# The Crystal and Molecular Structure of the Alkaloid Gerrardine 

By G. Gafner and L. J. Admiraal<br>Chemical Physics Group of the National Physical and National Chemical Research Laboratories, Council for Scientific and Industrial Research, Pretoria, South Africa

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#### Abstract

The structure of one enantiomorph of the alkaloid gerrardine $\left(\mathrm{C}_{11} \mathrm{H}_{19} \mathrm{O}_{2} \mathrm{~S}_{4} \mathrm{~N}\right)$ has been determined from diffractometer data. The final $R$ value is 0.066 after full-matrix least-squares refinement excluding hydrogen atoms. The molecule is found to be 1-methyl-2,5-bis-(4-hydroxy-1,2-dithiolan-3-yl)pyrrolidine and possesses a strong intramolecular $\mathrm{O}-\mathrm{H} \cdots \mathrm{N}$ hydrogen bond. One dithiolan ring is essentially planar with the central pyrrolidine ring and the other is at right angles to it. Intermolecular $\mathrm{O}-\mathrm{H} \cdots \mathrm{O}$ hydrogen bonds join molecules to form sheets normal to the $a$ axis.


## Introduction

The alkaloid gerrardine $\left(\mathrm{C}_{11} \mathrm{H}_{19} \mathrm{NO}_{2} \mathrm{~S}_{4}\right)$ was isolated, crystallized and submitted for structure analysis by Professor F. L. Warren and Dr W. G. Wright of the University of Natal, Pietermaritzburg, who have explained its chemical behaviour, mass- and infrared spectra in terms of its structure as determined here (Wright \& Warren, 1967). This is the second alkaloid from species of the genus Cassipourea which contains four sulphur atoms per molecule. The other is cassipourine (Cooks, Warren \& Williams, 1967; Gafner \& Admiraal, 1969). Structural similarities were expected for these two alkaloids but not found. No information on the possible structure of gerrardine was available when submitted for analysis other than its elemental analysis and formula weight.

## Experimental

The original structure analysis was carried out on visually estimated photographic intensity data. This analysis, although basically correct in the structure it gave, was considered too inaccurate for publication as $R$ was 0.17 and differences of up to $0.2 \AA$ were found between equivalent bonds. Intensities were thus measured diffractometrically when the necessary instrumentation became available. This redetermination of the structure is described here.

All X-ray measurements were made on a crystal which was cut to a cube of 0.5 mm edge from a needleshaped crystal. Preliminary cell constants, which were obtained using photographic methods, were refined using least-squares procedures on diffractometrically measured spot positions (Busing \& Levy, 1967).

The cell data are as follows:

$$
\begin{aligned}
& a=17.614 \pm 0 \cdot 002, b=12 \cdot 212 \pm 0 \cdot 002 \text {, (needle axis) } c= \\
& \quad 6 \cdot 79 \pm 0 \cdot 01 \AA \text {. } \\
& \text { Space group: } P 2_{1} 2_{1} 2_{1} \text { (absences } h 00,0 k 0 \text { and } 00 l \text { for } h, \\
& \quad k \text { and } l \text { odd respectively). }
\end{aligned}
$$

$$
Z=4, \varrho_{m}=1.49, \varrho_{c}=1.48 \mathrm{g.cm}^{-3}
$$

$$
\text { formula weight }=325 \cdot 5
$$

Densities were measured in $\mathrm{K}_{2} \mathrm{HgI}_{4} / \mathrm{H}_{2} \mathrm{O}$ and bromoform/methanol solutions using flotation methods.

Data were collected using the $\omega$ scan method on a Hilger \& Watts automatic single-crystal diffractometer. Zr -filtered Mo radiation was used ( $\lambda=0.70926 \AA$ ). Background was measured as a function of $\theta$ by counting at closely spaced points next to a central lattice row. The results obtained were found to agree satisfactorily with measurements made at the positions of the systematic absences. The 832 intensities in one octant with $\theta \leq 20^{\circ}$ were measured and, of these, 135 fell below the 'observed' criterion which was taken as three times the background count. These reflexions are marked with asterisks in the $F_{o}$ column of Table 2.
Lorentz and polarization corrections were applied to the measured intensities. Absorption corrections were obviated by the low $\mu R$ value of 0.14 which makes the absorption correction $<1 \%$ when intensities are measured at $\theta$ angles below $20^{\circ}$.

## Structure determination and refinement

The trial structure, excluding hydrogen atoms, was built up from four Fourier syntheses, the first of which was phased on the positions of the four sulphur atoms as established from a Patterson synthesis.
After preliminary refinement with isotropic temperature factors ( $R=0.12$ ), three cycles of full-matrix least-squares refinement using anisotropic temperature factors and equal weighting resulted in no further significant shifts and gave a final $R$ value of 0.066 . The program used was $O R F L S$ of Busing, Martin \& Levy (1962), which minimizes $\sum \omega\left(\left|F_{o}\right|-\left|F_{c}\right|\right)^{2}$, on an IBM 360/65 computer. The atomic scattering factors used were those of Hanson, Herman, Lea \& Skillman (1964).


The final atomic parameters and their estimated standard deviations are given in Table 1 with the $U_{i j}$ values from the expression

$$
\begin{aligned}
\exp [ & -2 \pi^{2}\left(h^{2} a^{* 2} U_{11}+k^{2} b^{* 2} U_{22}+l^{2} c^{* 2} U_{33}\right. \\
& \left.\left.+2 h k a^{*} b^{*} U_{12}+2 k l b^{*} c^{*} U_{23}+2 h l a^{*} c^{*} U_{13}\right)\right] .
\end{aligned}
$$

The inclusion of hydrogen atoms at their expected positions gave no significant reduction of $R$ before anisotropic refinement and their contribution is thus excluded from Table 2 which lists final calculated and observed structure factors after anisotropic refinement.

