Quemetco,Inc.

March 21, 2017

South Coast Air Quality Management District
21865 Copley Drive
Diamond Bar, CA 91765-4182

Attention: Mr. Ian MacMillan
               Planning and Rule Manager

Subject: Update to Rule 1402 Risk Reduction Plan
               Quemetco, Inc. (SCAQMD ID Number 008547)

Mr. MacMillan:

In response to your letter dated February 16, 2017, Quemetco, Inc. (Quemetco) is providing this update to its Rule 1402 Risk Reduction Plan which was originally submitted on November 16, 2016. This update addresses the items listed in the February 16, 2017 letter as well as our discussions during our February 17, 2017 meeting and our conference calls held on March 2, 2017 and March 10, 2017.

Arsenic Emission Limit:

Quemetco appreciates that South Coast Air Quality Management District (SCAQMD) agrees that the proposed arsenic limit of 6.50 pounds per year limit, based on a 30-day rolling average, as monitored by the Cooper Environmental Services (CES) Xact 640 continuous emissions monitoring system (CEMS) achieves a cancer burden at least 10% below the SCAQMD Rule 1402 threshold.

In-Stack Multi-Metal CEMS and QA/QC:

As SCAQMD notes in its February 16, 2017 letter, SCAQMD staff conducted a pilot study of CES' Xact 640 at Quemetco during 2015 and verified that the levels detected are within acceptable limits. In May, 2015 SCAQMD staff reported to the Governing Board that the preliminary results of the pilot study were "promising". SCAQMD has further indicated its confidence in CES' XRF technology by authorizing the expenditure of $290,000 for the purchase of two of CES' Xact 625 ambient metals monitoring systems (see Attachment 1) at its June, 2016 Governing Board Hearing.

Recordkeeping:

Except during periods of calibration, maintenance and testing, including RECLAIM CEMS maintenance which involves the WESP stack flow monitor, the Xact 640 will collect and analyze arsenic concentrations in the WESP stack on an hourly basis. The observed arsenic concentration will be used in conjunction with the corresponding flow recorded by the WESP stack flow monitor to calculate the hourly arsenic emission rate. The calculation will be performed using the Data Acquisition and Handling System (DAHS) Quemetco utilizes for NO_x and SO_x RECLAIM recordkeeping. Hourly arsenic emission rates accumulated over 30-day periods will be averaged and compared to a 7.42×10^{-4} pounds per hour average emission rate, which equates to an annual emission rate of 6.50 pounds per year.

Details regarding the Daily Upscale Calibration Drift Check, Daily Zero Calibration Drift Check, Daily Flow Calibration Drift Check, Quarterly XRF Audits and Annual Dynamic Spiking QAG RATAs are described in the Operation and Maintenance Manual prepared by Cooper Environmental Services. The Operation and Maintenance Manual is included as Attachment 2.

Daily Upscale Calibration Drift Check: This check evaluates the Xact 640's response to a standard once per day. During this check, a rod containing metals is inserted into the analysis area and the stability of the analyzer response is evaluated. The Xact 640 must report a value that is within 15% of the known value of the upscale standard to pass this daily check.

Daily Zero Calibration Drift Check: This check evaluates the Xact 640's response to a zero concentration produced by sampling a section of filter tape that has not been exposed to stack gas. The Xact 640 passes this evaluation if the result of the zero drift check for arsenic is less than 20% of the applicable emission limit. As specified by SCAQMD in its February 16, 2017 letter the arsenic emission limit applicable for this evaluation is 0.00114 pounds per hour.

Daily Flow Calibration Drift Check: This check evaluates the performance of the Xact 640's daily sample flow sensor and quality assurance flow sensors. The Xact 640 passes this evaluation if daily sample flow sensor and quality assurance flow sensor are less than 20% of the full scale value of the flow measurement.

Initial and Quarterly XRF Audit: An initial XRF audit was performed by SCAQMD and CES during the 2015 demonstration program. The first quarterly audit performed after final issuance of the permit revision including the Risk Reduction Plan provisions will also be considered the initial XRF audit. This audit evaluates the performance of the Xact 640 against a traceable-to-NIST thin film standard. SCAQMD has not specified a passing criterion for this evaluation. A passing criterion of 10% of the known concentration is proposed. CES points out that the arsenic thin film standard (made from a non-stoichiometric amalgam of GaAs) can slowly volatilize over time, changing the actual concentration of arsenic on the standard. The sensitivity of arsenic is validated at the time of instrument calibration using the physics of the X-ray Fluorescence response (i.e. sensitivity is a uniformly varying function of atomic number). The stability of the calibration can be checked by monitoring the response of another element analyzed in the same analysis condition. CES recommends evaluating a copper standard to validate the arsenic calibration.

Initial and Annual RATA Using the QAG: A series of QAG audits were performed by SCAQMD and CES during the 2015 demonstration program. The first annual RATA using the QAG performed following final issuance of the permit revision including the Risk Reduction Plan provisions will also be considered the initial RATA using the QAG. The dynamic spiking procedure requested by SCAQMD to fulfill this requirement includes challenging the Xact 640 at different spiked concentration levels with reference aerosols produced using CES' Quantitative Aerosol Generator (QAG). The Xact 640 reported concentration is plotted against QAG spiked concentration and a linear least squares regression analysis is performed. The Xact 640 is evaluated based on the slope, intercept and correlation coefficient. The Xact 640 passes this evaluation if the slope of the best fit line is between 0.85 and 1.15, if the correlation coefficient is 0.9 or greater and if the intercept is less than 20% of the applicable limit. Consistent with SCAQMD's specification in its February 16, 2017 letter, the arsenic emission limit applicable for this evaluation is 0.00114 pounds per hour.

Additionally:

1. Number of Concentration Levels – Spiking shall include a minimum of three concentration levels (not including a blank).
2. Specified number of data points at each concentration level – A minimum of five data points at each concentration level will be established.
3. Independent validation of solution concentration – The concentrations of the solutions used in the QAG will be independently analyzed for arsenic by a third party.
4. Procedures for data removal – CES performed dynamic spiking testing as part of the multi-metal CEMS demonstration program. During this testing the Xact 640 alternated between sampling unspiked stack gas (the background) for a sample period and QAG spiked stack gas for a sample period. The average in-stack background concentration for arsenic was subtracted from the Xact 640 reported arsenic concentration and this value was compared to the known QAG concentration. During time periods where the stack concentration was high relative to the spiked concentrations it was difficult to obtain agreement between the Xact 640 and the QAG. To account for this, CES recommends establishing a criterion for removing data where the in-stack background concentration is high relative to the spiking concentration. Consistent with the SCAQMD demonstration program, the removal of up to 25% of data points where the in-stack background concentration exceeds 20% of the spiked concentration will be allowed.
5. Spiking range – A spiking range from 1.0 micrograms per dscm to 5.0 micrograms per dscm will be employed.
6. Time frame to perform annual RATA – the annual dynamic spiking RATA will be performed during the same test mobilization required to perform the annual Rule 1420.1 arsenic compliance test. The notification provided to SCAQMD regarding the Rule 1420.1 compliance test will be deemed notification for the purposes of this RATA.

Reporting:

As requested by SCAQMD, Quemetco will update its website to include the 30-day arsenic average hourly emission rate with an accompanying indication of the 7.42×10^{-4} pounds per hour arsenic emission rate proposed in the Risk Reduction Plan. Quemetco will provide SCAQMD with a daily transmission of arsenic emissions measured by the Xact 640 through the existing RECLAIM reporting system. If SCAQMD's RECLAIM reporting system cannot accept the daily arsenic report, Quemetco will provide a monthly report of arsenic emissions as recorded by the Xact 640 to SCAQMD.

Availability Calculation:

The Xact 640 availability will be calculated on an ongoing basis with the first reported value 30 days after the facility permit is issued indicating approval of the Risk Reduction Plan by SCAQMD and incorporating the Risk Reduction Plan conditions in the permit. The percent data availability of the Xact 640 arsenic monitoring analyzer will be established according to the following procedures:

i. Calculate on a daily basis a rolling percentage of the reverberatory furnace operating hours that the Xact 640 was available for the period
ii. Record on a daily basis the percent concentration monitor availability using the following equation:

$$W = Y/Z \times 100\%$$

where:

W = the percent rolling Xact 640 availability

Y = the rolling operating hours for which the Xact 640 provided quality-assured data.

Z = the rolling operating hours of the reverberatory furnace.

The Xact 640 is guaranteed by CES to achieve 95% or greater up time. The parameters accumulated to develop the availability calculation will not be collected during the following periods:

- Xact daily Detector Energy Alignment.
- Xact daily flow and XRF checks.
- Interruptions necessary to perform any required Xact 640 QA/QC function.
- Xact 640 maintenance activities, including tape changes.
- WESP flow monitor interruptions associated with any required QA/QC function, maintenance or SCAQMD-required RECLAIM testing activities.

The 95% uptime requirement shall begin 30 days following final issuance of the permit revision including the Risk Reduction Plan provisions.

Contingency Plan:

Should Xact 640 availability fall below 95%, for each hour that the availability calculation is below 95% the average arsenic emission rate from the previous Rule 1420.1 arsenic compliance test shall be substituted in lieu of the arsenic emission rate calculated using the Xact 640. . The Rule 1420.1 arsenic emission rate result is subject to review by SCAQMD and is already used for compliance purposes. Data substitution will be discontinued once the availability returns to 95% or greater.

Should the Xact 640 fail any QA/QC check described in the Risk Reduction Plan, CES will be required to perform the maintenance necessary to correct the failure. For each hour that the QA/QC failure persists, the average arsenic emission rate from the previous Rule 1420.1 arsenic compliance test shall be substituted in lieu of the arsenic emission rate calculated using the Xact 640. The Rule 1420.1 arsenic emission rate result is subject to review by SCAQMD and is already used for compliance purposes. Data substitution will be discontinued once the Xact 640 passes the failed QA/QC check.

Description of Arsenic Reduction Measures:

Since the end of 2013 Quemetco has undertaken several measures which may have the potential to reduce arsenic emissions from the WESP:

- A redundant cooling tower was installed to service the WESP. This redundant cooling tower is reflected as Rule 219 exempt device E175 in the existing facility permit.
- The original WESP heat exchangers were replaced with larger units.
- The original WESP transformer/rectifier units were replaced with more energy efficient units that provide a more consistent voltage profile.
- The procedure for adding arsenic to refining kettles has been updated and is reflected in form 061-QCL-FRM-089 (ARSENIC ADD).

If you have any questions regarding this information. Please contact me at (714) 625-7020.

Sincerely,

Michael R. Buckantz

Michael R. Buckantz
Manager, Environmental Technical Support

Cc: Scott Bevans, Quemetco, Inc.

Attachments:

Attachment 1:   SCAQMD Purchase Approval for Cooper Environmental Xact 625 Monitors
Attachment 2:   Xact 640 Operation and Maintenance Plan Prepared by Cooper Environmental Services

ATTACHMENT 1

SCAQMD PURCHASE APPROVAL FOR
COOPER ENVIRONMENTAL XACT 625 MONITORS

BOARD MEETING DATE: June 3, 2016   AGENDA NO. 5

PROPOSAL: Recognize and Transfer Revenue and Appropriate Funds for
                 Monitoring Programs and Lab Analysis, Issue Solicitations and
                 Execute Contract and Purchase Orders for Field and Laboratory
                 Equipment, and Transfer Funds for Enhanced Particulate
                 Monitoring Program

SYNOPSIS: U.S. EPA has allocated Section 103 funds in the amount of
                 $731,010 for the PM2.5 Program. This action is to recognize
                 revenue and appropriate funds into Science and Technology
                 Advancement's FY 2015-16 Budget for the PM2.5 Monitoring
                 Program (as well as carry over any unexpended funds into the FY
                 2016-17 Budget), issue an RFP, execute a contract, and issue
                 purchase order for field and laboratory equipment. These actions
                 are to also transfer and appropriate $375,000 into Science and
                 Technology Advancement's FY 2016-17 Budget, re-issue an RFQ
                 and issue purchase orders for field and laboratory equipment.
                 Finally, this action is to transfer $175,000 into Science and
                 Technology Advancement's FY 2015-16 Budget between Major
                 Objects to realign expenditures for the FY 2015-16 Enhanced
                 Particulate Monitoring Program.

COMMITTEE: Administrative, May 13, 2016; On the motion of Chairman Burke,
                 the Committee unanimously approved all agenda items to be
                 forwarded to the full Board.

RECOMMENDED ACTIONS:
1. Recognize and appropriate upon receipt up to $270,010 awarded by U.S. EPA for
    the PM2.5 Monitoring Program into Science and Technology Advancement's FY
    2015-16 Budget ($461,000 was previously included in Salary and Employee
    Benefits within the FY 2015-16 Budget) as set forth in the attachment.

2. Appropriate any funds not expended by June 30, 2016, into Science and Technology
    Advancement's FY 2016-17 Budget, Services and Supplies and/or Capital Outlays
    Major Objects (Org 47).

3. Issue RFP #P2016-23 for Laboratory PM2.5 Weighing Room HVAC System
    Upgrade as part of the FY 2015-16 PM2.5 Monitoring Program.

4.   Authorize the Executive Officer to execute a contract, based on the results of RFP
    #P2016-23, for up to $65,000 for the Laboratory PM2.5 Weighing Room HVAC
    System Upgrade as budgeted in the attachment and described in this letter.

5. Authorize the Procurement Manager to issue a purchase order with Thermo Fisher Scientific, Inc. in an amount not to exceed $60,000 for the purchase of three PM2.5 continuous Federal Equivalent Method (FEM) monitors as budgeted in the attachment and described in this letter.

6. Transfer and appropriate $290,000 from the BP ARCO Settlements Projects Fund (46) and $85,000 from the Rule 1309.1 Priority Reserve Fund (36) into Science and Technology Advancement's FY 2016-17 Budget (Org 44), Capital Outlays Major Object, for the purchase of field and laboratory equipment.

7. Authorize the Procurement Manager, in accordance with SCAQMD Procurement Policy and Procedure, to:

   a. Issue a sole source purchase order in an amount not to exceed $290,000 from Science and Technology Advancement's FY 2016-17 Budget (Org 44), Capital Outlays Major Object, for two multi-metal ambient monitors as part of the Air Toxics Monitoring Program;

   b. Re-issue an RFQ for a thermogravimetric analyzer for the laboratory; and

   c. Issue a purchase order, based on the results of the RFQ, for a thermogravimetric analyzer in an amount not to exceed $85,000 from Science and Technology Advancement's FY 2016-17 Budget (Org 44), Capital Outlays Major Object.

8. Transfer $175,000 into Science and Technology Advancement's FY 2015-16 Budget from Salaries and Employee Benefits Major Object (Org 44), Salaries Account, to Services and Supplies Major Object (Org 47), Temporary Agency Account, to realign expenditures for the FY 2015-16 Enhanced Particulate Monitoring Program.

Wayne Nastri
Acting Executive Officer

MMM:LT:JCL:cv

Background

PM2.5 Program

Since 1998, U.S. EPA has provided funds under a Section 103 Grant for a comprehensive PM2.5 Air Monitoring Program. To date, there are 21 ambient SCAQMD monitoring stations operating 23 Federal Reference Method (FRM) PM2.5 monitors under U.S. EPA funding and 14 Federal Equivalent Method (FEM) PM2.5 continuous monitors. In addition, U.S. EPA has supported the expansion of the network to collect continuous PM2.5 mass and chemical speciation at several sites within the South Coast Air Basin. This augmentation substantially adds to the fine particulate data which will help in the characterization of PM2.5 sources, current air quality conditions and health impacts.

