Lead in Consumer Products

Section 101 XRF Technologies

Matt Kreiner

November 7, 2008

Oxford Instruments plc

Worldwide leader in installed EDXRF systems

- c. 1,300 people worldwide
- c. $350MM turnover (2006/07)
- 12 manufacturing sites in 6 countries
  - UK (3)
  - USA (4)
  - Germany (2)
  - Finland (1)
  - Denmark (1)
  - PR China (1)
- First technology spin-out from Oxford University (1959)

Structure

Oxford Instruments plc

Analytical

Nano Analysis	**EDXRF systems for electron microscopes**
XT	**X-ray tube/Detector manufacture**
Industrial Analysis	**Instruments for material analysis and quality control**
Plasma Technology	**Process solutions to enable the fabrication of nano structures**

Superconductivity

Nano Science	**Cryogenic systems Superconducting magnets**
OST	**Superconducting wire and cables. MRI support**
Austin Scientific	**Cryogenic vacuum pump systems**
Molecular Biotools	**Tools for the development of bioscience applications**

Innovation

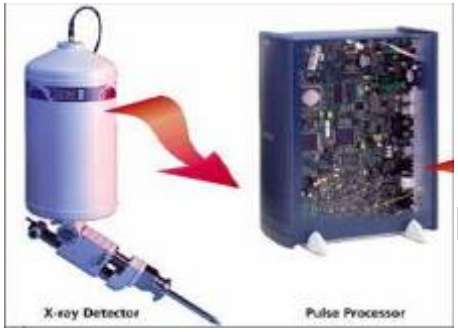
Incubator for new ideas, innovation, M&A