## Molecular geometry

This analysis indicated that the alkaloid is 1-methyl-2,5-bis-(4-hydroxy-1,2-dithiolan-3-yl)pyrrolidine. In contrast to cassipourine, in which the four sulphur atoms are contained in a $1,2,5,6$-tetrathiacyclo-octane ring, the sulphur atoms in gerrardine are in pairs in two dithioanyl rings. These dithioanyl rings are trans to one another. Atomic nomenclature, bond lengths and angles and deviations from the best plane containing the pyrrolidine ring and the $S(3), S(4)$ dithioanyl ring are given in Fig. 1. The equation to this plane is:

$$
-0.5101 x+0.8486 y-0.1403 z=2.9445
$$

with respect to the crystallographic axes. The deviations from this plane indicate coplanarity of these two


Fig. 1. (a) Bond lengths in $\AA$ and their standard deviations with reference to the last significant figure. Deviations from the plane through the central and right-hand side rings are given in the circles in $\AA$. (b) Atomic nomenclature, bond angles and their standard deviations in degrees.
rings to $0.37 \AA$. The other dithioanyl ring is essentially at right angles to this plane.

Average bond lengths are:

$$
\begin{aligned}
& \mathrm{S}-\mathrm{S}=2.07 \pm 0.01, \quad \mathrm{~S}-\mathrm{C}=1.83 \pm 0.02 \\
& \mathrm{C}-\mathrm{C}=1.51 \pm 0.01, \quad \mathrm{C}-\mathrm{N}=1.51 \pm 0.01, \\
& \mathrm{C}-\mathrm{O}=1.43 \pm 0.01 \AA .
\end{aligned}
$$

These agree satisfactorily with accepted values and no bond length differs significantly from the mean of the group into which it falls.

A strong intramolecular $\mathrm{O}-\mathrm{H} \cdots \mathrm{N}$ hydrogen bond of length $2.59 \AA$ exists between $\mathrm{O}(1)-\mathrm{H}$ and the nitrogen atom and this is presumably a determining factor in the mutual orientation of the two rings involved. This hydrogen bond length is close to the shortest $\mathrm{O}-\mathrm{H} \cdots \mathrm{N}$ bond length of $2 \cdot 62 \AA$ in guanine. $\mathrm{HCl} . \mathrm{H}_{2} \mathrm{O}$
listed by Pimentel \& McClellan (1960) and is shown by the stippled straight line in Fig. 2.

Bond angles are all as expected and no influence of the hydrogen bond is seen.

## Molecular packing

The molecules are arranged in stacks with stack axes parallel to $\mathbf{b}$ as shown in Fig. 2. Molecules within a. stack are related by twofold screw axes and each stack is surrounded by six further stacks in a near-hexagonal arrangement. Intermolecular $\mathrm{O}-\mathrm{H} \cdots \mathrm{O}$ hydrogen bonds are found between molecules in stacks which are in planes normal to the $a$ axis. One such bond is that which is shown in the lower right hand corner of Fig. 2 as a three-segment stippled line. The formation

