

## 5-Acetyl-6-amino-4-methylthio-2-phenylpyrimidine and its use in the synthesis of functionalized pyrido[2,3-d]pyrimidines and pyrimido[4,5-d]pyrimidines

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Schemes for the synthesis of MeS-substituted pyrido[2,3-d]pyrimidin-5-one, pyrimido[4,5-d]pyrimidine, and 4-methylenepyrimido[4,5-d]pyrimidin-2-one based on 5-acetyl-6-amino-4-methylthio-2-phenylpyrimidine, which was prepared from the adduct of benzoyl isothiocyanate with acetylketene *N*-benzoylaminal, were suggested.

**Key words:** 5-acetyl-6-amino-4-methylthio-2-phenylpyrimidine, pyrido[2,3-d]pyrimidin-5-one, pyrimido[4,5-d]pyrimidine, 4-methylenepyrimido[4,5-d]pyrimidin-2-one, acetylketene aminal, benzoyl isothiocyanate, phenyl isocyanate, dimethylformamide dimethylacetal, dimethylacetamide dimethylacetal.

One of the convenient ways for building fused nitrogen-containing cyclic systems involves annelation of a pyridine or pyrimidine ring to a molecule of a heterocyclic compound containing vicinal NH<sub>2</sub> and RCO groups. Previously we used this approach to prepare 1,2,3-triazolo[4,5-d]pyrimidines (8-azapurines)<sup>1</sup> from 5-acyl-4-aminotriazoles, pyrazolo[3,4-d]pyrimidines<sup>2</sup> from 4-acetyl-5-amino-1,3-dimethylpyrazole, and pyrido[2,3-d]pyrimidin-5-ones<sup>3,4</sup> from 5-acetyl-4-aminopyrimidines or from 3-acetyl-2-amino-6-phenylpyridin-4-one.

In the previous paper<sup>5</sup> it was shown that acetylketene *N*-benzoylaminal<sup>6</sup> reacts with benzoyl isothiocyanate as a C-nucleophile to give the corresponding adduct, *N*-benzoylthioamide (**1**), which was subsequently used to prepare 5-acetyl-6-benzoylamino-2-phenyl-3*H*-pyrimidine-4-thione (**2**) and its *S*-methyl derivative (**3**) (Scheme 1). It has been of interest to convert the latter compound into pyrimidine containing an unsubstituted NH<sub>2</sub> group, which can be used to annelate the second nitrogen-containing ring. However, when we attempted to debenzoylate compound **3** through the action of MeONa in MeOH, competing intramolecular cyclization involving the benzoyl group and giving 4-methylthio-2,7-diphenyl-8*H*-pyrido[2,3-d]pyrimidin-5-one occurred (in boiling BuOH, this process predominates<sup>5</sup>).

Nevertheless, in the present work we were able to carry out the conversion of thioamide **1** to pyrimidines with vicinal NH<sub>2</sub> and MeCO groups and further to synthesize derivatives of pyrido[2,3-d]pyrimidine and pyrimido[4,5-d]pyrimidine (Scheme 1). It turned out that when compound **1** is boiled with two equivalents of MeONa in MeOH, the closure of the pyrimidine ring is

accompanied by debenzoylation. The resulting salt (**4**) incorporating an unsubstituted NH<sub>2</sub> group can be easily converted into pyrimidinethione (**5**) or its *S*-methyl derivative (**6**) when treated with AcOH or MeI, respectively. Pyrimidinethione **2**, unlike 4-methylthiopyrimidine **3**, can be converted into compound **5** or **6** via salt **4**. In this case, no intramolecular cyclization occurs.

