

Full Paper

## Fast and Highly Efficient Solid State Oxidation of Thiols

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**Abstract:** A fast and efficient solid state method for the chemoselective room temperature oxidative coupling of thiols to afford their corresponding disulfides using inexpensive and readily available moist sodium periodate as the reagent is described. The reaction was applicable to a variety of thiols giving high yields after short reaction times. Comparison of yield/time ratios of this method with some of those reported in the literature shows the superiority of this reagent over others under these conditions.

**Keywords:** Solid state reaction, oxidation, periodate, thiols, disulfides.

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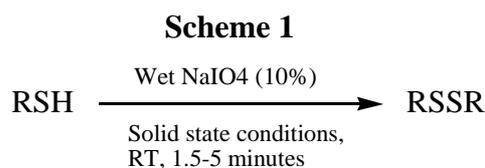
### Introduction

Oxidation of thiols to the corresponding disulfides is a characteristic functional group transformation, in which further oxidation(s) of the products to give disulfide S-oxides (thiolsulfonates), disulfide S-dioxides (thiolsulfonates), and sulfonic acids are possible, and consequently, considerable research has gone into controlling the initial oxidation. Weak S-S bonds in the disulfide products leads to high reactivity [1] and in natural products, the presence of these moieties and related cyclic analogues are associated with interesting biological activities and DNA-cleaving properties [2]. The vital role of the thiols and disulfides in living systems is due to their interconversion reactions so therefore oxidative coupling of the thiols under mild conditions has importance from both biological and practical points of view [3, 4].

Numerous methods have been reported for the selective oxidative coupling of thiols [5-22], but most of suffer from one or more disadvantages such as commercial unavailability toxicity or high cost of the required reagent(s), unfavorable conditions for preparing the reagent(s), long reaction times, over-oxidation, unpleasant work-ups or the need to use halogenated or other environmentally unfriendly solvents. Most reagents used are metal-based and toxic for the environment, while with others, such as halogens ( $\text{Br}_2$  and  $\text{I}_2$ ) and halogen containing reagents that are non-metallic, one encounters frequently difficulties in handling with the reagents and/or their separation from the products. In continuation of our recently reported work on the oxidation of thiols [23-26], herein we report a fast solid state method for controlled oxidation of the thiols to the corresponding disulfides using moist (10%) sodium periodate as an available, inexpensive and non toxic reagent.

## Results and Discussion

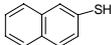
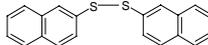
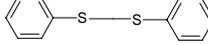
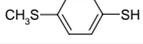
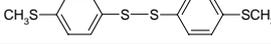
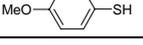
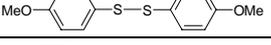
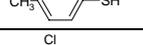
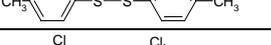
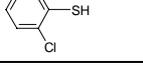
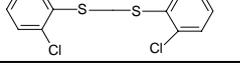
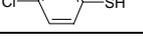
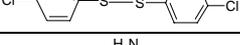
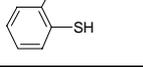
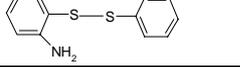
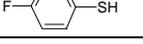
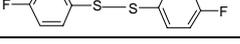
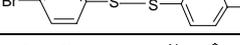
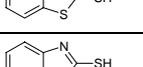
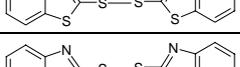
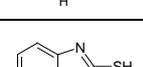
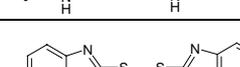
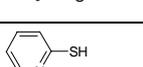
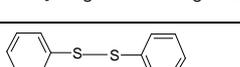
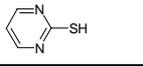
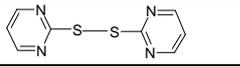
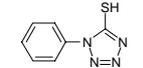
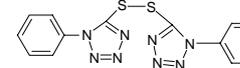
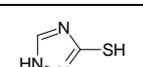
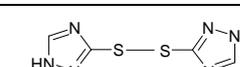
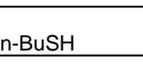
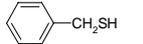
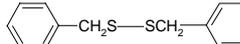
Oxidative coupling of a variety of thiols by treatment with moist sodium periodate (10% w/w) proceeds readily under solid state conditions by grinding together a mixture of the substrate with the wetted sodium periodate in a mortar (Scheme 1).



