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CsF-Celite, an Efficient Solid State Reagent for the Syntheses of Thioesters and Thioethers

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Summary. Coupling reactions of a number of aliphatic, aromatic, and heterocyclic compounds bearing an acidic hydrogen atom attached to sulfur, with alkyl, acyl, benzyl, or benzoyl halides in acetonitrile with cesium fluoride-Celite are described. This procedure is convenient, efficient, and practical for the preparation of thioethers and thioesters.

Keywords. Cesium fluoride-Celite, Thioethers, Thioesters.

Introduction

A variety of organic reactions have recently been reported to be catalysed by cesium fluoride-Celite. The syntheses of carboxylic esters [1], γ-lactones [2], N-alkylation of anilines, carboxamides, and nitrogen heterocyclic compounds [3], and ring opening of epoxides [4] are among the reactions which are facilitated.

The importance of the fluoride ion as a catalyst for the promotion of various types of base-catalyzed reactions in organic synthesis has been previously recognized [5–8]. In particular, the work of *Clark* and *Miller* [9–12] revealed that the fluoride ion effects the coupling reaction because of its high capability of hydrogen bond formation. As reagents, generating fluoride ions, potassium [6], cesium [1], and tetraalkylammonium fluorides were used so far. However, it is not easy to handle these hygroscopic reagents and the reproducibility of these reactions is invariably poor. Recently, poorly hygroscopic reagents generating fluoride ions were designed allowing cesium fluoride to be absorbed on Celite [1]. The effect of

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cesium fluoride-Celite might be two-fold [13]: (a) activation of the hydroxyl group by the fluoride ion whose ionic character is large owing to the low charge/surface area ratio of the cesium cation [14] and (b) activation of the alkyl or acyl halide groups by the *Lewis* acid type effect.

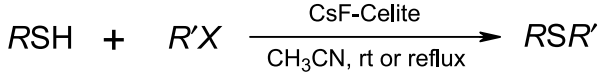
Thioethers are, in general, formed by reaction of thiols or thiophenols under basic conditions with alkyl or benzyl halides. Thioesters are formed and cleaved in the same way as oxygen esters, however, they are more reactive against nucleophilic substitution [15] and used as ''activated esters''. The conversion of thiols to thioethers is usually achieved by reaction of thiolates with organic halides [16]. The yields and reaction conditions depend on the solvent, the basic catalyst, and the acidity of the thiol. However, these reactions require often very long refluxing times and suffer from low yields [16].

Several other methods have been employed to prepare thioethers and thioesters, which include palladium(0)-mediated alkylation [17], phase-transfer catalysis [18], catalysis *via* platinum(II) complexes of bis(diphenylphosphino)methane [19], *via* bis(triphenylstannyl)-tellurides [20], palladium-catalyzed reactions of stannyl sulfides with aryl bromides [21], ligand-transfer reactions of aryl thiocyanates [22, 23], fluorodestannylation of organotin sulfides [24], montmorillonite clay-catalyzed acylation of thiols, *etc.* [25], or benzyl-type protection of thiols using trifluoroacetic acid [26]. Recently, *Yin* and *Pidgeon* [27] reported a high yield method for the preparation of unsymmetrical sulfides by using very strong basic *n*-butyllithium.

Results and Discussions

In extension of our work on the reactivity of CsF-Celite [2, 3, 28], we wish to report the utility of this reagent for the synthesis of thioethers and thioesters in good yields (Scheme 1). Tables 1 and 2 list a series of the CsF-Celite-assisted couplings of aliphatic and aromatic thiols into thioethers and thioesters using a variety of alkyl, acyl, benzyl, and benzoyl halides, catalyzed by this inexpensive, non-corrosive solid base, allowing simple work up of the reaction mixtures.

