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Practical Applications of Cyclopentadienyl Complexes of Transition Metals

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The review is devoted to the main practical applications of metallocenes (dicyclopentadienylmetal complexes); the main practical applications of ferrocene and its derivatives are dealt with most widely. The practical utilisation of other cyclopentadienylmetal complexes is only beginning and the number of studies in this field is small. Investigations published in 1977 are considered.
The bibliography includes 162 references.

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I. INTRODUCTION

The practical applications of dicyclopentadienylmetal complexes (metallocenes) have become possible owing to the successful development of synthetic research in this field. Ferrocene, which was discovered little more than 20 years ago, led to the foundation of the chemistry of metallocenes. Despite the fact that the cyclopentadienyl complexes of almost all the metals and many non-metals are known at present, the chemistry of the derivatives of metallocenes has so far been investigated most widely only for ferrocene[1,2]; that of osmocene and ruthenocene began to develop only comparatively recently and this is also true of cobaltocenium, nickelocenium, vanadocenium, etc. salts[2,3]. Derivatives of the dicyclopentadienyl complexes of titanium, zirconium, and to a lesser extent hafnium have been investigated in greater detail[4]. Among monocyclopentadienyl compounds, cyclopentadienylmanganese tricarbonyl (CMT) and cyclopentadienylrhenium tricarbonyl (CRT) have been most thoroughly investigated chemically[4].

The development of synthetic research on mono- and di-cyclopentadienyl derivatives of metals has served as the foundation on which the development of applied research in this field has been based. In the present review, which is devoted to the practical applications of metallocenes, attention is concentrated on the practical utilisation of ferrocene and its derivatives and studies associated with the cyclopentadienyl derivatives of other metals are only touched upon. The review constitutes a continuation of an earlier review[5], which was devoted to the practical applications of ferrocene and its derivatives.

A characteristic feature of the applications of metallocenes, which has become apparent recently, is the possibility of using them in various seemingly opposed fields; for example, ferrocene is used both as an inhibitor of the ageing of polymers and as a catalyst for their self-decomposition; or, for example, partly oxidised ferrocene derivatives are extremely effective semiconductors, while ferrocene serves at the same time as a successful additive in the manufacture of electrical insulators. The conditions used in the employment of metallocenes, their concentration, solvents, conditions of treatment, etc. apparently play a major role; for this reason, the application of such metal complexes requires an extremely careful adherence to the conditions developed for their utilisation. Wide scale studies on the properties of metallocenes involving all chemical and physicochemical methods are necessary; we may note that so far there have been virtually no quantitative investigations.

Studies on the mechanisms of the stabilisation of thermostable polymers under the influence of metal-containing inhibitors are only briefly mentioned in the review.

II. THERMO- AND PHOTO-STABILISERS

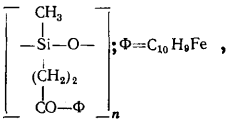
Effective photostabilisers for polymers, particularly *o*-hydroxybenzoylferrocene (I)[6,7], have been discovered comparatively recently and the possibility of coating spaceships with this compound has been mentioned[8]. The inhibiting effect of the ferrocenyl residue is apparently associated with the ability of compounds of this type to absorb light in both the visible and ultraviolet regions[9]. One cannot rule out the possibility that the effect is also enhanced by the aromatic component of the molecule, since benzoylbenzene derivatives are well known photostabilisers[10]. Other compounds containing the ferrocenyl fragment in the molecule, for example, phenylene substituted in the 4-position, can also serve as thermo- and photostabilisers[11]. Inhibitors of the light-induced ageing of polypropylene have been described[12].

The products of the interaction of diacetylferrocene with formaldehyde, which the authors called "diacetylferrocene-formaldehyde resins", have also been proposed for the modification of textile fibres based on poly(vinyl alcohol), for the improvement of their mechanical properties in spinning, and for the improvement of their resistance to the action of light[13]. The dimethyl ester of ferrocenedicarboxylic acid can likewise serve as a photostabiliser[14]. A decrease in mechanical fatigue strength in the spinning of fibre may be achieved by introducing into acetylcellulose 0.5–1 wt.% of formylferrocene[15], which also imparts resistance to radiation and increases the adhesion of polyamides to rubber[16,17].

