Efficient one-pot synthesis of (anthracen-9-yl) methyl sulfane derivatives

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Reshma Gopalakrishnan1, Jomon P. Jacob1, Ranjith Chirakandathil1, Perupparampil A. Unnikrishnan1 and Sreedharan Prathapan1*

Abstract: A series of unsymmetrical (anthracen-9-yl)methyl sulfanes have been synthesized using a one-pot reaction from (anthracen-9-yl)methyl alcohol, thiourea, and the corresponding alkyl halide and also by a base-promoted one-pot reductive coupling of tosylhydrazones with thiols.

Subjects: Applied & Industrial Chemistry; Chemistry; Organic Chemistry

Keywords: (anthracen-9-yl)methyl sulfanes; thiourea; tosylhydrazones; reductive coupling

1. Introduction

Organosulfur compounds are important intermediates for specialized organic synthesis (Bauld, Aplin, Yueh, & Loinaz, 1997; Chatgilialoglu, Bertrand, Ferreri, & Alfassi, 1999; Wrzyszczynski, Scigalski, & Paczkowski, 2000). These compounds are well known for their radical chemistry under thermal and photochemical conditions (Andrzejewska, Hug, Andrzejewski, & Marciniak, 1999; Bobrowski, Marciniak, & Hug, 1996; Kang & Friend, 2004; Kretzschmar, Friend, & Sigman, 2002; Marciniak, Andrzejewska, & Hugc, 1998; Steffen et al., 1991; Wiegand & Friend, 1992; Wiegand, Napier, Friend, & Uvdal, 1996; Wrzyszczynski, Scigalski, et al., 2000). Sulfur-centered radicals and radical ions play unique roles in diverse areas of chemistry. Thanks to their low ionization potentials, organic sulfides undergo fast one-electron oxidation reactions to give the corresponding radical cations. It is reported that organic sulfides are ideal precursors for sulfur-centered radical cations that can be used for probing mechanisms of electron transfer quenching of excited states as well as for monitoring the fate of the sulfur radicals (Bauld et al., 1997; Bisby, Cundall, Redpath, & Adams, 1976; Chatgilialoglu & Asmus, 1990a; Chatgilialoglu et al., 1999; Kosower, Kosower, & Pryor, 1976; Lalitha & Mittal, 1971; Thompson, Carroll, Watson, O'Donnell, & McGlynn, 1966; Wolff, Aldrich, Penner, &
Hunt, 1975; Wrzyszczynski, Filipiak, Hug, Marciniak, & Paczkowski, 2000). Photoexcited organic sulfides produce different responses which can lead to bond scissions or ionization channels (Anastasi, Broomfield, Nielsen, & Pagsberg, 1991; Callear & Dickson, 1970; Ohbayashi, Akimoto, & Tanaka, 1977). These sulfur radical cations are important intermediates in a variety of chemical processes (Baciocchi, Lanzalunga, & Pirozzi, 1997; Chatgilialoglu & Asmus, 1990b). Aliphatic sulfide radical cation \(1\) and radical cation complex \(2\) can form sulfoxide in oxygen-containing aqueous solution (Miller, Williams, & Schöneich, 1996; Scheme 1).

