

## FEDERAL UNIVERSITY OTUOKE

### The Eagle Eye of Nature, Fact, Figures and Concepts: An Analyst Perspective

## **An Inaugural Lecture**

By

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## The Eagle Eye of Nature, Fact, Figures and Concepts: An Analyst Perspective

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## **DEDICATION**

This 5<sup>th</sup> Inaugural Lecture series is dedicated to the Almighty God: O give thanks unto the Lord, for he is good: for his mercy endureth forever, Psalms 107:1

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### PROTOCOL

The Pro-Chancellor,

The Vice- Chancellor,

Members of the Governing Council,

Registrar and other Principal Officers of the University, Dean of the Postgraduate

School,

Deans of Faculties and Directors of Academic Institutes,

Distinguished Professors and Scholars,

Heads of Departments,

Coordinators of Academic Programmes, Staff

and Students of FUO, Distinguished Guests,

Ladies and Gentlemen

#### PREAMBLE

I feel greatly honoured and privilege to be given the opportunity to deliver this 'august' lecture in October. Jesus must be honoured in my life today for keeping me and my family alive.

I have a deep sense of appreciation, fully aware that, this will be the fifth Inaugural lecture in the series to be delivered from an experimental Chemist and fellow of the chemical Society of Nigeria in this University. I do know that the inaugural lectures are meant to serve as opportunity to use ones discipline to ginger, reflect and mentor others on matters that are of academic interest and sufficient importance and to the wider society. I wish, therefore to this tradition with the frame work of my chosen topic for this lecture: "The Eagle Eye of Nature: Facts, Figures and Concepts from An Analyst Perspective".

Mr. Vice Chancellor Sir, I have the singular honor to stand here today before you all, and my learned colleagues, erudite scholars to render as much as time permits my research work and findings that made me a chartered chemist, consultant on environmental pollution/waste management and finally a Fellow of the Chemical Society of Nigeria. *"It is the Lord's doing it is marvelous on my side"* (Psalms 118 vs 23).

Throughout my journey in existence, and contact with science in general and chemistry in particular I- have learnt a variety of terminologies, *Facts, Figures and Concepts*. Each new topic I encountered always provided its own collection of words and ideas – which at times, I may think to be endless. But each of these ideas within a particular topic is related in some way to the others; hence no concept in chemistry is isolated. Thus, it helped me to understand the topics clearly as well as the whole picture; that is the inter connectedness of all individual terms and ideas.

This became a more effective and satisfying way of learning and understanding chemistry rather than memorizing separate facts. Actually, this became also familiar process (routine) for me, even when I was not thinking about it that way. Many of the elements were analyzed in my daily life and activities by looking at relationships or connections. Example is by observing God's creation. *Genesis 1:* 26 says "And God said, let us make man in our own image (spirit) after our likeness (wisdom) and let them have <u>dominion</u> over the fish of the sea (marine environment), and the fowl of the air (atmospheric environment), and over the cattle and over all the earth (ecosystem and soil environment) and over everything that creepeth upon the earth (Holy Bible, KJV).

I learnt also how to organize information visually so as to see how it all fit together. One of the techniques for describing related ideas is called a Concept map (e.g. like the Periodic Table)

Group Period	<b>→</b> 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	1 H																	2 He
2	3 Li	4 Be											5 B	6C	7 N	8 0	9 F	10 Ne
3	11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 CI	18 Ar
4	19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
5	37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
6	55 Cs	56 Ba	57 La	* 72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 T1	82 Pb	83 Bi	84 Po	85 At	86 Rn
7	87 Fr	88 Ra	09	* 104 * Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn	113 Nh	114 Fl	115 Mc	116 Lv	117 Ts	118 Og
				* 58 Če	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu	
				* 90 * Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr	

Fig. 1: The periodic table

In a Concept map, an idea is represented by a word or phrase enclosed in a box (see Scheme below). Note a concept map should be so clear that if some of the terms are erased, the missing terms could easily be filled in following the logic of the concept map

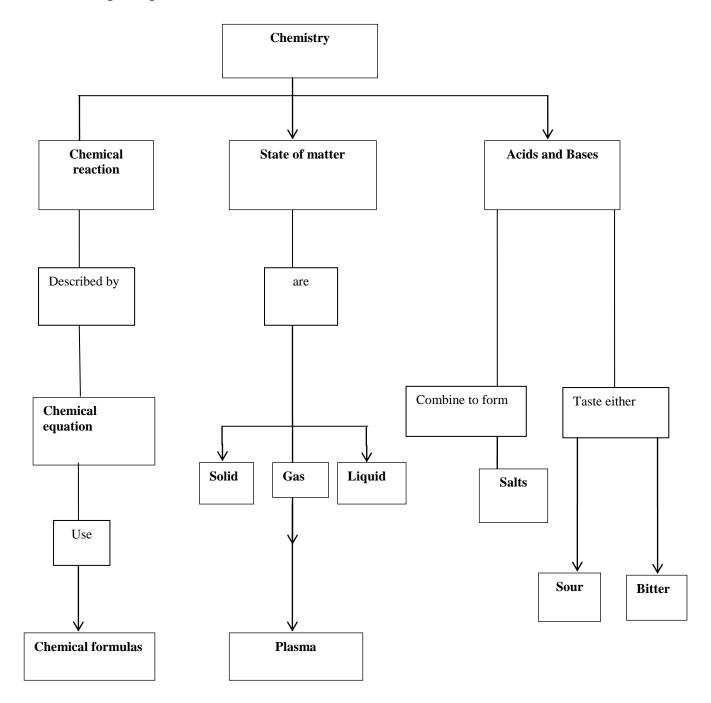


Fig. 2: Concept map (Source: Prentice Hall. Chemistry connections to charged atmosphere)

#### **1.0 INTRODUCTION**

Scientist today pursues knowledge with such specialization that an analyst, a physicist, a medical scientist and a psychologist are likely to find each other's work incomprehensible. But all scientists have a common foundation which may be understood by any of them and which is not difficult for a non-scientist to grasp. That foundation is not any particular body of factual knowledge but a way of thinking and acting which together form scientific method. Science, as a matter of fact, is what scientist does (whether in the field, classroom or laboratory). Scientific method is the study of how they do it and is properly a branch of psychology, particularly important to all practicing scientist (e.g. the analyst) and to anyone who is interested in scientists and science. We are well aware of scientific advances which are bringing spectacular changes to life. Biologist and engineers as well as technologist make the land provide more food, and doctors keep more people alive until old age than ever before. Now we can travel further and faster than our forefathers, fathers etc, and even speak instantaneously from one side of the world to the other (what a global village).

Materials unheard of before the industrial revolution are now woven into the texture of our life, and human labour has become a desolate source of energy (due to computer/ robotic age advancement). The earth is no longer the limit of human existence, and journey into space is no longer a fairy tale, it has advanced tremendously. Mr. Vice Chancellor sir, none of these achievements would be possible without an extraordinary increase in control of matter and energy. New technologies and drugs have been discovered only because enough basic knowledge (phyto-chemistry) has accumulated to solve all the problems involved in their discovery.

Mr. Vice Chancellor Sir, the title of the inaugural lecture may be a food for thought and an exhibition of curiosity of character of an experimenter or an analyst, the choice of the "eagle eye".

#### 1.1 The Eagle

The eagle (noun) 1. A large bird of prey (= bird that kills other creatures for food) with a sharp curved beak and very **good sight**: eagles soaring overhead. 2. (in golf ) a score of two strokes less than the standard score for a hole (Oxford Advanced Learner's dictionary International Student's Edition, 7<sup>th</sup> Edition ).

#### 1.1.1 Eagle 'eye' (noun)

If somebody has an eagle eye, they watch things carefully and good at noticing things (keen observer). The eagle eye is among the strongest in the animal kingdom, with an eye sight estimated at four to eight (4-8) times stronger than the average human (http://en.wikipedia.org>wiki>Ea...) An eagle is said to be able to spot a rabbit 3.2 km (2miles) away (http://en.wikipedia.org>wiki>Ea...). The question one would ask is what does the eagle represent? - The eagle with its keen eyes symbolizes courage, strength, and immortality, but is also considered king of the skies and messenger of the highest Gods (https://wwweagles.org>educate>g..). Another question comes handy, why are eagles so special? They flock together, they do not mix with other birds only enjoy flying at their high altitude. It is this characteristic that makes eagles unique birds. An eagle will never surrender to the size or strength of its prey. It will always give a fight to win its prey or regain its territory (<u>https://pgcpsmess.wordpress.com></u>). Going through the attributes required from the experimenter or analyst and drawing an analogy the question is ask are eagle smart? - golden eagles are cunning, intelligent, and bold, diving in front of the sun to blind their prey and using stealth-like tactics to cut off means of escape – they are **fierce** and **strong hunters** (https://wwweagles.org>golden-eag.. ) This means, therefore, that the experimenter or analyst to have an eagle eye must

have the ability to see or observe keenly; watch closely what is assigned to him /her and must not pre-empt judgment or being emotional when given a job that concerns both humans and the environment.

Mr. Vice Chancellor Sir, the journey of this lecture will take us to the interesting aspect of understanding nature and its activities either anthropogenic or natural which are sampling references based. Questions are bound to be ask and solutions proffered for a better Nigeria free of poverty; rancor but full of respect for human value; recognition of merit through commitment, hard work, functional education according to zeal, skills and create job satisfaction which will reduce borrowing with the natural resources we are endowed with.

### **1.2 Background Facts**

The first and foremost question we should ask ourselves – what is Nature? From the Oxford dictionary definition:

- The phenomena of the physical world collectively, including plants, animals the landscape, and other features and products of the earth, as opposed to humans or human creation. Synonyms: the natural world, the living world, mother nature, creation, the world, the environment, the earth, mother earth, the universe, the cosmos, natural, forces, etc.
- The basic or inherent features, character or qualities of something "helping them to realize the nature and their problems". Synonyms: essence, inherent/basic/essential characteristics; qualities; attributes; features, sum and substances, character, identity, complexion etc.

Nature, in the broadest sense (Wikipedia) is defined as the natural, physical or material world or universe. *"Nature"* can also be referred to the phenomena of the physical world, and also life in general. The study of nature is large, if not the only part of science (Wikipedia)

The other question that comes to the mind of the analyst or experiment is what is important about nature? – *Nature* is very important for humans in particular to survive and strive as provided by the natural world around us: food, water, medicine, material for shelter and even natural cycles.

The word nature is derived from the Latin word **'natura'** or "essential qualities, innate disposition", and in ancient times literally meant 'birth' (Haper, 2006)

Natura is a latin translation of the Greek word '**physis**' which originally related to the intrinsic characteristics of plants, animals and other features of the world, develop of their own accord (<u>https://wwwencyclopedia.com>no...</u>) The concept of nature as a whole, the physical *universe*, is one of several expansions of the original notion; it began with certain core application of the word *physis* by pre-Socratic philosophers, and steadily gained currency ever since. This usage continued during the advent of modern scientific method in the last several centuries (Naddaf, 2006, Archived March 5, 2011) at the Wayback Machine.; Harper, Retrieved September 20, 2006.) With the various uses of the word today, "nature" often refers to geology and wildlife. Nature can refer to the general realm of living plants and animals and in some cases the processes associated with inanimate objects – the way that particular types of things exist and change of their own accord, such as weather and geology of the Earth. It is often taken to mean the "natural environment" or wilderness. Wild animals, rocks, forest and in general those things that have not been substantially altered by human (*pristine*) intervention or which persist despite human intervention (anthropogenic) activities. For example, "human nature" or "the whole of nature". This more traditional concept of natural things which can still be found today implies a destruction between the natural and the artificial, with the artificial being understood as that which has been brought into being by human consciousness or a human mind.

Depending on the particular context, the term "natural" might also be distinguished from the unnatural or the supernatural.

The next information on the lecture Mr. Vice Chancellor sir, will be knowing and understanding our constituency the Earth. The Earth is the only **planet** known to support life, and its natural features are subject to many fields of scientific research. Within the **solar system**, it is the third closest to the sun; it is the largest **terrestrial planet** and the fifth largest overall. The most prominent climatic features are its two large polar regions, two relatively narrow temperate zones and a wide equatorial tropical to subtropical region ("World Climates." Blue- Planet Biomes. Archived from the original on December 17, 2008). With these features, precipitation varies widely with the location, from several meters of water per year to less than a millimeter. 71% of the Earth's surface is covered by salt-water oceans. The remainder consists of continents, and islands, with most of the inhabited land in the Northern Hemisphere, showing a lot of sample sites to explore.

From the features highlighted so far, its shows that the Earth has evolved through geological and biological processes that have left traces of the original conditions. The outer-surface is divided into several gradually migrating tectonic plates. The interior remains active, with a thick layer of plastic mantle and an iron-filed core which is composed of a solid inner phase, and a fluid outer phase. Convective motion in the core generates electric currents through dynamo-action, and these, in turn, generate the geomagnetic field.

The atmospheric conditions have been significantly altered from the original conditions by the presence of life-forms (Science Daily, 2005), which create an ecological balance that stabilizes the surface conditions. Despite the wide regional variations of a degree or two of average global temperature, have historically had

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major effects on the ecological balance, and on the actual geography of the Earth (Anderson & Walter, 1997; Spencer, 2011).

#### **1.3 Historical Perspective**

The Earth is estimated to have formed 4.5billion years ago from the **solar nebula** along with the sun and other planet (Brent, 1991). The moon formed roughly 20 million years later. Initially molten, the outer layer of the Earth cooled, resulting in the solid crust. Out gassing and volcanic activity produced the primordial atmosphere. Condensing water vapor, most or all of which came from ice delivered by comets, produced oceans and other water source (Morbidelli et al., 2000). The highly energetic chemistry is believed to have produced self-replicating molecules around 4 billion years ago (NASA Astrobiology Institute, 2001).

#### 1.3.1 Atmosphere, Climate and Weather

The Earth's atmosphere is a key factor in the sustainability of the ecosystem. The thin layer of gases that envelops the Earth is held in place by gravity. Air is mostly nitrogen, oxygen, water vapor, with much smaller amounts of carbon dioxide, argon, etc. The atmospheric pressure declines steadily with altitude. The ozone layer plays an important role in depleting the amount of ultra violet (UV) radiation that reaches the surface. As DNA is readily damaged by UV-light, this serves to protect life at the surface. The atmosphere also retains heat during the night, thereby reducing the daily temperature extremes. Terrestrial weather for instance occurs almost exclusively in the lower part of the atmosphere, and serves as a convective system for redistributing heat (Miller & Spoolman, 2007).

Ocean currents are another important factor in determining climate, particularly the major under water thermo-haline circulation which distributes heat energy from the equatorial oceans to the polar region. These currents help to moderate the differences in temperature between winter and summer in the temperate zones.

Also, without the redistribution of heat energy by the ocean currents and atmosphere, the tropics would be much hotter and polar regions much colder. Weather always has both beneficial and harmful effects in any environment. Extremes in weather, such as tornadoes or hurricanes and cyclones, can expend large amounts of energy along pathways, and produce severe devastation. Surface vegetation has evolved a dependence on the seasonal variation of the weather, and sudden changes lasting only a few years can have a dramatic effect, both on the vegetation and on the animals which depend on its growth for their food. Climate is a measure of the long-term trends in the weather. Various factors are known to influence the climate, including ocean currents, surface albedo, greenhouse gases, and variations in the solar luminosity, and changes to the Earth's orbit.