Table 2. Calculated and observed structure factors on an absolute scale

| $1 \mathrm{~L}_{0} \mathrm{P}_{\mathbf{c}}$ | $\mathrm{P}_{0} \mathrm{P}^{\text {c }}$ | $p$ 。 ${ }^{\text {c }}$ | $p_{0} y_{\text {c }}$ | $\mathrm{F}_{0} \quad \mathrm{P}_{6}$ | $1 \mathrm{~F}_{0} y_{c}$ | $p_{0}{ }^{\text {P }}$ | $\mathrm{P}_{0} \mathrm{P}_{\mathrm{c}}$ |  | $1 p_{0} p_{\text {c }}$ | L Po ${ }^{\text {c }}$ | $1{ }^{1}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 0. 0 , | - $1{ }_{59}{ }^{2,1} 6$ | ${ }^{37}$ | ${ }_{39}{ }^{3}{ }^{\text {2 }}{ }_{39}$ | 4 29 30 <br> 5 40  <br> 5   | 3 25 24 <br> 4 9 9 <br> 5   | $\begin{array}{lll}3 & 30 & 33 \\ 4 & 18 & 36 \\ & 19 & \\ & 17\end{array}$ | 7, 72, ${ }_{17}$ | 8, ${ }_{40}^{10, L_{4}}$ | ${ }_{0}^{10} \mathrm{i}_{2}{ }^{3, \mathrm{~L}}{ }_{13}$ | ${ }_{40}^{8,5}$ | ${ }^{13} 17{ }^{6,1}{ }^{4}$ |
| $\begin{array}{ll}24 & 24 \\ 21\end{array}$ | $\begin{array}{cc}106 \\ 57 & 102 \\ \\ 52\end{array}$ | $\begin{array}{ll}11 & 12 \\ 17 & 15\end{array}$ | 1 78  <br>  77  <br>  17 76 |  |  |  | $\begin{array}{llll}0 & 12 & 17 \\ 1 & 29 & 30 \\ 20 & 10\end{array}$ | 1 | $\begin{array}{llll}1 & 24 & 24 \\ 1 & 24 & \\ 2 & 12\end{array}$ | $\begin{array}{cc}25 & 25 \\ 7\end{array}$ | $\begin{array}{llll} \\ 2 & 19 & 17 \\ 2 & 12 & 11\end{array}$ |
| $21 \quad 20$ | $\begin{array}{lll}2 & 57 & 52 \\ 3 & 16 & 15 \\ 4 & 7\end{array}$ | 6 | $\begin{array}{lll}1 & 17 \\ 3 & 17 & 16 \\ 5 & 50 \\ 5 & 50\end{array}$ | 4,4 |  |  |    <br> 2 40 5 <br> 3 8  <br>    |  | $\begin{array}{ccc}2 & 13 & 12 \\ 3 & 9 & 9\end{array}$ |  |  |
| 0, 1, 1 | $\begin{array}{llll}4 & 5 & \\ 5 & 7 & 6 \\ 5 & 11 & 9\end{array}$ | 2, 3, 1 | $\begin{array}{llll}4 & 13 & 12 \\ 5 & 13 \\ 5 & 21 & 12 \\ 0\end{array}$ | $\begin{array}{lll} 0 & 6 \\ 1 & 54 & 54 \end{array}$ | $0_{0}{ }^{5} 57{ }^{5} \mathrm{l}_{68}$ | 6, 6, L | 4 4 ${ }^{\text {a }}$ |  | [ $\begin{array}{r}18 \\ \hline 8\end{array}$ | 22, ${ }^{12,1}$ |  |
| $\begin{array}{ccc}1 & 31 & 33 \\ 2 & 13 & 9 \\ 4 & 1 & \end{array}$ | $\begin{array}{lll}5 & 11 & 9 \\ 6 & 12 & 8\end{array}$ | ${ }^{21} 4{ }^{41}$ | $\begin{array}{ccc}5 & 21 & 20 \\ 6 & 7 & 6\end{array}$ | 1 54  <br> 2 39 54 | $1 \begin{array}{lll}1 & 13 & 9\end{array}$ | - 59.8 | 7, 8, | $\begin{array}{lll}2 & 19 \\ 3 & 30 & 19 \\ 4\end{array}$ | 5 50 3 | 0   <br> 1 7. 5 <br> 1 4  <br>    | $0^{13}{ }_{10}{ }^{\prime} L^{2}$ |
| $\begin{array}{llll}1 \\ 3 & 19 & 26\end{array}$ |  | $\begin{array}{ll}28 \\ 96 & 29 \\ 50\end{array}$ |  | $\begin{array}{llll}3 & 13 & 14 \\ 4 & 17 & \end{array}$ | $\begin{array}{llll}2 & 27 & 27 \\ 3 & 18 & 29\end{array}$ | $\begin{array}{llll}1 & 11 & 14 \\ 2 & 19 & 20 \\ & \end{array}$ |  |  | 10, 4, L | $\begin{array}{llll}2 & 14 & 14\end{array}$ | $1 \begin{array}{lll}11 & 10\end{array}$ |
| 4 30 1 <br> 5 20 1 <br>  20 21 | 1,3, 4 | $\begin{array}{ll}36 & 30 \\ 33\end{array}$ | 3, ${ }_{30}{ }^{2} L_{38}$ | $\begin{array}{lll}4 & 17 & 14 \\ 5 & 11 & 11\end{array}$ | $\begin{array}{llll}3 & 18 \\ 4 & 18 & 19 \\ 3 & 15\end{array}$ |  | 1 19819 | $5{ }^{4} \times$ | 0 515 | 3 10 4 <br> 4 21  <br>    <br> 10   |  |
| $\begin{array}{lll}5 & 20 & 21 \\ 0 & 13 & 13\end{array}$ | 71.70 | $7{ }^{7} 5$ | $\begin{array}{ll}30 & 38 \\ 58 & 56 \\ \end{array}$ |  | ${ }^{4}$ 11 10 | $\begin{array}{llll}4 & 14 & 13\end{array}$ | 2  <br> 3 19 <br> 10  <br> 40  |  | 1   <br> 1 19 19 <br> 2 18  |  | $0^{14} 16{ }^{16}$ |
