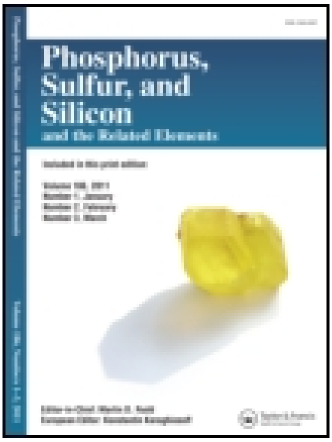

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Uronium Hydrogen Sulfate/Urea-Hydrogen Peroxide as a Green and Metal-Free Catalytic System for the Efficient, Chemo-, and Homoselective Oxidation of Sulfides to Sulfoxides

Alireza Hasaninejad [a] , Gholamabbas Chehardoli [b] , Mohammad Ali Zolfigol [c] & Abbas Abdoli [c]

[a] Department of Chemistry, Faculty of Sciences , Persian Gulf University , Bushehr, Iran

[b] School of Pharmacy , Hamedan University of Medical Sciences , Hamedan, Iran

[c] Faculty of Chemistry , Bu-Ali Sina University , Hamedan, Iran

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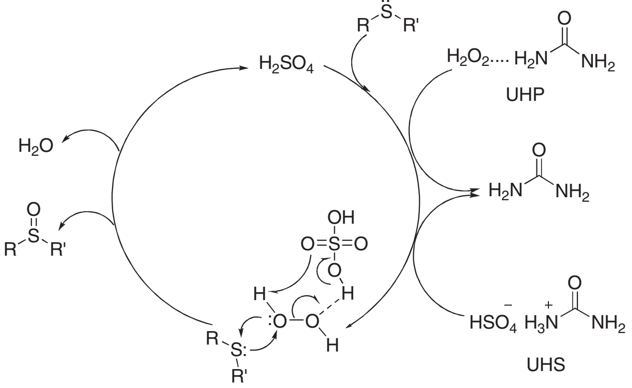
URONIUM HYDROGEN SULFATE/UREA-HYDROGEN PEROXIDE AS A GREEN AND METAL-FREE CATALYTIC SYSTEM FOR THE EFFICIENT, CHEMO-, AND HOMOSELECTIVE OXIDATION OF SULFIDES TO SULFOXIDES

Alireza Hasaninejad,[1] Gholamabbas Chehardoli,[2] Mohammad Ali Zolfigol,[3] and Abbas Abdoli[3]
[1]*Department of Chemistry, Faculty of Sciences, Persian Gulf University, Bushehr, Iran*
[2]*School of Pharmacy, Hamedan University of Medical Sciences, Hamedan, Iran*
[3]*Faculty of Chemistry, Bu-Ali Sina University, Hamedan, Iran*

GRAPHICAL ABSTRACT



Abstract *An efficient, chemoselective, and metal-free oxidation of sulfides is described using urea-hydrogen proxide (UHP) adduct as a stable, inexpensive, and easily handled reagent in the presence of uronium hydrogen sulfate (UHS). Both UHP and UHS release urea as an environmentally benign byproduct. The advantages of the described method are generality, chemoselectivity, short reaction time, low cost, high atom economy, and green chemistry protocols.*

Keywords Chemoselective; oxidation; sulfides; sulfoxides; urea hydrogen peroxide (UHP); uronium hydrogen sulfate (UHS)

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Address correspondence to Gholamabbas Chehardoli, School of Pharmacy, Hamedan University of Medical Sciences, 65178 Hamedan, Iran. E-mail: chehardoli@umsha.ac.ir, cheh1002@gmail.com

INTRODUCTION

The art of performing efficient chemical transformations by catalytic processes avoiding toxic byproducts, expensive catalysts, and difficult–to-handle reagents represents a fundamental target of modern organic synthesis.[1] Also, the search for safe and easily accessible sources of primary oxidants is of great interest in chemistry. In particular, reduction in the amount of hazardous wastes in the reactions would be welcome in term of green chemistry.[2]

