THE HISTORY OF POLYMERS : THE ORIGINS AND THE GROWTH OF A SCIENCE
Part I

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National Chemical Laboratory
Pune

September 10, 2011

International Year of CHEMISTRY 2011

- Celebrate the achievements of chemistry
- Improve public understanding of chemistry
- Champion the role of chemistry in addressing the critical challenges of our society
  - Food and nutrition
  - Clean water
  - Sustainable energy
  - Climate change
- Broader outreach and engagement
- Get younger people more interested
- in chemistry

Chemistry is the central, useful and creative science : Ronald Breslow

Madame Curie, Nobel Prize in Chemistry , 1911

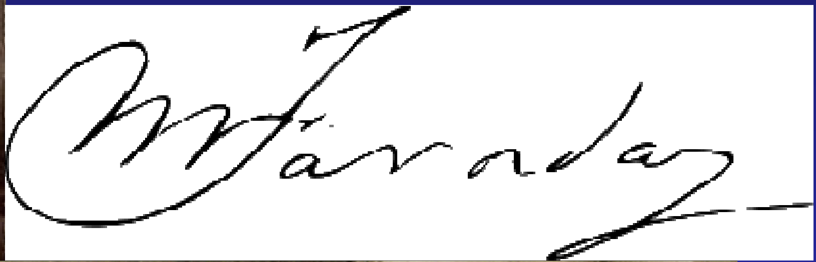
Earnest Rutherford, The Structure of the Atom. 1911

POLYMER SCIENCE : THREE PHASES OF EVOLUTION

- Post Industrial Revolution (1760-1900)
- World War I and II (1900-1950)
- The Era of Inexpensive Petroleum (1950- 2000)

- The beginnings of chemistry as a science (1800-1900)
- Atoms and molecules; understanding structure and the nature of the chemical bond (1900-1940)
- Understanding reactive intermediates in chemistry: The birth of physical organic chemistry (1940-60)

MICHAEL FARADAY (1791- 1867)



The most influential scientist in the history of science

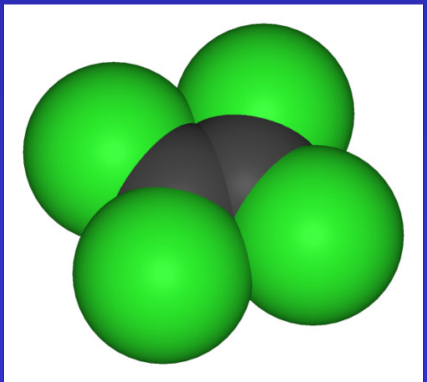
Faraday's Laboratory

- **One of the first scientists in the post – industrial revolution who established the methods of evidence based proof of hypothesis**
- **Contributed to both science and its applications; Studied pollution of river Thames, developed the first optical glass, studied the chemistry of flames and established that fine dust of coal can combust spontaneously**

MICHAEL FARADAY : A PIONEER IN CHEMISTRY

- **Discovery of Benzene**
- **Laws of electrolysis; concept of anode, cathode, electrode and ions**
- **First demonstration of decomposition of magnesium sulfate by applying electrical potential; Design of a voltaic pile consisting of seven half penny pieces, seven discs of zinc and filter paper soaked in salt water (1812)**
- **First synthesis of hexa-chloroethane and tetrachloro-ethylene (1820)**
- **Identification of isoprene as a constituent of natural rubber , now known as poly(isoprene) (1826)**

A Voltaic Pile



Tetrachloroethylene

Chemistry creates its own object. This creative power, similar to that of arts distinguishes it fundamentally from the other natural and historical sciences

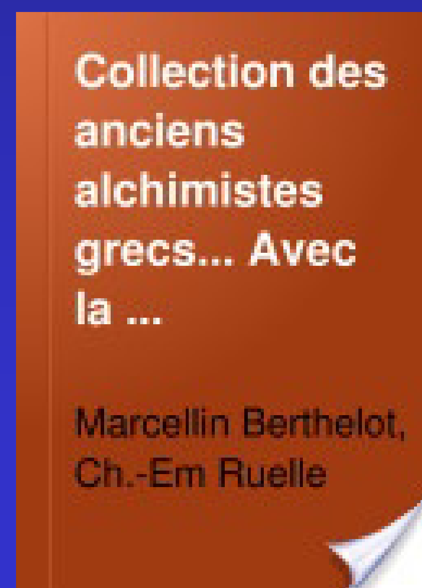
Marcellin Bertholet, 1860

(1827- 1907)

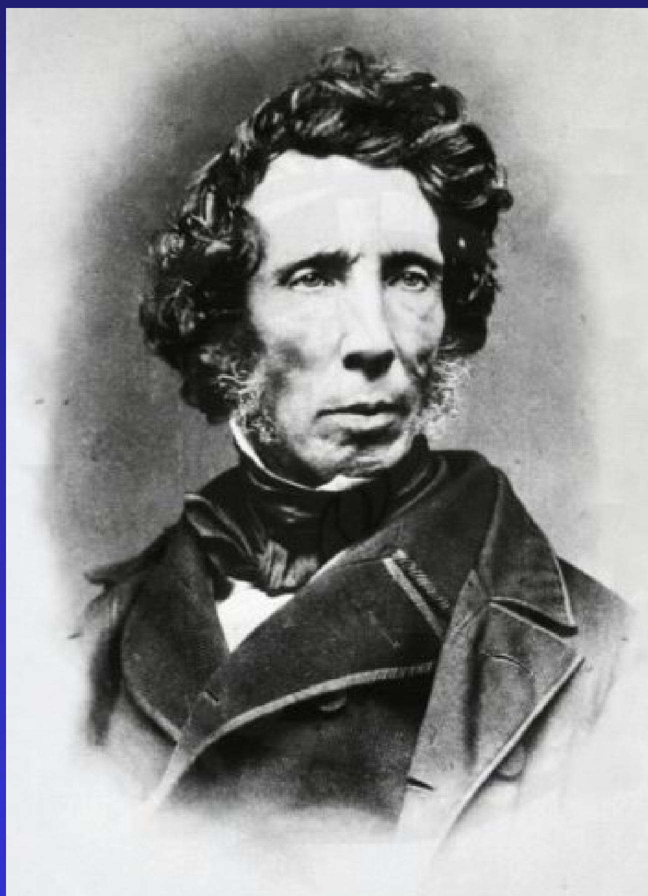
Bertholet gave the first general discussion on polymerism, that is, materials which have the same chemical composition, but differ only in their molecular weights