Based on historical records, the Earth is known to have undergone drastic climate changes in the past, including ice ages. The climate of a region depends on a number of factors, especially latitude. A latitudinal band of the surface with similar climatic attributes forms a climate region. There are a number of such regions, ranging from the tropical climate at the equator to the polar climate in the northern and southern extremes. Weather is also influence by the seasons which results from the Earth's axis being titled relative to its orbital plane. Thus, at any given time during summer or winter, one part of the Earth is more directly exposed to the rays of the sun. This exposure alternates as the Earth revolves in its orbits. At any given time, regardless of season, the northern and southern hemispheres experience opposite seasons. It should be noted that weather is a chaotic system that is readily modified by small changes to the environment, so accurate weather forecasting is limited to only few days (Harvey& Davidson, 2015) overall, two things are happening worldwide: 1. Temperature is increasing on the average 2. Regional climates have been undergoing noticeable changes (Science Daily, 2006).

- Water is one of the natural samples that are found in almost all part of the Earth. Water is a chemical substance that is composed of hydrogen and oxygen and is vital for all known form of life ("Water for Life", Un.org. 2005. Retrieved, May 14, 2011) in typical usage, water refers only to its liquid form or state, but the substance also has a solid state, ice and a gaseous state, water vapor or steam. Water covers 71% of the Earth's surface (World CIA- World Fact Book Retrieved, 2008).
- Ecosystems these are composed of a variety of *biotic* and *abiotic* components that function in an interrelated way (Michael, 2006). The structure and composition is usually determined by various environmental factors that are interrelated. Variations of these factors will initiate dynamic modifications to the ecosystems. Some of the more important components are: soil, atmosphere, radiation from the sun, water, and living organisms. Central to the ecosystem concept is the idea that living organisms interact with every element in their *local environment*. Within the ecosystem, species are connected and dependent on one another in the *food chain*, and exchange *energy* and *matter* between themselves as well their environment (Michael, 2006). The human /nature dichotomy and idea all the species are ecologically dependent on each other, as well as with the abiotic constituents of their biotope (Alam, 2011). A smaller unit of size is called a micro eco system, e.g. a micro ecosystem can be a stone and all the life under it, and a micro ecosystem might involve a whole *eco region*, with its drainage basin (Bailey, 2004). This situation typifies the Analyst strength of keen observation during sampling.
- Wilderness this is generally defined as areas that have not been significantly modified by human activity. Wilderness areas can be found in preserves, estates, farms, conservation preserves, ranches, national forest,

and national parks and even in urban areas along rivers, gulches, or otherwise underdeveloped areas. The analyst in such situation must develop an experimental and sampling technique for reliable results and easy interpretation of findings. However, wilderness areas and protected parks are usually considered important for the survival of certain *species*, ecological studies, *conservation* and solitude. Some nature writers believe wilderness areas are vital for the human spirit and creativity (Botkin, 2000) and some ecologist consider wilderness areas to be an integral part of Earth's self-sustaining natural ecosystem (the biosphere). They may also want to preserve historic genetic trait and provide habitat for wild flora and fauna that may be difficult or impossible to recreate in zoos or laboratories.

Human Interrelationship – although humans comprise only a *minuscule* proportion of the living biomass on Earth. Mr. Vice Chancellor Sir, the human effect on nature is disproportionately large. Because of the extent of influence, the boundaries between what humans regard as nature and 'made environment' is not clear cut except at the extremes. Even at the extremes, the amount of natural environment that is free of discernible human influence is diminishing at an increasing rapid pace. The development of technology by the human race has allowed the greater exploitation of natural resources and has helped to alleviate some of the risk from natural hazards. In spite of this progress, however, the fate of human *civilization* remains closely linked to changes in the environment. There exist a highly complex feedback loop between the use of advanced technology and changes to the environment that are only slowly becoming understood (Science Daily, 2006). Man-made threats to the Earth's natural environment such as pollution, deforestation, erosion, floods, land/ mud slides and disasters such as oil spills have given the analyst a greater food for thought on

*experimental designs, monitoring, and assessing, remediating and evaluating findings* due to the complexity of the environment. Humans however, have contributed immensely to the extinction of many plants and animals due to subsistent level of living in Nigeria in particular and Africa in general, poverty, and other anthropogenic activities. Humans also employ nature for both leisure and economic activities. The acquisition of natural resources for industrial uses remains a sizeable component of the world economic system (World Development Indicators (WDI), 2014). Some other activities such as poaching, hunting and fishing are used for both sustenance and leisure, often by different people and community.

Matter and Energy – some field of science Mr. Vice Chancellor sir, see nature as matter in motion, obeying certain laws of nature which science seeks to understand. For this reason the most fundamental science is generally understood to be 'physics' - the name for which is still recognizable as meaning that it is the study of nature. Matter is commonly defined as the substance of which physical objects are composed. It constitutes the observable universe. The visible components of the universe are now believed to compose only 4.9% of the total mass. The remainder it might interest us to know consist of 26.8% cold dark matter and 68.3% dark energy (Ade et al., 2013). The exact arrangement of these components is still unknown and under intensive investigation by physicists. The behavior of matter and energy throughout the observable universe appears to follow well-defined physical laws. These laws have been employed to cosmological models (space probes and satellite devices) that successfully explain the structure and the evolution of the universe we can observe.

The mathematical expressions of the laws of physics employ a set of twenty physical constants (Taylor, 1971) that appear to be static across the observable

universe (Varshalovich et al., 2000). However, the values of these constants have been carefully measured, but reason for specific values remains a mystery.

**1.3.2 The Nature of Chemistry** – Mr. Vice Chancellor Sir, what in the world is not chemistry? You may be thinking, what in the world does an ordinary candle have to do with the nature of chemistry? Surprise for you, one of the great English Chemist and Physicist Michael Faraday once wrote "There is no more open door by which you can enter into the study of natural philosophy (science), than by considering the physical phenomena of a *candle*". In 1860, Faraday devoted his annual lecture series of Christmas lectures to "The chemical History of a candle." As Faraday knew, that careful observations of even the simplest phenomena can tell you a lot about the world around you ('Eagle eye'). An introduction to the world of chemistry is but a prelude.

- Chemistry and you the first thing that always occurs to us is to think about how we have changed since we were born. Most especially the last few years, we may have noticed may be fantastic changes taking place in our body. The question we would normally ask ourselves is how did all these changes happen? (Note that: reaction is taking place since the body is made from the dust of the Earth (*Genesis 2: 7*), shows we are composed of elements *in vitro, in vivo* etc). These changes can fully be understood, when you look carefully at the substances that make up the body (it is assumed that the body of man is made up of *44 elements*). You would have to follow a particle of food as it is digested and then changed to living tissues; learn how light and sound waves changes to chemical signal that one's brain interprets as memories. In other words you would have to study chemistry (the foundation of sciences).
- Mr. Vice Chancellor Sir, we should ask: **what is chemistry**? chemistry can be said to be the study of all substances and the changes that they can

undergo ( both physically, spiritually; conversion of water into wine *John 2: 8-9*); medicine that cures diseases; fibre that goes into your clothing; even the complex substances that make up one's body – all are chemicals.

- Why the study of chemistry we have so far seen the role of chemistry in many areas of life, but there is another reason why we should learn chemistry. Chemistry is fun! Chemistry helps us to understand nature; what makes leaf change colour in the dry season; why ice bergs float, and how the food we eat turns into muscle ( sometimes fat ) in the body. Chemistry helps us also look at the world in ways you may never have imagined; its task is much easier if we concentrate on understanding concept than memorizing facts. (ii) Remembering that chemistry is first and foremost a process of discovery. Think of it as a journey into the unknown. Like all sciences, chemistry begins with curiosity, it also proves that something exist (elements). Questions will always prick our minds as to why does a match stick burst into flame when it is scraped against a rough surface? Why can a stain on your dress be removed by laundry bleach? And so on. As we all know, even ordinary substances can undergo dramatic changes.
- Chemistry in Action Chemistry Mr. Vice Chancellor Sir is a broad science that touches nearly all aspect of human life; it plays a great role in these professions and in many others as well. Chemistry has been called the *"central science"* because it over laps so many sciences. Whether your career interest are in science, engineering, public service, or some other occupation, chances are good that you will need the knowledge of chemistry. A hair stylist, e.g. need to know some chemistry so as to handle hair relaxers and permanent wave solutions safely and

correctly; construction engineers use chemistry when they select the right cement for a particular job (frequent collapse of building and road in the country); Biologist learn chemistry so that they understand the chemical processes that go on in living things; can you think of any other occupations in which the knowledge of chemistry might not be helpful?. I will encourage my audience, if they have a strong interest in chemistry, to venture into it for both the short and long term process which not only be a source of poverty alleviation, both small scale industries and quality products of our endowed natural resources to boost employment and less dependent on oil and white collar jobs (*look and learn philosophy*).

The process of accumulating (and simplifying) knowledge Mr. Vice Chancellor sir, can conveniently be called "Science", that blend of speculative thought and practical experiment which has preceded and accompanied all these familiar material development we observe. Science in this sense has been developing over 300years. Early science in the 17<sup>th</sup> and 18<sup>th</sup> centuries was mainly concerned with physics and chemistry, then the study of plants and animals did not go beyond observation and classification, and biology did not become experimental science to a greater extent before the middle of 19<sup>th</sup> century. Most of the triumphs of applied sciences were both for techniques and for understanding; the objective of human knowledge is now indispensible, the greater the physical power at any man's disposal, the more dangerous is its misuse, thus making the environment more stressful, and less convenient for peaceful coexistence. The survival of man depends not only on finding food and escaping disease but avoiding the hazards of nuclear weapons and other lethal devices culminating into (e.g. environmental pollution, contaminants, toxic heavy metal accumulation, pesticides/residue bioaccumulation etc.)

Mr. Vice Chancellor Sir, we notice that knowledge of human reaction and of circumstances which divert activity into beneficial or destructive course is urgently needed and the need adds to the reasons which promote the study of man himself. A distinction is sometimes made between *'hard'* and *'soft'* science, hard science in a sense being understood to be done in the laboratory with proper apparatus (referred lightly to as *'hard ware'*) and to provide hard facts as a result of hard reasoning. Soft science being referred to social studies and investigation of human behavior generally; the underlying methods necessary for any scientific research are the same, and invariably require development, alternative of experiment and ideas. We must know that it is more difficult to devise good experiment in the field than in the laboratory, and the chances of doing bad work are correspondingly greater.

Accordingly, scientific method is considered here in relation, to both hard and soft science; the subject treated in the course of this lecture informally, from the position of an analyst. We all know that scientists make discoveries in many ways, according to the subject they are studying, the means at their disposal and their individual temperaments; therefore, scientific method which is the theme of the eagle eye of nature from the analyst perspective will give a simplify account as much as possible of what goes on, or what might go on, in the process of making discoveries as the journey progresses. A description of scientific method is related to original research as grammar is related to everyday language, or even to poetry.

- The first step in scientific procedure is to *observe*, (the very concept and attribute of the *'eagle eye'* whatever thing or event that is being studied.
- The second step is to *think* about the *observations* and *notice* what *appears* to go together and what do not. One then makes more observations, choosing a situation in which conditions can be regulated *precisely* and *planned* experiments carried out. In most situations

deliberate experiments are also better than events not arranged by the scientist because it is easier to show casual connections; as well as giving evidence about causes, good experiments give qualitative information; it is always better whenever possible to *count* or *measure* what happens.

- A well designed experiment adds to knowledge, but either leaves some questions unanswered or suggest new questions (this will be the trend of this lecture). However, after an experiment or a series of experiments have been carried out, more thinking is always necessary.
- Sometimes thought can continue on the same line as before, but at times more facts are difficult to fit together and reconcile with existing ideas. At a point, a good scientist is ready to abandon much of his/her existing frame work of knowledge and look for a new and preferably simpler ways of treating all the available facts and predicting new ones correctly. The great leap in science have all involved replacing one set of *ideas* by another for example, the cosmology of Copernicus, Darwin's theory of evolution, Einstein's theory of relativity etc. Scientific thought is sharply distinct in respect from any authoritarian system of belief. Existing scientific ideas are liable to be replaced by a more general scheme of things, just as the solid atoms of 19<sup>th</sup> century Physics have given place to the system of protons and electrons and other sub-atomic particles which at present give the most workable account of the properties of matter.

#### 2.0 SCIENTIFIC METHOD

It involves an alternation between *thought* and *experiment*. The scientist usually forms definite *ideas* or *hypothesis* in the light of available knowledge, devices (apparatus, equipment and instruments) and performs experiments to test the

hypothesis. More knowledge is generated and the cycle continues indefinitely, never reaching absolute certainty but always approaching more generality, and giving an increased control of the environment. One of the simplest and most widely applicable scientific hypotheses is that given, the same conditions, the same events will occur. This hypothesis is not usually stated explicitly; it is applied practically in that scientist generally try to *control* the conditions in which they work and expect *reproducible* (precise) results, in well -controlled conditions (from classical to instrumental analysis).

- A laboratory and much laboratory apparatus exist for the purpose of controlling physical conditions, such as temperature, pressure or for the purpose of *purifying* materials, so that their composition is known exactly and unknown contaminants are excluded from interfering. With appropriate control of conditions, the experiments are found to be reproducible and there in fulfill a fundamental criterion of scientific work.
- The criterion of reproducibility (*precision*) is easier to apply in physical than in biological experiments, and most difficult in behavioral studies (psychology). This partly explains why science is easier in a laboratory than outside it, but it does not follow that scientific method can be pursued only in the laboratory. Good hypotheses, therefore, account only for the *occurrence* but also for the *magnitude* and *direction* of the *change*, and it is often impossible to *distinguish* conflicting hypotheses in *practice* except by *precision measurement*. The necessary observations must therefore, be quantitative, and sometimes must achieve very great precision. The degree of precision, however, needed will depend on the point at issue, and there is sometimes little virtue in greater precision than a particular situation may require, but some measurement

is nearly always *essential*. The statement of a hypothesis requires the use of *words* or *mathematical expression* or *symbols*; however, quantitative relations are generally put in mathematical terms because these terms are *simple* and *precise*. Again, mathematical terms are not familiar to everyone and give much difficulty when they are not understood. However, their precision is indispensible, and no branch of science can get very far without at least a trifling amount of algebra.

- Words differ from mathematical symbols in being blunder tools for the purpose of expressing ideas, but their use in scientific work still requires some restraints in logic and semantics. Most people in particular, take the use of words for granted and overlook quite important sources of confusion which arise from verbal misuse, many also on the other hand, reject all kinds of mathematics as freely as they are confident in their use of language (a case study in the discussion of results) renders the discussion most at times vague and pre mature casting much doubt on the observed values obtained in the course of experimentation. It must be recognized that scientific knowledge increases by two kinds of operation, the accumulation of single isolated facts and formation of general hypotheses, theories or laws which are consistent with large numbers of separate facts and from which further separate facts can be deduced.
- Mr. Vice Chancellor Sir, the basis of scientific method is the bringing together of *observation* and *hypothesis* or of facts and *ideas*. However, the process is usually cyclical, consisting alternatively of improving the ways in which observations are made and in revising the hypotheses. Observations are improved in two ways: by making them in deliberately designed circumstances i.e. as experiments, and by using apparatus or instrumental devices to produce special required circumstances.

