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ALKYLATION OF *N*-ARYLCYANAMIDES AND ELECTRON-DEFICIENT PHENOLS WITH (CHLOROMETHYL)THIIRANE

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Alkylation of N-arylcyanamides with (chloromethyl)thiirane in aqueous alkaline solution provides an easy synthetic approach to N-aryl-N-(thietan-3-yl)cyanamides. Yields vary from 34 to 76% and are lower in the case of electron-deficient aryl substituents. The reaction with phenols containing electron-withdrawing groups results in formation of 3-(aryloxy)thietanes in 19–45% yields.

Keywords: 3-aminothietanes, *N*-arylcyanamides, (chloromethyl)thiirane, phenols, thiirane-thietane rearrangement.

Small molecules comprise a vast majority of successfully designed, tested, and clinically approved drugs [1, 2]. Structure-activity relationship practices used in early steps of the molecular design call for a large variety of substituents that one should be able to install onto the desired scaffold for the screening purposes. Therefore, simple and selective methods for the functionalization of organic molecules (as can be illustrated by examples of *O*-alkylation of phenols and *N*-acylation and *N*-sulfonylation of amines) play an important role whenever it is necessary to quickly achieve high molecular diversity or optimize the substrate-target interaction by varying the substitution pattern of the substrate. The absence of transition metal catalyst in such reactions is an additional advantage if bioactivity tests are in view.

Small-ring heterocycles provide an attractive extension to the small alkyl and cycloalkyl set of substituents, as the presence of a heteroatom allows for an additional hydrogen bonding or dipole interactions between the substrate molecule and its target. Three-membered heterocycles, such as oxirane or aziridine fragments, often introduce unwanted reactivity because of the possibility of ring-opening reactions resulting in formation of electrophilic species. Four-membered heterocyclic fragments containing one heteroatom are inherently stable but notoriously difficult to synthesize. In this work, a simple and versatile method of introduction of 3-thietanyl substituent is discussed.

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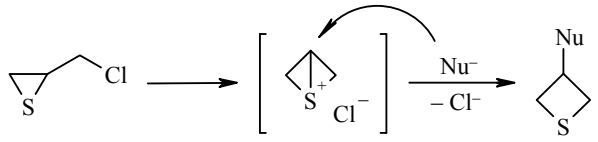
The thiirane-thietane rearrangement taking place on interaction of (α-haloalkyl)thiiranes and hard weak nucleophiles (most commonly, phenolates and carboxylates) in the presence of base has been known for over 40 years [3]. The postulated mechanism involves formation of 1-thioniabicyclobutane, which is then attacked at 3 position by a nucleophile [4]:



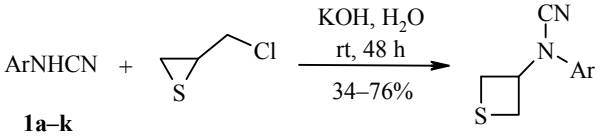
More recently, the reaction was successfully expanded to include aromatic nitrogen heterocycles (benzimidazoles [5, 6], xanthines [5], triazoles [7]) as nucleophilic partners. In our group, the reactions of (α-haloalkyl)thiiranes with *N*-arylsulfamides [8] and isatins [9] have been demonstrated to provide access to 3-substituted thietanes in satisfactory yields.

Our purpose was to further expand the scope of *N*-nucleophilic substrates suitable for the alkylation proceeding with thiirane-thietane rearrangement. *N*-Arylcyanamides have acidities similar to those of both phenols and *N*-arylsulfamides and are soluble in aqueous alkali metal hydroxides; they have also been successfully alkylated under various conditions [10, 11]. We therefore expected them to exert similar nucleophilic behavior towards (chloromethyl)thiirane.

