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THESES ABSTRACTS

Commencing with this issue we will be publishing extended abstracts of the theses accepted for the award of the Ph.D. and M.Sc. degrees of the Indian Institute of Science. This feature which will appear in every issue would provide an introduction to the research activity of the Indian Institute of Science.

Editor

Thesis Abstract (Ph.D.)

Nonstoichiometry of chemically-prepared lead zirconate titanate (PZT) solid solution

by R. Balachandran.
Research supervisor: T.R.N. Kutty.
Department: Inorganic and Physical Chemistry.

Solid solutions based on $PbTiO_3$-$PbZrO_3$ (PZT) ceramics with perovskite structure have gained increased importance because of their superior dielectric properties. Unlike the titanates and zirconates of alkaline earth metals, those containing Pb(II) have large nonstoichiometry arising from the removal of Pb and O through the escape of the more covalent PbO, resulting in coupled nonstoichiometry at Pb and O sublattices[1]. Since the physical properties are greatly affected by the nonstoichiometry, the understanding of the nature of lattice defects involved therein is of great importance. The PZT phases prepared by the chemical methods, as compared to the conventional ceramic technique, have better control of stoichiometry and inherent purification is possible[2]. Although atomic scale mixing is expected, and hence the high degree of homogeneity, it is possible that the materials prepared by these techniques are affected by the side reactions during their formation. The existing reports on the extent and the sign of nonstoichiometry with temperature are contradictory. Therefore, the present investigations have been undertaken with most homogeneous chemically-prepared PZT phases. They are prepared in the complete range of Zr/Ti ratios by direct syntheses from the oxalate precursors as well as by the hydrothermal methods. The thermolysis of lead zirconyl titanyl oxalates for the formation of PZT powders is investigated in detail, taking into account the reducing atmosphere generated during decompositions[3]. As an alternative technique, a procedure has been standardized for the direct precipitation of PZT by hydrothermal reactions[4].

Since the free PbO, but not PZT, alone is soluble in dilute acetic acid, a suitable analytical procedure could be devised for the PbO-excess compositions. Whereas for XO_2-excess (X = Ti or Zr) cases, the perovskite alone is soluble in hot hydrochloric acid, the nonstoichiometric phases approach stoichiometry according to the reaction

$$Pb_{1-\gamma}XO_{3-\gamma} + \gamma PbO = Pb_{1-\gamma}XO_{3-\gamma}. \qquad (1)$$

where γ' tends to zero on equilibrium annealing. If F is the mass fraction of XO_2, F_{SS} the mass fraction of XO_2 in PbO solid solution and F_{PX} the mass fraction in the nonstoichiometric perovskite, then according to Lever rule,

$$F' = (m_{SS} + m_{PX}) = m_{PX} + m_{SS} F_{SS} \tag{2}$$

So that,

$$F' \left(1 \mid \frac{m_{SS}}{m_{PX}}\right) = F_{PX} + F_{SS} \left(\frac{m_{SS}}{m_{PX}}\right) \tag{3}$$

is applicable. By plotting (m_{SS}/m_{PX}) against $F'(1+m_{SS}/m_{PX})$ for various initial compositions at a given temperature, we get a straight line with F_{PX} as the intercept and F_{SS} as the slope. The values of nonstoichiometry for PZT phases of varying Zr/Ti ratios can thus be obtained. The end-member phases have nonstoichiometry of $\gamma \sim 0.1$ whereas intermediate Zr/Ti phases have $\gamma \sim 0.025$. The γ-values decrease from 450 to 900°C and increase at higher temperatures. The samples prepared by the precursor technique have higher nonstoichiometry than those obtained from hydrothermal reactions.

How is the nonstoichiometry as high as 10 mole % maintained in monophasic perovskite PZT lattices? The measurements of physical properties as a function of γ-values have been carried out in a wide range of temperature and oxygen partial pressures (PO_2). These measurements include pycnometric density, electrical resistivity, Seebeck coefficient, lattice parameters, integrated intensity of X-ray powder diffraction lines and the electron paramagnetic resonance spectra of $Fe^{3+}-V_0$ defect complexes in PZT phases[5]. The theoretical dependence of these physical parameters based on various lattice defect models which include independent vacancies, interstitial cations and crystallographic shear (CS) planes are calculated. The experimental values show clear disagreement with the point defect models whereas they show better agreement with the CS model. The ABO_3 perovskite structure, with the removal of AO component approaches the ReO_3-type structure which is particularly prone to CS plane defects. These defects bring in more compaction relative to the initial volume. The stability of CS plane defect increases with Zr/Ti ratios. Kinetics and mechanism of the reaction between PbO and $Pb_{1-y}X_{3-y}$ in the solid state have also been carried out.

