



## Potassium phosphate catalyzed highly efficient synthesis of structurally diverse thioethers at ambient temperature

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Commercially available potassium phosphate has been demonstrated to be a highly efficient catalyst for the synthesis of thioethers employing two different routes *viz.* alkylation of thiols with alkyl/aryl halides and by Michael addition of thiols to conjugated alkenes.

**Keywords:** Potassium phosphate, thiols, thioethers, thia-Michael reaction, alkyl halides

Thioethers have emerged as an important class of organic compounds with useful applications as key intermediates in organic synthesis as well as bio-organic, medicinal and heterocyclic chemistry<sup>1-3</sup>. Amongst various available approaches for the synthesis of thioethers, base catalyzed nucleophilic displacement of halide ion by thiolate anion<sup>4-7</sup>, Michael addition of thiol to conjugated alkenes<sup>8-10</sup>, are the routinely preferred pathways. Additionally, deoxygenation of sulfoxides<sup>15,16</sup>, Indium (I) iodide promoted addition of thiolate anion to styrene<sup>17</sup>, intermolecular *s*-alkylation of thiol with alcohol<sup>18,19</sup> and transition metal catalyzed coupling of aryl halide with thiol<sup>20,21</sup> are a few other approaches of interest. Many of these reported protocols although furnish the desired thioethers in acceptable yields, a few of them are plagued with the use of expensive catalyst, elevated temperature, long reaction times, essentiality of non-ecofriendly or high boiling solvent as well as the formation of side products such as disulfides, sulfonium salts, *etc.* Although plethora of catalyst are available for the synthesis of thioethers, to the best of our knowledge there are no reports on the use of a single catalyst which could be useful in the synthesis of these molecules following two routinely preferred approaches<sup>4-14</sup>. Our interest in the synthesis of thioethers stems from the call for structurally diverse thioethers useful in our ongoing studies on controlled and chemoselective oxidation of thioethers. Such

thioethers either being very expensive or not being available commercially, it was surmised that the development of a mild, greener and practically simple protocol for the synthesis of thioethers would be in tune with our continued interest in the development of new synthetic methodologies<sup>22-26</sup>.

### Result and Discussion

Over the past few years, commercially available, non-toxic and relatively less expensive potassium phosphate has emerged as a useful base catalyst<sup>27,28</sup>. We have earlier explored its utility in Knoevenagel as well as Claisen-Schmidt condensation<sup>29,30</sup>, Henry reaction<sup>31</sup>, Michael addition reaction<sup>32</sup> and in the synthesis of tetrahydrobenzo [b] pyrans<sup>33</sup>. In continuation of these studies herein we report the usefulness of potassium phosphate as a highly efficient catalyst in synthesis of thioethers following two different routes (Figure 1). As regards the first route involving the alkylation of thiols, it was surmised that, potassium phosphate being basic enough to deprotonate a variety of thiols ( $pK_a = 7-11$ )<sup>28</sup>, the resulting thiolate anion on reaction with alkyl halide would easily furnish corresponding thioether by  $S_N$  pathway. Based upon this presumption, a model reaction was carried out at ambient temperature between thiophenol and benzyl bromide using potassium phosphate as a catalyst and dimethyl formamide as a dipolar aprotic solvent. The

desired thioether was obtained in quick time and excellent yield (30 min, 93%). With an aim to turn this initial success into a greener protocol, the reaction conditions were then optimized with respect to the choice of solvent as well as the amount of catalyst loading by performing a series of reactions using the same substrate combination. The results summarized in Table I clearly demonstrate the essentiality of 25 mol % potassium phosphate as a catalyst and ethanol as relatively less expensive and non toxic reaction medium for an optimum yield (93%) of benzyl phenyl sulfide (Table I). Under the established reaction conditions for thiophenol in hand, generality of the protocol was then investigated by reacting a series of aryl, aralkyl, cycloalkyl and alkyl thiols with a range of primary, secondary, allylic as well as propargylic bromide. In general, the reactions were smooth and furnished corresponding thioethers in high yields as well as purity (Table II). It is worth mentioning that, potassium phosphate-purged air combination has previously been used in oxidation of thiols to

