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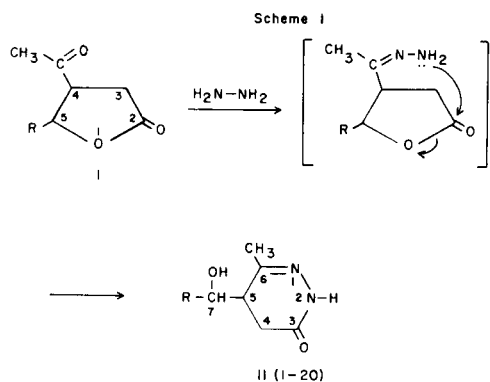
Received march 28, 1983

Reaction of hydrazine hydrate with 4,5-dihydro-3*H*-4-acetyl furan-2-ones **I** led to ring opening and rearrangement into 6-methyl-4,5-dihydro-2*H*-pyridazin-3-ones **II** (**1-20**), the structure of which was determined by spectroscopic methods (ir, <sup>1</sup>H and <sup>13</sup>C nmr). An X-ray crystal structure study of compound **2** supports the assignment of configuration *erythro* (*5RS*, *7SR*) for pyridazinones **II**. Stereochemical courses of this new synthetic route are discussed.

*J. Heterocyclic Chem.*, **21**, 305 (1984).

Pyridazine compounds have been reported to exhibit various pharmacological properties, especially in the CNS and cardiovascular areas [1-8]. Thus new pyridazine derivatives are of considerable interest in medicinal chemistry.

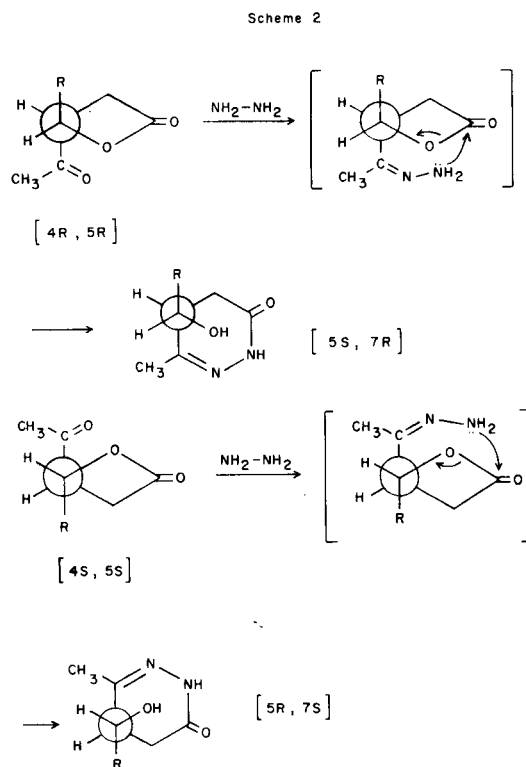
In the course of our studies on 3-acetyl-4-butanolides [9-11], our attention was focused on the reactivity of these compounds with nucleophilic agents. Treatment of dihydrofuranones **I** by hydrazine hydrate afforded a series of new 5-substituted 6-methyl-4,5-dihydro-2*H*-pyridazin-3-ones **II**, according to Scheme 1.



Analytical and spectral data of compounds **II** (**1** to **20**) are summarized in Tables 1 and 2.

An additional structural evidence is brought out by off resonance decoupled <sup>13</sup>C nmr spectra. The singlet at about  $\delta = 151$  ppm was assigned to C-6; the doublet at about  $\delta = 44$  ppm was attributed to C-5, while the triplet at  $\delta = 27$  ppm was assigned to C-4.

In the structure of these pyridazinones, there are two asymmetric centers, carbons 5 and 7, which can be related respectively to the carbons 4 and 5 of the starting furanones **I**. Yet only one of the two likely diastereoisomers was evidenced both by nmr spectra, and chromatographs (tlc,

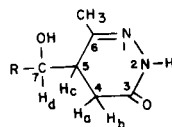


hplc). On the basis of X-ray analysis, configuration *erythro* (*5RS*, *7SR*) could be assigned to pyridazinones **II**. This statement provided support for structural evidence of furanones **I**, which also had been obtained in a stereoselective manner [11]. Configuration of furanones very likely was retained through conversion to pyridazinones; so only lactones *trans* (*4RS*, *5RS*) were able to provide *erythro* pyridazinones (Scheme 2).

