

[The important biological implications of alkali and alkaline earth cations ( $M^{2+}$ ) have led to an unprecedented interest in the interaction studies of these cations with electrically neutral molecules. Professor N. S. Poonia points out in this article that in vitro conditions can be created which lead to a mutual discrimination essentially through the incorporation of the anion and/or solvent effects. Professor Poonia further adds that mutual polarisation of the Fajans' type within the ion pair of a salt is strongly linked with its chemical reactivity including coordinative interaction irrespective of whether coordination involves a dominating polarisation of the cation (self-complexation) or of the donor atoms (ligand-complexation) and brings out a key role of the anion-effect of the counteranion during  $M^{2+}$ -ligand interaction.—Editor]

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## ALKALI AND ALKALINE EARTH CATIONS— INTERACTION PRINCIPLES

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SINCE the development of inorganic chemistry and until recently, the interaction chemistry of alkali ( $M^+$ ) and alkaline earth ( $M^{2+}$ ) cations (general abbr.  $M^{n+}$ ) has been considered to be of the ionic bond and discernible with the help of the simple ionic model<sup>1</sup>. Despite the availability of the information about the formation of stable stoichiometric species such as  $M(\text{halide})_2(\text{NH}_3)_n$  (1830-1923)<sup>2</sup>,  $M(\text{halide})_2(\text{hydroxylic solvent})_n$  (1913-1928)<sup>3</sup>,  $M(\text{halide})_2(\text{aliphatic ketone})_n$  (1907-1930)<sup>3</sup>, and  $M(\text{halide})_2(\text{ether or ester})_n$  (1907)<sup>3</sup>, the chemistry of  $M^{2+}$  had been confined essentially to the chemistry of their salts. This had probably a psychological bearing on the fact that  $M^{2+}$  ions are devoid of the directional partly filled  $d$ -orbitals and a direct link with the fact that interaction of  $M^{2+}$  with most electrically neutral nucleophiles is weak.

Undoubtedly, the  $M^{2+}$ -ligand bond cannot be as highly covalent and strong as for the analogous systems of the transition cations

(abbr.  $M^{n+}$ ). What precisely is the reason for the weakness has not been looked into details, although it has until now been related broadly to the absence of the partly filled  $d$ -orbitals. However, the Lewis acid status of the small  $M^{2+}$  is not always that diminished. For  $\text{Li}^+$  and  $\text{Mg}^{2+}$ , it may be even stronger<sup>4</sup> compared to the various  $M^{2+}$  ions of the  $d$ -series such as  $\text{Mn}^{2+}$  or to the  $p$ -block cations such as  $\text{Tl}^+$ ,  $\text{Pb}^{2+}$ , and  $\text{Bi}^{3+}$ .

Paradoxically, the interaction study of  $M^{2+}$ -salts with neutral ligands has been avoided in polar solvents especially in water because such solvents have been considered drastically hydrolytic towards the  $M^{2+}$ -ligand bond. Consequently, the study of most inorganic (high lattice energy) salts got automatically ruled out while the study of the organic salts, which can be solubilised even in the feebly polar media, had not essentially been resorted. Such factors superimposed on the difficulties due to nonavailability of potential ligands and the suitable  $M^{2+}$ -probe techniques have been

detering greatly the progress of the subject.

In the sixties, the awareness with respect to the role of  $M^{2+}$  in biological systems<sup>5</sup> and availability of the cyclic and acyclic multi-dentates as potential ligands, however, created an unprecedented interest<sup>6-8</sup> in the chemistry of the  $M^{2+}$  ions. Various groups around the world took a contemporary start employing solution (electrometric and calorimetric) and X-ray diffraction techniques. Even with the progress of the work<sup>7,8</sup> most workers, unfortunately, remained interested in technique- and/or the ligand-oriented type of the investigations while the present author maintained a consistent interest in understanding the interactive characteristics of the  $M^{2+}$  ions. This author and the associates undertook the investigations employing macrocyclic ligands (crown ethers) but to establish the chemical principles so discovered, and to evolve more, involved the use of various acyclic multi-dentates and the conventional ligands including the simple solvents in the later work.

Calorimetric and electrometric studies on the  $M^{2+}$  (anion) (ligand) (solvent) systems in solution and the X-ray crystallographic examination of the systems in the solid phase have revealed<sup>5-8</sup> beyond any doubt that  $M^{2+}$  possess coordinative properties in addition to their fundamental chemistry with anions and that  $M^{2+}$ -ligand interactions are unambiguously detectable in solution as well as in the solid phase. The  $M^{2+}$ -ligand interactions are by and large ion-dipole in nature, but they can be discriminated for the different  $M^{2+}$  ions under different anion- and solvent-effects. The overall state of knowledge in the subject gathered in our and other laboratories is outlined in the text to follow.

