CHAPTER IV

RESULTS

4.1. Determination of the effect of pH. Figure 4-1 showed the optimum pH range for mercury extraction by zinc dithizonate by plotting the activity of each organic phase against the pH of the solution. The result of the experiment was appeared in Table 4.1. It could be easily seen from Figure 4-1 that the suitable pH range for mercury determination was between 1 to 3.5.

4.2. Determination of the effect of zinc dithizonate chelating on mercury. Result of this experiment was shown in Table 4-2. The activity in count per minute for organic extract was plotted against the amount of zinc dithizonate, as showed in Figure 4-2. The volume of 4×10^{-5} % zinc dithizonate chelating with 0.6466 microgram of mercury was three millilitre.

4.3. Determination of the effect of time for reaching equilibrium. The result was shown in Table 4-3. Figure 4-3 showed the suitable equilibrium time for mercury extraction by plotting counts per minute of organic extract against time of shaking. It was indicated that the equilibrium time was reached after shaking for 30 seconds.

4.4. Determination of the selectivity of substoichiometric extraction of mercury. The data was tabulated in Table 4-4 and 4-5. It was shown that under the pH range 1 to 3.5, copper (II) both in micro and macro scale interfered the extraction while thallium (III) and thallium (I) would interfere only macro scale.

4.5. Determination of the effect of masking agent on substoichiometric extraction of mercury. The result in using EDTA as masking agent for copper (II), thallium (III) and thallium (I) was shown in Table 4-6. The result indicated that only 8 millilitre of EDTA solution was enough to mask copper (II), thallium (III) and thallium (I) both in micro and macro scale under the extract conditions.

4.6. Determination of experimental reproducibility of substoichiometric extraction of mercury. Result of this study was shown in Table 4-7. It was clearly indicated that the experiment was in good agreement and was well reproducible.

4.7 Determination of the reliability of substoichiometric extraction of mercury. The reliability was tested and the result was shown in Table 4-8. The average value of calculated mercury was 1.070 ± 0.030 microgram compared to the added value of 1.007 microgram. The investigation was therefore satisfactory.

4.8. Determination of the effect of amount of hydroxylamine hydrochloride on substoichiometric extraction of mercury. The data was presented in Table 4-9 indicating that only 4 millilitre of hydroxylamine hydrochloride was enough to reduce any oxidants occurring in the combustion step.

4.9. Determination of the reproducibility of the developed technique. The result of this experiment was shown in Table 4-10. It was clearly seen that the developed technique was satisfied.

4.10. Determination of the reliability of the developed technique. It was checked by the good precision and accuracy for analyses of the Kale Standard Sample, Orchard Leaves and Bovine Liver which contained a wide variety of elements, most of which are present in a large excess relative to the mercury content. The results were shown in Table 4.11.

Table 4-1. Effect of pH on the extraction of mercury with zinc dithizonate

pH	counts per minute
<1	502
1.0	54,284
1.5	53,957
2.0	54,641
2.5	54,788
3.5	54,239
5.0	53,874
6.5	52,369
8.0	49,231
9.0	45,824

Table 4-2 Effect of zinc dithizonate chelating on 0.6466 microgram of mercury.

ml. of 4×10^{-5} % zinc dithizonate	counts per minute
2.0	14,920
2.5	28,746
2.8	39,465
3.0	47,847
3.2	45,781
3.5	46,992