EDXRF Instruments

Micro spot
X-Strata Series

Bulk XRF
Lab-X

Handheld Portable
X-MET 5000

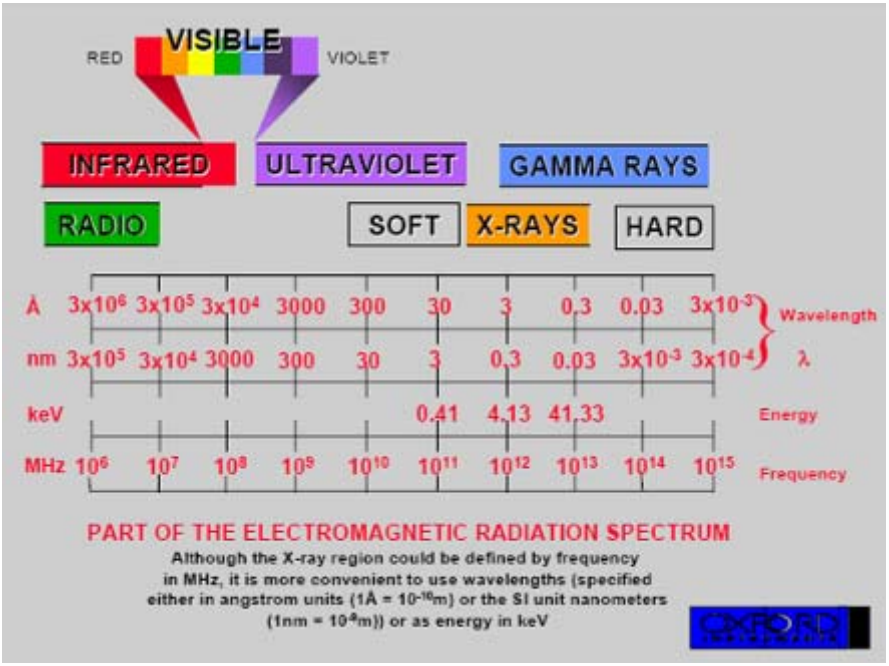


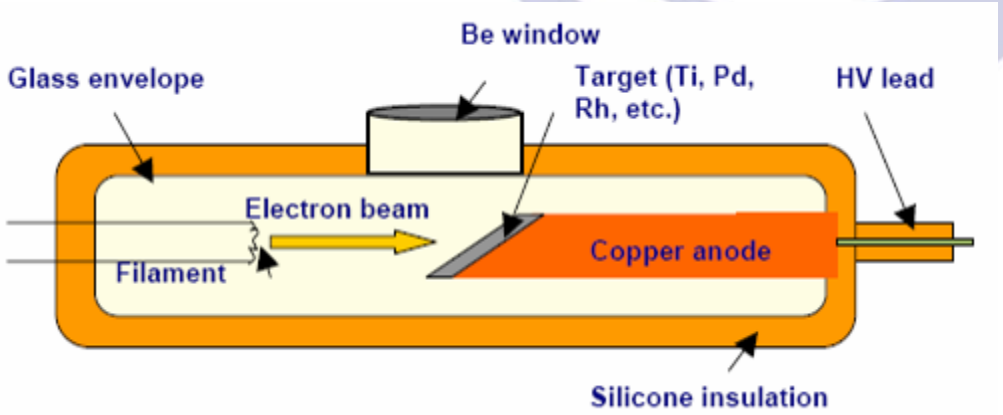
Micro/Nano Analysis
Inca Series

Bulk XRF
ED2000

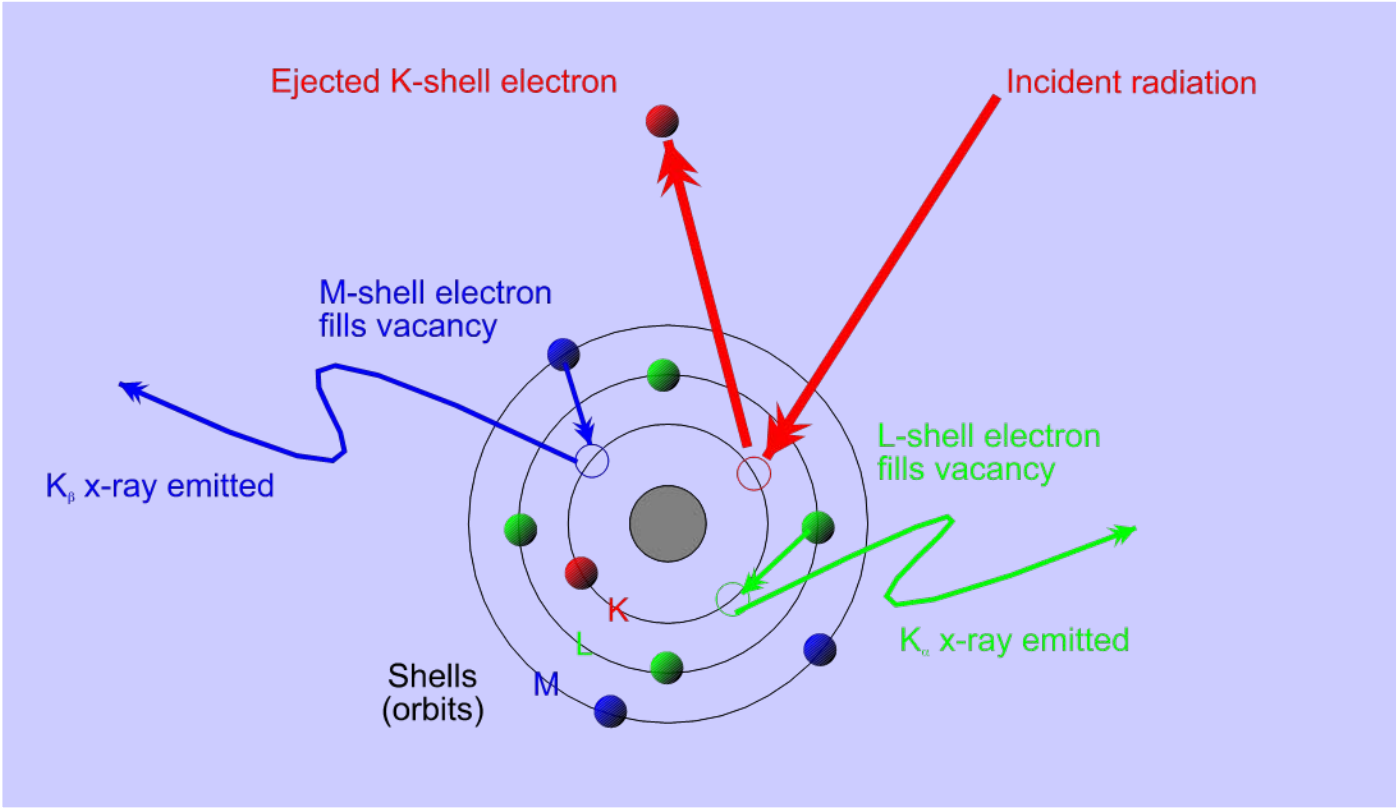
Bulk XRF
Twin-X

Generation of X-rays

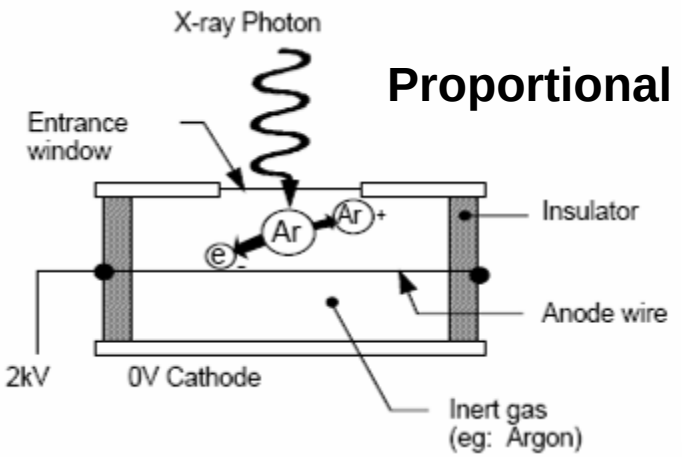




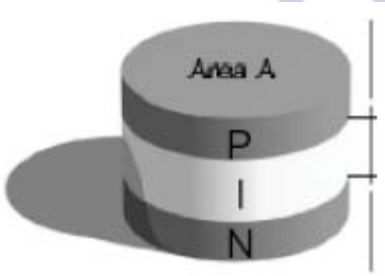
Generation of X-rays

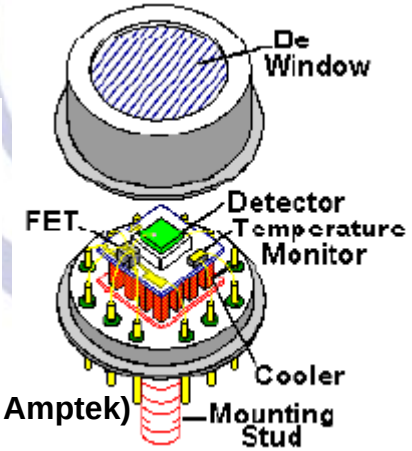


Detection of X-rays

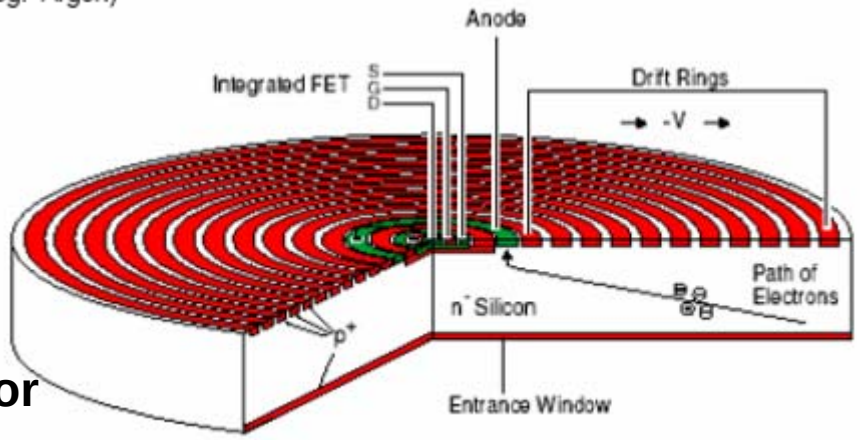


Proportional Counter



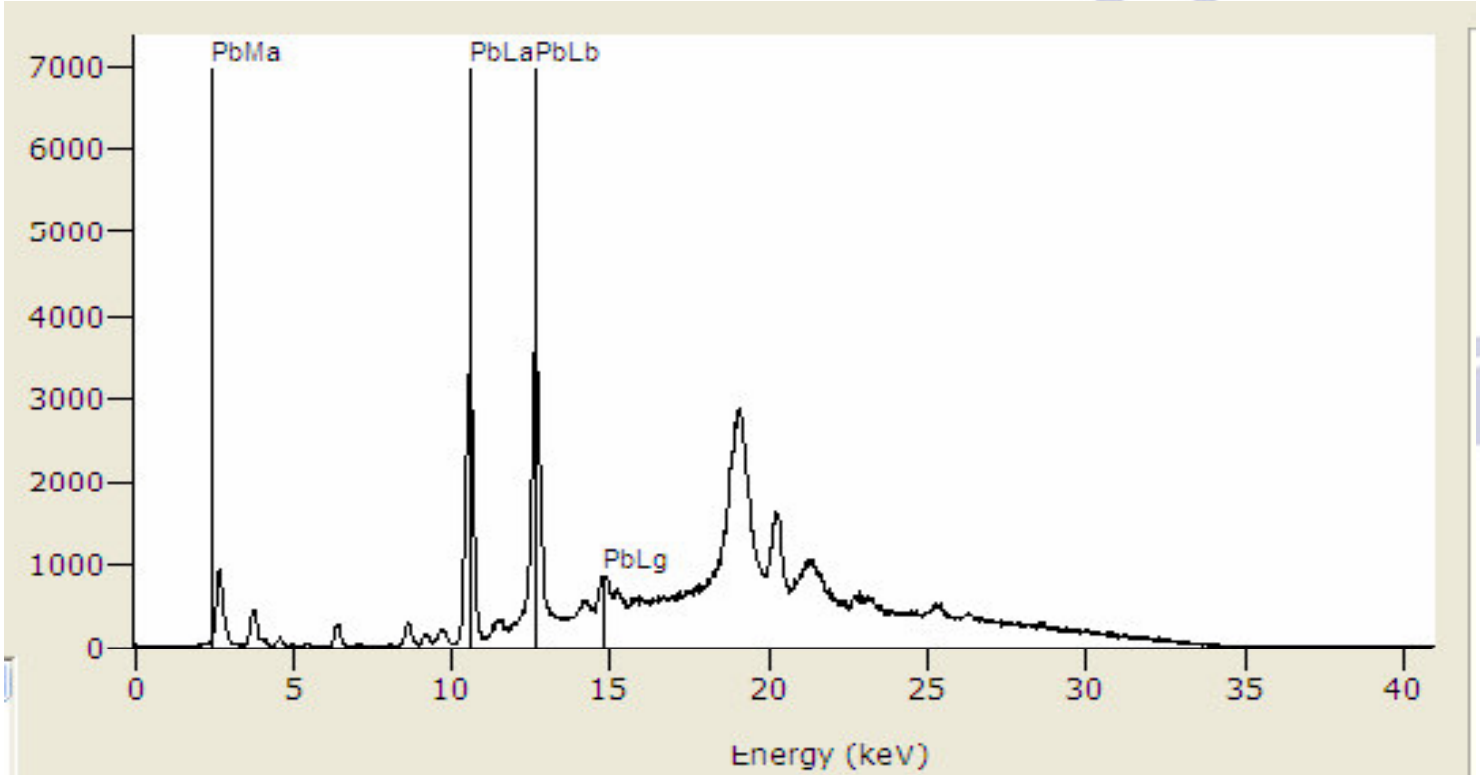


PIN Diode (courtesy of Amptek)



Silicon Drift Detector

Spectra



Information in Spectrum

•Position determines the element

•Peak size determines concentration

Detector Characteristics

	Proportional Counter	PIN Diode	SDD
Background	**Better** →		
Resolution	**Better** →		
Detection Limit	**Better** →		
Price	**Higher** →		

