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# International Symposium on Ionic Polymerization IP'97

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Conference Report

## International Symposium on Ionic Polymerization: IP'97

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IP'97



Paris: 7-11 July/Juillet 1997

The International Symposium on Ionic Polymerization was held in Paris from July 7 to July 11, 1997. It was held under the auspices of the International Union for Pure and Applied Chemistry (IUPAC), the European Polymer Federation (EPF)

the Centre National de la Recherche Scientifique (CNRS), the Groupe Française des Polymères (GFPO), the Division Polymères et Elastomères de la SFC and the Université Pierre et Marie Curie (UPMC).



Przemyslaw Kubisa



Otto Vogl



**Jean Pierre Vairon**



**Symposium Opening**

**Pierre Sigwalt**

**Jean Pierre Vairon**

**President of  
Université Pierre et Marie Curie**

The Symposium was held under the Cochairmanship of Hervé Cheradame and Jean-Pierre Vairon with Pierre Sigwalt as the Honorary Chairman.

The meeting which was unusually well organized and arranged, was attended by about 190 participants from 25 countries. 45 Main Lectures were presented; 48 contributions were given orally and 40 papers were presented in poster form. The contributed papers were equally divided between cationic, anionic and ring-opening polymerizations. If there were any criticism, of the way the 45 Lectures were organized, it might be that there could have been about 5 "Plenary" Lectures in which some of the general subjects were summarized by established authorities with vision. We now have in these fields a few scientists who have very general knowledge that could have given such lectures not a summary of their own research.



**Otto Vogl**

**Joseph P. Kennedy**



**At the Reception**

The following were presented:

**Joseph P. Kennedy** of the University of Akron, Akron, OH, U.S.A.: *Quo Vadis Ionic Polymerization?* The progress in the field of cationic polymerization was presented in broad perspectives with special emphasis on objectives of academic and industrial research. The most recent advances made in the author's laboratory were outlined.

**Bruce M. Novak** of the University of Massachusetts, Amherst, MA, U.S.A.: *Building Chiral Molecular Architectures Using Living Transition-Metal Initiating Polymerizations.* The living polymerization leading to extended-chain, helical polymers (polymerization of carbodiimides with initiators of CpTiCl<sub>2</sub>X type) was described. It was shown, that annealing leads to the transformation of the helical conformation formed under kinetic control into thermodynamically controlled conformation.

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**Thio E. Hogen-Esch** of the University of Southern California, Los Angeles, CA, U.S.A.: *Ylide-Mediated Polymerizations of Methyl Methacrylate. Aspects of Initiation and Termination Reactions.* The living character of methyl methacrylate polymerization initiated with tetraphenylphosphonium carbanions was discussed. The related initiating system were less suitable for the polymerization of acrylates (e.g. butyl acrylate); side reaction leading to chain termination was identified as intramolecular Claisen reaction.

**Stanislaw Penczek** of the Center of Molecular and Macromolecular Studies of the Polish Academy in Lodz, Poland: *Intra- and Intermolecular Chain Transfer to Macromolecules with Chain Scission. The Case of Cyclic Ethers.* The general treatment of the kinetics of ionic polymerization of heterocyclic monomers involving propagation and intermolecular chain transfer to polymer was presented. The analytical solution of the set of kinetic equations allowed the determination of  $k_p/k_t$  ratio from the dependence of  $M_w/M_n$  on conversion. The validity of the treatment was confirmed by analysis of the kinetics of L-lactide polymerization.

**Krzysztof Matyjaszewski** of the Carnegie Mellon University, Pittsburgh, PA, U.S.A.: *Novel Block Copolymers by Transformation of "Living" Carbocationic to "Living" Radical Polymerization.* The mechanism of ATRP (Atom Transfer Radical Polymerization) was discussed and the possibilities of preparing complex polymer architectures by this approach were indicated and illustrated by several examples.

**Bernadette Charleux** of University Pierre and Marie Curie, Paris VI, Paris, France: *Model Kinetic Study of  $TiCl_4$ -Induced Ionization of Polyisobutene Capped with Diphenylethylene. Application to the Synthesis of Block Copolymers.* The ionization of model compound: 1-methoxy-1,1-diphenyl-3,3,5,5-tetramethylhexane (model of active species of isobutylene polymerization capped with diphenylethylene) was studied, allowing the conclusions on the capping efficiency and stability of the cation.