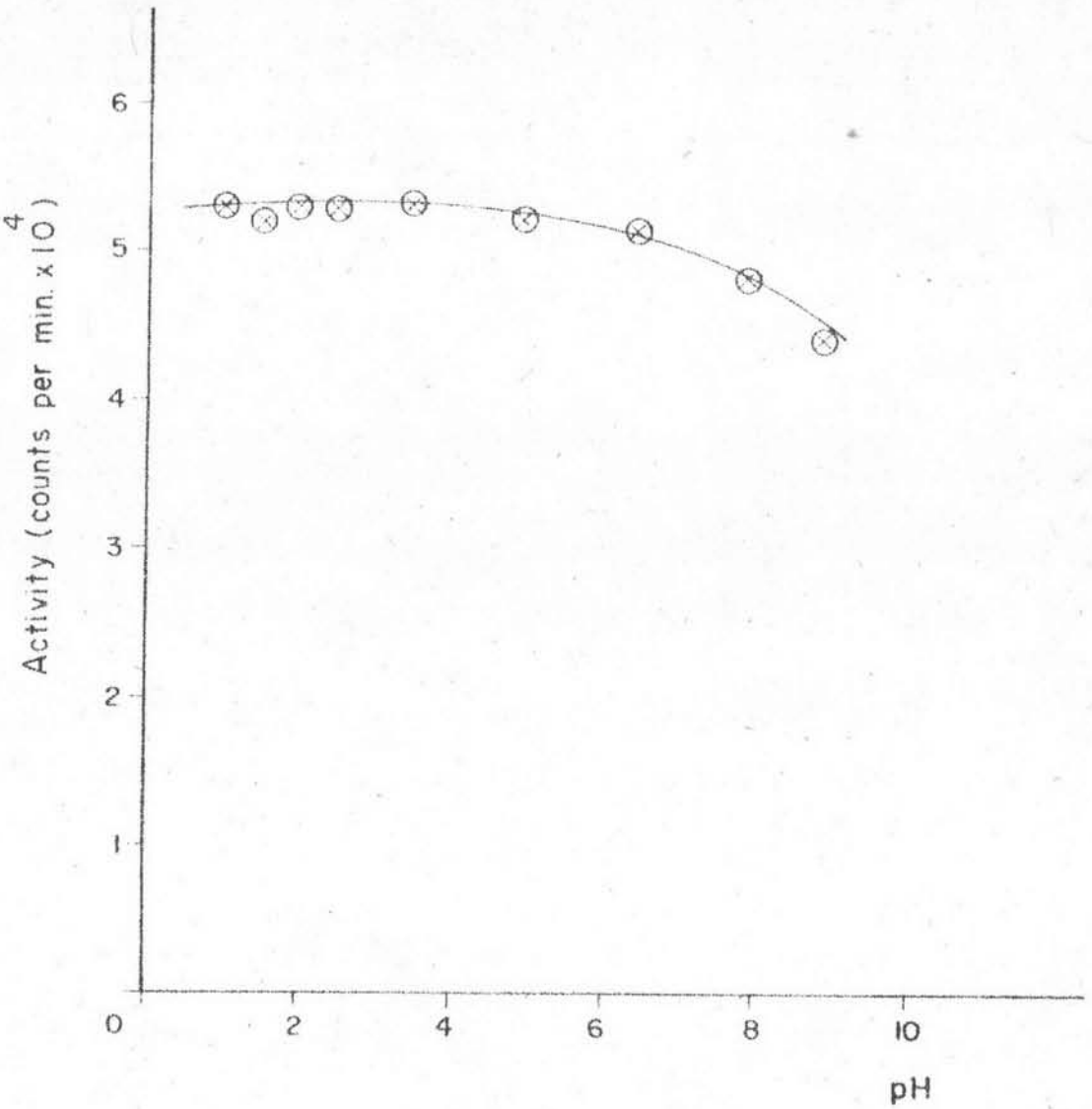

Figure 4-1 EFFECT OF pH ON THE EXTRACTION OF MERCURY WITH ZINC DITHIZONATE.

