

Inorganic Chemistry

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Unit- 19 Metal Cluster

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- Introduction
- Boranes
- Nomenclature
- Classification
- Wade's Rules
- Styx Numbers
- Diborane
- Structure and Bonding
- Reactions
- Carboranes
- Synthesis and Reactions
- Carborane Ligands
- Metalloboranes
- Metallacarboranes
- Compounds with metal-metal multiple bonds
- Polyoxometalates (Isopoly and Heteropoly acids)
- Summary
- Terminal Questions
- Answers

* OBJECTIVES:

The chapter covers boranes, carboranes, metalloboranes, metallocarboranes along with various aspects of isopoly and heteropoly acids. This chapter aims at providing a good understanding of metal cluster compounds with respect to their synthesis, structure and properties. Typesboranes, carboranes their synthesis and various bonding aspects in these class of compounds will be focused.

***INTRODUCTION**

Clusters are a collections of atoms or molecules which are intermediate in size between a molecule and bulk solid. They may have varied nuclearities and stoichiometry. They can be considered as link between atoms, molecules and bulk material. The properties of clusters changes with size. Metal clusters have metal-metal bonds. This chapter first deals with boron compounds and then gradually leads to clusters such carboranes, metalloboranes, metallocarboranes and isopoly-heteropoly acids. These complex materials finds various applications in the synthesis of various organic derivatives and industry.

***BORANES**

Boron occurs naturally as borax Na₂B₄O₅(OH)₄.8H₂O and as kerniteNa₂B₄O₅(OH)₄.2H₂O. Some other ores of boron are Colemanite, Urexite and Kernite. Borate minerals contain trigonal BO₃ or tetrahedral BO₄ units. Boron has two stable isotopes, ⁵B₁₁ (80.4% abundance) and ⁵B₁₀ (19.6%), of which ⁵B₁₀ has a very high neutron absorption cross section. Boron is a non-metal belonging to group 13 (III A) whereas the other elements of this group show metallic properties. Thus the chemistry of boron is quite different from other elements of the group. Boron resembles carbon and silicon in its tendency to form covalent compounds using its sp^2 hybridized orbitals. No ionic compounds containing B³⁺ cations are formed because the energy of ionization is

> Boron Hydrides or Boranes: Boron forms several hydrides known as Boranes, composed solely of boron and hydrogen bonds and may be neutral or ionic. They are electron deficient species possess fewer valence electrons than are required for a localized bonding scheme. Their chemistry was first studied by Alfred Stock. Typical boranes are B_2H_6 , B_4H_{10} , B_9H_{15} , $B_{10}H_{14}$, and $B_{20}H_{16}$.

***NOMENCLATURE:**

The nomenclature of boranes is somewhat simple. As mentioned before boranes exist as neutral and ionic species.

Neutral Boranes: For neutral boranes prefix di, tri etc. are used before 'borane' to indicate the number of boron atoms. The number of hydrogen atoms are indicated by writing the Arabic numeral in the parentheses at the end. For example:

BH_3	borane (3)
B_2H_6	diborane (6)
B_3H_7	triborane (7)
B_4H_{10}	tetraborane (10)
B_5H_9	pentaborane (9)
B_5H_{11}	pentaborane (11)
B_6H_{10}	hexaborane (10)
$B_{10}H_{14}$	decaborane (14)
$B_{10}H_{16}$	decaborane (16)
$B_{20}H_{16}$	icosaborane (16)

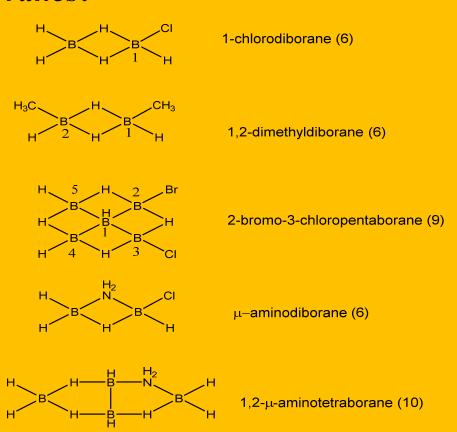
It is to be noted that these names are based on the molecular formula and not on their structures. Generally the numbers in the bracket are not written.