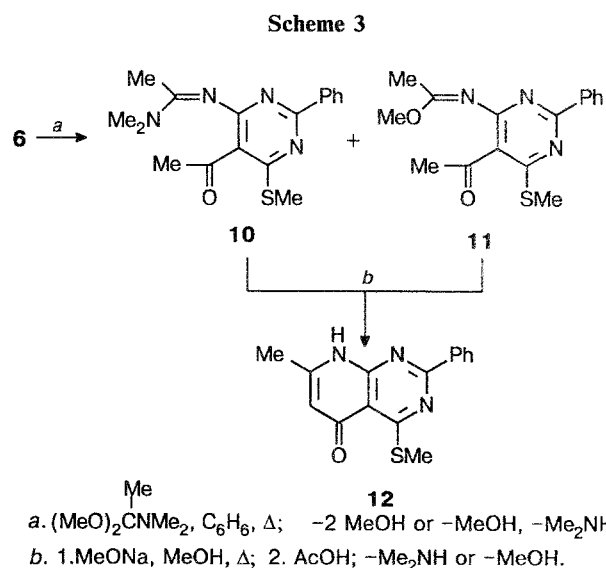
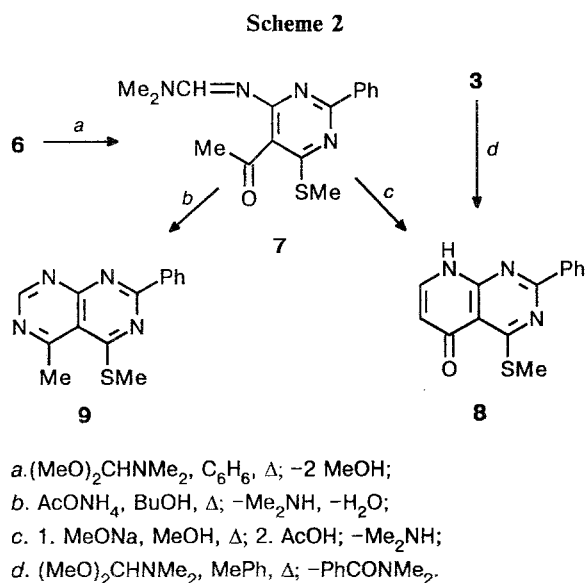
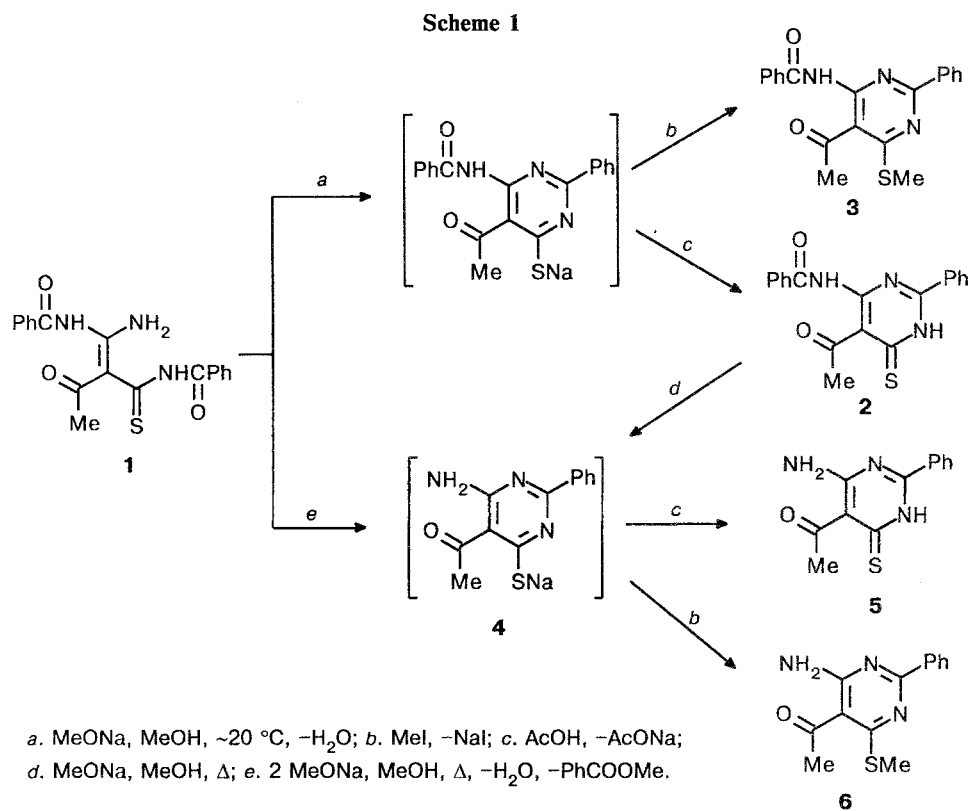
The structures of the products were confirmed by spectral methods.