The reaction times are usually between 1.5-5 minutes. Initially, we decided to explore the role of moisture in a solventless system using the oxidation of 2-pyrimidine thiol as a typical substrate. We found that in the presence of moist sodium periodate (10% w/w) 2-pyrimidine thiol was oxidized to give bis(2-pyrimidyl)disulfide almost quantitatively in less than 2 minutes, whereas in the absence of moisture, the oxidation was observed to require longer reaction times (ca. 15 minutes) to go to completion. To optimize the oxidant, stoichiometry several molar ratios of sodium periodate to thiol including 1:1, 1:2, 1:3 and 1:4 were used. An optimum 1:2 molar ratio of sodium periodate to thiol was established as other ratios led to incomplete reactions or extended reaction times. For comparison purposes the same oxidation reaction was performed on 2-pyrimidine thiol in solution. It was interesting to note that the reaction was completed in nearly 12 minutes, that is, six times longer than under solid state conditions.

Regarding the stoichiometry of the reaction, with a 1:2 molar ratio of sodium periodate to thiol it is reasonable to assume that 1 mol of sodium periodate reacts with 2 mol of thiol to produce 1 mol of disulfide, 1 mol of  $\text{H}_2\text{O}$  and 1 mol of sodium iodate. Since sodium iodate is generally insoluble in the most organic solvents, the products were easily isolated by washing the reaction mixtures with a suitable solvent such as dichloromethane, acetonitrile and/or methanol depending on the solubility, followed by filtration, and drying of the filtrate on sodium sulfate. Evaporation of the filtrate under vacuum, followed by flash chromatography, often produced pure disulfides in high yields (Table 1). In the cases that methanol is used the filtration through silica-gel can be used to remove sodium iodate from the reaction mixture.

**Table 1.** Solid state oxidation of thiols to corresponding disulfides using moist (10%) sodium periodate at room temperature.

Entry	Thiol	Disulfide	Time (min.)	Yield <sup>a,b</sup> (%)	m.p.(°C) [lit.]
1			2	94	142-145 [18b]
2			2	90	61-62 [21]
3			2	95	40-43 [26]
4			2	90	76-80 [21]
5			2	96	43-44 [18b]
6			2	94	78-92 <sup>c</sup>
7			2	91	70-73 [25]
8			2	70	89-91 [25]
9			2	75	Oil [16c]
10			2	93	90-92 [18b]
11			2	94	178-179 [18b]
12			2	90	199-201 [25]
13			2	95	92-94 [140-150 (dec.)] [16d]
14			1.5	96	55-56 [18b]
15			1.5	95	143-145 [18b]
16			2	96	58-60 [21]
17			2	92	179-183 (dec) [16f]
18	n-BuSH	(n-BuS) <sub>2</sub>	4	93	Oil [18b]
19	c-C <sub>6</sub> H <sub>11</sub> SH	(c-C <sub>6</sub> H <sub>11</sub> S) <sub>2</sub>	5	92	Oil [18b]
20			2	91	68-70 [18b]

<sup>a</sup>The products were identified by comparison of their melting points and spectral data with those reported in the literature; <sup>b</sup> Refers to isolated yields; <sup>c</sup> The product was identified by IR, <sup>1</sup>H-NMR and MS spectra (see Experimental).