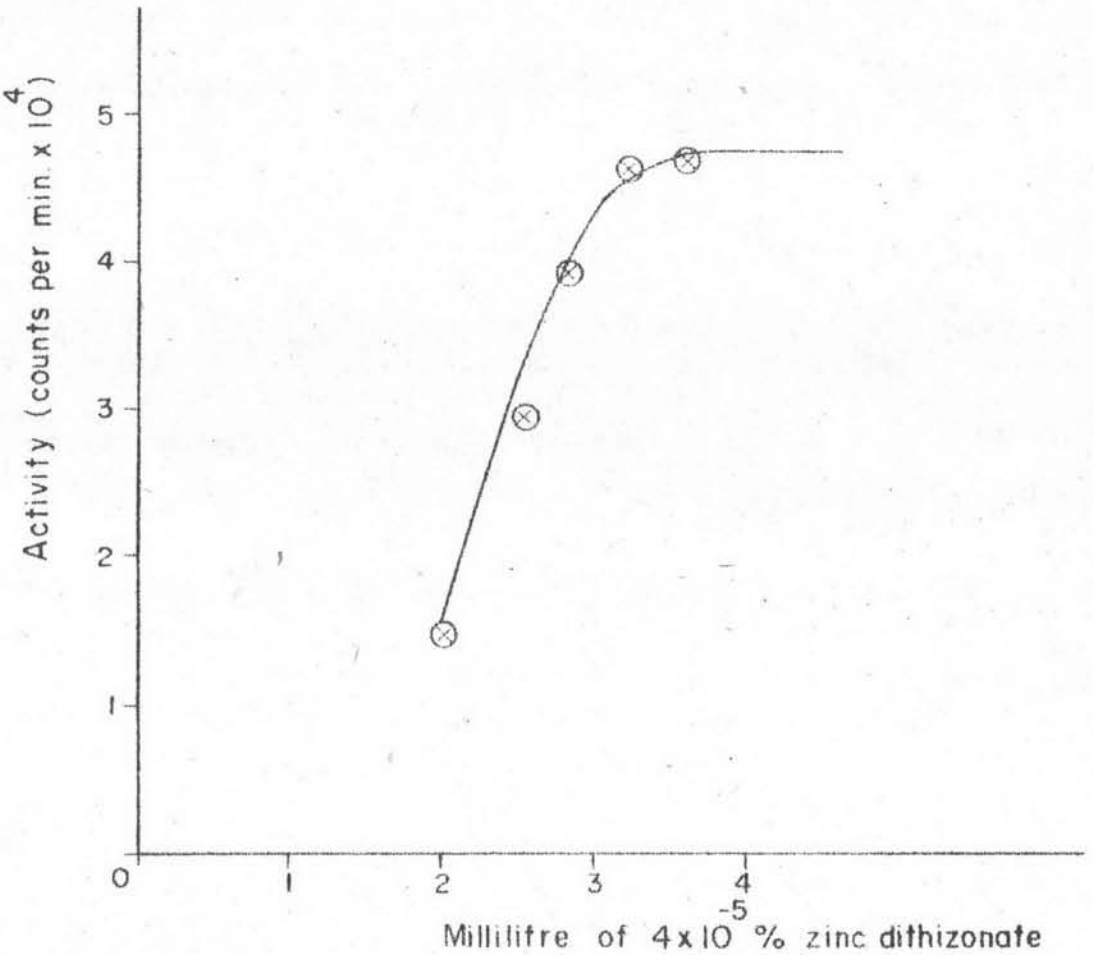

Figure 4-2 EFFECT OF ZINC DITHIZONATE CHELATING ON 0.6466 MICROGRAM OF RADIOMERCURY.

Table 4-3. Effect of time for reaching equilibrium on the extraction of mercury.

time of shaking sec.	counts per minute
10	39,273
15	40,834
20	50,941
25	54,233
30	54,385
40	53,784
50	54,825
60	54,429