Ferrocene has been proposed as a thermostabiliser for polyethylene: tests on shields 2 mm thick (involving heating to 100°C for three days) showed that the tensile

strength decreases to 82.2% compared with 69.3% for the control specimen[18]. The addition of small amounts of ferrocene (0.5 wt.%) has a similar effect on ethylene–propene and diene rubbers[19]. A polyethylene film is stabilised in relation to ultraviolet radiation by the addition of 10% of the benzaldehyde–ferrocene copolymer in methylene chloride[20].

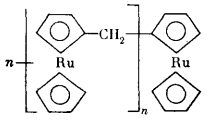
Polyferrocenylsiloxanes of the type



where the side chain contains the same ferrocenyl residue, have been described[5] as absorbers of ultraviolet light and photostabilisers.

Among other ferrocene derivatives proposed as thermostabilisers and antioxidants, *p*-aminoarylferrocenes have been mentioned[21]. Organosilicon derivatives of ferrocene in which the silicon atom is linked directly to the ferrocene ring constitute an interesting group of antioxidant stabilisers for polymers: $C_{10}H_4Fe[Si(CH_3)_3]_2$, $C_{10}H_8Fe[Si.(CH_3)_2OC_2H_5]_2$, $C_{10}H_8Fe[Si(CH_3)_2.OC_8H_{17}]_2$, $C_{10}H_8Fe[Si.(CH_3)_2OC_6H_5]_2$, and $C_{10}H_8Fe[Si(CH_3)_2N(C_2H_5)_2]_2$. In this series the most effective compounds are the diethylaminosilyl derivatives[22], which can be used as antioxidants for heat exchangers in refrigerators[23].

Polymers based on ruthenocene



proved to be resistant to heat and radiation[24]. They have been proposed as special protective coatings.

Two general ways of inhibiting polymer decomposition processes were examined in a recently published review[25]: (1) inhibition of chain processes by introducing into the system substances causing chain termination; (2) the removal from the system of chemical agents (present in the polymer composition or formed as a result of ageing), weak bonds, etc. It is shown in the same review that the deactivation of active centres under the influence of metal-containing inhibitors takes place as a non-chain inhibition process. The $M-C_5H_5$ bond dissociation energy probably constitutes a measure of the thermal stability of the compounds. Thermal decomposition can result in the formation of either pure finely dispersed metals or readily oxidisable products, which are involved in the chain termination reactions in the course of thermochemical degradation.

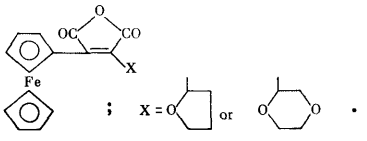
III. DYES AND PHOTOGRAPHIC MATERIALS

Few data on ferrocene dyes have appeared in the literature of recent years: this can be explained primarily by their low stability to light; early studies in this field have already been discussed[5].

Data have been published recently concerning the possibility of using organometallic compounds in photographic and particularly electrophotographic processes. Thus light-sensitive compositions containing organoelemental compounds, particularly cyclopentadienylmetal complexes, have been proposed[26]. An electrophotographic material is obtained by pouring compositions containing photoconductors, polyhalogeno-substituted compounds (for the formation of free radicals under the influence of light), the base form of the dye (for the formation of a sensitising dye under the influence of free radicals) and organoelemental additives onto conducting polyvinyl supports. After preliminary activation of these compositions by light or after pouring the same compositions onto polyvinyl supports and subsequent irradiation of the entire conducting polyvinyl layer with light, a positive image is formed. The material is then charged by a corona discharge source, irradiated with light from a tungsten lamp, and developed using a positive liquid developer, which yields the positive image. Tetraethyl-lead is used as an organometallic additive; cyclopentadienylmetal complexes have been mentioned as possible additives.

