

Experiment no. 4

Aim: Analysis of high explosives (picric acid) by instrumental technique (X-ray Fluorescence-XRF).

Requirements:

- a) Test Sample: Picric acid (synthesized-solid) and standard (liquid).
- b) Materials: Beaker(s), P1 Ucup, mylar sheet, lint free wipes.
- c) Instruments: XRF (Make: PANalytical B.V.; Model; Epsilon 1)

Principle:

1. The sample is irradiated with a beam of primary X-ray knocks out an electron from the K shell. An electron in the L shell drops into the K shell with concurrent emission of radiation of characteristic wavelength, the secondary or fluorescent radiation.
2. An electron in the M shell drops into the vacancy in the L shell with the emission of radiation. The process is repeated as an electron in the N shell drops into the newly created vacancy in the M shell.
3. Hence obtain spectrum consist of well-defined peaks due to these transitions, which are called $K\alpha$, $K\beta$, $K\gamma$, $L\beta$, and $L\gamma$ for the transition $L \rightarrow K$, $M \rightarrow K$, $N \rightarrow K$, $M \rightarrow L$ and $N \rightarrow L$.
4. These peaks may have shoulders, because the electron in the L or M shell can have one of three or five energies depending whether it is in a, s, p or d subshell but quantum mechanical selection rule allows two $K\alpha$ and two $K\beta$ transitions.
The energy differences within a shell are very small compared with the differences between various shells and these transitions show up. If at all, as extra peaks on the main peaks, they are labelled $K\alpha_1$, $K\alpha_2$ and $K\beta_1$ etc.
5. The X-ray spectrum is thus highly characteristic of an element.

According to Moseley for a series of the same type the observed wave length is related to the atomic number of the element. This leads to two conclusions of analytical importance:

- (1) A spectrum of unknown sample will have a series of peaks whose maximum wavelength is directly related to atomic number of the element in the sample, i.e., quantitative analysis is simple.
- (2) The peaks are so characteristic of an element that quantitative analyses can be carried out without prior separation, i.e., method is specific.

- (3) Furthermore, because the transitions are between inner shells, the valence state of the element is of little consequence so that the total amount of an element can be readily determined regardless of its chemical form.

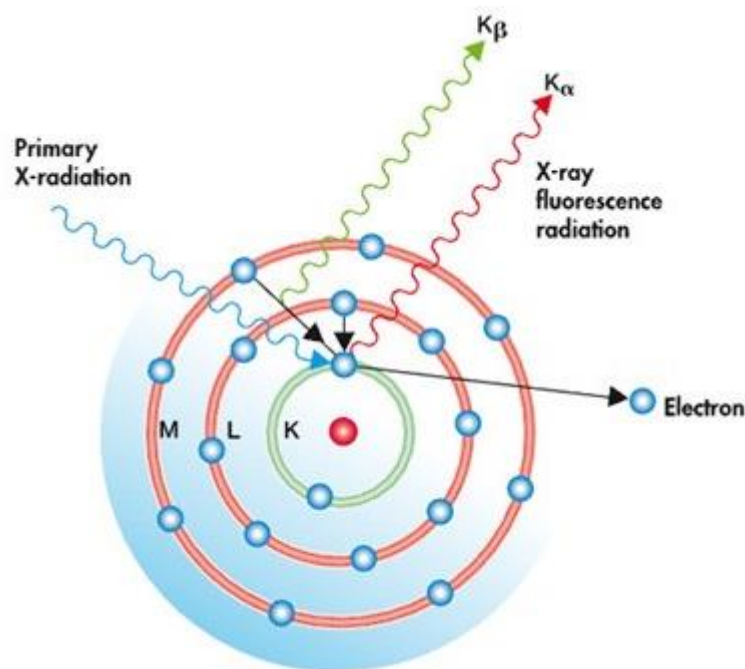


Figure illustrates X-Ray Fluorescence (on blank side)

Procedure:

1. The sample holder or P1 U cup was prepared by positioning it in the sample ring, then 3.6 μ m thin mylar film was placed over sample cup. The mylar film was placed such that it's free off any crease.
2. Then the sample to be analysed was loaded in the prepared P1 U cup and insert it in the sample dock.
3. The XRF spectrometer was turned on with the key.
4. The Epsilon 3 software was clicked to start and advanced mode was selected.
5. A pop-up tab appeared, then select the 'Omnian' sample.
6. The sample name input as sample 1.
7. The calibration for quantification was selected as Omnian.
8. Then the sample parameter (type) was selected as Liquid.
9. Clicked the measure button to start the process
10. The quantification may take approximately 20-30 minutes or more.
11. After the completion, the result and graph were saved as pdf file format, to be printed.

12. Repeated the similar procedure for solid sample.

13. The P1 U cup was removed and the spectrometer turned off with the key.

Conclusion:

Thus, on the basis of XRF analysis the heavy metal quantitation was done in sample synthesized and identified on comparison with data to standard sample.