Macrocyclic compounds containing three pyrimidine fragments

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The reactions of disodium salts of *N*,*N*′-bis(2-mercapto-6-methylpyrimidin-4-yl)alkylenediamines with 1,3-bis(ω-bromoalkyl)-6-methyluracils afforded a series of macrocyclic compounds containing three pyrimidine fragments with different numbers of methylene groups in linking bridges.

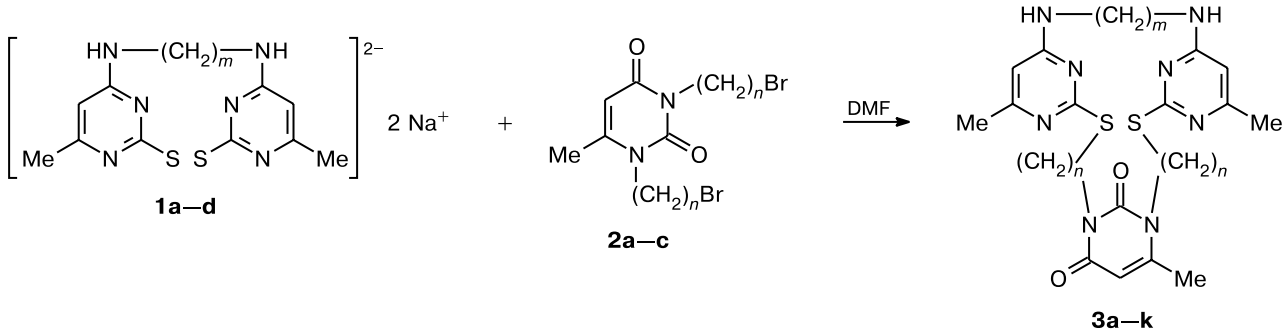
Key words: macrocyclic compounds, pyrimidinophanes.

In most of known pyrimidinophanes,[1] two uracil or 2,4-dithiouracil fragments are linked to each other by hydrocarbon bridges through the N(1) and N(3) atoms or the S atom. In recent years, pyrimidinophanes containing three[2,3] or four uracil fragments[4,5] have been synthesized. However, macrocyclic compounds containing fragments of other pyrimidine derivatives, in particular, of aminopyrimidines, have been much less studied. The reactions of dibromoalkanes with disodium salts of *N*,*N*′-bis(2-mercapto-6-methylpyrimidin-4-yl)alkylenediamines[6] afforded pyrimidinophanes containing two 4-amino-2-mercapto-6-methylpyrimidine fragments. Earlier, we have also reported[7] the synthesis of first macrocyclic compounds containing two 4-amino-2-mercapto-6-methylpyrimidine fragments and one uracil fragment. In the present study, we report the synthesis of a series of pyrimidinophanes with similar structures and describe their selected properties.

The reactions of disodium salts of *N*,*N*′-bis(2-mercapto-6-methylpyrimidin-4-yl)alkylenediamines **1a—d** with 1,3-bis(ω-bromoalkyl)-6-methyluracils **2a—c** in DMF gave rise to pyrimidinophanes **3a—k** (Scheme 1), which were isolated by column chromatography on aluminum oxide.

Pyrimidinophanes **3a—k** occur as crystalline compounds characterized by a broad range of melting points varying from 85 to 246 °C (Table 1). These compounds are soluble in $CHCl_3$. The smaller is the length of the polymethylene bridge $N(CH_2)_mN$, the lower is the solubility of such compounds. The solubility of compounds containing the bridge with the same number of units sharply decreases as the number of methylene groups in

Scheme 1



1a—d **2a—c** **3a—k**

1: *m* = 4 (**a**), 5 (**b**), 6 (**c**), 7 (**d**); **2:** *n* = 4 (**a**), 5 (**b**), 6 (**c**)

3	**a**	**b**	**c**	**d**	**e**	**f**	**g**	**h**	**i**	**j**	**k**
m	4	4	4	5	5	5	6	6	7	7	7
n	4	5	6	4	5	6	4	5	4	5	6

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Table 1. Physicochemical characteristics of compounds **3a—k**[a]