Similarly to the previously described 5-acetyl-4-aminopyrimidines,<sup>3</sup> 4-methylthiopyrimidine **6** is converted into the corresponding amidine (**7**) by boiling in benzene with DMF dimethylacetal. The treatment of amidine **7** with MeONa in MeOH results in cyclization to give 4-methylthio-2-phenylpyrido[2,3-d]pyrimidin-5-one (**8**) (Scheme 2). The latter is also formed from *N*-benzoylamino-4-aminopyrimidine **3** and DMF acetal.<sup>5</sup>

Boiling amidine **7** with ammonium acetate in BuOH gave 5-methyl-4-methylthio-2-phenylpyrimido[4,5-d]pyrimidine (**9**), whose structure was confirmed by spectral data. Condensation of compound **5** with DMF dimethylacetal is accompanied by the methylation at the *S* atom, which also affords amidine **7** (for methylation of pyrimidinethiones with amide acetals, see Refs. 5 and 7).

The reaction of aminopyrimidine **6** with dimethylacetamide (DMA) acetal under conditions similar to those of its reaction with DMF acetal gives, in addition to acetamide (**10**), acetimidate (**11**) (Scheme 3). Compounds **10** and **11** were isolated by chromatography in 55 and 36 % yields, respectively.

The competing elimination of dialkylamino and alkoxy groups is not typical of transformations of amide acetals.<sup>8</sup> However, it has been shown recently<sup>9</sup> that the condensation of DMA diethylacetal with indolin-



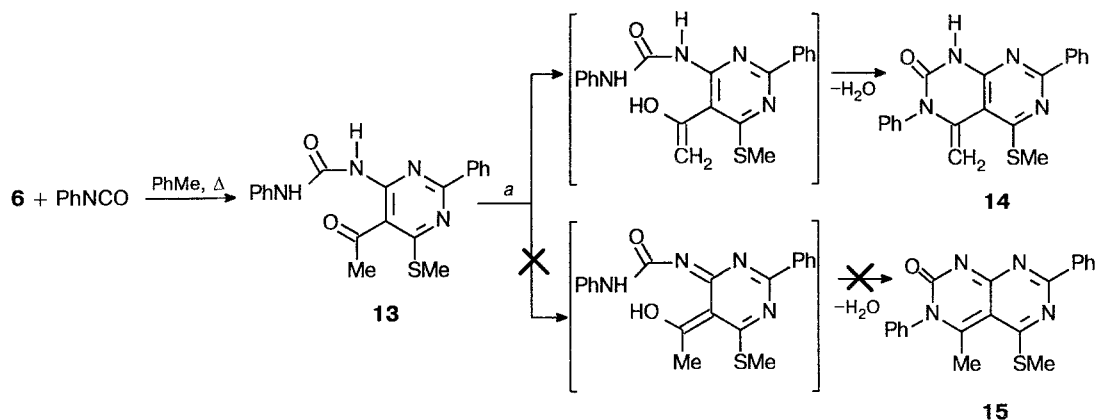
2-one predominantly occurs with the elimination of  $\text{Me}_2\text{NH}$ .

The above-mentioned peculiarity of the reaction of compound **6** with DMA acetal does not interfere with its efficient use in the construction of the pyridopyrimidine system, because amidine **10** and imidate **11** when treated by MeONa in boiling MeOH give the same product of cyclization, *viz.*, 7-methyl-4-methylthio-2-phenylpyrido-[2,3-d]pyrimidin-5-one (**12**) (Scheme 3).