## EXPERIMENTAL

Melting points were determined with a Reichert microscope apparatus and are uncorrected. Infrared spectra were recorded on a Beckman 4240 spectrophotometer as potassium bromide pellets. The <sup>1</sup>H and <sup>13</sup>C nmr

Table 1  
5-Substituted-6-methyl-4,5-dihydro-2H-pyridazin-3-ones



Compound No.	R	% Yield	Mp °C	Empirical Formula	Molecular Weight	Elemental Analysis %					S	
						Calcd.	(Found)	C	H	N		Cl
1	Phenyl	74	95	C <sub>12</sub> H <sub>14</sub> O <sub>2</sub> N <sub>2</sub>	218	66.05 (66.12)	6.42 (6.45)	12.84 (12.65)				
2	<i>o</i> -Chlorophenyl	90	205	C <sub>12</sub> H <sub>13</sub> ClO <sub>2</sub> N <sub>2</sub>	252.5	57.03 (57.16)	5.15 (5.08)	11.08 (11.26)	14.06 (14.02)			
3	<i>o</i> -Fluorophenyl	60	165	C <sub>12</sub> H <sub>13</sub> FO <sub>2</sub> N <sub>2</sub>	236	61.01 (60.92)	5.51 (5.50)	11.86 (11.83)			8.05 (7.90)	
4	<i>o</i> -Nitrophenyl	76	235	C <sub>12</sub> H <sub>13</sub> O <sub>4</sub> N <sub>3</sub>	263	54.75 (54.88)	4.94 (5.10)	15.97 (15.91)				
5	<i>m</i> -Chlorophenyl	72	152	C <sub>12</sub> H <sub>13</sub> ClO <sub>2</sub> N <sub>2</sub>	252.5	57.03 (57.23)	5.15 (5.03)	11.08 (11.15)	14.06 (13.98)			
6	<i>m</i> -Nitrophenyl	89	188	C <sub>12</sub> H <sub>13</sub> O <sub>4</sub> N <sub>3</sub>	263	54.75 (54.46)	4.94 (4.97)	15.97 (15.90)				
7	<i>m</i> -Trifluoro- methylphenyl	88	140	C <sub>13</sub> H <sub>13</sub> F <sub>3</sub> O <sub>2</sub> N <sub>2</sub>	286	54.54 (54.29)	4.54 (4.64)	9.79 (9.80)			19.93 (19.75)	
8	<i>p</i> -Chlorophenyl	76	215	C <sub>12</sub> H <sub>13</sub> ClO <sub>2</sub> N <sub>2</sub>	252.5	57.03 (57.11)	5.15 (5.25)	11.08 (11.25)	14.06 (14.08)			
9	<i>p</i> -Fluorophenyl	61	170	C <sub>12</sub> H <sub>13</sub> FO <sub>2</sub> N <sub>2</sub>	236	61.01 (61.08)	5.51 (5.57)	11.86 (11.83)			8.05 (7.89)	
10	<i>p</i> -Nitrophenyl	96	210	C <sub>12</sub> H <sub>13</sub> O <sub>4</sub> N <sub>3</sub>	263	54.75 (54.73)	4.94 (4.75)	15.97 (15.70)				
11	<i>p</i> -Methylphenyl	80	180	C <sub>13</sub> H <sub>16</sub> O <sub>2</sub> N <sub>2</sub>	232	67.24 (67.07)	6.89 (6.76)	12.07 (12.18)				
12	<i>p</i> -Methoxyphenyl	80	169	C <sub>13</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub>	248	62.90 (62.76)	6.45 (6.54)	11.29 (11.30)				
13	2,6-Dichloro- phenyl	90	211	C <sub>12</sub> H <sub>12</sub> Cl <sub>2</sub> O <sub>2</sub> N <sub>2</sub>	287	50.17 (50.35)	4.18 (4.23)	9.75 (9.58)	24.74 (24.69)			
14	2-Chloro-6-fluoro- phenyl	63	192	C <sub>12</sub> H <sub>12</sub> ClFO <sub>2</sub> N <sub>2</sub>	270.5	53.23 (53.30)	4.44 (4.57)	10.35 (10.29)	13.12 (13.11)	7.02 (6.89)		
15	3,4-Dimethoxy- phenyl	80	165	C <sub>14</sub> H <sub>16</sub> O <sub>4</sub> N <sub>2</sub>	278	60.43 (60.17)	6.47 (6.40)	10.07 (10.05)				
16	3,4-Methylenedi- oxyphenyl	78	106	C <sub>13</sub> H <sub>14</sub> O <sub>4</sub> N <sub>2</sub>	262	59.54 (59.61)	5.34 (5.25)	10.69 (10.78)				
17	2,4,5-Trimethoxy- phenyl	93	190	C <sub>15</sub> H <sub>20</sub> O <sub>5</sub> N <sub>2</sub>	308	58.44 (58.42)	6.49 (6.50)	9.09 (9.03)				
18	2-Thienyl	40	161	C <sub>10</sub> H <sub>12</sub> O <sub>2</sub> N <sub>2</sub> S	224	53.57 (53.60)	5.36 (5.42)	12.50 (12.42)				14.28 (14.16)
19	2-Furyl	77	138	C <sub>10</sub> H <sub>12</sub> O <sub>3</sub> N <sub>2</sub>	208	57.69 (57.72)	5.77 (5.88)	13.46 (13.43)				
20	Cyclohexyl	84	192	C <sub>12</sub> H <sub>20</sub> O <sub>2</sub> N <sub>2</sub>	224	64.29 (64.31)	8.93 (9.20)	12.50 (12.44)				

