

## Synthesis of 9-Substituted Derivatives of tert-Butyl 6-(9H-Purin-6-Ylthio) Hexylcarbamate

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### Abstract

We report herein the synthesis of series of 9-substituted derivatives of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate by the reaction of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate with different acid chlorides using triethylamine in Dichloromethane solvent.

**Keywords:** 6-mercaptapurine; Tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate; Acylation; Dichloromethane; Boc-deprotection

### Introduction

The purine ring system possesses undisputed biological importance and it is considered to be one of the most important heterocyclic rings in nature [1]. Cells obtain purine nucleotides through two separate metabolic pathways, de novo purine synthesis and salvage of extracellular purine bases and nucleotides [2]. Therefore, de novo purine synthesis and several enzymes involved in the purine metabolism pathway are important targets for antimetabolites [2]. Purine antimetabolites have been used in the development of many potent medicinal agents, which exhibited antineoplastic, antileukemic, antiviral, antibacterial, and antifungal activities [1-4]. The purine nucleoside analogs are also used in the treatment of autoimmune diseases [5]. 6-mercapto purine is used therapeutically as an immunosuppressive agent [6] and inhibits the growth of bacterial and mammalian cells [7]. Other 6-mercapto purine, mercapto pyridine and mercapto pyrimidine derivatives also exhibit antibacterial activity, and have been studied as agents for targeting melanoma [8], reducing cholesterol and as vasodilators [9]. As part of our present research work we are preparing acylated derivatives of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate. According to the Neiman and Bergmann [10] work we confirmed that acylation at the 9- position.

### Materials and Methods

All <sup>1</sup>H NMR spectra were recorded on 400 MHz Varian FT-NMR spectrometers. All chemical shifts are given as  $\delta$  value with reference to Tetra methyl silane (TMS) as an internal standard. Melting points were taken in open capillaries. The IR spectra were recorded on a PerkinElmer 257 spectrometer using KBr discs. Products were purified by flash chromatography on 100-200 mesh silica gel. The chemicals and solvents were purchased from commercial suppliers either from Aldrich, Spectrochem and they were used without purification prior to use.

### Scheme of synthesis

The synthesis of the title compounds (8a-8i) were prepared by the acylation of compound 6 by using different acid chlorides. The main intermediate compound 6 was prepared from 6- mercaptapurine.

### Experimental

#### Synthesis of tert-butyl 6-hydroxyhexylcarbamate (2)

To a stirred solution of compound 1 (5 gm, 42.73 mmol) in THF (50 ml) was added (Boc)<sub>2</sub>O (9.8 mL, 42.73 mmol) at 0°C for 30 min and at rt for over 1 h. The reaction mixture was evaporated in vacuum until

dryness. The obtained crude reaction product 2 (8 gm) was used to next step without any further purification.

#### Synthesis of 6-(tert-butoxycarbonylamino) hexyl methane-sulfonate (3)

To a stirred solution of compound 2 (8 gm, 36.86 mmol) in THF (50 ml) was added DIPEA (20.3 mL, 110.5 mmol), methane sulfonyl chloride (4.5 mL, 55.29 mmol) at 0°C for 30 min and at rt for over 2 h. Reaction was monitored by TLC. When reaction was completed the mixture was diluted with ethyl acetate and washed with H<sub>2</sub>O, brine solution. Organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure. Crude compound was purified by column chromatography to obtained compound 3 (9 g, 71%) as a light yellowish gummy solid. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>):  $\delta$  6.76 (m, 1H), 4.17 (t, J=8.4 Hz, 2H), 3.15 (s, 3H), 2.89 (q, J=8.4 Hz, 2H), 1.66-1.59 (m, 2H), 1.36 (s, 9H), 1.33-1.25 (m, 6H); (LC-MS) m/z: 196.1 [M-100(Boc)]<sup>+</sup>.

