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Croatian Symposium of Chemistry and Macromolecular Chemistry

Otto Vogl

University of Massachusetts - Amherst, vogl@polysci.umass.edu

Zvonimir Janovic

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Conference Report

Croatian Symposium of Chemistry and Macromolecular Chemistry

Zvonimir Janović, University of Zagreb, Zagreb, Croatia and Otto Vogl
Polytechnic University, Brooklyn, NY 11201, U.S.A



Zvonimir Janovic



Otto Vogl



Dragomir Fleš

A joint Meeting, the XIV. Symposium of the Croatian Chemical Society and the IV. Croatian Symposium of the Chemical and Technological Macromolecular Society was held in Zagreb, Croatia from February 6 to February 8, 1995. It was the first joint meeting of the two Societies and was attended by more than 500 participants. They came primarily from Croatia but participants from 12 other countries attended. The organization of the Meeting consisted of 17 plenary lectures and 370 papers that were presented in poster form. The plenary lectures represented the major subject area of chemistry and macromolecular chemistry and the poster session was also divided into several categories: a.) Inorganic Chemistry and Solid State Chemistry, b.) Organic Chemistry—this section was by far the largest section and included synthesis, reaction mechanisms and natural compounds; c.) Physical Chemistry—thermodynamics, kinetics and structure; d.) Theoretical Chemistry, e.) Chemistry and Instrumentation f.) Chemical and Biochemical Engineering, g.) Industrial Processes and Quality Control, h.) Environmental Protection and i.) Chemical Education.

The sessions of the IV. Croatia Symposium on Chemistry and Technology of Macromolecules included, synthesis, processes characterization and application.

In this article we will comment specifically only on the

plenary lectures and on the poster sessions of the IV. Symposium on Chemistry and Technology of Macromolecules.

The Symposium was under the Chairmanship of Professor Zvonimir Janović of the University of Zagreb, who was the chairman of the entire Symposium and of the organizing committee. The meeting was opened by the Minister of Science, Mr. B. Jeren, the rector of the University of Zagreb, Professor M. Sunjič, representatives of the Ministry of Education, the Ministry of Industry, the Croatian Chemical Society and of the Croatian Academy of Arts and Sciences.

After the Opening Ceremony, the Symposium was opened with a plenary lecture by Otto Vogl of Brooklyn, NY USA entitled **Macromolecular Architecture and Design**. He emphasized that polymer science and technology will have a continuing and increasing impact on the well being of mankind. Many needs that are essential for our existence and for the improvement of human life will be influenced by polymer science and technology. Specifically mentioned were clothing,



Emblems of the conference



Zagreb



In the Old City

food and nutrition, shelter and housing, health care and medicine, construction, transportation, energy and communication.

Commodity plastics have reached a level of maturity in industrialized countries as can be seen from the drop or their relative growth rate. A number of important global companies have shifted their thrust of involvement from commodity plastics into more profitable ventures, such as products that serve health care, communication and transportation.

The speaker discussed selected areas that are now receiving considerable attention and pointed out that the economic synthesis of monomers and polymers will require more and efficient catalytic processes. In technical polymer synthesis, the importance of the production of polymeric materials with narrow and broad molecular weight distribution "on demand" for both homo- and copolymers are needed as is the concern of polymer production vs. the disposal.

Living cationic, anionic and now even radical polymerizations (at least in some important cases!) still show new avenues of development. Important examples are the methacrylate polymerization by group transfer techniques, macromolecular engineering by controlled carbocationic polymerization and methacrylate polymerization by cobalt complexes. We have learned and are still learning how to make star copolymers, hyperbranched polymers and dendrimers.

Fiber reinforced plastic materials play an important part in many polymer applications using ultrahigh molecular weight highly drawn polyethylene, carbon and aramide fibers. Reinforced epoxy laminates are starting to be used in

construction, for repair and for the salvation of bridges and buildings.

More efficient antioxidants and ultraviolet stabilizers for polymers and better ways of applying them are being developed. Most prominent are stabilizers, bound to the polymers; they are compatible, non-fugitive and capable of surface assembly of the polymer chains, now sometimes called morphology engineering.

