

# RoHS Test Methods

Presented by:  
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# Motivation for Presentation

- The EU RoHS directive takes effect July 1, 2006
- Customer confusion about -
  - Scope of directive
  - How much testing needs to be performed
  - Analytical Testing Techniques Available
  - Expected Costs



# Scope of RoHS Directive

## What is included:

- Applies to any product which is dependent on electric current or electromagnetic fields in order to work properly. Includes all non-electronic parts of the product.
- Voltage rating of product is less than 1000VAC or 1500VDC

## What is NOT included:

- Spare parts for the repair , or the reuse, of electrical and electronic equipment put on the market prior to July 1, 2006
- Military Equipment
- Medical Equipment
- Measurement and Control Equipment
- Large scale stationary industrial tools



# Specific Exemptions from RoHS

- Mercury in compact fluorescent lamps (with restrictions)
- Lead in cathode ray tubes, light bulbs and fluorescent tubes
- Lead as an alloying element in steel (< 0.35%), Aluminum (< 0.40%), Copper Alloy (< 4%)
- Lead in high melting temperature solders
- Lead in solders for servers, storage and storage array systems
- Lead in solders for network infrastructure equipment for switching, signaling, transmission and network management
- Lead in ceramic parts
- Cadmium plating
- Hexavalent chromium as an anti-corrosion of the carbon steel cooling system in absorption refrigerators



# Definitions

- **Unit –**

The smallest part of an electrical or electronic equipment that can be separated from the equipment by using ordinary tools, without destroying the function of the part when it is removed.

- **Mechanically Disjointed –**

Dismantling of a unit by simple processes (such as screwing, disconnecting and/or desoldering) using ordinary tools (i.e. not applying chemicals, cutting, grinding and/or polishing) without destroying the function of the unit.

- **Homogeneous Material –**

A homogeneous material cannot be “mechanically disjointed” into different materials. “Mechanically disjointed” means that the materials cannot in principal be separated into other materials by mechanical methods such as unscrewing, cutting, crushing, grinding or abrasive processes



# RoHS Enforcement

- RoHS is EU directive
  - Each member state will use the directive as model for local legislation
  - Each member state will be responsible for enforcement testing
- Enforcement testing will be regional and therefore may vary



# How Do I Comply with RoHS?

- It is up to each company to:
  - Assess risk
  - Build risk mitigation policy
  - Establish “due diligence” defense in case of litigation



# RoHS Concentration Limits

Substance	Allowed Limit (% by weight)
Lead (Pb)	0.1 (1000ppm)
Mercury(Hg)	0.1 (1000ppm)
Cadmium (Cd)	0.01 (100ppm)
Hexavalent Chromium (Cr6+)	0.1 (1000ppm)
Polybrominated biphenyls (PBB)	0.1 (1000ppm)
Polybrominated diphenyl ethers (PBDE)	0.1 (1000ppm)