Conventional method used for the synthesis of organic sulfides involves the reaction of a thiol (or disulfide) with a halide in the presence of a strong base (Smith & March, 2001). This method is robust but requires handling of malodorous thiols and reactive halides that are difficult to handle. Moreover, not many thiols are commercially available. Reported method for the synthesis of \((\text{anthracen}-9\text{-yl})\) methyl methyl sulfane involves reaction of methyl iodide with sodium salt of \((\text{anthracen}-9\text{-yl})\) methyliodide. The reaction condition is somewhat tedious because of the use of malodorous thiol, reactive sodium metal, and inert atmosphere in the procedure (Stein & Gellman, 1992). Recently, a one-pot synthesis of symmetrical and unsymmetrical benzyl sulfanes from benzyl halides using thiourea has been developed. The synthetic scope of this reaction is limited by its use of highly reactive benzyl halides (Eccles, Ecloate, Lawrence, & Maguire, 2010). There are many reports on the formation of aryl and alkyl sulfanes (Dunn et al., 2012; Gundermann & Röker, 1976) using cross-coupling reactions of aryl or alkyl halides with various nucleophilic compounds, but these reactions require more forcing conditions such as use of transition metal catalyst (Ham, Yang, & Kang, 2004; Huang et al., 2013; Jammi, Barua, Rout, Saha, & Punniyamurthy, 2008; Prasad & Sekar, 2011; Takagi, 1986) or photochemical activation (Argüello, Schmidt, & Peñéñory, 2003). High cost and toxicity of some transition metal catalysts and ligands restrict their applications in large-scale processes. Recent modifications such as metal free reactions have shown the same levels of efficiency as metal-catalyzed reactions (Borisova, Reshetova, & Ustynyuk, 2007). Ding et al. reported the synthesis of substituted benzyl phenyl sulfides via the metal-free reductive coupling of tosyl hydrazones with substituted benzene thiols (Ding, Cao, Yuan, Liu, & Peng, 2011). Since none of the reported methods satisfied our requirements to generate \((\text{anthracen})\)methyl sulfanes in sufficient quantities, we explored the possibility of adopting a new strategy.

Herein, we describe a robust and convenient procedure for the synthesis of \((\text{anthracen}-9\text{-yl})\) methyl alkyl sulfanes or substituted \((\text{anthracen}-9\text{-yl})\) methyl alkyl sulfane \(16–24\) by a one-pot reaction using readily accessible \((\text{anthracen}-9\text{-yl})\) methyl alcohol or substituted \((\text{anthracen}-9\text{-yl})\) methyl alcohol \(3\), thiourea \(4\), and the corresponding alkyl halide \(7–15\) adapting a reported procedure for the synthesis of thiols (Vetter, 1998). Conspicuous advantage of this procedure is avoidance of direct use of thiols. \((\text{Anthracen}-9\text{-yl})\) phenyl sulfane \(31\) and \((\text{anthracen}-9\text{-yl})\) methyl \(p\)-tolyi sulfane \(32\) were prepared by the reductive coupling between \(9\)-anthraldehyde tosyl hydrazones \(27\) and benzene thiol \(29\) or \(p\)-tolyi thiol \(30\) following the protocol developed by Ding et al. (2011). Though, we synthesized a handful of naphthylmethyl and benzyl sulfanes (Ding et al., 2011; Eccles et al., 2010; Santoni et al., 2010) using these procedures, in this article, we restrict our discussion to the synthesis of a few \((\text{anthracen})\)methyl sulfane derivatives.

2. Results and discussions
Anthracenemethyl thiol is conveniently prepared by the reaction between \((\text{anthracen}-9\text{-yl})\) methyl alcohol and thiourea (Miller, Amidon, & Tawney, 1955). We reasoned that the thiolate ion generated as an intermediate can be intercepted by a suitable alkyl halide to give the corresponding sulfane in a one-pot reaction. Applying this strategy, we synthesized \((\text{anthracen}-9\text{-yl})\) methyl alkyl sulfanes \(16–24\) by the one-pot reaction of \((\text{anthracen}-9\text{-yl})\) methyl alcohol \(3\), thiourea \(4\), and the
corresponding alkyl halide. Reaction of 3 with 4 under acidic conditions affords the isothiouronium salt 5 which upon treatment with a strong base generates the thiolate 6. This intermediate is further reacted in situ with a series of alkyl halides 7–15 to generate the (anthracen-9-yl)methyl alkyl sulfanes 16–24 (Scheme 2). Here the isolation of intermediate thiol or isothiouronium salt is not required, thereby significantly simplifying this synthetic method. The reaction took place in good yields (Table 1). Steps involved in the synthesis of (anthracen-9-yl)methyl alkyl sulfanes 16–24 are presented in Scheme 2. A notable feature of this strategy is the flexibility available here: we could select the alcohol and halide substrates based on their availability and ease of handling. This procedure is compatible with several organic halides and substituted anthracenes.

