Silica-Supported Perchloric Acid ($HClO_4$-SiO_2): A Versatile Catalyst for Tetrahydropyranylation, Oxathioacetalization and Thioacetalization

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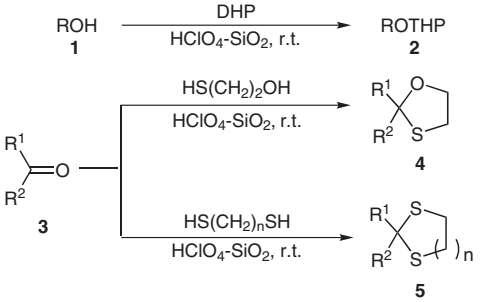
This work is dedicated to Prof. D. N. Buragohain, former Director of IITG, on the occasion of his 65th birthday.

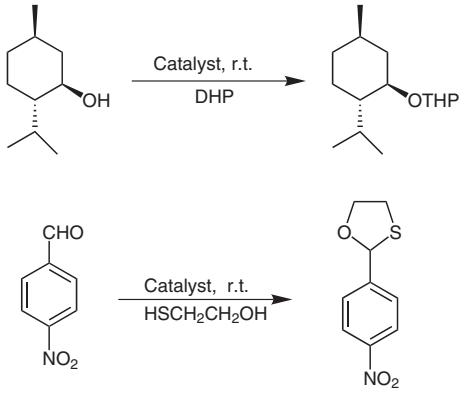
Abstract: A simple and convenient synthetic protocol for the protection of hydroxyl group as tetrahydropyranyl ether as well as carbonyl functionality as oxathioacetal and thioacetal has been achieved using a catalytic amount of silica-supported perchloric acid under solvent-free conditions. This protocol has many advantages compared to currently available methodologies in terms of simplicity, yield, reaction time, reusability of the catalyst and economic viability.

Key words: alcohols, phenols, THP ethers, carbonyl compounds, oxathioacetals, thioacetals



R = alkyl, aryl; R^1= alkyl, aryl; R^2 = H, alkyl, aryl; n = 1, 2

Scheme 1



Scheme 2

Recently silica-supported catalysts[1] as well as perchloric acid impregnated on silica gel has gained considerable attention in current organic synthesis due to its ease of preparation, high efficiency, reusability of the catalyst and its economic viability. The versatility and overall synthetic utility of this reagent is exemplified by the following applications such as acetylation of phenols, thiols, alcohols and amines,[2] peracetylation of carbohydrates,[3] acetalation followed by acetylation,[4] for glycosylation,[5] Ferrier rearrangement of glycals,[6] and synthesis of heterocycles[7] as well as selective cleavage of isopropylidene and trityl ethers.[8] Recently we found that the same catalyst is useful for *gem*-diacylation of aldehydes[9] as well as for Michael addition of thiols to the electron-deficient alkenes.[10] In our earlier preliminary communication,[11] we reported that 70% aqueous perchloric acid is an effective catalyst for oxathioacetalization of carbonyl compounds. In continuation of our work on solvent-free conditions and utilization of heterogeneous catalysis, herein report silica-supported perchloric acid as a catalyst for the protection of hydroxyl and carbonyl compounds as THP ethers and oxathioacetals or dithioacetals, respectively at room temperature under mild conditions as shown in Scheme 1.

The catalyst $HClO_4$-SiO_2 was prepared by following the literature procedure.[2] To prove the better catalytic activity of silica-supported perchloric acid over aqueous perchloric acid as well as to find out an optimal condition for both the transformations, i.e. tetrahydropyranylation and oxathioacetalization, a set of reactions were studied under different catalytic conditions. Menthol and 4-nitrobenzaldehyde were chosen as model substrates for tetrahydropyranylation and oxathioacetalization, respectively (Scheme 2). For both the transformations the same sets of experiments were carried out and the results are summarized in Table 1.