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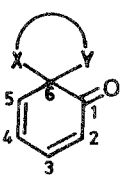
Thesis Abstract (Ph.D.)

Chemistry of spironaphthalenones by G. Madhusudhan Reddy.

Research supervisor: T.R. Kasturi.
Department: Organic Chemistry.

Introduction

Spirodienones, a class of compounds which occur widely in nature, are important in bio-synthesis, and have a fascinating chemistry. The topic is of current interest to many organic chemists and is an area for future development[1]. Fungal metabolites like geodin, erdin, geodoxin, proaporphine alkaloids (*e.g.* pronuciferine, stepharine, orientalinone, etc.), homoproaporphine alkaloids (*e.g.* kreysiginone) are compounds, though isolated from different sources, having a common spirodienone moiety. The term spirodienone is used in this context to describe derivatives of cyclohexa-2,4-and 2,5-dienone (I and II) in which carbon atoms 6 and 4 respectively are spiro centres.



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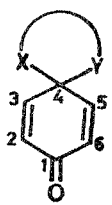

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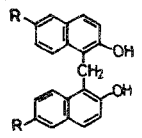
Of greater significance than the natural occurrence of spirodienones is their role in the biosynthesis of alkaloids, isoflavones, etc. Thus the suggestion made by Barton and Cohen[2] in 1957 that spirodienones might be intermediates in the bio-synthesis of certain types of isoquinoline alkaloids has received convincing support from subsequent labelling experiments[3]. Another reason why spirodienones have attracted the attention of organic chemists is that they react or rearrange on treatment with many reagents to give thermodynamically more stable benzene derivatives[1,4]. In view of this, we have synthesized some spironaphthalenones of this class of compounds and studied their fascinating chemistry.

Results and discussion

Oxidation reaction of various substituted bisnaphthols (1) with 2,3-dichloro-5, 6-dicyano-1, 4-benzoquinone (DDQ) was studied in detail. This reaction gave a novel *cis*-& *trans*-dispironaphthalenones (2) & (3) along with naphthoquinonemethide dimers (4) and spiroketones (5). All these compounds were characterized on the basis of spectral properties. The complex structure of *cis*-dispironaphthalenone (2) was further confirmed by X-ray crystal structure analysis and also by an independent synthesis.

X-ray crystal structure analysis[5] of *cis*-dispironaphthalenone (2) revealed that 2′, 3′ carbon-carbon bond is unusually elongated (1.616 Å instead of 1.54 Å of normal C_{sp^3}-C_{sp^3} bond). In the mass



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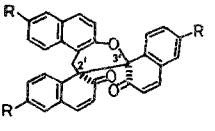

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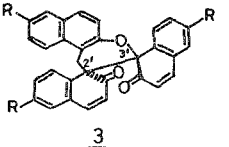

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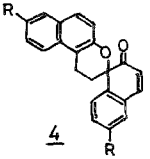

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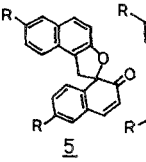

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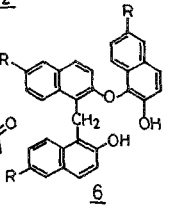

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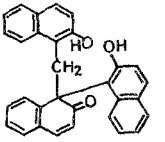

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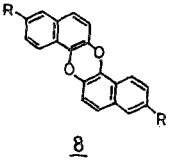

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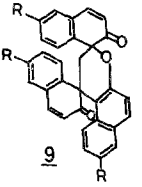

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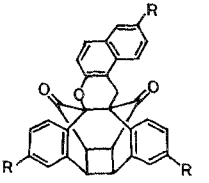

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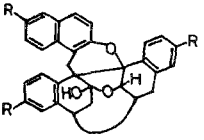

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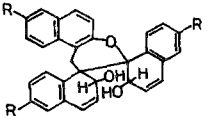

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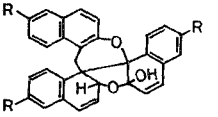