As can be inferred from the mechanism presented in Scheme 2, phenols cannot react with thiourea to generate isothiouronim salts analogous to 5. Additionally, the reaction fails with halobenzenes. Thus, a major limitation of the procedure shown in Scheme 2 is that this method is not suitable for the preparation of aryl sulfanes such as 31 and 32. Hence, we adopted the metal-free reductive coupling reaction reported by Ding et al. (2011) for the generation of aryl sulfanes. Reaction of benzene thiol (29) and p-tolyl thiol (30) with tosylhydrazone 27 derived from 9-anthraldehyde (25) proceeded smoothly to give (anthracen-9-yl)methyl phenyl sulfane (31) and (anthracen-9-yl)methyl p-tolyl sulfane (32) in high yields (Scheme 3). The proposed reductive coupling mechanism involves the initial generation of an intermediate carbene 28 via the base-promoted thermal decomposition of the tosylhydrazone (Barluenga, Tomás-Gamasa, Aznar, & Valdés, 2010; Liu, Yan, & Lu, 2013) 27. Insertion of incipient carbene 28 into the S–H bond of the benzene thiol (29) and p-tolyl thiol (30), giving rise to (anthracen-9-yl)methyl phenyl sulfane (31) and (anthracen-9-yl)methyl p-tolyl sulfane (32). Structure of 16–24, 31, and 32 was established on the basis of analytical and spectral data. 1H and 13C NMR spectra of these compounds were in agreement with the expected structure and they exhibited acceptable elemental analysis and mass spectral data.

3. Experimental section

3.1. General techniques

All reactions were carried out using oven dried glasswares. All experiments were done with distilled and dried solvents by using standard protocols. All starting materials were purchased from either Sigma-Aldrich or Spectrochem Chemicals and were used without further purification. Progress of the reaction and chromatographic separations were monitored by dried and activated silica gel TLC plates (aluminum sheets coated with silica gel, E. Merck). Visualization of TLC plates was acquired by exposure to iodine vapors or UV lamp. Separation and purification of compounds were done by column chromatography using silica gel (Spectrochem Chemicals, 60-120 mesh). The products were further purified by recrystallization from suitable solvent systems. Solvent eluted from the column chromatography was concentrated using Heidolph rotary evaporator. Melting points were determined on a Neolab melting point apparatus. Infrared spectra were recorded using Jasco 4100 and ABB Bomem (MB Series) FT-IR spectrometers. The 1H and 13C NMR spectra were recorded at 400 MHz on a Bruker Advance III FT-NMR spectrometer with tetramethylsilane (TMS) as internal standard. Chemical shifts (δ) are reported in parts per million (ppm) downfield of TMS. Elemental analysis was performed using Elementar Systeme (Vario EL III). Molecular mass was determined by electron impact method using GC-MS (Agilent GC-7890A, Mass-5975C) and fast atom bombardment using JMS 600 JEOL mass spectrometer. All the required starting materials are commercially available and were used as received.
**Table 1.** List of (anthracen-9-yl)methyl sulfanes synthesized using a one-pot reaction from (anthracen-9-yl)methyl alcohol, thiourea, and alkyl halide*