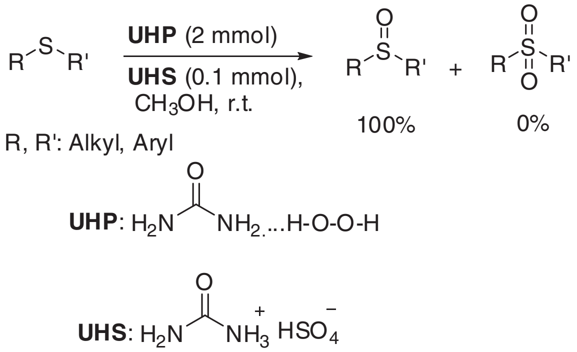
Sulfoxides are fascinating organic molecules. These compounds have immense applications in the field of sulfur-substituted natural products and also in pharmaceuticals.[3] Because of the multifarious conversions of sulfoxides into a myriad of organosulfur compounds, they are extremely important to organic chemists. Much exploration has been carried out to understand their importance as chiral auxiliary in asymmetric synthesis[4] and particularly in carbon–carbon bond forming reactions.[5] Oxidation of sulfides is a general method for synthesis of sulfoxides.[6] Several methods have been introduced for this purpose.[7] However, most of the existing methods use sophisticated reagents, complex catalysts, toxic metallic compounds, or rare oxidizing agents that are difficult to prepare. Also, many of these procedures also suffer from poor selectivity.[7]

Urea hydrogen peroxide (UHP) is a white crystalline solid, formed by strong hydrogen bonding between urea and hydrogen peroxide in a 1:1 proportion[8]; it is easily handled, without the need for special precautions. It is commercially available, cheap, and can be stored for months at +4°C without any change in the oxygen content, and, in contrast to other oxidizing agents, UHP is the most attractive oxidant from an environmental viewpoint and shows safety in operation.[9] However, UHP alone cannot oxidize sulfides to sulfoxides at room temperature.[10] Therefore, there is a need to improve the ability of UHP for the selective conversion of sulfides to sulfoxides. Uronium hydrogen sulfate (UHS) is a very cheap and stable solid acid whose application in synthetic methodology has already been reported. Another advantage of the using of UHS is reduction of toxic byproducts, because the uranium cation is a green and safe waste.[11]

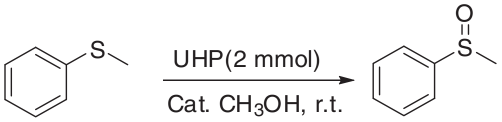
RESULTS AND DISCUSSION

In continuation of our interest in the development of synthetic methodologies for organic functional group transformations,[12] H_2O_2 adducts, catalytic oxidation reactions,[13,14] and the application of UHS in organic synthesis,[11] in the present work we have used a stable solid urea/hydrogen peroxide addition compound (UHP) as the safe and eco-friendly oxidant for selective oxidation of thioethers into the corresponding sulfoxides in the presence of a catalytic amount of UHS as a very cheap, stable, and recyclable solid acid catalyst (Scheme 1).

Initially, in order to optimize reaction conditions, the oxidation of methyl phenyl sulfide into methyl phenyl sulfoxide using UHP in methanol was selected as a model reaction (Scheme 2). The results are summarized in Table 1. At first, a model run was carried out with methyl phenyl sulfide in the absence of catalyst. It was found that even using a 10-fold excess of H_2O_2, the reaction was not completed after a long reaction time (4 h) (Table 1, entries 1 and 2). Then, the effects of various amounts of UHS as a catalyst on the reactivity of UHP for the oxidation of methyl phenyl sulfide in methanol at room temperature have been studied. The results show that UHSis an effective catalyst for the sulfoxidation reaction with high yield and short reaction time. Also in this study, the effects



Scheme 1



Scheme 2

of various amounts of UHP were checked (Table 1, entries 3–7). As is shown in Table 1, higher yield and acceptable reaction time for sulfoxidation reactions were obtained when 0.1 mmol UHS was used for the reaction of 1 mmol methyl phenyl sulfide and 2 mmol UHP in 5 mL CH_3OH at room temperature (Table 1, entry 4).