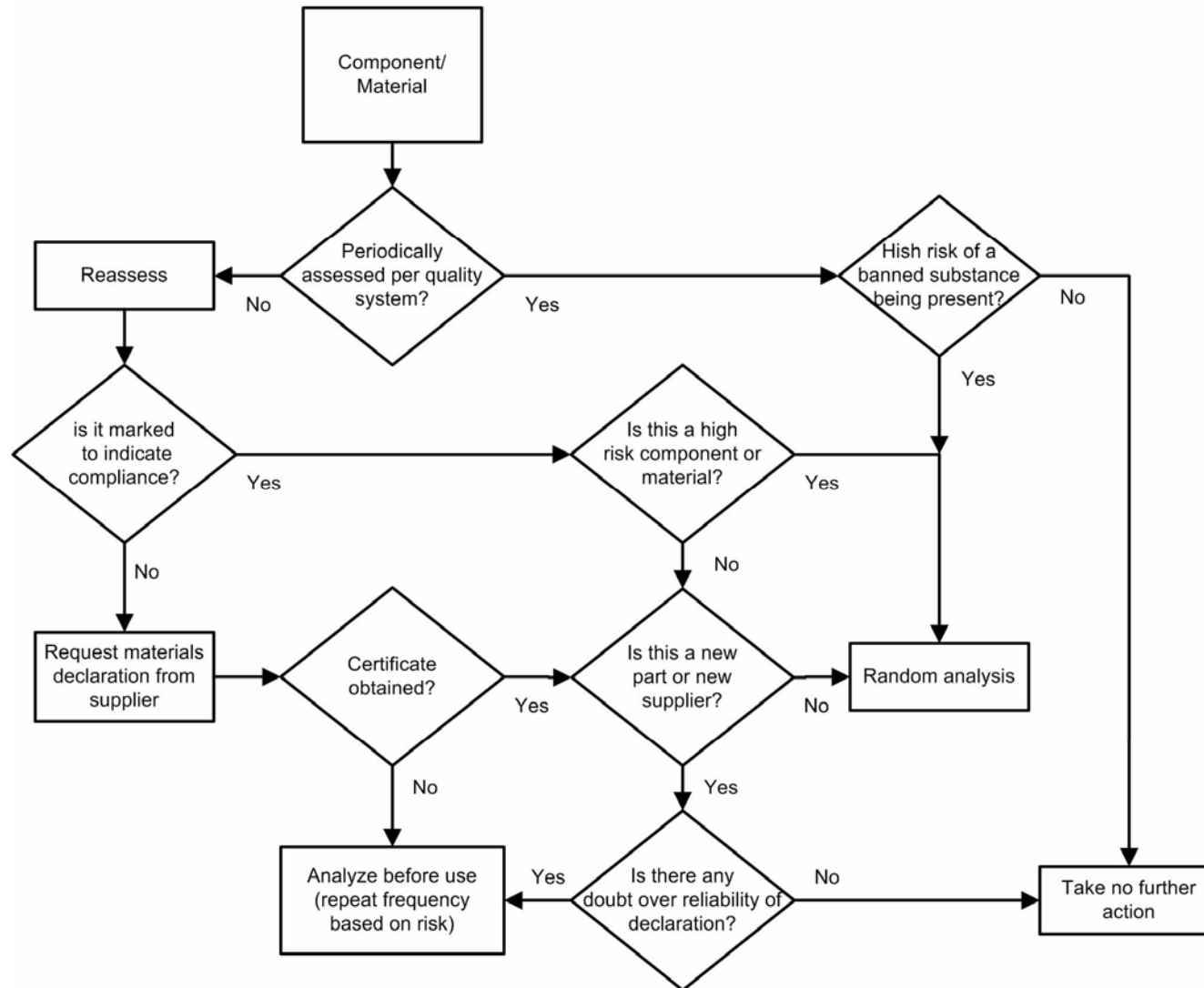




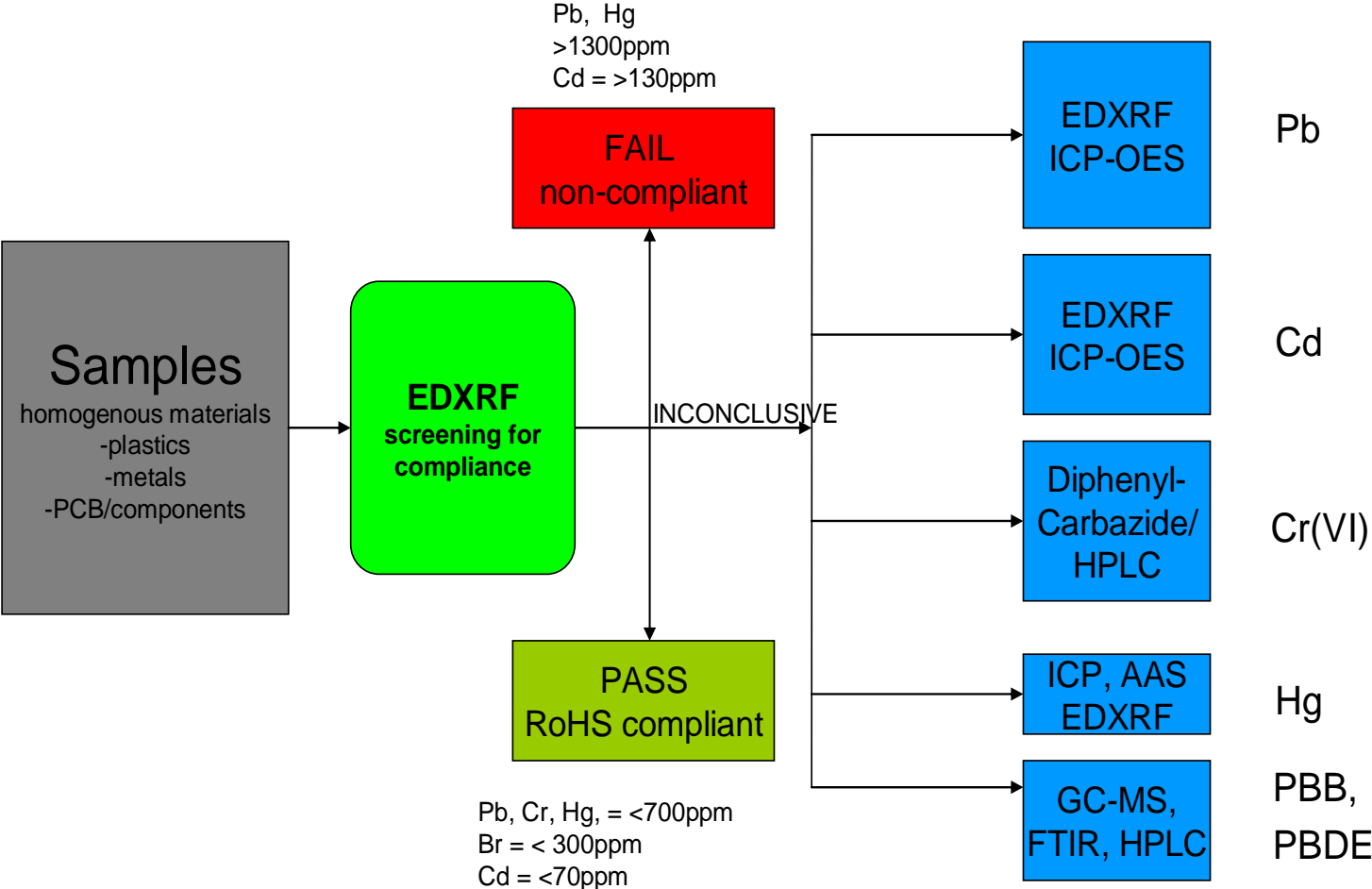
# Other RoHS Green Initiatives

- RPCEP (Regulation for Pollution Control of Electronic Products – July 1, 2006)
- JGPSSI (Japan Green Procurement Survey Standardization Initiative – July 1, 2006)
- SB20 (Electronic Waste Recycling Act of 2003 – California, Jan 1, 2007)
- “China RoHS” – March 1, 2007
- Also adopting EU RoHS directive: Australia, Canada, Korea, Taiwan