XRF Screening

- Portable
  - Go to your suppliers factory
- Non-destructive
  - No sample preparation required
- Rapid Test
  - 10-100 sec measurement times
- Ease of Use
  - Pass/Fail testing
- Good detection limits

What Screening Results Tell You

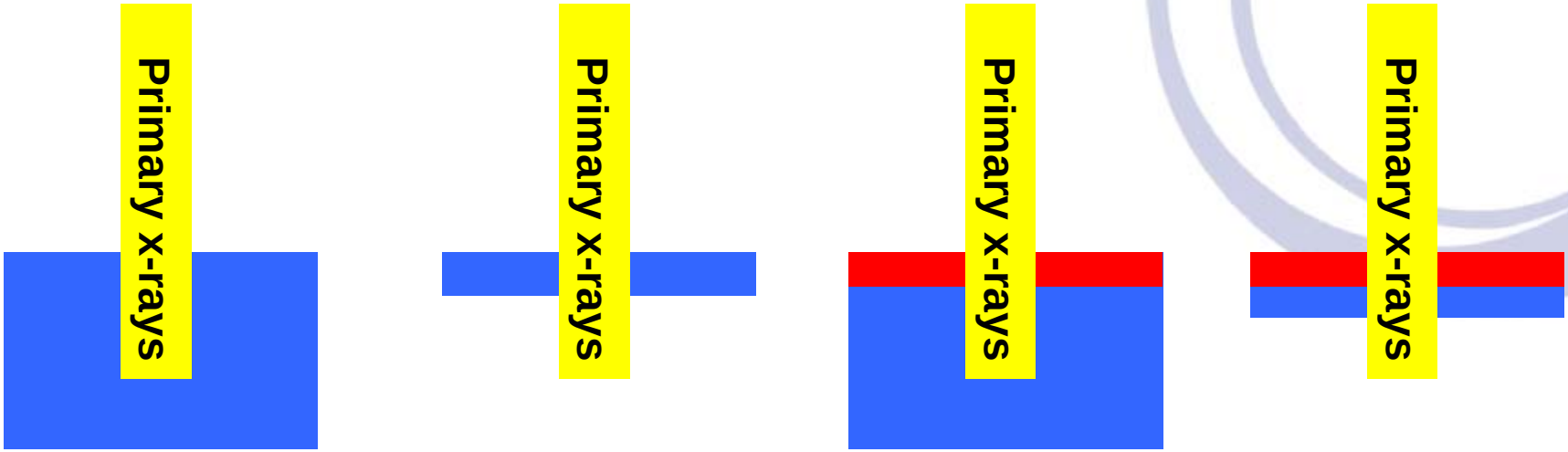
- With any XRF screening device…
- …if you have homogenous, flat, infinitely thick samples that completely cover the excitation area, you can get results that correlate to destructive techniques

Since consumer goods don't look like that...

What Screening Results Tell You

Scenario	Screening Result
No lead in product	No lead detected
Lead only in paint	Lead detected, but result is lower than actual concentration
Lead only in substrate	Lead detected, result is comparable to actual concentration
Lead in paint and substrate	Lead detected, result in between actual concentration in paint or substrate

Measurement Volume



"Infinitely thick"	Thin sample	Painted, thick	Painted, thin
Results OK	Results OK if corrected	Results mix paint and substrate	Results mix paint and substrate, and need correction

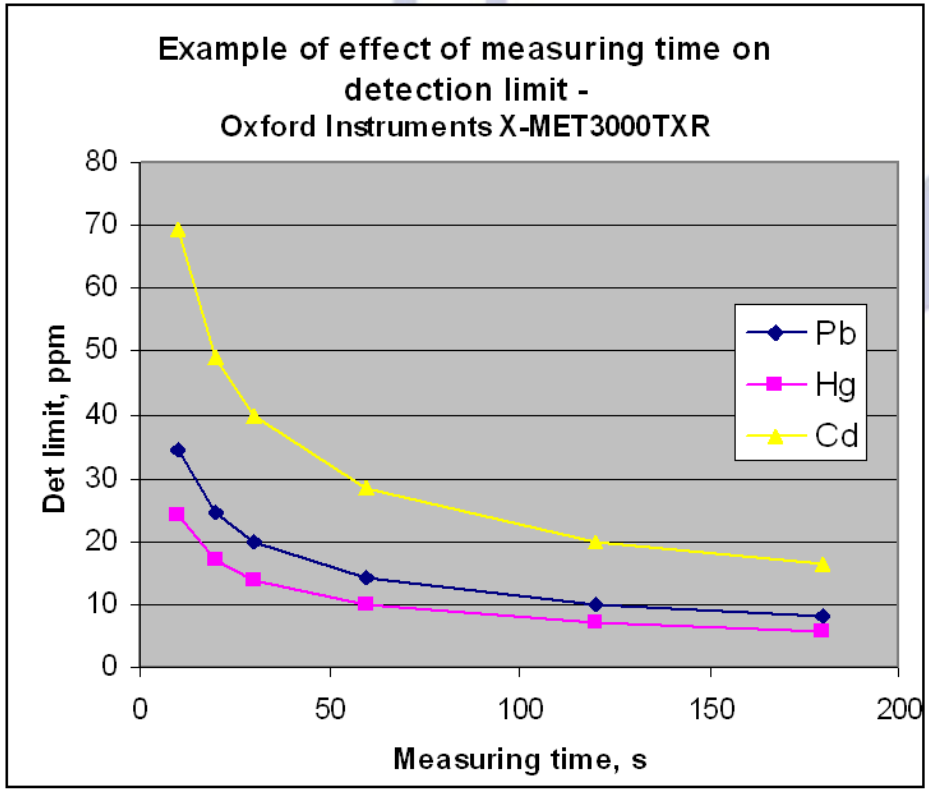
Sample Judgment

Result	Judgment	Action
Pb < 0.002 %	Pb not detect	Part accepted
0.002 % < Pb < 0.009 %	Pb detected	Needs further investigation
Pb > 0.009 %	Pb detected	Part rejected

Results from screening complex materials is qualitative or semi-quantitative. If you make judgments based only on CPSIA limits, you risk accepting bad material.

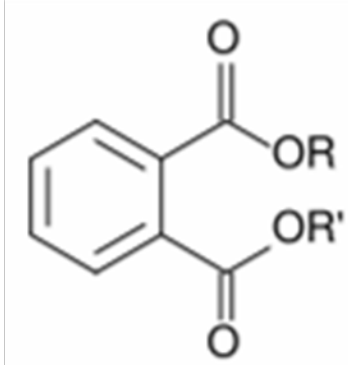
Effect of accumulated counts on detection limit

- Precision of measurement improves as the number of counts increases
- Counts depend on power, spot size and measurement time
- For a given instrument counts increase as measurement time increases
- Choose measurement time based on a balance of throughput and reliability



Regarding Phthalates

- No XRF device can directly measure phthalates
- Phthalates are used as a plasticizer for PVC (vinyl) and other polymers
- Detection of chlorine can be an *indication* that PVC or phthalates may be present



Types of XRF Instrumentation

- Handheld
  - Mainly Screening
  - Large spot; portable; ease of use
  - X-MET5000
- Micro spot
  - Screening and quantitative analysis
  - Allow measurement of small spots on sample
  - X-Strata 980
- Bulk Analysis
  - Quantitative analysis of bulk samples
  - Large spot; ultimate detection limits
  - Twin-X

Handheld Instrumentation

- Advantage
  - Ease of use
  - Low Cost
  - Short measurement time
  - Portability

X-MET® History

2008 X-MET5000 series

2006 X-MET3000+ series
2005 X-MET3000TXR RoHS
2004 X-MET3000TX METAL and SOIL

2003 X-MET 3000TA analyzer

2002 METOREX C-100 SXT sulfur-in-oil process on-line analyzer

2002 Low background EDXRF detector also used in Oxford's Twin X (OEM)

2002 ARC-MET 8000 MobileLab Analyzer

2002 X-MET 3000T analyzer

1999 - 2000 X-MET 1000 & 800 MetalMaster bench-top analyzers

1998 High performance bench-top analyzer with Si-PIN detector

1997 portable X-MET 970 analyzer for coatings

1996 PC-based portable analyzer with semiconductor detector - X-MET 960->X-MET 2000 MetalMaster

1994 on-line sulfur-in-oil analyzer - METOREX C-10SXT

1993 portable ARC-MET 930 Analyzer

Metorex 1994

1992 PC based bench-top X-MET 920 analyzer

1991 portable XRF analyzer for ENVI(Si(Li))