Table 1 Oxidation of benzyl phenyl sulfide (1 mmol) to the corresponding sulfoxide using UHP in the presence of representative catalyst in CH_3OH (5 mL) at room temperature

Entry	Catalyst (mmol)	UHP (mmol)	Time (h)	Yield of sulfoxide (%)[a]
1	—[b]	2	4	Trace
2	—[b]	10	4	30
3	UHS (0.1)	1	50 (min)	97
4	UHS (0.1)	2	30 (min)	95
5	UHS (0.2)	2	25 (min)	97
6	UHS (0.4)	4	20 (min)	95
7	UHS (0.1)	4	25 (min)	86
8	$AlCl_3$ (0.5)	2	3	—[c]
9	$Al(HSO_4)_3$ (0.5)	2	3	—[c]
10	Na_2HPO_4 (0.5)	2	3	Trace
11	$ZnCl_2$ (0.3)	2	3	35
12	$CaCl_2.2H_2O$ (0.5)	2	3	20
13	Al_2O_3 (0.5 g)	2	3	Trace
14	SiO_2 (0.5 g)	2	3	20
15	$ZrCl_4$ (0.2)	2	3	Trace
16	$NaHSO_4$	2	3	Trace
17	ZnO (0.5)	2	3	Trace
18	MgO (0.5)	2	3	Trace
19	NH_2SO_3H	2	3	–[c]

[a] Isolated yield.
[b] In the absence of catalyst.
[c] Reaction did not complete.

Table 2 Solvent effect in the oxidation of benzyl phenyl sulfide using UHP in the presence of UHS as catalyst

Entry	Solvent	Time (h)	Yield (%)[a]
1	CH_2Cl_2	5	0
2	$CHCl_3$	5	0
3	EtOAc	5	Trace
4	Acetone	24	35
5	*n*-Hexane	5	0
6	THF	5	0
7	CH_3CN	24	40
8	DMF	5	0
9	CH_3OH	30 (min)	100
10	C_2H_5OH	24	50
11	H_2O	24	Trace

[a]Conversion.

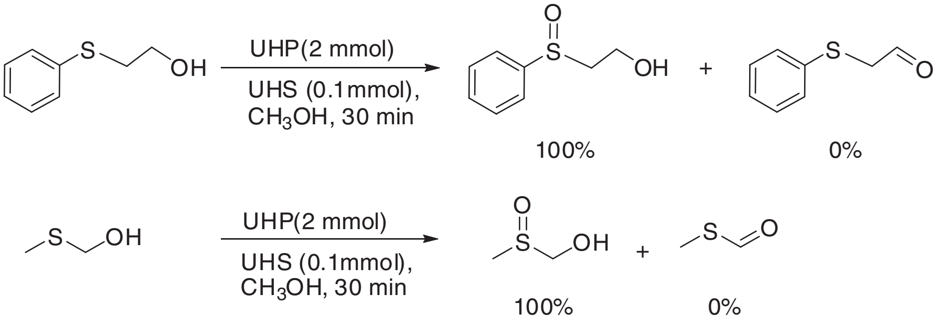
In the next step, we investigated the effect of various solvents on the progress of reactions. As is evident from Table 2, the best choice for the selective sulfoxidation by the described procedure is methanol.

In order to investigate the scope and limitations of this procedure, the oxidation of other sulfides was also studied. A good range of substrates such as aryl alkyl, diaryl, and dialkyl sulfides have been selectively oxidized to their corresponding sulfoxides (Table 3).

CHEMO- AND HOMOSELECTIVITY[14g]

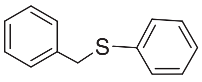
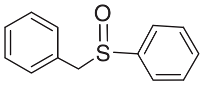
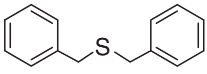
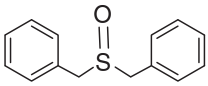
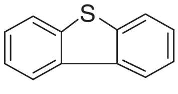
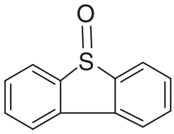
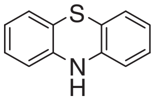
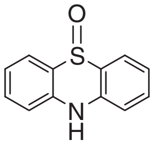
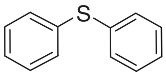
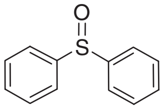
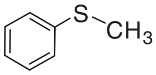
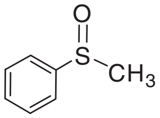
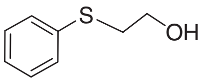
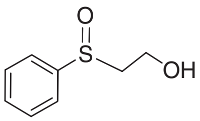
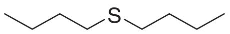
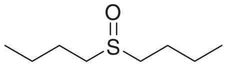
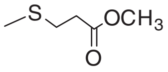
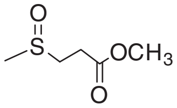
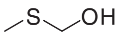
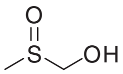
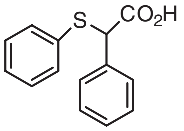
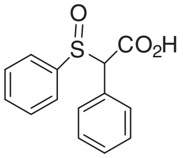
It is noteworthy that sulfides containing functional groups such as alcohol, acid, and ester are oxidized selectively without any interference from these groups (Table 3, entries 7, 9, and 10). For example, two sulfides containing a hydroxyl group were subjected to the sulfoxidation reaction, but alcohol oxidation did not occur under mentioned conditions, and the primary hydroxyl group remained intact in the course of the reaction (Scheme 3).

