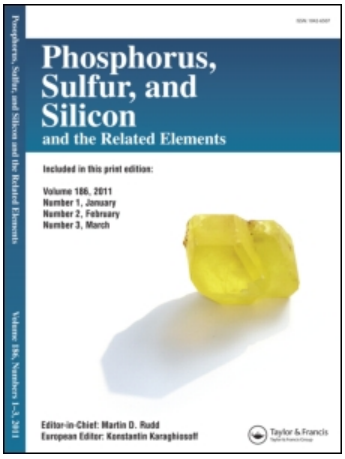

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Efficient Chemoselective Mild Deprotection of S,Sand S,O-Acetals and Ketals with Electrophilic Halogens

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Efficient Chemoselective Mild Deprotection of S,S- and S,O-Acetals and Ketals with Electrophilic Halogens

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A novel and simple method for the chemoselective deprotection of S,S- and S,O-acetals and ketals in the presence of their O,O-analogs with electrophilic halogens to their corresponding carbonyl compounds is described using N-bromosuccinimide, N-chlorosuccinimide, 2,4,4,6-tetrabromo-2,5-cyclohexen-1-one, trichlorocyanuric acid, or molecular bromine in aqueous acetonitrile. The use of these reagents in the presence of hydrated silica gel provide efficient, novel, and mild procedures for the deprotection of cyclic and acyclic O,O-, S,S-, and S,O-acetals and ketals in excellent yields in short reaction times.

Keywords Chemoselective; deprotection; electrophilic halogens; hydrated silica gel; S,O-acetals and ketals; S,S-acetals and ketals

Thioacetals and ketals as important intermediates in multistep natural product synthesis[1,2] are stable compounds in both acidic and basic conditions, and their deprotection to carbonyl groups, especially under mild reaction conditions, are considered an important transformation reaction in organic synthesis.

The methods employed for the deprotection of S,S-acetals include (a) hydrolysis in the presence of different transition-metal ions,[3–10] (b) oxidation of sulfur to a higher oxidation state with various oxidizing agents,[11–18] and (c) hydrolysis[19–21] and use of alkylating agents.[21b]

A review has been published on the methods of preparation and cleavage of 1,3-dithiolanes.[22]

Although some of the reported methods in the literature can be applied for the chemoselective deprotection of O,O- acetals and ketals in

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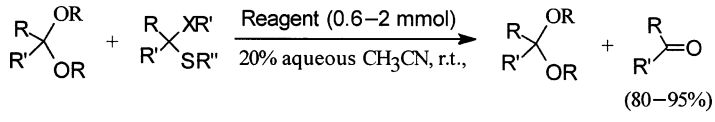
the presence of S,S- and S,O- analogs, to the best of our knowledge, there is no report available in the literature concerning reverse chemoselectivity. In our previous research, we reported catalytic and chemoselective deprotection of S,S- and S,O-acetals and ketals in the presence of their O,O-analogs with electrophilic halogens under neutral conditions using DMSO as oxygen transfer.[23]

Now we report a novel and a highly chemoselective protocol for the deprotection of S,S- and S,O- acetals and ketals in the presence of their O,O- analogs. In this method, N-Bromosuccinimide (NBS), N-Chlorosuccinimide (NCS), 2,4,4,6-Tetrabromo-2,5-Cyclohexadien-1-One (TABCO), Trichlorocyanuric Acid (TCCA), or bromine are used as sources of electrophilic halogens in aqueous acetonitrile. Halogens carried by these reagents interact with the soft sulfur atom in S,S- and S,O- acetals and ketals and water as a hydrolyzing agent and source of oxygen. The ease of halogenation of the sulfur atom in S,S- and S,O-acetals and ketals in comparison with the oxygen in the O,O-analogs let the reaction occur with high chemoselectivity for the deprotection of S,S- and S,O-acetals and ketals at r.t. (Scheme 1).