Stable and Persistent Bis(ketenes): Novel Chemical Intermediates of Theoretical and Practical Importance were the subject of Thomas T. Tidwell, Toronto, Canada. The study of ketene chemistry has become of renewed interest worldwide. The effect of substituents on the ketene structure and reactivity and the origin of the extraordinary stabilizing effect of silyl substituents on ketenes has been recently elucidated by *ab initio* molecular orbital calculations. The authors have prepared silylated cyclobutenediones where the substituents are trialkylsilyl groups. Thermolysis of these compounds gives the more stable 1,2-bis(ketene) as the only observable product. 1,2-Bis(ketenes) are reactive toward a variety of reagents, including oxygen, water, alcohols, and electrophiles.



Opera, Zagreb

Cyclodextrins as Tools in Scientific Research and Industrial Applications was discussed by Wolfram Saenger, Berlin, Germany. Cyclodextrins are obtained by enzymatic degradation of starch. They are cyclic oligosaccharides consisting of 6 to 10 glucose units with $\alpha(1-4)$ linkages. Due to their angular structure, they, like common crown ethers, are able to form inclusion complexes even in solution. Cyclodextrins are used as excellent models for a number of biological processes where noncovalent intermolecular interactions are desired. The formation of these inclusion compounds has been followed by different spectroscopic techniques.

Bernard Schrader, Essen, Germany presented his recent work on **New Applications of NIR FT Raman Spectroscopy**. Since its discovery in 1928, Raman spectroscopy, a valuable tool in science, has never become a method for routine analysis in the industrial environment. This is caused by the fact that most samples contain impurities that are inherent in the samples or products of oxidation. Of special importance are the products of photochemical and thermal reactions which fluoresce after excitation with the usual exciting radiation for Raman spectroscopy at 488 or 515 nm. It has now become possible to

strength, the ultimate toughness of polymeric materials, another important engineering property, is poorly understood as demonstrated by the numerous papers with conflicting views. Toughness is in first approximation proportional to the total energy involved in deformation up to fracture, i.e. the area under the stress-strain curve. On a microscopic level (i.e. inside deformation zones) a satisfying correlation has been established between the local strain and the strain-to-break based on stretching the molecular network to its full extension.



Zvonimir Janović

Helena Jasna Mencer

J. Villadsen, Lyngby, Denmark presented his talk on the **Reaction Engineering and Microbial Physiology—The Concept of The "Cell Factory"**. Microorganisms are able to produce valuable metabolites and proteins that cannot economically be synthesized by other routes. Antibiotics and a number of other pharmaceuticals are examples of high value added products, ethanol and acids such as lactic acid and gluconic acid are examples of bulk chemicals which can be produced from agricultural waste at a cost which may be economically attractive.

Three plenary lectures addressed the General Problem of treatment of wastes. **Biological Purification of Waste Gases Technical Applications—Process Technology Fundamentals** was discussed by K. Kirchner, Karl Winnacker Institut der DECHEMA e. V., Frankfurt, Germany. The biological treatment of waste gases is being increasingly used in various industries. It is applied especially in those areas where waste gases can develop that contain pollutants in small concentrations, e.g. odor emissions. Biological methods rely on the degradation of the pollutants by bacteria. The pollutants must be soluble in water. One advantage of the biological treatment of exhaust gases is the relatively low reaction temperature. On the other hand, the presence of certain substances can deactivate or kill bacteria. This means that only exhaust gases of known composition can be purified biologically, as is the case with the catalytic treatment of exhaust gases.

Chemical Reaction Engineering Principles In Solving Environmental Problems: Unconventional Waste Water Treatment Technologies was addressed by J. Levec, Ljubljana, Slovenia. Increasing demand for the re-use of water and



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Igor Čatič

increasingly stringent water quality regulations lead to the need for treating all of the various waste stream containing pollutants. Waste waters originating from industrial activities contain organic pollutants which are either toxic or biodegradable, consequently direct biological treatment is not feasible.

Computer Integrated Plant Operations In Chemical & Refining Industry was presented by V. Mahalec, Cambridge, MA. Plant models throughout plant life cycle (from design to plant operation to revamp), including decision making in process control, on-line optimization, production scheduling and production planning were examined. Such models become the basis for models used in all aspects of the plant decision making: a.) Design modifications; b.) Design of Advanced Process Control Strategies; c.) On-line Optimization and d.) Production Planning and Scheduling.