Multi-Metal Ambient Monitors
In May 2014, the Board approved the acquisition of one ambient near real-time metals monitor for the Air Toxics Monitoring Program from Cooper Scientific Instruments using $290,000 transferred from the BP ARCO Settlement Projects Fund (46) into that fiscal year's budget. This acquisition was subject to successful completion of a technology demonstration indicating satisfactory performance of the monitor. In 2014 and 2015, staff rented and deployed one of these multi-metal Xact 625 ambient monitors to conduct a six-month instrument evaluation at Quemetco, Inc. and TAMCO/Gerdau to assess its performance. Measurements from the rented Xact 625 multi-metal monitor were compared to measurements from FRM and established method measurements at the two facilities, and the Xact measurements compared well with the two techniques. Based on the performance results, staff had intended to move forward with the acquisition. However, subsequent to issuance of a purchase order but prior to equipment delivery, Cooper Environmental Services, LLC, submitted a revised quote for two (2) Xact 625 multi-metal ambient monitors at a discounted cost of $289,567 including sales tax.

Thermogravimetric Analyzer
In July 2015, the Board approved issuance of RFQ #Q2016-10 for a new thermogravimetric analyzer for laboratory testing. The Board also approved the transfer and appropriation of $85,000 from the Rule 1309.1 Priority Reserve Fund (36) into the current fiscal year budget to purchase the thermogravimetric analyzer based on the results of the RFQ. However, while three bids were received, all were deemed unresponsive because the proposers failed to provide requested technical details and/or necessary product references.

Enhanced Particulate Monitoring Program
SCAQMD has been providing enhanced particulate monitoring support including sample collection as part of a national monitoring program since 2003 and will continue for the foreseeable future.

Proposal
PM2.5 Program
SCAQMD staff anticipates a U.S. EPA award of $731,010 in Section 103 Grant funds for the continuation of the PM2.5 Program through March 31, 2017. This action is to recognize revenue up to $270,010 and appropriate funds, upon receipt, into Science and Technology Advancement's FY 2015-16 Budget ($461,000 was previously included in Salary and Employee Benefits within the FY 2015-16 Budget) as set forth in the attachment. U.S. EPA concurs with staff's proposed allocation. Any funds not expended by June 30, 2016, will be appropriated into Science and Technology Advancement's FY 2016-17 Budget.

Issue RFP

The U.S. EPA Section 103 Grant award includes one-time funding of $65,000 for the purchase of an HVAC upgrade to the SCAQMD laboratory's PM2.5 weighing room. Per 40 CFR Part 50, Appendix L, there are specific temperature and humidity criteria that are required for conducting PM2.5 pre- and post-weighing operations in an environmentally controlled room. In a continued effort to improve the reliability of SCAQMD's PM2.5 laboratory analysis, an upgrade to the HVAC system and humidifier will improve day-to-day stability of the environmental controlled weighing room and increase the cooling capacity of the HVAC system. This would provide longer term reliability and performance. This action is to issue RFP #P2016-23 to solicit formal bids, in accordance with SCAQMD Procurement Policy and Procedure, for the purchase of an HVAC upgrade.

Execute Contract

Laboratory PM2.5 Weighing Room HVAC System Upgrade
Based on the results of the above RFP, this action is to execute a contract for an HVAC system upgrade in an amount up to $65,000 to help enhance existing analytical capabilities. U.S. EPA concurs with staff's proposed expenditure. This item is budgeted in the Proposed PM2.5 Program Expenditures for FY 2015-16 (attached).

Issue Purchase Order

Three PM2.5 Continuous FEM Monitors
The U.S. EPA Section 103 Grant award includes one-time funding of $60,000 for the purchase of three FEM PM2.5 continuous monitors. Many of the FEM continuous monitors in SCAQMD's PM2.5 Air Monitoring Program have been in operation since 2001 and are in need of replacement. On October 4, 2013, RFQ #Q2014-02 was released in accordance with SCAQMD's Procurement Policy and Procedure, and Thermo Fisher Scientific, Inc. was chosen as the successful bidder at the conclusion of the evaluation process. Thermo Fisher Scientific, Inc. has agreed to honor the last price from that RFQ process. Since the SCAQMD's Procurement Policy and Procedure allows purchases based on a prior bid or last price, this action is to issue a purchase order with Thermo Fisher Scientific, Inc. for three FEM PM2.5 Monitors in an amount not to exceed $60,000 as budgeted in the Proposed PM2.5 Program Expenditures for FY 2015-16 (attached).

Multi-Metals Ambient Monitors

In light of the revised substantially lower quote submitted by Cooper Environmental Services, LLC, staff proposes to issue a new purchase order to acquire two (2) Xact 625 multi-metal ambient monitors in an amount not to exceed $290,000 including sales tax. This action would also include the transfer and appropriation of $290,000 from the BP ARCO Settlement Projects Fund (46) into Science and Technology Advancement's FY 2016-17 Budget (Org 44) to facilitate this purchase out of next fiscal year's budget. The $290,000 does not include the cost of the two mobile trailers that will be required to

house and deploy the multi-metal ambient monitors. An appropriation for the two mobile trailers will be requested in a separate Board action next fiscal year.

Thermogravimetric Analyzer
As a result of the nonresponsive bids, staff proposes to have the RFQ re-issued to seek new bids for the thermogravimetric analyzer. Thermogravimetric analysis (TGA) is used to determine compliance of metal working fluid and lubricant samples with Rule 1144. It is also useful in determining the volatility of various compounds and products. The lab has a TGA instrument; however, the instrument isn't functional for long periods of time. The current TGA instrument breaks down frequently and is costly to repair, often requiring that it be sent to Germany. The repairs, in addition to being costly, remove the instrument from the laboratory for months at a time. This action is to authorize the Procurement Manager, in accordance with SCAQMD Procurement Policy and Procedure, to re-issue the RFQ.

Furthermore, this action would include the transfer and appropriation of $85,000 into the Science and Technology Advancement FY 2016-17 Budget (Org 44) from the Rule 1309.1 Priority Reserve Fund (36). And finally, based on the results of the above RFQ, this action is to issue a purchase order for a thermogravimetric analyzer in an amount up to $85,000 from Science and Technology Advancement FY 2016-17 Budget (Org 44).

Enhanced Particulate Monitoring Program
The SCAQMD already received and recognized funding from the U.S. Government for the ongoing Enhanced Particulate Monitoring Program for FY 2015-16. Revenue in the amount of $2,836,157 for this grant was previously included in the FY 2015-16 Budget. This action is to transfer $175,000 in Science and Technology Advancement's FY 2015-16 Budget from Salaries and Employee Benefits Major Object (Org 44), Salaries Account, to Services and Supplies Major Object (Org 47), Temporary Agency Account, to realign expenditures for the FY 2015-16 Enhanced Particulate Monitoring Program.

Sole Source Justification
Section VIII.B.2 of the Procurement Policy and Procedure identifies four major provisions under which a sole source award may be justified. Cooper Environmental Services, LLC, is the only manufacturer of the Xact 625 multi-metal ambient monitor. Consequently, the request for sole source purchases through Cooper Environmental Services, LLC, is made under Section VIII.B.2.c(1): The unique experience and capabilities of the proposed contractor or contractor team. Additionally, Section VIII.B.2.d(6) of the SCAQMD's Procurement Policy and Procedure allows for sole source purchases in which: "Other circumstances exist which in the determination of the Executive Officer require such waiver in the best interests of the SCAQMD. Such circumstances may include but are not limited to: Projects requiring compatibility with existing specialized equipment."

ATTACHMENT 2

XACT 640 OPERATION AND MAINTENANCE PLAN
PREPARED BY COOPER ENVIRONMENTAL SERVICES

Cooper Environmental Services Xact 640 Operation and Maintenance Program for Quemetco City of Industry Facility

Cooper Environmental Services, LLC

9403 SW Nimbus Ave

Beaverton, OR 97062

March 21, 2017

1. Introduction
1.1. Document Description

Quemetco, Inc., has proposed using the Xact 640 multi-metals continuous emissions monitoring system (CEMS) as a part of Rule 1402 risk reduction plan. Under the proposed plan the Xact would be monitoring for arsenic to ensure that Quemetco is meeting its new lower emission limit for that element. This document outlines the instrument operation and maintenance program at Quemetco's City of Industry Facility

1.2. Xact Theory of Operation

The Xact 640 continuously collects samples of smokestack emissions on reel-to-reel filter tape and then analyzes the filter deposit in real-time by nondestructive X-ray fluorescence (XRF) analysis for metal concentrations. The Xact determines metal concentrations through two basic steps: first, the volume of the sampled gas collected is measured, and second, metals mass in the sample is quantified by XRF analysis. The flow diagram for the basic Xact 640 operation is shown here.
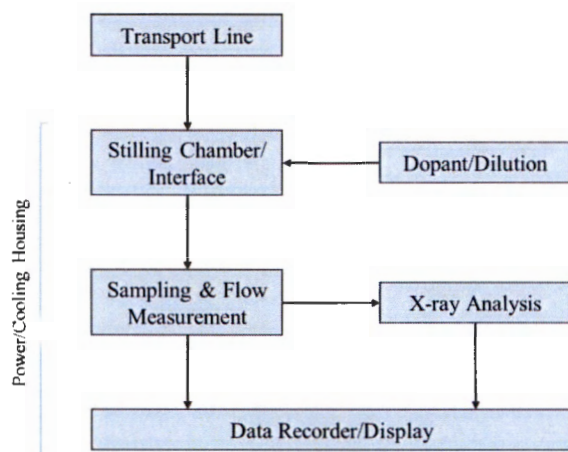
The air from the Quemetco Wet Electrostatic Precipitator (WESP) stack is drawn through the temperature controlled inlet into the sampling component at approximately 200 lpm for subsampling. Approximately 1.4 lpm subsample of gas is then taken in isokinetic manner and the remaining flow is returned to the stack as exhaust. The particulate and vapor phase metals are then deposited onto the reactive filter tape and the total sample flow through the tape is measured using a mass flow sensor. At the start of the next

sampling period, the filter tape with mass deposit is advanced into the XRF analysis chamber where it is analyzed for metals mass while the next sample is simultaneously collected on the filter tape.
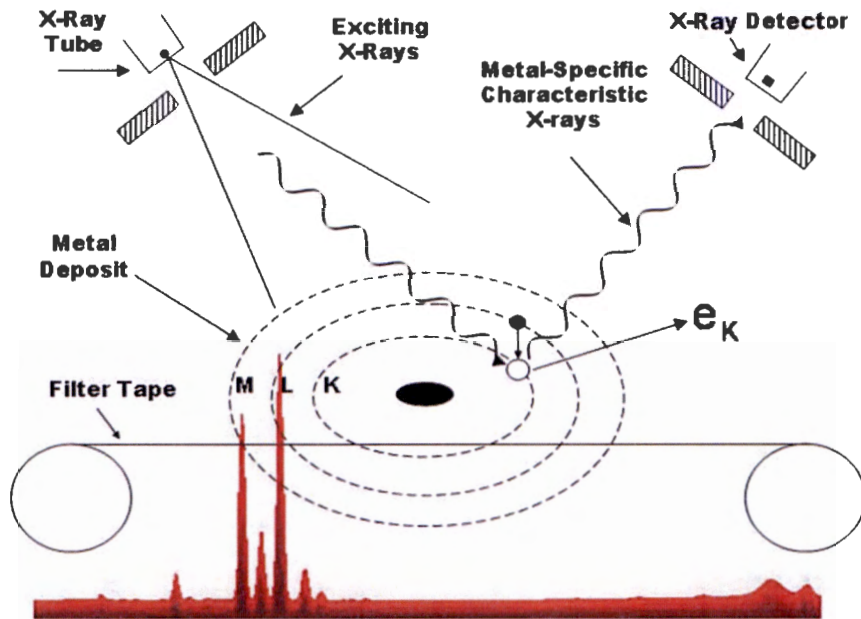
The figure above describes the metal detection principle of the XRF technology. In the XRF chamber, the X-ray tube emits high energy X-rays that bombard the filter tape deposit. The metal atoms in the tape deposit are excited by the incoming radiation and emit X-rays with energies characteristic of the elements present in the sample. These sample X-rays are detected and the resulting pulses are processed by a digital pulse processing unit. The digital pulse processor relays the counts/channel/second to a Spectrum Analysis and Quantitation (SAQ) software package located on the computer. Each spectrum, plotted as intensity versus energy, is interpreted by the SAQ software's least-squares fitting package to determine the metals contributing to the spectral peak intensities of the sample deposit. This spectral deconvolution process uses multiple reference spectra stored in an electronic reference spectra library to fit the unknown spectrum.

The quantitative calibration information - generated during routine calibration typically performed once every year - is used to correlate the intensity to the metals concentration. The spectral deconvolution program (XRS-FP) employed by the Xact requires the

acquisition of reference spectra for every element of interest as well as interfering elements that could contribute to an unknown analysis spectrum. Each element has a characteristic peak shape arising from different energy transitions within the inner shell electrons. XRS-FP uses a library of these characteristic elemental peak shapes to identify the amount of intensity each element is contributing to an unknown spectrum. Each intensity identified relates to a finite amount of a particular metal within the analysis area.

The metal concentrations are reported in $\mu g/dscm$ (C_i) as the ratio of XRF-determined metal mass (M_i) to the volume of the sample (V). Concentrations of the measured metals are automatically recorded at the end of the analysis period (same as the set sampling period). Concentration data can be immediately viewed at the instrument and/or saved on the computer as a comma delimited text file.