Compound	Yield (%)	M.p. /°C	^{1}H NMR ($CDCl_3$), δ (*J*/Hz)[b,c]
3a	19	225—227	1.74—1.81 (m, 8 H, 4 CH_2); 1.71—1.74 (m, 4 H, 2 CH_2'); 2.24 (s, 3 H, $C(6)_{ur}CH_3$); 2.25, 2.26 (both s, 3 H each, 2 $C(6)_{pyr}CH_3$); 3.04 (m, 4 H, 2 SCH_2); 3.44 (m, 4 H, 2 $NHC\underline{H}_2$); 3.82 (m, 2 H, NCH_2, *J* = 11.0); 3.90 (m, 2 H, NCH_2, *J* = 15.7); 5.20 (br.s, 2 H, 2 NH); 5.58 (s, 1 H, $C(5)_{ur}H$); 5.86, 5.87 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3b	12	193—194	1.51 (m, 4 H, 2 CH_2); 1.63—1.83 (m, 8 H, 4 CH_2); 1.69—1.73 (m, 4 H, 2 CH_2'); 2.22 (s, 3 H, $C(6)_{ur}CH_3$); 2.25, 2.26 (both s, 3 H each, 2 $C(6)_{pyr}CH_3$); 3.02 (m, 2 H, SCH_2, *J* = 15.8); 3.04 (m, 2 H, SCH_2, *J* = 14.1); 3.36 (m, 4 H, 2 $NHC\underline{H}_2$); 3.81 (m, 2 H, NCH_2, *J* = 14.9); 3.93 (m, 2 H, $N_{ur}CH_2$, *J* = 14.9); 5.58 (s, 1 H, $C(5)_{ur}H$); 5.83, 5.84 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3c	11	165—167	1.43—1.46, 1.65—1.73 (both m, 8 H each, 4 CH_2 each); 1.69—1.73 (m, 4 H, 2 CH_2'); 2.23 (s, 3 H, $C(6)_{ur}CH_3$); 2.26 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.03 (m, 4 H, 2 SCH_2); 3.39 (m, 4 H, 2 $NHC\underline{H}_2$); 3.79, 3.90 (both m, 2 H each, NCH_2, *J* = 14.8); 5.06 (br.s, 2 H, 2 NH); 5.57 (s, 1 H, $C(5)_{ur}H$); 5.86 (br.s, 2 H, 2 $C(5)_{pyr}H$)
3d	25	240—242	1.51 (m, 2 H, CH_2'); 1.66 (m, 4 H, 2 CH_2'); 1.80 (m, 8 H, 4 CH_2); 2.24 (s, 3 H, $C(6)_{ur}CH_3$); 2.23 (s, 6 H, 2C $(6)_{pyr}CH_3$); 3.07 (m, 2 H, SCH_2, *J* = 15.4); 3.10 (m, 2 H, SCH_2); 3.40 (m, 4 H, 2 $NHC\underline{H}_2$); 3.82 (br.m, 2 H, $N_{ur}CH_2$, *J* = 10.4); 3.93 (m, 2 H, $N_{ur}CH_2$, *J* = 12.9); 4.92 (br.s, 2 H, 2 NH); 5.56 (s, 1 H, $C(5)_{ur}H$); 5.84, 5.85 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3e	10	195—197	1.38—1.46 (m, 10 H, 2 CH_2, 3 CH_2'); 1.63—1.74 (m, 8 H, 4 CH_2); 2.21 (s, 3 H, $C(6)_{ur}CH_3$); 2.23 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.03, 3.07 (both m, 2 H each, 2 SCH_2); 3.35 (m, 4 H, 2 $NHC\underline{H}_2$); 3.80 (m, 2 H, NCH_2, *J* = 14.5); 3.91 (m, 2 H, NCH_2, *J* = 14.4); 5.53 (br.s, 2 H, 2 NH); 5.56 (s, 1 H, $C(5)_{ur}H$); 5.88 (s, 2 H, 2 $C(5)_{pyr}H$)
