**California Environmental Protection Agency** 



# COMMUNITY AIR MONITORING BRANCH

STANDARD OPERATING PROCEDURE

FOR

# CONTINOUS DETERMINATION OF METALS IN AMBIENT AIR USING AN XACT 625i AMBIENT METALS MONITOR

CAMB SOP 450

First Edition

Monitoring and Laboratory Division November 2018

DISCLAIMER: Mention of any trade name or commercial product in this Standard Operating Procedure does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedure are for equipment used by the Air Resources Board laboratory.

## **California Environmental Protection Agency**



## **Approval of Standard Operating Procedure**

Title: Continuous Determination of Metals in the Ambient Air Using an Xact 625i Ambient Metals Monitor

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## Acronyms

ADAPT	Automated Data Analysis & Plotting Toolset
CES	Cooper Environment Services
EPA	Environmental Protection Agency
EC	Energy Condition
ECAL	Energy Calibration
l/m	liter per minute
m	Meter
µg/dscm	Microgram per Dry Standard Cubic Meter
MDL	Minimum Detection Limit
ng m <sup>-3</sup>	Nanogram per Cubic Meter
NIST	National Institute of Standards and Technology
PM	Particulate Matter
PM10	Particles with Aerodynamic Diameters ≤10 µm
PM2.5	Particles with Aerodynamic Diameters ≤2.5 μm
PM1	Particles with Aerodynamic Diameters ≤1 µm
QA	Quality Assurance
XRF	X-ray Fluorescence
XRS-FP	X-ray Spectrometry-Fundamental Parameter Program

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## 1.0 Scope

This document describes a method using the Xact 625i continuous ambient multi-metals monitor for continuous determination of metals in ambient particles and a custom designed Automated Data Analysis & Plotting Toolset for quick and easy examination of collected data (Cooper Environment Services, Oregon, U.S.A.). This document also covers safety and maintenance required to use this instrument. In this document, both terms of "this instrument" and "Xact 625i" refer to the Xact 625i ambient metals monitor. Please see the manufacturer's operating manual for a more detailed description of this monitor.

## 2.0 Summary of Method

- 2.1 This instrument is based on reel-to-reel filter tape sampling followed by nondestructive X-ray fluorescence (XRF) analysis of metals in the collected particulate matter (PM) deposit. This approach allows this instrument to analyze the present sample by the XRF system while the next sample is being collected. This concurrent operations of sampling and analysis enable this instrument to provide continuous, near-real-time metals concentrations. The instrument specifications are attached as Appendix 1. The front, back and interior views of this instrument are showed in Figures 2.1, 2.2 and 2.3.
- 2.2 This instrument can simultaneously measure up to 46 elements with an atomic number between Aluminum (Al) and Uranium (U). The metals measured by this instrument are showed in Table 2.1 as "A" (Analyte). The letter of "I" refers to the interference. The minimum detection limits for these compounds are provided in Appendix 2.
- 2.3 During sampling, this instrument draws ambient air through a PM size-selective inlet (such as PM2.5 and PM1) and across a filter tape at the sampling rate of 16.7 l/min. The sampling inlet tube is heated above dew point. The ambient particles are collected on the filter tape. The sampling time can be set in a range between fifteen minutes and four hours.
- 2.4 The collected PM on the tape is automatically advanced to be analyzed by the XRF system for selected metals at the completion of the sampling. The filter deposit is irradiated by an X-ray tube. This irradiation generates fluorescing X-rays. The energy of each of generated X-ray is specific to the element present in the sample which it is emitted from. The observed X-rays are counted and sorted by energy levels to produce a spectrum, a plot of intensity versus energy. The spectrum is then analyzed by an X-ray spectrometry-fundamental parameter (XRS-FP) program to determine metal mass concentrations in ng m<sup>-3</sup>.

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2.5 This instrument also includes a custom designed Automated Data Analysis & Plotting Toolset (ADAPT) and a meteorological sensor. ADAPT can automatically create intuitive and user-friendly data analysis graphics allowing for quick and easy examination of a large amount of collected data, such as the average concentration, diurnal profile for a user selected sampling period. ADAPT can further combine the collected metal data with the meteorological data to identify directionality of high metals concentrations occurring at the monitoring site.



Figure 2.1 Overview of Xact 625i components on the instrument front.

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Figure 2.2 Overview of Xact 625i components on the instrument back.

Inlet Fan

Inlet Fan

4

5

6

11

12

3

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Figure 2.3 Overview of Xact 625i sampling and X-ray analysis components.

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**Table 2.1** Elements labeled as "A" can be quantified by this instrument. The letter "X" marks the element measured under each energy condition. The letter of "A" refers to "Analyte" and the letter of "I" refers to "Interference".

				)	(act <sup>™</sup> 625	Calibration	Guide (6	526-00-17	'0101j)					
Element of Interest	Atomic Number	Compound on Micromatter Standard	Standard ID	Meas. Conc. µg/cm <sup>2</sup>	Meas. Mass ng	Condition 1	Purpose 1	Run Time (Sec)	Condition 2	Purpose 2	Run Time (Sec)	Condition 3	Purpose 3	Run Time (Sec)
Al	13	Al	42538	49.3	60519	×	Α	4400						
Si	14	SiO2	42539	36.8	45207	×	Α	2700						
Р	15	GaP	42552	20.6	25315	x	Α	2700						
S	16	InSx	42540	14.0	17185	×	Α	2700						
Cl	17	KCI	42541	24.4	30007	x	Α	360						
к	19	KCI	42541	25.3	31040	x	Α	360						
Ca	20	CaF2	42542	28.6	35180	×	Α	180						
Ti	22	Ti	42543	47.8	58689	x	Α	63						
v	23	v	42544	44.2	54223	x	Α	63						
Cr	24	Cr	42545	41.8	51291	x	Α	63						
Mn	25	Mn	42546	52.9	64938	x	Α	63	x	1	65			
Fe	26	Fe	42547	52.5	64472	x	1	63	x	Α	65			
Со	27	Со	42548	49.2	60396	x	1	63	x	Α	65			
Ni	28	Ni	42549	44.4	54497				x	Α	65			
Cu	29	Cu	42550	47.6	58412				x	Α	65			
Zn	30	ZnTe	42551	14.9	18334				x	Α	180			
Ga	31	GaP	42552	40.6	49898				x	Α	65			
Ge	32	Ge	42553	46.7	57363				x	Α	65			
As	33	GaAs	42554	30.1	36948				x	A	65			
Se	34	Se	42555	46.2	56734				x	Α	65			
Br	35	CsBr	42569	19.5	23908				x	Α	65			
Rb	37	Rbi	42556	20.4	25008				x	Α	65			
Sr	38	SrF2	42557	29.0	35552				x	Α	65			
Y	39	YF3	42558	30.6	37534				x	A	65			
Zr	40	ZrF4	42559	30.2	37048				x	Α	65			
Nb	41	Nb2O3	42560	41.9	51423				x	Α	65			
Mo	42	MoO3	42561	28.6	35151				x	Α	65			
Pd	46	Pd	42562	43.3	53128							x	Α	1400
Ag	47	Ag	42563	51.3	63042							x	Α	1200
Cd	48	CdSe	42564	29.6	36336							x	Α	2700
In	49	In	42565	53.1	65265							x	Α	1800
Sn	50	Sn	42566	57.3	70326	x	1	65				x	Α	1800
Sb	51	Sb	42567	49.4	60684	x	1	65				x	Α	1800
Те	52	Те	42568	51.7	63540	x	Α	64						
1	53	Rbi	42556	30.0	36846	x	Α	65						
Cs	55	CsBr	42569	31.6	38848	x	Α	65						
Ba	56	BaF3	42570	36.7	45019	x	Α	65						
La	57	LaF3	42571	36.8	45153	x	Α	65						
Ce	58	CeF3	42572	35.7	43875	x	Α	65						
w	74	WO3	42573	43.4	53284				x	Α	65			
Pt	78	Pt	42574	47.1	57897				x	Α	65			
Au	79	Au	42575	50.0	61460				x	Α	65			
Hg	80	AgHg	42576	26.7	32788				х	Α	65			
TI	81	TICI	42577	42.0	51558				x	Α	65			
Pb	82	Pb	42578	50.2	61635				x	Α	65			
Bi	83	Bi	42579	50.1	61542				x	Α	65			
As	33	PSR	PSR	N/A	N/A				х	1	65			

## 3.0 Personnel Qualification

Operation of this instrument does not require a technical background. However, the new operators need to undergo hands-on training provided by the current operator. The new operators also need to be familiar with the safety issues described in **4.0 Safety.** 

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## 4.0 Safety

Xact 625i has been designed to meet all applicable safety requirements for electrical and radiation devices, but potential hazards exist in the operation and service of this equipment. Any installations or repairs must be performed by Cooper Environmental Services (CES) employees or other properly trained technicians. Operators must receive safety training, including specific training on the hazards of radiation prior to operating the instrument.

