Mohd Tariq

The Science and Art of Kushtasazi (Taklees)

Basic concepts and analytical Theory

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The Science and Art of *Kushtasazi* (*Taklees*)

(Basic concepts and Analytical Theory)

Preface

Kushtasazi today is considered as an essential element of liberal education at all levels. A well educated person is expected to be familiar with various areas of calcination. So, it is not surprising that calcination has emerged as a major section in the field of Unani pharmacy (Ilmul Saidla). *Kushta* (calcined product) is the dosage form concerned with rejuvenation, longitivity, virility, vitality. Works on *kushtajat* are now read by a much wider audience and now is no longer confined to historicians. The need of serious treatment is this subject is now felt more widely. So far no attempt has been made to study this type of dosage form in a scientific manner. This is because of a lack of communication among traditional healers, physicians and scientists and the unavailability of the literature in English. The preparation procedures of *kushtas* have always been a practical problem. So, the present book has been designed keeping in view the fact that students require greater depth and breadth of knowledge to tackle complex nature of calcination technology and with the self effacing aim in the direction of development and understanding of *kushtasazi*. All efforts have been made to make this book as informative as possible. However, the author would welcome the suggestions for further improvement of the book. The author would be really happy to know about the other topics that can be covered in future editions of the book.

Dr. Mohd Tariq (M.D)

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Introduction

There are three principal systems of medicine practiced in India: Unani-Tibb, Ayurveda and Siddha. These systems utilize drugs of natural origin constituting plants, animals and mineral preparations. The medicines made under these systems use drug material of plant, mineral and animal origin. **(Kapoor RC, 2010) (Hussain GD et al, 2010)** While research on medicinal plants has received considerable attention, the metallic and mineral preparations have relatively been neglected. Studies on the role of elements in health and disease have now become of global importance with spurt of research activity in the last two decades. **(Bajaj S and Vohora SB, 2000) (Anonymous, 1996)**

The Horizon of Unani Tibb

Unani medicine originated from Greece under the patronage of Hippocrates (460 BC–377 BC) and from the fusion of diverse thoughts and experiences of nations with ancient cultural heritages and advanced with the wealth of scientific thought of that age. **(Chaudhary RR and Rafei UM, 2001)** *Unani Tibb* was introduced in India by Arab and Persian settlers. The Unani physicians paid special attention to the medicinal herbs found in India and wrote books on the therapeutic qualities of these herbs. **(Chaudhary RR and Rafei UM, 2001)** It was developed by Arabs into an elaborate medical science based on the frame work of the teaching of Hippocrates and Galen. Since that time Unani Medicine has been known as Greco-Arab medicine. Unani is one of the most well known traditional medicine systems and draws on the ancient traditional systems of medicine of China, Egypt, India, Iraq, Persia and Syria.

Most of the recent investigations on the Indian indigenous drugs have been confined to drugs of vegetable origin. The reason for this is not far to seek. The vegetable drugs from the very early times have formed a predominant portion of the *materia medica* of both the *Hindu* and the *Mohammedan* medicine in this country. **(Chopra RN et al, 1982)**

In the recent times, public interest in the world has shifted towards traditional medicine for safety concerns, namely, about the adverse reaction and side effects that entailed use of many allopathic formulations. Unani medicine and herbal products are gradually more being used in many countries. The indigenous systems use mineral preparations mostly in calcined forms: *Kushtas* in Unani-Tibb, *Bhasmas* in Ayurveda and *Parpams* in Siddha. *Kushta* is an important class of Unani medicinal preparation obtained by calcinations of metal, mineral and animal drugs. The usage includes even those elements which are otherwise considered toxic and not administered internally in conventional system of medicine.

After the development of *Kushtasazi* technique, metals like Gold, Silver, Copper, Iron etc were found therapeutically useful after processing them by various pharmaceutical processes such as *tasfiya* etc. These dosage forms gained importance because of their smaller dose and quick relief as compared to herbal drugs in various ailments. **(Devanathan R, 2011)** Traditional calcination techniques are specialized processes wherein herbal juices are incorporated during preparation. It is claimed that these processes purify the metal and make it therapeutically effective and safe.**(Bajaj S and Vohora SB, 2000)** *Rasayana* (Rejuvenation Therapy) mainly deals with the preservation and promotion of health. It promotes longevity and prevents or delays the aging process. *Rasayana* promotes resistance against infections and other causative factors for the disease. The *Rasayana*, if administered at an early age, also helps the body metabolism in

such a way that the genetic predisposition for a particular disease is avoided and the intensity of the symptoms of a particular disease is greatly reduced. Rejuvenation therapy, in addition to dealing with the preventive and promotive aspects of health rather than merely with the disease condition, acts also by improving microcirculation in the body leading to better bio-availability of nutrients to tissues. The use of *Rasayana* drugs and other measures produces longevity, improves memory, intelligence and good health, promotes youthfulness, good lusture, complexion and voice, promotes optimum strength of the body and the sense organs, efficiency in talk and humility, and makes the personality attractive. Rasayana drugs having an immuno-protective effect is helpful in improving the immunity and general well-being of patients. **(Chaudhary RR and Rafei UM, 2001)** *Kushta* being metal preparation are inorganic in nature. *Kushtas* of iron, calcium, copper, tin silver, gold and zinc are commonly used. **(Dubey N et al, 2008)**

Synonym

Kushta **(Kareem BH and Ferozuddin CH, YNM)**, *Rasayana* **(Kareem BH and Ferozuddin CH, YNM: Mahdihassan S, 1979)**, Elixir **(Kareem BH and Ferozuddin CH, YNM)**, *Kimiya*(**Kareem BH and Ferozuddin CH, YNM: Mahdihassan S, 1979)**, *Ikseer*(**Kareem BH and Ferozuddin CH, YNM: Mahdihassan S, 1979)**, *Bhasma* **(Bajaj S and Vohora SB, 2000)**.

Etymology and definition

***Kushta*:** From Persian *kushtan* 'to kill' (کشتن), *Kushta* (کشته) past participle of *kushtan* means "killed" or "conquered".

Kushta is a metallic, non-metallic or mineral substance, converted through thermal treatment to an effective and rapid acting drug, which produces prompt

pharmacological effect after entering in the body and blending with blood. **(Kabeeruddin HM, YNM)**

Bhasma: **(bhäs··ma),** From Sanskrit word *bhasma,* literally means 'disintegration'. *Bha* implies *bhartsanam* (to destroy), while *sma* implies *smaranam* (to remember). *Bhasmas* are unique Ayurvedic dosage form based on metallic/mineral preparations, treated with herbal juice or decoction and exposed for certain quantum of heat. **(Sarkar PK and Chaudhary AK, 2010)**

Bhasma: *n* in Ayurveda, a method of medicine preparation in which purified minerals and animal ingredients (e.g., horn, shell) are macerated and ground with herbal extracts. The resulting mixture is then heated until only the residual ash remains. **(Jonas, 2005)**

Rasayana /ra·sa·ya·na/ (rah″sah-yah´nah) **(रसायन) (Mahdihassan S, 1979)**

Prefix: *Rasa*

Rasa = mercury (Sir PC Ray); gold, juice (Alberuni); *Pran* means life essence or soul (Sanskrit Bengali dictionary Amartha Chandrika); *Swarna* means gold (Bengali to Bengali dictionary). Its actual meaning is juice extracted from a plant.

Suffix: *Ayana*

Ayana= way, path (Sir PC Ray); *ashra* means shelter hence also abode and vehicle (Bengali to Bengai Dictionary of Mitra).

Thus *Rasayana*= way to mercury, way to gold, way to life, path of essence, vehicle of juice etc. **(Mahdihassan S, 1979)**

Any of a group of herbal remedies with antioxidant properties used in Ayurveda to promote health, provides defense against disease and promotes longevity. **(Dorland, 2007: http://medical-dictionary.thefreedictionary.com/rasayana)**

***Iksir*:** From Arabic *Iksir* = *kimiya, iksir* means one-soul incorporated. **(Mahdihassan S, 1979)**

Elixir: Derives from Arabic *al-'iksir*; al, *the* + *'iksir, elixir* Middle English, a substance of transmutative properties, from old French *elissir*, from Medieval Latin *elixir*, from Arabic al-*'iksir*: *al, the* + *'iksir, elixir*

(probably from Greek *xerion*, desiccative powder, from *xeros*, dry).]

A substance or medicine believed to cure all ills, a substance believed to maintain life indefinitely, a panacea and universal remedy. **(http://www.thefreedictionary.com/elixir)**

***Kimyah*:** From Chinese *Chin* 'gold'; *I* or *yeh* 'plant juice.' *Chin-I* or *Chin-Yeh,* literally means gold cum plant juice. In Fukin dialect *Chin-I* is *Kim-Iya* which entered in Arabic as *kimiya*. It was transliterated into Greek as *CHEMIA* but pronounced exactly as the Arabs did. This was about 200 BC. **(Mahdihassan S, 1987)** *Kimyah* means gold making juice/soul, synonym of elixir, as *rasayana*. **(Mahdihassan S, 1979)**

Mukallas (مكلس)**:** From Arabic *kils* 'lime', *Kalas* 'to calcine, calcify'; *mukallas* 'calcined or calcified.**(Cowan M, 1980)**

Historical Background of *Kushtasazi*

(*Taklees ka tareekhi pase manzar*)

The use of calcined metals in Unani and Ayurveda is very old. It is often said that calcined form of the metals (*kushta*) is specifically a method proposed and developed by Ayurveda as claimed by some Ayurvedic scholars (**Rasheed A et al, 2011: Chitnis K and Stanley A, 2011, Devanathan R, 2011: Chaudhury RR and Rafei UM, 2001**) and also by some Unani scholars (**Kabeeruddin HM, YNM**). To find the fact we must go through the history.

Uses of metals and minerals are dates back to the time immemorial. It should be known that for long ago, this dosage form was in practice in Greece and Italy and *Hermis* is regarded as the father of alchemy. (**Hafeez A, YNM**)

Gold, the most precious of all, has ever had an attraction for man. It is probable that it was one of the earliest metals of which man made, use, and may, indeed, have been the first discovered, as it was to be found free in nature, in rocks as well as in the sands of rivers. In Ethiopia and Nubia it must have been known at a period of great antiquity, and there is evidence that quartz-crushing and gold-washing were known in Egypt before 2500 B.C (**Thompson CJS, 2002**).

Copper, which was associated with the planet Venus, was known and used before 9000 BC in Middle East before iron. It was smelted from the ores by primitive man. As the copper ores were frequently found associated with other metals it was probably soon discovered that by alloying, it could be rendered hard enough to fashion into tools. It was known to the Greeks in the time of *Homer*, for he tells us that the shield of Achilles was composed of gold, silver, tin and copper, while the arms of the heroes were of copper.

Bronze was used by the Egyptians as early as 2000 B.C. and was employed for making vases, statuettes; mirrors etc. and the alloy consisted of from 80 to 85 % copper with between 20 and 15 per cent of tin.

The metal-workers in ancient times obtained the red and black copper oxides by heating copper to redness and allowing it to cool in the air. They distinguished between the scales which fell off during the cooling and those that could be obtained by heating the metal. These oxides were used for colouring glass. **(Thompson CJS, 2002)**

The oxy-acetate of copper or verdigris was known and used as a pigment at least five thousand years ago while later it was employed by the Egyptians, Greeks and Romans as a remedy for various affections of the eyes. The Egyptians prepared it by covering plates of copper with the refuse of grapes. **(Thompson CJS, 2002)**

However to locate the origins of alchemy, we must travel back to Egypt in the first century of the Christian era when Egypt had come under the influence of Greek culture following its conquest by the Alexander the great during his vast military campaigns of 334-323 BC. **(Principle LM, 2013)** Many technical operations, fundamentals of alchemy had been developed well before its emergence. The smelting of metals such as silver, tin, copper and lead from their ores had been practiced already for 4000 years. The making of alloys (such as bronze, brass etc.) and various techniques for metallurgy and metal work had been developed to a fairly high degree. **(Principle LM, 2013)**

Papyri are the original documents currently known to survive from the Greco-Egyptian period. Despite many books about alchemy written during that time the only surviving testimony of that distant era comes in the form of

anthologies. (i.e. collection of excerpts copied from the original texts that are now lost) These anthologies are collectively called *corpus alchemicum graecum.* The oldest surviving copy dates from around the start of 11th cent. It contains scripts from about two dozen books mostly dating from 2nd to 8th cent. A.D and is now preserved in Venice. **(Principle LM, 2013)**

The earliest text within the *corpus alchemicum graecum* dates from about late 1st or 2nd cent.AD. It carries the title *physica kai.* Its authors name is Democritus and it records workshop recipes. In fact it uses the same fourfold division of processes into those for gold, silver, gems and dyes. **(Principle LM, 2013)**

The another old chemical documents is of Greco-Roman period and probably dated from sometime around the late 3rd or early 4th century AD Leyden papyrus and Stockholm papyrus written in Greek. Leyden papyrus focus primarily on the preparation of various metal alloys, many of which are intended to imitate the appearance of either gold or silver, for use in making jewellery, in gilding or in metallic writing while a few others deal instead with dyes of various sorts. The contents of the Stockholm papyrus have the same form, but focus more on dyeing and the imitation of various precious stones and gems. However few chapters of Leyden papyrus, dealt almost exclusively with chemical recipes.[45] These papyri contains about 250 practical workshop recipes **(Principle LM, 2013)** and includes process of identification, gliding, manufacture and purification of metals, minerals and precious stones. **(Caley ER, 2008)**

If we focus our attention on the *corpus alchemicum graecum*, it is clear that medicine played an important role in the origins of alchemy, which took its first

steps in the Graeco-Roman Egypt from the 1st to the 4th century AD. **(http://www.crassh.cam.ac.uk/uploads/documents/abstracts.pdf.)**

The first Greek writer who mentioned the transmutation of metals is Baraus, who lived about the fifth Century A.D. but Zosimus of Panopolis, who flourished about the fifth century A.D, was the first alchemist of whom any authentic record is present. **(Thompson CJS, 2002)**

It is a historical truth that old Unani physicians used some remedies containing certain minerals, such as salts or oxides of copper and lead. **(Magner LN, 2005)** This may also be proved by some historical facts. Galen (129-200 AD) states that the efficacy of burnt lead is unparallel in cancer and copper was burnt before its use because it is harmful for the body when used as it is **(Tariq M et al, 2013)**. Aribasus (326-403 A.D) **(Nigrami SMH, 2004)** used ash of animal. **(Usaiba IA, 1990)**

Indian alchemy

Indian alchemy begins probably about 500 AD. The early historian of alchemy such as Von Lippmann believed that alchemy was brought to India by Muslims. **(Mahdihassan S, 1979)** The use of mercury is well known in India but it is only found in the latter literature and cannot be traced back any further than the earliest *tantric* text of 5th to 6th cent AD. In medicinal works mercury is mentioned only once in *Charaka* treatise (year highly variable) once in Bower manuscript of 4th cent. AD and twice in *Sushruta* (year highly variable). It is entirely unknown to earlier literature. **(Mahdihassan S, 1979)**

The Arabian Alchemists

With the conquest of Egypt, Syria, and Persia by the Arabs and the rise of Islam the centre of scientific learning changed. The Arabians were eager seekers after knowledge and became the most cultivated people in the world. (**Thompson CJS, 2002**) The Arabs were master in alchemy. Prior to the rise of Islamic civilization, the subject of alchemy and its basic characteristics were well established by the ancient Hellenistic sages. From the very beginning Muslim scholars worked on the alchemical principle formulated by the Alexandrians and further restructured it and aligned it with their own interest and need of the time. The knowledge of alchemy was as a result considered as supernatural science that dealt with the attributes of matter whose origin could not be visualized by the senses. The first impulse given to the desire for knowledge of the wisdom of the Greeks came from the Umayyad Prince Khalid, who had a passion for alchemy. According to the Fihrist this prince assembled the Greek philosophers in Egypt about the eighth century A.D. (**Thompson CJS, 2002**) and translated Greek books on chemistry into Arabic. (**Islam A, 2011**)

Around 800 AD lived another master of Indian medicine known as Vagbhatta. His treatise has eight divisions called *astang* in which two are of *rasayana tantra* or doctrine of elixers and *vajikarana* tantra or the doctrine of aphrodisiacs. (**Mahdihassan S, 1979**)