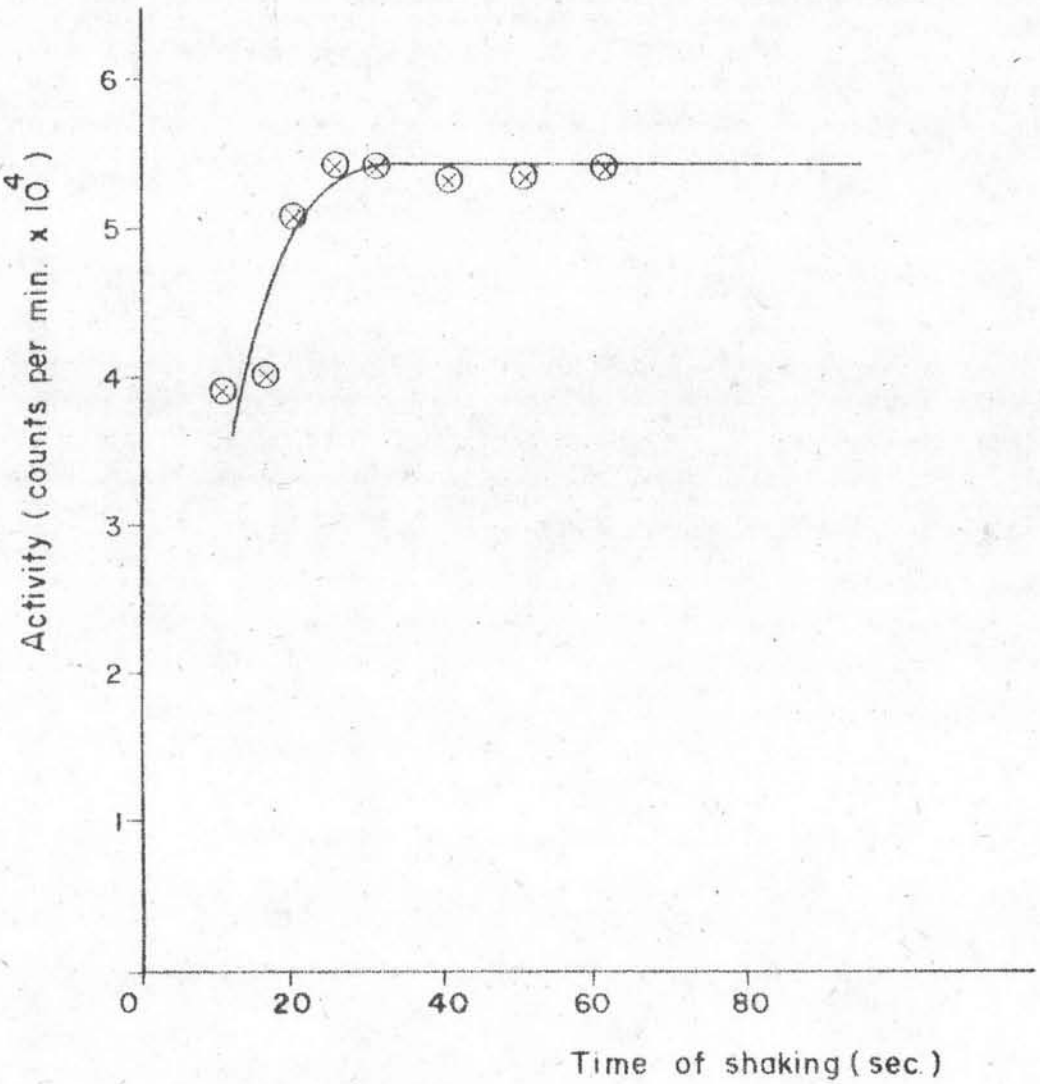

Figure 4-3 EFFECT OF TIME FOR REACHING EQUILIBRIUM ON THE EXTRACTION OF MERCURY

Table 4-4. Selectivity of substoichiometric extraction of mercury in the presence of micro-amount of different foreign metals.

Element Added	Activity isolated counts per minute
None	14,914
Co (II)	15,012
Mn (II)	14,831
Cu (II)	5,380
Ag (I)	15,070
Fe (III)	14,784
Cd (II)	14,919
In (III)	14,570
Pb (II)	14,650
Bi (III)	14,614
Ni (II)	14,683
Sn (II)	14,922
Tl (III)	14,809
Tl (I)	14,872

Table 4-5. Selectivity of substoichiometric extraction of mercury in the presence of macro-amount of different foreign metals.

Element added	Activity isolated counts per minute
None	14,914
Co (II)	14,532
Mn (II)	15,236
Cu (II)	1,832
Ag (I)	14,473
Fe (III)	14,536
Cd (II)	14,296
In (III)	14,418
Pb (II)	14,950
Bi (III)	14,287
Ni (II)	14,900
Sn (II)	14,385
Tl (III)	4,508
Tl (I)	3,137

Table 4-6. Effect of EDTA on selectivity of substoichiometric extraction of mercury.

EDTA added ml.	Activity isolated in the absence of interfering substances	Activity isolated in the presence of interfering substances counts per minute			
		Cu (II)		Tl(III)	Tl(I)
		amount of 200 µg.	amount of 10 mg.	amount of 10 mg.	amount of 10 mg.
None	14,914	5,380	1,832	4,508	3,137
5.0	14,837	14,740	9,382	7,354	9,121
8.0	14,925	14,811	14,628	14,632	14,542
10.0	14,929	14,728	15,011	14,677	14,288

Table 4-7. The experimental reproducibility of substoichiometric extraction of mercury.

Mercury added (μg)	Mercury founded (μg)
0.10	0.103±0.025
0.20	0.206±0.034
0.51	0.539±0.025
1.01	1.055±0.077
5.04	5.026±0.169

Table 4-8. The reliability of substoichiometric extraction of mercury.

Mercury added (μg)	Radiomercury added (μg)	$\frac{A}{A_x}$	Mercury calculated (μg)
1.007	0.6466	2.624	1.050
1.007	0.6466	2.729	1.118
1.007	0.6466	2.721	1.113
1.007	0.6466	2.516	0.980
1.007	0.6466	2.697	1.097
1.007	0.6466	2.639	1.060
		average	1.070±0.030

Table 4-9. Effect of hydroxylamine hydrochloride on the substoichiometric extraction of combustion product of mercury.

The activity of organic extract of radiomercury solution

(no combustion) = 28,151 cpm.

amount of 20 % hydroxylamine hydrochloride (ml.)	counts per minute
None	2,613
2.0	27,734
4.0	27,118

Table 4-10. The experimental reproducibility of the developed technique.

Mercury added μg	Mercury founded μg
0.208	0.215±0.026
0.300	0.265±0.021
0.520	0.438±0.024
0.832	0.765±0.038

Table 4-11. The reliability of the developed technique.

Types of sample	Concentration in one gram of sample (ppm)	
	Certified Value	Result of this work
Kale	0.1667 ± 0.023 *	0.1695 ± 0.003
Orchard Leaves	0.155 ± 0.015 **	0.1545 ± 0.024
Bovine Liver	0.016 ± 0.002 **	0.0141 ± 0.002

* Certified value from Dr. Bowen

** Certified value from NBS.