Several synthetic approaches to *N*-arylcyanamides, which are generally not commercially available, have been reported in the literature. Desulfurization of arylthioureas in aqueous base requires stoichiometric amounts of a heavy metal salt (usually $Pb(OAc)_2$ [12], but mercury(II) [13], copper(II) [14] or silver(I) [15] salts may be used instead), which is both toxic and possess significant danger to the environment. Direct cyanation of anilines involves the use of highly toxic cyanogen bromide or chloride [11, 16, 17], or alkali metal cyanides [18, 19]. Aryl isothiocyanates also afford *N*-arylcyanamides in high yields when treated with NaHMDS in THF at room temperature [20]. More recently, an efficient method for the synthesis of 1-aryltetrazoles has been proposed [21]. The latter compounds produce *N*-arylcyanamides when treated with KOH in DMSO [22, 23].

The series of *N*-arylcyanamides **1a-k** was prepared using either of these methods. Many of the compounds **1a-k** were found to have limited stability at room temperature, gradually forming products that were insoluble in aqueous alkali (likely dimers or polymers [24, 25]), and were therefore either used immediately upon drying or stored only briefly in the freezer.

Alkylation of *N*-arylcyanamides **1a-k** with (chloromethyl)thiirane in aqueous potassium hydroxide over 48 h resulted in formation of the target *N*-aryl-*N*-(thietan-3-yl)cyanamides **2a-k** in moderate to good yields (Tables 1, 2).



1a–k **2a–k**

a Ar = Ph, **b** Ar = 2,6-$Me_2C_6H_3$, **c** Ar = 4-$MeOC_6H_4$, **d** Ar = 1-naphthyl, **e** Ar = 2-ClC_6H_4, **f** Ar = 4-ClC_6H_4, **g** Ar = 4-BrC_6H_4, **h** Ar = 3-FC_6H_4, **i** Ar = 2-$O_2NC_6H_4$, **j** Ar = 3-$O_2NC_6H_4$, **k** Ar = 4-$O_2NC_6H_4$

The products **2a**,**b** are viscous colorless oils, compounds **2c-k** are crystalline solids, and all are stable at room temperature.

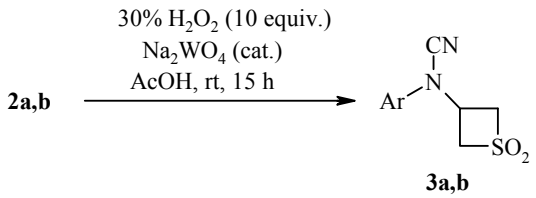
TABLE 1. Characteristics of the Synthesized *N*-Aryl-*N*-(thietan-3-yl)cyanamides **2a-k**

Compound	Empirical formula	mp, °C	Yield, %	Appearance
2a	$C_{10}H_{10}N_2S$	—	72	Yellowish oil
2b	$C_{12}H_{14}N_2S$	—	34	Colorless oil
2c	$C_{11}H_{12}N_2OS$	67–68	59	Colorless crystals
2d	$C_{14}H_{12}N_2S$	114–115	38	Colorless crystals
2e	$C_{10}H_9N_2SCl$	48–50	63	Colorless crystals
2f	$C_{10}H_9N_2SCl$	104–105	76	Colorless crystals
2g	$C_{10}H_9BrN_2S$	116–117	69	Colorless crystals
2h	$C_{10}H_9N_2FS$	65–66	74	Colorless crystals
2i	$C_{10}H_9N_3O_2S$	79–80	44	Yellow crystals
2j	$C_{10}H_9N_3O_2S$	98–99	59	Yellow crystals
2k	$C_{10}H_9N_3O_2S$	167–168	38	Yellow crystals

