7.87 (d, 1, H_1 or H_3), 7.94 (d, 1, H_5), 8.02 (d, 1, H_{12}), 9.13 (d, 1, H_7), 9.30 (s, 1, H_6) ($J_{2,3}$ = 7.8, $J_{4,5}$ = 8.6, $J_{7,8}$ = 8.8, $J_{11,12}$ = 8.7 Hz).

Acknowledgment. This research was supported by Grant No. CA 11968 from the National Cancer Institute, DHHS. The 500-MHz NMR spectrometer was funded in part through the University of Chicago Cancer Research Center Grant No. CA 14599. We also gratefully acknowledge the expert assistance of Dr. Hongmee Lee in the interpretation of the 500-MHz proton NMR spectra.

Registry No. 1, 202-94-8; 2, 201-42-3; 3, 81380-82-7; 4, 84877-34-9; 5b, 84877-35-0; 5c, 84877-36-1; 6, 84877-37-2; 7a, 84895-09-0; 7b, 84877-38-3; 8, 84877-39-4; 9, 78618-70-9; 10, 84877-40-7; 11, 84877-41-8; 12, 84877-42-9; 1,2-acenaphthylenedione, 82-86-0; *N,N*-diethylbenzamide, 1696-17-9; *N,N*-diethyl-1-naphthamide, 5454-10-4.

σ Adducts of 5-Nitropyrimidines with Liquid Ammonia and Their Oxidation into Aminonitropyrimidines[1]

Henk C. van der Plas,* Valery N. Charushin,[§] and Beb van Veldhuizen

Laboratory of Organic Chemistry, Agricultural University, Wageningen, The Netherlands

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The reactivity of azines toward potassium amide or liquid ammonia to form anionic σ complexes or neutral σ adducts has been the subject of numerous publications.[2] Highly electrophilic azines such as pteridines,[3] tetrazines,[4] or quaternary pyrimidinium salts[5] were found to react with liquid ammonia (free of amide ions) to give the corresponding amino adducts. These dihydro compounds can be regarded as intermediates in the nucleophilic displacement of hydrogen by ammonia.[6] Indeed, it has recently been shown that when 3-(*R*)-1,2,4,5-tetrazines[7] or pteridines[8] interact with liquid ammonia in the presence of potassium permanganate, the intermediate amino σ adducts were immediately oxidized to the corresponding amination products (Scheme I).

Because of the rather mild conditions that can be applied (low temperatures, no need to use the strong nucleophilic potassium amide) and good yields, this modification of the Chichibabin amination seems to be a very attractive and promising procedure for direct amination of azines.

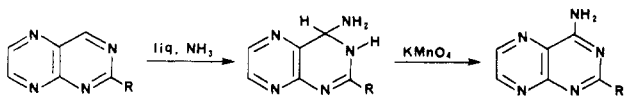
This paper describes the further successful application of this method in pyrimidine chemistry.

Results and Discussion

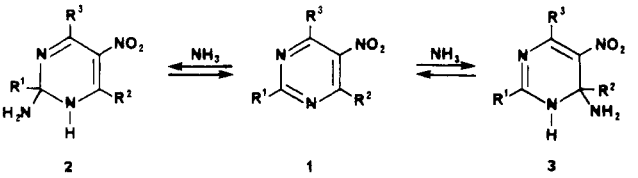
σ Adducts of 5-Nitropyrimidine with Ammonia. A solution of 5-nitropyrimidine (**1a**) in liquid ammonia was found to give, in the temperature range between −60 and −33 °C, two different σ adducts, i.e., **2a** and **3a** (Scheme II), as shown by NMR spectroscopy.

A. ^{1}H NMR Data. The ^{1}H NMR spectrum of a solution of **1a** in liquid ammonia at −60 to −40 °C recorded immediately after preparation of the solution consists mostly of two intense peaks at δ 8.18 (d, 2 H) and 5.33 (t, 1 H) with a small coupling constant J = 1.3 Hz (Figure 1a, Table I). A large upfield shift was found for the resonance signal of one of the protons of **1a** (Δδ 4.17) while the two other protons are shifted much less (Δδ 1.41). This result can only be explained if addition of ammonia to C-2 of

