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# XRF Analyser

Instruction manual

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## 1 About this manual

Important information is listed under the following symbols:



Caution



Note

## 2 Introduction

This manual covers the use and operation of the Parker Kittiwake XRF (X-Ray Fluorescence) Analyser. The instrument is rugged, compact, stable and easy to operate. Besides being able to accurately and rapidly measure the concentration of sulphur in marine fuels, it can determine the concentration of wear metals in oil.

Sulphur in fuel will continue to be a hugely important issue in the marine shipping industry for many years to come. Current regulations at time of writing, set out in Marpol Annex VI, prevent the use of fuel with a sulphur concentration above 3.5% in much of the World's international waters (unless the vessel in question is fitted with a scrubber). From 1 January 2020 this limit will be reduced to 0.5%. The same regulations will continue to ensure that only fuel with a sulphur concentration of less than 0.1% can be burnt in designated Emission Control Areas (ECAs), such as the Baltic Sea.

The most cost effective way of meeting the 0.5% sulphur limit will be to blend the minimum amount of expensive, low sulphur, distillate with the maximum amount of cheap, high sulphur, residual fuel oil. These fuels will surf a very fine line between economics and legislative compliance. Going forwards, ships will need to be armed with the best technology to ensure that they only buy and burn compliant fuel; anything else is likely to lead to significant fines and a loss of reputation.

Condition monitoring of machinery lubricants is an established method of predicting and avoiding impending machinery breakdown. Component wear and oil contamination can be identified early and rectified before any serious damage occurs. Production can be maintained, machinery life extended and the return on capital investment increased.

### 3 Product description



## 4 XRF safety

While the XRF Analyser is designed to be usable without any special training or qualification, appropriate safety precautions must still be taken.

If the XRF Analyser is used in any manner or for any purpose, other than that described in this manual, safety protection may be impaired.



Warning! This unit is not approved for use in an explosive atmosphere.



Warning! This unit produces ionising radiation and must be operated in accordance with local regulations (see Sections 4.1 and 4.2 for details).

### 4.1 Radiation safety on land

Employers who expect workers to operate the XRF Analyser must determine and apply the ionising radiation regulations that apply in their country of operation. For example, UK employers must ensure compliance with IRR17. One of the requirements of IRR17 is that employers appoint a Radiation Protection Supervisor to provide advice on safe operation of the unit. Local regulations will also likely stipulate requirements for maintenance programs to be set up by the employer and may include regular servicing by qualified persons.

### 4.2 Radiation safety at sea

Ships are subject to the maritime laws of their flag state. The flag state has the authority and responsibility to enforce regulations over its vessels. As an example, a UK-registered ship will need to obey the radiation safety rules set out by the (UK) Maritime and Coastguard Agency. MGN451 covers the risk of radiation exposure on board ships. This document specifically says that “the Ionising Radiations Regulations 17, do not generally apply to ships when they are outside Great Britain. MGN 197 (M+F) sets out the relevant legislation for ships.” MGN197 lists five different pieces of legislation that, once considered, should ensure maritime “compliance as applied to ship owners/operators” with the EU directive 96/29 Euratom. Of these five pieces of legislation, it is The Merchant Shipping and Fishing Vessels (Health and Safety at Work) Regulations 1997 SI 1997 No 2962 that has particular relevance to the Parker Kittiwake XRF Analyser. Given that ships must already comply with these regulations, the extra work involved in incorporating radiation safety measures is fairly minimal.

Ships also fall under port state control. Port state control (PSC) is an internationally agreed regime for the inspection of foreign ships in ports other than those of the flag state. PSC officers are required to investigate compliance with the requirements of international conventions written by bodies such as the IMO and ILO. Both of these bodies are sponsors of the IAEA radiation safety document entitled “Safety Fundamentals SF-1”. However, the Parker Kittiwake XRF Analyser is exempted from specific regulation by the IAEA because, under normal operating conditions, it does not cause an ambient dose rate of  $> 1 \mu\text{Sv}$  per hour at a distance of 0.1 m from the unit.

### 4.3 Health and safety

The design of the XRF Analyser is consistent with the principles outlined in ISO 12100:2010 (Safety of machinery – General principles for design - Risk assessment and risk reduction). A risk assessment of the final design has been conducted to highlight actions that should be taken to minimise operator risk.

