Selective H₂ and CO production with rhenium(I) biscarbonyl complexes as photocatalyst

Tatsuki Morimoto • Junji Tanabe • Kazuhiko Sakamoto • Kazuhide Koike • Osamu Ishitani

Received: 28 January 2012/Accepted: 6 March 2012/Published online: 29 June 2012 © Springer Science+Business Media B.V. 2012

Abstract Rhenium(I) biscarbonyl complexes with two phosphine ligands photocatalyzed not only CO₂ reduction under CO₂ atmosphere but also H₂ evolution under Ar. The reductant 1-benzyl-1,4-dihydronicotinamide (BNAH) worked only as a one-electron donor, and it was quantitatively converted to its corresponding oxidized dimer (BNA₂). The photocatalytic reactions required addition of a base such as triethanolamine, because deprotonation from the oxidized BNAH (BNAH^{•+}) is essential for the suppression of the back electron transfer from the reduced rhenium(I) complex to BNAH^{•+}. ¹H, ¹³C, and ³¹P NMR studies under vacuum or ¹³CO₂ atmosphere indicated that the rhenium(I) complex is relatively stable under the CO₂ reduction conditions, but it is converted to some other complexes under the H₂ evolution conditions.

T. Morimoto · O. Ishitani

T. Morimoto PRESTO, Japan Science and Technology Agency (JST), 4-1-8 Honcho, Kawaguchi-shi, Saitama 332-0012, Japan

J. Tanabe · K. Sakamoto Graduate School of Science and Engineering, Saitama University, 255 Shimo-Okubo, Saitama 338-8570, Japan

K. Koike National Institute of Advanced Industrial Science and Technology, 16-1 Onogawa, Tsukuba, Ibaraki 305-8569, Japan

O. Ishitani (⊠) ALCA, Japan Science and Technology Agency (JST), 4-1-8 Honcho, Kawaguchi-shi, Saitama 332-0012, Japan e-mail: ishitani@chem.titech.ac.jp

Department of Chemistry, Graduate School of Science and Engineering, Tokyo Institute of Technology, O-okayama 2-12-1-NE-1, Meguro-ku, Tokyo 152-8551, Japan

Keywords Photochemistry \cdot CO₂ reduction \cdot H₂ evolution \cdot Rhenium complex

Introduction

Solar energy use has attracted much attention in recent years owing to both the shortage of fossil resources and global warming. In particular, the conversion of solar energy to chemical energy should be one of the most promising solutions. The number of studies on photocatalytic systems for CO_2 reduction and for reduction and oxidation of water have been increasing over the past few years [1–4]. Photocatalysis by transition metal complexes is unique because of the high selectivity of the product(s) and efficiency as compared to photocatalysis by organic and semiconductor compounds.

In particular, rhenium(I) tricarbonyl complexes fac-[Re(N^N)(CO)₃L]ⁿ⁺ $(N^N = \text{dimine ligand}, L = \text{halide anion}, SCN^-: n = 0; PR_3, pyridine derivatives:$ n = 1) have exhibited attractive photocatalytic abilities for CO₂ reduction [5–8]. The reaction mechanism is unique. These complexes can work both as a photosensitizer, which initiates a photochemical one-electron transfer, and as a catalyst, which introduces two electrons into CO_2 , selectively giving CO [9]. In contrast, photocatalytic systems of other metal complexes used for CO_2 reduction are mostly constructed with at least two components. Another unique property they possess is their high selectivity of the product. The photocatalytic reactions yield CO as the sole product even in the presence of water in the reaction solution. Furthermore, only a few reports have described photocatalytic production of H_2 by using rhenium(I) tricarbonyl complexes. Tajik and Detellier [10] have reported that $[Re(bpy)(py)(CO)_3]^+$ (bpy = 2,2'-bipyridine; py = pyridine) incorporated into the layers of hectorite clay can photocatalyze H₂ production. Pac et al. [11] have observed that H₂ becomes the main product during the photocatalytic reaction using fac-[Re(bpy)(CO)₃Br] only in ether solutions such as tetrahydrofuran, even under CO₂ atmosphere.

We have successfully synthesized a new series of rhenium(I) complexes that have only two CO ligands, i.e., *cis*, *trans*- $[Re(X_2bpy)(CO)_2(PR_3)L]^{n+}$ (Chart 1: $X_2bpy = 4,4'-X_2$ -bpy) [12]. Photocatalysis by these complexes might be interesting because some of them can emit at room temperature in solution and can efficiently absorb the visible light [13, 14]. Herein, we report the peculiar photocatalytic ability of the rhenium(I) biscarbonyl complexes, some of which photocatalyze H₂ evolution under Ar atmosphere and selective CO formation under CO₂ atmosphere.