Jabir (b.831. A.D), regarded as "father of chemistry" (**Nigrami SMH, 2004**) was an Alchemist (**Ibn Nadeem, YNM**) and wrote many books on alchemy. (**Qifti J, 1945**) He was not only universally recognized in the Muslim world but in the West as well. Jabir's science was based upon the Hellenistic view that all metals are basically the identical matter, but with varying impurities. His writing encompasses different areas including philosophy, theology, metaphysics, medicine and technology etc., but the predominant discipline is alchemy where he

provides a rational basis for the expansion of chemistry and pharmacy. He was the founder of experimental chemistry and was outstanding in his laboratory work. Jabir was the first to prepare sulphuric acid by distillation, and he prepared mercury oxide and nitric acid. According to *Ibn Nadim,* Jabir wrote 306 books on chemistry, but most of them have vanished, still eighty of these are preserved in various libraries. Most of his books were translated into Latin in the 12th century by Robert Alshestry (d. 1144), Girard Alcremony (d.1187) and others. **(Islam A, 2011)** Jabir, in his book *'Nakhbe Jabri'* has mentioned the use and method of preparation of *kushtaj* of *faulad, qalai, naushadar* and *abrak, taseed* of *seemab* and *gandhak.***(Thompson CJS, 2002)**

Razi (d.925. A.D) an another reputed personality in chemistry **(Qifti J, 1945)** was equally renowned in the field of alchemy too. He gave more preference to experimental chemistry rather than theoretical and magical style. In his book *Sirr al-asrar* (the Latin *Liber secretorum bubacaris*) Razi divides his subject matter into three categories: the first, on the acquaintance and identification of drugs from plant, animal and mineral origins and its use in treatment; the second, understanding of equipment and tools used; the third, the familiarity of the seven alchemical procedures and techniques like sublimation and condensation of mercury, precipitation of sulphur and arsenic, calcinations of minerals, salts, glass, talc, shells and waxing. His alchemical texts: *Al-asrar* (The secrets), and *Sirr al-asrar* (Secret of secrets) are the most famous of his alchemical Works. According to *Ibn Nadim*, Razi's alchemical writings number 115 books and 30 epistles. **(Islam A, 2011)** Razi, wrote a book *''Kitab ul akseer''* on *kushtasazi*. **(Ibn Nadeem, YNM)**

A treatise on *taklees 'Risaiala fil hikmah al mastoorah'* known as *Kitab al taklees* has also been written by Ibn e Sina, *Risala dar azkare tanqia wa taklees*

(Irshad S, 2009), *Risala fi al taklees* **(Rehman SZ, 1999)** are rare books on *kushtasazi*. Other books though not dealing specifically with calcination include a chapter on the Taklees.

Majmooat al sanae by *Shamsuddin undulusi, Haqaiq asrar al tib* by *Masood bin mohammad al sanjary, Makhzan al hikmat* by *Ismail, Tibbe aurang shahi* by *Hakim durvash mohammad aminabadi, Qarabadeene masumi* by *Masoom bin kareemuddin sherazi, Majma al bahrain, Mufradat kitab dastoor al hunood* and *Ganj bad award* by *Amanullah khan, Matla al nayyaran by Baragi khan, Majmoa ziai by Zia mohammd , Ahwal al jawahar* by *Mohammd bin mansoor* etc. can be cited here for the contribution and legacy of Unani physician in *kushtasazi* **(Irshad S, 2009)** Another important book written on *kushtasazi* is *"Kitab tib wa kimiya"* written by Barakalus. It has been translated from Greek to Persian and is preserved in Kutub Khana Nadva, Lucknow and kutub khana Asfia, Hydrabad.**(Qasmi IA, YNM)**

The use of *kushtajat* is also seen in *Tibb nabwi* where the ashes of *bori* were used for the treatment of soldiers fighting the battles for Islam. **(Qasmi IA, YNM)** The use of *kushtajat* is also evident in Umayyad dynasty where cohen Ablak treated a girl named Am-ul-gilan with *ikseer* and later got married to her. The use of metals is evident from the epics of *Homer*. **(Qasmi IA, YNM)**

As far as Ayurveda is concerned, the drugs of animal origin were occasionally used by the Hindu physicians. As regards the drugs of mineral origin, their use is also comparatively limited. It appears that the ancient Hindus were not quick in learning the art of adopting the metals and metallic compounds for medicinal purposes.

Charaka: As far as *Charaka* is concerned, the time period of *Charaka* is highly variable in different books and articles like (900 B.C) (**Ali M, 2008**), 7th century BC (**Prakash B, 1997**), *Charaka samhita* was written around 5th Century B.C. (**Chaudhury RR and Rafei UM, 2001**), 3rd-2nd cent B.C (**Murlidhar P and Byadgi PS, 2011**) or 2nd century A.D. (**Mahdihassan S, 1979**) It is also well known that one of the earliest works on Ayurvedic medicine by *Charaka* does not deal at all with any mineral origin drug. (**Chopra RN et al , 1982**)

Sushruta samhita written at latter period (year highly variable), only mentions the use of a few natural salts such as sodium chloride, impure carbonates of sodium and potassium, borax and some salts of iron, silver, copper, tin and lead as well as some precious stones.[19] but the time period of *Sushruta* and his book i.e. *Sushruta samhita* is also quite variable. According to Traditional medicine in Asia *Sushruta samhita* was written around 6th – 5th Century BC.[41] and according to Roy PN *et al* the medical treatise *"Sushruta samhita"* was written approximately 800 years before the birth of Christ.[55] It means *Sushruta* came even before *Charaka*. Secondly as we know that *Sushruta* was a surgeon, so he might have used some natural salts but being a surgeon he might have used those externally not for internal purpose i.e. not in calcined form. Thirdly it is also important to note that being a surgeon was *Sushruta* able to prepare a *kushta* of a metal or not, because *kushtasazi* is entirely different from surgery. Fourthly it is clear from Chopra's indigenous drug of India that only writers of considerably later periods gave descriptions of calcination and purification of compounds and other process of converting such metals as gold, silver, iron, copper, mercury and arsenic into suitable forms for use as medicaments.[19] It means up to the time of *Charaka* and *Sushruta* process of purification i.e. *tasfiya* was not present. So one assume that if the process of purification or *tasfiya* was not present till their time then process of calcination was also not present till their time.

Nagarjuna

According to Ayurveda, *Nagarjuna* is considered as the father of Indian alchemy and *Rasa shastra.* (8-9 cent AD).**(Prakash B,1997)** It is true but he is the father of only Indian alchemy not alchemy as a whole, as it is clear from above collected data mentioning use of metals by *Hermes* (B.C). **(Hafeez A, YNM)** Galen (200 A.D), use of ash of animals by *Aribasus* (326-403 A.D) **(Nigrami SMH, 2004)** is much before than *Nagarjuna.*

So by the help of the data collected one may reach to the conclusion that the history of use of calcined materials in Unani medicine is much older than Ayurveda. **(Irshad S, 2009)**

Calcination: Definition, Aims and Objective, Scope

Kushtasazi / Calcination is the process of heating a substance at high temperature (but below the melting point) causing loss of moisture, reduction or oxidation. The process of *Kushtasazi* is carried out using cowdung cakes or in the furnaces, *bhattis* or kilns. It is a thermal treatment process applied to metals like Gold, Silver, Copper, Tin and Iron; to *Hajriyat* or precious stones like *Zamarud, Yaqoot, Yashab, Marwarid, Marjan, Hajrul Yahud* and non-metals like *Raskapoor, Shangraf, Sankhya* and *Darchikna*. This process is carried out in order to bring about a thermal decomposition or removal of a volatile fraction from the ingredient drugs. The calcination process normally takes place at temperatures below the melting point of the product. The process of calcination derives its name from its most common application, the decomposition of calcium carbonate (limestone) to calcium oxide (lime) and carbon dioxide. The product of calcination is usually referred to in general as "calcine */kushta*" irrespective of the actual metals, minerals or precious stones, undergoing thermal treatment

Calcination Reaction

For example, in limestone calcination, a decomposition process, the chemical reaction taking place is as follows-

$CaCO_3 = CaO + CO_2$ **(Khaleefatullah M, 2009)**

In the burning of the limestone the water in the stone evaporates, and the carbon dioxide is given off as a gas, leaving the oxides of calcium and magnesium. **(Anonymous, 1993)**

The prepared *kushta* gives different effects depending upon the method of its preparation. E.g *Kushta Sim Ul Far* when prepared with *Doodh, Ghee* and *Baiza*

Murgh acts as *Muqawwi Baah* (Aphrodiasiac), when prepared with *Afyoon* , *Dhatura* then acts as *Mumsik* (Retention of semen by Making it thick.), when prepared with *Barg Paan, Adrak* then it acts as *Muqawwi Meda* (Tonic to stomach), when prepared with *Chobcheeni, Shahtarah* then it acts as *Musaffi Khoon* (Blood purifier), when prepared with *Hanzal* then it acts as *Qawi Mushil* (Purgative). **(Kareem BH and Ferozuddin CH, YNM)**

Very few reports are available where attempts have been made to understand the physico-chemical properties of bhasma. The literature reveals the need of scientific methods for assessing and maintaining the quality of this preparation. **(Dubey N et al, 2009)**

Aims of *Taklees* (Calcination)

1. To use the *lateef johar* of *dawa* for example. **(Maseehi I, Ynm; Hubal I, 2005)**
2. To remove undesirable parts of the drug, for example crab and stag horn are burnt to remove undesired fluids contain in them. **(Khan MS, 2006; (Maseehi I, Ynm)**
3. To make a hard drug soft enough to pound easily. **(Arzani H A, 1998; Hubal I, 2005; Rais S A, 1998)**
4. To remove toxic effect of drug, for example, scorpion and snake are burnt to remove their poison. **(Arzani H A, 1998; Hubal I, 2005; Rais S A, 1998)**
5. To decrease the intensity of the effect of a drug. **(Arzani H A, 1998; Hubal I, 2005; Rais S A, 1998)**
6. To increase the efficacy and potency of a drug. . **(Arzani H A, 1998; Hubal I, 2005; Rais S A, 1998)**

Advantages of *kushtajat*

1. Low dose, highly efficacious in action, **(Kabeeruddin HM, YNM)** and can be taken by the patient in diseases where the intake of large doses of medicine is not possible.
2. Use is easy as compared to other dosage forms. **(Kabeeruddin HM, YNM)**
3. Some *kushtajat* are unparalleled as aphrodisiac. **(Kabeeruddin HM, YNM)**
4. Most of the *kushtajat* stimulates innate heat of the body. **(Kabeeruddin HM, YNM)**

5. *Kushtajat* is more stable than other dosage forms. (**Kabeeruddin HM, YNM**) Shelf life of *Kushta* is believed to be infinite and they became more potent with the advent of time.
6. Only dosage form in which a bio-incompatible substance is made biocompatible. **(Kabeeruddin HM, YNM)**
7. Only dosage form of Unani system of medicine that can be given in acute conditions.
7. It is a dosage form which induces the change in temperament of the drug e.g. the temperament of *sadaf* is *sard wa khushk* but its *Kushta* has temperament *garm wa khushk.* **(Rehman Q A, 2003)**
8. All the *kushtajat* are very potent by their actions. This property of *kushtajat* is used to treat severe diseases. **(Qasmi, 2010)**
9. The older the *kushtajat* the better the effect. **Anonymous, 2006)**

General Precautions to be taken during Calcination Process

1. The weight of the drug mentioned in the formulation should always be followed and should not be changed. (**Kabeeruddin HM, YNM)**
2. If the intensity of heat used in preparation of *kushta* is not known then it should be experimentally determined starting from giving low intensity of heat then gradually increasing the intensity of heat. (**Kabeeruddin HM, YNM)**
3. While preparing *kushta* using classical method it is advised that half of the cow dung cakes are placed first in pit then followed by drug and then it is covered with remaining half of the cow dung cakes. While preparing *kushta* from modern method the sample of drug should be placed in crucible and directly kept in MF.
4. The pellet of triturated dry sample should be placed in center of the crucible.
5. The prepared *kushta* should be stored in airtight bottle not in any pouch made of paper. Since on size reduction, the surface area of a drug is increased. **(Gupta AK, 2009)** The surface area of *kushta* is more as compared to other dosage forms. So if *kushta* is not stored properly or due to any other reason is exposed to atmospheric conditions then is it is very easy for *kushta* to absorb moisture and degradation occurs in such *kushta*.
6. The most important aspect of calcinations is heating so it should be done very carefully. While preparing *kushta* from classical method, care of wind should be taken, as the place chosen for preparation of *kushta* should be *mehfooz ul hawa* (Protected from wind) as wind may cause uneven distribution of heat and result in incomplete preparation of *kushta*. While preparing *kushta* in muffle furnace one should not consider this point

because furnace itself is a closed chamber so this is one of the advantages of modern method preparation over classical method preparation of *kushta*. (**Kabeeruddin HM, YNM)**

7. While *kushta* preparation using classical method one should take care about *istelehat* (terminologies) like *gajput*, *kukatput* etc. **(Kabeeruddin HM, YNM)** While preparing *kushta* from modern method the important aspect is implementing the heat pattern received by using pyrometer and recording the thermogram and then implementing that pattern on furnace.
8. *Kushta* should be prepared by an experienced person only **(Kabeeruddin HM, YNM)** because there are some basics which should be followed during the preparation of *kushta* for example if a person is preparing *kushta* by classical method then one very basic thing is how to ignite the cow dung cakes. Only experienced person know that all the cow dung cakes should be burnt simultaneously from all directions. Heat should not be started from one side of the group of cake; they should be ignited from all the directions. So this very basic thing might alter the physicochemical properties of the prepared *kushta*.
9. The drug of which *kushta* is to be prepared should be of best quality and it is better to purify it. (**Kabeeruddin HM, YNM)** Here one thing should be kept in mind that it is advised to purify a drug before making its *kushta* but it is not a must or mandatory to do it.

   For example if one purchase a metal from a registered jeweler shop with ISI mark quality having 99.99 percent purity so in this case one should not wait for the detoxification. On the other hand if a metal let lead is purchased from ordinary dealer shop then this metal will definitely contains some impurities and it should be always subjected to the process of *tasfiya* or detoxification before making its *kushta*.

10. The weight of the drug mentioned in the formulation should always be followed and should never be changed. (**Kabeeruddin HM, YNM)**

Why let's take an example : In a formulation it is mentioned that for sake of preparation of *kushta sadaf* 60 gm *sadaf* is subjected to heat of 15 kg cow dung cakes, so that means that 15 kg cakes will give sufficient amount of heat to calcinize 60 gm of *sadaf*. If the person making *kushta* let increases the weight and took 70 gm of *sadaf*, now the heat of 15 kg cannot calcinize this 70 gm because the weight of *sadaf* is more than normal, so the end product will be *kushta kham* (Incomplete *kushta*).

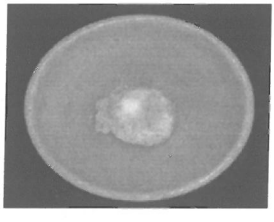
Now take the other case,

In a formulation it is mentioned that for sake of preparation of *kushta sadaf* 60 gm *sadaf* is subjected to heat of 15 kg cow dung cakes. If the person making *kushta* let decreases the weight and took 50 gm of *sadaf* , now the intensity of heat of 15 ser uplas is more than usual and it will result in overheating of *sadaf* and the end product will again be *kushta kham.*

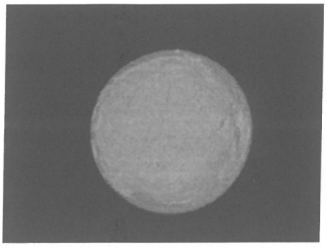
11. The herbs used in preparation should also be of good quality and there *murawwaq* should be used in preparation. **(Kabeeruddin HM, YNM)** When a drug is triturated in *murawwaq* of a *booti* then it should not be put in the fire until completely dry because the moisture will lead to boiling and may crack the *boota*. It is similar to cracking of *aatishi sheeshi* in oil extraction if the drug placed inside the *sheeshi* is wet. But if a person is preparing *kushta* in muffle furnace using silica crucible then this principle can be avoided because the boiling water can't crack the silica crucible.
12. Recently prepared *kushta* should never be used specially if it is prepared form toxic drugs. If needed then the *kushta* should be placed inside *namnaak*

zameen (wet soil) for 3-4 days and then it should be used. **(Kabeeruddin HM, YNM)** We also know that older the *kushta*jat the better the effect. **(Anonymous, 2006 ; Kabeeruddin HM, YNM)** It is written in the essentials of *Ayurveda* that the shelf life of *kushta* is infinite and the efficacy of *kushta* increases with the advent of time. But this point should be first proven scientifically then it should be believed. If the efficacy of a dosage form is increasing with the advancement of time that means the recently prepared *kushta* must have lesser efficacy as compared to a *kushta* which has been stored in an airtight bottle for 10 yrs. Until not scientifically validated these two points are contrasting each other.