#### INTERACTION PRINCIPLES

The essential principles involved with respect to the coordination chemistry of  $M^{2+}$ , viz., coordination geometry of the complex and the coordination number of the cation therein, are not as much recognisable for the  $M^{2+}$

ions as for the  $M^{3+}$  ions. Broadly it can be said, however, that the number of the interacting donor sites increases with the size of the  $M^{2+}$  ion and for the reason related to steric problems, the ligands tend to be so distributed around the cation as to make the complex display an approximate geometry which can be used while conveying an idea about the coordination state of the cation. Some complexes of calcium, viz.,  $\text{CaNa}(\alpha\text{-D-galacturonate})_3 \cdot 6\text{H}_2\text{O}$  (9, tricapped trigonal prism)<sup>9a</sup>,  $\text{CaKAsO}_4 \cdot 8\text{H}_2\text{O}$  (8, square antiprism)<sup>9b</sup>,  $\text{Ca}(\text{IO}_3)_2 \cdot 6\text{H}_2\text{O}$  (8, distorted Archimedian antiprism)<sup>9c</sup>,  $\text{Ca}(\text{Pic})_2 \cdot 3\text{H}_2\text{O}$  (7, distorted pentagonal bipyramid)<sup>9d</sup>,  $\text{Ca}(1,3\text{-diphenyl-1,3-propanedionate}) \cdot \frac{1}{2} \text{EtOH}$  (7 and 6)<sup>9e</sup>,  $\text{CaBr}_2 \cdot 6\text{MeOH}$  (6, semiregular octahedron)<sup>9f</sup>, and  $\text{CaCl}_2 \cdot 2\text{MeOH}$  (6, octahedron)<sup>9g</sup>, illustrate the variation range of the coordination geometry and the coordination number shown in respective parenthesis, for instance. The  $M^{2+}$ -ligand interaction is in general weak while the work essentially in our laboratories has revealed that  $M^{2+}$  ions behave fairly anionophilic for the counteranion even in the presence of a chelating ligand and the anion-effect, in turn, becomes a unique feature of their chemistry.

#### Anion-Effect vis-a-vis Anionphilicity

Coordination chemists until now have by and large been familiar with the coordination characteristics of the soft  $M^{2+}$  ions with respect to which the effect of the hard counteranions is quite diminished and rather unimportant. Unfortunately, this psychology was carried forward also for the  $M^{2+}$  ions so that the anion-effect has not been highlighted despite their hard character; with respect to the  $M^{2+}$ -ligand complexation the all-important contribution of the counteranion had in fact not been recognised and not investigated consciously. Consequently, weak complexation of  $M^{2+}$ , in addition to the lack of partly filled *d*-orbitals, has been thought to be mainly due to the hydrolytic effect of the polar medium which has usually always to be used for dissolution of an  $M^{2+}$ -salt. This is the



main point, infact the main mistake, which has largely been coming in the way of understanding the subject of  $M^{z+}$ -complexation. Detailed solution stability investigations in our laboratories for the different  $M^{z+}$ -B15C5 (B15C5, benzo-15-crown-5) systems have shown, for example, that in the poorly solvating media in particular, the  $M^{z+}$ -B15C5 interaction becomes weak<sup>10</sup> for most cations as the counteranion is changed from the most self-stabilised and the least competing 2,4,6-trinitrophenolate (Pic) to 2,4-dinitrophenolate (Dnp) to the most nucleophilic 2-nitrophenolate (Onp); careful scrutiny of the literature reveals such an effect also for some ligands such as Octamethylphosphoramidate<sup>11</sup>, with respect to inorganic anions.

Synthesis possibility of solvated as well as unsolvated  $M^{z+}$ (anion)(phenacylkojate)<sup>12</sup> complexes has been found to be related to the dissociation constant ( $K_a$ ) of the  $M^{z+}$ (anion) pair in the concerned solvent—favoured for moderately dissociated pairs while unfavoured for too highly dissociated as well as too highly associated pairs. In the case of potential chelators such as B15C5<sup>13</sup> and 1,10-phenanthroline<sup>14</sup> addition of water to the solution of  $M^{z+}$  (anion) (ligand) in an organic synthesis medium has led to a facilitated synthesis of the complexes as well as favourable to an increased ligand/cation ratio<sup>15</sup> for the  $M^{z+}$  in the crystallized phase; water understandably stabilises (bonds with) the anion and helps in “lifting” the anion-effect.