**Reimund Stadler** of the University of Bayreuth, Bayreuth, Germany: *Anionic Homo- and Copolymerization of  $N,N$ -Dialkylaminoisoprenes.* The new synthesis of amino functionalized monomer: 5-N, N-diisopropylamino isoprene was described and the kinetics of its polymerization was analyzed. Copolymerization of the monomers that were studied led to block copolymers: strong influence of the amino group on the copolymerization behavior and the microstructure of copolymers was observed.

**Zbigniew Jedlinski** of the Center of Polymer Chemistry, Polish Academy, Zabrze, Poland: *Regio- and Stereoselective Anionic Polymerization of Lactones.* The mechanism of anionic polymerization of lactones initiated by alkali metals complexed with crown ethers was discussed with special emphasis on the regio- and stereoselectivity of ring-opening reactions.

**Shokyoku Kanaoka** of the University of Shiga Prefecture, Hikone, Japan: *The Propagating Species in Living Cationic Polymerization: Living Nature and Steric Structure of Polymers.* The living polymerization of isobutyl vinyl ether at different conditions was discussed from the point of view of the influence of solvent polarity on the stereochemistry of propagation.

**Marcel van Beylen** of the University of Leuven, Heverlee, Belgium: *Influence of Salts on the Reactivity of Polystyryllithium in Etheral Solvents.* The complex role of LiCl as additive in



The Pantheon

the anionic polymerization of styrene was studied. It was shown, that at low concentration LiCl suppresses the dissociation of polystyryllithium, whereas at higher concentrations, it increases the dissociation, yielding triple and quintuple ions.

**Philippe Guerin** of the University Paris XII, Thiais, France: *Optically Active Polymers via 3-Alkylmalic Acids: Contribution of the Bioconversion for Diversifying the Chiral Precursors.* The enzymatic route to optically pure derivatives of aspartic acid was described. These monomers could be polymerized to optically active isotactic polyesters.

**Daniel J. Brunelle** of the General Electric Company, Schenectady, NY, U.S.A.: *Semi-Crystalline Polymers via Ring-Opening Polymerization: Preparation and Polymerization of Alkylene Phthalate Cyclic Oligomers.* The synthesis of cyclic oligomeric alkylene phthalates with >85 % yield by pseudo-high dilution method and their polymerization to high molecular weight polyester, crystallizing at the polymerization temperature 180–200 °C, was described.

**Hsien C. Wang** of the Exxon Chemical Co., Baytown, TX, U.S.A.: *New Commercialized Isobutene-Based Elastomers.* The methods of improving processability of butyl rubbers, by broadening the molecular weight distribution as the result of formation of star-like macromolecules were discussed. The synthesis, characterization and properties of new isobutylene based elastomers, developed and commercialized recently by Exxon Chemical Co. were presented.

**Christophe Novarro** of Elf Atochem LACQ, France: *Ligated Anionic Polymerization: An Innovative ELF-ATOACHEM Technology for the Polymerization of (Meth)Acrylates.* A recent Elf Atochem process using alkoxyalkoxides – based initiating systems for ultra-fast anionic polymerization of (meth)acrylates was presented. Applications of the process for the synthesis of syndiotactic poly(methyl methacrylate) and block copolymers were discussed.

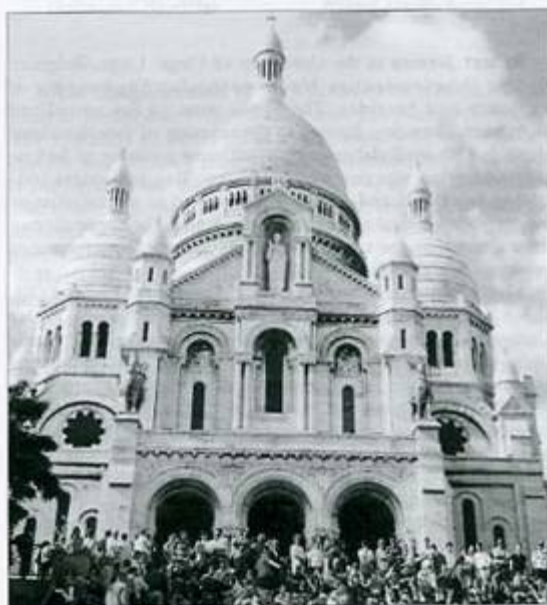


Boulevard St. Michel



Judit Puskas

Gyorgy Deak



Sacré Coeur

**Konrad Knoll** of BASF A.G., Ludwigshafen, Germany: *From Highly Processable Thermoplastic Elastomers to Transparent High Impact Polystyrene – The Gamut of BASF's Anionic Styrene Butadiene Block Copolymers.* Several new brands of commercial BASF styrene-butadiene thermoplastic elastomers, including symmetrical triblock copolymer (Styroflex®) in which the soft segment is a statistical SB sequence and star block copolymer (Styrolux®) was described.