#### Actions

- Set up a controlled-access area around the instrument.
- Only allow trained/authorised people to operate the instrument.
- Carry out a visual inspection of the analyser prior to each use.
- Use a calibrated radiation monitor to perform annual radiation leak testing.
- Equip operators with a dosimeter and record cumulative measurements.
- Review dosage records at regular intervals.
- Inspect the safety functions (especially the lid interlock, lid release button and lights) of the analyser prior to use.
- Prepare local rules that require the unit to be returned in the event of suspected damage.
- Maintain the analyser in line with manufacturer guidance.
- Perform regular electrical testing of the power supply.
- Protect external cable from damage in use.
- Do not run the analyser without a sample in place.
- Use appropriate PPE when handling hazardous samples.
- Lift the unit only by the integrated handle when necessary.
- Local rules should state that operators must inform management if they become pregnant.
- For absolute safety, redeploy any operator who reports a pregnancy.
- Keep the XRF in cool, dry conditions.
- When not in use, the XRF should be stored in a secure location, under lock and key, to prevent unauthorised access and use. As well as being good practice, this may be a requirement of local regulations covering the use of ionising radiation. See sections 4.1 and 4.2 for more details.

## 5 Chemical compatibility

The XRF Analyser is only intended to measure liquid hydrocarbons, such as lubricating oils and fuel samples.

Any liquids that are chemically incompatible with polyester may breach the Mylar film used to retain samples and destroy the X-ray detector.

## 6 Technical specification

Rated Input Voltage	9 V D.C.
Operating temperature	15 to 30 °C
Operating humidity	≤ 70 %
Instrument weight	4.5 kg
Test time	130 seconds (typical)
Unit dimensions	212 mm (w) × 258 mm (h) × 258 mm (d)
X-ray tube	40 kV, 100 µA
Detector type	Silicon drift
Measurable elements	Sulphur, calcium, titanium, vanadium, chromium, manganese, iron, nickel, copper, zinc, molybdenum, silver, cadmium, lead

### 6.1 Sulphur measurement precision

The Parker Kittiwake XRF Analyser is factory calibrated according to ISO 8754 and makes field sulphur concentration measurements that correlate strongly with ISO 8754 laboratory measurements. The repeatability of the instrument, established at three different sulphur concentrations using accepted laboratory and statistical techniques, is actually better than that provided in ISO 8754. Given this information, the reproducibility of the unit should also be no worse than that stated in ISO 8754.

Sulphur conc. / %	<sup>1,2</sup> Repeatability / %	<sup>1,3</sup> Reproducibility / %
0.500	± 0.010	± 0.037
1.000	± 0.017	± 0.065
3.000	± 0.045	± 0.179

1 19 out of 20 repeated measurements fall within the stated range for each sulphur concentration.

2 Measured instrument repeatability.

3 Inferred instrument reproducibility.

## 6.2 Wear metal measurement precision

The repeatability of wear metal concentration measurements has been established using accepted laboratory and statistical techniques. Instrument repeatability has been determined for a subset of measurable elements at a variety of concentrations.

Metal	Conc. / ppm	Repeatability / ppm
Calcium	11,249	± 150
Vanadium	49	± 4
Chromium	6	± 2
Iron	6	± 2
Nickel	17	± 1
Copper	2	± 1
Zinc	204	± 4

## 7 Manufacturer information

Please contact the manufacturer for service, consumables, spare parts and repairs.

## 8 Spares/consumables

Part no.	Item	Contents
ACCK04001	Consumables pack	Syringes (100) Sample cups (100) Copper targets (5) Reel of Mylar (30 m) Labels for printer (10 m)
ACCK04002	Check standards	Pack of 3 standards



## 9 Instrument set-up



Warning! It is critically important that the XRF Analyser is connected to the local mains power supply using the power adaptor and cable provided. Damage to internal circuitry caused by accidental use of the incorrect power supply is not covered by the warranty.

For best results, the XRF Analyser should be operated:

- On a flat, level surface such as a workbench or a desk.
- In temperature-stable environments.
- Away from drafts and heat sources such as air conditioning or heaters.
- Away from strong magnetic fields, such as large electronic motors or generators, power transformers, loudspeakers or microwave ovens (these can even affect the instrument through walls).



Before each use of the XRF instrument, a series of safety checks should be carried out to ensure safety features are functional. This will likely be a requirement of local regulations – see section 4. At a minimum, these checks should include:

- Visual checks for damage to the XRF exterior
- Visual check for cracks of the glass lid.
- Check the lid interlock - the lid must only open by pressing the button.

## 10 Sample preparation

### 10.1 Sample handling

*Please read this section to familiarise yourself with sample preparation before using the XRF Analyser for the first time.*

In order to ensure a reliable measurement, the following steps should be taken to ensure that the measured sample is representative of the main body of fluid:

- Any containers used to store or collect fluid samples should be clean and free of contamination before use.
- The sample cups themselves should be kept in a clean dry environment.
- Fluid samples should be mixed thoroughly by shaking or stirring.
- Any air bubbles introduced during homogenisation should be allowed to disperse before dispensing the fluid into an XRF sample cup.

## 10.2 Constructing and filling a sample cup



Liquid samples **MUST** be put into a prepared sample cup before being placed inside the spectrometer.

