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Synthesis and structure of $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$ and its catalytic application to anti-Markovnikov addition of carboxylic acids to terminal alkynes

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ABSTRACT

The compound, $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$ (**1**) has been synthesized from the precursor complex, $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$. The complex has been structurally characterized. This complex has been found to be an efficient catalyst for the *anti*-Markovnikov addition of carboxylic acids to alkynes.

1. Introduction

Coordinatively unsaturated ruthenium complexes have received much attention due to their involvement in transition metal catalyzed organic transformations. Ruthenium can adopt wide range of coordination geometry and oxidation states and because of this ruthenium compounds are found to be effective catalysts for variety of organic reactions [1–3]. It has been emphasized in the literature that, 16 electron cationic species are the active catalyst in many cases. Thus, in recent years, some 16 electron cationic stable species have been isolated and characterized structurally [4–6]. Ruthenium(II) phosphine complexes are widely used as catalyst for various types of organic transformations. A large number reports deal with synthesis of ruthenium complexes of tertiary phosphines of all types, including bi and tridentate phosphines [7–15]. For example, diphenylphosphinoethane complex of ruthenium of the type, $[Ru(dppe)_2Cl_2]$ has been synthesized from the reaction of $RuCl_2(DMSO)_4$ with the diphosphine ligand [16]. However, reaction of $RuCl_2(DMSO)_4$ with the diphenylphosphinoethane affords mixture of *cis*- and *trans*- isomers [16]. The corresponding cationic complexes of the type, and $[Ru(dppe)_2Cl]PF_6$ and *cis*-$[Ru(dppe)_2(CH_3CN)Cl]PF_6$ have been synthesized from the parent complex, *cis*-$[Ru(dppe)_2Cl_2]$ [17]. It is worth mentioning here that, *cis*-$[Ru(dppe)_2(CH_3CN)Cl]PF_6$ has not been structurally characterized and their catalytic properties still remains unexplored.

Ruthenium complexes with diphosphine ligands have been shown to be effective catalysts for various organic transformations [18–20]. Control of stereo and regioselectivity of metal catalyzed organic transformations has been shown to be dependent on the ligand environment around the metal center. Thus, ligand controlled metal catalysis has received much attention in recent years [21–24].

Rotem and Shvo reported the first ruthenium catalyzed addition of carboxylic acid to alkyne using $Ru_3(CO)_{12}$ as catalyst [25,26], which was followed by a large number of reports on ruthenium catalyzed addition of carboxylic acid to alkynes [27–36]. Addition of carboxylic acid to alkynes affords three possible isomers (Scheme 1) and most of the reported reactions afford more than one isomers. Ruthenium catalyzed addition of carboxylic acid has been studied extensively by Dixneuf and coworkers [27,32,33] and Mitsudo and coworkers [29–31] and catalytic systems for *anti*-Markovnikov addition as well as Markovnikov addition product have been developed. For *anti*-Markovnikov addition, the catalysts used were synthesized from sensitive organometallic compounds like, bis(cyclootadienyl)Ru [29,30] or bis(2-methylpropenyl)-(cyclooctadienyl)Ru [27]. Recently Goossen et al. have reported *anti*-Markovnikov addition of carboxylic acid to alkynes using $[(p\text{-cumene})RuCl_2]_2$ and phosphenes like PPh_3, $P(Fur)_3$, or $P(p\text{-Cl-}C_6H_4)_3$ in the presence of organic bases like pyridine or (4-dimethylamino)pyridine [28]. We have been interested in the

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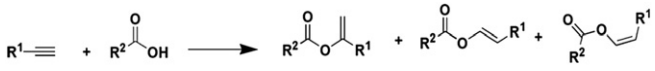


Scheme 1. Three possible products of the reaction of alkyne with carboxylic acid.

Table 1
Crystal data and structure refinement for **1**.