**Judit E. Puskas** of the University of Western Ontario, Canada: *Multiarm-Star Polyisobutylenes by the "Core First" Method.* The synthesis of multiarm-star high molecular weight polyisobutylene ( $M_w$  up to 400 000) with narrow molecular weight distribution ( $M_w/M_n = 1.2$ ) containing up to 23 arms by the "core first" method was presented.

**Junzo Masamoto** of Kyoto Institute of Technology, Matsugasaki, Kyoto, Japan: *Specific Phenomena During Copolymerization of Trioxane and Ethylene Oxide.* The Asahi Chemicals process for manufacturing the acetal resins by cationic copolymerization of 1,3,5-trioxane with ethylene oxide has been described and the formation of cyclic oligomers during the induction period was discussed.

**Bela Ivan** of the Central Research Institut for Chemistry of the Hungarian Academy of Sciences, Budapest, Hungary: *Open Problems of the Mechanism of Living Carbocationic Polymerizations.* The existing views on the mechanism of living carbocationic polymerization was reviewed and the role of added nucleophiles was critically analyzed.

**Yves Gnanou** of the University of Bordeaux, Talence, France: *New  $\sigma$ - $\mu$ - Ligands for the Anionic Polymerization of Methyl Acrylates in Apolar Medium.* The role of chelating agents in the anionic polymerization of methyl methacrylate was discussed and stereochemical arguments were presented, indicating that chelating agents not only interact with counterions but also modify the nature of anionic active species.

**Stanislaw Slomkowski** of the Center of Molecular and Macromolecular Studies of the Polish Academy of Sciences, Lodz, Poland: *Mechanism of Particle Formation and Kinetics of the Dispersion Polymerization of Cyclic Esters.* The direct preparation of polyester microspheres by anionic polymerization of  $\epsilon$ -caprolactone and lactides in 1,4-dioxane was described. Mechanism and kinetics of the dispersion polymerization were discussed; due to the high concentration of active species in microspheres, the dispersion polymerization is considerably faster than polymerization in solution.

**Alain Deffieux** of the University of Bordeaux, Talence, France: *Design of New Polymer Architectures by Combination of*

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*Anionic and Cationic Living Polymerization Techniques.* The general, step-by-step route for the preparation of polystyrene and polybutadiene with the chain architecture of increasing complexity based on successive and repeated interconversion of poly(chloroethyl vinyl ether) blocks obtained by cationic polymerization and polystyrene or polybutadiene blocks obtained by anionic polymerization, was described.

**Axel H. E. Müller** of the University of Mainz, Mainz, Germany: *Novel Initiating Systems for the Living Polymerization of Acrylates and Methacrylates.* The formation of coordinative polymer network due to coordination of the growing chain ends with an ether group of the other chain was observed in the anionic polymerization of methyl methacrylate in the presence of aluminum alkyls in toluene. In the presence of Lewis bases the formation of networks was prevented and the system showed all the features of a living system.

**Hans R. Kricheldorf** of the Institute of Technical and Macromolecular Chemistry, Hamburg, Germany: *Cationic Polymerization of Aliphatic Cyclocarbonates.* Mechanism of cationic polymerization of aliphatic cyclocarbonates with various initiators was studied.  $\text{BuSnCl}$  was found to be a particularly useful initiator for synthetic applications; a mechanism of polymerization initiated by tin compounds (cationic polymerization vs insertion) was discussed.

**Julian Chojnowski** of the Center of Molecular and Macromolecular Studies, of the Polish Academy of Sciences, Lodz, Poland: *Selectivity of the Polymerization of Cyclic Siloxanes with Mixed Siloxane Units.* The cationic and anionic polymerization of model cyclotrisiloxanes containing two different types of siloxane units were studied. The mechanism of polymerization was deduced on the basis of the analysis of polymerization kinetics, sequence distribution and NMR studies of active centers.