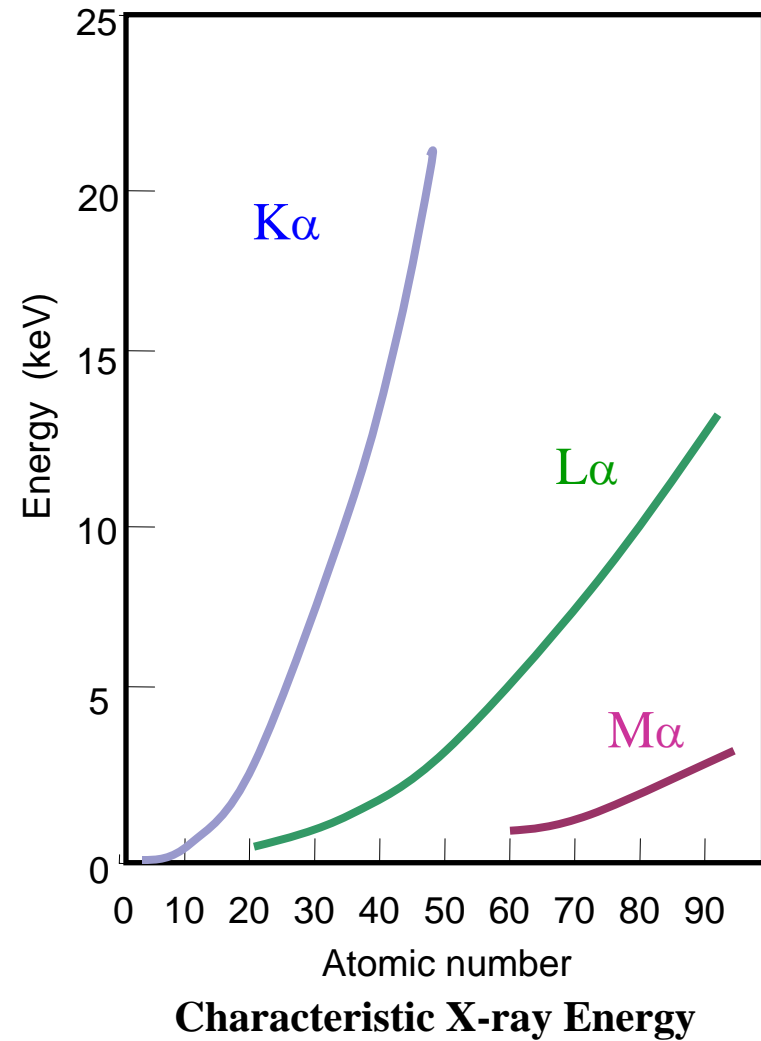
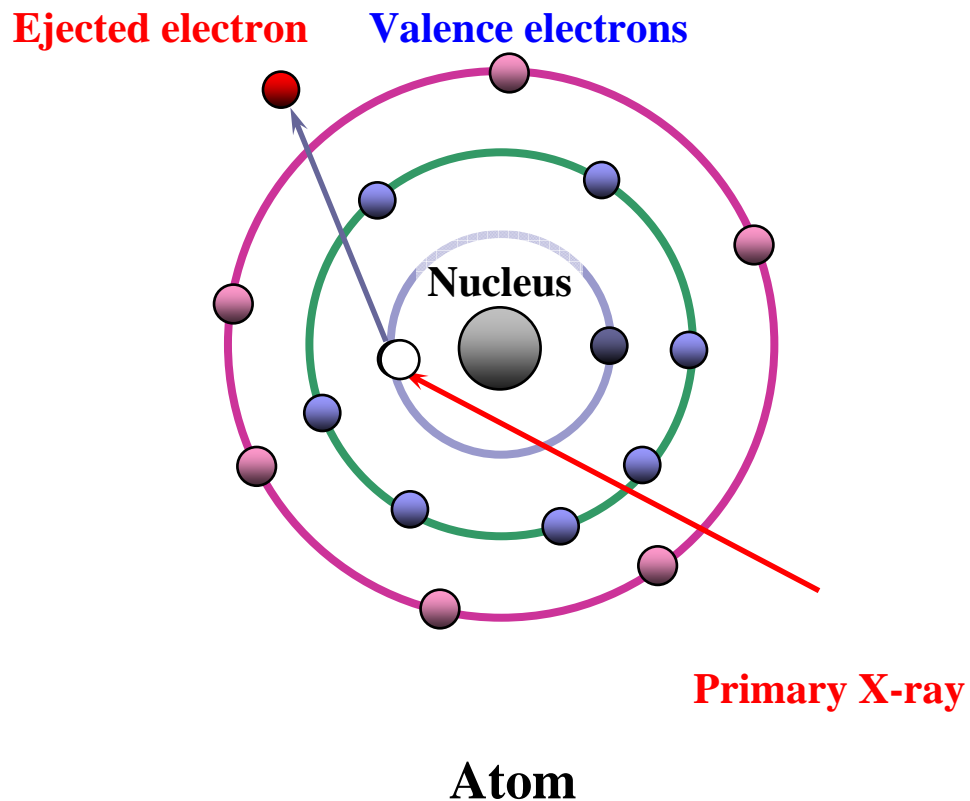
# RoHS Testing Decision Flowchart