- 3.1 Lifting Hazard. Do NOT lift Xact 625i using the handles on the front/rear of the instrument. These handles are designed to stabilize the instrument during lifting, instead of bearing the instrument weight. If used for lifting, the instrument can fall. Xact 625i is designed to be lifted by the two handles built into the sides of the instrument (Figure 2.1).
- 3.2 Electrical Shock Hazard. Xact 625i is designed to use up to 20 amps during normal operations. Voltages and currents used in electrical equipment can cause severe injury or death from electrocution. Avoid accidental contact with live circuit components. If electrical problems occur, contact an authorized distributor or CES. Do not attempt electrical repair.
- 3.3 Burn Hazard. Xact 625i has one heater that could present a potential burn hazard. The heater is installed in the sampling inlet and located immediately above the inlet hub (Figure 2.1). Allow sufficient cool down time after the instrument has stopped before any further interaction.
- 3.4 Radiation Hazard. The XRF instrument generates X-rays. Exposure to X-rays is injurious to health. Xact 625i is safe to use and is designed with multiple safeguards to prevent accidental radiation exposure.
  - 3.4.1 The safety features of Xact 625i include the following:
    - a. The X-ray source and detector are installed in a copper mounting block and enclosure to provide shielding from X-ray radiation.
    - b. The system access points, consisting of tape drive access door and system cover, are protected by interlock circuitry, which will shut down the X-ray source if opened or removed.
    - c. A removable enable/disable key can be used by the operator to control the power access to the XRF system.
    - A red X-RAYS ON light on the front of the instrument indicates the interlock circuit is energized and Xact 625i is producing X-ray radiation.
       If the X-ray bulb burns out, the X-ray circuit is disabled.
    - e. Factory radiation checks meet the above regulations prior to shipment. To maintain International Atomic Energy Agency (Specific Safety

Guide No. SSG-11), replacement parts may ONLY be obtained from the manufacturer.

- 3.4.2 Do not tamper with or attempt to defeat the interlock circuits. If any safety features are not operating correctly or have been damaged, do not operate Xact 625i. Contact CES personnel if further information is needed.
- 3.4.3 Do not energize the X-ray tube if the safety features of the instrument have been damaged or defeated.

## 5.0 Interferences

Wireless communications transmitters have occasionally been found to affect the X-ray detector, resulting in instrument malfunction. This instrument needs to be isolated from the communications hardware as much as possible.

## 6.0 Instrument Installation

- 6.1 Xact 625i should be installed in a secured and enclosed environment- protected shelter with temperatures controlled to 20±3°C (68±5.4°F). Dramatic changes in temperature can cause sampling and analysis readings to fluctuate.
- 6.2 Xact 625i should be connected to a filtered and EMI-shielded 110-230 VAC/ 50-60 Hz power source from the AC IN plug on the back of the instrument using the provided cable.
- 6.3 The diaphragm pump provided with Xact 625i resides outside the instrument and must remain within the shelter. It should be connected to the back of the module using port J4 (it is both a power and signal connection).
- 6.4 In accordance with U.S.EPA guidelines, the PM inlet should be at least 1 m above its supporting structure and 2 to 15 m off the ground with a minimum of 2 m of unobstructed air flow in all horizontal directions. The sampling tube should not exceed 5 m in length. Supporting braces for the inlet tube should be installed on the shelter roof if there is potential for swaying from high wind conditions.
- 6.5 The meteorological sensor provided with Xact 625i should be installed based on U.S.EPA guidelines on a meteorological tower using the strap and bracket provided at least 10 m above ground level and away from any vertical obstructions. The meteorological sensor should not be located near the shelter, Xact sampling inlet, or other obstructions (trees, buildings etc.).

- 6.6 The latitude and longitude values of Xact 625i location should be recorded for input into the ADAPT software provided.
- 6.7 The following checks (**Table 6.1**) should be performed before ambient sampling starts.

QA Check	During the Installation	See Section
QA Blank Check	х	9.2
Leak Check	х	9.3
Flow Check	Х	9.4
XRF Calibration Check	Х	9.6

Table 6 1	Xact 625i	Installation	$\cap \Delta$	Checks
	Add 0201	Installation	QA	CHECKS

# 7.0 Ambient Measurements: Sampling and Chemical Analysis

- 7.1 Ensure that Xact 625i is properly installed and connected.
- 7.2 Ensure that the filter tape has been properly installed.
- 7.3 The main breaker on the back of the instrument labeled MAINS (above the power switch) should be in the ON position.
- 7.4 Flip the power switch on the back of the instrument to the ON position (the switch will illuminate).
- 7.5 Push the power button on the front of the instrument (the button will illuminate). This switch powers the computer and cooling fans.
- 7.6 When prompted by Windows operating system, enter the following login information: Username: CES Password: ces
- 7.7 Click or tap the Xact Control icon on the instrument user interface desktop to launch the Xact Control program. The icon launches the Xact Control program and the associated XRS-FP program, which will run in conjunction with Xact Control in the background.
- 7.8 To set the Sampling Time for Xact 625i, navigate to Setting page (Xact Control>> Settings) and follow the instructions as highlighted in **Figure 7.1**:
  - 7.8.1 Click on "Enable Changes" button.
  - 7.8.2 Select the desired Sample Time. Sample Time options: 15, 30, 60, 120, 180 or

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#### 240 minutes.

7.8.3 Click on "Save" button.

Control									1.0.0.45	
Status	Data	System Settin	ngs	Calibration	Maint	enance		Adapt		_
Sample Time	15 🔻	Enable Changes							Save	
EC1 kV	25	EC2 kV	48	EC3 kV		48				
C1 uA	2000	EC2 uA	1000	EC3 uA		1000				
C1 Run Time %	20	EC2 Run Time %	40	EC3 Run 1	ime %	40				
C1 Run Time (s)	0	EC2 Run Time (s)	0	EC3 Run 1	ime (s)	0				
C1 Filter	Al 💌	EC2 Filter	Pd	▼ EC3 Filter		Cu	-			
C1 Auto Current	OFF 💌	EC2 Auto Current	OFF	The EC3 Auto	Current	OFF	*			
EC1 Filter Pos	430	Upscale Cr	900	Motor C	om Port	7				
C2 Filter Pos	830	Upscale Cd	900	Wind Co	m Port	1				
C3 Filter Pos	1230	Upscale Pb	900	Modbus	Com Port	3	_			
Upscale Pos	3250	11	10							
Ecal Pos	3400	Upscale Alarm %	10	_						
Ecal kV	27		900							
Ecal uA	1850	Flow Control	Act	*						
cal Time (s)	180	Flow Sum	Act	- Inlet Hea	ter %rh	45				

Figure 7.1 Setting Xact 625i Sample Time.

7.9 To set Xact 625i to begin sampling, navigate to home screen (Xact Control>> Status). Click on the "Run" button (**Figure 7.2**).

Status	Data	System	Settings	Calibration	Maintenance	Adapt	
	Stu	atus			Run		
peration				Xray Stab	u		
ogress				Interiock			
ter Wheel							
mamic Rod							

Figure 7.2: Setting Xact 625i to Run mode

- 7.9.1 During Run mode, Xact 625i operational parameters can be viewed by navigating to the System Page (Xact Control >> System >> Input/Output).
- 7.9.2 Elemental data reported by Xact 625i for each Sample Time can be

viewed/downloaded by navigating to Data page (Xact Control >> Data) (**Figure 7.3**). The "Back" and "Forward" navigation buttons enable viewing of archived ambient datasets.