As may be seen, the method offers a simple, mild, and efficient solid state route for the oxidation of a variety of thiols to the corresponding disulfides. Notably, disulfides did not undergo further oxidation to their corresponding disulfide S-oxides (thiolsulfinates), disulfide S-dioxides (thiolsulfonates), and/or sulfonic acids under the reaction conditions. It must also be noted that no severe exothermal or other safety problems were observed during the mixing of the starting materials and the reactions themselves.

To illustrate the applicability of the method on larger laboratory scales, three reactions were carried out on gram scale for the oxidation of 2-mercapto-benzothiazole (10 mmol, 1.67 g), 2-naphthalenethiol (10 mmol, 1.60 g) and 2-bromothiophenol (10 mmol, 1.89 g) as typical substrates that afforded bis(benzothiazoyl)disulfide, bis(2-naphthyl)disulfide and bis(4-bromophenyl)disulfide in 94%, 92% and 90% isolated yield, respectively.

In order to show the advantages of the procedure, the results of the oxidation of some thiols by the current method have been compared to several ones previously reported in the literature (Table 2). As shown in this table, the yields of the products in all cases presented are similar and/or better whereas in all cases the reaction times are substantially shorter compared to the reported methods. Overall the results showed that wet sodium periodate in the solid state seems to be superior to the reported reagents in terms of non-toxicity, reagent availability, higher yields, shorter reaction times, milder conditions and easy work up at room temperature.

**Table 2.** Comparison of yield%/time (min.) (y/t) for the oxidation of some thiols by present method with those reported in the literature.

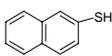
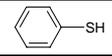
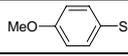
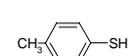
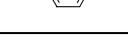
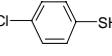
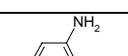
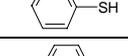
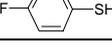
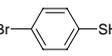
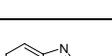
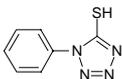
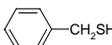
Entry	Thiol	y/t <sup>a</sup>	y/t <sup>b</sup>	y/t <sup>c</sup>	y/t <sup>d</sup>	y/t <sup>e</sup>	y/t <sup>f</sup> (y/t <sup>g</sup> )	y/t <sup>h</sup>	y/t <sup>i</sup>
1		94/2	95/5	93/60	76/45	-	91/15(93/2)	-	90/10, 90/10,
2		90/2	88/10	97/210	87/60	99/20	95/8(96/1)	87/5	-
3		90/2	-	-	86/45	-	-	86/5	-
4		96/2	95/5	91/60	84/45	-	91/6(95/0.75)	88/6	91/10, 91/10
5		91/2	85/10	-	-	70/1	92/10(93/1.5)	82/5	90/20, 90/30
6		78/2	95/15	-	-	-	-	88/6	-
7		75/2	-	83/90	-	92/35	-	-	75/60, -
8		93/2	-	95/60	-	-	92/10(94/1.5)	-	95/10, 95/10
9		94/2	90/5	-	75/120	-	92/18(94/3)	87/6	65/90, 65/30
10		90/2	85/5	89/240	-	-	-	86/5	60/90, 63/60
11		96/1.5	-	-	78/30	-	91/10(95/1.5)	85/5	80/40, 78/40

Table 2. Cont.

Entry	Thiol	y/t <sup>a</sup>	y/t <sup>b</sup>	y/t <sup>c</sup>	y/t <sup>d</sup>	y/t <sup>e</sup>	y/t <sup>f</sup> (y/t <sup>g</sup> )	y/t <sup>h</sup>	y/t <sup>i</sup>
12		95/1.5	-	-	-	-	92/12(93/2)	83/5	82/30, 76/60
13		96/2	-	-	75/60	-	-	-	-
14	n-BuSH	93/4	92/15	-	90/60	92/30	92/8(94/1.25)	88/6	
15	c-C <sub>6</sub> H <sub>11</sub> SH	92/5	90/15	77/330	-	99/60	90/9(90/1.5)	-	-, 92/20
16		91/2	88/20	70/300	82/30	91/40	91/7(94/1)	-	90/20, 90/20