Unlike imidate **11**, which is readily soluble almost in all of the organic solvents, amidine **10** is poorly soluble in petroleum ether. The structures of compounds **10**–**12** was confirmed by spectral methods.

Further we considered the route to the pyrimido-[4,5-d]pyrimidine system starting from pyrimidine **6** and phenyl isocyanate. We found that boiling these reactants in toluene results in the formation of urea (**13**) (Scheme 4). However, it has been shown previously that

Scheme 4



a. MeONa, MeOH,  $\Delta$ .

5-acetyl-4-aminopyrimidin-2-ones, monosubstituted at the exocyclic N atom, react with isocyanates under similar conditions to directly give 4-methylene-1*H*,3*H*,6*H*-pyrimido[4,5-*d*]pyrimidin-2,7-dione derivatives.<sup>10</sup> We expected that intramolecular cyclization of urea **13** through the action of MeONa in MeOH would yield pyrimido[4,5-*d*]pyrimidin-2-one (**15**) (preparation of type **15** compounds from 4-*RNH*-5-acetylpyrimidin-2-ones and isocyanates is impossible). However, 4-methylene-5-methylthio-3,7-diphenyl-1*H*,3*H*-pyrimido[4,5-*d*]pyrimidin-2-one (**14**) was isolated as the reaction product in 74 % yield (Scheme 4).

The structure of product **14** was confirmed by spectral methods. The **6**→**13**→**14** transformation indicates that the approach to the synthesis of pyrimido[4,5-*d*]pyrimidines with an exocyclic methylene group based on the reaction of pyrimidines with vicinal MeCO and NHR groups with isocyanates is a more general method.

The possibility of replacing the MeS group in the pyrimidine ring makes bicyclic compounds **8**, **9**, **12**, and **14** useful starting reactants for preparing new derivatives of pyrido[2,3-*d*]pyrimidine and pyrimido[4,5-*d*]pyrimidine. Heterocyclic compounds of these classes exhibit diversified biological properties.

### Experimental

<sup>1</sup>H NMR spectra were recorded on a Bruker WM-250 instrument and <sup>13</sup>C NMR spectra were obtained on a Bruker AM-300 spectrometer. IR spectra were run on UR-20 (in solutions) and Perkin-Elmer 577 (in KBr pellets) instruments. Mass spectra were obtained on a Varian MAT-311A mass spectrometer (EI, 70 eV). Preparative-scale TLC was carried out using Kieselgel 60F<sub>254</sub> plates (Merck). 2-Acetyl-3-amino-3-benzoylamino-*N*-benzoylthioamide of acrylic acid **1** was synthesized according to the known procedure.<sup>5</sup>

#### 5-Acetyl-6-amino-2-phenyl-3*H*-pyrimidine-4-thione (**5**).

a. A mixture of thioamide **1** (0.367 g, 1 mmol) and MeONa (2 mmol) in 10 mL of MeOH was boiled for 1.5 h. The

solvent was evaporated *in vacuo*, and 15 mL of H<sub>2</sub>O and AcOH were added until pH ~5. The precipitate was filtered off and dried to give 206 mg (84 %) of pyrimidinethione **5** as a yellow crystalline solid with m.p. 241–244 °C (dec.) (from ethanol), slightly soluble in EtOH, C<sub>6</sub>H<sub>6</sub>, and CHCl<sub>3</sub> and insoluble in H<sub>2</sub>O. Found (%): C, 58.85; H, 4.63; N, 16.61; S, 12.58. C<sub>12</sub>H<sub>11</sub>N<sub>3</sub>OS. Calculated (%): C, 58.75; H, 4.52; N, 17.13; S, 13.07. IR (CHCl<sub>3</sub>),  $\nu/\text{cm}^{-1}$ : 3470 (NH); 3370 (NH); 3280 (NH); 1623 (CO); 1600; 1578; 1525. <sup>1</sup>H NMR (CDCl<sub>3</sub>),  $\delta$ : 3.00 (s, 3 H, Me); 6.02 (br.s, 1 H, NH); 7.57 (m, 2 H, Ph); 7.65 (m, 1 H, Ph); 7.99 (m, 2 H, Ph); 9.90 (br.s, 1 H, NH); 10.12 (br.s, 1 H, NH). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>),  $\delta$ : 32.85 (Me); 111.90 (C(5)); 128.37; 128.60; 130.76; 132.36 (Ph); 156.42 and 159.73 (C(2) and C(6)); 180.41 (C(4)); 201.64 (CO). MS,  $m/z$  ( $I_{\text{rel}}(\%)$ ): 245 [M]<sup>+</sup> (95), 244 [M-H]<sup>+</sup> (100), 230 [M-Me]<sup>+</sup> (21).