2. Operation
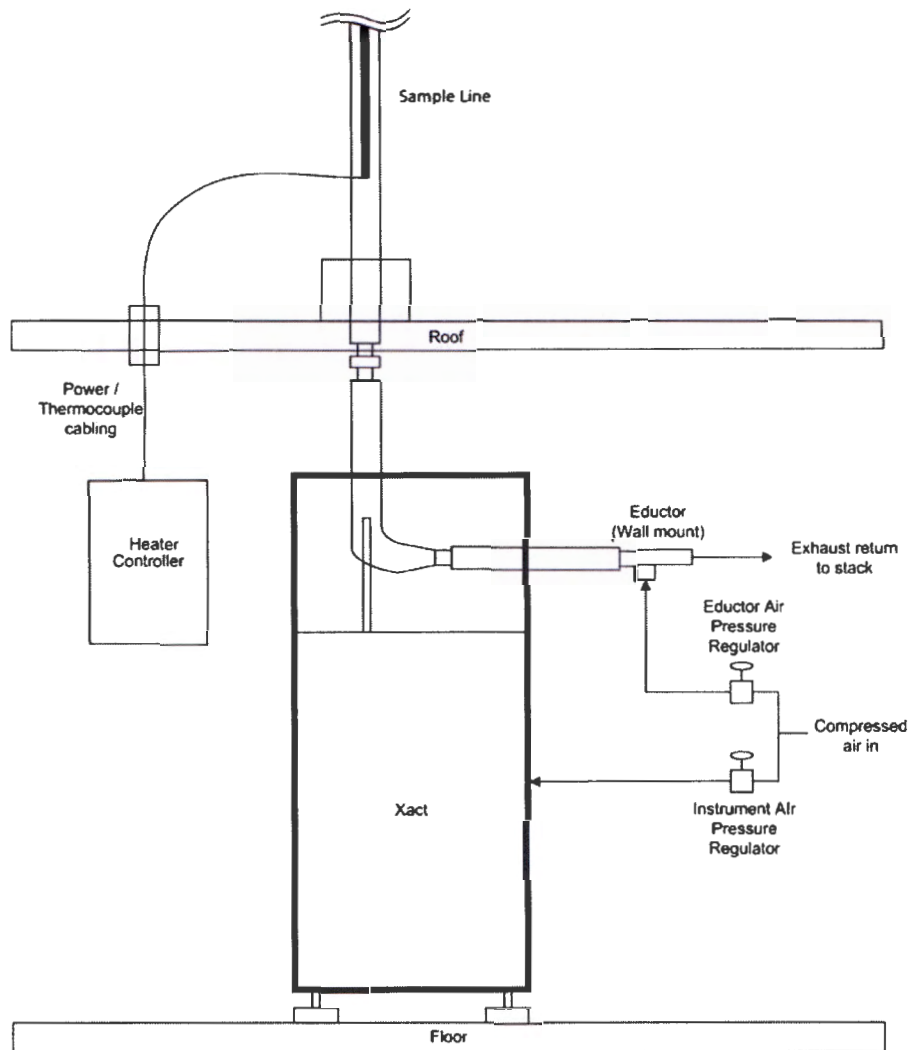### 2.1. Safety

Users need to be familiar electrical, burn and radiation hazardous and safety features of the instrument. These features are detailed in Section 1.4 of the Xact 640 Operating Manual.

2.2. Installation

Installation of Xact 640 should be performed by CES employee or authorized technicians/distributors only.

Xact 640 must be installed in a secure environment with temperature controlled to ~21±3 °C (~70±5 °F). The Xact 640 requires external heater controls for ensuring heated sample transport line. Sample line heater controller must be set to a temperature higher than the dew point of the stack gas (A good target is 180 °C). The setup also requires access to compressed air. The pressure regulator on the eductor line should be adjusted so that the stack flow rate on the Xact Controller reads ~160 lpm. The pressure regulator on the air line into Xact should read ~80 psi.

2.3. Start Up

Instrument start-up procedure is recommended if Xact is switched on:

(a) For the first time.

(b) After a long instrument shut-down period (> 4 hours).

(c) Following a power loss (> 5 min) to the instrument.

During the start-up procedure, it is critically important to allow the XRF Control program to successfully initialize all hardware components before setting the Xact 640 for sampling. This program launch and initialization process may take up to 5 minutes.

1. Make sure the Xact is plugged into the power-conditioned outlet or a clean power source.
2. Turn *Main Disconnect* switch located on the outer right side on.
3. The Heater Controls Interface should be already logged in. Open the program Specview 32.
4. Make sure Xact1point5 is selected, and click *Go Online Now*.
5. Begin ramping up the heaters using the SP button in the program to edit the set point for the following heaters: Stilling Chamber (350F), Dil. Maze (300F), Dil. Xport (325F), Return to Stack (300F), Return Line 1 and Return Line 2 (250F)
6. Turn on the Host PC with the login and password: *ces*.
7. Once Windows XP® has loaded, locate and open the *CES_XRF_Control* program (shortcut on desktop).

8. The XRF Control program and XRS-FP will be launched and numerous hardware devices will be initialized. Initialization of Digital Pulse Processor inside the Analysis Cabinet may result in a series of clicking noises.

9. Watch the LED lights (right figure), indicative of the home position of the two devices: X-ray filter disc and Linear Actuator (figure below) during the initialization period. Both devices will move away from the home positions (LED unlit) and move back to home positions (LED lit).

Linear Actuator &
QA Rod Upscale

X-Ray Filter
Disc Assembly
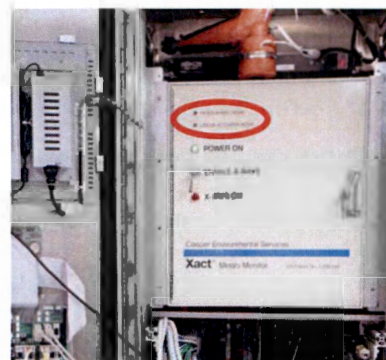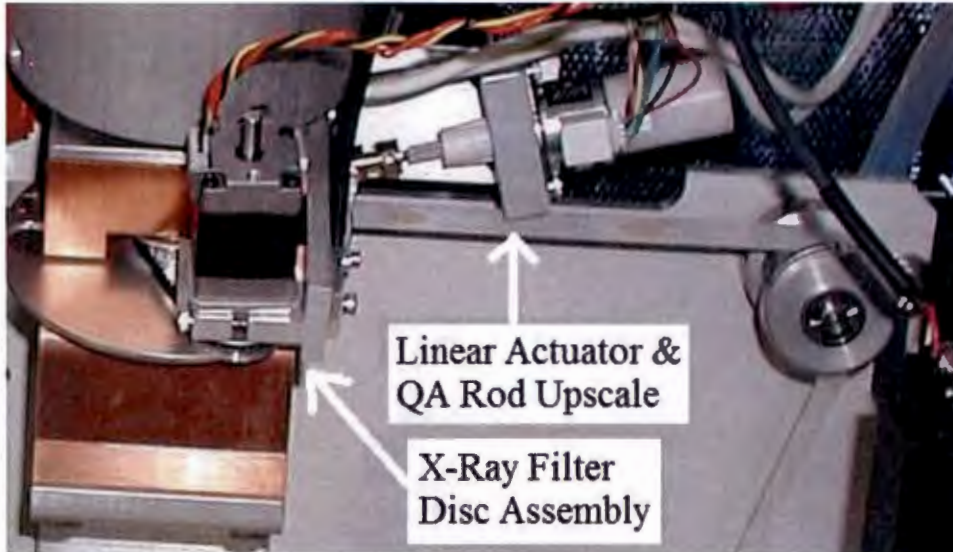
10. In the XRF Control program's *Acquisition Control* tab, acknowledgement messages will be displayed once the initialization is complete (example shown here).
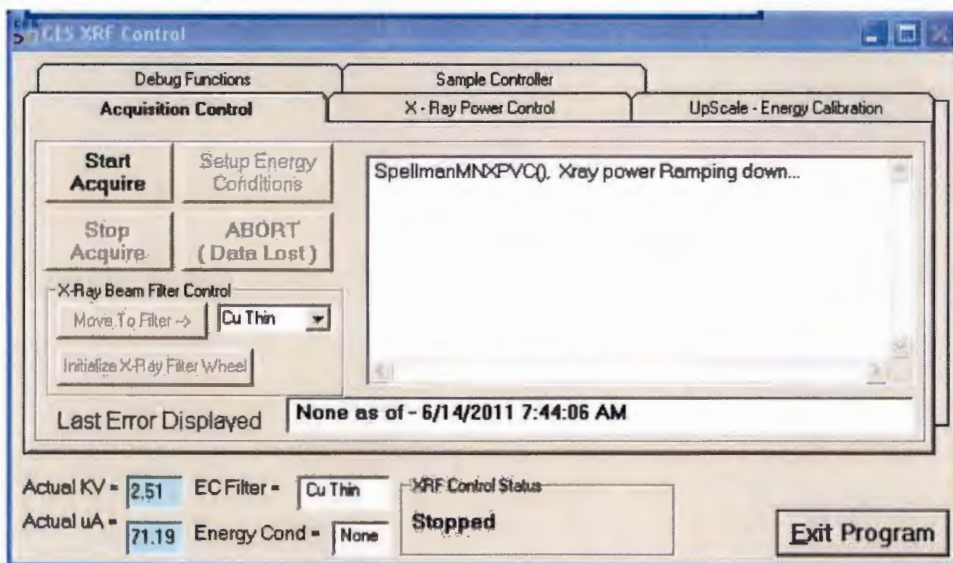
11. If the *Debug Message Window* option is enabled, a small window with messages may be displayed (shown here). This window will also show the successful completion of the Linear Actuator and X-Ray filter disc initialization.
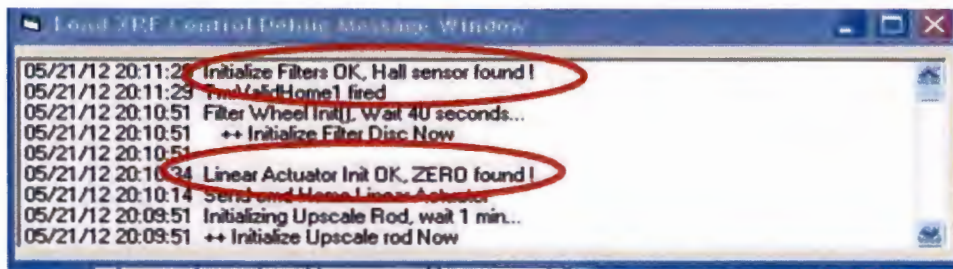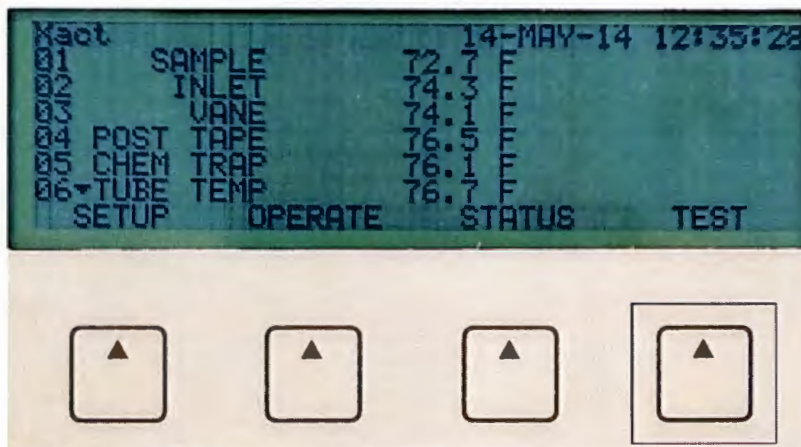
12. Following the initialization routine, the XRF Control may take up to 60 sec to complete the XRF Control hardware initialization sequence.

13. Ensure that the time stamp on Xact Controller and the Host PC are synchronized.

Allow the XRF Control program to successfully initialize all the hardware components before performing any action on the Xact Controller. The Xact launch and initialization process may take up to 5 minutes, concluding with the Linear Actuator and X-Ray Filter Disc moving to the home position.

2.4. Filter Tape Change
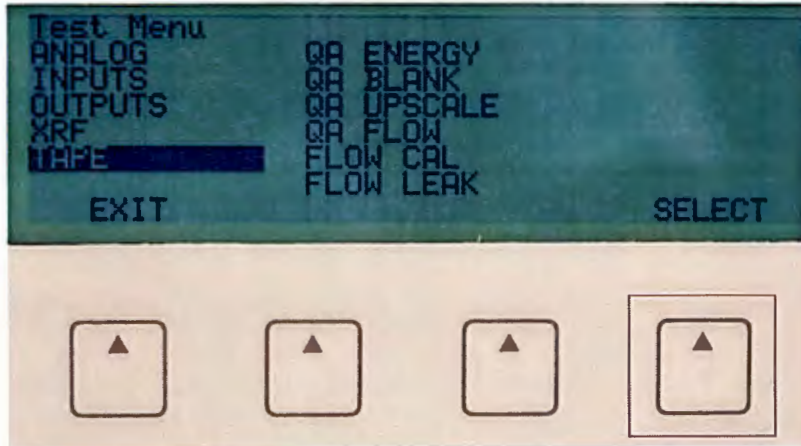
Quemetco uses 1 hour sampling and analysis mode. At this sampling time interval the filter tape will be changed every 28 days (4 weeks) to avoid downtime due to filter tape running out. Filter tape changing procedures are outlined below.
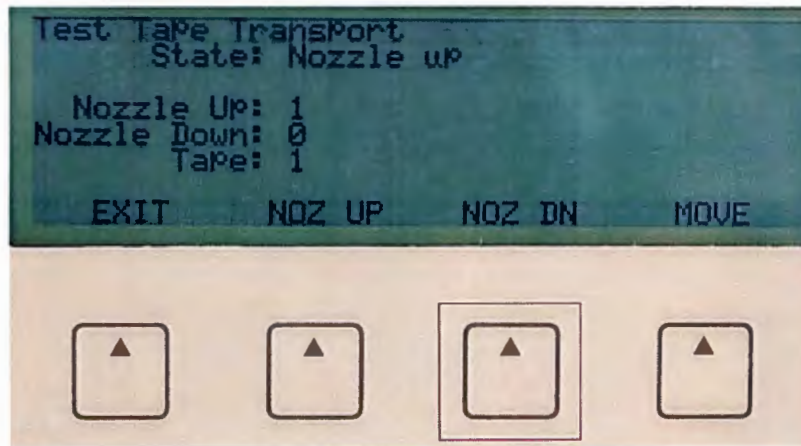
1. Place Xact Controller in STANDBY mode.

2. Press TEST on the Xact controller main screen.

3. Use the arrow keys to highlight *TAPE* and press SELECT.

Test Menu
ANALOG   QA ENERGY
INPUTS   QA BLANK
OUTPUTS   QA UPSCALE
XRF   QA FLOW
TAPE   FLOW CAL
             FLOW LEAK
    EXIT   SELECT

4. Press *NOZ DN*.

Test Tape Transport
        State: Nozzle up

    Nozzle Up: 1
  Nozzle Down: 0
         Tape: 1

    EXIT   NOZ UP   NOZ DN   MOVE

Nozzle Down value will change from 0 to 1.

Test Tape Transport
        State: Nozzle up

    Nozzle Up: 0
  Nozzle Down: 1
         Tape: 0

    EXIT   NOZ UP   NOZ DN   MOVE
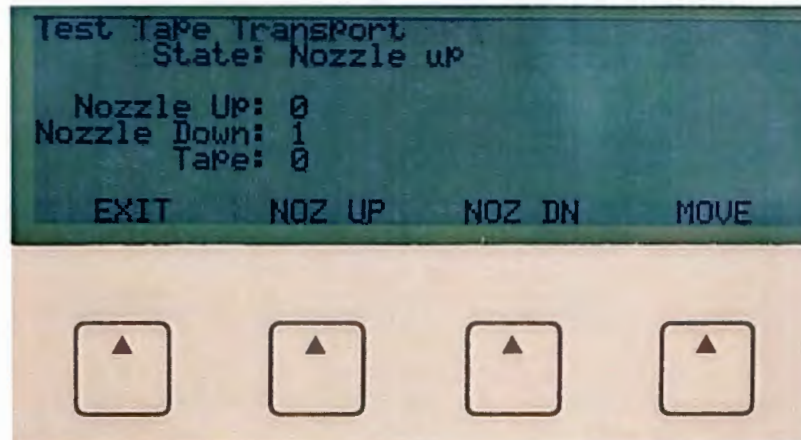
5. Tape drive components inside the Xact Analysis Cabinet are shown here. Open the Xact Analysis Cabinet and remove the reel covers.

