**Keywords:** coordination polymers · crystal engineering · network structures · nonlinear optics · solid-state structures

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[10] X-ray single crystal diffraction data for both **1** and **2** were collected on a Siemens SMART CCD diffractometer. Crystal data for **1**: crystal size $0.16 \times 0.22 \times 0.34$ mm, orthorhombic, space group $P2_12_12_1$, $a = 8.2149(1)$, $b = 11.6635(2)$, $c = 12.7420(1)$ Å, $U = 1220.9(1)$, $Z = 4$, $\rho_{calcd} = 1.68\ g\,cm^{-3}$, $\rho_{obs} = 1.65(1)\ g\,cm^{-3}$, $T = 123$ K, $Mo_{K\alpha}$ radiation ($\lambda = 0.71073$ Å). Least-squares refinement based on 2755 reflections with $I > 3\sigma(I)$ and 173 parameters led to convergence, with a final value of $R = 0.026$ and $R_w = 0.031$. Crystal data for **2**: crystal size $0.14 \times 0.40 \times 0.40$ mm, monoclinic, space group $Cc$, $a = 12.0137(2)$, $b = 22.3435(4)$, $c = 7.7738(1)$ Å, $\beta = 123.509(1)^\circ$, $U = 1739.9(1)$ Å$^3$, $Z = 4$, $\rho_{calcd} = 1.63\ g\,cm^{-3}$, $\rho_{obs} = 1.63(1)\ g\,cm^{-3}$, $T = 198$ K, $Mo_{K\alpha}$ radiation ($\lambda = 0.71073$ Å). Least-squares refinement based on 2034 reflections with $I > 3\sigma(I)$ and 226 parameters led to convergence, with a final value of $R = 0.039$ and $R_w = 0.043$. – Further details of the crystal structure investigation(s) can be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany (fax: (+49) 7247-808-666; e-mail: crysdata@fiz-karlsruhe.de), on quoting the depository numbers CSD-391070 and CSD-391071.

[11] Interestingly, when attempts were made to synthesize the $Cd^{II}$ analogue of **1**, a twofold diamondoid structure with a formula [Cd(isonicotinate)$_2$(EtOH)][EtOH] was obtained. The formation of twofold (but not threefold) diamondoid structure is presumably a consequence of the larger size of $Cd^{II}$ versus $Zn^{II}$. [Cd(isonicotinate)$_2$(EtOH)][EtOH] adopts a centrosymmetric structure (space group *Pbca*, SHG-inactive) due to the twofold interpenetration: W. Lin, R. Xiong, O. Evans, Z. Wang, unpublished results.

[12] Identical X-ray powder diffraction patterns were obtained for samples of **2** before and after the removal of water guest molecules.

# Highly Efficient Ruthenium-Based Catalytic Systems for the Controlled Free-Radical Polymerization of Vinyl Monomers**

François Simal, Albert Demonceau,* and Alfred F. Noels

The ability to control molecular architecture constitutes a major challenge for synthetic polymer chemists.[1] Controlled free-radical polymerization (also referred to as "living" or "pseudoliving") has in recent years revitalized the rather mature field of radical olefin polymerization in an unprecedented way, and has provided access to well-defined polymers and copolymers. Stable free radicals, such as nitroxides, have been introduced for control of radical polymerization.[1] Recently, the groups of Matyjaszewski, Sawamoto, Jérôme, and others have replaced the stable nitroxide free radical with transition metal species to obtain inter alia a variety of copper-,[2] iron-,[3] nickel-,[4] palladium-,[5] or rhodium-mediated[6] controlled free-radical polymerization systems, a methodology which goes by the name of atom transfer radical polymerization (ATRP).

Ruthenium was introduced by Sawamoto et al. for the polymerization reaction,[7] but $[RuCl_2(PPh_3)_3]$ (the most widely used ruthenium complex) requires the presence of a Lewis acid activator. We now report on the exceptional efficacy of new catalytic systems based on well-defined and fully characterized $[RuCl_2(p\text{-cymene})(PR_3)]$ complexes (*p*-cymene = 4-isopropyltoluene) for promotion of the controlled free-radical polymerization of vinyl monomers *without* cocatalyst activation. These readily prepared and air-stable catalysts compare favorably with the most active ATRP catalysts reported to date.