|  | $2{ }^{2}$ 45 46 |  | $47 \quad 47$ | ${ }_{0}{ }^{4}, 55^{5} \mathrm{~L}$ |  | 55 |  | ${ }_{0} 9.1$ | ${ }_{3}^{3} 817.16$ | 12. ${ }^{12} \mathrm{l}^{2}$ | 1 20 21 <br> 2 13 15 <br>  18  |
|  | 3 57 58 <br>    |  | 3 16 <br> 4 14 <br>  18 | $\begin{array}{llll}0 & 75 & 73 \\ 1 & 23 & 22\end{array}$ |  | 6. 7.2 | ${ }^{7}{ }_{18} 9{ }^{1}{ }^{17}$ | $\begin{array}{llll}1 & 32 & 29\end{array}$ |  | $\begin{array}{llll}0 & 12 . & 11 \\ 1 & 4 . & 4\end{array}$ | $\begin{array}{llll}3 & 13 & 15 \\ 3 & 40 & 4\end{array}$ |
| $\begin{array}{llll}0 & 4 & 12 \\ 1 & 4 & 12\end{array}$ | $\begin{array}{lll}4 & 20 & 19 \\ 5 & 21 & 20\end{array}$ | 2, 4, L | $\begin{array}{lll}4 & 14 \\ 5 & 15 & 14\end{array}$ | $\begin{array}{llll}1 & 22 & 23 \\ 3 & 20 & 23 \\ 3 & 20 & 19\end{array}$ | $\begin{array}{lll}1 & 19 & 18 \\ 2 & 19 & 20\end{array}$ | $\begin{array}{lll}0 \\ 1 & 44 & 48 \\ 1 & 22 & \\ 28\end{array}$ |  | 2 34 38 <br> 3 32 38 <br> 5   | ${ }^{10,55}{ }^{5} \mathrm{~L}^{27}$ | ${ }_{2}{ }^{1} 787$ |  |
| 35 34 <br> 54  <br> 4  | 8 |  | ${ }_{5}{ }^{5}$ | $\begin{array}{lll}3 & 20 \\ 4 & 20 & 19 \\ 4 & 21 & 21\end{array}$ | $\begin{array}{lll}2 & 19 & 20 \\ 3 & 6 & 7\end{array}$ | $\begin{array}{llll}1 \\ 2 & 22 & 22 \\ 2 & 16 & 17\end{array}$ | $\begin{array}{llll} \\ 2 & 18 & 12 \\ 3 & 72 & 7\end{array}$ | $\begin{array}{llll} \\ 4 & 17 & 19 \\ 4 & 15 & 19\end{array}$ | $\begin{array}{lll}0 & 25 & 27 \\ 1 & 37 & 36\end{array}$ |    <br> 3 8 8 <br> 4 7 8 <br>    |  |
| 3 54 54 <br> 4 20 19 <br> 5 28  |  | 29 28 <br> 67 63 |  | 51111 | ${ }_{5}^{4}$ 13. 14 | $\begin{array}{llll}3 & 14 & 13 \\ 4 & 8 & 8\end{array}$ | 3 1212 | 5 15 25 | $\begin{array}{llll}1 \\ 2 & 37 & 38 \\ 2 & 31\end{array}$ |  | $1 \begin{array}{llll}1 & 26 & 25\end{array}$ |
| $\begin{array}{ccc}5 & 28 . & \\ 6 & 4 . & \\ \end{array}$ | ${ }^{12} 2^{4} \cdot{ }_{1}$ | $14 \quad 13$ | 14.16 |  | 5 4* | $4{ }^{4} 8$ | 7, 10, 6 |  | $\begin{array}{lll}3 & 22 & 23 \\ 4 & 48\end{array}$ | ${ }^{12,}{ }_{40}{ }^{2} \mathrm{~L}_{5}$ | 2   <br> 3 21 21 <br>  10  |
|  | 50 So |  | $\begin{array}{lll}1 & 24 & 23 \\ 2 & 43 & 23 \\ 4 & 17\end{array}$ |  | 5 | , |  | $0{ }^{0} 5$ |  | 0    <br> 1 40 40 3 |  |
| $0,3, \mathrm{~L}$ | $\begin{array}{ll}26 & 27 \\ 18 & 16\end{array}$ | 5 $4^{\circ}$ 5 <br> 0 5  <br> 5 5  | $\begin{array}{lll}2 & 43 & 47 \\ 3 & 12 & 10\end{array}$ | 1 19 <br> 1 19 | 1 | $\bigcirc 8$ | $\begin{array}{ccc}1 & 13 & 14 \\ 2 & 7 & 5\end{array}$ | $1{ }^{1} 1248$ | .6, | $\begin{array}{llll}2 & 11 & 11\end{array}$ | 14, 2, 1 |
| ${ }^{2} 1$ | 4 <br> 4 <br> 4 |  | 21.21 | ${ }_{23}^{22} \quad 2 \begin{array}{ll}24 \\ 24\end{array}$ | $\begin{array}{lll}1 \\ 1 & 23 & 26 \\ 2 & 15 & 15\end{array}$ | 1 32 31 <br> 2 13 16 <br>    |  | 2   <br> 3 27 28 <br>  28  <br>  28  | $0{ }^{0} 86$ | $3{ }^{3} 121$ |  |
| $\begin{array}{ll}31 & 32 \\ 10 & 11\end{array}$ | ${ }^{5} 80$ | $0^{2.3 .5}{ }^{5}{ }^{5}$ | $10 \quad 11$ | 97 | $3{ }^{1} 311$ | , 40 5 |  |  | $\begin{array}{llll}1 \\ 2 & 18 & 17 \\ 2 & 15 & 15\end{array}$ | $\begin{array}{lll}4 & 12 & 12\end{array}$ | 1 17  <br> 2 4.  |
| $10 \quad 10$ | 11. |  | 3,6, L | $5{ }^{5} 46$ | $\begin{array}{llll}4 & 14 & 13\end{array}$ | $4{ }^{4}$ | ${ }^{815}{ }^{815} \mathrm{~L}_{42}$ | $5{ }^{\circ} \mathrm{C}$ | $\begin{array}{ccc}1 & 15 & 17 \\ 3 & 7 & 5\end{array}$ | 12, 3, ${ }^{\text {c }}$ | $3 \begin{array}{lll}3 & 17 & 17\end{array}$ |