Empirical formula, Formula weight	$C_{78}H_{71}BClNP_4Ru$, 1293.57
Temperature, K	293(2)
Wavelength (Å)	0.71073
Crystal system, Space group	Triclinic, P-1
a (Å), b (Å), c (Å)	12.3230(13), 13.8160(14), 19.274(2)
α, β, γ (°)	93.776(3), 97.575(3), 94.302(3)
V (Å^3)	3234.1(6)
Z	2
$D_{calc.}$ (Mg/m^3)	1.328
μ (Mo-Kα) (mm^{-1})	0.428
F(000)	1344
θ range (°)	1.07–27.40
Reflections collected, Unique reflections	43,614, 14,108
R(int)	0.0727
Restraints, parameters	0, 776
Goodness of fit (F^2)	0.991
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0530, wR2 = 0.1109
R indices (all data)	R1 = 0.1236, wR2 = 0.1440

synthesis of simple catalytically active ruthenium(II) cationic compound containing phosphine ligands and labile N donor ligands, which can easily afford 16 electron species in solution. Recently, we have reported synthesis, structure and catalytic properties of the cationic complex, $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ [37–40]. The cationic complex was found to be an active catalyst for condensation of carboxylic acid and alkynes in the presence of catalytic amount of $BF_3{\cdot}Et_2O$ [40]. The major product was found to be Markovnikov addition product. Dixneuf and coworkers have suggested that, for the anti-Markovnikov addition diphosphine complexes are more efficient and also, it has been suggested that, the addition takes place via external attack of carboxylic acid to the electrophilically activated coordinated alkyne. We wanted to synthesize a cationic ruthenium complex containing diphosphine ligand having only one vacant coordination site. This complex is expected to form a vinylidene complex on reaction with terminal alkynes. The carboxylic acid is expected to attack the C2 carbon of the vinylidene moiety, leading to the formation of anti-Markovnikov addition product. Herein we report synthesis, structure and catalytic properties of the cationic ruthenium complex, $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$ (**1**).

2. Results and discussion

2.1. Synthesis and characterization

The complex has been synthesized from the reaction of $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ with diphenylphosphinoethane (dppe) in 1:1 ratio in acetonitrile. The complex has been characterized by elemental analyses, IR, and ^1H and ^{31}P NMR spectroscopy.

The elemental analyses agree well with the formulation of the compound. The ^1H NMR spectrum of the compound in $CDCl_3$ solution shows a broad signal at 2.70 ppm due to the four $-CH_2$ protons of the bonded dppe ligand and $-CH_3$ protons of the coordinated acetonitrile ligand, which is in good agreement with other ruthenium dppe complexes [16,17]. The signals for the aromatic protons appear as multiplets in the region 6.56–7.88 ppm. Along with these signals a singlet appears at 2.07 ppm. This is due to the $-CH_3$ protons of free acetonitrile. The appearance of the signal for free acetonitrile clearly shows that, the complex undergoes dissociation and produces a five coordinate species in solution. At −40 °C the signal at 2.70 ppm splits into two broad signals, which appear at 2.75 and 2.65 ppm. The signal for free acetonitrile ligand appears at 2.07 ppm. Thus it is clear that, even at low temperature the complex undergoes dissociation in solution to give a five coordinate species. The ^{31}P NMR spectrum of the complex in $CDCl_3$ solution shows a sharp singlet at 48.7 ppm.

2.2. Single crystal X-ray structure

The complex crystallizes in triclinic space group P-1 (Table 1). The asymmetric unit contains one cationic ruthenium complex, $[Ru(dppe)_2(CH_3CN)Cl]^+$, and one tetraphenylborate anion. The