- 7.9.3 To monitor instrument performance, daily QA Upscale data reported by Xact 625i for each day can be viewed/ downloaded by navigating to System page (Xact Control >> System >> QA Upscale).
- 7.9.4 Automated trends and directionality analysis of high metal concentrations can be examined by navigating to ADAPT page (Xact Control >> ADAPT). Please see Section 8.0 for a detailed description of features of the ADAPT.
- 7.9.5 Further data analysis can be performed by exporting the data in the comma delimited file (.txt) using the data page (Xact Control >> data) (Figure 7.3). The user desired sampling period can be selected using the "Start Data" and "End Date" boxes. The text box at the bottom of the screen displays the folder directory path for accessing the downloaded dataset.

Status	Data	System	Settings	Calibration	Maintenance	Adapt	
Back				Forward			
ample Start art of EC1: S Sym, At No, 13,592.0550 14,9.5085,10 15,0.0000,0 16,33.9125, 17,27.2865; 19,0.0000,0 ,20,17.3566, 22,0.0000,0 23,0.0275,1	Time = ,05/05 tartTime,05-05 Mass, Uncerta ,833.5587,2639 11.7983,42.390 0000,0.0000,, 0000,0.0000,, 3.7616,77.378 .3.7616,77.378	/2017 10:00:00 -17 10:17:34, inty, Concentra 9.4652,, 2,, 167,, 71,, 2,,	, tion				
		End Date					
Start Date							

Figure 7.3 Xact Control data page.

## 8.0 On-Site Data Analysis

- 8.1 The data can be quickly examined and analyzed using the included custom designed data analysis program of ADAPT (Xact Control >> ADAPT).
- 8.2 The navigation menu under ADAPT allows the user to select and view different analysis report pages (**Figure 8.1**). Each analysis report page

displays data from a user-selected time period. If the time period selected includes data collected using different sampling durations, ADAPT will exclude data that was collected with a sample time that differs from what Xact 625i is currently set to during the data analysis/examination.



Figure 8.1 ADAPT navigation menu.

8.2.1 Time period selection for data examination can be done in pages other than the Home Page (**Figure 8.2**). In addition, the selected data can be downloaded by clicking on the download icon. The description of each feature under ADAPT can also be found by clicking the help tool icon.



**Figure 8.2** Locations of the download and help icons and boxes for selection of the user desired sampling period.

- 8.2.2 The home page of the ADAPT software automatically populates with real-time data acquired from Xact 625i. However, the home page can only generate reports with data that was collected using the same sampling duration that is currently in use by Xact 625i. The home page includes a Metal Distribution Chart, which illustrates metals concentrations and wind data from the most recent sample. A Time Series Graph is also included, which displays cumulative metal concentration data over the past 48 hours of sampling for one or more selected metals.
- 8.2.3 The Average Concentration page displays average metal concentrations for a user-defined time period in a pie chart and the distribution of metal concentrations in a histogram.
- 8.2.4 The Temporal Trends page displays the time series of metal concentrations over a specified time period.
- 8.2.5 The Daily Distribution page displays the daily average concentration of each metal over a specified time period
- 8.2.6 The Multi-element Temporal page illustrates the time series of oneor-more metals normalized to each metal's highest concentration within the user- selected time period.
- 8.2.7 The Time of Day Trends page illustrates the distribution of metal concentrations for each sampling duration within a 24-hour period. The user-selected time period defines the data to be displayed.
- 8.2.8 The Day of Week Trends page illustrates the distribution of metal concentrations for each sampling duration across the week through a heat map feature. The user-selected time period defines the data to be displayed.
- 8.2.9 The Correlation Matrix page presents a series of scatter plots based on data from a user-defined time period. The correlation matrix plots the selected metal's concentration against the concentration of all other sampled metals. Each plot also displays the coefficient of correlation (R<sup>2</sup>) between the two corresponding metals.
- 8.2.10 The Meteorological Correlation graph plots a selected metal's concentration against wind direction. The graph can also be projected onto a Google geographic map. The latitude and longitude values in the map settings should be updated if the instrument is relocated.

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8.2.11 The Concentration Directionality graph illustrates a selected metal's frequency of occurrence from each wind direction. The graph can also be projected onto a Google geographic map. The latitude and longitude values in the map settings should be updated if the instrument is relocated.

## 9.0 Instrument Shutdown

- **9.1** On the Xact Control home screen (Xact Control >> Status), click on the "Stop" button to place the instrument on Stop mode (**Figure 9.1**). Wait for the 'Xray Status' message bar to indicate that the X-ray is ramping down. The red X-ray light on the front door will stop illuminating when ramp down is completed.
- **9.2** Close the Xact Control program and then close the XRF-FP program running in the background.
- 9.3 Shut down the on-board computer on Xact 625i using the start menu.
- **9.4** Flip the power switch on the back of the instrument to OFF position (the switch will stop illuminating).
- **9.5** Switch the main breaker on the back of the instrument labeled MAINS (above the power switch) to the OFF position.

Xad	ct Control								1.0.0.45	- 0	×
_	Status	Data	System	Settings	Calibratio	n Mai	ntenance	Adapt			_
					E					1	
		St	atus				Stop				
	Operation	Stoping acquisit	tion pump		,	Kray Status					
	Progress				1	nterlock					
	Filter Wheel										
	Dynamic Rod										
	Nozzle				1	Tape Remaining					

Figure 9.1 Xact control page for instrument shutdown.

## 10.0 Quality Assurance

To optimize the performance and life expectancy of the instrument, the routine checks and maintenance are recommended to perform at the following schedule.

Preventative Maintenance	Monthly	3 Months	Annually
QA Upscale Tracking	Х		
QA Blank Check	Х		
Leak Check <sup>1</sup>	Х		
Flow Check	Х		
Flow Calibration		Х	
XRF Calibration Check		Х	
General Cleanup <sup>2</sup>			Х
Xact Recalibration <sup>3</sup>			Х

#### Table 10.1 Preventative maintenance schedule.

Notes: 1. Perform anytime a connection break occurs in the flow system.

- 2. Examine tubing, enclosure, and components for particle build-up, rust or damage. Replace parts as needed.
- 3. Perform after new X-ray tube is installed.

#### 10.1 QA Upscale Tracking

- 10.1.1 Xact 625i performs daily automated QA checks to monitor the stability and performance of critical XRF processes. The following is a list of daily automated QA checks performed by the Xact 625i:
  - Energy Calibration: Performed once a day (00:00-00:15)
  - QA Upscale: Performed once a day (00:15-00:30)
- 10.1.2 Automated Energy Calibration is performed daily during 00:00-00:15. This process uses a Chromium (Cr) and Niobium (Nb) rod to ensure that the spectral peaks for each element are at the correct energy levels.
- 10.1.3 Automated QA Upscale is performed immediately after Energy Calibration daily during 00:15-00:30. This process uses chromium (Cr), lead (Pb), cadmium (Cd) and Niobium (Nb).
- 10.1.4 Daily QA Upscale data for these metals should not deviate by more than 10% outside the CES set point. These values are reported by Xact 625i for the four most recent days. They can be viewed by navigating to System page (Xact Control >> System >> QA Upscale)
- 10.1.5 Daily QA Upscale data can also be downloaded from this page by selecting the time period of interest.
- 10.1.6 Xact 625i users are recommended to routinely monitor QA Upscale values

to ensure that any instrument issues are promptly identified. If these value deviate by more than 10% outside the CES set point, Xact Control program will display an alarm flag.

- 10.1.7 Slow decline in QA Upscale value is expected over a long period of Xact 625i usage (1-3 years) due to decrease in excitation from the X-ray tube.
- 10.1.8 If the QA Upscale metal concentrations continue to deviate outside the specified limits over time, perform an XRF Calibration Check. If Xact 625i fails the XRF Calibration Check, contact a CES personnel or authorized distributor for instrument repair.