Photographic materials based on vinylferrocene, organic halides, and aromatic amines or their derivatives have been recently proposed[27] as photographic materials. A negative image has been obtained on paper following the illumination with light from a mercury lamp of a light-sensitive composition consisting of the styrene–vinylferrocene copolymer, tribromomethane, *p*-nitrophenyl sulphone, and diphenylamine.

Compounds containing the products of the condensation of maleic anhydride with tetrahydrofuran (THF), or 1,4-dioxan have been proposed as antidetonating agents and as inks soluble in organic solvents[28]:



IV. ADDITIVES TO FUELS AND OILS

Derivatives of ferrocene and cyclopentadienylmanganese tricarbonyl serve as regulators of various oxidation processes occurring in the fuel. They can be used as antismoke and anticoking agents, antidetonating agents, and regulators of the rate of combustion, can promote more complete oxidation of the fuel and decrease the amount of harmful exhaust gases resulting from combustion, and can even inhibit combustion processes, imparting fire resistance to the relevant substances. In view of the urgency of all the problems enumerated, the large scale manufacture of fuels of various types, and the importance of the problem of the protection of the environment, the possibility of using metallocenes in this field is of considerable practical interest.

1. Antismoke Additives

Ferrocene, diethylferrocene, tris(acetylacetonato)-iron[29], CMT, and polymers based on vinyl-CMT and methyl-CMT have been proposed as antismoke additives to motor fuel[30].

The successful use of organometallic antismoke additives is possible only in those cases where small amounts of the additive to the fuel (0.05–0.1 wt.%) can ensure a very effective increase of the degree of combustion of the fuel and a decrease of the amount of smoke. An increase in the amount of the antismoke additive can lead to other

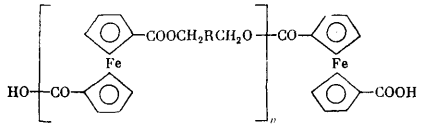
undesirable phenomena–the formation of a deposit on the working components of the engine, etc.

2. Antidetonating Agents

CMT† and ferrocene derivatives are effective antidetonating agents. However, fairly extensive deposit formation accompanying the use of ferrocene derivatives has so far precluded the practical employment of these compounds. Tests on the antidetonating properties of compounds of this class have shown[31,32] that ferrocenylmethyltrimethylammonium iodide, diferrocenylpropenone, and ferrocenylfluorophenylpropenone are most effective. Compounds containing a halogen and nitrogen form the smallest amount of deposit but it was not discovered whether nitrogen oxides are formed in the exhaust gases.

3. Regulators of the Rate of Combustion

Among new studies in this field, mention should be made of polymeric ferrocene-containing substances, for example, the products of the polycondensation of the heteroannular ferrocenedicarboxylic acid and diglycols (IX)[33,34], i.e.



(I)

the copolymers of isopropylferrocene or 1-divinylferrocene with butadiene, isoprene, and chloroprene[35], and the copolymers of butylferrocene with formaldehyde[36] and of vinylferrocene with butadiene[37] or chloroprene[38,39].

An involatile combustion catalyst, obtained by condensing t-butylferrocene with methylal[40] and added to a carboxylate butyl-rubber, the vulcanising agent, ammonium perchlorate, and dioctyl adipate, ensures an increase of the rate of combustion by a factor of 2–2.5.

Intensive research on the elimination of exhaust gases from air is proceeding in various countries. Thus it has been suggested that ferrocene be used as an additive to fuel (0.05 wt.%), which promotes the further combustion of CO to CO_2.[41] Compositions which yield less harmful substances on combustion have been obtained from poly(vinyl chloride), an aliphatic plasticiser, and $CaCO_3$ with small amounts of added ferrocene as well as lead stearate and sulphate.