#### Synthesis of 9H-purine-6-thiol (5)

To a stirred solution of compound 4 (20 gm, 129.39 mmol) in DMF (200 ml) was added potassium thioacetate (29.5 gm, 258.79 mmol) at room temperature. The reaction mixture was stirred for an additional 6 h at 80°C. Reaction was monitored by TLC. When reaction was completed the mixture was cooled to room temperature and diluted with water (200 mL), ethyl acetate (200 mL) and stirred for 1 h at RT. Reaction mixture was filtered and obtained solid was dried under vacuum to get compound 5 (16 g, 80%) as a brown colour solid. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>):  $\delta$  13.78 (s, 1H), 13.66 (s, 1H), 8.41 (s, 1H), 8.20 (s, 1H); (LC-MS) m/z: 153.06 [M+H]<sup>+</sup>.

#### Synthesis of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate (6)

To a stirred solution of compound 5 (5 gm, 32.89 mmol) in DMF (50 ml) was added potassium carbonate (4.5 gm, 32.89 mmol) and compound 3 (9.7 gm, 32.89 mmol) at room temperature. The reaction mixture was stirred for an additional 16 h at RT. Reaction

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was monitored by TLC. When reaction was completed the mixture was diluted with ethyl acetate and washed with H<sub>2</sub>O, brine solution. Organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure. Crude compound was purified by column chromatography to obtained compound 6 (6.5 g, 56%) as an off-white solid, compound 7 (600 mg) as a light brown solid. Compound 6-<sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ 13.50 (s, 1H), 8.71 (s, 1H), 8.42 (s, 1H), 6.78 (s, 1H), 2.95-2.89 (m, 2H), 1.72-1.68 (m, 2H), 1.38 (s, 9H), 1.36-1.25 (m, 8H); (LC-MS) m/z: 352.2 [M+H]<sup>+</sup>. Compound 7-<sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ 8.70 (s, 1H), 8.47 (s, 1H), 6.77-6.75 (m, 2H), 4.24-4.21 (m, 2H), 3.60 (br s, 1H), 2.90-2.85 (m, 4H), 1.83-1.68 (m, 5H), 1.36 (s, 18H), 1.36-1.24 (m, 12H); (LC-MS) m/z: 551.4 [M+H]<sup>+</sup>.

### General procedure for compound 8a-8i

To a stirred solution of compound 7 (1 mmol) in Dichloromethane (3 ml) was added TEA (3 mmol), Acid chloride (1.2 mmol) at 0°C for 30 min and at rt for over 4h. Reaction was monitored by TLC. When reaction was completed, the reaction mixture was diluted with DCM and washed with H<sub>2</sub>O, brine solution. Organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure. Crude compound was purified by column chromatography to get compound 8a-8i.