BERTHELOT AND THE BIRTH OF CHEMISTRY

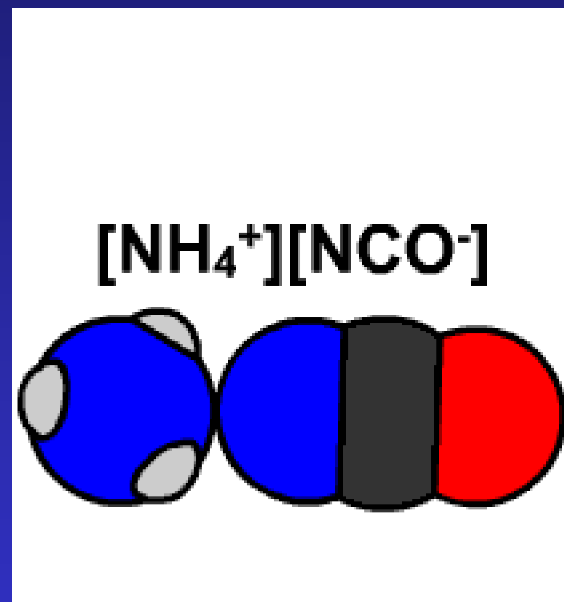
- **One of the greatest chemists of all time**
- **Was the bridge between Alchemists interpretation of matter and the modern science; Extensively studied the practice of Alchemie and wrote a book “Les Origines de l`Alchemie”, Paris, 1885**
- **Disproved the theory of Vitalism, the belief that organic compounds are infused with a unique force, not amenable to human interventions**
- **The beginning of the philosophy of reductionism in science**
- **The belief became prevalent that all problems can be solved by breaking them into parts**

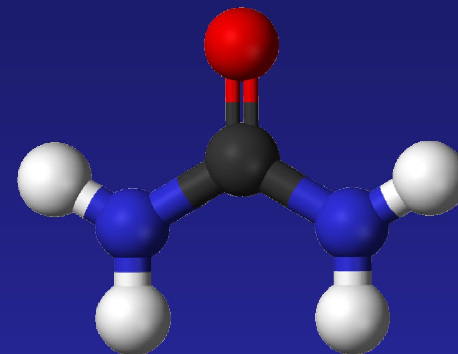


CHEMICAL REVOLUTION : EARLY BEGINNINGS



Friedrich Wohler (1800 – 1882)





" I must tell you that I can make urea without the use of a kidney of either man or dog" ***: Wohler in a letter to his teacher Berzelius***

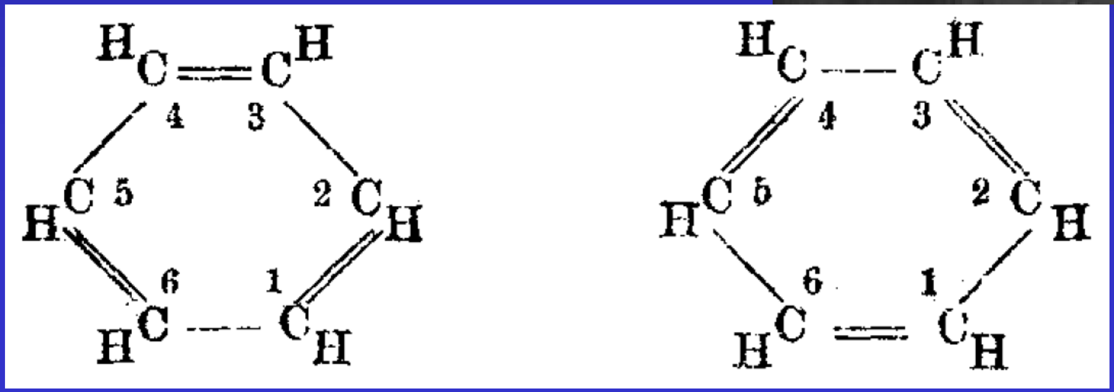
Annalen der Physik und Chemie, 88(2), 253-256 (1828)

CHEMICAL REVOLUTION : UNDERSTANDING CHEMICAL STRUCTURES

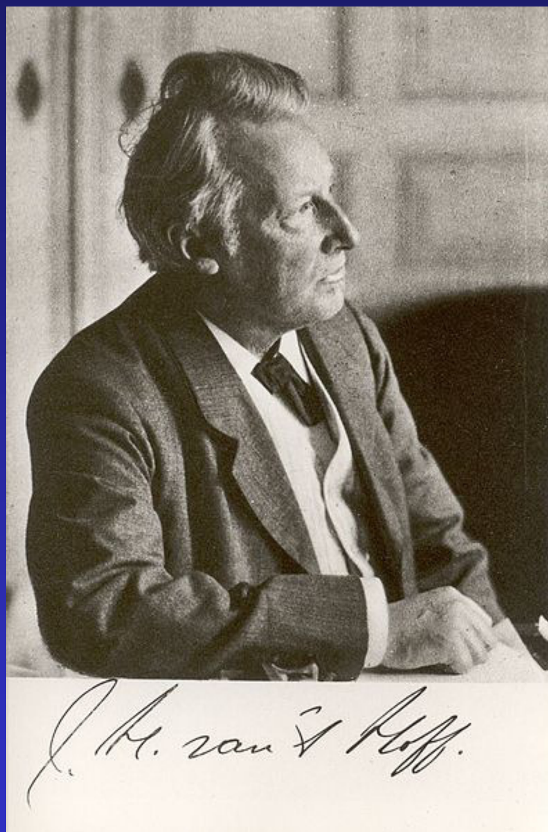
- The Theory of Chemical Structure (1857-58)

- Structure of Benzene published in *Bulletin de la Society Chimique de Paris, 3(2), 98-110 (1865)*

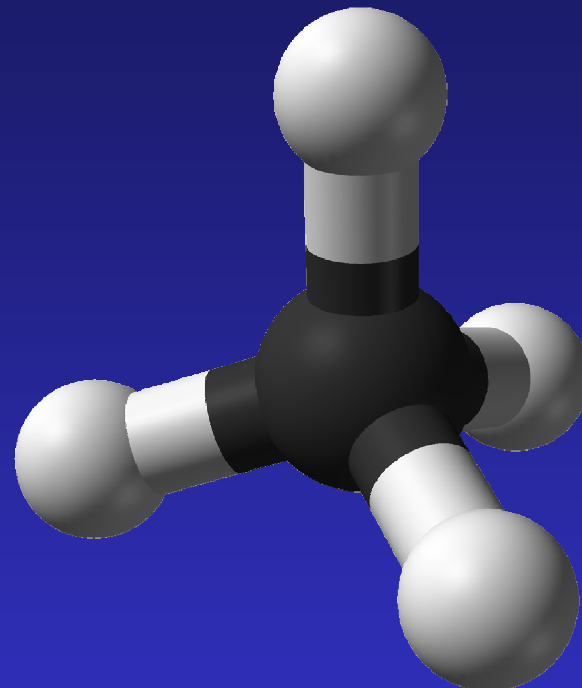
Auguste Kekule (1829 -1896)