Anionic Boranes: For anions the name ends with 'ate' and the number of hydrogen and boron atoms are specified along with the charge. Example:

$Na[B_2H_7]$	sodium heptahydrodiborate(1 -)
$Na_2[B_2(C_6H_5)_6]$	sodium hexaphenyldiborate(2 -)
$Ca[B_3H_8]_2$	calcium octahydrotriborate(1 -)
$Na_2[B_{10}Cl_{10}]$	sodium decachlorodecaborate(2-)
$Na_{2}[B_{10}H_{10}]$	sodium decahydrodecaborate(2-)
$Na[B_{10}H_9NH_3]$	sodium amminenonahydrodecaborate(1-)
$B_{10}H_8(NH_3)_2$	diammineoctahydrodecaboron
$[B_{10}H_7(NH_3)_3]^+$	triammineheptahydrodecaboron(1+) ion
$[B_{10}H_8(COOH)_2]^{2-}$	dicarboxyoctahydrodecaborate(2-) ion
$Na_2B_4O_7$	sodium heptaoxotetraborate(2-) or sodium tetraborate

Certain prefixes such as 'closo, nido and arachno are used. This classification will also be discussed. Eg. $Na_2[B_{10}H_{10}]$ sodium decahydro-closo-decaborate(2-), $[B_{12}H_{11}Cl]^2$ -chloroundecahydro-closo-dodecaborate (2-)ion.

Substituted Boranes:



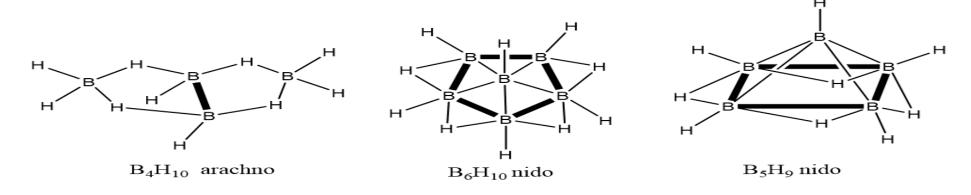
2.2 CLASSIFICATION OF BORANES

Higher boranes or borane clusters possess deltahedral structures which are somewhat complex in terms of their 3c-2e bonding. A deltahedron is a polyhedron that possesses only triangular faces, e.g., an octahedron. The boranes are mainly classified as:

- 1. *closo*-B_nH_{n+2} (*closo*-Greek for cage), *Closo* pertains to the most symmetrical form that is a closed polyhedra
- 2. nido-B_nH_{n+4} (nido-Greek for nest), nido has one of the vertex removed from a regular polyhedral; (n + 1)-cornered polyhedron.
- 3. arachno-B_nH_{n+6} (arachno-Greek for spider web).Arachno is a higher version of nido, with most highly connected boron vertex on the open face removed. Thus,the arachno form has two vertices missing; (n + 2)-cornered polyhedron.

- 4. Hypho- B_nH_{n+8} (Greek: net like): They have the most open clusters in which the B atoms occupy n corners on an (n + 3)-cornered polyhedron.
- 5. *Conjuncto-* B_nH_{n+10} (Latin: join together): They have structures formed by linking together of two or more of the above mentioned type of clusters.

They can also be written as having the general formula BnHn²⁻, BnHn⁴⁻, BnHn⁶⁻, BnHn⁸⁻, BnHn¹⁰⁻ for closo, nido, arachno, hypho and conjuncto respectively. That is formally subtracting the number of H⁺ ions from the formula to make the number of boron and hydrogen atoms equal.



***WADE'S RULES:**

A set of rules were laid down by Kenneth Wade to predict the shape of boron clusters. These rules correlate skeletal structures of boranes, carboranes, heteroboranes and their anions (closo, nido, arachno, hypho) with the number of skeletalelectron pairs present in them.