4. Oxidation Inhibitors

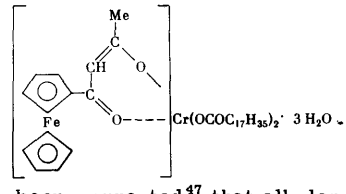
Ferrocene derivatives can serve not only as catalysts but also as inhibitors of oxidation. Thus fire-resistant compositions in which ferrocene and its derivatives are combined with halogen-containing polymeric materials have been proposed in a number of studies[42-44]. For example, fire-resistant shields (2 mm thick) are made from a composition containing polychlorovinyl-bis-(2-ethylhexyl) phthalate, lead sulphate and stearate, ferrocene, and $MgCO_3$. Such shields are also made from ferrocene-containing copolymers with anisyl aldehyde and *pp'*-benzoquinone dioxime combined with butyl-rubber and zinc oxide. Similar fire-resistant compositions, based on polyethylene with added ferrocene, have been proposed[42]. It may be that, oxidation inhibitors are formed from ferrocene and its derivatives on heat treatment in the presence of the components of the given fire-resistant compositions.

5. Accelerators of the Decomposition of Polymers

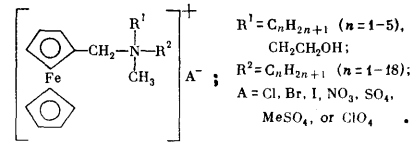
The problem of the contamination of the environment by waste products from the synthesis of polymers, which are difficult to decompose, is now becoming increasingly acute. Ferrocene and its derivatives proved not only to inhibit the ageing of polymers but also to catalyse their decomposition[44].

6. Antistatic Additives to Fuel

The transport of fuel over long distances and its storage for a fairly long time constitute an urgent problem. The necessity to find antistatic additives to fuel arises in this connection. Among ferrocene and CMT derivatives, salt-like compounds containing a chromium atom in addition to iron or manganese atoms have been proposed[45,46]:



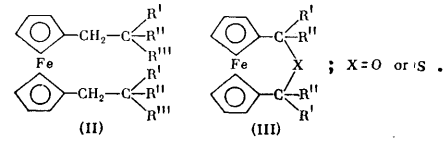
It has recently been suggested[47] that alkylammonium salts of the dialkylaminomethylferrocene type be used as antistatic additives[47]:



V. MEDICINAL SUBSTANCES

Ferrocene compounds proved to be effective agents in the fight against diseases caused by iron deficiency in the organism, i.e. against various types of anaemia.

Dialkylferrocenes (II)[48-51] and oxygen-containing[52,53] and sulphur-containing[54,55] ferrocenophanes of type (III) exhibit antianaemic activity:



(II) (III)

The oxygen-containing compounds also exhibit a blood-liquefying activity.

The sodium salt of *o*-carboxybenzoylferrocene (ferrocerone)[56-60] is the first ferrocene-containing medicinal preparation whose widescale use in medical practice is permitted. Its characteristic features are non-toxicity,

†This problem is discussed in detail in a monograph[30].

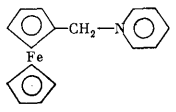
ready solubility in water, and rapid assimilation by the organism. Its application does not require the use of acids and it is therefore particularly suitable for administration to patients suffering from diseases of the gastrointestinal system as well as children. It can be used in the treatment of various forms of anaemia and a severe disease of the nasopharynx—ozena, which hitherto has been virtually incurable.

Substituted ferrocenylcyclopentenes have been proposed for the treatment of iron deficiency anaemia[61]:

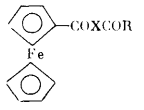

; R=Ph, $PhCH_2$, n- ClC_6H_4CO, or m- $CH_3C_6H_5CO$.

A complex medicine consisting of (1,1-diethyl-$\alpha\alpha$-thiobiscyclopentadienyl)iron and iron-dextran has been suggested recently[64]. It was found that the iron in this preparation, like that in ferrocerone[56-60], is absorbed in the liver.

