Synthesis and Crystal Structure Characterization of two new Antibacterial Agents: 5-[1-(2,3- and 2,4-Dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidines

H. Allouchi [a], Y. Fellahi [b], C. Hébert [b], C. Courseille [c] and Y. Frangin [b]

[a] Laboratoire de Chimie Physique - PIMIR E.A. 2098, Faculté des Sciences Pharmaceutiques, 31 avenue Monge, 37200 Tours, France.
[b] Département de Chimie, Faculté des Sciences et Techniques, Parc de Grandmont, Université François Rabelais, 37200 Tours, France
[c] Unité de Biophysique Structurale, UMR 5471-CNRS, Avenue des Facultés, Bâtiment de Biologie Végétale, 33405 Talence, France.
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The synthesis of 5-[1-(2,3-dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4a**) and 5-[1-(2,4-dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4b**) is described. These compounds were prepared by chlorination of the corresponding 5-substituted barbituric acids obtained by treatment of the 5-benzylidenebarbituric acids with benzylzinc bromide in the preceding step. These new trichloropyrimidines belong to a series of pyrimidine derivatives which show *in vitro* antibacterial activity against the undesirable human bacterial flora of the axilla and foot. The characterization of these compounds were performed by spectroscopy and X-ray structure determination. Compounds **4a** and **4b** crystallize in the monoclinic system. In the crystal of **4a** there are two conformers A and B both of which are the same type of conformation, and are different from that found in the structure of **4b**. The crystal cohesion results from numerous very week Van der Waals interactions.

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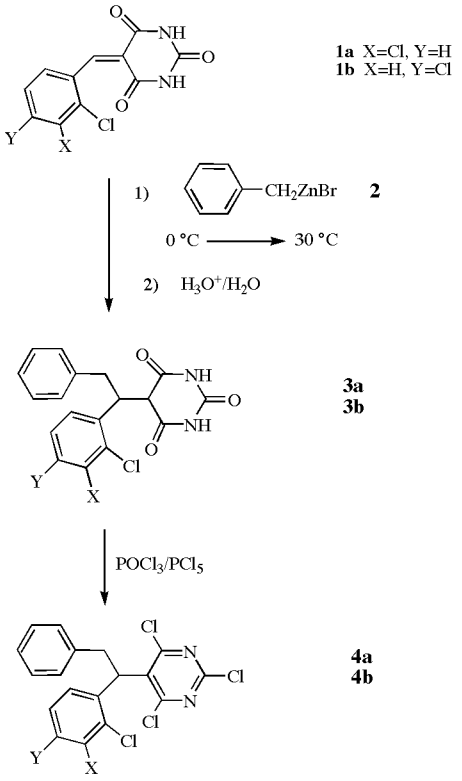
The synthesis of pyrimidines has attracted the attention of chemists because of their potential biological properties. Over the last few years there has been an increased interest for pyrimidine derivatives having antibacterial activity. In the scope of our research, we have described in a previous report the synthesis and the antibacterial activity of 5-(1,2-diarylethyl)-2,4,6-trichloropyrimidines [1]. Among these compounds, 5-[1-(4-chlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine was the most active against the undesirable human bacteria of both the foot and the axilla. In continuation with our research work, 5-[1-(2,3-dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4a**) and 5-[1-(2,4-dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4b**) were synthesized according to the previously described method [1,2], with the aim to study their crystal structures and their potential antibacterial activity. Compounds **4a** and **4b** were characterized by the usual spectroscopic techniques and by X-ray structure analysis.

The synthetic method for preparing compounds **4a** and **4b** is shown in Scheme 1. The precursors of trichloropyrimidines **4a** and **4b** were barbituric acids **3a** and **3b** respectively. Compounds **3a** and **3b** were obtained following 1,4-addition of benzylzinc bromide (**2**) to the corresponding benzylidenebarbituric acids **1a** and **1b**. Products **1a** and **1b** were easily prepared by condensing barbituric acid with 2,3-dichlorobenzaldehyde and 2,4- dichlorobenzaldehyde according to a method described with benzaldehyde [3]. In our previous paper [1], we proved that the organozinc reagent **2** also undergoes hydrogen-metal exchange with both NH sites of the benzylidenebarbituric acids. The reaction therefore only led to substantial yields of product **3a** or **3b** if three molecular equivalents of organozinc reagent **2** were used. Compounds **3a** and **3b** were purified by sodium hydroxide treatment before use in trichloropyrimidine synthesis. The reaction yielded 70 %