CHEMICAL REVOLUTION : UNDERSTANDING CHEMICAL STRUCTURES



Jacobus van't Hoff
(1852-1911)



The tetrahedral nature of carbon
(La Chimie dans l'espace, 1874)

First Nobel Prize in 1901

THREE EARLY EXPERIMENTS

1805, John Gough	**Natural rubber heats up when stretched; a phenomena which took a century thereafter for formulating an understanding**
1826, Michael Faraday	Determined the elemental composition of natural rubber; in his note book he also recorded in passing a reaction of rubber with sulfur
1839, E.Simon	First isolation of styrene from a natural resource and observed that upon distillation styrene left a residue

BERTHOLET AND THE POLYMER HYPOTHESIS

- Bertholet came to a remarkable understanding of the conversion of vinyl compounds into polymeric chain molecules. He reasoned that upon addition of an olefin to a chain with a terminal double bond, the unsaturation would be retained, so that there was no reason why long chains should not be produced. Bertholet isolated the dimer, trimer and tetramer of pentene.
- In 1853, Bertholet reported the thermal and catalytic polymerization of pinene ; 1869 he published his results on polymerization of ethylene, propylene, pentene and pinene.
- His prescience is all the more remarkable, because the only techniques available to him were, density and boiling point measurement and softening temperature of solids
- He presented his results in a long lecture titled " la polymerie" presented at the Chemical Society of Paris in 1863
- We should admire his insight, given that chemistry was just then beginning to be understood in terms of structure and bonding.
- His imagination ran wild. He proposed that his principles also applies to atoms and suggested that sulfur can be viewed as a dimer of oxygen and tellurium as a tetramer of oxygen !

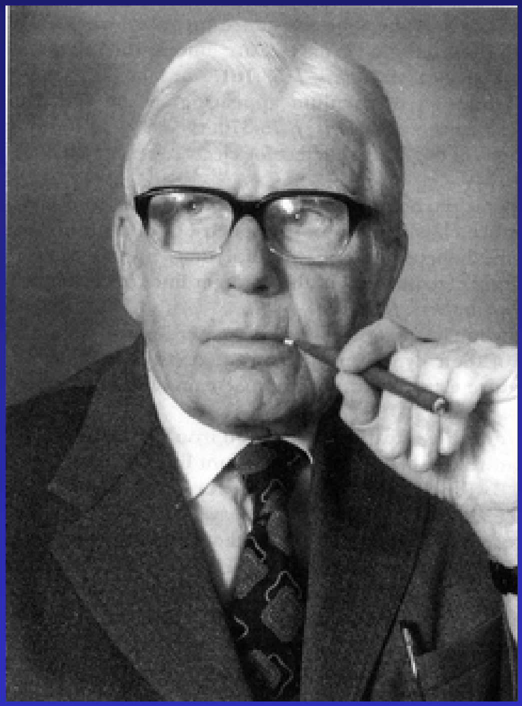
WHAT IS THE ORIGIN OF THE TERM POLYMER ?

- Faraday in 1826 was puzzled by the fact that ethylene and butene differed in their gas density , but had the same elemental composition
- Berzelius was astounded by Faraday`s observation and suggested that butene be referred to as a “polymer” of ethylene (1827, 1832). All through the nineteenth century, there are references to styrene being a polymer of acetylene and lactic acid as a polymer of formaldehyde
- Staudinger adopted this definition of Berzelius. For Staudinger, polystyrene was a polymer of styrene. However, he objected to the use of this term for products of polycondensation
- It was Carothers in 1929 who gave a general definition of the term. He defined them as substances” whose structures may be represented by R-R-R- where -R- are bivalent radicals which in general are not capable of independent existence” *(J.Am.Chem.Soc., 51, 2548 , 1929)*

MISCONCEPTIONS AND ARGUMENTS AGAINST THE EXISTENCE OF POLYMERS

- **The major opposition to the concept of polymers came from the emergence of the concept of colloids**
- **Much of the behaviour of polymers were identical to those observed in colloids, using the concepts of Thomas Graham . Micellar self assembly of small molecules in the absence of covalent bond accounted for most of the properties of polymers**
- **Harries, between 1902 to 1907 proposed that natural rubber was composed of aggregates of dimethylcyclooctadiene, held together by non covalent interactions (π – π) between the two double bonds**
- **Leading scientists of the day (example , Brill) argued that since a large molecule cannot be fitted into the crystallographic unit cell and, therefore, cannot exist. It was believed , based on some early evidence, that only compounds with molecular weight varying between 500-600 daltons can fit into the dimensions of a unit cell**

THE TALE OF TWO HERMAN(N)'S : THE POLYMER PIONEERS



X Ray Crystallography of Macromolecules to show that a molecule could be larger than its unit cell (1926-28)

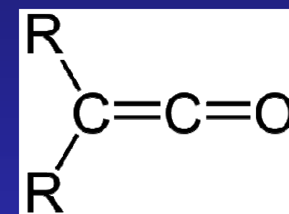
Herman Mark (1895-1992)

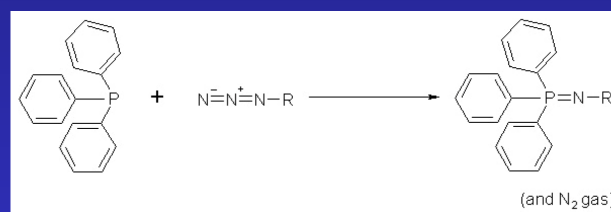
Concept of macromolecules as large molecules linked together by covalent bonds (1920)

Hermann Staudinger (1881-1965)
Nobel Laureate 1953

STAUDINGER : EARLY YEARS

- Volhard initiated him in chemistry; PhD in 1903 with Prof Vorlander in Halle
- Discovered ketenes as a new class of organic compounds in 1905 at Strasbourg during his habilitation with Thiele
- Began his scientific career at Karlsruhe in organic chemistry; Discovered the Staudinger Reaction





- Appointed to the faculty of ETH, Zurich in 1912 as successor to Richard Willstatter
- Between 1910 -20, extensively studied chemical constituent of natural products (chrysanthemum flowers, pepper, coffee) and prepared chrysanthemic acids, pyrethrins, synthetic piperidine and furfurylthiol as a coffee flavour). One of his students from these days Ruzicka won the Nobel Prize in chemistry in 1939

STAUDINGER : EARLY YEARS

- During this period, Staudinger was a typical practitioner of mainstream organic chemistry, which was already a highly sophisticated and respected science, led by chemists such as Adolf von Baeyer, Emil Fischer, and Richard Willstatter. By 1914, organic chemists had prepared more than 100,000 synthetic compounds used for various applications, including dyes and pharmaceuticals. Although not yet 40, Staudinger was considered a leading organic chemist.