Feed Reel   **Uptake Reel**

Labels: Pinch Roller, Sample Tube, X-ray Block, Tape, Reel Covers
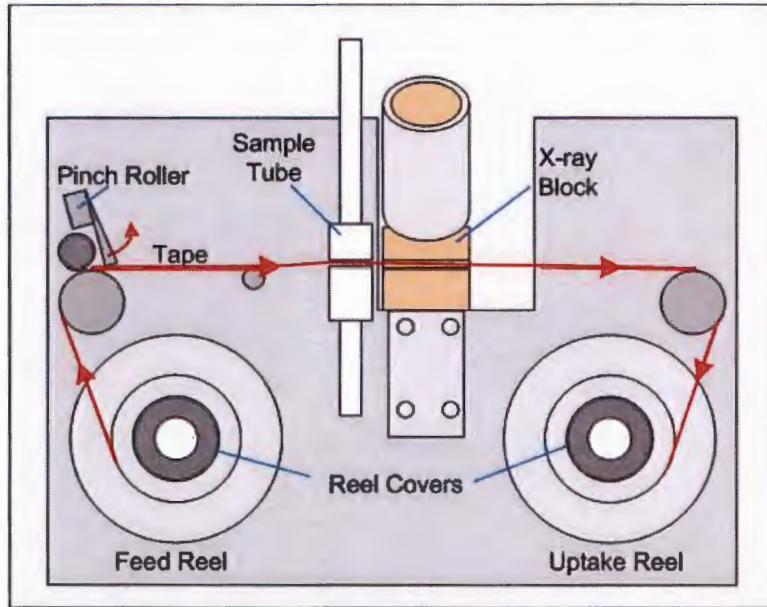
6. Place spool containing clean tape on the feed reel and an empty spool on the uptake reel.

Labels: X-Ray Detector, X-Ray Tube, Tape Slot (also slot for standard), Palladium Rod & Screw, Mounting Block
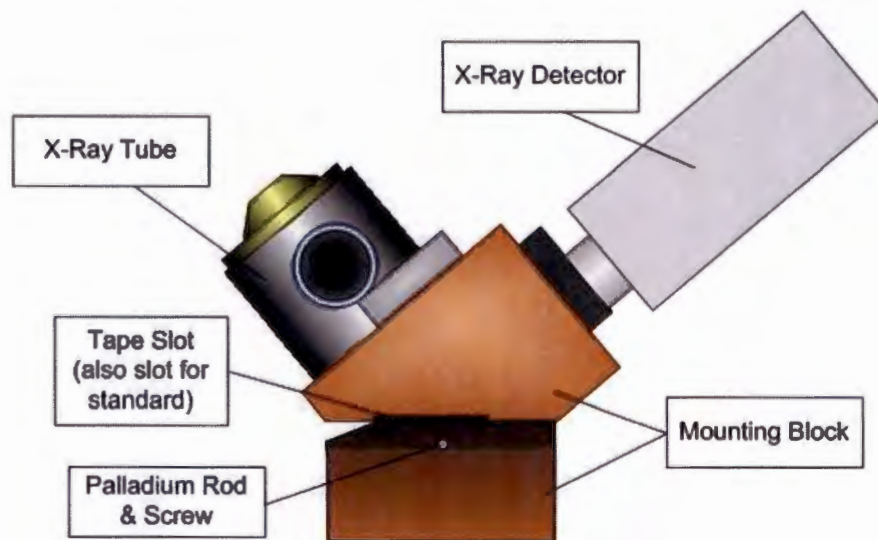
7. Feed the filter tape through the rollers, sample tube and the tape slot in the X-ray block. Tape the filter tape onto the uptake reel. Visually confirm the proper placement of the tape as below the shield plate at the nozzle.

8. Replace the reel covers.

9. Press *NOZ UP*.

```
Test Tape Transport
      State: Nozzle up

  Nozzle Up: 0
Nozzle Down: 1
       Tape: 0

   EXIT      NOZ UP      NOZ DN      MOVE
```
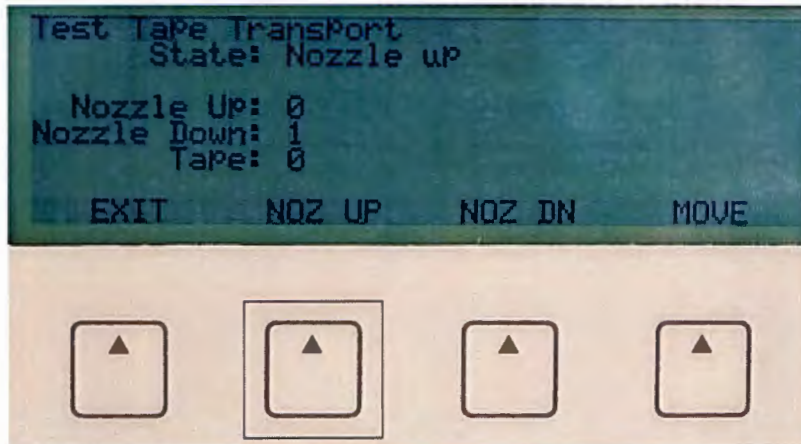
Nozzle Up value will change from 0 to 1.

```
Test Tape Transport
      State: Nozzle up

  Nozzle Up: 1
Nozzle Down: 0
       Tape: 0

   EXIT      NOZ UP      NOZ DN      MOVE
```
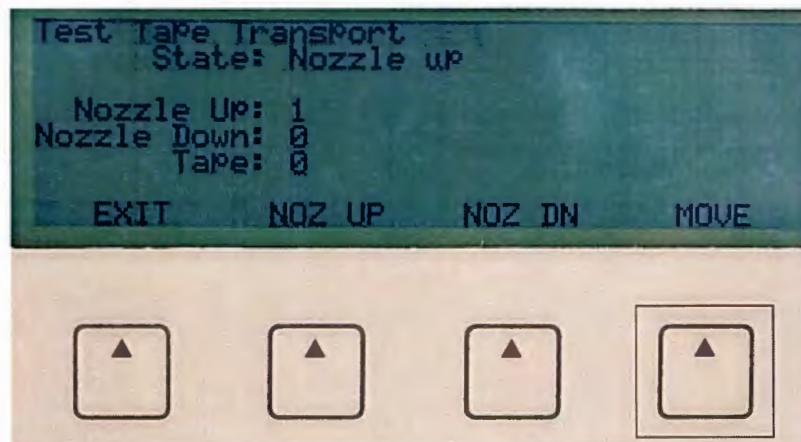
10. Press *MOVE* to advance the filter tape.

```
Test Tape Transport
      State: Nozzle up

  Nozzle Up: 1
Nozzle Down: 0
       Tape: 0

   EXIT      NOZ UP      NOZ DN      MOVE
```

11. Watch the movement of both tape reels carefully during the operation. The *Nozzle Down*
    value will change from 0 (false) to 1 (true). The *Tape* value, which senses the movement of

feed reel encoder, will change state twice and finally return to 1. *Nozzle Up* value will also return to 1.
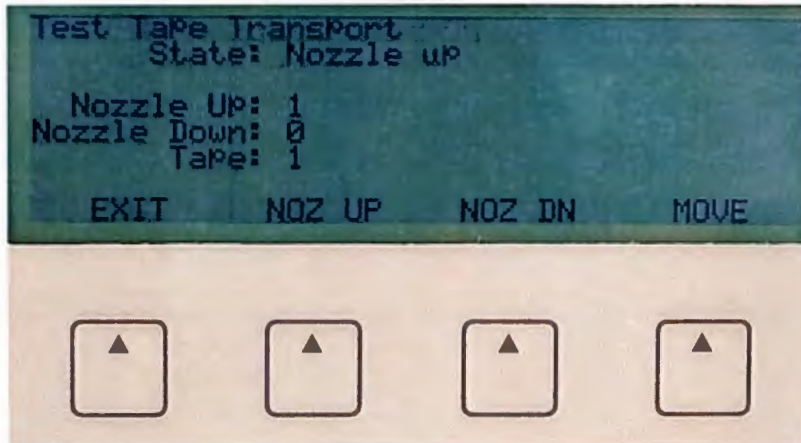
12. Press EXIT or HOME (F1) to return to the main screen.

13. Close and lock the sample/analysis cabinet door.

2.5. Setting the instrument to Run

The procedure for setting the Xact for routine sampling is provided here.

1. On the Xact controller home screen, press OPERATE.
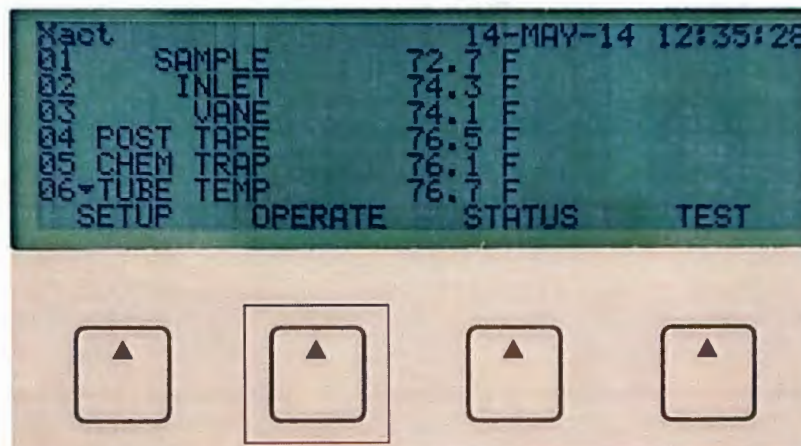
2. Use the arrow keys to highlight *Xact Mode* and press EDIT.
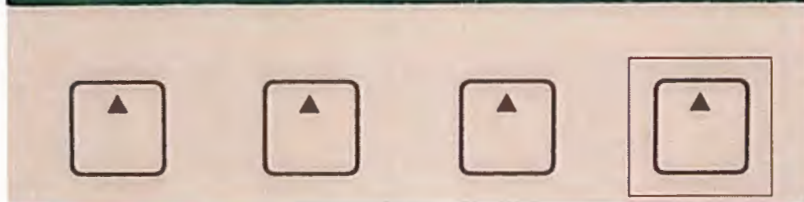
3. Use the arrow keys to highlight *STANDBY* and press OK. The *XRF Status* will change to *Stopped*.
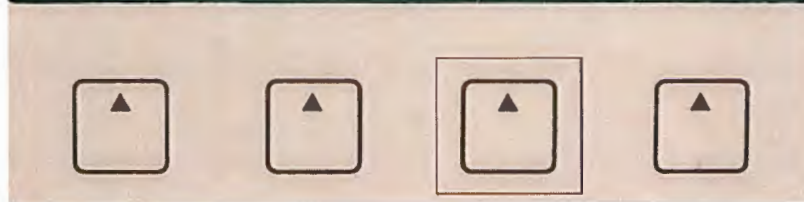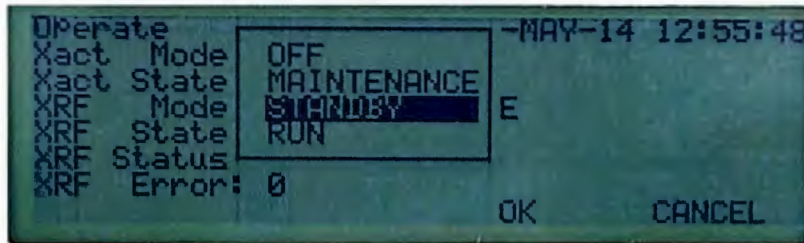
4. In XRF Control on the Host PC, open the *Sample Controller* tab and verify that the Modbus Read is incrementing to ensure a continuous serial connection from the Sample Controller to the Host PC.

5. On the Xact controller home screen, ensure that the sampling time is correct and synchronized with the XRF Control on the Host PC.

6. On the Xact Controller home screen, press OPERATE.

```
Xact                    14-MAY-14 12:35:28
01      SAMPLE        72.7 F
02       INLET        74.3 F
03        VANE        74.1 F
04 POST TAPE          76.5 F
05 CHEM TRAP          76.1 F
06▼TUBE TEMP          76.7 F
   SETUP      OPERATE      STATUS      TEST
```

7. Use the arrow keys to highlight *Xact Mode* and press EDIT.

```
Operate                 14-MAY-14 12:55:46
Xact   Mode:▼STANDBY
Xact   State: 0 STOP
XRF    Mode: 2 XRF_OPERATE
XRF    State: 25 FAILED
XRF    Status: DISCONNECTED
XRF    Error: 0
   EXIT                          EDIT
```
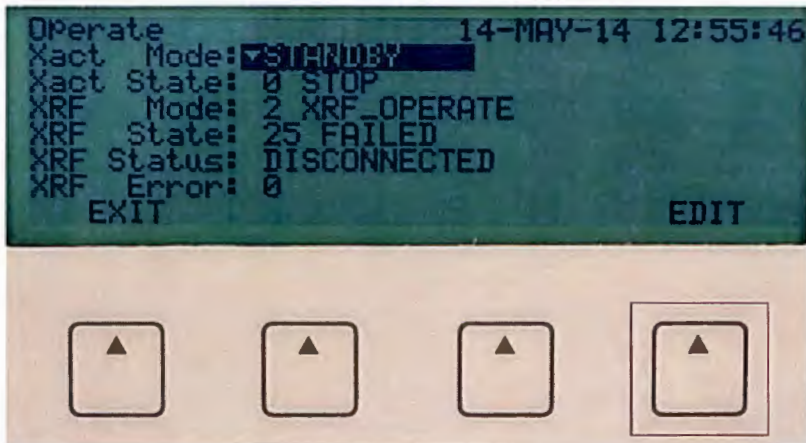
8. Use the arrow keys to highlight *RUN* and press OK.

```
Operate                 -MAY-14 12:55:48
Xact   Mode   OFF
Xact   State  MAINTENANCE
XRF    Mode   STANDBY      E
XRF    State  RUN
XRF    Status
XRF    Error: 0
                    OK        CANCEL
```
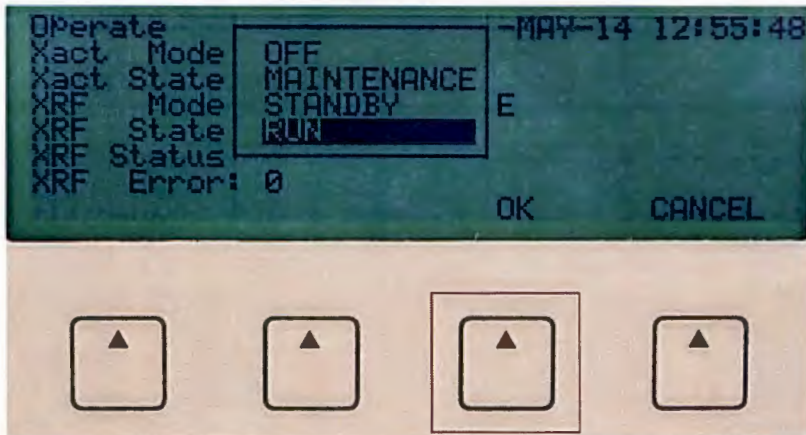
Xact will cycle a window of filter tape and start the pump at the beginning of the next sample start time

9. Press EXIT or Home (F1) to return to home screen.

2.6. Data Collection

The Xact data can be viewed on the LCD screen, downloading the data to a CSV file that can be exported into excel or by downloading the data to an external data acquisition system. Details on each procedure can be found in Section 2.5 of the Xact 640 Operation Manual

2.7. Instrument Shutdown

If sampling must be discontinued, placing Xact 640 in STANDBY mode is recommended over shutting down the system. Complete shut-down of Xact is only recommended if the instrument will not be used for prolonged period (>5 days) or will be moved.

