CHAPTER 5

DETERMINATION OF TRACE LEAD IN TOOTH PASTE

The most substances containing in tooth paste are calcium carbonate precipitated, glycerin, magnesium carbonate and flavoring. Heavy metals could be ones of the trace components that contaminate tooth paste during the production process. However, no publication has reported the method for determination of trace heavy metals in tooth paste. This is a contributing reason for the present study to investigate the technique of IR spectroscopy and anodic stripping analysis for determination of trace lead in tooth paste.

5.1 Decomposition of sample

Various methods were tried for dissolution of Pb (II) from tooth paste. First, the sample was treated with a few milliliters of concentrated HNO_3 and the mixture was heated to dryness. Thrice deionized water was added and the solution was warmed up. Residues were removed by filtration and the filtrate was concentrated to about 1 cm^3. The 2% Na-TMDTC solution was gradually added to the filtrate until precipitates occurred. The sulfide smell and black precipitates with instability in the solution were observed, the acid decomposition of Na-TMDTC can cause these problems. Though the precipitate was immediately filtered, its IR spectrum showed different absorption

peaks. Thus, these precipitates must not be $Pb(TMDTC)_2$.

Neutralization of the filtrate before addition of Na-TMDTC was also performed and white gelatinous precipitates were formed. These white gelatinous precipitates were filtered out. The filtrate gave no precipitate with 2% Na-TMDTC. Even preconcentration of the mixture was performed, no precipitate obtained.

Secondly, the tooth paste sample was treated with 20 cm^3 3 M HNO_3 and warmed up to aid the solution. Thrice deionized water was added to the mixture and the mixture was warmed and stirred for 30 minutes. The mixture was cooled down to room temperature and the residue was filtered out of solution (It took a day for this filtration). The filtrate was concentrated to about 1 cm^3 and was neutralized. The white precipitates formed were filtered out. The filtrate obtained gave no precipitate with 2% Na-TMDTC.

Thirdly, the treatment of sample with 20 cm^3 1 M HNO_3 was tried and the steps for precipitation of $Pb(TMDTC)_2$ were followed as mentioned above. No precipitate of $Pb(TMDTC)_2$ was resulted.

Finally, the method for determining lead in evaporated milk (11) was tried. The procedure was mentioned in 2.3.5. After addition of Na-TMDTC to the filtrate, preconcentration of the mixture was done, no precipitate obtained.

The reason for no $Pb(TMDTC)_2$ precipitate formation could come from the trace concentration of Pb(II) in tooth paste as well as the product of concentration of Pb(II) and TMDTC did not exceed the solubility product of $Pb(TMDTC)_2$. Thus, no IR spectrophotometric analysis of Pb(II) in tooth paste was performed.

5.2 Anodic stripping analysis

To prevent any adsorption process at the electrode surface, the decomposition of organic matters in tooth paste is necessary. Thus, the modifying method for determination lead in evaporated milk was chosen. The procedure for decomposition tooth paste, dissolution and determination of lead are mentioned in 2.3.5.

Six tooth paste samples were investigated. Four of them are from local production, there are Colgate (CT); Darkie (DAT); Denza (DET); and White Lion (WLT). The other two samples are Colgate DMF (CA), US production; and Vademecum Menthy (VMS), Swedish production. The large-size-tooth paste tube (ca. 150 g) which was enough for 6 trials (a 25 g sample/trial) was used. Thus, lead in a 25 g tooth paste sample was dissolved in a 25.0 cm^3 solution. A 15.0 cm^3 aliquot sample and a standard addition method were used for anodic stripping analysis. The conditions for anodic stripping analysis of lead in the samples are 20 minute-deposition in 1.00×10^{-5} M $Hg(NO_3)_2$ and 0.10 M in KNO_3 at -0.80 V and stripping Pb(Hg) out by anodic voltammetry, using the scan rate of 0.8 V/minute. The result of trace lead in tooth paste are listed in Table 9. To avoid high charging current effect, the sample solution was pH controlled at ca. 1.5. As seen in Table 9, lead in tooth paste of the local production ranges from 1.18 to 7.06 μg for a 100 g sample. The anodic stripping voltammograms of lead from 15 g DET sample as well as 4.00×10^{-7} M Pb(II) and lead from 15 g DET sample are shown in Figure 9.

Table 9 Data of anodic stripping analyses for lead (II) in tooth paste samples

Tooth paste sample	Date of purchase	Date of determination	$i_{p,a}^{a}$ sample, μa	$i_{p,a}^{a}$ sample+std Pb(II), μa	Conc. of std Pb(II) M added	μg of Pb(II)[b] in 100 g sample
CA	May 15, 76	June 17, 76	0	0.17±0.01	4.00×10^{-7}	0
	June 23, 76	July 26, 76	0	0.38±0.01	6.00×10^{-7}	0
CT	May 15, 76	May 21, 76	0.38±0.03	0.82±0.12	4.00×10^{-7}	7.06±0.31
	June 23, 76	July 15, 76	0.22±0.02	0.68±0.02	6.00×10^{-7}	6.01±0.37
DAT	May 15, 76	June 5, 76	0.18±0.01	0.55±0.01	2.00×10^{-7}	2.02±0.08
	June 23, 76	July 19, 76	0.05±0	0.49±0.02	5.00×10^{-7}	1.18±0
DET	May 15, 76	May 27, 76	0.29±0	1.00±0	4.00×10^{-7}	3.39±0
	June 23, 76	July 24, 76	0.25±0.05	1.04±0.09	4.00×10^{-7}	2.61±0.41
VMS	June 23, 76	July 24, 76	0.42±0	1.77±0.03	4.00×10^{-7}	2.63±0
	July 30, 76	July 31, 76	0.10±0	1.25±0.08	4.00×10^{-7}	0.67±0
WLT	May 15, 76	June 9, 76	0.05±0	0.21±0.03	2.00×10^{-7}	1.29±0
	June 23, 76	July 20, 76	0.10±0	0.60±0	4.00×10^{-7}	1.66±0