spectra were measured, respectively, on a Perkin-Elmer R 24 60 MHz and a Jeol FX 60 spectrometer, using DMSO-d<sub>6</sub> as the solvent. Chemical shifts are expressed in parts per million with respect to TMS: s, singlet; d, doublet; m, multiplet. Microanalyses for C, H, N, Cl, F and S were performed by the Service Central d'Analyses du CNRS (Thiais-France).

#### 5-Substituted 6-Methyl-4,5-dihydro-2H-pyridazin-3-ones **1** to **20**.

The general procedure consisted in dissolving 0.01 mole of 4-acetyl-5-substituted-4,5-dihydro-3H-furan-2-one I in a small volume of hot ethanol. Then 0.01 mole of hydrazine hydrate was added. The reaction mixture was refluxed under stirring for 2 hours. In most cases the expected

pyridazinone precipitated on cooling. Otherwise the solvent was removed *in vacuo* and the residue was washed with anhydrous ether. The crude products were recrystallized from ethanol.

#### X-Ray Analysis of Compound **2**.

Single crystals of the titled compound were grown from ethanol solution.

#### Data Collection.

Compound **2** crystallized as pale yellow blocks in the monoclinic space group P2<sub>1</sub>/n with eight molecules per unit cell (two independent mole-

Table 2  
Spectroscopic Data of Pyridazinones 1 to 20

No.	Infrared Spectrum (KBr), $\nu$ $\text{cm}^{-1}$			$^1\text{H}$ NMR Spectrum (DMSO- $d_6$ ), $\delta$ ppm						
	O-H, N-H	C=O	C=N	CH <sub>3</sub>	Ha, Hb	Hc	Hd	OH	NH	R
1	3400, 3100	1680	1630	1.60 (s)	2.35 (m)	3.30 (m)	4.75 (m)	5.60 (d)	10.15 (s)	7.20 (5H, arom)
2	3280	1670	1640	1.75 (s)	2.20 (m)	2.60 (m)	4.85 (m)	5.85 (d)	10.35 (s)	7.30 (4H, arom)
3	3260	1670	1640	1.80 (s)	2.25 (m)	2.65 (m)	4.90 (m)	5.15 (d)	10.20 (s)	7.35 (4H, arom)
4	3400, 3210	1670	1640	1.95 (s)	2.25 (m)	2.75 (m)	5.55 (m)	6.02 (d)	10.30 (s)	7.90 (4H, arom)
5	3380, 3200	1660	1630	1.78 (s)	2.30 (m)	2.60 (m)	4.90 (m)	5.95 (d)	10.30 (s)	7.30 (4H, arom)
6	3450	1665	1640	1.85 (s)	2.30 (m)	2.70 (m)	5.05 (m)	5.92 (d)	10.20 (s)	7.90 (4H, arom)
7	3460, 3320	1665	1630	1.80 (s)	2.32 (m)	2.67 (m)	4.95 (m)	5.90 (d)	10.35 (s)	7.58 (4H, arom)
8	3400, 3220	1650	1625	1.70 (s)	2.30 (m)	2.60 (m)	4.80 (m)	5.70 (d)	10.25 (s)	7.28 (4H, arom)
9	3280	1665	1640	1.83 (s)	2.33 (m)	2.72 (m)	5.20 (m)	5.68 (d)	10.32 (s)	7.25 (4H, arom)
10	3460, 3240	1650	1630	1.85 (s)	2.25 (m)	2.70 (m)	5.08 (m)	6.05 (d)	10.35 (s)	7.95 (4H, arom)
11	3350, 3250	1665	1640	1.65 (s)	2.45 (m)	3.15 (m)	4.75 (m)	5.40 (d)	10.20 (s)	7.70 (4H, arom) 2.35 (s, CH <sub>3</sub> )