b. A mixture of pyrimidinethione **2** (0.349 g, 1 mmol) and MeONa (1 mmol) in 10 mL of MeOH was boiled for 1 h. Pyrimidinethione **5** (0.21 g, 87 %) was isolated as described in procedure a.

#### 5-Acetyl-6-amino-4-methylthio-2-phenylpyrimidine (**6**).

a. A mixture of thioamide **1** (0.51 g, 1.4 mmol) and MeONa (2.8 mmol) in 10 mL of MeOH was boiled for 1.5 h and cooled to ~20 °C. MeI (0.35 mL, 5.6 mmol) was added, and the mixture was stirred for 30 min. The precipitate was filtered off to give 0.32 g (88 %) of pyrimidine **6** as a colorless crystalline solid, m.p. 152–153 °C (from a heptane/benzene mixture, 4 : 1) readily soluble in CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, and Me<sub>2</sub>CO and slightly soluble in alcohols. Found (%): C, 60.24; H, 5.26; N, 15.80; S, 12.22. C<sub>13</sub>H<sub>13</sub>N<sub>3</sub>OS. Calculated (%): C, 60.21; H, 5.05; N, 16.20; S, 12.36. IR (CH<sub>2</sub>Cl<sub>2</sub>),  $\nu/\text{cm}^{-1}$ : 3490 (NH); 3340 (NH); 1640 (CO); 1585; 1530; 1520. <sup>1</sup>H NMR (CDCl<sub>3</sub>),  $\delta$ : 2.76 (s, 3 H, Me); 2.82 (s, 3 H, Me); 7.25 (br.s, 2 H, NH<sub>2</sub>); 7.42–7.58 (m, 3 H, Ph); 8.43 (m, 2 H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>),  $\delta$ : 14.62 (q, SMe, <sup>1</sup>J = 142 Hz); 33.53 (q, COMe, <sup>1</sup>J = 128 Hz); 109.49 (C(5)); 128.35; 128.87; 131.30; 137.09 (Ph); 161.89 (t, C(2), <sup>3</sup>J = 3.8 Hz); 163.12 (C(6)); 171.66 (q, C(4), <sup>3</sup>J = 3.8 Hz); 199.52 (q, CO, <sup>2</sup>J = 6.1 Hz). MS,  $m/z$  ( $I_{\text{rel}}(\%)$ ): 259 [M]<sup>+</sup> (20), 244 [M-Me]<sup>+</sup> (100), 226 [M-SH]<sup>+</sup> (27).

b. A mixture of pyrimidinethione **2** (0.349 g, 1 mmol) and MeONa (1 mmol) in 7 mL of MeOH was boiled for 1 h. Then the reaction mixture was worked-up as described in procedure a to give 0.21 g (83 %) of pyrimidine **6**.