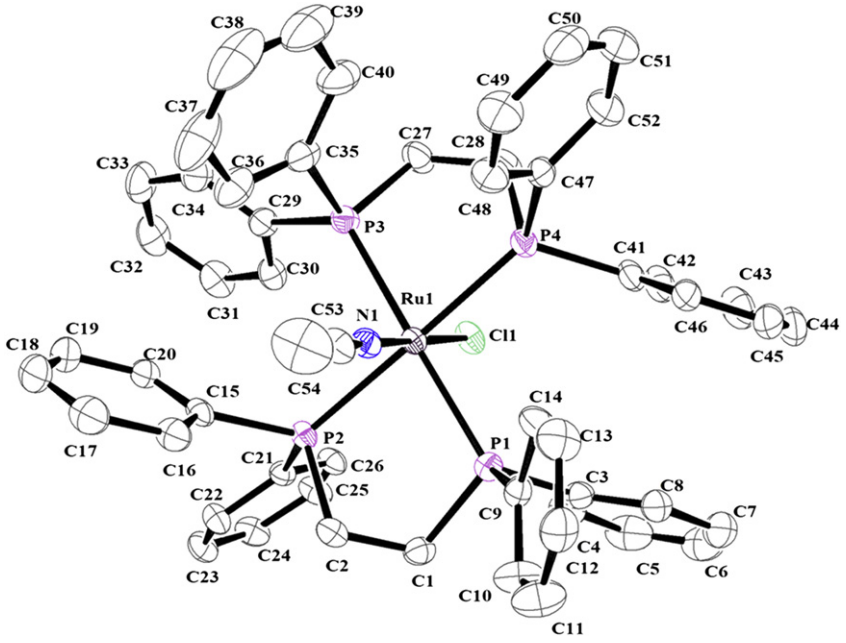


Fig. 1. ORTEP view of $[Ru(dppe)_2(CH_3CN)Cl]^+$ cation. Hydrogen atoms have been omitted for clarity.

Table 2
Important bond distances [Å] and bond angles [°] of **2** and **3**.

Bond distances [Å]		Bond angles [°]			
N(1)-Ru(1)	2.006(3)	N(1)-Ru(1)-P(2)	92.59(10)	N(1)-Ru(1)-P(1)	99.03(10)
P(1)-Ru(1)	2.3940(12)	P(2)-Ru(1)-P(1)	80.29(4)	N(1)-Ru(1)-P(4)	89.68(10)
P(2)-Ru(1)	2.3510(12)	P(2)-Ru(1)-P(4)	177.71(4)	P(1)-Ru(1)-P(4)	99.09(4)
P(3)-Ru(1)	2.4106(11)	N(1)-Ru(1)-P(3)	82.58(10)	P(2)-Ru(1)-P(3)	98.22(4)
P(4)-Ru(1)	2.3964(12)	P(1)-Ru(1)-P(3)	177.83(4)	P(4)-Ru(1)-P(3)	82.33(4)
Cl(1)-Ru(1)	2.4241(10)	N(1)-Ru(1)-Cl(1)	178.02(10)	P(2)-Ru(1)-Cl(1)	88.20(4)
		P(1)-Ru(1)-Cl(1)	79.31(4)	P(4)-Ru(1)-Cl(1)	89.52(4)
		P(3)-Ru(1)-Cl(1)	99.11(4)		

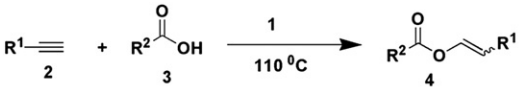
cationic complex has a ruthenium(II) center bonded to one acetonitrile and a chloride in a *trans* fashion and two diphenylphosphinoethane ligands in *cis* fashion (Fig. 1). The BPh_4^- anion is found outside the coordination sphere and does not show any bonding interaction with ruthenium(II) center. All the four Ru-P distance are not equal [P1–Ru1 = 2.3940(12) Å, P2–Ru1 = 2.3510(12) Å, P3–Ru1 = 2.4106(11) Å, P4–Ru1 = 2.3964(12) Å]. The ∠N1-Ru1-Cl1 angle is less then 180° [178.02(10)]. Similarly, the ∠P(1)-Ru(1)-P(3) and ∠P(2)-Ru(1)-P(4) angles are found to be 177.83(4)° and 177.71(4)°, respectively (Table 2). Thus, the ruthenium(II) center is found to be in a distorted octahedral coordination environment.