Piperidinomethylferrocene exhibits a physiological activity[67]:

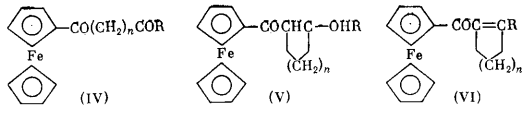


Ferrocene derivatives of the type



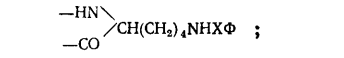
have been suggested for the treatment of disorders caused by iron deficiency in the organism. Here X is a saturated C_2–C_{10} hydrocarbon group and R is an alkyl group, a thiophen, furan, or benzene ring, or a benzene ring substituted by one or several halogen atoms or alkynyl groups.

Monoketones (IV), diketones (V), and hydroxyketones (VI) of the type[63]


(IV) (V) (VI)

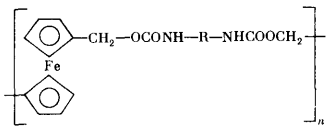
have also been suggested[63].

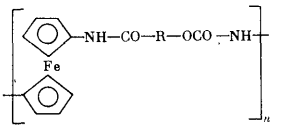
Molecules of the type


; X=CH_2, CS—NH—CH_2, or CO $(CH_2)_2$.

based on ferrocenylacetic and ω-ferrocenylcaproic acids, have been obtained by grafting to the lysine residues in proteins[66]. Modified proteins of this type can be used in fluorescence microscopy; owing to their enhanced electron density, they are visible under the electron microscope.

Polymers obtained on the basis of ferrocenylenediacetic acid and diaminoferrocene can be used to study gas diffusion through membranes[67]:

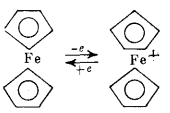

; R = $(CH_2)_4$, or $(CH_2)_6$


; R = $(CH_2)_2$, $(CH_2)_4$, or $(CH_2)_6$.

They can also be used to study the following micro-organisms: *Candida pseudotropical*, *Mycobacteria BCG*, and *Mycobacterium foctuitum*. It has been noted[69] that compounds in which there is a possibility of ready elimination of a hydrogen halide showed the greatest bactericidal efficiency; there is a possibility of the formation of a double bond in the position conjugated to the CO group. It is striking that 3-chloropropionyl and 4-chlorobutyrylferrocenes are altogether ineffective.

VI. ION-EXCHANGE RESINS, REDOX SYSTEMS, SEMICONDUCTORS, AND INSULATORS

The use of ferrocene and its derivatives in various redox systems



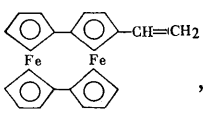
is based on their ability to undergo reversible oxidation to ferrocenium salts.

Electron-exchange resins based on ferrocene were first obtained by Nesmeyanov and coworkers[70,71]. A ferrocene electrode, ferrocene-containing polymers (redoxites), and liquid redox systems based on alkylferrocenes have been proposed[72-77]. Styrylferrocene–styrene copolymers exhibit electron-exchange properties[78]. Ion-exchange resins have been synthesised by grafting ferrocenyl residues (in the form of the amide of ferrocenedicarboxylic acid or formylferrocene) onto a fibre based on poly(vinyl alcohol)[81]. A biological redox system based on the reversible ferrocene–ferrocenium reaction at pH 7 has been used to convert cytochrome *c* into cytochrome *c* oxidase[80] and conversely.

Anion-exchange resins of a new type, based on cobaltocenium salts, have been suggested[81].

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It has been shown that carefully purified polymers based on ferrocene do not exhibit paramagnetic properties[1,82,84]. Studies on partly oxidised ferrocene-containing polymers, which exhibit semiconducting properties, for example, polymers obtained from vinylferrocene and carbazole, constitute a new field in the chemistry of ferrocene[85]. Partial (to the extent of 60%) oxidation of the polymer obtained from vinylbis(fulvalene)iron



gives rise to a product which has the highest specific magnetic susceptibility among the known organic semiconducting materials[86]. It appears that there is at present a possibility of employing ferrocene and its derivatives as organometallic semiconductors; investigators in a number of countries are carrying out vigorous researches in this field[84,87,88].