#### 10.2 **QA Blank Check**

- 10.2.1 To perform QA Blank Check, ensure that the tape is properly installed and that the instrument is in Stop mode.
- 10.2.2 Navigate to Maintenance page (Xact Control >> Maintenance)

10.2.3 Within 'XRF Check' section (Figure 10.1)

- a. Select "Manual" and "EC1".
- b. Enter the acquisition time for EC1 in seconds (Table 10.2).
- c. Click on "Run Acquisition" button.

	Data Sy	ystem S	ettings C	alibration	Maintenance	Adapt	
Nozzle Control	Up Down	·		XRF Check	Ti	me (s) 30	
				O Manual	○ EC1	Run Acquisiti	on
Tape Control	Reset Tape Remaining	2		<ul> <li>Ecal</li> <li>Upscale</li> </ul>	<ul> <li>EC2</li> <li>EC3</li> </ul>	Auto Analyz	e
				Status			
Dynamic Rod Home	Fwd	Rev 0		Save Spectra	c:/temp/output.c	es	
MOVE ROD TO		оркак		Init Xrs-fp	status		
Filter Wheel Home	Fwd	Rev		Xray Contro	4		
Move Filter To	1 2	3		Target kV	25	Actual kV 0.00	
				Target uA	250	Actual uA 0.00	

Figure 10.1 Settings for XRF Check.

10.2.4 Recommended acquisition time breakdown for each energy condition based on Xact 625i sample times are provided in **Table 10.2**.

Sample	Acqu	Acquisition Time (seconds)							
Time	EC1	EC2	EC3						
15	180	360	360						
30	360	720	720						
60	720	1440	1440						
120	1440	2880	2880						
180	2160	4320	4320						
240	2880	5760	5760						

 Table 10.2 Recommended acquisition time for each energy condition.

10.2.5 After acquisition is completed, the 'Status' window of 'XRF Check' section will prompt to save the spectra. Enter the folder path, save the file with .ces extension (example: c:/temp/EC1\_Blank.ces) and click "Save Spectra" (Figure 10.2).

Status	Data	System	Setting	gs Ca	alibration	Maintenance		Adapt		
Nozzle Control	Up	Down			XRF Check		Time (s)	30		
					O Manual	O EC1		Run A	cquisition	
Tape Control	Move Half	Move Full			O Ecal	O EC2				
	Reset Tape Res	maining			O Upscale	O EC3		Auto	Analyze	
					Status					1
Dynamic Rod Home	Fwd	Rev	0		Save Spectra	c:/temp/outp	ut.ces			1
Move Rod To	Ecal	Upscale			Init Xrs-fp	status				1
Filter Wheel Home	Fwd	Rev	0		Xray Contro	i -				
Move Filter To	1	2 3			Target kV	25	A	ctual kV	0.00	
					Target uA	250	Ac	tual uA	0.00	
Interlock	Open	Inlet	Heater	On	Ramp To Tar	get		Ra	mp To Zero	
	-			0.00						

Figure 10.2: Settings for saving spectra.

Status Da	ta Sy	stem	Setti	ngs	Calibratio	on	Maintenance		Adapt		
Nozzle Control	Up Down					KRF Check		Time (s)	30		]
						O Manual	○ EC1		Run A	cquisition	
Tape Control Mor	ve Half Move F	ull				O Ecal	○ EC2				'n.
	Reset Tape Remaining	)				O Upscale	○ EC3		Auto	o Analyze	
						Status					1
Dynamic Rod Home	Fwd	Rev	0			Save Spectra	c:/temp/outp	ut.ces			j
Move Rod To	Ecal	Upscale				Init Xrs-fp	status				
Filter Wheel Home	Fwd	Rev	0			Xray Contro	e.				
Move Filter To	1 2	3			_	Target kV	25	,	ctual kV	0.00	]
						Target uA	250	A	ctual uA	0.00	]
Interlock	Open	Inlet	Heater	On		Ramp To Tar	get		R	amp To Zero	
	Close			Off							

10.2.6 Wait until the status indicates that the X-rays have started to ramp down, then click "Auto Analyze" (**Figure 10.3**).

Figure 10.3: Automated Analysis of saved spectra.

- 10.2.7 The resulting EC1 elemental masses for Blank can be viewed in the process data file located at C:\process\_data\data1.txt
- 10.2.8 Repeat the process for EC2 and EC3.
- 10.2.9 Routinely monitor the QA Blank values to capture any systematic deviations with time.

#### 10.3 Leak Check

- 10.3.1 The following items are required for Leak Check procedure:
  - Sample inlet ball valve (included with the Xact 625)
  - Xact 625i Flow and Leak Calibration Check Form (Provided in Appendix; Contact CES personnel for self-calculating electronic version)
- 10.3.2 To perform a Leak Check, ensure that the filter tape is properly installed, the nozzle is in the UP position and that the instrument is in Stop mode.
  - a. Remove all PM inlets. Put the sample inlet ball valve over the PM inlet tube.
  - b. From Xact Control home screen, navigate to Calibration page (Xact

Control>> Calibration) and click on "Enable Changes" button.

- c. Click on "Flow Leak Check" toggle to ON (**Figure 10.4**). Slowly close the sample inlet ball valve.
- d. Wait until the pump turns off (**Figure 10.4**). Close Xact 625i's pump ball valve and click "Pump Off".
- e. Wait for the software to report the leak rate. The rate should be less than 150 mm Hg/min.

Status	Data	System	Settings	Calibration (Flow)	Maintenance	Adapt
Enat	le Changes					
Flow	Calibration	Flow Leal	k Check			Pump Control Pump On
Ambient Te	mp (C) 19.95	Ambient Pr	essure (mmHg) 764.	51 Flow 25 (L/m)		○ 15.0 (L/m
		Sample Pre	ssure (mmHg) 783.	79 Flow Std (L/m)		○ 16.7 (L/m
				Flow Act (L/m)		○ 18.4 (L/m
				Control Delta		

Figure 10.4 Settings for Leak Check.

#### 10.4 Flow Check

10.4.1 The following items are required for the Flow Check procedure:

- a. NIST certified reference flow meter
- b. Sample inlet ball valve (included with the Xact 625i)
- c. Flexible test tubing
- d. Xact 625i Flow and Leak Calibration Check Form (Provided in Appendix; Contact CES personnel for self-calculating electronic version)
- 10.4.2 To perform a Flow Check, ensure that the filter tape is properly installed, the nozzle is in the UP position and that the instrument is in Stop mode.
  - a. Remove all PM inlets. Put the sample inlet ball valve over the PM

inlet tube. The sample inlet ball valve should be in the open position.

- b. Connect the flow meter to the sample inlet ball valve using the flexible test tubing.
- c. From the Xact Control home screen, navigate to the Calibration page (Xact Control>> Calibration) and click on "Enable Changes" button.
- d. Select Xact 625i flow rate of 16.7 l/m (Figure 10.5) and wait for the pump to adjust the sample flow rates. When the "Control Delta" box reads 0.000±0.002, the flow has stabilized. This may take 15-20 seconds.
- e. Click on "Pump On" (Figure 10.5).

Status	Data	System Settin	ngs (	Calibration (Flow)	Maintenance	Adapt
Ena	ble Changes	)				
Flow	Calibration	Flow Leak Check				Pump Control
			764.54	٦		Fumption
Ambient T	emp (C) 19.95	Ambient Pressure (mm	Hg) 764.51	Flow 25 (L/m)		이 15.0 (L/m)
		Sample Pressure (mmH	lg) 783.79	Flow Std (L/m)		0 16.7 (L/m)
				Flow Act (L/m)		이 18.4 (L/m)
				Control Delta		



- f. Record the "Flow Act (I/m)" value from the Xact Control program into 'Xact 625i Flow and Leak Calibration Check Form'.
- g. Record the reference flow rate from the flow meter into 'Xact 625i Flow and Leak Calibration Check Form'.
- h. Calculate and record the percentage difference between the flow rates into 'Xact 625i Flow and Leak Calibration Check Form'.
- i. Repeat Steps *e h* for Xact 625i flow rate of 15.0 l/m and 18.4 l/m.
- j. Repeat Steps *e i* twice for a total of three readings per Xact 625i flow

rate.

 k. Calculate the average of all the recorded percentage differences. If the average percentage difference is less than 1%; and, temperature and pressure are within 10% of the reference values, Xact 625i has passed the Flow Check. If Xact 625i fails the Flow Check, perform Flow Calibration.