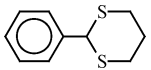
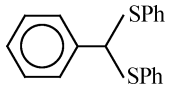
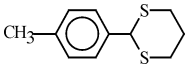
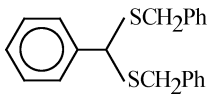
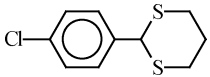
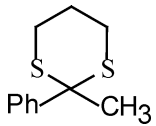
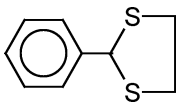
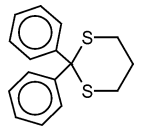
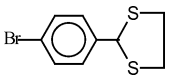
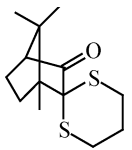


Reagent = NBS, TABCO, NCS, TCCA, Br_2;
R = R' = Aryl, Alkyl
R''= Aryl, Alkyl, [-$(CH_2)_n$-, n = 2, 3; X = O, n = 2]
X = O, S

SCHEME 1

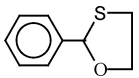
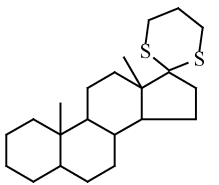
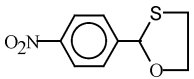
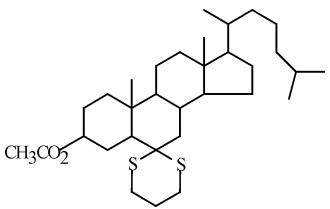
We first performed the deprotection reaction of 2-phenyl-1,3-dithiane using NBS, NCS, TABCO, TCCA, and Br_2 reagent in aqueous acetonitrile (Scheme 1, condition A) at r.t. It was observed that the rate of the reactions and the molar ratio of dithiane/reagent (1/1.3) with these reagents were nearly the same and the reactions were completed after 15 min to produce benzaldehyde in a 91–95% yield. Therefore, we applied similar reaction conditions to other S,S- and S,O- acetals and ketals. Experimentally it was observed that at least one molar equivalent of electrophilic halogens for S,S- acetals and ketals and half an equimolar amount for S,O- acetals and ketals were required. The results of this investigation are shown in Table I. By these methods, cyclic and acyclic thioacetals and also 1,3-oxathiolanes carrying electron-withdrawing or electron-donating groups on their aromatic rings were converted into their corresponding carbonyl compounds easily at r.t. within 10–80 min. 2,2-diphenyl-1,3-dithiane, and mono-1,3-dithiane of camphor quinone

TABLE I Deprotection of S,S- and S,O-Acetals and Ketals With TABCO in Aqueous Acetonitrile at r.t.

Entry	Substrate	Sub./TABCO (ratio)	Time (min)	Yield (%)[a]	Entry	Substrate	Sub./TABCO (ratio)	Time (min)	Yield %[a]
1		1/1.3	15	92	8		1/1.3	10	92
2		1/1.3	15	93	9		1/1.4	10	91
3		1/1.3	20	91	10		1/1.2	30	89[b]
4		1/1.3	15	90	11		1/2.0	45	87
5		1/1.3	25	89	12		1/1.8	50	89

(Continued on next page)

TABLE I Deprotection of S,S- and S,O-Acetals and Ketals With TABCO in Aqueous Acetonitrile at r.t. *(Continued)*

Entry	Substrate	Sub./TABCO (ratio)	Time (min)	Yield (%)[a]	Entry	Substrate	Sub./TABCO (ratio)	Time (min)	Yield %[a,]
6		1/0.8	15	94	13		1/1.8	55	85[c]
7		1/1.0	30	95	14		1/2.0	80	80[c]

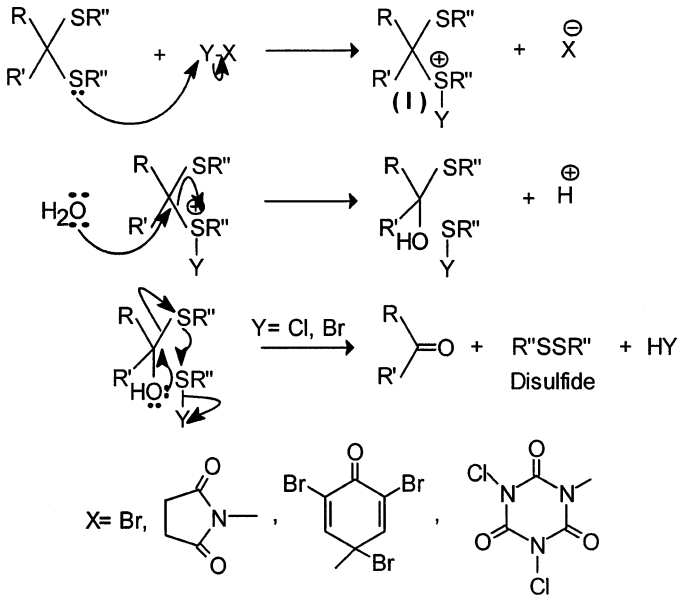
[a]Isolated yield.