Sometimes the protection of hydroxyl group as its THP ether is preferred due to easy installation, low cost of the reagent DHP, stability towards a wide variety of reaction conditions as well as easy removal at the later stage. Tetrahydropyranylation of alcohols and phenols is traditionally carried out in the presence of PTSA,[12] PPTS,[13] K-10 clay[14] and $BF_3 \cdot OEt_2$.[15] Recent alternatives include $ZrCl_4$,[16] I_2,[17] LiBr,[18] acetonyltriphenylphosphonium bromide (ATPB),[19] tetrabutylammonium tribromide (TBATB),[20] aluminum chloride hexahydrate,[21] $Bi(OTf)_3$,[22] dialkylimidazolium tetrachloroaluminates,[23] $InCl_3$ immobilized in ionic liquid,[24] H_2O,[25] bromodimethylsulfonium bromide,[26] cupric sulfate pentahydrate,[27] and bismuth nitrate pentahydrate.[28] Although most of the methods provide

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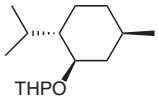
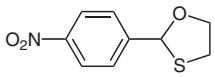
Table 1 Tetrahydropyranylation of Menthol and Oxathioacetalization of 4-Nitrobenzaldehyde

Entry	Catalyst				
		Time	Yield (%)[a]	Time	Yield (%)[a]
1	No catalyst	12 h	0	12 h	0
2	SiO_2 (20 mg/mol)	12 h	5	12 h	10
3	Aqueous $HClO_4$ (10 mol%)	10 min	76	1 h	35[11]
4	$HClO_4$-SiO_2 (20 mg/mmol, 1 mol%)	5 min	91[b]	30 min	65

[a] Isolated yield.
[b] The catalyst can be recycled up to consecutive three cycles without lose of its activity and provided 85% and 82% yields within 5 and 10 min, respectively, for THP protection.

good yields, still some of them require harsh reaction conditions, long reaction times, involvement of expensive and moisture-sensitive catalysts. Hence cheap heterogeneous catalyst, which work under mild conditions are desirable.

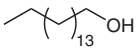
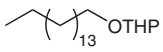
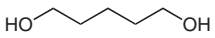
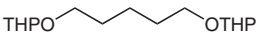
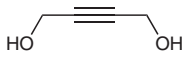
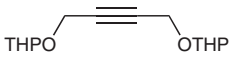
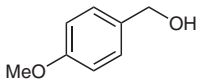
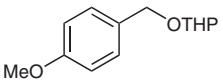
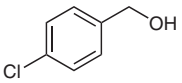
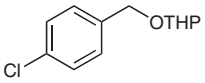
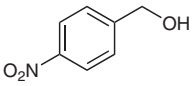
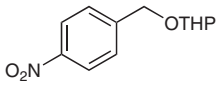
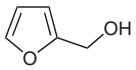
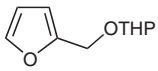
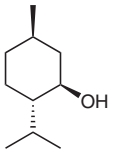
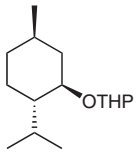
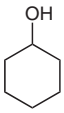
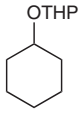
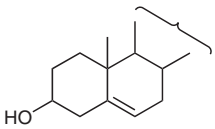
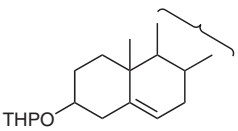
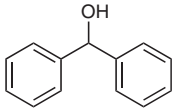
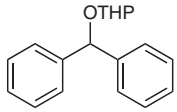
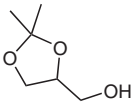
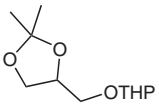
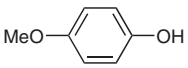
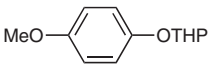
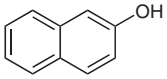
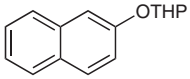
To verify the scope of this reagent for tetrahydropyranylation of alcohols and phenols, firstly cetyl alcohol was treated with 1.1 equivalents of 3,4-dihydro-2*H*-pyran (DHP) under solvent-free conditions with 1 mol% of $HClO_4$-SiO_2 at room temperature. It was smoothly converted to the corresponding THP ether within five minutes in 91% yield. Similarly, a wide variety of other aliphatic as well as benzyl alcohols also gave the THP ethers within a very short time. It is worthwhile to mention that this protocol is faster and provide better yields compared to recently reported methods. Likewise, various secondary alcohols such as menthol, cyclohexanol, cholesterol and even hindered alcohol benzhydrol also underwent tetrahydropyranylation without any difficulty. Moreover, isopropylidine-protected glycerol provided the desired THP ether under similar experimental conditions keeping the acid-sensitive protecting group intact. 4-Methoxyphenol and β-naphthol were also converted to the required THP ethers in good yields (Table 2).