The studied rhenium(I) biscarbonyl complexes are represented by **1a–1i**, as shown in Chart 1.

Experimental

General procedures

IR spectra were recorded on a JEOL JIR-6500 spectrometer with a TGS detector or on a JASCO FT/IR-610 with an MCT detector. UV–Vis spectra were recorded using

		Х	PR ₃	L	n
	1a	Н	PPh ₃	PPh ₃	1
, ¬n+	1b	н	P(OEt) ₃	PPh_3	1
X T	1c	Me	P(OEt) ₃	PPh ₃	1
S=N, CO	1d	н	P(OEt) ₃	P(OEt) ₃	1
	1e	Me	P(OEt) ₃	P(OEt) ₃	1
X~ 00	1f	н	P(OEt) ₃	ру	1
PR ₃	1g	н	P(OEt) ₃	CH ₃ CN	1
-	1h	н	P(OEt) ₃	CI⁻	0
	1i	CF_3	P(OEt) ₃	P(OEt) ₃	1

Chart 1 Rhenium(I) biscarbonyl complexes 1a-1i

a Hitachi 330 spectrophotometer or an Otsuka-Denshi Photal-2000 multichannel spectrophotometer with a $D_2(25 \text{ W})/I_2(25 \text{ W})$ mixed lamp. Emission spectra at 25 °C were measured with a Hitachi F-3000 fluorescence spectrophotometer. ¹H, ¹³C, and ³¹P NMR spectra were recorded on a JEOL JNM-LA 500 FT-NMR spectrometer. Emission lifetimes were measured with a Horiba NAES-1100 time-correlated single-photon counting system (the excitation source was a nanosecond H₂ lamp, NFL-111, and the instrumental response time was less than 1 ns).

Materials

N,*N*-dimethylformamide (DMF) was dried over 4A molecular sieves and distilled under reduced pressure. Triethanolamine (TEOA) was distilled under reduced pressure. Triethylamine (TEA) was distilled under Ar atmosphere. These compounds were stored under Ar atmosphere before use. Commercially available reagents (reagent grade) were used without further purification. The rhenium(I) biscarbonyl complexes **1a–1i** were synthesized according to the methods used previously [12]. 1-Benzyl-1,4-dihydronicotinamide (BNAH) and the corresponding 4,4'- and 4,6'-dimers (BNA₂) were prepared according to the literature [15–17].

Photocatalytic reactions

Photocatalytic reactions were carried out with a solution of the photocatalyst (1.0 mM) in DMF (4 mL) in the presence of TEOA (0.5 M) and BNAH (50 mM). After bubbling the reaction mixture with CO₂ or Ar for 20 min, the solution was irradiated for 6 h in a merry-go-round irradiation apparatus at $\lambda > 400$ nm using a 300 W Matsushita tungsten-halogen lamp with a NaNO₂ solution filter (4 mol/L, d = 1 cm). The evolved CO and H₂ were analyzed by a Yanagimoto G3800 GC with a TCD detector and an active carbon column (60/80 mesh, 2 m). The turnover number (TN) is defined as the (amount of evolved CO or H₂)/(amount of the photocatalyst). BNAH and BNA₂ were quantified by a JASCO HPLC system comprising a PU-980 pump with a DG-980-80 degasser, a CO-965 column oven, a UV-970 detector, and an octadecylsilyl (ODS) column (Nomura Chemical Develosil Packed Column ϕ 4.6 × 250).

Results and discussion

Photocatalytic reaction under Ar or CO₂ atmosphere

As a typical run, a DMF solution (4 mL) containing **1a** (1.0 mM), TEOA (0.5 M), and BNAH (50 mM) as a sacrificial electron donor was bubbled with a gentle stream of Ar for 20 min, and then irradiated with >400 nm light. Irradiation for 6 h yielded 60.4 µmol of H₂ (TN_{H₂} = 15.1). On the other hand, the formation of H₂ was almost suppressed and photocatalytic CO formation was observed when the reaction mixture was bubbled with CO₂ instead of Ar keeping the other reaction conditions unchanged (TN_{CO} = 16.5).