- During the 1920s, Staudinger decided to leave the safe and prestigious haven of classical organic chemistry to embark on the stormy high seas of polymer science. Staudinger's pioneering spirit drove him to break away from the typical thinking of traditional organic chemists and to advance new and revolutionary ideas.

- In 1926, he was appointed to a chair at Albert Ludwigs University in Freiburg, where he dedicated all his efforts to establishing and expanding the frontiers of polymer science. His research topics included natural rubber, cellulose, and synthetic polymers such as polyoxymethylene, polystyrene, and polyethylene oxide, which Staudinger considered to be model systems for the much more complex biopolymers. As well as making synthetic polymers, Staudinger tried to determine the molecular weights of polymers by using end-group analysis, by measuring the viscosity of polymer solutions, and by using electron microscopy analysis.

STAUDINGER AND THE ORIGIN OF MACROMOLECULAR CHEMISTRY

- He propounded the revolutionary concept, that macromolecules can be formed by linking of a large number of small molecules by means of covalent bonds
- Created the " molecular blueprint " that led to the explosive growth of man made organic materials
- Through sheer audacity of intuition and imagination, he proposed that polymers were composed of large number of base units linked together by covalent bonds *(Ber. Dtsch. Chem. Ges., 53, 1073 (1920).* At that time he had no experimental evidence for his hypothesis.
- Wieland told him " Dear colleague, abandon your idea of large molecules; organic molecules with molecular weights exceeding 5000 do not exist. Purify your products and they will turn out to be low molecular weight materials "
- His ideas met with much resistance and criticism from eminent chemists of the period, notable amongst them, Emil Fischer. People called the chemistry being pursued by Staudinger as "Grease Chemistry". After on of the lectures given by Staudinger in Zurich in 1925, one of the speakers termed Staudinger`s championship of long chain molecules as akin to some traveler in Africa reporting that he had seen a zebra 400 meters long !
- Staudinger persevered in spite of being ostracized by the scientific community. From 1926, he abandoned research in organic chemistry and shifted exclusively to macromolecules.

STAUDINGER AND THE ORIGIN OF MACROMOLECULAR CHEMISTRY

- First experimental evidence for existence of long chains came in 1922.Hydrogenation of natural rubber was not accompanied by the formation of volatile cyclic hydrocarbons *(Staudinger and Fritschi,Helv.Chim.Acta,5, 785 (1922)*

- First time the term “macromolecules” wass used in chemistry

- The first definition of macromolecules: “For such colloid particles, in which the molecule is identical with the primary particle, and in which the individual atoms of this colloid molecule are linked together by covalent bonds, we propose for better differentiation the name macromolecule *(Staudinger,Ber.Dtsch.Chem.Ges.,57,1203(1924)*

- Much of the rigorous proof for the existence of macromolecules will come from physical measurements (viscosity measurements, molecular weight measurements by ultracentrifuge, osmometry and light scattering as well as X Ray diffraction)
- But being quintessentially an organic chemist, Staudinger was skeptical of physical measurements. Staudinger believed polymers to possess “rigid rod like structure”, a belief he will not abandon for several years. When evidence began building up that polymers have flexible chains, Staudinger ignored them.

STAUDINGER: THE POLYMER PIONEER

(R.Mulhaupt, Angew. Chem. Int. Ed. Engl., 43, 1054, 2004)

- **Hermann Staudinger always maintained a close relationship with industry to obtain funds for his research and to act as a technical consultant for firms interested in plastics and rubber. For many years, the "Forderverein" (association of supporters) of the Institute for Macromolecular Chemistry linked the research managers of the various companies who sponsored polymer research in Freiburg.**

- **Staudinger's research was published in more than 800 publications amounting to more than 10,000 printed pages. He summarized his research in his autobiography, *Arbeitserinnerungen (From Organic Chemistry to Macromolecules)* published in 1970. His collected works, entitled *Das Wissenschaftliche Werk von Hermann Staudinger (The Scientific Contributions of Hermann Staudinger)*, were edited by Magda Staudinger and published between 1969 and 1976.**

- **For many years, Staudinger's textbook, entitled *Die Hochmolekularen Organischen Verbindungen Kautschuk und Cellulose (The High Molecular Weight Organic Compounds Rubber and Cellulose)*, published in 1932 by Springer in Berlin, was the "bible" of many academic and industrial scientists. In 1947, Staudinger inaugurated the new journal *Makromolekulare Chemie* with Wepf & Company, publishers in Basel. For more than 60 years, this journal has provided an excellent forum for scientific exchanges and has promoted the expansion of polymer science.**

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HERMAN MARK AND THE EVOLUTION OF THE STRUCTURE OF POLYMERS

- Mark, along with Staudinger and Carothers can be credited as a cofounder of Polymer Science

- Mark was trained as an organic chemist. His PhD thesis was on the chemistry of free radicals under the supervision of Schlenk

- With Polanyi , Mark began to explore the technique of Crystallography (X Ray and electron diffraction)for the study of organic molecules at Kaiser Wilhelm Institute

- One of the materials chosen was cellulose fiber. They found that cellulose fiber upon stretching leads to increase in modulus

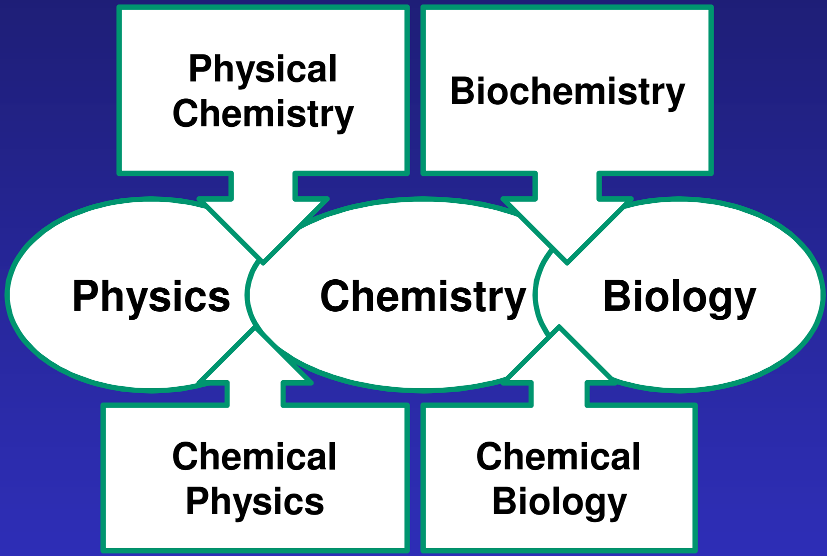
Mark presents his results in a meeting of the Society of German Natural Scientists at Dusseldorf in 1926 ; He says that important information can be obtained from unit cells and space groups, even if detailed molecular structures are not known; He proposes that in polymers "lattice forces are comparable to intramolecular forces and the entire crystallite behaves like a large molecule"

