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Indian Society of Analytical Scientists
Delhi Chapter

e-Bulletin

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Contents

Page No

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Editorial Board

From Editor's Desk…….

Launch of the First Issue of ISAS-DC e-Bulletin

It gives us immense pleasure to release the first issue of the e-bulletin by ISAS-DC. The e-bulletin aims to share advancements, foster collaboration, and highlight the innovations in analytical sciences for global knowledge exchange. The field of analytical sciences has evolved rapidly, with technological breakthroughs driving major advances across various industries, from pharmaceuticals to environmental monitoring. These innovations are not only enhancing research capabilities but also paving the way for economic and social progress globally, including in India.

The "Atmanirbhar Bharat" (Self-reliant India) initiative has significantly accelerated growth in the field of analytical sciences. The integration of cutting-edge technologies like Artificial Intelligence (AI), machine learning, and automation into analytical tools has enhanced data accuracy and research efficiency. India has made significant strides in nanotechnology, with tools such as atomic force microscopy (AFM) and scanning electron microscopy (SEM) allowing researchers to study materials at the molecular level. One of India's standout achievements has been in high-resolution mass spectrometry (HRMS), revolutionizing the analysis of complex chemical compounds. This advancement, along with progress in chromatography, has paved the way for faster and more precise analyses crucial in areas like drug development, environmental monitoring, and energy sectors, including the oil industry.

The oil industry, essential to the global economy, benefits immensely from these advancements. Analytical techniques like chromatography, mass spectrometry, and spectroscopy are being used to optimize fuel quality, detect impurities, and improve refining processes. These innovations enhance petroleum product quality, ensure compliance with environmental standards, and support sustainable energy solutions.

As these technologies continue to evolve, India, by fostering innovation and collaboration, can contribute significantly to global scientific progress, helping to create a safer, healthier, and more interconnected world.

Since this is our maiden initiative, suggestions and feedback are welcome for further improvement. We hope this e-bulletin will serve as a true reflection of major accomplishments in the related fields, and we encourage more members of ISAS-DC, as well as associates and allied stakeholders, to engage with it, fostering greater interest and collaboration in these transformative fields.

(Editorial Team)

Message from
Director (R&D), IndianOil
Chairman, ISAS Delhi Chapter

It is with great pleasure and immense pride that I welcome you to the first edition of our e-bulletin. As we embark on this exciting new venture, we take a moment to reflect on the growth and evolution of the field of analytical chemistry in India and its vital role in shaping the future of science, technology, and industry.

The discipline of analytical chemistry has always been at the forefront of scientific advancements. It plays a critical role in numerous sectors, from environmental monitoring and healthcare to food safety, pharmaceuticals, and beyond. As our world faces increasingly complex challenges, analytical chemistry remains a cornerstone of innovation and discovery.

The formation of the Indian Society of Analytical Chemistry (ISAC)-Delhi Chapter has been a monumental step in uniting professionals, researchers, educators, and students from across the country. We have strived to create a platform where knowledge can be shared, skills can be honed, and collaborations can be fostered. Our society continues to grow, driven by the dedication and passion of its members, and this e-bulletin is a testament to that collective energy.

This inaugural edition is a celebration of our achievements, while also looking ahead to the future. It features the latest developments in the field, highlights from recent events, and contributions from our esteemed members. We are also excited to share upcoming initiatives and opportunities for involvement, ensuring that ISAC-DC remains a vibrant hub for analytical chemistry in India.

As we continue to innovate and advance, it is crucial that we stay connected. This e-bulletin will serve as a medium for communication, allowing us to stay informed about key trends, research breakthroughs, and upcoming events. We invite you to actively participate by sharing your insights, research, and experiences, as together, we continue to push the boundaries of knowledge and excellence.

On behalf of the entire executive committee, I would like to express our gratitude to all those who have contributed to this first edition of the e-bulletin. Your unwavering support and commitment to the society’s goals are what make ISAC a thriving community.

We hope that this publication becomes a valuable resource for all members and serves as a platform for future collaboration and growth. Thank you for your continued support. Together, let’s shape the future of analytical chemistry in India.

(Dr Alok Sharma)

Message from
Executive Chairman, ISAS-Delhi Chapter
Convener, Editorial Committee

It is with great pleasure, we launch the inaugural edition of e-bullrin by ISAS – Delhi Chapter. After years of meticulous planning and with the unwavering support of our Executive Committee members, we are proud to release the first issue in April 2025.

On behalf of the Editorial Committee of ISAS-Delhi Chapter, I thank all contributors who submitted articles for this inaugural issue. I extend my sincere appreciation and thanks to the Editorial committee members for their efforts in bringing this e-bulletin to fruition.

In our endeavor with humble beginning, members are encouraged to submit research articles, updates on analytical sciences, and relevant news articles from reputable sources with proper citations. We eagerly anticipate receiving more articles for our next issue, scheduled for release in July 2025. We aim to obtain an ISBN number for our e-bulletin after publishing few issues, enhancing its reach and recognition.

We invite scientists and researchers to join our professional society and engage with the latest advancements in analytical sciences. **Avail** the opportunity, **Attune** yourself with recent excitement of analytical sciences and **Achieve** excellence in your sphere of activity. We look forward to your active participation and contributions for the growth of analytical science.

For more information and insights into our activities, please visit our website: www.isasdelhi.org

(Dr. J Christopher)

About ISAS - Delhi Chapter

Greetings from Indian Soceity of Analytical Scientists – Delhi Chapter.

ISAS-Delhi Chapter (www.isasdelhi.org), was established in 1992 with headquarters at Indian Oil R&D Centre, Faridabad, Haryana to promote the growth of analytical sciences in the northern part of our country. Delhi Chapter of ISAS covers Delhi, Chandigarh, Punjab, Haryana, UP, Uttranchal, Jammu & Kashmir and other contiguous area. Among the 12 chapters of ISAS all over India, ISAS-Delhi Chapter is one of the most active Chapter. At present Delhi Chapter itself has more than 1600 life members from more than 100 research institutes, national laboratories, universities, industry etc.

The Chapter has organized more than 60 technical programs like one / two / three -days technical Seminars / Symposia / Conference / Workshops on various contemporary topics of science at diverse locations like Shimla, Agra, Dehradun, Palampur, Rohtak, Faridabad, Delhi, Ballabgarh, Noida, Dharamshala, Leh/Ladakh. The society also organizes summer and winter workshops on various analytical techniques for the benefit of students, researchers and faculty members. ISAS-Delhi Chapter has been adjudged as the Best Chapter by the parent body four times in the past based on our activities to promote analytical science in the country. Our Chapter also provides partial financial assistance of Rs. 25,000/- to students to present research work in international conferences abroad. We have also initiated Lifetime achievement award and young researcher award once in 2 years.

ISAS Delhi Chapter provides a common platform for analytical scientists to exchange ideas, keep themselves abreast of the latest developments, thus promoting growth of analytical sciences in the country. ISAS-DC is now an established scientific forum with sound financial and intellectual backing. We invite all scientists and researchers in the field of science to be part of this professional scientific society and achieve excellence in your sphere of activity.

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Editorial Series ………….. Nobel Laurite

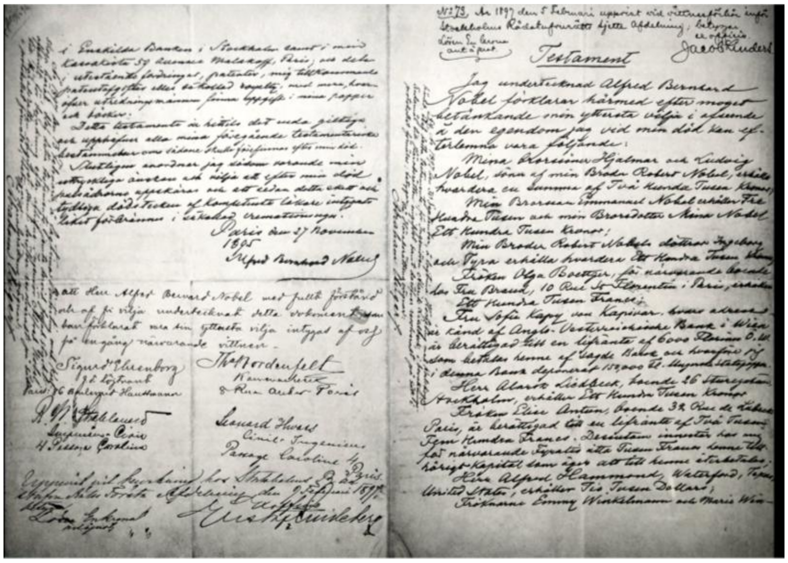
Dear Members,

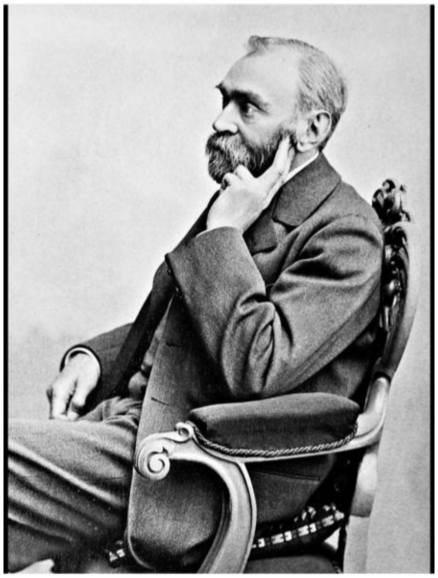
In this editorial series, we would like to cover Nobel Laurates in Science especially Chemistry with details of their significant contributions in their sphere of research.