13. It is said that the pellet of triturated dry sample should be placed in center of the crucible as seen in fig



But the preparation of *kushta* in this case depends upon pellet thickness as if the pellet thickness is more then on heating only the outer surface of the pellet will be white and the inner surface will be blackish or grayish depending upon formulation. But such cases can be modified as seen in the following figure.



In this technique the triturated sample is made adhered to the surface of the crucible and then dried in oven and then it is placed in MF as in this case

(a) The surface area will be more.

(b) The heat distribution will be more uniform and even.

But before going for preparation of *kushta*, analysis of raw metal or mineral should also be done because it gives a lot of information about that particular metal or mineral. It gives us detail about physicochemical constants of metal or mineral as well as the composition and impurities analysis by chemical methods as well as by AAS present in raw metal or mineral.

So by doing the physical and chemical analysis of raw metal or mineral one can use the data obtained for the sake of identification of the metal or mineral. Secondly as the quantitative analysis of the raw material also inform us about nature and quantity of impurity, so once these levels are estimated and recorded then these drugs are subjected to purification for the sake of preparation of *kushta* then the actual aim behind the process of purification can also be known. Because once analyzing the metal or mineral in non detoxified form and then comparing it with the detoxified form will give us very important and valuable information for the validation of the process of detoxification.

Difference between *Ihraq* and *Taklees*

IHRAQ (Ignition)

When a drug is burnt to the extent that it is reduced to ashes then it is regarded as *Ihraq*. The term *Ihraq* is a very broad term. If the drug is reduced to coal then also it is regarded as *Ihraq* and the substance left is called as *Muharak*.

When a drug is burnt to the extent that it resembles lime then it is regarded as *taklees* and the substance left is called as *Mukallas*.

Sometimes cow-dung cakes are used and sometimes furnaces are used in the processes of *Ihraq* and *Taklees*. **(Kabeeruddin HM, 2003)**

The term *'Taklees'* is taken from the word *'Kils'*, which means lime, It Is applied to a process by which a substance is converted in a form that looks like lime, known as *Mukallas* or *Kushta*. Technically, this term is applied to a process in which hard elements are made soft and converted into white powder. **(Ghani N, 2009)**

In Unani system of medicine, the term *kushta* (calcined product) is employed for a dosage form that is blend of metals, metallic oxides, non metals and their compounds and minerals, used in small quantity and one that is immediately effective. The ingredients are oxidized through the action of heat. The preparation of a *kushta* results in increase efficacy of a medicine, and after affecting its entry into the body, the *kushta* discharges its curative role promptly and effectively. **(Said HM, 1997; Ali M, YNM)** It is the finest powder form of the medicinal preparations obtained by calcination of metal, minerals and animal

origin drugs. *Kushta* is easily absorbed in human body and is highly efficacious in its action. **(Anonymous, 2006)**

Sometimes cow dung cakes and sometimes furnaces are used in the processes of *ihraq* and *taklees*. The process is conducted in presence of air or in tightly closed vessels according to the method mentioned in classical texts. **(Akhtar S, 2005)**

The process of *ihraq wa takless* is mentioned in most classical Unani text. *Ali bin Abbas Majoosi* in *Kamilus sana* **(Majoosi IA, 2010)** mentioned the method of *ihraq* of *qalai*, *bichcho*, *sapaid mohrah*, *sartan* and *abgenah* etc. *Ismail Jurjani* in *Zakhira khwarzam shahi* **(Jurjani AH, 2010)** described the process of *ihraq busd*, *sartan* and *bichcho*. In *Firdaus ul hikmat* **(Tabri R, 2002)** *Rabban Tabri* mentioned the action of ash of *seemab* and burnt crab. He also mentioned the temperament of *tamba muharraq* and *samandar jhag muharraq*. *Ihraq zarneekh and ihraq zaj* is mentioned in *Tohfatul momineen.* **(Momin HMM, Ynm)** Further it is described that the process eliminates toxicity of drugs. *Ihraq wa takless* of various drugs are also described in *Qarabadeen qadri*, **(Arzani H A, 1998)** *Khazain ul advia* **(Ghani N, 2011),** *Bayaaz khas*. **(Khan MS, 2006)** Thus it can be said that the terms were used as a synonym, at the place of each other.(**Kabeeruddin HM, YNM)**

Scope of Calcination

1. In the modern world reduction in quantity of dose is very important and special emphasis is always given to this. The objective is achievement of maximum effect with minimum dosage. This property is found only in *kushtajat*.
2. *Kushtajat* are the only dosage form which can be used in acute condition on USM due to its high bioavailability. So work on them should be encouraged and more done scientifically.
3. *Kushtajat* are usually used in serious illness or when the disease is acute. So this property of *kushta* separates them from other dosage forms.
4. It is the only dosage form which is concerned with elimination of all the debilities and problems as this is mentioned in the classics. As soon as it enters the body it affects the *hararat garizah* (innate heat) and the patient immediately feels active. Nagarjuna once said that he will free the entire world from all diseases by using *kushta paara*.
5. *Kushtajat* are also known as *shababawar* (aphrodiasiac). It means they have very high caliber as aphrodisiac that is why it is written in the classical literature that the efficacy of *kushtajat* is unparallel as aphrodisiac and even *Majoon* having lots of ingredients cannot compete with them.
6. Using *kushtajat* is very easy for the patient. Its intake is patient friendly.
7. In certain diseases like pharyngitis or laryngitis when the patient cannot take medicine easily even in this case he can take *kushta* very easily because its dose is very small.
8. The shelf life of *kushtajat* is supposed to be infinite and they became more and more potent with the advent of time whereas other dosage form becomes deteriorated with the advancement of time.

9. In the state of oxidation metal possesses very strong action upon body which is absent in metallic form. In this dosage form this metal is changed into its oxide form so that it could positively affect our body, this implies that it is the only dosage form in which a bio-incompatible substance is changes into biocompatible one.

10. Metals play an important role in human body, the deficiency of which leads to various disorders. In Ayurveda, seven metals such as gold, silver, copper iron, tin, lead and zinc are described as essential elements for the body. It has been described that metal based formulations, called *kushta*, are highly effective in prevention and cure of various diseases related to the organ where they are naturally found. It is written in *Rasaratan Samuchai* written by *Vagbhatt* and is also mentioned in *Miftah Ul Khazain.*

According to the table 1 given below **(Rasheed A et al, 2011)**

Table 1. Metals and their presence in human body

Metal	Effects on human body
Gold	Present in trace amounts in blood, semen, eyes, heart, upper layer of skin and intestines. Imbalance affects vision, causes general weakness in the body, dullness of intellect, loss of imagination power, voice and general disposition of an individual.
Silver	Present in bone marrow, upper layer of bones, gall bladder, pancreas, inner layer of the skin, lungs, flesh, blood vessels, meanings (membrane which surrounds the brain), audio receptive glands and septum of nose. Imbalance affects mind and disposition, neurological disturbances, problems related to teeth, cataract, sores, and absorption from gut.
Copper	Present in upper and inner layer of skin, mucosa of soft tissue, large glands, eye pupil, hair, pleura and pericardium. Imbalance causes defects in cardio-vascular, central nervous and skeletal system. Deficiency affects production or red blood cells and hair keratization. Imbalance causes chronic inflammatory disorders in soft tissues
Iron	Major constituent of blood, present in the villi of the Intestine, eye pupil, hair and in small quantity in all tissues of the body. Has special effect on elderly people Imbalance causes arteriolosclerosis, anemia and general debility.
Tin	Present in every tissue, however more in abdominal muscles, blood and blood vessels, synovial membrane, outer layer of uterus, Imbalance causes malformation in bones, diseases of reproductive tract, affects formation of urine, polyurea, increased perspiration.
Lead	Present in blood and lymphatic tissue. Imbalance causes anemia, disturbance in gastro-intestinal tract due to poor secretion of digestive juices, hemolytic anemia and ascitis.
Zinc	Present in Blood, brain, sensory tissues and flesh. Imbalance causes problems related to nervous system like despondency, anxiety, dullness of intellect, extreme forgetfulness, irritable temperament, somniloquism.

Gold is present in heart, silver is present in brain, copper is present in nerves, iron is present in liver and kidney, tin is present in urinary bladder, lead is present in intestine, zinc is present in eyes. **(Kareem BH, YNM)**

- On combining all three (Miftah Ul Khazain, Rasaratan Samuchai and journal of pharmacy research) one may come to the conclusion that in treating diseases related to heart *Kushta Tila* is highly effective medicine.
- In treating diseases related to brain *Kushta Nuqra* is highly effective medicine.
- In treating diseases related to eyes *Kushta Jast* is highly effective medicine.

Because these metal are naturally present at these sites.

11. It is believed that calcination process converts the metal into its specially desired chemical compound which eliminates the toxicity of the metal and has the necessary medicinal benefits. **(Kabeeruddin HM, YNM; Tripathi YB et al, 2003; Wadekar MP et al , 2005)**
12. It is the only dosage form which induces the change in temperament of the drug e.g. the temperament of *Sadaf* is *Sard Wa Khushk* but its *kushta* has temperament *Garm Wa Khushk*. (**Rehman Q A, 2003**)
13. It is the only dosage form which is concerned with rejuvenation, longitivity, virility, vitality and this concept is not theoretical only because *attibba* have described a *booti* named *somlata* which can got all these properties. **(Kareem BH, YNM)**
14. Work on alchemy is now read by a much wider audience and now is no longer confined to historicians.
15. The need of serious treatment is this subject is now felt more widely.

16. The search for the true understanding of alchemy is today related to the general quest on the part of many well intentioned people for rediscovery of tradition and recovery of harmonious relationship of man with nature.
17. Calcinations itself is so vast. It is the science of cosmos and souls, the process of spiritual realization and hence traditional psychology, medicine, metallurgy, chemistry and also art.

Maul Hayat

It's the technique by which *kushta* of metallic origin drug is rejuvenated resulting in obtainment of pure metal.

The three common formulas used as maul hayat are

1. *Shahed, Suhaga, Ghee.*

2. *Muqil, Rai, Gudh, Choti Machliyan, Bhed Ki Oon.*

3. *Muqil, Rai, Gudh, Shehad, Dodh.* **(Qasmi IA, 2009)**

Principle

- All living things contain carbon. Carbon is a non metallic element. The word is derived from latin word *carbo*, which means charcoal. The bodies of all organisms -plant or animal contains this element.

CHARCOAL

- It is made from wood by the process of destructive distillation. In destructive distillation of wood chemical changes occur at 350 degree and charcoal is formed.
- TYPES OF CHARCOAL
- Wood charcoal is commonly known as charcoal. On combustion of wood one gets wood charcoal which is commonly known as charcoal. Similarly on combustion of bone we get animal charcoal.
- On destructive distillation of sugar we get sugar charcoal.

- The charcoal obtained from sugar, being a very pure form of carbon is used for obtaining metals from their oxides.

$$ZnO + C \longrightarrow Zn + CO$$

It is the property of charcoal to adsorb the oxide from metal and yield pure metal again. **(Pant KM, 2003)**

In the above three formulas

- Wood charcoal will be the product of combustion of *Muqil, Rai.*
- Animal charcoal will be the product of combustion of *Choti Machliyan.*
- Sugar charcoal will be the product of combustion of *Shahed, Gudh.*

By combustion of all the drugs the end product will always be charcoal whether animal or plant or sugar in origin and this product charcoal is responsible for the conversion of metallic oxide into pure metal.

Our modern day periodic table is expanded beyond Mendeleev's initial 63 elements. Most of the current periodic tables include 108 or 109 elements.

It is also important to notice how the modern periodic table is arranged. The elements of what we now call a "period" were listed vertically by Mendeleev. Chemical "groups" are now shown vertically in contrast to their horizontal format in Mendeleev's table.

Note also that Mendeleev's 1871 arrangement was related to the atomic ratios in which elements formed oxides, binary compounds with oxygen; whereas today's periodic tables are arranged by increasing atomic numbers, that is, the number of protons a particular element contains. Although we can imply the formulas for oxides from today's periodic table, it is not explicitly stated as it was in Mendeleev's 1871 table. The oxides ratio column was not shown in earlier Mendeleev versions.

GROUPS

The modern periodic table of the elements contains 18 groups, or vertical columns. Elements in a group have similar chemical and physical properties because they have the same number of outer electrons. Elements in a group are like members of a family--each is different, but all are related by common characteristics.

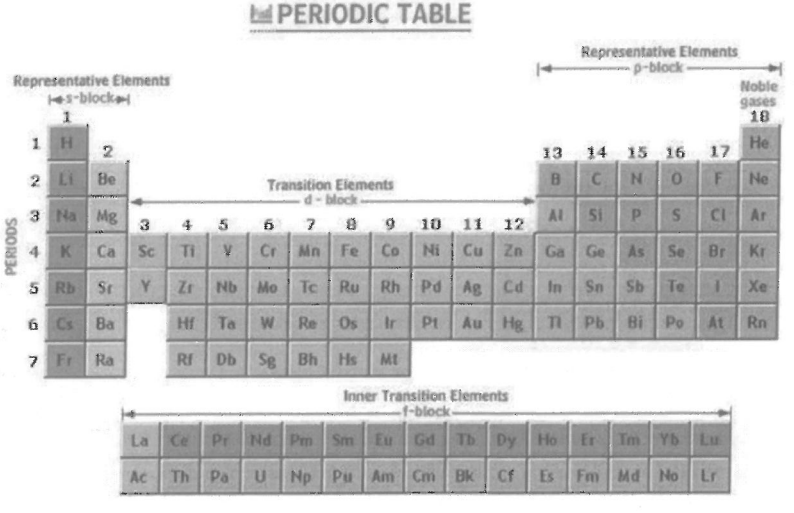
PERIODS

Each of the table's horizontal rows is called a period. Along a period, a gradual change in chemical properties occurs from one element to another. For example, metallic properties decrease and nonmetallic properties increase as you go from

left to right across a period. Changes in the properties occur because the number of protons and electrons increases from left to right across a period or row. The increase in number of electrons is important because the outer electrons determine the element's chemical properties.

The periodic table consists of seven periods. The periods vary in length. The first period is very short and contains only two elements, hydrogen and helium. The next two periods contain eight elements each. Periods four and five each have 18 elements. The sixth period has 32 elements. The last period is not complete yet because new exotic or man-made elements are still being made in laboratories.

CLASSIFICATION OF GENERAL PROPERTIES



The general properties of elements allow them to be divided into three classifications—metals, nonmetals and metalloids.

METALS

The vast majority of the known elements are metals. Many metals are easily recognized by non-chemists. Common examples are copper, lead, silver and gold. In general, metals have a luster, are quite dense and are good conductors of heat and electricity. They tend to be soft, malleable and ductile (meaning that they are easily shaped and can be drawn into fine wires without breaking).

All of these properties are directly related to the fact that solid metals are crystals formed from positive ions surrounded by mobile electrons. This mobility allows electrons to absorb and reflect light in many wavelengths, giving the metals their typical luster. It also permits electrons to absorb thermal and electrical energy from the environment or neighboring electrons and transfer this energy to other electrons; in this way, heat and electricity can be conducted throughout the metal.

These mobile electrons hold the positive metallic ions so tightly that even when the metal sample is only a few layers thick, as in gold foil, the sample stays intact. So, the density, malleability, and ductility of metals are also due to electron mobility.

The Group I Alkali Metals and the Group II Alkaline Earths have more metallic characteristics than elements farther right whose square are colored blue, especially those that border on the metalloid elements. Generally speaking, the most metallic metals are in the bottom left corner. As you move toward the upper right on the periodic table, elements become less metallic in property.

ALKALI METALS

Alkali metals are so chemically reactive that they are never found in the element form in nature. All these metals react spontaneously with gases in the air, so they must be kept immersed in oil in the storeroom. They are so soft that they can be cut with an ordinary table knife, revealing a very "buttery," silvery metal surface that

immediately turns dull as it reacts with water vapor and oxygen in the air. The chemical reactivity of alkali metals increases as the atomic number increases.