Detailed studies on the physicochemical properties of bis(fulvalene)iron and its mono- and di-cations yielded evidence that the monocation is a delocalised resonance-stabilised ion[89,90]—a representative of the so called mixed valence compounds. Thus the role of the ferrocene–ferrocenium system in the generation of semiconducting properties is evident.

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The study of partly oxidised ferrocene derivatives as semiconductors apparently stimulated research on the use of diamagnetic ferrocene compounds as insulators. Thus the addition of ferrocene (or its homologues) as well as polymers based on ketones and aldehydes derived from ferrocene to polyethylene increases the breakdown potential of the resulting dielectrics from 9.8 to 14.0 and 18.6 kV respectively[92]. An even more effective electrical insulator has been obtained from polyethylene, ferrocene, and isoprene GM.[93]

VII. FERROMAGNETIC MATERIALS BASED ON FERROCENE DECOMPOSITION PRODUCTS

On heating to 70–300°C with subsequent slow cooling to 25°C, ferrocene polymers[94] acquire ferromagnetic properties. Powders, solid materials, and thin shields made from these materials can be used in magnetic cores and in teleprinting and tele-reproducing devices and in magnetically shielded apparatus. The applications of organometallic compounds in electronics are described in greater detail in a monograph[95].

VIII. CHEMICAL REACTIONS INVOLVING METALLOCENES

1. Applications in Analytical Chemistry and in Methodological Chemical Research

Ferrocene has recently found increasing applications in analytical chemistry. The use of ferrocene in the potentiometric determination of molybdenum in metals, alloys, and steels[96] and the photometric determination of molybdenum in an aqueous organic medium[97] have been reported. The possibility of employing ferrocene in mixtures of glacial acetic and concentrated hydrochloric acids has been demonstrated in the direct amperometric oxidation-reduction titration of rhenium(VII) in aqueous organic media; many cations and anions do not interfere with the analysis[98].

Ferrocene has not become merely a generally accepted reagent of considerable importance in practice. Very recently it has come to be used as a methodological material for the training of students in procedures for the preparation of pure materials, the use of the infrared spectroscopic method in the determination of structure, in the gas–liquid chromatographic method, polarography, potentiometry, etc.[99]

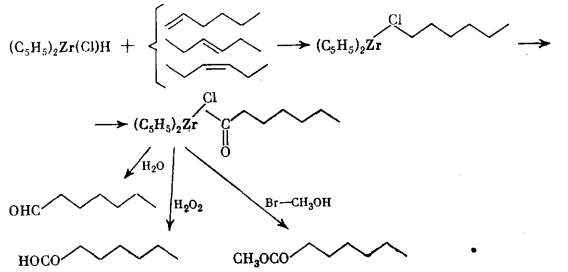
2. Catalysts of Chemical Processes

The oxidation of phenol by hydrogen peroxide in the presence of ferrocene or diacetylferrocene entails the formation of hydroquinone and catechol in proportions of 1 : 2.3 and in an almost quantitative yield[100]. Ferrocene–dichlorobenzene copolymers are capable of dehydrogenating and dehydrating alcohols[101].

When a solution of azulene is irradiated in the presence of sodium acetate and ferrocene in a carbon tetrachloride–ethanol mixture[102], the product is 1-ethoxycarbonylazulene. It has been noted that the irradiation of the same mixture in the absence of ferrocene or, conversely, in the presence of ferrocene derivatives with electron-donating (ethylferrocene) or electron-accepting (acetylferrocene) substituents does not yield satisfactory results.

Nickelocene $(C_5H_5Ni)_2$ catalyses many chemical processes. Thus a complex of the type $(C_5H_5)_2Ni.MX_n$ (where M = Al, Ti, or Nb; X = Cl; n = 3–5) polymerises butadiene with formation of polybutadiene containing *cis*-1,4-units[103,104]. Nickelocene in benzene or cyclohexane solution catalyses the hydrolysis of lignin to phenol[105]. The hydrolysis of lignin has become very important at the present time in connection with the problem of the elimination from the environment of the waste products of the cellulose industry. Reductive complex-formation reactions between phosphine sulphides and nickelocene have been described[106].