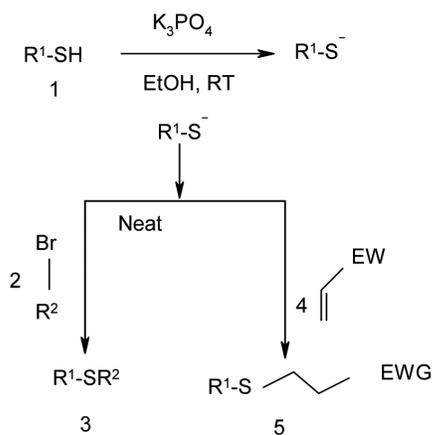


Figure 1 — Synthesis of thioethers following two different routes

Table I — Synthesis of benzyl phenyl sulphide under various conditions<sup>a</sup>

Entry	Catalyst (mol %)	Solvent	Yield (%) <sup>b,c</sup>
1	—	DMF/CH <sub>3</sub> CN/EtOH	30 <sup>c</sup>
2	5	DMF	40 <sup>c</sup>
3	5	EtOH	35 <sup>c</sup>
4	10	DMF	50 <sup>c</sup>
5	10	EtOH	45 <sup>c</sup>
6	20	DMF	80 <sup>c</sup>
7	20	EtOH	76 <sup>c</sup>
8	25	DMF	93 <sup>d</sup>
9	25	EtOH	91 <sup>d</sup>

<sup>a</sup> Reaction conditions: Thiophenol (2 mmol), benzyl bromide (2 mmol), catalyst, solvent, RT. <sup>b</sup> Isolated yield. <sup>c</sup> Yields after 6 h. <sup>d</sup> Yields after 3 h.

disulfides<sup>28</sup> however; we did not observe the formation of any disulfide to a detectable extent (tlc). This is possibly because, in the absence of purged air, rate of nucleophilic displacement of halide ion by thiolate anion is much faster than that for its oxidation to yield corresponding disulfide. While examining the scope of the present protocol it was noticed that, the reaction of thiophenol with 1, 2 – dibromoethane and that of ethane 1, 2-dithiol with benzyl bromide furnished corresponding dithioethers. (Entries h, i, Table II) It was further gratifying to note that the protocol developed was also applicable in chemoselective S vs O or N alkylation with the choice of 2-mercaptoethanol (2n) and 2-amino thiophenol (2o) or 2-mercatobenzimidazole (2q) as representative bifunctional thiols (Table II). Our major aim behind the present study was not alone to develop a method for the synthesis of simple thioethers but was to develop a practically simple protocol for the synthesis of thioethers bearing a variety of functional groups such as OH, -CN, -CONH<sub>2</sub>, -COOR, *etc.* In this context, although we were successful in synthesizing a few thioethers of such variety (Entry 1c, 1f, 1n, Table II), higher cost of starting bromo compounds such as, bromoacetonitrile, bromoacetamide, bromoester, *etc.* was the major limiting factor in using this new protocol. To circumvent this limitation, it was then planned to extend our earlier experience