Hypotheses, however, are improved by making them *simple*, *quantitative* and *general*; the process of successive refinement, both *experimentally* and *theoretically*, has no evident end. Scientific method is an empirical method of acquiring knowledge that has characterized the development of science from the 17<sup>th</sup> century; it involves careful observation, applying rigorous skepticism about what is observed, given that cognitive assumptions can distort how one interprets the observation (Wikipedia ).

• A diagrammatic illustration is given below on the activities expected by the analyst when carrying out a scientific method

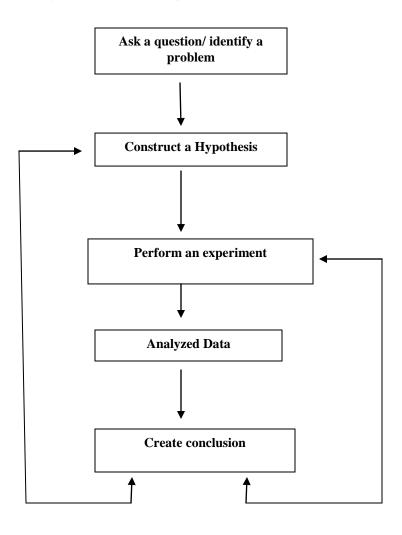


Fig.3: Scientific method

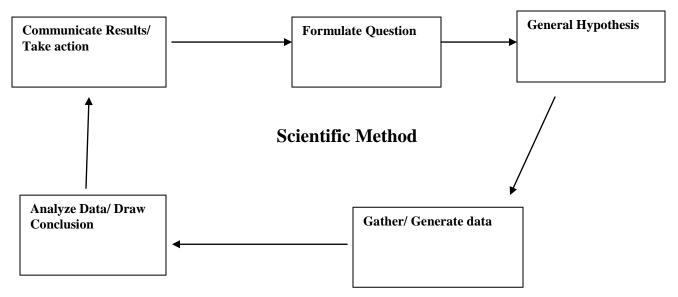


Fig.4: Steps to using data

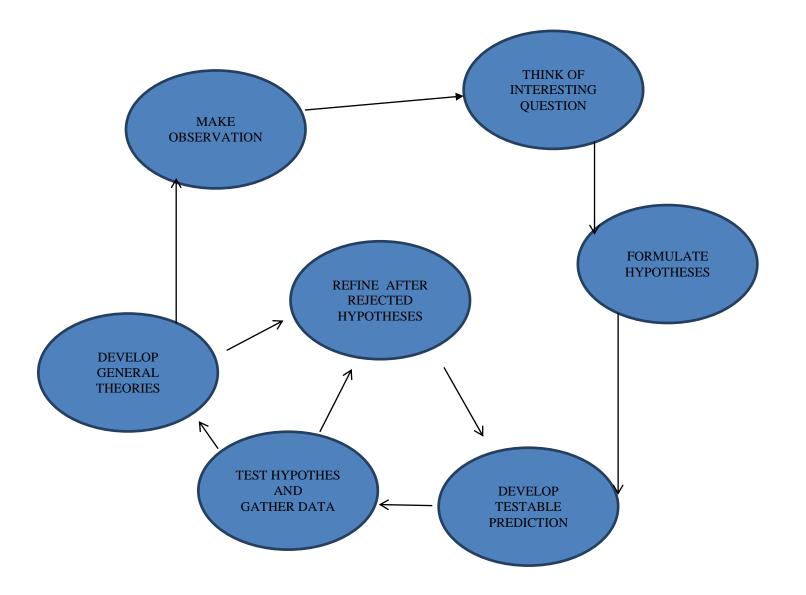


Fig. 5: The Scientific method as an on-going process

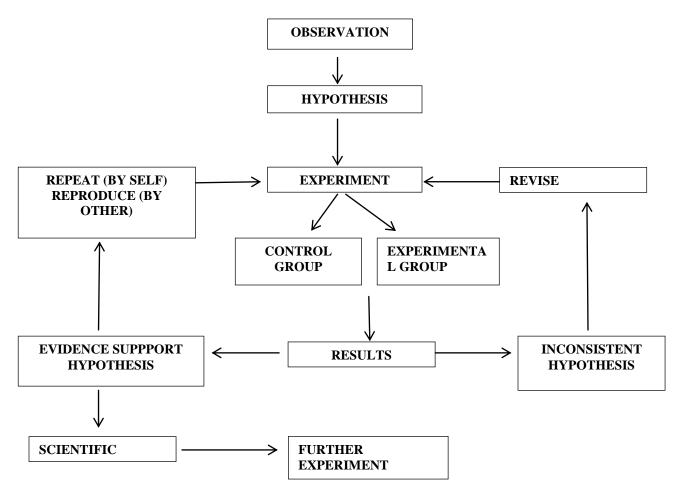


Fig. 6: The Scientific method

The tremendous potential of science comes from the way in which scientific knowledge is gathered (precisely and distinct for easy and continuous transmission to generation unborn). Scientists are known to follow an orderly and systematic approach (by first principle) called *scientific method*, *protocol*, *procedure* to gather knowledge. With such approach, new ideas about the world are constantly checked against reality. What is scientific method if I may ask? – is a way of answering questions about the world we live in.

- Steps of scientific method This consists of a series of steps that can be summarized as follows:-
  - 1. First a scientist makes an *observation* (*eagle eye*).
  - 2. The observation leads to question ( because science begins with curiosity, questions may come first )
  - 3. Thinking about the question produces a *hypothesis*, a tentative answer to the question.
  - 4. The scientist test the hypothesis with an experiment; as part of an experiment, the scientist *carefully* (no assumption, or pre-empting judgment) records and analyze the data obtained, or information on the event were necessary, gathered in the experiment.
  - 5. The experiment produces a *result*, or a *conclusion*, which the scientist *interprets carefully.* The result may raise new questions and lead to new hypotheses as well as new experiments.
  - 6. After, a number of experiments,, the scientist may be able to summarize the results in a natural law, which describes how nature behaves but does not explain, why nature behave in a particular way.
  - 7. Finally, the scientist may be able to formulate a *theory*. The theory explains why nature behaves in the way described by the natural law. It answers, not only the original question, but also any other questions that were raised during the process. The theory also *predicts* (*forecast*) the result of further experiments, which is how it is checked.
    - Mr. Vice Chancellor Sir, it would interest us to know that the choice of the lecture is to assist our youth who are aspiring to be good and committed scientist will be able to bail out this nation from induced poverty, joblessness and defeat because we have the platform with the endowments of both natural and human

resources and the mind to create wealth without dependency on oil. This lecture will not be complete without understanding this important parameter of nature; *observation*.

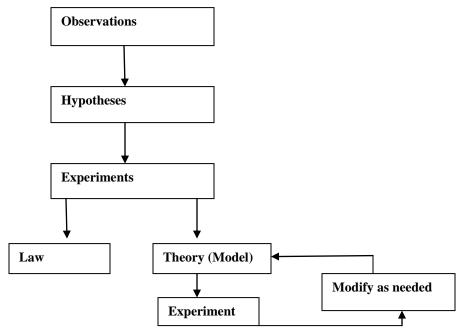


Fig. 7: The steps in the Scientific method

#### **2.1 Observation** (noun)

By definition 1. The action or process of clearly observing or monitoring something or someone. 2. A statement based on something one has seen, heard or noticed; it is also said to be, the active acquisition of information from a primary source in living beings, observation employs the senses. In science, observation can also involve the perception and recording of data via the use of scientific instrument (Wikipedia).

#### • Observation as the sample of the Universe

If we knew everything, we should not need to make discoveries; if we could find out about everything at once, problems of science as we know them would disappear; in fact we can never study everything at once, but only a sample of the universe. However, one basic scientific problem is to decide how far the tiny

sample that we may access represent the whole that we believe to exist. As this cannot be done by mere *observation*, it is necessary done by some sort of mental process, usually called *inference*. Ordinarily inferences are made unthinkably, and this requires some effort to analyze the steps involved when inferences are made. It is also much easy to make false inferences, such that some familiarity with the elements of logic is desirable in order to decide whether a particular reasoning is sound or not. In scientific research the observed facts, can be stated as particular propositions; particular propositions cannot prove general ones. The process of arguing from the particular to the general is called *inductive reasoning* or generalizing. It is invaluable as a stimulus to the imagination, but wholly invalid as a process of proof. All that can be done, however, is to disprove some propositions and say 'this anyway is not true' and to advance other general propositions or hypothesis and show that they are consistent with facts. Mr. Vice Chancellor Sir, having said this we should ask ourselves, what kind of propositions or hypotheses is suitable for scientific research? The most important property of a hypothesis is that it can be tested experimentally; it must have definite practical consequences, which can be deducted rigorously from it. If the hypothesis is true, then such and such a result will occur when the appropriate experiment is done. If this does not occur, then the hypothesis is not true. This is being emphasized, because this aspect of scientific method is almost totally omitted or neglected, thus affecting the quality of research. In this part of the world, setting forward a hypothesis, however, which is capable of being tested by experiment and which may become useful advancement in knowledge, some general considerations are important; most hypotheses usually involves one or more of the following:-

- Simple logical relationships
- Quantitative relationships
- Spatial relationships

- Generality
- Simplicity

These elements, however, overlap considerably.

### 2.2Sampling

This provides a means of gaining information about the population without the need to examine the population in its entirety; it also provides a valid, defensible methodology, but it is important to match the type of sample needed to the type of analysis required. The analyst should always take care to check the quality of information from which the sample is to be drawn; if the quality is poor, sampling may not be justified.

The question one would ask is why sample? It is often necessary to draw a sample of information from whole population to enable the detailed examination required to take place. Samples can also be drawn for several reasons: for example to draw inferences across the entire population; or to draw illustrative examples of certain types of behavior

### 2.2.1 Sample design

The aim of the design is to achieve a balance between the required precision and the available resources. Sample design covers the method of selection, the sample structure and plans for analyzing and interpreting the results. Sample design can also vary from simple to complex and depend on the type of information required and the way the sample is selected.

• *The design* will impact on the size of the sample and the way in which analysis is carried out. In simple terms the tighter the required precision and the more complex the design, the larger the sample size.

The design may make use of the characteristics of population, but it does not have to be proportionally representative e.g. it may be necessary to draw larger sample than would be expected from some parts of the population; to select e.g. more from a minority grouping to ensure that sufficient data for analysis for such groups is obtained. Many designs are usually built on random selection; this permits justifiable inference from the sample to the population, at quantified levels of precision. Given due regard to other aspects of design, random selection guards' bias in a way that selecting by judgment or convenience cannot.

The first step in good sample design is to ensure that the specification of the target population is as clear and complete as possible ensure that all elements within the population are represented

#### 2.2.2 Sample size

For any sample design deciding on the appropriate *sample size* will depend on *five factors*. It is important to consider these factors together to achieve the right balance and ensure that the objectives are met.

### Margin error or precision ; Variability in the population; Confidence level; Population size and Population proportion

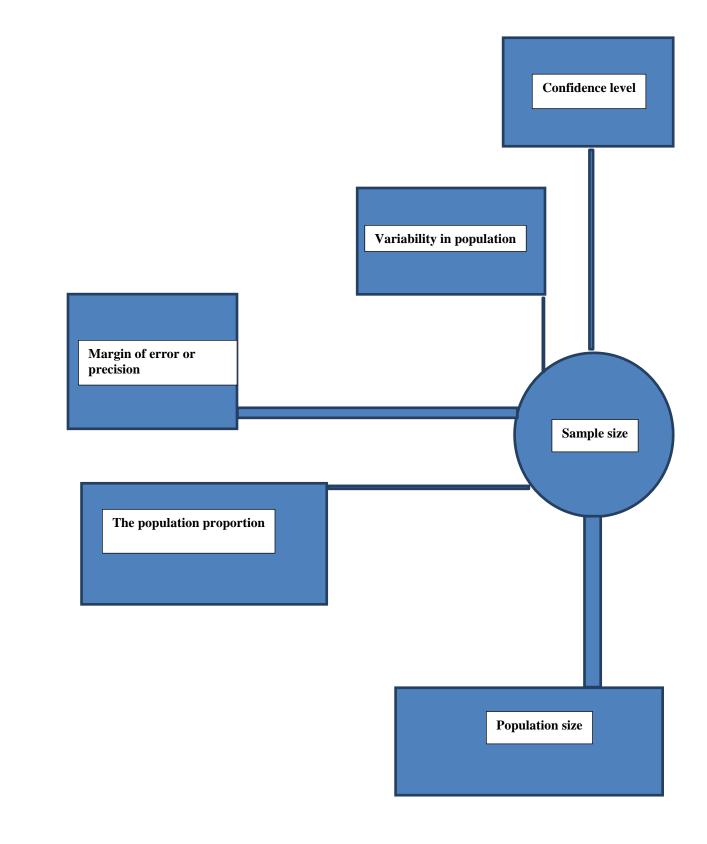


Fig. 8: Sample size

As a general rule, a statistical sample should contain 50 to 100 cases for each sample or sub-group to be analyzed.

#### 2.2.3 Weighted Sample

If a normal sample is insufficient to reflect the population characteristics then it may be necessary to look for ways in which this can be improved. One way of doing this is to weight the sample. If one is looking to sample three locations and they have varying workloads, one may want to the sample to reflect the workload at each location; while this does not reflect the population characteristics, it may still be perfectly valid if one is interested in the locations as well as work load, from the Table 1 below. In the last column the sample has been weighted to reflect the population characteristics. This approach would be more suitable if one is interested more in the actual cases than the locations. The method of calculating the results for a weighted sample are different than for the simple random sample.

**Table 1:** Example of a weighted sample

Location	Population	%	of	А	simple	The e	effect on	
	workload	population		random		sample size at		
		workload		sample	of the	each	location	
				total wo	orkload	when	the	
						sampl	e is	
						weigh	ted	
North	50,000	13%		128		51		
South East	250,000	67%		153		256		
South West	75,000	20%		103		77		
TOTAL	375,000	100%		384		384		

A weighted sample more accurately reflects the workloads at the regional locations.

2.2.4 Post- weighted Sample: - should this weighting be required, but had not taken place at the sample selection stage then it is possible to weight the sample in the results phase. This is done by applying the population proportions to the results of the un- weighted result of adjusted result. In this case of un weighted result of 37% becomes a weighted proportion of 49% as shown in the Table 2 below.

Location	Simple	Displaying	Unweighted	Location	Weighted
	Random	Required	Proportion	Percentage	Proportion
	Sample	Attribute			
	(A)	(B)	(B/A)	(C)	(B/A)×C
North	128	21	16%	13%	2%
South East	153	98	64%	67%	43%
South West	103	23	22%	20%	4%
TOTAL	384	142	37%	-	49%

A weighted sample more accurately reflects the workloads at the regional locations

**2.3Sampling Methods**: - Methods, their use and limitations. There are different ways in which a sample can be selected. Nine of the most common methods are illustrated below (see Table 3).