Also, it is noteworthy that the mentioned oxidizing system allowed the homoselective[14g] oxidation of *bis*-(methyl thio)-methane to its monosulfoxide derivative without further oxidation to *di*-sulfoxide or sulfone (Scheme 4, entry 12).



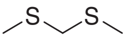
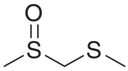
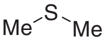
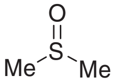
Scheme 3

Table 3 Chemoselective oxidation of sulfides (1 mmol) to their corresponding sulfoxide using UHP (2 mmol) in the presence of UHS (0.1 mmol) as catalyst in CH_3OH (5 mL) at room temperature

Entry	Substrate	Product	Time (min)	Yield (%)[a]	Ref.[b]
1			50	92	14d
2			30	95	7j
3			6h	85	14e
4			3h	—[c]	—
5			120	95	7k
6			30	95	14e
7			50	88	14e
8			40	80	7m
9			20	82	7j
10			45	91	14f
11			50	90	14d

(Continued on next page)

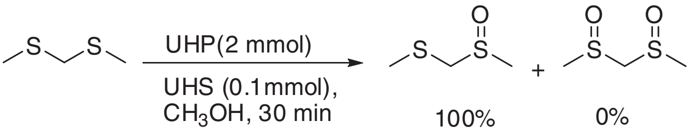
Table 3 Chemoselective oxidation of sulfides (1 mmol) to their corresponding sulfoxide using UHP (2 mmol) in the presence of UHS (0.1 mmol) as catalyst in CH_3OH (5 mL) at room temperature *(Continued)*

Entry	Substrate	Product	Time (min)	Yield (%)[a]	Ref.[b]
12			20	87	14f
13			25	97	14e

[a]Isolated yields.

[b]All products are known and were identified using comparison of their physical and spectral data (IR, NMR, or MS) with those reported in the literature.

[c]Undesired products.



