of detection under the conditions used was 0.005 ppm.

Actually, at the beginning of the study, two types of dry combustion process, in an oxygen flask and in a tube furnace, were first studied utilizing mercury-203 as the tracer. The results from both techniques showed the good recovery yield of mercury of 96 % with a relative standard deviation of $\pm$ 1.55 % for the first method compared with 97.3 % and $\pm$ 1.7 % for the latter. The advantages of using two methods were about the same, in consideration with the factors of, time of combustion, cost per analysis and equipment required. However, the disadvantages were found more in the latter case, since it required inactive mercury

as a carrier for preventing it from tube absorption which resulted the decreasing of the experimental reliability and sensitivity. Besides, the liquid nitrogen was required for the condensation of the mercury vapor. Consequently, the combustion in the oxygen flask was selected for this study.

Further studies on this technique showed that mercury was not remained in the ash although the incomplete combustion was indicated. In addition, mercury was never found in the rubber balloon and the wire basket.

The sensitivity of the developed technique could be increased by previous enrichment of the biological samples, either using freeze drying technique or drying the samples, at $45^{\circ}C$ for about 2-3 hours in normal oven or vacuum-oven. The first technique was tested by La Fleur (72) using Hg-203 as tracer and reported no losses of mercury was found. The latter was studied here also indicated no loss of mercury.

The proposed method was really fast, with using automatic shaker, a set of three samples and a standard could be accomplished in only one hour. It was also applicable for routine analysis and to various types of sample matrices as well. This method will eventually substitute atomic absorption spectrophotometry and neutron activation analysis, hence it is simple, inexpensive, fast and easier to automate, even less health hazards and more suitable for routine work in common chemistry laboratories.