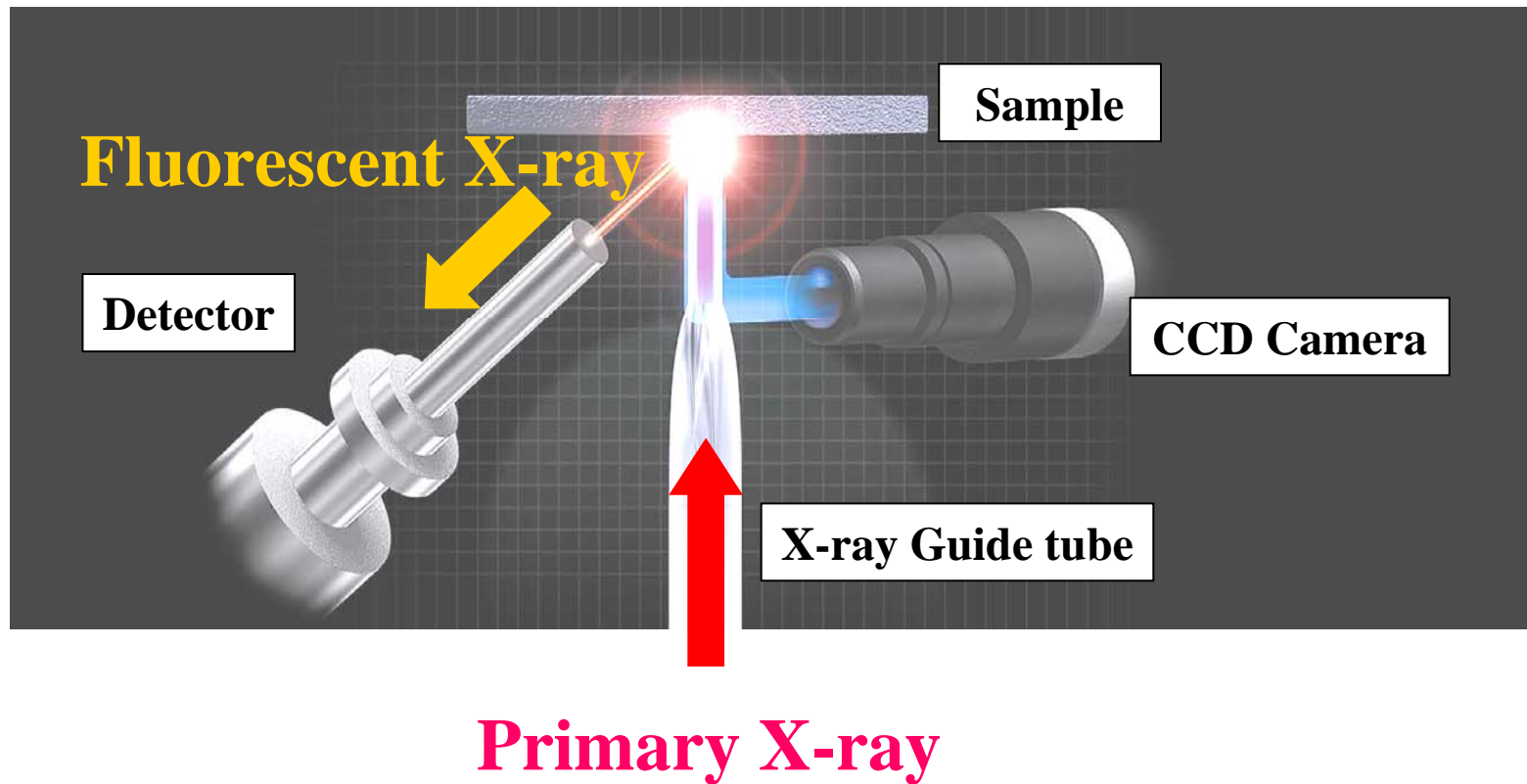
# RoHS Analytical Method Flowchart



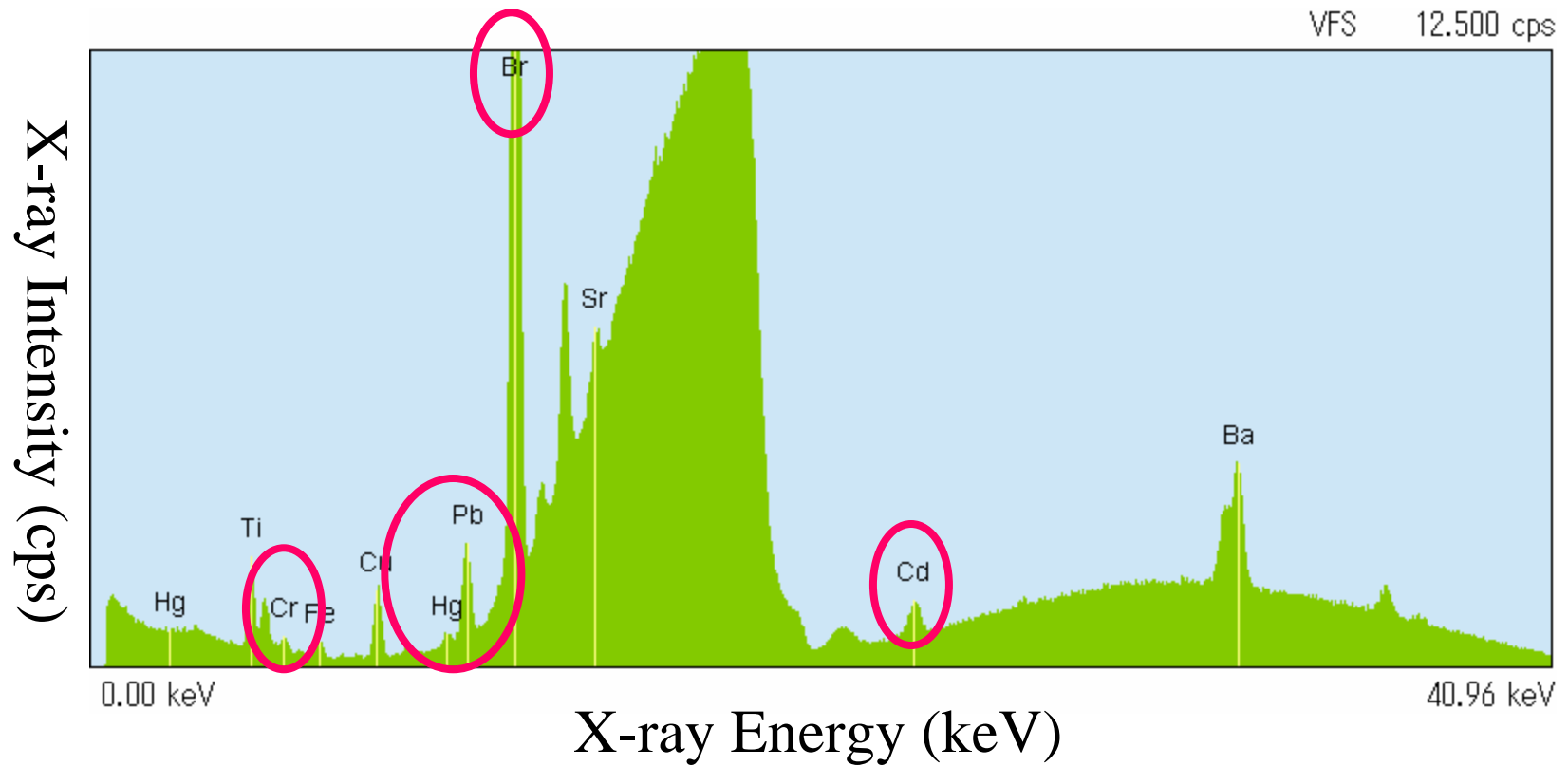
# Energy Dispersive X-Ray Fluorescence



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# Energy Dispersive X-Ray Fluorescence

Detector Technology	Advantage	Limitation
Proportional Counter	Fast Pb only screening Inexpensive Measure plating composition	Won't analyze all RoHS elements High limit of detection Good for Pb only
PIN diode	Good resolution (275eV) Does not need LN2 Can be used for portable systems	Resolution may not be good enough for some samples Sn $L\alpha = 3.440\text{KeV}$ , Cd $L\alpha = 3.134\text{KeV}$ ( $\Delta = 306\text{eV}$ ) Limit of quantification for Cd close to RoHS limit
Si, Si(Li)	Best resolution (150eV) Best limit of detection for all RoHS elements	More expensive LN2 required