1. On the Xact Controller's home screen, press OPERATE.
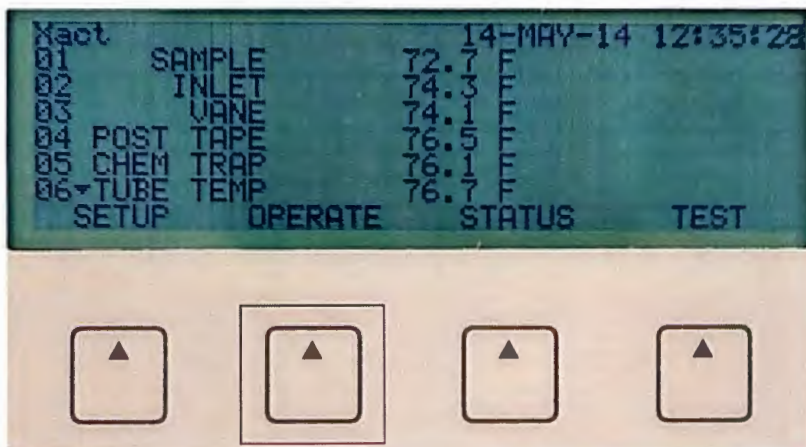
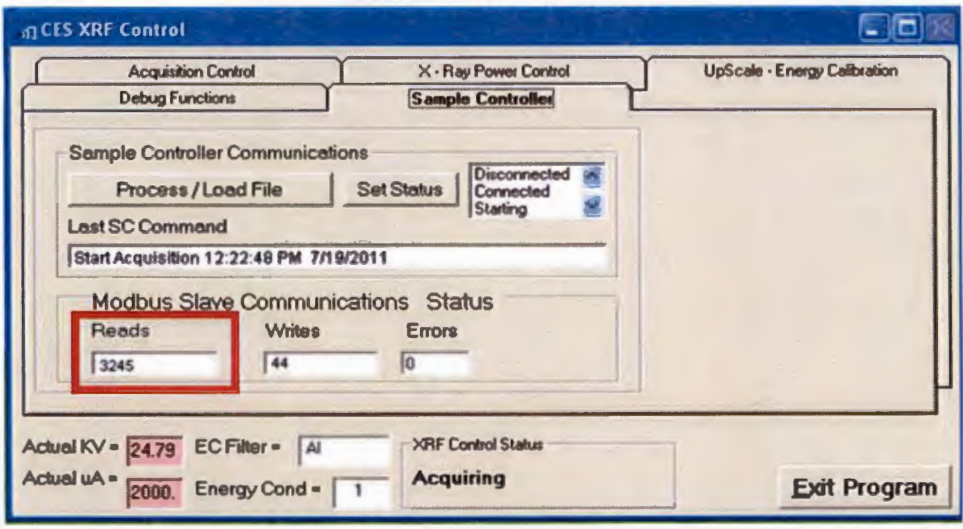2. Use the arrow keys to select *Xact Mode* and press EDIT.
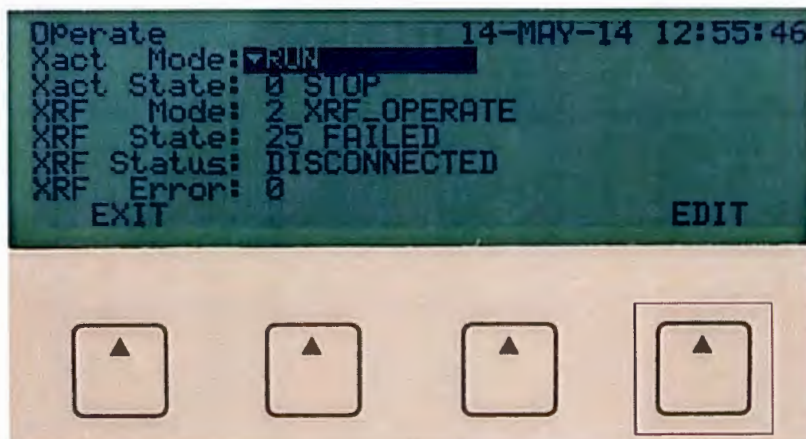
3. Use the arrow keys to highlight *STANDBY* and press OK.
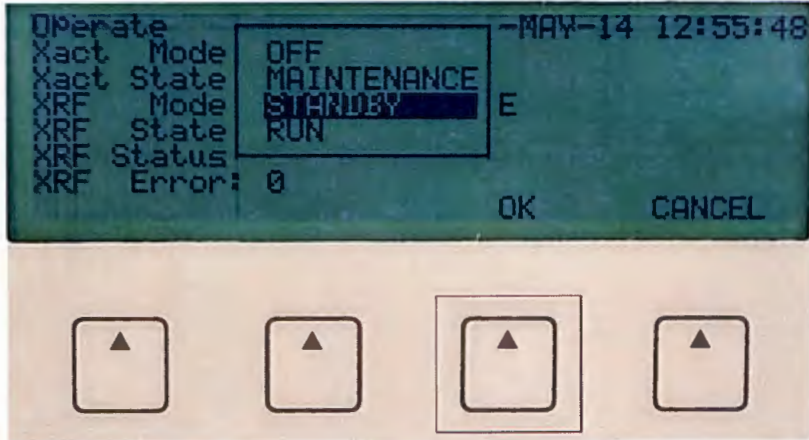
4. The instrument will prompt user-input for stopping after finishing the present sample or stop immediately. Select as desired. Once the instrument goes into STANDBY mode, the pump will stop immediately.

Complete shut-down of Xact 640

1. Ensure that Xact is in STANDBY mode.

2. On the Xact Controller's home screen, press OPERATE.
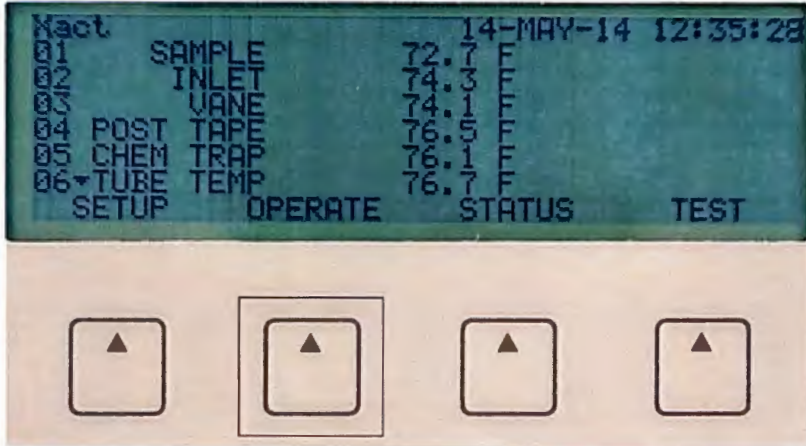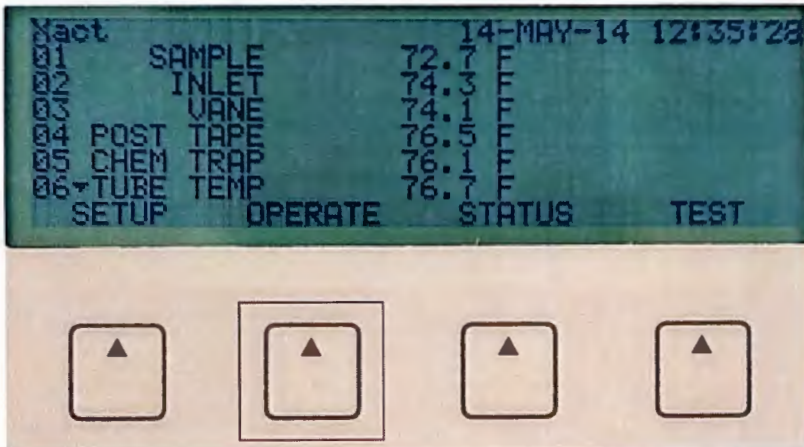

3. Use the arrow keys to highlight *Xact Mode* and press EDIT.
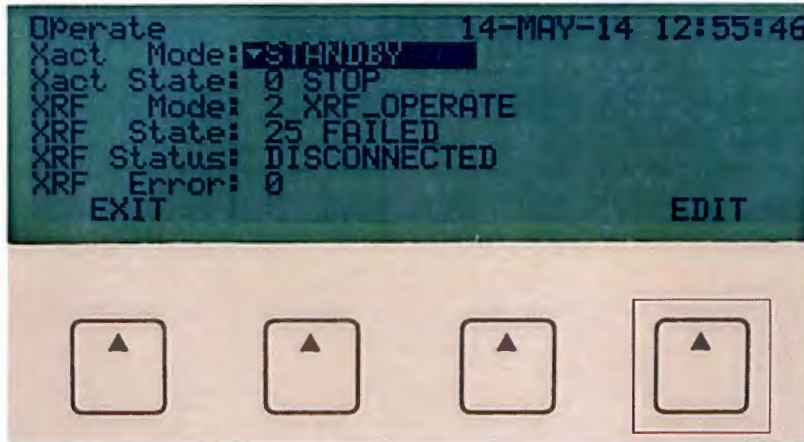
4. Use the arrow keys to highlight *OFF* and press OK.
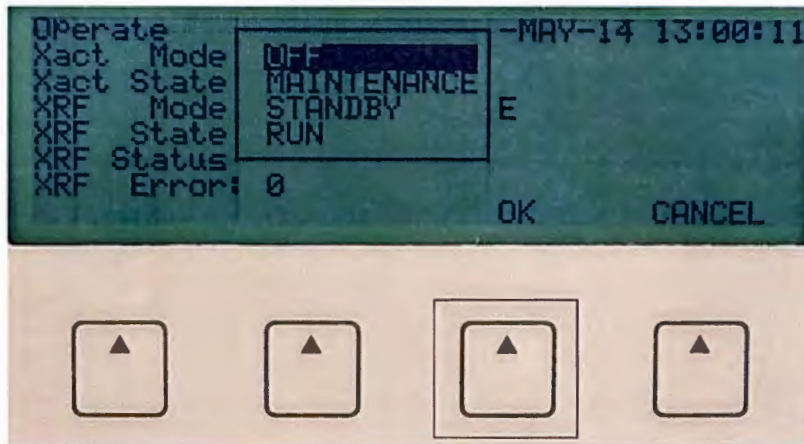
5. On the Host PC, exit the XRF Control program and ensure that all of its windows close down (may take few seconds).

6. On the Heater Controller Interface, set all heater set points to 0 and close the program.

7. Shut down the Heater Controller Interface.

8. Switch off all the breakers in the power distribution cabinet.

9. Inside the shed, turn off the heater controller.

10. Outside the shed, turn off the compressed air and close the sampling valve.

2.8. QA/QC Flags

The table below shows the data flags for the Xact 640 and their meaning.

Code	Alarm Flag Definitions
1	Stilling chamber temperature out of bounds
2	Inlet tube temperature out of bounds
3	Vane temperature out of bounds
4	Post tape temperature out of bounds

5	Chemical trap temperature out of bounds
6	Dilution temperature out of bounds
7	Sample flow out of bounds
8	Dilution flow out of bounds
9	Not Used
A	Not Used
B	Pressure too low
C	Dopant concentration too low
D	Palladium concentration out of bounds
E	QA energy data record
F	QA flow out of bounds
G	QA energy calibration failed
H	QA blank data record
J	Cr QA blank out of bounds
K	As QA blank out of bounds
L	Pb QA blank out of bounds
M	Cd QA blank out of bounds
N	Hg QA blank out of bounds
P	QA upscale data record
Q	Cr QA upscale out of bounds
R	As QA upscale out of bounds
S	Pb QA upscale out of bounds
T	Cd QA upscale out of bounds
U	Hg QA upscale out of bounds
V	Not Used
W	Not Used
X	X-Ray Tube
Y	8255 Reset

3. Quality Assurance
3.1. Daily Energy Alignment

The Xact performs an automatic daily energy alignment one time per day. This procedure assures that the spectral peaks are in the correct position. Failure to successfully complete this test does not necessarily invalidate instrument data.

3.2. Daily Upscale Check

This check evaluates the precision of the Xact's XRF systems response to a standard once per day. During this check, a rod containing metals is injected into the analysis area and the stability

of the analyzer response is evaluated. The CEMS must report a concentration that is within 15% of known value of the upscale standard each day. The rod contains three elements – one element analyzed under each of the three analysis conditions used by the Xact 640. At Quemetco the instrument is being used to evaluate compliance with arsenic emission limits. Arsenic is analyzed under analysis condition 2. On the upscale rod the accuracy and repeatability of energy condition 2 is evaluated using lead (Pb).

Upscale calibration drift is calculated using equation 1 below.

$$CD = \frac{|R - A|}{L} x100 \qquad \text{Equation 1}$$

Where:

CD = Upscale Drift

R = Either the upscale reference value

A = Either the Xact CEMS response to the upscale

L = The known concentration of the upscale standard

The value for L is established immediately following calibration of the Xact 640 by running at least three replicate analyses of the upscale rod.

3.3. Daily Zero Check

This check evaluates the Xact's XRF system response to a zero concentration produced by analyzing an un-sampled section of filter tape. The instrument passes the test if the result of the zero drift check for each monitored metal is less than 20% of the applicable emission limit. The zero drift can be calculated using Equation 2. This check is performed daily.

$$CD = \frac{|R - A|}{L} x100 \qquad \text{Equation 2}$$

Where:

CD = zero calibration drift.

R = zero standard reference value

A = Xact CEMS response to the zero standard

L = The emission limit expressed in the $\mu g/m^3$

3.4. Daily Flow Check

The Xact utilizes a quality assurance flow sensor to check the flow reading from sample flow sensor once per day. The difference flow between the quality assurance flow sensor and the sample flow sensor must be less than 20% of the full scale value of the flow measurement device to pass the test. The flow drift is calculated according to equation 3. The drift check is performed daily.

$$FD = \frac{|F_P - F_{QA}|}{FS} x100 \qquad\qquad \text{Equation 3}$$

Where:

FD = The flow drift

F_p = The flow as measured by the primary flow measurement device in the Xact.

F_{QA} = The flow as measured by the CEMS's secondary quality assurance flow meter.