Begin by setting a sample cup base on a flat surface.



Extract roughly 2 ml of sample fluid using one of the supplied syringes and dispense it into the sample cup base.



Cut off a roughly-square piece of Mylar sheet from the roll provided and lay it over the sample cup base.



The substitution of Parker-Kittiwake-supplied Mylar film with any other product (including other types of Mylar) will cause measurement inaccuracies.



Identify the flanged side of a sample cup ring.



Flanged edge of the sample cup ring

Press the flanged side of the sample cup ring down over the Mylar and snap it onto the sample cup base.



At this stage the sample cup can be safely inverted in preparation for insertion into the XRF Analyser.



## 11 General Maintenance and cleaning

Wipe the instrument body with a dry cloth to remove most oily marks. The removal of particularly stubborn stains can be assisted by the use of a mild hydrocarbon solvent such as isopropyl alcohol. Other solvents or cleaning products may damage the instrument.



Replace Mylar film covering detector aperture every month and also whenever it appears to be dirty or compromised in any way.



Replace Mylar film carefully, since procedure exposes the fragile detector. Physical damage to the detector is not covered by the warranty.

Depress the oval silver button on the front of the unit to open the lid. Undo the screw nearest the front of the XRF Analyser within the sample compartment. Lift the screw completely out of the compartment and store safely.



Lift front end of retaining plate using a small screwdriver and hinge backwards.



Carefully remove the existing Mylar sheet from over the detector window.



Cut a square of Mylar film from the roll provided and rest it gently over the aperture.



Lower the hinged metal flap back into its original position, insert the screw back into the vacant hole and tighten carefully. Examine the Mylar film to ensure that it is free of wrinkles and tears.



The XRF Analyser does not have any user serviceable parts and any attempt to dismantle the unit will invalidate the warranty.

Local regulation may require annual safety checks by a suitably qualified organisation, such as a Radiation Protection Officer.

For all servicing and repairs, please contact the manufacturer (see section 7). Please retain all original instrument packaging for return shipping. The manufacturer will not be liable for damage to returned goods resulting from inadequate packaging.



## 12 Method of operation

### 12.1 Switching the unit on

Prior to switching the unit on, depress the oval silver button to open its lid. Place a copper target centrally over the detector aperture and reclose the lid.



Turn on the power switch and wait roughly 1 min for the HOME screen to appear.

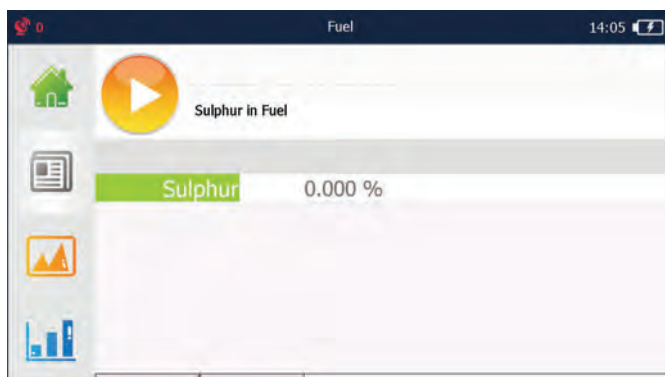


## 12.2 Making a measurement

Depress the oval silver button to open the lid and replace the copper target with the sample of interest (prepared according to the instructions in section 10.2). Place the sample centrally over the detector before closing the lid.



Press the FUEL button on the HOME screen if you wish to determine the sulphur content in a fuel or the LUBE button if you wish to determine the wear metal content in a lubricant. The unit will present a START screen.



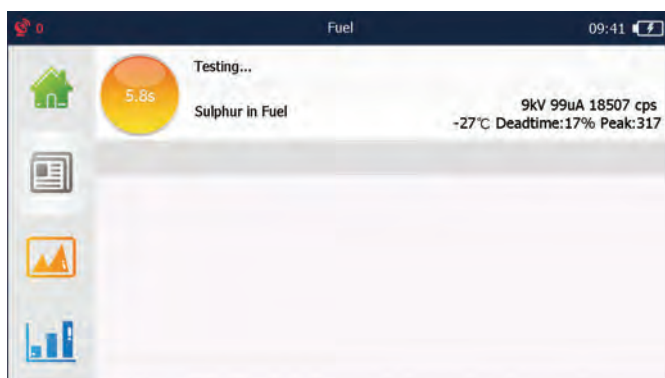
Press the PLAY button on START screen to reveal SAMPLE ID screen.