Table II — Potassium phosphate catalyzed synthesis of thioethers

Entry	R <sup>1</sup> (1)	R <sup>2</sup> (2)	Yield (%) <sup>b</sup>	Ref.
a	Ph	PhCH <sub>2</sub> Br	91	7
b	Ph	4-F-PhCH <sub>2</sub> Cl	84	—
c	Ph	Br-H <sub>2</sub> CCO <sub>2</sub> Et	86	11
d	Ph	H <sub>2</sub> C=CHCH <sub>2</sub> Br	93	6
e	Ph	HCCCH <sub>2</sub> Br	75 <sup>d</sup>	—
f	Ph	HOH <sub>2</sub> CCH <sub>2</sub> Br	87	52
g	Ph	(CH <sub>3</sub> ) <sub>2</sub> CHI	90	4
h	Ph <sup>c</sup>	BrH <sub>2</sub> CCH <sub>2</sub> Br	89	52
i	HSH <sub>2</sub> CCH <sub>2</sub> SH	PhCH <sub>2</sub> Br	93	52
j	n-C <sub>4</sub> H <sub>9</sub>	4-Cl-PhCH <sub>2</sub> Br	79	7
k	4-Me Ph	PhCH <sub>2</sub> Br	86	48
l	4-Cl Ph	4-NO <sub>2</sub> -PhCH <sub>2</sub> Br	93	7
m	Cyclohexane	PhCH <sub>2</sub> Br	83	47
n	HSH <sub>2</sub> CCH <sub>2</sub> OH	4-F-PhCH <sub>2</sub> Cl	84	—
o	2-Aminophenyl	PhCH <sub>2</sub> Br	90	47
p	PhCH <sub>2</sub>	PhCH <sub>2</sub> Br	96	48
q	2-Mercaptobenzimidazole	PhCH <sub>2</sub> Br	98	49

<sup>a</sup> Reaction conditions: Thiol (2 mmol), halide (2 mmol), potassium phosphate (25 mol %), ethanol (3 ml), RT. <sup>b</sup> Yield refers to pure isolated product. <sup>c</sup> 2 equ. <sup>d</sup> Solution of propargyl bromide in DCM was used. <sup>e</sup> Commercially available.

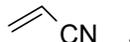
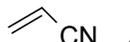
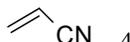
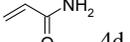
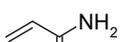
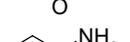
with Michael addition of thiols to enones<sup>32</sup>, towards other Michael acceptors such as conjugated esters, amides, nitriles, *etc.* A survey of literature interestingly revealed that a variety of catalysts<sup>34-40</sup> have already been demonstrated to be useful in Michael addition of thiols to enones. However, only a few of them<sup>38-40</sup> have been used in reactions with the afore-mentioned Michael acceptors.

A representative reaction was then carried out between thiophenol, 1a and methyl acrylate, 4a, employing the reaction conditions established earlier for the preparation of benzyl phenyl sulfide. The desired thia-Michael addition product, 5aa, resulted very quickly (3-4 min) in almost quantitative yield. Further studies regarding the optimization of yield with respect to the catalyst loading revealed that, only 10 mol % potassium phosphate was sufficient to drive the reaction to completion under solvent-free condition with only a slight compromise in time (8-10 min vs. 3-4 min). So as to demonstrate the versatility of the protocol, representative alkyl, 1j, cycloalkyl, 1m and aralkyl thiol, 1p, were initially scrutinized with methyl acrylate and following success to these reactions, the protocol was extended towards addition of these thiols to other Michael acceptors (Table III). In all the cases studied, the resultant product in excellent yield (>95%) and purity (NMR) was isolated by simply stirring the reaction mixture with dichloromethane followed by filtration of concentrated organic extract through a short column of silica gel.

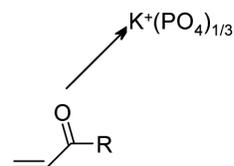
From a comparison view point, it is worthy to note that, synthesis of thioethers by SN pathway required slightly longer time (2-3 h) while the same following thia Michael pathways required very short reaction times. In context of the remarkable rate acceleration, on the basis of our earlier experience<sup>32</sup> we propose that, the primary role potassium phosphate in these C-S bond formation reactions is to deprotonate thiol to corresponding thiolate ion. In addition, due the presence of strong electron withdrawing counter anion *viz.*  $[(\text{PO}_4)^{-3}]$ ,  $\text{K}^+$  ion in potassium phosphate is sufficiently oxophilic and can form a strong coordinate bond with oxygen atom in enones as well as other Michael acceptors (Figure 2). Consequently, C- $\beta$  in conjugated alkenes becomes enough electrophilic to accelerate the addition of thiolate anion (thia-Michael pathway).