According to the rule suppose a cluster has say 'n' skeletal atoms (that is the vertices) then it will adopt *closo* structure if it contains n+1 skeletal bonding electron pairs. Similarly, *nido* if n+2, *arachno* if n+3 and *hypho* if n+4 skeletal bonding electron pairs respectively and so on.

To determine this one needs to know the number of skeletal electron pairs in a cluster which can be determined by following the electron count for various donating units as given below.

Each BH unit gives 2 skeletal bonding electrons

B as such gives three skeletal electrons

Each C-H unit of a carboranecontributes 3 skeletal bonding electrons

Each additional H furnishes 1 skeletal bonding electron

Ionic charges must be included in the electron count

For borane clusters with other hetero-elements,

C, Si, Ge and Sn of a cluster is replaced with a BH unit;

N, P and As with a BH2 unit and

S and Se with a BH₃ unit for counting purpose

$$B_5H_9$$
 $B_2H_7^ C_2B_{10}H_{12}$
 $C_2B_9H_{11}^2$

 5 BH = 5x2 2 BH = 2x2 = 2 CH= 3x2 = 6e
 2 CH= 2x3= 6e

 =10e
 4e

 4 H = 4x1 = 4e
 5 H = 5x1 = 5e
 10 BH= 2x10 = 9
 BH= 9x2

 20e
 =18e

 14 e = 7pair
 1(-) = 1e
 26e = 13 pair
 2(-) = 2e

 n = 5; n+2 = 10 e = 5 pair
 n= 12; n+1 = 26 e = 13 pair

 nido
 closo

 n = 2; n+3=
 n= 11; n+2 = nido

 (MeSi)₂B₁₀H₁₀
 CPB₁₀H₁₁

 2 MeSi = 2x3 = 6e
 Consider C=BH; PB₁₁H₁₂

 10 BH = 10x2 = 20e
 Consider P=BH₂; B₁₂H₁₄

 26 e = 13 pair
 12BH = 12x2= 24e

 n = 12; n+1 = closo
 2H = 2e

 26e = 13 pair; n= 12; n+1= closo

***STYX NUMBERS:**

The Styx rules were given by Lipscomb to determine what type of bonds are possible for a given formula of boron hydride. It is assumed that one

B–H bond is present for each boron atom.

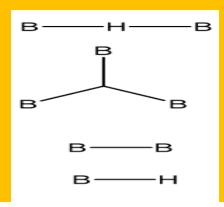
i. The centre 3c-2e, B-H-B bond is labelled as 'S'

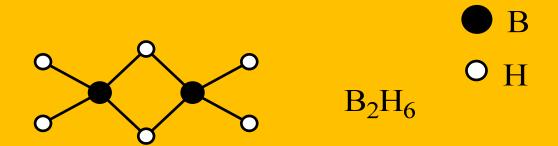
ii. Closed or open 3c-2e B-B-B bond is labelled as 'T'

iii. The 2c-2e B-B bond is labelled as 'Y'

iv The B–H terminal bond (2c-2e) labelled as 'X' or simply we can say number of BH₂units

The B–B–B bond may be open or closed but it is the closed bonds that are always considered.





Total valence electrons two B and six H = 12 STYX number is 2002

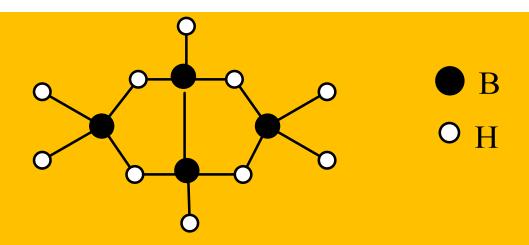
$$B$$
— H — B (Bridging) Electrons used = $2x2 = 4$; $S = 2$

$$T = 0$$

$$\mathsf{B} \longrightarrow \mathsf{B} \qquad \mathsf{Y} = 0$$

Four terminal BH bonds, electrons used =
$$4x2=8$$

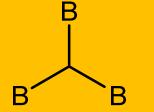
Extra BH $X=2$



 B_4H_{10}

Total valence electrons; four B and ten H, total = 12 + 10 = 22 STYX number is 4012