[b]When the reaction was performed in the presence of pyridine (1.5 equiv.), only 7% of phenacyl bromide was formed. In the absence of pyridine, phenacyl bromide was formed in 45% yield.

[c]The reaction was performed in the presence of pyridine (1.5 equiv.).

as examples of hindered dithioacetals were also converted into benzophenone and camphor quinone, respectively, with good yields (Table I, Entries 11, 12). In the case of S,S- and S,O-acetals and ketals carrying enolizable hydrogens, the addition of pyridine (1.5 equimolar) to the reaction mixture greatly reduced the formation of α-brominated carbonyl compounds in the reaction mixtures (Table I, entries 10, 13, and 14).

The deprotection of structurally different thioacetals produced the corresponding aldehydes or ketones together with structurally different organo-sulfur compounds. The production of disulfides from acyclic dithioacetals in the presence of electrophilic halogens were also detected and isolated in high yields from the reaction mixtures under this study. The structure of the isolated disulfides (II) was identified by comparison of their physical data with authentic samples.[24,25] We have proposed a mechanism for the production of the disulfides and carbonyl compounds from acyclic dithioacetals (Scheme 2).



SCHEME 2

Easily polymerizable 1,2-dithiacyclopentane, which is stable in dilute solutions, is produced in the reaction of 1,3-dithianes in the presence of electrophilic halogens. The structure of this compound was identified by comparison of its ^{1}H and ^{13}C NMR spectral data with those reported in the literature[24] and also by the synthesis of

1,2-dithiacyclopentane by an intramolecular coupling of 1,3-propanedithiol by the known procedure.[25]

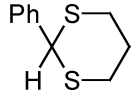
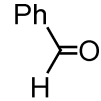
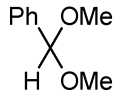
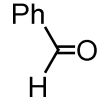
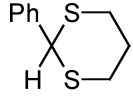
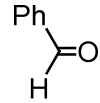
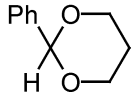
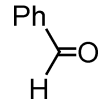
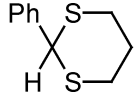
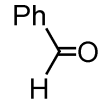
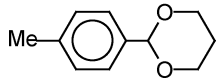
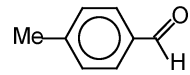
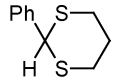
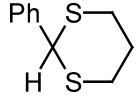
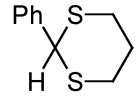
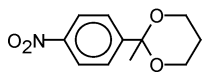
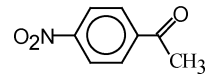
Cyclic 1,3-oxathiolanes under similar reaction conditions, in addition to carbonyl compounds, bis(2-hydroxyethyl) disulfide, was also produced in high yields. The structures of the isolated bis(2-hydroxyethyl) disulfide was identified by comparison of its physical data with those reported in the literature[24,25] and also by its synthesis from 2-mercaptoethanol by the reported procedure.[26]

1,3-dithiolanes in the process of deprotection reactions produce a cyclic compound with four sulfur atoms. Easily polymerizable 1,2,5,6-tetrathiacylooctane was detected in the reaction mixture and identified by spectroscopic data and also by its synthesis from the coupling reaction of 1,2-ethanedithiol.[24,25] In this mechanism, 1,2,5,6-tetrathiacylooctane can be produced either by the intermolecular reaction between (I) and a molecule of 1,3-dithiolane or through the formation of unstable intermediate 1,2-dithiacyclopentane[24,25] that is produced in the reaction by intramolecular coupling of (I).

We have also studied the possibility of using these reagents for the chemoselective deprotection of S,S-, and S,O-acetals and ketals in the presence of their O,O-analogs in aqueous acetonitrile in the presence of pyridine as an acid scavenger. Pyridine does not show a considerable effect on the rate of the reactions but greatly prevents deprotection of O,O-acetals and ketals in the presence of their S,S- and S,O-analogs by the removal of the produced acid. The results of this investigation using TABCO as a representative reagent are shown in Table II. By this method, 2-phenyl-1,3-dithiane was deprotected in the presence of benzaldehyde dimethyl acetal with the ratio of 100/8 (Table II, entry 1). When we used 2-phenyl-1,3-dioxane that is more stable than benzaldehyde dimethyl acetal, excellent chemoselectivity was observed with the ratio of 100/5, and in the presence of even more stable 4-nitrophenyl-1,3-dioxane, the ratio of 100/2 was obtained (Table II, entries 2, 4). The deprotection of the S,S-acetal and S,O-acetal in the presence of O,O-ketals also can be performed with excellent chemoselectivity (Table II, entries 5–9).