***N*-(5-Acetyl-6-methylthio-2-phenylpyrimidin-4-yl)-*N,N'*-dimethylformamide (7).** A mixture of pyrimidine 6 (0.33 g, 1.3 mmol) and DMF dimethylacetal (0.25 mL, 1.9 mmol) in 6 mL of benzene was boiled for 3 h. The solvent was evaporated *in vacuo*, and the residue was recrystallized from a 2 : 1 hexane/benzene mixture to give 0.285 g (71 %) of amidine 7, m.p. 136–137 °C. Found (%): C, 61.25; H, 5.87; S, 10.42.  $C_{16}H_{18}N_4OS$ . Calculated (%): C, 61.12; H, 5.77; S, 10.20. IR (KBr),  $\nu/cm^{-1}$ : 1610 (CO); 1510.  $^1H$  NMR ( $CDCl_3$ ),  $\delta$ : 2.64 (s, 3 H, Me); 2.70 (s, 3 H, Me); 3.14 (s, 3 H) and 3.22 (s, 3 H) ( $NMe_2$ ); 7.42–7.55 (m, 3 H, Ph); 8.42–8.55 (m, 2 H, Ph); 8.88 (s, 1 H, CH).

**4-Methylthio-2-phenyl-8*H*-pyrido[2,3-*d*]pyrimidin-5-one (8).** A mixture of amidine 7 (0.314 g, 1 mmol) and MeONa (1 mmol) in 7 mL of MeOH was boiled for 4 h, cooled to 20 °C, acidified with AcOH, and evaporated to dryness. The residue was washed with 15 mL of  $H_2O$ , dried, and washed with 5 mL of  $CHCl_3$  to give 0.24 g (88 %) of compound 8, m.p. 342–345 °C (dec.). The spectral characteristics of the compound obtained coincide with those reported for pyridopyrimidinone 8,<sup>5</sup> synthesized from pyrimidinethione 2 or pyrimidine 3.

**5-Methyl-4-methylthio-2-phenylpyrimido[4,5-*d*]pyrimidine (9).** A mixture of amidine 7 (0.314 g, 1 mmol) and  $AcONH_4$  (0.77 g, 10 mmol) in 10 mL of butanol was boiled for 2 h. The solvent was evaporated *in vacuo* and the residue was chromatographed on a column with  $SiO_2$  (using  $CHCl_3$  as the eluent) to yield 0.166 g (62 %) of pyrimidine 9, m.p. 170–172 °C (dec.), readily soluble in organic solvents except petroleum ether. Found (%): C, 62.49; H, 5.00; N, 21.20; S, 11.37.  $C_{14}H_{12}N_4S$ . Calculated (%): C, 62.66; H, 4.51; N, 20.88; S, 11.95. IR ( $CH_2Cl_2$ ),  $\nu/cm^{-1}$ : 1560; 1540.  $^1H$  NMR ( $CDCl_3$ ),  $\delta$ : 2.86 (s, 3 H, SMe); 3.17 (s, 3 H, Me); 7.45–7.62 (m, 3 H, Ph); 8.70 (m, 2 H, Ph); 9.28 (s, 1 H, H(7)).  $^{13}C$  NMR ( $CDCl_3$ ),  $\delta$ : 15.11 (q, SMe,  $^1J = 143$  Hz); 28.05 (q, Me,  $^1J = 130$  Hz); 115.66 (C(4a)); 128.44; 129.41; 132.07; 136.27 (Ph); 160.32 (d, C(7),  $^1J = 203$  Hz); 162.30 (d, C(8a),  $^3J = 10.8$  Hz); 163.72 (C(2)); 169.45 (q, C(5),  $^2J = 6.3$  Hz, d,  $^3J = 9.1$  Hz); 174.51 (q, C(4),  $^3J = 3.9$  Hz). MS,  $m/z$  ( $I_{rel}(\%)$ ): 268 [ $M$ ]<sup>+</sup> (33), 253 [ $M-Me$ ]<sup>+</sup> (100).