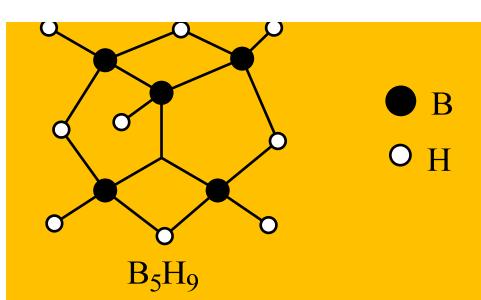
 $B \longrightarrow H \longrightarrow B$ (Bridging) Electrons used = 4x2=8; S=4



$$T = 0$$

One B-B bond; electrons used = 2; Y = 1

Six terminal BH bonds, electrons used = 6x2 = 12; X = 2



Total valence electrons; five B and nine H, total = 15 + 9 = 24 STYX number is 4120

$$B \longrightarrow H \longrightarrow B$$
 (Bridging) Electrons used = $4x2 = 8$; $S = 4$

 $\mathbb{R}^{\mathbb{R}}$

One closed B-B-B bond; 3c-2e; electrons used = 2; T = 1

B—B Two B-B bond; 2c-2e; electrons used = 4; Y = 2

B—H Five B-H bonds, electrons used = 5x2 = 10; X = 0

SYNTHESIS of DIBORANE(6)

Diborane(6), B₂H₆ is a gas and like most boranes it is air-sensitive and catches fire. It is spontaneously hydrolyzed by water into hydrogen and boric acid.

One of the simplest method is hydride abstraction from BH_4^- .

At industrial scale diborane is prepared by reduction of BF₃ with sodium hydride

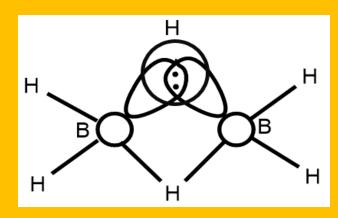
$$2 BF_3 + 6 NaH \longrightarrow B_2H_6 + 6 NaF$$

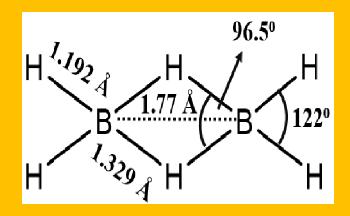
Diborane acts as a very versatile reagent in synthesis of organoboranes. It also acts as a reducing agent for certain functionnal groups such as nitriles and aldehydes.

2.6 STRUCTURE AND BONDING

In the earliest stages of decoding the structure of diboranemany errors and contradictions led to the establishment of the structure known today. The imminent deficiency of electrons in their structural formula indicates that there cannot be conventional 2c-2e (two center-two electron, two electrons are shared between two atoms) bonds. Thus in the efforts to rationalize the structure of boranes, earliest concepts of multicenter bonding were developed. Languet-Higgins proposed the theory of 3c-2e (three center-two electron) bonds which greatly helped in the understanding of bonding in boranes. This implies that a pair of electrons can share more than two atomic centers.

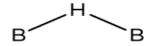
The simplest borane i.e. diborane B₂H₆, is two electron short for bonding when compared with its electron-precise analogue C₂H₆. Thus, diborane is two electron short of conventional 2c-2e bonding. It was proposed that BH₃ exists as dimer to form B₂H₆. This was later confirmed by X-ray diffraction studies. These studies revealed the presence of *terminal* and bridging hydrogen atoms. The two terminal hydrogen on each boron atom formed the conventional 2c-2e bonds thus utilizing eight electrons out of the total of 12 electrons. The remaining two H atoms are bridging in nature. They link together the two boron centers (B-H-B) as shown below. The bridging bonds are electron deficient utilizing only 4 electrons and form 3c-2e bonds.