Entries refer to <sup>a)</sup> the method described herein and other reported procedures using <sup>b)</sup> bipyridinium hydrobromide perbromide [22], <sup>c)</sup> 1,4-diazabicyclo[2.2.2]-octane-di-N-oxide diperhydrate [20], <sup>d)</sup> nitric acid [21], <sup>e)</sup> silica phosphoric acid/NaNO<sub>2</sub> [15c], <sup>f)</sup> n-butyltriphenylphosphonium dichromate (under M.W. irradiation) [18], <sup>g)</sup> tetrabutylammonium peroxydisulfate [16a], <sup>h)</sup> iron (III) – tetraphenylporphyrin using UHP and <sup>i)</sup> maleic anhydride/UHP [23-24].

## Conclusions

We have presented a new application of moist (10%) sodium periodate as an effective reagent for the solid state oxidation of the thiols that offers potential green chemistry advantages. It is important to note that most of the previously reported methods for the oxidation of thiols use synthesized or toxic reagents and/or suffer from other disadvantages such as long reaction times and low yields, but this method uses a readily available reagent and additionally offers the advantages of high yields, very short reaction times (1.5-5 min.), no side reactions, mild reaction conditions, a solid state reaction and simple isolation of the products. Consequently, this methodology should represent a good addition to list of methods already available for this purpose.

## Experimental Section

### General

All chemicals were purchased from Merck, Fluka and Aldrich and used without further purification. The products were characterized by a comparison of their physical (melting point) and spectral data with those reported in the literature [5-26]. All yields refer to isolated products. <sup>1</sup>H-NMR spectra were recorded on a Bruker DPX 250 MHz in CDCl<sub>3</sub>, CD<sub>3</sub>OD or DMSO-d<sub>6</sub> relative to TMS as an internal standard. IR spectra were run on a JASCO FT-IR-680 spectrophotometer. Mass spectra were recorded on an AMD 604 spectrometer, EI-mode at 70 ev, FT-mode at 0.005 V. All of the reactions were carried out in the solid state conditions in a fume hood with strong ventilation at room temperature.

*Typical procedure for the solid state oxidation of thiol: oxidation of 2-pyrimidinethiol*

Moist sodium periodate (10% w/w, 1 mmol, 0.237 g) was added to 2-pyrimidinethiol (2 mmol, 0.224 g) placed in a mortar. The reaction mixture was ground by pestle at room temperature under solvent-free condition for 1.5 minutes. After the disappearance of starting material as monitored by TLC, the mixture was washed twice with dichloromethane (2 × 8 mL) and filtered. The filtrate was evaporated under reduced pressure and the resulting crude material was purified by flash chromatography on SiO<sub>2</sub> (eluent: CH<sub>2</sub>Cl<sub>2</sub>) to afford bis 2-pyrimidine disulfide (95%); m.p = 132-135°C (Lit. [24] 134-136°C); <sup>1</sup>H-NMR (CDCl<sub>3</sub>): δ= 8.64 (m, 4H), 7.16 (t, 2H) ppm; MS m/z: 224 (M<sup>+</sup>), 222, 158, 143, 114, 112, 79.

The other substrates were treated similarly. Although a known compound, the analytical data for the product of entry 6 in Table 1, (2,6-dichlorophenyl) disulfide, was not readily accessible and is therefore reported here: IR (KBr, cm<sup>-1</sup>): 3076 (w), 1563 (s), 1552 (s), 1501 (w), 1483 (w), 1425 (vs), 1399 (sh), 1253 (w), 1188 (s), 1147 (m), 1085 (m), 1040 (w), 778 (vs), 705 (m), 516 (m), 486 (m), 475 (m); <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) δ ppm: (7.44, t, 4H), (7.15, q, 2H); MS (m/z): 354, 321, 284, 249, 214, 177, 142, 107, 69.

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*Sample Availability:* Samples of the compounds are available from authors.

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