In Table 3, menthol and 2,3-*O*-isopropylidene-D-(±)-glycerol are used as model substrates for comparison of this protocol with some of the recently reported methods. We have done the comparison with respect to mol% of catalyst used, reaction timing and yields obtained.

Several methods are known in the literature[29] for both oxathioacetalization and thioacetalization. Conventionally, oxathioacetals are prepared from the corresponding carbonyl compounds and 2-mercaptoethanol by using equimolar amount of Lewis acid such as $BF_3{\cdot}OEt_2$[30] or $ZnCl_2$.[31] Some of the recent procedures for similar transformation are: montmorillonite K-10,[32] bromodimethylsulfonium bromide[33] and silica-supported tungstophosphoric acid.[34] In our preliminary communication we reported the use of 70% aqueous perchloric acid for the same transformation.[11] Notably, we got relatively low yield in case of 4-nitrobenzaldehyde as mentioned in Table 1. $HClO_4$-SiO_2 is a better catalyst as shown in Table 4. It is much more effective in terms of reaction time and yield. Both aromatic aldehydes and ketones can be protected as oxathioacetals using this protocol without any difficulty. All the products were isolated by distillation under reduced pressure.

Various new methods have also been developed for thioacetalization of carbonyl compounds. Among them, some of the recent methods employ: *N*-bromosuccinimide,[35] bromodimethylsulfonium bromide,[36] acetyl chloride,[37] copper (II) tetrafluoroborate,[38] ruthenium(III) chloride,[39] PTSA/SiO_2,[40] silica-supported polyphosphoric acid[41] and other catalysts.[42] Interestingly, a large number of carbonyl compounds underwent dithioacetalization using 1 mol% $HClO_4$-SiO_2 under solvent-free conditions at room temperature. Most of the reactions were complete within 2–30 min in very good yields as shown in Table 5. Remarkably, aldehydes having electron-donating or -withdrawing substituents in the aromatic ring underwent thioacetalization smoothly in good yields although the yield for the 1,3-dithiane of 4-nitrobenzaldehyde is less than that of other protected aldehydes. It is noteworthy to mention that the same procedure is applicable to aliphatic aldehydes also. The acid-sensitive 2-furaldehyde can also be protected to the desired 1,3-dithiane derivatives without any difficulty. All the products were fully characterized by recording ^1H NMR, ^{13}C NMR and elemental analysis. Remarkably, all solid dithioacetals can be obtained by direct recrystallization after filtering the catalyst. The generality and scope of this catalyst can be ascertained from its recyclability, which we have tested by the following way. For example, a mixture of 4-hydroxybenzaldehyde and propane-1,3-dithiol was treated with $HClO_4$-SiO_2. After completion of the reaction, anhydrous diethyl ether was added to the mixture and the catalyst was filtered off, dried and used for a second time. The recovered catalyst can be recycled for another two more cycles without any loss of its catalytic activity: yields were 92% and 90% respectively after 2 and 5 minutes.

Table 2 Tetrahydropyranylation of Alcohols and Phenols Using $HClO_4$-SiO_2 as Catalyst

Entry	Substrate **1**	Reaction time (min)	Product **2**[a]	Yield (%)[b]
a		5		91[27]
b		5		90
c		5		87
d		5		95[25]
e		5		92
f		5		83[28]
g		15		96[13]
h		5		91[19]
i		5		91[13]
j		15		97[19]
k		5		95
l		15		84[25]
m		5		80[13]
n		30		83[28]

[a] Products were characterized by recording IR, ^1H and ^{13}C NMR spectra and elemental analysis.
[b] Isolated yield.