The *double-action neutral ligands*<sup>16</sup>, which simultaneously stabilise the cation through coordination and the counteranion through bonding, are capable of lifting the anion-effect themselves and of ensuring a high ligand/cation ratio in the resulting product. The amides, which are under a keen examination by Rao and associates<sup>17</sup> in this country, are outstandingly double-action in character and hence useful for complexation studies with  $M^{z+}$ . The *reinforced ligands*<sup>18</sup>, such as the pyridine oxides, wherein the donor site is based on an electronegative atom, are also powerful in their interaction behaviour. For the ligands

of this type the ligand/cation ratio with the highly polarising cations such as  $Li^+$  and  $Mg^{2+}$  can be as high as 6 and they, like the double-action ligands, frequently cause charge separation of the complexing salt.

The cation-anion association in solution as well as in the solid state is invited also by the  $M^{z+}$  ion. This contribution of the cation is a reflection of its anionphilicity which, in turn, relates itself to its charge/radius ratio. For a given anion, the anionphilicity should in principle increase in the orders  $M^+$  to the similar-sized  $M^{2+}$ ,  $Cs^+$  to  $Li^+$ , and  $Ba^{2+}$  to  $Mg^{2+}$  as evidenced by the lattice energy trends of salts of a halide, for instance<sup>18</sup>.

In case the foreign ligand is only a solvent even then the anion-effect for the  $M^{z+}$ (anion)-solvent system is operative. A detailed solution work<sup>19</sup> on all the  $M^{z+}$  ions has shown that the anion *vis-a-vis* solvent preferences of these cations do not grade monotonously from  $Li^+$  to  $Cs^+$  and  $Mg^{2+}$  to  $Ba^{2+}$ . Though much remains to be learnt in this regard but to us it appears that the mysterious discrimination between the seemingly alike pairs ( $K^+/Na^+$  and  $Ca^{2+}/Mg^{2+}$ ) in biological systems should have a strong bearing on this aspect of their solution chemistry.

#### *Anionphilicity vis-a-vis Fajans' Effect*

Anionphilicity of the cation and anion-effect exercised by the counteranion aid tight pairing for the ions of comparable charge density. Such ion pairs, for which mutual polarisation within the constituting ions is not favoured, display a chemical inertness; the salts  $K^+ClO_4^-$ ,  $Ba^{2+}SO_4^{2-}$ , and  $Li_3^+PO_4^{3-}$ , which are composed of the ions of the same charge or comparable charge density, display a noted water-insolubility<sup>20</sup>.

For a pair involving mutual polarisation of the type introduced by Fajans<sup>21</sup> such as the one in  $Li^+I^-$ ,  $Cs^+F^-$ , or  $K_3^+PO_4^{3-}$ , not only that the ionic bond develops covalency but the salt also develops chemical reactivity<sup>18,20</sup>. The polarised (imbalanced) electronic system of the larger, *i.e.*, the low charge density ion in the pair becomes the site of invitation to



the nucleophilic species—solvent molecules (dissolution) or the neutral ligands (coordination). This vital fact has not only been substantiated with the help of extensive solubility data<sup>3,20</sup> but also experimentally employing ligands such as lower oligoethylene glycols<sup>22</sup> and phenacylkojate<sup>13</sup>.

It is a pity that the chemist community has not until now resorted to the all important mutual-polarisation phenomenon for understanding the chemical reactivity of the ionic systems. We hope to establish that mutual-polarisation of the Fajans' type is a basic phenomenon that determines the chemical reactivity of inorganic salts—fundamental chemistry as well as coordination chemistry—the latter because interaction of a neutral ligand with an ion pair in a melt or on the surface of a solid exploits directly the electronic imbalance in the pair while in solution it substitutes those solvent molecule(s) which interact with the pair because of this imbalance.