**Axel Müller**



**Moulin Rouge**

**Robert Jerome** of the University of Liege, Liege, Belgium: *Recent Development in Macromolecular Engineering of Lactones and Lactides.* The application of functionalized aluminum alkoxides, formed in the reaction of functionalized alcohols with trialkylaluminum, as efficient initiators of lactone polymerization was described. The resulting polyesters containing functional end-groups may be used as macroinitiators for controlled radical or ionic polymerization. The application of these systems for the synthesis of comb, graft and hyperbranched polymers, was discussed.

**Virgil Percec** of Case Western University, Cleveland OH, U.S.A.: *Controlling the Shape of the Polymer Chain by Living Cationic Ring-Opening Polymerization of Monodendritic Cyclic Imino Ethers.* Cationic polymerization of monodendrons containing cyclic iminoether polymerizable groups, was described. In such systems, the polymerizable group is jacketed by a cylindrical or spherical coat generated from the dendritic part of the molecule. These supramolecular reactors produce polymerization mechanisms different from polymerizations in solutions.

**Eric J. Goethals** of the University of Ghent, Ghent, Belgium: *Poly(vinyl ethers) as Building Blocks for New Materials.* The synthesis of poly(vinyl ether)-methacrylate macromonomers and their homo- and copolymerization leading to star-shaped or highly branched polymers was described. Corresponding bis-methacrylate terminated poly(vinyl ethers), polymerized in the presence of styrene or (meth)acrylates, gave segmented polymer networks.

**David M. Haddleton** of the University of Warwick, Coventry, UK: *Polymethacrylate Star Polymers from Living GTP. Anionic and Radical Polymerization.* Synthesis of methacrylate star polymers by the addition of ethylene glycol dimethacrylate to living polymethacrylates was presented. Star polymers with block copolymer arms were also described and the effect of reaction conditions on the molecular weights of the star polymer was studied.



Reimund Stadler

Nikos Hadjichristidis

**Nikos Hadjichristidis** of the University of Athens, Athens, Greece: *Model Block Copolymers with Complex Architecture*. The synthesis of novel block copolymers with the structure (BA)<sub>n</sub>B and related star or H-shaped architecture, in which B is polystyrene and A is polyisoprene-1,4, was achieved. The microphase separation, studied by different methods, showed the influence of the architecture in the phase boundaries of different morphologies in the phase diagram.

**Herbert Mayr** of the Ludwig-Maximilian University Munich, Munich, Germany: *Alkyl Chloride - Boron Trichloride-Initiated Polymerization of Isobutylene: Detailed Analysis of the Initial Propagating Steps*. The studies of the kinetics of the reaction between cumyl chloride and isobutylene in the presence of BCl<sub>3</sub> was studied as a model of cationic isobutylene polymerization. It was shown, that the rate of propagation at the early stage increases with increasing length of the chain, leading to narrowing of the molecular weight distribution.

**Swaminathan Sivaram** of the National Chemical Laboratory, Pune, India: *Functionalization of Anionic and GTP Chain Ends in Methacrylate Polymerization: Problems and Solutions*. The problem of reactivity of enolate chain end in the polymerization of methacrylates was discussed in the context of the efficiency of chain ends functionalization.

**Przemyslaw Kubisa** of the Center of Molecular and Macromolecular Studies of the Polish Academy of Sciences, Lodz, Poland: *Copolyethers with Controlled Structure: Mechanism of Formation and Microstructure*. Cationic copolymerization of tetrahydrofuran with ethylene oxide in the presence of diols was described in terms of contribution of *Active Chain End* and *Activated Monomer* propagation mechanisms. Influence of kinetic and thermodynamic factors on the microstructure of the copolymers was discussed.

**Rudolf Faust** of the University of Massachusetts in Lowell, Lowell, MA, U.S.A.: *The Role of Pyridine Derivatives in Living Carbocationic Polymerization: Lewis Base or Nucleophile?*. The



The Louvre

role of nucleophiles in the living cationic polymerization of isobutylene was discussed. It was shown, that nucleophiles act as scavengers of protic impurities, in some cases, however, may lead to proton elimination, affecting the living character of the process.