**tert-butyl 6-(9-(2-fluorobenzoyl)-9H-purin-6-ylthio) hexylcarbamate 8a:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.62 (s, 1H), 8.56 (s, 1H), 7.78-7.68 (m, 2H), 7.38 (t, J=7.6 Hz, 1H), 7.21 (t, J=9.2 Hz, 1H), 4.51 (br s, 1H), 3.36 (t, J=7.2 Hz, 2H), 3.12-3.10 (m, 2H), 1.82-1.75 (m, 2H), 1.58-1.37 (m, 6H), 1.43 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3377, 2934, 1723, 1692, 1577, 1522, 1381, 1167, 899, 748, 633; *Anal.* Calcd. For C<sub>23</sub>H<sub>28</sub>FN<sub>5</sub>O<sub>3</sub>S: C, 58.33; H, 5.96; F, 4.01; N, 14.79; O, 10.14; S, 6.77. Found: C, 57.87; H, 5.86; N, 14.59. (LC-MS) m/z: 474.11 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(2-methylbenzoyl)-9H-purin-6-ylthio) hexylcarbamate 8b:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.73 (s, 1H), 8.34 (s, 1H), 7.57-7.53 (m, 1H), 7.43-7.31 (m, 3H), 4.51 (br s, 1H), 3.37 (t, J=7.2 Hz, 2H), 3.12-3.09 (m, 2H), 2.46 (s, 3H), 1.83-1.75 (m, 2H), 1.61-1.33 (m, 6H), 1.43 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3384, 2932, 1708, 1693, 1519, 1378, 1246, 1169, 897, 733, 633; *Anal.* Calcd. For C<sub>24</sub>H<sub>31</sub>N<sub>5</sub>O<sub>3</sub>S: C, 61.38; H, 6.65; N, 14.91; O, 10.22; S, 6.83. Found: C, 60.29; H, 6.86; N, 14.89. (LC-MS) m/z: 470.21 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(2-trifluoromethylbenzoyl)-9H-purin-6-ylthio) hexylcarbamate 8c:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.55 (s, 1H), 8.51 (s, 1H), 7.86 (d, J=7.6 Hz, 1H), 7.79-7.70 (m, 2H), 7.52 (d, J=7.6 Hz, 1H), 4.49 (br s, 1H), 3.35 (t, J=7.2 Hz, 2H), 3.11-3.09 (m, 2H), 1.81-1.73 (m, 2H), 1.56-1.36 (m, 6H), 1.43 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3392, 2932, 2860, 1719, 1569, 1368, 1316, 1170, 1138, 900, 771, 634; *Anal.* Calcd. For C<sub>24</sub>H<sub>28</sub>F<sub>3</sub>N<sub>5</sub>O<sub>3</sub>S: C, 55.06; H, 5.39; F, 10.89; N, 13.38; O, 9.17; S, 6.12. Found: C, 55.09; H, 5.26; N, 13.21. (LC-MS) m/z: 524.17 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(2-methoxybenzoyl)-9H-purin-6-ylthio) hexylcarbamate 8d:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.68 (s, 1H), 8.39 (s, 1H), 7.65-7.61 (m, 2H), 7.15 (t, J=7.6 Hz, 1H), 7.01 (d, J=8.8 Hz, 1H), 4.50 (br s, 1H), 3.62 (s, 3H), 3.36 (t, J=7.2 Hz, 2H), 3.12-3.10 (m, 2H), 1.82-1.79 (m, 2H), 1.54-1.33 (m, 6H), 1.43 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3380, 2936, 1742, 1689, 1581, 1520, 1387, 1180, 890, 752, 632; *Anal.* Calcd. For C<sub>24</sub>H<sub>31</sub>N<sub>5</sub>O<sub>4</sub>S: C, 59.36; H, 6.43; N, 14.42; O, 13.18; S, 6.60. Found: C, 59.21; H, 6.40; N, 14.31. (LC-MS) m/z: 486.26 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(4-methoxybenzoyl)-9H-purin-6-ylthio) hexylcarbamate 8e:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.73 (s, 1H), 8.49 (s, 1H), 7.88 (d, J=8.8 Hz, 2H), 7.03 (d, J=8.8 Hz, 2H), 4.52 (br s, 1H), 3.93 (s, 3H), 3.39 (t, J=7.2 Hz, 2H), 3.13-3.11 (m, 2H), 1.84-1.77 (m, 2H), 1.54-1.38 (m, 6H), 1.44 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3382, 2928, 1690, 1557, 1514, 1368, 1264, 1172, 899, 762, 634; *Anal.* Calcd. For C<sub>24</sub>H<sub>31</sub>N<sub>5</sub>O<sub>4</sub>S: C, 59.36;