#### Anionphilicity vis-a-vis Ligand- and Self-Complexation

While dissolution of an  $M^{n+}$ -salt in a solvent derives the advantage of the Fajans' effect within the salt ion pairs, the solvent/ligand exchange involves mutual polarisation which is between the  $M^{n+}$  ion and the donor atoms of the ligands as permitted by the counteracting charge neutraliser. From the studies on the systems  $M^{n+}$ -B15C5<sup>10,13,23-25</sup>  $M^{n+}$ -DB30C10<sup>23,26</sup> (DB30C10, dibenzo-30-crown-10) in particular, we recognised<sup>27</sup> that the complexation process with the neutral ligand, especially under a rather weak counteracting effect of anions such as iodide and picrate, involves two complimentary processes, viz., *ligand-complexation* and *self-complexation*; the former involves polarisation of the cation by the donor atoms and dominates during complexation of a low charge density cation like  $K^+$  while the latter involves polarisation of the donor atoms by the cation and dominates for a high charge density cation like  $Mg^{2+}$ <sup>27</sup>. For a medium charge density cation

like  $Na^+$  or  $Ca^{2+}$ , for which operation of both ligand- and self-complexation is comparatively weak, a distinct anionphilicity is displayed towards the charge neutraliser<sup>24,28,29</sup>.

X-ray structural results of the  $K^+$ -complexes  $K(B15C5)_2I$ <sup>24</sup>,  $K(B15C5)_2Pic$ <sup>25</sup>, and  $K(DB30C10)I$ <sup>28</sup> as against those of  $NaI(B15C5)$ . aq.<sup>28</sup>,  $(NaNCS)_2$  (DB30C10)<sup>29</sup>, and  $Ca(Pic)_2(B15C5)$ . 3aq<sup>28</sup> illustrate the point some of which are schematically displayed in Fig. 1. Scrutiny of the literature results reveals<sup>18</sup> that the ligand- and self-complexation processes can also be recognized for the conventional ligands as well as for solvents with respect to dissolution<sup>18</sup> of a salt.

Various recent results support our concepts about complexation *vis-a-vis* anionphilicity. Thus, in a given complex the self-complexing  $Mg^{2+}$  can be seen exclusively within the neutral ligand environment while the anionphilic  $Ca^{2+}$  in that very system can be noted totally within the anionic environment—type of a contrast the significance of which needs to be recognised by other workers, too.