Table 1 summarizes the yields of H₂ and CO obtained by photocatalytic reactions with **1a–1i** during 6 h irradiation. The rhenium(I) complexes with two phosphine ligands (**1a–1e**) exhibited higher photocatalytic abilities for producing both H₂ and CO as compared to the others (**1f–1h**), which have only one phosphine ligand. For example, the most efficient photocatalyst (**1a**), which contained two triphenylphosphine ligands, showed $TN_{H2} = 15.1$ and $TN_{CO} = 16.5$. The only exception was **1d**, which could photocatalyze the CO₂ reduction but gave a relatively small amount of H₂ under Ar atmosphere. Moreover, the efficiencies of the photocatalytic reactions were highly dependent on the substituents present on the diimine ligand. The complex coordinated by electron-donating substituents displayed H₂ evolution and CO₂ reduction more efficiently than those with electron-withdrawing substituents, i.e., **1e** > **1d** \gg **1i**.

Stoichiometry of the photocatalytic reactions

In addition to the reduced products, the products of oxidation of the sacrificial electron donor (BNAH) were also analyzed in order to determine the stoichiometry

[Re(4,4'-X2-bpy	TN					
Complexes	Х	PR ₃	L	n	$\overline{H_2}^a$	CO ^b
1a	Н	PPh ₃	PPh ₃	1	15.1	16.5
1b	Н	P(OEt) ₃	PPh ₃	1	12.9	12.3
1c	Me	P(OEt) ₃	PPh ₃	1	13.8	13.0
1d	Н	P(OEt) ₃	P(OEt) ₃	1	4.8	9.2
1e	Me	P(OEt) ₃	P(OEt) ₃	1	6.8	11.5
1f	Н	P(OEt) ₃	ру	1	1.2	7.9
1g	Н	P(OEt) ₃	MeCN	1	0.34	1.2
1h	Н	P(OEt) ₃	Cl^{-}	0	0.04	0.05
1i	CF ₃	P(OEt) ₃	P(OEt) ₃	1	0.00	0.00

 Table 1
 Photocatalytic ability of 1a–1i under CO2 and Ar atmospheres

A DMF solution (4 mL) containing a complex (1.0 mM), TEOA (0.5 M), and BNAH (50 mM) was irradiated with >400 nm light for 6 h $\,$

^a Under Ar atmosphere

^b Under CO₂ atmosphere

of electrons. There are two oxidation pathways of BNAH: one-electron oxidation, deprotonation, and dimerization give the BNA dimers (BNA₂); two-electron oxidation via the electrochemical–chemical–electrochemical (ECE) mechanism gives BNA^+ (Scheme 1).

Production of the oxidized species (BNA⁺ and BNA₂) as well as consumption of BNAH can be analyzed by HPLC with an ODS column. A DMF solution containing **1d** (4.0 µmol, 1.0 mM), TEOA (0.5 M), and BNAH (2.0×10^2 µmol, 50 mM) was irradiated for 20 h using >400 nm light under Ar or CO₂ atmosphere. The results are summarized in Table 2. In both cases, BNAH was almost quantitatively oxidized and converted to its oxidized and dimerized forms 4,4'-BNA₂ and 4,6'-BNA₂ [17], whereas BNA⁺ was not detected. H₂ or CO in a similar amount to that of BNA₂ was also obtained. According to the material balance, BNAH served as a one-electron reductant, and therefore, two BNAH gave two electrons to produce one molecule of H₂ or CO. Deprotonation of BNAH^{•+} should give BNA[•], which is quantitatively converted to its dimerized form BNA₂ (Scheme 2).

Reaction mechanism of the photocatalytic reactions

Table 3 summarizes the emission lifetimes of $\mathbf{1}$ (τ_{em}) and behavior of emission quenching with BNAH. The complexes with two phosphine ligands exhibited not only longer excitation lifetime but also a higher quenching rate constant with BNAH, and therefore, they showed a relatively high quenching efficiency (η_q), which was calculated from Eq. 1, and should be strongly related to the efficiency for the excited state of **1** to be transformed into the corresponding one-electron reduced species.

$$\eta_{q} = 1 - \left(1 + k_{q}\tau \times [\text{BNAH}]\right)^{-1} \tag{1}$$

The photocatalytic ability of 1 thus should depend on the fact that the complex having a higher quenching efficiency is prone to photocatalyze reduction reactions



Scheme 1 Two oxidation pathways of BNAH: one-electron oxidation, deprotonation, and dimerization giving the BNA dimers (*top*) and two-electron oxidation via the ECE mechanism giving BNA⁺ (*bottom*)