<table>
<thead>
<tr>
<th>Entry</th>
<th>Alkyl Halide $^\cdot$-$X$</th>
<th>Thiolate intermediate, 6</th>
<th>Product</th>
<th>Yield (%)</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CH$_3$I</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td>74</td>
<td>30 min</td>
</tr>
<tr>
<td>2</td>
<td>CH$_3$CH$_2$I</td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
<td>78</td>
<td>30 min</td>
</tr>
<tr>
<td>3</td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
<td><img src="image7.png" alt="Image" /></td>
<td>78</td>
<td>1 h</td>
</tr>
<tr>
<td>4</td>
<td><img src="image8.png" alt="Image" /></td>
<td><img src="image9.png" alt="Image" /></td>
<td><img src="image10.png" alt="Image" /></td>
<td>72</td>
<td>1 h</td>
</tr>
<tr>
<td>5</td>
<td><img src="image11.png" alt="Image" /></td>
<td><img src="image12.png" alt="Image" /></td>
<td><img src="image13.png" alt="Image" /></td>
<td>59</td>
<td>30 min</td>
</tr>
<tr>
<td>6</td>
<td><img src="image14.png" alt="Image" /></td>
<td><img src="image15.png" alt="Image" /></td>
<td><img src="image16.png" alt="Image" /></td>
<td>74</td>
<td>1 h</td>
</tr>
<tr>
<td>7</td>
<td><img src="image17.png" alt="Image" /></td>
<td><img src="image18.png" alt="Image" /></td>
<td><img src="image19.png" alt="Image" /></td>
<td>61</td>
<td>30 min</td>
</tr>
<tr>
<td>8</td>
<td><img src="image20.png" alt="Image" /></td>
<td><img src="image21.png" alt="Image" /></td>
<td><img src="image22.png" alt="Image" /></td>
<td>52</td>
<td>1 h</td>
</tr>
<tr>
<td>9</td>
<td>BrCH$_2$C≡CH</td>
<td><img src="image23.png" alt="Image" /></td>
<td><img src="image24.png" alt="Image" /></td>
<td>85</td>
<td>30 min</td>
</tr>
</tbody>
</table>

*Note: Only a few typical examples are listed herein.
3.2. General procedure for the synthesis of (anthracen-9-yl)methyl alkyl sulfanes 16–24

To a solution of (anthracen-9-yl)methyl alcohol (2 g, 9.6 mmol, 1 equiv.) or (10-ethylanthracen-9-yl)methyl alcohol (2 g, 8.5 mmol, 1 equiv.) in 15 mL of methanol, thiourea (4 equiv.) and 10 mL of 5 N HCl were added and the mixture was stirred at RT for 10 h. To this mixture, NaOH pellets (3 equiv.) were added and the mixture was stirred vigorously for 30 min. On addition of NaOH, yellow color of the solution turned into gray. At this point, 1.1 equiv. of alkyl halide was added and the mixture was stirred for one hour. After the completion of the reaction, the reaction mixture was poured into water and extracted with dichloromethane. Organic layer was separated, washed with water, and dried over anhydrous Na$_2$SO$_4$. Solvent was removed under reduced pressure and the product obtained was passed through a silica gel column to purify (anthracen-9-yl)methyl alkyl sulfanes 16–23 and (10-ethylanthracen-9-yl)methyl prop-2-ynyl sulfane (24). The solid obtained upon removal of solvent was purified by recrystallization from a mixture (1:3) of hexane and dichloromethane.

3.3. General procedure for the synthesis of (anthracen-9-yl)methyl aryl sulfanes, 31 & 32

To a solution of 9-anthraldehyde (25) (1.0 g, 4.8 mmol) in 5 mL of dioxane, tosyl hydrazide (26) (1.8 g, 9.6 mmol) was added and the mixture was stirred for 1 h. To this mixture, benzene thiol (29) (0.98 mL, 9.6 mmol) or $p$-tolyl thiol (30) (1.19 g, 9.6 mmol) and K$_2$CO$_3$ (2 g, 14.4 mmol) were added and the mixture was stirred at 100°C for 2 h. After completion of the reaction, as indicated by TLC, the reaction mixture was cooled to room temperature. Ethyl acetate (25 mL) was added and the organic phase was washed with brine, dried over anhydrous Na$_2$SO$_4$ and the solvent was removed under reduced pressure. The product obtained was purified by column chromatography on silica gel.