Their reactions with halogens, elements in Group VIIA, are especially spectacular because some of them emit both light and heat energy. They react with other nonmetals, forming compounds that are very stable. They also react with acids, forming hydrogen gas and salts; with water they form hydrogen gas and metallic hydroxides, which are sometimes called bases. They react with hydrogen to form metallic hydrides, which form strong bases in water. In all these reactions, the metals form ionic compounds, in which each metal atom loses one electron to form a positively-charged ion or cation.

All compounds of alkali metals are soluble in water. These compounds are widely distributed. Large mineral deposits of relatively pure compounds of sodium and potassium are found in many parts of the world. Sodium and potassium chlorides are among the most abundant compounds in sea water. Potassium compounds are found in all plants and sodium and potassium compounds are essential to animal life—including human life.

ALKALINE EARTH METALS

The alkaline earth (IIA) metals exhibit the typical metal characteristics of high density, metallic luster and electrical and thermal conductivity. Rocks and minerals containing silica, magnesium, and calcium compounds are widely distributed. These chemicals are also abundant as compounds in sea water. Their chlorides are abundant in sea water. Radium, the largest of the alkaline earths, is a radioactive element that occurs naturally only in very small quantities. Chlorophyll, the green coloring in plants, is a magnesium-containing compound. Calcium is a major component of animal bones, teeth and nerve cells.

The chemical reactivity of these elements increases with size. Calcium, strontium, and barium react with water forming hydrogen and alkaline compounds. Magnesium reacts with steam to produce magnesium oxide. Common oxides of alkaline earth metals include lime (CaO) and magnesia (MgO), which react with water to produce strongly alkaline solutions.

TRANSITION METALS

The transition (or heavy) metals have most of the usual properties of metals. Their densities are greater than the other metals Transition metals are also called heavy metals because their atoms are relatively small and their large numbers of protons and neutrons give them relatively large masses. There is a great variance in the chemical reactivity of transition metals. All the transition elements react with halogens and most react with sulfur and oxygen.

Copper, silver and gold are sometimes known as coinage metals because they can be found naturally in the free state and because they tarnish slowly. Since prehistoric times, they have been used in coins, utensils, weapons, and jewelry.

Although many transition metals have very high melting and boiling points, mercury (Hg) has such a low melting point that it is a liquid at room temperature.

All the transition metals are electrical conductors, with copper, silver and gold being among the best; they vary from very good to only fairly good thermal conductors.

Many heavy metal compounds, such as those of mercury, cadmium, zinc, chromium and copper, are poisonous. When transition metal ions are present in even small percentages in crystalline silicates or alumina, the minerals become gems. Rubies are gems in which small numbers of chromium ions are substituted

for aluminum ions in aluminum oxide. Chromium substitution for a small number of aluminum ions in another clear crystal, beryllium aluminum silicate, forms the green gem known as emerald.

RARE EARTH METALS

The rare earth metals consist of the lanthanide series and the actinide series. Because they are difficult to find, they are termed rare earths. They often appear to be an add-on to the rest of the periodic table, but actually, they should be shown in the center of the table. Uranium (U) is the most well-known naturally occurring member of this group of elements. Mendelevium (Md),element number 101, is named for Dmitri Mendeleev, the Russian chemist who first arranged the elements in a table in order of increasing atomic mass.

OTHER METALS

Other metals include heavier elements of Groups IIIA, IVA, and VA. They form a staircase inside the periodic table. The metals in Group IIIA are aluminum (Al), gallium (Ga), indium (In), and thallium (Tl). The metals in Group IVA are tin (Sn) and lead (Pb), and the only metal in Group VA is bismuth (Bi).As atomic number decreases within each group, their metallic character gets weaker.

METALLOIDS

The metalloids include boron (B), silicon (Si) and germanium (Ge), arsenic (As) and antimony (Sb), tellurium (Te) and polonium, (Po). Note that they are arranged in stair steps between the metals and nonmetals.

Metalloids have some of the properties of metals and nonmetals—and each metalloid has its own unique mixture. A few are shiny like metals, but do not really have a metallic luster. Some metalloids have very high melting and boiling points; others do not. Others conduct electricity—but their electrons are mobile in only certain directions, so they are called semi-conductors. This makes them useful in designing transistors and other solid state electronic components.

NONMETALS

The nonmetallic elements are in the upper right portion of the Periodic Table. At room temperature and pressure, many of them exist as gases, but one is a liquid. Others are either the hardest or the softest of solids. The nonmetals have few chemical properties in common. They range from fluorine, the most active nonmetal, to the most nonreactive of the elements, the noble gases. Millions of compounds formed from carbon, hydrogen, oxygen, sulfur and nitrogen are known as organic chemicals.

Oxides of sulfur and nitrogen have been identified as atmospheric pollutants. Nonmetallic compounds also include salts as well as many acids and bases. Many of these salts are found in soil or dissolved in ocean water. Any ions formed by nonmetals are negatively charged. Almost eighty percent of our atmosphere is made up of nitrogen gas and most of the rest is oxygen, which is necessary for human respiration and metabolism. There are negligible amounts of noble gases in our atmosphere.

Many of the nonmetals are colored, including yellow sulfur, red and yellow phosphorus, yellow-green fluorine, pale yellow chlorine, red-brown bromine, and violet-black iodine. Others, like oxygen, nitrogen, and the noble gases are colorless. Only sulfur is found as a free element in nature.

Physical properties of Metal and Mineral

1. **Nature**
2. **Colour**
3. **Streak**
4. **Cleavage or parting**
5. **Fracture**
6. **Lusture**
7. **Tenacity**
8. **Transparency**
9. **Magnetism**
10. **Hardness**
11. **Fluorescence**
12. **XRD analysis**

1. NATURE

Being natural chemical compounds, minerals may occur in any aggregation state, though most of them are known to belong to solid crystalline substances. Amorphous minerals are scarce. As such, natural occurrence of minerals may be in the following forms-

i) Crystalline (columnar, bladed, fibrous and tabular etc.)

ii) Amorphous (massive, earthy and resinous etc.)

iii) Opaque, transparent and translucent

iv) Aggregate (granular, incrusted, micaceous etc.)

1.1 State of Aggregation

Under favorable conditions, minerals assume a definite crystal form. The following crystal forms are known-

a. Crystallized: A term denoting that the mineral occurs as well developed crystals. Most of the beautiful mineral specimens in museums are of crystallized minerals.

b. Crystalline: A term denoting that no definite crystals are developed, but that a confused aggregate of imperfect crystal grains have formed interfering with one another during their growth.

c. Crypto-crystalline: A term denoting that the mineral possesses traces of crystalline structure.

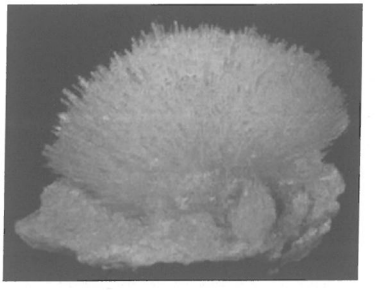
d. Amorphous: A term used to describe the complete absence of crystalline structure, a condition common in natural rock glasses, but rare in minerals.

1.2. Crystal Habit

The development of an individual crystal or an aggregate of crystals to produce a particular external shape is described as its habit and this depends upon the conditions during formation.

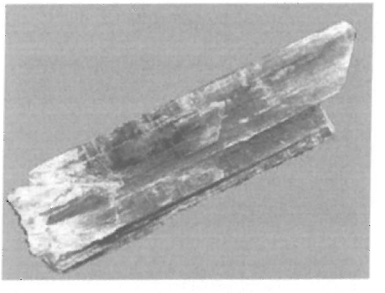
A. Individual crystals

a. Acicular:



Fine needle like crystals

b. Bladed:

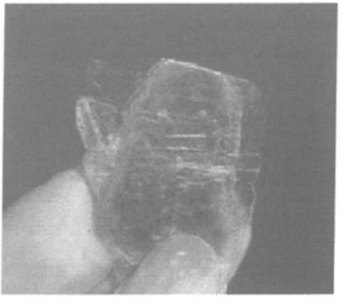


Shaped like a knife blade

c. Fibrous

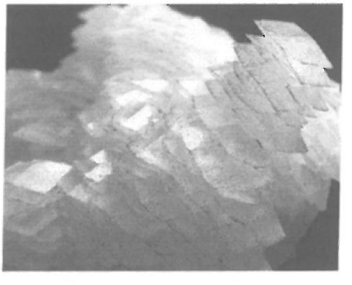
Consisting of tine thread like strands

d. Foliated



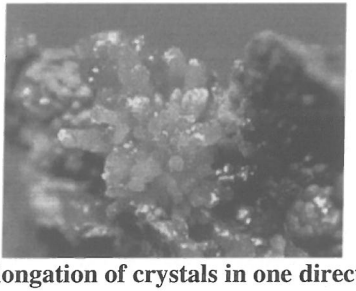
Consisting of thin and separate lamellae or leaves as is shown by mica

e. Lamellar



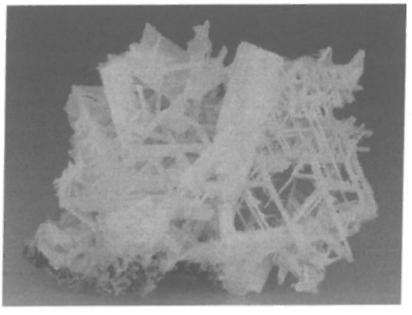
Consisting of separate plates or leaves as in wollastonite

f. Prismatic



Elongation of crystals in one direction

g. Reticulated

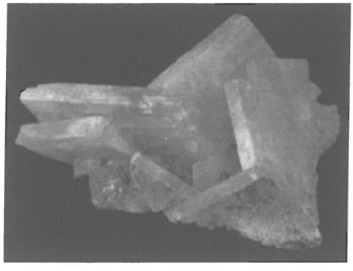


Crystal in a cross-mesh pattern, like a net

h. Scaly

In small plates

i. Tabular

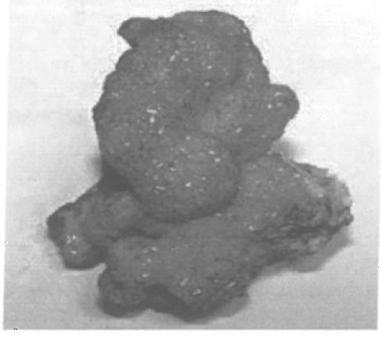


Broad, flat, thin crystals

B. Crystal Aggregates

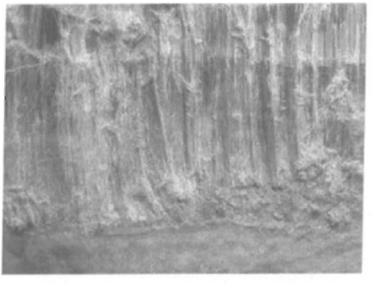
There may be aggregates of the crystals of which individuals can be seen with the naked eye or massive aggregates of minerals in which individual crystals are too small to be seen with the naked eye.

a. Botryoidal



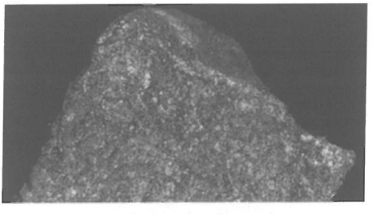
Spherical aggregates resembling a bunch of grapes

b. Columnar



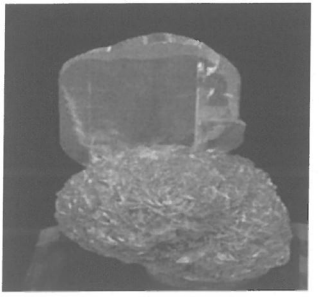
Massive aggregations in slender columns

c. Granular



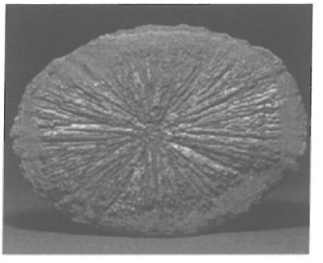
Coarse or fine grains

d. Lenticular



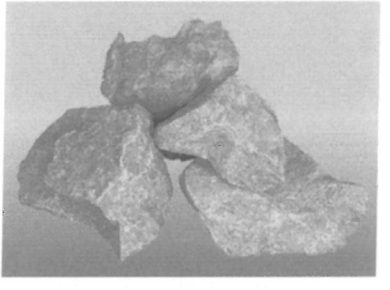
Flattened balls or pellets shown by many concretionary and nodular minerals

e. Radiating or divergent



Fibers arranged around a central point

f. Lump



An uneven mass / piece of mineral / ore /rock

2. COLOUR

A mineral gives a constant distinctive colour, when observed on freshly broken surface. Though this property is not clear in many minerals, commercial minerals, by and large, are distinguished by their typical colour.

3. STREAK

The tine powder of a mineral shows more constant colouration than the same mineral in a massive specimen. This phenomenon is used for identification of a mineral by rubbing the mineral on a rough surface. A white unglazed porcelain plate commonly known as streak plate is generally used to see the colour of the powder of a mineral, Streak is a more reliable distinguishing feature of minerals with a semi-metallic and sub-metallic luster.

Method of determination

Take sample in question and a streak plate then draw a line or rub across the white surface of the streak plate with the sample. Now observe the colour of the powder left by the trail. This colour is the streak of that particular mineral.

Tools: Streak Plate

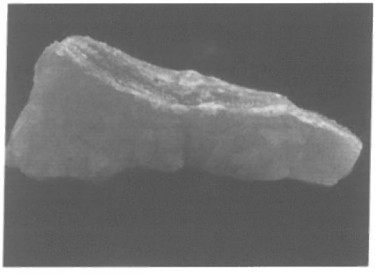
4. CLEAVAGE OR PARTING

Cleavage or parting is the tendency of minerals to split along certain definite planes. The cleavage plane is closely related to crystalline form and internal atomic structure and therefore is generally parallel to crystal faces. Minerals may show several cleavages, which are described by stating the crystallographic directions of each cleavage and also the degree of perfection of each cleavage plane. Cleavage may be described in order of quality as under:

a. Perfect or eminent

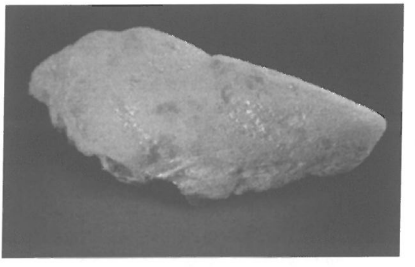
Cleavage planes are seen perfectly in the minerals

b. Good or distinct



Cleavage planes can be seen with little effort

c. Poor or indistinct



Cleavage planes indistinctly visible

Method of determination

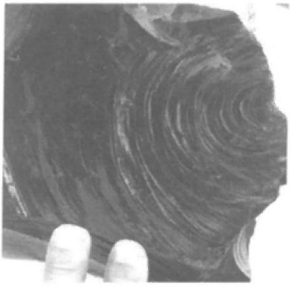
Take the mineral and break it with a hammer gently. If the mineral separates along parallel planes each time when it is repeatedly broken, it is said to possess cleavage. Observe the quality of the cleavage as defined above. Observe some minute step—like planes or a wavy pattern on the surface of the mineral. Smooth parallel cracks or directional cracks are indication of cleavage direction.

Tools: Small (about % kg) hammer.