Methyl methacrylate (MMA) was chosen as a model substrate, and polymerization was initiated by ethyl 2-bromo-2-methylpropionate in the presence of various $[RuCl_2(p\text{-cymene})(PR_3)]$ complexes at 85 °C. From the results summarized in Table 1, it appears that only phosphanes which are both strongly basic (the p$K_a$ being taken as a reasonable measure of the σ-donating ability of the ligand) and which possess a well-defined steric bulk ($160^\circ < \theta < 170^\circ$, $\theta$ = cone angle of the phosphane) present both high catalytic activity and high control of the polymerization process (high initiation efficiency *f*, and molecular weight distribution $M_w/M_n = 1.1$). A polydispersity as narrow as $M_w/M_n = 1.07$ is observed when the catalyst is prepared in situ from the ruthenium dimer $[\{RuCl_2(p\text{-cymene})\}_2]$ and tricyclohexylphosphane in the ratio $Ru:PCy_3 = 2:1$.

Under these experimental conditions, all the criteria of living polymerization are fulfilled. Indeed, the plots of

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Table 1. Ruthenium-catalyzed controlled atom transfer radical polymerization of methyl methacrylate.[a]

| Catalyst | $\theta$ [°] | p$K_a$ | $T_D$ [°C][b] | Yield [%] | $M_n$[c] | $M_w/M_n$ | $f$[d] |
|---|---|---|---|---|---|---|---|
| $[RuCl_2(p\text{-cymene})(PR_3)]$ | | | | | | | |
| $PR_3 = P(OPh)_3$ | 128 | −2.0 | 195 | 0 | – | – | – |
| $PR_3 = PMe_3$ | 118 | 8.65 | 216 | 26 | 157000 | 1.75 | 0.07 |
| $PR_3 = P(n\text{Bu})_3$ | 132 | 8.43 | 198 | 44 | 36000 | 1.6 | 0.5 |
| $PR_3 = PBn_3$ | 165 | 6.0 | 223 | 30 | 21000 | 1.6 | 0.55 |
| $PR_3 = PPh_3$ | 145 | 2.73 | 213 | 20 | 25000 | 1.6 | 0.3 |
| $PR_3 = PPh_2Cy$ | 153 | 5.05 | 211 | 58 | 41000 | 1.25 | 0.55 |
| $PR_3 = PPhCy_2$ | 161 | 7.38 | 189 | 90 | 60500 | 1.10 | 0.6 |
| $PR_3 = PCy_3$ | 170 | 9.7 | 163 | 100 | 41500 | 1.12 | 0.95 |
| $PR_3 = Pi\text{Pr}_3$ | 160 | 9.0 | 172 | 80 | 40500 | 1.10 | 0.8 |
| $[\{RuCl_2(p\text{-cymene})\}_2] + PCy_3$ (1:1) | – | – | – | 98 | 51500 | 1.07 | 0.75 |
| $[RuCl_2(=CHPh)(PCy_3)_2]$ | – | – | – | 95 | 66000 | 1.28 | 0.6 |

[a] $[MMA]_0:[initiator]_0:[Ru]_0 = 800:2:1$ (for details, see the Experimental Section). [b] Temperature at which the arene ligand is liberated as determined by TGA. [c] Determined by size-exclusion chromatography (SEC) with PMMA calibration. [d] Initiation efficiency $f = M_{n,theor.}/M_{n,exp.}$ with $M_{n,theor.} = ([MMA]_0/[initiator]_0) \times M_w(MMA) \times$ conversion. $\theta$ is the cone angle of the phosphane ligand;[16] TGA = thermogravimetric analysis, $p$-cymene = 4-isopropyltoluene, Bn = benzyl, Cy = cyclohexyl.

$\ln([M]_0/[M])$ versus time (Figure 1) and of $M_n$ versus monomer conversion (Figure 2) are linear. The linear time dependence of $\ln([M]_0/[M])$ indicates that the concentration of the active species remains constant during polymerization. The lack of transfer reactions is supported by the linearity of the plot of $M_n$ versus conversion. Furthermore, control of MMA polymerization was confirmed by the addition of a second equivalent of MMA feed to the completely polymerized system. This second polymerization reaction is also quantitative, and only a slight increase in polydispersity is observed (Figure 3). Under similar reaction conditions (MMA, neat or in toluene), high molecular weight PMMA is obtained ($M_n = 150000$) with polydispersities that remain relatively low ($M_w/M_n = 1.35 - 1.45$).