Advances In Polymer Blends, was the subject of the talk by Roberto Greco, Napoli, Italy representing E. Martuscelli. Syntheses of new polymers are still being developed in order to meet the growing demand of materials tailored for specific technological needs. In recent years and, specifically, in the last decade, multicomponent polymeric systems became the subject of intensive study. New properties could be achieved by the proper matching of the properties of already existing polymers.

Multicomponent polymer systems are made from a thermoplastic or thermosetting matrix intimately mixed to one or more minor polymer phases. The influence of the molecular structure of components (tacticity and constitution), composition, procedures of blending and processing and crystallization conditions on the kinetic and thermodynamic factors of the crystallization process, phase structure in the melt and the condensed state and morphology has been investigated for different type of polymer blends containing crystallizable and amorphous components.

The blend types may be assembled, according to the mutual miscibility of components in the melt and in the amorphous state as follows: a) Miscible, b) Phase separated, c) Immiscible. The speaker was particularly interested in: a.) *Melt miscible blends*; b.) *Melt phase separated blends*; c.) *Melt immiscible blends and d.) Reactive blending technologies*. Interesting materials with improved properties were realized starting with the following types of polymer pairs.



Zagreb, Chamber of Commerce



Statue, Josip Jelačić

record Raman spectra, using laser emitting radiation at 1064 nm, in a relatively short time, if interferometers are used. The application of Raman spectroscopy in the field of biochemistry is especially rewarding, since water, the main constituent of biomaterials, which absorbs heavily in the infrared range, gives only a weak Raman spectrum. Of special importance for this utilization is the effectiveness of NIR FT Raman spectroscopy in medical diagnostic; here the recording

conditions are especially delicate.

Polymerization of p-Methylstyrene was presented by F. Tudos, Budapest, Hungary. Radical bulk polymerization of p-methylstyrene was investigated at 50°C. with AIBN as initiator. The results of measurements were compared with the relevant literature data. The overall rate constant of p-methylstyrene was practically equal to that of styrene, but vinyl toluene polymerizes at about a 20% higher rate. The inhibitive effect of p-nitroso aniline derivatives and other aromatic nitroso compounds (s-trinitrobenzene, o- and p-dinitrobenzene) was studied on the bulk polymerization of p-methylstyrene. The inhibitive effects of p-benzoquinone and its derivatives (toluquinone, o-xyloquinone) was also studied in bulk and solution polymerization. All these compounds were found to be effective inhibitors.

Ultimate Properties of Synthetic Polymer Systems was the subject of the plenary lecture of Peter J. Lemstra, Eindhoven, The Netherlands. Polymer Science and Technology is the paradigm of an integrated discipline in which Chemistry, Physics, Processing and Design constitute a "chain-of-knowledge". In order to exploit the intrinsic potential, aiming at ultimate properties of synthetic polymeric materials, the various elements in this "chain-of-knowledge" need to be linked and be of equal strength.

Creative chemistry requires creative processing and examples were presented concerning novel processing routes of intractable, high T_g polymers, using reactive solvents. Apart from sophisticated chemistry, substantial progress has been made in the past two decades concerning physical aspects of polymer science and technology. A striking example in this respect is the development of high-modulus and high-strength fibers based on the "primus inter pares" of simplicity in synthetic polymers, polyethylene. Besides the development of high-strength/high-modulus fibers based on flexible macromolecules, another important parallel line of research was the development of high performance fibers based on intrinsically rigid macromolecules, i.e. the aromatic polyamide fibers.

In contrast with the ultimate properties like stiffness and



At Lunch

strength, the ultimate toughness of polymeric materials, another important engineering property, is poorly understood as demonstrated by the numerous papers with conflicting views. Toughness is in first approximation proportional to the total energy involved in deformation up to fracture, i.e. the area under the stress-strain curve. On a microscopic level (i.e. inside deformation zones) a satisfying correlation has been established between the local strain and the strain-to-break based on stretching the molecular network to its full extension.