12	3450, 3270	1660	1630	1.65 (s)	2.32 (m)	2.70 (m)	4.70 (m)	5.50 (d)	10.25 (s)	7.00 (4H, arom) 3.78 (s, OCH <sub>3</sub> )
13	3460, 3250	1655	1635	1.30 (s)	2.55 (m)	3.30 (m)	5.30 (m)	5.95 (d)	10.55 (s)	7.32 (3H, arom)
14	3450, 3240	1650	1630	1.50 (s)	2.50 (m)	3.10 (m)	5.15 (m)	5.90 (d)	10.55 (s)	7.23 (3H, arom)
15	3460, 3350	1660	1640	1.68 (s)	2.30 (m)	2.60 (m)	4.75 (m)	5.70 (d)	10.40 (s)	6.95 (3H, arom) 3.75 (s, 2 OCH <sub>3</sub> )
16	3200, 3100	1675	1640	1.72 (s)	2.35 (m)	2.65 (m)	4.72 (m)	5.55 (d)	10.25 (s)	6.82 (3H, arom) 6.00 (s, CH <sub>2</sub> )
17	3480, 3220	1655	1635	1.90 (s)	2.30 (m)	2.65 (m)	4.55 (m)	5.20 (d)	10.20 (s)	6.85 (2H, arom) 3.80 (s, 3 OCH <sub>3</sub> )
18	3280, 3150	1660	1640	1.75 (s)	2.35 (m)	2.70 (m)	4.10 (m)	5.05 (d)	10.32 (s)	7.20 (3H, arom)
19	3250, 3150	1660	1640	1.70 (s)	2.35 (m)	2.77 (m)	4.70 (m)	5.70 (d)	10.30 (s)	7.00 (3H, arom)
20	3260, 3180	1670	1645	1.95 (s)	2.32 (m)	2.62 (m)	3.45 (m)	4.60 (d)	10.28 (s)	1.50 (m, 11H)

cules). Accurate lattice parameters (Table 3) were determined by least-squares refinement of 25 reflections measured on a CAD-4 Enraf-Nonius automatic diffractometer. Systematic extinctions (hol,  $h + 1 = 2n + 1$ ; oko,  $k = 2n + 1$ ) were determined by preliminary intensity measurements.

Table 3

## Physical and Crystal Data of Pyridazinone 2

Chemical Formula	$\text{C}_{12}\text{H}_{13}\text{ClO}_2\text{N}_2$
Formula Weight	252.5
Crystal dimensions	$0.2 \times 0.2 \times 0.1 \text{ mm}^3$
Symmetry	Monoclinic
Space Group	$P2_1/n$
Unit Cell dimensions	$a = 17.445 (3) \text{ \AA}$ $b = 7.079 (1)$ $c = 18.814 (4)$ $\beta = 91.96 (2)^\circ$ $v = 2322.1 (3) \text{ \AA}^3$
Density (calculated)	$1.445 \text{ g cm}^{-3}$
Number of molecules per unit cell, Z	8 (2 independent molecules)
X-radiation used for data collection	$\lambda (\text{CuK}\alpha) = 1.54178 \text{ \AA}$
Total number of reflections with $\theta < 60^\circ$	3540
Number of reflections with $I \geq 3\sigma(I)$	2464
Disagreement index, R	0.041

Intensities for 3540 unique reflections ( $\theta < 60^\circ$ ) were measured at room temperature by a  $\omega\theta$  scan procedure (scan width,  $1^\circ$ ; speed ratio  $\theta/\omega = 1$ ). No deviations in the intensities of check reflection were observed during the data collection. Corrections for Lorentz and polarization effects were made; absorption was ignored.