3f	9	148—150	1.43—1.49 (m, 14 H, 4 CH_2, 3 CH_2'); 1.71 (m, 8 H, 4 CH_2); 2.23 (s, 3 H, $C(6)_{ur}CH_3$); 2.27 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.03 (m, 2 H, SCH_2, *J* = 13.0); 3.06 (m, 2 H, SCH_2, *J* = 13.9); 3.37 (m, 4 H, 2 $NHC\underline{H}_2$); 3.80 (m, 2 H, NCH_2, *J* = 15.3); 3.91 (m, 2 H, NCH_2, *J* = 14.8); 5.30 (br.s, 2 H, 2 NH); 5.57 (s, 1 H, $C(5)_{ur}H$); 5.89 (s, 2 H, 2 $C(5)_{pyr}H$)
3g	19	208—210	1.42, 1.62 (both m, 4 H each, 2 CH_2' each); 1.79 (m, 8 H, 4 CH_2); 2.22 (s, 3 H, $C(6)_{ur}CH_3$); 2.23, 2.24 (both s, 3 H each, 2 $C(6)_{pyr}CH_3$); 3.07, 3.09 (both m, 2 H each, 2 SCH_2); 3.37 (m, 4 H, 2 $NHC\underline{H}_2$); 3.82, 3.93 (both m, 2 H each, 2 NCH_2); 4.84 (br.s, 2 H, 2 NH); 5.56 (s, 1 H, $C(5)_{ur}H$); 5.82, 5.84 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3h	12	192—193	1.40 (m, 4 H, 2 CH_2); 1.49 (m, 8 H, 4 CH_2'); 1.61—1.78 (m, 8 H, 4 CH_2); 2.21 (s, 3 H, $C(6)_{ur}CH_3$); 2.25 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.03, 3.07 (both m, 2 H each, 2 SCH_2); 3.30 (m, 4 H, 2 $NHC\underline{H}_2$); 3.79, 3.92 (both m, 2 H each, 2 NCH_2); 5.57 (s, 1 H, $C(5)_{ur}H$); 5.83, 5.85 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3i	12	232—234	1.40—1.64 (m, 10 H, 5 CH_2'); 1.63—1.80 (m, 8 H, 4 CH_2); 2.23 (s, 3 H, $C(6)_{ur}CH_3$); 2.25 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.08, 3.10 (both m, 2 H each, 2 SCH_2); 3.34 (m, 4 H, 2 $NHC\underline{H}_2$); 3.83, 3.94 (both m, 2 H each, NCH_2, *J* = 10.3); 4.88 (br.s, 2 H, 2 NH); 5.56 (s, 1 H, $C(5)_{ur}H$); 5.83, 5.84 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3j	10	244—246	1.38—1.65 (m, 14 H, 5 CH_2', 2 CH_2); 1.65—1.78 (m, 8 H, 4 CH_2); 2.22 (s, 3 H, $C(6)_{ur}CH_3$); 2.26 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.04 (m, 2 H, SCH_2, *J* = 15.9); 3.08 (m, 2 H, SCH_2, *J* = 14.8); 3.31 (m, 4 H, 2 $NHC\underline{H}_2$); 3.79 (m, 2 H, NCH_2, *J* = 15.2); 3.92 (m, 2 H, NCH_2, *J* = 14.8); 5.10 (br.s, 2 H, 2 NH); 5.57 (s, 1 H, $C(5)_{ur}H$); 5.83, 5.84 (both s, 1 H each, 2 $C(5)_{pyr}H$)
3k	22	85—87	1.40—1.65 (m, 18 H, 5 CH_2', 4 CH_2); 1.65—1.73 (m, 8 H, 4 CH_2); 2.22 (s, 3 H, $C(6)_{ur}CH_3$); 2.25 (s, 6 H, 2 $C(6)_{pyr}CH_3$); 3.03, 3.06 (both m, 2 H each, SCH_2, *J* = 13.6); 3.32 (m, 4 H, 2 $NHC\underline{H}_2$); 3.79 (m, 2 H, NCH_2, *J* = 15.1); 3.90 (m, 2 H, NCH_2, *J* = 14.6); 4.91 (br.s, 2 H, 2 NH); 5.56 (s, 1 H, $C(5)_{ur}H$); 5.83, 5.84 (both s, 1 H each, 2 $C(5)_{pyr}H$)