4. Characterization data

(anthracen-9-yl)methyl methyl sulfane (16) (Stein & Gellman, 1992): Yellow solid, Yield: 1.70 g, 74%; mp: 72–74°C; IR $\nu_{\text{max}}$ (KBr): 3055, 2958, 2846, 1619, 1598, 1392, 719 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.31–7.38 (m, 9H), 4.65 (s, 2H), 2.06 (s, 3H); $^{13}$C NMR (CDCl$_3$): $\delta$ 130.6, 128.9, 128.3, 128.2, 126.2, 125.1, 124.0, 123.2, 37.5, 17.2; MS: m/z 238 ($M^+$), 191; Anal. Calcd for C$_{16}$H$_{14}$S: C, 80.63; H, 5.92; S, 13.45; Found: C, 80.58; H, 5.85; S, 13.39.

(anthracen-9-yl)methyl ethyl sulfane (17) (Stein & Gellman, 1992): Yellow solid, Yield: 1.88 g, 78%; mp: 68–70°C; IR $\nu_{\text{max}}$ (KBr): 3055, 2958, 2846, 1619, 1598, 1392, 719 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.31–7.38 (m, 9H), 4.65 (s, 2H), 2.06 (s, 3H); $^{13}$C NMR (CDCl$_3$): $\delta$ 130.6, 128.9, 128.3, 128.2, 126.2, 125.1, 124.0, 123.2, 37.5, 17.2; MS: m/z 238 ($M^+$), 191; Anal. Calcd for C$_{17}$H$_{16}$S: C, 80.63; H, 5.92; S, 12.71; Found: C, 80.81; H, 5.32; S, 12.65.

(anthracen-9-yl)methyl propyl sulfane (18): Yellow solid, Yield: 1.99 g, 78%; mp: 92–94°C; IR $\nu_{\text{max}}$ (KBr): 3058, 2961, 2856, 1620, 1596, 1388, 1221, 716 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.41–7.38 (m, 9H), 4.75 (s, 2H), 2.80 (t, J = 7.3 Hz, 2H), 1.82–1.70 (m, 2H), 1.00 (t, J = 7.3 Hz, 3H); $^{13}$C NMR (CDCl$_3$): $\delta$ 131.44, 131.40, 129.01, 127.62, 125.03, 124.89, 67.15, 54.59, 53.70; MS: m/z 266 ($M^+$), 191; Anal. Calcd for C$_{18}$H$_{18}$S: C, 81.15; H, 6.81; S, 12.03; Found: C, 81.19; H, 6.78; S, 12.03.
(anthracen-9-yl)methyl butyl sulfane (19) (Stein & Gellman, 1992): Yellow solid, Yield: 1.94 g, 72%; mp: 52–54°C; IR $\nu_{\text{max}}$ (KBr): 2960, 1466, 1217, 888, 842, 727 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.31–7.37 (m, 9H), 4.68 (s, 2H), 3.19–3.09 (m, 1H), 1.37 (d, 6H, J = 6.8 Hz); $^{13}$C NMR (CDCl$_3$): $\delta$ 130.6, 128.9, 128.3, 128.2, 126.2, 125.1, 124.0, 123.2, 35.7, 26.9, 22.6; MS: m/z 280 (M$^+$), 191; Anal. Calcd for C$_{26}$H$_{20}$S: C, 85.67; H, 5.53; S, 8.80; Found: C, 85.58; H, 5.46; S, 8.76.

(anthracen-9-yl)methyl isopropyl sulfane (20): Yellow solid, Yield: 1.52 g, 59%; mp: 62–64°C; IR $\nu_{\text{max}}$ (KBr): 3053, 2957, 2858, 1620, 1597, 1384, 722 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.31–7.37 (m, 9H), 4.68 (s, 2H), 3.29–3.22 (m, 1H), 4.74 (s, 2H), 2.67 (t, 2H, J = 7.2 Hz), 1.69 (m, 2H), 1.44 (m, 2H), 0.91 (t, 3H, J = 7.3 Hz); MS: m/z 280 (M$^+$), 191; Anal. Calcd for C$_{15}$H$_{10}$S: C, 81.32; H, 7.19; S, 11.43; Found: C, 81.32; H, 7.14; S, 11.32.