[a] average anodic peak current of three 15.00 cm^3 sample solutions ± mean deviation

[b] calculated by equation 5

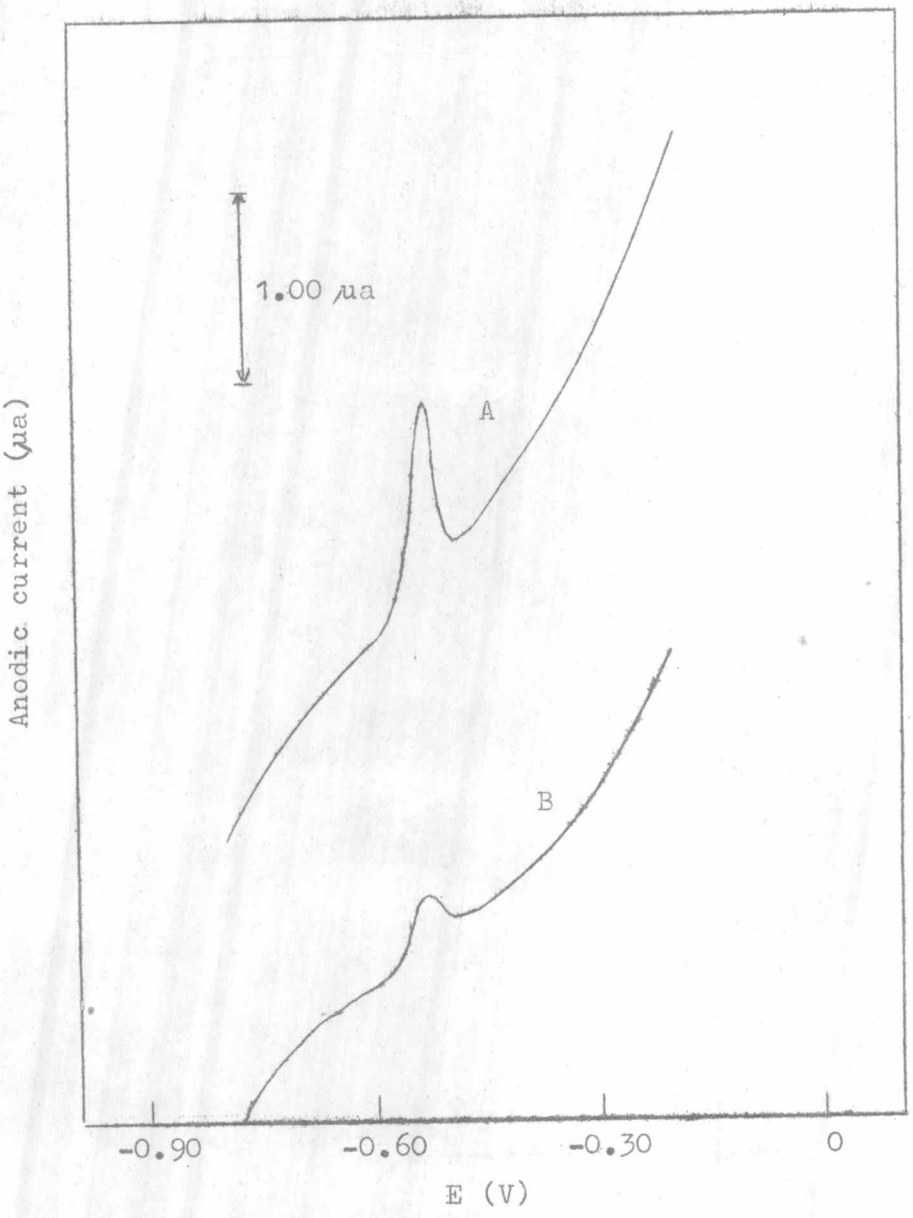

Figure 9 The anodic voltammograms of lead from 15 g DET sample (A) as well as 4.00×10^{-7} M Pb(II) and lead from 15 g DET sample (B), using the scan rate of 0.8 V/minute.

From two sets of tooth paste samples, difference in purchasing time, the amounts of lead in tooth paste samples from each manufacturer are different. Trace lead in tooth paste could come from the aluminum tube (container) and the impured substances used in making tooth paste. Though the amount of trace lead in tooth paste examined is not over the maximum permission value, it should be controlled. The maximum permission value for lead in tooth paste laid by the Ministry of Industry of Thailand is 200 µg for 100 g tooth paste (35) .