Atmosphere	Consumption of BNAH $(\mu mol)^a$	Formation (µ	Formation (µmol)			
		BNA ₂ ^a	${\rm H_2}^{\rm a}$	CO ^a		
Ar	74.2 (200)	36.7 (99)	28.7 (77)	-		
CO ₂	161.4 (200)	75.2 (93)	_	63.0 (78)		

Table 2 Consumption of BNAH and formation of BNA2 and H2(CO) under Ar(CO2) for 20 h

^a The numbers in parentheses denote the relative amount of materials consumed or formed

Scheme 2 Material balances of the photocatalytic reactions for H₂ evolution and CO₂ reduction

more efficiently. Since the complexes with only one phosphine ligand have only short lifetimes of the triplet metal-to-ligand charge transfer (³MLCT) excited state, the initiation process of the photocatalytic reactions, namely, the electron transfer from BNAH to the excited state of **1**, could not proceed.

The material balance of the products shows that BNAH is the only source of electrons. Although BNA₂ is a stronger electron donor than BNAH [$E_{1/2}$ (BNA₂/BNA₂⁺) = -0.03 V and $E_{1/2}$ (BNAH/BNAH^{•+}) = +0.28 V versus Ag/AgNO₃], BNA₂ should not act as the reductant in the photocatalytic reactions. This is probably because the corresponding oxidized species BNA₂⁺⁺ is stable enough for the back electron transfer process, as shown in Scheme 3.

As a result of the competitive quenching process of the excited state of **1** with BNA₂, the quenching efficiency with BNAH should decrease after the accumulation

[Re(4,4'-X ₂ -bpy)(CO) ₂ (PR ₃)L] ^{+ or 0}				$\tau_{\rm em}~({\rm ns})$	$k_{\rm q}^{\rm b} \times 10^8 \; ({\rm M}^{-1} \; {\rm s}^{-1})$	$\eta_{\mathrm{q}}^{\mathrm{c}}\left(\% ight)$
Complexes	Х	PR ₃	L			
1a	Н	PPh ₃	PPh ₃	640	12.7	98
1b	Н	P(OEt) ₃	PPh ₃	350	8.0	93
1c	Me	P(OEt) ₃	PPh ₃	560	9.9	97
1d	Н	P(OEt) ₃	P(OEt) ₃	250	4.2	84
1e	Me	P(OEt) ₃	P(OEt) ₃	340	-	-
1f	Н	P(OEt) ₃	ру	12	-	-
1g	Н	P(OEt) ₃	MeCN	nd ^a	-	-
1h	Н	P(OEt) ₃	Cl^{-}	nd ^a	-	-
1i	CF_3	P(OEt) ₃	P(OEt) ₃	20	-	-

Table 3 Emission lifetimes of 1^a and quenching with BNAH

^a Measured at room temperature in DMF

^b Quenching rate constants of the emission with BNAH

 $^{\rm c}$ Quenching efficiency $\eta_{\rm q}$ shows what percentage of the emission was quenched in the presence of 50 mM of BNAH

$$1^* + BNA_2 \longrightarrow 1^- + BNA_2^{*+} \xrightarrow{fast} 1 + BNA_2$$

BNA⁺ + BNA^{*}

Scheme 3 An efficient back electron transfer from the reduced photocatalyst 1^- to the oxidized species $BNA_2^{\bullet+}$

of BNA_2 in the reaction solution and the formation rates of CO and H_2 also slow down.

Role of TEOA: a suitable base for the photocatalytic reactions

Kinetic considerations indicated that only 1 % of the excited state of **1d** was quenched by TEOA whereas BNAH quenched 84 %. This is because TEOA has a lower reduction power as compared to BNAH [$E_{1/2}$ (TEOA/TEOA^{+•}) = +0.67 V and $E_{1/2}$ (BNAH/BNAH^{•+}) = +0.28 V versus Ag/AgNO₃]. However, TEOA is an essential additive for efficient photocatalytic reactions (Table 4). In similar experimental conditions except for the absence of TEOA, **1d** could not work as the photocatalyst for both H₂ and CO production. Addition of a protic additive (1.5 M of proton source), i.e., EtOH and H₂O, or a weaker base (0.5 M), 2,6-di-*tert*butylpyridine (pyBu₂) and pyridine (py), could not improve the photocatalysis of **1d**. In contrast, in the presence of a stronger base (0.5 M), i.e., TEA and 2,2,6,6tetramethylpiperidine (pipMe₄), the photocatalytic formation of H₂ and CO proceeded. These results strongly indicate that the photocatalytic system requires a relatively strong base. The deprotonation process of the one-electron-oxidized BNAH (BNAH^{•+}) giving BNA[•] should be essential for promoting the photocatalytic reactions, because, if the process is slow, back electron transfer from the