1991 mobile OES analyzer (ARC-MET 900)

1988 on-line EDXRF process analyzer ¨- COURIER 10

1988 portable X-MET 880 analyzer

1984 portable X-MET 840 analyzer

1982 bench-top X-MET 820 lab analyzer

1975 high-resolution X-ray proportional counter

1974 portable X-MET 740 analyzer

1968 On-stream X-ray analyzer - COURIER 300

1968 1970 1980 1990 2000 2006

X-Met 5000 Features

- Penta PIN or SDD gives good detection limits
- Removable PDA allows for easy data viewing and file transfer to PC via USB or Bluetooth
- FP calibration: plastics, alloys
- 7mm diameter spot size
- < 2W x-ray tube power
- Software controlled primary filter changer

Radiation Safety

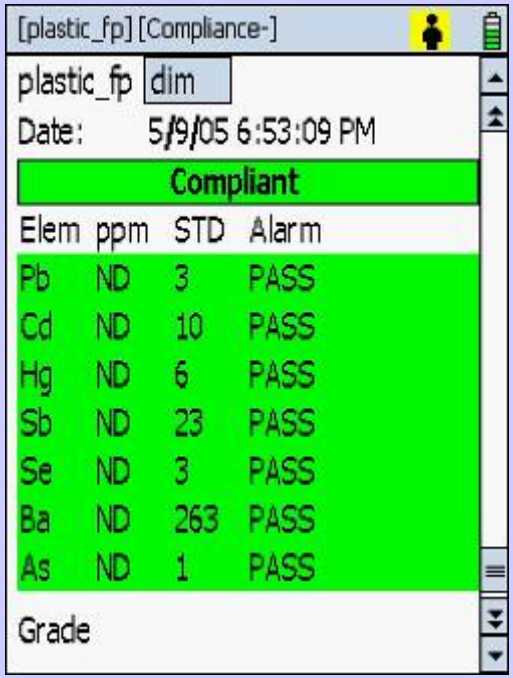
- Handheld – portable x-ray tube
  - No Radioactive Material
  - No Transportation Restrictions
  - Low dose at measurement site
  - No dose behind instrument
  - Safety interlocks

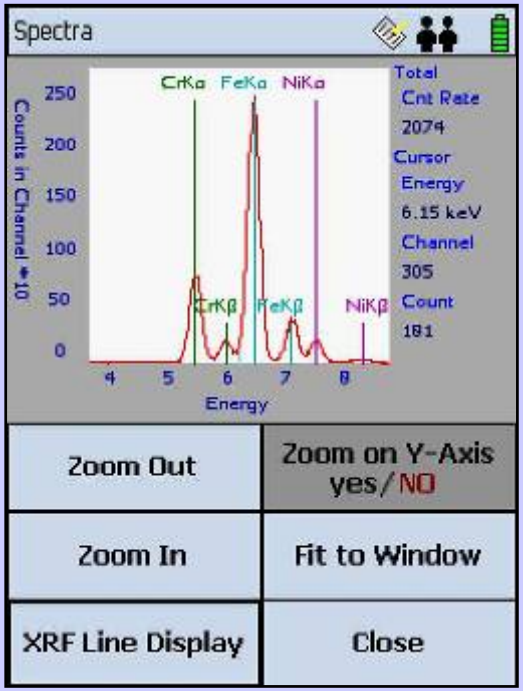
- Bench Top mode
  - Completely interlocked
  - Essentially no radiation outside of chamber

Bench-top Stand

- Optional sample stand for convenient and safe bench-top use
- Simplified and more comfortable operation when measuring many samples
- Easier to position small samples than in hand-held operation
- Enclosed chamber enables closed beam operation
  for increased radiation safety
- Equipped with Safety interlock
- Can accommodate up to
  21 cm x 21 cm samples

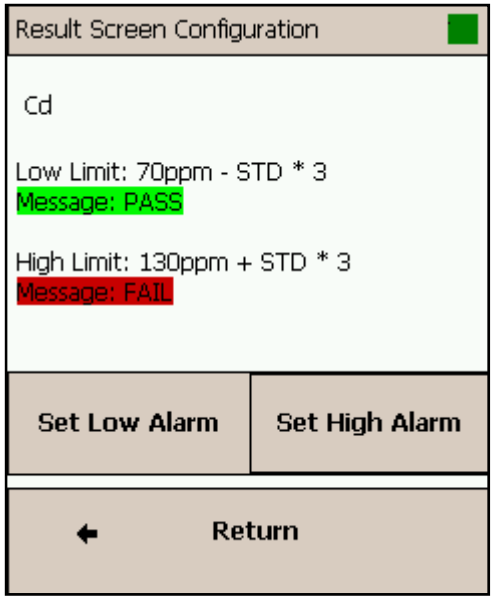
PDA Screens

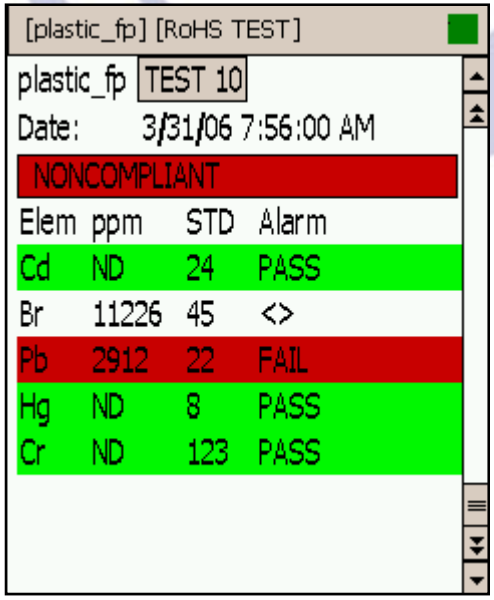




User programmable interface for Compliance testing

- Fully configurable alarm limits
- Report only selected elements or all detected elements
- Build own tests according to adjustable limits
- Save unlimited number of different tests





Easy reporting – Time saving and trouble-free

- Bar-code reader to input the sample name
- Recording of results can be automated if desired
- Measurement data is easily transferred to a PC by via Bluetooth connection for further inspection and report generation
- Over 100,000 spectra and results can be stored for future processing
- Optional PC Report Generator is available for easy and versatile formatting of final QC reports
- Microsoft Excel, Word and Access can be used to store and process the data and to generate custom reports

X-MET5000 Plastic analysis

- FP, universal calibration for all main heavy elements in polymers
  - For all different plastic types: PVC, PE, ABS, PS, Epoxy etc
  - Thickness correction to compensate effect of different sample thickness, min thickness 1mm.
  - Following 24 elements are measured with Plastic FP: Cl, Ca, Br, Ba, Sn, Sb, Ti, Cr, Mn, Fe, Ni, Cu, Zn, As, Pb, Bi, Se, Cd, Hg, Sr, Ag, Au, Ta

X-MET5000 Metal analysis

- FP universal calibration for all alloy types for quality control and lead free screening
- Measures Pb-free and SnPb solders
- Concentration of total 31 elements analyzed:
  - Ag , As, Au, Bi, Br, Cd , Co, Cr, Cu, Fe, Hg, In, Ir, Mn, Mo, Nb, Ni, Pd, Pb, Pt, Sb, Se, Sn, Sr, Ta, Ti, V, W, Y, Zn, Zr

