Chapter 4

We saw in chapters 3 and 4 within the book that in order to solve an analytical problem, the sample type with identified analyte, the sample preparation step and the measurement technique are all interconnected. They can in some ways be considered as a "hub" within the 'analytical approach'. When the sample and the analyte(s) within it are defined as shown in chapter 3 and figure 3.1 (for identification and/or measurement purposes), the process of selection for this 'hub' is underway. However, before we go on to consider certain measurement problems and their solutions, it is worth considering a fundamental concept with regard to the "measurement" step itself. We have seen a number of measurement techniques presented already in chapter 4 and many rely on some form of instrument (if not all techniques, based upon the definition of how a measurement is actually achieved). If we reduce the process down to those measurements based upon an instrument that fundamentally involves an energetic process then, schematically, we are considering the following:

Stimulus: →	**Sample** Interacts with →	**Detector** sensitive to →	**Measurement**
Energy input based:	Stimulus:	: stimulus or emitted	and read-out
Upon:	Absorption / emission /	radiation from sample	
↓	Change of state =>	or physical / chemical	
	Physical or chemical	change of state	

EMR
Electrical (Voltage or Current)
Heat, Chemical….etc.

How each of these parts within the schematic may be explained is best illustrated using an example technique we have already encountered, that of UV-visible spectrophotometry. Here the energetic input is from the visible and ultra-violet part of the electromagnetic (EM) spectrum. The **analyte** from the sample **interacts with this EM radiation** and **absorbs** the energetic radiation (**raising the analyte's energy** system accordingly). The **sensitive detector** system **responds to the intensity of the selected energy** both before and after its interaction with the analyte of interest and **presents a read-out**, allowing **a measurement** to be made – and thereby **allowing a comparison** to be made **between the presence and absence of the sample's analyte**. This process can allow both a qualitative and a quantitative measurement of the analyte if selection of the radiation to be measured and the detector to be used are carefully matched / controlled (e.g. wavelength of EMR, band-width of radiation, CCD, PDA, etc.).

Problem 1

If we return to Question 1 in our on-line problems from chapter 1 and the contaminant found in a loaf of bread purchased by a member of the public; further examination of the loaf by the analytical team finds three transparent, colourless strips of a thin, very flexible solid material, each having a smooth surface and nearly identical in size (50 mm by 15 mm). They have the appearance of an inert plastic / polymer material, possibly from the machinery used in the manufacturing process.

What instrumental technique(s) would you consider to quickly identify the three contaminants found in the bread?
Consider Table 4.6 and Figure 4.12 first and then Tables 4.7 and 4.9 to slowly focus your thoughts.

Problem 2

If we return to Question 2 in our on-line problems from chapter 1 and the possible issue of an imported children's toy not meeting consumer regulations because of the paint used in its manufacture.

What instrumental technique(s) would you consider to quickly identify if there is a consumer problem associated with the paint used in the toy's manufacture?

Consider Table 4.6 and Figure 4.12 first and then Tables 4.7 and 4.9 to slowly focus your thoughts.

Problem 3

If we return to Question 3 in our on-line problems from chapter 1 and the bottles of sunflower oil for cooking that have been withdrawn from the shelves in a local supermarket by the store manager because they "don't look right" and appear to have "gone off".

The sample presented to you demonstrates the problem. The 'suspect' sunflower oil batch is slightly darker in colour, is cloudy and with a faint brown tint compared with the acceptable batch which is a clear, light straw yellow colour.

What procedures and instrumental technique(s) would you consider using to help identify the problem with the sunflower oil?

Hint: Consider the physical changes that are evident first, how they might contribute to the observed changes and how you might prove / confirm your hypothesis as to their contribution to the problem.

Problem 4

In the on-line questions given in Chapter 3, you were asked to consider the following scenarios.

i) The presence of (the NSAID) Phenylbutazone in meats sold for human consumption.
ii) (Methyl) and other alkyl Parabenzoic Acid additives in face cream
iii) Manuka Factor in honey to check adulteration or Faked quality goods
iv) Levels of Bisphenol A in printed Till and Card Receipts
v) Levels of Radio-nuclides in Plants and Soils from old uranium mining activities. (U, Ra, Pb, etc.)
vi) Concentration of titanium dioxide in sunscreen
vii) The composition of scrap metal salvaged from a waste facility, for sorting and selling
viii) The determination of elemental composition of mineral tablets sold on the Food Supplement market

ix) The determination of the (range of) fatty acid content of Soya beans.

x) The determination of ammonium ions in a processed waste-water discharged to a river.

Which analytical technique(s) could you use to determine the analytes stated above, having been obtained for the sample types shown?

What conditions are required (Direct or after processing) in order to make these measurements successful? As before, the first in the series (Phenylbutazone , PBZ) is given as a guide in the solutions section. Remember to consult section 4.3 in Chapter 4, Figure 4.12 and Tables 4.1, and 4.4 to 4.9 to help guide your decision making process

Problem 5

We saw in the on-line problems for Chapter 1 that '<u>Problem 7</u>' asked you to consider recycled plastics and secondary plastics products destined for use by the general consumer as a potential problem because of certain metal and metalloid elements being present as well as certain non-metallic elements (other than the more common backbone elements of carbon, hydrogen, oxygen, nitrogen and sulphur). With this in mind, we asked you to consider the scenario (in Question 3 of chapter 3 on-line) where you are a county public analyst and have been sent a yellow-coloured plastic drinking mug from the Trading Standards authority. The mug is imported from a non-EU country and the only details you have are the country of origin, the importer, that the 'plastic' material itself is possibly constituted from recycled sources but stated to be HDPP and "suitable as a drinking vessel".

a) How might you identify the type (and possible range) of plastic material it is made from?
b) How might you identify the suitability of the yellow plastic mug for its stated use as a "drinking vessel"

Hint: See the general feedback to Problem 1 in this section to aid part a), the specific feedback to question 3 in chapter 3 on-line for parts a) and b) and the feedback to Problem 7 in chapter 1 on-line; all for guidance.