Table 3 Comparisons of Some of the Recent Methods for Tetrahydropyranylation with $HClO_4$-SiO_2 Protocol

Catalyst	Mol%	Menthol		2,3-*O*-Isopropylidene-D-(±)-glycerol	
		Time	Yield (%)[a]	Time	Yield (%)[a]
$CuSO_4 \cdot 5H_2O$[27]	20	1 h	89	50 min	85
TBATB[20]	0.1	1 h	74	–	–
$Bi(OTf)_3 \cdot 4H_2O$[22]	0.1	2 h	74	3.25 h	78
$Bi(NO_3)_3 \cdot 5H_2O$[28]	5	22 min	90	20 min	84
$HClO_4$-SiO_2	1	5 min	91	10 min	86

[a] Isolated yield.

In conclusion we have devised a simple and efficient methodology for tetrahydropyranylation, oxathioacetalization and thioacetalization using a very cheap, readily accessible and recyclable catalyst. The notable advantages of this protocol are non-aqueous work-up; very rapid and simple procedure, very good yields. Therefore, we believe this methodology will be a new addition in organic synthesis.

Melting points were recorded on a Büchi B-545 melting point apparatus and were uncorrected. IR spectra were recorded in KBr or neat on a Nicolet Impact 410 spectrophotometer. ^{1}H NMR spectra and ^{13}C NMR spectra were recorded on a Varian 400 MHz spectrometer in $CDCl_3$ using TMS as internal reference. Elemental analyses were carried out in a PerkinElmer 2400 automatic analyzer. Column chromatographic separations were done on SRL silica gel (60–120 mesh).

Silica-Supported Perchloric Acid ($HClO_4$-SiO_2)[2]

$HClO_4$ (1.8 g, 12.5 mmol, as a 70% aq solution) was added to a suspension of SiO_2 (230–400 mesh, 23.7 g) in Et_2O (70 mL). The mixture was concentrated and the residue was heated at 100 °C for 72 h under vacuum to furnish $HClO_4$-SiO_2 (0.5 mmol/g) as a free-flowing powder (50 mg = 0.025 mmol of $HClO_4$).

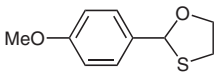
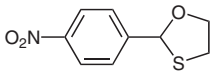
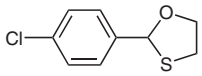
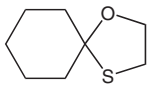
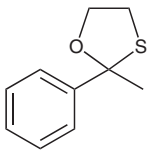
$HClO_4$-SiO_2 Catalyzed Tetrahydropyranylation of Alcohols and Phenols; General Procedure

To a mixture of alcohol or phenol **1** (1 mmol) and 2,3-dihydropyran (DHP) (1.1 mmol, 100 μL) was added $HClO_4$-SiO_2 (0.01 mmol, 20 mg) and the mixture was stirred at r.t. After completion of the reaction as checked by TLC, the crude mixture was directly passed through a short basic alumina column to obtain the desired pure THP ether **2** (Table 2).

$HClO_4$-SiO_2 Catalyzed Oxathioacetalization and Thioacetalization of Carbonyl Compounds; General Procedure