In general, to a stirred solution of thiol (1.0 mol) and CsF-Celite (1.5 mol) in acetonitrile, alkyl, acyl, benzyl, or benzoyl halides (2.0 mol) were added. Then, the mixture was stirred at room temperature or reflux up to completion of the reaction (TLC analysis). The reaction mixture was filtered and the solvent evaporated. The product was purified, whenever necessary, by column chromatography on silicagel using appropriate solvent systems like dichloromethane and petroleum ether, *etc.* as eluents, to afford pure thioethers or thioesters (Scheme 1).



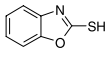
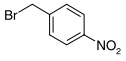
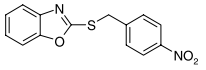
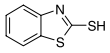
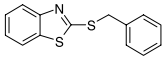
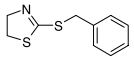
R = alkyl or aryl

X = Cl, Br, or I

R′ = alkyl, acyl, benzyl, or benzoyl

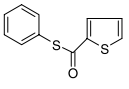
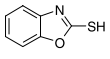
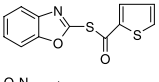
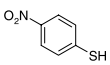
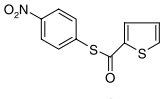
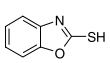
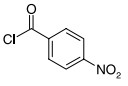
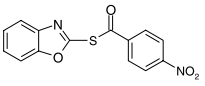
Scheme 1

Table 1. Synthesis of thioethers using CsF-Celite

No.	Substrate	Reagent	Product	Comp.	Yield/%
1	CH_3CH_2SH	$CH_3O_2CCH_2CH_2Br$	$CH_3O_2CCH_2CH_2SCH_2CH_3$	**1**	60[b]
2	$CH_3(CH_2)_4SH$	$C_6H_5CH_2Cl$	$CH_3(CH_2)_4SCH_2C_6H_5$	**2**	81[b]
3	$CH_3(CH_2)_3SH$	$C_6H_5CH_2Cl$	$CH_3(CH_2)_3SCH_2C_6H_5$	**3**	88[b]
4	$C_6H_5CH_2SH$	$CH_2{=}CHCH_2Br$	$C_6H_5CH_2SCH_2CH{=}CH_2$	**4**	66[b]
5	C_6H_5SH	$C_6H_5CH_2Br$	$C_6H_5SCH_2C_6H_5$	**5**	85[b]
6	C_6H_5SH	CH_3CHICH_3	$C_6H_5SCH(CH_3)_2$	**6**	78[b]
7	4-$CH_3OC_6H_4SH$	$C_6H_5CH_2Br$	4-$CH_3OC_6H_4SCH_2C_6H_5$	**7**	81[b]
8	$CH_3C_6H_4SH$	$C_6H_5CH_2Br$	$CH_3C_6H_4SCH_2C_6H_5$	**8**	92[b]
9	4-$NO_2C_6H_4SH$	CH_3CHICH_3	4-$NO_2C_6H_4SCH(CH_3)_2$	**9**	61[b]
10	4-$NO_2C_6H_4SH$	$CH_2{=}CHCH_2Br$	4-$NO_2C_6H_4SCH_2CH{=}CH_2$	**10**	75[b]
11	4-$NO_2C_6H_4SH$	CH_3CH_2I	4-$NO_2C_6H_4SCH_2CH_3$	**11**	60[b]
12	4-$NO_2C_6H_4SH$	$C_6H_5CH_2Cl$	4-$NO_2C_6H_4SCH_2C_6H_5$	**12**	77[b]
13	4-$CH_3OC_6H_4SH$	4-$CH_3OC_6H_4CH_2Cl$	4-$CH_3OC_6H_4SCH_2C_6H_4OCH_3$-4	**13**	86[b]
14				**14**	90[b]
15				**15**	74[a]
16				**16**	81[a]