(Courtesy: Wikipedia)

The Nobel Prize is an international award given to people who have made significant contributions to humanity. The prizes are awarded in the fields of physics, chemistry, physiology or medicine, literature, economics, and peace. The person or organization that receives the Nobel Prize is called a Nobel Prize laureate. The word "laureate" comes from the laurel wreath, which was given to victors in ancient Greece. The Nobel Prize is considered one of the most prestigious awards for intellectual achievement. The prize includes a medal, a personal diploma, and a cash award.

The Nobel Prize was established in the will of Swedish industrialist Alfred Nobel in 1895. On November 27, 1895, Alfred Nobel signed his third and last will at the Swedish-Norwegian Club in Paris. When it was opened and read after his death, the will caused a lot of controversy both in Sweden and internationally, as Nobel had left much of his wealth for the establishment of a prize. His family opposed the establishment of the Nobel Prize, and the prize awarders he named refused to do what he had requested in his will. It was five years before the first Nobel Prize could be awarded in 1901.





(Courtesy: Wikipedia)

In this excerpt of the will, Alfred Nobel dictates that “All of my remaining realisable assets are to be disbursed as follows: the capital, converted to safe securities by my executors, is to constitute a fund, the interest on which is to be distributed annually as prizes to those who, during the preceding year, have conferred the greatest benefit to humankind. The interest is to be divided into five equal parts and distributed as follows: one part to the person who made the most important discovery or invention in the field of physics; one part to the person who made the most important chemical discovery or improvement; one part to the person who made the most important discovery within the domain of physiology or medicine; one part to the person who, in the field of literature, produced the most outstanding work in an idealistic direction; and one part to the person who has done the most or best to advance fellowship among nations, the abolition or reduction of standing armies, and the establishment and promotion of peace congresses.

The Nobel Foundation in Stockholm, Sweden administers the Nobel Prize. In 1968, Sveriges Riksbank (Sweden’s central bank) established the Sveriges Riksbank Prize in Economic Sciences in Memory of Alfred Nobel. Nobel Peace Prize is awarded in Norway because in his will Alfred Nobel laid out who he wanted to be responsible for the selection of the Nobel Prize laureates. The prizes were to be awarded by Swedish institutions – apart from the peace prize, whose award was to be decided by a committee of five persons elected by the Norwegian Parliament.

A Nobel Prize can be shared by up to three individuals, or in the case of the peace prize, it can also be awarded to an organisation. The rule that a prize can only be awarded to three people comes from the statutes of the Nobel Foundation, which is responsible for fulfilling the intentions of Nobel’s will. It specifically states: “In no case may a prize amount be divided between more than three persons.” When an organisation is awarded the Nobel Peace Prize the organisation becomes the laureate, not the individuals working there. Therefore, those people connected to or working for a Nobel Prize awarded organisation cannot in any way refer to themselves personally as a Nobel Prize laureate.

Nobel Prize cannot be awarded posthumously. However, since 1974, if the recipient dies after the prize has been announced they can still be awarded it. Previously, a person could be awarded a prize posthumously if they had already been nominated before 1 February of the same year,

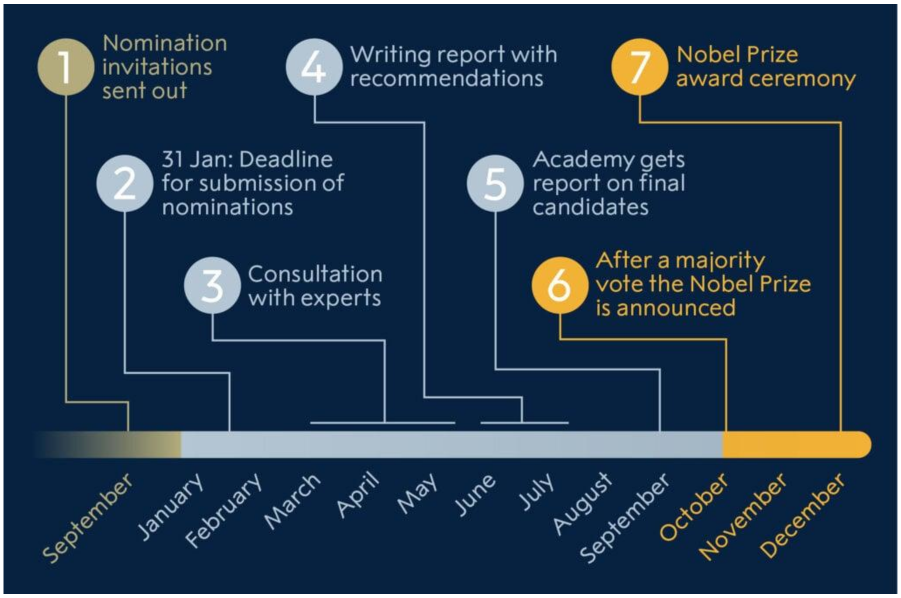
Nomination to the Nobel Prize in Chemistry is by invitation only. The Nobel Committee for Chemistry sends confidential forms to persons who are competent and qualified to nominate. The right to submit proposals for the award of a Nobel Prize in Chemistry shall, by statute, be enjoyed by:

1. Swedish and foreign members of the Royal Swedish Academy of Sciences;
2. Members of the Nobel Committees for Chemistry and Physics;
3. Nobel Prize laureates in chemistry and physics;
4. Permanent professors in the sciences of Chemistry at the universities and institutes of technology of Sweden, Denmark, Finland, Iceland and Norway, and Karolinska Institutet, Stockholm;

5. Holders of corresponding chairs in at least six universities or university colleges selected by the Academy of Sciences with a view to ensuring the appropriate distribution over the different countries and their centers of learning; and
6. Other scientists from whom the Academy may see fit to invite proposals.

The Royal Swedish Academy of Sciences is responsible for the selection of the Nobel Prize laureates in chemistry from among the candidates recommended by the Nobel Committee for Chemistry. The Nobel Committee is the working body that screens the nominations and selects the final candidates. It consists of five members, but for many years the Committee has also adjunct members with the same voting rights as members.

The candidates eligible for the Chemistry Prize are those nominated by qualified persons who have received an invitation from the Nobel Committee to submit names for consideration. No one can nominate himself or herself. nominators must fulfil the criteria set out by the awarding institutions. Below is a brief of the process involved in selecting the Nobel Prize laureates in chemistry.



Nobel Prize process in chemistry. © Nobel Prize Outreach. Ill. Niklas Elmehed

The statutes of the Nobel Foundation restrict disclosure of information about the nominations, whether publicly or privately, for 50 years. The restriction concerns the nominees and nominators, as well as investigations and opinions related to the award of a prize.

References:

https://www.nobelprize.org
https://en.wikipedia.org/wiki/Nobel_laurea
https://www.britannica.com/topic/Nobel-Prize

Continue ….

Lithium-Sulfur Batteries: A Practical Solution for Sustainable Energy Storage Devices

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Abstract

Lithium-sulfur (Li-S) batteries represent one of the most promising candidates for next-generation energy storage systems. With a theoretical energy density of 2600 Wh/kg, they hold the potential to outperform conventional lithium-ion batteries (LIBs) in terms of energy density, cost, and sustainability. However, despite their advantages, several key challenges, such as poor cycle life, low conductivity of sulfur, the polysulfide shuttle effect, and volume expansion, hinder the commercial viability of Li-S batteries. This review aims to provide a detailed analysis of the current state of Li-S battery technology, highlighting recent advancements, material innovations, and strategies to overcome these challenges. We also discuss the prospects of Li-S batteries for various applications, including electric vehicles (EVs), grid energy storage, and portable electronics.

Introduction

Lithium-ion batteries (LIBs) have dominated the field of energy storage for decades, but their relatively modest energy densities (around 150 Wh/kg) and limited resource availability have driven research into alternative battery technologies. Lithium-sulfur (Li-S) batteries have emerged as one of the most promising candidates due to their high theoretical energy density, low cost, and the abundance of sulfur. Sulfur, a naturally abundant element, is inexpensive and environmentally friendly, making it an attractive alternative to the metals used in conventional LIBs. However, despite the remarkable potential of Li-S batteries, their commercialization has been impeded by challenges such as poor sulfur conductivity, polysulfide dissolution, and poor cycle life [1-2]. This review will provide a comprehensive examination of the advancements made in Li-S battery technology, the current challenges, and the various strategies employed to improve their performance and cycle stability. We will also discuss future research directions and the potential applications of Li-S batteries.

Basic Working Principle of Lithium-Sulfur Batteries

A Li-S battery consists of a lithium anode and a sulfur-based cathode. During discharge, sulfur is reduced to lithium sulfide (Li_2S), while lithium ions migrate from the anode to the cathode through an electrolyte. The high energy density of Li-S batteries arises from the high specific capacity of sulfur (1675 mAh /g), which far exceeds that of traditional cathode materials like cobalt oxide (Co_xO_2). Despite this, the practical implementation of Li-S batteries has been hindered by several factors, which are discussed in the following sections.