HERMAN MARK AND THE POLYMER INDUSTRY

- In 1926, Mark becomes the Director of research at IG Fabenindustrie in Ludwigshafen, a large producer of rayon and cellulose acetate. Here he develops the first commercial process for producing styrene
- Mark and Meyer solve the crystal structure of Cellulose in 1928, the first crystal structure of a polymer which reconciles the X-Ray pattern with the chemical composition.
- Using X Ray, Mark establishes that natural rubber is a polymer of isoprene, namely, 1,4-polyisoprene and that isoprenes are in a *cis* configuration in the polymer chain
- It is interesting that although Mark provided the most unequivocal support to the macromolecular hypothesis of Staudinger, he and Staudinger were not in good terms. Staudinger felt that macromolecular chemistry was his field and he looked upon physicists and physical chemists as interlopers, who stole his ideas.
- Mark left IG Farben in 1932 and returned to the University of Vienna . It was in Vienna that he along with Eugene Guth proposed the statistical theory of rubber elasticity and developed the Mark Houwink relationship, relating dilute solution viscosity of a polymer solution to its molecular weight
- One of his famous students in Vienna was Max Perutz who went on to found the Medical Research Council at Cambridge which became a centre of excellence in protein crystallography

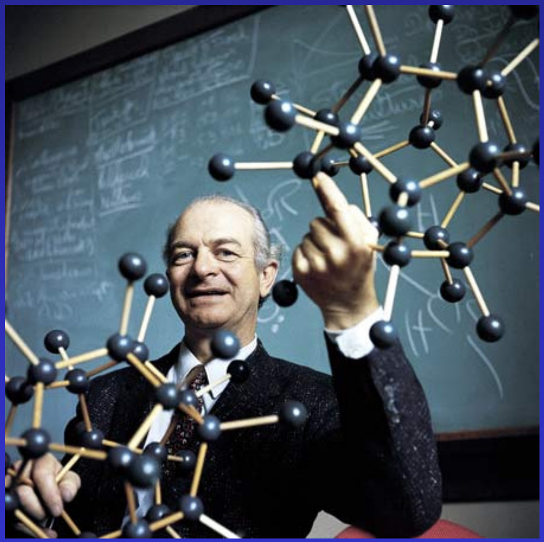
LINUS PAULING AND THE NATURE OF THE CHEMICAL BOND

- **Established chemistry as an overarching science that bridges physics on one side and biology on the other**



Created a new discipline called Quantum Chemistry

Retrieved chemistry which was slipping into the hands of the early twentieth century physicists

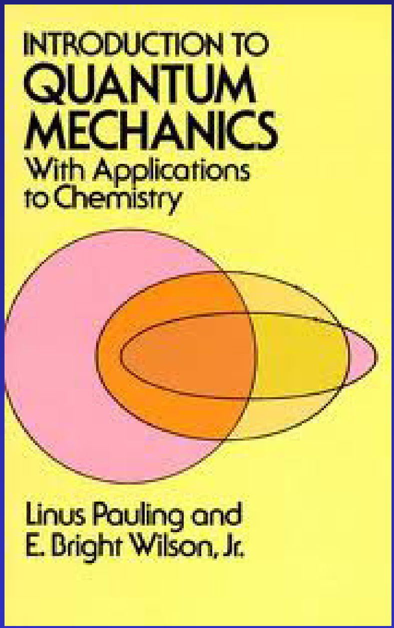


1901- 1994
Nobel Laureate, 1954

MARK AND HIS INFLUENCE ON PAULING

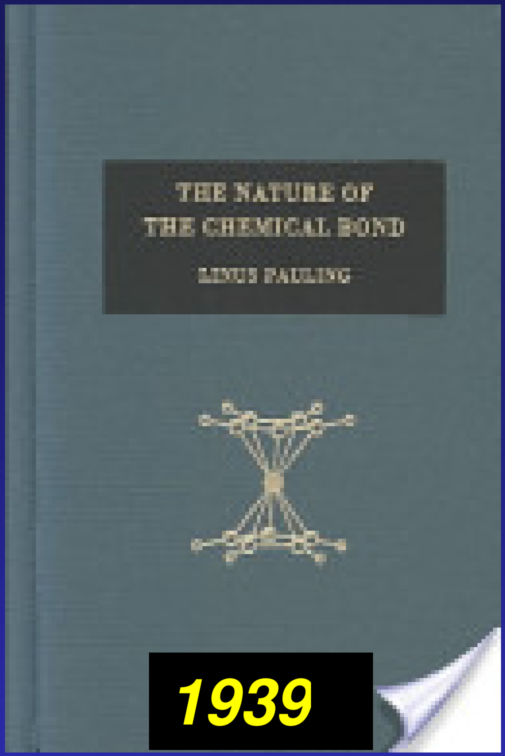
Reproduced from" Linus Pauling in His Own Words",1995

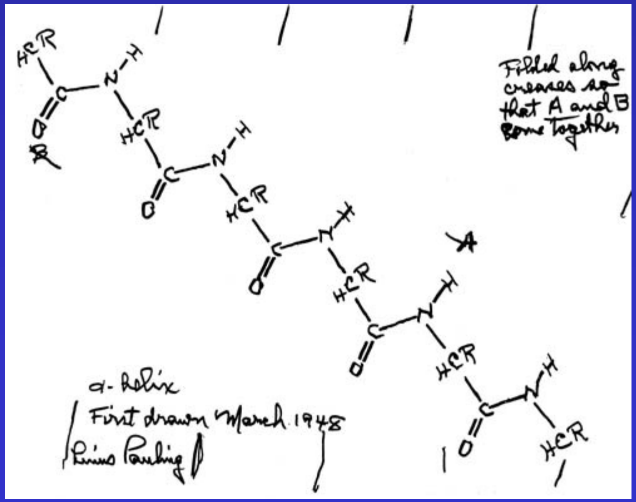
" By 1932 I felt reasonably well satisfied with my understanding of inorganic compounds , including such complicated ones like silicate minerals. The possibility of getting a better understanding also of organic compounds then presented itself. Very little information on bond lengths and bond angles were available for organic compounds. In 1930, when I visited Germany, I learned about a new method of determining the structure of molecules that had been invented by Dr Herman Mark. This was the method of electron diffraction "