| $\begin{array}{llll}5 & 27 & 25 \\ 6 & 14 & 13\end{array}$ | 1, 5, 1 | 67.67 |  |  | 5.8, | , 9, | $\begin{array}{llll}1 & 61 & 62 \\ & 3 & & \\ 3\end{array}$ | 9,3, | 450 | 28.29 |  |
|  | 13 14 | $\begin{array}{ccc}3 & 33^{*} & 12 \\ 4 & 5 & 4\end{array}$ | $\begin{array}{llll}1 & 3 \\ 2 & 38 & 38\end{array}$ | $\begin{array}{llll}0 & 29 & 30\end{array}$ | 27 27 27 | 0 4: | $\begin{array}{cccc}2 \\ 2 & 33 & 34 \\ 3 & 4 . & 2 \\ 5\end{array}$ |  | 10, 7. L | 910 | 0 is 14 |
| ${ }^{0}{ }^{4}{ }^{4}$. | $17 \quad 18$ | 5 8 | 3 28 27 | $\begin{array}{ccc}1 & 20 & 20 \\ 2 & 13 & 11 \\ & \end{array}$ | $\begin{array}{lll}33 & 22 \\ 24\end{array}$ |  | $\begin{array}{lll}4 & 16 & 16\end{array}$ | $\begin{array}{llll}1 \\ 2 & 16 & 14 \\ 18\end{array}$ | ${ }^{0}{ }^{40}$ | $\begin{array}{lll}3 & 12 & 10 \\ 4 & 10\end{array}$ | $\begin{array}{llll}1 & 22 & 22 \\ 2 & 15 & 15 \\ & 15 & \end{array}$ |
| $\begin{array}{rl}129 & 127 \\ 27 & 30\end{array}$ | $\begin{array}{ccc}3 & 5 & 4 \\ 4 & 19 & 4\end{array}$ |  | $\begin{array}{lll}4 & 10 & 10 \\ 5 & \text { is } & 15\end{array}$ | $\begin{array}{llll}3 & 4 . & 6 \\ 3 & 4 . & \end{array}$ |  | 3 | $5{ }^{5} 1313$ | 3 25 25 <br>  14  <br>    <br>    | $\begin{array}{llll}1 & 16 & 16 \\ 2 & 22 & 22 \\ 20\end{array}$ |  | $\begin{array}{llll}2 \\ 3 & 4 . & \\ 3 & 4\end{array}$ |
| ${ }^{46} 46$ | $\begin{array}{ccc}4 & 19 \\ 5 & 7 & 21 \\ & 7 & 6\end{array}$ | ${ }^{2} \mathrm{ib}_{6}^{6, L_{17}}$ |  | $\begin{array}{lll}4 & 27 \\ 5 & \\ 5\end{array}$ | 4 4* 4 |  | . 1, | $\begin{array}{cccc}4 & 14 & 14 \\ 5 & 8 . & 14 \\ \end{array}$ | $\begin{array}{llll}3 & 16 & 16\end{array}$ |  |  |
| ${ }^{38} 188$ |  | $25 \quad 25$ |  | 5 \% 5 | 5, 9, 1 | ${ }^{-1}{ }_{21} 10,23$ | , | 5 ¢ |  | 0 29 31 <br> 1 19 19 | ${ }^{14}{ }_{22}{ }^{4,} L^{2}$ |
| 15. 15 | ${ }_{12}^{14}{ }^{6} \mathrm{C}_{14}$ | 17818 |  | 4, 8 , | $25 \quad 26$ | $\begin{array}{llll}1 & 12 & 13\end{array}$ | $15 \quad 12$ | 9. $4, \mathrm{~L}$ | ${ }_{0}{ }_{18}{ }^{8} 8^{8}$ | $\begin{array}{llll}1 & 19 & 19 \\ 2 & 17 & 16\end{array}$ | $16 \quad 16$ |
| T | $12 \quad 11$ | 19.18 | $11 \quad 15$ | 25 25 <br> 35 36 <br> 15  | 1 12  <br> $\frac{12}{12}$ 11  <br>  14 15 | $2 \quad 10 \quad 10$ |  |    <br> 0 30 30 <br> 1 23 32 <br> 1   | 14.6 | 3 13 13 | 278 |
| 0,5 , | $\begin{array}{lll}54 & 35 \\ 33 & 32\end{array}$ | 5 | $\begin{array}{ll}21 & 22 \\ 17 & 16\end{array}$ | $\begin{array}{llll}2 & 15 & 19 \\ 2 & 15\end{array}$ | 11 is |  | 4 4* | $\begin{array}{lll}2 & 12 & 11\end{array}$ |  | $5 \cdot$ |  |
| 7 | $10 \quad 9$ | , | $\begin{array}{llll}5 & 15 & 14\end{array}$ | $\begin{array}{ccc}3 & 11 & 13 \\ 4 & 6\end{array}$ |  | 10 |  | 1   <br> 3 21 20 <br> 4 10 20 <br> 5   | 10, 9, L | 22, 3, 2 |  |
| $\begin{array}{ccc}2 \\ 3 & 8 & 8 \\ 3 & 78 \\ 5 & 79\end{array}$ | 5 10 5 |  |  | 45 | $\bigcirc{ }^{10} 10.10$ |  | ${ }_{62}^{8,2, L}$ | $5{ }_{5} 50 \quad 5$ | (18) | $\begin{array}{lll}0 \\ 1 & 15 & 3 \\ 15\end{array}$ | $\begin{array}{lll}1 & 22 & 20\end{array}$ |
|  | 1, 7, | $21 \quad 23$ |  | ${ }^{4} 8_{8}^{9,}{ }^{\text {L }} 7$ |  | 7, o, ᄂ | $\begin{array}{llll}0 & 62 & 58 \\ 1 & 46 & \\ 47\end{array}$ |  |  | $\begin{array}{llll}2 & 4 & 4 \\ 3 & 12 & 4\end{array}$ |  |
| $14 \quad 12$ | $3{ }^{\circ}$ | $16 \quad 19$ | 1 17 17 <br> 2 10  | $\begin{array}{lll}1 \\ 1 & 11 & 11\end{array}$ |  | 1   <br>  46 47 <br> 2 51 55 <br>    | 24 38  <br> 3 35  <br>  16  |  |  | $\begin{array}{llll}3 & 12 & 11\end{array}$ | 14. 6, L |