Scheme I



Scheme II



2 1 3

	R¹	R²	R³
a)	H	H	H
b)	H	D	D
c)	CH_3	H	H
d)	SCH_3	H	H
e)	C_6H_5	H	H
f)	CH_3SO_2	H	H
g)	H	OCH_3	OCH_3
h)	H	OCH_3	H

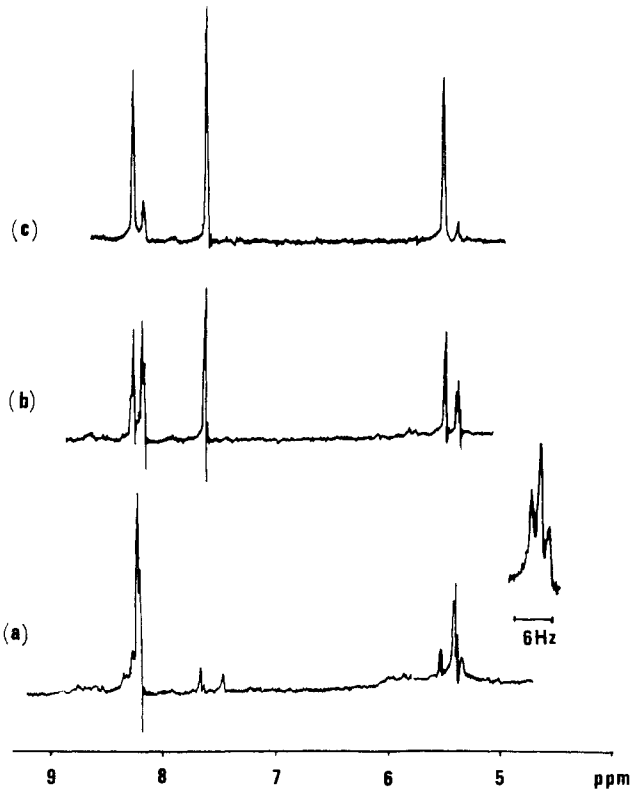


Figure 1. ^{1}H NMR spectra of 5-nitropyrimidine in liquid ammonia at −40 °C: (a) immediately after preparation of a solution, (b) after 15 min, (c) after 1 h.

[§] Postdoctoral fellow. Permanent address: The Department of Organic Chemistry, Urals Polytechnical Institute, Sverdlovsk, 620002, USSR.

(1) Part 94 on pyrimidines. For part 93, see: Charushin, V. N.; van der Plas, H. C. *Tetrahedron Lett.* **1982**, 3965. Part 30 on S_N(ANRORC) mechanism. For part 29, see: Rykowski, A.; van der Plas, H. C. *J. Heterocycl. Chem.* **1982**, *47*, 2856.

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0022-3263/83/1948-1354$01.50/0

Table I. ^{1}H NMR Data of 5-Nitropyrimidines

starting pyrimidine	solvent	temp, °C	adduct	chemical shifts, δ H-2	H-4	H-6	R
1a	$CDCl_3$	+20		9.50	9.50	9.50	
1a	CD_3OD	+20		9.50	9.59	9.59	
1a	NH_3	−40	2a	5.33, t	8.18, d, J = 1.3 Hz	8.18, d	
			3a	7.57	5.45	8.22	
1b	NH_3	−45	2b	5.38			
			3b	7.60			
1c	$CDCl_3$	+20			9.43	9.43	2.91 (3 H)
1c	NH_3	−55	3c		5.32	8.25	1.95 (3 H)
1d	$CDCl_3$	+20			9.28	9.28	2.68 (3 H)
1d	NH_3	−55	3d		5.38	8.15	2.17 (3 H)
1e	$CDCl_3$	+20			9.54	9.54	7.5-7.8 (m, 3 H), 8.4-8.7 (m, 2 H)
1e	NH_3	−45	3e		5.55	8.47	7.2-7.6 (m, 3 H), 8.0-8.3 (m, 2 H)
1f	$CDCl_3$	+20			9.63	9.63	3.43 (3 H)
1f	NH_3	−45	3f		5.48, d, J = 1.5 Hz	8.25, d	3.12 (3 H)
1g	$CDCl_3$	+20		8.46			4.12 (6 H)
1g	NH_3	−40	*a*				
1h	$CDCl_3$	+20		8.93	9.17		4.22 (3 H)
1h	NH_3	−55	2h	5.44, d, J = 1.3 Hz	8.30, d		3.51 (3 H)

[a] Failed to register because of low solubility.