в-----Н

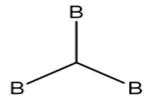
2c-2e boron-hydrogen bond



3c-2e bridging boron-hydrogen-boron bond

в-----в

2c-2e boron-boron bond



3c-2e closed boron bond



3c-2e open B-B-B bond

Various types of bonds found in boranes.

*****CARBORANES:

Carboranes are large family of clusters which contain boron and carbon atoms. They are mixed hydrides of carbon and boron having electron deficient molecular structure. The carboranes are considered to be derived from BnHn² by replacement of BH unit(s) with isoelectronic (since C atom has one more electron than B so CH moiety is isoelectronic with BH and isostructural CH unit(s).

Each C-H group is regarded as donating 3 electrons to the framework electrons. So a replacement of two BH will give a neutral molecule having general formula $B_{n-2}C_2H_n$. Carboranes having n=5 to n=12 are known. They have delocalized electrons in the boron framework having one or more carbon atoms. Carboranes with higher boron content are dominant. The most studied boranes are $B_{10}H_{10}^{2-}$ and $B_{12}H_{12}^{2-}$ thus for carboranes the best studied system is $C_2B_{10}H_{12}$ that is isoelectronic with $[B_{12}H_{12}]^{2-}$. Some other boranes and their corresponding carboranes are

pentaborane(5) $B_5H_5^{2-}$ [$C_2B_3H_5$],hexaborane(6) $B_6H_6^{2-}$ [$C_2B_4H_6$] heptaborane(7) $B_7H_7^{2-}$ [$C_2B_5H_7$], octaborane(8) $B_8H_8^{2-}$ [$C_2B_6H_8$], nonaborane(9) $B_9H_9^{2-}$ [$C_2B_7H_9$], decaborane(10) $B_{10}H_{10}^{2-}$ [$C_2H_8H_{10}$] dianions $B_{11}H_{11}^{2-}$ [$C_2B_9H_{11}$].

Similar to boranes, carboranes are also classified as *closo*, *nido* and *arachno* which can be regarded as derivatives of B_nH_{n+2} (or related anion $B_nH_n^{2-}$), B_nH_{n+4} and B_nH_{n+6} respectively.

i. *Closocarboranes*: General formula $C_xB_{n-x}H_{n-x+2}$ bonded by n+1 skeletal pair of electrons. Eg. Most common with x=2, $C_2B_{n-2}H_n$. One or both C atoms can be replaced by isoelectronic B^- ion to give $CB_{n-1}H_{n+1}$.

ii. *Nidocarboranes*: General formula $C_x B_{n-x} H_{n-x+4}$ bonded by n+2 skeletal pair of electrons. Here also C can be replaced by B⁻ or BH to give compounds that have their skeleton isostructural with $B_n H_{n+4}$

iii. *Arachnocarboranes*: General formula $C_x B_{n-x} H_{n-x+6}$ bonded by n+3 skeletal pair of electrons. These are structurally related to $B_n H_{n+6}$ (i.e. x =0); for example $C_2 B_7 H_{13}$ is related to $B_9 H_{15}$ and $B_9 H_{14}$

3.2 CARBORANE LIGANDS

The carborane anion or dicarbollide anion $[B_9C_2H_{11}]^{2-}$ has been used as ligand and numerous studies have been done on its derivatives. This anion forms sandwich compounds with many metal ions known as bis(dicarbollides). When the 1,2-and 1,7-dicarba-closo-dodecaboranes are heated with alkoxide ions, degradation of parent carborane occurs to form isomeric *nido*carborane anions $(B_9C_2H_{12})^-$.

$$C_{2}B_{10}H_{12} + MeO^{-} + MeOH \xrightarrow{85^{\circ} C} [C_{2}B_{9}H_{12}]^{-} + H_{2} + B(OMe)_{3}$$

$$[C_{2}B_{9}H_{12}]^{-} + NaH \xrightarrow{} [C_{2}B_{9}H_{11}]^{2^{-}} + H_{2} + Na^{+}$$