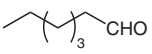
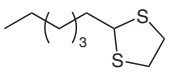
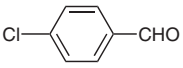
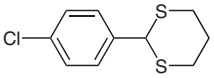
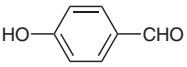
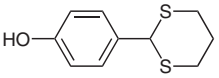
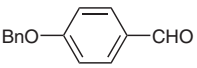
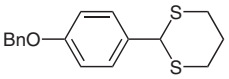
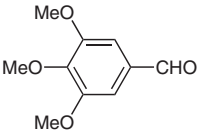
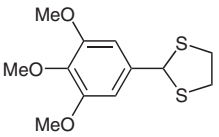
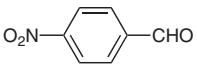
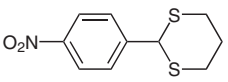
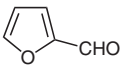
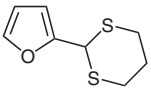
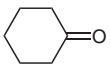
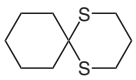
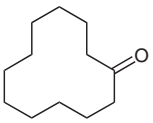
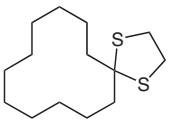
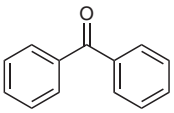
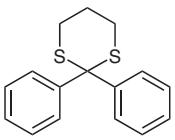
A stirred mixture of carbonyl compound **3** (10 mmol) and 2-mercaptoethanol (12 mmol, 0.84 mL) was treated with $HClO_4$-SiO_2 (200 mg, 0.1 mmol) at r.t. The progress of the reaction was monitored by TLC. After completion of the reaction, the pure product **4** was obtained directly by distillation (Table 4). In the case of dithioacetalization, a mixture of carbonyl compound **3** (5 mmol) and ethane-1,2-dithiol (A) or propane-1,3-dithiol (B) (6 mmol) was treated with the same catalyst. After completion of reaction as checked by TLC, it was diluted with EtOAc and the catalyst was fil-

Table 4 Oxathioacetalization of Carbonyl Compounds Using Catalytic Amount of $HClO_4$-SiO_2 versus Aqueous $HClO_4$ under Solvent-Free Conditions

Entry	Product **4**	Method A[a]		Method B[b]		Bp (°C/mm Hg) or Mp (°C)
		Time (min)	Yield (%)[c,d]	Time (min)	Yield (%)[c,d]	
a		20	68	10	74[33]	135/1
b		60	35	30	65[33]	78
c		–	–	25[e]	90[33]	125/5
d		30	76	30	85[33]	85/5
e		90	60	60	75[33]	110/5

[a] Method A = reactions were carried out using 10 mol% aq $HClO_4$ (Ref. 11).
[b] Method B = reactions were carried out using 1 mol% $HClO_4$-SiO_2.
[c] Isolated yield.
[d] All the products were colorless oils, except **4b** (solid), and were characterized by recording ^{1}H NMR, ^{13}C NMR and elemental analysis.
[e] The catalyst was recycled up to consecutive three cycles without loss of its activity and it provided 80% and 78% yields within 25 and 30 min, respectively.

Table 5 Thioacetalization of Carbonyl Compounds Using Catalytic Amount of $HClO_4$-SiO_2 under Solvent-Free Conditions

Entry	Substrate **3**	Thiol[a]	Reaction time (min)	Product **5**[b]	Yield (%)[c]	Mp (°C)[d]
a		A	15		98[36]	liquid
b		B	10		81[35]	91
c		B	2		94[36]	157 (158)[36]
d		B	5		98[37]	77–78 (78)[37]
e		A	15		98	94
f		B	30		61[42b]	148 (141–142)[42b]
g		B	15		96[36]	liquid
h		B	30		93[36]	liquid
i		A	5		91[36]	88–89 (88)[36]
j		B	40		96[37]	109 (108–109)[37]

[a] A = ethane-1,2-dithiol, B = propane-1,3-dithiol.
[b] All products were solids, except **5a**,**g**,**h** (liquids), and were characterized by recording ^{1}H NMR, ^{13}C NMR and elemental analysis.
[c] Isolated yield.
[d] Reported mps are given in parentheses.

tered off. Then the filtrate was concentrated and either kept for recrystallization by adding hexane if the product **5** is solid or purified by silica gel column chromatography (Table 5).

THP Ether of Pentane-1,5-diol 2b
Colorless viscous liquid; yield: 0.245 g (90%).

IR (neat): 2940, 2879, 1455, 1445, 1352, 1265, 1204, 1132, 1035 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.42–1.88 (m, 18 H, CH_2), 3.39 (dt, *J* = 6.8, 9.6 Hz, 2 H, OCH_2), 3.46–3.53 (m, 2 H, OCH_2), 3.74 (dt, *J* = 6.8, 9.6 Hz, 2 H, OCH_2), 3.83–3.90 (m, 2 H, OCH_2), 4.56 (dd, *J* = 6.8, 9.6 Hz, 2 H, OCH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.71 (2 C), 23.00, 25.55 (2 C), 29.61 (2 C), 30.79 (2 C), 62.23 (2 C), 67.41 (2 C), 98.67 (2 C).