Table II. Carbon-13 Chemical Shifts of Some 5-Nitropyrimidines, δ

starting pyrimidine	solvent	temp, °C	adduct	C-2	C-4	C-5	C-6	R
1a	$CDCl_3$	+20		162.4	152.4	142.4	152.4	
1a	NH_3	−50	2a	80.8	151.3	116.1	151.3	
			3a	157.1	62.0	118.3	152.1	
1d	$CDCl_3$	+20		179.7	152.4	138.8	152.4	15.0
1d	NH_3	−40	3d	171.7	62.8	120.4	153.3	13.6
1h	$CDCl_3$	+20		160.9	161.7		154.2	55.6
1h	NH_3	−50	2h	81.6	156.9	109.5	153.5	51.6

5-nitropyrimidine to form **2a** has taken place. Besides these two intense peaks, there are three small ones present in the spectrum which increase gradually at the expense of a simultaneous decrease of the 2-amino adduct (**2a**) signals (Figure 1b). Allowing the solution to stand for 1 h at −40 °C results in almost complete disappearance of the signals attributed to **2a**, and the presence of only three one-proton singlets is observed (Figure 1c). The same result can be obtained if the reaction mixture is allowed to stand for 5 min at room temperature (sealed thick-wall tube). No further changes in the ^{1}H NMR spectra were observed when the solution was kept for several hours at −33 °C. The signals at δ 5.45, 7.57, and 8.22 are unquestionably ascribed to H-4, H-2, and H-6 of **3a**, respectively. All the absorptions could be assigned unequivocally by measuring the ^{1}H NMR spectra of [4,6-$^2H_2$]-5-nitropyrimidine (**1b**) in liquid ammonia (Table I). The observed ^{1}H NMR parameters of **2a,b** and **3a,b** are in good agreement with those reported for 5-nitropyrimidine–methoxide σ adducts.[9]

B. ^{13}C NMR Data. Since 2-amino adduct **2a** is not stable and even at −60 °C converts gradually into 4-isomer **3a**, ^{13}C NMR spectra of the reaction mixture at −50 °C display signals of both σ adducts **2a** and **3a**. Observing changes in spectra in the course of the reaction makes it possible to distinguish signals of isomers and to assign them correctly (Table II). The characteristic feature of the ^{13}C NMR spectra of **2a** and **3a** is the great difference between chemical shifts of the sp^3 carbon of C-2 in **2a** (δ 80.8) and C-4 in **3a** (δ 62.0) (Table II). These values are quite close to those found for C-2 (δ 84.6 and C-6 (δ 63.7) in the σ adducts formed between 4-phenylpyrimidine and potassium amide.[10]

^{1}H and ^{13}C NMR data show clearly that the formation of 2-amino adduct **2a** in the low-temperature reaction of **1** with ammonia is a kinetically controlled reaction but that the concurrent addition of ammonia at C-4 leads to the more stable σ adduct **3a**. That σ adduct **3a** is more stable than σ adduct **2a** can possibly be explained by an extended conjugation between the NH group in the ring and the nitro group, being less in **2a**. Some intramolecular hydrogen bonding between the amino protons and the nitro group may also contribute to the higher stability of **3a**. Similar factors effecting the stability of 1,5-naphthyridine–amide ion σ adducts have recently been considered.[11]

2. Substituted 5-Nitropyrimidines. In order to investigate the influence of substituents on the formation of amino adducts **2** and **3**, we measured ^{1}H and ^{13}C NMR spectra of some substituted 5-nitropyrimidines in liquid ammonia (Tables I and II).

When position 2 of the pyrimidine ring is substituted by a methyl (**1c**), methylthio (**1d**), phenyl (**1e**) or me-

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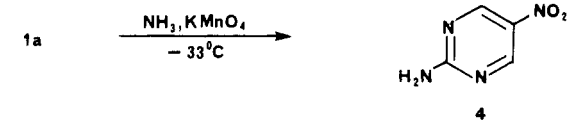
Table III. Amination of 5-Nitropyrimidines by Liquid Ammonia in the Presence of $KMnO_4$[a] (1 Redox Equiv)