Method	Definition		Uses	Limitations
Cluster sampling	Units in	the	• Quicker,	• Larger
	population	can	easier and	sampling

	Often be found in	cheaper than	error than
		-	
	geographical	other forms	other forms
	groups or clusters	of random	of random
	e.g. Schools,	sampling.	sampling.
	households etc.	• Does not	• If clusters
		require	are not small
		complete	it can
		population	become
		information.	expensive.
		• Useful for	• A larger
		face-to-face	sample size
		interviews.	may be
		• Works best	needed to
		when each	compensate
		cluster can	for greater
		be regarded	sampling
		as a	error.
		microcosm	
		of the	
		population.	
Convenient	Using those who	• Readily	Sample
sampling	are willing to	available.	results
	volunteer, or cases	• The larger	cannot be
	which are	the group,	extrapolated
	presented as	the more	to give
	sample	information	population

		is gathered.	results.
			• May be
			prone to
			volunteer
			bias.
Judgment	Based on	Normal	• It is prone to
sampling	deliberate choice	application	bias
	and excludes any	is for small	• The sample
	random process.	samples	is small and
		from a	can lead to
		population	credibility
		that is well	problems.
		understood	• Sample
		and there is	results
		a clear	cannot be
		method for	extrapolated
		picking the	to give
		sample.	population
		• Is used to	results.
		provide	
		illustrative	
		examples or	
		case studies.	
Multi-stage	The sample is	• Usually the	• Complex
sampling	drawn in two or	most	calculation
	more stages (e.g. a	efficient and	of the

	selection of offices	practical	estimates
	at the first stage	way to carry	and
	and a selection of	out large	associated
	claimants at the	survey of	precision.
	second stage).	the public.	I
Probability	Sample are drawn	• Where you	• Can be
proportional to size	in proportion to	want each	expensive to
	their size giving a	element (e.g.	get the
	higher size of	claimants at	information
	selection to the	an office) to	to draw the
	larger items (e.g.		
	the more the	have a equal	sample
		chance of	• Only
		selection	appropriate
	office the higher	rather than	if you are
	the office's chance	each	interested in
	at selection).	sampling	only the
		unit (e.g.	element
		offices)	
Quota sampling	The aim is to a	• It is a quick	• Not random
	sample that is	way of	so strong,
	representative of	obtaining a	possibility
	the population	sample.	of bias.
	The population is	• It can be	• Good
	stratified by	fairly cheap.	knowledge
	important variables	• If there is no	of
	and the required	sample	populations

	quota is obtained	frame it	characteristi
	from each stratum.	could be the	c is
		only way	essential.
		forward.	• Estimates of
		• Addition	sample error
		information	and
		may	confidence
		improve the	limits
		credibility of	probably
		the results	can't be
			calculated
Simple random	Ensures every	Produces	• Need
sampling	member of the	defensible	complete
	population has	estimates of	and accurate
	equal chance of	the	population
	selection	population	listing
		and	• May not be
		sampling	practicable
		error.	if a country-
		• Simple	wide sample
		sample	would
		design and	involve lots
		interpretatio	of audit
		n.	visits.
Stratified sampling	The population is	• Ensures	• Selecting the
	sub-divided into	units from	sample is

	homogeneous	each main	more
	groups, for	group are	complex and
	example regions,	included and	requires
	size or type of	may	good
	establishment.	therefore be	population
	The strata can have		information.
		more	
	equal sizes or you	reliably	• The
	may wish a higher	representativ	estimates
	proportion in	e.	involve
	certain strata.	• Should	complex
		reduce the	calculation
		error due to	
		sampling.	
Systematic	After randomly	• Easier to	• Can be
sampling	selecting a starting	extract the	costly and
	point in the	sample than	time
	population	simple	consuming
	between 1 and n,	random.	if the sample
	every n <sup>th</sup> unit is	• Ensures	is not
	selected, where n	cases are	conveniently
	equals the	spread	located.
	population size	across the	• Can't be
	divided by the		used where
	sample size.	population	
	sample size.		there is
			periodicity
			in the

			population.
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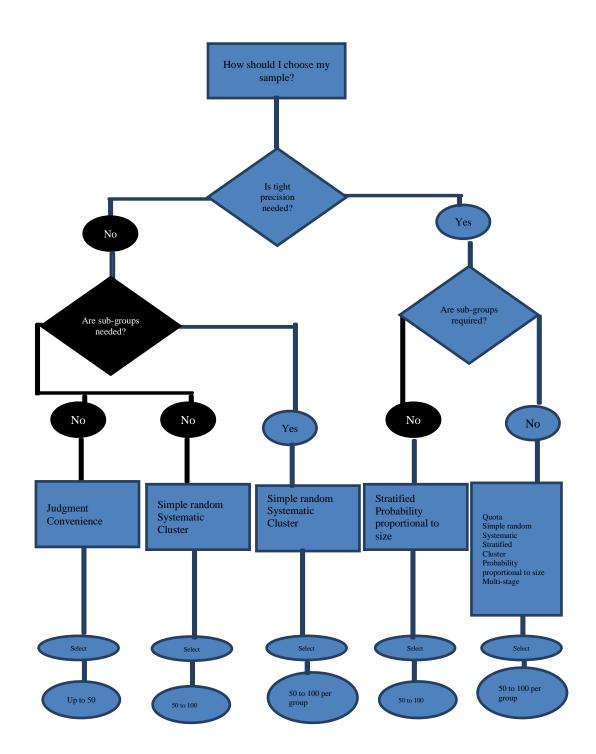


Fig.9: Selecting an appropriate method

- Selecting an appropriate method: as one can see there are many methods use with varying degrees of complexity. Certain methods suit circumstances better than others and the following diagram is designed to help one select an appropriate method.
- Extracting the sample: Simple random sampling it is possible to use Excel or SPSS, to select the sample. It is also possible to use other statistical menus to extract the sample. If the population is not held electronically then an interval sample from a random starting point could be used as an alternative. If one is not intending to use a simple random sample then one should sought advice from the professional expert on how to extract the sample and also to boost the quality of work and obtain a reliable result.

## **3.0 INTERPRETING AND REPORTING THE RESULTS**

The choice of sample design and how well it mimics the population will impact on the results obtained. The closer the sample designs to the population characteristics the more precise the estimate from the sample. It is therefore, important to match the calculation of the results from the sample to the design of the sample. To obtain the sample estimate for a simple random sample one can use a package such as Excel or SPSS and other suitable ones which will return not only the average but also the standard deviation, and the precision at 95% confidence level.

### **3.1Glossary of terms:**

• **Confidence level**: - The certainty with which the estimate lies within the margin of error.

- **Margin of error**: A measure of the difference between the estimate from the sample and the population value.
- **Population**: The number of items from which to draw your sample.

Population proportion:- The proportion of items within the population which exhibit the characteristics you are seeking to examine, this is only required when sampling for attributes.

- **Precision**: A measurement of the accuracy of the sample estimate compared to the population value.
- **Sample**: A selection of items from which you may estimate a feature of the population.
- **Sample size**: The number of items in the sample.
- **Standard deviation**: A measure of the variability in the population values, this is only required when sampling for values.

Appendix 1: Relevant formulae for simple random sampling

# **3.2 Randomization**

The basic element of a control observation is that it is like the treated observation in every respect that which constitutes the treatment. If the analyst is working with *stable* and *uniform* material, it is easy to divide the material into identical parts and treat one but not the other. But experimental material is often neither stable nor uniform, particularly when individuals are studied and not large numbers in which average effects obscure individual variation (samples are supposed to be representative enough for repeatability and reliability of results). However, answers to the questions of comparative sampling will be closely associated with the way results are analyzed. At the end of the experiment all samples will be measured, and from these measurements it will be possible to calculate two means (one for the control and one for the treated ) and one or more estimates of the variability between the samples ( within- and between- batches ). Appropriate statistical tests will now be used to decide whether the difference between the means is likely to have arisen simply because of the inherent variability of the material. If this cause is unlikely, the difference will be judged significantly, and the treatment can be supposed effective.

This inference, however, is justified only if the groups were initially comparable, in the sense that there was no significant difference between the mean sizes of control and treated groups at the start of the experiment (if a significant difference had existed before the treatment was applied, may not be due to the treatment). In order to restrict the difference in this way, all initial variation between the control and treated groups must be due to the same indeterminate factors as operate within the groups. It is also known Mr. Vice Chancellor Sir, that the measurement of variation within the groups will then be a valid measure of the variation or difference to be expected between groups before treatment (and also after treatment, if the treatment is ineffective). Comparison of this variation with the observed difference after treatment will give a satisfactory test of the significance of the observed effect. The essential point here, is that individual allocation of treatment must be unpredictable from any properties of the test material, nor by any preferences of the experimenter; such allocation is usually described as 'random'

• **Controls**: - Experiments involve comparison between treated and appropriate controls. Their purpose is to see what consequence follows the treatment; it is essential to know also what happens if the treatment is not applied (blank or pristine), so that even if treatment is not given; or are not wrongly attributed to the treatment. At first sight it is not difficult to arrange control observations, and if the system being studied is sufficiently stable it becomes true; it is then easy to see which controls ought to be included

when the facts are understood, while, the essence of research is working with facts which are not understood, and with situations where the crucial variable to be controlled is not always apparent. The question that rings in one's mind is how then, should an experiment be planned so that the results do not lead to false inferences; and also that time and labour are not wasted on confirming the obvious and displaying familiar negatives.

- Factorial design: It is a known fact that scientific experiments consist of observing what happens when a single factor is changed. This has been the argument adopted so far. Many experiments are indeed so arranged, but it is not necessary always desirable to be simple; in most circumstances, numerous factors are likely to affect the events being studied, and it may be extremely laborious to study each one separately.
- Interaction between factors: Is as important as the effects of the factors separately, and it is essential to design experiments so that interaction can be detected. A factorial design is often used by scientist wishing to understand the effects of two or more independent variables on a single dependent variable (<u>http://explorable.com.factorial-de</u>...). What are the key features of a factorial design?- according to Cozby (2009), a factorial design has more than one independent variable and key features which include: all levels of the independent variables; factorial research designs are a form of true experiment, where multiple factors ( the researcher-controlled independent variables ) are manipulated or allowed to vary, and they provide researchers two main advantages: can test limits; to test whether an independent variable affects different kinds of people or people in different situations, the same way ( https://quizlet.com.h-12-pys..350..). In statistics, a full factors, each with discrete possible values or levels across all such factors

(Wikipedia). When choosing an experimental design, on important consideration is which one delivers the most statistical power with the fewest subjects. If the research questions calls for direct comparison of individual experimental conditions, as is required when treatment packages are being compared, then this design will be an RCT – Randomized Controlled Trials. If the research questions call for assess the effects of individual components of an intervention, then, this design will usually be a factorial experiment (Collins et al., 1991).

Factorial design in Chemistry: - an experiment of such in chemistry requires the full factorial design; in this design, the treatments are all possible combinations of all levels of all factors. The simplest full factorial experiment consist of two factors, each with two levels( https://chemical statistician.wordpress.com...). An experimenter may seek to determine the causal relationships between G *factors* and the *response*, where G > 1. One first instinct, one may be tempted to conduct G separate experiments, each using the completely randomized design with 1 factor. Often, however, it is possible to conduct 1 experiment with G factors at the same time; this is better than the first approach because, it is *faster*; = it uses less *resources* to answer the same questions; = the interactions between the G factor can be examined; such an experiment requires also full factorial design; in this design, the treatment are all possible combinations of all levels of all factors. After controlling for con founding variables and choosing the appropriate range and numbers of levels of the factor, the different treatments are applied to the different groups, and data on the resulting responses are collected. The simplest full factorial experiment consists of two factors, each with two levels; such an experiment would result in  $2x^2 = 4$  treatments, each being a combination of 1 level from the first factor and 1 level from the

second factor. Since this is a full factorial design, experimental units are independently assigned to all treatments. One further advantage of factorial types of experiment is that each isolation of a component of the overall variation reduces the residual, unexplained variation which is the component on which all statistical test of significance depends.

The 2- factor ANOVA model is the most commonly used to analyze data from such designs, From Eric Cai – The Chemical Statistician (http://chemicalstatistician.com.wordpress.com>...).

**3.3 Reduction of Experimental Error**:- These are errors caused by the way in which the experiment was conducted; in other words, they are caused by the design of the system. Mr. Vice Chancellor Sir, this has been a source of worry to me since I ventured into the world of an experimental chemist; I found out that error (s) is either overlooked or taken for granted; this has become a night mare to quality research, presentation of findings and carrying out routine experimentation both in the field and the laboratory.

- One of experimental errors (systematic errors) cannot be eliminated by averaging in principle, they can always be eliminated by changing the way in which the experiment was done. To reduce the systematic error of a data set, one must identify the source of the error and remove it. Unless that is done, one will never reduce the systematic error by more measurements (https://www.nap.edu>oneuniverse.). The question now ask – how can one minimize errors in measurements? This takes us to the ways to reduce measurement error (Observational Error)
- ✓ Double check all measurement for accuracy
- $\checkmark$  Double check that your formulas use are correct
- $\checkmark$  Make sure observers and measurement takers are well trained

- ✓ Make the measurement with the instrument that has the highest precision
- ✓ Take the measurements under controlled conditions. Measurement error from these highlights is called *bias* and is also the difference between a *measured quantity* and *true value*; this includes:-*random error* ( naturally occurring errors that are not expected with any experiment ) and *systematic errors* (caused by a miscalibrated instrument that affects all measurements); having shown this one must know the different measures of error for a precise and reliable results which includes :-
- ✓ Absolute Error: The amount of error in one's measurement
- ✓ Greatest Possible Error: Defined as one half of the measuring unit
- ✓ Instrument Error: Error caused by an inaccurate instrument (like a scale that is off or a poorly worded questionnaire )
- ✓ Margin of Error: An amount above or below one's measurement
- Measurement Location Error: Caused by instrument being place somewhere it should not, like a thermometer left out in the full sun.
- ✓ Operator Error (personal): Human factors that cause error, like reading a scale incorrectly or colour blindness.
- Percent error: Another way of expressing measurement error :defined as

Percent Error = (measured value – actual value) / actual value

• **Relative Error**: The ratio of the absolute error to the accepted measurement, as shown

$$E_{relative} = \frac{E_{absolute}}{E_{measured}}$$

## **3.4 Statistical Procedures to Access Measurement Error:**

The following methods access "absolute reliability ":

- Standard Error of Measurement (SEM): Estimate how repeated measurements taken on the same instrument are estimated around true score.
- Coefficient of Variation (CV): A measure of the variability of a distribution of repeated scores or measurements. Smaller values indicate a smaller variation and therefore, values closer to the true score.
- Limits of Agreement (LOA): Gives an estimate of the interval where

   a proportion of the differences lie between measurements
   (https://wwwstatisticshowto.datasciencecentral.co
- Experimental Error and Uncertainty: Experimental errors, on the other hand, are inherent in the measurement process and cannot be eliminated simply by repeating the experiment no matter how carefully it is performed. There are two types of errors as stated earlier; systematic errors are errors that affect the accuracy of a measurement (www2.ece.rochester.edu>err... PDF )
- Measurement and Error Analysis : A former head of the Federal Reserve, US Central Bank – Alan Greenspan, once said I quote 'It is better to be roughly right than precisely wrong'' unquote. This takes us to the uncertainty of measurements: some numerical statements may be considered exact, however, all measurements have degree of uncertainty that may come from a variety of sources; the process of evaluating the uncertainty associated with a measurement is often called uncertainty analysis or error analysis. The complete statement, however, of a measured value should include an estimate of the level of

confidence associated with the value. Properly reporting an experimental result along with its uncertainty (standard deviation) allows others to make judgment about the quality of the experiment and it facilitates meaningful comparison with other similar values or a theoretical prediction. Without an uncertainty estimate, it is impossible to answer the basic scientific question, Does my result agree with a theoretical prediction or results from other experiments? This is a fundamental question for deciding if a scientific hypothesis is confirmed or refuted.