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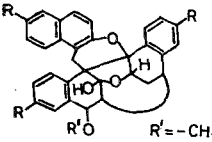

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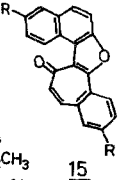
R′ = −CH3
−CH2CH3
−CH(CH3)2



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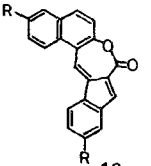

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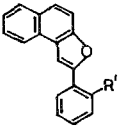

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a) R′ = CH=CH−CH2OH
b) = CH2CH2CH2OH
c) = CH=CH−COOCH3

R = H ; Br ; C(CH3)3 ; OCH3 etc.

spectrum of this compound also, most of the fragments arise from the initial cleavage of this bond indicating that this particular bond is weak and could be broken easily. This is further substantiated by hydrogenation experiment and zinc-acetic acid reduction. Thus, hydrogenation of *cis*- & *trans*-dispironaphthalenones (2) & (3) with 10% palladium carbon in dry ethyl acetate afforded a dihydroxy compound (6). This is apparently formed by cleavage of the C(2')– C(3') bond instead of the C(3')-O bond, which would lead to compound (7) normally expected on the basis of hydrogenolysis of α-keto-spiroquinol ethers. Reduction of the dispironaphthalenone (2) with zinc and acetic acid also gave through cleavage of the C (2') - C (3') bond the same dihydroxy compound (6), identified by mixed m.p., IR and ^{1}H NMR. Because of the weakness of the C (2')– C (3') bond in dispironaphthalenone (2), we thought that this compound could be a useful model for less known photochemical β-C-C cleavages in enones. Crystal structure of this compound also reveals that the two olefinic π-bonds are fairly well oriented for 2+2 cycloaddition which can lead to cage-type products. With these expectations, photochemical irradiations of *cis*-dispironaphthalenones (2) were studied. All the compounds upon irradiation resulted in the formation of products (3), (4), (8), (9) and (10). These compounds are believed to arise from the lowest excited state of compound (2) via β-C-C cleavage [compounds (4), (8) and (3)], β-C-O cleavage [compound (9)] and intramolecular cycloaddition [compound (10)]. Cleavage of the particular C (2') - C (3') carbon-carbon bond was also demonstrated under pyrolytic conditions in dispironaphthalenones (2) & (3) to give compounds (4) and (8).

Further, borohydride reduction of the compound (2) was found to give interesting results. The reductions of the compound (2) in tetrahydrofuran gave a novel intramolecular reductive C-C coupled product (11) along with biallylic alcohol (12) and hemiketal (13). The above reduction under alkaline conditions in the presence of alcohols resulted in the formation of alkoxy incorporated intramolecular reductive C-C coupled product (14) along with corresponding hemiketal (13). The above reaction was carried out with various substituted dispironaphthalenones in the presence of alcohols like methanol, ethanol and isopropanol. The structure assigned for alkoxy intramolecular C-C coupled products was finally confirmed by X-ray crystal structure analysis of compound (14) (R = Br; R' = CH_3).

The fascinating rearrangements of spiroketones (5) were studied. Reaction of spiroketones (5) with DDQ in dry benzene resulted in the formation of a novel furanotropone and benzfulvene derivatives (15) & (16). The structure assigned for compound (15) was also confirmed by X-ray crystal structure analysis. The benzfulvene derivative (16) underwent an unusual C-C bond cleavage under various reaction conditions. Thus, sodium borohydride reduction of benzfulvene derivative gives an alcohol for which 2 [2' (−CH=CH−CH_2OH) phenyl] naphthofuran structure (17a) has been assigned. This alcohol on hydrogenation with 10% Pd-C gives a dihydroalcohol (17b). The same dihydroalcohol was also obtained from benzfulvene derivative itself by lithium aluminium hydride reduction. Further, the benzfulvene derivative was shown to give an ester (17c) on base hydrolysis followed by methylation with dimethyl sulphate.

The structures of all the compounds isolated in the above reactions were characterised on the basis of thorough spectral analysis like IR, UV, mass (high resolution), ^{1}H NMR (including proton decoupling experiments) and ^{13}C NMR and also by chemical transformations. Suitable mechanisms have been proposed for all the above reactions studied.

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Thesis Abstract (Ph.D.)

A study of DNA methylation in the mealybug, *Planococcus lilacinus* by K. Muralidharan.