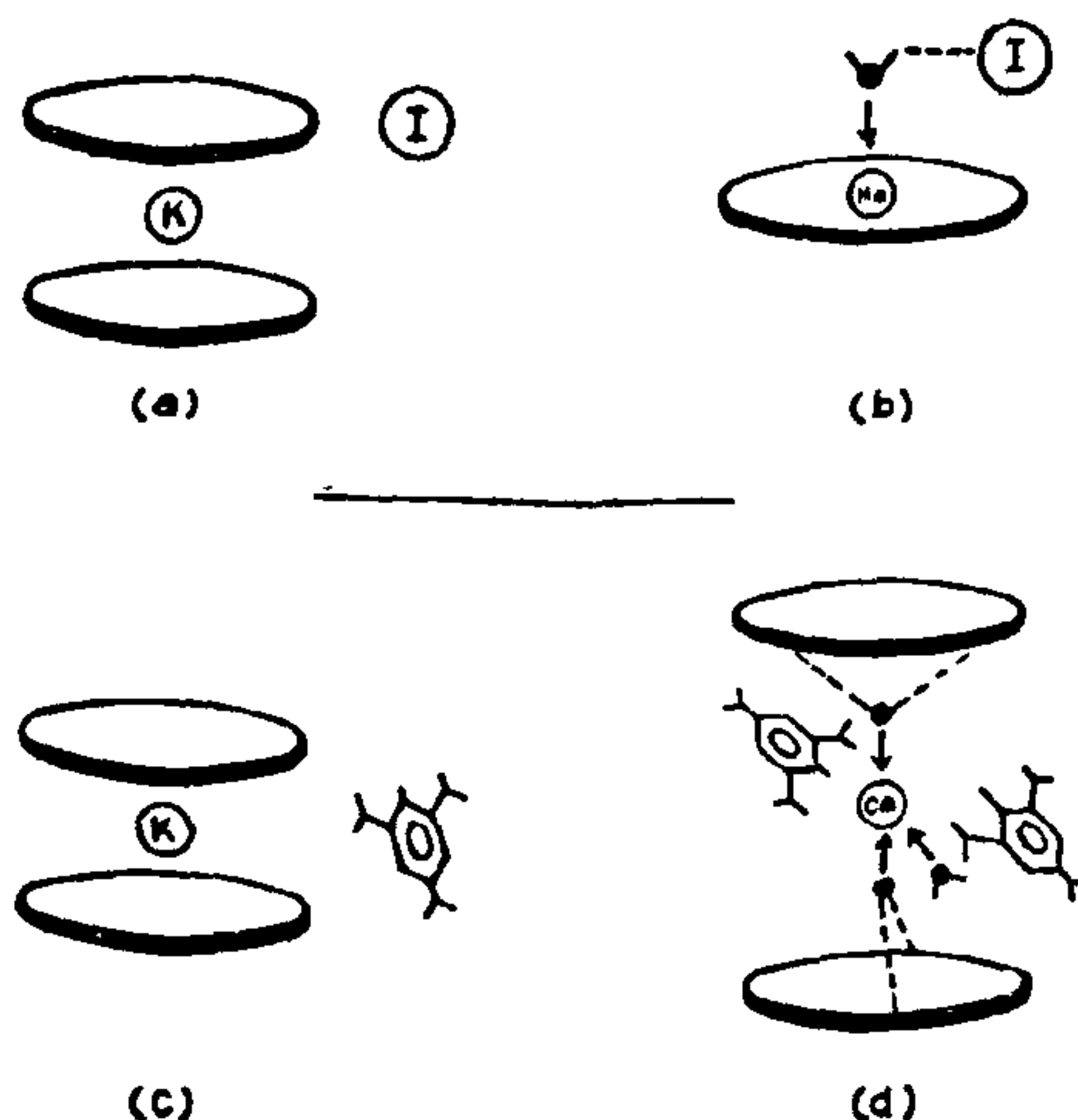


FIG 1. Showing schematically crown-complexation versus anionphilicity of potassium as against that of sodium and calcium in (a)  $K(B15C5)_2I$ , (b)  $NaI(B15C5)$ . aq, (c)  $K(B15C5)_2Pic$ , and (d)  $Ca(Pic)_2(B15C5)$ . 3aq.



## PRESENT STATUS AND FUTURE TRENDS

The understanding of the subject is becoming satisfactory to the extent that a degree of prediction about the interactive behaviour of a given  $M^{n+}$  (anion) (ligand) (solvent) system is possible<sup>16</sup>. The Lewis acid status of different  $M^{n+}$  ions has been familiarised to a stage that the efficiency and route of an organic reaction can be controlled through a deliberate participation of an  $M^{n+}$  ion in reaction<sup>30</sup>—a new field which may prove immensely useful not only for understanding the mechanism of such reactions but also for controlling the efficiency of industrial synthesis of various organics and stereoselective synthesis of important biomolecules such as peptides.  $M^{n+}$ -ligand compatibility has been invoked such that template synthesis<sup>31</sup> of macro (especially the macrocyclic) molecules is becoming a common practice and the use of active (naked) counteranions<sup>32</sup> in organic synthesis is drawing everincreasing attention. The reaction selectivities of  $M^{n+}$  with acyclic multidentates (in particular have been understood to the extent that ion-selective electrodes based on diverse multidentates<sup>33</sup> have been standardised for most  $M^{n+}$  which are useful in their analytical chemistry.

Finer understanding of interaction chemistry of the  $M^{n+}$  ions as well as of their mysterious role in biological systems, calls for further elaborated work, however. Since anionphilicity and solventphilicity of  $M^{n+}$  do not appear to grade monotonously from  $Li^+$  to  $Cs^+$  and from  $Mg^{2+}$  to  $Ba^{2+}$ , it is expected that a detailed solution work of  $M^{n+}$  under different anion and solvent effects will reveal some specific and interesting anion *vis-a-vis* solvent preferences for each cation which may ultimately be related to their chemical differentiation as noted for the natural systems. Work with acyclic multidentates<sup>34</sup>, which carry the partial advantage of both the cyclic macromolecules and the conventional bi- and tridentates, may add information to the extent that  $Na^+$ ,  $K^+$ , and  $Mg^{2+}$  and  $Ca^{2+}$  may be followed in natural systems through their

own chemistry rather through the use of probes which may not<sup>35</sup> be a dependable approach under most conditions.

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### ALL INDIA INSTITUTE OF ANIMAL CELL CULTURE AND DIFFERENTIATION

The Department of Zoology, University of Poona, Pune-7 will be organizing an All India Institute on 'Animal Cell Culture and Differentiation', sponsored by the UGC and DST, from 20th November to 15th December 1981. The teaching programme of the Institute includes lectures and laboratory experiments on cell-tissue-culture techniques and their applications as well as aspects of cell structure and function. The

staff of the Institute include eminent scientists in the field from many Indian laboratories.

Research workers from Universities and other National Laboratories in India desirous of participating in the institute should contact Prof. S. P. Modak, Department of Zoology, University of Poona, Pune 411 007. Participants will be paid TA and DA at rates admissible by the University.