Table 4

Atomic Positions as Fractional Coordinates ( $\times 10^4$ , H atoms  $\times 10^3$ ) and Equivalent Isotropic Parameters with the Estimated Standard Deviations in Parenthesis

$$\text{Beq} = 4/3 \sum_i \beta_i a_i \cdot a_i$$

	Molecule I			
	Atoms	x/a ( $\sigma$ )	y/b ( $\sigma$ )	z/c ( $\sigma$ )
C(1)	1898(2)	840(5)	8390(2)	2.3(1)
C(2)	1852(2)	1064(5)	7652(4)	2.7(1)
C(3)	2492(2)	1227(5)	7239(2)	3.4(2)
C(4)	3212(2)	1176(6)	7570(2)	3.6(2)
C(5)	3284(2)	955(6)	8300(2)	3.5(2)
C(6)	2630(2)	799(5)	8707(2)	3.0(2)
C(7)	1186(2)	622(5)	8827(2)	2.6(1)
O(8)	1406(1)	-190(4)	9499(1)	3.3(1)
C(9)	794(2)	2576(5)	8937(2)	2.5(1)
C(10)	1276(2)	3934(5)	9408(2)	2.7(2)
C(11)	1156(2)	3690(5)	10189(2)	2.7(2)
N(12)	486(2)	2919(4)	10380(1)	2.9(1)
N(13)	-112(2)	2346(4)	9914(2)	2.8(1)
C(14)	17(2)	2218(5)	9250(2)	2.5(1)
C(15)	-631(2)	1627(6)	8757(2)	3.6(2)
O(16)	1625(1)	4249(4)	10645(1)	3.5(1)
Cl(17)	952(1)	1116(2)	7211(0)	4.0(0)
H(70)	80(2)	-25(5)	855(2)	3.5
H(90)	71(2)	322(5)	845(2)	3.9

Atoms	Molecule II			Beq/Bi ( $\text{\AA}^2$ )
	x/a ( $\sigma$ )	y/b ( $\sigma$ )	z/c ( $\sigma$ )	
C(51)	-1202(2)	40(5)	5895(2)	2.4(1)
C(52)	-1303(2)	-110(5)	5154(2)	2.9(2)
C(53)	-934(2)	1063(6)	4691(2)	3.7(2)
C(54)	-429(2)	2411(6)	4960(2)	4.0(2)
C(55)	-299(2)	2584(5)	5695(2)	3.6(2)
C(56)	-694(2)	1412(5)	6153(2)	3.0(2)
C(57)	-1634(2)	-1226(5)	6393(2)	2.5(1)
O(58)	-1621(1)	-405(4)	7085(1)	3.1(1)
C(59)	-1263(2)	-3226(5)	6447(2)	2.3(1)
C(60)	-514(2)	-3243(5)	6902(2)	2.6(1)
C(61)	-649(2)	-3354(5)	7691(2)	2.5(1)
N(62)	-1314(2)	-4116(4)	7887(1)	2.9(1)
N(63)	-1878(2)	-4886(4)	7424(2)	3.0(1)
C(64)	-1838(2)	-4546(5)	6759(2)	2.6(1)
C(65)	-2418(2)	-5466(6)	6258(2)	3.8(2)
O(66)	-159(1)	-2871(4)	8140(1)	3.5(1)
Cl(67)	-1909(1)	-1842(2)	4793(1)	4.4(0)
H(570)	-220(2)	-139(5)	621(2)	3.6
H(590)	-113(2)	-367(5)	595(2)	3.0