Research supervisors: H. Sharat Chandra and T.M. Jacob.

Department: Molecular and Cell Biology Laboratory.

The mealybug *Planococcus lilacinus* possesses an unusual genetic system. In most nuclei of males of this organism the paternal chromosomes are heterochromatic and genetically inactive. In females, on the other hand, both paternal and maternal sets are euchromatic and genetically or potentially active. The genetic and molecular basis of such imprinting of a set of chromosomes as paternal and the relationship of such chromosome inactivation to sex determination are questions of obvious interest. Although a great deal of information is available on the cytogenetics of this and related organisms[1], it is only recently that the tools of molecular biology are being applied towards understanding these systems. The present thesis is an attempt in this direction.

In the introduction, the mealybug genetic system is discussed. Two other related genetic systems, *Comstokiella* and *Diaspidid*, are also included for comparison. Molecular mechanisms that have been proposed to explain such differential regulation of homologous chromosomes are mentioned. A brief review of DNA methylation and its possible role in chromosome inactivation is included. The Sager-Kitchin model[2] envisages that the egg cytoplasm contains a modifying system and that the sex of the embryo is dependent on whether or not the sperm chromosomes are methylated at particular sites. If methylated, the sperm chromosomes turn euchromatic. If unmethylated, they are acted upon by a 'restriction'-type system which would lead to base changes in DNA and heterochromatization.

We have attempted to compare the DNAs from males and females of *Planococcus lilacinus*. In addition to looking for possible differences in base composition we looked for modified bases in the DNA of the two sexes and further compared the levels of modified bases in the DNA of the two sexes.

The diploid chromosome number of *P. lilacinus* is ten. The nuclear DNA content, as determined by Feulgen microdensitometry, was 0.23 ± 0.012 picograms per diploid nucleus. Thermal denaturation of DNA yielded a G+C value of 33.2% in both sexes. In CsCl buoyant density gradient centrifugation, DNA from males appeared to have a slightly different buoyant density than female DNA (about 1.6938 g cm^{-3} in male as compared to 1.6862 g cm^{-3} in female). Analysis of male and female DNAs by fine melting and CsCl-Actinomycin-D buoyant density gradient centrifugation did not show any differences. There was no indication of major satellites.

Significant amounts of three modified bases—5 methylcytosine (5mC), 6 methyladenine (6mA) and 7 methylguanine (7mG)—were detected in DNA samples from both sexes. Evidence for the presence of 5mC in male and female DNA came from Msp I/Hpa II digestion, two-dimensional thin layer chromatography (TLC), high performance liquid chromatography (HPLC) and antibodies raised against 5-methyl cytidine. Evidence for the presence of 6mA was obtained by TLC, HPLC and immunochemical assay using 6mA-specific antibodies. Evidence from TLC, HPLC and the piperidine reaction indicated the presence of 7mG.

Dinucleotide analysis of DNA indicated that in this organism 5mC, 6mA and 7mG are found in a variety of dinucleotide combinations. Consequently, it appeared to us that the commonly used Msp I/Hpa II digestion as well as Dpn I/Sau 3A/Mbo I digestion may not be sufficient to detect differences, if any, in the level of modified bases in the two sexes. A kinetic-enzyme-linked immunosorbant assay (K-ELISA) using biotin-avidin-peroxidase was therefore devised. The specificity and affinity of the 5mC and 6mA antibodies were determined. When male and female DNAs were studied by K-ELISA, male DNA appeared to contain lower levels of 5mC than female DNA; 6mA appeared to be more common in male DNA than in female DNA. Piperidine digestion of male and female DNA seemed to indicate higher levels of 7mG in male than in female DNA.

Loewus *et al*[3] using paper chromatography were unable to detect any modified bases in the DNA of a related mealybug, *P. citri*. Our results are also in contrast to those of Scarbrough *et al*[4] who could detect, by HPLC, 5mC but neither 6mA nor 7mG in two other species of mealybugs. Further, they found that male DNA contains more 5mC than female DNA.