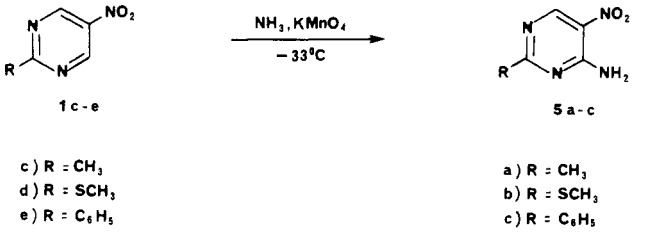
starting pyrimidine	reaction temp, °C	product	mp, °C (lit. mp, °C)	yield, %	^{1}H NMR data in Me_2SO-d_6, δ
1a	−33	4	234-236 (236)[c]	45	9.06 (s, 2 H) 8.20 (br s, 2 H)
1c	−33	5a	decomp above 230[d] (277-278)[16]	53	9.00 (s, H-6), 8.0-9.0 (br, 2 H), 2.45 (s, 3 H)
1d	−33	5b	183-184	72	9.00 (s, H-6), 7.9-8.6 (br, 2 H), 2.42 (s, 3H)
1e	−33	5c	213-216	50	9.28 (s, H-6), 8.2-8.5 (m, 2 H), 7.3-7.7 (m, 3 H), 7.2-8.7 (br, 2 H)
1g	−33	6	186-187	53	7.60 (br, 2 H), 3.90 (s, 6 H)
1h	−65	8	225-227	50	8.87 (s, H-6), 8.00 (br, 2 H), 3.98 (s, 3 H)
1h	+20[b]	7	210-212 (212-214)[16]	65	9.13 (s, H-6), 8.67 (s, H-2), 8.0-9.2 (br, 2 H)

[a] Satisfactory analytical values (±0.3%, for C and H) and consistent mass spectral data were reported for all compounds in the table. [b] Without $KMnO_4$. [c] Reference 12. [d] When preheating a microscope up to 240 °C crystals were not melted, but changed around 250 °C.

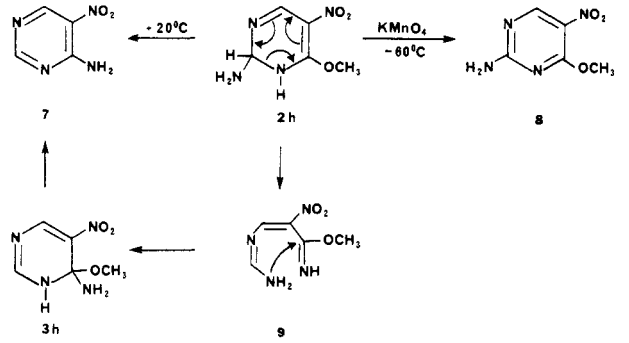
Scheme III



Scheme IV



Scheme V



thylsulfonyl group (**1f**), only the peaks of 4-amino adducts **3c–f** appear in the NMR spectra. The adduct **3e** is formed only in a small concentration because of low solubility. The adduct **3c** is not stable and decomposes rapidly even at −55 °C, but adducts **3d,f** are quite stable at −55 to −40 °C (Tables I and II). No adducts are formed when 2-amino-5-nitropyrimidine is dissolved in liquid ammonia. The 2-amino adduct **2g** is supposed to form when 4,6-dimethoxy-5-nitropyrimidine (**1g**) reacts with ammonia (see data on the oxidation of σ adducts presented below), although it cannot be seen in the ^{1}H NMR spectrum due to its low solubility.

When 4-methoxy-5-nitropyrimidine (**1h**) is dissolved in liquid ammonia, only 2-amino adduct **2h** is formed, which is quite stable at temperatures below −33 °C (Tables I and II).

3. Oxidation of σ Adducts 2 and 3. As mentioned before, potassium permanganate is a very useful reagent to oxidize dihydroazines. For oxidizing the σ adducts **2** and **3** at low temperatures into the corresponding 2-amino- or 4-aminopyrimidines, potassium permanganate was also found to be appropriate.

Since the reaction of **1a** with ammonia results in σ adducts **2a** and **3a**, the sequence in which reagents are mixed is of importance. When 5-nitropyrimidine (**1a**) was added to a solution of potassium permanganate in liquid ammonia at −33 °C, 2-amino-5-nitropyrimidine (**4**) could be isolated in 45% yield. The product **4** was identified with an authentic sample[12] by IR and ^{1}H NMR spectra and also mixed-melting-point determination (Scheme III).

All attempts to obtain 4-amino-5-nitropyrimidine by addition of potassium permanganate to a solution of **1a** in liquid ammonia, being preliminary kept for 1–3 h at −33 °C to convert **2a** into **3a**, have failed; a complex mixture of unidentified products was formed from which only 2-amino-5-nitropyrimidine could be isolated in poor yield (5%). Thus, immediate oxidation of the kinetically controlled σ adducts appears to be the method to apply for amination of pyrimidines. 4-Aminopyrimidines **5a–c** are formed in good yield when 2-substituted pyrimidines **1c–e** are added to liquid ammonia containing potassium permanganate (Table III, Scheme IV).

