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## Nucleosides, Nucleotides and Nucleic Acids

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### Nucleic Acid Components and Their Analogues: New Synthesis of Bicyclic Thiopyrimidine Nucleosides

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**NUCLEIC ACID COMPONENTS AND THEIR ANALOGUES:  
NEW SYNTHESIS OF BICYCLIC THIOPYRIMIDINE  
NUCLEOSIDES**

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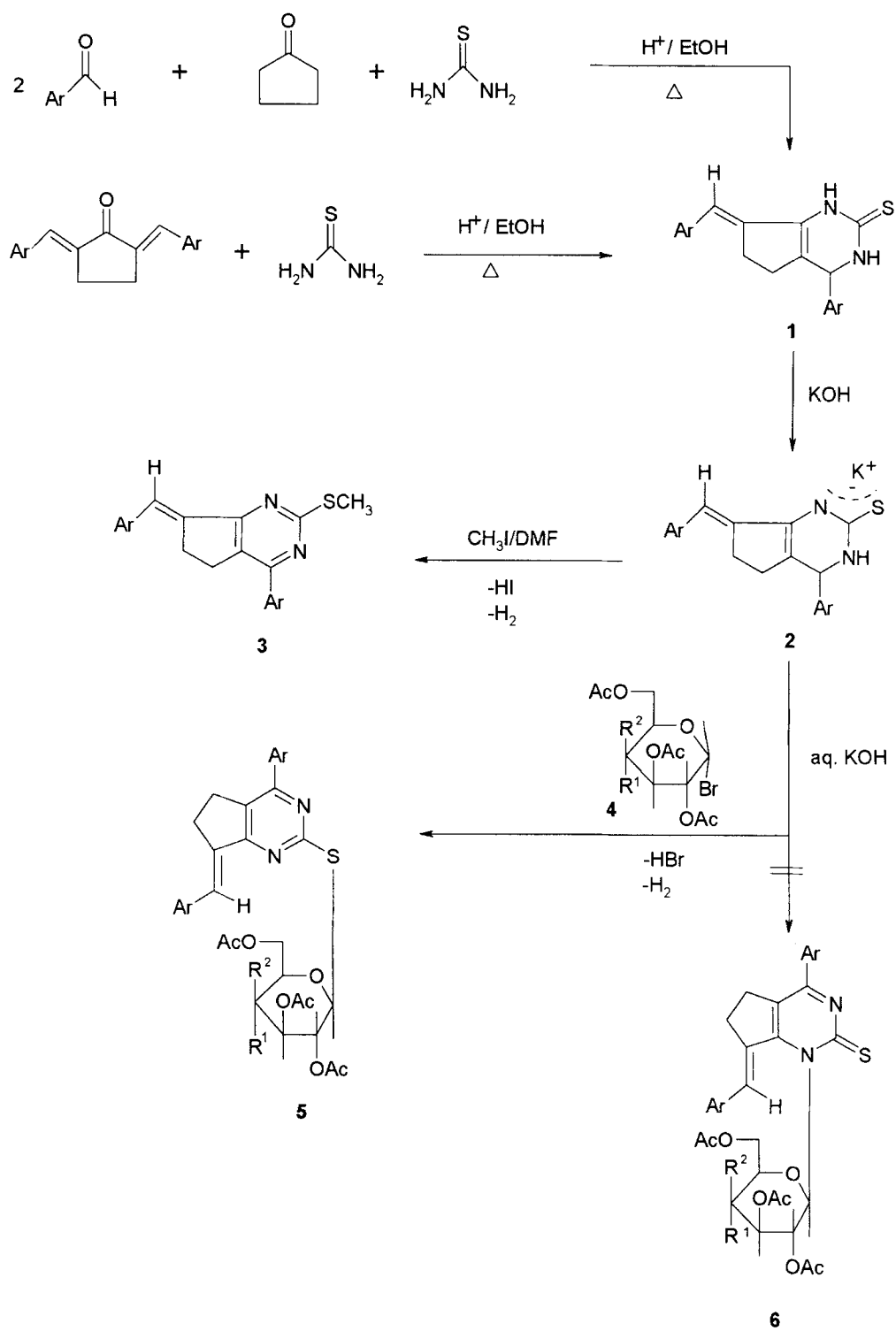
**Abstract-** A novel synthesis of condensed bicyclic thiopyrimidine glycosides utilising 1H-cyclopentapyrimidine-2(3H)-thiones and  $\alpha$ -bromoglucose or  $\alpha$ -bromogalactose tetraacetate as starting components is described.