Base or additive	p <i>K</i> a	TN	
		$\overline{{\rm H_2}^{\rm a}}$	CO ^b
_		0.3	0.0
EtOH ^c		_	0.0
H_2O^d		0.6	0.1
pyBu ^e ₂	3.58	-	0.0
py ^e	5.19	0.4	0.0
TEOA ^e	7.82	10.2	8.2
TEA ^e	10.87	6.0	2.4
pipMe ₄ ^e	11.07	0.7	10.2

Table 4 Photocatalytic ability of 1d under Ar or CO2 for 6 h in the presence of an amine

^a Under Ar atmosphere

^b Under CO₂ atmosphere

^c 1.5 M EtOH was adopted

^d 0.75 M H₂O was adopted

e 0.5 M base was adopted

one-electron reduced complex $1^{\bullet-}$ to BNA^{•+} competes with the following reaction processes of $1^{\bullet-}$, giving either H₂ or CO [18–20]. The results and considerations described above lead to the conclusion that TEOA mainly works as a base and BNAH is the main reductant.

Detection of reaction intermediate in the photocatalytic reactions traced by NMR

¹H, ¹³C, and ³¹P NMR measurements were conducted to follow changes in **1** under the photocatalytic reaction conditions. A DMF- d_7 solution containing **1d** (40 µmol, 10 mM), TEOA (0.5 M), and BNAH (2.0 × 10² µmol, 50 mM) was irradiated with a >400-nm light under vacuum or under ¹³CO₂ (ca. 500 Torr) atmosphere. The solutions obtained were subjected to NMR measurements after they were kept under dim light for several hours until the paramagnetic species disappeared.

In the case of vacuum, the starting complex **1d** was not detected and some aromatic protons of unidentified products were observed in the ¹H NMR spectrum of the sample after 18-h irradiation (Fig. 1). Furthermore, the ³¹P NMR spectrum suggested presence of free $P(OEt)_3$ (138.3 ppm). Some unidentified signals at 114.8, 121.5, and 123.6 ppm were also observed in addition to a strong signal (116.8 ppm), which was similar to that of **1d** (Fig. 2). From these observations, it was concluded that the starting rhenium(I) complex **1d** was converted into other rhenium(I) species without the original aromatic diimine ligand, and some portions of the phosphine ligands were removed from the complex during the photocatalytic reaction.

The ¹³C NMR spectrum of the sample after 14 h of irradiation under ¹³CO₂ atmosphere showed not only an intense signal of ¹³CO₂ (125.0 ppm) but also a new



Fig. 1 ¹H NMR spectrum of a DMF- d_7 solution containing the complex 1d (12.5 mM), TEOA (0.625 M), and BNAH (62.5 mM) irradiated for 18 h under vacuum



Fig. 2 ³¹P NMR spectrum of a DMF- d_7 solution containing the complex 1d (12.5 mM), TEOA (0.625 M), and BNAH (62.5 mM) irradiated for 18 h under vacuum

signal at 184.6 ppm, corresponding to ¹³CO (Fig. 3). This clearly indicates that the carbon source of the CO formed is CO₂. The ¹H NMR spectrum of the irradiated sample showed the signals of the starting photocatalyst **1d** and smaller signals in the aromatic proton region caused by the other rhenium(I) complexes (Fig. 4). It also displayed the complete consumption of BNAH and formation of BNA₂. In the ³¹P NMR spectrum of the same sample (Fig. 5), the strong signal (116.8 ppm) of the complex **1d** remained as the main peak, with new and smaller signals observed at 138.3 and 116.0 ppm. The former is assigned to the free P(OEt)₃ ligand released from **1d**, and the latter might be attributed to another rhenium(I) complex with a



Fig. 3 13 C NMR spectrum of a DMF- d_7 solution containing 1d (10 mM), TEOA (0.5 M), and BNAH (50 mM) irradiated for 14 h under 13 CO₂ atmosphere



Fig. 4 ¹H NMR spectrum of a DMF- d_7 solution containing **1d** (10 mM), TEOA (0.5 M), and BNAH (50 mM) irradiated for 14 h under ¹³CO₂ atmosphere. + Represents new small signals in the aromatic proton region