Precision is often reported quantitatively by using relative or fractional uncertainty:

**Relative Uncertainty** = [uncertainty/measured quantity]

E.g. If m=  $75.5 \pm 0.5$ g, has a fractional uncertainty of 0.5/75.5 =

0.66

- Accuracy is often reported quantitatively by relative error:
- Relative Error (%) = measured value expected value / expected value

If the expected value is: say m = 80.0g, then the relative

error is

$$75.5 - 80.0 / 80.0 = -4.5 / 80.0 = -0.056 = -0.6\%$$

(Note: The negative sign indicates that the measure value is less than the expected value). When analyzing experimental data, it is important that one understand the difference between precision and accuracy. Precision indicates the quality of the measurement without any guarantee that the measurement is *'current''* while, Accuracy, on the other hand, assumes that there is an ideal value, and tells how far one's answer is from that ideal *'right''* answer. These concepts are directly related to random and systematic measurement errors; measurement, therefore,

may be express as *measurement* = (*best estimate*  $\pm$  *standard uncertainty*) units measurement. For instance: where the  $\pm$  standard uncertainty indicates approximately a 68% confidence interval (Bevington & Robinson, 1991).

Another interesting aspect of this journey was the graphical presentation of results which highlights the important effects observed during the cause of field experiment or data treatment for forecasting.

# 3.5Graphs

These are pictures designed to express words, particularly the connection between two or more quantities. A simple graph usually shows the relationship between of numbers the form two or measurements in a grid. (https://simple.m.wikipedia.org>wiki ); in mathematics, graph theory is the study of graphs, which are mathematical structures used to model pair wise relations Graphs show visually the relation between measurements. between objects. Conventional graphs consist of two axes drawn at right angles to each other and a grid of lines representing the intersecting scale divisions on each axis. In preparing any graph it is always necessary to decide on the following:-

- Which measurement is to appear on each axis
- What range to display
- What origin to use
- What scale interval to apply
- Whether the quantities shown are continuous or discontinuous
- Whether theoretical relationships are to be displayed as well as actual observations. If algebraic symbols are used to represent the measurements, values of x are plotted on the horizontal axis or *abscissa* and values of y on the vertical axis or *ordinate*. The principal axes intersect at the origin, which is the point where x=0 and y=0, but it is not always necessary or desirable to include

the origin in a graph intended to display relationships between x and y if none of the values come near to zero. A graph containing both experimental points and a precise theoretical line is the keystone of research; as well as showing how nearly the theory or hypothesis fits the facts, it can show how far there are no facts in some regions to which the hypothesis can be extended. The choice of hypothesis can often be reduced to the choice of the theoretical relationship between x and y which best fits the plotted observations; the problem of goodness of fit are of great and wide spread importance and their solution involves many aspects. A graphical display which distinguishes clearly between facts and hypothesis is valuable as a statement of a particular problem of this kind. Arbitrary joining of experimental points by undefined lines obscures this distinction and on the whole should be avoided.

#### **4.0 INSTRUMENTATION**

Cost and ease of operation are also important state measurements can be made with direct reading instrument, but dynamic measurements need automatic recording, counting event involves different procedures from measuring continuous variables and this distinction has its counterpart in all computing systems, are either digital or analogical.

The processing of instrumental design, measuring, recording, calculating and controlling are all related to each other, and all are fundamental to scientific investigation. However, apparatus for research i.e. often very costly and the choice of apparatus needs to be considered in relation to alternative procedures and available resource.

Instruments are devices for clear observations. Direct observation: the events can be observed directly or the use of aided sense is very small fraction of the known range of physicals phenomena. Observation of any event involves not only the detection of stimuli by sense organs but also the observers brain of the information transmitted from the sense organs. This interpretation is affects by the observer previous experience so that a trained observer can detect what an experience notice would miss and also sometimes a trained observer overlook or dismisses irrelevant some change c would be conspicuous to the expert witness.

Similarly the execution of experiments is subject to physical limits of human performance. Practically all scientific work involves the use of instrument to overcome their limits. The physical properties of the instruments used contribute to what happens and is recorded in a experiment and these properties must therefore, be studied as part of any scientific investigation.

It is however, essential to recognize that the behaviors of any of suppliers are to substitute for experimental analysis of the performance of an instrument in the hand of its user

## **4.1 Basic Principles of Instrumentation**

The choice of apparatus for any particular experiment always involves answering certain questions

- 1. How accurately must the experiment be done?
- 2. What resources are available?
- 3. Are the quantities to be measured fixed or changing in the course of the experiment?
- 4. Are the results wanted as the experiment proceeds, or not until, later and are wanted in a form suitable for human inspection or for mechanical interpretation
- 5. How much disturbance of the experiment will the apparatus create?
- 6. How well the apparatus adapted to the operators is needs?

# **\*** Accuracy

It is a fundamental and expensive mistake to believe that experiments must always be done with the greatest attainable accuracy.

- The degree of accuracy required depends on the hypothesis being tested and the variability of all the phenomena involved.
- Alternatively, considerable error is unimportant in detecting a particular effect, particularly if the experimental conditions are selected intelligently.
- The labour and cost of making measurements increases steeply as increasing accuracy is required
- Most experiments involve more than one kind of measurements and the result depends on relationship between measurements.
- Another point in assessing accuracy lies in the range were variation is important. Measurements arte commonly comparative and the range over which they vary is small in comparison with the absolute value of the measurement
- It is important to choose apparatus with right range of sensitivity than with the greatest accuracy. The error of reading a pointer is equally greater with a small or a large deflection, and so the proportional error becomes less as full-scale deflection is approached.

# \* Resources

The apparatus, people and money available for any research is always limited. Good research involves getting the most out of the available resources. Apparatus of great accuracy is generally slow in use and requires special skill to handle it and keep it in working order.

If the accuracy is not needed, simpler apparatus will give that is needed quickly and leave more for other work.

### **\*** Static and Dynamic Measurement

Different techniques of measurement are needed when object or event to be measured is static and when it is changing during experiment.

Static measurements are usually made with apparatus which includes e.g. a scale and pointer and the result is obtained by visual observation when the pointer comes to rest ( non-electronic balances are used).

### \* Availability of Results

Results can be presented many ways. Some of which are open to immediate inspection and some only after a delay. Direct reading instruments (e.g. UV-VIS spectrophotometers) involve no delay but give permanent record and are unsuitable for dynamic measurements

Recording instruments are usually more expensive and capable of introducing additional sources of error if the design is inappropriate for the kind of record required: but for many dynamic measurements they are indispensable.

## ✤ Effect of Instruments on the System Studied

It is difficult in any circumstances to study a system without disturbing it. Measurement generally draws some energy from the object measured. Apparatus is selected or designed for particular purposes so that such effects are avoided by using null methods.

Many measurements physically possible cannot be made without interference incompatible with life and others are of limited value because the surrounding are disturbed.

## ✤ Helping the Observer

A last basic principle in instrumentation is that a good instrument minimizes difficulty for the operator or observer. If the operator is going to remain at an instrument, he will probably work better sitting down. If he going to continually to and fro between an instrument and other equipment, he will probably prefer to work standing.

The choice affects the design of the instrument because the best position for the controls is near the hands and the best position for scales or other display is in line of direct vision.

The size of knobs, levers, switches, the closeness or otherwise of placing and their particular pattern of their arrangement all affect the speed and reliability of the observer.

# \* Measuring Instruments

Measurement involves comparison of object being measured with a standa5rd size or scale or unit of measurement.

Accuracy of measurement depends on having a suitable reference standard which does not vary in the conditions in which it is used and for practical purposes divisions of the standard unit are desirable, so that objects can be measured to function or multiples of unit.

Automatic measurement and recording system which is fashion now are expensive and not often available and for most laboratory purposes more conventional methods are necessary.

In any practical experimental work, it is important to know how reliable are the measuring instruments in use and not to accept uncritically their graduation. The process of checking the accuracy of apparatus is called calibration. It may be done directly, by comparing the scale under test with a reliable standard.

Measuring time presents some interesting special problems. A standard of time cannot be preserved like a standard meter or kilogram but must depend on some physical process with recognizable recurrent events.

Measuring long periods of time retrospectively raises fundamental problems in historical sciences such has geology and some aspects of biology.

#### **\*** Recording Instruments

Dynamic measurement usually is the use of automatic recording apparatus.

The principal limitation of any recording system is the extent to which it is nonlinear, that is to the size of the charge being recorded. Especially in quantitative determinations much labour may be wasted through failure to appreciate the response characteristics of the recording system in use. It is not always easy to establish whether a recording system is strictly linear, unless the object being recorded can be replaced by an artificial system which behaves in a known way (internal standard) and so can be used to test the recording system (testing a spectrophotometer – that is to divide a light beam into separate components of different wavelengths). It is essential to have light sources of known wavelengths in order that the calibration of the instrument can be verified whenever it is used.

The other limitation is that elaborate recording apparatus like accurate measuring instruments

Is apt to be costly and the value of any proposed apparatus must be assessed in relation to the resources available and the results which can be obtained by other means.

### Calculating Machines and Computers

All the calculating devises fall into two categories according to whether they represent quantities by a physical dimension or by countable units.

Any physical process which can be made to take place with sufficient reproducibilit5y and in accord with known mathematical equations can be used on a basis for a computer (analog or digital).

One basic advantage of a digital computer is its capacity to perform arithmetic with extreme speed and accuracy. It is especially suitable for problems involving repetitive calculation; either as part of the required process (e.g. in making successive approximation) or because the same calculation is wanted repeatedly. It

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is also especially valuable for handling large size quantities of data, even when any requisite arithmetic is very simple.

The principal limitation is that programming is an exacting and laborious operation. In comparison, an analog computer can be programmed more quickly but the results obtained have a lower precision. For many purposes, the precision of an analog computer is sufficient and the simpler programming may make it preferable.

## 4.2 Separating and Analysis

It is easy to study a simple system than a complicated one. An early step in examining anything is to separate it into parts and to inspect each one in the hope that it will be simpler. Chemistry in particular, consists of separating mixtures of substance and isolating pure compounds or elements.

Separation is always achieved by taking advantage of the different physical properties of the components of a mixture: e.g. solubility in different solvents, volatility at different temperatures, differences in absorbability etc. As a rule, no separation process is completely efficient. Adaptation of procedures so that they are automatically repetitive greatly increases their efficiency. A separation can therefore, be achieved by adding adsorbent to a solution, shaking so that the adsorbent comes into contact with all the solution and filtering (extraction process).

#### \* Synthesizing

Analysis consists of taking something apart and identifying what is present. Synthesis is the opposite process and apart from its obvious practical creative uses, it is valuable confirmation of the results of chemical analysis.

If the parts identified by analysis can be reconstituted to make a substance indistinguishable from that initially analysed, then the analysis is complete and nothing has been missed. If reconstitution fails, the fault may be in the process of

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synthesis, or it may indicate that the analysis is incomplete and an essential component remains to be discovered.

The apparatus needed for experimental syntheses does not require particular description. Much of it depends on the specific process involved.

# **\*** Controlling Instruments

An essential part of many experiments is to hold constant all the variables which might affect the outcome, but which are not at the moment being investigated.

Control of temperature is particularly important because physical and chemical processes are usually accelerated by a rise in temperature.

Control of electrical potential is also fundamental. Even if the actual experiment is not concern with electrical events, the recording apparatus is likely to depend on electricity supplies and to vary in its response if the voltage fluctuates.

A control system therefore requires:

- A device for detecting or measuring the property to be controlled in the apparatus, e.g. temperature
- An element for comparing the measurement which the value is to be maintained
- A means of supplying the property into be controlled in the apparatus, e.g. a heating coil
- A regulator in the supply line

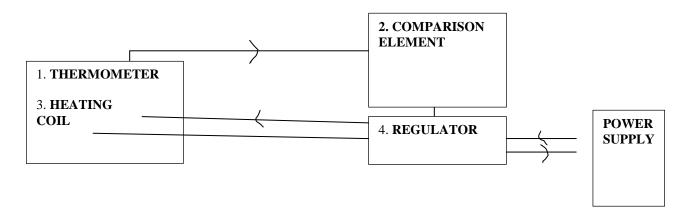


Fig.10: Components necessary for control of temperature in a hot water bath

These components are illustrated in the figure above, as applied to a constant temperature water bath. The principle involved is perfectly general. It is known as feedback or closed loop control because information about the state of the system is feedback to influence the system. No system however, can be controlled without information about its state at the time, and feedback mechanism operates not only in scientific and industrial apparatus but everywhere in physiological regulation and also in social control processes.

Control system does not operate instantaneously and are always imperfect because of time lags. The control cannot be better than the response of the detecting instrument permits and fluctuations occur at least over the range necessary to activate it.

Control systems have extensive applications in engineering and their behavior is intimately associated with the machinery they govern.

The simple system shown in figure above becomes expanded to a variety of inputs and outputs and the comparison element becomes an electronic computer, adjusting all the outputs according to pre-determined calculations and currently available inputs.

## **4.3 Interpreting Results**

A set of experimental results often consists of a number of measurements of a variable  $\mathbf{y}$  when another variable  $\mathbf{x}$  is altered. A mathematical relationship between  $\mathbf{x}$  and  $\mathbf{y}$  can be put forward, either on theoretical basis which the experiment is designed to test (a priori) or after looking at the results in order to discover a theoretical basis for them (posteriori).

More complex relationships, involving logarithms or some power of  $\mathbf{x}$ , can often be transformed to straight lines and it is generally, better to look for straight line relationships in transformed measurements than to fit curves directly.

Periodic processes can be described by trigonometric functions. Limitation and hazard, especially of interpolation and extrapolation are important.

# **4.3.1 Theoretical and Empirical Interpretation**

There are two ways of accounting for an observed set of points. One, the theoretical is to speculate on how the relationship might arise and deduce from this speculation or theory, what sort of mathematical expression to fit the facts.

The other way of accounting for a relationship, the empirical, is to look at the points and suggest: 'I think those points lie more or less on a straight line or those points look as though they lie on a curve which might have such and such an equation and to use this general relationship (once it has been established by proper curve fitting) to lead to ideas about how such a relationship might arise.

The terms theoretical and empirical, indicate that the equations are derived from theory and from experience or experiment respectively.

Agreement between experimental results and an equation with a definite theoretical basis is important as evidence supporting the theory or hypothesis. It does not prove the theory to be right because another theory might also lead to the same equation.

Familiarity with simple mathematical relationships between two variables is therefore, an essential part of scientific research.

# • Transformation

When the departures of experimentally determined points from a straight line are too large to be attributed to experimental variation, or when the deviations are systematic, some kind of curve must be considered. Fitting curves is less simple than fitting straight lines because any number of curves may be devised to go through a limit set of point. To avoid the difficulty, the process is called transformation and is often desirable.

If particular curvilinear relationships are suspected, values appropriately derived from the experimental results are plotted instead of the results themselves and if the suspected relationship holds, a straight line will be obtained.

In general, it is better to transform results and try to fit straight lines than to try and fit curves directly. However, it is first necessary to be from curve with some curvilinear relationships in order to know what transformation is appropriate.