H, 6.43; N, 14.42; O, 13.18; S, 6.60. Found: C, 59.19; H, 6.42; N, 14.39. (LC-MS) m/z: 486.16 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(3-chlorobenzoyl)-9H-purin-6-ylthio) hexylcarbamate 8f:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.70 (s, 1H), 8.51 (s, 1H), 7.85 (s, 1H), 7.71 (t, J=7.6 Hz, 1H), 7.53-7.49 (m, 2H), 4.51 (br s, 1H), 3.38 (t, J=7.2 Hz, 2H), 3.12-3.10 (m, 2H), 1.82-1.78 (m, 2H), 1.55-1.37 (m, 6H), 1.43 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3381, 2942, 2868, 1714, 1570, 1342, 1310, 1178, 1136, 910, 774, 631; *Anal.* Calcd. For C<sub>23</sub>H<sub>28</sub>ClN<sub>5</sub>O<sub>3</sub>S: C, 56.37; H, 5.76; Cl, 7.24; N, 14.29; O, 9.80; S, 6.54. Found: C, 56.30; H, 5.69; N, 14.18. (LC-MS) m/z: 490.12, 492.14 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(1-naphthoyl)-9H-purin-6-ylthio) hexylcarbamate 8g:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.70 (s, 1H), 8.55 (s, 1H), 8.40 (s, 1H), 8.02-7.91 (m, 4H), 7.72-7.62 (m, 2H), 4.51 (br s, 1H), 3.40 (t, J=7.2 Hz, 2H), 3.13-3.11 (m, 2H), 1.84-1.80 (m, 2H), 1.56-1.38 (m, 6H), 1.44 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3293, 2929, 1689, 1558, 1369, 1283, 1190, 911, 806, 756, 633; *Anal.* Calcd. For C<sub>27</sub>H<sub>31</sub>N<sub>5</sub>O<sub>3</sub>S: C, 64.14; H, 6.18; N, 13.85; O, 9.49; S, 6.34. Found: C, 64.02; H, 6.21; N, 13.59. (LC-MS) m/z: 506.16 [M+H]<sup>+</sup>.

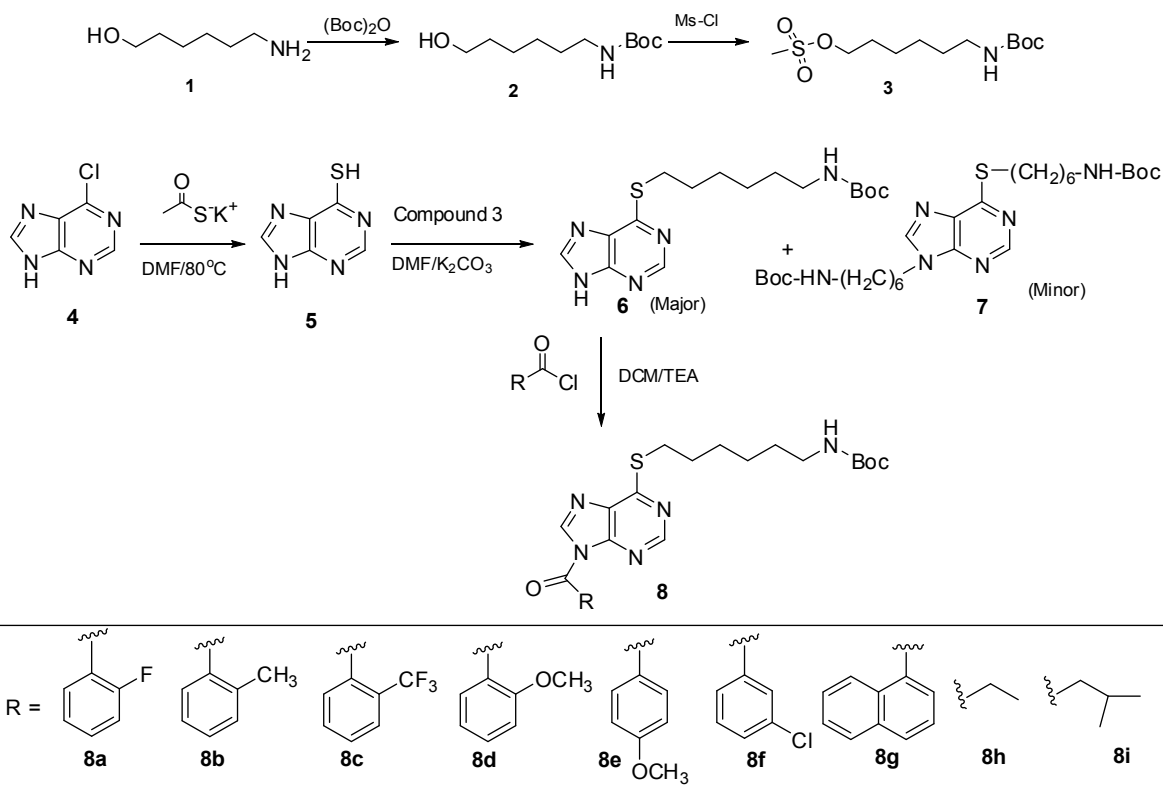
**tert-butyl 6-(9-propionoyl-9H-purin-6-ylthio)hexylcarbamate 8h:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.77 (s, 1H), 8.69 (s, 1H), 4.51 (br s, 1H), 3.47 (q, J=7.2 Hz, 2H), 3.37 (t, J=7.6 Hz, 2H), 3.12-3.10 (m, 2H), 1.83-1.79 (m, 2H), 1.56-1.34 (m, 9H), 1.44 (s, 9H); IR (KBr, cm<sup>-1</sup>): 3371, 2921, 1748, 1675, 1563, 1516, 1366, 1240, 1183, 909, 729, 602; *Anal.* Calcd. For C<sub>19</sub>H<sub>29</sub>N<sub>5</sub>O<sub>3</sub>S: C, 56.00; H, 7.17; N, 17.18; O, 11.78; S, 7.87. Found: C, 56.09; H, 7.11; N, 17.02. (LC-MS) m/z: 408.09 [M+H]<sup>+</sup>.