[a] The concentrations of the compounds were 1—5 mmol L^{-1}.

[b] The methylene groups in the $NH—(CH_2)_m—NH$ chains are denoted as CH_2'; pyr is the 4-amino-2-mercapto-6-methylpyrimidine fragment, ur is the 6-methyluracil fragment.

[c] Either ranges of chemical shifts or chemical shifts of the centers of the corresponding multiplets and sums of the vicinal coupling constants ($^3J_{AX} + {}^3J_{AX'}$) are given.

the $N(CH_2)_nS$ fragments decreases (maximum concentration of **3a** in chloroform is 1 mmol L^{-1}).

The compositions and structures of compounds **3a—k** were confirmed by high-resolution mass spectrometry and IR and ^{1}H NMR spectroscopic data.

In the high-resolution mass spectra (EI) of compounds **3a—k**, molecular ions peaks are most intense. The main fragmentation ions in the heavy-mass region are formed due to elimination of the Me, MeS, and HS groups from the molecular ion. The mass spectra of all pyrimidino-

phanes have intense peaks of the ions with compositions R^1CH_2SH (*m/z* 154), R^1Me (*m/z* 122), $R^1(CH_2)_2SH$ (*m/z* 168), $R^1C_3H_4SH$ (*m/z* 180), $R^1(CH_2)_3SH$ (*m/z* 182), R^2Me (*m/z* 139), R^2H_3 (*m/z* 127), where $R^1 = C_5H_5N_3$, $R^2 = C_5H_4N_2O_2$. In addition, these compounds are characterized by the formation of the doubly charged ions $[M]^{2+}$.

The IR spectra of compounds **3a—k** (in KBr) have absorption bands at 1655—1660, 1705, and 1710 cm^{-1} (ν(C=O)) and also at 3140—3410 cm^{-1} (ν(N—H)).

The ^1H NMR spectroscopic data for pyrimidinophanes are given in Table 1. The assignment of the signals was made based on their multiplet structures and integral intensities as well as on the 2D ^1H COSY spectrum of compound **3g**, for which the complete assignment of resonances was made. The signals of the CH_2 groups are observed as a spectrum of the AA´ fragment of the AA´XX´ system for the terminal $N_{ur}CH_2$ groups and the AA´ fragment of the MM´AA´XX´ system for the CCH_2C groups. It should be noted that the C(5)H atoms and the Me groups of the 4-amino-2-mercapto-6-methylpyrimidine fragments are nonequivalent (in the spectra of compounds **3a**,**b**,**g**, the signals of the Me groups are observed as three singlets). The chemical shifts of the broadened signals of NH (δ 4.84—5.53) have different values, some spectra having no signals of NH. These characteristic features of the spectra result, apparently, from both the asymmetry of the uracil fragment and intramolecular interactions (in particular, intramolecular hydrogen bonding). The presence of these interactions is supported by the data from IR spectroscopy in solutions.[8]

Experimental

The IR spectra (in KBr) of the compounds synthesized were recorded on a Specord IR-75 spectrophotometer (Carl Zeiss, Jena) and a Vector 22 Fourier spectrometer (Bruker). The ^1H NMR spectra were measured on WM-250 (250.13 MHz, Bruker) and MSL-400 (400.62 MHz) spectrometers at 298 K with Me_4Si as the internal standard. The mass spectra (EI) were obtained on a mass-spectrometic MKh-1310 complex with high-precision processing at V_{ion} = 60 V (direct inlet).

The melting points were measured on a Boetius hot-stage apparatus and were not corrected. Chromatography was carried out in thin films on Silufol-254 plates; visualization was performed with UV light using a 10 : 1 ethyl acetate—triethylamine mixture as the eluent. Column chromatography was carried out on neutral aluminum oxide (Brockmann activity II).

All solvents and reagents used in the study were dehydrated.