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A Small Church in Zagreb

Blends with a thermoplastic polymer as matrix: 1) Polyamide-6/Modified ethylene-propylene rubbers; 2) Polybutylene terephthalate/Modified ethylene-propylene rubbers; 3) Polymethylmethacrylate/Ethylene-vinyl acetate copolymers; 4) Polyhydroxybutyrate/Polybutylacrylate.

Blends with a thermosetting polymer as matrix: 1) Unsaturated polyesters/reactive liquid rubbers; 2) Unsaturated polyesters/Thermosetting polyimides 3) Highly cross-linked epoxies/Bisphenol-A-polycarbonate.

Polymeric Membranes were discussed by B. Kunst, Zagreb, Croatia. A membrane, an intervening phase separating two fluid



Medvedgrad Castle

phases, plays a determining role in a transport of matter during membrane separation operations. Because the membranes for both the separation operations and for the physico chemical studies are usually built of polymeric materials, their properties depend greatly on the proper choice of the polymer. Asymmetric membranes are most often prepared by the phase inversion technique. The procedure for the proper addition in the membrane preparation and some new ideas on the formation of a "defect-free" skin, was presented.

Polystyrene-Block-Poly(Methyl Methacrylate) Micelles By Size Exclusion Chromatography/Low Angle Laser Light Scattering was discussed by Z. Grubisic-Gallot, Strasbourg, France. Block copolymers in selective solvents (i.e. thermodynamically a good solvent for one block and at the same time a poor solvent for the other block) form multimolecular associates—called micelles—having a core formed by blocks of poor solubility and a protective shell formed by blocks of good solubility. Micelles are formed via so-called closed association, which is characterized by an equilibrium between micelles, with a narrow molecular-weight and size distribution and a molecularly dissolved copolymer—unimer. Size exclusion chromatography (SEC) is known to be a powerful method of characterizing polymer molecules in solution based on the separation of macromolecules according to the hydrodynamic volume.

An example of SEC results obtained for micellar system poly(styrene-*b*-methyl methacrylate) diblock copolymer in the mixed solvent system 1,4-dioxane/cyclohexane was reported. Good separation of peaks of micelles achieved for this system enabled a direct molecular-weight characterization of micelles by a low angle laser scattering detector. Experiments with changing flow rate and concentration of injected sample solutions showed moderately fast unimer-micelles re-equilibration in the course of separation.

The Mechanism of Singlet Photoreactions was presented by M. Klessinger, Munster, Germany. The reaction pathway of a nonadiabatic photochemical reaction begins on the excited-state potential energy surface at the Franck-Condon region or on a spectroscopic minimum of the reactant. It finishes at the product minimum on the ground-state surface. Conical intersections are a common feature in most nonadiabatic singlet photoreactions. Geometries at which conical intersections are likely to occur can now be predicted by means of simple models.

HPLC, NMR and Chiroptics in Stereochemistry: Axial Chirality of Hindered Amides was the subject of the lecture by A. Mannschreck, Regensburg, Germany. The following physical methods, essential for organic stereochemistry, particularly enantioselective ¹H NMR, enantioselective HPLC, circular dichroism and polarimetry techniques were used to study sterically hindered amides. The demonstration of their axial chirality, the analytical and semipreparative separation of their enantiomers and/or diastereomers, the determination of their relative configuration and the measurement of their barriers to stereoisomerizations was discussed. The latter was found to occur by the following processes: rotation about the aryl-N and aryl-C bonds in dialkylaminobenzoic and dialkyl-naphthoic acid amides and the rotation about the =C-C= and =C-N bonds in benzoic, naphthoic and acrylic thioamides. Acrylic thioamides represent a novel class of axially chiral compounds.

Recent Progress In the Investigation of Carbocations was

discussed by Hans-Ullrich Siehl, Tübingen, Germany. He pointed out that the award of the 1994 Nobel Prize in Chemistry to George Olah underscores the importance of carbocation chemistry. The latest progress in this field can be characterized by the fruitful interplay of advanced experimental methods and quantum chemical methods for the characterization.

This had been particularly for disubstituted carbocations (vinyl cations) and silyl substituted carbocations which were up-to-now not accessible in solution. Using special experimental techniques to avoid cationic polymerization the authors successfully protonated suitably substituted alkynes and allenes.