***N*-(5-Acetyl-6-methylthio-2-phenylpyrimidin-4-yl)-*N,N'*-dimethylacetamide (10) and *N*-(5-acetyl-6-methylthio-2-phenylpyrimidin-4-yl)-*O*-methylacetimidate (11).** A mixture of pyrimidine 6 (0.13 g, 0.5 mmol) and DMA dimethylacetal (0.15 mL, 1 mmol) in 4 mL of benzene was boiled for 2 h. The solvent was evaporated *in vacuo*, and the residue was chromatographed on a column with  $SiO_2$  (using a 1 : 1 hexane/benzene mixture as the eluent) to isolate 0.057 g (36 %) of imidate 11, and then (with  $C_6H_6$  and a 1 : 1  $C_6H_6/CHCl_3$  mixture as eluents) 0.09 g (55 %) of amidine 10, m.p. 99–100 °C (from hexane). Found (%): C, 62.58; H, 6.52; N, 16.89; S, 9.03.  $C_{17}H_{20}N_4SO$ . Calculated (%): C, 62.17; H, 6.14; N, 17.06; S, 9.76. IR (KBr),  $\nu/cm^{-1}$ : 1650 (CO); 1580; 1500.  $^1H$  NMR ( $CDCl_3$ ),  $\delta$ : 2.22 (s, 3 H, MeCN); 2.58 (s, 3 H, Me); 2.65 (s, 3 H, Me); 3.15 (s, 6 H,  $NMe_2$ ); 7.37–7.55 (m, 3 H, Ph); 8.39–8.52 (m, 2 H, Ph). MS,  $m/z$  ( $I_{rel}(\%)$ ): 328 [ $M$ ]<sup>+</sup> (89), 313 [ $M-Me$ ]<sup>+</sup> (72), 295 [ $M-SH$ ]<sup>+</sup> (52); 285 [ $M-COMe$ ]<sup>+</sup> (100).

Imidate 11, m.p. 78–79 °C. Found (%): C, 60.84; H, 4.99; N, 13.50; S, 10.62.  $C_{16}H_{17}N_3O_2S$ . Calculated (%): C, 60.93; H, 5.43; N, 13.32; S, 10.17. IR (KBr),  $\nu/cm^{-1}$ : 1670 sh; 1645 (CO); 1520; 1500.  $^1H$  NMR ( $CDCl_3$ ),  $\delta$ : 2.07 (s, 3 H, MeCN); 2.59 (s, 3 H, Me); 2.69 (s, 3 H, Me); 3.88 (s, 3 H, OMe); 7.43–7.58 (m, 3 H, Ph); 8.42–8.52 (m,

2 H, Ph).  $^{13}C$  NMR ( $CDCl_3$ ),  $\delta$ : 13.66 (q, SMe,  $^1J = 140$  Hz); 17.70 (q, Me,  $^1J = 130$  Hz); 31.42 (q, COMe,  $^1J = 128$  Hz); 54.19 (q, OMe,  $^1J = 146$  Hz); 120.85 (C(5)); 128.42; 128.63; 131.18; 137.13 (Ph); 162.73 and 163.32 (C(2) and C(4)); 166.04 (q, Me–C=N,  $^2J = 6.1$  Hz,  $^3J = 3.8$  Hz); 169.53 (q, C(6),  $^3J = 2.5$  Hz); 200.58 (q, CO,  $^2J = 4.0$  Hz). MS,  $m/z$  ( $I_{rel}(\%)$ ): 315 [ $M$ ]<sup>+</sup> (41), 300 [ $M-Me$ ]<sup>+</sup> (100), 282 [ $M-SH$ ]<sup>+</sup> (93).

**7-Methyl-4-methylthio-2-phenyl-8*H*-pyrido[2,3-*d*]pyrimidin-5-one (12).** A mixture of amidine 10 (0.164 g, 0.5 mmol) and MeONa (0.5 mmol) in 5 mL of MeOH was boiled for 4 h. Similarly to compound 8, 0.08 g (56 %) of pyridopyrimidinone 12 was isolated, m.p. 352–356 °C (dec.). The compound is poorly soluble in organic solvents. Found (%): C, 63.03; H, 5.21; N, 14.27; S, 10.88.  $C_{15}H_{13}N_3SO$ . Calculated (%): C, 63.58; H, 4.62; N, 14.83; S, 11.32. IR (KBr),  $\nu/cm^{-1}$ : 3150–2500 (NH, CH); 1660 (CO); 1625; 1595; 1578; 1560; 1528.  $^1H$  NMR ( $DMSO-d_6$ ),  $\delta$ : 2.28 (s, 3 H, Me); 2.52 (s, 3 H, SMe); 6.01 (s, 1 H, H(6)); 7.45–7.67 (m, 3 H, Ph); 8.40–8.58 (m, 2 H, Ph). MS,  $m/z$  ( $I_{rel}(\%)$ ): 283 [ $M$ ]<sup>+</sup> (88), 250 [ $M-SH$ ]<sup>+</sup> (100).