Key Challenges in Lithium-Sulfur Batteries

Low Electrical Conductivity of Sulfur

Sulfur has a relatively low electrical conductivity ($\sim10^{-14}$ S/cm), which limits its effectiveness as a cathode material. To improve conductivity, sulfur is often incorporated into conductive carbon matrices or composites. For example, mesoporous carbon, graphene, and carbon nanotubes (CNTs) have been explored as sulfur hosts. These materials provide a conductive network that facilitates electron transfer and enhances the performance of the sulfur cathode. However, the inherent conductivity of sulfur remains a significant issue at high discharge rates, necessitating further advancements in sulfur-carbon composites [3-5]

Polysulfide Shuttle Effect

One of the most prominent challenges in Li-S batteries is the dissolution of polysulfides (Li_2S_x, where $4 \leq x \leq 8$) formed during discharge into the electrolyte. These soluble polysulfides migrate to the anode, causing the loss of active material and reducing the efficiency of the battery. This phenomenon, known as the polysulfide shuttle effect, significantly diminishes the cycle life and performance of Li-S batteries. Researchers have developed various strategies to mitigate this issue, such as incorporating polysulfide-adsorbing materials in the cathode. Additionally, functionalized separators and advanced electrolyte formulations have been developed to inhibit polysulfide dissolution [6-8]

Volume Expansion and Mechanical Instability

Sulfur undergoes a substantial volume change (up to 80%) during cycling, which leads to mechanical stress and degradation of the cathode structure. This results in poor cycling stability and capacity fading. To mitigate this issue, sulfur is often encapsulated in carbon materials or composites that can accommodate the volume expansion and provide structural support (Li et al., 2019). Furthermore, sulfur-carbon composites have been designed with porous structures to reduce the mechanical stress and enhance the cycling stability [9-11]

Low Coulombic Efficiency

Coulombic efficiency, which refers to the ratio of discharge capacity to charge capacity, is often low in Li-S batteries due to side reactions such as the formation of a solid electrolyte interphase (SEI) layer and polysulfide dissolution. These side reactions reduce the overall efficiency and lifespan of the battery [12]. By optimizing the electrolyte composition and interface engineering, it is possible to improve the coulombic efficiency and prolong the cycle life of Li-S batteries [13-14].

Recent Advances in Lithium-Sulfur Battery Technology

Advances in Cathode Materials

A key area of research has been the development of advanced cathode materials that can overcome the limitations of sulfur. Some of the most promising approaches include:

1. **Sulfur-Carbon Composites**: Incorporating sulfur into highly conductive carbon matrices, such as mesoporous carbon, graphene, or carbon nanotubes, has been shown to improve conductivity and reduce the dissolution of polysulfides [10]
2. **Polysulfide-Adsorbing Materials**: Various materials, such as metal-organic frameworks (MOFs), graphene oxide, and conductive polymers, have been explored for their ability to adsorb and stabilize polysulfides, thereby mitigating the polysulfide shuttle effect [12].
3. **Sulfur Nanocomposites**: Nanostructured sulfur, such as sulfur nanoparticles or sulfur-polymer composites, offer high surface area and better interaction with the electrolyte, which enhances sulfur utilization and performance .

Electrolyte Optimization

The electrolyte plays a crucial role in the performance of Li-S batteries. Recent research has focused on developing electrolytes that minimize polysulfide dissolution while maintaining high ionic conductivity. These include:

1. **Ionic Liquid Electrolytes**: Ionic liquids have been used to stabilize polysulfides and enhance the electrochemical performance of Li-S batteries [8].
2. **Gel Polymer Electrolytes**: Gel polymer electrolytes offer advantages in terms of safety and stability, preventing dendrite formation and improving cycle life (Zhao et al., 2019).
3. **Solid-State Electrolytes**: Solid-state electrolytes, such as lithium phosphorus oxynitride (LiPON) or sulfide-based materials, have been explored for their potential to stabilize polysulfides and improve the overall performance and safety of Li-S batteries [9].
4.

Separator and Membrane Technology

Separators are critical in Li-S batteries for preventing the migration of polysulfides to the anode. Recent innovations in separator design include:

1. **Polysulfide-Adsorbing Membranes**: Materials such as graphene oxide, carbon nanofibers, and polymer composites have been developed to absorb polysulfides and prevent their migration [10].
2. **Functionalized Separators**: Researchers have also developed separators that are functionalized with hydrophilic or polar groups to interact with polysulfides and reduce shuttle effects [8].

Lithium Anode Protection

The lithium anode is prone to dendrite formation, which can cause short circuits and battery failure. Several strategies have been proposed to mitigate this issue, including:

1. **Solid Electrolyte Interphase (SEI) Formation**: Controlling the formation of the SEI layer can improve the stability of the lithium anode [7].
2. **Lithium Alloying**: Using lithium alloys or lithium composites can reduce dendrite formation and enhance the cycle life of Li-S batteries [7]
3. **Protective Coatings**: Protective coatings, such as polymer films or ceramic layers, have been developed to inhibit dendrite growth and improve the safety of Li-S batteries [8].

Future Directions and Applications

While Li-S batteries have shown considerable promise, there are still significant challenges that need to be addressed before they can be commercially viable. The future of Li-S batteries will likely focus on:

1. **Enhancing Sulfur Utilization**: Strategies to increase sulfur loading and utilization are critical for achieving the high energy density required for practical applications.
2. **Improving Cycle Stability**: Research into advanced sulfur-carbon composites and improved electrolytes will be essential for increasing the cycle life and efficiency of Li-S batteries.
3. **Solid-State Li-S Batteries**: Solid-state electrolytes offer the potential for safer and more stable Li-S batteries with higher energy densities [14-15].

Li-S batteries have the potential to play a significant role in applications such as electric vehicles (EVs), grid energy storage, and portable electronics. Their high energy density makes them ideal candidates for applications where weight and energy capacity are critical factors [15].

Conclusion

Lithium-sulfur batteries offer a promising alternative to traditional lithium-ion batteries due to their high theoretical energy density, low cost, and the abundance of sulfur. Despite several challenges, significant progress has been made in developing strategies to address issues such as poor conductivity, polysulfide dissolution, and low cycle life. Advanced cathode materials, optimized electrolytes, improved separators, and lithium anode protection techniques have all enhanced the performance of Li-S batteries. Continued research and development will be crucial to overcoming the remaining challenges and unlocking the full potential of Li-S batteries for next-generation energy storage applications.

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An Early Prototype of Point of Detection (POD) Device for the Estimation of Morpholine: a Xenobiotic Pollutant

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Background

Xenobiotic pollutant is synthetic chemical substances of anthropogenic origin found with in an environment and organism that is not naturally produced. Examples include but not limited to pesticides, plastics, pharmaceuticals, industrial chemicals, fumigants, solvents, fuels, cosmetics, flavorings, fragrances, food additives, disinfectants, industrial by-products, venoms, and bacterial toxins and many more. In general, xenobiotic pollutants are considered to recalcitrant meaning that they are highly resistant to biodegradation and can persist in the environment for longer period of time; because the enzymes that are used to break down haven't been evolved in their evolutionary period. Therefore they are not recognized by existing metabolic pathway making them "foreign to the organism with unfamiliar structure" lead to toxic effects by disrupting biological processes through mechanisms involving their biotransformation to reactive chemical species by the ingress event of biomagnifications in the food chain of each tropic level. It has been estimated that humans are exposed to 1-3 million xenobiotics in their lifetimes [1]. More than 52 million organic and inorganic substances have been synthesized of which over 39 million are commercially available [2] to potential human exposures via the diet, air, drinking water, drug administration, and lifestyle choices etc. Morpholine is one of important versatile xenobiotic volatile organic chemical (VOC) widely used in plethora of industries, including textile, paper, glass, agriculture, personal care, rubber, pharmaceuticals, and water treatment.

Novel Microscopic Assay of Morpholine

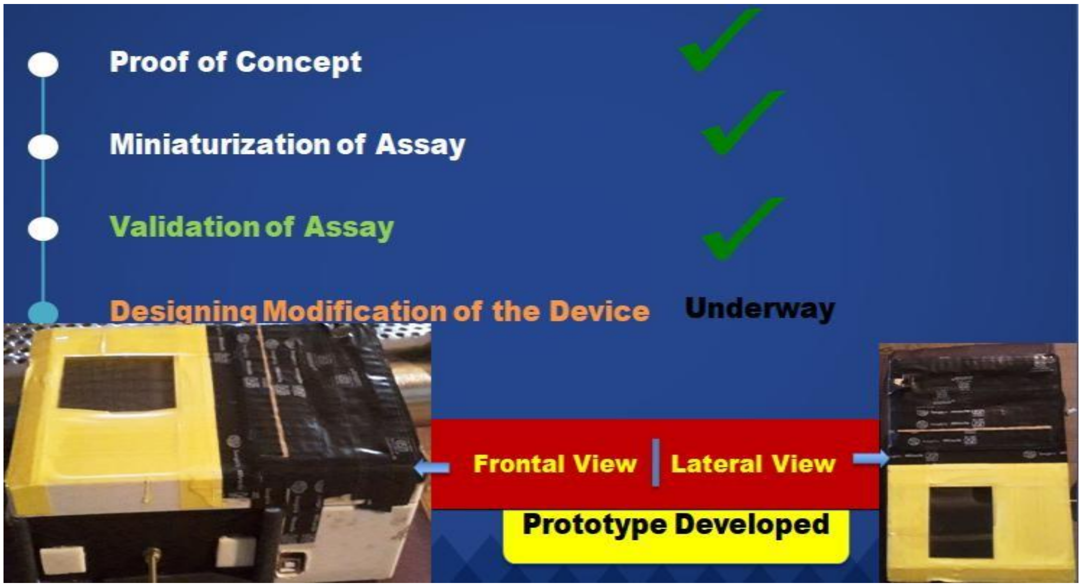
Chemically it is six-membered heterocyclic compound (C_4H_9NO) that features of having secondary amine and ether functionalities. These functional groups are characteristics feature to estimate the presence of morphine. However, many less sensitive and indirect methods based on titration [3] and chemical conversion to oxides- chemical oxygen demand (COD) [4] have been also used in the past 2–3 decades. Several other methods based on analytical instruments such as capillary isotachophoresis [5, 6], gas chromatography (GC) [7] high pressure liquid chromatography (HPLC) [8] ion/cation exchange chromatography [9] are in use for quantification of morpholine. Each analytical method has its own advantage and limitation. In this regard, a microscopic assay has been developed based on the chemical reaction of in which morpholine reacts with sodium salt of 1, 2-naphtoquinone 4-sulphonic acid (NPQ/NQS) under alkaline conditions at room temperature in a span of 20 min (in dark) to form a red/orange coloured product named 4-(4-morpholinyl) naphthalene-1,2-dione which was detected at 480 nm [10]. With the help of this micro-assay, a direct correlate the morpholine concentration with the colour intensity of the product formed in the aforesaid assay reaction is established to quantitate the concentration of morpholine in industrial effluents. It has been shown that untreated gray textile effluents (1: 1000) and treated effluent (1:100) have a concentration of 5000 and 340 ppm morpholine respectively estimated through said assay (Table 1).