Finally, an interesting work was presented by M. Eckert-Maksic, Zagreb, Croatia, entitled **Electronic Structure and Reactivity of Fused 7-Oxanorbornenes**. 7-Oxanorbornenes, linked either via common carbon-carbon aromatic moiety, are of great interest as potential building blocks of highly interesting molecules such as polarofacial spacers and macromolecules with extended π -conjugation. They represent also a great challenge from both spectroscopic and theoretical points of view as they provide a unique framework for exploring transannular orbital interactions.

The **Poster Session** provided a considerable number of interesting scientific developments: A list of the posters is presented: "Synthesis and Characterization of Acryloyloxy Azomethyne Monomer" by N. Koprivanac, A. Metes, S. Papic and Z. Janovic. "Polyhydroxamic Acids—Synthesis and Iron (III) Complexes" by M. Jakopovic, B. Zoric, M. Birus and I. Butula. "Synthesis and Characterization of some Ferrocene Amines and Ferrocene Oligo-Amides" by Z. Zoric, V. Rapic and S. Lisac. "Conversion of (1,1'-Ferrocenylene) Bis(Mercapto-Aliphatic Acids) to Dimeric and Oligomeric Products" by S. Lisac and V. Rapic. "Copolymerization and Copolymers of Vinyl Acetate with Some Brominated Phenylmaleimides" by Z. Janovic, M. Podkrcanski, Lj. Tomasek, F. Ranogajec and K. Saric. "Copolymerization and Copolymers of Tribromostyrene with Styrene or Acrylonitrile" by Z. Janovic, Lj. Tomasek and T. Malavasic. "Emulsion Polymerization of Styrene Acrylonitrile and Methyl Methacrylate Mixture in the Presence of a Polybutadiene Latex" by M. Kopic. "In-Source Monitoring of Radiation Curing of Unsaturated Polyester Resins by DC-Electrical Conductivity Measurement" by I. Pucic and F. Ranogajec. "Influence of Electrical Field and Dose Rate on



Poster Session



Poster Session

Radiation Curing of Unsaturated Polyester Resins" by I. Pucic and F. Ranogajec. "The Influence of Accelerators of Sulphur Vulcanization on the Structure of Crosslinking" by Lj. Vrban, J. Travas-Sejdic, J. Jelencic and M. Bravar. "Reaction Mechanisms in the Electrochemical Synthesis of Polyaniline" by Lj. Dacic, S. Kovac and Z. Mandic. "Novel Polysiloxanes: Synthesis and Properties" by A. Vlahov, J. M. Maud, A. R. Hepburn, J. M. Marshall and D. M. Goldie. "Studies on Diluted Solutions of Poly(Styrene-co-Acrylonitrile)" by M. Rogosic, Z. Jurasinovic and H. J. Mencer. "Solution Properties of Partially Sulfonated Alternating Copolymers of Maleic Acid and Styrene in Polar Solvent" by N. Segudovic, S. Sertic, M. Kovac-Filipovic and V. Jarm. "Application of the Group-Contribution Methods to Polymer Solution Thermodynamics. Vapor-Liquid Equilibria" by G. Bogdanic and A. Fredenslund. "Polymer Microstructure of Cyclo-Pentene/Ethene Copolymers with 1D and 2D NMR" by A. Jerschow and N. Muller. "Local Order in Lamellar Structure of Block Copolymers: a Deuterium NMR Study" by S. Valic, B. Deloche, Y. Gallot and A. Skoulios. "Miscibility in the Copolymer Blends of Sulphonylated Poly(2,6-Dimethyl-1,4-Phenylene Oxide) and Poly(O(P)-Fluorostyrene-co-O(P)-Chlorostyrene) Copolymers" by R. Vukovic, G. Bogdanic, A. Erceg, N. Segudovic and D. Fles. "Study of Phase Separation in Styrene-Butadiene Diblock Copolymers by ESR Method" by V. Bozicevic, B. Rakvin, Z. Vekšli and Y. Gallot. "Study of Molecular Motion and Phase Separation in Styrene-Acrylonitrile Copolymers by ESR Method" by M. Andreis, Z. Vekšli, M. Rogosic, and H. J. Mencer. "X-Ray Investigations of Bicomponent Injection Molded Polypropylene" by J. Travas Sejdic, P. Zipper and F. Eigl. "WAXS Structural Analysis of Polypropylene Fibers Spun from Two Different Polymer Grades" by D. Gregor Svetec, P. Zipper and A. Janosi. "Parameters of Supramolecular Structure of Polyester Fibers" by M. Ziberna Sujica and M. Sfiligoj Smole. "Supercritical Fluids and Fiber Structure" by M. Sfiligoj Smole, P. Zipper, S. Jeler and Z. Knez. "Effects of PP on Rheology of Dynamically Crosslinked Alloys PP/EPDM" by T. Marinovic, M. Sustar and A. Pertot. "Mechanical Properties and Morphology of PP/SAN/SEBS Blends" by G. Radonjic and V. Musil. "Matrix Morphology and Mechanical Properties of Crosslinked Natural Rubber" by S. Valic, M. Andreis, T. Marinovic, F. Ranogajec and Z. Vekšli. "Investigation of Thermal Properties of Blends Based on Polyurethanes" by M. Ulcnik, B. Zerjal and T. Malavasic. "Polyurethane Flexible Foams on the Base of