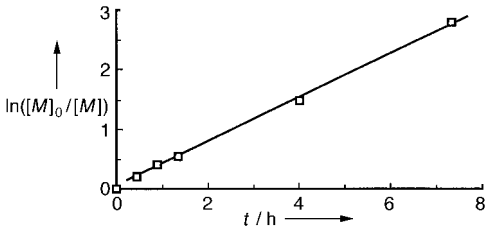


Figure 1. Time dependence of $\ln([M]_0/[M])$ at 85 °C where $[M]_0$ and $[M]$ are the MMA concentration at times 0 and $t$ ($y = 3.60 \times 10^{-2} + 0.375x$; $r^2 = 0.999$). Reaction conditions are the same as in Table 1 (catalyst = $[RuCl_2(p\text{-cymene})(PCy_3)]$).

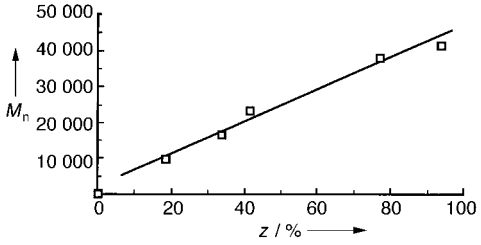


Figure 2. Dependence of the PMMA molecular weight $M_n$ on monomer conversion $z$ ($y = 1655 + 449.5x$; $r^2 = 0.985$). Reaction conditions are the same as in Table 1 (catalyst = $[RuCl_2(p\text{-cymene})(PCy_3)]$).

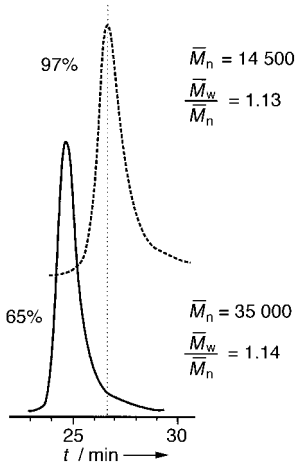


Figure 3. Size-exclusion chromatograms of the PMMA after a first feed of MMA (solid line; $[MMA]_{0(1)}:[initiator]_0:[RuCl_2(p\text{-cymene})(PCy_3)]_0 = 200:2:1$; conversion = 97%), and after a second feed of MMA (dashed line; $[MMA]_{0(2)}:[initiator]_0:[RuCl_2(p\text{-cymene})(PCy_3)]_0 = 400:2:1$; conversion = 65% (230% when expressed according to Sawamoto et al.[15])).

A further advantage of this new catalytic system is that it is highly soluble in neat MMA. $[RuCl_2(p\text{-cymene})(PR_3)]$ complexes are also quite soluble in common organic solvents including heptane, which is the solvent used for precipitation of the polymer. This yields white PMMA as opposed to pale green or light brown PMMA precipitated from reaction mixtures of nickel-[3] or iron-mediated[8] polymerization reactions.

The polymerization mechanism is likely to be radical since the PMMA tacticity (typically $rr:rm:mm = 57.8:36.8:5.4$, $\rho = 0.99$) fits the tacticity known for a radical polymerization reaction. Furthermore, galvinoxyl (5 equiv relative to the initiator), a well-known radical inhibitor, inhibits the MMA polymerization, and reaction mixtures in air also fail to polymerize. Surprisingly, the best catalyst systems for ATRP of MMA have been shown to be also the most active ones for the ring-opening metathesis polymerization (ROMP) of strained and low-strain cycloolefins.[9] In both reactions, the same stereoelectronic requirements for the phosphane ligand of the ruthenium complex (sterically demanding phosphanes, typically tricyclohexylphosphane $PCy_3$) have been demonstrated. Furthermore, the ease with which the arene ligand is disengaged from the different $[RuCl_2(p\text{-cymene})(PR_3)]$ complexes reported in Table 1 (as quantified by standardized thermogravimetric measurements[10] and $^1H$ NMR spectroscopy at 85 °C) indicates a direct relationship between arene ligand lability and catalyst activity in both reactions. This suggests that the $p$-cymene ligand is released in the process and the question arises about the possible coordination of the monomer during the ATRP reaction. Hence, $[RuCl_2(=CHPh)(PCy_3)_2]$, the Grubbs ruthenium–carbene complex commonly used for olefin metathesis,[11] was tested as a catalyst for polymerization of MMA (Table 1). This catalyst was found to be even more active ($k_{app} = 1.95 \times 10^{-4}\,s^{-1}$) than $[RuCl_2(p\text{-cymene})(PCy_3)]$ ($k_{app} = 1.05 \times 10^{-4}\,s^{-1}$), $[RuCl_2(p\text{-cymene})(Pi\text{Pr}_3)]$ ($k_{app} = 5.65 \times 10^{-5}\,s^{-1}$), or $[RuCl_2(p\text{-cymene})(PPhCy_2)]$ ($k_{app} = 5.2 \times 10^{-5}\,s^{-1}$), but to the detriment of polymer control ($M_w/M_n = 1.28$; $f = 0.60$). The synthesis of potential ATRP catalysts based on ruthenium–carbene complexes is presently in progress.[12]