Anal. Calcd for $C_{15}H_{28}O_4$ (272.38): C, 66.14; H, 10.36. Found: C, 66.21; H, 10.40.

THP Ether of But-2-yne-1,4-diol 2c
Colorless viscous liquid, yield: 0.221 g (87%).

IR (neat): 2940, 2873, 1445, 1399, 1352, 1271, 1209, 1127, 1025, 968 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.46–1.79 (m, 12 H, CH_2), 3.43–3.48 (m, 2 H, OCH_2), 3.73–3.79 (m, 2 H, OCH_2), 4.19 (d, *J* = 17.6 Hz, 1 H, OCH_2), 4.22 (d, *J* = 12.4 Hz, 2 H, OCH_2), 4.27 (d, *J* = 14.2 Hz, 1 H, OCH_2), 4.74 (t, *J* = 3.4 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.88 (2 C), 25.20 (2 C), 30.07 (2 C), 54.19 (2 C), 61.81 (2 C), 81.81 (2 C), 96.66 (2 C).

Anal. Calcd for $C_{14}H_{22}O_4$ (254.32): C, 66.12; H, 8.72. Found: C, 66.21; H, 8.69.

THP Ether of 4-Chlorobenzyl Alcohol 2e
Colorless viscous liquid; yield: 0.209 g (92%).

IR (neat): 2943, 2867, 1592, 1464, 1358, 1132, 1075, 1038 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.54–1.89 (m, 6 H, CH_2), 3.52 (d, *J* = 11.2 Hz, 1 H, OCH_2), 3.87 (t, *J* = 8.4 Hz, 1 H, OCH_2), 4.44 (d, *J* = 12.0 Hz, 1 H, ArCH), 4.67 (br s, 1 H, OCHO), 4.72 (d, *J* = 12.4 Hz, 1 H, ArCH), 7.29 (br s, 4 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.40, 25.50, 30.58, 62.13, 67.99, 97.70, 128.30 (2 C), 128.89 (2 C), 133.02, 136.65

Anal. Calcd for $C_{12}H_{15}ClO_2$ (226.70): C, 63.58; H, 6.67. Found: C, 63.38; H, 6.59.

THP Ether of Benzhydrol 2k
White solid; yield: 0.255 g (95%); mp 50–51 °C.

IR (KBr): 2942, 2903, 2877, 1490, 1199, 1121, 1025, 977, 916 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.52–1.97 (m, 6 H, CH_2), 3.47–3.52 (m, 1 H, OCH_2), 3.85–3.91 (m, 1 H, OCH_2), 4.66 (t, *J* = 3.2 Hz, 1 H, OCHO), 5.79 [s, 1 H, $(Ar)_2CH$], 7.17–7.36 (m, 10 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.27, 25.65, 30.66, 61.99, 78.07, 95.37, 126.67 (2 C), 126.88 (2 C), 127.43 (2 C), 127.53 (2 C), 128.03 (2 C), 128.29 (2 C).

Anal. Calcd for $C_{18}H_{20}O_2$ (268.35): C, 80.57; H, 7.51. Found: C, 80.71, H, 7.59.

2-[3′,4′,5′- Trimethoxyphenyl]-1,3-dithiolane (5e)
White solid; yield: 1.335 g (98%); mp 53–54 °C.

IR (KBr): 1586, 1505, 1127 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.31–3.38 (m, 2 H, SCH_2), 3.46–3.54 (m, 2 H, SCH_2), 3.82 (s, 3 H, OCH_3), 3.86 (s, 6 H, 2 × OCH_3), 5.60 (s, 1 H, ArC*H*), 6.76 (s, 2 H, ArH).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 40.18 (2 C), 56.12 (2 C), 56.86, 60.78, 104.71, 104.85 (2 C), 135.14, 152.85 (2 C).

Anal. Calcd for $C_{12}H_{16}O_3S_2$ (272.37): C, 52.92; H, 5.92; S, 23.54. Found: C, 52.98; H, 5.89; S, 23.61.

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