5. FRACTURE

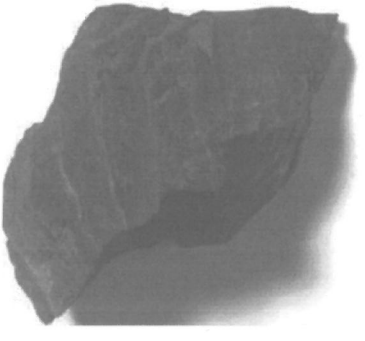
The character of the fracture displayed on the broken or chipped surfaces of a mineral is an important property. The fracture surface is not the smooth surface of a cleavage plane but is an irregular surface, usually totally independent of cleavage. The types of fractures, known are given here.

a. Conchoidal



The mineral breaks with a curved concave or convex fracture. This often shows concentric and gradually diminishing undulations towards the point of impact, somewhat resemble the growth lines on a seashell.

b. Even

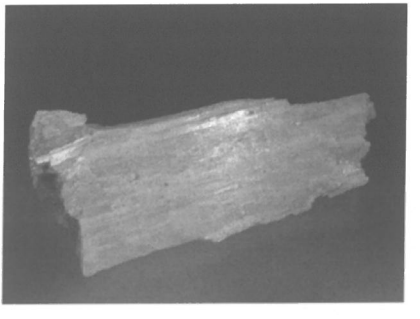


The fractured surface is flat.

c. Uneven

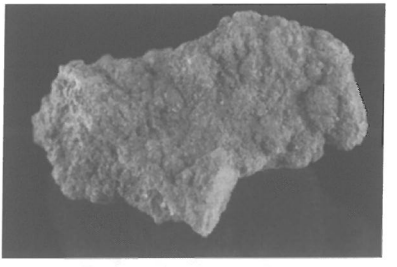
The fractured surface is rough by reason of minute elevations and depressions. Most minerals have an uneven fracture.

d. Hackly



The broken edge shows sharp and jagged projections / elevations like serrations.

e. Earthy



The dull fractured surface of chalk or clay minerals.

Method of determination

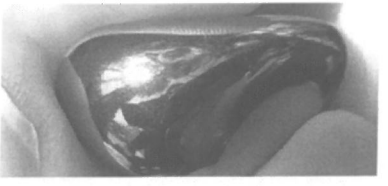
Take the mineral and break it with hammer in two pieces in such a way that it does not get shattered. Observe the broken surface (other than the cleavage plane/direction) and decide the type of fracture as defined above.

Tools: Small (about % kg) hammer.

6. LUSTURE

Lusture is one of the most regular and easily observable properties of a mineral. As a rule, a mineral reveals, at first glance, its lusture which is produced by the light reflected or refracted from the faces of crystals, cleavage planes and from freshly fractured faces of the mineral. Lusture is directly dependent on the light reflected from the surface.

Splendent



Reflects objects distinctly like a mirror

Shining

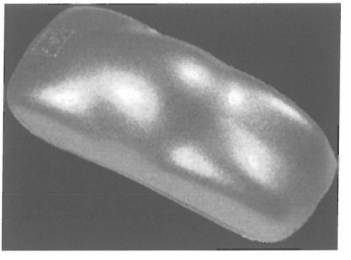
Objects are reflected indistinctly

Dull

When the surface has no reflection or refraction

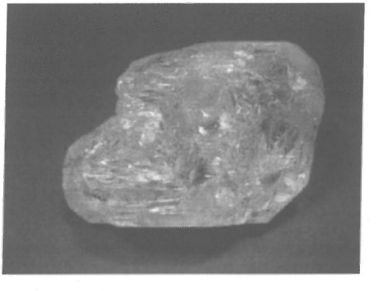
Various types of luster are:

a. Metallic:



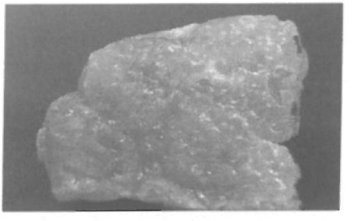
This is the ordinary lusture of metals. When feebly displayed, this type of lusture is termed sub- metallic and when not displayed at all, is termed as dull Based on the intensity and degree of reflection frefraction, metallic lustre is termed as Metallic, Sub-Metallic, Dull and Non-Metallic.

b. Vitreous



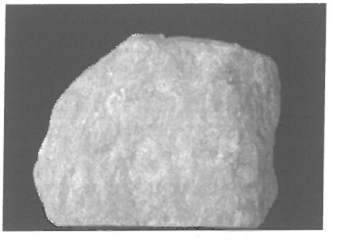
The lusture of broken glass, when less well developed, it is termed sub-vitreous and when not developed at all is dull

c. Resinous



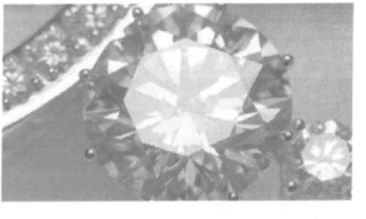
The lusture of resin

d. Pearly



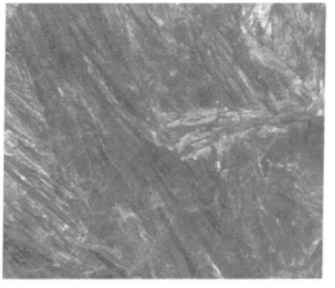
The lustre of Pearl, it is shown by the surfaces parallel to which the mineral is separated in to thin plates, similar to the condition of a pile of thin glass sheets such as cover glasses on microscopic slides.

e. Adamantine



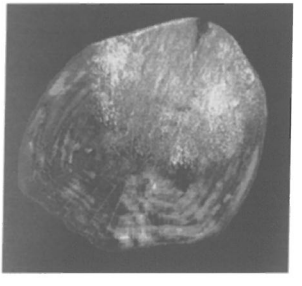
The lusture of Diamond

f. Silky



The lusture of silk, this lusture is peculiar to minerals possessing a fibrous structure.

g. Iridescence



Play of colours of rainbow, as those formed in soap bubbles due to interference of light waves.

Method of determination

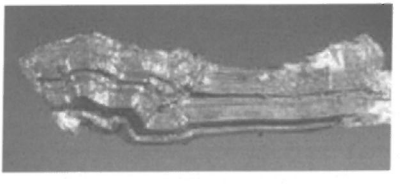
Observe the quality of natural light reflected on the faces of crystals, cleavage planes or fresh fracture of a mineral then decide the type of lustre as defined above.

Tools: Visual observa

7. TENACITY

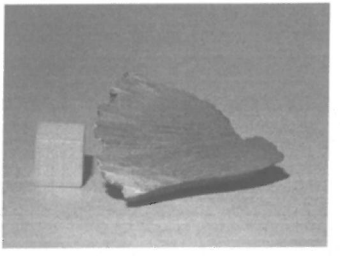
This is a measure of how a mineral deforms when it is crushed or bent. In fact, tenacity or tensile strength is the resistance that a mineral offers to mechanical deformation. In other words, it is the resistance that the atoms or ions of a substance offer to being subjected to processes that tend to cause bending, breaking, crushing or cutting. Tenacity is termed as follows:-

a. Sectile



The mineral can be cut with a knife and the resulting slice breaks up under a hammer.

b. Malleable

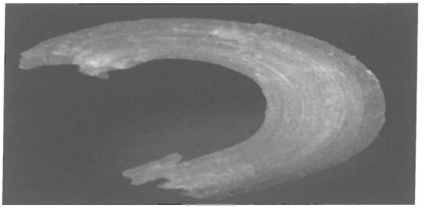


A slice cut from the mineral/metal can be hammered into thin flat sheets.

c. Brittle

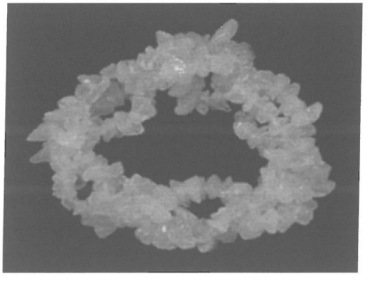
The mineral crumbles or shatters easily

d. Flexible



The mineral or thin plates or laminae of the mineral can be bent, remains bent and does not return to its original position even if that pressure is removed.

e. Elastic



The mineral or thin plates or laminae of the mineral can be bent and returns to its original position alter the pressure is removed.

f. Ductile: The mineral can be drawn out in to thin wires.

Method of determination

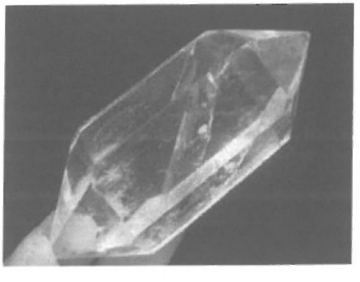
Take a hammer and hit the substance under question. Repeat the process several times till the substance breaks into pieces, this will change its shape, either will be bent or will be crushed. Observe the type of deformation (Tenacity) as defined above.

Tools: Small (about % kg) hammer.

8. LIGHT TRANSMISSION (TRANSPARENCY)

Transparency is a property of the substance to transmit light. In other word; the ability of a substance, to allow light to pass through it, is called transparency (Diaphaneity). Based on their capability and varying degrees of Transparency. The materials are said:

Transparent



i.e. materials capable of transmitting light and through which an object can be seen easily with its sharp, clear and distinct outlines.

Translucent

i.e. materials capable of transmitting light but through which an object cannot be seen, except an outline, generally distorted and blurred.

Opaque i.e. materials that are incapable of transmitting light.

Method of determination

Take the sample and observe whether one can see through it or not. Then decide the degree of transparency as defined above.

Tools: Visual observation

9. MAGNETISM

A few minerals / metals, in their natural state, are capable of being attracted by a hand magnet. They are called magnetic. In other words, if a mineral / metal gets attracted by a horse-shoe magnet or deflects the needle of a compass, it is said to possess magnetism. A substance may be strongly magnetic, weakly magnetic or non- magnetic.

Method of determination

There are two methods to know whether a substance is magnetic.

1. Take a simple horse—shoe magnet. Bring the mineral near both the ends of magnet and feel whether the mineral is attracted by the magnet or vice-versa. If attraction is felt, the mineral is said to be magnetic.

2. Take a compass. Bring the sample under question near the compass. If the needle of the compass deflects due to placement of the sample around the compass, the sample is said to be magnetic.

Feel the degree of magnetism of the substance and decide whether it is non-magnetic (no attraction at all), weakly magnetic (little attraction felt) and strongly magnetic (strong attraction felt).

Tools: Simple horse-shoe magnet or Compass

Horse Shoe Magnet: A horse shoe magnet is a magnetized small iron bar shaped as horse shoe. This has a property of attracting the substances possessing magnetism.

Compass: A compass is usually a rectangular (7.5 cm X 7.5 cm) or circular (8.5 cm diameter) small box having about 2 cm height with a glass cover on it. A magnetized needle is pivoted at the centre inside the box in such a way that it rotates freely in all directions. The compass also has a dial marked on it and graduated from 0^0 to 360^0 with indication of directions (i.e. 0° - North, 90^0 — East, 180°-South and 270^0 - West). The needle always indicates North - South direction, when it is at rest. To identify North Pole of the needle that end indicating north is usually painted in different colour. The magnetic needle shows deflection towards the substances possessing magnetism, when brought around the compass.

10. HARDNESS

Hardness of a mineral is its resistance to scratching. It is a relative parameter. In mineralogy relative hardness is always determined by using a set of minerals

known as the Moh's Scale of Hardness. This scale consists of 10 reference minerals, each of which is assigned a number in order of increasing hardness from 1 (the lowest hardness, that of Talc) to 10 (the highest hardness, that of Diamond). The Moh's Scale of Hardness, with reference minerals, is given here.

Moh's Scale of Hardness

Hardness	Mineral
1	Talc
2	Gypsum
3	Calcite
4	Fluorite
5	Apatite
6	Orthoclase
7	Quartz
8	Topaz
9	Corundum
10	Diamond

Method of determination

Take the mineral and select a smooth/clear surface. Scratch this smooth surface firmly with sharp edge of a reference mineral, specified in Moh's Scale of Hardness. Observe, whether this reference mineral can scratch the mineral under question. If not, take another reference mineral with higher hardness and observe

the scratch again. Repeat the scratching process with reference minerals in increasing order of hardness, one by one, to determine nearer to correct hardness of the mineral under question.

Tools: Set of reference minerals of Moh's Scale of Hardness (Reference minerals can be obtained from scientific stores dealing with geological instruments).

Important

1. Always start scratching with softer reference mineral and proceed upward one by one with reference mineral until just a visible scratch is seen.

2. Examine the scratch with a hand lens to ensure that it is a scratch and not the powder form the reference mineral, If it is a scratch, rubbing would not make it disappear, whereas if only a sneak, it can be rubbed off.

11. SPECIFIC GRAVITY (DENSITY)

Specific gravity is the relative weight of a mineral compared to that of the weight of an equal volume of purified water. The weight of an equal volume of purified water is the same as the loss of weight of the mineral in purified water. Specific gravity is calculated by dividing the weight of the mineral in air by the loss of weight in purified water. The specific gravity of natural minerals is known to vary from 0.8 to 21.

Method of determination

There are several methods of determining specific gravity namely (1) By normal chemical balance (2) By Jolly's spring balance (3) By Walker's steelyard balance (4) By using pycnometer and (5) By using liquids of high density. The first method, which is quite simple, is being dealt with here:

Determination of specific gravity with the chemical balance

Weigh accurately a small piece of mineral under question in air on a good, non-digital chemical balance of sensitivity 0.001 g and record the weight (w_1). Then put a beaker half filled with purified water on a wooden bridge placed over the scale pan of the balance. Suspend the mineral by a horse-hair or a silk thread from one arm of the balance in the beaker filled with purified water. Now, weigh the mineral, immersed in purified water and record the weight (w_2).

The specific gravity of the mineral is determined by the formula

$$\frac{\text{Weight of mineral in air } (w_1)}{\text{Loss of weight of mineral in purified water } (w_1 - w_2)}$$

Tools: Chemical balance of 0.001 g sensitivity, beaker, wooden bridge, horse-hair or silk thread and puryied water.

Geological Survey of India, western region, Jaipur has a specific gravity measuring kit, which calculates the specilic gravity and gives its value straightway.

12. FLUORESCENCE

This is the property of substances, which emit light when subjected to irradiation with ultra-violet (UV), cathode or other short wave rays. Such luminescence remains only till the substance is kept under irradiation. It is a characteristic feature of some minerals.

Ultra—violet radiation has a range of wavelength from 200 nm to 400 nm. The range from 200 nm to 300 nm is called short wave (or far UV rays) and from 300

to 400 nm is called long wave (or near UV rays). Short wave UV lamp has been used in present determinations.

Method of determination

Place the sample in dark viewing cabinet carrying the UV source of radiation, irradiate for about 5 minutes. Then observe if there is any fluorescence visible. Turn the sample in different directions with observation. The sample is positive, if under these conditions a bright colour different from the original colour of sample develops under irradiation and disappears if source of irradiation is removed.

Tools: Ultra—violet (UV) lamp of 200 to 400 nm.

13. XRD ANALYSIS

X · ray diffraction is one of the methods which can be employed to identify the crystalline matters such as minerals / metals and inorganic / organic compounds. It is more useful in mixture or intergrowth of minerals including species identification by identifying its crystal structure and cell size.

Principle of X-Ray Diffraction

A beam of X - rays when strike a crystalline material or mineral, it causes electrons in its path to vibrate and scatter. Such X - rays when reinforce each other, the process is called diffraction. The diffracted X - rays strike equally spaced parallel rows of identical atoms or planes and behave as if they have been reflected from planes with in a crystal. Diffraction takes place from parallel (atomic) planes only, if they satisfy certain conditions simplified in Bragg's equation / law viz;

$n\lambda = 2d \sin\theta$

The diffraction effect is not from a single or a few such atomic planes but from almost infinite number of planes each reinforcing and contributing to increase intensity (of a particular plane or face) for recording.

The X - rays are collimated and allowed to fall on the powdered sample. A goniometer and a detector are placed in the diffracted path of the X – rays synchronized to measure the angle and detect the signals simultaneously. The signal (counts per second) is processed electronically and converted to digital mode on panel and simultaneously move the recorder pen to produce a chart / diffractogram in a synchronized chart drive. Each face of a crystal thus would be represented by a peak. The diffractometre would provide a recorded history of 2 Theta angles vis-a—vis the peaks (faces of crystals) as per their atomic configuration. Alternatively, the whole data can be stored in a computer and can be conveniently retrieved later.

Each mineral has its characteristic atomic configuration and thus a characteristic set of faces/peaks. Most of the minerals with a few exceptions have unique'd' spacings and could be interpreted and identified by using certain parameters and comparison with published standard data cards (ICDD Cards or international Center for Diffraction Data Cards) which are used as reference patients.

Chemical Analysis of Metal or Mineral Samples (Gravimetric -- Wet Analysis Method)

1. Preparation of powdered sample

Prepare finely pulverized homogenous (-) 200 # (mesh) size powdered sample by taking proper care.

The conversion of an ore / rock sample into desired soluble or insoluble species is most important. The method constitutes the fusion of sample rock with solid fluxes to convert complex mineral compound into simple chemical compound. Silicate rock samples are generally analyzed for LOI (Loss of Ignition), SiO_2, Fe_2O_3, Al_2O_3, CaO, MgO, TiO_2, Na_2O and K_2O.