## Experimental Section

All chemicals were obtained from Lancaster or Aldrich Chemical Corporation and were used without

Entry	Thiol (1) R =	Alkene (4)	Product (5)	Yield (%) <sup>Ref</sup>
1	Ph 1a	 4a	5aa	94 <sup>38</sup>
2	n-Bu 1j	 4a	5aj	98 <sup>37</sup>
3	Cyclohexyl 1m	 4a	5am	94
4	PhCH <sub>2</sub> 1p	 4b	5bp	96
5	Ph 1a	 4c	5ca	95 <sup>38</sup>
6	n-Bu 1j	 4c	5cj	97
7	Cyclohexyl 1m	 4c	5cm	93
8	Ph 1a	 4d	5da	96 <sup>38</sup>
9	n-Bu 1j	 4d	5dj	94 <sup>37</sup>
10	Cyclohexyl 1m	 4d	5dm	92
11	PhCH <sub>2</sub> 1p	 4d	5dp	92

Reaction conditions: Thiol (2 mmol), alpha beta unsaturated compound (2 mmol), potassium phosphate(25 mol %), RT, neat.



R = alkyl, cycloalkyl, aryl, etc.

Figure 2 — Formation of coordinate bond between potassium ion and oxygen

purification. IR spectra were recorded on a Perkin-Elmer-793 instrument. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded as CDCl<sub>3</sub> solutions on a Bruker Avance spectrometer at 300 and 75.4 MHz, respectively, using TMS as an internal standard. Chemical shifts are expressed in  $\delta$  units. Elemental analysis was carried out using EURO EA analyzer.

## Representative procedure

### (a) Preparation of thioether by alkylation of thiol

To 0.55 g thiophenol (5 mmol) in 5 cm<sup>3</sup> ethanol was added 0.26 g (25 mol %) of potassium

phosphate. After stirring the mixture for about 10 min, 0.60 g (5 mmol) allyl bromide was added. The stirring was continued and on completion of the reaction (tlc) water (20 cm<sup>3</sup>) was added. The reaction mixture was extracted with 3×20 cm<sup>3</sup> of diethyl ether. The ether extract was washed with water (3×15 cm<sup>3</sup>) and dried over anhydrous sodium sulfate. The removal of solvent followed by filtration of the resultant oil through a short column of silica gel afforded 0.730 g (93%) **2d** as an oil.

#### (b) Preparation of thioether by thia-Michael reaction

To a mixture of 0.55 g thiophenol (5 mmol) and 0.11g (10 mol %) potassium phosphate, 0.430 g (5 mmol) methyl acrylate was added. The reaction mixture was stirred till completion of the reaction. (tlc, 8-10 min). The resultant product was extracted with 3×15 cm<sup>3</sup> dichloromethane. The organic extract was concentrated under vacuum and the resultant residue upon filtration through a short column of silica-gel furnished 1.39 g (94%) **4a** as an oil.

#### Spectral data of the representative compounds

**(Prop-2-ynylsulfanylmethyl) benzene, 3e** (C<sub>9</sub>H<sub>8</sub>S): IR (Neat): 3010, 2221, 1447, 763, 699 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 2.32 (t, *J* = 2.4 Hz, 1H), 3.09 (d, *J* = 2.4 Hz, 2H), 3.89 (s, 2H), 7.24-7.36 (m, 5H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 35.24 (SCH<sub>2</sub>), 71.33 (CH), 79.86 (C), 127.21 (ArCH), 128.58 (ArCH), 129.04 (ArCH), 137.45 (ArC). Anal. Calcd for C<sub>9</sub>H<sub>8</sub>S: C 72.93, H 5.44. Found: C 72.19, H 5.12%.