TABLE 2. NMR Spectra of Cyanamides **2a-k**

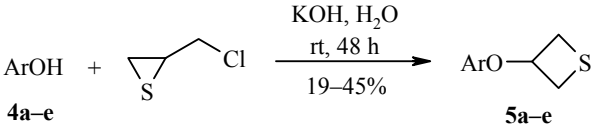
Com-pound	^{1}H NMR spectrum, δ, ppm (*J*, Hz)	^{13}C NMR spectrum, δ, ppm (J_{C-F}, Hz)
2a	3.23-3.32 (2H, m, CH_2); 3.69-3.79 (2H, m, CH_2); 4.87-5.01 (1H, m, CHN); 7.00-7.13 (3H, m, H-2,4,6); 7.27-7.36 (2H, m, H-3,5)	34.2; 54.2; 111.8; 117.1; 125.0; 130.3; 139.3
2b	2.30 (6H, s, Me); 3.21-3.28 (2H, m, CH_2); 3.88-3.95 (2H, m, CH_2); 4.24-4.35 (1H, m, CHN); 7.04-7.09 (2H, m, H-3,5); 7.10-7.15 (1H, m, H-4)	18.1; 34.9; 58.5; 112.8; 128.6; 129.0; 135.5, 137.1
2c	3.23-3.32 (2H, m, CH_2); 3.72-3.82 (2H, m, CH_2); 3.76 (3H, s, MeO); 4.77-4.91 (1H, m, CHN); 6.83-6.91 (2H, m, H-2,6); 6.97-7.05 (2H, m, H-3,5)	34.2; 55.7; 56.0; 112.8; 115.5; 120.1; 132.5, 157.6
2d	3.25-3.35 (2H, m, CH_2); 3.90-4.00 (2H, m, CH_2); 4.68-4.82 (1H, m, CHN); 7.37-7.50 (2H, m, H Ar); 7.54-7.67 (2H, m, H Ar); 7.82-8.00 (3H, m, H Ar)	34.5; 59.2; 114.2; 122.2; 123.3; 125.9; 127.5; 128.0; 129.0; 129.2; 129.5; 135.1; 135.9
2e	3.25-3.32 (2H, m, CH_2); 3.84-3.91 (2H, m, CH_2); 4.63-4.73 (1H, m, CHN); 7.27-7.34 (3H, m H Ar); 7.45-7.50 (1H, m, H-3)	34.2; 58.2; 112.3; 127.0; 128.3; 129.5; 130.5; 131.2; 136.5
2f	3.28-3.36 (2H, m, CH_2); 3.76-3.85 (2H, m, CH_2); 4.89-5.00 (1H, m, CHN); 6.98-7.05 (2H, m, H-2,6); 7.29-7.36 (2H, m, H-3,5)	33.5; 53.9; 110.8; 117.9; 129.8; 129.9; 137.4
2g	3.27-3.36 (2H, m, CH_2); 3.75-3.83 (2H, m, CH_2); 4.89-5.00 (1H, m, CHN); 6.92-6.98 (2H, m, H-2,6); 7.42-7.49 (2H, m, H-3,5)	33.5; 53.8; 110.7; 117.3; 118.1; 132.7; 137.9
2h	3.33-3.39 (2H, m, CH_2); 3.81-3.88 (2H, m, CH_2); 4.95-5.05 (1H, m, CHN); 6.79-6.92 (3H, m, H-2,4,6); 7.31-7.39 (1H, m, H-5)	33.5; 53.7; 104.2 (26.7); 110.6; 111.4 (21.4); 111.9 (3.1); 131.3 (9.5); 140.4 (9.9); 163.4 (248.3)
2i	3.26-3.33 (2H, m, CH_2); 3.82-3.92 (2H, m, CH_2); 4.57-4.67 (1H, m, CHN); 7.43 (1H, dd, *J* = 1.2, *J* = 7.9, H-6); 7.51 (1H, dt, *J* = 1.2, *J* = 7.9, H-4); 7.69 (1H, dt, *J* = 1.2, *J* = 7.9, H-5); 8.01 (1H, dd, *J* = 1.2, *J* = 7.9, H-3)	34.4; 59.2; 111.4; 126.3; 127.5; 129.1; 133.0; 134.7; 144.0
2j	3.37-3.44 (2H, m, CH_2); 3.81-3.89 (2H, m, CH_2); 5.05-5.15 (1H, m, CHN); 7.45-7.50 (1H, m, H-6); 7.59 (1H, t, *J* = 8.3, H-5); 7.89 (1H, t, *J* = 2.5, H-2); 7.97-8.02 (1H, m, H-4)	33.4; 53.6; 109.8; 110.7; 119.1; 122.2; 131.0; 140.0; 149.0
2k	3.36-3.44 (2H, m, CH_2); 3.82-3.89 (2H, m, CH_2); 5.08-5.18 (1H, m, CHN); 7.18-7.24 (2H, m, H-2,6); 8.24-8.30 (2H, m, H-4,5)	33.3; 53.3; 109.3; 115.8; 125.8; 143.8; 144.0