| 0, 6, L | $\begin{array}{ll}22 & 21 \\ 15 & 16\end{array}$ | $\begin{array}{cc}16 & 15 \\ 6 & 7\end{array}$ | $\begin{array}{lll}3 & 18 & 17 \\ 3 & 18 & 17\end{array}$ |  |  | $\begin{array}{cccc}2 & 51 & 55 \\ 3 & 30 & 3\end{array}$ | $\begin{array}{lll}3 & 16 \\ 4 & 16 & \\ 4 & 24 \\ & 26\end{array}$ |  | $\begin{array}{ccc}1 & 4 & 6\end{array}$ | 12, 6, L, | $\begin{array}{lll}0 & 9 & 8 \\ 1 & 13 & 14\end{array}$ |
| 333 | $15 \quad 14$ |  | 6 | $\begin{array}{llll}3 & 12 & 11\end{array}$ | 122 | : ${ }^{16}{ }^{17}$ | 54. | $3{ }^{2} 302015$ | $\begin{array}{ccc}3 & 40 \\ 4 & 9 & 11\end{array}$ | $\begin{array}{ccc}0 \\ 1 & 10 & 71\end{array}$ |  |
| 21820 |  | ${ }^{2} 15^{8,1}{ }^{1} 17$ |  | ${ }^{12}$ 10, 2 |  |  |  | 4 |  | $\begin{array}{llll}1 & 8 & \\ 2 & 6 & 6\end{array}$ | 15, 0.1 |
| $\begin{array}{lll}3 & 9 & 11 \\ 4 & 6 & 6\end{array}$ |  | 88 | ${ }_{0} \quad 1819$ | $\begin{array}{ccc}0 & 15 & 15 \\ 1 & 9 & 6\end{array}$ |  | 7.1, L | $0_{0}^{8}{ }_{31}{ }^{\text {a }}$ |  | ${ }_{0}^{11,}{ }_{46}{ }^{1} \mathrm{~L}$ | 3109 | $\begin{array}{lll}2 & 40 \\ 2 & 16 & 2 \\ & 15\end{array}$ |
| $5{ }^{4 .} 5$ | ${ }_{18}^{18} 8^{8,} \mathrm{l}_{20}$ | $\begin{array}{ll}17 & 17 \\ 21 & 21\end{array}$ | 1 7  <br> 2 16  <br>  17  | $\begin{array}{llll}2 & 13 & 12\end{array}$ | ${ }_{21}^{61}{ }^{0}{ }_{20}$ | $\begin{array}{lll}0 & 59 & \\ 1 & 46 \\ 1 & 4 & 40\end{array}$ | $\begin{array}{llll}1 & 30 \\ 2 & 30 \\ 24 & 33 \\ 23\end{array}$ | $0{ }^{1} 12{ }^{12}$ | $\begin{array}{llll}0 & 46 & 47 \\ 1 & 9 & 12\end{array}$ |  | 3 5. ${ }^{1}$ |
| $\mathrm{O}_{38}{ }^{7}{ }^{\text {, }} \mathrm{L}_{40}$ | $\begin{array}{ll}18 & 20 \\ 16 & 19\end{array}$ | $\begin{array}{\|cc\|}21 & 21 \\ 6 & 6\end{array}$ | $\begin{array}{rrrr}1 & 1 \\ 3 & 7 & 7\end{array}$ | $15 \quad 13$ | 24 | 1   <br>  42  <br> 2 22 80 <br>  38  | $\begin{array}{llll}2 & 24 \\ 3 & & \\ 14 & 23 \\ 5 & 15\end{array}$ | $\begin{array}{llll}1 & 30 & 32 \\ 2 & 27 & 32 \\ & 27\end{array}$ | $\begin{array}{llll}2 & 4 & 5 \\ 3 & 4 & 5 \\ 4 & 4 & 5\end{array}$ | $\bigcirc 10$ |  |
| $\begin{array}{lll}1 & 26 & 26\end{array}$ | $20 \quad 22$ |  | 8 |  | $\begin{array}{ccc}2 \\ 3 & 21 \\ 3\end{array}{ }_{3}{ }^{23}$ | $\begin{array}{lll}3 & 35 & 36 \\ 1 & 25 & 36 \\ 1 & 8 & \end{array}$ | $\begin{array}{llll}4 & 10 \\ 5 & 11\end{array}$ | $\begin{array}{lll}3 & 23 & 14 \\ 4 & 4 . & \end{array}$ | ${ }^{3}{ }_{4}$ | (1809 |  |
| $\begin{array}{lll}1 & 31 & 31 \\ 4 & 16 & 31 \\ 4 & 16 & 17\end{array}$ | $\begin{array}{lll}3 & 27 & 25 \\ 4 & 13 & 11\end{array}$ | ${ }_{29}^{2,9} \mathrm{~L}_{30}$ |  | 19 | $4{ }^{4} 9$ | $\begin{array}{ccc}1 & 25 & 26 \\ 5 & 8 & 7\end{array}$ |  | 4 - |  |  | ${ }^{9}$ 9 ${ }^{9}$ |
| $\begin{array}{llll}4 & 16 & 17 \\ 5 & 4 . & 2\end{array}$ |  | $\begin{array}{ll}25 & 25 \\ 21\end{array}$ | 23 |  | $\begin{array}{llll}5 & 40 \\ 6 & 50 \\ 50\end{array}$ |  | 8, 4, 4 | 9,7 | ${ }^{11} \mathrm{C7}^{2} \mathrm{l}^{\mathrm{L}}{ }_{48}$ | ${ }_{5}^{12,8.8}$ |  |
|  | $0^{1,9}{ }^{\text {a }}$, | $\begin{array}{ll}23 & 22 \\ 8 & 10\end{array}$ | $\begin{array}{llll}2 & 20 & 20\end{array}$ | 5, ${ }^{0}$ |  | - ${ }_{19}{ }^{2+}{ }^{21}$ | $16 \quad 17$ |  | $23 \quad 21$ | $5 \cdot$ |  |
| ${ }_{17}{ }^{8}, 4$ | $19 \quad 20$ | 111 | 50.4 | $24 \quad 24$ | $6_{29}{ }^{1,1} L_{29}$ |  | $18 \quad 19$ | $\frac{1}{2}$ | $\begin{array}{lll}2 & 21 & 20 \\ 3 & 11 & 12 \\ 3 & 1 & \end{array}$ |  | ${ }_{0}{ }^{15} \mathrm{id}_{4} \mathrm{~L}_{1} \mathrm{~L}_{13}$ |