Table 1: Quantitative estimation of morpholine in industrial effluent of textile origin

Sample	Dilution factor	Calculated concentration (ppm)	Actual concentration (ppm)
Positive control (Morpholine 0.1%)	1:100	10000.00	1000
Untreated gray effluent	1:1000	5.00	5000
Treated effluent	1:100	3.40	340

Development of POD Device

Based on this microscopic assay, point of detection (POD) device has been prototyped that would quite sensitive enough against the concentration of 2-10 ppm morpholine in industrial effluents at a lab cost of < 0.015 USD ($) only. The beauty of the early POD is that it is simple, rapid and feasible to estimate morpholine. Also, it is requiring minimal reagent (only 2 common reagents of 2µL each), small sample volume (300µL), efforts, infrastructure and skill sets. The further refinement of the proposed POD is underway in order to give a final design to make it miniaturize to fit into pocket and has a potential to enable it to handle large number of industrial samples in a single run (Fig.1).



Proof of Concept ✓

Miniaturization of Assay ✓

Validation of Assay ✓

Designing Modification of the Device Underway

Frontal View | Lateral View

Prototype Developed

Fig.1: Early prototype of the POD device for the estimation of morpholine

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Energy Transition in Indian Oil & Gas Sector: Net Zero Goals and Role of Technology

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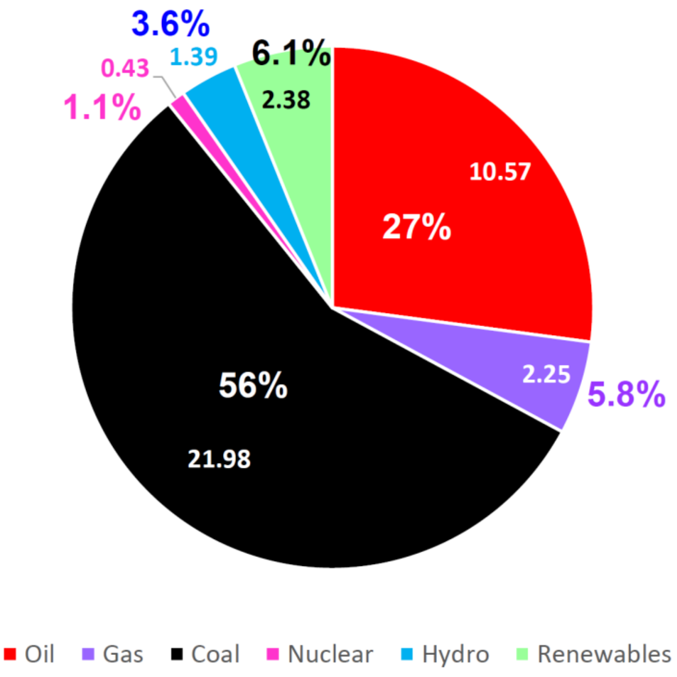
India's announcement that it aims to reach net zero emissions by 2070 has set the agenda for the Indian Industry for next 5-decades. To meet fifty percent of its electricity requirements from renewable energy sources by 2030 is a hugely significant moment for the global fight against climate change. Coal and oil have so far served as bedrocks of India's industrial growth and modernisation, giving a rising number of Indian people access to modern energy services. The rapid growth in fossil energy consumption has also meant India's annual CO_2 emissions have risen to become the third highest in the world

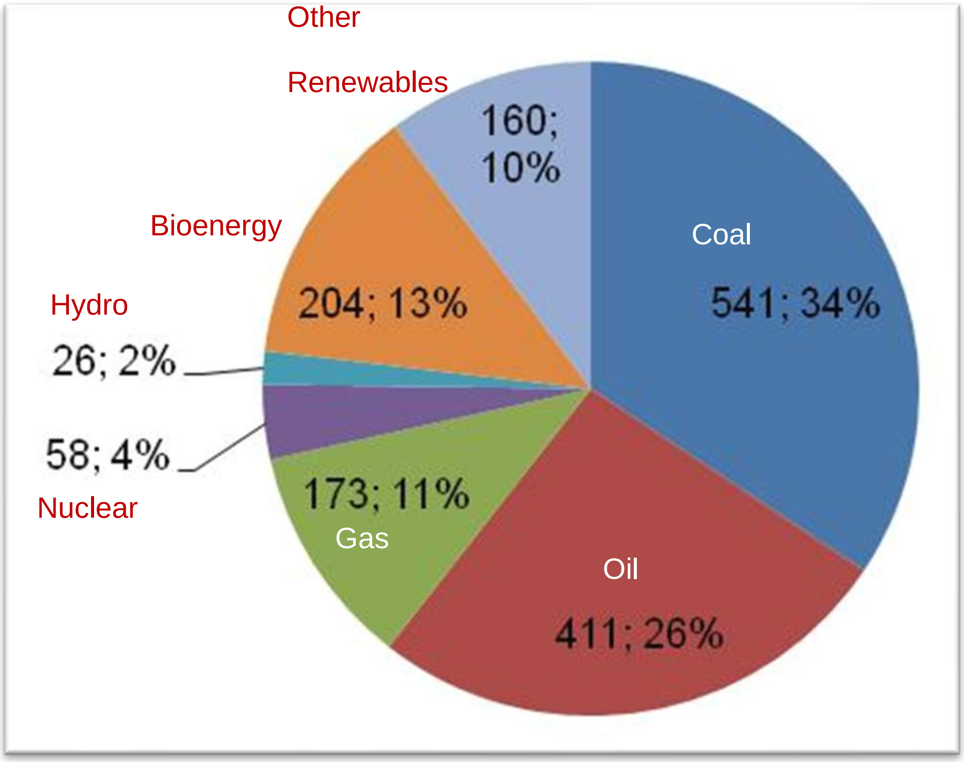
India's Energy Transition (2023-2040)



India is 3rd largest energy consuming country in the world, with a share of 6.4%. India total primary energy demand stands at 39 ExaJoules (EJ) i.e. 936 Mtoe in 2023. Coal (56%) a dominant share in energy mix, way above the global average (27%) followed by Oil & gas. Share of oil & gas at 33% is way below the global average of 50%. Only 17% of total energy consumption in India is in the form of electricity.

However. the scale of transformation in India is stunning. India's sheer size and its huge scope for growth means that its energy demand is set to grow by more than that of any other country in the coming decades. Its economic growth has been among the highest in the world over the past two decades, lifting of millions of people out of poverty. Every year, India adds a city the size of London to its urban population, involving vast construction of new buildings, factories and transportation networks. This includes adding new electricity connections for 50 million citizens each year over the past decade. Extra 300 Mn vehicles are expected to be added by 2040 and energy demand for road transport to double by 2040 (220 mtoe).

Therefore, by 2040, India's total primary energy demand expected to grow to 1527.9 Mtoe, with a global share of 9.2%.

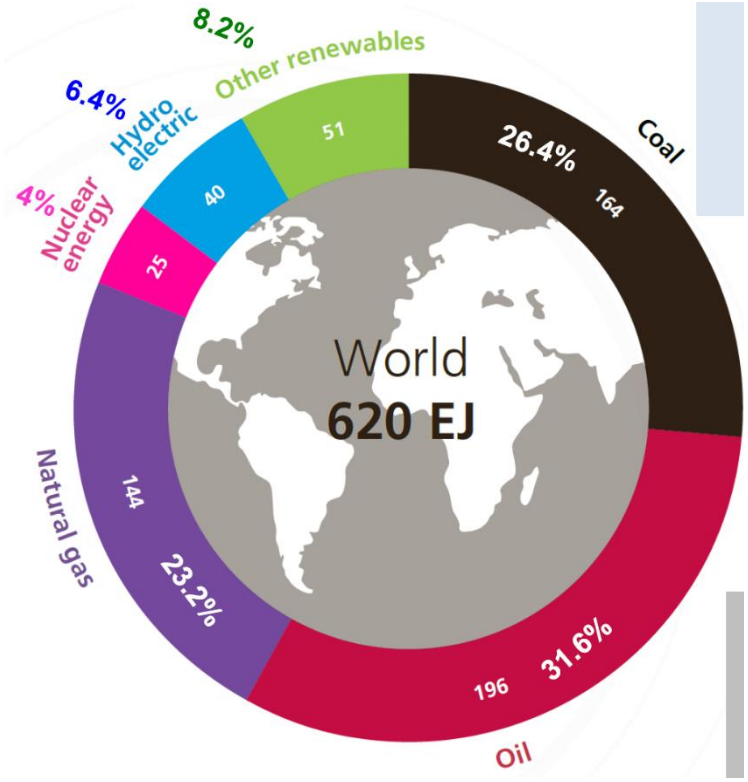


The salient features are:

Share of coal declines from 56% in 2023 to 34% in 2040. Oil & Gas continues to dominate a fast-growing transport sector, with share increasing from 32% to 37% mainly driven by increased gas penetration. India's crude oil demand will rise by almost 4 mb/d to reach 8.7 mb/d in 2040, the largest increase of any country. Gas penetration, renewables, bio-energy show significant potential but to go a long way.