#### 10.5 Flow Calibration

- 10.5.1 The following items are required for the Flow Calibration procedure:
  - a. NIST certified reference flow meter
  - b. Sample inlet ball valve (included with the Xact 625)
  - c. Flexible test tubing
- 10.5.2 To perform the Flow Calibration, ensure that the filter tape is properly installed, the nozzle is in the UP position, and the instrument is in Stop mode.
- 10.5.3 Remove all PM inlets. Put the sample inlet ball valve over the PM inlet tube. The sample inlet ball valve should be in the open position.
- 10.5.4 Connect the flow meter to the sample inlet ball valve using the flexible test tubing.
- 10.5.5 From Xact Control home screen, navigate to Calibration page (Xact Control>> Calibration) and click on the "Enable Changes" button.
- 10.5.6 Click on "Flow Calibration" to switch to ON and then click on "Pump On" (**Figure 10.6**).
- 10.5.7 The Xact Control program will provide the directions for the process in the notification box to enter values manually in the input box (Figure 10.6).
  - a. When prompted in "Notification Box", input the reference ambient temperature from the flow meter in "Data Input Box" and click on "Enter Data".
  - b. When prompted in "Notification Box", input the reference ambient pressure from the flow meter in "Data Input Box" and click on "Enter Data".
  - c. The pump will adjust the sample flow rates to each of the three predefined calibration flow rates (15.0 l/m, 16.7 l/m and 18.4 l/m). Wait for the flow to stabilize i.e., wait for the "Control Delta" box to read 0.000±0.002; this may take 15-20 seconds. When prompted, input the

reference flow rate from the flow meter and click on "Enter Data".

10.5.8 Click on "Pump Off" and then click on "Flow Calibration" to switch to OFF. Xact Control program will automatically calibrate Xact 625i's flow system.

Control						1.0.0.57
Status	Data	System	Settings	Calibration (Flow)	Maintenance	Adapt
Enat	ble Changes					
Flow Off	Calibration	Flow Lea	k Check			Pump Control
Ambient Te	emp (C) 19.95	Ambient P	ressure (mmHg) 764	.51 Flow 25 (L/m)		○ 15.0 (L/m)
		Sample Pro	essure (mmHg) 783	.79 Flow Std (L/m	)	이 16.7 (L/m)
				Flow Act (L/m	)	이 18.4 (L/m)
				Control Delta		
No	tification Box					
Da	ta Input Box		Enter Data			

Figure 10.6 Setting for Flow Calibration.

#### **10.6 XRF Calibration Check**

- 10.6.1 Element Standards are pure element standards and are free from any interfering elements. These standards contain a known amount of each element on a Nucleopore substrate that is mounted on a plastic ring. Items needed for this procedure include:
  - a. Xact 625i Calibration Guide (Provided with Xact 625i). <u>The calibration</u> <u>guide is specific to each standard set and needs to be updated when a</u> <u>new standard set is provided.</u>
  - b. Element Standards and holder (Provided with Xact 625i)
  - c. CES Xact 625i XRF Calibration Check Form (Provided in Appendix)
  - d. Nitrile gloves
- 10.6.2 'Xact 625i Calibration Guide' provides the Energy Condition and Acquisition Time to be used for each Element Standard during this procedure. <u>A sample of the Xact 625i Calibration Guide is shown in Table</u>
   <u>10.7. The calibration guide used for calibration should be provided with the</u>

standard set.

10.6.3 Each Element Standard is marked as an Analyte (A) or Interfering (I) element. This procedure should only be performed with Element Standards marked with A for analyte. The procedure should be performed with at least 5 recommended standards, 2 each for EC1 and EC2 and 1 for EC3 energy condition, are recommended. The user can perform the check with additional standards to populate 'Xact 625i XRF Calibration Check Form'.

	Xact <sup>TM</sup> 625 Calibration Guide (626-00-170101j)													
		Compound on		Meas.	Meas.			Run Time			Run Time			Run Time
Element	Atomic	Micromatter	Standard ID	Conc.	Mass	Condition 1	Purpose 1	(Sec)	Condition 2	Purpose 2	(Sec)	Condition 3	Purpose 3	(Sec)
of Interest	Number	Standard		µg/cm²	ng			(500)			(000)			(000)
Al	13	Al	42538	49.3	60519	×	A	4400						
Si	14	SiO2	42539	36.8	45207	×	A	2700						
Р	15	GaP	42552	20.6	25315	×	A	2700						
S	16	InSx	42540	14.0	17185	×	A	2700						
Cl	17	KC	42541	24.4	30007	×	A	360						
К	19	KCI	42541	25.3	31040	×	A	360						
Ca	20	CaF2	42542	28.6	35180	×	A	180						
11	22	11	42543	47.8	58689	X	A	63						
V	23	V	42544	44.2	54223	X	A	63						
Cr Mar	24	Cr M-	42545	41.8	51291	X	A	63	~					
IVIN	25	IVIN	42546	52.9	64938	×	A	63	×		65			
Fe	20	Fe	42547	52.5	64472	×	<u> </u>	63	×	A	65			
NI	27	CO Ni	42548	49.2	54407	<u> </u>	<u> </u>	05	- ÷	A 	65			
- NI Cu	28	NI CII	42549	44.4	54497				~	A	65			
70	29	ZnTo	42550	47.0	19224				~ ~	A 	180			
63	21	GaP	42551	14.5	10554				~	A .	100			
Ge	32	Ga	42552	46.7	57363				×	A	65			
Δε	32	GaAs	42554	30.1	36948				x	Δ	65			
Se	34	Se	42555	46.2	56734		<u> </u>		X	Δ	65			
Br	35	CsBr	42569	19.5	23908				x	Δ	65			
Rh	37	Bhi	42556	20.4	25008				x	Δ	65			
Sr	38	SrE2	42557	29.0	35552				x	Δ	65			
Y	39	YF3	42558	30.6	37534				x	Δ	65			
7r	40	7rF4	42559	30.2	37048				x	Δ	65			
Nb	41	Nb2O3	42560	41.9	51423				x	A	65			
Mo	42	MoO3	42561	28.6	35151				x	A	65			
Pd	46	Pd	42562	43.3	53128							x	Α	1400
Ag	47	Ag	42563	51.3	63042							x	A	1200
Cd	48	CdSe	42564	29.6	36336							x	A	2700
In	49	In	42565	53.1	65265							x	Α	1800
Sn	50	Sn	42566	57.3	70326	x	1	65				x	Α	1800
Sb	51	Sb	42567	49.4	60684	x	1	65				x	Α	1800
Те	52	Те	42568	51.7	63540	x	А	64						
1 I.	53	Rbi	42556	30.0	36846	x	Α	65						
Cs	55	CsBr	42569	31.6	38848	x	Α	65						
Ba	56	BaF3	42570	36.7	45019	x	Α	65						
La	57	LaF3	42571	36.8	45153	x	Α	65						
Ce	58	CeF3	42572	35.7	43875	x	Α	65						
w	74	WO3	42573	43.4	53284				x	Α	65			
Pt	78	Pt	42574	47.1	57897				x	Α	65			
Au	79	Au	42575	50.0	61460				x	Α	65			
Hg	80	AgHg	42576	26.7	32788				x	Α	65			
TI	81	TICI	42577	42.0	51558				x	Α	65			
Pb	82	Pb	42578	50.2	61635				x	Α	65			
Bi	83	Bi	42579	50.1	61542				x	Α	65			
As	33	PSR	PSR	N/A	N/A				x	1	65			

Table 10.7 Xact 625i Calibration Guide with 'Mass (ng)' highlighted

- 10.6.4 To perform XRF Calibration Check, ensure that the instrument is in Stop mode.
- 10.6.5 Check to confirm if the instrument underwent the Xact 625i Daily Automated QA Checks (performed every midnight) within the past 24 hours by navigating to System page (Xact Control >> System >> QA Upscale). If more than 24 hours have lapsed since the last Xact 625i Daily Automated QA Checks, manual Energy Calibration must be performed before proceeding. The procedure for performing manual Energy Calibration is provided here:
  - a. Navigate to the Maintenance page (Xact Control >> Maintenance)
  - b. Within 'XRF Check' section, select "Ecal" (Figure 10.7).
  - c. Click on "Run Acquisition" button. The status bar will display operational messages.