Scheme 1



Synthesis of compounds **4a** and **4b**.

of barbituric acid **3a** and 81 % of barbituric acid **3b**. The reaction of the mixture of phosphorus oxychloride and phosphorus pentachloride with barbituric acids **3a** and **3b** according to the procedure of Gershon [4], gave the corresponding 2,4,6-trichloropyrimidines **4a** and **4b**, which were purified first by sodium hydroxide treatment to remove the residual starting material **3a** or **3b**. The purity was checked on the basis of their elution profile in a capillary gas chromatography procedure. Gas chromatography was coupled with a mass spectrometer to analyse compounds **4a** and **4b**. Synthesis yielded 52 % of compound **4a** and 64 % of compound **4b**.

^{1}H nmr analysis at 200MHz of 5-substitutedbarbituric acids displayed for **3a** two singlets (7.97 and 8.17 ppm) and for **3b** two singlets (8.33 and 8.55 ppm) assigned to the two NH protons. The tertiary proton CH of the ethyl chain gave a multiplet at 4.72 ppm for **3a** and at 4.54 ppm for **3b**. As expected, the characteristic signals of the barbituric acids **3** were not observed in the ^{1}H nmr spectra of the corresponding trichloropyrimidines **4**. Moreover, the ^{1}H nmr signals of the ethyl chain was modified. In particular, the tertiary proton exhibited a triplet for **4a** (5.10 ppm with a coupling constant ^{3}J = 8.15 Hz) and for **4b** (5.17 ppm with a coupling constant ^{3}J = 8.2 Hz) while the two protons of the methylene group gave a doublet for **4a** (3.48 ppm with the coupling constant ^{3}J = 8.15 Hz) and for **4b** (3.59 ppm with a coupling constant ^{3}J = 8.2 Hz). The downfieldshift of the tertiary proton resonance for trichloropyrimidines **4** com-

Table 1

Crystal Data, Data Collection and Structure Refinement for Compounds **4a** and **4b**

	Compound **4a**	Compound **4b**
Crystal Data		
Formula	$C_{18}H_{11}Cl_5N_2$	$C_{18}H_{11}Cl_5N_2$
Molecular weight (g.mol^{-1})	432.54	432.54
Temperature (K)	298	298
Wavelength (Å)	1.54178	1.54178
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/n$
Z	8	4
Unit cell dimensions		
a (Å)	15.702(1)	11.910(2)
b (Å)	13.276(2)	13.065(1)
c (Å)	17.726(2)	12.985(2)
β (°)	92.58(1)	110.91(1)
Volume of cell(Å^3)	3691.3(7)	1886.4(4)
Calculated density (g.cm^{-3})	1.557	1.523
F(000)	1744	872
Absorption coefficient (mm^{-1})	7.189	7.035
Data Collection		
Scan mode	ω-2θ	ω-2θ
Absorption correction mode	ψ scan	ψ scan
Limiting indices		
h_{min}/h_{max}	0/17	0/13
k_{min}/k_{max}	0/14	0/12
l_{min}/l_{max}	-19/19	-12/12
Reflections collected	4675	1894
Limit of collection θ_{max} (°)	60	60
Refinement		
Refinement method	On F^2	On F
Parameters	453	227
Final R indices [I>2σ(I)]	R = 0.0574	R = 0.0253
	wR = 0.1315	wR = 0.0653
Goodness-of-fit	1.153	1.092
Weighting factor	$1/[\sigma^2(F_o^2)+(0.0130P)^2+8.1397P]$	$1/[\sigma^2(F_o^2)+(0.0271P)^2+1.1801P]$
	where $P=(F_o^2+2F_c^2)/3$	where $P=(F_o^2+2F_c^2)/3$
Extinction coefficient	8.4(7)	0.0031(2)
Δr_{min} / Δr_{max} (e.Å^{-3})	-0.407 / 0.493	-0.152 / 0.147
$(\Delta/r)_{max}$	0.029	<0.001