Thietanes **2a**,**b** were oxidized to crystalline sulfones **3a**,**b** using hydrogen peroxide in acetic acid in the presence of Na_2WO_4 catalyst:



3 **a** Ar = Ph (86%), **b** Ar = 2,6-$Me_2C_6H_3$ (75%)

Serious problems were previously encountered when *N*-nucleophiles bearing strong electron-withdrawing group were alkylated with (chloromethyl)thiirane. Thus, almost no thietanes were formed in the reactions of *N*-(4-nitrophenyl)methanesulfonamide or benzenesulfonamides with (chloromethyl)thiirane [8]. In the same manner, 5-nitroisatin was not alkylated by this reagent in aqueous media [9]. Fortunately, the presence of an electron-withdrawing group conjugated with the reaction center does not show such pronounced effect on the reaction with *N*-arylcyanamides. For this reason, we have decided to reinvestigate the thietanylation of nitrophenols **4a**–**e** under identical conditions. As shown in the scheme below, this reaction provides the corresponding 3-(aryloxy)thietanes **5a**–**e** in acceptable yields:



a Ar = 2-$O_2NC_6H_4$, **b** Ar = 3-$O_2NC_6H_4$, **c** Ar = 4-$O_2NC_6H_4$,
d Ar = 5-Me-2-$O_2NC_6H_3$, **e** Ar = 3-Me-4-$O_2NC_6H_3$

We have shown the synthetic utility of the thiethanylation reaction for *N*-arylcyanamides and electron-deficient phenols. These substrates provide convenient access to novel thietane derivatives. Further studies on the synthetic approaches to thietanes, their ring opening reactions and their possible applications in medicinal chemistry are currently underway.

EXPERIMENTAL

The ^1H and ^{13}C NMR spectra were recorded on a Bruker DPX-300 spectrometer in $CDCl_3$ using $CHCl_3$ residual signal (δ 7.26 ppm) as internal standard for ^1H NMR and $CHCl_3$ (δ 77.0 ppm) for ^{13}C NMR. Elemental analyses were performed on a Hewlett-Packard HP-185B CHN-analyzer. Low resolution mass spectra (EI

TABLE 3. Characteristics of the Synthesized 3-(Aryloxy)thietanes **5a–e**

Com-pound	Empirical formula	Found, *m/z* Calculated, *m/z*	mp, °C	Yield, %	Appearance
5a	$C_9H_9NO_3S$	211.0294 $[M]^+$ 211.0303	44-45	35	Yellowish crystals
5b	$C_9H_9NO_3S$	211.0305 $[M]^+$ 211.0303	57-58	33	Yellowish crystals
5c	$C_9H_9NO_3S$	211.0306 $[M]^+$ 211.0303	99-100	39	Yellowish flakes
5d	$C_{10}H_{11}NO_3S$	248.0352 $[M+Na]^+$ 248.0357	86-87	45	Yellowish crystals
5e	$C_{10}H_{11}NO_3S$	473.0811 $[2M+Na]^+$ 473.0817	63-64	19	Yellowish crystals