Dry the sample at 110^0 in oven for 1 hour before analysis. Cone and quarter the dried sample and preserve half quantity for future reference.

2. Determination of Loss on Ignition (LOI)

Weigh 1.0 g finely powdered sample in platinum/silica crucible then heat to constant weight in electric muffle furnace at $950\text{-}1000^0$ for an hour and allow to cool and weigh.

$$\text{LOI \%} = \frac{W3-W2}{W2 \cdot W1} \times 100$$

W1 = weight of empty crucible

W2 = weight of crucible + sample

W3 = weight of crucible + sample after ignition

3. Determination of Silica (SiO2)

Procedure

Weigh 0.5 g (in case of high silica) or 1.0 g (low silica) finely powdered and dried sample in a platinum/silica crucible (Wt). Add 3-5 gm anhydrous sodium carbonate into the crucible, mix thoroughly and cover the crucible with lid, if necessary place the crucible in muffle furnace, allow the temperature to rise gradually to reach 900-950 degree and keep on this temp for about half hour to complete the fusion. Take out the crucible and allow cooling at room temperature. Extract the cooled mass in 25-30 ml dilute hydrochloric acid in 250 ml beaker followed by heating on hot plate/burner to dissolve the contents. Wash the crucible with purified water and keep the beaker on purified water bath. Allow to dry the mass to make powder.

Take out the beaker and allow cooling at room temp and adding 25-30 ml dilute hydrochloric acid diluting upto 100 ml with purified water. Boil the content and allow cooling then filter through whatman's's 40 No. filter paper. Wash the residue with hot purified water 6-8 times then place the residue along with filter paper in platinum crucible. Ignite at 900- 950° for 2-3 min. Allow to cool and weigh (W2).

$$\% \ SiO_2 = \frac{\text{Initial Wt (W1) - Final Wt (W2)}}{\text{Weight of sample (W1) X 100}}$$

Note: Fuse the residue after hydro-fluorization with 2-3 g of potassium pyrosulphate on burner and allow to cool and extract the mass with dilute hydrochloric acid in original filtrate of the sample. Boil to dissolve the residue, if any. Cool the content at room temperature and make up the volume of filtrate to

desired volume in 250 ml volumetric flask. Use the stock solution for the determination of other additional radicals as required in the sample.

4. Determination of Iron (Fe)

Solutions

• Stannous chloride solutions- Dissolve 5.00 mg $SnCl_2$ (A.R.) in 25 ml hydrochloric acid and dilute to 100 ml (5% solution).

• Mercuric chloride » Saturated solution in purified water,

• Sulphuric acid + ortho-phosphoric acid mixture — take 60 ml purified water, add 15 ml Sulphuric acid and I5 ml phosphoric acid cool and dilute to 1000 ml.

• Diphenyleamine barium sulphonate - Dissolve 0.25 g in 100 ml purified water.

• 0.1N standard potassium dichromate solution. Dissolve 4.9035 g A.R. grade in purified water and dilute to 1000 ml.

Procedure

Take a suitable aliquot from the stock solution in 250ml-conical flask in duplicate and dilute to about 100 ml with purified water and add 1-2 drops of methyl red indicator followed by 1-2 g of ammonium chloride and dilute ammonia solution was added till brown precipitate appears and solution with precipitate is boiled for 4-5 minutes then the content cooled and filtered through whatman's's 41 No. filter paper. Wash the residue with hot purified water 4-6 times. Dissolve the residue in dilute hydrochloric acid in 250 ml beaker and make the volume upto 100 ml approx, then boil the solution on burner to reduce the Fe^{+3} to Fe^{+2} by adding stannous chloride solution drop wise till solution becomes colorless. Add 1-2 drops stannous chloride in excess and cool the content in purified water, then add 10-15

ml 10% solution of mercuric chloride, 25ml acid mixture and 2-3 drops of diphenyl amine barium sulphonate indicator. Add purified water, if required and titrate against standard potassium dichromate solution; appearance of violet color shows end point.

Calculation: 1 ml 1N $K_2Cr_2O_7$ = 0.05585 g Fe

= 0.7985 g Fe_2O_3

$$\% \text{ Fe} = \frac{0.05585 \times \text{normality of } K_2Cr_2O_7 \times \text{aliquot} \times \text{ml } K_2Cr_2O_7 \times 100}{\text{Weight of sample} \times \text{total volume}}$$

5. Determination of Calcium Oxide (CaO-EDTA) – Complexometric method)

Solutions:

• 20% potassium hydroxide solution — Dissolve 200 g potassium hydroxide (A.R.) in purified water and make upto 1000 ml.

• Ammonia buffer solution 9.5 pH- Dissolve 67.5 g NH_4Cl (A.R.) in 300 ml purified water, add 570 ml ammonia solution (Analytical Reagent) and dilute to 1000 ml.

• Ethylene diamine tetra acetic acid (EDTA) solution 0.05 M- dissolve 18.6120 g of sodium salt of Ethylene diamine tetra acetic acid (EDTA) (Analytical Reagent) in purified water and make up to 1000 ml.

• Triethanolamine 20% solution — 200 ml of triethonolamine, add 800 ml purified water and make up to 1000 ml.

• Eriochrome Black T indicator 0.1% solution — Dissolve 0.10 g indicators in 100 ml of methanol.

• Patterns and Reeders indicators 0.1% solution — Dissolve 0.10 g indicators in 100 ml of methanol.

Procedure

Take one part of filtrate reserved from iron estimation, add 5 ml triethanoiamine 20% solution and a pinch of hydroxylamine hydrochloride then add 25-30 ml potassium hydroxide 20% solution. Addition of 4-5 drops of Patterns and Reeders indicator, imparts rose-red color. Titrate the solution against standard ethylene diamineterraacetic acid (EDTA) solution. The color changes from rose- red to Prussian blue indicates end point.

Calculation: 1 ml of 1M EDTA = 0.05608 g CaO

$$Cao = \frac{0.5608 \times \text{normality EDTA} \times \text{Aliquot} \times 100 \times \text{ml EDTA}}{\text{Weight of sample} \times \text{total volume}}$$

6. Determination of Magnesium Oxide (MgO-EDTA) - Complexometric method

Take another part of filtrate resented from Fe estimation; add 5ml triethanolamine solution and a pinch of hydroxylamine hydrochloride followed by 25-30 ml ammonia buffer (9.5 pH) and 4-5 drops of Eriochrome black- T indicator. Titrate

against standard ethylene diaminetetraacetic acid (EDTA) solution appearance of rose- red to blue marks indicates the end point.

Calculation- 1ml of 1M EDTA = 0.0409 g MgO

$$\% \text{ MgO} = \frac{0.0409 \times \text{normality EDTA} \times \text{aliquot} \times 100 \times \text{ml EDTA}}{\text{Weight of sample} \times \text{total volume}}$$

7. Determination of Alumina (Al_2O;-EDTA) - Complexometric method

Solutions:

• 10% sodium hydroxide solution — dissolve 100 g sodium hydroxide in 100 ml purified water.

• EDTA solution 0.05 M- dissolve 18.6120 g of sodium salt of EDTA (A.R.) in purified water and make up to 1000 ml.

• Zinc acetate solution 0.05M- dissolve 10.9690 g of zinc acetate (AR.) in 50 ml purified water and a few drops of glacial acetic acid and dilute to 1000 ml.

• Acetate buffer 5.5 pH- dissolve 21.5 g of sodium acetate (Analytical Reagent) in 300 ml purified water containing 2 ml glacial acetic acid and dilute to 1000 ml.

• Xylenol orange indicator — dissolve 0.2 g of xylenol orange indicator in 100 ml purified water with 2 ml acetic acid.

Procedure

Take suitable aliquot from the stock solution in 250 ml-beakers. Take 50 ml of 10% sodium hydroxide solution in another beaker neutralize the aliquot with sodium hydroxide solution and transfer the 10% sodium hydroxide solution to aliquot with constant stirring add a pinch of sodium carbonate into the solution. Boil the content on burner, cool and filter through whatman's 40 No. filter paper with pulp in 600 ml beaker, wash the precipitate with hot purified water 6-8 times then acidify the filtrate with dilute hydrochloric acid and adjust pH 5.5, normally 25 ml 0.05M ethylene diamine tetra acetic acid (EDTA) solution add in excess and 25 ml of acetate buffer solution boil, cool and again adjust the pH to 5-5.5 and add 5-6 drops of xylenol orange indicator. Change in color from golden yellow to orange red indicates end point. Take 25 ml 0.05M ethylene diamine tetra acetic acid (EDTA) solution and run a blank.

Calculation: 1 ml 1 M Ethylene diamine tetra acetic acid (EDTA) = 1 ml 1M Zinc acetate = 0.05098 g Al_2O_3

$$\% \ Al_2O_3 = \frac{0.05098 \times \text{normality Zn acetate} \times \text{aliquot} \times 100 \times \text{ml Zn acetate}}{\text{Weight of sample} \times \text{total volume}}$$

8. Determination of Sulphur (total) - gravimetric method

Solutions:

- Carbon tetrachloride saturated with Bromine
- Barium chloride — 10% solution in purified water

Procedure:

Take 0.5-1.0 g powdered sample in 250 ml beaker. Add 10 ml carbon tetra chloride saturated with bromine keep in cold condition in fume chamber over night and add 10-15 ml nitric acid. Digest on water bath then add 10 ml hydrochloric acid digest it to expel NO_2 fumes till syrupy mass is obtained. Cool and extract with hydrochloric acid make volume upto 100 ml, boil and filter through whatman's's 40 No. filter paper, wash the residue with hot purified water. Treat the filtrate with ammonia solution for R_2O_3 precipitations, here R stands for Fe and Al. Filter through whatman's 41 No, filter paper in 500ml—beaker. Acidify the filtrate with hydrochloric acid and add 20 ml of 10% barium chloride solution Stir the solution and digest on burner. Allow the precipitate to settle for overnight. Filter the precipitate through whatman's 42 No. filter paper and wash the precipitate with purified water. Ignite the precipitate in muffle furnace in pre weighed platinum crucible upto 850 and allow to cool and weigh. Calculate the weight of sulphur by multiplying weight of precipitate with 0.13734.

Calculation

$$\% \text{ sulphur} = \frac{\text{Weight of precipitate} \times 0.13734 \times 100}{\text{Weight of sample}}$$

9. Determination of Lead, Zinc and Copper — complexometric method

Solutions:

• Acetic acid — ammonium acetate buffer 5.5 — 6.0 pH, take 200 g ammonium acetate, add 30 ml glacial acetic acid and make up to 1000 ml.

• EDTA solution 0.05 M - Dissolve 18.6120 g of sodium salt of Ethylene diamine tetra acetic acid (EDTA) (Analytical Reagent) in purified water and make upto 1000 ml.

• Xylenol orange indicator - Dissolve 0.2 g indicator in 100ml purified water, add 2 drops acetic acid.

• Thio urea (Analytical Reagent)

• Ascorbic acid (Analytical Reagent)

• Urea (Analytical Reagent)

• Sodium fluoride (Analytical Reagent)

Procedure

Take 1.0 g powdered sample in a beaker and add 30 ml aquaregia [HNO_3: HCl, (1:3)] digest it on hot plate. After 15 minutes add 15 ml sulphuric acid (1:1), and evaporate the solution to dryness. Dissolve the residue in 10 ml sulphuric acid (1:1) and make volume to 100 ml with purified water. Boil and filter through whatman's's 40 No, filter paper. Wash the residue with hot purified water and make up the filtrate to 250 ml in volumetric flask and reserve for determination of zinc and copper.

9. i. Determination of Lead

Procedure

Take the residue along with filter paper in a beaker and add 50 ml acetic acid-ammonium acetate buffer. Boil and filter through whatman's's 40 No. filter paper, wash the residue with hot purified water. To the filtrate, add a pinch of thiourea and ascorbic acid and 3 drops of xylenol orange indicator. Titrate the solution

against standard 0.05 M EDTA solution. Purple red color solution changes to lemon yellow color at the end point. Calculate lead content from the EDTA used up in titration.

Calculate lead value against 1 ml of EDTA solution titrating against standard 1000 ppm lead solution.

Calculation 1ml 0.05 N EDTA = 10. 3605 mg ofPb

$$\% \text{ Lead} = \frac{10.3605 \times \text{Nomiality of EDTA} \times \text{ml of EDTA} \times \text{Aliquot} \times 100}{\text{Weight of sample (mg)} \times \text{Total volume}}$$

9.ii. Determination of Zinc

Procedure

Take suitable aliquot in a beaker from stock solution and add ammonia solution to neutralize the acidity. Add 2 g urea and boil vigorously followed by filtration through whatman's's 40 No. filter paper. Wash the residue with hot water and acidify the filtrate with acetic acid and add 0.5 g ascorbic acid and thiourea each. Add 25 ml acetic acid- ammonium acetate buffer and 4-6 drops of xylenol orange indicator. Titrate against standard 0.05 N EDTA solution. The color of solution changes from purple red to lemon yellow at the end point. Calculate the zinc content from EDTA used up in the titration.

Calculate zinc value against 1 ml of EDTA solution titrating against standard 1000 ppm Zinc solution.

Calculation: 0.1 ml 0.05 N EDTA =3.269 mg of Zn

$$\% \text{ Zinc} = \frac{3.269 \times \text{Normality of EDTA} \times \text{ml of EDTA} \times \text{Aliquot} \times 100}{\text{Weight of sample (mg)} \times \text{Total volume}}$$

9. iii. Determination of Copper

Solutions

- Standard 0.1 N sodium thiosulphate solution.
- Potassium iodide (AR.)
- Starch 1% solution —dissolve 1 g in purified water, boil and make up to 100ml.

Procedure

Take suitable aliquot from the stock solution in a beaker and add approx. 1.0 g sodium fluoride. Add ammonia solution till precipitation occurs and add acetic acid to dissolve the precipitate, boil and cool in water bath. Add approximately 1.0g potassium iodide and titrate the liberated iodine against 0.1N sodium thiosuphate solution by adding starch solution as indicator in iodine flask. The color changes from blackish brown to white indicates end point. Calculate copper value against 1 ml of sodium thoisulphate solution titrating against standard 1000-ppm copper solution.

Calculation: 1ml N $Na_2S_2O_3$=0.06354 g of Cu

$$\%Cu = \frac{0.06354 \times \text{Normality of } Na_2S_2O_3 \times \text{ml of } Na_2S_2O_3 \times \text{Aliquot} \times 100}{\text{Weight of sample} \times \text{Total volume}}$$

Chemical Analyses of Metal or Mineral Samples by Instrumental Methods

1. Determination of Silver by Atomic Absorption Spectrophotometer (A.A.S.) (Anonymous, 2008)

Solutions:

- Prepare standard 1000 ppm silver solution from Ag metal or Silver nitrate.
- Prepare standard solutions of silver of 1 ppm, 3ppm and 5 ppm.

Procedure:

Weigh 0.5 g — 1.0 g powdered sample in 250 ml beaker and add 25 - 30 ml, reverse aquaregia [HNO_3: HCI (3:1)], digest on hot plate, till syrupy mass is obtained.

Neutralize the syrupy digested mass with ammonia solution make up the volume to 25-50 ml Make dry filtration through whatman's 40 No.filter paper and take observations on 328.1 nm by atomic absorption spectrophotometer

Calculation —

$$\text{Ag ppm} = \frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$$

2. Determination of Copper, Lead, Zinc, Nickel, Cobalt and Cadmium by Atomic Absorption Spectrophotometer:

Solutions

i) Copper

- Prepare 1000 ppm copper standard solution from Cu metal.

• Prepare 1 ppm, 3 ppm and 5 ppm standard Cu solutions.

• Wavelength — 324.7 nm.

ii) Lead

• Prepare 1000 ppm Lead standard solution from Pb.

• Prepare 1 ppm, 5 ppm and 10 ppm standard Pb solution.

• Wavelength — 283.3 nm.

iii) Zinc

• Prepare 1000 ppm zinc standard solution from Zn metal,

• Prepare 0.5 ppm, 1.0 ppm and 1.5 ppm standard Zn solution.

Wavelength —213.9 nm.

iv) Nickel

• Prepare 1000ppm nickel standard solution from Ni metal.

• Prepare 1 ppm, 3ppm and 5 ppm standard Ni solution.