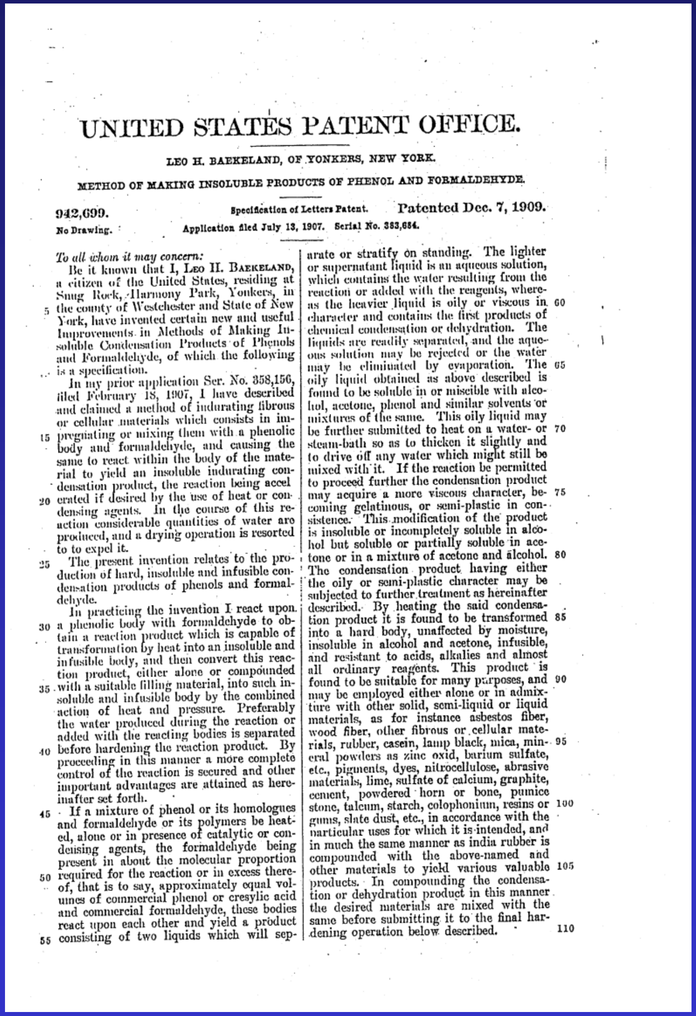
- *“ Herman mark had been good enough to say that he was not planning to continue work along the lines and that he would be glad to see it done in Caltech. He also gave me the drawings showing how the instrument can be constructed. My student Brockway began in 1930 to construct the first electron diffraction apparatus for studying organic molecules that had been built anywhere but in Mark`s laboratory*

  Within a few years we had amassed a large amount of information, leading to new ideas in structural chemistry, such as the theory of resonance, orbital hybridization and valence bonds. These results were in accordance with the results of quantum mechanical calculations and it became clear by 1935 that a far more extensive, precise and detailed understanding of organic compounds have been developed than that had been available to chemists in earlier decades”





THE DAWN OF THE CHEMICAL INDUSTRY: THE MANUFACTURE OF BAKELITE



UNITED STATES PATENT OFFICE.

LEO H. BAEKELAND, OF YONKERS, NEW YORK.

METHOD OF MAKING INSOLUBLE PRODUCTS OF PHENOL AND FORMALDEHYDE.

942,699. Specification of Letters Patent. Patented Dec. 7, 1909.

No Drawing. Application filed July 13, 1907. Serial No. 383,684.

To all whom it may concern:

Be it known that I, LEO H. BAEKELAND, a citizen of the United States, residing at Snug Rock, Harmony Park, Yonkers, in the county of Westchester and State of New York, have invented certain new and useful Improvements in Methods of Making Insoluble Condensation Products of Phenols and Formaldehyde, of which the following is a specification.

In my prior application Ser. No. 358,156, filed February 18, 1907, I have described and claimed a method of indurating fibrous or cellular materials which consists in impregnating or mixing them with a phenolic body and formaldehyde, and causing the same to react within the body of the material to yield an insoluble indurating condensation product, the reaction being accelerated if desired by the use of heat or condensing agents. In the course of this reaction considerable quantities of water are produced, and a drying operation is resorted to to expel it.

The present invention relates to the production of hard, insoluble and infusible condensation products of phenols and formaldehyde.

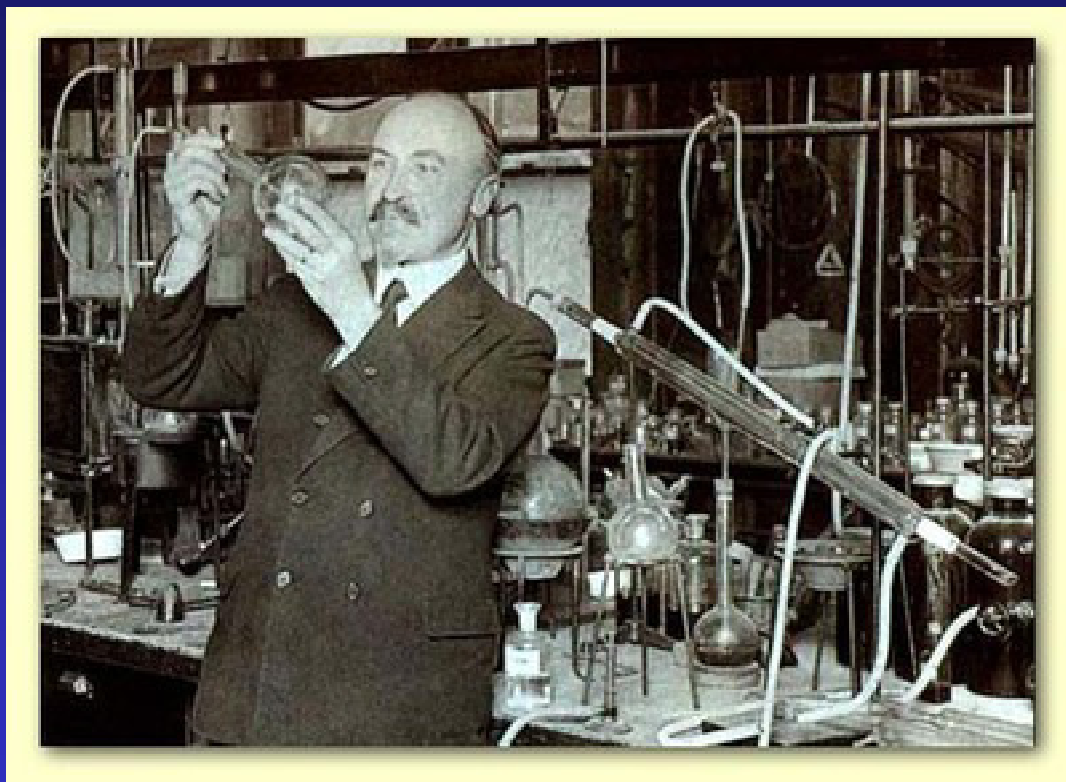
In practicing the invention I react upon a phenolic body with formaldehyde to obtain a reaction product which is capable of transformation by heat into an insoluble and infusible body, and then convert this reaction product, either alone or compounded with a suitable filling material, into such insoluble and infusible body by the combined action of heat and pressure. Preferably the water produced during the reaction or added with the reacting bodies is separated before hardening the reaction product. By proceeding in this manner a more complete control of the reaction is secured and other important advantages are attained as hereinafter set forth.