**2-[(4-Fluorobenzyl)sulfanyl] ethanol, 3n** (C<sub>9</sub>H<sub>11</sub>OFS): IR (Neat): 3420, 2963, 1455, 804 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 2.43 (br s, OH, 1H), 2.60 (t, *J* = 6.0 Hz, 2H), 3.67 (t, *J* = 5.7 Hz, 2H), 3.69 (s, 2H), 6.99 (t, *J* = 8.7 Hz, 2H), 7.24-7.29 (m, 2H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 34.22 (SCH<sub>2</sub>), 35.05 (SCH<sub>2</sub>), 60.34 (OCH<sub>2</sub>), 115.29 (ArCH), 115.58 (ArCH), 130.32 (ArCH), 130.42 (ArCH), 133.76 (ArCH), 133.80 (ArC), 161.91 (ArC, <sup>1</sup>*J*<sub>CF</sub> = 22.4 Hz). Anal. Calcd for C<sub>9</sub>H<sub>11</sub>OFS: C 58.04, H 5.95. Found: C 57.85, H 5.47%.

**Methyl 3-(cyclohexylsulfanyl) propanoate, 5am** (C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>S): IR (Neat): 2930, 1741, 1436, 1244 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.10-1.40 (m, 5H), 1.60-1.62 (m, 1H), 1.71-1.77 (m, 2H), 1.91-1.98 (m, 2H), 2.58 (t, *J* = 7.5 Hz, 2H), 2.59-2.69 (m, 1H), 2.79 (t, *J* = 7.5 Hz, 2H), 3.68 (s, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 24.96 (CH<sub>2</sub>), 25.76 (CH<sub>2</sub>), 26.04 (CH<sub>2</sub>), 35.03 (SCH<sub>2</sub>), 43.56 (CH<sub>2</sub>), 51.74 (OCH<sub>2</sub>), 172.49 (CO). Anal. Calcd for C<sub>10</sub>H<sub>18</sub>O<sub>2</sub>S: C 59.37, H 8.97. Found: C 58.87, H 8.14%.

**Methyl-3-(benzylsulfanyl)-2-methylpropanoate, 5bp** (C<sub>12</sub>H<sub>16</sub>O<sub>2</sub>S): IR (Neat): 3028, 1738, 1455, 701 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.21 (d, *J* = 6.9 Hz, 3H), 2.47 (dd, *J* = 12.6 & 6.3 Hz, 1H), 2.65 (sextet, *J* = 6.9 Hz, 1H), 2.76 (dd, *J* = 12.6 & 7.2 Hz, 1H), 3.69 (s, 3H), 3.72 (s, 2H), 7.31-7.33 (brs, 5H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 16.85 (CH<sub>3</sub>), 34.55 (CH<sub>2</sub>), 36.62 (SCH<sub>2</sub>), 39.82 (CH<sub>2</sub>), 51.79 (OCH<sub>3</sub>), 127.06 (ArCH), 128.48 (ArCH), 128.51 (ArCH), 138.13 (ArC), 175.55 (CO). Anal. Calcd for C<sub>12</sub>H<sub>16</sub>O<sub>2</sub>S: C 64.25, H 7.19. Found: C 63.86, H 6.97%.

**3-(Cyclohexylsulfanyl) propanenitrile, 5cm** (C<sub>9</sub>H<sub>15</sub>NS): IR (Neat): 2930, 2250, 1449, 1265, 740 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.2-1.4 (m, 5H), 1.60-1.63 (m, 1H), 1.75-1.77 (m, 2H), 1.94-1.98 (m, 2H), 2.61 (t, *J* = 6.9 Hz, 2H), 2.67-2.75 (m, 1H), 2.79 (t, *J* = 6.9 Hz, 2H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 19.26 (CH<sub>2</sub>), 25.58 (CH<sub>2</sub>), 25.64 (CH<sub>2</sub>), 25.92 (CH<sub>2</sub>), 33.49 (SCH<sub>2</sub>), 43.76 (CH<sub>2</sub>), 118.45 (CN). Anal. Calcd for C<sub>9</sub>H<sub>15</sub>NS: C 63.85, H 8.93, N 8.27. Found: C 63.16, H 8.27, N 7.87%.