pared to their barbituric acid precursors **3** characterized the out-magnetic anisotropy effect of the pyrimidine ring of the compounds **4**. The mass spectrum (electronic ionization at 70 eV) of compounds **4a** and **4b** gave five molecular peaks at m/z 430, 432, 434, 436 and 438 due to the presence of the isotopes 35 and 37 of chlorine in the molecular formula $C_{18}H_{11}Cl_5N_2$. For the same reason, the fragment $(C_{11}H_4Cl_5N_2)^+$ resulting from loss of the benzyl group was assigned to four peaks at m/z 339, 341, 343 and 345.

For crystal structure analysis, the crystal data, the data collection conditions and the refinement last indexes are given in Table 1.

The molecular conformation, atomic labeling of non-hydrogen atoms and ellipsoid representation [6] are shown in Figure 1 for the two conformers A and B of **4a** (there are two independent molecules per asymmetric unit in the

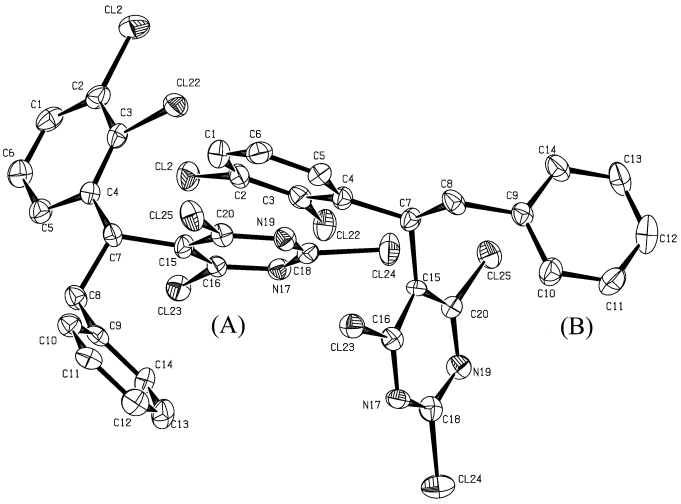


Figure 1. ORTEP View of compound **4a**: conformers **A** and **B**. Thermal ellipsoids are drawn at the 20% probability level.

crystal structure of **4a**) an Figure 2 for **4b**. The thermal ellipsoid representation of both conformers A and B for **4a** does not show any significant deformation of the electronic distribution. The C-C, C-N and C-Cl bond lengths in the three aromatic rings are close to 1.40(1), 1.32(1) and 1.73(1) Å for **4a** and 1.377(4), 1.331(4), 1.734(3) Å for **4b**.

The three aromatic rings (atoms C1 to C6), (atoms C9 to C14) and (atoms C15, C16, N17, C18, N19 and C20) are found roughly planar for **4a** and perfectly planar for **4b** with chlorine atoms slightly out of the mean planes.

The molecular conformations of the positional isomers **4a** and **4b** are perfectly described by the torsion angles around the C4-C7, C7-C8, C8-C9 and C7-C15 bonds (see Table 2). The crystalline conformations are only very weakly perturbed by their molecular environment and should be very close to that found in the liquid phase

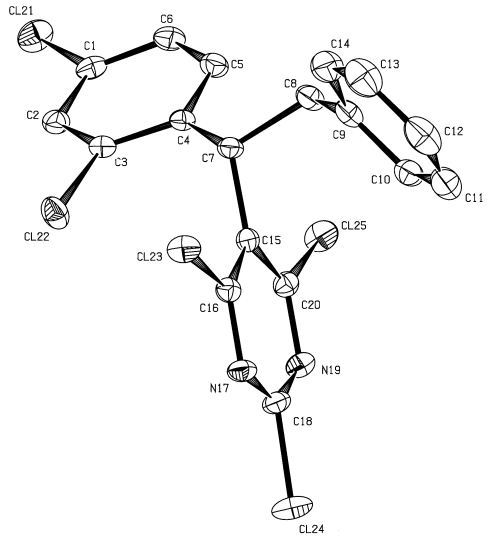


Figure 2. ORTEP View of compound **4b**. Thermal ellipsoids are drawn at the 20% probability level.