[a] rt for 1–8 h; [b] reflux at 82°C for 2–48 h

Table 2. Syntheses of thioesters using CsF-Celite

No.	Substrate	Reagent	Product	Comp.	Yield/%
1	C_6H_5SH	C_6H_5COCl	$C_6H_5SOCC_6H_5$	**17**	88[a]
2	$CH_3C_6H_4SH$	C_6H_5COBr	$CH_3C_6H_4SOCC_6H_5$	**18**	75[b]
3	C_6H_5SH	CH_3COCl	$C_6H_5SOCCH_3$	**19**	85[a]
4	4-$CH_3OC_6H_4SH$	$CH_3O_2CCH_2CH_2COCl$	4-$CH_3OC_6H_4SOCCH_2CH_2CO_2CH_3$	**20**	89[b]
5	4-$CH_3OC_6H_4SH$	CH_3COCl	4-$CH_3OC_6H_4SCO_2CH_3$	**21**	88[b]
6	4-$CH_3OC_6H_4SH$	4-$CH_3OC_6H_4COCl$	4-$CH_3OC_6H_4SOCC_6H_4OCH_3$-4	**22**	81[b]
7	4-$CH_3OC_6H_4SH$	C_6H_5COCl	4-$CH_3OC_6H_4SOCC_6H_5$	**23**	71[b]
8	4-$NO_2C_6H_4SH$	C_6H_5COCl	4-$NO_2C_6H_4SOCC_6H_5$	**24**	49[b]
9				**25**	83[b]
10				**26**	79[b]
11				**27**	75[b]
12				**28**	80[b]

[a] rt for 1–8 h; [b] reflux at 82°C for 2–4 h

When thiophenol and benzyl bromide were allowed to react in *DMF* or acetonitrile, *S*-benzylthiophenol could be collected in excellent yield (92% or 85%) after reflux for 28 h. Most of the reactions were carried out in acetonitrile, but sometimes *DMF* was used as a solvent (Table 1, entries 5 and 9). These experiments indicate that the cesium fluoride-Celite-catalyzed reaction is not very much dependent on the solvent used. Application of this method on some heterocyclic compounds with mercapto groups, *e.g.* 2-mercaptobenzoxazole or 2-mercapto-2-thiazoline, supplied the corresponding thioethers in very good yields proving that the methodology is equally applicable on aromatic as well as heterocyclic compounds (Table 1, entries 14–16). We also extended our present discovery to the formation of thioesters by using CsF-Celite to catalyze the reaction between mercaptanes, thiophenols, and acyl groups in very good yield (Table 2).

In conclusion, the CsF-Celite-assisted reactions provide an easy access to thioethers as well as thioesters in good yields. In most cases the products were obtained just by filtration and evaporation of the filtrates, while in some cases (**7**, **24**–**26**) they were isolated by column chromatography. In short, this approach is an efficient, convenient, inexpensive, non-corrosive, and practical method for preparing thioethers and thioesters. As this methodology has several advantages, it is a valuable addition to existing methods for preparing thioethers and thioesters. The physical properties and NMR spectra of the prepared compounds agreed with those reported in literature [29–44], and were furthermore confirmed by comparing the data with those of authentic samples. The hitherto unknown compounds were characterized through their sharp melting points, spectroscopic techniques, and their elemental analyses.

Experimental

Melting points were determined with a Büchi SMP-20 apparatus. IR spectra (KBr discs) were recorded on a Bruker FT-IR IFS 48 spectrometer and EI mass spectral data on a Varian MAT 711 (70 eV) spectrometer (data are tabulated as m/z). ^{1}H and ^{13}C NMR spectra were performed in $CDCl_3$ containing *ca.* 1% *TMS* as internal standard on a Bruker AC 250 (250 MHz and 62.9 MHz) spectrometer. Chemical shifts are reported in δ (ppm) and coupling constants are given in Hz. The progress of all reactions was monitored by TLC on 2.0×5.0 cm Al sheets, precoated with silicagel $60F_{254}$ to a thickness of 0.25 mm (Merck, Darmstadt, Germany). The chromatograms were visualized under ultraviolet light (254–366 nm). Elemental analyses were found to agree favourably with the calculated values (C, H, S).