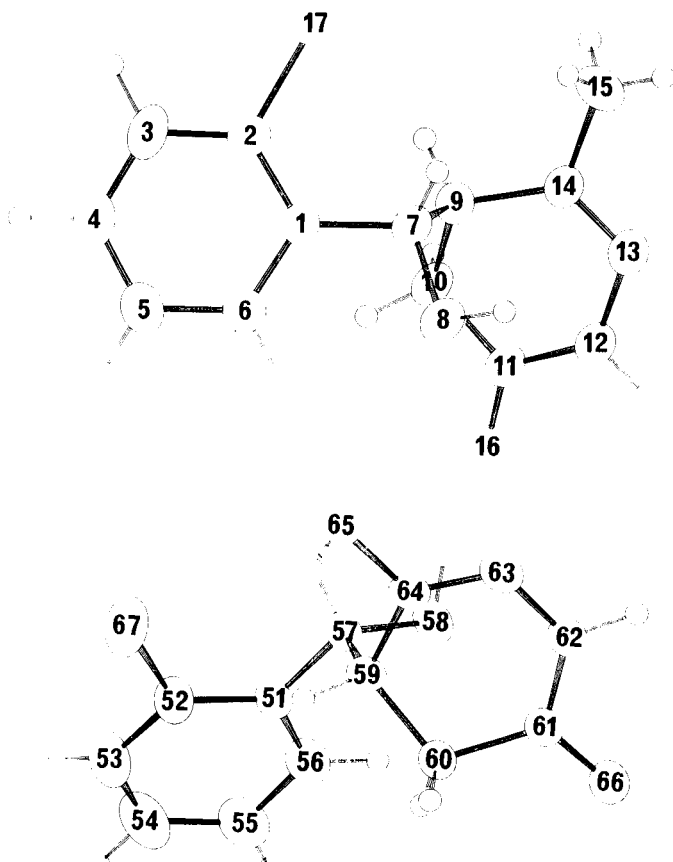


Figure 1. Perspective views of the two independent molecules showing the numbering of atoms. Molecule I is numbered from 1 to 17. X(m) atoms in Molecule II are numbered such that  $m = m + 50$  of the corresponding X in Molecule I. H atoms are numbered such that H(mn) is the nth H on X(m) (X = C, N or O). Atoms 8, 16, 58 and 66 are oxygens, atoms 12, 13, 62 and 63 are nitrogens, atoms 17 and 67 are chlorines. The bared numbers are for carbon atoms.

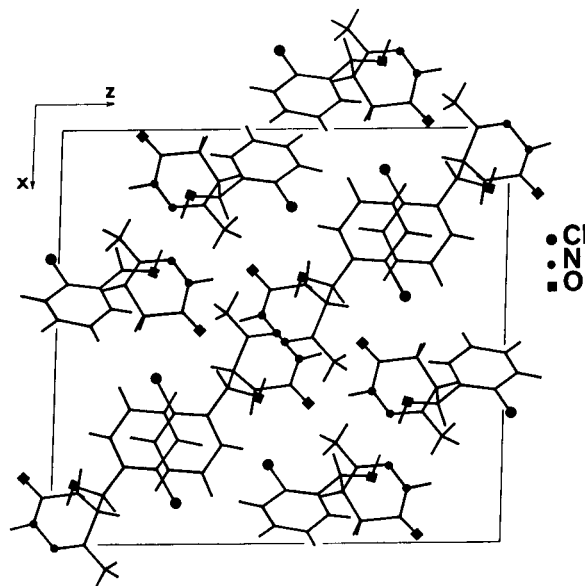


Figure 2. Projection of the structure on (010).

Table 5

Bond Lengths ( $\text{\AA}$ ) (e.s.d.'s in parenthesis)

Molecule I		Molecule II	
C(1)–C(2)	1.396(5)	C(51)–C(52)	1.403(5)
C(1)–C(6)	1.392(5)	C(51)–C(56)	1.390(5)
C(1)–C(7)	1.519(5)	C(51)–C(57)	1.515(5)
C(2)–C(3)	1.387(5)	C(52)–C(53)	1.378(5)
C(2)–Cl(17)	1.751(4)	C(52)–Cl(67)	1.741(4)
C(3)–C(4)	1.384(6)	C(53)–C(54)	1.382(6)
C(4)–C(5)	1.384(6)	C(54)–C(55)	1.399(6)
C(5)–C(6)	1.400(5)	C(55)–C(56)	1.396(5)
C(7)–O(8)	1.430(4)	C(57)–O(58)	1.425(4)
C(7)–C(9)	1.560(5)	C(57)–C(59)	1.558(5)
C(9)–C(10)	1.537(5)	C(59)–C(60)	1.538(5)
C(9)–C(14)	1.518(5)	C(59)–C(64)	1.506(5)
C(10)–C(11)	1.503(5)	C(60)–C(61)	1.511(5)
C(11)–N(12)	1.349(5)	C(61)–N(62)	1.342(4)
C(11)–O(16)	1.230(4)	C(61)–O(66)	1.230(4)
N(12)–N(13)	1.400(4)	N(62)–N(63)	1.402(4)
N(13)–C(14)	1.280(4)	N(63)–C(64)	1.277(5)
C(14)–C(15)	1.496(5)	C(64)–C(65)	1.507(5)

#### Determination of the Structure.

All non hydrogen atoms were located in the E-map generated from the most consistent set of phases from MULTAN 78 [12]. Following the refinement of all positional and anisotropic thermal parameters of these atoms ( $R = 0.068$ ) a difference Fourier map was calculated from which it was possible to locate all the hydrogen atoms; then the positions and isotropic thermal parameters of these atoms were refined. Refinement of all parameters based on 2464 reflections having  $I \geq 3\sigma(I)$  led to the final disagreement index  $R = 0.041$ .