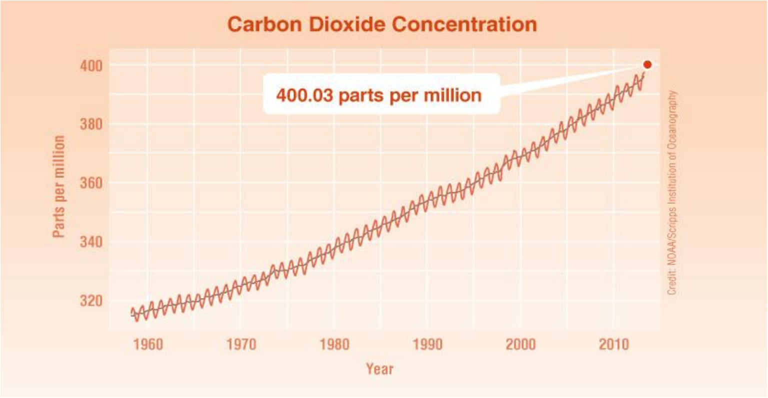
Global Primary Energy Demand:

The global primary energy consumption today is about **620 Exajoules (1x10^{18} Joules),** with the following breakup. The share of coal and oil is predominant ~ 58%, followed by gas which is about 24%. This means world's heavy dependence on fossil energy, which has resulted in current CO_2 emissions trajectory.



The Emissions Story and Global Warming:

Post industrialisation, there has been unprecedented rise in the atmosphere CO_2 concentration with the result that the world todays face the greatest climate challenges of all the times. The CO_2 concentration in the atmosphere has reached about 418 ppm in 2020, compared to about 280 ppm in 1960, the pre-industrial era, which is on an average an increase of 2.3 ppm per year. The last time, CO_2 concentration were this high was in the Pliocene period, about 3-5 million years ago. At that time, there were no people, sea level was about five to 40 meters higher than normal and the planet was much hotter and wetter, it was roughly 3 to 4°C higher than the average global temperature today.



Global carbon dioxide emissions from fossil fuels and industry were 37 billion metric tons ($GtCO_2$) in 2022. The two biggest contributors to global emissions are China and the United States, who produced 11.47 and 5.01 $GtCO_2$ in 2022, respectively. India with a population of 1.4 bn constitutes 17% of global population but its contribution to the emissions has only been ~7%. By 2040, India will be world's 2nd largest emitter after China, but per capita emission at 2.4t remains much lower than others.

The Greenhouse gas (GHG) emissions which include: carbon dioxide, methane and nitrous oxide from all sources, including agriculture and land use change are much higher and stands today at a level of 55 billion metric tons. They are measured in carbon dioxide-equivalents over a 100-year timescale.

Technological Options for Indian Oil & Gas Sector to achieve Net Zero

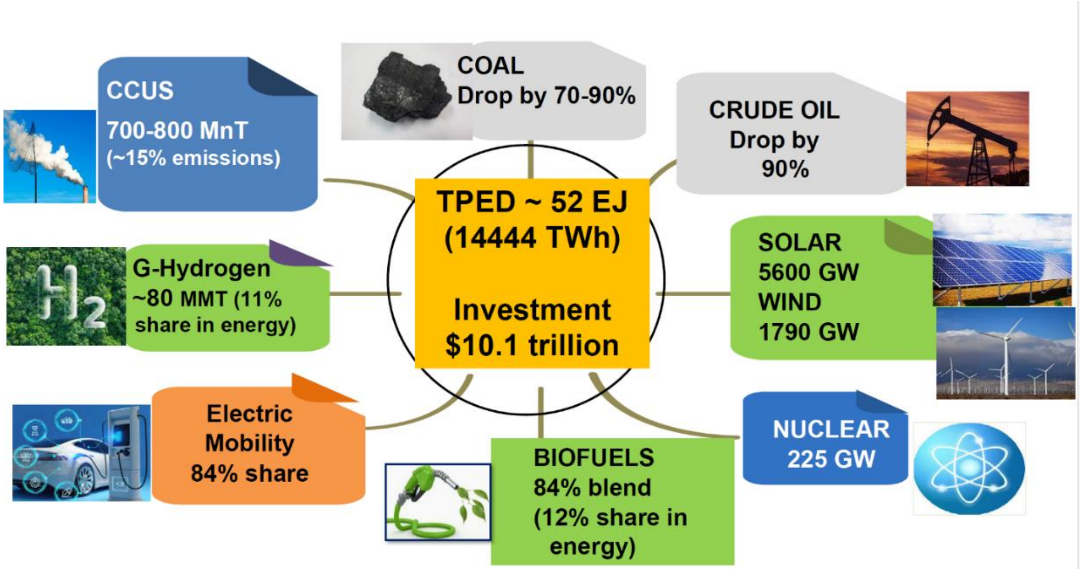
India remains a energy hungry country i.e. India Needs More Energy… but with Less Carbon in order to meet the stated goal of Net Zero by 2070. Indian Oil & Gas sector is in a unique position due to robust projected growth and strong financials to contribute towards achieving Net Zero goal. While staying strong in the traditional business, companies have the multiple options:

1. Refinery Capacity Expansion- Sustainable Refineries including bio-refineries, producing Sustainable Aviation Fuel (SAF)
2. Accelerating Efforts Towards A Gas-based Economy
3. Waste to Energy and Bioenergy (1G, 2G & 3G Technologies Ethanol technologies), CBG
4. Increased Petrochemical Intensity coupled Plastic Circularity & Neutrality
5. Carbon Capture, Storage and Utilization (CCSU)
6. Renewable Power & Clean Cooking Fuel
7. Hydrogen and Fuel Cell – Production technologies with much reduced cost of production, Green Hydrogen @ Refineries, Electric Mobility thru Fuel Cell applications

8. Electrolyser Technologies coupled with Solar and wind energy
9. EVs and Advanced Chemistry Cell (ACC) Battery Storage infrastructure and technology development suiting to India conditions
10. Material & Energy Efficiency
11. Digitalization for efficient energy delivery

Getting India to Net Zero by 2070

By 2070, India's primary energy demand is expected to reach 52 EJ (14444 TWh). As per a simulation study by CEEW, in this scenario, achieving net zero emissions means that India's coal usage should drop by 70-90%, crude oil drop by 90%, Solar and wind capacities required would be 5600 GW & 1790 GW respectively, Nuclear should reach 225 GW, Biofuel blending should increase to 84%, Electric mobility share should increase to 84%, green hydrogen production should reach 80 Mnt. This off course would require huge financial out go to the tune of 10 Trillion USD.



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Correlation of Chloride analysis in Reforming Unit Catalyst between Potentiometric Titration & novel method of Energy Dispersive X-ray Fluorescence spectroscopy

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Introduction

Continuous Catalyst Regeneration Unit (CCRU) of Reforming Unit produces high Octane reformate from straight run naphtha through catalytic reforming process. The activity of the catalyst gets reduced with time during unit operation because of carbonaceous coke deposition and chloride loss. The catalyst is periodically regenerated or restored by in situ high temperature oxidation of the coke followed by chlorination.

Chloride content, depending on its deposition concentration on catalyst, impacts the effectiveness of the catalyst which in-turn affects the refining process. Therefore, by monitoring chlorine concentration in catalyst helps refiners to determine how long their catalysts can be used before becoming spent or needing regeneration.

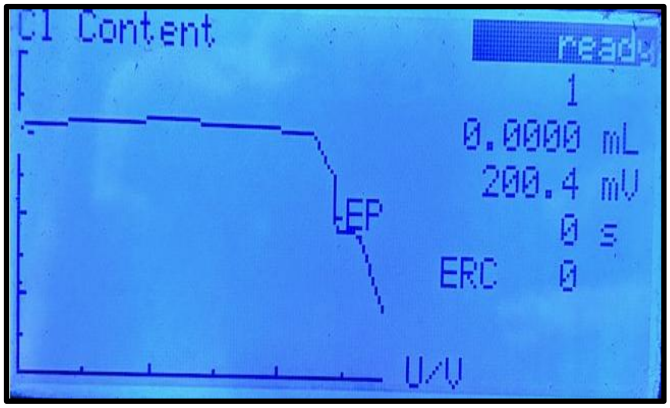
Inorder to monitor the catalyst deposition while unit operation, chloride concentration is checked regularly in refineries by UOP 291[1] method for determining the total chloride content of regenerated and spent alumina-supported catalysts.

The present study aims at devising alternate method for chloride analysis, which is accurate in terms of repetition and reproducibility, fast technique than existing UOP method. Thus Energy Dispersive X-ray Fluorescence (ED-XRF) offers fast (single step procedure), accurate and better alternate analysis technique which is user friendly and does not involves use of hazardous chemicals. This work aims in establishing correlation between the two analytical technique for determining chloride in spent and regenerated catalyst of reforming units as well as in catalyst of Penex Unit.

Material and Method

A. Conventionally, chloride content is determined for alumina and silica-alumina catalyst through potentiometric titration method of UOP-291.