X-MET5000 Detection Limits

Elemental Detection limits on Polymers (Polyethylene plastic), ppm

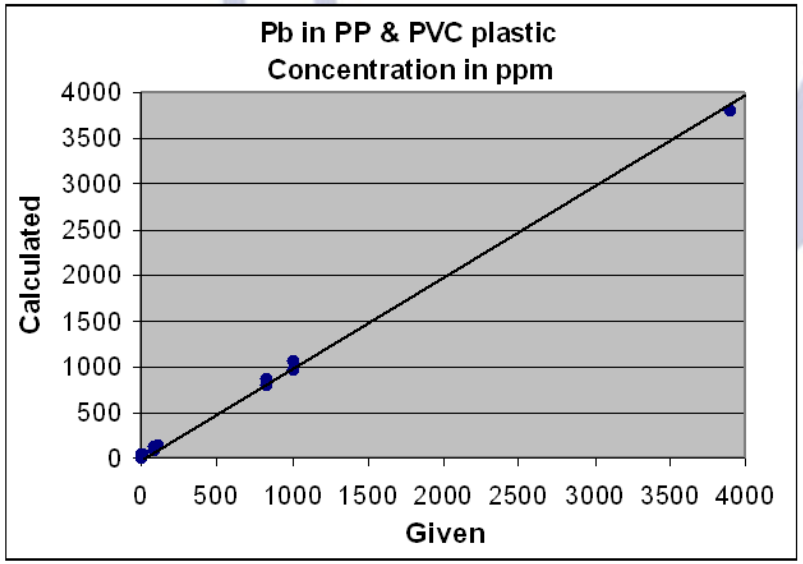
Meas. Time	Cr	Br	Cd	Hg	Pb
30s	35	< 5	18	6	< 5
60s	25	< 5	13	< 5	< 5
120s	18	< 5	9	< 5	< 5

Elemental Detection limits on different Metal alloys, ppm

Matrix	Meas. Time	Cr	Cd	Pb
Al-matrix	120s	140	15	15
Cu-matrix	120s	200	59	95
Fe-matrix	120s	200	70	82
Sn-matrix	120s	170	100	52

Optional Calibrations

- Optional empirical calibration for optimal accuracy and speed
  - Possibility to make customer specific calibrations with customer samples
  - Cd, Hg, As, Cr, Pb, Se etc.
- Complete Metal calibration which provides Metal ID and Assay
- Chlorine in plastics

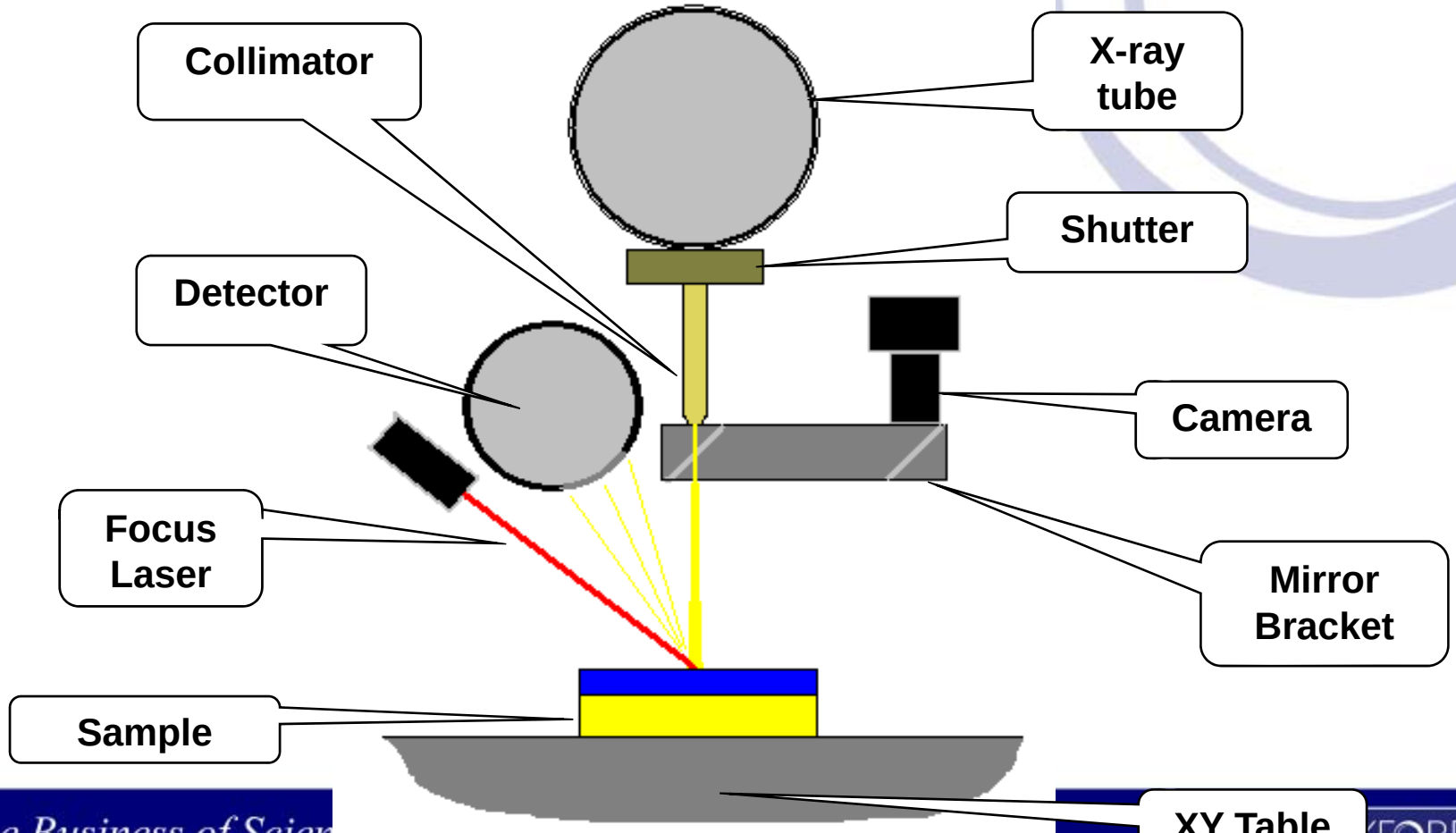


Chlorine detection limit in PE plastic	
30s	240 ppm
60s	170 ppm
120s	120 ppm

Micro-Spot Instrumentation

- Measures small spot
- Measures features on complex samples
- Measure in recesses
- Measures composition in layers
- Programmable multipoint assay

X-Strata 980 Components

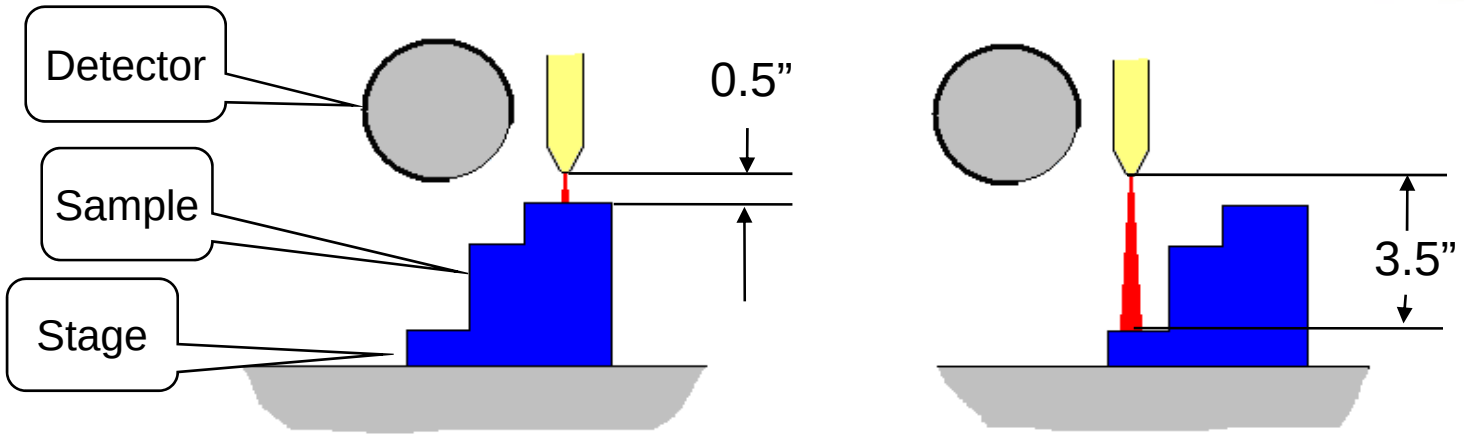


Advantages of X-Strata

- Designed to measure small features on complex samples
  - 4 Spot Sizes: 1.2, .3, .2, .1 mm
  - 50 kV, 100W
    - Gives good detection limits on small spot
  - Camera
    - Allows easy alignment
    - Allows image capture
  - Motorized Stage
    - Allows easy alignment
    - Allows programmed measurements
    - Allows mapping
  - Top-down measurement allows variable focal distance