The hydrozirconation reactions in the presence of di(ηs-cyclo)pentadienyl(chloro)alkylzirconium hydride are being extensively investigated at the present time[107,108]. Carbon monoxide is inserted in the carbon–zirconium bond, olefins undergo oxidative hydrogenation, and, depending on the mode of the subsequent decomposition of the organozirconium compound, aldehydes, acids, or carboxylate esters may be obtained.



Cyclopentadienylzirconium(hafnium) dicarbonyls, $C_5H_5Zr(CO)_2$ and $C_5H_5Hf(CO)_2$, have been synthesised for the first time and tested as diphenylacetylene hydrogenation catalysts; their catalytic activity has been demonstrated. However, comparative analysis of the effect of equimolar amounts of these catalysts and cyclopentadienyltitanium dicarbonyl in the hydrogenation of phenylacetylene by hydrogen under a pressure of 3 atm showed that the latter is more effective[109].

Among organic derivatives of Group IV metals, mention should be made of dicyclopentadienyltitanium dichloride, which is widely used as a Ziegler–Natta catalyst in the polymerisation of ethylene and other mono- and poly-olefins[110,111]. Dicyclopentadienyltitanium dichloride in a 1 : 1 complex with triethylaluminium is also a hydrogenation catalyst: for example, it is used to convert oct-1-ene into n-octane[112]. The same complexes may be used as igniting agents for pyrotechnic compositions and as sources of free radicals[110].

The ability of dicyclopentadienyltitanium compounds to be reduced to compounds containing titanium in the lowest valence state, involving the intermediate formation of complexes with alkylaluminium halides, which ultimately leads to the appearance of free radicals, is believed[111] to be the main cause of the processes underlying the most important

technological methods for the polymerisation of ethylene and other olefins.

Dicyclopentadienyltitanium dichloride also catalyses the fixation of molecular nitrogen[112-115]. Various systems are used in this process (for example $(\pi\text{-}C_5H_4)_2TiCl_2$, EtMgBr, $(\pi\text{-}C_5H_5)_2TiCl_2$, n-BuLi, etc.)

Vanadocene compounds proved to be active catalysts for the polymerisation of ethylene at room temperature[116]. The $(C_5H_5)_2VCl_2-CH_3AlCl_2$ and $C_5H_5VOCl_2-CH_3AlCl_2$ systems have been tested. In the first case polymerisation begins solely in the presence of oxygen as the initiator of chain transfer and termination. It has been suggested[116] that complexes of alkylated vanadium in the highest valence state are catalytically active.

An interesting report has been published recently concerning the use of cyclopentadienylcopper in the synthesis of acrylonitrile, methyl methacrylate, etc. polymers. The complex $C_5H_5Cu \leftarrow C{\equiv}N\text{-}t\text{-}Bu$ is particularly effective in the polymerisation of acrylonitrile. The catalytic effect is appreciably enhanced in the presence of an excess (1 : 1) of t-BuCN and other ligands[117,118].

The use of cyclopentadienylmetal complexes in various chemical reactions constitutes a new rapidly developing field of homogeneous catalysis, which in the future may help us approach an understanding of the stereospecific processes occurring in the living organism at enormous rates at atmospheric pressure and at room or similar temperatures. The future applications of metallocenes will probably be found in this field, at the boundary between organic, organoelemental, and biological chemistry.

During the preparation of the manuscript for the press, a number of new reports were published on thermo- and photo-stabilisers[119-124], on photographic materials[125-131], on additives to fuels and oils[132-140], on medicinal substances[141-147], on semiconductors and insulators[148-154], on ferromagnetic materials[155], on applications in analytical chemistry[156-163], and on catalysts of chemical processes[164,165].

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