Apart from gene regulation, DNA methylation also appears to play a role in chromosome regulation. Mealybugs offer a situation where one can possibly resolve the role methylation plays in gene regulation from the role methylation may play in chromosome regulation. The mealybug vitellogenin genes seem to offer an unusual opportunity for investigating this problem. In an Appendix to this thesis, we describe preliminary studies on the isolation, purification and characterization of mealybug vitellogenins. These proteins are lipoglycoproteins, synthesised in mature females, and stored in the egg. A genomic library of *P. lilacinus* DNA was made in Lambda Charon 4a. It should be possible to pick out the vitellogenin sequences and use them to study the pattern of methylation of these genes in the various structural and activity states of mealybug chromosomes.

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Thesis Abstract (Ph.D.)

Systems of learning automata: Estimator algorithms and applications by P.S. Sastry.

Research supervisor: M.A.L. Thathachar.
Department: Electrical Engineering.

Introduction

Learning is an important aspect of intelligent behaviour. Colloquially, a person is called intelligent if he is capable of coping with a new situation both effectively and efficiently. In most cases this capability is a cumulative result of past experiences. This ability of people, commonly known as learning, has been the subject of speculation by philosophers for many centuries now. A systematic study of learning was started only around the turn of the century by psychologists. These results from mathematical psychology are well documented[1,2]. In engineering sciences, interest in learning is of a more recent origin[3,4]. Scientists working in systems engineering and lately in artificial intelligence are trying to analyse and model learning so as to be able to incorporate such behaviour in engineering systems[5,6].

From an engineering view point, a system can be called learning if it improves its performance through experience. This improvement is often brought about by changes in the internal structure effected by the system itself in the light of the experience gained by it. There are many probabilistic and fuzzy models proposed which exhibit simple learning behaviour[5,7]. Interconnection of such models can result in complex learning behaviour. Stochastic variable structure automata operating in random environments are among the various probabilistic models proposed to realize learning behaviour. In this context, they are referred to as learning automata. Presented in this thesis is a systematic investigation of the behaviour of collectives of such learning automata.

Learning automaton

A learning automaton model is what is known as choice behaviour in psychology. At each instant, the automaton makes a choice from a finite set of alternatives available to it and the environment responds to this choice by a random reward. The automaton learns to make a choice that maximises the expected reward by processing the random environmental responses through a learning algorithm[8].

The automaton makes its choice probabilistically. The choices are represented by a finite set of actions $A = \{\alpha_1, ..., \alpha_r\}$. The state at instant k, Q(k), of the automaton includes an action probability distribution $p(k) = [p_1(k) \ldots p_r(k)]^T$, wehre $p_i(k)$ is the probability of choosing α_i at k. The learning algorithm, which is also called the reinforcement scheme, updates Q(k) and hence $p(k)$, knowing $\alpha(k)$, the action selected, and $\beta(k)$, the random reward obtained at k. The environment is characterized by a set of probability distributions, $\mathscr{F} = \{F_i, ..., F_r\}$, where F_i is the conditional distribution of $\beta(k)$ given $\alpha(k) = \alpha_i$. If F_i are independent of k then the environment is said to be stationary; otherwise, it is non-stationary. In stationary environments d_i denotes the expectation of F_i. It is assumed that $\beta(k)$ (and hence d_i) takes values in a subset of $[0, 1]$. If $d_m = \max \{d_i\}$ then α_m is called the optimal action. It is desired that asymptotically $p(k) \to e_m$ where e_m is the r-dimensional unit vector with mth component unity.

Within this framework, the automaton functions as follows. At each instant k, the automaton selects an action at random depending on the probability distribution $p(k)$. For this choice, it gets a response from the environment. Then using the learning algorithm, it updates its state Q(k) and the cycle repeats.

Introduced in the thesis is a new class of learning algorithms which are termed estimator algorithms.

These algorithms use some estimated quantities regarding the random characteristics of the environment in updating $p(k)$. These estimates are obtained in the normal functioning of the automaton and are stored in the state Q(K) and hence are updated at each instant through the learning algorithm. For the simplest case of a single automaton acting in a random environment, this results in $Q(k) = (p(k), \hat{d}(k))$, where $\hat{d}(k)$ are the estimated reward probabilities at k. $\hat{d}(k)$ are obtained as sample means of the rewards received. When action α_i is selected the updating of $p(k)$ is as follows. All actions α_j such that $d_j > d_i$, get their probabilities increased and the others get their probabilities decreased in proportion to their current estimated worth. These changes should be properly designed so that $p(k+1)$ is still a probability vector.