| $13 \quad 16$ | 2 14 17 <br> 3 9 8 <br>  13 12 |  |  | $25 \quad 26$ |  |  | $\begin{array}{lll}3 & 16 & 17 \\ 4 & 17 & 13\end{array}$ | 3 | $\begin{array}{llll}4 & 15 & 15\end{array}$ | ${ }_{38}{ }^{\circ}$ | $4{ }^{4 \cdot}$ |
| $\begin{array}{llll}2 \\ 3 & 24 & 24 \\ 3 & 13 & \\ 13\end{array}$ | $\begin{array}{lll}4 & 13 \\ 4 & 13\end{array}$ | ${ }^{2,70,4} 5$ | $0_{1}{ }_{4}{ }_{4}$ | $\begin{array}{llll}4 & 15 & 16 \\ 5 & 4 . & \\ 3\end{array}$ | $\begin{array}{lll}2 & 30 & 31 \\ 3 & 44\end{array}$ | $\begin{array}{llll}4 & 29 & 31 \\ 4 & 4 & 4\end{array}$ | $\begin{array}{ccc}4 & 6 & 13 \\ 5 & 6 & 6\end{array}$ |  |  | $2{ }^{2}$ 16 16 | 2808 |
| $\begin{array}{rrr} \\ 4 & 13 & 13 \\ 4 & 7 & 6\end{array}$ |  | 40  <br> 20 18 <br> 18  | $\begin{array}{ccc}1 & 9 & 9 \\ 2 & 13 & 14\end{array}$ |  | $\begin{array}{llll}3 & 48 \\ 4 & 21 & 45 \\ & 22\end{array}$ | 540 |  | - ${ }_{21}{ }^{21} 82$ | ${ }_{25}^{1,3, L_{24}}$ | $\begin{array}{ccc}3 & 11 & 11 \\ 4 & 8 & 6\end{array}$ | 15, 3, 4 |
|  | $\bigcirc{ }^{1,10,2}$ | 20  <br> 7 7 <br> 18  |  |  | 59 | 7, 3, L | ${ }_{\text {is }}{ }^{5,4}{ }^{1}$ | $\begin{array}{ll}1 \\ 2 & 15 \\ 2 & 19\end{array}$ | $23 \quad 23$ |  | ${ }^{8} 8$ |
| $\mathrm{ig}^{9} \mathrm{l}_{17}$ |  |  | ${ }_{41}{ }^{0,2}{ }_{41}$ |  | 6 5- |  | 113 | ${ }^{1} \cdot{ }^{\circ}$ | $\begin{array}{lll} \\ 2 & 26 & 27 \\ 3 & 26 & 27 \\ 3 & 10 & 10\end{array}$ | $0_{0}^{13,1, ~}{ }^{15}$ | $\begin{array}{llll}2 & 11 & 10\end{array}$ |
| ${ }_{2}{ }^{1} 8.8$ | $\begin{array}{lll} \\ 3 & 40 & 3 \\ & 9 & 10\end{array}$ | $2_{7}{ }^{11} .{ }^{\text {L }}$, | $\begin{array}{lll}41 & 41 \\ 38 & 34\end{array}$ | $\begin{array}{lll}0 & 49 & 48 \\ 1 & 51 & 50\end{array}$ | 6, 2, L | $\begin{array}{ccc}1 & 52 & 33 \\ 2 & 7 & 7\end{array}$ | 2 11 12 <br> 3 6 5 <br> 3 5  |  | - 21 | $\begin{array}{llll}1 & 39 & 39\end{array}$ |  |
| $\begin{array}{lll}3 & 40 & 2 \\ 4 & 5 & 3\end{array}$ |  | $12 \quad 13$ | 448 | $\begin{array}{llll}1 & 57 & 50 \\ 2 & 23 & 22\end{array}$ |  | 3 2222 | $\begin{array}{llll}4 & 18 & 16 \\ 4\end{array}$ | ${ }_{12}{ }_{12} \mathrm{P}^{4} \mathrm{~L}_{12}$ |  |  | $7_{7}^{4,}{ }^{\text {L }} 8$ |
|  |  | $15 \quad 14$ | $\begin{array}{lll}46 & 47 \\ 17 & 16\end{array}$ | $\begin{array}{ccc}1 & 3 & 50 \\ 3 & 36 & 35 \\ 4 & 8 & 9\end{array}$ | $\begin{array}{lll}33 \\ 10 & 10\end{array}$ | $\begin{array}{lll}4 & 32 & 33 \\ 3 & 13\end{array}$ | $3{ }^{11} 12$ | 12.10 |  | 41615 | $1 \begin{array}{lll}10 & 8\end{array}$ |
| $\bigcirc{ }^{\circ}$ is ${ }^{2}$ | $\begin{array}{lll}1 & 15 & 13\end{array}$ | 3. 0,1 | $8{ }^{8}$ | $5{ }_{5} 989$ | $3 \begin{array}{lll}3 & 18 & 19\end{array}$ |  |  |  | ${ }^{11} 13^{4},{ }^{2}$ |  | 10.10 |
| $1{ }^{1} 86.6$ | $25^{\circ} 4$ |  | 10 | $6 \quad 9 \quad 10$ | ${ }_{5}^{4} \times 14.017$ |  | $\begin{array}{llll}0 & 11 & 12\end{array}$ | 10,0 | $28 \quad 28$ | ${ }_{12}{ }_{12}{ }^{2}$, ${ }_{11}$ | , |
| $\begin{array}{lll}2 & 4 * & 5 \\ 3 & 4 & 6\end{array}$ | $2,0, \mathrm{~L}$ | $\begin{array}{lll}59 & 59 \\ 24 & 59 \\ 29\end{array}$ | 4, 1 |  | 67 | ${ }^{1.4}{ }^{4}$ | ${ }^{36} 4$. |  | 2 13 14 <br> 3 37  <br> 7   <br> 7   | 76 | 50, ${ }^{5}$ |
|  | $7{ }^{7}$ | $3{ }^{3} 1$ |  | ${ }_{37} 7^{29}$ |  |  | $3{ }^{1} 808$ | $\begin{array}{lll}1 & 15 & 14 \\ 2 & 46 & 47\end{array}$ | $\begin{array}{ccc}3 & 7 & 27 \\ 4 & 6 & 6\end{array}$ | $\begin{array}{ccc}2 & 14 & 14 \\ 3 & 8 & 7\end{array}$ | $11 \quad 11$ |