Disodium salts of *N*,*N*´-bis(2-mercapto-6-methylpyrimidin-4-yl)alkylenediamines (1a—d) and 1,3-bis(ω-bromoalkyl)-6-methyluracils (2a—c) were synthesized according to known procedures.[6,9]

Synthesis of pyrimidinophanes 3a—k (general procedure). A mixture of disodium salt **1a—d** (0.01 mol) and uracil **2a—c** (0.01 mol) was stirred in DMF (400 mL) at 20 °C for 30 h. The DMF was evaporated *in vacuo*. The residue was chromatographed on a column with Al_2O_3 using successive elution with hexane, diethyl ether, and ethyl acetate. The target compounds **3a—k** were isolated from the fractions obtained with the use of ethyl acetate (see Table 1).

10,19,30-Trimethyl-13,27-dithia-2,7,11,18,22,29,32,33-octaazatetracyclo[26,3,1,$1^{8,12}$,$1^{18,22}$]tetratriaconta-1(32),8(33),9,11,19,28,30-heptaene-21,34-dione (3a), R_f 0.14. Found (%): C, 56.71; H, 6.50; N, 19.72; S, 11.30. $C_{27}H_{38}N_8O_2S_2$. Calculated (%): C, 56.84; H, 6.66; N, 19.64; S, 11.22. Found: *m/z* 570.253 $[M]^+$. $C_{27}H_{38}N_8O_2S_2$. Calculated: M 570.2559.

10,20,32-Trimethyl-13,29-dithia-2,7,11,19,23,31,34,35-octaazatetracyclo[28,3,1,$1^{8,12}$,$1^{19,23}$]hexatriaconta-1(34),8(35),9,11,20,30,32-heptaene-22,36-dione (3b), R_f 0.17. Found (%): C, 58.30; H, 7.10; N, 18.65; S, 10.61. $C_{29}H_{42}N_8O_2S_2$. Calculated (%): C, 58.19; H, 7.02; N, 18.72; S, 10.70. Found: *m/z* 598.295 $[M]^+$. $C_{29}H_{42}N_8O_2S_2$. Calculated: M 598.2872.

10,21,34-Trimethyl-13,31-dithia-2,7,11,20,24,33,36,37-octaazatetracyclo[30,3,1,$1^{8,12}$,$1^{20,24}$]octatriaconta-1(36),8(37),9,11,21,32,34-heptaene-23,38-dione (3c), R_f 0.19. Found (%): C, 59.41; H, 7.40; N, 17.75; S, 10.10. $C_{31}H_{46}N_8O_2S_2$. Calculated (%): C, 59.33; H, 7.33; N, 17.86; S, 10.20. Found: *m/z* 626.315 $[M]^+$. $C_{31}H_{46}N_8O_2S_2$. Calculated: M 626.3185.

11,20,31-Trimethyl-14,28-dithia-2,8,12,19,23,30,33,34-octaazatetracyclo[28,3,1,$1^{9,13}$,$1^{19,23}$]pentatriaconta-1(33),9(34),10,12,20,29,31-heptaene-22,35-dione (3d), R_f 0.21. Found (%): C, 57.32; H, 6.74; N, 19.21; S, 11.03. $C_{28}H_{40}N_8O_2S_2$. Calculated (%): C, 57.51; H, 6.84; N, 19.17; S, 10.95. Found: *m/z* 584.275 $[M]^+$. $C_{28}H_{40}N_8O_2S_2$. Calculated: M 584.2716.

11,22,33-Trimethyl-14,30-dithia-2,8,12,20,24,32,35,36-octaazatetracyclo[29,3,1,$1^{9,13}$,$1^{20,24}$]heptatriaconta-1(35),9(36),10,12,21,31,33-heptaene-23,37-dione (3e), R_f 0.23. Found (%): C, 59.02; H, 7.10; N, 18.22; S, 10.38. $C_{30}H_{44}N_8O_2S_2$. Calculated (%): C, 58.82; H, 7.18; N, 18.30; S, 10.45. Found: *m/z* 612.3059 $[M]^+$. $C_{30}H_{44}N_8O_2S_2$. Calculated: M 612.3029.