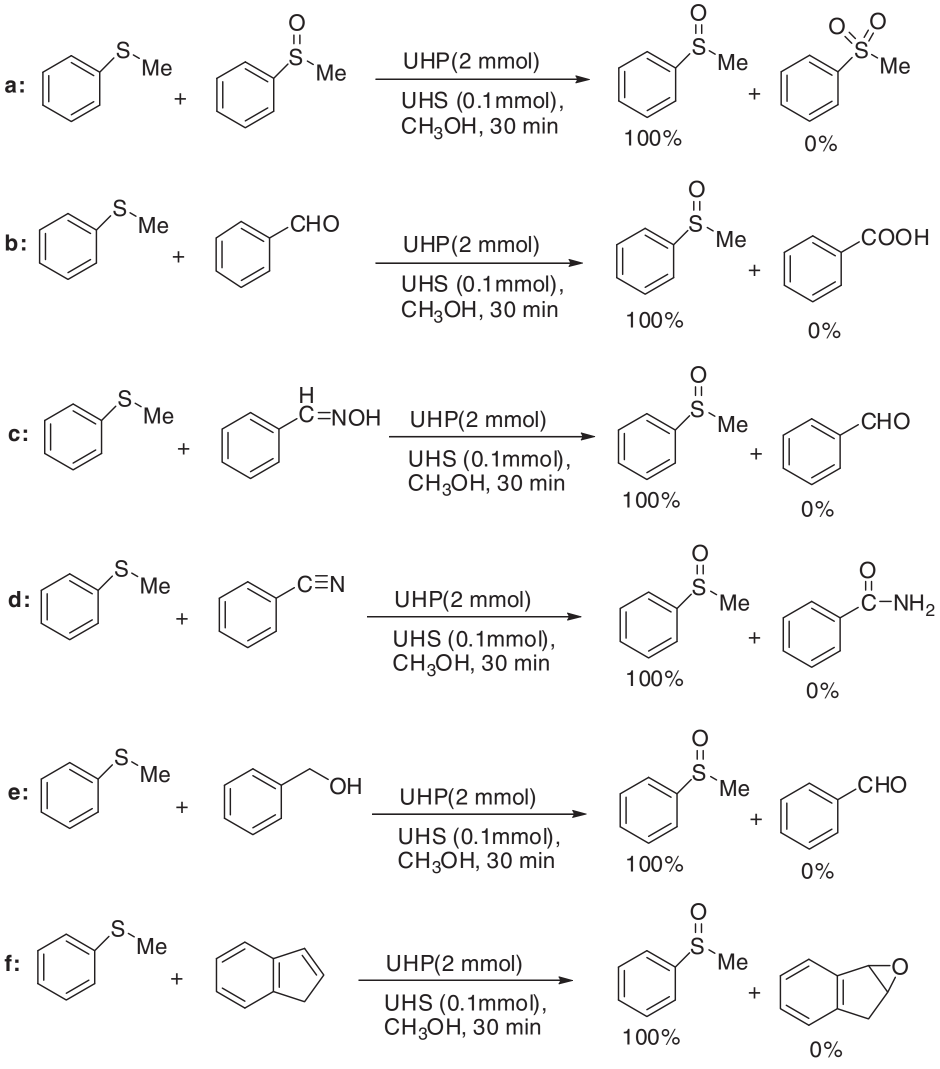
Scheme 4

To further determine the chemoselectivity of the described system, some competitive reactions were designed. We have monitored competitive oxidation of sulfides in the presence of sulfoxide, aldehyde, oxime, nitrile, benzylic alcohol, and alkene. The results show that this method is applicable for the chemoselective oxidation of sulfides to sulfoxides in the presence of the previously mentioned functional groups and can be considered as a useful practical achievement in oxidation of sulfides to sulfoxides without the overall oxidation (Scheme 5).

In Table 4, the results of the oxidation of benzyl phenyl sulfide by our method are compared with those reported by other methods. As can be seen, this method is superior to some previously reported methods in terms of chemoselectivity, yields, reaction times, and amount of the catalyst/reagent for successful oxidation without the need of complex catalysts, hazardous and unstable oxidants, microwave, or toxic metallic compounds.

The observation of oxidation of benzyl phenyl sulfide and dibenzyl sulfide to the corresponding sulfoxide (Table 3, entries 1 and 2) indicates that the reaction proceeds via an oxygen transfer mechanism. If the reaction involved electron transfer instead of oxygen transfer, substantial amounts of benzaldehyde would have been formed.[15] According to a literature survey,[7k,16] the proposed mechanism for the oxidation of sulfide to the corresponding sulfoxide using UHP in the presence of UHS is outlined as shown in Scheme 6.

In summary, we found that UHS efficiently catalyzed chemo- and homoselective oxidation of sulfides to sulfoxides by the UHP adduct at room temperature without overoxidation to sulfone. Moreover, the simplicity of the operation, low cost of the reagents, and high yields of sulfoxides are the advantages of this method. Also, its compatibility with sensitive functionalities such as ester, aldehyde, oxime, nitrile, and double bonds with regard to economic and ecological consideration allows us to believe that this method represents a valuable alternative to the existing reagents reported in the literature.