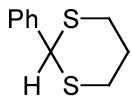
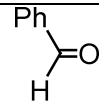
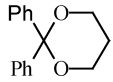
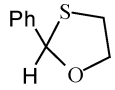
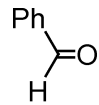
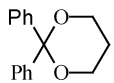
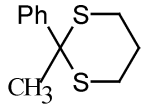
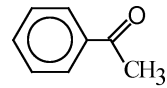
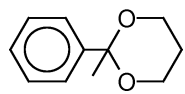
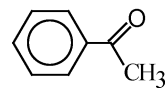
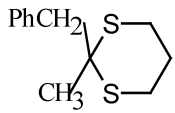
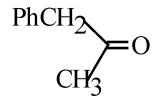
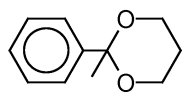
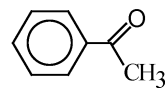
In order to extend the applicability of these reagents for the deprotection of O,O-acetals and ketals, we developed a very simple method and used these reagents in the presence of hydrated silica gel in dichloromethane. In this procedure, silica gel (2.5 g) and water (1.5 mL) were stirred vigorously until a free-flowing powder was obtained. A solution of O,O- , O,S-, or S,S-acetals or ketals (1.0 mmol) in CH_2Cl_2 (5 mL) was added to the resulting silica gel. To this mixture, a solution of TABCO, NBS, NCS, TCCA, or Br_2 (Table III) in CH_2Cl_2 (5 mL) was added and stirred at r.t. for 10 min. During this period, TLC and GC

TABLE II Selective Deprotection of Cyclic and Acyclic O,O-Acetals and Ketals in the Presence of S,S- and S,O-Acetals and Ketals with TABCO in Aqueous Acetonitrile at r.t.

Entry	Substrate 1 Substrate 2	Product 1 Product 2	Time (min)	Yield (%)[a,b,c]
1			8	100
				8
2			12	100
				5
3			12	100
				7
4			25	100
				2
5			25	100
				1

(*Continued on next page*)

TABLE II Selective Deprotection of Cyclic and Acyclic O,O-Acetals and Ketals in the Presence of S,S- and S,O-Acetals and Ketals with TABCO in Aqueous Acetonitrile at R. T. (*Continued*)

Entry	Substrate 1 Substrate 2	Product 1 Product 2	Time (min)	Yield (%)[a,b,c]
6			30	100
				0
7			25	98
				2
8			35	98
				5
9			40	97
				7

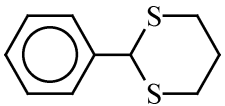
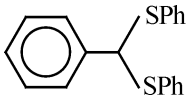
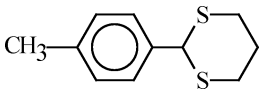
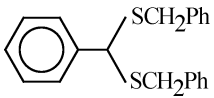
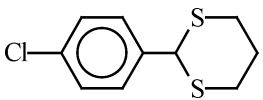
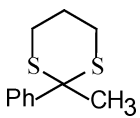
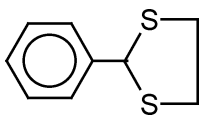
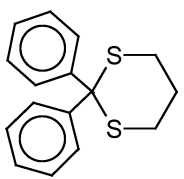
[a]Yields based on analysis by NMR and GC (n-heptane was used as internal standard).

[b]The ratio of S,S-acetal/O,O-acetal/TABCO is 1/1/1.2.

[c]The reaction was performed in the presence of two molar equivalents of pyridine.

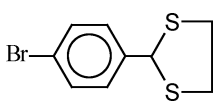
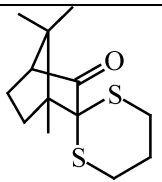
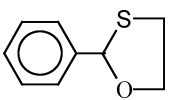
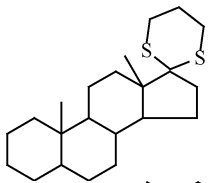
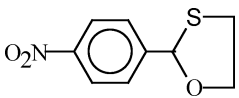
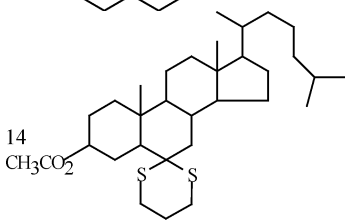
analysis confirmed the complete disappearance of the reactant. Simple filtration and chromatography on a short silica gel column afforded the corresponding carbonyl compounds in excellent yields. The results of this study are also shown in Table III.