#### • Statistics

When all the known sources of variation or error in a measurement have been controlled, some difference is still often found between repeated measurements made under the same conditions. This variation shows a characteristic bell shaped frequency distribution. A theoretical curve, the Normal or Gaussian distribution, results from supposing that a large number of small factors operate independently, some tending to increase and some to decrease the individual measurement. This theory provides a starting point for statistical analysis of variation and be used to measure variation (standard deviation and variance) and the accuracy of averages (standard error of the mean). When repeated samples are drawn from the same population, their averages are not always identical and test of significance are necessary to estimate the odds on a particular difference arising by the changes of sampling.

What changes in one variable depends on the magnitude of changes in another experimentally controllable, variable, the dependence is shown by fitting regression lines and when two variables appear to have some association the strength of the association is measured by means of a correlation coefficient. Both correlation and analysis of variance can be extended to apply to multivariate systems.

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# **\*** Factors Contributing to Personal Variation

It will be convenient here to note the main sources of personal variation in scientific experiments. These are:

- Limits of sensation and perception (genetically determined limitations e.g. colour-blindness)
- Limit of time of reaction (the variable reaction time makes human observation liable to errors of a magnitude which is serious when events last a few seconds)
- Misinterpretation (e.g. digit preference, misleading scales) due to expectations.
- Effect of repetition ('practice' and 'fatigue'). Experiment done more than once and some steps in an experiment may have to be repeated many times in the course
- Irrelevant stimulation (distraction). Visitors, telephones or casual conversation, lack of concentration
- Selective, pre-conceived observation.
- Memory and time lapses in recording
- Motivation: the direction in which personal errors occur is not random. Some are due to unsatisfactory apparatus or condition of use and be consistently in one direction. Others result from expectation of the observer and these errors more commonly favour the expectation than oppose it.

A scientific experiment involves human intervention at some stages, at least in setting up the apparatus and in reading the results. Human performance is variable, and experiments can be designed to isolate and measure the contribution of human factors to experimental variation. The opinion of the experimental about his reliability or accuracy is often misleading: also the good intentions of the experimental have no simple connection with the accuracy of his performance. Human observation is possible only over a limited range of physical conditions and with a finite time of reaction.

Observation is usually more variable at the extremes of ranges. Performance improves with practice but also becomes less reliable if continued over a long period, or if the performer is exposed to distracting stimuli. Time expectations of the observer have a considerable influence on his/her actions and perceptions. Readings made blind i.e. without information about previous values of the same reading and without knowledge of the experimental group (control or treated) to which the reading belong, avoid this bias. Memory is selective and record made of the time of observation are more likely to be accurate than records made later. Transcribing and editing of original notes introduces variation which depends on the bias of the editor. Such variation is open to experimental study just as is variation in making actual observations. Knowledge of previous results sometimes reduces or eliminates certain kinds of errors and sometimes contributes to persistence of other errors. Consequently trained and experienced observers are generally more accurate than untrained personnel, though also more to bias.

## Criteria for Selection of Method for Analysis

- Choice of method is usually influenced by several factors such as speed, convenience, accuracy, sensitivity, availability of instruments, amount of sample, level of analysis. Of these the last consideration is of paramount importance.
- Unfortunately one cannot set a hard and fast rule for the selection methods. The choice of method is a matter of judgment. Such judgment is difficult and can come only from one's personal experience. It is unhealthy to suggest only one selected method of analysis for a particular element.

• The knowledge of fundamental or basic concepts of analytical chemistry certainly provides and develops such a judgment. Thus, the analytical chemist should have an extensive knowledge and understanding of basic concepts underlying methods of analysis.

## 4.4 Chemical Analysis and Analytical Chemistry

Chemical analysis establishes the quantitative composition of the materials. The constituents to be detected or determined are elements, radicals, functional groups, compounds or phases. Analytical chemistry is concerned with much narrower and more specific aspects of analysis. The determination of one constituent in the presence of several other similar materials is essential e.g. the careful control of conditions such as pH, complexation, change in oxidation state of metals etc. Several advances in analytical chemistry have been made possible due to the spectacular progress in separation science. Analysis is generally composed of quantitative analysis and qualitative analysis is carried out before quantitative analysis. The constituents can be quickly detected by spectrographic methods or by spot test with selective, specific and sensitive organic reagents.

### ✤ Quantitative Analysis with Scale of Operation

- The amount of sample and range of relative amount of constituent determinable are important characteristics of quantitative analytical methods. The methods can be termed as macro, semi micro, or micro on the basis of the mass of the sample taken.
- A macro sample will be defined as one whose weight is greater than 0.100 gram and a semi micro as one between 0.100-0.010 gram and any sample less than 0.010 is termed as a micro sample.
- In general a concentration between 0.01-0.001 grams is termed a micro sample; while samples weighing less than 0.002 gram can be called sub-

micro or ultra-micro samples. Components which are between 100-1% of a sample are termed major constituents and those between 1-100% are minor constituents. Those below the range of 0.01% are termed as trace concentrations.

A spectrophotometric determination requires a macro sample but spectrographic determinations can be carried out with micro samples. Nowadays one can analyse fema (10<sup>-15</sup>) or ato levels (10<sup>-18</sup>) of analysis.

# \* Steps in Quantitative Analysis

The important steps involved in determination are:

- Procurement of samples
- Its conversion to measurable state
- Measurement of desired constituent
- Calculation and interpretation of numerical data. The most difficult problem is the isolation, before the measurement step.

The important steps encountered are:

- *Sampling*: It should be representative of the mass of the material. This is possible provided the material is reasonable pure and is homogenous in nature.
- *Conversion of desired constituent to measurable form:* This step involves method of separation. The selection of the separation technique for a specific situation will depend upon a number of factors. Such selection is generally decided on the basis of accuracy and the precision required.
- *Measurement of desired constituent:* Any physical or chemical property can be used as a means of qualitative identification and quantitative measurement or both. If the property is specific and selective for

measurement, then separation and pretreatment of the sample can be minimized, e.g. analysis involving atomic absorption spectroscopy.

• *Calculation and interpretation of analytical data:* An analysis is not complete until the results have been expressed in such a manner that the person for whom the results are intended can understand their significance. In recent years greater attention has been paid to statistical techniques both in development and in assaying the value of the final analytical results. This has led to the establishment of a new branch of science termed as Chemometrics.

# \* Methods of Analytical Determination

The chemical methods of determination are composed of volumetric and gravimetric methods of analysis, as they involve chemical reactions. The methods based on measurement of physical properties are called physicochemical methods. Physical methods of analysis are those methods which do not necessarily involve use of chemical reaction. Most of the physical methods of measurement are instrumental methods of analysis.

## ✓ CLASSICAL METHODS

- Separation of analytes by precipitation, extraction, or distillation.
- Qualitative analysis by reaction of analytes with reagents that yielded products that could be recognized by their colours, boiling or melting points, solubilities, optical activities, or refractive indexes.
- Quantitative analysis by gravimetric or by titrimetric techniques.

In the early year of Chemistry, most analyses were carried out by separating components of interest in a sample by precipitation, extraction, or distillation. For quantitative analyses, the amount of analytes of determined by gravimeric or by titrimetric measurement. **Gravimetric Methods** – The masss of the analyte or some compound produced from the analyte was determined.

**Titrimetric Methods** – the volume or mass of a standard reagent required to react completely with the analyte was measured.

• *Chemical methods:* These methods comprise of gravimetric and volumetric methods of analysis. They are generally based upon stoichiometric equations of the type:

 $aC + bR C_a + R_b$ 

In gravimetry, excess of the reagent (R) reacts with the constituent (C) to form a product  $(C_aR_b)$  which is solid and is weigh table. The chemical reaction and separation should be quantitative and loss should be minimum i.e. with recovery of approximately 99.9% for the major components.

However in volumetric reagent (R) is added to constituent (C) until  $C_aR_b$  is formed, the end point of the reaction is shown by an indicator. Many times the end point is reached before or after the equivalent point. Various reactions such as neutralization, oxidation-reduction, precipitation and complex formation can be used in such titrations. The speed of analysis is of great importance. Both gravimetric and volumetric methods are useful for the determination of major components. For multi analysis or for samples many in number, titrimetry are most useful. On the whole, both methods are considered precise, accurate and practicable in routine analysis.

• *Physical methods:* The popularity that these physical methods or physicochemical methods enjoy arises from the new kind of determinations that they make possible due to the determination of minor or trace concentrations of elements or constituents in preference to the major constituent of the sample. It is generally believe that relative precision or

accuracy is usually not so great as compared with chemical methods. However, this is not always true. As far as speed of analysis is concerned, physical and chemical methods cannot be compared as the former are very rapid and accurate. The majority of methods require the use of standards containing a known amount of the constituent which serves as the basis for comparison in the measurements as reference material.

### \* Role of Instrumentation

It is essential to distinguish between instrumentation for analytical techniques and operation of instruments. The former is of utmost importance to the analytical chemist. The chemist in general should understand the fundamental relation between chemical species and their characteristics. A good analytical chemist may not be an electronics expert but should know the scope and applicability of analysis and also the limitations of measurement in analysis. During analysis methods which demand the least skill from the analytical chemist are preferred. However, knowledge of the chemical reactions in a particular system is most essential. When the chemist resorts to the use of instruments, he should not end up as a black box operator. It is absolutely essential that he is able to interpret data obtained and arrive at the logical conclusion regarding the composition or structure of the analyte.

## Present Day Instrumental Analysis

- Better and faster
- More Data (images)
- Miniaturization
- Better data processing methods
- Chemo metrics

Analytical Chemistry deals with methods for determining the chemical composition of samples of matter. A qualitative method yields information about composition of samples of matter. A qualitative method yields information about the identity of atomic or molecular species or the functional groups in the sample; a quantitative method, in contrast, provides numerical information as to the relative amount of more of these components.

Analytical methods are often classified as being either classical or instrumental. The classification is largely historical with classical methods, sometimes called wet-chemical methods, preceding instrumental methods by a century or more.

# ✤ Instrumental Methods

Measurement of physical properties of analytes, such as conductivity, electrode potential, absorption, or emission, mass to charge ratio, and flurescence, began to be used for quantitative analysis of a variety of inorganic, organic, and biochemical analyte. High efficient chromatographic and electrophoretic techniques began to replace distillation, extraction, and precipitation for the separation of components of complex mixtures prior to their qualitative or quantitative determination. These newer methods for separating and determining chemical species are known collectively as instrumental methods of analysis.

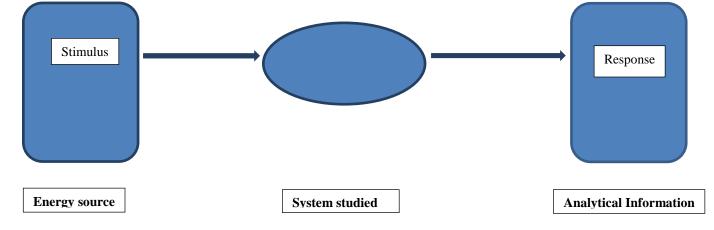
- Measurement of physical properties of analytes such as conductivity, electrode potential, light absorption or emission, mass-to-charge ratio, and fluorescence-began to be employed for quantitative analysis of inorganic, organic, and biochemical analytes.
- Efficient chromatographic separation techniques are used for the separation of components of complex mixtures.
- Instrumental Methods of analysis (collective name for newer methods for separation and determination of chemical species).
- Define the Problem

In order to select an analytical method intelligently, it is essential to define clearly the nature of the analytical problem. Such a definition requires answers to the following questions.

- What accuracy and precision are required?
- How many samples are available
- What is the concentration range of the analyte?
- What component of the sample will cause interference? What else is present?
- What is the origin of the sample?
- What are the physical and chemical properties of the sample matrix?
- When was it released or discharged?
- How/ under what circumstances will certain reactions occur?

Instrumentation is necessary to decipher these values. The challenge for the instrumental scientists is to mimic the 5 sense. Substances have physical and chemical fingerprints with unique thresholds. The object is to detect a chemical substance with a matrix and selectively perturb the substance of interest. Signals must be readable (in a voltage or electrical signal).

#### **Analytical Instruments**



#### Fig. 11: Analytical instrument

# \* Analytical Signals

The table below lists the names of instrumental methods that are based upon various analytical signals.

Signal	Instrumental Methods
Emission of radiation	Emission spectroscopy (X-ray, UV,
	visible, electron, Auger); fluorescence,
	phosphorescence, and luminescence (X-
	ray, UV, and visible)
Asorption of radiation	Spectrophotometry and photometry (X-
	ray, UV. Visible, IR); photoacoustic
	spectroscopy, nuclear magnetic
	resonance and electron spin resonance
	spectroscopy
Scattering of radiation	Turbidimetry; nephelometry; Raman
	spectroscopy
Refraction of radiation	Refractometry; interferometry
Diffraction of radiation	X-ray and electron diffraction methods
Rotation of radiation	Polarimetry; optical rotary dispersion;
	circular dichroism
Electrical potential	Potentiometry; chronopotentiometry
Electrical charge	Coulometry
Electrical current	Polarography; amperometry
Electrical resistance	Conductometry

**Table 4:** Signals employed in instrumental methods

Mass-to-charge ratio	Mass spectrometry
Rate of reaction	Kinetic methods
Radioactivity	Activation and isotope dilution methods

### **\*** Desirable Characteristics for an Analytical Method

- Speed
- Ease and convenience
- Skill required of operator
- Cost and availability of equipment
- Per-sample cost

## ✤ Selecting an Analytical Method

- Required accuracy
- Amount of sample
- Concentration range(s) of analyte(s)
- Possible interferences
- Chemical and physical properties of matrix
- Number of samples

## ✤ Numerical Criteria for Selecting an Analytical Method

- Precision
- Absolute standard deviation
- Relative standard deviation
- Coefficient of variation
- Variance
- Bias
- Absolute systematic error
- Relative systematic error

- Sensitivity
- Calibration
- Analytical
- Detection limit
- Blank plus three time's standard. Development of blank
- Concentration range
- Limit of quantitation (LOQ)
- Limit of linearity (LOL)
- Selectivity
- Effect of interferences
- Coefficient of selectivity

## Precision

- Precision of analytical data is the degree of mutual agreement among data that have been obtained in the same way. Precision provides a measure of the random, or indeterminate, error of analysis. Figures of merit for precision include: absolute standard variation, relative standard deviation, coefficient of variation, and variance.
- \* Sensitivity
- Ability to discriminate between small difference is analyte concentration

## Calibration sensitivity, $\mathbf{S} = \mathbf{M}\mathbf{C} + \mathbf{S}_{bl}$

- $\mathbf{S}$  = measured signal
- $\mathbf{C} =$ concentration of analyte

 $\mathbf{S}_{bl}$ = instrument signal for a blank

 $\mathbf{M} =$ slope of the straight line

### Analytical sensivity, $y = m/S_s$

**Y** =analytical sensitivity

**m** = slope of straight line

 $S_s$ = standard deviation of the signals

#### Detection limit

- The minimum concentration or weight of analyte that can be detected at a known confidence level
- The useful range of an analytical method ranges from the lowest concentration at which quantitative measurements can be made (LOQ) to the concentration at which the calibration curve departs from linearity (LOL).

$$\mathbf{C}_{\mathbf{m}} = \frac{Sm - Sbl}{m}$$

 $C_m$ = detection limit

 $S_m$ = minimum distinguishable analytical signal

**Sbl** = mean signal of blank

 $\mathbf{m} =$  slope of calibration

#### **\*** Selectivity

• The degree to which it is free from interference by other species contained in the sample matrix

### \* Bias

• Bias provides a measure of the systematic, or determinate, error of an analytical method

Bias = g - xt

Where: **g**is the population mean for the concentration of analyte in a sample that has a true concentration of **xt** 

### **5.0 SAMPLING IN ANALYSIS**

### Definition of Terms

- A Sample is a portion of material selected which possesses essential characteristics of the bulk of the original material.
- A sampling Procedure consists of various steps.
- A sampling Unit is defined as the minimum sized package in the consignment of material taken from any bulk sample.
- A gross sample is one which is prepared by mixing various portions or increments of samples.
- A sub sample is smallest portion of the main sample just like a gross sample.
- The analyte is the amount of sample taken for analysis

### Theory of Sampling

An ideal sample would be identical in all its respective properties of the bulk material from which it is removed. The factors to be noted are mainly the cost of the test and the value of the products, the permitted variation in the material, the accuracy of test method and the nature of the material used.