2.3. Catalytic addition of carboxylic acids to alkynes

We were interested in studying the efficacy of **1** as catalyst for the addition of carboxylic acids to alkynes. The reaction of phenylacetylene with benzoic acid in toluene was chosen as a model reaction to establish the reaction condition. Benzoic acid (5 mmol) was mixed with phenylacetylene (5 mmol) and **1** (0.05 mmol) in toluene (5 cm^3) and the reaction was monitored by HPLC. To begin with the reaction was carried out for 12 h at room temperature. However, we could not detect any product formation. Gradually the temperature was raised to 70 °C, but even at that temperature no reaction took place. However, at 90 °C we could observe product formation and at 12 h complete conversion was achieved. The product obtained was a mixture of *E* and *Z* isomers (25:75) of *anti*-Markovnikov product (60%) and Markovnikov addition product (40%) (Table 3). When the temperature was raised to 110 °C, the regioselectivity of the addition reaction changed and a mixture (5:95) of Markovnikov and *anti*-Markovnikov addition product was obtained. The ratio of the *E* and *Z* isomers of the *anti*-Markovnikov product was found to be 20:80.

Having established the reaction condition, the scope of the reaction was tested. The reactions of various alkynes with aromatic and aliphatic carboxylic acids were carried out (Scheme 2, Table 4). The reaction was found to be effective in the cases of both aromatic and aliphatic carboxylic acids as well as alkynes. The products were isolated in 80–90% yield with moderate turnover number. The products were characterized by ^1H and ^{13}C NMR spectroscopy. In addition, the newly synthesized compounds, **4e**, **4h**, **4k** and **4l** have been characterized by high resolution mass spectrometry (HRMS).

In contrast to the $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ catalyzed addition of carboxylic acids to terminal alkynes, which afford Markovnikov addition product [40], in the present catalytic system, the major product is anti-Markovnikov addition product. Also, in $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ catalyzed reaction addition of Lewis acid, BF_3 is necessary. However, in the present case, addition of Lewis acid is not required. No improvement on the conversion could be observed upon addition of BF_3Et_2O. Apart from these, the turnover numbers were found to be slightly better compared to the $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ catalyzed reaction. In the complex, $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ the number of possible vacant coordination sites is more, as a result we could observe coordination of both the substrates to the ruthenium center [40]. However, the number of possible vacant coordination site in $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$ is only one and thus, in the in situ NMR studies (*vide infra*)



1 = $[Ru(dppe)_2Cl(NCCH_3)][BPh_4]$

R^1 = -Ph (a); R^1 = -$(CH_2)_3CH_3$ (b); R^1 = -cyclo-(C_6H_{11}) (c)
R^2 = -CH_3 (a), R^2 = -CH_2Cl (b), R^2 = -CH_2CH_3 (c), R^2 = -Ph (d), R^2 = -pClC_6H_4 (e),
R^2 = -CH=CHPh (f) R^2 = -o(OH)C_6H_4 (g), R^2 = -$CH(CH_3)NHC(O)O^tBu$ (h),

Scheme 2. Reaction of various alkynes with different carboxylic acids catalyzed by **1**.

Table 3
Temperature dependence of the product distribution of **1** catalyzed reaction of phenylacetylene and benzoic acid acid.[a]