Operator	Operator A
Sample Point	Position A
Vessel / Plant Name	Vessel 1
IMO	123456789
Fuel / Fluid ID	test30000R6
Location	#120
Engine Hours	100000
<div>OK</div> <div>Return</div>	

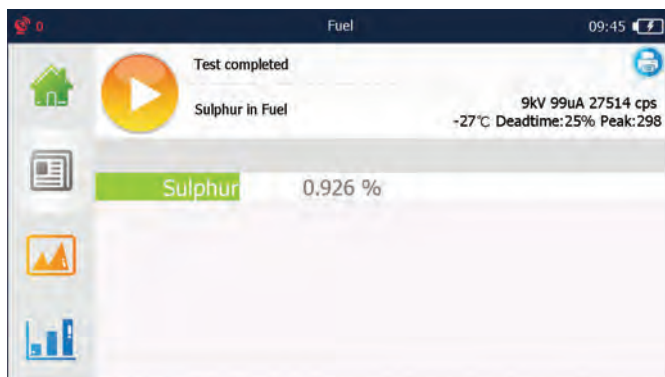
Press any of the fields on the right of the sample ID table to reveal a soft keyboard that can be used to change their contents.

Operator	Operator A
Sample Point	Main engine
Vessel / Plant Name	Vessel 1
IMO	123456789

Press the ENTER key on the keyboard to complete any changes and then press OK to begin the test. The screen will show the time elapsed since starting the measurement during the following 130 seconds.



An alert will sound at the end of the test and the results will be presented.

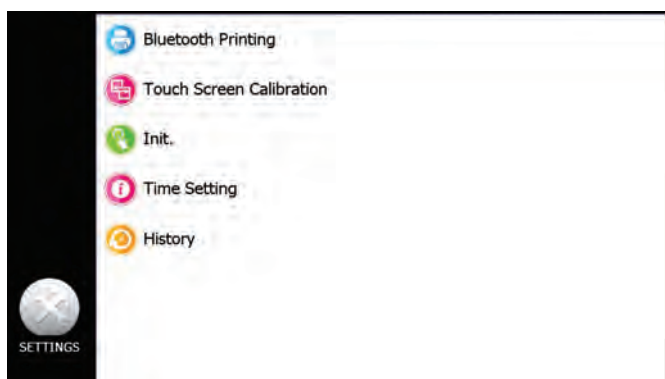


### 12.3 User settings

Starting from the HOME page, press the SETTINGS button to reveal the SETTINGS screen.



Press the USER SETTINGS button to show a USER SETTINGS screen.

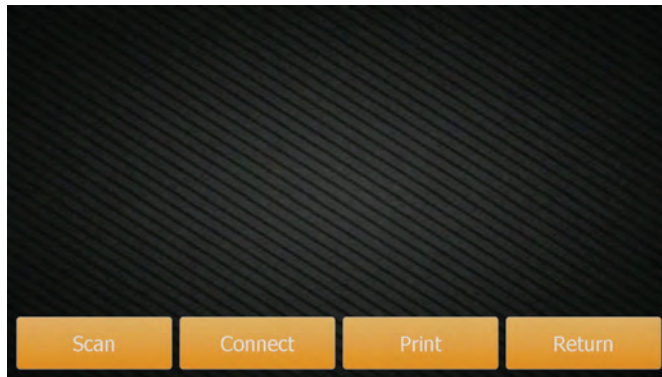


## 12.4 Bluetooth printing

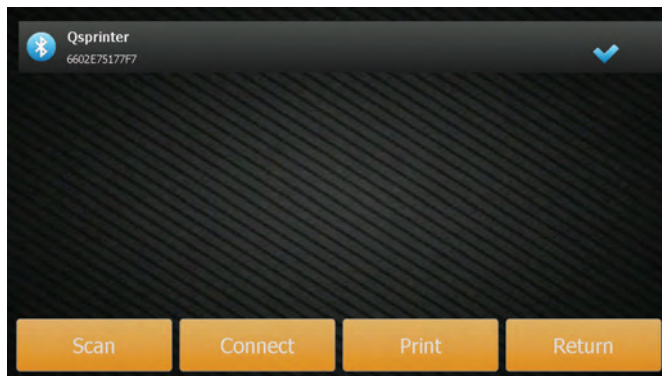
Plug the XRF printer into a local power supply and hold down the power button for a second or two until it lights up green.



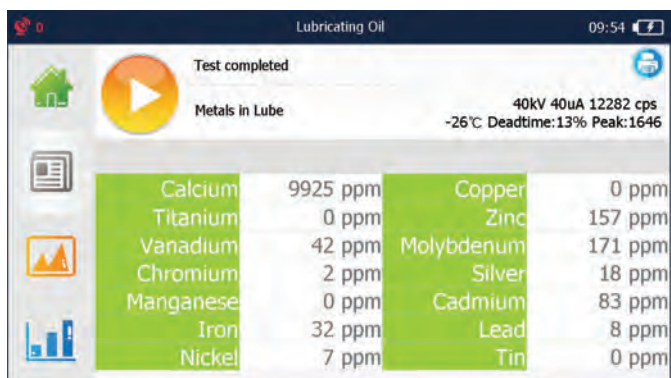
Starting from the USER SETTINGS screen on the XRF Analyser, press BLUETOOTH PRINTING.