Table 2

Significative Torsion Angles (°) for Compounds **4a** and **4b**

	Compound **4a**		Compound **4b**
	Conf. **A**	Conf. **B**	
C3-C4-C7-C8	*trans* [a]	166.3(8)	-167.4(3)
C4-C7-C8-C9	*trans*	*trans*	168.8(3)
C4-C7-C15-C16	-50.4(12)	-55.0(11)	-128.0(3)
C7-C8-C9-C10	87.7(10)	-91.5(11)	92.4(3)

[a] Differing by less than 10° from the *trans* conformation.

because the Van der Waals intermolecular interactions are very weak in the crystals.

Preliminary pharmacological results were quite different since compounds **4a** and **4b** demonstrated a better antibacterial activity than 5-[1-(4-chlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine [7].

In conclusion, we synthesized two new pyrimidine derivatives, 5-[1-(2,3-dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4a**) and 5-[1-(2,4-dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4b**), according to a method used for preparing the 5-[1-(4-chlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine. These compounds were well characterized by spectroscopic analysis and X-ray structure determination. Moreover, the analytical results allowed the structural comparison since these compounds differ by the chlorine atoms on a phenyl ring. It appears that this difference improves the antibacterial properties with the new pyrimidine derivatives **4a** and **4b.** These preliminary satisfactory results should encourage the continued development this series of trichloropyrimidines.

EXPERIMENTAL

2,3-Dichlorobenzaldehyde, 2,4-dichlorobenzaldehyde, barbituric acid, benzyl bromide, zinc, phosphorus oxychloride and phosphorus pentachloride were purchased from Acros organics. All reagents were used without purification and all solvents were usually redistilled and dried. ^{1}H nmr spectra were recorded at 200 MHz and ^{13}C nmr spectra were recorded at 50 MHz on a Brüker Avance DPX 200 instrument. Chemical shifts were measured as δ units (ppm) relative to tetramethylsilane. Infrared spectra were recorded from 4000 to 600 cm^{-1} on a Perkin-Elmer ftir Paragon 1000 spectrometer. Samples for ir studies were prepared as fused potassium bromide disc. Ultraviolet spectra were performed on a Secomam Anthelie spectrometer using ethanol as solvent. Mass spectra were obtained on a Hewlett Packard HP 5989A spectrometer. The purity of the synthesized compounds was verified by gas chromatography (gc, HP 5890A, II) coupled with a mass spectrometer. A 25 m x 0.2 mm fused silica capillary column OVI (HP1) Hewlett Packard was directly inserted into the ion source of the HP quadrupole mass spectrometer through a heated (250°C) interface box. Helium was used as carrier gas, with a flow rate through the column of 0.7 ml/min. The temperature remained at 70°C for 1 min and was then programmed up to 300 °C at 10 °C/min. The final time was 60 min. The temperature of the ion source was 200 °C and the energy of bombarding electrons was 70 eV. Melting points were determined with a Kofler hot stage apparatus and are uncorrected. The results of elemental analyses (C, H, N, Cl) were within ±0.4% of theoretical values. Microanalyses were carried out at the service central d'analyse CNRS, Vernaison, France.

X-ray Structure Determination.

The X-ray diffraction study allows us to obtain the spatial conformation of the compounds **4a** and **4b**. The crystals of these compounds were obtained by slow evaporation of solution in ethanol and choloroform mixtures. The crystals are colourless prisms. Intensity data were collected on an Enraf-Nonius CAD-4 diffractometer equipped using monochromated Cu(kα) radiation at room temperature (298 K) with an ω-2θ scan. The structure was solved by direct methods *SHELXS-97* [8] and refined with *SHELXL-97* [8]. Scattering factors were taken from International Tables for Crystallography [9]. The hydrogen atoms were introduced in their theoretical positions and allowed to ride with the atoms to which they are attached. The crystal data, the data collection conditions and the structure refinement, are given in Table 1. Figures were prepared from *PLATON-99* [6].

General Procedure for Preparation of 5-Benzylidenebarbituric Acids (**1**).