Conference Report



At the Banquet

Polymer Polyols" by J. Sadadinovic, V. Rek, and K.C. Frisch. "Isothermal Degradation of Poly(Vinyl Chloride)/Methyl Methacrylate-Butadiene-Styrene Blends" by B. Baric and T. Kovacic. "Study of Thermochemical Degradation System of Copolymer/Butadiene-g-Styrene" by D. Paje-Liplin, D. Haco and V. Kovacevic. "Investigation of Thermooxidative Degradation of PVC/ABS Blends by Infrared Spectroscopy" by I. Klaric and U. Roje. "The Effect of Molecular Weight of Polyols on Photostability in Polyurethane Elastomers" by E. Govorcin-Bajsic, V. Rek, A. Sendjarevic, V. Sendjarevic and K. C. Frisch. "Effect of Molar Volume of Solvent and Vulcanizate Structure on Interactions in the System Solvent-Crosslinked Polymer" by Z. Hrnjak-Murgic, J. Jelencic, M. Bravar and M. Marovic. "Chemorheology of Unsaturated Polyester Resin" by M. Opalicki, M. M. Kenny and L. Nicolais. "Rheological Behaviour Comparison of PVC Plastisols on the Basis of Emulsion and Microsuspension PVC" by Z. Spehar and V. Rek. "Adsorption of Polyacrylic Acids on the Hydrophobic Surface" by M. Plavsic and B. Cosovic. "Water Absorption Effects of PA 6 on DSC Measurements" by D. Spanicek and Z. Smolic Zerdik. "Standardization in the Fields of Plastics and Rubber in Croatia" by I. Catic and D. Spanicek. "Annealing Process in Segmented Polyurethane" by V. Rek and E. Govorcin-Bajsic. "The Effect of Polymers in Formulations of Engine Lubricant" by M. Orsic. "Paraffin Wax-Polymer Blends"

by J. Jakopovic. "Polymer Materials in Shipbuilding" by Z. Hell and Z. Mrlkic. "Weathering of Polypropylene (PP) Fibers During Usage" by E. Pezelj, R. Cunko and M. Andrassy. "The Influence of CaCO₃-Filler on the Properties of Poly(Vinyl Acetate) Based Polymers" by S. Lucic and V. Kovacevic. "The Polymer Painting Ground" by S. Horvat-Kurbegovic and D. Turkalj. "Enzymatic Starch Hydrolysis Kinetics in Nutrition Media for Oxytertracycline Biosynthesis" by K. Mihaljevic, I. Markovic, A. Stroj, and M. Halusek.

On **Monday evening**, February 6th, a very nice reception was given which allowed the people to become acquainted. It gave the participants, especially the foreign visitors the opportunity to meet their Croatian colleagues and some of the many young scientists and students who participated in the Symposium. A banquet was held on Tuesday evening for the invited speakers and a number of the key organizers and scientists of Croatia.

Zagreb as a City presented itself at its best with sunshine during the entire meeting period. This allowed some of the participants also to see the beautiful sights of this most interesting city, the cultural center of this part of Europe.

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We would like to thank Meifang Qin for her assistance during the preparation of this manuscripts.