TABLE III Deprotection of S,S- and S,O-Acetals and Ketals with TABCO (A), NBS (B), NCS (C), TCCA (D), and Br_2 (E) on Hydrated Silica Gel in CH_2Cl_2 at r.t.

Entry	Substrate	Sub./Reagent (ratio)	Yield %	Entry	Substrate	Sub./Reagent (ratio)	Yield %[a,b]
1		1/1.3 (A)	94	8		1/1.3 (A)	94
		1/1.2 (B)	95			1/1.2 (B)	95
		1/1.4 (C)	93			1/1.3 (C)	93
		1/1.5 (D)	92			1/1.5 (D)	92
		1/1.2 (E)	97			1/1.2 (E)	96
2		1/1.3 (A)	93	9		1/1.4 (A)	93
		1/1.2 (B)	94			1/1.33(B)	94
		1/1.5 (C)	90			1/1.6 (C)	92
		1/1.6 (D)	91			1/2.0 (D)	91
		1/1.2 (E)	95			1/1.2 (E)	96
3		1/1.3 (A)	94	10		1/1.2 (A)	89
		1/1.2 (B)	96			1/1.2 (B)	87
		1/1.5 (C)	91			1/1.4 (C)	89
		1/1.6 (D)	92			1/1.5 (D)	87
		1/1.3 (E)	97			1/1.2 (E)	90
4		1/1.3 (A)	93	11		1/1.5 (A)	90
		1/1.2 (B)	94			1/1.5 (B)	90
		1/1.4 (C)	91			1/2.0 (C)	89
		1/1.5 (D)	91			1/2.5 (D)	87
		1/1.2 (E)	94			1/1.4 (E)	92

(*Continued on next page*)

TABLE III Deprotection of S,S- and S,O-Acetals and Ketals with TABCO (A), NBS (B), NCS (C), TCCA (D), and Br_2 (E) on Hydrated Silica Gel in CH_2Cl_2 at r.t. (*Continued*)

Entry	Substrate	Sub./Reagent (ratio)	Yield %	Entry	Substrate	Sub./Reagent (ratio)	Yield %[a,b]
5		1/1.3 (A) 1/1.2 (B) 1/1.5 (C) 1/1.7 (D) 1/1.2 (E)	92 92 91 90 93	12		1/1.8 (A) 1/1.7(B) 1/2.0 (C) 1/3.0 (D) 1/1.5 (E)	87 89 85 85 89
6		1/0.8 (A) 1/0.7 (B) 1/1.0 (C) 1/1.2 (D) 1/0.6 (E)	94 95 93 93 96	13		1/1.8 (A) 1/1.7(B) 1/2.0 (C) 1/3.0 (D) 1/1.5 (E)	89 90 89 87 90
7		1/1.0 (A) 1/0.9 (B) 1/1.1 (C) 1/1.3 (D) 1/0.7 (E)	95 96 93 92 98	14		1/2.0 (A) 1/2.0(B) 1/2.5 (C) 1/3.5 (D) 1/2.0 (E)	90 90 87 85 88

[a]Isolated yield.

[b]The reaction mixture was stirred at r.t. for 10 min.

We believe hydrated silica gel plays the following important roles in the reactions we have studied: (a) Silica gel acts as an acid scavenger, and by the absorption of hydrogen halides, enolization of the carbonyl compounds is minimized; consequently, the amount of α-halogenated carbonyl compounds is drastically reduced; (b) the water in the hydrated silica gel acts as an oxygen an transfer source and hydrolyzes the sulfonium intermediate easily; (c) deprotection of S,S- and S,O-acetals and ketals in the presence of hydrated silica gel occurs much faster than that observed in aqueous acetonitrile but with a lack of selectivity; and (d) using this method, the efficient mild deprotection of cyclic and acyclic O,O-, S,S-, and S,O-acetals and ketals to their carbonyl compound was achieved in excellent yields at r.t. High yields of desired products, short reaction times, and easy work up in comparison with aqueous acetonitrile media are the advantage of this system (Tables I and III).