### ✤ Pitfalls in Sampling

- It is dangerous to collect sample without some background knowledge of its history
- Random choice is one of the sources of bias.
- Separation base on size can introduce a serious problem
- Operation(s) that change the composition of the sample should be avoided
- Adhesive tapes give erroneous information about the presence of zinc oxide when stuck on minerals or ores samples ordinary

### ✤ Techniques of Sampling

Various techniques for sampling solids, liquids and gases are available. Particle size differences are an associated variable with the sampling of solids. Finely ground sample is usually preferable to heterogeneous solids. Hand picking should be discouraged. It is better to avoid excessive grinding of sample as sometimes coarse granules are one which differs in composition. Mechanical sampling machines are used extensively because they are more accurate and faster than hand sampling. In the sampling of pure liquids a simple procedure is employed. Gases are however sampled by flushing, displacement with a liquid and expansion into an evacuated container. The physical state of the contaminant will determine to some extent, the method of sampling.

It is usually advisable that the bulk material to be sampled should be stratified into real and imaginary units with analysis of variance as a tool of figure of merit or a vehicle to either continue or discontinue stratification. A gross sample should be collected and reduced to appropriate size for analysis.

#### **\*** Statistical Criteria of Ideal Sample

The sampling scheme can be explicitly expressed as the sample mean which should provide an unbiased estimate of the population mean. The sample should also provide an unbiased estimate of population variance. This is possible provided every possible unit of pre-selected size has equal opportunity of being drawn. The sampling procedure should lead to estimates of the central value and dispersion that are accurate as possible. This is possible by stratified procedure. The estimation of required sample size of particulate material is another important factor. For solid particulate material the weight of sample is taken at random from bulk must be increased with increase in variation in composition, considering accuracy of analysis and nature of particle size. The main criterion is that the amount of sample should be drawn for which sampling error is less than the required limit.

#### Stratified sampling Versus Random Sampling

When the material to be sample is divided into sampling units, there are two ways of random sampling. A number of samples can be chosen at random from the bulk of material or sampling can be stratified by first choosing from the amount of the sampling units and then sampling within the units, again by random procedure. The stratified procedure gives results as precise as the simple random scheme and gives best results whenever the variance among sampling units is remarkable compared with the variance within such units. In order to reduce the sampling in the nonrandom sampling procedure, the population is divided by random procedure into a number of subdivisions and if the variance among the section is large compared with variance in sections, a more accurate results will be obtained by accurately sampling each section. Such a stratified procedure includes a risk of bias. However, such risk can be eliminated by making the procedure representative as far as possible.

Statistically, if the whole population to be sampled is homogenous then simple random sampling is as good as stratified sampling but if we have a different subpopulation which cannot be described by a single set of factors, then sampling the subpopulation by stratified procedure is accepted. If an estimate of the population mean is to be unbiased with the minimum variance in the estimate then the number of samples taken for each stratum is taken in relation to the size of the stratum also its standard deviation i.e.,

$$\frac{n_r}{n} = \frac{W_r(\sigma_o)\mathbf{r}}{\sum W_r(\sigma_o)\mathbf{r}}$$

From the 'r' stratum, 'n' is the total number of samples, 'W' is the weight of stratum and  $(\sigma_o)_r$  standard deviation with rth stratum. In calculation of population

mean of the stratum must be the weighted average in relation to the size of strata. Thus

$$\bar{x} = \frac{\sum W_{rx_r}}{\sum W_r}$$

If the  $\bar{x}$  is the mean of determined values in the 'r' stratum,  $\bar{x}$  is estimate of population mean. If strata differ in size but not in variance,  $\sigma_r$  is the same for all.

$$\frac{n_r}{n} = \frac{W_r}{\sum W_r}$$

i.e. the number of samples within strata should be related to the size of the strata. This is called representative sampling which gives an unbiased estimate, unless the variance is uniform in all the strata. The variance of the mean is independent of the variance among strata. Stratified procedure gives excellent results if the variance among the strata is appreciable compared with the variance within the strata.

#### Minimization of Variance in Stratified Sampling

Consider a bulk of material to be analyzed by taking  $(n_1)$  start, each of which provides  $(n_2)$  samples and  $(n_3)$  determinations are to be carried out on each sample. If strata are equal in size and in variance within strata, it is essential to look into minimization of cost of determining an estimate of the population mean to a certain variance. If  $(C_1)$  is the relative cost of selecting the strata, is the cost within the sampling unit  $(C_3)$  and is the cost of performing determinations, the total cost (C)is:

#### $\mathbf{C} = \mathbf{n}_1 \mathbf{C}_1 + \mathbf{n}_1 \mathbf{n}_2 \mathbf{C}_2 + \mathbf{n}_1 \mathbf{n}_2 \mathbf{n}_3 \mathbf{C}_3$

Now if  $S_1^2 S_2^2 S_3^2$  are quantities with  $\sigma_1^2$ ,  $\sigma_2^2$  and  $\sigma_3^2$  as corresponding estimates then we have:

$$\sigma^2 = rac{\sigma_1^2}{n_1} + rac{\sigma_2^2}{n_1 n_2} + rac{\sigma_3^2}{n_1 n_2 n_3}$$

Now it can be proved that to minimize the cost for (C) for fixed value of  $\sigma^2$  the values are given as

$$n_{1} = \sqrt{\frac{\sigma_{1}^{2}/c_{1}}{\sigma^{2}}} (\sqrt{\sigma_{1}^{2}}C_{1} + \sqrt{\sigma_{2}^{2}}C_{2} + \sigma_{2}^{3}C_{3};$$
$$n_{2} = \sqrt{\frac{\sigma_{2}^{2}/c_{1}}{\sigma_{1}^{2}c_{1}}}; n_{3} = \sqrt{\frac{\sigma_{3}^{2}/c_{2}}{\sigma_{2}^{2}c_{2}}}$$

Here the optimum allocation of sampling after the first stage is independent of the desired overall variance,  $\sigma^2$ .

#### **\*** Transmission and Storage of Samples

During the transportation, the container, moisture, oxygen, carbon dioxide, light, heat and other environmental pollutants in the atmosphere may contaminate the nature of the sample. It is therefore necessary to consider both the nature of the material that is being sampled and the tests that are to be made on the samples, while transporting it. Utmost care should be taken while drawing a sample from a population with a finite number of units to be examined. Since the process of sampling involves various operations like crushing, grinding and subdivision, no contamination can be permitted during storage; in addition to each operation, storage and transportation also contributes to the overall variance. It is essential to recognize the problem of storage otherwise efforts may be expended on the sampling operation either increasing the cost or failing to achieve the desired level of accuracy and precision.

#### Improving the Accuracy of Analysis

There are several ways of improving the accuracy of analysis and have been highlighted as follows:

- One can use small blanks along will the sample during determination. The use of large blanks reduces the precision;
- Give a correction e.g. weight of precipitate unreacted or unignited;
- Use controls by use of standard addition, where a known amount of constituent is added to a sample which is to be analyzed for the total amount of constituent present

### \* Statistical Analysis

The evaluation and interpretation of analytical data is very important. Statistical methods are very useful in analytical chemistry in several ways, as they measure the performance of an analytical procedure as there is need to have some measure of confidence in an analytical procedure. Such confidence depends upon the inherent performance, amount of information pertaining to performance and number of results that are used to get the average. Statistical manipulation helps to recognize changes in composition or performance due to changes or differences in a set of data.

### \* Criteria Used to evaluate Analytical Data

- Absolute and Relative difference in values
- Mean value deviation
- Average deviation of a single measurement
- Average deviation of mean
- Standard deviation
- Standard deviation of mean
- Mean value
- Median value
- Range
- Relative standard deviation

#### **\*** Criteria for Rejection of Results

- Q-Test: the most reliable basis of rejection
- The differences (δ) ≥ 4d rejection is justified, if the deviation of the suspected value from the mean is at least four times the average deviation of the retained value.

Where:  $\delta$  is the deviation from the mean, d is the actual difference between the given values and the mean value in a particular case

#### Presentation of Data

Rounding off the data to get rid of uncertainty in the last place is not always advisable for presenting data as every measurement can be used to contribute to the information and estimate. It sometimes conceals the original difference and throw away information on variation e.g. 20.38 and 20.53 has a difference of 0.15 units but on rounding off it appears 10.4 and 10.5 with a difference of 0.10 unit which is not a good representation of values.

#### ✤ Confidence Limit

If 'n' is smaller than 'S' or s, it will not be reliable and will be uncertain. By calculating 90% confidence, a limit for 'S' upper and lower limits of confidence limits can be obtained by dividing  $(n-1)S^2$ by two numbers taken from Tables of chi square e.g. if n = 7, (n - 1) = 6, then the upper confidence limit be:

Upper confidence limit =  $3.67S^2$ 

 $= \frac{(n-1)S^2}{chi \, square \, value \, at \, 95\% \, confidence \, limit}$ Lower confidence limit = 0.47S<sup>2</sup>  $= \frac{(n-1)S^2}{chi \, square \, value \, at \, 90\% \, confidence \, limit}$ 

(Where: S – standard deviation, s – standard deviation of mean) at 90% limit. For 'n' value chi square values of 95% or 90%, confidence limit are 3.94 and 18.1 respectively.

#### \* Q-TEST for Reject of Result

It can be found at 90% level by using equation for upper limit,

Range, 
$$\boldsymbol{Q}_n = \left(\frac{M_{2-M_1}}{M_n - M_1}\right) = lower limit = \frac{M_{n-M_{n-1}}}{M_n - M_1}$$
.

If  $Q_1$ ,  $Q_n$  is larger than given values in Table, it must be rejected.

#### Standard 't' Test

First standard deviation, S is calculated and a value 't' is obtained from 't' Tables. The limits are given by confidence level by an expression:  $(mean \pm \frac{ts}{\sqrt{n}})$ 

#### **6.0Research Interest**

- Developing techniques for sensitivity-Analysis of Environmental model
- Study of behavioural Responses of Target Organisms to residue Stress
- Analytical Toxicology of non-beneficial Metals, Fine Chemicals in the Environment Systems
- Application of mixed Ligand complexes in the Analysis of Environment, Biological and Agricultural Systems
- Energy management: through Environmental Waste Conversion Approach.
- Studies of Heavy/Trace metal contaminants in Human, Ecological, Environmental Systems
- Computer Modeling on Environmental Residues: Heavy/trace metals; Pesticides; chemical; Contaminants and Pollutants

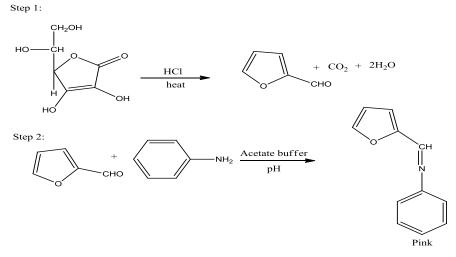
- Impact assessment of waste, waste-wasters, food/Biological, Risk and Disaster in the Environment
- Waste management Design/Recycling, Disaster and Risk management
- Development of Analytical Techniques to enhance Sensitivity, Selective, Specificity and accuracy of methods

### 7.0RESEARCH CONTRIBUTIONS AND HIGHLIGHTS

Use of Aniline and Sensitive Spectrophotometric reagent (based on furfural formation) for Trace determination of L-ascorbic acid.Journal of Applied Sciences 4(4): 2233 – 2241. **E.O. Ekanem** and D.C. Nzewi (2001).

- Equation for the reaction between pure furfural and pure aniline solutions (1:1 mole ratio)
- Principle the method is based on the condensation of L-abscorbic acid using concentrated HCl to obtain a quantitative yield of furfural, which is then analyzed using redistilled aniline in acetate buffer of pH 3.6. the pink colour obtained is then measured for maximum absorbance at 520 nm. The estimation of L-abscorbic acid is obtained by the expression y = -0.000363 + 1.83x where y is expressed in (mg) and x is the absorbance. -0.000363 represents the regression constant and 1.83 represents the regressions coefficient reaction.

**Reaction Mechanism:** 



 Use of Mixed Ligand Complex as Sensitive Titrant for Trace Analysis of Lascorbic acid in Green 'Leafy' Vegetables. Journal of Chemical Society of Nigeria, 35(2): 127-133. E.O. Ekanem et al., 2010

Estimation of Pure ascorbic acid Content by Mixed Ligand Complex (MLC) Solution During Standardization.

**Proposed Expression:** 

Half reaction:  $C_6H_8O_6 \longrightarrow C_6H_6O_6 + 2H^+ + 2e^-$  (OXIDATION)  $[Fe(Phen)(CN^-)_4]^{-1} + e^- \longrightarrow [Fe(Phen)(CN^-)_4]^{-2}$ (REDUCTION)

- Bioaccumulation and mobility of Cadmium(Cd), Lead (Pb) and Zinc (Zn) in Green Spinach Grown on Dumpsite Soils of Different pH Levels. 4(1): 85-91. M. O. Eze and E.O. Ekanem, 2014
  - The average accumulation factors of heavy metals in Green spinach is greater than unity
  - Green spinach is a potential hyperaccumulator especially in soil with low pH values
  - Metals analyzed were highly mobile from soil to leaves via the roots and stems

- 10. Analytical assessment of 'Amora'tuber (*Teacaleonto petaloides*) as a potential Raw Material in Adhesive Production. Journal of Applied Sciences, 5(1): 2433 2438. E.O. Ekanem et al., 2002
  - Amora flour (from the tuber) is suitable for adhesive formulation
  - The adhesive values (viscosity and both tensile and shear strength ) decrease with increase in temperature
  - Amora flour when converted to Dextrin gives a high adhesive value i.e. a better tack with moderate concentration and viscosity with good aging characteristics
  - Interference alter the adhesive value as observed in one of the experiment.

Detailed information could be obtained in Journal of Applied Science 5(1):2433 - 2438.

- 11. The Persistence of Herbicide Residues in Fadama and UpLand Soils in Plateau State, Nigeria. Journal of Environmental and Earth Science, 2(10):148-156. J. S. Gushit et al., 2012
  - Atrazine, 4,4-byridine, chlorophenoxy acetic acids , oxadiazon representing the residues of atrazine, paraquat dichloride, 2,4-dichlorophenoxy acetic acids and oxadiazon respectively
  - It was observed that 'atrazine' was stable among all the herbicides under investigation in the soil due to its persistent nature and long history of usage in the study area
  - No wide distinct difference was observed for the persistence of the herbicides in the Fadama and the Upland soils
- 12. Analysis of Herbicide Residues and organic Priority Pollutants in Selected Roots and Leafy Vegetable Crops in Plateau State, Nigeria.