Table 6

Bond Angles ( $^{\circ}$ ) (e.s.d.'s in parenthesis)

Molecule I		Molecule II	
C(2)–C(1)–C(6)	116.7(3)	C(52)–C(51)–C(56)	117.2(3)
C(2)–C(1)–C(7)	121.9(3)	C(52)–C(51)–C(57)	121.4(3)
C(6)–C(1)–C(7)	121.4(3)	C(56)–C(51)–C(57)	121.5(3)
C(1)–C(2)–C(3)	123.1(3)	C(51)–C(52)–C(53)	122.4(3)
C(3)–C(2)–Cl(17)	117.4(3)	C(53)–C(52)–Cl(67)	117.9(3)
C(2)–C(3)–C(4)	118.9(3)	C(52)–C(53)–C(54)	119.4(4)
C(3)–C(4)–C(5)	120.0(4)	C(53)–C(54)–C(55)	120.1(4)
C(4)–C(5)–C(6)	120.2(4)	C(54)–C(55)–C(56)	119.4(4)
C(1)–C(6)–C(5)	121.2(3)	C(51)–C(56)–C(55)	121.5(3)
C(1)–C(7)–O(8)	108.7(3)	C(51)–C(57)–O(58)	109.3(3)
C(1)–C(7)–C(9)	110.6(3)	C(51)–C(57)–C(59)	111.3(3)
O(8)–C(7)–C(9)	110.1(3)	O(58)–C(57)–C(59)	108.5(3)
C(7)–C(9)–C(10)	113.4(3)	C(57)–C(59)–C(60)	112.7(3)
C(7)–C(9)–C(14)	107.8(3)	C(57)–C(59)–C(64)	107.9(3)
C(10)–C(9)–C(14)	111.1(3)	C(60)–C(59)–C(64)	110.0(3)
C(9)–C(10)–C(11)	113.6(3)	C(59)–C(60)–C(61)	113.0(3)
C(10)–C(11)–N(12)	117.2(3)	C(60)–C(61)–N(62)	117.0(3)
C(10)–C(11)–O(16)	122.2(3)	C(60)–C(61)–O(66)	122.2(3)
N(12)–C(11)–O(16)	120.5(3)	N(62)–C(61)–O(66)	120.7(3)
C(11)–N(12)–N(13)	125.9(3)	C(61)–N(62)–N(63)	125.5(3)
N(12)–N(13)–C(14)	118.6(3)	N(62)–N(63)–C(64)	118.3(3)
C(9)–C(14)–N(13)	123.7(3)	C(59)–C(64)–N(63)	123.9(3)
C(9)–C(14)–C(15)	118.2(3)	C(59)–C(64)–C(65)	117.7(3)
N(13)–C(14)–C(15)	118.0(3)	N(63)–C(64)–C(65)	118.3(3)

Table 7

Torsion Angles ( $\pm 1^{\circ}$ )

Molecule I		Molecule II	
C(2)–C(1)–C(7)–C(8)	161	C(52)–C(51)–C(57)–C(58)	199
C(2)–C(1)–C(7)–C(9)	–78	C(52)–C(51)–C(57)–C(59)	79
C(1)–C(7)–C(9)–C(10)	–67	C(51)–C(57)–C(59)–C(60)	75
C(1)–C(7)–C(9)–C(14)	169	C(51)–C(57)–C(59)–C(64)	197
C(7)–C(9)–C(10)–C(11)	–86	C(57)–C(59)–C(60)–C(61)	82
C(7)–C(9)–C(14)–N(13)	98	C(57)–C(59)–C(64)–N(63)	268

The scattering factor tables for non-hydrogen atoms and the anomalous dispersion corrections for Cl were taken from the International Tables for X-ray Crystallography [13] whereas the scattering factors for hydrogens were taken from [14].

Positional parameters and isotropic (or equivalent) thermal parameters are given in Table 4.

This study establishes the configuration as *erythro* (5*RS*,7*SR*) for pyridazinone **2**.