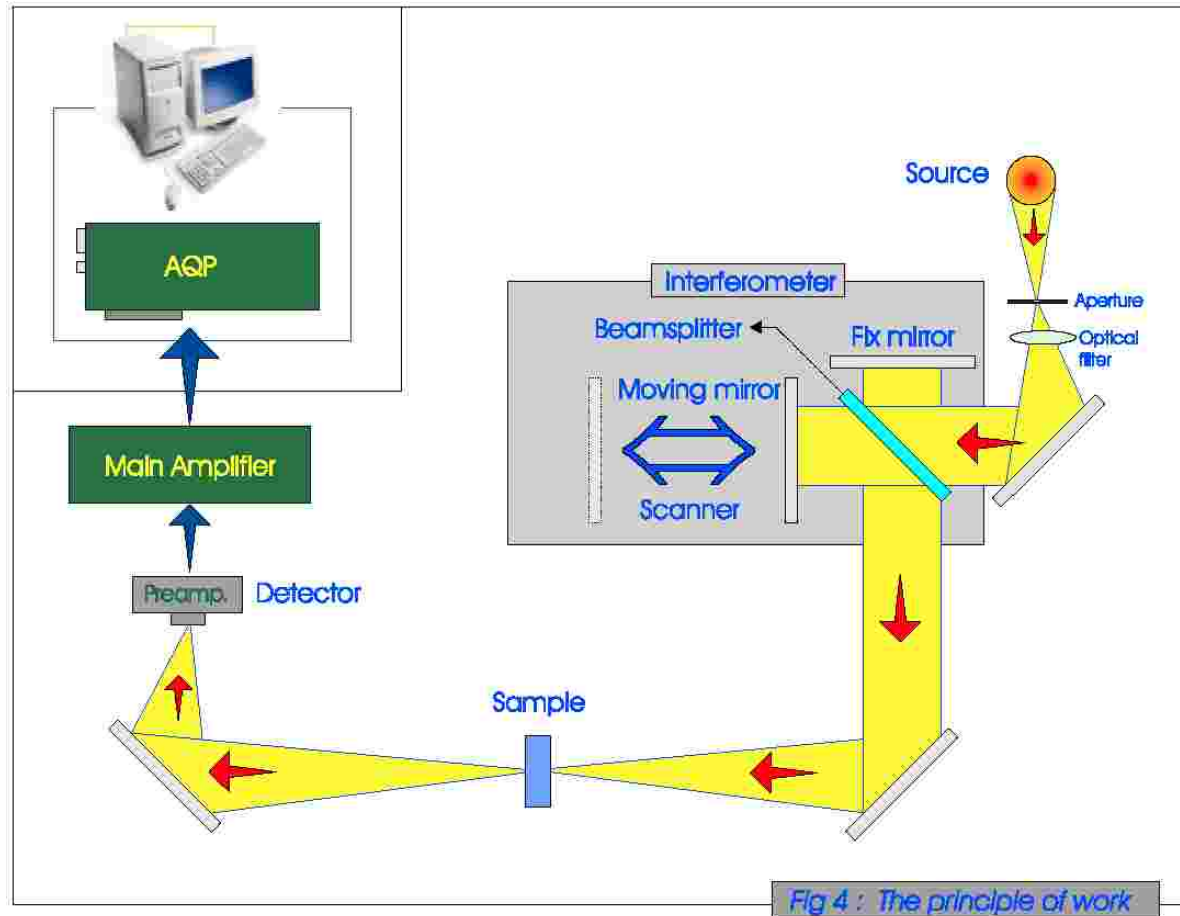


# Energy Dispersive X-Ray Fluorescence

- Other desired characteristics for XRF
  - Small X-ray spot size
  - Capillary optics in favor of collimators
  - Evacuated X-ray detector path for light element detection



# Fourier Transform Infra-Red Spectrometer (FTIR)

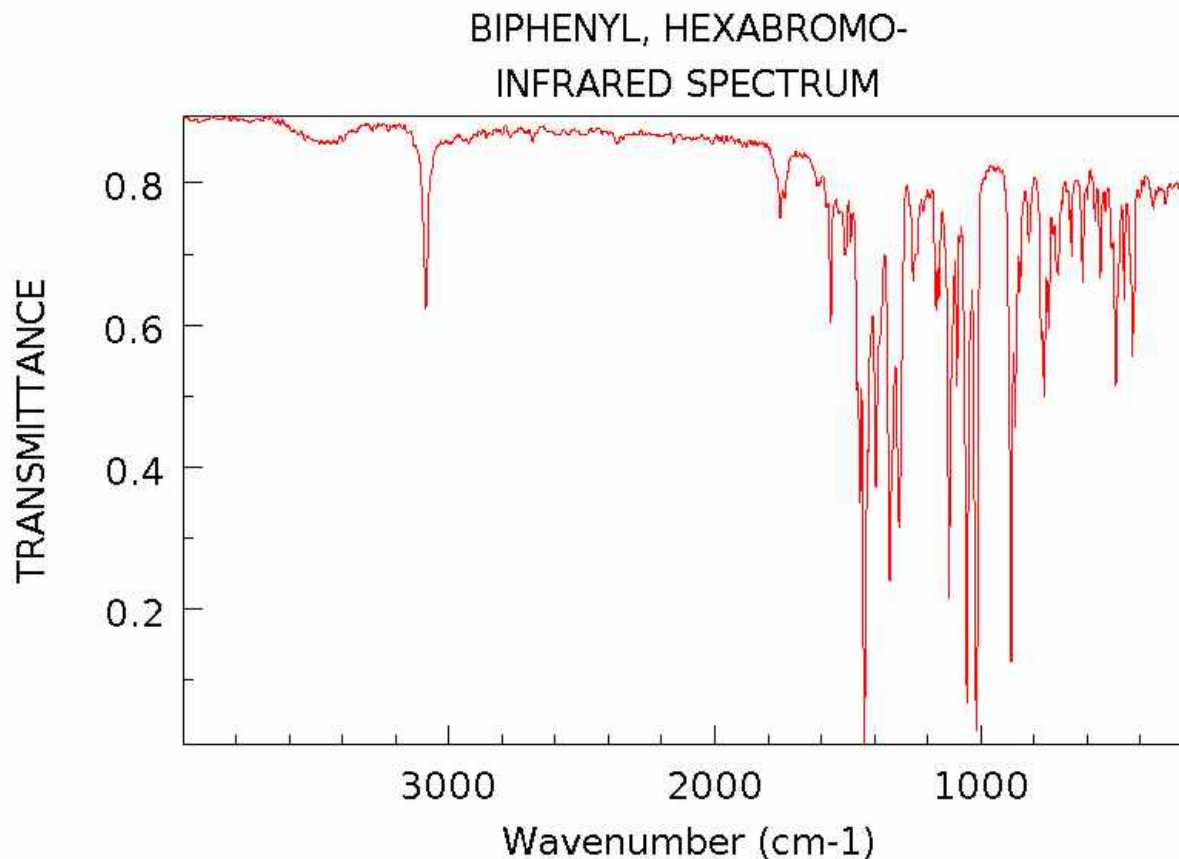




# Fourier Transform Infra-Red Spectrometer (FTIR)

- Many sampling techniques
  - Attenuated total reflectance (ATR)
  - Diffuse reflectance
  - KBr pellets
  - Transmission or Reflectance using FTIR microscope
- Detection limits are 30,000ppm
  - Materials containing PBBs and PBDEs will generally have concentrations exceeding 5% (50,000ppm)