FS = The full scale value of the flow sensor – the full scale value of the flow sensor for the Xact 640 is 5slpm

3.5. Periodic Leak Check

A leak check should be performed on the instrument at the time of installation, and quarterly thereafter. To perform a leak check:

1. Place Xact in STANDBY mode.
2. On the Xact Controller home screen, press TEST.

3. Highlight *TAPE* and press SELECT.
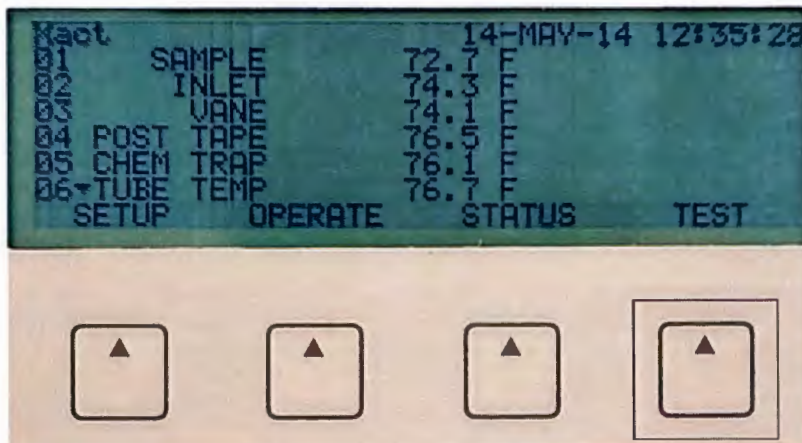
Test Menu
ANALOG   QA ENERGY
INPUTS   QA BLANK
OUTPUTS   QA UPSCALE
XRF   QA FLOW
TAPE   FLOW CAL
              FLOW LEAK
    EXIT   SELECT
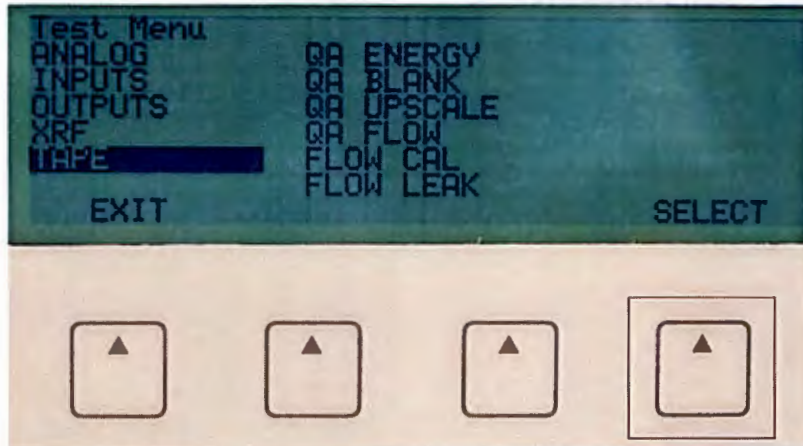
4. Press *Noz Dn* to release the filter tape from the tape slot in the mounting block (*Nozzle Down* value changes from 0 to 1).

Test Tape Transport
       State: Nozzle up

   Nozzle Up: 1
Nozzle Down: 0
       Tape: 1
    EXIT   NOZ UP   NOZ DN   MOVE
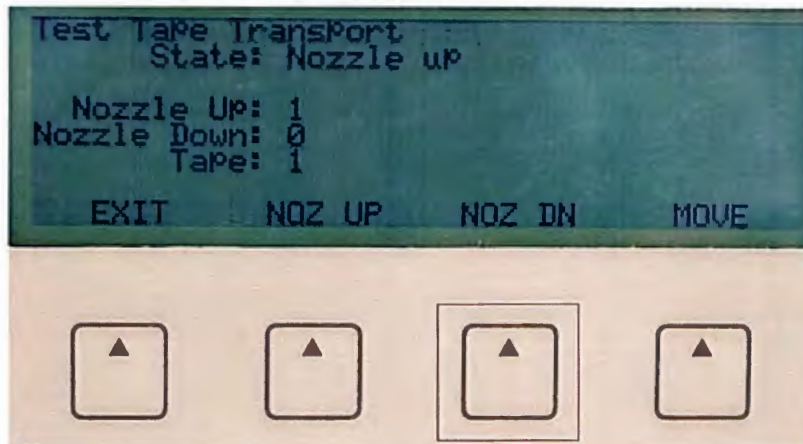
5. Remove the filter tape from the tape drive.

6. Place a piece of thin rubber (typically part of a nitrile glove) on the nozzle that seals on the tape.

7. Press *NOZ UP* to create a seal (*Nozzle Up* value changes from 0 to 1)

Test Tape Transport
       State: Nozzle up

   Nozzle Up: 0
Nozzle Down: 1
       Tape: 0
    EXIT   NOZ UP   NOZ DN   MOVE

8. Press EXIT and return to the *TAPE* menu.


```
Test Tape Transport
        State: Nozzle up

   Nozzle Up: 1
 Nozzle Down: 0
        Tape: 1

   EXIT      NOZ UP      NOZ DN      MOVE
```
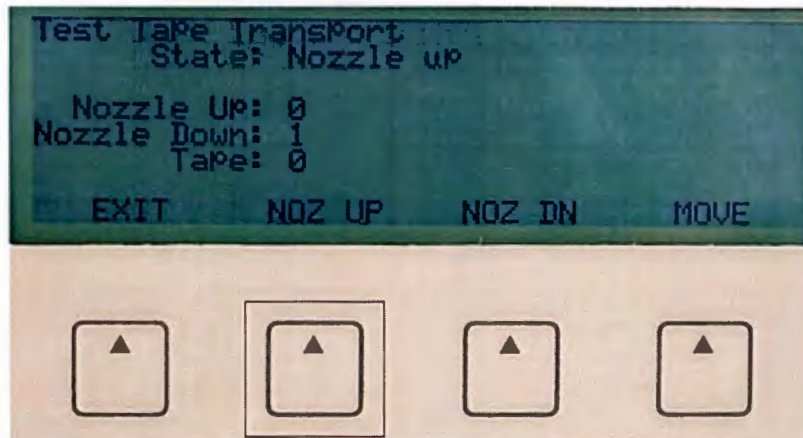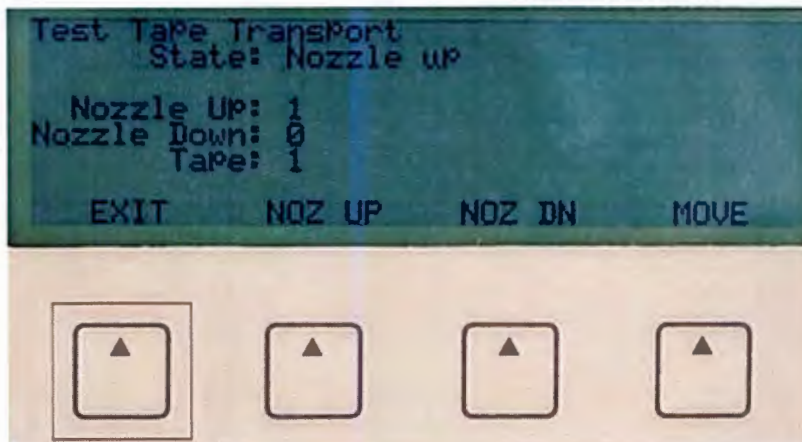
9. Highlight *FLOW LEAK* and press SELECT.


```
Test Menu
ANALOG          QA ENERGY
INPUTS          QA BLANK
OUTPUTS         QA UPSCALE
XRF             QA FLOW
TAPE            FLOW CAL
                FLOW LEAK

    EXIT                        SELECT
```
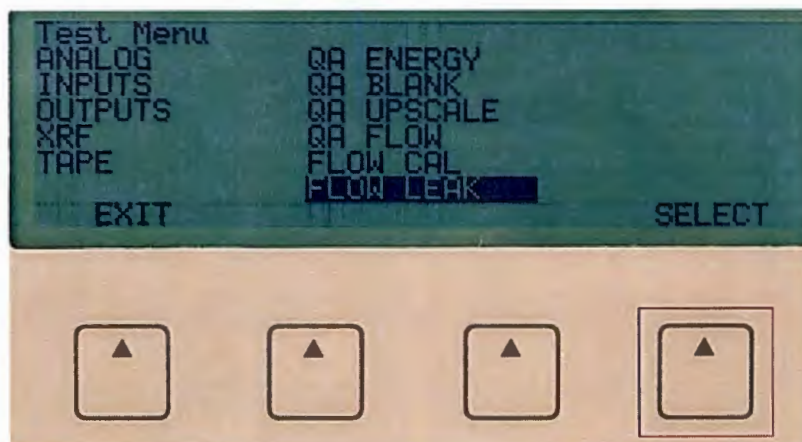
10. Turn the pump on using the menu controls.

11. Wait for the Tape Pressure to decrease to 5 psi, then turn off the pump and close the shut-off valve on the Flow Module (Section 1.5.4).

12. Monitor the psi for 60 seconds. If the change in psi is less than 1 psi, then Xact has successfully passed the leak check.

13. Open the shut-off valve in the Flow Module.

14. Press EXIT and return to the *TAPE* menu.

15. Highlight *TAPE* and press SELECT.

```
Test Menu
ANALOG          QA ENERGY
INPUTS          QA BLANK
OUTPUTS         QA UPSCALE
XRF             QA FLOW
TAPE            FLOW CAL
                FLOW LEAK
    EXIT                    SELECT
```
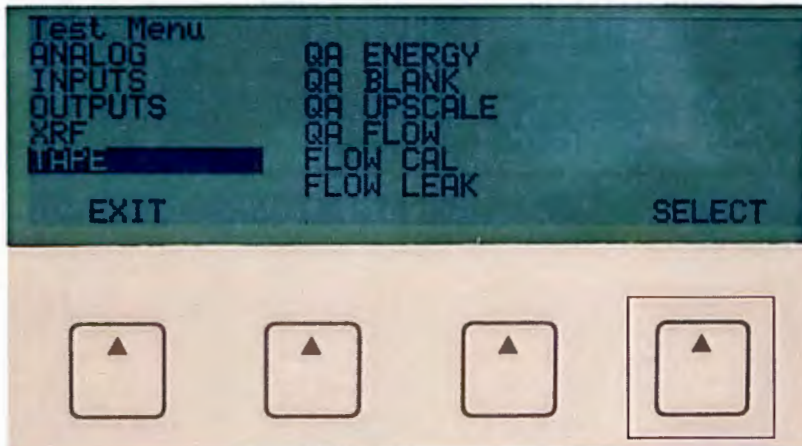

16. Press *Noz Dn* and remove the piece of rubber from the tape drive.

```
Test Tape Transport
        State: Nozzle up

   Nozzle Up:  1
 Nozzle Down:  0
        Tape:  1
   EXIT      NOZ UP    NOZ DN    MOVE
```


17. Replace the filter tape and press *Noz Up*.

```
Test Tape Transport
        State: Nozzle up

   Nozzle Up:  0
 Nozzle Down:  1
        Tape:  0
   EXIT      NOZ UP    NOZ DN    MOVE
```
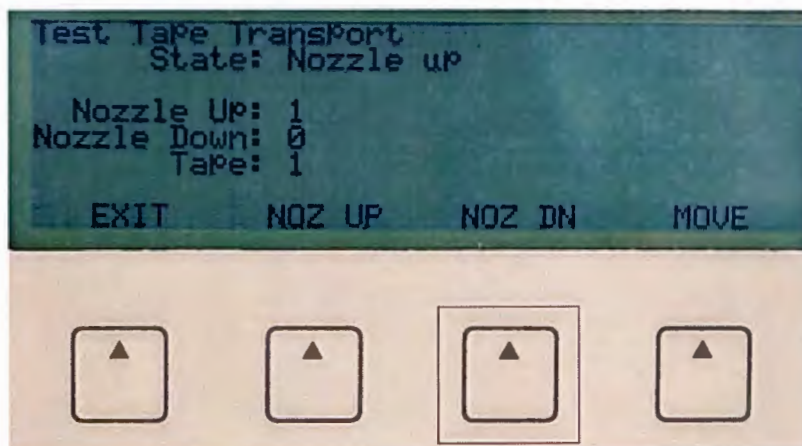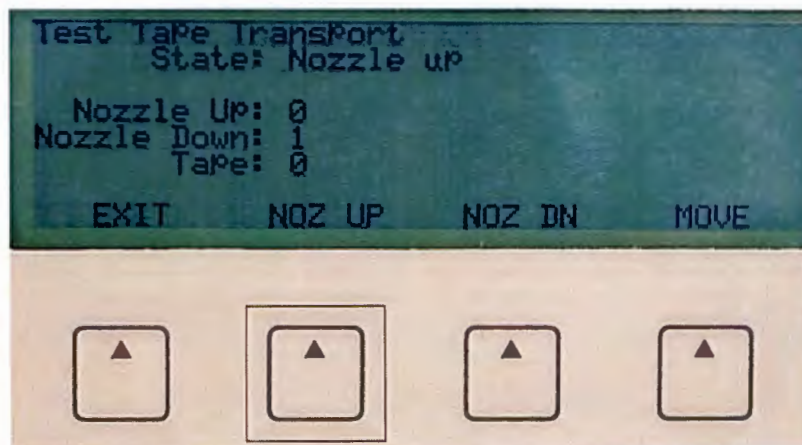

18. Press EXIT or HOME (F1) to return to the home screen.

3.6. XRF Check

XRF checks should be performed on a quarterly basis on the elements of interest to the user.

These checks are performed using traceable to NIST thin film standards that are recommended

by the US EPA in the Inorganic Compendium of Methods for the analysis of air filter samples by XRF. For Quemetco, arsenic is the element of most interest and the GaAs thin film standard would be used to evaluate the instrument for accuracy. Unfortunately, the GaAs standard can volitalize slowly over time, resulting in a change in the actual concentration of arsenic on the standard. Typically this accounted for at the time of calibration by using several elements with atomic numbers near arsenic and taking advantage of the physics of the XRF (i.e. that sensitivity is a uniformly varying function of atomic number).

Since the arsenic standard will volatize over time the XRF check of the Xact 640 will be performed using copper – which is another element analyzed under the same energy condition as arsenic. The procedure for performing an XRF check is outlined below.

1. Place the Xact in MANUAL mode on the Host PC:

a. Start CES XRF Control program on the Host PC.

b. Open the *Sample Controller* Tab and click on DISABLE.
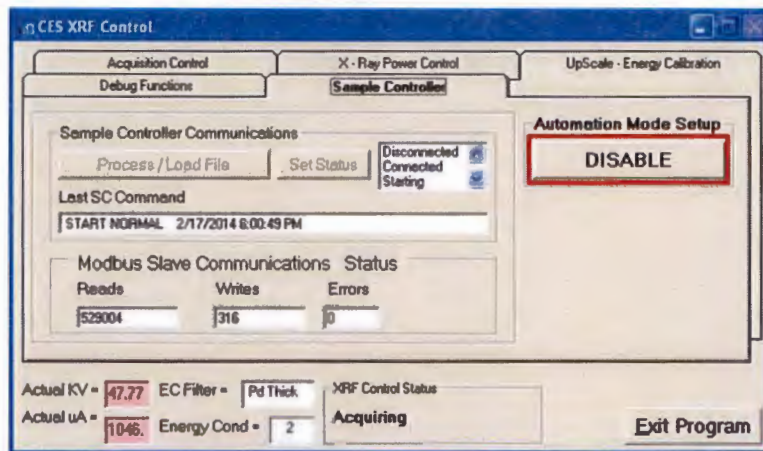
c. The XRF Control program will automatically close. Restart the XRF Control.