Press the SCAN button and wait until "Qsprinter" appears as a local Bluetooth device. Press "Qsprinter" to highlight the device and then press CONNECT. Once the XRF unit and printer are connected, a blue tick will appear next to Qsprinter.



Press PRINT to print out the results of the most recent test or press RETURN to go back to the USER SETTINGS page. Once the printer and XRF Analyser have been connected by Bluetooth, it is possible to print out results by pressing the blue icon that appears on the RESULTS page of every subsequent test.



## 12.5 Touchscreen calibration

Under normal circumstances there is no need to adjust the screen calibration. However, if finger presses do not correspond with activation of the underlying button, recalibration may be worthwhile.

From USER SETTINGS page, press TOUCH SCREEN CALIBRATION.



Carefully press and briefly hold finger on the centre of the target (cross). Repeat this process until the target disappears; press the screen once again to return to the USER SETTINGS page.

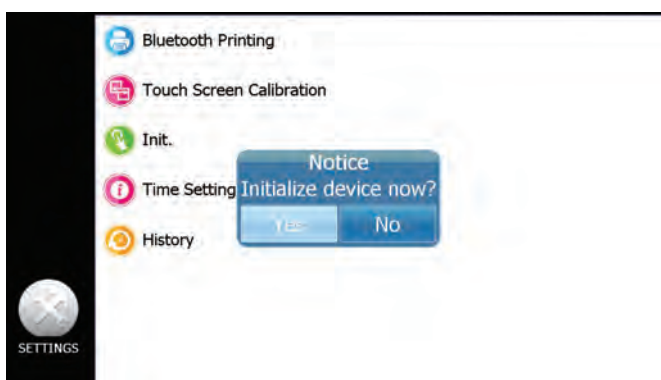


## 12.6 Initialisation (Init.)

Under normal circumstances the XRF Analyser is initialised during start up (see section 11.1). However, the following procedure may be used to re-initialise a running unit as an alternative to switching it on and off.

Depress the oval silver button on the XRF Analyser to open its lid. Place a copper target centrally over the detector aperture and reclose the lid.

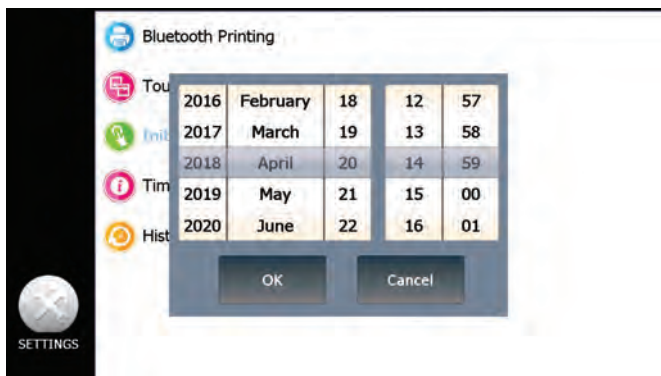
From the USER SETTINGS screen, press the Init. button. A dialogue box will open to confirm that you really wish to initialise the device (press YES).



A bell will sound after a few seconds to confirm successful re-initialisation.

## 12.7 Time setting

From the USER SETTINGS screen, press the TIME SETTING option to reveal a calendar/clock.



Use upward or downward finger strokes to highlight the desired year, month, day, hour and minutes and press OK to return to the USER SETTINGS screen.

## 12.8 History

From the USER settings screen, press the HISTORY option to reveal the HISTORY screen.

	SpecName	Date	S (%)	Ca (PPM)	Ti (PPM)	V (PPM)
<input type="checkbox"/>	test30000R7	04-20 15:08		9750	4709	1
<input type="checkbox"/>	test30000R7	04-20 15:03	0.033			
<input type="checkbox"/>	test30000R7	04-20 15:03	0.028			
<input type="checkbox"/>	test30000R7	04-20 15:02	0.031			
<input type="checkbox"/>	test30000R7	04-20 15:01	0.032			

Export
Delete
Clear
Return

The HISTORY screen lists out all of the test result files stored by the instrument. If you wish to export the contents of all files as a .CSV spreadsheet, simply press EXPORT and then OK on the displayed confirmation dialogue box.