They were obtained according to a described method [3] by condensing the dichlorobenzaldehyde (8.75 g, 50 mmol) with barbituric acid (6.4 g, 50 mmol) dissolved in hot water (60 mL).

5-(2,3-Dichlorobenzylidene)barbituric Acid (**1a**).

The reaction was performed with 2,3-dichlorobenzaldehyde to yield compound **1a** as a yellow solid, 12.04 g (85 %), mp 253-255 °C; ir (potassium bromide): ν 3252 (NH), 3102, 2806, 1770, 1745, 1729, 1714, 1694, 1684 (C=O), 1614, 1571, 1555, 1520, 1500 (aromatic ring), 1428, 1372, 1319, 1272, 1258, 1214, 1185, 1050, 948, 916, 787, 754, 721 cm^{-1}; uv (ethanol) λmax 281 nm (ε 3,800), 314 nm (ε 5,500); ^{1}H nmr (dimethyl-d$_6$ sulfoxide): δ 7.41 (t, 1H, J = 7.8 Hz, ArH), 7.60 (dd, 1H, J = 7.8 Hz, J = 1.6 Hz, ArH), 7.72 (dd, 1H, J = 7.8 Hz, J = 1.6 Hz, ArH), 8.26 (s, 1H, ArCH), 11.32 (s, 1H, NH), 11.54 (s, 1H, NH).

5-(2,4-Dichlorobenzylidene)barbituric Acid (**1b**).

The reaction was performed with 2,4-dichlorobenzaldehyde to yield compound **1b** as a yellow solid, 12.79 g (90 %); ir (potassium bromide) : ν 3200 (NH), 3072, 1760, 1710, 1692 (C=O), 1590, 1577 (aromatic ring), 1438, 1380, 1326, 1260, 1223, 1176, 1107, 1050, 945, 838, 791, 757, 670 cm^{-1}; uv (ethanol) λmax 204 nm (ε 12,500), 259 nm (ε 7,300), 326 nm (ε 500); ^{1}H nmr (dimethyl-d$_6$ sulfoxide): δ 7.50 (dd, 1H, J = 8.5 Hz, J = 2 Hz, ArH), 7.76-7.80 (m, 2H, ArH), 8.23 (s, 1H, ArCH), 11.32 (s, 1H, NH), 11.53 (s, 1H, NH).

Benzylzinc Bromide (**2**).

According to a procedure described by Gaudemar [5] benzylzinc bromide (**2**) was obtained by reaction between benzyl bromide (14.36 g, 84 mmol) and zinc (5.5 g, 84 mmol) in dry THF (40 mL), blanketed under nitrogen gas at 25-30 °C. The organozinc reagent **2** was used *in situ* for the synthesis of compounds **3a** and **3b**.

General Procedure for Preparation of 5-[1-(Dichlorophenyl)-2-phenylethyl]barbituric Acids (**3**).

According to a previously described method [1,2], a solution of the benzylzinc bromide (**2**) (84 mmol) in THF was cooled at 0 °C and the 5-benzylidenebarbituric acid (**1a**) or (**1b**) (5.46 g, 19 mmol) was added with stirring and cooling. The temperature of the mixture quickly rose to 30 °C. When it began to fall the cooling bath was removed. After stirring at room temperature for 1 h, the mixture was hydrolysed with crushed ice (60 g) and concentrated hydrochloric acid (10 mL); ether (60 mL) was then added. The two phases were separated and the aqueous layer was extracted with ether (3 x 40 mL). The combined organic phase was washed with brine (100 mL), dried with anhydrous sodium sulfate and evaporated to give the crude solid product **3**. For purification, the crude product was dissolved in aqueous 0.5 *N* sodium hydroxide (240 mL). The aqueous layer was washed with ether (3 x 40 mL). Strong hydrochloric acid (15 mL) was then added to precipitate product **3**. This product was dissolved in ether (120 mL), washed with brine (4 x 25 mL) and dried with anhydrous sodium sulfate. The solvent was removed under vacuum.

5-[1-(2,3-Dichlorophenyl)-2-phenylethyl]barbituric Acid (**3a**).