**Christo B. Tsvetanov** of the Institute of Polymers, Bulgarian Academy of Sciences, Sofia, Bulgaria: *Quaternary Ammonium Salts as Additives in Anionic Polymerization of Methacrylates*. The mechanism of anionic polymerization of methyl methacrylate in the presence of adducts of Bu<sub>3</sub>Mg and tBuOLi with quaternary ammonium salts (R<sub>3</sub>N<sup>+</sup>X<sup>-</sup>) was discussed with special emphasis on the effect of size of ligand R on polydispersity and tacticity of the polymers.

**Patrick Hemery** of the University Pierre and Marie Curie, Paris VI, Paris, France: *Ionic Polymerization in Aqueous Emulsion*. The mechanisms of ionic polymerization of cyclosiloxanes in aqueous emulsions were discussed. The main reactions take place at monomer/water interface which affects the side reactions. In the case of octamethylcyclotetrasiloxane polymers with M<sub>n</sub> up to 40 000 with a polydispersity index 1.1-1.5 were prepared.

**Yusuf Yagci** of the Technical University of Istanbul, Maslak, Istanbul, Turkey: *Addition-Fragmentation Type Initiator for Cationic Polymerization*. The method of initiation of the cationic polymerization of e.g., cyclohexene oxide by radical cations generated by addition of radicals formed by thermal decomposition of radical initiator to the addition-fragmentation agent (AFA) (allyl onium salts), with the subsequent fragmentation of AFA was described.

**Pierre J. Lutz** of the Institute Charles Sadron, Strasbourg, France: *Anionic Polymerization and Copolymerization of Macromonomers: Kinetics, Structure Control*. The synthesis and polymerization of polystyrene and poly(ethylene oxide) (PEO) macromonomers was described. The dependence of the molecular weight of the macromonomer on the propagation rate constant was determined. Amphiphilic "dumbbell" polymers, containing a central polystyrene block linking two branched PEO parts, were obtained.

**Shiro Kobayashi** of Kyoto University, Kyoto, Japan: *Enzymatic Ring-Opening Polyaddition for Chitin Synthesis: A Cationic Mechanism in Basic Media?* The first synthesis of the naturally occurring polysaccharide - chitin - by ring-opening

## Conference Report



Yusuf Yagci

Mrs. van Beylen

polymerization of a sugar oxazoline monomer catalyzed by an enzyme, was described. The process was stereo- and regio-specific and gave high molecular weight chitin in quantitative yield.

**Guangying Wu** of Beijing University of Chemical Technology, Beijing, P.R. China: *The Role of Electron Donors in the Cationic Polymerization of Esters (or Ethers, Alkanols)/Lewis Acids/Monomers/Polar Diluents (or Solvents) Systems*. The role of electron donors in cationic polymerization was discussed from the point of view of competition between different complexation processes.

**Roderic P. Quirk** of the University of Akron, Akron, OH, U.S.A.: *Polymer Synthesis Using Functionalized Alkylolithium Initiators*. The application of alkylolithium initiators containing protected hydroxyl groups for the synthesis of  $\alpha$ -hydroxy and  $\alpha,\omega$ -dihydroxy polybutadienes was described. This approach was used also for the preparation of hydroxy functionalized star-branched polymers and random, tapered block and block copolymers of styrene and dienes.

**Takeshi Endo** of the Tokyo Institute of Technology, Nagatsuta-cho, Yokohama, Japan: *Cationic Equilibrium Ring-Opening Polymerization of Bicyclic Monomers and its Application to Chemical Recycle*. Cationic polymerization of spiroorthoester type monomers leading to reversible cross-linking was described. In one of the studied systems, a methacrylate with a spiroorthoester substituent was synthesized and polymerized radically to a linear polymer, which could be reversibly crosslinked by opening of the spiroorthoester ring.

**Koichi Hatada** of Osaka University, Toyonaka, Osaka, Japan: *Preparation of Stereoregular Uniform PMMA Architectures and their Stereocomplex Formation*. The synthesis of narrow molecular weight distribution isotactic and syndiotactic poly(methyl methacrylate) and the highly efficient separation of the products by supercritical fluid chromatography, was described. The uniform samples were used to study the mechanism

of the formation of stereocomplexes. Hydroxy terminated uniform polymers were reacted with acid dichlorides to form uniform stereoblock copolymers and the formation of intra- and intermolecular stereocomplexes was studied.