**tert-butyl 6-(9-(3-methylbutanoyl)-9H-purin-6-ylthio) hexylcarbamate 8i:** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.77 (s, 1H), 8.67 (s, 1H), 4.51 (br s, 1H), 3.39-3.33 (m, 4H), 3.12-3.10 (m, 2H), 2.35-2.32 (m, 1H), 1.81-1.75 (m, 2H), 1.54-1.37 (m, 6H), 1.44 (s, 9H), 1.09 (d, J=6.8 Hz, 6H); IR (KBr, cm<sup>-1</sup>): 3379, 2934, 1732, 1689, 1527, 1198, 1170, 916, 639; *Anal.* Calcd. For C<sub>21</sub>H<sub>33</sub>N<sub>5</sub>O<sub>3</sub>S: C, 57.91; H, 7.64; N, 16.08; O, 11.02; S, 7.36. Found: C, 57.89; H, 7.56; N, 16.11. (LC-MS) m/z: 436.25 [M+H]<sup>+</sup>.

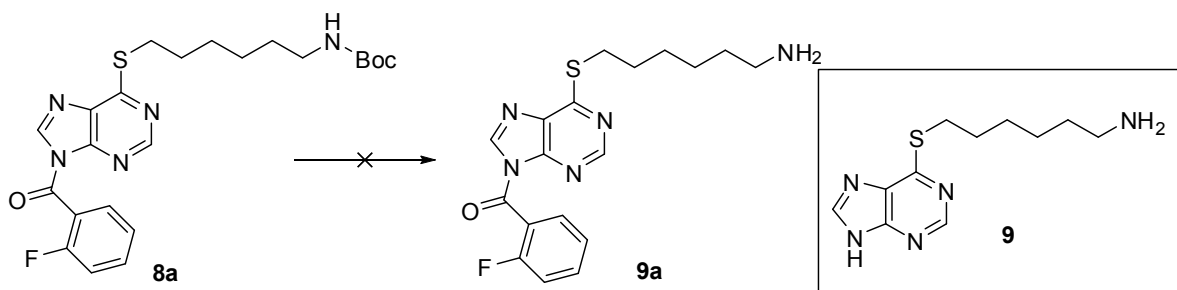
**Synthesis of 6-(9H-purin-6-ylthio) hexan-1-amine 9:** To a stirred solution of compound 8a (200 mg, 0.42 mmol) in Dichloromethane (3 ml) was added TFA (0.034 mL, 0.42 mmol) at 0°C for 30 min. Reaction was monitored by TLC. When reaction was completed, the reaction mixture was concentrated under reduced pressure. Crude compound was purified by column chromatography to get compound 9 as brown colour solid (70 mg, 66%). The same result was obtained from Dioxane-HCl method, and spectral data indicates absence of compound 9a. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ 8.68 (s, 1H), 8.44 (s, 1H), 4.2 (br s, 2H), 3.35 (t, J=6.8 Hz, 2H), 2.79-2.75 (m, 2H), 1.75-1.68 (m, 2H), 1.59-1.36 (m, 6H); (LC-MS) m/z: 252.21 [M+H]<sup>+</sup>.