# FTIR Spectrum of PBBs and PBDEs



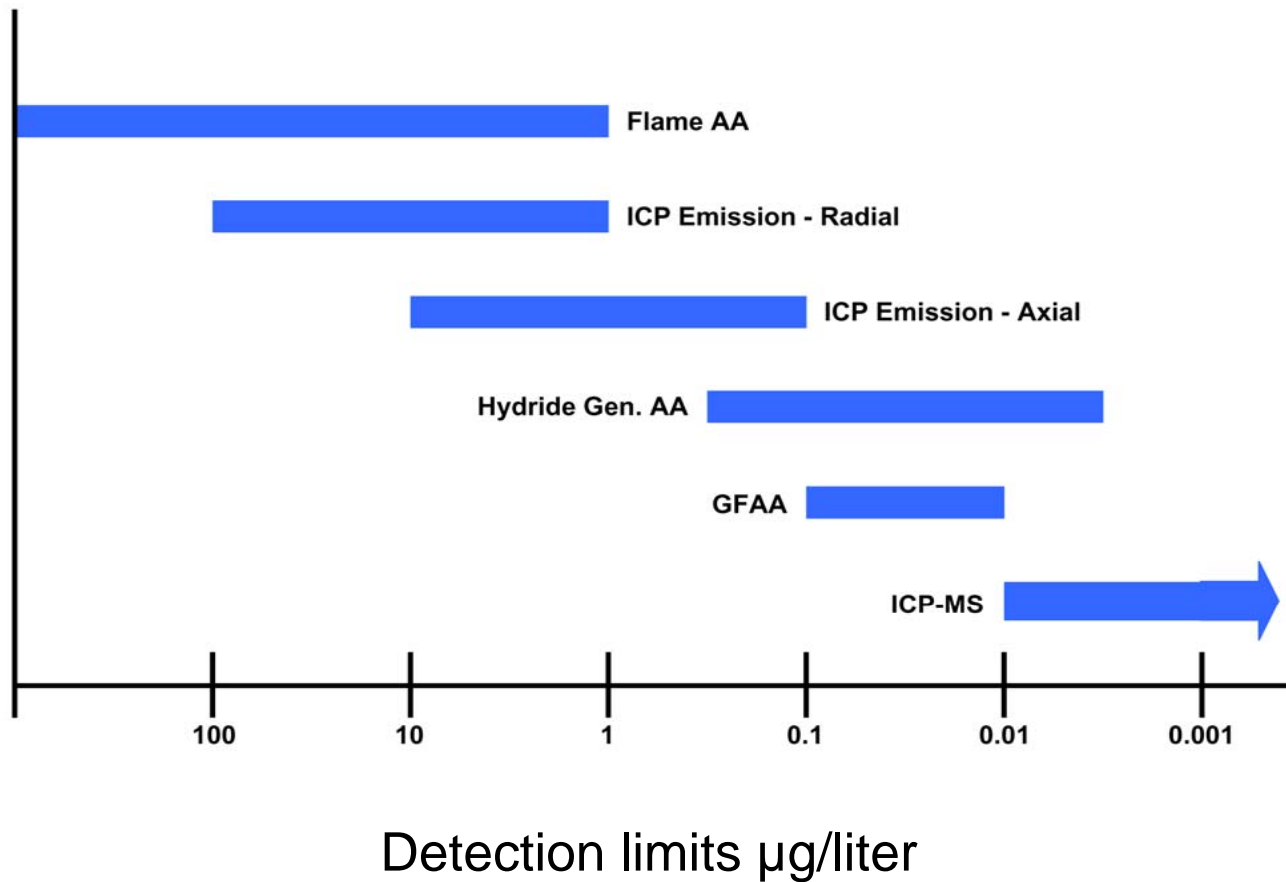
“Fingerprint region” for PPBs and PBDEs are in the  $1000\text{cm}^{-1}$  to  $1500\text{cm}^{-1}$  range



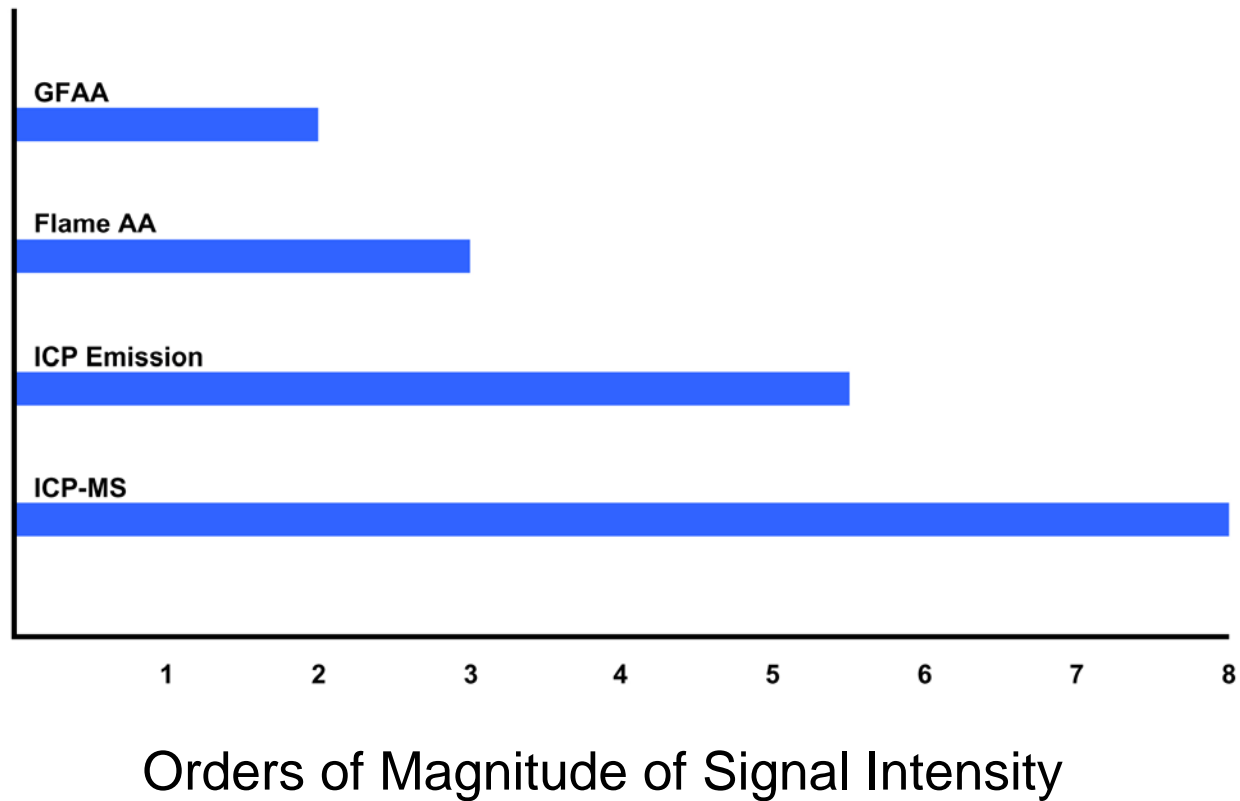
# Cr6 determination using “spot test”

- Based on ISO-3613
- Uses test solution with 1,5-diphenylcarbazide
- Test solution must be prepared not more than 8 hours prior to use
- Place 1 to 5 drops on clean sample
- A red/violet color will appear in a few minutes if Cr6 is present – ignore any color that appears much later
- Negative result concludes Cr6 testing, positive result requires more testing for Cr6 quantification