There is increasing interest in the synthesis of nucleoside analogues and their incorporation into DNA sequences for the study of ligand-DNA and protein-DNA interactions<sup>1</sup>. A variety of nucleoside derivatives has been

prepared that result in the deletion, or change in the nature of the functional groups present on the heterocyclic bases. Such analogues permit the synthesis of oligodeoxynucleotides in which a single functional group at a preselected position has been deleted or otherwise altered. For the pyrimidine nucleosides, the base analogues prepared have generally involved the deletion or alteration of the N<sup>4</sup>-amino group, the O<sup>4</sup>-carbonyl or the C<sub>5</sub>-methyl group<sup>2</sup>. Also, the 2- and 4-thiopyrimidine derivatives were prepared as a route to alter the hydrogen bonding character of the O<sup>4</sup>-carbonyl as an alternative to deletion of the functional group<sup>3</sup>. As a part of our program of research on the synthesis of deazapyrimidine nucleosides and other antimetabolites,<sup>4-7</sup> we report here a novel and convenient method for the synthesis of condensed 2-thiopyrimidine carbocyclic nucleosides utilizing the condensed pyrimidinethiones **1** as starting materials. Compounds **1** were prepared by the reaction of cyclopentanone with aldehydes and thiourea in boiling ethanol containing catalytic amounts of hydrochloric acid. Compounds **1** could also be prepared by the condensation of 2,6-diarylmethylenecyclopentanones with thiourea in refluxing ethanolic hydrochloric acid. The structures of the reaction products **1** were established and confirmed on the basis of their elemental analyses and spectral data (MS, IR, UV, <sup>1</sup>H NMR, <sup>13</sup>C NMR). Thus, the analytical data for **1d** revealed a molecular formula C<sub>20</sub>H<sub>16</sub>Cl<sub>2</sub>N<sub>2</sub>S (m/z 387). The <sup>1</sup>H NMR spectrum showed a singlet at δ 5.22 ppm assigned for a saturated pyrimidine H-4 proton and two broad singlets at δ 8.96 and 10.08 ppm assigned for two NH groups. The <sup>13</sup>C NMR spectrum of **1d** was characterized by a signal at δ 178 ppm corresponding to the thione carbon. Compounds **1** bearing latent functional substituents were found useful for the synthesis of interesting S-alkyl and S-glycoside derivatives. Thus, it has been found that **1** reacted with methyl iodide in DMF-KOH and successfully obtained the corresponding S-alkyl derivatives **3** through methylation and aromatization under the reaction

conditions. The structure of **3** was established by mass spectroscopy and IR and  $^1\text{H}$  NMR data. The  $^1\text{H}$  NMR spectra for **3b** showed a band at  $\delta = 2.7$  ppm assigned to  $\text{SCH}_3$  group. Similarly, compounds **1** reacted with tetra-*O*-acetyl- $\alpha$ -D-glucopyranosyl bromides **4** in the presence of aqueous potassium hydroxide to give *S*-glycosylated pyrimidines **5** through hydrogen bromide elimination and aromatization under the reaction conditions. The chemical formula and molecular structure of the formed glycosides **5** were identified using elemental analyses and spectral data (MS, IR, UV,  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR). The  $^1\text{H}$  NMR spectrum showed a doublet at 6.08 ppm assigned to the anomeric proton of the glucose moiety with a spin-spin coupling constant equal to 10.24 Hz which corresponds to a diaxial orientation of H-1' and H-2' protons indicating the presence of only  $\beta$ -configuration and  $^4\text{C}_1$  (D) conformation for this glucoside. The other protons of the glucopyranosyl ring resonates at  $\delta$  4.04-5.21 ppm region, while the four acetoxy groups appear as four singlets at  $\delta$  1.80-2.13 ppm region. The  $^{13}\text{C}$  NMR spectrum of **5b** was characterized by a signal at  $\delta$  81.2 ppm corresponding to the C-1' atom of the  $\beta$ -D-glucopyranose. It may be argued that the coupling reaction of **1** with **4** happened on the nitrogen atom to give the corresponding N-glycosides **6**. The formation of **5** was proven using  $^{13}\text{C}$  NMR which revealed the absence of the thione carbon at  $\delta$  178 ppm and appearance of C-2 carbon at  $\delta$  161 ppm of the same value of the corresponding *S*-alkyl derivatives **3**. The UV spectrum of **5b** proved that the reaction had led selectively to the formation of *S*-glucosyl derivatives since the *S*-methyl derivative of **1f** and its glucoside **5b** give the same UV absorption maxima at 298 and 367 nm. Moreover, attempt removal of protecting groups by methanolic ammonia has not resulted in formation of the corresponding free glycosides.