Ethanethiol, thiophenol, 1-pentanethiol, 4-methoxythiophenol, 4-nitrothiophenol, 2-mercaptobenzoxazole, 2-mercaptobenzothiazole, 2-mercapto-2-thiazoline, benzyl bromide, 4-nitrobenzyl bromide, allyl bromide, acetyl chloride, benzoyl chloride, 4-methoxybenzoyl chloride, CsF, Celite 521, and other chemicals are commercially available (Fluka, Aldrich, Munich, Germany). Anhydrous acetonitrile was purchased from Merck, Darmstadt, Germany, and used without purification. The CsF-Celite was prepared by stirring an aqueous solution of CsF with Celite 521 at room temperature for 20 min [1].

Typical Procedure for Syntheses of Thioethers and Thioesters

To a stirred solution of 1.0 mol thiol and 1.5 mol CsF-Celite in 20 cm^3 of acetonitrile, 2.0 mol alkyl, acyl, benzyl, benzoyl halides, *etc.* were added. Then the mixtures were stirred at room temperature or reflux up to completion of the reactions, indicated by monitoring. The reaction mixtures were filtered, the solvents evaporated, and the residues dissolved in ethyl acetate. Precipitates were filtered off, washed with 20 cm^3 of ethyl acetate and the filtrates evaporated under reduced pressure. The products

were purified whenever necessary by column chromatography on silicagel using various solvent systems like CH_2Cl_2 and petroleum ether, *etc.* as eluents, to afford pure thioethers or thioesters.

Benzyl phenyl sulfide (**5**)
Solid; mp 42–43°C (Ref. [31] 43–44°C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 4.3$ (s, 2H), 7.45–7.25 (m, 10H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 39.25$, 126.45, 127.12, 128.44, 128.89, 129.52, 136.40, 137.40 ppm; EIMS: $m/z = 200$ (M^+).

2-Propyl 4-nitrophenyl sulfide (**9**)
Solid; mp 46–47°C (Ref. [34] 46–47°C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.40$ (d, $J = 6.65$ Hz, 6H), 3.60 (m, 1H), 7.45 (d, $J = 8.92$ Hz, 2H), 8.23 (d, $J = 8.92$ Hz, 2H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 22.15$, 36.64, 124.38, 127.62, 145.13, 147.55 ppm; EIMS: $m/z = 197$ (M^+).

Allyl 4-nitrophenyl sulfide (**10**)
Solid; mp 38–39°C (Ref. [35a] 38–39°C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 3.57$ (m, 2H), 5.14–5.55 (m, 2H), 5.94 (m, 1H), 7.54 (d, $J = 9.10$ Hz, 2H), 8.08 (d, $J = 9.12$ Hz, 2H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 53.12$, 119.01, 123.54, 125.92, 131.71, 145.00, 146.58 ppm; EIMS: $m/z = 195$ (M^+).

Ethyl 4-nitrophenyl sulfide (**11**)
Solid; mp 40–42°C (Ref. [35b] 42–43°C); ^{1}H NMR ($CDCl_3$): $\delta = 1.40$ (t, $J = 7.53$ Hz, 3H), 3.00 (q, $J = 7.36$ Hz, 2H), 7.31 (d, $J = 8.93$ Hz, 2H), 8.21 (d, $J = 8.80$ Hz, 2H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 13.73$, 25.15, 123.77, 126.29, 144.63, 147.81 ppm; EIMS: $m/z = 183$ (M^+).

Benzyl 4-nitrophenyl sulfide (**12**)
Solid; mp 128–129°C (Ref. [33] 128–129°C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 4.22$ (s, 2H), 7.36 (d, $J = 9.10$ Hz, 2H), 7.00–7.51 (m, 5H), 8.43 (d, $J = 8.67$ Hz, 2H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 37.41$, 122.80, 126.35, 127.62, 128.88, 129.27, 135.42, 145.22, 147.35 ppm; EIMS: $m/z = 245$ (M^+).