**Phillipe Teyssie** of the University of Liege, Liege, Belgium: *Living Anionic Polymerization of (Meth)acrylic Esters: New Mechanistic Concepts and Resulting Materials*. The new developments in the area of anionic polymerization of (meth)acrylates were discussed, including application of ligated anionic polymerization for the synthesis of highly isotactic and highly syndiotactic polymers, synthesis of tailored PMMA-polybutadiene-PMMA triblock copolymers and a method allowing the initiation of MMA (also styrene and diene) polymerization by weakly nucleophilic groups such as alcoholates or silanolates.

**James V. Crivello** of Rensselaer Polytechnic Institute, Troy, NY, U.S.A.: *Tandem Transition Metal Catalyzed Isomerization and Cationic Polymerization*. The polymerization of mono- di- and multifunctional allyl ethers by new transition metal catalyzed tandem isomerization and cationic polymerization was described. The applied catalyst induced first the isomerization of allylic ether to the corresponding vinyl or enol ether, which in the second step polymerized by cationic mechanism.

**Pierre Sigwalt** of the Université Pierre and Marie Curie, Paris VI, Paris, France: *Some Still Unsolved Problems in Carbocationic Polymerization*. The critical review of the state of art in the carbocationic polymerization with special emphasis on the reactivities of free and paired ions, the effect of nucleophiles and electron donors and the possibilities of controlling the transfer and termination reactions, were presented.

On Sunday, July 11, a welcoming reception was hosted by the organizers of the conference in the foyer of the Ministry of Commerce where the participants had the opportunity to meet old friends and make new acquaintances.

On Monday evening the President of the Université Pierre et Marie Curie, Paris VI, invited the participants to a party at the top of the famous tower of the Université Paris VI. This location is certainly one of the most exciting and spectacular places and



At the Banquet





*Mrs. Vairon    Norbert Bikales    Mrs. Tsvetanov*

sights in Paris. The weather was clear and perfect: the view over Paris was breathtaking, and, even for those that knew Paris, it was exciting and unique.

Tuesday evening a buffet dinner was enjoyed on the boat on the Seine during a four hour cruise up and down the Seine. The river Seine divides Paris but, at the same time, it is the hearth and life line of Paris, and the center of much of the life of Paris. For these four hours the participants of the Conference enjoyed the spirit of Paris on the boat and then sailed to the end of the practical life of Paris. What memorable experience!

Wednesday, the banquet of the conference was held in the Restaurant of the Louvre. While sailing on the Seine the evening before was an exciting experience of the present day Paris, the banquet took us three to four hundred years back in history. It had been arranged that the banquet was held in the restaurant

in the Louvre. It was next to the Paris of the middle ages "below Paris". This old part of Paris City had been recently restored and is well preserved. The restaurant is very extensive, much of it located below the Louvre. What an exciting experience! The banquet at this center of art and culture at the Louvre, with its exquisite cuisine, wine and camaraderie led by the people that had been the mainstay of these conferences on ionic, formerly cationic, and ring-opening polymerizations was the high point of the conference.

Now that the regular organization of Symposia on "Ionic Polymerization" is well under way, plans have been made for the next conference. For 1999, the Symposium on "Ionic Polymerization" will be held in Kyoto under the Chairmanship of Professor Shiro Kobayashi. The Ionic Symposium in Paris could be called the Second Ionic Symposium on Ionic Polymerization, the Symposium in Istanbul being the First Symposium on Ionic Polymerization. For political reasons it was agreed on not numbering at this time the newly established combined meetings on "Ionic Polymerizations". Still there is the fact that Istanbul was the first agreed combined meeting of "Ionic Polymerizations" [for the history of these meetings see *Polymer News* **21**(10), 352-359 (1996); *Progress in Polymer Science*, **22**(6), 1381-1395 (1997)].

It also has been informally agreed upon that the next Symposium on Ionic polymerization which is scheduled to be held in Kyoto in 1999 include the most important subject of coordinative polymerization that leads to polyolefins. Especially the discussions of the catalysts that lead to polyolefins of controlled molecular weight molecular weight distribution, living nature of the polymerization stereospecificity of polymerization copolymers and polymers of the proper particle size for direct fabrication is highly desirable.

The participants are looking forward to the Conference in Kyoto and indicated that they will be working diligently to provide new results, new insights and a more complete venue of all aspects of ionic polymerizations. The subsequent symposium on Ionic Polymerization is planned in Crete, Greece.