• Wavelength- 232.0 nm.

v) Cobalt —

• Prepare 1000ppm cobalt standard solution from Co metal (A 240.7 nm).

vi) Cadmium-

• Prepare l000ppm Cadmium standard solution Cd metal (X 228.8 nm).

Procedure

Weigh 0.10g - powdered sample in 250 - ml beaker and add 25-30 ml aquaregia, [HNO_3:HCl (1:3)] digest on hot plate till syrupy mass is obtained .Filter through whatman's 40 No. iilter paper in 100 ml volumetric flask. Maintain 1% acidity with nitric acid and wash with hot water making volume to 100 ml. Take observations on Atomic Absorption Spectrophotometer on appropriate wavelength as follows:-

Calculation

$$\text{Ppm (metal)} = \frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$$

3. Determination of Tin by Atomic Absorption Spectrophotometer

Solutions

- Prepare 1000 ppm Tin standard solution from Sn metal.
- Prepare 22 ppm, 5 ppm and 10 ppm standard Sn solutions.
- Prepare 7.5% potassium chloride solution in purified water as buffer.

Procedure

Weigh 0.5g - 1.0 g powdered sample in 250ml-beaker and add 25-30 ml aquaregia [HNO_3: HC1 (3:1)], digest on hot plate, till syrupy mass is obtained. Extract in hydrochloric acid maintain 20% hydrochloric acid acidity followed by filtration through whatman's 40 No. filter paper, wash with hot water add 2 ml 7.5%

potassium chloride solution and make up to 100 ml in volumetric flask. Take observations by Atomic Absorption Spectrophotometer on 286.3 nm wavelength.

Calculation Tin Ppm = $\frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$

4. Determination of Antimony by Atomic Absorption Spectrophotometer

Solution

- Prepare 10% tartaric acid solution in purified water.
- Prepare 7.5% potassium chloride solution in purified water.
- Prepare 1000 nm standard Sb solution from Sb metal.
- Prepare 2 ppm, 5 ppm and 10 ppm standard Sb solutions.

Procedure

Weigh 1.0 g powdered sample in 250ml-beaker and add 25-30 ml aquaregia [HNO_3: HC1 (3:1)], digest for one hour on hot plate till syrupy mass is obtained. Filter through whatman's 40 No. paper in 100ml-volumetric flask. Wash with hot water and add 10 ml of 10% tartaric acid, add 2 ml of 7.5% potassium chloride solution. Add hydrochloric acid drop wise to get clear solution and make up to 100ml volume. Take observation by Atomic Absorption Spectrophotometer on 217.6 nm wavelengths.

Calculation

$$\text{Antimony Ppm} = \frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$$

5. Determination of Bismuth by Atomic Absorption Spectrophotometer

Solutions

- Prepare 7.5% potassium chloride solution in water.
- Prepare 1000 ppm standard Bismuth solution from Bi metal.
- Prepare 10 ppm, 20 ppm and 40 ppm standard Bi solutions.
- Prepare 2 ppm, 5 ppm and l0 ppm standard Sb solutions

Procedure

Weigh 1.0 g powdered sample in 250ml-beaker and add 25-30 ml aquaregia [HNO_3: HC1 (3:1)], digest for one hour on hot plate till syrupy mass is obtained. Extract with 10 ml hydrochloric acid. Maintain 20% acidity and Filter through whatman's No. 40 paper in 100 ml volumetric flask. Wash with hot water and then .add 2 ml of 7 5% potassium chloride solution make up the volume to 100 ml. Take observation by Atomic Absorption Spectrophotometer on 223.1 nm wavelength

Calculation

$$\text{Bismuth Ppm} = \frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$$

6. Determination of Gold by Atomic Absorption Spectrophotometer

Solutions

- Equilibriated MIBK (methyl isobutyl ketone solution) · 20% hydrochloric acid solution mixes with MIBK in 1·1 ratio. Transfer in separating funnel and shake

well to remote heavy metals present as impurities. Separate organic layer of MIBK.

- 5% hydrochloric acid solution for washings
- Standard 1000 ppm gold solution from Au metal
- Prepare 1 ppm, 2 ppm and 3 ppm standard Au solution in MIBK medium.
- Conc. Hydrochloric acid saturated with bromine

Procedure

Weigh 10-20 g of100 mesh powdered sample in 500 mi conical flask and add 20 ml hydrochloric acid saturated with bromine. Keep in (cold condition) for two hours and add 50 ml aquaregia [HNO_3: HC1 (3:1)], digest on hot plate slowly till syrupy mass is obtained. Extract with l0 ml hydrochloric acid and heat it. Filter extracted mass through whatman's No. 40 filter paper wash with hot water. Transfer the filtrate into a separating funnel and add 25 ml MIBK solution in 3 fractions. Add 5 ml aquaregia into separating funnel and shake the separating funnel 2-3 minutes. Drain off aqueous layer from the separating funnel and wash the organic layer by 5 ml of 5% hydrochloric acid three times drain off aqueous layer. Transfer the organic layer in dry 25 ml volumetric flask. Take observations by Atomic Absorption Spectrophotometer on 242.8 nm wavelength in MIBK medium.

Calculation

$$\text{Gold Ppm} = \frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$$

7. Determination of Chromium by Atomic Absorption Spectrophotometer

Solutions

- Prepare 1000 ppm standard chromium solution from Cr Metal or from $K_2Cr_2O_7$.
- Prepare standards 10 ppm, 20 ppm and 40 ppm.
- Prepare 2 ppm, 5 ppm and 10 ppm standard Sb solutions.

Procedure

Weigh 0.10-0.20 g powdered sample in 250 ml-beakers and add 10 ml of 30% solution of hydrogen peroxide, add 25-30 ml perchloric acid digest for one hour on hot plate till syrupy mass is obtained. Again add 4-5 ml hydrogen peroxide and 10 ml perchloric acid digest on hot plate till complete digestion. Filter through Whatman's No. 40 filter paper wash with hot water and make up in 100ml-volumetric flask. Take observations by Atomic Absorption Spectrophotometer on 357.9 mm

Calculation

$$\text{Chromium Ppm} = \frac{\text{Reading (conc.) x original volume}}{\text{Weight of sample}}$$

8. Determination of Arsenic - Gutzeit method

Solutions:

- Potassium iodide 4% solution · dissolve 4 g potassium iodide in purified water and make upto 100 ml volume.

• Stannous chloride 0.75% solution - dissolve stannous chloride in hydrochloric acid, boil it and maintain 100 ml volume.

• Mercuric chloride 25% solution · dissolve in ethyl alcohol in warm condition. Prepare whatman's filter paper circles of apparatus size. Soak the circles in mercuric chloride solution and dry it.

• Lead acetate 15% solution - dissolve lead acetate in water with a few ml of acetic acid and maintain volume to 100ml. Soak Whatman's filter paper pieces. Dry it to keep in glass tube of the apparatus.

• Standard Arsenic solution - Prepare standard Arsenic solutions of 2.5, 5.0 and 10 ppm.

Procedure

Weight 10 g — powdered sample in 250ml-beaker and add 10 ml bromine in hydrochloric acid keep it for overnight then a add 5 ml nitric acid, digest on water bath at controlled temperature till syrupy mass is obtained. Add 5 ml conc. hydrochloric acid and digest it. Extract the mass in hydrochloric acid. Filter through Whatman's No. 40 filter paper wash it with hot water and make up to 100 ml. Take 50 ml aliquot in Gutzeit apparatus. Add 6 ml of potassium iodide solution then add 10 ml of stannous chloride solution to reduce Arsenic, add 2-3 g zinc metal in the container. Add conc. hydrochloric acid drop wise from the stopper cork keep mercuric chloride soaked filter paper circles in between the stopper and lower glass tube. Keep lead acetate soaked filter paper in lower tube join and tight both the portion of apparatus till completion of reaction for 15-20 minutes. AsH_3 gas liberated gives yellow spot on mercuric chloride soaked filter paper. Prepare yellow spots of 1,2,4,5 ppm standard Arsenic and compare with unknown samples.

9. Determination of Mercury (Hg) by cold vapour Atomic Absorption Spectrophotometcr (Mercury Analyzer)

Reagents and Standards

• Stannous chloride solution (20 % w/v); Dissolve 20 g of stannous chloride. 2 water in 25 ml hydrochloric acid by boiling; cool and dilute to 100 ml. Add 1—2 g of tin metal.

• Potassium dichromate solution (1% w/v): Dissolve 1 g potassium dichromaie in 100 ml water.

• Potassium permanganate solution (5% w/v): Dissolve 5 g potassium permanganate in 100 ml sulphuric acid (1%v/v)

• Sulphuric acid (1%) — dilute 1 ml of con. sulphuric acid to 100 ml by adding purified water.

• Nitric acid (10 % v/v) — dilute 50 ml of nitric acid to 500 ml by adding purified water.

• Sodium hydroxide (20% w/v)- dissolve 20 g of sodium hydroxide pellets in purified water and make volume to 100 ml.

• Sulphuric acid (1:1 H_2SO_4) - add 100 ml of Sulphuric acid to cold purified water and make volume to 200 ml.

• Standard Mercury solution:

A. Dissolve 0.1354 g of mercuric chloride in 25 ml of 5% nitric acid. Add 1 ml of potassium dichromate solution and make upto 100 ml with 5% nitric acid.

1 ml of this solution = 1.0 mg Hg/ml = 1000 µg Hg/1 ml: 1000 ppm Hg

B. Standard 0.10 µg Hg/ml (=100ng Hg / ml):

Prepare 100ng Hg/ml standard solution by successive serial dilution of the 1000 µg/ ml Hg solution (10000 times) maintaining 5 % nitric acid and 0.01% potassium dichromate concentration. This solution is generally stable for 30 days, Prepare this solution preferably fresh every day, Measure absorbance of 0.2 ml, 0.4 mi, 0.6 ml 0.8 ml and 1.0 ml of the above, 100 ng Hg / ml, dilution solution which corresponds to 20, 40, 60, 80 and 100 ng respectively. Plot the calibration curve of nano-gram mercury versus absorbance. Use this calibration curve for computation of mercury content in unknown sample.

Procedure

Weigh 2-5g dry tine powder of sample (150 mesh) in a 500 ml conical flask and add 15 ml nitric acid and 5 ml sulphuric acid then leave flask in ice bath for 90 minutes. Heat on purified water bath for 30 minutes and add 150 mg potassium permanganate and 3 ml mercury free hydrochloric acid, Put a funnel and just heat and then boil gently for 5 minutes, cool and transfer into a 50 ml plastic volumetric conical tube and make upto volume.

Centrifuge: Use clear solution for determination of mercury content. Run a reagent blank through the procedure. Pipette out 10 ml aliquot into the reaction vessel R_2 of mercury analyzer. Follow procedure for reading mercury content absorbance as described in instruction/operation manual of the instrument.

Calculation

Mercury content (ng) =

Total volume of (From calibration graph) ng X aliquot (50 ml)

Weight of the sample (g) X Volume of aliquot for measurement (10 ml)

Methods of Other Important Chemical Studies

1. Determination of Acid- Insoluble ash

Take about 1 g pre dried sample (150 mesh) in 250ml-beaker. Add 50 ml dilute hydrochloric acid. Stir the solution and cover the beaker by watch glass. Heat on the hot plate at 150 degree and digest for two hours. Then cool, filter, wash by water and ignite at 900°, Weigh the residue and calculate residue % and report as acid insoluble.

2. Reaction with Hydrochloric acid, Nitric Acid and Sulphuric Acid:

Put 1 g fine powdered (150 mesh) sample in a glass test tube, Add 10 ml concentrated acid. Take observations for change of colour, dissolution, evolution of any gas and increase or decrease in temperature. Then heat the test tube and again record all the observations stated above.

3. Effect of Heat

The observations of effect of heat are recorded as "effect of heat in open" and "effect of heat in closed". The effect of heat of mineral is observed by heating 1 g powdered sample in open test tube at 900 degree. Take observations for change in colour and volume, evolution of any gas and purified water etc. For effect of heat in closed, use penfield tube and repeat the same process and observations.

4. Qualitative Test for Carbonate and Sulphate

Carbonate

Take about 0.5 g powdered sample in a test tube and add 10-15 ml of dilute hydrochloric acid to it. Observe effervescence of CO_2, lime purified water turns milky white.

Sulphate: Take about 0.5 g powdered sample in a test tube and add 10-15 ml dilute hydrochloric acid and boil then add 10% barium chloride solution drop wise to the solution it shows white precipitate of $BaSO_4$.

STANDARDIZATION OF *KUSHTA*

Standardization is a measurement for ensuring the quality and is used to describe all measures, which are taken during the manufacturing process and quality control leading to a reproducible quality. Standardization is not an easy task as numerous factors influence the bio efficacy and reproducible therapeutic effect. In order to obtain quality oriented products, care should be taken right from the process of preparation. **(Shrikumar S, 2006; Shinde, 2009)**

- Standardization of *kushta* is utmost necessary to confirm its identity and to determine its quality and purity. It will also make sure the safety, effectiveness and acceptability of the product. But the most important challenges faced by these formulations are the lack of complete standardization by physicochemical, microbiological and analytical evaluation.

STANDARDIZATION OF *KUSHTA*

The various physicochemical evaluation include organoleptic properties, physical properties, chemical properties, preliminary testing's of prepared *kushta* and its physicochemical parameter evaluation like pH, fineness, loss on drying at 105 degree Celsius, total ash, acid insoluble ash, water soluble ash and particle size, mesh test. **(Prakash B,1997; Singh SK, 2009; Bhowmick TK, 2009; Sarkar PK, 2010)**. Tests for heavy/toxic metals should be carried out.

The standardization of *kushta* begins with

a. Preliminary testing's
b. Parameters for identity

c. Parameters for purity.

d. Parameter for quantity.**(Dubey N et al, 2009)**

PRELIMINARY TEST OF *KUSHTA*

a) **WARITARA**: Ideal Bhasma or *kushta* will float on the surface of water, indicating lightness.

This parameter is given in number of books but one important thing about this point is written in *Miftah ul Khazain* that floating of a *kushta* over the surface of water indicates its lightness only and does not tell us that our *kushta* is ideal (*kamil*) or not. So it is not a reliable parameter which can be taken as standard. **(Kareem BH and Ferozuddin CH, YNM)**

b) **NIRDHUM** (Smokeless): Finished Bhasma will not emit any smoke when put over fire, while the impure form emits smoke.

One important thing about this parameter is that one ideal and *kamil kushta* of non metal can also give smoke when placed on fire, so it would be unfair to consider a *kushta* of non metal as *kham* if it gives smoke when put on fire. **(Kareem BH and Ferozuddin CH, YNM)**

c) **REKHAPURNA** (Lines/full): The *kushta* should be fine when taken between thumb and forefinger. It should occupy the lines of pores of fingers.

d) **UTTANIA**: A grain of rice, barley, etc. will float over the *kushta* like a swan on a lake.

e) **NISCHANDRA** (Sparking): There should be no shining or sparkly particles in the prepared *kushta*. This parameter is particularly applicable to metallic *kushtajat*.

f) **NISWADU** (Taste): The prepared *kushta* should be completely tasteless.

g) **AWAMI**: when put on the tip of the tongue, there should be no effect. Impure *kushta* will cause nausea or vomiting.

h) **AMLA**: When *kushta* is put with citrus juice, especially lemon, it should retain its color and original form.

An ideally prepared *kushta* should stick to the wall when thrown at it.

i) **LOSS OF METALLIC STATE OR NIRUTTA**: This involves heating of a very thin silver sheet (600 nm thickness along with a small quantity of *bhasma* or *kushta* to red hot for about 5 min. After cooling the sheet to room temperature, no traces of this sample should permanently stick to the silver sheet indicating no alloy formation takes place, thus confirming the metal has totally transformed into *bhasma*, its oxide form. **(Mohaptra S, 2010)**

However, these qualitative tests do not provide any quantitative information about the composition and the structure of the final drug. For any drug containing heavy metals (for example lead, mercury), such structural information is an absolute necessity.

f) Parameters for Identity:

1. Macroscopic: luster, color etc.

   Physical properties: Bulk density ,Tapped density, Particle size range, Angle of repose, Carr's index, Hausners ratio, Loss on drying, loss on ignition, XRPD Characteristic (X ray powder diffraction),TGA (Thermogravimetric analysis),IR spectroscopy. **(Dubey N et al, 2009)** Extractive value **(Rasheed A, 2011)**

g) Parameters for purity: (Dubey N et al, 2009)

1. Arsenic

2. Cadmium
3. Lead
4. Mercury
5. Microbial evaluations

h) parameter for quantity: (Dubey N et al, 2009)

1. Quantitative analysis by chemical methods or by AAS.

MACROSCOPIC PROPERTIES

Organoleptic properties like appearance, colour, taste, texture and smell are noted.