World Journal of Analytical Chemistry, 1(2):23-28. J.S. Gushi et al., 2013

- Herbicide residues and organic priority pollutants in the root and leafy vegetable crop samples were determined using GC-MS
- The average concentration of the herbicide residues: atrazine (0.04 mg/kg) and 2,4- dichlorophenoxy acetic acids (0.02 mg/kg) were discovered to be more in the root and nuts (cassava, yam, potato, groundnut)
- The distribution of herbicide residues in leafy vegetables under investigation was very scanty as it was not detected in most of the samples
- The organic priority pollutants identified includes: organochlorine, bezene amine, polyaromatic hydrocarbons (PAHs), phthalate and phenols. They were found to occur in the samples in the rate of 6.98, 16.25, 4.67 and 9.28% respectively.
- Herbicides should not be applied when root crops are almost ready for harvest
- Bioaccumulation of Heavy Metals in mechanic Workshop. International Journal of Mathematics and Physical Sciences, 2(1):58-65. C.U. Okeke et al., 2014
  - Atomic absorption spectrophotometer was employed in the investigation of phytoaccumulation potentials of heavy metals in *Azadirachta indica* plants growing in two workshops and one farm site (control) within Bauchi Metropolis

- The weighed means of the various metals (Cu, Zn, Mn, Pb, Cr and Cd) were higher in the mechanical workshops than those obtained in the control site and were all within normal range except Cd
- ANOVA results showed that significant different in the metals studied existed among some of the aerial part of the studied plant
- The accumulation ability of *A.indica* for metals except for Cd and Cr were observed to be moderate
- Phytoaccumulation Potentials of *Tamarindus indica*. International Journal of innovation and Scientific Research, 11(1): 72-78. Okeke et al., 2014
  - The weighted means of the various metals in the plant under investigation were observed to be higher than those obtained in the control site
  - Cd has the highest translocation ratio while
  - Zn tends to accumulate mostly in the barks and roots of *Tamarindus indica* and this would therefore decrease their transfer probabilities to secondary consumers.
- Monitoring of Industrial Waste Water Channel in Kano Metropolis Kano State, Nigeria. Chemistry and Material Research. 8(6): 20-24. T. Akpomie et al., 2016.
  - The concentrations of four, 4 heavy metals (Fe, Cd, Cu and Pb) on the determination of pollutants were modeled using the Minitab statistical software
  - The model showed that the concentrations of the respective heavy metals were increasing in a quadratic manner with time

- It was predicted that an increase in concentrations from 20.45, 3.58, 3.78 and 2.87 mg/l in the year 2008 to 15, 56, 30 and 35times higher than their initial concentration for Fe, Cd, Cu and Pb respectively at 95% confidence level. The predicted concentrations would be indicative of the degree of bioaccumulation of these metals by vegetables if irrigated from this waste water source
- Estimating the Degration half-life of Herbicides in Soil Using computer Developed Models. World Journal of Anal. Chem., 4(2): 17-18. Apomie et al., 2016
  - The research was carried out with the aim of developing an appropriate model for estimating the degradation of half-life of soil applied herbicide.
  - Minitab computer programme was used to develop the mode:  $T_{1/2} = 175 5.63C_{(ppm)} 12pH$
  - The model gave a remarkable results at estimating the extent of persistence of the herbicide in the soil, water or even plant uptake
- 17. Computer Modeling of Heavy Metals in Artificial Boring. World Journal of Analytical Chemistry, 4(1): 6-10. T. Akpomie et al., 2016
  - Mintab computer software, time series and multi-regression models were employed for each of the metals: Fe, Zn, Cd, Cu and Pb in a deep well (bore hole)
  - The obtained models were of the form:  $y = a + bt + ct^2$  and y = a + b[pH] + C[pH].
  - These models were shown to be reliable at 95% confidence interval
  - The stimulation revealed that bioaccumulation was on the increase in Cu and Cd while biodegradation was the case with Fe, Zn and Pb.

This observation was a clear indication that underground seepage activities were going on, contrary to the believe, especially by rural dwellers, that boreholes (deep wells) water are pure and fit for drinking

- 18. Health Risk assessment of Exposure of Some Heavy metals via drilling Water from Dakinkowa Dan and River Gombe Abba in Gombe State, North east Nigeria. World Journal of Anal Chem 4(1): 6-10. A.Y. Magari et al., 2016.
  - The concentrations of eight heavy metals (Fe, Mn, Cu, Pb, Cd, Ni, Co and Zn) were determined by atomic absorption spectrophotometer
  - The human health risk assessment was performed by determining the chronic intake, CDI, hazard quotient, HQ and total hazard index, TDI of the metals through human oral consumption for both adult and children
  - The HQ of iron, manganese, nickel and cobalt in water from the study sites were all greater than unity and thus pose a potential health risk for both adult and children
  - Cobalt was the only heavy metal of concern in water as its HQ was greater than one
  - The THI of water from all the sampled sites assessed were of high risk
  - Further monitoring of these sites is recommended as well as biomedical experts to ascertain the exact adverse effects that metal contamination of water make might induce in human particularly among individuals in vulnerable populations such as children.

- Multivariate Statistical Analysis of Ground Water Chemistry Data from Hadijia Local Government Area of Jigawa State, Nigeria. Global Journal of Advanced Research, 3:8-30, 2016
  - Hydrochemistry i.e. Chemistry of water, was investigated using the multivariate statistical tools cluster and principal component analysis result
  - Cluster analysis results grouped twenty, 20 sampling points into five, 5 statistically significant clusters based on their similarities
  - Principal component analysis was used to investigate the origin of each water quality parameters and yielded five, 5 varimax factors/ components with 75.28% total variance, indicating the major variations are related to anthropogenic activities and natural processes.
- 20. Kinetic Modeling of Vitamin C (Ascorbic Acid) Degradation in Blanched Commonly Consumed Salad Vegetables Using Computer Simulating Analysis. IOSR Journal of Applied Chemistry, 10(40): 59-66.
  - Vitamin C (ascorbic acid is one of the most important and popular vitamins, and is contained in most fruits and vegetables. The problem with vitamic C is its easy degradation during processing
  - The degradation kinetics of vitamin C was determined in Lettuce and Cabbage, and the processing treatment was blanched at 70 °C of water differently for 5, 10, 15, 20, 25, 30, 35 and 40 mins.
  - Samples were dried (15-20 °C) and ground to fine dust and HPLC was used for determination of the abscorbic acid of vegetable salad samples with UV-Visible detection at 245 nm

- The rate constants, using integrated rate law method and half life were found to be 0.099 min<sup>-1</sup> and 0.088 min<sup>-1</sup> and 420.0892 and 472.6004 seconds respectively for both lettuce and cabbage under the same blanching conditions: 0.099 min<sup>-1</sup> and 0.088 min<sup>-1</sup> respectively
- Relationship between Cadmium Accumylation and Soil EC (A Case Study of Five Automobile Workshops). International Journal of Advanced Research. C.U. Okeke et al., 6(1): 764-770.
  - Resulting coefficient of correlation established a linear relationship between variables, with  $R^2$  of between 0.869 0.955 in all the study sites
  - Three different models: Linear, Logarithmic and Quadratic were employed and found to suit the data obtained from the correlation between Cd and the soil electrical conductivity.
  - Regression models obtained can be used to forcast the impact of Cd in the various workshop
- 22. In vitro Evaluation of Free Radical Scavenging Ability of root Bark Extract of Massularis acuminate Spp. International journal of Advanced Research, 4(3): 1428-1432. U.S. Ukekpe et al., 2016
  - The outcome of this investigation has shown the root bark of Massularia acuminate contains phytochemicals that are antioxidant in nature and are capable of scavenging free radicals reasonably.
  - It has been established that there is a good relationship between extracting solvent and antioxidant property of extract as it affects the phytocomposition.
  - Methanol was observed to be a good solvent for extracting antioxidant phtochemicals from the root bark of *Massularis acuminate* Spp.

- 23. Comparative Evaluation of *Luffa aegyptiaca* Seed Oils as Insulating oil in the Nigeria Power Sector. Chemistry Research Journal , 3(2), ISSN: 2455-8990. A. B.Mustapha et al., 2018
  - The comparative (α = 0.05) evaluation between *Luffa aegyptiaca* Seed Oil, LASO and Mineral Insulating Oil, MIO shows that the ester oil, LASO is better than MIO in terms of environmental safety and electrical properties
  - MIO was better in terms of shelf-life
- 24. Conversion of Domestic Wastes (Saw Dust, Rice Husk, Corn Cob and Plastics) to Industrial Products, 2012.
  - Saw dust, rice husk, corn cobs and plastic waste particle board of cliff combination ratio were produced with 20 cm<sup>3</sup> of 4% melanine urea formaldehyde as adhesive, 10 cm<sup>3</sup> of 20% NH<sub>4</sub>Cl and 10 cm<sup>3</sup> of 20% (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> as harderness. Data were statistically analysed and comparison of products were performed by series of tests. Generally, the results and mechanical properties of the particle board. In this study, the best condition was obtained by the use of 20 cm<sup>3</sup> of 40% melanine urea formaldehyde and 10 cm<sup>3</sup> of 20% NH<sub>4</sub>Cl with particle board combination ratio of 5% saw dusk, 5% rice husk, 5% corn cobs and 85% plastic waste. Tensile strength, thickness, swelling and water absorption were the series of tests carried out on the particle board.
- 25. Fate of Organochlorine pesticides in the Savanna Soils of Bauchi State, Nigeria. Journal of Agriculture, Biotechnology and Ecology, 3(3): 83-91. E.O. Ekanem et al., (2010).
- Chemical Analysis of Some Metals in selected Nigeria coals. Journal of Chemical Society of Nigeria, 36(1): 152-156. E.O. Ekanem et al., (2011).

- 27. Effect of pH on recoveries of Commonly Used Organochlorine Herbicides from Soil in Bauchi State, Nigeria. International Journal of Modern Analytical and Separation Sciences, 2(2):71-79. E.O. Ekanem et al., (2013).
- 28.Evaluation of Toxic Heavy metals in Savanna Soils of Bauchi State, Nigeria: Agronomical and Environmental Aspect. International Journal of Modern Analytical and Separation Sciences, Modern Scientific Press, Florida, USA. 2167-77782920:80-90
- 29.Simulating the Concentration of Some Heavy metals in Mista-Ali River in Jos Nigeria. International Journal of Scientific and Technological Research, Vol. 3. Issue 9. T.M. Apomie et al., 2014
- 30.Physiochemical and Electrical properties of Pre-processed Ester Oils. Chemical Research Journal, 2(6):261-272. A.B. Mustapha et al., (2017).

#### 8.0 CONCLUSIONS

Mr. Vice Chancellor Sir, the following conclusions were drawn from the title of the inaugural lecture as follows:

- That the scientific method in today's researches and journal paper presentation are not strictly follow such as observation, experimental design, hypothesis, data treatment and interpretation of results which makes some of the scientific work in term of discussion shallow.
- Others that have raised serious research concern in the mode of sampling, details steps of sampling many at times are usually not followed hence confusion in presentation of results. As you heard from the lecture observation is the sample of the universe, this means that sampling design must follow individual segments of the environmental system to enhance reproducibility and reliability of results.

- Graphical impressions are of utmost important to ease in the interpretation and discussion of the observed result and enhance mature and professional discussion in the subject matter.
- Finally, this lecture will encourage the present scientific, experimental and professional analysts as they would use their eagle eye to proffer solution to long term and short term remediation of the environment and also create a standard for use by the various agencies such as: environmental, health and communities where there are disaster, natural or anthropogenic to make the place habitable.

#### 9.0 RECOMMENDATIONS

Mr. Vice Chancellor Sir, having gone through the journey, I wish to recommend without delay, the following:

- We should make Science and indeed Chemistry have a human face in this country to encourage quality research and development; by going back to the rudiments, to bring those steps that would assist us break the poverty line and by reeducating ourselves and mentoring the youth (future leader, the act of following instructions, committing them to a chosen career and depending on what they have acquired to face any challenges they may see in the near future: "Rome was not built in a day".
- Making Science a literature tool for national development; creating ; an enabling environment for research, development and productivity; considering the enormous resources both human and material in our environment systems
- Sensitize the public by revisiting the vocational and technical studies, to equip our middle level manpower and not being over dependent on foreign

skilled labourers and over borrowing (Paris Club). This will reduce dependency in buying and selling of crude materials e.g. oil, minerals which usually increases unemployment and over bloat the economy, with youth restiveness.

- Teach and mentor our youths especially the graduates to be less dependent on white collar jobs and reduce the get rich syndrome, than be more creative in challenging their immediate environment with exploration and exploitation of our rich resources (source of creating wealth) and technocracy.
- Advice the undergraduate to engage on collaborative researches by: "Do it Yourself Concept" to understand our environment and generate employment no matter how small it may be (invention and discovery drive).
- I also should not only be well equipped but should have well trained, committed and experimental Scientist to educate and maintain the facilities and mentor the youth in laboratory management
- Create greater awareness in cleanliness and precautions for safety and health environment
- Instructional procedures on operating the equipment/ instrument must always be placed on the wall by the instrument to avoid accident or damage when in used and be guided by trained personnel.
- Data obtained from an experiment should not be mutilated but kept for further treatment.
- Train the future Chemists in particular to be confident in carrying out any experiment or field work for the reliability of results

#### **10.0 ACKNOWLEDGEMENTS**

Firstly I am grateful to my Creator, mentor and teacher who made me what I am today. His name is JESUS who is always by my side and speaks silently to me with these words of encouragement and strength and inspiration: "Fear thou not; for I am with you (Eno): be not dismayed; for I am your God: I will strengthen you (Eno); yes, I will uphold you( Eno) with the right hand of my righteousness" – Isaiah 41: 10 (KJV, Holy Bible). "For God has not me (Eno) the spirit of fear; but of power, and of love, and of sound mind" - 2 Timothy 1: 7; and finally, He spoke to me saying "you (Eno) can do all things, you choose to do through Him (Christ ) who strengthened me (Eno)" - Philippians 4: 13. Secondly, to appreciate the numerous people on my way through this journey, some encouraging, others despiteful in all challenging thus, bringing out the best in me this journey of my I would at this moment express my profound gratitude to my academic career. late parent Prince Okon Ekanem and Madam Arit O. Ekanem of blessed memory for their upbringing, never accepting defeat, love, always going for the best and personal sacrifices, made my life meaningful and educational career possible.

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ibom State I learnt to be alert, always prepared and confident in what I say and do, this makes me remember Reverend Father Umoh, principal, Justice Enefiok Udoh, my guidance at Stella Maris college Port Harcourt and teacher in Holy Family College Oku Abak, who had a watchful eye over me and other staff of the college you are greatly appreciated.

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#### APPENDIX

Appendix 1: Relevant formulae for simple random sampling

• Absolute standard deviation  $S = \sqrt{\frac{\sum_{N=1}^{N} (x_{i-\overline{x}})^2}{N-1}}$ 

• Relative standard deviation  $RDS = \frac{S}{\bar{X}}$ 

- Coefficient of variation  $CV = \frac{s}{\bar{x}} \times 100\%$
- Standard deviation of mean  $S_m = \frac{S}{\sqrt{n}}$

Where n = number of times the mean was determined