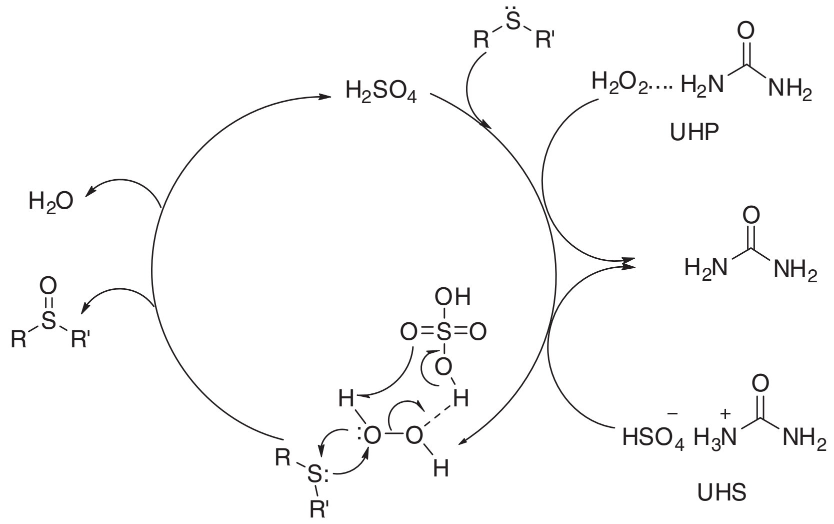
Scheme 5

EXPERIMENTAL

Chemicals were purchased from Fluka, Merck, and Aldrich chemical companies. Yields refer to isolated pure products. The oxidation products were characterized by comparison of their spectral (IR, ^{1}H NMR, and ^{13}C NMR) and physical data with those authentic samples, which were produced by other reported procedures.[7,14]

General Procedure for the Oxidation of Sulfides

A mixture of sulfides (1 mmol), UHP (2 mmol), and UHS (0.1 mmol) in methanol (5 mL) was vigorously stirred for the appropriate time (see Table 3). The progress of the reaction was monitored by TLC. When the reaction was completed, the reaction mixture



Scheme 6 Proposed mechanism for the oxidation of sulfide to the corresponding sulfoxide with urea hydrogen peroxide (UHP) in the presence of uronium hydrogen sulfate (UHS) as catalyst.

was filtered and the residue was washed with CH_3OH (2 × 5 mL). Then anhydrous Na_2SO_4 (4 g) was added to the filtrate and filtered off after 15 min. The solvent was evaporated, and the crude product was purified by a short column chromatography on silica gel with EtOAc/*n*-hexane (1:5 to 1:2).

Table 4 Comparison of oxidation of benzyl phenyl sulfide (1 mmol) by the UHP/UHS system with some of those reported methods in the literature

Entry	Reagent (oxidant/substrate)	Time (min)	Yield (%)		Ref.
			Sulfoxide	Sulfone	
1	UHP/UHS/CH_3OH/rt (2:0.1)	50	92	—[a]	This work
2	BTPPDC/$AlCl_3$/CH_3CN/reflux (1.5:1)	90	93	—[a]	7i
3	BTPPDC/$AlCl_3$/MW (1.2:1)	2	94	—[a]	7i
4	$NaIO_4$/wet SiO_2/MW (1.7:1)	2.5	83	—[a]	7o
5	$PhCH_2PPh_3HSO_5$/CH_3CN/reflux (1.5:1)	12 (h)	88	—[a]	7p
6	$Ba(MnO_4)_2$/CH_3CN/reflux (6:1)	4 (h)	88	—[a]	7q
7	CAN/wet SiO_2/CH_2Cl_2/rt (2:1)	45	96	—[a]	7r
8	BTPPDC/$AlCl_3$/MW (2.5:1)	2.5	—[a]	97	7i
9	CPCC/$AlCl_3$/CH_3CN/reflux (1:1)	75	93	—[a]	7j
10	CPCC/$AlCl_3$/MW (0.8:1)	1.5	92	—[a]	7j
11	H_2O_2/$ZrCl_4$/CH_3OH (7:2)	2	96	—[a]	7n
12	H_2O_2/Silica sulfuric acid/CH_3CN/rt (1:0.1 g)	40	96	—[a]	7k
13	H_2O_2/Amberlyst 15/CH_3OH/rt (2:0.5)	6.5 (h)	95	—[a]	7l
14	H_2O_2/Amberlyst IR-400/CH_3OH/rt (3:0.5)	7.5 (h)	92	—[a]	7l
15	HIO_3/wet SiO_2/solvent-free 50°C (1:3)	170	93	—[a]	7m
16	H_2O_2/Silica-based tungstate/CH_2Cl_2, CH_3OH (4:0.1)	4 (h)	83	—[a]	7s

[a] None reported.

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