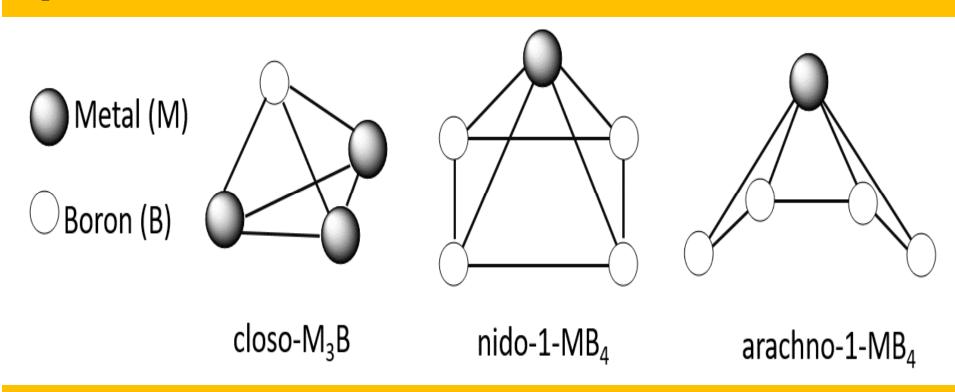
nido 7,8-
$$[C_2B_9H_{11}]^{2-}$$
 + $FeCl_2$ \longrightarrow $\eta^5[7,8-(C_2B_9H_{11})]_2Fe$

4.0 METALLOBORANES

Compounds in which metal atom bound with boron hydride group form the class of compounds known as metalloboranes. They can be classified into following categories:

- 1. Ionic hydroborates: Such as NaBH₄, Ba(BH₄)₂
- 2. Metal hydroborates: Contain hydrogen bridge bonds, M–H–B. Eg: Al(BH₄)₃, (Ph₃P)₂CuBH₄.
- 3. Metal Carbaboranes: Include π -bonded 'sandwich compounds'.
- 4. Compounds containing metal-boron bonds except those in the above category (3).

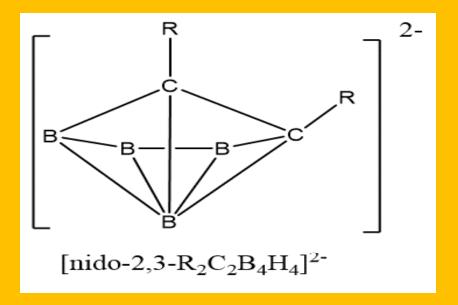
Like boranes and carboranes, metalloboranes can also be structurally classified as *closo*, *nido*, *arachno*, *crypto* and *commo*. Some of them are depicted below.



5.0 METALLACARBORANES

Metallacarboranes or metallacarboboranes are metal complexes where carboranes or heteroboranes act as ligands with at least one metal atom incorporated in the cage framework. They are inorganic polyhedral clusters which contain carbon, boron, hydrogen and metal ion in different combinations. Hawthorne and co-workers first synthesised metallacarboranesin the mid-1960s. A typical example metallacarborane is the $[C_2B_9H_{11}]^{2-}$ (dicarbollide) cluster having a metal atom sandwiched between two dicarbollide units. Here the dicarbollide, binds as η^5 , thus considered to be isolobal with cyclopentadienyl ligand, somewhat similar to ferrocene. Hawthrone suggested that $[C_2B_9H_{11}]^{2-}$ is isoelectronic with C₅H₅ and therefore it should be capable of acting as

There are two different types of metallacarboranes, endo- and exo. In endo- type the metal centre is sandwiched between two carborane clusters are two different types of metallacarboranes, endo- and exo. In endo- type the metal centre is sandwiched between two carborane clusters are two different types of metallacarboranes, endo- and exo.



The other highly investigated metallacarborane is nido-[2,3- $R_2C_2B_4H_4$]₂(R=H or a cage carbon substituents), having pentagonal bipyramidal structure.