Fig. 5 ³¹P NMR spectrum of a DMF- d_7 solution containing **1d** (10 mM), TEOA (0.5 M), and BNAH (50 mM) irradiated for 14 h under ¹³CO₂ atmosphere. + Represents a new signal at 116.0 ppm

phosphine ligand, which may have formed as a product of a minor process or processes during the photocatalytic cycles. These results strongly indicate that 1d is stable during the photocatalytic reduction of CO₂. Since no significant increase in the intensity of the peaks corresponding to the two CO ligands at 199.6 ppm was

observed, the exchange of the CO ligands with the reduction product of CO_2 is considered as a minor process during the photocatalytic reaction.

It is noteworthy that, since the H_2 evolution was almost completely suppressed under CO_2 atmosphere, there should be common precursors between the photocatalytic H_2 formation and CO_2 reduction.

Conclusion

The photocatalytic properties of a series of rhenium(I) biscarbonyl complexes were investigated under Ar and CO₂ atmospheres. The rhenium(I) complexes behaved as "chameleon-like" photocatalysts, i.e., they can photocatalyze H₂ evolution under Ar atmosphere and CO₂ reduction under CO₂ atmosphere with no H₂ evolution. The photocatalytic abilities were highly dependent on the ligands of the complexes, which can be mainly rationalized by the quenching efficiencies of the ³MLCT excited state of the complexes with BNAH. Mechanistic studies clearly indicated that BNAH works as a one-electron donor and TEOA mainly works as a base; both of them are necessary for the photocatalytic reactions.

References

- 1. H. Takeda, K. Koike, T. Morimoto, H. Inumaru, O. Ishitani, Adv. Inorg. Chem. 62, 137 (2011)
- 2. T. Yui, Y. Tamaki, K. Sekizawa, O. Ishitani, Top. Curr. Chem. 303, 151 (2011)
- 3. H. Takeda, O. Ishitani, Coord. Chem. Rev. 254, 346 (2010)
- 4. A.J. Morris, G.J. Meyer, E. Fujita, Acc. Chem. Res. 42, 1983 (2009)
- 5. A. Kudo, Y. Miseki, Chem. Soc. Rev. 38, 253 (2009)
- 6. K. Maeda, K. Domen, J. Phys. Chem. C 111, 7851 (2007)
- 7. H. Ozawa, K. Sakai, Chem. Commun. 47, 2227 (2011)
- 8. J.L. Dempsey, B.S. Brunschwig, J.R. Winkler, H.B. Gray, Acc. Chem. Res. 42, 1995 (2009)
- 9. H. Takeda, K. Koike, H. Inoue, O. Ishitani, J. Am. Chem. Soc. 130, 2023 (2008)
- 10. M. Tajik, C. Detellier, J. Chem. Soc. Chem. Commun. 1824 (1987)
- 11. C. Pac, K. Ishii, S. Yanagida, Chem. Lett. 765 (1989)
- K. Koike, J. Tanabe, S. Toyama, H. Tsubaki, K. Sakamoto, J.R. Westwell, F.P.A. Johnson, H. Hori, H. Saitoh, O. Ishitani, Inorg. Chem. 39, 2777 (2000)
- H. Tsubaki, A. Sekine, Y. Ohashi, K. Koike, H. Takeda, O. Ishitani, J. Am. Chem. Soc. 127, 15544 (2005)
- H. Tsubaki, A. Sugawara, H. Takeda, B. Gholamkhass, K. Koike, O. Ishitani, Res. Chem. Intermed. 33, 37 (2007)
- K. Kalyanasundaram, T. Colassis, R. Humphry-Baker, P. Savarino, E. Barni, E. Pelizzetti, M. Graetzel, J. Am. Chem. Soc. 111, 3300 (1989)
- 16. K. Kano, T. Matsuo, Bull. Chem. Soc. Jpn. 49, 3269 (1976)
- 17. Y. Ohnishi, M. Kitami, Bull. Chem. Soc. Jpn. 52, 2674 (1979)
- 18. F.M. Martens, J.M. Verhoeven, Recl. Trav. Chim. Pays-Bas 100, 228 (1981)
- 19. C. Pac, Y. Miyauchi, O. Ishitani, M. Ihama, M. Yasuda, H. Sakurai, J. Org. Chem. 49, 26 (1984)
- 20. O. Ishitani, S. Yanagida, S. Takamuku, C. Pac, J. Org. Chem. 52, 2790 (1987)