Measure a sample with different focal planes

- **Working focal range is 0.5” to 3.5” or 13mm to 90mm**
- **Works with XYZ programmable mode**
- **Maximum sample height 9”**



Giant Closed Chamber Design

- Large chamber with standard 8.5” motorized Z travel
- **Closed chamber design provides a**dded safety and radiation protection when measuring light matrix (plastics) samples

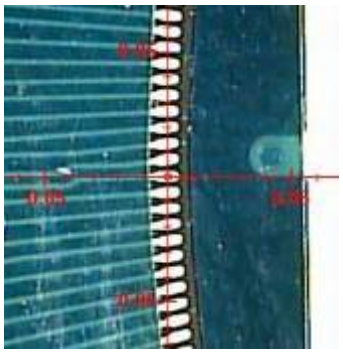
Interior XYZ Dimensions:

23”x 20”x 9”

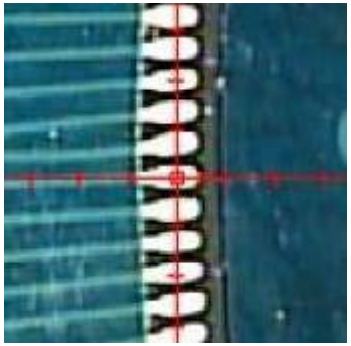
(580mm x 510mm x 230mm)

Small Spot Size

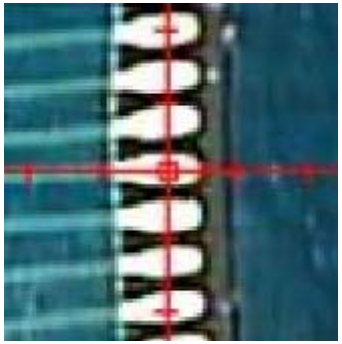
- 2X~4X digital zoom option is available to help aligning small collimators
- Advanced image interpolation software makes the image quality at higher magnification clean