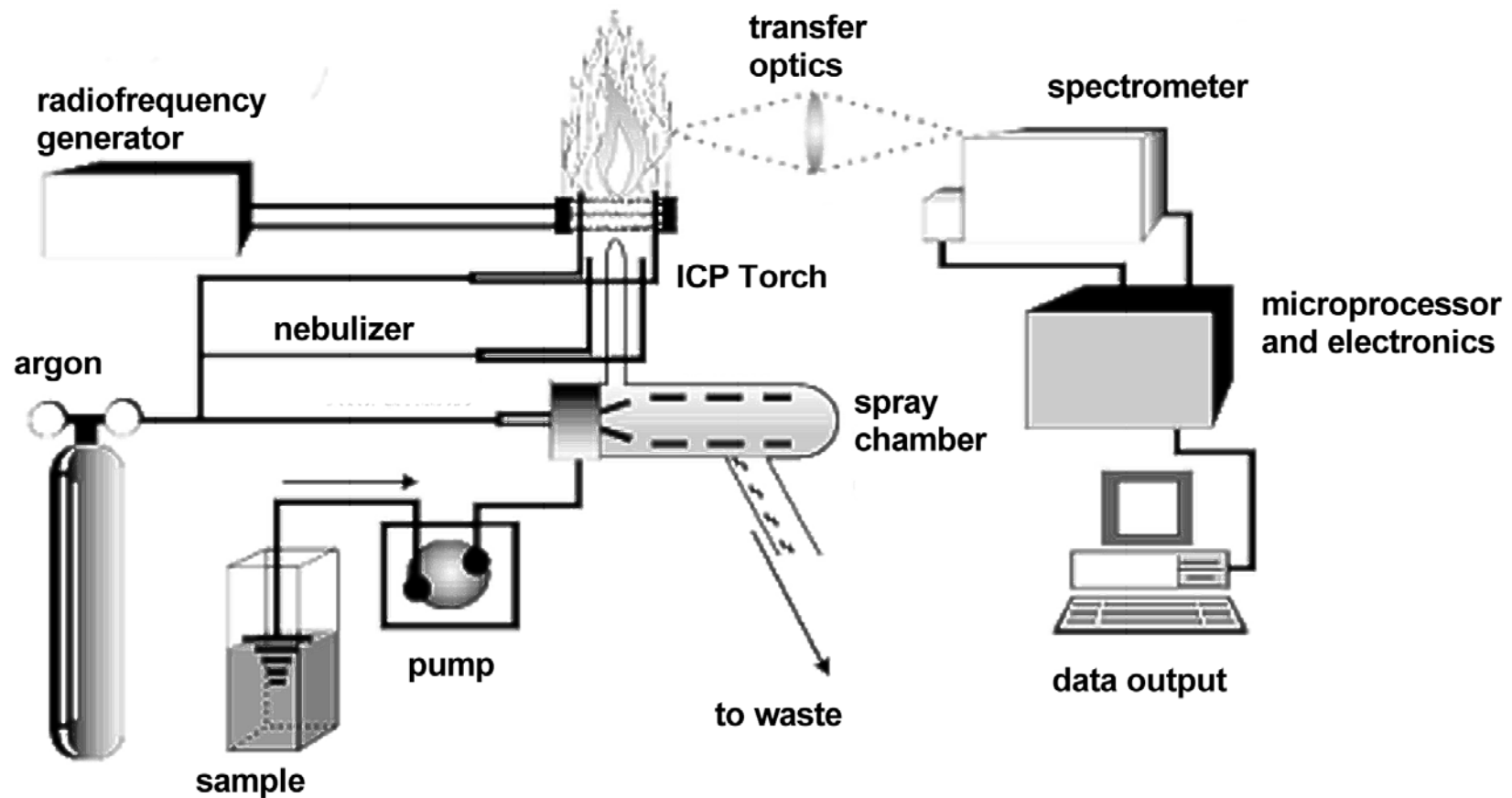
# Atomic Spectrometry Equipment

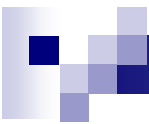


# Atomic Spectrometry Equipment



# Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES)



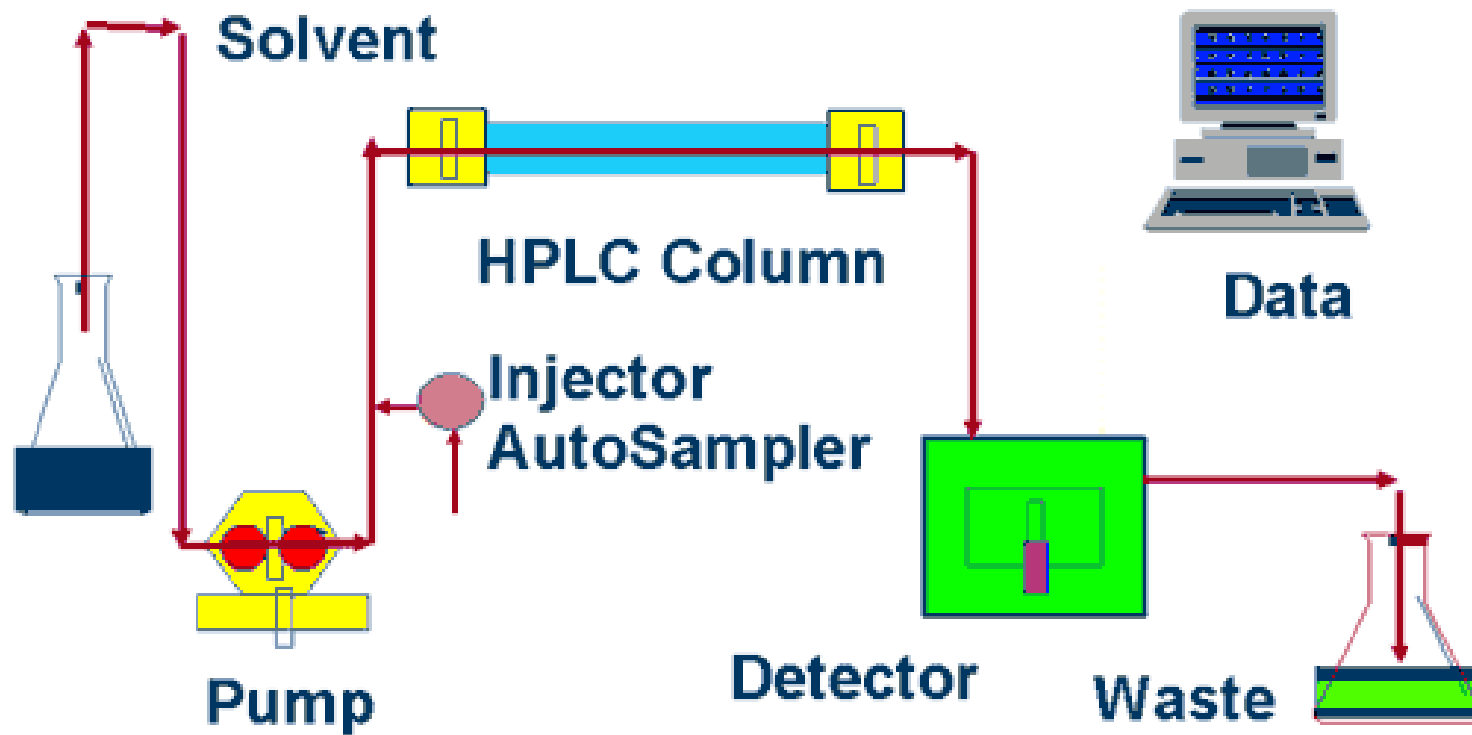


# Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES)