This method is also successful in the case where the concentration of a σ adduct is too small to be registered by means of ^{1}H NMR spectroscopy. Thus, the addition of 4,6-dimethoxy-5-nitropyrimidine (**1g**) to a solution of potassium permanganate in liquid ammonia results in 2-amino-4,6-dimethoxy-5-nitropyrimidine (**6**) in good yield. The structures of compounds **5a–c** and **6** have been confirmed by IR, ^{1}H NMR, and mass spectroscopy data as well as elemental analyses (Table III).

The adduct **2h**, resulting from the addition of ammonia at C-2 of 4-methoxy-5-nitropyrimidine (**1h**), can be oxidized by potassium permanganate at −60 to −70 °C into the corresponding 2-amino-4-methoxy-5-nitropyrimidine (**8**). However, if a solution of **1h** in liquid ammonia is allowed to stand for 5 min at room temperature, 4-amino-5-nitropyrimidine (**7**) is formed. On the basis of our results obtained in earlier studies,[2] we suggest that the conversion of **2h** → **7** occurs according to the S_N(AN-RORC) process involving the intermediates **9** and **3h** (Scheme V).

In conclusion it is clear that low-temperature oxidative amination is a very effective method for the introduction of an amino group into the pyrimidine ring even in the presence of readily replaceable groups.

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Experimental Section

Melting points are uncorrected. The ^1H NMR spectra were recorded on a Hitachi Perkin-Elmer R 24B spectrometer and Varian EM 390 spectrometer equipped with a Varian EM 3940 variable-temperature controller. Me_4Si was used as internal standard (δ = 0 ppm). In liquid ammonia the solvent peak was used as the standard (δ = 0.95 ppm). The ^{13}C NMR spectra were recorded at 75.46 MHz on a Bruker CXP-300 spectrometer equipped with a B-VT 1000 variable-temperature controller. For measurements in anhydrous liquid ammonia, thick-wall 5- and 10-mm o.d. tubes were used for ^1H and ^{13}C NMR spectroscopy, respectively. The latter contained an internal 3-mm capillary with acetone-d_6. This was used both for the lock signal and as internal standard (δ = 29.8 ppm). Spectral parameters: 15000-Hz spectral width, 0.27 s acquisition time, 1.2 s pulse delay. IR spectra were recorded on a Hitachi EPG-3 spectrometer.

Starting Materials. 5-Nitropyrimidine,[13] 2-methyl-5-nitropyrimidine,[14] 2-(methylthio)-5-nitropyrimidine,[15] 2-phenyl-5-nitropyrimidine,[12] 2-(methylsulfonyl)-5-nitropyrimidine,[15] and 4-methoxy-5-nitropyrimidine[16] were synthesized as described in the literature.

2-Amino-5-nitropyrimidine (4). Potassium permanganate (440 mg, 1.05 redox equiv) was dissolved in liquid ammonia (ca. 20 mL), and 5-nitropyrimidine (**1a**) (500 mg, 4 mmol) was added to this solution in one portion with stirring. After 15 min, methanol (25 mL) was slowly added through a dry ice/acetone condenser, and the reaction mixture was allowed to stand at room temperature for 1 h. Purification by column chromatography on silica gel using ethanol as eluent gave crystals (250 mg, 45%): mp 234–236 °C (lit.[12] 236 °C) ^1H NMR (Me_2SO-d_6) δ 9.06 (s, 2 H), 8.20 (br s, 2 H).

Aminopyrimidines 5a–c and 6 were obtained analogously to the procedure described above. Analytically pure compounds were isolated by TLC on silica gel plates with chloroform/ethyl acetate (20:1) followed by recrystallization from ethanol. All data of **5a–c** and **6** are collected in Table III.

Reaction of 4-Methoxy-5-nitropyrimidine (1h) with Ammonia. A. Without $KMnO_4$. 4-Methoxy-5-nitropyrimidine (310 mg, 2 mmol) was dissolved in 5 mL of liquid ammonia, and the reaction mixture was allowed to stand at room temperature for 10 min (sealed tube was used). After cooling, ammonia was evaporated slowly and the residue recrystallized from ethanol, yielding 210 mg (65%) of **7**: mp 210–212 °C (lit.[16] 212–214 °C) (Table III).