In summary this simple step provide a novel and efficient synthetic method



Ar			Ar			Ar		
<b>1a</b> , C <sub>6</sub> H <sub>5</sub>			<b>1 f</b> , 4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>			<b>3 a</b> , C <sub>6</sub> H <sub>4</sub>		
<b>b</b> , 3-Br-C <sub>6</sub> H <sub>4</sub>			<b>g</b> , 4-CH <sub>3</sub> O-C <sub>6</sub> H <sub>4</sub>			<b>b</b> , 3-Br-C <sub>6</sub> H <sub>4</sub>		
<b>c</b> , 2-Cl-C <sub>6</sub> H <sub>4</sub>			<b>h</b> , 3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>			<b>c</b> , 4-Cl-C <sub>6</sub> H <sub>4</sub>		
<b>d</b> , 4-Cl-C <sub>6</sub> H <sub>4</sub>			<b>i</b> , 4-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>			<b>d</b> , 3-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>		
<b>e</b> , 3-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>			<b>j</b> , 4-(CH <sub>3</sub> ) <sub>2</sub> N-C <sub>6</sub> H <sub>4</sub>			<b>e</b> , 4-CH <sub>3</sub> O-C <sub>6</sub> H <sub>4</sub>		

Ar		R <sup>1</sup>	R <sup>2</sup>	Ar		R <sup>1</sup>	R <sup>2</sup>
<b>5 a</b> , 4-Cl-C <sub>6</sub> H <sub>4</sub>		OAc	H	<b>5d</b> , 4-Cl-C <sub>6</sub> H <sub>4</sub>		H	OAc
<b>b</b> , 4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>		OAc	H	<b>e</b> , 4-CH <sub>3</sub> -C <sub>6</sub> H <sub>4</sub>		H	OAc
<b>c</b> , 4-CH <sub>3</sub> O-C <sub>6</sub> H <sub>4</sub>		OAc	H	<b>f</b> , 4-CH <sub>3</sub> O-C <sub>6</sub> H <sub>4</sub>		H	OAc

for a new class of nonclassical pyrimidine nucleosides, with regard to experimental convenience and overall yield.

## Experimental

All evaporations were carried out under reduced pressure at 40 °C. Melting points are uncorrected. TLC aluminium sheets silica gel 60F254 (Merck) was used for thin layer chromatography. detection as effected by viewing under a short-wavelength UV lamp. IR spectra were obtained (KBr disc) on a Pye Unicam Spectra-1000. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were measured on a Wilmad 270 MHz or on a Varian 400 MHz spectrometer for solutions in (CD<sub>3</sub>)<sub>2</sub>SO using SiMe<sub>4</sub> as internal standard. Mass spectra were recorded on a Varian MAT 112 spectrometer. Analytical data were obtained from the Microanalytical data Center at Cairo University.

#### 4-Aryl-7-(substitutedbenzylidene)-4,5,6,7-tetrahydro-1H-cyclopentapyrimidine-2(3H)-thiones 1a-j

##### General Procedure:

**Method A:** To a mixture of aromatic aldehyde (0.02 mol), cyclopentanone (0.01 mol) and thiourea (0.01 mol) in ethanol (50 ML), conc. HCl (2 ML) was added. The mixture was heated under reflux for 4 h and then left to stand overnight. The resultant precipitate was filtered off and crystallized from EtOH-DMF to afford yellow crystals.

**Method B:** To a mixture of 2,6-diarylmethylenecyclopentanones (0.01 mol) and thiourea (0.01 mol) in ethanol (50 ML), conc. HCl (2 ML) was added. The reaction mixture was heated under reflux for 3 h and then left to stand overnight. The resultant precipitate was filtered off and recrystallized from EtOH-DMF to afford yellow crystals.