Control						1.0.0.45
Status	Data	System	Settings	Calibration	Maintenance	Adapt
Nozzle Control	Up C	own		XRF Check	۲	Time (s) 30
				O Manu	al O EC1	Run Acquisition
Tape Control	Move Half Mo	ive Full		O Ecal	O EC2	Auto Analyze
	кезестаре кеша	aung		Status		
Dynamic Rod Home	Fwd	Rev	0	Save Spectra	c:/temp/output	.ces
Move Rod Io	CCar	Opscale		Init Xrs-fj	p status	
Filter Wheel Home	Fwd	Rev	0	Xray Cor	itrol	
Move Filter To	1	2 3		Target kV	25	Actual kV 0.00
				Target u	A 250	Actual uA 0.00
Interlock	Open	Inlet H	leater	On Ramp Te	o Target	Ramp To Zero
	Close			Off		

d. Wait for the X-rays to ramp down. The process may take ~3-10 minutes.

Figure 10.7 Settings for performing Energy Calibration manually.

- 10.6.6 Unlock the front door of Xact 625i to access the interior of the instrument. If the Xact 625i has been recently analyzing samples, the sample tube may be hot! Wait for sample tube to cool down.
- 10.6.7 Remove the filter tape from the analysis block (Figure 10.8).

10.6.8 Place the first Element Standard into the element standard holder; the deposit is on the side of the support that is attached to the ring. Align the index mark on the Element Standard with the index mark on the element standard holder. The Element Standard should be oriented with the plastic ring up (**Figure 10.8**).



Figure 10.8 Element Standard Placement

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10.6.9 Place the element standard holder in the slot under the filter wheel in the analysis zone (**Figure 10.9**). The element standard holder takes the place of the filter tape in the analysis block. Be sure to place the element standard holder as far into the analysis zone as possible.



Figure 10.9 Element Standard Placement

- 10.6.10 Close and lock the front door of Xact 625i.
- 10.6.11 On Xact Control program, navigate to Maintenance page (Xact Control >> Maintenance). 'Xact 625i Calibration Guide' provides the Energy Condition and Acquisition Time to be used for each Element Standard during this procedure. The steps described here are for Ca Element Standard only.
- 10.6.12 Within 'XRF Check' section (**Figure 10.10**)
  - a. Select "Manual" and "EC1" for the Ca Element Standard.
  - b. Enter '65' acquisition time into the time text box.
  - c. Click on "Run Acquisition" button.

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Status	Data	Sys	tem	Setti	ngs	Calibration	Maintenance	Adapt		
										\
Nozzle Control	Up	Down				XRF Check	[	Time (s) 30		1
						O Manua	O EC1	Run A	cquisition	
Tape Control	Move Half	Move Ful				Cal	O EC2			
	Reset Tape R	emaining				O Upscal	e O EC3	Auto	Analyze	
Durantic Red Home						Status				
Dynamic Kod Home	rwa		NEV	0		Save Spectra	c:/temp/output	ces		Ϊ
Move Rod To	Ecal		Upscale			Init Xrs-fp	status			
Filter Wheel Home	Fwd		Rev	0		Xray Cont	rol			
Move Filter To	1	2	3			Target kV	25	Actual kV	0.00	
						Target uA	250	Actual uA	0.00	
Interlock	Open		Inlet	Heater	On	Ramo To	Target	Ra	mp To Zero	

d. To initiate acquisition, X-rays will ramp to full power (~1 minute).

Figure 10.10 Settings for XRF Calibration Check for Ca Element Standard

10.6.13 After acquisition is completed, the 'Status' window of 'XRF Check' section will prompt to save the spectra. Enter the folder path, save the file with .ces extension (example: C:/temp/Ca\_data.ces) and click "Save Spectra" (Figure 10.12).

Control											1.0.0.4	45
Status	Data	S	/stem	Setti	ngs	Calibrat	ion	Maintenance		Adapt		
Nozzle Control	Up	Down	1				XRF Check		Time (s)	30		]
							O Manual	O EC1		Pup A	quisition	
Tape Control	Move Half	Move I	ull				O Ecal	O EC2		Kull A	cquisition	
	Reset Tape	Remainin	9				O Upscale	⊖ EC3		Auto	Analyze	
						. [	Status					
Dynamic Rod Home	Fv	rd	Rev	0			Save Spectra	c:/temp/outp	ut.ces			]
Move Rod Io		a	opscale				Init Xrs-fp	status				
Filter Wheel Home	Fi	vd	Rev	0			Xray Contro	a.				
Move Filter To	1	2	3				Target kV	25	А	ctual kV	0.00	]
						1	Target uA	250	A	ctual uA	0.00	]
Interlock	Op	en	Inlet	Heater	On		Ramp To Ta	rget		Ra	mp To Zero	
	Clo	ise			off							

Figure 10.11 Settings for saving spectra.

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10.6.14 Wait until the status indicates that the X-rays have started to ramp down, then click "Auto Analyze" (**Figure 10.12**).

Status D	ata	System	Settings	Calibrat	ion	Maintenance		Adapt	<b>i</b>	_
					XRE Check		Time (s)	20		
Nozzie Control	Up Do	own					1111e (3)			
					O Manual	O EC1		Run A	Acquisition	
Tape Control M	ove hair Mov	re Full			O Ecal	O EC2		Auto	o Analyze	
	Reset Tape Remain	ning			O Upscale	O EC3				Ŀ.
				_	Status					
Dynamic Rod Home	Fwd	Rev	0		Save Spectra	c:/temp/outp	ut.ces			
Move Rod To	ECal	Opscale			Init Xrs-fp	status				
Filter Wheel Home	Fwd	Rev	0		Xray Contro	ы				
Move Filter To	1	2 3			Target kV	25	Ac	tual kV	0.00	
					Target uA	250	Ac	tual uA	0.00	
Interlock	Open	Inlet H	eater	On	Ramp To Ta	rget		R	amp To Zero	
	Class			Off						

Figure 10.12 Automated analysis of saved spectra.

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Figure 10.13 Sample process file with reported mass (ng).

- 10.6.15 Navigate to C:/process\_data/data1.txt to obtain the reported mass. For example, Ca reported mass in **Figure 10.13** is 35577.75 ng. The Ca Element Standard acquired in EC1 energy condition will be saved in data1.txt. Data for EC2 and EC3 will be saved in data2.txt and data3.txt respectively.
- 10.6.16 Record this value in the 'Reported Mass (ng)' column into the 'Xact625i XRF Calibration Check Form'. On Xact Control, click "Cancel Acquisition".
- 10.6.17 Copy the value from 'Mass (ng)' column for Ca from 'Xact 625i Calibration Guide' into the 'Mass (ng)' column into the 'Xact 625i XRF Calibration Check Form'.
- 10.6.18 Repeat the process for Element Standards listed in 'Xact 625i XRF Calibration Check Form'.
- 10.6.19 Calculate and record the percentage difference between corresponding mass (ng) values. If percentage difference is within ±10%, then the XRF Calibration Check is successful. If Xact 625i fails the XRF Calibration Check, contact a CES personnel or authorized distributor.

## 11.0 Maintenance

#### 11.1 Filter Tape Replacement.

11.1.1 Xact 625i requires the user to manually install new filter tape rolls. The frequency of filter tape changes depends on the sampling time, as shown in **Table 11.1**. The filter tape change takes approximately 15 minutes to complete.

Sample Time in Minutes	Approximate Frequency of Tape Change
15	Once every 6 days
60	Once every 25 days
240	Once every 3 months

#### **Table 11.1** Approximate frequency of sampling tape change.

- 11.1.2 The instructions for changing filter tape are provided below.
  - a. On the Xact Control home screen (Xact Control >> Status), click on the "Stop" button to place the instrument in Stop mode (Figure 11.1). Wait for the 'Xray Status' message bar to indicate that X-ray is ramping down. The red X-ray light on the front door will no longer be illuminated when ramp down is completed.