### Results and Discussions

The synthesis of 9-substituted derivatives of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate (8a-8i) is described in scheme 1. Compound 3 was prepared by the Boc protection of 6-amino- hexanol and then followed by mesylation [11] in THF as a solvent (Scheme 2). 6-chloro purine was reacted with potassium thioacetate in DMF at 80°C for 6 h to get 6-mercaptopurine [12]. The obtained 6-mercaptopurine was reacted with compound 3; the reaction was carried out in anhydrous DMF in the presence of one equivalent of anhydrous potassium carbonate at room temperature for 16 h to get mixture of only mono alkylated as major, di-alkylated derivative as minor amounts 6 and 7. Another one more reaction was tried in two equivalent of anhydrous potassium carbonate to get 1: 1 ratio of mono and di-alkylated compounds. Compound 6 was reacted with different acid chlorides in presence of Triethylamine and DCM to afford the titled compounds



**Scheme 1:** Synthesis of 9-substituted tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate derivatives.



**Scheme 2:** Boc deprotection of 9-substituted tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate derivative.

compound	Molecular formula	Molecular weight	Melting range °C	Yield (%)
8a	C <sub>23</sub> H <sub>28</sub> FN <sub>5</sub> O <sub>3</sub> S	473.56	79-81	56
8b	C <sub>24</sub> H <sub>31</sub> N <sub>5</sub> O <sub>3</sub> S	469.60	98-101	58
8c	C <sub>24</sub> H <sub>28</sub> F <sub>3</sub> N <sub>5</sub> O <sub>3</sub> S	523.57	52-54	54
8d	C <sub>24</sub> H <sub>31</sub> N <sub>5</sub> O <sub>4</sub> S	485.60	84-86	62
8e	C <sub>24</sub> H <sub>31</sub> N <sub>5</sub> O <sub>4</sub> S	485.60	78-80	64
8f	C <sub>23</sub> H <sub>28</sub> ClN <sub>5</sub> O <sub>3</sub> S	490.02	100-102	53
8g	C <sub>27</sub> H <sub>31</sub> N <sub>5</sub> O <sub>3</sub> S	505.63	115-118	61
8h	C <sub>19</sub> H <sub>29</sub> N <sub>5</sub> O <sub>3</sub> S	407.53	97-99	51
8i	C <sub>21</sub> H <sub>33</sub> N <sub>5</sub> O <sub>3</sub> S	435.58	92-94	62

**Table 1:** Physical data of the newly synthesized 9-substituted derivatives of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate (8a-8i).

of 9-substituted derivatives of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate (8a-8i) in good yields. The purity of compounds 8a-8i was confirmed by using TLC, LCMS purity and their NMR spectral data. In other side we are tried for the deprotection of Boc group of

compound 8a by using different conditions like Trifluoroacetic acid in Dichloromethane, Dioxane-HCl at cold condition causes deprotection not only of the Boc group but also fission of acylated derivative to give the 6-(9H-purin-6-ylthio)hexan-1-amine 9. The compound 9 was

confirmed by <sup>1</sup>H NMR and Mass spectral data. The obtained result indicates that the derivatives of 9-substituted tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate are acid sensitive (Table 1).

## Conclusion

In conclusion, we have synthesized 9-substituted derivatives of tert-butyl 6-(9H-purin-6-ylthio) hexylcarbamate in good yields and characterized by <sup>1</sup>H NMR, IR and LCMS spectral data.

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