PROCEDURE NUMBER	APPROVED	DATE
*12.10.3		06/21/83
VOLUME NAME: 12 CHEMISTRY PROCEDURES		
SECTION: 12.10 POST ACCIDENT SAMPLING AND ANALYSIS		
TITLE: *12.10.3 DETERMINATION OF CHLORIDES		

12.10.3.1 Purpose

This procedure describes a method of determining low level chloride concentrations on post accident liquid sample diluted 1 ml to 10 ml final volume.

12.10.3.2 Precautions/Prerequisites

A. Analysis are to be performed when conditions are ALARA.

B. Direct waste lines from the ion chromatograph to shielded container in a hood.

C. Dionex System 2 valves must be toggled to:

1. Separator 2

2. Suppressor 2

3. Eluent 2

D. Dionex System 2 flow rate should be approximately 3.0 mls/min.

E. HP 3390 Integrator Settings

1. Run Parameters setting

Zero = 0

ATT2 = 8

Cht. SP = 0.5

Pk. WD. = 0.40

Thrsh = 7

Ar Rej = 0

2. Report Optns Settings

RF UNC PKS = 0.0 E + 0

Mul Factor = Scale Setting from Dionex conductivity meter

Pk Height Mode Yes

Extend. RT No

RPRT UNC Pks Yes

3. Time TBL

20.00 STOP

F. Appropriate dose rate meters must be present and calibrated. Survey all equipment for exposure rates continuously during analysis and sample handling.

12.10.3.3 Reagents - (Prepare when required)

A. Eluent 2 - 0.0015 M $NaHCO_3$

Dissolve 0.5040 gms $NaHCO_3$ in one liter demineralized water then dilute to 4 liters. Label as per PPM 12.2.6.4.

B. Regeneration Solution - 1N H_2SO_4 to approximately 800 mls of demineralized water add slowly 111 mls concentrated sulfuric acid. Allow to cool to room temperature then dilute to 4 liters. Label as per PPM 12.2.6.4.

C. Chloride Standard - 10 ppb

From a stock 1000 ppm standard dilute serially to 10 ppb. Label as per 12.2.6.4.

12.10.3.4 Equipment

A. Fast run anion separator column

B. Anion suppressor

C. 3cc or larger, disposable syringes

D. Evacuated 14cc gas vial

12.10.3.5 Procedure

A. Fill a disposable syringe with air and attach to the system 2 injection port.

B. Place the load/inject switch to load.

C. Inject air into the sample loop to remove any liquid from the sample line.

D. Place the sample needle into the 10 ppb chloride standard solution.

E. Using the syringe, draw the standard into the sample loop until liquid is seen entering the syringe.

F. Enter the conductivity scale setting into the integrator multiplication factor. Push OP then 6, enter scale setting number, then press enter.

G. Simultaneously place the load/inject switch to inject and press the integrator start button. Place the load/inject switch to load after approximately 10 seconds.

H. After the chromatogram has run calibrate the integrator using the following steps:

Del Calib

Calib ESTD

Ref RTW

RTW

Cal 1 RT = (enter Cl retension time in minutes)

AMT = (enter Cl standard amount, 10 ppb)

ENTER

I. With the load/inject switch in load, flush the sample loop with demineralized water.

J. Flush the sample loop with air.

K. Place the sample needle into the post accident sample.

L. Enter scale setting times 10 into the integrator multiplication factor. Push OP then 6, enter conductivity meter scale setting times 10, then press enter.

M. Draw sample into sample loop until liquid is detected entering into the syringe.

N. Simultaneously switch load/inject switch to inject and press integrator start. Switch load/inject to load position after 10 seconds. The integrator will report ppb Cl.

O. Remove syringe and dispose of as high level radioactive waste.

P. Carefuly remove the syringe needle from the sample vial and insert into the 14 ml evacuated vial. Dispose of vial as high level radioactive waste after excess liquid has been drawn from the sample room.