									~
Xact Control							1.0.0.45		
Status	Data	System	Settings	Calibration	Maintenance	Adapt			_
_								1	
	Sta	itus			Stop	,			
								J	
Operation	Stoping acquisit	ion pump		Xray Statu	5				
Progress				Interlock					
Filter Wheel									
Dynamic Rod									
Nozzle				Tape Rema	aining				

Figure 11.1 "Stop" button to place Xact 625i in Stop mode.

b. Unlock the front door of Xact 625i to access the interior of the instrument. If the Xact 625i has been recently analyzing samples, the sample tube may be

hot! Wait for the sample tube to cool down.

c. Remove the Feed and Uptake reel covers of the filter tape (Figure 11.2).



Figure 11.2 Schematic of filter wheel assembly for changing filter tape.

d. If there is tape remaining on the feed roll, cut the tape at the right side of the analyzer block. Wind the remaining tape to the uptake spool. Remove the used tape before inserting a fresh roll.

- e. Place the spool containing the clean tape on the feed reel.
- f. Place an empty spool on the uptake reel to collect the used tape.
- g. Place the filter tape from left to right along the filter wheel assembly shown in **Figure 11.2** and in the order listed below:
  - a) Feed reel
  - b) Encoder wheel and first guide wheel
  - c) Through nozzle assembly
  - d) Through tape slot under filter wheel on analyzer block
  - e) Over second guide wheel
  - f) Uptake reel
- h. Attach the filter tape to the uptake spool on the feed reel using adhesive tape.
- i. Replace the reel covers. Do not overtighten.
- j. Close and lock the Xact 625i front door.
- Navigate to the Xact Control home screen (Xact Control >> Status). Click on the "Run" button to set Xact 625i to Run mode.

#### 11.2 Filter Tape Break

If the filter tape breaks, Xact 625i will automatically enter Stop mode and issue an alarm on the Xact Control home screen. The filter tape can be repaired by attaching the broken ends together with adhesive tape.

- 11.2.1 Follow Steps *a c* from Section 11.1.2.
- 11.2.2 Cut clean edges on the unused tape and the used tape to repair jagged edges from the break.
- 11.2.3 Follow Step *h* in **Section 11.1.2** to feed the clean, unused tape remaining on the spool through the filter reel assembly.
- 11.2.4 Overlap the used and unused sections of tape by approximately 2-3 inches. Apply adhesive tape to the upper and lower portions of the overlapping filter tape.
- 11.2.5 Roll the uptake spool clockwise to secure the repaired tape to the uptake spool.
- 11.2.6 Follow Steps *i-k* in Section 11.1.2.

#### 11.3 Change the group of target metals

Xact recalibration is performed when a new X-ray tube is installed or if the user wants

to change the Xact 625i setting to monitor elements different than those the instrument was calibrated for at the factory. Since Xact 625i is a precisely calibrated instrument for user requested elements of interest, the entire instrument must be recalibrated. Such Xact recalibration must be performed by CES personnel or authorized distributor only. For details on Xact recalibration, contact CES personnel or an authorized distributor.

## 12.0 Alarms and Troubleshooting

- **12.1** The Xact Control program displays color-coded alarms for Xact 625i on the status button of the home screen.
  - The Status button on the Xact Control home page displays alarm codes (100-110) in RED for system errors.
  - The Status button on the Xact Control home page displays alarm codes (200-203) in YELLOW for system warnings.
- **12.2 Table 12.1** provides the list of alarm codes, notification messages from the Xact Control program, and their possible resolutions. If system errors cannot be resolved, contact a CES personnel or authorized distributor.

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Table 12.1	Xact 625i	Alarm	Codes.
------------	-----------	-------	--------

Code	e Definit	ion	Xact	Control	Recommended Resolutior	
			Notificati	on Message	Procedure	
		Actual Xra	ay Voltage too	) hi	Hardware failure or software bug; contact your service representative	
100	Yray M				<ul> <li>Check the interlock circuit is closed (Front door is closed; enclosure cover is installed and screwed in place; Enable X-rays key is in ON position; X- RAYS ON light is installed)</li> </ul>	
100	xray v	Actual Xray Voltage too low			<ul> <li>Check the x-ray tube cables are connected to the High Voltage Power Supply (HVPS). This</li> </ul>	
					includes the large, black high voltage cable and the 2 white filament cables	
		Actual Xra	ay Current to	o hi	Hardware failure or software bug; contact your service representative	
					<ul> <li>Check the interlock circuit is closed (Front door is closed; enclosure cover is installed and screwed in place; Enable X-rays key is in ON position; X- RAYS ON light is installed)</li> </ul>	
101	Xray I	Actual Xra	ay Current to	o low	<ul> <li>Check the x-ray tube cables are connected to the High Voltage Power Supply (HVPS). This</li> </ul>	
					includes the large, black high voltage cable and the 2 white filament cables	

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 Table 12.1 Xact 625i Alarm Codes (continued).

Code	Definition	Xact Control Notification Message	Recommended Resolution Procedure
102	Tube Temp	Error: Tube Temp > 45 C. Tube Temp = NNN.NN	<ul> <li>Check the temperature of the shelter is being properly controlled between 20 and 25 C</li> <li>Make sure the tubing between the x-ray blower fan and the x-ray tube is properly installed</li> <li>Check the blower fan filter for excess dust buildup. Clean if necessary</li> <li>Check fan filters on the back of the instrument for excess dust buildup. Clean if necessary</li> <li>Check the x-ray tube Thermistor is connected to the x-ray tube</li> <li>Check the x-ray tube thermistor is not broken or disconnected</li> </ul>
		Warning: Tube Temp > 40 C	
103	Enclosure Temp	Error: Enclosure Temp > 40 C. Enclosure Temp = NNN.NN	<ul> <li>Check the temperature of the shelter is being properly controlled</li> <li>Check the fan filters on the back of the instrument for excess dust buildup. Clean if necessary</li> <li>Check the Enclosure Temp Thermistor is connected to the frame of the instrument</li> <li>Check the Enclosure Temp thermistor is not broken or disconnected</li> </ul>
104	Таре	Error: Move tape Full/Half	<ul> <li>Tape is broken</li> <li>Check tape is properly installed</li> <li>Try moving manually and watch for tape spool movement</li> <li>Check all connectors on EZ-AXIS board are properly seated</li> </ul>
105	Pump	Error: Unable to Start/Stop pump	<ul> <li>Check the pump power cable is properly connected</li> </ul>

Code	Definition	Xact Control Notification Message	Recommended Resolution Procedure		
106	Filter wheel	Error: Move FW (pos-NNN)	<ul> <li>Check Filter Wheel power cable is properly connected between motor and EZ-AXIS board</li> <li>Check Filter Wheel home sensor cable is properly connected between sensor board and EZ-AXIS board</li> <li>Check EZ-AXIS board power connector and usb connector</li> </ul>		
107	Dynamic Rod	Error: Move DR Failed	<ul> <li>Check Dynamic Rod power cable is properly connected between motor and EZ-AXIS board</li> <li>Check Dynamic Rod home sensor cable is properly connected between sensor board and EZ-AXIS board</li> <li>Check EZ-AXIS board power connector and usb connector</li> <li>Check that Dynamic Rod is sticking during movement</li> </ul>		
108	Nozzle	Error: Move Nozzle Up/Dn	<ul> <li>Check spring below vane has not seized or broken</li> <li>Check EZ-AXIS board power connector and usb connector</li> <li>Check all connectors on EZ-AXIS board are properly seated</li> </ul>		
109	Ecal	Error: Invalid Ecal Gain = NNN.NN Offset = NNN.NN Gain must be [-100,100], Offset [0,50]	<ul> <li>Check usb and power cable to detector are properly connected</li> <li>Check that Dynamic Rod is sticking during movement</li> </ul>		
110	Software	Error: Internal State (N)	<ul> <li>Possible software failure. Shut down and restart instrument</li> </ul>		
200	Upscale Cr	Warning: Cr upscale value hi/low. Cr = NNN.NN			
201	Upscale Pb	Warning: Pb upscale value hi/low. Pb = NNN.NN	<ul> <li>Perform an XRF Calibration Check if problem persists</li> </ul>		
202	Upscale Cd	Warning: Cd upscale value hi/low. Cd = NNN.NN	<ul> <li>Check that Dynamic Rod is sticking during movement</li> </ul>		
203	Upscale Nb	Warning: Nb upscale value hi/low. Nb = NNN.NN			