TABLE 4. NMR Spectra of 3-(Aryloxy)thietanes **5a–e**

Compound	^{1}H NMR spectrum, δ, ppm (*J*, Hz)	^{13}C NMR spectrum, δ, ppm
5a	3.33-3.43 (2H, m, CH_2); 3.60-3.68 (2H, m, CH_2); 5.31-5.42 (1H, m, CHO); 6.93 (1H, d, *J* = 8.3, H-6); 7.06 (1H, t, *J* = 7.7, H-4); 7.45-7.53 (1H, m, H-5); 7.78-7.83 (1H, m, H-3)	35.2; 72.4; 115.3; 121.3; 125.7; 134.0; 140.1; 149.4
5b	3.38-3.45 (2H, m, CH_2); 3.53-3.60 (2H, m, CH_2); 5.30-5.39 (1H, m, CHO); 7.13 (1H, ddd, *J* = 0.8, *J* = 2.5, *J* = 8.3, H-6); 7.42 (1H, t, *J* = 8.3, H-5); 7.60 (1H, t, *J* = 2.5, H-2); 7.81 (1H, ddd, *J* = 0.8, *J* = 2.5, *J* = 8.3, H-4)	35.0; 71.6; 109.3; 116.3; 121.7; 130.2; 149.0; 156.6
5c	3.36-3.45 (2H, m, CH_2); 3.52-3.62 (2H, m, CH_2); 5.31-5.42 (1H, m, CHO); 6.81-6.89 (2H, m, H-2,6); 8.10-8.18 (2H, m, H-3,5)	34.9; 71.5; 114.7; 125.9; 141.7; 161.0
5d	2.38 (3H, s, CH_3); 3.35-3.41 (2H, m, CH_2); 3.60-3.67 (2H, m, CH_2); 5.30-5.39 (1H, m, CHO); 6.70 (1H, s, H-6); 6.84 (1H, d, *J* = 8.0, H-4); 7.74 (1H, d, *J* = 8.0, H-3)	21.8; 35.3; 72.4; 115.9; 122.0; 125.9; 137.6; 145.7; 149.7
5e	2.59 (3H, s, CH_3); 3.38-3.49 (2H, m, CH_2); 3.53-3.60 (2H, m, CH_2); 5.30-5.40 (1H, m, CHO); 6.66-6.72 (2H, m, H-2,6); 8.01-8.06 (1H, m, H-5)	21.6; 35.0; 71.4; 112.5; 118.3; 127.6; 137.2; 142.5; 159.5

ionization, 70 eV) were obtained on an MKh-1321 instrument. High-resolution mass spectra (ESI or FAB) were obtained on an IonSpec Fourier transform mass spectrometer. All melting points are uncorrected. Reactions were monitored by thin-layer chromatography carried out on Macherey-Nagel ready-to-use plates AluGram Alox N/UV_{254} using UV visualization or basic $KMnO_4$ solution as a developing agent. Silica gel SiliCycle (60 mesh) was used for flash chromatography. All reagents and solvents were commercially available and were used as supplied without additional purification.

The starting *N*-arylcyanamides **1a–k** were synthesized according to literature methods (**1a** [11], **1b** [26], **1c**,**f**,**g** [17], **1d** [23], **1e** [12], **1h** [27], **1i** [28], **1j**,**k** [20]).

Preparation of *N*-aryl-*N*-(thietan-3-yl)cyanamides 2a–k (General Method). The corresponding *N*-arylcyanamide **1a–k** (20 mmol) was added in one portion to a stirred solution of KOH (1.35 g, 24 mmol) in water (80 ml). Then (chloromethyl)thiirane (2.40 g, 22 mmol) was added in one portion to the resulting solution, and the reaction mixture was stirred at room temperature for 48 h. After that, $CHCl_3$ (100 ml) was added, the organic layer was separated, and the aqueous layer was extracted with $CHCl_3$ (2×30 ml). The combined organic phases were washed with 5% aqueous NaOH (2×50 ml), 100 ml water and 50 ml brine, and dried over $MgSO_4$. The solution was passed through a plug of silica gel (2 cm) eluting with $CHCl_3$ (200 ml). The solvent was removed on a rotary evaporator (bath temperature < 50°C) under reduced pressure, and the residue was recrystallized from $CHCl_3$–hexane (**2c–k**) or dried at 1 mm Hg (**2a**,**b**).

***N*-Phenyl-*N*-(thietan-3-yl)cyanamide (2a)**. Mass spectrum, *m/z* (I_{rel}, %): 190 $[M]^+$ (21), 144 (8), 104 (12), 91 (8), 77 (21), 73 (100), 65 (9), 51 (14), 45 (51), 39 (18).

***N*-(2,6-Dimethylphenyl)-*N*-(thietan-3-yl)cyanamide (2b).** Found, *m/z*: 241.0770 $[M+Na]^+$. $C_{12}H_{14}N_2NaS$. Calculated, *m/z*: 241.0775.