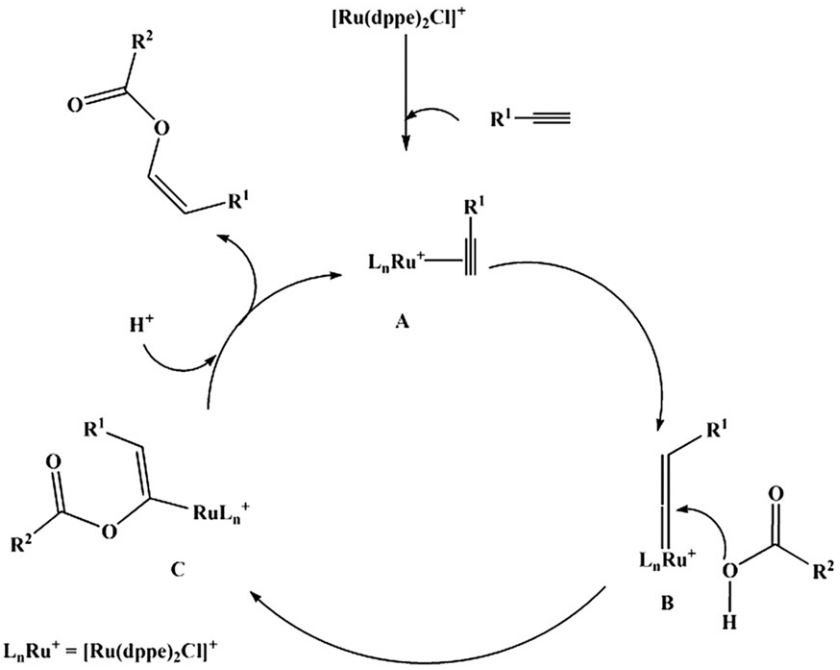
Entry	*T* (°C)	Yield (%) (Markovnikov)[b]	Yield (%) (Anti-Markovnikov)[b] (E/Z)[c]
1	90	40	60 (25:75)
2	110	5	95 (20:80)

[a] Reaction condition: 5 mmol benzoic acid, 5 mmol phenylacetylene in 5 cm^3 of toluene and 0.05 mmol of catalyst, time 12 h.
[b] Isolated.
[c] HPLC and ^1H NMR.

Table 4
Reaction of alkynes with carboxylic acid catalyzed by **1**.[a]

Entry	Alkyne	Acid	Product	Yield (%)[b]	E/Z	TON[C]
1	2a	3a	4a	90	10/90	180
2	2a	3b	4b	85	20/80	170
3	2a	3c	4c	87	20/80	174
4	2a	3d	4d	95	20/80	190
5	2a	3e	4e	80[d]	25/75	160
6	2a	3f	4f	85[d]	35/65	170
7	2a	3g	4g	82[d]	15/85	164
8	2a	3h	4h	75[e]	5/95	150
9	2b	3d	4i	80	25/75	160
10	2b	3e	4j	85[d]	20/80	170
11	2c	3d	4k	83	25/75	166
12	2c	3c	4l	80	35/65	160

[a] Reaction condition: 2 mmol carboxylic acid, 2 mmol alkyne in 5 cm^3 of toluene and 0.01 mmol of catalyst, time 12 h.
[b] Isolated.
[c] mol of product/mol of catalyst.
[d] Time 15 h.
[e] Time 17 h.



Scheme 3. Plausible mechanism of reaction of alkyne and carboxylic acid catalyzed by **1**.

we could observe only coordination of alkyne. This may be the reason for difference in regioselectivity of the addition reaction.

2.4. Mechanistic investigations

We have carried out *in situ* ^{31}P as well as ^{1}H NMR spectral studies in order to get an insight into the mechanism of the reaction. As discussed earlier, the ^{1}H NMR spectrum of the complex clearly show that, in solution, it affords a pentacoordinated ruthenium complex cation, $[Ru(dppe)_2Cl]^+$.

The ^{31}P NMR spectrum of the complex in toluene-d^8 shows a singlet at 49.1 ppm. The spectrum remains unchanged when the solution is heated up to 110 °C. However, when phenylacetylene is added to the solution and is heated to 110 °C, a new intense signal appears at 38.1 ppm. The ^{1}H NMR spectrum of this solution shows a new signal at 3.40 ppm (singlet). This can be assigned to the ruthenium coordinated vinylidene proton of the species, $[Ru(=C=CHPh)(dppe)_2Cl]^+$. This assignment is in good agreement with a structurally characterized ruthenium vinylidene compound, [(*p*-Cymene)Ru(μ-Cl)$_3$RuCl(=C=CHPh)(IMes)] {IMes = 1,3-dime-setylimidazole-2-yilidene}, the ^{1}H NMR spectrum of which shows a singlet at 3.41 ppm due to the vinylidene proton [41]. It may be noted that, a similar ruthenium vinylidene intermediate has been suggested in the ruthenium catalyzed synthesis of vinylic carbamates by the reaction of secondary amines, CO_2 and terminal alkynes [27].