**1a:** Yield 60%, mp 232°C; IR 3150 (NH), 3050 (NH), 1680 (C=C), 1616 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 2.72 (s, 2H,  $\text{CH}_2$ ), 2.88(s, 2H,  $\text{CH}_2$ ), 5.20(s, 1H, CH), 6.85 (s, 1H, =CH), 7.44 (m, 10H,  $2\text{C}_6\text{H}_5$ ), 8.96 (s, 1H, NH), 10.06 (s, 1H, NH); m/z 318 (Found: C,75.7; H,5.8; N,8.9.  $\text{C}_{20}\text{H}_{18}\text{N}_2\text{S}$  requires C,75.5; H, 5.7; N, 8.8% ).

**1b:** Yield 63%, mp 220 °C; IR 3300 (NH), 3150 (NH), 1670 (C=C), 1628 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 2.66 (s, 2H,  $\text{CH}_2$ ), 2.92 (s, 2H,  $\text{CH}_2$ ), 5.28 (s, 1H, CH), 6.86 (s, 1H, =CH), 7.43 (m, 8H,  $2\text{C}_6\text{H}_4$ ), 9.08 (s, 1H, NH), 10.14 (s, 1H, NH); m/z 476 (Found: C, 50.7; H, 3.6 ; N, 6.1.  $\text{C}_{20}\text{H}_{16}\text{N}_2\text{SBr}_2$  requires C, 50.4; H, 3.4; N, 5.9% ).

**1c:** Yield 54%, mp 195 °C; IR 3330 (NH), 3300 (NH), 1650 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 2.65 (s, 2H,  $\text{CH}_2$ ), 2.98 (s, 2H,  $\text{CH}_2$ ), 5.34 (s, 1H, CH), 6.71 (s,

1H, =CH), 7.39-8.22 (m, 8H, 2C<sub>6</sub>H<sub>4</sub>), 9.21 (s, 1H, NH), 10.25 (s, 1H, NH); <sup>13</sup>C NMR 28.1 (CH<sub>2</sub>), 29.2 (CH<sub>2</sub>), 58.2 (C-4), 118.2-134.5 (Ar-carbons), 138.6 (C-5), 142.5 (C-6), 175.1 (C=S); m/z 387 (Found: C, 62.3; H, 4.3; N, 7.5. C<sub>20</sub>H<sub>16</sub>N<sub>2</sub>SCl<sub>2</sub> requires C, 62.0; H, 4.1; N, 7.2% ).

**1d:** Yield 57%, mp 235 °C; IR 3340 (NH), 3190 (NH), 1666 (C=C), 1626 (C=C) cm<sup>-1</sup>; <sup>1</sup>H NMR 2.74 (s, 2H, CH<sub>2</sub>), 2.85 (s, 2H, CH<sub>2</sub>), 5.22 (s, 1H, CH), 6.90 (s, 1H, =CH), 7.36 (m, 8H, 2C<sub>6</sub>H<sub>4</sub>), 8.96 (s, 1H, NH), 10.08 (s, 1H, NH); m/z 387 (Found: C, 62.2; H, 4.1; N, 7.4. C<sub>20</sub>H<sub>16</sub>N<sub>2</sub>SCl<sub>2</sub> requires C, 62.0; H, 4.1; N, 7.2% ).

**1e:** Yield 65%, mp 235 °C; IR 3400 (NH), 3210 (NH), 1668 (C=C), 1630 (C=C) cm<sup>-1</sup>; <sup>1</sup>H NMR 2.16 (s, 2H, CH<sub>2</sub>), 2.32 (2s, 6H, 2CH<sub>3</sub>), 2.85 (s, 2H, CH<sub>2</sub>), 5.24 (s, 1H, CH), 6.92 (s, 1H, =CH), 7.28 (m, 8H, 2C<sub>6</sub>H<sub>4</sub>), 8.96 (s, 1H, NH), 10.08 (s, 1H, NH); m/z 346 (Found: C, 76.5; H, 6.5; N, 8.3. C<sub>22</sub>H<sub>22</sub>N<sub>2</sub>S requires C, 76.3; H, 6.4; N, 8.1% ).