	SpecName	Date	S (%)	Ca (PPM)
<input type="checkbox"/>	test30000R7	2018-04-27 16:43		152
<input type="checkbox"/>	test30000R7	2018-04-27 16:41		85
<input type="checkbox"/>	test30000R7	2018-04-27 16:38	2.466	
<input type="checkbox"/>	test30000R7		0.994	
<input type="checkbox"/>	test30000R7		1.939	
<input type="checkbox"/>	test30000R7			56
<input type="checkbox"/>	test30000R7	2018-04-27 12:00	1.917	
<input type="checkbox"/>	test30000R7	2018-04-27 11:04	1.91	

Notice  
Are you sure to export?  
OK Cancel

Export Delete Clear Return

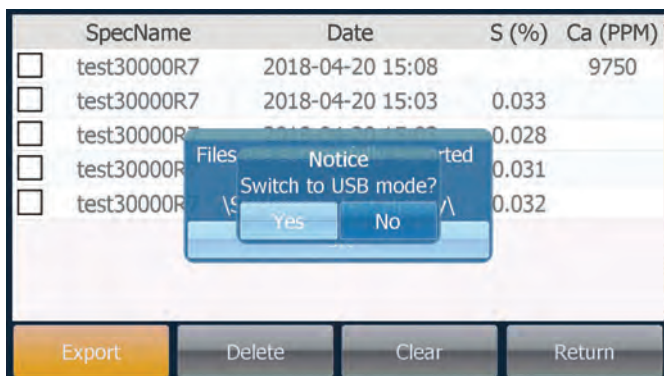
After a few seconds the instrument will report that the files have been successfully exported.

	SpecName	Date	S (%)	Ca (PPM)	Ti (PPM)	V (PPM)
<input type="checkbox"/>	test30000R7	04-20 15:08		9750	4709	1
<input type="checkbox"/>	test30000R7	04-20 15:03	0.033			
<input type="checkbox"/>	test30000R7	04-20 15:03	0.033			
<input type="checkbox"/>	test30000R7	04-20 15:03	0.033			
<input type="checkbox"/>	test30000R7	04-20 15:03	0.033			

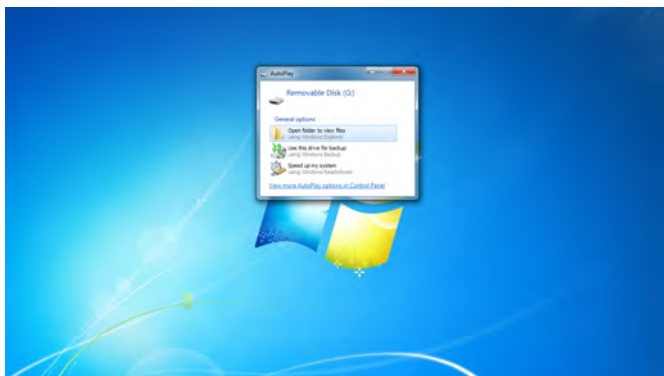
Files are successfully exported  
to  
\Storage Card\Test History\  
OK

Export Delete Clear Return

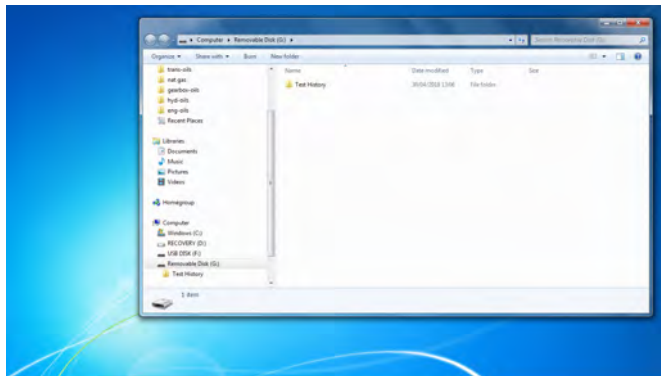
Once OK has been pressed, the unit will ask for confirmation that you wish to switch to USB mode (press YES).



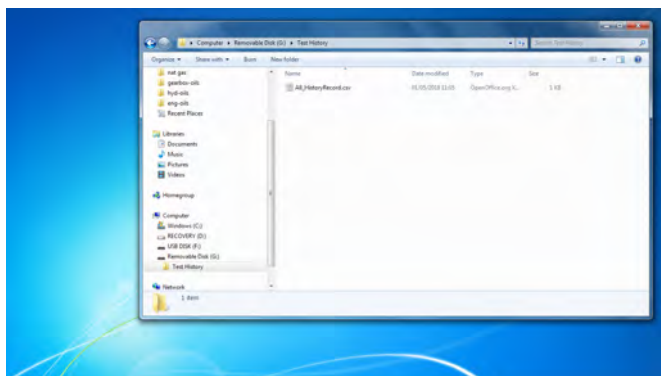
Connect the XRF Analyser to a laptop or PC using the USB port on the rear of the instrument (see Section 3). Windows will recognise the XRF Analyser as a USB mass storage device.