To the solution of the complex and phenylacetylene, when acetic acid is added and heated, a new signal (singlet) appears at 2.28 ppm in the ^{1}H NMR spectrum. This singlet can be assigned to the $-CH_3$ protons of the ester group. Based on the above *in situ* NMR studies a plausible mechanism has been suggested, which has been shown in Scheme 3. The first step is the coordination of alkyne to $[Ru(dppe)_2Cl]^+$ (**A**) followed by the rearrangement of the coordinated alkyne to give alkylidene species, $[Ru(=C=CHPh)(dppe)_2Cl]^+$ (**B**). This is followed by the external attack by the carboxylic acid to produce the species **C**, which is followed by the release of the product by protonolysis (Scheme 3).

3. Conclusion

In summary, we have synthesized a simple cationic ruthenium complex, $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$, using commercially available cheap chemicals. The complex is not air or moisture sensitive. This complex can catalyze *anti*-Markovnikov addition of carboxylic acids to alkynes, in a stereoselective manner affording *Z*-vinyl esters. The catalyst loading is low (0.5 mol%) and the reaction does not require any harmful additives like pyridine.

4. Experimental

Chemicals and solvents used were reagent grade products. The ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on Bruker Avance II (^{1}H frequency = 400 MHz and 200 MHz) spectrometers. HRMS of the newly synthesized compounds, **4e**, **4h**, **4k** and **4l** were recorded on TOF MS in ESI^+ mode in methanol water mixture.

4.1. Preparation of $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$ (**1**)

A sample of $[(PPh_3)_2Ru(CH_3CN)_3Cl][BPh_4]$ (1.1 g; 1 mmol) and dppe (0.76 g; 2 mmol) was dissolved in acetonitrile (10 cm^3) and the reaction solution was refluxed for about 2.5 h. The reaction solution was then filtered. The filtrate was concentrated on water-bath, where upon a yellow crystalline solid separated out. The separated solid was filtered and washed with hexane (3–4 times) and dried in vacuo. The compound was finally recrystallized from acetonitrile. Yield: 1.02 g; 79%. Anal. Calc. for $C_{78}H_{71}BClNP_4Ru$ (Mol. Wt. = 1293.63): C; 72.42, H; 5.53, N; 1.08. Found: C; 71.08, H; 4.72, N; 0.98. IR (cm^{-1}): 730, 745, 930, 1434, 1483, 3053. ^{1}H NMR ($CDCl_3$) (in ppm): 2.70 (br, 11H) 6.55–7.82 (m, 60H). ^{31}P NMR ($CDCl_3$) (in ppm): 48.7.

4.2. $[Ru(dppe)_2(CH_3CN)Cl][BPh_4]$ (**1**) catalyzed reaction of alkynes with carboxylic acid

The alkyne (2 mmol), acid (2 mmol), and the catalyst (0.01 mmol) were taken in 5 cm^3 toluene in a 25 ml round

bottomed flask fitted with a reflux condenser. The reaction mixture was stirred for desired time (12–17 h) at 110 °C. The reactions were monitored by TLC. After the completion of the reaction, the solvent was removed under vacuum. The products were purified by column chromatography. The products were characterized by ^{1}H and ^{13}C NMR spectroscopy. The new compounds, **4e**, **4h**, **4k** and **4l**, have been further characterized by HRMS. The spectral data and the spectra are given in supplementary material.

4.3. Single crystal data collection and refinements

Single crystal X-ray data of **1** were collected on Bruker Smart APEX system that uses graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). The structure was solved by direct method and refined by least square method on F^2 employing WinGx [42] package and the relevant programs (SHELX-97 [43] and ORTEP-3 [44]) implemented therein. Non-hydrogen atoms were refined anisotropically and hydrogen atoms on C-atoms were fixed at calculated positions and refined using a riding model. The crystal data is given in Table 1 and important bond distances and bond angles are given in Table 2.

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Appendix. Supplementary material

Supplementary data related to this article can be found online at doi:10.1016/j.jorganchem.2011.11.017.

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