Pyridopyrimidinone 12 (0.07 g, 52 %), whose spectral characteristics correspond to those of the compound prepared from amidine 10, was synthesized from imidate 11 (0.158 g, 0.5 mmol) in a similar way.

**4-Methylene-5-methylthio-3,7-diphenyl-1*H*,3*H*-pyrimido[4,5-*d*]pyrimidin-2-one (14).** A mixture of pyrimidine 6 (0.064 g, 0.25 mmol) and  $PhNCO$  (0.054 mL, 0.5 mmol) in 5 mL of toluene was boiled for 6 h and allowed to stand for 12 h at ~20 °C. The precipitate was filtered off to give 0.054 g (57 %) of 5-acetyl-4-methylthio-2-phenyl-6-(*N*-phenylureido)pyrimidine 13, m.p. 173–174 °C.  $^1H$  NMR ( $CDCl_3$ ),  $\delta$ : 2.80 (s, 3 H, Me); 2.85 (s, 3 H, Me); 7.12 (m, 1 H, Ph); 7.39 (m, 2 H, Ph); 7.51–7.70 (m, 5 H, Ph); 8.36 (m, 2 H, Ph); 10.75 (br.s, 1 H, NH); 11.87 (br.s, 1 H, NH). MeONa (0.25 mmol) in 5 mL of MeOH was added to the adduct 13 synthesized, and the mixture was boiled for 1 h, cooled to ~20 °C, and acidified with 0.2 mL of AcOH. The solvent was evaporated, and 20 mL of  $CHCl_3$  was added to the residue. The precipitate of  $AcONa$  was filtered off, the filtrate was concentrated, and the residue was purified by preparative-scale chromatography on a TLC plate (using  $CHCl_3$  as the eluent) to give 0.038 g (74 %) of colorless crystalline compound 14, m.p. 244–245 °C (from MeCN), readily soluble in  $CHCl_3$ ,  $Me_2CO$ , or EtOH. Found (%): C, 66.90; H, 4.82; N, 15.86; S, 9.10.  $C_{20}H_{16}N_4OS$ . Calculated (%): C, 66.65; H, 4.47; N, 15.55; S, 8.90. IR ( $CH_2Cl_2$ ),  $\nu/cm^{-1}$ : 3410 (NH); 1710 (CO); 1615; 1590; 1570; 1535.  $^1H$  NMR ( $CDCl_3$ ),  $\delta$ : 2.76 (s, 3 H, Me); 4.37 (d, 1 H,  $J = 3.3$  Hz) and 5.36 (d, 1 H,  $J = 3.3$  Hz) ( $CH_2=$ ); 7.25–7.60 (m, 8 H, Ph); 8.02 (br.s, 1 H, NH); 8.41 (m, 2 H, Ph).  $^{13}C$  NMR ( $CDCl_3$ ),  $\delta$ : 14.36 (q, SMe,  $^1J = 142$  Hz); 98.96 (t,  $CH_2=$ ,  $^1J = 162.46$  Hz); 106.71 (C(4a)); 128.47; 128.56; 129.20; 130.01; 131.26; 136.55; 139.22 (2 Ph); 137.60 (m, C(4)); 149.94; 153.72; 166.11 (C(2), C(7), C(8a)), 166.44 (q, C(5),  $^3J = 3.7$  Hz). MS,  $m/z$  ( $I_{rel}(\%)$ ): 360 [ $M$ ]<sup>+</sup> (56), 345 [ $M-Me$ ]<sup>+</sup> (100).

Along with pyrimidopyrimidinone 14, 0.008 g of unidentified compound that contained compound 14 as an impurity was isolated.

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