#### Discussion.

The ORTEP [15] drawings of the two independent molecules are depicted in Figure 1. The bond lengths and angles are listed in Tables 5 and 6. Some torsion angles are presented in Table 7. Least squares planes are listed in Table 8. The angle between plane 1 and plane 2 is  $19(1)^{\circ}$  in Molecule I and  $11(1)^{\circ}$  in Molecule II.

The crystalline cohesion is ensured by van der Waals contacts and by intermolecular hydrogen bonds. Data for these interactions are listed in Table 9.

Table 8

Least-squares Planes and the Displacements of Atoms from the Plane

Molecule I	
Plane 1 – $0.0317 X + 0.9930 Y + 0.1140 Z = 2.2927$	
Atoms used for the calculation of the equation of the plane	Deviations of the atoms from the plane ( $\text{\AA}$ )
C(1)	0.008(3)
C(2)	0.009(4)
C(3)	–0.002(4)
C(4)	–0.005(4)
C(5)	–0.007(4)
C(6)	0.008(4)
C(7)	–0.011(4)
Cl(17)	–0.001(1)
Other atoms	
O(8)	–0.448(3)
Plane 2 + $0.2425 X - 0.9611 Y + 0.1321 Z = 0.6614$	
Atoms used for the calculation of the equation of the plane	Deviations of the atoms from the plane ( $\text{\AA}$ )
C(9)	0.003(4)
C(14)	–0.010(3)
C(15)	0.004(4)
N(13)	0.003(3)
Other atoms	
C(11)	–0.310(4)
N(12)	–0.025(3)
C(7)	1.472(4)
O(8)	2.274(3)
Molecule II	
Plane 1 – $0.7572 X + 0.6531 Y + 0.0119 Z = 2.0214$	
Atoms used for the calculation of the equation of the plane	Deviations of the atoms from the plane ( $\text{\AA}$ )
C(51)	0.003(3)
C(52)	0.014(4)
C(53)	0.035(4)
C(54)	0.010(4)
C(55)	–0.030(4)
C(56)	–0.016(4)
C(57)	0.023(3)
Cl(67)	–0.011(1)
Other atoms	
O(58)	0.434(2)
Plane 2 – $0.6285 X + 0.7708 Y + 0.1042 Z = 1.1433$	
Atoms used for the calculation of the equation of the plane	Deviations of the atoms from the plane ( $\text{\AA}$ )
C(59)	0.003(3)
C(64)	–0.011(3)
C(65)	0.004(4)
N(63)	0.004(3)
Other atoms	
C(61)	–0.446(3)
N(62)	–0.086(3)
C(57)	1.489(3)
O(58)	2.086(2)

Full hydrogen atoms coordinates, bond distances and angles involving hydrogen atoms as well as observed and calculated structure factors are available from unpublished data [16].

Table 9

## Intermolecular Hydrogen Bonds

Symmetry Code			
(i) x, y, z	(ii) $\bar{x}$ , $\bar{y}$ , $2 - z$	(iii) $-\frac{1}{2} - x$ , $\frac{1}{2} + y$ , $\frac{3}{5} - z$	
Hydrogen bonds (distances in Å, angles in degrees)			
Molecule I - Molecule I			
O(8 <sup>i</sup> )...N(13 <sup>ii</sup> )	2.968(4)	H(80 <sup>i</sup> )...N(13 <sup>ii</sup> )	1.98(4)
		O(8 <sup>i</sup> )-H(80 <sup>i</sup> )...N(13 <sup>ii</sup> )	170(3)
Molecule II - Molecule II			
O(58 <sup>i</sup> )...N(63 <sup>ii</sup> )	2.831(4)	H(580 <sup>i</sup> )...N(63 <sup>ii</sup> )	1.93(4)
		O(58 <sup>i</sup> )-H(580 <sup>i</sup> )...N(63 <sup>ii</sup> )	170(4)
Molecule I - Molecule II			
N(12 <sup>i</sup> )...O(66 <sup>ii</sup> )	2.862(4)	H(120 <sup>i</sup> )...O(66 <sup>ii</sup> )	1.89(4)
		N(12 <sup>i</sup> )-H(120 <sup>i</sup> )...O(66 <sup>ii</sup> )	173(3)
O(16 <sup>i</sup> )...N(62 <sup>ii</sup> )	2.835(4)	O(16 <sup>i</sup> )...H(620 <sup>ii</sup> )	1.84(4)
		O(16 <sup>i</sup> )-H(620 <sup>ii</sup> )-N(62 <sup>ii</sup> )	179(3)

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