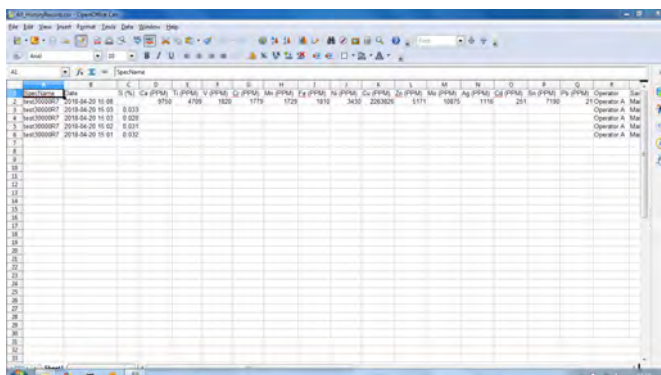
Opening the folder to view files option will reveal the contents of the device.



Open the Test history folder to reveal a file entitled All\_HistoryRecord.csv.



Double click on the All\_HistoryRecord.csv file to open the spreadsheet.



Save the spreadsheet to your laptop and then disconnect the USB lead between the XRF analyser and the computer. Now press RETURN on the XRF Analyser screen to continue working with the unit.

The contents of particular files can be exported by pre-selecting the corresponding tick boxes on the left of the HISTORY screen.

	SpecName	Date	S (%)	Ca (PPM)	Ti (PPM)	V (PPM)
<input type="checkbox"/>	test30000R7	04-20 15:08		9750	4709	1
<input checked="" type="checkbox"/>	test30000R7	04-20 15:03	0.033			
<input checked="" type="checkbox"/>	test30000R7	04-20 15:03	0.028			
<input type="checkbox"/>	test30000R7	04-20 15:02	0.031			
<input type="checkbox"/>	test30000R7	04-20 15:01	0.032			

Export

Delete

Clear

Return

Selected files can be deleted from the HISTORY screen list by ticking them and pressing delete. Press OK when the unit asks for confirmation. Similarly, all of the listed files can be removed from the HISTORY screen by pressing CLEAR.

It is possible to return to the USER SETTINGS from the HISTORY screen at any point by pressing RETURN.

## 12.9 Expert settings

The SETTINGS screen has the option to select EXPERT SETTINGS (besides the USER SETTINGS displayed in section 11.3). However, access to the EXPERT SETTINGS menu screen is restricted by password and intended for factory diagnostic use only.

## 13 Interpretation of results

If an XRF Analyser measurement is significantly different than expected, it is recommended that the sample is retested. The following steps may help:

- Replace the Mylar sheet that covers the detector aperture.
- Ensure sample is thoroughly mixed before testing.

The XRF Analyser is designed to operate between 15°C and 30°C as stated in the Technical Specification table. Operation of the XRF Analyser at the limits of, or outside of this range, may affect the measurement and uncertainty of tests.

It is very important to remember that scientific instruments are unable to report exactly the same values for consecutive measurements of the same sample. The expected variation in repeated measurements of the same sample made by the same operator on the same instrument in the same environment is known as the repeatability. For example, repeated measurements of a 1.000 % sulphur standard using the Parker Kittiwake XRF Analyser typically fall within the range 0.983 – 1.017 % nineteen times out of twenty. Of course this means that some measurements will fall outside this range.

Measurements of identical samples made using different instruments, different operators, and different environments show a variability characterised by reproducibility. For example, measurements of the same 1.000 % sulphur standard made using different XRF analysers on different ships and labs by different operators throughout the world would be expected to fall within the range 0.935 - 1.065 % nineteen times out of twenty. From this perspective it is easy to see how a sample sent to a laboratory could test at, for example, 0.95 %, while yielding the result of 1.05 % when measured on a ship.

Please note that the repeatability and reproducibility of sulphur measurements made using the Parker Kittiwake XRF Analyser are at least as good as those provided in ISO8754. A degree of result uncertainty is unavoidable.

Although the XRF Analyser reports wear metal concentrations, there is no advice on suitable pass/fail limits because they tend to be very engine-specific.

## 14 Recalibration

The XRF Analyser has been designed for long term accuracy of measurements. It is recommended that sulphur check standards are purchased and benefits from periodic recalibration to ensure the most accurate results.



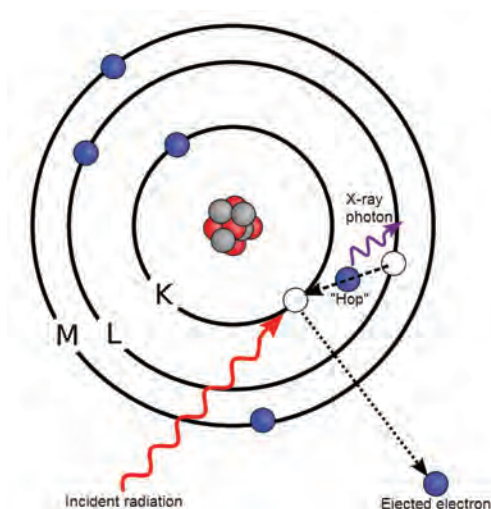
Contact the manufacturer for details on service and recalibration (see page 32).

