UNCON TROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC. ANALYTICAL CHEMISTRY METHOD ANALYTICAL AND PROCESS CHEMISTRY KOH FUSIONS Approved by: C. W. McVay, Manager Analytical and Process Chemistry

Part I

1.0 PURPOSE

Prepare potassium hydroxide fusions on samples in a controlled, reproducible manner for analysis by inductively coupled plasma spectroscopy (ICP).

2.0 APPLICATION

The potassium hydroxide fusion can be used on most solid samples to get them into a soluble form. A separate sample using an alternate digestion procedure must be used if potassium must be determined. Liquid slurry samples are dried in the crucible prior to the actual fusion.

3.0 DISCUSSION

Certain chemical species are very resistant to acid digestion, even under increased pressure. Using an alkali fusion is often an alternative method which will work. This is usually the method of choice for glass and feed samples if the use of hydrofluoric acid is to be avoided.

4.0 REFERENCES

PNL Standard Procedure PNL-SP-12, Rev. 0, 07/19/85.

Part II

5.0 EQUIPMENT

5.1 Muffle furnace or bunsen burner

<u>.</u>	5-89102400-36	2		Page 1 of 4	
	PDR PROJ	891025			
	11 32	PDC	, i		

ACM-FUSION-3801, Rev. 3 Effective Date: 08/02/89

5.2 Crucibles: nickel, zirconium, or graphite

- 5.3 Hot plate
- 5.4 Volumetric glassware
- > 5.5 Analytical Balance

6.0 REAGENTS

6.1 Potassium hydroxide, reagent grade

. . .

- 6.2 Potassium nitrate, reagent grade
- 6.3 Potassium oxalate solution (0.2 percent) Dissolve 2.0 g of potassium oxalate in distilled water and dilute to one litre. Label and prepare according to ACP 8.1. This will have a shelf life of one year.
- 6.4 ASTM Type II Reagent Grade Water

7.0 SAFETY

Standard laboratory safety practices should be followed. (See ACP 7.2)

8.0 RECORDS

•

All blanks should be run on the ICP and elemental values recorded in the QC book.

9.0 CALIBRATION AND CONTROL

A blank sample should be run with every set of samples or every 10 samples (whichever is less) to determine impurity level of the flux. See ACM-ICP-1001.

10.0 PROCEDURE

- 10.1 Finely ground or liquid slurry samples are placed in a crucible. A sample weight of 50 to 150 mg is used.
- 10.2 If necessary, dry the sample on a hot plate, being careful not to allow any spattering.
- > 10.3 Add \pm 0.05 g KOH to the crucible and melt the flux on a hot plate set on high.
 - 10.4 Complete the fusion by heating the sample on the bunsen burner or in the muffle furnace at $500 + 50^{\circ}$ C for about ten minutes.
 - 10.5 Cool the melt to room temperature.

BLB0453:ENG-362

Page 2 of 4

100 N

- 10.6 Leach the melt with 20 mL potassium oxalate solution for ten minutes. Transfer the sample to 100 mL disposable beaker, or other suitable container. Add 3 mL of nitric acid to the crucible to dissolve the residual fusion product. Transfer to the container with the leachate.
- 10.7 If necessary, gently heat to aid in dissolution. Add an additional 20 mL of potassium oxalate solution if the sample remains cloudy. Repeat the fusion if the solution will not clear.
- 10.8 Cool and transfer to a 100 mL volumetric flask. Add the internal standard, and dilute to the mark and mix. (See ACM-ICP-1001)

11.0 CALCULATIONS

N/A

: *

12.0 ATTACHMENTS

Attachment (Fusion Worksheet)

Page 3 of 4

ACM-FUSION-3801, Rev. 3 Effective Date: 08/02/89

ATTACHMENT A

Page ____ of ____

KOH FUSION WORKSHEET

SAMPLE NAME	LOG NUMBER
SPECIAL INSTRUCTIONS	
INSTRUMENTS USED	

>SAMPLE ID		 	 	 	l	
ELEMENT		[[[]	[[
(B) FUSED SAMPLE WT.(G)		! [l 1	[
(C) DIGESTED SAMPLE WT. (G)		 {	l l	l I	 	
(A) PPM FUSION		l	i 1			
CORRECTED PPM	 	l L	1	l L		

CORRECTED PPM - $\frac{C \times A}{B}$

ANALYST	DATE
APPROVED	DATE

.

.

•