A typical potentiometric curve is depicted in Graph-1 where the EP represents the End Point of the titration which is generally the median of the curve. The quantity of silver nitrate consumed and the milli volt generated can be seen in the Graph-1.



Graph:-1 Potentiometric Graph of Chloride Determination

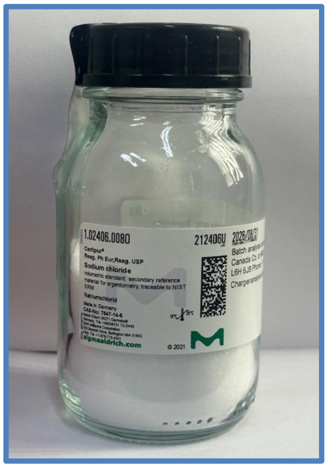


Fig:-1) Sodium Chloride CRM

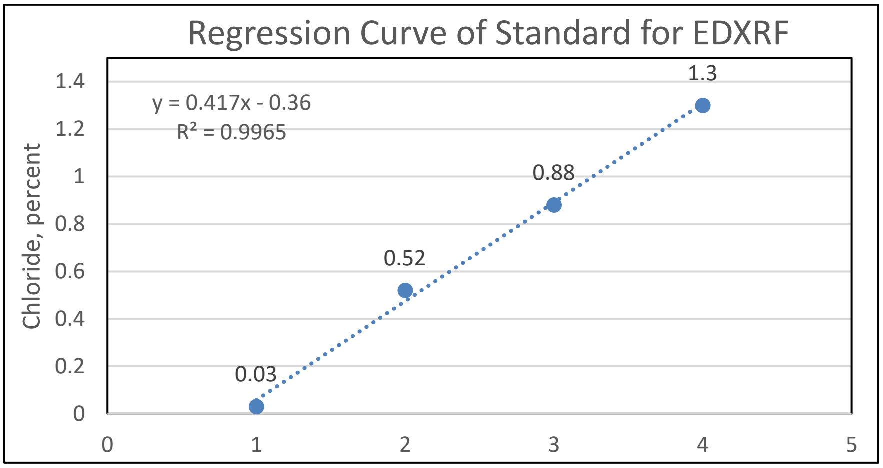
Catalyst contains both organic and inorganic chloride content. Inorganic chloride content can be directly determined by titration, however the organic chloride needs to be converted into inorganic chloride which is then estimated through titration. Thus the UOP method determines inorganic chloride as total chloride in the catalyst after converting organic into inorganic chloride. Titration Silver Nitrate ($AgNO_3$) was standardized using Merck make Sodium Chloride (NaCl) (CAS No. 7647-14-5).

B. XRF or X-ray fluorescence spectroscopy, is an analytical technique used to determine the elemental composition of a material. It is non-destructive, reliable, accurate, requires very little time, easy sample preparation and is suitable for all forms of solid, liquid and powdered samples. These qualities make XRF technique a better choice for determination of chloride content in reforming unit catalyst. The technique consist of introducing sample to X-ray source, where the element in the sample gets excited simultaneously and an energy dispersive detector is used to simultaneously collect the fluorescence radiation emitted from sample. The detector then separate different energies of the characteristic radiation from each of the different sample elements. Each sample was analyzed in

Triplicate and the average of the results was considered for comparison with the potentiometric method. Same procedure was utilized for analysis of chloride in both spent and regenerated catalyst. For each analysis approximately 4.0 gm of sample was used in finely grounded power form and total time taken for complete analysis was approximately 15 minutes with results reported in weight percent.

The regression line in ED-XRF is established using a series of matrix-matched catalyst samples (Spent catalyst). The primary concentrations determined for reference chloride were obtained by UOP 9792,3 vis-a-vis UOP 291. The standard deviation of both the methods were determined to check the repeatability of ED-XRF technique.

Graph:1- Calibration curve using catalyst concentration of 0.03 % 0.52%, 0.88%, 1.30%, was drawn in ED-XRF.



The results of spent and regenerated catalyst obtained from EDXRF were subjected for cross verification through different ED-XRF instruments at different IOCL locations, by different user. The results obtained from different IOCL locations were compared with the results of Mathura Refinery (Table 6). All the results obtained were within the reproducibility limit of the method / technique.

Result and Discussion

Under this study, the results obtained from EDXRF were compared and correlated with the results obtained from potentiometric titration method. Correlation of conventional titration method with ED-

XRF technique for chloride determination of reforming units' catalyst was done on 168 number of samples, 84 for each type of Catalyst i.e. spent and regenerated.

It is observed, from table-2 that chloride concentration reported by ED-XRF technique is in close relation with the Potentiometric titration method for Regenerated catalyst (Table-2, Graph-1). In most of the cases the results of ED-XRF techniques are well within the reproducibility of Potentiometric Titration method (UOP 291) i.e. ± 0.06. The Pearson's Coefficient r = 0.94 (n = 84) suggests a close resemblance with the results obtained from both the methods.

The chloride content of spent catalyst was compared using both the techniques (i.e. Potentiometric Titration and ED-XRF). From table -4, it is observed, that chloride concentration by ED-XRF technique for spent catalyst also is in close relation with the concentration by Potentiometric titration method (Table-2, Graph-1). In most of the cases the results of ED-XRF techniques are well within the reproducibility of Potentiometric Titration method (UOP 291) i.e. ± 0.06. The Pearson's Coefficient r = 0.95 (n = 84) further suggests a close resemblance of the results obtained from both the methods.

In both the cases (regenerated and spent catalyst the results by ED-XRF method are found well within the reproducibility of limit of UOP-291. To establish the reliability of the ED-XRF technique for determination of chloride of spent catalyst by ED-XRF technique, the same sample was analyzed under the same set of similar conditions viz. instrument, analyst, ED-XRF film, time, location, environmental conditions and within short gap, using standard deviation σw.

The samples of spent and regenerated catalyst were further analyzed on different ED-XRF (ED-XRF-2) instrument, at different time, location and under different environmental condition and the results were compared:

Table 6: Chloride results in reforming unit catalyst at Mathura Refinery and other location

S. NO.	Chloride results in reforming unit catalyst at Mathura Refinery and other location			
	Regenerated Catalyst		Spent catalyst	
	ED-XRF-1 (MR)	ED-XRF-2 (Other Location)	ED-XRF-1 (MR)	ED-XRF-2 (Other Location)
1	1.27	1.23	0.99	1.05
2	1.19	1.15	1.22	1.26
3	1.25	1.30	1.13	1.16
4	1.24	1.29	1.17	1.22
5	1.22	1.18	1.25	1.21

6	1.12	1.09	1.01	0.98
7	1.11	1.07	1.06	1.11
8	1.00	0.95	0.97	1.01
9	1.15	1.11	0.92	0.98
10	1.17	1.15	1.02	1.05

The standard deviation of ED -XRF data for spent and regenerated catalyst was found to be 0.06, which shows less variation from the mean value.

Table 7: Chloride results of Penex catalyst by Potentiometric Titration and ED-XRF

S. No.	Titration Results	ED-XRF
1	3.83	3.80
2	3.85	3.81
3	3.83	3.79
4	3.84	3.80
5	3.85	3.80

Conclusion

Therefore the above it is inferred that that ED-XRF is an excellent alternative test method for determination of chloride in Reforming catalyst as well other Penex catalyst. This method is hassle free, less time consuming per analysis (20-25 minute) than potentiometric method (approx 4 hours). Apart from these, the method does not require solvents and chemicals, no infrastructure for catalyst digestion which has certain hazard associated with it, which can be eliminated by using EDXRF method. Moreover, ED-XRF method does not results in any liquid waste generation and does not require handling of any acid as well. It is much cleaner method, which is easy, robust and faster.

Colours of Hydrogen

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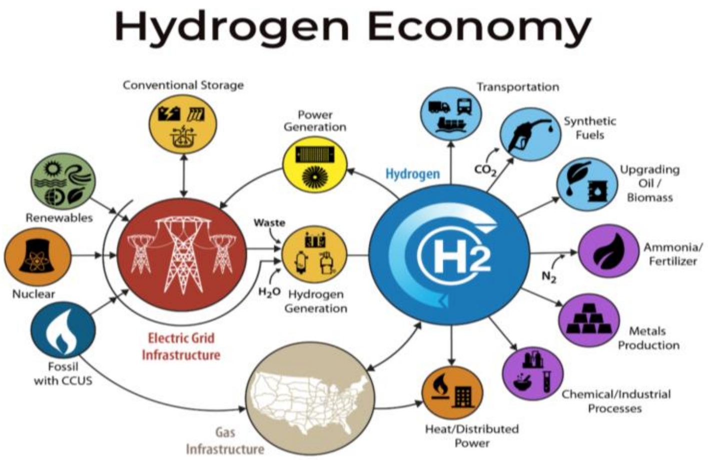
Generally, Hydrogen gas has been supplied in different purity levels with three grades, namely, pure hydrogen (purity ≥ 99.99%), high pure hydrogen (purity ≥ 99.999%), and ultrapure hydrogen (purity ≥ 99.9999%). We all know that Hydrogen has No colour. No smell and No taste due to which lot of precautions to be followed while using hydrogen for various applications. In the energy transition scenario, focus has been turned towards usage hydrogen for various applications. Hydrogen is a clean and efficient fuel and has a wide range of applications across various industries due to its versatility as an energy carrier.



n recent years, hydrogen has been decorated with various colours based on the source and technology of production. Though hydrogen is colourless, colour codes are being used in the energy industry to differentiate different types of hydrogen. In this article, the characteristics of various colour coded hydrogen used in energy industry are discussed.