In conclusion, high chemoselectivity that has been observed in the presence of pyridine using NBS, NCS, TCCA, TABCO, and Br_2 in aqueous acetonitrile is an excellent practical achievement in organic synthetic methods. The use of NBS, NCS, TCCA, TABCO, and Br_2 as sources of electrophilic halogens in the presence of hydrated silica gel in CH_2Cl_2 provide efficient, novel, and mild procedures for the deprotection of cyclic and acyclic O,O-, S,S-, and S,O-acetals and ketals. In addition, in both of procedures, cheapness and availability of the reagents, simplicity of the procedures, short reaction times, and excellent yields can be considered strong practical points for the presented protocols.

EXPERIMENTAL

All yields refer to isolated products. The products were purified by column chromatography and the purity determination of the products were accomplished by GLC on a Shimadzu model GC-8A instrument or by TLC on Silica-gel polygram SIL G/UV 254 plates. Mass spectra were run on a Shimadzu GC MS-QP 1000EX at 75 eV. IR spectra were recorded on a Perkin-Elmer 781 spectrophotometer. The NMR spectra were recorded on a Bruker Avance DPX 250 MHz instrument.

General Procedure for the Deprotection of Cyclic and Acyclic Dithioacetals and 1,3-Oxathiolanes to Their Parent Carbonyl Compounds Using Electrophilic Halogens in Aqueous CH_3CN

To a solution of dithioacetals or 1,3-oxathiolanes (1.0 mmol) in 20% aqueous acetonitrile (5 mL) a solution of TABCO, NBS, NCS, TCCA, or

Br_2 (Table I) in CH_3CN (5 mL) was added. The resulting mixture was stirred at room temperature for 30–50 min (Table II). The progress of the reaction was monitored by TLC and GC analysis. Upon completion of the reaction, CH_2Cl_2 (25 mL) was added to the reaction mixture. The solution was washed in an aqueous solution of NaOH (5%, 20 mL) followed with brine solution (10 mL) and water (2×10 mL). The organic phase was separated and dried over anhydrous sodium sulfate. After evaporation of the solvent followed by chromatography on short silica gel column (hexane/EtOAc 8/2), the desired carbonyl compound(s) was obtained in excellent yield(s) (Table I).

General Procedure for the Deprotection of Cyclic and Acyclic Dithioacetals and 1,3-Oxathiolanes to Their Parent Carbonyl Compounds Using Electrophilic Halogens and Hydrated Silica Gel in Dichloromethane

Silica gel (2.5 g) was placed in a round-bottom flask (100 mL) equipped with a magnetic stirrer and a loosely fitted rubber septum. Water (1.5 g) was added to the vigorously stirred silica gel. After complete addition of water, stirring continued until a free-flowing powder was obtained (5 min). A solution of dithioacetals or 1,3-oxathiolanes (1.0 mmol) in CH_2Cl_2 (5 mL) was added to the hydrated silica gel. To this mixture, a solution of TABCO, NBS, NCS, TCCA, or Br_2 (Table III) in CH_2Cl_2 (5 mL) was added. The mixture was stirred at r.t. for 10 min. The mixture was then filtered through a sintered glass funnel, and the solid residue was washed with CH_2Cl_2 (5×10 mL). Removal of the solvent under vacuum gave the crude product(s). After chromatography on silica gel using n-hexane/EtOAc 8/2 as eluent, the carbonyl compound(s) were obtained in excellent yield(s). The product was characterized by comparison of its physical data (m.p. or b.p.), IR, and ^{1}HNMR spectral data with those of known aldehydes and ketones.

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[26] **Bis(2-hydroxyethyl) disulfide**: ^{1}HNMR (DMSO-d_6); δ(ppm): 4.9(t, 2H), 3.6(m, 4H), 2.8 (t, 4H); ^{13}CNMR (DMSO-d_6); δ(ppm): 59.5, 41.1; MS, 75eV m/z (%): 156.0(M + 2, 4), 155.0(M+1, 3), 154.0(M, 49), 92.0(90), 79.0(43), 64.0(51), 45.0(100), 31.0(38); IR (liquid film, cm^{-1}): 3500–3150, 2924, 2914, 2876, 1463, 1418, 1400, 1287, 1218, 1156, 1062, 1066, 1046, 1008.