Physicochemical Properties

1. BULK DENSITY, TAPPED DENSITY, CARR'S INDEX, HAUSNER RATIO

30 gm of *kushta* should be taken and filled and carefully into cylinder with the aid of a funnel without any loss. The cylinder is tapped by a digital tap density apparatus. The initial volume was noted and the *kushta* is again then tapped until no further reduction in volume is noted.

Bulk density = Mass/Bulk volume

Tapped density = Mass/Tapped volume

Carr's Index:

Carr's index is calculated according to following equation.

Carr's index (% compressibility) = $100 \times (1 - D_b / D_t)$

Where D_b = Bulk density, D_t = Tapped density

Hausner Ratio

Hausner ratio is calculated according to following equation.

Hausner ratio = D_t / Db. Where D_b = Bulk density and D_t = Tapped density.

The bulk and tapped densities are calculated. **(Lachman L et al, 1991; Anonymous, 1968)**

2. **Extractive values: (Rasheed A, 2011; Rajshekaran A et al, 2002; Anonymous, 1991)**

   The **Extractive value** is a parameter for detecting the adulteration in any drug. The amount of the extract that the drug yields in a solvent is often an approximate measure of the amount of certain constituents that the drug contains. Therefore, for establishing the standards of any drug these extractive values play an important role.

Determination of Extractive values

A) Successive Extractive Value

The extractive values of *kushtas* in different solvents viz. Petroleum ether, chloroform and alcohol should be carried out by percolation in soxhlet apparatus. The extracts should be filtered using filter paper (Whatman's No.1) and after evaporation of the solvents on water bath, the extracts values should be determined with reference to the weight of drug (% w/w). The procedure should be repeated three times and the mean value for each extract should be calculated. **(Anonymous, 2006)**

B) Non-Succesive Extractive Value

In this method the extractive values of *kushta* in different solvents viz. alcohol and water are carried out separately by percolation in soxhlet

apparatus. The heat is applied for six hours on a water bath for alcohol and heated directly on a heating mantel for water. *Kushta* is taken and subjected to separate extraction with each solvent (% w/w). The extracts are then filtered using filter paper (Whatman's No.1) and after evaporation of the solvents on water bath, the extracts values are determined with reference to the weight of drug. The procedure should be repeated three times and the mean value for each extract should be calculated. **(Anonymous, 2006)**

3. **Loss of weight on drying at 105°C**

Two gram of drug should be taken, spread uniformly and thinly in a shallow petri dish. Heat it at a regulated temperature of 105°C and then cool in a desiccator and weigh. Repeat the process many times till two consecutive weights are constant. **(Anonymous, 2006; Afaq SH et al 1994)**

1. **Determination of pH in 1% solution and 10% solution:**

The **pH value** of various dosage forms may also be considered a parameter for the purity of a drug. In aqueous solutions these compounds dissociate into their respective ions. The concentration of hydronium ions influences the concentrations of the anions, cations, and undissociated molecules present in solution. In turn, these chemical species often affect the stability, therapeutic activity (though drug absorption), and pharmaceutical elegance of medicinal agents in aqueous preparations. The pH and hydronium ion concentration also play an important role in the study of drug receptor-site interactions, an area of research which has gained considerable impetus during recent years. **(Afaq SH et al 1994; Lachman L et al, 1991)**

The pH value of 1% solution

Dissolve an accurately weighed 1 gm of *kushta* in accurately measured 100 ml of distilled water, filter and measure pH with a pH meter.

The pH value of 10% solution

Dissolve an accurately weighed 10 gm of *kushta* in accurately measured 100 ml of distilled water, filter and measure pH with a pH meter. **(Anonymous, 2006)**

2. **Ash value** is the residue that remains after complete incineration of the drug. Ash value plays an important role in ascertaining the standard of a drug, because the dust, earthy and unrequired matters are generally added for increasing the weight of a drug resulting in the higher ash percentage. Therefore, the ash value determination furnishes the basis of judging the identity and cleanliness of a drug and give information related to its adulteration. **(Fransworth NR, 1997)**

a. Total Ash

A sample of 2 gm of *kushta* should be incinerated in a silica dish at a temperature not exceeding 450°C until free from carbon, cooled and weighed and the percentage was calculated with reference to air dried drug. **(Afaq SH et al, 1994; Anonymous, 1968; Anonymous, 2011)**

b. Acid insoluble Ash

The ash is boiled with 25ml of dilute hydrochloric acid for 5 minutes. The insoluble matter is collected on an ash less filter paper washed with hot water and ignited at a temperature not exceeding 450°C and weighed after cooling. The percentage of acid insoluble ash is calculated with reference to the air dried drug. **(Afaq SH et al, 1994; Anonymous, 1968; Anonymous, 2011)**

c. Water soluble Ash

The ash is boiled with 25 ml of distilled water for 5 minutes. The insoluble matter is collected on an ash less filter paper, washed with hot water and ignited. The weight of insoluble ash is subtracted from the weight of the total ash, giving the weight of the water soluble ash. The percentage of water soluble ash is calculated with reference to air dried drug. **(Afaq SH et al, 1994; Anonymous, 1968; Anonymous, 2011)**

Acid insoluble ash can also be calculated by the method mentioned in API.

Determination of Acid- Insoluble ash

Take about 1 g pre dried sample (150 mesh) in 250 ml-beaker. Add 50 ml dilute hydrochloric acid. Stir the solution and cover the beaker by watch glass. Heat on the hot plate at 150 degree and digest for two hours. Then cool and filter, wash by water and ignite at 900°, Weigh the residue and calculate residue % and report as acid insoluble. **(Anonymous, 2008)**

A. Parameters for purity and parameter for quantity

These tests Can be done by the chemical methods as mentioned in the chemical properties of metal or minerals.

WHO Limit: Normal permissible concentration

Heavy Metals	**Blood**
Mercury	0.2-3 ng/ml
Arsenic	1.1 µgm/Lit.
Lead	0.02-1.0 µgm/Lit.

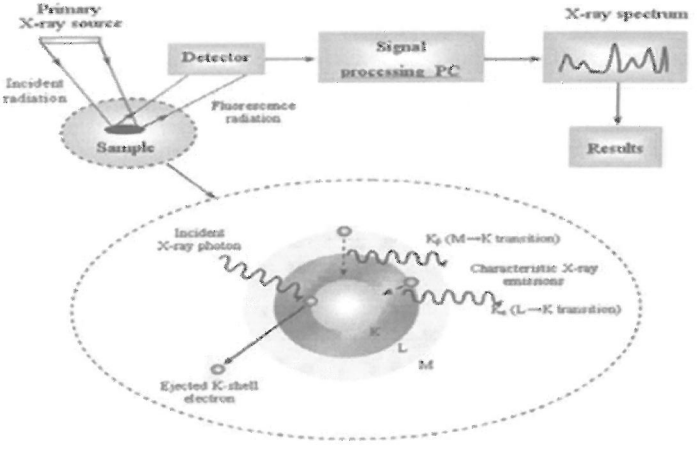
Copper	100-200 µgm/dl
Aluminium	1-10 ng/ml
Nickel	0.05-1 ng/ml
Magnesium	1.8-3 mg/dl
Zinc	50-150 µgm/Lit.
Iron	50.175 µgm/Lit.

Analytical Techniques and their Purpose for Analysis

1. EDX Or Energy Dispersive X Ray –

USE: Chemical Nature, Size and Morphology of Particles. It is used mainly with SEM (Scanning electron microscopy).

EDX- APPARATUS

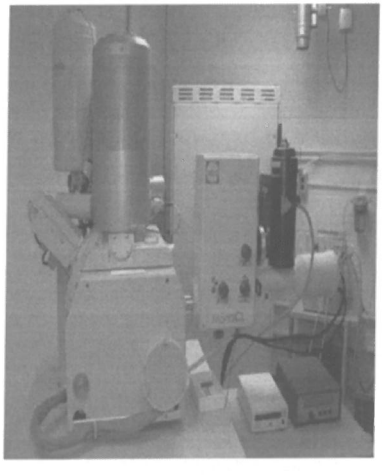


EDX PRINCIPLE

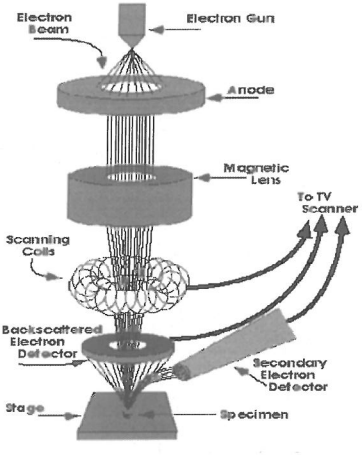
(EDS or EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons (see PIXE), or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam excites an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured. **(Goldstein J, 2003)**

SEM - Scanning Electron Microscopy SEM

- It is a type of electron microscope that images the sample by scanning it with a high energy beam of electron in a scan pattern.
- It is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with electrons in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer.



SEM APPARATUS



SEM PRINCIPLE

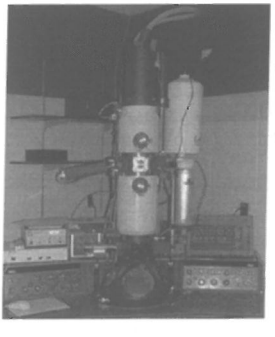
2. TEM-TRANSMITTION ELECTRON MICROSCOPY

- USE: Particle Size And Size Distribution

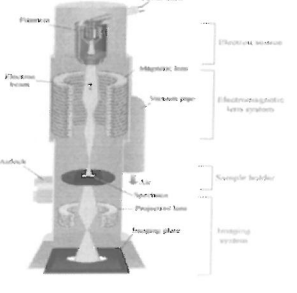
- A beam of electron is transmitted through an ultrathin specimen and an image is formed from interaction of an electron transmitted through a specimen and then it is magnified.

Transmission electron microscopy (**TEM**) is a microscopy technique whereby a beam of electrons is transmitted through an ultra thin specimen, interacting with the specimen as it passes through. An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film, or to be detected by a sensor such as a CCD camera.

TEMs are capable of imaging at a significantly higher resolution than light microscopes. This enables the instrument's user to examine fine detail—even as small as a single column of atoms, which is tens of thousands times smaller than the smallest resolvable object in a light microscope. TEM forms a major analysis method in a range of scientific fields, in both physical and biological sciences.



TEM APPARTUS



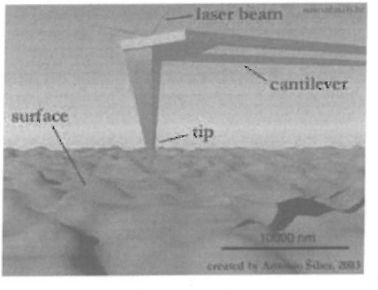
TEM PRINCIPLE

3. **AFM -ATOMIC FORCE MICROSCOPY**

Discovered by Rohrer and won the noble prize in 1986.

USE: particle size and size distribution.

AFM APPARATUS



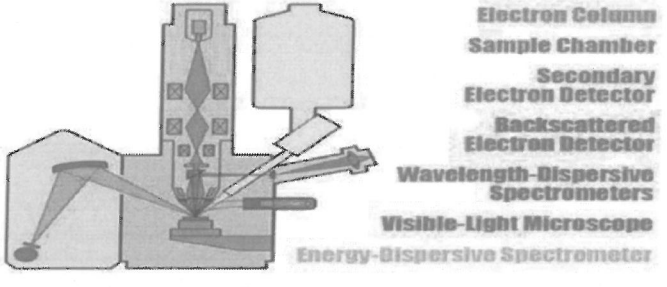
AFM PRINCIPLE

- Forces between the tip of cantiliver and the sample lead to deflection and this deflection is measured by using a laser reflected from top of cantiliver to detector.

CANTILIVER

4. EPMA- Electron Probe Micro analyser

- EPMA is also called an **electron microprobe**, or just **probe**. It is fundamentally the same as an SEM, with the added capability of chemical analysis.
- The primary importance of an EPMA is the ability to acquire precise, quantitative elemental analyses at very small "spot" sizes (as little as 1-2 microns).



EPMA PRINCIPLE

5. XRD- Powder X Ray Diffraction

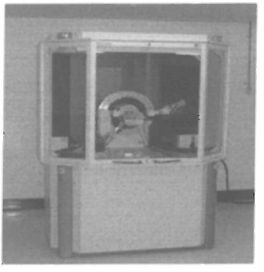
X-ray powder diffraction is most widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds).

Other applications include:

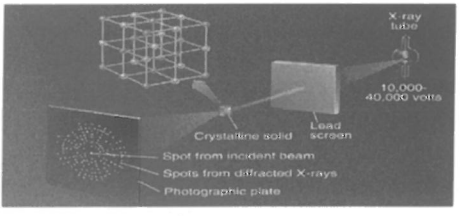
- characterization of crystalline materials
- identification of fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically
- determination of unit cell dimensions
- measurement of sample purity

With specialized techniques, XRD can be used to:

- determine crystal structures.
- determine of modal amounts of minerals (quantitative analysis)
- characterize thin films samples by:
  - determining lattice mismatch between film and substrate and to inferring stress and strain
  - determining dislocation density and quality of the film by rocking curve measurements
  - measuring superlattices in multilayered epitaxial structures
  - determining the thickness, roughness and density of the film using glancing incidence X-ray reflectivity measurements
- make textural measurements, such as the orientation of grains, in a polycrystalline sample



XRD APPARATUS

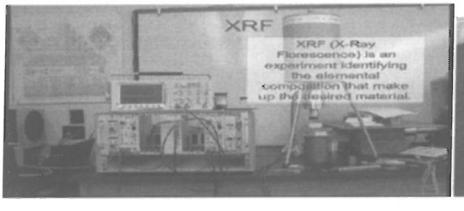


PRINCIPLE XRD

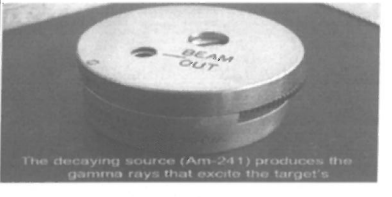
6. XRF – X RAY FLORESCENCE AND PIXE – PROTON INDUCED X RAY EMISSION

XRF

- The XRF uses Gamma rays from the decaying source (Am 241) to excite the target atoms.



XRF APPARATUS



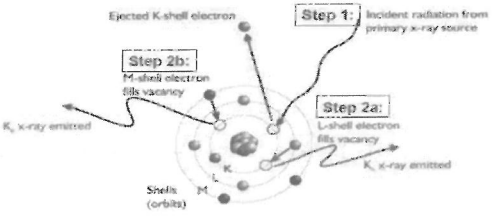
DECAYING SOURCE

XRF – A PHYSICAL DESCRIPTION

Step 1: When an X-ray photon of sufficient energy strikes an atom, it dislodges an electron from one of its inner shells (K in this case)

Step 2a: The atom fills the vacant K shell with an electron from the L shell; as the electron drops to the lower energy state, excess energy is released as a K_α X-ray

Step 2b: The atom fills the vacant K shell with an electron from the M shell; as the electron drops to the lower energy state, excess energy is released as a K_β X-ray



XRF PRINCIPLE

PIXE- PROTON INDUCED X RAY EMISSION

- Proton Induced X Ray Emmision is similar to XRF but uses a proton beam instead of Gamma rays to stimulate x ray emmision.
- The PIXE is also more accurate than the XRF experiment because PIXE uses a beam instead of a decaying source.

7. ESCA- Electron Spectroscopy For Chemical Analysis

- It gives quantitative information on the chemical composition of a surface (atomic composition and chemical state).

8. Single crystal XRD –

To confirm exact molecular structure of crystalline intermediates or products

9. **Extraction and Chromatography** -To extract out organic matter if any

12. **HPLC, NMR, IR, MALDI & ESI - MS** - Characterization of organic matter (if > 20% wt/wt)

(Tate PM et al, 2009; Wadekar M.P wt al, 2006; Wadekar M.P et al, 2005; Smita D, 2012)

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