(anthracen-9-yl)methyl prop-2-ynyl sulfane (24): Yellow solid, Yield: 1.85 g, 61%; mp: 74–76°C; IR $\nu_{\text{max}}$ (KBr): 3084, 2952, 2857, 1619, 1598, 1399, 723 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.36–7.32 (m, 14H), 4.60 (s, 2H), 3.89 (s, 2H); $^{13}$C NMR (CDCl$_3$): $\delta$ 136.4, 130.5, 129.1, 128.5, 128.1, 127.5, 127.1, 126.8, 126.5, 125.1, 124.1, 123.4, 42.8, 35.9; MS: m/z 314 (M$^+$), 191; Anal. Calcd for C$_{18}$H$_{16}$S: C, 81.07; H, 6.76; S, 10.95; Found: C, 81.07; H, 6.76; S, 11.0.

(anthracen-9-yl)methyl naphthylmethyl sulfane (23) (Stein & Gellman, 1992): Yellow solid, Yield: 1.82 g, 52%; mp: 94–96°C; IR $\nu_{\text{max}}$ (KBr): 3080, 3046, 2931, 2860, 1598, 1380, 716 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.35–7.32 (m, 16H), 4.65 (s, 2H), 4.33 (s, 2H); $^{13}$C NMR (CDCl$_3$): $\delta$ 136.49, 133.55, 131.35, 130.8, 128.9, 128.3, 127.4, 127.3, 126.1, 125.0, 125.1, 124.1, 123.5, 42.8, 35.9; MS: m/z 364 (M$^+$), 191; Anal. Calcd for C$_{24}$H$_{20}$S: C, 85.77; H, 5.77; S, 10.20; Found: C, 85.82; H, 5.68; S, 10.9.

(anthracen-9-yl)methyl phenyl sulfane (31): Yellow solid, Yield: 1.02 g, 69%; mp: 103–105°C; IR $\nu_{\text{max}}$ (KBr): 3299, 3119, 3090, 2953, 2860, 2127, 1597, 1382, 1272, 1024, 890, 728, 686, 632 cm$^{-1}$; $^1$H NMR (CDCl$_3$): $\delta$ 8.45–7.49 (m, 9H), 4.92 (s, 2H), 3.65 (q, J = 7.6 Hz & 15.2 Hz, 2H), 3.37 (d, J = 2.8 Hz, 2H), 2.44 (t, J = 2.4 Hz & 4.8 Hz, 1H), 1.46 (t, J = 7.6 Hz & 15.2 Hz, 3H); $^{13}$C NMR (CDCl$_3$): $\delta$ 137.63, 130.17, 129.08, 126.88, 125.67, 125.14, 125.10, 124.95, 80.56, 71.35, 28.98, 21.39, 20.56, 15.52; MS: m/z 290 (M$^+$), 191; Anal. Calcd for C$_{21}$H$_{18}$S: C, 85.67; H, 5.53; S, 8.80; Found: C, 85.58; H, 5.46; S, 8.76.

8. Conclusion

We have developed an efficient, inexpensive, and operationally simple procedure for the synthesis of (anthracen-9-yl)methyl alkyl sulfanes using commercially available compounds. This method is superior to other reported procedures due to its inherent flexibility, simplicity, and avoidance of less accessible and malodorous thiols.
Supplementary material
Supplementary material for this article can be accessed here http://dx.doi.org/10.1080/23312009.2015.1033820.

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Author details
Reshma Gopalakrishnan1
E-mail: reshmag21@gmail.com
Jomon P. Jacob1
E-mail: jacobbjomon@yahoo.com
Ranjith Chirakandathil1
E-mail: cranjith85@gmail.com
Perupparampil A. Unnikrishnan1
E-mail: paunni@gmail.com
Sreedharan Prathapan1
E-mail: prathapans@gmail.com
1 Department of Applied Chemistry, Cochin University of Science and Technology, Cochin 682 022, Kerala, India.

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