Phenylthio acetate (**19**, C_8H_8OS)
Liquid [42a, 42b]; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 2.41$ (s, 3H), 7.41–7.43 (m, 5H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 30.11$, 127.87, 129.12, 129.35, 134.37, 193.90 ppm; EIMS: $m/z = 152$ (M^+).

Methyl 4-[(4-methoxyphenyl)thio]-4-oxobutanoate (**20**, $C_{12}H_{14}O_4S$)
Liquid; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 2.51$ (t, $J = 6.00$ Hz, 2H), 3.21 (t, $J = 6.10$ Hz, 2H), 3.56 (s, 3H), 3.62 (s, 3H), 6.98–7.15 (m, 4H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 31.21$, 40.51, 52.22, 55.54, 111.75, 117.69, 136.87, 160.22, 172.50, 196.23 ppm; EIMS: $m/z = 254$ (M^+).

4-Methoxyphenylthio benzoate (**23**)
Solid; mp 96–98°C (Ref. [40] 97.5–99.5°C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 3.80$ (s, 3H), 6.96–7.41 (m, 4H, H-2,3,5,6), 7.45–7.60 (m, 3H, H-3′,4′,5′) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 55.45$, 115.15, 117.53, 127.42, 128.34, 133.35, 135.59, 136.59, 160.84, 190.95 ppm; EIMS: $m/z = 231$ (M^+).

4-Nitrophenylthio benzoate (**24**)
Solid; mp 125–127°C (Ref. [40] 126–127°C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 7.58$–7.66 (m, 3H, H-3′,4′,5′), 7.72 (m, 2H, H-2,6), 8.03 (m, 2H, H-2′,6′), 8.30 (m, 2H, H-3,5) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 123.88$, 127.78, 128.69, 135.10, 135.80, 135.85, 136.25, 148.30, 188.01 ppm; EIMS: $m/z = 259$ (M^+).

1,3-Benzoxazol-2-yl thiophene-2-carbothioate (**26**, $C_{12}H_7NO_2S_2$)
Solid; mp 110–112°C; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 7.01$ (dd, $J = 3.74$, 4.8 Hz, 1H), 7.44 (dd, $J = 1.17$, 4.75 Hz, 1H), 8.22 (dd, $J = 1.29$, 3.59 Hz, 1H), 7.35 (m, 2H), 8.21 (m, 2H) ppm; ^{13}C NMR

(63 MHz, $CDCl_3$): $\delta = 111.51$, 120.51, 124.12, 125.87, 131.25, 132.24, 142.55, 146.90, 152.84, 157.26, 163.50, 176.41 ppm; EIMS: $m/z = 261$ (M^+).

4-Nitrophenyl thiophene-2-carbothioate (**27**, $C_{11}H_7NO_3S_2$)
Liquid; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 6.71$ (dd, $J = 3.67$, 4.8 Hz, 1H), 7.42 (dd, $J = 1.27$, 4.8 Hz, 1H), 8.02 (dd, $J = 1.27$, 3.69 Hz, 1H), 7.55 (m, 2H), 8.15 (m, 2H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 126.87$, 129.12, 131.25, 135.24, 138.90, 146.45, 155.25, 177.50 ppm; EIMS: $m/z = 265$ (M^+).

1,3-Benzoxazol-2-yl 4-nitrobenzenecarbothioate (**28**, $C_{14}H_8N_2O_4S$)
Liquid; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 7.25$ (m, 2H), 7.55 (m, 1H), 7.95–8.12 (m, 3H), 8.32 (m, 2H) ppm; ^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 110.12$, 120.11, 125.02, 125.22, 125.57, 130.25, 143.54, 144.60, 148.76, 153.30, 163.44, 188.22 ppm; EIMS: $m/z = 300$ (M^+).

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