Grey Hydrogen

Grey hydrogen is the most common form of hydrogen production. It is produced from natural gas, or methane, using steam methane reforming (SMR) process using natural gas and water in the form of steam. The output is hydrogen, but contains carbon dioxide as a by-product. This hydrogen is supplied as such without capturing the greenhouse gases like CO_2 made in the process and therefore, contributing significantly to greenhouse gas emissions and climate change.

Blue Hydrogen

Blue hydrogen is also produced by steam reforming process used for grey hydrogen but CO_2 is captured by carbon capture and storage (CCS) process therefore, environment friendly and does not contribute to climatic change. It is also called as low-carbon hydrogen as it contains almost half the amount of CO_2 compared to grey hydrogen.

Green Hydrogen

As the name suggests, green hydrogen is the most climate-friendly approach for hydrogen production. Green hydrogen is made by using clean electricity from surplus renewable energy sources, such as solar or wind power, to electrolyse water. Electrolysers use an electrochemical reaction to split water into its components of hydrogen and oxygen, emitting zero-carbon dioxide in the process but it is an expensive process

Brown Hydrogen

Gasification of brown coal (lignite) is the process used for production of brown hydrogen. In this process, coal is converted to hydrogen and oxides of carbon by reacting it with steam and oxygen at high temperatures. The gasification process releases substantial amounts of CO_2 and CO into the atmosphere, therefore not environmentally friendly and causes damage to the atmosphere significantly

Black Hydrogen

This is also produced by gasification process, but black coal (anthracite) is used in this process. Like brown hydrogen, the environmental impact of black hydrogen is considerable and making it less favorable process.

Pink Hydrogen

Pink hydrogen is generated through electrolysis powered by nuclear energy. This is also called as purple hydrogen or red hydrogen. This method does not emit greenhouse gases during production, making it environmentally friendly like green hydrogen. However, the process is relatively expensive due to the costs associated with nuclear reactors.

Turquoise Hydrogen

Turquoise hydrogen is made using a process called methane pyrolysis to produce hydrogen and solid carbon without emission of CO_2 and CO. The solid carbon can be utilized for various applications, like tires, coatings, and batteries. This approach is likely to dominant in future and compete with green hydrogen.

Yellow Hydrogen

Yellow hydrogen is a relatively new phrase for hydrogen made through electrolysis using electricity generated from solar power to split water molecules into hydrogen and oxygen, resulting in a clean and sustainable energy source. The process is almost similar to green hydrogen.

White Hydrogen

White hydrogen is a naturally occurring hydrogen found in earth's crust and formed through geological processes like reaction of minerals with water. It is also called as gold hydrogen or natural hydrogen. This is the latest colour added to the rainbow of hydrogen. This hydrogen is expected to be completely free from carbon and compete with green hydrogen. White hydrogen presents a promising avenue for future energy production, however, at present, white hydrogen industry is still in its early stages and not exploited fully for its usage.

There is a wide opportunity for hydrogen, as a clean fuel with high energy intensity, to play a significant role various industrial and transport sectors. However, overcoming the challenges like low cost of production, safe transportation, storage and usage will make hydrogen as the preferred energy carrier. The hydrogen rainbow with different colours will play a significant role in reaching net zero target set by all counties,

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Alzheimer's Disease (AD): A Major Public Health Problem

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Alzheimer's Disease (AD) is a neurodegenerative disease and comes under the umbrella of dementia. Neurodegenerative diseases are classified as chronic conditions that result in the gradual loss of neurons [1]. AD is a predominant form of dementia which is becoming a world-wide health challenge. Approximately 80% of dementia patients are known to suffer from AD. This disease results in a loss of motor, cognitive, and autonomic functions and have a significant impact on the quality of life of affected individuals [2].

Dr. Alois Alzheimer, a German psychiatrist and neuropathologist, first time observed related symptoms in his patient Auguste Deter in her early 50s. The patient died at the age of 55 but left a question mark to Dr. Alzheimer. After she died, Dr. Alzheimer performed a post-mortem examination of her brain and noted two characteristic abnormalities: amyloid plaques and neurofibrillary tangles, which are presently known to be defining features of AD [3]. Symptoms of AD generally appear gradually and increase over time. Early symptoms often involve, memory loss, language difficulties, confusion, behavioral changes etc. [4]. As the illness advances, patients may develop more severe cognitive impairments, such as:

- Inability to recognize familiar faces or carry out everyday activities.
- Increased confusion and difficulty with abstract thinking.
- Personality changes, e.g., agitation or aggression.

As the time passed, more and more people throughout the globe started reporting the symptoms of forgetfulness, behavioral changes, and other symptoms as given above. According to statistical data by WHO, some facts about dementia are summarized in Table 1 [5].

Throughout the years, many hypotheses have been formulated to account for its pathogenesis, each of which has helped us understand the disease. Some of the principal hypotheses are as follows [6]:

1. Amyloid Hypothesis: Based on amyloid hypothesis, accumulation of amyloid-beta (Aβ) peptides in the brain resulted in the development of plaques that initiate a series of neurodegenerative processes. The hypothesis was developed early in the 1990s when scientists detected genetic mutations responsible for Aβ production and deposition [7].

Table 1. Statistical data and facts about dementia [3].

S. No.	Facts	World-wide data
1.	Current global prevalence	Over 55 million
2.	Annual new cases	10 million
3.	Expected global prevalence by 2030	78 million
4.	Expected global prevalence by 2050	139 million
5.	Disproportionate Impact	Over 60% of people live in low- and middle-income countries
6.	Gender	Women disproportionately affected

2. Tau Hypothesis: The tau hypothesis targets the involvement of hyperphosphorylated tau protein in forming neurofibrillary tangles inside neurons. Such tangles impair neuronal function and lead to cell death. The tau pathology has traditionally been regarded as a downstream consequence of amyloid deposition, but current evidence indicates that it could also possess independent pathogenic mechanisms [8].

3. Cholinergic Hypothesis: This early hypothesis suggests that the degeneration of cholinergic neurons in the basal forebrain leads to cognitive deficits observed in AD. The loss of acetylcholine, a neurotransmitter crucial for learning and memory, correlates with the severity of dementia symptoms. While cholinesterase inhibitors have been used to treat symptoms, they do not halt disease progression [9].

4. Inflammatory Hypothesis: Neuroinflammation has also been implicated as a primary cause in the progression of AD. According to this hypothesis, chronic brain inflammation due to activated microglia and astrocytes contributes to damage and deterioration of neurons and cognitive impairment. The inflammatory process could be instigated by Aβ deposition or other mechanisms that propagate each other through a vicious cycle exacerbating neurodegeneration [10].

Treatments available

Traditionally, AD treatment was confined to drugs that only controlled symptoms and not causes. The advent of disease-modifying therapies (DMTs) represents a significant change in the paradigm. In recent years, there have been a number of hopeful drugs [11-13] available that slow the progression of the diseases.

- Aducanumab (Aduhelm): Approved in 2021 as the first to reduce amyloid plaques in the brain. While its effectiveness and approval were met with controversy, it's a landmark treatment for Alzheimer's.
- Lecanemab (Leqembi): Approved in early 2023, this treatment has demonstrated to slow cognitive deterioration in early AD patients.
- Donanemab (Kisunla): Another potentially promising DMT for targeting amyloid proteins.
- Remternetug: A new drug currently under investigation that also targets amyloid accumulation.
- Donepezil, Galantamine, and Rivastigmine: These are acetylcholinesterase (AChE) inhibitors, licensed for the treatment of people with mild to moderate AD.

The available drugs only slow down the progression of the diseases. The challenges remain regarding their complete cure or reversal of the disease.

In conclusion, AD is a formidable challenge in the world of neurodegenerative diseases. Cognitive dysfunction and memory decline are the two prominent symptoms. This disease becomes more fatal with comorbidities. The growing death rates emphasize the imperative need for successful therapies. Although present-day drugs mostly treat symptoms, new developments bring hope for more potent interventions addressing the disease's root mechanisms. As research advances, there is promise for breakthroughs that not only enhance treatment outcomes but also the quality of life for patients and caregivers. Modifying risk factors through lifestyle changes may also play a role in preventing or delaying the development of AD. The combined efforts of researchers, clinicians, and policymakers will be critical in charting this multifaceted landscape toward better care and outcomes for individuals with AD.

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Analytical Method Validation - Importance and Concepts

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Analytical method validation is having immense importance in most of the industries viz petroleum, pharma, health, environmental, forensic, chemical etc. Methods used for technology / process optimisation and product development need to be validated to meet the intended use requirements. Non-validated analytical methods lead to technology or product failures and need further investments of time / labour / money for root cause analysis aspects. Test method with more accuracy not only helps the technology for developing better performing product but helps in lowering the technology or product costs to get the better market advantage.

- **Qualitative analysis** is a method that determines if a substance is present in a sample. It is also used to identify non-numerical information about a chemical reaction or species (Inference: Pass or Fail / Classification or Type)

- **Quantitative analysis** is a method for determining the amount or concentration of a substance in a sample. It involves measuring the sample's physical properties and performing calculation

For qualitative and quantitative analysis, we can follow

- **Classical methods** of analysis use chemical reactions to analyze a material. They are also known as wet chemical analysis, e.g., Volumetric Titrations, Gravimetry Analysis, Flame Test etc.