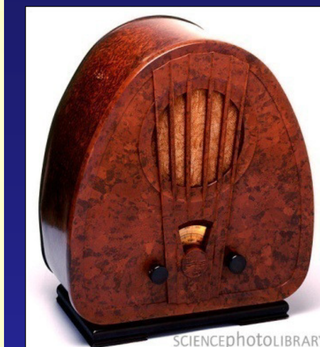
If a mixture of phenol or its homologues and formaldehyde or its polymers be heated, alone or in presence of catalytic or condensing agents, the formaldehyde being present in about the molecular proportion required for the reaction or in excess thereof, that is to say, approximately equal volumes of commercial phenol or cresylic acid and commercial formaldehyde, these bodies react upon each other and yield a product consisting of two liquids which will separate or stratify on standing. The lighter or supernatant liquid is an aqueous solution, which contains the water resulting from the reaction or added with the reagents, whereas the heavier liquid is oily or viscous in character and contains the first products of chemical condensation or dehydration. The liquids are readily separated, and the aqueous solution may be rejected or the water may be eliminated by evaporation. The oily liquid obtained as above described is found to be soluble in or miscible with alcohol, acetone, phenol and similar solvents or mixtures of the same. This oily liquid may be further submitted to heat on a water- or steam-bath so as to thicken it slightly and to drive off any water which might still be mixed with it. If the reaction be permitted to proceed further the condensation product may acquire a more viscous character, becoming gelatinous, or semi-plastic in consistence. This modification of the product is insoluble or incompletely soluble in alcohol but soluble or partially soluble in acetone or in a mixture of acetone and alcohol. The condensation product having either the oily or semi-plastic character may be subjected to further treatment as hereinafter described. By heating the said condensation product it is found to be transformed into a hard body, unaffected by moisture, insoluble in alcohol and acetone, infusible, and resistant to acids, alkalies and almost all ordinary reagents. This product is found to be suitable for many purposes, and may be employed either alone or in admixture with other solid, semi-liquid or liquid materials, as for instance asbestos fiber, wood fiber, other fibrous or cellular materials, rubber, casein, lamp black, mica, mineral powders as zinc oxid, barium sulfate, etc., pigments, dyes, nitrocellulose, abrasive materials, lime, sulfate of calcium, graphite, cement, powdered horn or bone, pumice stone, talcum, starch, colophonium, resins or gums, slate dust, etc., in accordance with the particular uses for which it is intended, and in much the same manner as india rubber is compounded with the above-named and other materials to yield various valuable products. In compounding the condensation or dehydration product in this manner the desired materials are mixed with the same before submitting it to the final hardening operation below described.

US Patent 942, 699, December 7, 1909

- Baekland set out to discover a substitute for Shellac, then wholly supplied by India to the world
- In the process he made the first man made material, heralding the age of plastics, a discovery considered as revolutionary
- Heat resistant and insulating, demand from the burgeoning electrical goods industry
- Baekland named his new material Novolak
- He founded a company called General Bakelite Corporation in 1910 to manufacture the product

THE DAWN OF THE CHEMICAL INDUSTRY: THE MANUFACTURE OF BAKELITE

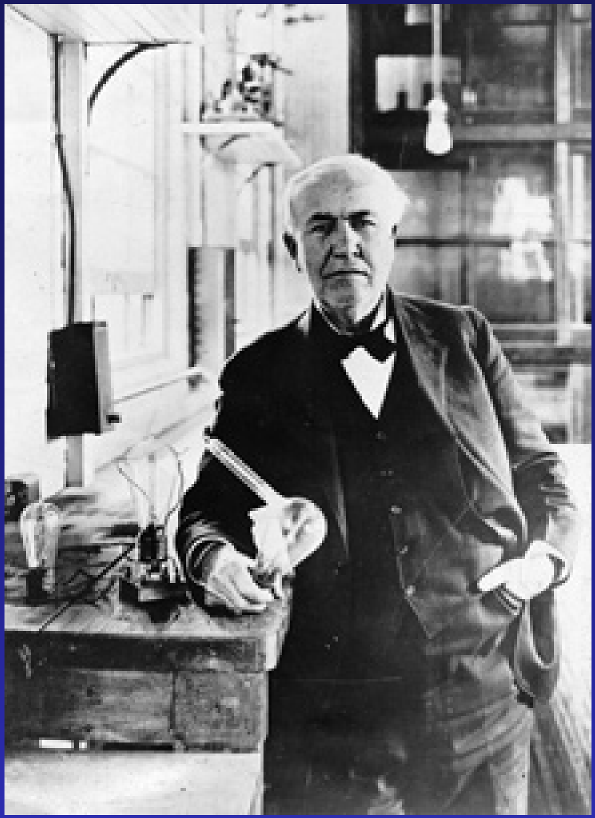




Leo Baekland (1863-1944)

When asked why he chose to work in the field of synthetic resins, he replied, "to make money"

EDISON AND MENLO PARK : THE BIRTH OF INVENTION FACTORY



Thomas Alva Edison (1847-1931)

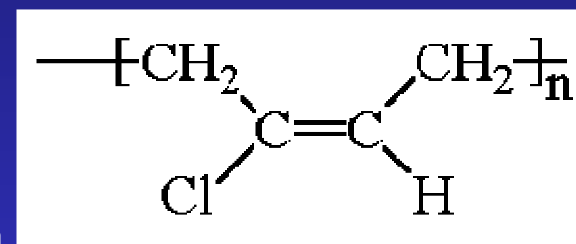
Menlo Park, NJ Laboratory

- **Most prolific inventor in the history**
- **Edison holds the record for the largest number of patents granted to an individual inventor, 1093**
- **Inventor of phonograph, incandescent bulb, motion picture camera, alkaline battery and many others**
- **First to organize and manage research , a forerunner to the later day corporate research laboratories of companies**
- **Assembled a cross functional global team of coworkers, from Germany(glass blowing), Switzerland (watch making),mathematicians, chemists , carpenters and machinists**
- **In 1900 Menlo Park employed over hundred people who were inventing for a salary and living**