B. In the Presence of $KMnO_4$. 4-Methoxy-5-nitropyrimidine (310 mg, 2 mmol) was added in small portions to a solution of potassium permanganate (220 mg, 1.05 redox equiv) in 20 mL of ammonia at –60 to –70 °C with stirring. The reaction mixture was kept for 15–20 min at –60 °C, and then methanol (20 mL) was added through a dry ice/acetone condenser. 2-Amino-4-methoxy-5-nitropyrimidine (170 mg, 50%) was isolated by column chromatography on silica gel with ethanol: mp 225–227 °C; ^{13}C NMR ($(CD_3)_2SO$) δ 163.8, 162.7 (C-2 and C-4), 158.2 (C-6, $^1J_{C\text{-}H}$ = 189.1 Hz), 123.0 (C-5). For comparison, $^1J_{C\text{-}H}$ values of 5-nitropyrimidine are 207 Hz for C-2 and 192 Hz for C-4(6).

Acknowledgment. We are indebted to Dr. M. A. Posthumus for providing mass spectra and to Mr. H. Jongejan for microanalyses.

Registry No. **1a**, 14080-32-1; **1b**, 84928-77-8; **1c**, 14080-34-3; **1d**, 14001-70-8; **1e**, 68906-00-3; **1f**, 65735-65-1; **1g**, 15846-14-7; **1h**, 15579-58-5; **2a**, 84928-78-9; **2b**, 84928-79-0; **2h**, 84944-06-9; **3a**, 84928-80-3; **3b**, 84944-07-0; **3c**, 84928-81-4; **3d**, 84928-82-5; **3e**, 84928-83-6; **3f**, 84928-84-7; **4**, 3073-77-6; **5a**, 15579-59-6; **5b**, 84928-85-8; **5c**, 84928-86-9; **6**, 84928-87-0; **7**, 15568-46-4; **8**, 84928-88-1; NH_3, 7664-41-7.

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Direct Synthesis of Thioethers from Thiols and Alcohols

Yvan Guindon,* Richard Frenette,* Réjean Fortin, and Joshua Rokach

Merck Frosst Canada Inc., Pointe-Claire/Dorval, Québec, Canada H9R 4P8

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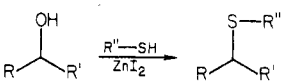
Herein we report a very simple and convenient synthesis of a wide range of thioethers. It is based on the finding that thiols in the presence of zinc iodide react smoothly with activated alcohols to give the corresponding thioethers in excellent yields, according to the following scheme:



R = aryl, alkenyl; R' = H, alkyl, aryl; R'' = alkyl, aryl

This work grew out of our research into methods for the synthesis of the leukotrienes,[1] related analogues,[2] and potential antagonists or biosynthetic inhibitors, in which it was necessary to develop mild procedures for the formation of a variety of substituted thioethers. In this regard we have developed approaches based on the sulfenyllactonization reaction[2] and the reaction of thiosilane reagents with epoxides.[3]

The present reaction, involving the use of zinc iodide and alcohols, is attractive for its simplicity, effectiveness, and the very mild conditions employed. These advantages were demonstrated dramatically in the synthesis of [1-(phenylthio)pentyl]benzene. Thus reaction of the phenyl thiolate anion with (1-chloropentyl)benzene (Williamson's ether synthesis conditions[4]) lead mainly to elimination of HCl from the alkylating agent, while attempts to prepare the tosylate of 1-phenyl-1-pentanol yielded the ether of the starting alcohol as the principal product. These experiments underline the sensitivity of such systems (benzylic or allylic alkylating agents) to either base or nucleophile, which renders the "classical" approach difficult.

In sharp contrast, with use of the new conditions herein described, thiophenol reacted with 1-phenyl-1-pentanol to yield the desired thioether in 88% yield (Table I, entry 3). In this same context, it is worth pointing out that even a tertiary benzylic alcohol gives a high yield (81%) of thioether, without significant olefin formation (Table I, entry 6).

Although acid-catalyzed displacement of alcohols by thiols has found some isolated examples in the literature,[5] no effort has been made to develop this methodology into a sound and efficient approach to sulfides (thioethers) and to determine its scope and limitations.

Results and Discussion

The choice of Lewis acid was based on the following observations: the alcohols and thiols used are stable to zinc

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