- **Instrumental methods** of analysis are analytical techniques that use machines to study the composition of substances. They can be used to identify elements, compounds, and their molecular structures. Eg Spectroscopy, Microscopy Chromatography (Gas & Liquid), Thermal Analysis

Test methods for qualitative analysis (pass or fail or classification) not having much significance in regulatory environment and does not require to demonstrate the validation aspects. However, all the test methods produce quantitative information need to be demonstrated for analytical method validation parameters viz specificity or selectivity, linearity or range, detection limit, quantification limit, precision (repeatability or reproducibility), accuracy or bias. In recent years, most of the industries are using sophisticated analytical techniques to meet the regulatory compliance and validation of these analytical methods becomes more complex compared to conventional or non-sophisticated analytical test methods. Most of the standard test methods (ASTM / ISO / BIS / IP / UOP / Pharmacopeia) are matrix specific and use of these methods other than specified matrix results in erroneous estimations.

For effective planning of method validation program with any analytical technique requires the sound knowledge of definitions and data interpretations through statistics for validation parameter performance evaluation. Considering the importance of validation parameter, in-house acceptance criteria can be made stringent to meet the customer accuracy requirements. More accurate or repeatable method development or validation process requires more investment of money / time / labour. There is no need of stringent acceptance criteria for validation parameters for the methods where customer is not requires the more accurate results.

Definitions of validation parameters and more relevant statistical parameters useful for performance evaluation given below

- **Specificity:** Specificity of the test method is a measure of method capability to distinguish analyte response in presence of sample matrix. Different levels of analyte spiked to sample matrix and response should be linear to meet the test method specificity.
- **Linearity:** Linearity is defined as the correlation of instrument response (y- axis) and analyte concentration (x-axis). Correlation factor or R2 value more than 0.99 is considered as acceptable correlation for using regression parameters (slope and intercept) in analyte predictions. Number of standards used in linearity is more important and preferably 6 or more concentrations.
  - For assay test methods linearity concentration : 20 to 120% of spec level
  - For impurity test methods linearity concentration rage: 50 to 150% of spec level

  Note: 100% = Analyte specification level

Regression Parameters

a) Pearson correlation coefficient:

$$r = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum (x_i - \bar{x})^2 \sum (y_i - \bar{y})^2}}$$

xi = concentration in standard, $\bar{x}$ = mean of concentrations,
yi = response of instrument, $\bar{y}$ = mean of response

b) STEYX = Standard Error of predicted Y (Response) for each X (Concentration)

c) Slope = r *(S_y / S_x)

- r is the correlation coefficient between x and y.
- S_y is the standard deviation of the y-values.
- S_x is the standard deviation of the x-values.

- **Detection Limit (DL):** Detection Limit defined as lowest concentration of the analyte that can be reliably detected by the analytical method. This can be calculated from the 3.3 times to STEYX / Slope. %RSD is calculated for DL level preparations and typical acceptance criteria is not more than 30.

- **Quantitation Limit (QL):** Quantification Limit (QL) defined as lowest concentration of the analyte that can be quantified with acceptable precision and accuracy. This can be calculated from the 10 times to STEYX / Slope. %RSD is calculated for QL level preparations and typical acceptance criteria is not more than 10.

- **Precision:** Precision defined as evaluation of the method performance against small deliberate variation in method parameters (Intra lab results). Six preparations of QL level or Assay level are tested with proposed method. % RSD (Relative Standard Deviation) of the six measurements of LOQ level and Assay level should meet the acceptance criteria. Precision measurements

  % RSD = 100 * STDEV of measured analyte conc / Average of measured concentration

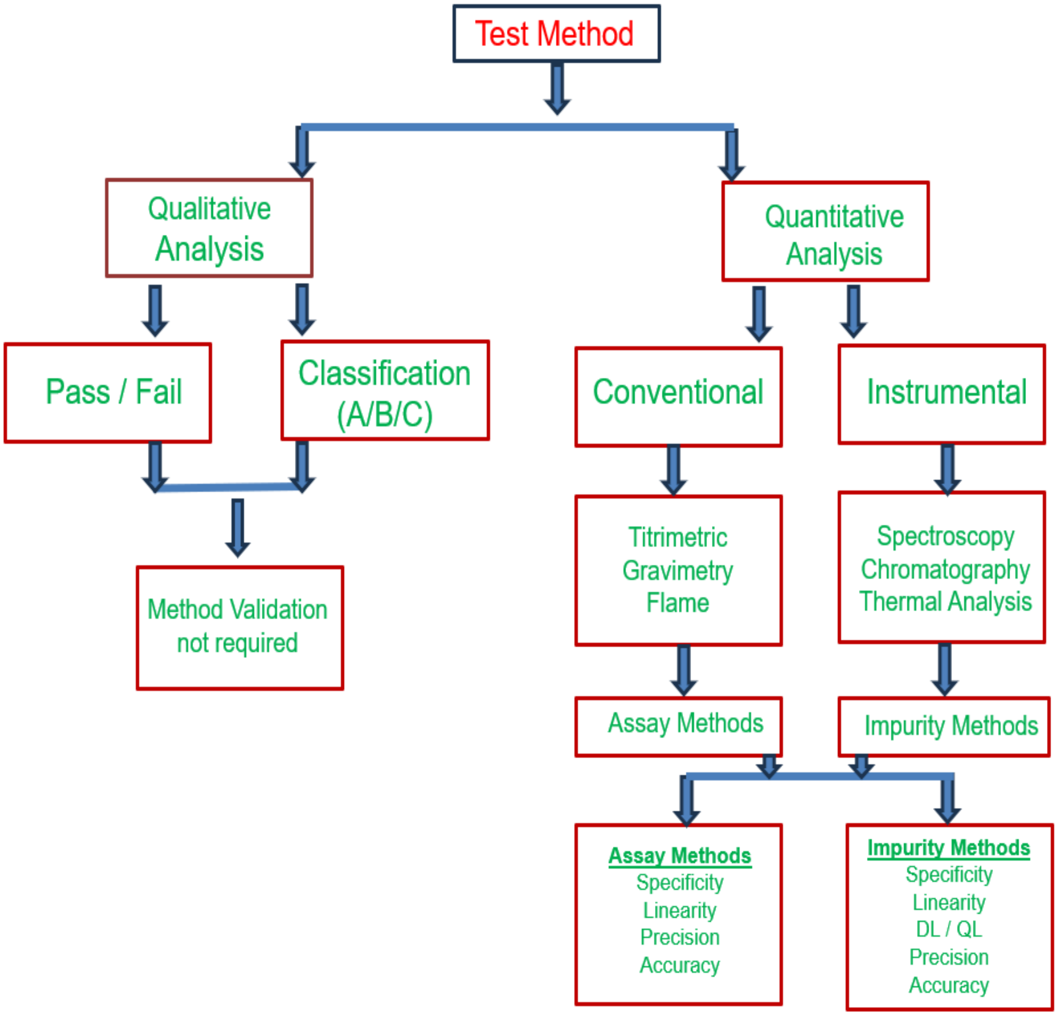
  % RSDs are reported against the acceptance criteria:
  10% Max for QL level (Impurity test)
  2% or 5% for Assay level tests

- **Accuracy / Bias:** Assessment of the difference between the measured value and true value. For accuracy experiments, certified reference materials (CRM) of analyte in similar matrix are tested with the proposed method and the difference of observed and certified value is reported as bias. If the CRM's with similar matrix of interest is not available then certified analyte spiked to sample matrix at different levels. For impurity tests QL level spiking is preferred whereas for assay tests specification level assay amount is preferred. % Recoveries are reported against acceptance criteria 80 to 120% for QL level and 95 to 105% for Assay level).

$$\% \text{ Recovery} = 100 * (C_{SS} - C_{S}) / C_{SPK}$$

C_{SS} = Observed analyte concentration in spiked sample
C_{S} = Observed analyte concentration in sample
C_{SPK} = Analyte conc used for spiking study

Flow chart for method validation parameters selection



References:

1) ICH Guideline - Q2 (R2) Validation of Analytical Procedures
2) ASTM E177 Standard Practice for Use of the Terms Precision and Bias in Test Methods
3) ASTM E1488 Guide for statistical procedures to use in developing and applying test methods
4) ASTM E2586 Practice for calculating and using basic statistics

Forthcoming international events in Analytical Science

- **MassSpecMeet 2025: European Congress on Mass Spectrometry & Analytical Techniques**,
  Berlin, Germany, April 14-15, 2025.
- **International Conference on Forensic Analytical Chemistry, Chemometrics and Statistics:**
  Male, Maldives, on April 19, 2025.
- **International Conference on Analytical Chemistry & Applied Spectroscopy. ICACAS-25**
  Florence, Italy, April 21-22, 2025.
- **12th Analytical Biosciences Group Early Career Researcher**
  Sheffield, United Kingdom, on April 24-25, 2025.
- **6th International Workshop on Analytical Metabolomics:**
  Thessaloniki, Greece, on May 5-6, 2025.
- **10th Conference for Analytical Sciences in Ireland:**
  Cork, Ireland, July 2-4, 2025.
- **Analytical Acta 2025: Global Summit on Analytical and Bioanalytical Techniques,**
  France, July 17-18, 2025.
- **International Conference on Analytical Chemistry (ICAC):**
  Zurich, Switzerland on July 29-30, 2025.
- **AOAC INTERNATIONAL Annual Meeting & Exposition**:
  San Diego, California, USA, from August 23 to 28, 2025,
- **Euroanalysis 2025:**
  Barcelona, Spain. August31 to September 4, 2025
- **14th International Conference on “Instrumental Methods of Analysis”**
  Kefalonia, Greece, September 14-17, 2025.
- **Analytical Research Forum 2025 (ARF25):**
  Dates and locations not yet announced

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