It has been shown that for this class of algorithms $p(k)$ can be made to converge to e_m as desired by using sufficiently small learning parameter (which controls the step size in $p(k)$ updating). It is seen that these algorithms converge many times faster than the conventional learning algorithms. Also, with a finite memory estimator, these algorithms exhibit good tracking ability in environments whose random characteristics switch in time.

It is shown that using the concept of estimator algorithms, the automaton can be made to learn any relevant characteristics of the environment. The methodology is quite powerful and readily extends to tackle many different systems of learning automata.

Systems of learning automata

The thesis considers, in detail, three different systems of learning automata. These are hierarchical systems of learning automata with multiple reactions, two person zero-sum games and N-person cooperative games played by automata players.

Hierarchical systems with multiple reactions are natural models for many sequential learning process. Such structures are useful, for example, while learning in the context of Markovian decision processes. Estimator algorithms to be used by individual automata in the hierarchy are developed using which the hierarchy converges to its optimal path. In cases where the optimal path satisfies some local optimality condition, the reinforcements at different levels of the hierarchy can be decoupled and hence can be performed concurrently. When this is not the case, the algorithm needs information transfer from the lower to the higher levels of the hierarchy and it finds the globally-optimal path. In addition to this being the first algorithm which can learn the globally-optimal path in a general hierarchy, the flow direction of information here well captures the idea behind hierarchical decomposition of any problem.

Competitive and cooperative games are models widely used in Systems Science to study multiperson interactions. An estimator algorithm is devised for use by the automata players involved in a two-person zero-sum sequential stochastic game with incomplete information. Using this the players can learn the optimal mixed strategy in a general $m \times n$ matrix game. It is shown that the players converge to optimal strategies in the sense of weak convergence even when optimal strategy may not be unique. The thesis also considers cooperative games of many automata when each automaton gets the same pay-off. Estimator algorithms are designed for such games with common pay-off played by N-automata so that each of the players converges to his optimal strategy.

The general features of estimator algorithms noted in the context of a single automaton, namely, superior speed of convergence and generality as regarding learning the relevant characteristics of the environment are shown to carry over to the case of systems consisting of multiple automata also.

Applications

Two applications of these models are also considered in detail.

The first one is the problem of learning optimal discriminant functions in pattern recognition problems with completely unknown class conditional densities and prior probabilities. The problem is posed as a game with common pay-off played by a number of automata. Using no more than a set of labelled-training samples, the automata team is shown to learn the discriminant function which minimises the probability of misclassification. It is assumed that the functional form of the desired discriminant function is known. This is a common assumption to all approaches that learn the discriminant function without assuming the class conditional densities[9]. The assumption here is that it is easier to decide on the type of discrimination sought rather than guess the class conditional densities. The noteworthy feature of the automata approach is that the team can learn discriminant functions that are nonlinear in their parameters also. This is in contrast to all the existing methods for learning in pattern recognition.

The second problem that is considered in detail is that of relaxation labelling in computer vision. A general labelling problem is posed in terms of sets of labels and objects along with a neighbour relation over objects and some constraints on possible labels at neighbouring objects. In any image understanding task, one wishes to assign interpretations to various positions in the picture array. Due to noise and variations of illumination, reflectance, etc., in the scene, it may not be possible to uniquely interpret every portion of the image. But using neighbour information and domain specific knowledge in the form of constraints, many problems in computer vision can be posed as labelling problems[10]. In the thesis, the general labelling problem is formulated in terms of a cooperative game of learning automata. In the paradigm of the algorithm, at each instant the team puts forward a tentative labelling of the scene. A critic process (or the environment) evaluates the labelling locally and supplies a stochastic or noisy response to each of the automata depending on the local consistency of their choices. The team cooperatively evolves the globally-consistent labelling. The model can accommodate a range of uncertain knowledge about the local constraints unlike any of the other existing algorithms. The method performs well even when the consistency constraints are assumed completely known. The method can be viewed as a probabilisitic algorithm for the continuous-labelling problem. A local convergence result is proved around every consistent unambiguous labelling. The algorithm has inherent parallelism and can be efficiently implemented on an array processor.

A novel feature of analyses presented in the thesis is the extensive use of weak convergence techniques for analysing learning algorithms. Using these techniques, an asymptotic approximation to the stochastic difference equation representing the learning algorithm can be obtained in the form of an ordinary differential equation. This allows for easier analysis of the asymptotic behaviour of the learning system.

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