**1f:** Yield 64%, mp 184 °C; IR 3250 (NH), 3050 (NH), 1666 (C=C), 1616 (C=C) cm<sup>-1</sup>; <sup>1</sup>H NMR 2.18 (s, 2H, CH<sub>2</sub>), 2.30 (2s, 6H, 2CH<sub>3</sub>), 2.86 (s, 2H, CH<sub>2</sub>), 5.45 (s, 1H, CH), 7.08 (s, 1H, =CH), 7.52 (m, 4H, C<sub>6</sub>H<sub>4</sub>), 8.26 (m, 4H, C<sub>6</sub>H<sub>4</sub>), 9.18 (s, 1H, NH), 10.25 (s, 1H, NH); m/z 346 (Found: C, 76.6; H, 6.4; N, 8.2. C<sub>22</sub>H<sub>22</sub>N<sub>2</sub>S requires C, 76.3; H, 6.4; N, 8.1% ).

**1g:** Yield 50%, mp 201 °C; IR, 3310 (NH), 3160 (NH), 1692 (C=C), 1620 (C=C) cm<sup>-1</sup>; <sup>1</sup>H NMR 2.24 (m, 2H, CH<sub>2</sub>), 2.80 (m, 2H, CH<sub>2</sub>), 3.82 (2s, 6H, 2OCH<sub>3</sub>), 5.14 (s, 1H, CH), 6.98 (m, 1H, =CH and 4H, C<sub>6</sub>H<sub>4</sub>), 7.26 (m, 4H, C<sub>6</sub>H<sub>4</sub>), 8.86 (s, 1H, NH), 9.92 (s, 1H, NH); m/z 378 (Found: C, 70.1; H, 5.9; N, 7.6. C<sub>22</sub>H<sub>22</sub>N<sub>2</sub>SO<sub>2</sub> requires C, 69.8; H, 5.8; N, 7.4% ).

**1h:** Yield 46%, mp 230 °C; IR, 3380 (NH), 3168 (NH), 1668 (C=C),



1630 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 2.18 (s, 2H,  $\text{CH}_2$ ), 2.86 (s, 2H,  $\text{CH}_2$ ), 5.50 (s, 1H, CH), 7.14 (s, 1H, =CH), 7.72 (m, 4H,  $\text{C}_6\text{H}_4$ ), 8.12 (m, 4H,  $\text{C}_6\text{H}_4$ ), 9.16 (s, 1H, NH), 10.20 (s, 1H, NH);  $m/z$  408 (Found: C, 59.0; H, 4.1; N, 13.9.  $\text{C}_{20}\text{H}_{16}\text{N}_4\text{SO}_4$  requires C, 58.8; H, 3.9; N, 13.7%).

**1i:** Yield 48%, mp 228 °C; IR, 3320 (NH), 3180 (NH), 1668 (C=C), 1628 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 2.22 (s, 2H,  $\text{CH}_2$ ), 2.90 (s, 2H,  $\text{CH}_2$ ), 5.46 (s, 1H, CH), 7.08 (s, 1H, =CH), 7.54 (m, 4H,  $\text{C}_6\text{H}_4$ ), 8.22 (m, 4H,  $\text{C}_6\text{H}_4$ ), 9.16 (s, 1H, NH), 10.28 (s, 1H, NH);  $m/z$  408 (Found: C, 58.9; H, 4.0; N, 13.9.  $\text{C}_{20}\text{H}_{16}\text{N}_4\text{SO}_4$  requires C, 58.8; H, 3.9; N, 13.7%).

**1j:** Yield 53%, mp 215 °C; IR 3340 (NH), 3300 (NH), 1645 (C=C)  $\text{cm}^{-1}$ ;  $m/z$  404 (Found: C, 71.6; H, 7.0; N, 14.2.  $\text{C}_{24}\text{H}_{28}\text{N}_4\text{S}$  requires C, 71.3; H, 6.9; N, 13.9%).

#### 4-Aryl-7-(substitutedbenzylidene)-5,6-dihydro-2-(methylthio)-7H-cyclopentapyrimidines 3a-e

##### General procedure:

Condensed pyrimidine-2-thiones **1** (0.01 mol) was suspended in a solution of sodium ethoxide (0.01 mol). An excess of methyl iodide (0.015 mol) was added dropwise to the resulting mixture. The precipitate obtained after 2 h stirring at room temperature was filtered off and recrystallized from ethanol to afford yellow crystals.