- Used for quantifying Pb, Cd, Hg and Cr when XRF results are in the “uncertain range”
- Lowest detection limits (ppb to ppt range)
- No matrix interferences
- Analytical range good for over 5 orders of magnitude (less need for dilution)
- Requires sample digestion (turn into liquid) with strong acids – use microwave digestion system to accelerate process
- Instrument requires calibration against reference samples – good linearity in analytical range (fewer calibration points)
- Sensitivity of instrument may require sample dilution to prevent detector overload



# High Pressure Liquid Chromatography (HPLC)

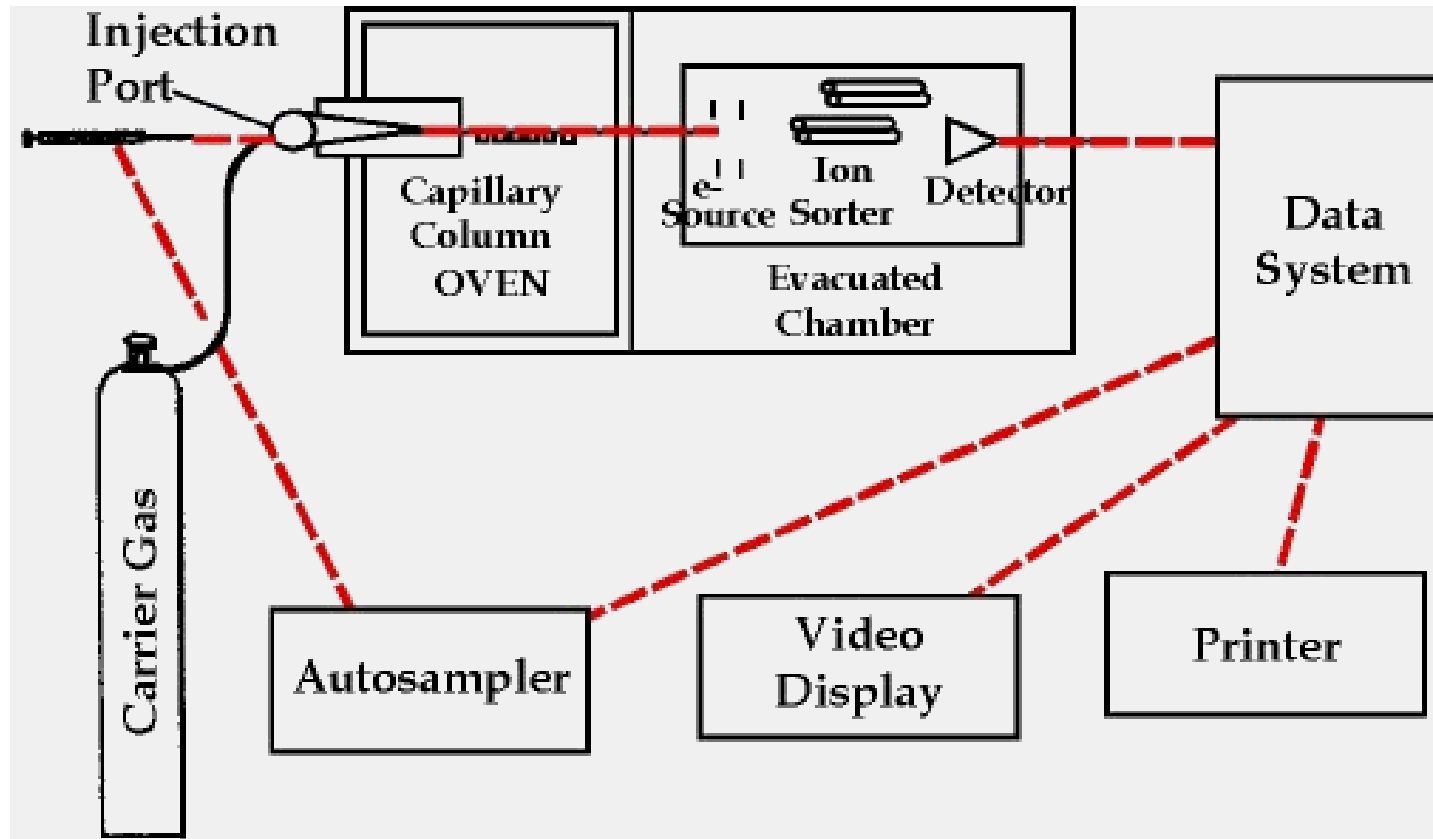




# High Pressure Liquid Chromatography (HPLC)

- Used for determination and quantification of PBBs, PBDEs when XRF total Bromine concentration is above limits
- Need extraction of PBBs and PBDEs from sample matrix – use FTIR first to identify matrix material for solvent determination
- Requires calibration against reference standards
- Used for quantification of Cr6 when XRF total Chrome concentration (Cr6 + Cr3) is above limits - use reference standards or XRF results for quantification
- Need extraction of Cr from matrix
- Use photodiode array detector for best results

# Gas Chromatograph – Mass Spectrometer (GCMS)





# Gas Chromatograph – Mass Spectrometer (GCMS)

- Use for quantification and identification of PBBs and PBDEs when XRF total Bromine concentration is above limits
- Most accurate analytical technique for quantification of PBBs and PBDEs
- Need extraction of PBBs and PBDEs from sample matrix – use FTIR first to identify matrix material for solvent determination
- Requires calibration against reference standards

# Analytical Testing Techniques

Analytical Technique	Advantages	Limitations	Cost
ED XRF	No sample preparation Non-destructive Can use FP mode	Matrix interferences Sample absorption	Low
FTIR	Little sample prep No reference samples	High detection limits ( $\geq 30,000$ ppm)	Low
Diphenylcarbazide Test	Fast go/no go test of Cr6	No quantification	Low
HPLC for Cr6	Best for Cr6 quantification Can be used with XRF results	Requires Cr ion extraction	Med
HPLC for PPBs and PPDEs	Better detection limits than FTIR	Needs sample extraction Destructive test	High
ICP-OES/ ICP-MS	No matrix interferences Low detection limits High resolution	Needs sample digestion Destructive test May need to dilute sample	High
GCMS	Low detection limits High resolution	Needs sample extraction Destructive test	High
AA/AAS	No matrix interferences Low detection limits High resolution	Needs sample digestion Destructive test Single element test	High



# Thank You!

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