2. Place Xact in STANDBY mode and ensure that X-Rays are turned off.

3. Open the XRF cabinet door in the instrument and remove the filter tape.

4. Place the first standard in the black standard holder with the plastic ring on standard facing upwards.

5. Place the standard holder in the tape slot of XRF analysis block, as far into the analysis block as it will fit.

Standard

Standard in Standard Holder
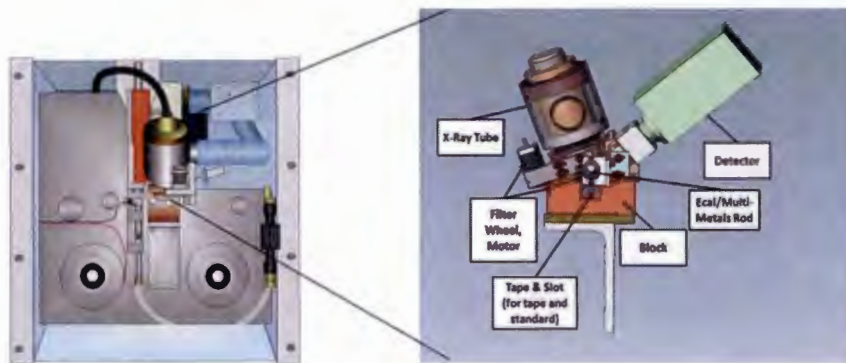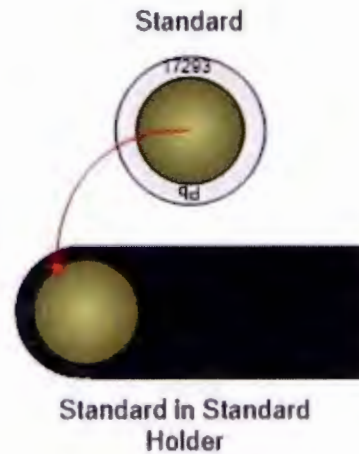
6. Close the XRF cabinet door and the instrument.
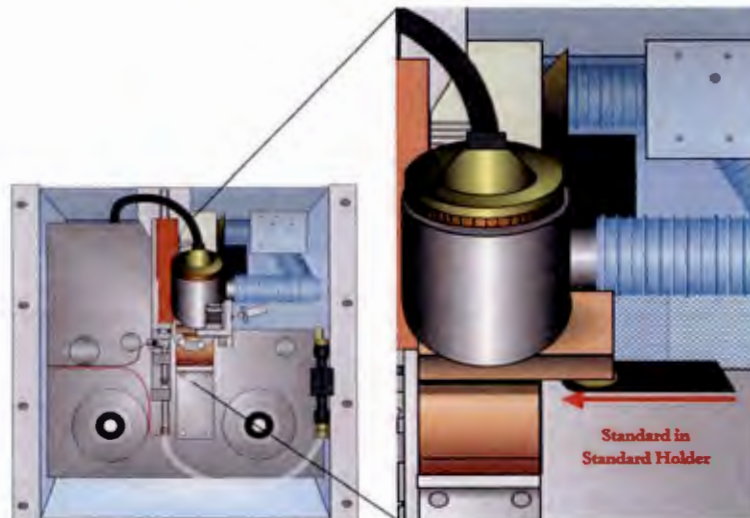
7. On the Host PC, go to XRF Control program. In the *Acquisition Control* tab, select *Setup Energy Conditions*.
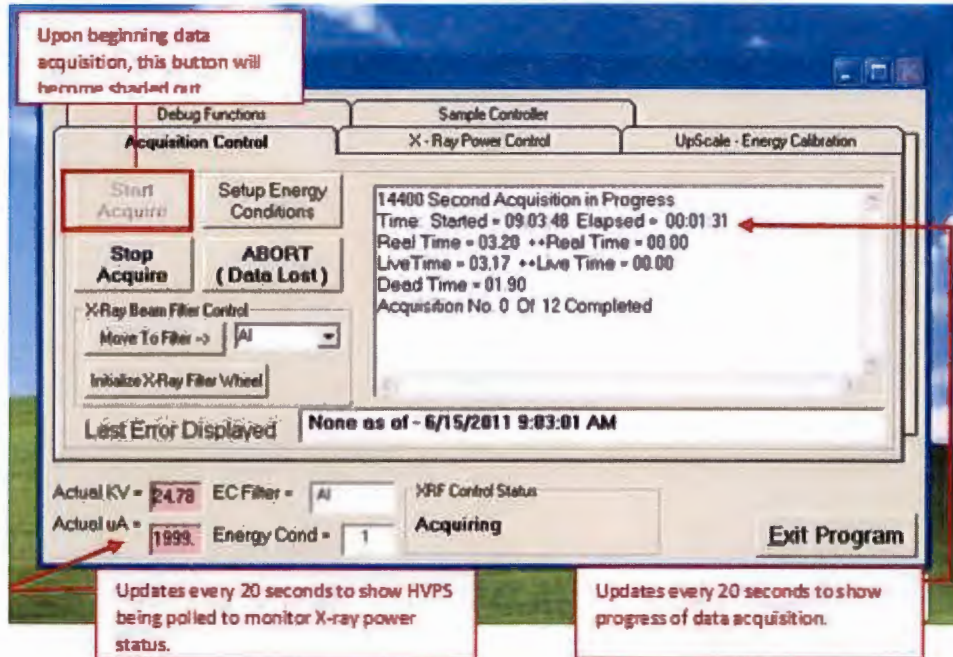
8. Set the excitation and filter to the appropriate settings for the analysis condition required by the chosen standard. Consult the Calibration Table provided by CES to determine the correct setting. For copper the analysis time is 90 seconds.

a. Copper is run under Energy Condition 2, set EC1 to the lowest % of RunTime, since that time is essentially wasted.

9. If needed, change the Total Sample time parameter to achieve a correct setup. Don't alter the kV, Filter, or AutoCurrent parameters.

10. Always exit the Parameter Setup form by selecting *Apply Changes*. Ignore messages relative to running in AUTOMATION mode.

Form1

Sample Time [120 ▾] Min <— This value MUST match Sample Controller's <— for Automation Mode to function properly

Energy Condition 1 Parameters

[20 ▾] % Run Time = EC1 Run Time of [1440] Sec. ☑ Auto Current ?

X-Ray Filter [Al ▾] Target kV [25 ▾] Target uA [990]

Energy Condition 2 Parameters

☑ Enabled ? [40 ▾] % Run Time = EC2 Run Time of [2880] Sec. ☑ Auto Current ?

X-Ray Filter [Pd Thick ▾] Target kV [46 ▾] Target uA [505]

Energy Condition 3 Parameters

☑ Enabled ? [41 ▾] % Run Time = EC3 Run Time of [2952] Sec. ☑ Auto Current ?

X-Ray Filter [Cu Thin ▾] Target kV [46 ▾] Target uA [405]

Energy Condition 4 Parameters

☐ Enabled ? [C ▾] % Run Time = EC4 Run Time of [ ] Sec. ☑ Auto Current

X-Ray Filter [CmbxEC4Fil ▾] Target kV [C ▾] Target uA [500]

[Reset all Parameters] [Apply Changes] [Save Parameters] [Exit] [Exit AND Save]

11. Once the correct parameters are setup, select *Exit and Save*.

12. In the XRF Control, go to the *Acquisition Control* tab and press *Start Acquire*. XRF Control will run the selected setup.

13. Monitor the *Target kV* and *Target uA* text boxes during the run, they are updated as XRF Control changes the excitation parameters to run the standard. When the run is finished, the X-ray power will ramp down.

14. Using the Automation form of XRS-FP, select the *Expert Mode* button and the XRS-FP Main form will open as shown below.

XRS FP

File Acquire Setup Calibrate Process Help

| Acquire | kV: 46.9 (4->200) | uA: 999.0 (1->2000) | Preset: 180 | Time: 15.91 | %DT: 0.0 | X: 0 | Y: 0 | Z: 0 |

Specimen Component Table:

#	Component	Type	Conc.	Error	Units	Mole%	Error
1	Si	Calc	######	100.000	ug/cm2	100.000	239.095
2	K	Calc	86.573	36.980	ug/cm2	148.664	63.502
3	Ca	Calc	29.501	27.077	ug/cm2	49.421	45.360
4	Ti	Calc	0.000	0.000	ug/cm2	0.000	0.000
5	V	Calc	0.000	0.000	ug/cm2	0.000	0.000
6	Cr	Calc	2.940	6.220	ug/cm2	3.797	8.032
7	Mn	Calc	0.000	0.000	ug/cm2	0.000	0.000

Thickness Information:

	Layer						Normalize
Thick.	Type	Error	Units	Density	Fixed	OK	Total
0.000	Bulk	0.000	mg/cm2	0.000			100.00

Global Threshold Settings
n-sigma ▼ 2.000 Clear Conc Method ☐

Element Table: ⦿ Normal ○ Coefficients

#	Element			Measurement							Threshold		Intensity	Ratio	ROI (keV)		Chi2	Quant	Calibration
	Elmt	Line	Code	Intensity	Error	Backgr.	Conc.	Error	MDL	Atom%	Value	Conc	Method	Method	Low	High	Fit	Method	TCC Coeff
1	Si	Ka	1	0.20759	0.01	213.45	#####	#####	#####	#####	0.000	☑	Reference	Current	1.636	1.843	2.83	LSQ	0.00
2	K	Ka	1	0.02223	0.01	173.41	86.573	36.980	#####	148.664	0.000	☑	Reference	Current	3.201	3.424	7.28	LSQ	0.00
3	Ca	Ka	1	0.00949	0.01	134.00	29.501	27.077	#####	49.421	0.000	☑	Reference	Current	3.577	3.803	0.73	LSQ	0.00
4	Ti	Ka	1	0.00000	0.00	105.03	0.000	0.000	0.0000	0.000	0.000	☑	Reference	Current	4.390	4.626	0.00	LSQ	0.00
5	V	Ka	1	0.00000	0.00	99.37	0.000	0.000	0.0000	0.000	0.000	☑	Reference	Current	4.828	5.069	0.00	LSQ	0.00
6	Cr	Ka	1	0.00330	0.01	102.95	2.940	6.220	#####	3.797	0.000	☑	Reference	Current	5.287	5.533	1.01	LSQ	0.00

Measurement & Processing Conditions: ⦿ Measurement ○ Processing   Active Condition Code: 1

Code	X-Ray Source					Detector			Chamber	Time (secs)		Monitor
	Target	Filter	Thick(um)	kV	uA	Type	Filter	Thick(um)	Atmos	Preset	Actual	Intensity
1	Mo	None	0.00	47.0	999.8	Si-pin	None	0.00	Air	100.0	15.9	0.0

Status:

2 | Off | 2048 | 20 | 31 | No

Comment: Command String

15. Check the column for the element of interest from the selected standard. If the Column appears as #####, simply widen it with the mouse to see the value in micrograms detected on the XRF Standard.

The data file written for each energy condition can be viewed at C:\Process_Data. This ASCII text file lists the values for ALL the metals detected for each Excitation Condition. Move this file to another folder location with appropriate name, such as C:\Xact QA\Jan2011\. Record the values in a QA Spreadsheet each time an XRF Standard Check of the metals analyzer is performed.

16. Open the door to the sampling/analysis cabinet. Make sure the X-ray tube is off. Remove the standard and replace it with the next standard to be analyzed. Close the cabinet door.

17. Place the Xact in AUTOMATION Mode on the Host PC.

The error for the audit will be calculated using Equation 4. If the error exceeds 10% than the test should be repeated two more times. If the average error for all three runs exceeds 10% than the Xact should be evaluated problems with the XRF system.

$$AE = \frac{|C_S - C_c|}{C_s} x100$$

Equation 4

Where:

AE = XRF Analyzer Error

C_S = Concentration (or mass) of the metal analyte on the traceable to NIST standard

C_c = Concentration (or mass) of the metal analyte as reported by the Xact

3.7. Dynamic Spiking RATA
3.7.1. Introduction

As part of the performance specifications and on-going quality assurance requirements for compliance measurements on hazardous waste incinerator, periodic relative accuracy test audits (RATA) were performed on the Xact. The RATA procedures involved dynamically spiking sampled stack gas with an aerosol of a known concentration. These reference aerosols were generated using CES' Quantitative Aerosol Generator (QAG). The guidelines for these procedures have been recognized by EPA in Other Test Methods 16 and 20.

There are several benefits to dynamic spiking. One benefit is the ability to test the instrument's response over a wide range of concentration levels rather than just relying on whatever the native stack gas concentrations are. In addition, dynamic spiking, as opposed to spiking with a clean dry air, challenges the instrument and tests for any potential effects of the stack matrix.

There are some differences between the hazardous waste incinerator source and this Secondary Lead Smelter in the source operation that provide additional challenges to the dynamic spiking procedure that CES intends to do. The hazardous waste incinerator was able to burn waste during the RATA procedure that did not contain significant metal concentrations. This means that there was no need to correct for background concentrations of the spiked metals. The secondary lead smelting facility included in this study is not be able to provide such a metals free background. To resolve this issue CES intends to alternate between background (non spiked) and spiked concentrations during its dynamic spiking procedures. This procedure is similar to approach that EPA has taken to dynamic spiking procedures on HCl CEMS (PS-18).

3.7.2. Spiking Arrangement

Figure 1 shows the arrangement for Quantitative Aerosol spiking. Stack effluent is drawn into the spike mixing chamber. The spike aerosol flow (approximately 50 Lpm) is introduced to the approximately 250 Lpm flow of stack effluent, is allowed to mix and transported to the heated flexible sample line. After traveling through the heated sample line the aerosol is transported to

the Xact stilling chamber where approximately one liter per minute of aerosol is drawn through the reactive filter tape. The majority of the spiked stack effluent passes through the stilling chamber and then through a carbon trap which removes metals and particulate matter. Finally, the flow is measured using a calibrated Rosemount Mass Flow Sensor.
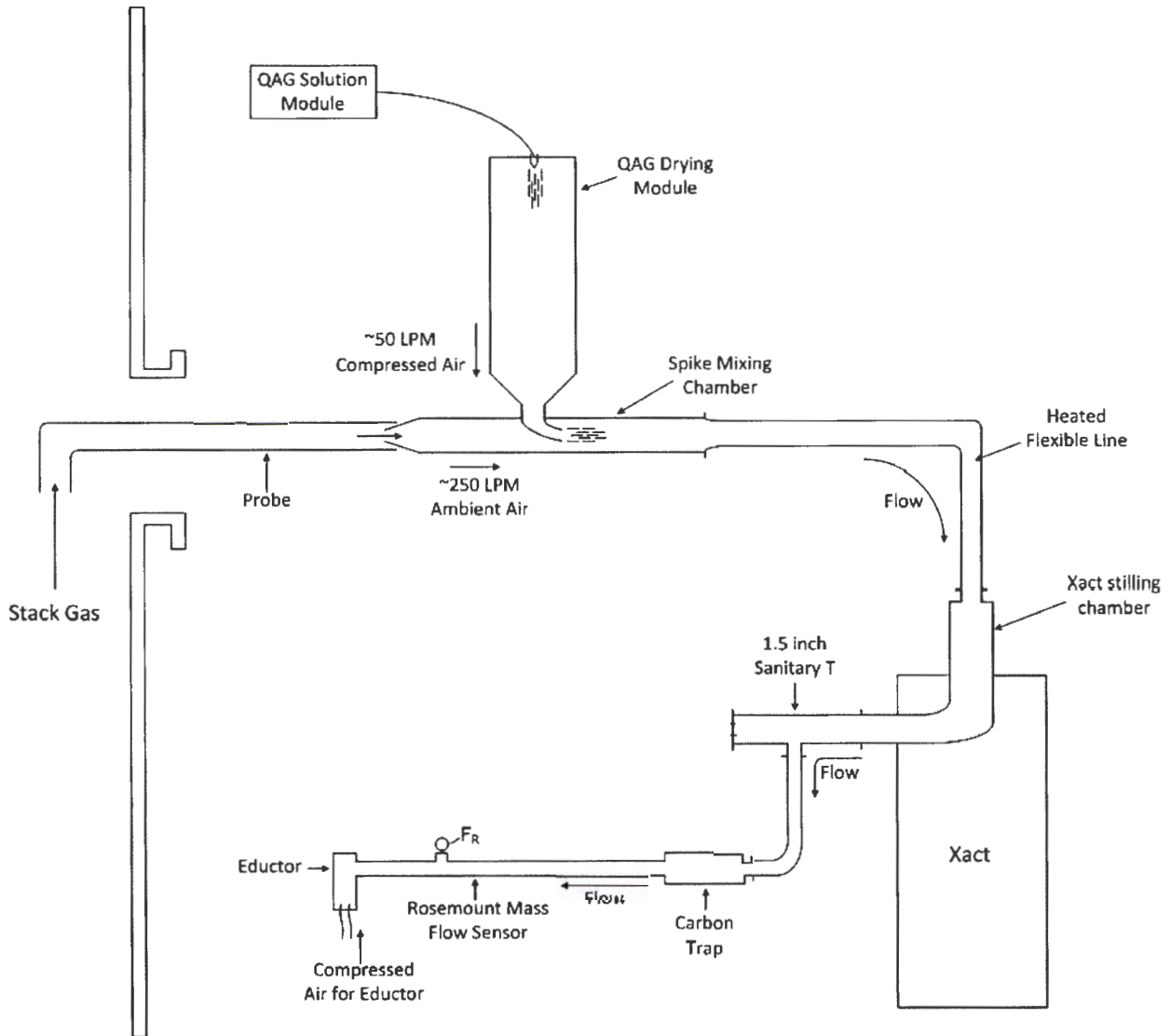
Figure 1. Dynamic Spiking Arrangement