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## 13.0 References

Xact 625i Ambient Metals Monitor Operation Manual, Cooper Environmental Service, 2017. (The electronic version of the operational manual is not currently available on the manufacturer's website)

U.S. Environmental Protection Agency. *Quality Assurance Guidance Document* 2.12: Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods. Research Triangle Park, NC. January 2016. (EPA-454/B-16-001) (https://www3.epa.gov/ttnamti1/files/ambient/pm25/qa/m212.pdf)

U.S. Environmental Protection Agency. *Meteorological Monitoring Guidance for Regulatory Modeling Applications*. Research Triangle Park, NC. February 2000. (EPA-454/R-99-005) (https://www3.epa.gov/scram001/guidance/met/mmgrma.pdf)

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# Appendices

# Appendix 1. Xact 625i Specifications

Measurement method	Based on EPA Method IO 3.3: Determination					
	of Metals in Ambient PM Using XRF					
Key applicable elements	Sb, As, Ba, Cd, Ca Cr, Co, Cu, Fe, Pb, Hg, Mn,					
	Ni, Se, Ag, Sn, Ti, Tl, V, Zn, and more available					
Measurement range	Up to 60 μg/dscm and higher					
Measurement resolution	15, 30, 60, 120, 180, or 240 minutes					
Calibration stability check frequency	Automatically with each sample analyzed					
Estimated recalibration	Annually, when CES' operating					
Sample flow rate	16.7 liters/minute					
Linearity	Correlation coefficient >0.99					
Size	19" w x 20" d x 30" h (48 x 51 x 76 cm)					
Weight	130 pounds (59 kilograms)					
Setup Options	19 inch (483 mm) rack-mountable or tabletop					
Operating environment	Enclosed with temperature controlled					
	to 20±5°C (68°F)					
Power requirements	120 VAC/60 Hz @ 20 amp or					
	220 VAC/50 Hz 10 amp circuit					
Outputs	RS232 Modbus protocol					
	Reporting of all metals that the system is calibrated to measure					
Options	Change or add elements					
	Enclosures (NEMA 4, 4x, 12, or 12x)					
	Inlets (PM10, PM2.5, PM1, low volume TSP)					

# Appendix 2. Xact 625i Minimum Detection Limits

	Xact 625i Minimum Detection Limits													
	(ng/m³)													
			(	58%Confi	dence Le	vel (C <sub>10</sub> )	pe	er US EPA	103.3 an	d Currie	*			
Element		Sam	oling/Anal	ysis Time	(min)			Element		Sam	oling/Anal	ysis Time	(min)	
(At. No.)	15	30	60	120	180	240		(At. No.)	15	30	60	120	180	240
Al (13)	1500	500	170	61	33	22		Sr (38)	3.1	1.1	0.38	0.13	0.072	0.047
Si (14)	260	89	31	11	5.9	3.8		Y (39)	3.9	1.4	0.48	0.17	0.092	0.060
P (15)	76	26	9.0	3.2	1.7	1.1		Zr (40)	4.7	1.6	0.57	0.20	0.11	0.071
S (16)	46	16	5.5	1.9	1.0	0.68		Nb (41)	5.8	2.0	0.70	0.25	0.13	0.088
Cl (17)	25	8.6	3.0	1.1	0.57	0.37		Pd (46)	31	11	3.8	1.3	0.73	0.48
K (19)	17	5.8	2.0	0.71	0.39	0.25		Ag (47)	27	9.5	3.3	1.2	0.64	0.42
Ca (20)	4.4	1.5	0.52	0.18	0.099	0.064		Cd (48)	36	12	4.4	1.5	0.84	0.54
Ti (22)	2.3	0.79	0.28	0.097	0.053	0.034		In (49)	44	15	5.4	1.9	1.0	0.67
V (23)	1.7	0.60	0.21	0.073	0.040	0.026		Sn (50)	58	20	7.1	2.5	1.4	0.88
Cr (24)	1.7	0.58	0.20	0.071	0.038	0.025		Sb (51)	73	26	9.0	3.2	1.7	1.11
Mn (25)	2.1	0.71	0.25	0.087	0.047	0.031		Te (52)	8.7	3.0	1.0	0.36	0.20	0.13
Fe (26)	2.5	0.85	0.30	0.11	0.057	0.037		I (53)	7.1	2.4	0.85	0.30	0.16	0.11
Co (27)	2.0	0.68	0.24	0.085	0.046	0.030		Cs (55)	5.4	1.9	0.65	0.23	0.12	0.080
Ni (28)	1.4	0.47	0.17	0.059	0.032	0.021		Ba (56)	5.7	1.9	0.67	0.24	0.13	0.083
Cu (29)	1.1	0.39	0.14	0.048	0.026	0.017		La (57)	5.3	1.8	0.63	0.22	0.12	0.078
Zn (30)	0.95	0.33	0.12	0.041	0.022	0.014		Ce (58)	4.4	1.5	0.52	0.18	0.10	0.064
Ga (31)	0.83	0.29	0.10	0.036	0.019	0.013		Pt (78)	1.7	0.58	0.20	0.072	0.039	0.025
Ge (32)	0.79	0.28	0.097	0.034	0.019	0.012		Au (79)	1.4	0.50	0.18	0.062	0.034	0.022
As (33)	0.90	0.31	0.11	0.039	0.021	0.014		Hg (80)	1.7	0.60	0.21	0.074	0.040	0.026
Se (34)	1.2	0.40	0.14	0.050	0.027	0.018		TI (81)	1.7	0.57	0.20	0.071	0.039	0.025
Br (35)	1.5	0.52	0.18	0.064	0.035	0.023		Pb (82)	1.8	0.63	0.22	0.078	0.042	0.027
Rb (37)	2.7	0.95	0.33	0.12	0.064	0.042		Bi (83)	1.8	0.64	0.23	0.080	0.043	0.028

$$\sigma_o = \frac{\sqrt{N_b}}{St} = \frac{\sqrt{R \cdot t \cdot I}}{S \cdot t \cdot I}$$
  
R = Background Count Rate

I = Current t = Live Time S = Sensitivity

$$MDL_{\sigma_o} = \frac{\sigma_o}{V}$$

 $\sigma_o=68\%$  Confidence Level Detection Limit

V = Sample Period Volume

# Appendix 3. Xact 625i Leak & Flow Check Form

## LEAK CHECK

Initial Sample Pressure (mmHg)	
Final Sample Pressure (mmHg)	
Elapsed Time (sec)	
Leak Rate (mmHg/min)	

## **FLOW CHECK**

Target Flow	Refere	ence	Xact Flow	% Difference
15.0				
16.7				
18.4				
15.0				
16.7				
18.4				
15.0				
16.7				
18.4				

MONITORING SITE: _	 TECHNICIAN:
SIGNATURE:	 _ DATE:

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## Appendix 4. Xact 625i XRF Calibration Check Form

Element	Atomic Number	Standard Compound	Standard ID	Energy Condition	Acq. Time	Mass (ng)	Reported Mass	% Diff
Ca	20	CaF2		1	63			
Mn	25	Mn		1	63			
Se	34	Se		2	63			
Pb	82	Pb		2	63			
Cd	48	CdSe		3	300			

## **OPTIONAL**

Element	Atomic Number	Standard Compound	Standard ID	Energy Condition	Acq. Time	Mass (ng)	Reported Mass	% Diff

NOTE: Use only elements in Xact 625i Calibration Guide marked as A in Purpose column.

MONITORING SITE:	TECHN	ICIAN:
SIGNATURE:	DATE:	