***N*-(4-Methoxyphenyl)-*N*-(thietan-3-yl)cyanamide (2c).** Mass spectrum, *m/z* (I_{rel}, %): 220 $[M]^+$ (14), 174 (5), 147 (7), 73 (100), 45 (43), 39 (9). Found, %: C 60.09; H 5.65; N 12.65. $C_{11}H_{12}N_2OS$. Calculated, %: C 59.98; H 5.49; N 12.72.

***N*-(1-Naphthyl)-*N*-(thietan-3-yl)cyanamide (2d).** Mass spectrum, *m/z* (I_{rel}, %): 240 $[M]^+$ (20), 115 (13), 73 (100), 45 (18). Found, %: C 70.06; H 5.06; N 11.78; $C_{14}H_{12}N_2S$. Calculated, %: C 69.97; H 5.03; N 11.66.

***N*-(2-Chlorophenyl)-*N*-(thietan-3-yl)cyanamide (2e).** Found, *m/z*: 224.0174 $[M]^+$. $C_{10}H_9ClN_2S$. Calculated, *m/z*: 224.0175.

***N*-(3-Fluorophenyl)-*N*-(thietan-3-yl)cyanamide (2h).** Found, *m/z*: 208.0467 $[M]^+$. $C_{10}H_9FN_2S$. Calculated, *m/z*: 208.0470.

***N*-(3-Nitrophenyl)-*N*-(thietan-3-yl)cyanamide (2j).** Found, *m/z*: 258.0308 $[M+Na]^+$. $C_{10}H_9N_3NaO_2S$. Calculated, *m/z*: 258.0313.

Preparation of Sulfones 3a,b (General Method). $Na_2WO_4 \cdot 2H_2O$ (20 mg) was dissolved in a minimal amount of water, and the resulting solution was added to a stirred solution of cyanamide **2a**,**b** (5.3 mmol) in 20 ml glacial acetic acid. Then 30% aqueous H_2O_2 (6.0 g, 53.0 mmol) was added over 5 min, and the reaction mixture was stirred for 15 h and poured into 120 ml cold water. The crude product was filtered off, washed with water, and dried in air. Pure sulfones **3a**,**b** were obtained by reprecipitation from $CHCl_3$ solution with hexane.

***N*-(1,1-Dioxothietan-3-yl)-*N*-phenylcyanamide (3a).** Yield 1.01 g (86%). Colorless crystals, mp 127-128°C. ^{1}H NMR spectrum, δ, ppm: 4.43–4.75 (5H, m, $2CH_2$, CHN); 7.07–7.16 (2H, m, H-2,6); 7.20–7.30 (1H, m, H-4); 7.34–7.50 (2H, m, H-3,5). ^{13}C NMR spectrum, δ, ppm: 39.7; 69.9; 110.8; 117.7; 126.1; 130.7; 138.6. Mass spectrum, *m/z* (I_{rel}, %): 222 $[M]^+$ (25), 144 (100), 118 (15), 117 (16), 104 (77), 91 (21), 77 (62), 65 (31), 51 (35), 41 (93), 39 (84). Found, %: C 53.99; H 4.52; N 12.62. $C_{10}H_{10}N_2O_2S$. Calculated, %: C 54.04; H 4.53; N 12.60.

***N*-(2,6-Dimethylphenyl)-*N*-(1,1-dioxothietan-3-yl)cyanamide (3b).** Yield 0.99 g (75%). Colorless crystals, mp 168–170°C (subl. above 150 °C). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.37 (6H, s, CH_3); 4.31–4.45 (5H, m, $2CH_2$, CHN); 7.13 (2H, d, *J* = 7.5, H-3,5); 7.21 (1H, t, *J* = 7.5, H-4). ^{13}C NMR spectrum, δ, ppm: 18.3; 42.4; 68.9; 112.1; 129.6; 129.7; 135.0; 136.3. Found, *m/z*: 251.0849 $[M+H]^+$. $C_{12}H_{15}N_2O_2S$. Calculated, *m/z*: 251.0854.

Preparation of 3-(aryloxy)thietanes 5a–e. These compounds were prepared in accordance with the general procedure from the corresponding nitrophenols **4a**–**e**. For physical properties of compounds **5a**–**e**, see Tables 3 and 4.

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