11,22,35-Trimethyl-14,32-dithia-2,8,12,21,25,34,37,38-octaazatetracyclo[31,3,1,$1^{9,13}$,$1^{21,25}$]nonatriaconta-1(37),9(38),10,12,22,33,35-heptaene-24,39-dione (3f), R_f 0.30. Found (%): C, 59.91; H, 7.46; N, 17.41; S, 9.89. $C_{32}H_{48}N_8O_2S_2$. Calculated (%): C, 60.00; H, 7.50; N, 17.50; S, 10.00. Found: *m/z* 640.332 $[M]^+$. $C_{32}H_{48}N_8O_2S_2$. Calculated: M 640.3342.

12,21,32-Trimethyl-15,29-dithia-2,9,13,20,24,31,34,35-octaazatetracyclo[28,3,1,$1^{10,14}$,$1^{20,24}$]hexatriaconta-1(34),10(35),11,13,21,30,32-heptaene-23,36-dione (3g), R_f 0.33. Found (%): C, 58.26; H, 7.05; N, 18.68; S, 10.59. $C_{29}H_{42}N_8O_2S_2$. Calculated (%): C, 58.19; H, 7.02; N, 18.72; S, 10.70. Found: *m/z* 598.286 $[M]^+$. $C_{29}H_{42}N_8O_2S_2$. Calculated: M 598.2872.

12,22,34-Trimethyl-15,31-dithia-2,9,13,21,25,33,36,37-octaazatetracyclo[30,3,1,$1^{10,14}$,$1^{21,25}$]octatriaconta-1(36),10(37),11,13,22,32,34-heptaene-24,38-dione (3h), R_f 0.28. Found (%): C, 59.44; H, 7.29; N, 17.79; S, 10.25. $C_{31}H_{46}N_8O_2S_2$. Calculated (%): C, 59.33; H, 7.33; N, 17.86; S, 10.20. Found: *m/z* 626.319 $[M]^+$. $C_{31}H_{46}N_8O_2S_2$. Calculated: M 626.3185.

12,23,36-Trimethyl-15,33-dithia-2,9,13,22,26,35,38,39-octaazatetracyclo[32,3,1,$1^{10,14}$,$1^{22,26}$]tetraconta-

1(38),10(39),11,13,23,34,36-heptaene-25,40-dione (3i), R_f 0.26. Found (%): C, 58.94; H, 7.16; N, 18.20; S, 10.41. $C_{30}H_{44}N_8O_2S_2$. Calculated (%): C, 58.82; H, 7.18; N, 18.30; S, 10.45. Found: *m/z* 612.302 $[M]^+$. $C_{30}H_{44}N_8O_2S_2$. Calculated: M 612.3029.

13,23,35-Trimethyl-16,32-dithia-2,10,14,22,26,34,37,38-octaazatetracyclo[31,3,1,$1^{11,15}$,$1^{22,26}$]nonatriaconta-1(37),11(38),12,14,23,33,35-heptaene-25,39-dione (3j), R_f 0.21. Found (%): C, 60.20; H, 7.56; N, 17.60; S, 9.95. $C_{32}H_{48}N_8O_2S_2$. Calculated (%): C, 60.00; H, 7.50; N, 17.50; S, 10.00. Found: *m/z* 640.3319 $[M]^+$. $C_{32}H_{48}N_8O_2S_2$. Calculated: M 640.3341.

13,24,37-Trimethyl-16,34-dithia-2,10,14,23,27,36,39,40-octaazatetracyclo[33,3,1,$1^{11,15}$,$1^{23,27}$]hentetraconta-1(39),11(40),12,14,24,35,37-heptaene-26,41-dione (3k), R_f 0.23. Found (%): C, 61.19; H, 7.85; N, 16.69; S, 10.08. $C_{34}H_{52}N_8O_2S_2$. Calculated (%): C, 61.07; H, 7.78; N, 16.76; S, 9.58. Found: *m/z* 668.365 $[M]^+$. $C_{34}H_{52}N_8O_2S_2$. Calculated: M 668.3654.

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