5.1 SYNTHESIS

1. Preparation of $[C_2B_9H_{11}]^{2-}$ (dicarbollide)

$$C_2B_{10}H_{12} + MeO^- + 2 MeOH \longrightarrow [C_2B_9H_{12}]^- + H_2 + B(OMe)_3$$

$$[C_2B_9H_{12}]^- + NaH \longrightarrow [C_2B_9H_{11}]^{2-} + H_2 + Na^+$$

2. Preparation of metallacarboranes

$$2[C_2B_9H_{11}]^{2^-} + FeCl_2 \longrightarrow [(C_2B_9H_{11})_2Fe]^{2^-} + 2Cl^-$$

$$[C_2B_9H_{11}]^{2^2} + C_5H_5^2 + FeCl_2 \longrightarrow [C_2B_9H_{11}FeC_5H_5]^2 + 2Cl^2$$

$$[C_2B_9H_{11}]^{2^-} + BrMn(CO)_5$$
 \longrightarrow $[C_2B_9H_{11}Mn(CO)_3]^- + Br^- + 2CO$

***COMPOUNDS WITH METAL-METAL MULTIPLE BONDS:**

Metal clusters are those compounds which have metal-metal bonds. They are as old as chemistry itself. The halide and oxide metal clusters will be discussed here. In these clusters the metal ion is in higher formal oxidation state +2 to +3 and frequently comprise of transition metals on the left side of periodic table (early second row and third row of transition metals). Such as Zr, Nb, Tc, Ru, Rh, Hf, Ta, W, Re, Os, Ir and Pt.

The d orbitals play a crucial role in the formation of these clusters since effective overlap of d orbitals is necessary for the stability of metal cluster. Contraction of d orbitals will cause destabilizing effect on the cluster. It is owing to these reasons that the transition metals of first row even in +2 or +3 oxidation states are unable to extend the d orbitals sufficiently for effective overlap.

> Dinuclear compounds:

A typical example of dinuclear metal compound is $[Re_2X_8]^{2-}$ ion. It can be synthesized as shown below. Reducing agents such as H_2 , H_3PO_2 or PhCOCl can be used. The ion is formed as a royal blue colored product.

2
$$ReO_4^- \xrightarrow{HX} [Re_2X_8]^{2-} X = Cl, Br, I, NCS$$

This compounds shows two very interesting structural features. Firstly, the Re-Re distance in the compound is 224 pm which is shorter than the average Re-Re distance in rhenium metal (at 275 pm) or in Re₃Cl₉ (248 pm). Secondly, the chlorine atoms are present in eclipsed configuration at 330 pm from Re which less than the sum of their van der Waals radii (340-360 pm). So, in such a situation a staggered form would be preferable. Both the above features were explained by F. A. Cotton by proposing a quadruple bond.

*POLYOXOMETALATES (ISOPOLY AND HETEROPOLY ACIDS):

Acid catalysis is an important area of heterogeneous catalysis. Among several acid catalysts polyoxometalates(POM) also known as heteropoly acids (HPAs) hold an important position owing to their catalytic properties. HPAs are used as catalysts in different reactions as acid, redox and bifunctional catalysts in homogeneous and heterogeneous reaction systems owing to their high solubility in polar solvents and thermal stability. They are also used as heterogeneous and homogeneous catalyst in medicine as antiviral and antitumoral agents.

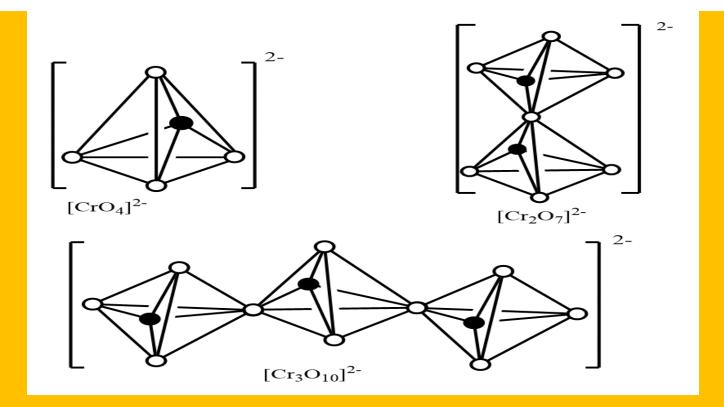
> THE ISOPOLYOXOMETALATES OR ISOPOLY ACIDS:

Some important isopolymetalates are discussed in this section. The basic unit of isopolychromate is CrO_4 tetrahedra. They exist both in solution & as solids. Chromates are stable only at high pH, on lowering the pH protonation and dimerisation occurs and $(Cr_2O_7)^{2-}$. In acidic solution $(Cr_2O_7)^{2-}$, predominates whereas in basic solution $(CrO_4)^{2-}$ predominates.

$$2(CrO_4)^{2-} + H^+ \longrightarrow 2[CrO_3-OH]^- \longrightarrow \left[O_3Cr - O - CrO_3\right]^{2-} + H_2O$$
Basic solution

$$n/2 Cr_2O_7^{2-} + n H^+ \longrightarrow (CrO_3)_n + n/2 H_2O$$

The CrO₃ structure consists of infinite linear chains of CrO₄ tetrahedra.



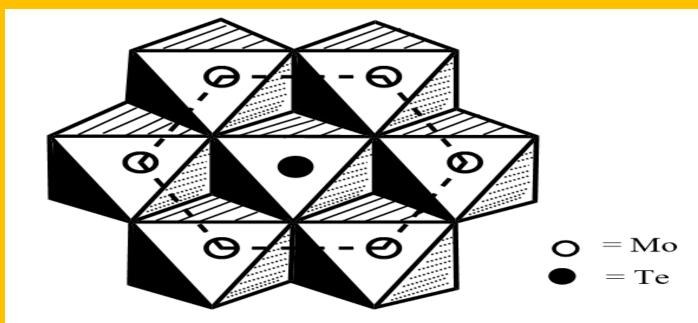
There are certain reports of polymerization such as the Tri chromates $(Cr_3O_{10})^{2-}$ and tetra chromates $(Cr_4O_{13})^{2-}$. Polymerization does not go beyond $(Cr_4O_{13})^{2-}$. These anions as well as dichromate ion are formed by corner sharing CrO_4^{2-} tetrahedra. The tendency for Cr to form large number of polyacids is limited. The small size of Cr^{6+} evidently limits it to the tetrahedral rather than octahedral coordination with oxygen.

> THE HETEROPOLYOXOMETALATES:

determined

Their general formula is $[X_x M_m O_y]^{n-}(x \le m)$; M is usually Mo or W and sometimes V, Nb or Ta. X is the heteroatom which may be located at the centre of polyanion. The heteroatom can be any ele"ment in the periodic table some of the most frequently encountered ions are P^{5+} , As^{5+} , Si^{4+} , Ge^{4+} , B^{3+} etc.

Heteropolyanion are by far mostly important for catalysis and other applications hence they will be discussed in detail. Molybdenum and tungsten polyoxometalates are extensively studied and most easily formed because of the accessibility of their d orbitals forming metal-oxygen π bonds. The structure of the related phosphotung state anion was determined in 1934, and is generally called the Keggin structure after its discoverer. In the period following this other fundamental structures, e.g. the Wells-Dawson ion, were discovered, and their chemistry and applications as catalysts were



6-molybdotellurate anion ; $[TeMo_6O_{24}]^{6-}$

***** TERMINAL QUESTIONS:

- Q1. Sketch the molecule B_2H_6 and show the different bonding between the hydrogen and boron atoms?
- Q2. Which structures (closo, nido, arachno) do the molecules B_5H_8 , $B_4H_4^{2-}$ and $B_{10}H_{14}$ have?
- Q3. What are Boron Hydrides or Boranes? Write a short note on the classification of Boranes with examples.
- Q4. What are metal clusters? Explain the structure of $[Re_2X_8]^2$ ion.
- Q5. Give some examples of metal clusters having metal-metal multiple bonds. Draw structure of any two.
- Q6. What are polyoxometallates? Write short note on Isopoly and heteropolyoxometallates.
- Q7. Explain why polymerization stops in isopoly acids?
- Q8. Write short note on Keggin type polyoxometalate

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THANK