WALLACE CAROTHERS AND THE BIRTH OF RATIONAL POLYMER SYNTHESIS

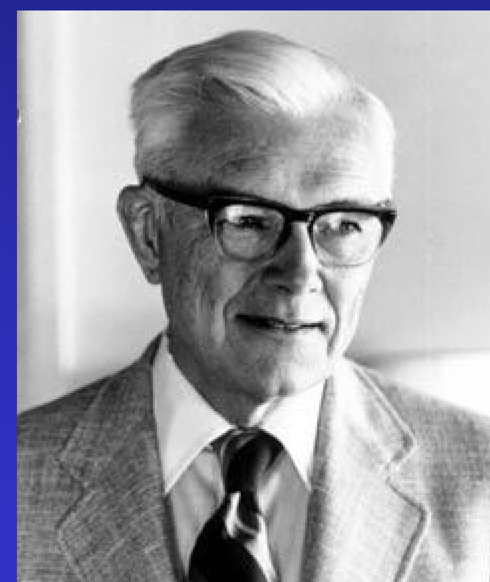
1896-1937

- Trained as an organic chemist with Roger Adams, PhD, 1924
- Hired as a faculty at Harvard
- DuPont lured him to Wilmington Delaware to lead a fundamental research programme in organic chemistry and polymers
- by 1931, he had synthesized chloroprene and polymerized to a new synthetic rubber, called by DuPont as Neoprene
- Publishes his seminal paers in JACS in 1929 where in he establishes the equivalence of organic and polymer forming reactions, namely esterification and polyesterification



Poly(chloroprene)

WALLACE CAROTHERS AND THE BIRTH OF RATIONAL POLYMER SYNTHESIS



A young man joins Carothers at DuPont in 1934who will go on to make history

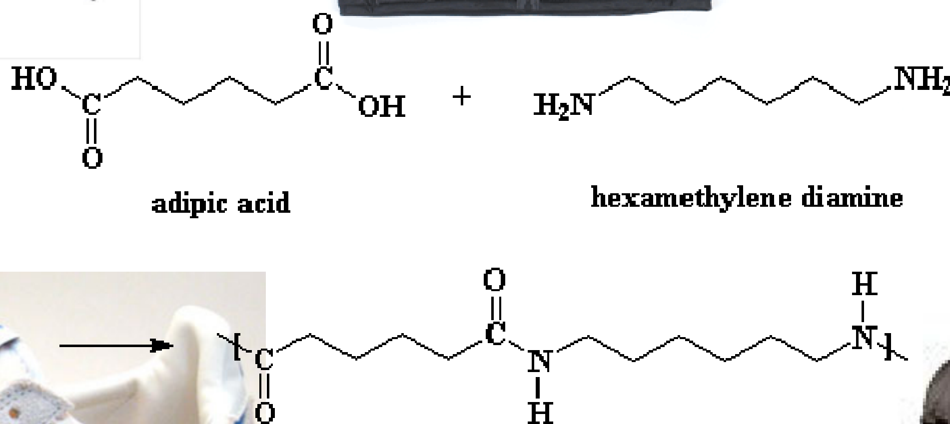
With Julian Hill, extends the reaction to adipic acid and hexamethylene diamine, a polyamide forming reaction , leading to the first synthesis of Nylon-66 in 1934. Nylon-66 goes into production in 1939

Develops a theoretical understanding of the polycondensation reaction relating the average degree of polymerization to fractional conversions (Carother`s Equation)

Carothers had been troubled by periods of mental depression since his youth. Despite his success with nylon, he felt that he had not accomplished much and had run out of ideas

His unhappiness was compounded by the death of his sister, Isobel, and on the evening of April 28, 1937 he checked into a Philadelphia hotel room and committed suicide by drinking a cocktail of lemon juice laced with potassium cyanide

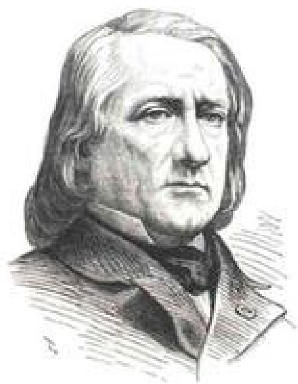
His daughter, Jane, was born seven months later on November 27, 1937.



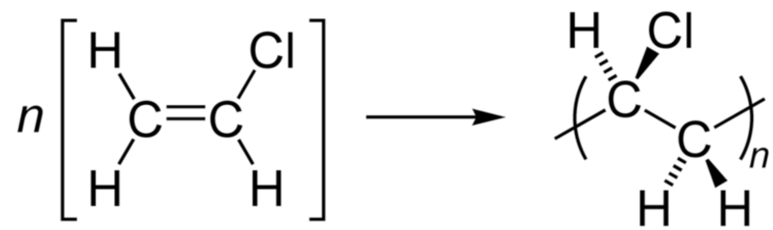
nylon 6,6

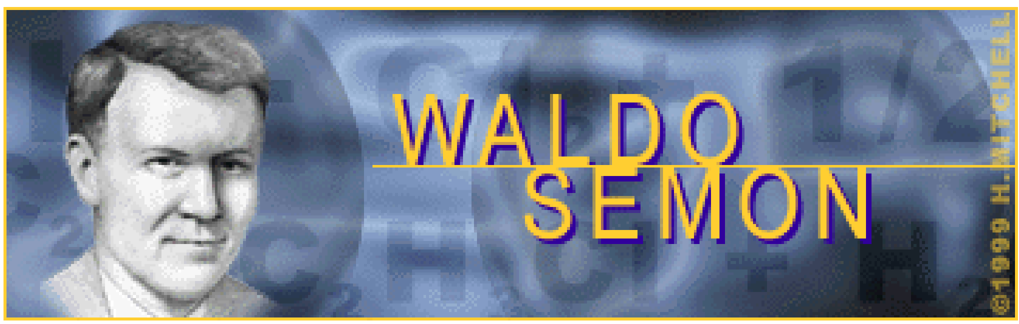
NYLONS ARE ALSO FOUND IN MANY ARTICLES OF EVERY DAY USE

PVC : ORIGINS AND HISTORY

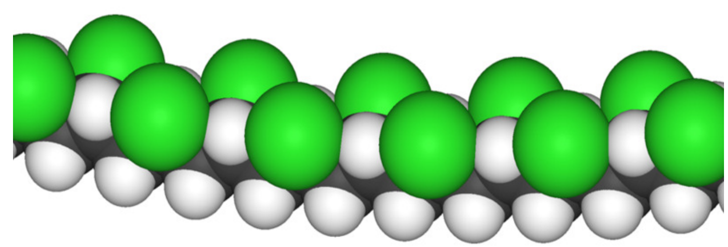


Henri Victor Regnault , 1835





On December 11, 1935, in Akron , Ohio Dr. Semon made flexible PVC by using a plasticizer, paving the way for its large scale applications



PVC : REPLACEMENT FOR A RENEWABLE RESOURCE

78 rpm Gramaphone records

Shellac, a natural resin secreted by the female lac bug on trees; Main constituent : Aleuritic acid; In the early part of twentieth century , India was the largest supplier of Shellac to the world

33 rpm long playing records (vinyls)

I am inclined to think that the development of polymerization is, perhaps, the biggest thing that chemistry has done, where it has the biggest effect on everyday life

Lord Alexander Todd (1907-1997)

Nobel Laureate, 1957

NCL

1950 - 2010

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THANK YOU