The reaction of benzylzinc bromide (**2**) with 5-(2,3-dichlorobenzylidene)barbituric acid (**1a**) yielded compound **3a** as a colorless solid, 5.02 g (70 %), mp 105-107 °C; ir (potassium bromide): ν 3218 (NH), 3092, 2847, 1730, 1715, 1694 (C=O), 1612, 1592, 1582, 1496 (aromatic rings), 1353, 1228, 1180, 1104, 786, 700 cm^{-1}; uv (ethanol): λmax 278 nm (ε 4,200); ^{1}H nmr (deuteriochloroform): δ 3.13 (dd, 1H, J = 14.0 Hz, J = 6.7 Hz, ArC*H*H), 3.32 (dd, 1H, J = 14.0 Hz, J = 9.8 Hz, ArCH*H*), 3.62 (d, 1H, J = 3.0 Hz, COCHCO), 4.72 (m, 1H, ArCH), 7.13 - 7.41 (m, 8H, ArH), 7.97 (s, 1H, NH), 8.17 (s, 1H, NH).

5-[1-(2,4-Dichlorophenyl)-2-phenylethyl]barbituric Acid (**3b**).

The reaction of benzylzinc bromide (**2**) with 5-(2,4-dichlorobenzylidene)barbituric acid (**1b**) yielded compound **3b** as a colorless solid, 5.77 g (81 %), mp 88-90 °C; ir (potassium bromide): ν 3218 (NH), 3100, 2860, 1730, 1710, 1694 (C=O), 1590, 1550,

1490 (aromatic rings), 1470, 1410, 1360, 1105, 819, 700 cm^{-1}; uv (ethanol): λmax 220 nm (ε 28,000), 265 nm (ε 7,200); ^1H nmr (deuteriochloroform): δ 3.05 (dd, 1H, J = 13.7 Hz, J = 7.4 Hz, ArC*H*H), 3.24 (dd, 1H, J = 13.7 Hz, J = 9.6 Hz, ArCH*H*), 3.54 (d, 1H, J = 2.8 Hz, COCHCO), 4.54 (m, 1H, ArCH), 7.24 (m, 6H, ArH), 7.31 (d, 1H, J = 2.2 Hz, ArH), 7.37 (d, 1H, J = 8.5 Hz, ArH), 8.33 (s, 1H, NH), 8.55 (s, 1H, NH).

General Procedure for Preparation of 5-[1-(Dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidines (**4**).

According to a previously described method [4], a mixture of barbituric acid **3** (3.77 g, 10 mmol) and phosphorus oxychloride (3.06 g, 20 mmol) was heated under reflux (105 °C) overnight. After cooling to room temperature, phosphorus pentachloride (6.24 g, 30 mmol) was added. Refluxing was then continued overnight. After cooling, the reaction mixture was poured onto ice and allowed to stand 30 min. Product **4** was extracted with ether (3 x 40 mL), decolorized with charcoal and then filtered. The organic layer was treated by excess of 2 *N* sodium hydroxyde (20 mL, 40 mmol) and washed with brine until neutral. The organic phase was then dried with anhydrous sodium sulfate and the solvent was removed. The residue was purified by recrystallization.

5-[1-(2,3-Dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4a**).