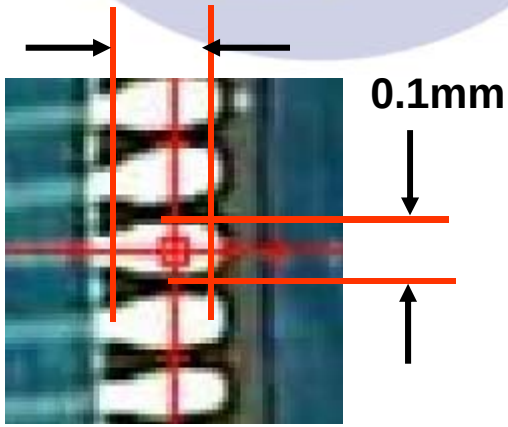
15x



30x

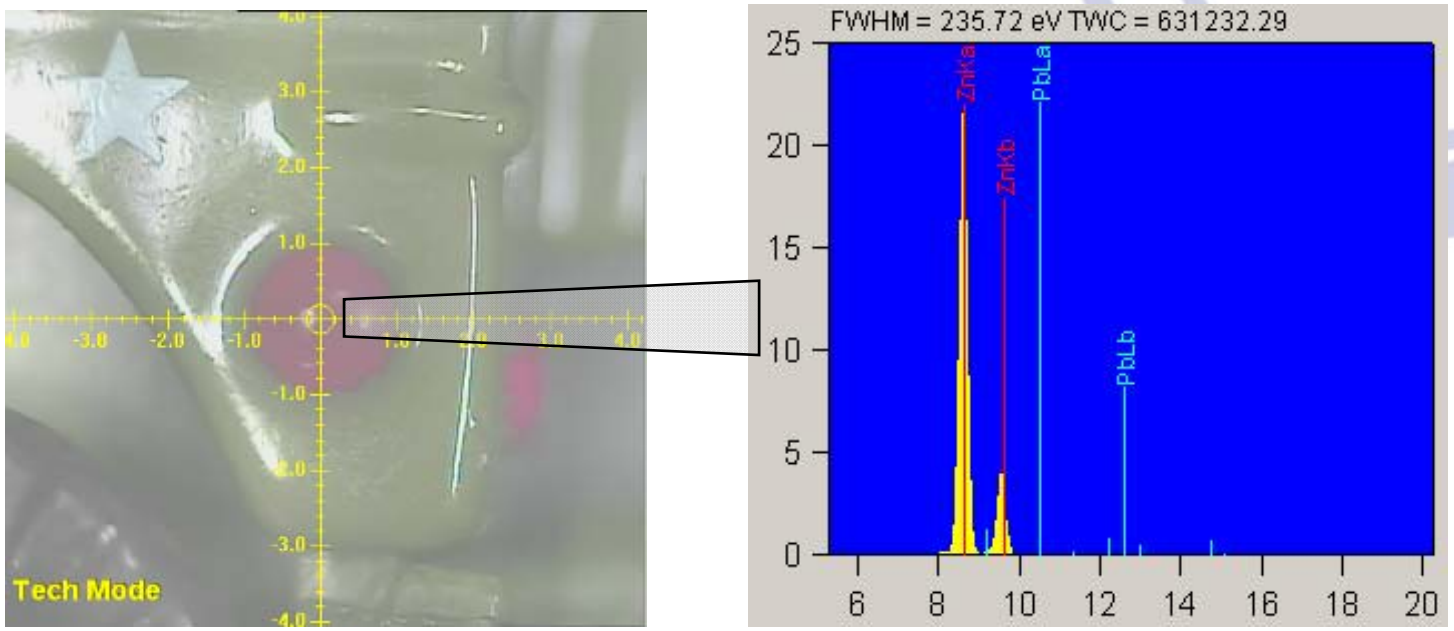


45x



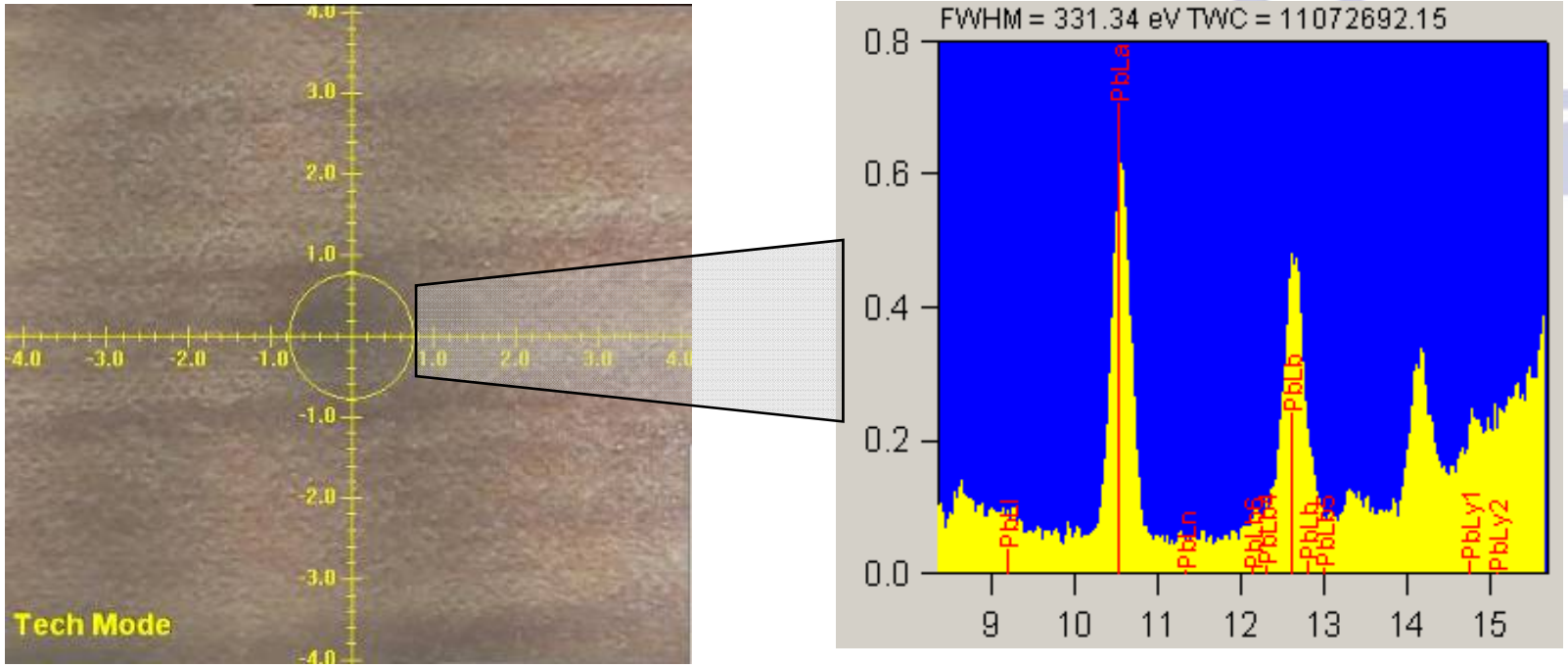
60x

Small Spot Example

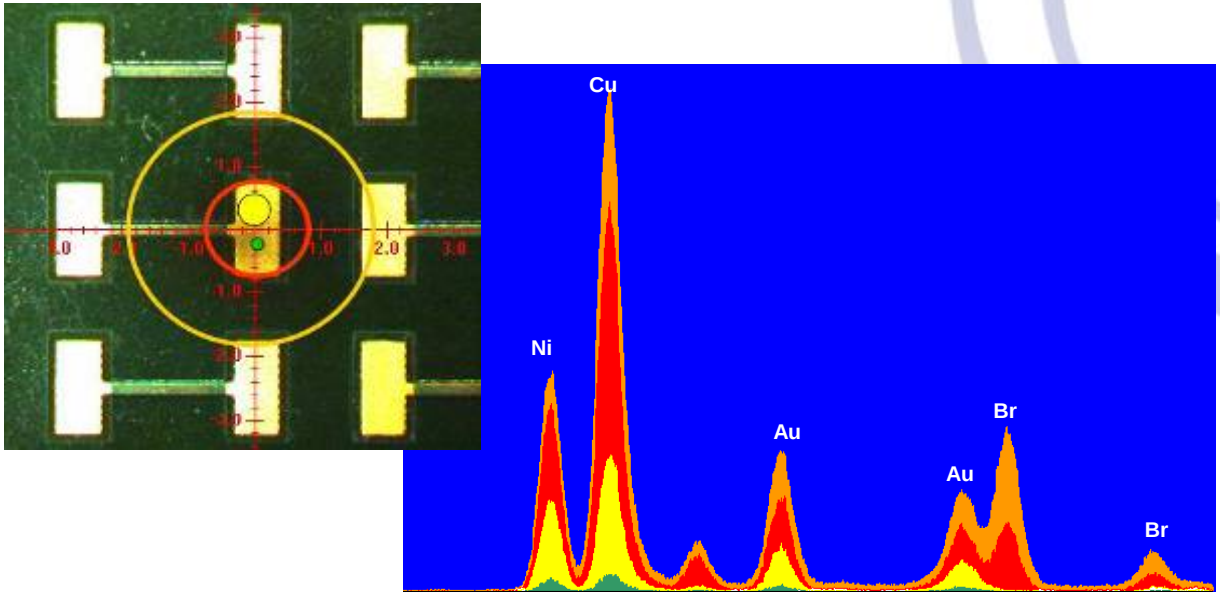


Note: measurement spot size 0.1 mm

Lead containing Coating

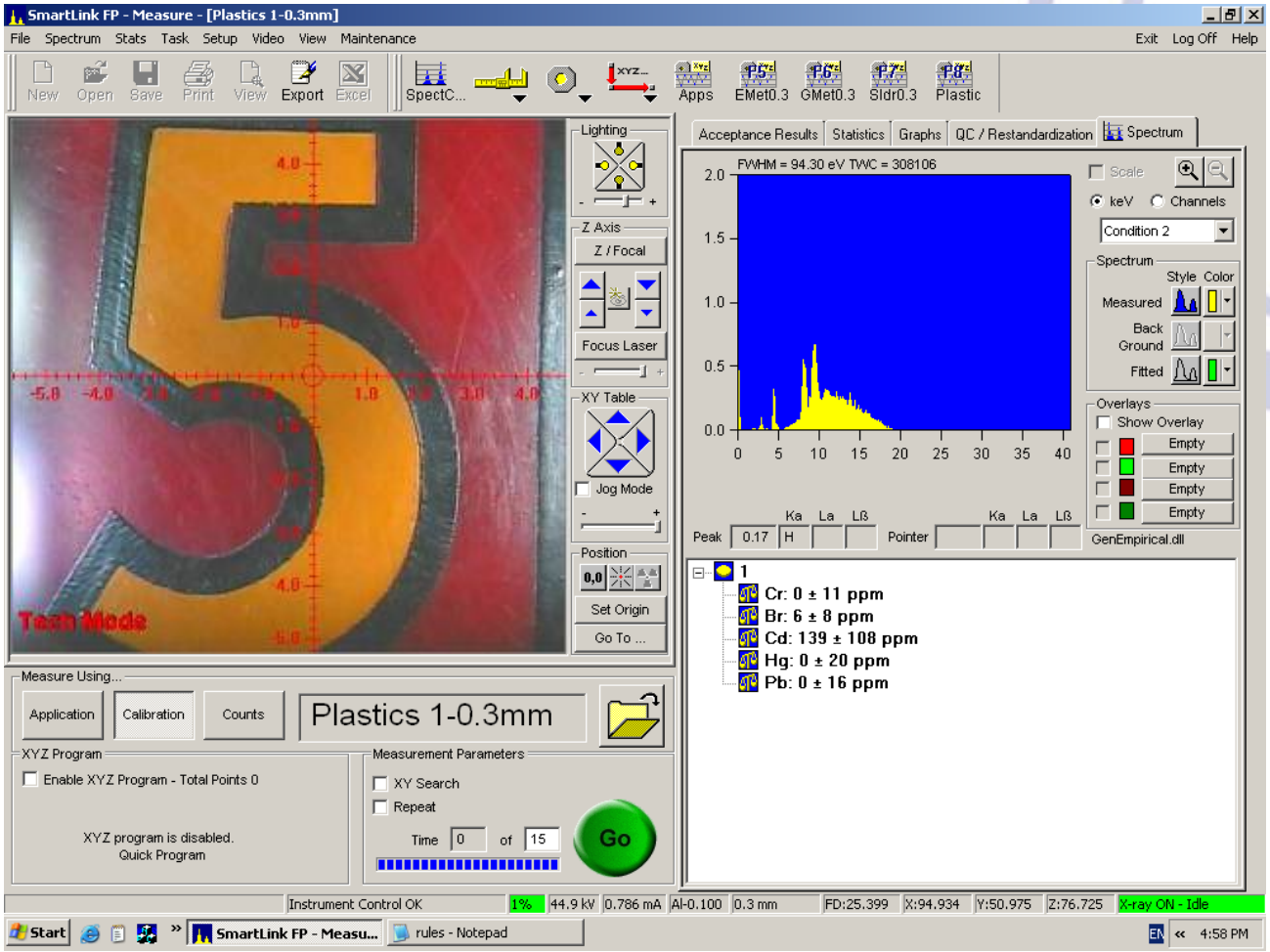


Effect of Spot Size



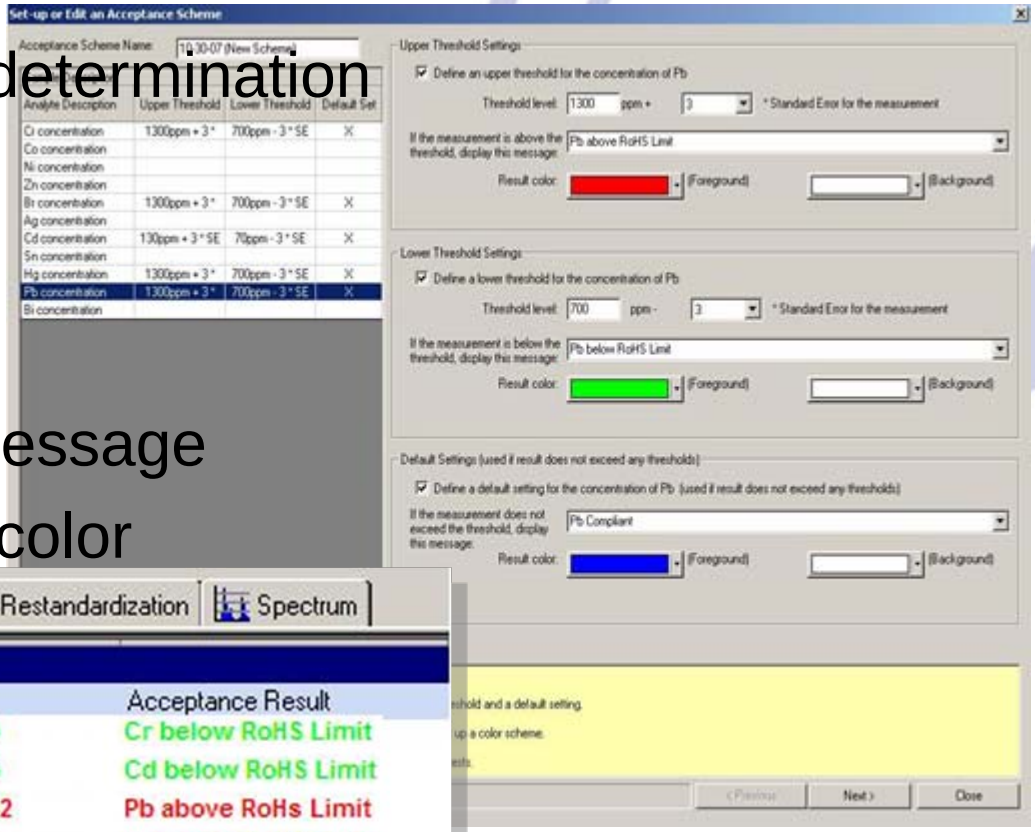
3.0mm spot (Handheld): too much interference (Br), poor accuracy
1.27mm coll: too much interference (Br), poor accuracy
0.3mm coll: ideal as it yields the optimal count rate
0.1mm coll: too small area, precision is compromised

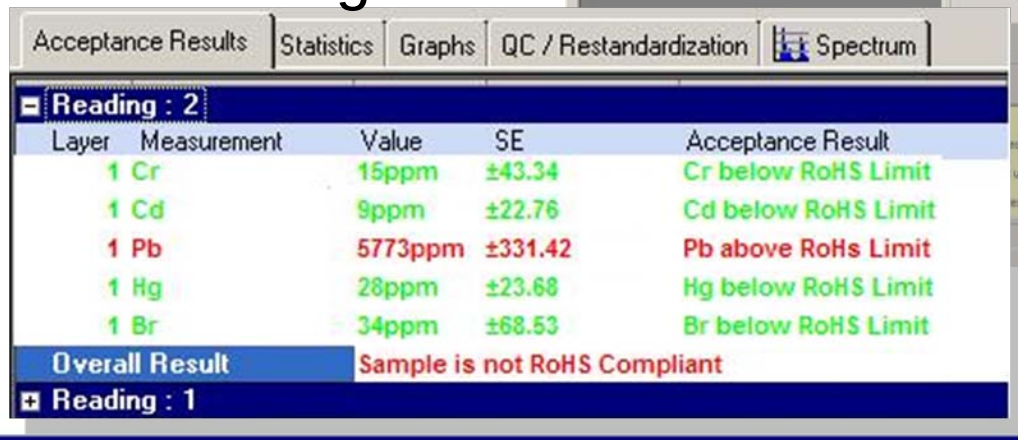
Testing Small Quantities of Paint



Acceptance Scheme

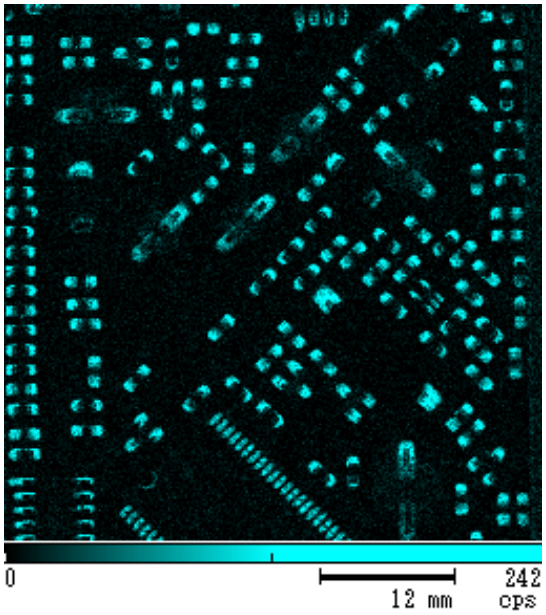
- Easy to make PASS/FAIL determination
- User define
  - Threshold
  - Rules
  - Overall determination message
  - Result text/background color



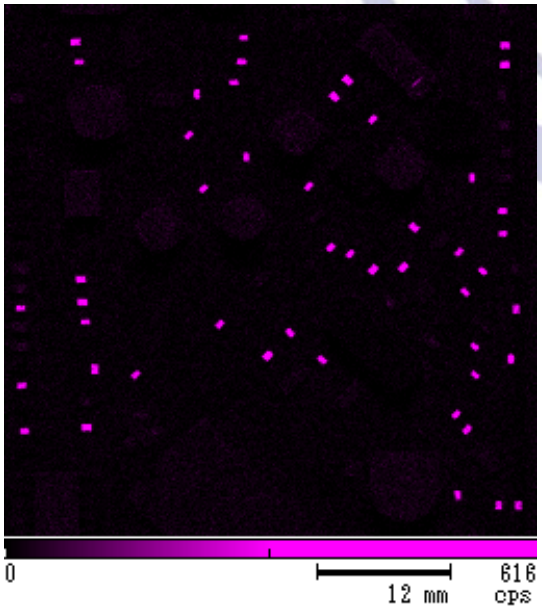


Mapping

Visual identification of problem area



Sn



Pb

X-Strata 980 Detection Limits

Typical Limit of Detection for RoHS Element Analysis (100 sec /Condition)

RoHS	Cr	Cd	Br	Hg	Pb
Cu Metals	255	75	N/A	N/A	145
SAC Alloys	N/A	160	N/A	120	110
Aluminium	15	14	N/A	15	5
Cables	4	7	6	7	9
Plastics	6	13	1	3	3

Bulk Analysis XRF

- Advantage
  - Ultimate detection limits
  - Good for raw material inspection
    - Paint prior to application
    - Raw plastic prior to molding
- Disadvantage
  - Not optimized for finished goods

Twin-X

- Bench top operation
- Good detection limits
- Automatic Sample changer
- Good for raw materials
  - Paint prior to application
  - Plastic prior to molding

Conclusion

- Impractical to completely analyze a toy
- Choose high risk areas
- Establish testing guidelines
  - Number of measurements per sample
  - Number of samples per lot
  - Locations to measure

Conclusion

- Understand what the screening device tells you
  - Local support from the manufacturer is key
- Choose instrumentation that complements your compliance scheme
  - Handheld for in-field
  - Microspot or Bulk for centralized testing