## 15 Technical explanation

An X-ray fluorescence (XRF) spectrometer irradiates samples with high energy electromagnetic radiation and then detects the X-rays they fluoresce as a result. The wavelengths and intensities of this fluorescence are characteristic of the chemical elements present and their concentration within the sample, respectively.

On a more detailed level, XRF spectrometers generate a range of electromagnetic wavelengths that carry sufficient energy to expel electrons from constituent atoms within a sample. Atoms that lose an electron from an orbital close to their nucleus are particularly unstable and spontaneously rearrange their electronic structure as a consequence. Electrons that occupy orbitals further from the nucleus tend to hop into the recently vacated hole left by the departed electron in the inner orbital. This electronic transition is accompanied by the release of an X-ray photon with a wavelength characteristic of the separation between the two orbitals in question. Given that different chemical elements have different orbital separations, the wavelength of the emitted X-rays can be used to diagnose their presence in a sample. Furthermore, the number of X-ray photons detected at a particular wavelength is proportional to the number of atoms that generated them, and this can be used to calculate the concentration of an element within a sample. The key processes involved in X-ray fluorescence are best shown in a diagram.

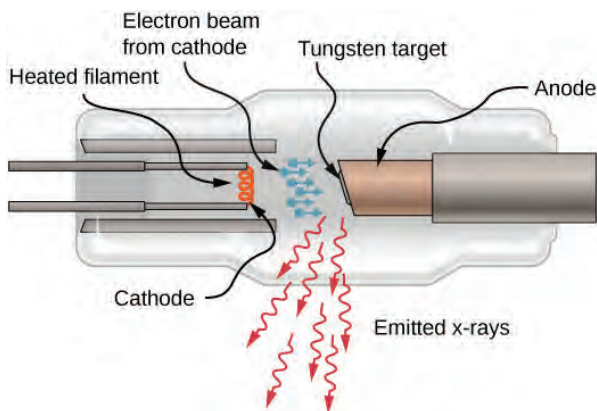




The key processes in X-ray fluorescence

The two most important components of an XRF spectrometer are the vacuum tube, which generates the probing electromagnetic radiation (usually X-rays), and the detector, which responds to the X-ray photons released by the sample as a result of the radiation.

Vacuum tubes (in the form of the cathode ray tube) are familiar to many people as the technology that used to be at the heart of every television. X-ray tubes operate along exactly the same lines. In the most common design, a cathode is electrically heated to stimulate the emission of electrons which are accelerated through an evacuated glass tube towards a metal anode. The rapid deceleration of the electrons when they hit the anode, which is usually made from copper, tungsten, or molybdenum, is accompanied by the emission of X-rays. The energy of the emitted X-ray photons increases with the voltage applied to accelerate the electrons whereas the number of photons increases with the current flow. X-rays usually exit the tube through a beryllium window and are directed towards the sample. The key elements of an x-ray tube are shown in the following diagram.



The key elements of an X-ray tube.

Energy-dispersive XRF spectrometers usually incorporate either a silicon drift (SDD) or silicon pin (Si-PIN) detector. The substantially greater cost of an SDD is justified in higher end units, such as the Parker Kittiwake XRF Analyser, by better resolution and sensitivity.

An SDD is a semiconductor device built around high-resistivity silicon that absorbs X-ray photons fluoresced by the sample under consideration. Electrons, ejected from the silicon as a result of photon absorption, drift through the silicon under the influence of an electric field applied by a series of concentric reverse-biased strips. The electrons are eventually collected by a small anodic ring that sits at the centre of the strips and generate an electrical current that can be measured. X-ray photons create a current pulse at the anode that is proportional to the energy they carry, thus identifying the element responsible for their birth. A relatively large number of pulses with the same height implies a high concentration of that element within the sample. The high purity of the silicon used within SDDs means that low noise levels can be achieved very simply using Peltier cooling.

## 16 EU Declaration of Conformity

Product Name:	XRF Analyser
Part Number	XRF6111

Complies with the essential requirements of the following Directives:

2006/42/EU	Machinery Directive
2014/53/EU	Radio Equipment
2011/65/EU	Restriction of the use of certain hazardous substances in electrical and electronic equipment.

The following harmonised and international standards have been applied:

EN 12100:2010  
EN 61010-1: 2010  
EN 61010-2-061: 2018  
EN 61000-3-2:2014  
EN 61000-3-3:2013  
EN 50581:2012  
EN 301 489 V 1.9.2.  
EN 55011

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Place of issue: Littlehampton, England

Date of issue: 07 December 2018