**3a:** Yield 47%, mp 136 °C; IR 1650 (C=C)  $\text{cm}^{-1}$ ;  $m/z$  330 (Found: C, 76.5; H, 5.6; N, 8.8.  $\text{C}_{21}\text{H}_{18}\text{N}_2\text{S}$  requires C, 76.4; H, 5.5; N, 8.5%).

**3b:** Yield 50%, mp 159 °C; IR, 1690 (C=C), 1626 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 2.66 (s, 2H,  $\text{CH}_2$ ), 3.05 (s, 2H,  $\text{CH}_2$ ), 3.48 (s, 3H,  $\text{SCH}_3$ ), 7.68 (m, 1H,

=CH and 8H, 2C<sub>6</sub>H<sub>4</sub>); m/z 488 (Found: C, 51.9; H, 3.4; N, 5.8. C<sub>21</sub>H<sub>16</sub>N<sub>2</sub>SBr<sub>2</sub> requires C, 51.6; H, 3.3; N, 5.7%).

**3c:** Yield 52%, mp 166 °C; IR, 1680 (C=C), 1628 (C=C) cm<sup>-1</sup>; <sup>1</sup>H NMR 3.04 (m, 4H, 2CH<sub>2</sub>), 3.55 (s, 3H, SCH<sub>3</sub>), 7.12 (m, 1H, =CH and 4H, C<sub>6</sub>H<sub>4</sub>), 7.78 (m, 4H, C<sub>6</sub>H<sub>4</sub>); m/z 399 (Found: C, 63.5; H, 4.2; N, 7.1. C<sub>21</sub>H<sub>16</sub>N<sub>2</sub>SCl<sub>2</sub> requires C, 63.2; H, 4.0; N, 7.0%).

**3d:** Yield 45%, mp 110 °C; IR, 1686 (C=C), 1630 (C=C) cm<sup>-1</sup>; m/z 358 (Found: C, 77.3; H, 6.2; N, 8.0. C<sub>23</sub>H<sub>22</sub>N<sub>2</sub>S requires C, 77.1; H, 6.1; N, 7.8%).

**3e:** Yield 50%, mp 162 °C; IR 1664 (C=C), 1635 (C=C) cm<sup>-1</sup>; <sup>1</sup>H NMR 2.60 (s, 2H, CH<sub>2</sub>), 3.02 (s, 2H, CH<sub>2</sub>), 3.44 (s, 3H, SCH<sub>3</sub>), 3.85 (2s, 6H, 2OCH<sub>3</sub>), 7.05 (m, 1H, =CH and 4H, C<sub>6</sub>H<sub>4</sub>), 7.62 (m, 4H, C<sub>6</sub>H<sub>4</sub>); m/z 390 (Found: C, 71.1; H, 5.6; N, 7.3. C<sub>23</sub>H<sub>22</sub>N<sub>2</sub>SO<sub>2</sub> requires C, 70.8; H, 5.6; N, 7.2%).

#### 4-Aryl-7-substitutedbenzylidine-5,6-dihydro-2-(2,3,4,6-tetra-*O*-acetyl-β-D-glycopyranosylthio)-7H-cyclopentapyrimidines 5a-f.

##### General coupling procedures

To a solution of 1H-cyclopentapyrimidine-2(3H)-thiones **1** (0.01 mol) in aqueous potassium hydroxide [0.56 g, 0.01 mol, in 6 ML of distilled water], was added a solution of 2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl bromide **4** (0.011 mol) in acetone (30 ML). The reaction mixture was stirred at room temperature until reaction was judged complete by TLC (2-3 h), using chloroform: ether 4:1, v/v (R<sub>f</sub> 0.68-0.74 region), then evaporated under reduced pressure at 40 °C and the residue

was washed with distilled water to remove the potassium bromide formed. The product was dried and recrystallized from EtOH to afford pale yellow crystals.

**5a:** Yield 61%, mp 130 °C; IR, 1748 (C=O ester), 1630 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 1.79-1.97 (4s, 12H, 4CH<sub>3</sub>CO), 2.34 (s, 2H, CH<sub>2</sub>), 2.84 (s, 2H, CH<sub>2</sub>), 3.97 (m, 2H, 2H-6' and 1H, H-5'), 4.88 (m, 1H, H-4'), 5.03 (m, 1H, H-3'), 5.15 (t, 1H, H-2'), 5.98 (d,  $J_{1,2} = 10.50$  Hz, 1H, H-1'), 6.96 (s, 1H, =CH), 7.42 (m, 8H, 2C<sub>6</sub>H<sub>4</sub>); m/z 715 (Found: C, 57.3; H, 4.6; N, 4.1. C<sub>34</sub>H<sub>32</sub>N<sub>2</sub>SCl<sub>2</sub>O<sub>9</sub> requires C, 57.1; H, 4.5; N, 3.9%).