Other vinyl monomers such as methacrylates, $n$-butyl acrylate, and 4-substituted styrenes have also been success-

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fully polymerized (see Table 2), although with a somewhat lesser control (the reactions were not optimized). Vinyl acetate, a substrate known to be reluctant to undergo ATRP, is not polymerized under the same reaction conditions.

Table 2. [$RuCl_2$(*p*-cymene)($PCy_3$)]-catalyzed polymerization of various vinyl monomers.[a]

| Monomer | Yield [%] | $M_n$[b] | $M_w/M_n$ | $f$[c] |
|---|---|---|---|---|
| methyl methacrylate | 100 | 41500 | 1.12 | 0.95 |
| *tert*-butyl methacrylate | 80 | 33500 | 1.2 | 0.95 |
| isobornyl methacrylate | 70 | 25000 | 1.2 | 1.1[d] |
| *n*-butyl acrylate | 80 | 37500 | 1.95 | 0.85 |
| styrene | 64 | 28500 | 1.3 | 0.9 |
| vinyl acetate | 0 | – | – | – |

[a] Reaction conditions same as in Table 1, except for styrene (initiator, (1-bromoethyl)benzene; temperature, 110 °C). [b] Apparent $M_n$ for poly(*tert*-butyl methacrylate), poly(isobornyl methacrylate), and poly(*n*-butyl acrylate) determined with PMMA calibration. For poly(methyl methacrylate) and polystyrene, PMMA and PS calibrations were used, respectively. [c] Initiation efficiency $f = M_{n,theor.}/M_{n,exp.}$ with $M_{n,theor.} = ([MMA]_0/[initiator]_0) \times M_w(MMA) \times$ conversion. [d] An initiation efficiency higher than 1 could mean that the PMMA calibration is not suitable for poly(isobornyl methacrylate).

However, similar reactions with methacrylic acid (MA) and 2-hydroxyethyl methacrylate (HEMA) are successful, as well as controlled copolymerizations (95% MMA/5% MA and 90% MMA/10% HEMA) ($M_w/M_n = 1.24$ and 1.17). Since ATRP requires a suitable adjustment between the structure of the monomer, initiator, and atom (or group of atoms) to provide reversible termination, the catalyst has to be fine-tuned to each monomer. This has been exemplified for *n*-butyl acrylate. For this monomer, the molecular weight distribution dropped from 1.9 to 1.4 simply by the use of P*i*$Pr_3$ as the phosphane (instead of $PCy_3$), which demonstrates the versatility of the catalyst system.

*Experimental Section*

All reagents and solvents were dried, distilled, and stored under nitrogen at −20 °C with conventional methods. Ruthenium complexes were synthesized and purified according to the literature.[9, 13, 14] Grubbs catalyst, [$RuCl_2$(=CHPh)($PCy_3$)$_2$], was used as received (Strem).

Polymerization of MMA: Ruthenium complex (0.0116 mmol) was placed in a glass tube containing a bar magnet and capped by a three-way stopcock. The reactor was purged of air (three vacuum–nitrogen cycles) before methyl methacrylate (1 mL, 9.35 mmol), and the initiator (ethyl 2-bromo-2-methylpropionate 0.1M in toluene, 0.232 mL) were added. All liquids were handled with dried syringes under nitrogen. The mixture was heated in a thermostated oil bath for 16 h at 85 °C and, after cooling, dissolved in THF and the product precipitated in heptane. The polymer was filtered off and dried overnight at 80 °C under vacuum.

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## Modeling the Selectivity of Potassium Channels with Synthetic, Ligand-Assembled π Slides**

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Since Hodgkin and Huxley's demonstration almost fifty years ago that nerve signals originate from selective flux of $Na^+$ and $K^+$ ions across cell membranes, the mechanism of ion selectivity, particularly that of $K^+$ channels, has remained a fascinating and central question in life sciences.[1–3] The classical view of amide oxygen atoms serving as selective $K^+$ binding sites has received substantial support from site-

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