3.8. Spiking Procedures

Dynamic spiking shall include at least three concentration levels not including a blank ranging in concentration from 1 to 5 μg/dscm. Aliquots of each solution used during spiking shall be provided to a third party selected by Quemetco for independent analysis. The Xact sampling

time shall be set so that the predicted spiked concentration is at least 10 times the detection limit. This means that for spiked concentrations under 2 µg/dscm hour long sampling and analysis intervals will be used and for concentrations between 2 and 5 µg/dscm 30 minute sampling and analysis times will be used. At least five data points will be gathered at each concentration level.

Because dynamic spiking involves stack effluent it is likely that arsenic will be present at appreciable concentrations relative to the concentrations being spiked. To evaluate these background concentration levels the testing procedures will alternate between sampling un-spiked stack effluent and spiked stack effluent. The average concentration of arsenic for the two un-spiked time periods immediately before and immediately after each spiked concentration will be calculated and this average concentration will be subtracted from the Xact reported aerosol concentration. The result will be compared to the expected QAG aerosol concentration. If the un-spiked native stack concentration for arsenic exceeds 20% of the expected spiked concentration then that data point may be removed. Up to 25% of the data points may be removed in this way.

After spiking the Xact reported aerosol (with native stack concentrations removed) will be plotted against the QAG reference aerosol concentration. A linear least squares fit will be generated using Excel or a similar commercially available program. The slope of the best fit line must be between 0.85 and 1.15 with a correlation coefficient of at least 0.9 and an intercept that is less than 20% of the applicable emission limit.

4. Maintenance
4.1. Cleaning
4.1.1. Probe and Transport Line Rinse

The Xact 640 probe and transport line will be cleaned at 6 to 8 week intervals, or more frequently, as was done during the demonstration program. In this procedure the probe is removed from the stack and decoupled from the transport line. Both the line and the probe are allowed to cool for several minutes. The end is capped and rinsed with a 2 to 5% nitric acid solution. After rinsing the rinsate is poured into an empty container.

Similarly, the transport line is rinsed by pouring one to two liters of 2 to 5% nitric acid solution into the transport line after capping one end. The other end is then capped (2 inch tri-clover

sanitary end caps) and the acid is swished back and forth in the line. The rinsate is then poured into an empty container and the transport line and tube are reinstalled.

4.1.2. Stilling Chamber Cleaning

The stilling chamber will also be cleaned on an annual basis (during the annual maintenance) following procedures outlined below.

1. Place Xact Controller in STANDBY mode and after 5 minutes set Xact Controller in OFF mode.
2. Switch off the power supply by setting MAIN DISCONNECT to OFF.
3. Remove the XRF Cabinet from the Xact enclosure (**Section 2.1.1**).
4. Disconnect the 1/4" Dilution Transport line from the stilling chamber.
5. Disconnect all power and thermocouple connectors for the stilling chamber heaters.
6. Loosen the roof clamp on the top of the system enclosure.
7. Slowly pull the stilling chamber down through the roof of the enclosure.
8. Using a 1% nitric acid solution, rinse the inside of the stilling chamber thoroughly.
9. Use DI-water to thoroughly wipe the inside of the stilling chamber to remove the nitric acid.
10. Let the stilling chamber dry thoroughly.
11. Reinstall the XRF Cabinet into the Xact 640 enclosure and ensure that all connections are properly connected.
12. Switch on the instrument by setting MAIN DISCONNECT to ON.

4.1.3. Cumulative Dust Build up Cleaning

As part of the routine preventive maintenance, it is strongly advised to inspect the instrument for dust build-up which could potentially interfere with components of the Xact system. The most common places prone to dust build-up inside the XRF Cabinet are the floor, around the sample deposition area, behind the tape drive, and around the high voltage power supply. Inside the system enclosure, dust may build-up on the floor, around the transformer and around the components of the flow module. Routine cleanup of the instrument is recommended
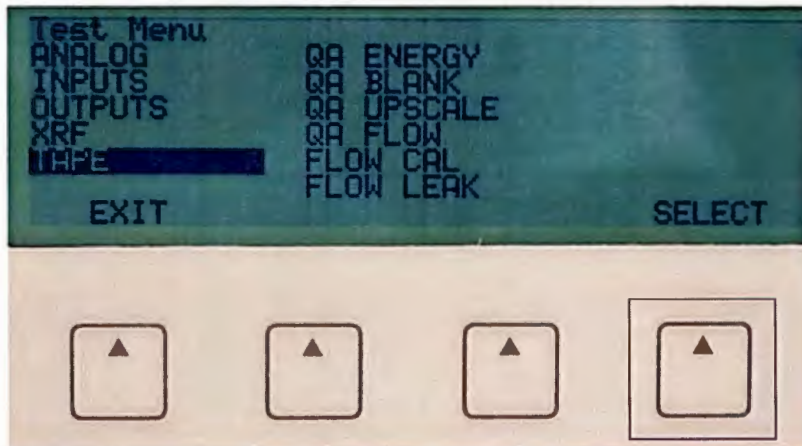
4.1.3.1. Sample Deposition Area Cleaning

The sample deposition area must be cleaned at least once 6 months (at the annual and semi-annual maintenance interval) to prevent possible build-up of dust/contaminants.
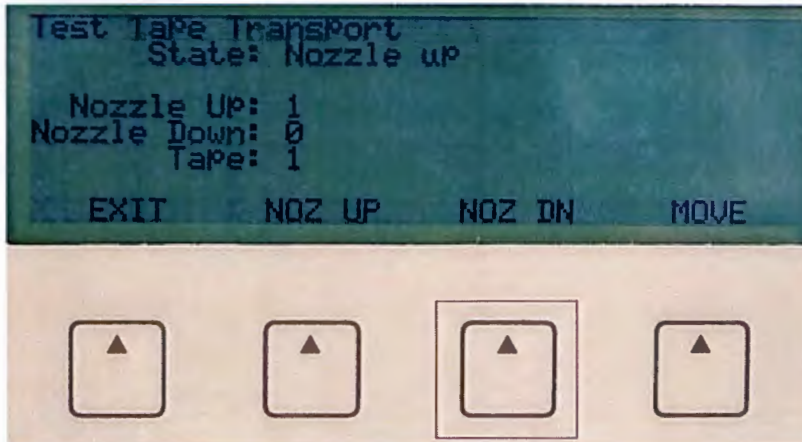
1. Place Xact Controller in STANDBY mode.
2. After X-ray power ramps down, set Xact Controller in OFF mode and wait for the vane and inlet heaters to cool down.
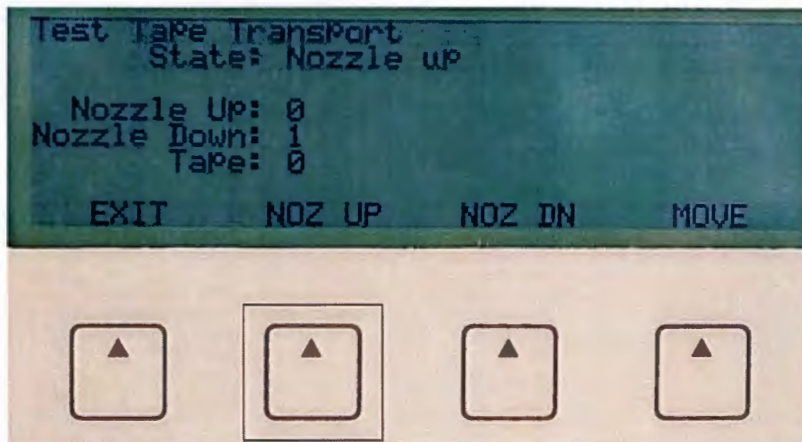3. On the Xact Controller home screen, press TEST.
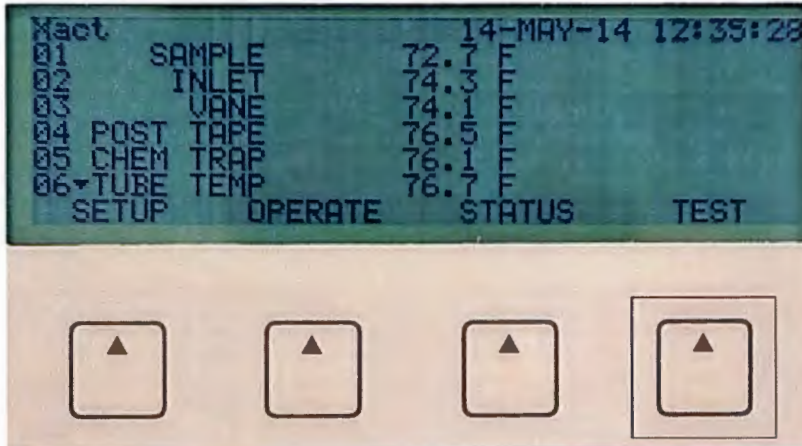
4. Highlight *TAPE* and press SELECT.

5. Press *Noz Dn* to release the filter tape from the tape slot in the mounting block.

Test Tape Transport
State: Nozzle up

Nozzle Up: 1
Nozzle Down: 0
Tape: 1

EXIT   NOZ UP   NOZ DN   MOVE

6. Remove the filter tape.

7. Wet an extra-low lint wipe (such as Kimwipe) or other lint-free cloth with deionized water (DI-water) and gently wipe the vane area to remove any dirt or grime that may have built up over Xact operation.

8. Replace the filter tape and press *NOZ UP*.

Test Tape Transport
State: Nozzle up

Nozzle Up: 0
Nozzle Down: 1
Tape: 0

EXIT   NOZ UP   NOZ DN   MOVE

9. Press EXIT or HOME (F1) to return to the home screen.

4.1.3.2. Fan Guard Dust Cleaning

The fan dust guard is located on the back of the XRF Cabinet (figure on right), which is located in the Xact 640 Enclosure. It is prone to dust accumulation and requires periodic cleaning depending on the environment Xact is operated in. The fan dust guard must be cleaned at least once every 6 months to prevent possible build-up of dust/contaminants.

1. Place Xact Controller in STANDBY mode and after 5 minutes set Xact Controller in OFF mode.
2. Switch off the power supply by setting MAIN DISCONNECT to OFF.
3. Remove the XRF Cabinet from the enclosure (**Section 2.1.1**).
4. On the back of the XRF cabinet remove the plastic bracket holding the dust guard in place.
5. Pull out the dust guard and clean with compressed air or water (if using water, make sure dust guard is completely dry before re-installing).
6. Re-install dust guard and plastic bracket on to the XRF Cabinet.
7. Reinstall the XRF Cabinet into the Xact 640 enclosure and ensure that all connections are properly connected.
8. Switch on the instrument by setting MAIN DISCONNECT to ON.

4.1.3.3. Sample Inlet Tube Cleaning

The sample inlet tube located in the sampling/analysis module should be cleaned annually to prevent contamination from any dust build-up.

1. Place Xact Controller in STANDBY mode and after 5 minutes set Xact Controller in OFF mode.
2. Switch off the power supply by setting MAIN DISCONNECT to OFF.
3. Remove the XRF Cabinet from the Xact enclosure.
4. Place a paper towel or clean rag over the vane in the sample area of the tape drive.
5. Use a bore brush to clean the inside of the inlet tube.

6. When finished, reinstall the XRF Cabinet into the Xact 640 enclosure and ensure that all connections are properly connected.

7. Switch on the instrument by setting the MAIN DISCONNECT to ON.

4.1.4. Cleaning the Air Conditioning Filters

The air filter for the air conditioning unit on the outside of the enclosure should be cleaned every month.

4.2. Maintenance and Parts Replacement Schedule

Table 1 below outlines the maintenance and parts replacement schedule for the Xact 640.

Activity	Sub Activity	Parts Replaced
Audit Q1 + Annual Maintenance	XRF Check	Pump Diaphram
	Flow Check	Carbon Trap
	Leak Check	Flow Control Valve
	Blank Check	Dry Sample Flow meter
	Air Conditioning Filter Clean	Clean Filters
	Instrument Inspection	
	Replace Parts	Clean Venturi Flow Sensor
	X-ray Tube	X-ray Tube Replace
	Air Compressor Filters	Air Compressor Replace
	Run Standards For Calibration	
	Create Calibration File	
	Replace Cu X-ray Filter	Cu Filter
	Travel	
Audit Q2	XRF Check	None Unless Visual Inspection Reveals a problem
	Flow Check	
	Leak Check	
	Blank Check	
	Air Conditioning Filter Clean	
Audit Q3 and Semi-annual maintenance	XRF Check	Pump Diaphram
	Flow Check	Carbon Trap
	Leak Check	Flow Control Valve
	Blank Check	Dry Sample Flow meter
	Air Conditioning Filter Clean	Clean Filters
	Instrument Inspection	
	Replace Parts	Clean Venturi Flow Sensor
	Travel	
Audit Q4	XRF Check	None Unless Visual Inspection Reveals a problem
	Flow Check	
	Leak Check	
	Blank Check	
	Air Conditioning Filter Clean	

4.3. Data Archive

The CES Data Archive program automatically archives the data files for all the samples into a zip file. This enables saving space on the computer hard drive. The program opens when the user logs in on the Host PC; the program can also be performed manually. The program should be run after every filter tape change. The following instructions detail how to run the CES Archive program manually.

1. Place Xact Controller in STANDBY mode.

2. On the Host PC, navigate to the folder C:/Program Files/Xact 640/CESArchive1

3. Locate and run CESArchive.exe.

4. Select *Zip Now* and specify the file name and location.

5. Press *OK* and all the Data Files, Log Files, and Process Data Archive files will be stored in a compressed file.

6. Close the CES Data Archive program