**5b:** Yield 63%, mp 123 °C; IR, 1756 (C=O ester), 1625 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 1.80-2.13 (4s, 12H, 4CH<sub>3</sub>CO), 2.24 (s, 3H, CH<sub>3</sub>), 2.32 (s, 2H, CH<sub>2</sub>), 2.37 (s, 2H, CH<sub>2</sub>), 4.04 (m, 2H, 2H-6' and 1H, H-5'), 4.96 (m, 2H, H-4' and H-3'), 5.2 (t, 1H, H-2'), 6.08 (d,  $J_{1,2} = 10.24$  Hz, 1H, H-1'), 7.01 (s, 1H, =CH), 7.54 (m, 8H, 2C<sub>6</sub>H<sub>4</sub>); m/z 674 (Found: C, 64.2; H, 5.7; N, 4.3. C<sub>36</sub>H<sub>38</sub>N<sub>2</sub>SO<sub>9</sub> requires C, 64.1; H, 5.6; N, 4.2%).

**5c:** Yield 64%, mp 161 °C; IR, 1750 (C=O ester), 1628 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 1.78-2.12 (4s, 12H, 4CH<sub>3</sub>CO), 3.02 (s, 2H, CH<sub>2</sub>), 3.10 (s, 2H, CH<sub>2</sub>), 3.88 (s, 3H, OCH<sub>3</sub>), 3.94 (s, 3H, OCH<sub>3</sub>), 4.11 (m, 2H, H-6' and 1H, H-5'), 4.58 (s, 1H, H-4'), 5.21 (t, 1H, H-3'), 5.64 (m, 1H, H-2'), 6.05 (d,  $J_{1,2} = 10.46$  Hz, 1H, H-1'), 7.18 (m, 1H, =CH and 4H, C<sub>6</sub>H<sub>4</sub>), 7.66 (m, 4H, C<sub>6</sub>H<sub>4</sub>); m/z 706 (Found: C, 61.4; H, 5.4; N, 4.1. C<sub>36</sub>H<sub>38</sub>N<sub>2</sub>SO<sub>11</sub> requires C, 61.2; H, 5.4; N, 4.0%).

**5d:** Yield 60%, mp 154 °C; IR 1740 (C=O ester); m/z 715 (Found: C, 57.2; H, 4.5; N, 4.1. C<sub>34</sub>H<sub>32</sub>N<sub>2</sub>SCl<sub>2</sub>O<sub>9</sub> requires C, 57.1; H, 4.5; N, 3.9%).

**5e:** Yield 63%, mp 135 °C; IR 1760 (C=O ester); m/z 674 (Found: C, 64.3; H, 5.7; N, 4.3. C<sub>36</sub>H<sub>38</sub>N<sub>2</sub>SO<sub>9</sub> requires C, 64.1; H, 5.6; N, 4.2%).

**5f:** Yield 65%, mp 146 °C; IR, 1754 (C=O ester), 1626 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR 1.69-2.15 (4s, 12H, 4CH<sub>3</sub>CO), 3.04 (s, 2H, CH<sub>2</sub>), 3.14 (s, 2H, CH<sub>2</sub>), 3.81(s, 3H, OCH<sub>3</sub>), 3.85 (s, 3H, OCH<sub>3</sub>), 4.07(m, 2H, 2H-6' and 1H, H-5'), 5.17(t, 1H, H-4'), 5.42 (s, 1H, H-3'), 5.58 (t, 1H, H-2'), 6.08 (d,  $J_{1-2}=10.60$  Hz, 1H, H-1'), 7.05 (m, 1H, =CH and 4H, C<sub>6</sub>H<sub>4</sub>), 7.63 (d, 2H, Ar-H), 8.07 (d, 2H, Ar-H); m/z 706 (Found: C, 61.3; H, 5.5; N, 4.1. C<sub>36</sub>H<sub>38</sub>N<sub>2</sub>SO<sub>11</sub> requires C, 61.2; H, 5.4; N, 4.0%).

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