**3-(Butylsulfanyl)propanamide, 5dj** (C<sub>7</sub>H<sub>15</sub>NOS): Solid, M.P. 106 °C; IR (KBr): ν = 3358, 3185, 1657, 1423, 682 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 0.90 (t, *J* = 7.2 Hz, 3H), 1.38 (sextet, *J* = 7.2 Hz, 2H), 1.57 (quintet, *J* = 7.5 Hz, 2H), 1.98 (brs, 2H), 2.49 (t, *J* = 7.8 Hz, 2H), 2.54 (t, *J* = 7.2 Hz, 2H), 2.80 (t, *J* = 7.2 Hz, 2H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 13.66 (CH<sub>3</sub>), 21.95 (CH<sub>2</sub>), 27.44 (CH<sub>2</sub>), 31.61 (CH<sub>2</sub>), 32.03 (CH<sub>2</sub>), 35.99 (SCH<sub>2</sub>), 173.87 (CO). Anal. Calcd for C<sub>7</sub>H<sub>15</sub>NOS: C 52.14, H 9.38, N 8.69. Found: C 52.06, H 9.18, N 8.37%.

**3-(Cyclohexylsulfanyl) propanamide, 5dm** (C<sub>9</sub>H<sub>17</sub>NOS): Solid, M. P. 79 °C; IR (KBr): 3356, 3187, 1654, 1420, 1303, 682 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.21-1.31 (m, 5H), 1.5-1.6 (m, 1H), 1.73-1.74 (m, 2H), 1.92-2.0 (m, 2H), 2.47 (t, *J* = 7.5 Hz, 2H), 2.60-2.67 (m, 1H), 2.79 (t, *J* = 7.2 Hz, 2H), 6.0 (brs, 2H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 25.47 (CH<sub>2</sub>), 25.73 (CH<sub>2</sub>), 26.02 (CH<sub>2</sub>), 33.57 (CH<sub>2</sub>), 36.26 (SCH<sub>2</sub>), 43.82 (CH<sub>2</sub>), 174.21 (CO). Anal. Calcd for C<sub>9</sub>H<sub>17</sub>NOS: C 57.71, H 9.15, N 7.48. Found: C 57.27, H 8.93, N 7.13%.

**3-(Benzylsulfanyl) propanamide, 5dp** (C<sub>10</sub>H<sub>13</sub>NOS): Solid, M. P. 101 °C; IR (KBr): ν = 3354, 3182, 1634, 1413, 702 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 2.41 (t, *J* = 7.2 Hz, 2H), 2.73 (t, *J* = 7.2 Hz, 2H), 3.75 (s, 2H), 5.72 (brs, 2H), 7.30-7.33 (m, 5H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 26.95 (CH<sub>2</sub>), 36.57 (SCH<sub>2</sub>), 36.69 (CH<sub>2</sub>), 127.15 (ArCH), 128.59 (ArCH),

128.84 (ArCH), 138.21(ArC), 173.59 (CO). Anal. Calcd for C<sub>10</sub>H<sub>13</sub>NOS: C 61.15, H 6.71, N 7.17. Found: C 61.03, H 6.49, N 6.87%.

### Conclusions

In conclusion, we have demonstrated the usefulness of potassium phosphate as a highly efficient catalyst for the synthesis of a variety of thioethers following two different routes. The operational simplicity, substrate compatibility, chemoselectivity, very high regioselectivity, excellent yields and easy work up procedures are a few other noteworthy features of the developed protocols to have general applicability.

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