The reaction with barbituric acid **3a** yielded compound **4a** which was crystallized from ethanol-chloroform as colorless prisms, 2.24 g (52 %), mp 61-63 °C; ir (potassium bromide): ν 3041, 3029, 2929, 2867, 1929, 1883, 1638, 1592, 1561, 1528, 1496 (aromatic rings), 1458, 1449, 1408, 1328, 1269, 1208, 1158, 1077, 1048, 954, 862, 808, 776, 752, 700 cm^{-1}; uv (ethanol): λmax 283 nm (ε 2,800), 300 nm (ε 1,400), 311 nm (ε 1,100); ^1H nmr (deuteriochloroform): δ 3.48 (d, 2H, J = 8.15 Hz, $ArCH_2$), 5.10 (t, 1H, J = 8.15 Hz, ArCH), 7.00 (m, 2H, ArH), 7.15 (m, 4H, ArH), 7.36 (d, 1H, J = 8.0 Hz, ArH), 7.55 (d, 1H, J = 7.8 Hz, ArH); ^{13}C nmr (deuteriochloroform): δ 35.4 (CH_2), 45.2 (CH), 127.1 (arom. CH), 128.7 (arom. CH), 128.9 (arom. CH), 132.5 (arom. C), 133.2 (arom. C), 137.4 (arom. C), 137.6 (arom. C), 157.2 (C-Cl), 163.4 (C-Cl); gc/ms: Rt = 35.4 min; ms: m/z 438 $[M + 8]^+$ (0.1), 436 $[M + 6]^+$ (1.4), 434 $[M + 4]^+$ (4.1), 432 $[M + 2]^+$ (6.1), 430 M^+ (3.6), 345 $[C_{11}H_4N_2{}^{35}Cl_2{}^{37}Cl_3]^+$ (1.1), 343 $[C_{11}H_4N_2{}^{35}Cl_3{}^{37}Cl_2]^+$ (2.8), 341 $[C_{11}H_4N_2{}^{35}Cl_4{}^{37}Cl]^+$ (5.0), 339 $[C_{11}H_4N_2{}^{35}Cl_5]^+$ (3.4), 305 (2.1), 269 (3.6), 208 (3.9), 178 (1.7), 138 (1.4), 92 (44.3), 91 $[C_7H_7]^+$ (100), 65 (46.4).

Anal. Calcd. for $C_{18}H_{11}Cl_5N_2$: C, 49.98; H, 2.56; N, 6.47; Cl, 40.98. Found: C, 50.20; H, 2.63; N, 6.33; Cl, 41.25.

5-[1-(2,4-Dichlorophenyl)-2-phenylethyl]-2,4,6-trichloropyrimidine (**4b**).

The reaction with barbituric acid **3b** yielded compound **4b** which was crystallized from ethanol-chloroform as colorless prisms, 2.76 g (64 %), mp 76-78 °C; ir (potassium bromide): ν 3045, 3026, 2908, 2847, 1934, 1852, 1653, 1592, 1582, 1556, 1529, 1496 (aromatic rings), 1469, 1444, 1372, 1329, 1275, 1214, 1103, 1050, 1014, 945, 882, 820, 754, 699 cm^{-1}; uv (ethanol): λmax 283nm (ε 3,000), 300 nm (ε 1,700), 311 nm (ε 1,400); ^1H nmr (deuteriochloroform): δ 3.59 (d, 2H, J = 8.2 Hz, $ArCH_2$), 5.17 (t, 1H, J = 8.2 Hz, ArCH), 7.10-7.15 (m, 7H, ArH), 7.68 (d, 1H, J = 8.4 Hz, ArH); ^{13}C nmr (deuteriochloroform): δ 36.7 (CH_2), 44.2 (CH), 127.2 (arom. CH), 127.8 (arom. CH), 129.3 (arom. CH), 129.7 (arom. CH), 130.4 (arom. CH), 131.4 (arom. C), 132.4 (arom. CH), 134.7 (arom. C), 135.5 (arom. C), 135.6 (arom. C), 137.3 (arom. C), 157.6 (C-Cl), 163.9 (C-Cl); gc/ms: Rt = 33.6 min; ms: m/z 438 $[M + 8]^+$ (0.1), 436 $[M + 6]^+$ (1.4), 434 $[M + 4]^+$ (3.7), 432 $[M + 2]^+$ (5.4), 430 M^+ (3.6), 345 $[C_{11}H_4N_2{}^{35}Cl_2{}^{37}Cl_3]^+$ (0.8), 343 $[C_{11}H_4N_2{}^{35}Cl_3{}^{37}Cl_2]^+$ (2.8), 341 $[C_{11}H_4N_2{}^{35}Cl_4{}^{37}Cl]^+$ (5.0), 339 $[C_{11}H_4N_2{}^{35}Cl_5]^+$ (3.3), 305 (3.2), 269 (2.8), 208 (3.6), 178 (1.4), 138 (1.4), 92 (33.6), 91 $[C_7H_7]^+$ (100), 65 (37.8).

Anal. Calcd. for $C_{18}H_{11}Cl_5N_2$: C, 49.98;H, 2.56; N, 6.47; Cl, 40.98. Found: C, 50.19; H, 2.81; N, 6.60; Cl, 41.29.

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