| $0_{4}{ }^{11}$, L ${ }_{5}$ | ${ }^{116} 7113$ | ${ }_{4}^{27}$. | 30 28 <br> 61 63 | ${ }^{59} 5$ | 6, ${ }^{\text {3, }}$ L 0 |  | $4 \quad 1210$ | $\begin{array}{llll}3 & 34 & 35 \\ 4 & 8 & 3\end{array}$ |  | $\begin{array}{llll}4 & 8 & 8\end{array}$ | 16. ${ }^{\circ}$ |
| $\begin{array}{llll}2 & 11\end{array}$ | ${ }_{3}{ }^{3} \mathrm{~J} \cdot \mathrm{i}$ |  | 3436 | $\begin{array}{llll}3 & 3 \\ 3 & 24 & & \\ 5\end{array}$ |  | $\begin{array}{llll}3 & 9 & 10 \\ 4 & 21 & 19\end{array}$ |  | $\begin{array}{lcc}4 & 8 \\ 5 & 82 & 7 \\ & 20\end{array}$ | $0^{11.4 .5 .5}$ |  |  |
|  | ${ }_{5}^{4} 88$ | ', | 37 | ${ }_{5}^{4} 89.8$ | 39 31 30 32 | 51011 | $\begin{array}{llll}0 & 37 \\ 1 & 34\end{array}$ |  | $\begin{array}{lll}1 & 16 & 17\end{array}$ | $0_{0}^{13,3 .}{ }_{4}$ | $\begin{array}{llll}2 & 16 & 16\end{array}$ |
| ${ }_{87}^{1 \cdot 0,2}{ }_{87}$ | $\begin{array}{lll}5 & 10 \\ 0 & 11 & 10\end{array}$ |  | ${ }_{8}^{20} \quad 20$ | $\begin{array}{ccc}5 & 4^{40} & 5 \\ 0 & 10 & 11\end{array}$ | $\begin{array}{llll}4 & 21 & 32 \\ 5 & 21 & 22 \\ 5 & 10\end{array}$ |  | 1 19  <br> 2 19 17 <br>  19 18 | ${ }^{10}{ }_{30}{ }^{1}, L_{4}^{4}$ | $\begin{array}{ccc}20 \\ 2 & 13 & 13 \\ 3 & 8 & 8\end{array}$ | $\begin{array}{lll}1 & 12 & 11 \\ 2 & 12 & \\ 121\end{array}$ |  |
| ${ }_{7}{ }^{8}$ |  | $\begin{array}{ll}65 & 65 \\ 11 & 12\end{array}$ | , 2 |  | $5 \quad 10 \quad 9$ | ${ }_{25}^{7,}{ }^{3,} L^{26}$ | 3 14 18 <br>  9  |  | 4 | 2 21  <br> 3 4. 21 | $0{ }^{1614}{ }_{14} \mathrm{l}^{2} \mathrm{~L}$ |
| $\begin{array}{llll}3 & 3 * & \\ 4 & 13 & 12\end{array}$ | ${ }^{2} 10$ | $\begin{array}{ll}11 & 12 \\ 23 & 23 \\ 23\end{array}$ | $\begin{array}{lll}62 & 62 \\ 56 & 88 \\ & \\ \end{array}$ | 5., 3. ${ }^{\text {d }}$ \% |  | $\begin{array}{llll}1 & 39 & 41 \\ 1 & 15 & 15 \\ 5 & 15 & \end{array}$ | 49 | $\begin{array}{ccc}2 & 7 & 6 \\ 3 & 22 & 22 \\ 5 & 10\end{array}$ |  |  | $\begin{array}{llll}1 & 19 & 17 \\ 2 & 90 & 7\end{array}$ |
| 13 8 | 10 8 <br> 32 27 | $\begin{array}{ll}23 & 23 \\ 23\end{array}$ | $\begin{array}{ll}56 & 38 \\ 15 & 14 \\ 14\end{array}$ | ${ }^{5} 5808$ |  | 2 14 15 <br> 3 6 6 |  | $\begin{array}{lll}3 & 22 & 22 \\ 4 & 19 & 15\end{array}$ | $0^{11}{ }^{19} 9^{6, L}{ }_{20}$ | 13. 4,5 | 2907 |
| ${ }^{4 *}$ | $\begin{array}{lll}2 & 33 & 33 \\ 3 & 10 & \end{array}$ | ${ }^{4}$ | 2319 | ${ }^{36} 8$ | $\begin{array}{lll}36 & 36 \\ 28\end{array}$ | : $30 \quad 30$ | 0 16 <br> 1 14 <br> 1 14 | $\begin{array}{llll}5 & 7 & 9\end{array}$ | $\begin{array}{llll}1 & 27 & 27 \\ & & 17 & \end{array}$ | $\begin{array}{lll}0 & 10 & 10 \\ 1 & 26 & 26\end{array}$ | , 2 |
|  | $\begin{array}{ccc}3 & 10 & 9 \\ 4 & 20 & 22\end{array}$ | 2, | ${ }^{7}{ }^{7}{ }^{8} 8$ | $\begin{array}{lll}3 & 30 & 6 \\ 4 & 12 & 12\end{array}$ | ${ }^{3}$ 11, 11 | ; 1412 | 1 19  <br> 2 9 9 <br>  9 9 |  | $\begin{array}{lll}2 & 17 & 16 \\ 3 & 12 & 15\end{array}$ | $\begin{array}{lll}2 & 7 & 7\end{array}$ | ${ }_{9}{ }_{8}$ |
| $\bigcirc$ | $5{ }_{5} 23 \quad 24$ | 1818 | $10 \quad 9$ | 5 5 1817 | 4   <br> 5 $\therefore$ 5 |  | $\begin{array}{llll}3 & 10 & 10\end{array}$ | $10,{ }^{2} \cdot$ |  |  |  |
| $\begin{array}{llll}1 & 40 \\ 2 & 20 & 40 \\ & 23 \\ & 30\end{array}$ |  | 11 28 |  |  |  |  |  |  | ${ }^{11}{ }_{4} 7^{4}{ }^{1}$ | ${ }^{13} 3_{27}{ }^{5} \mathrm{~L}_{27}$ | ${ }_{23} 3^{3}{ }^{2}$ |
| 3 39 39 <br> 4 36 39 <br> 8   <br> 49   | , 2 , | 10 | 36 |  |  | $\begin{array}{rrrr}1 \\ -1 & 9 & 7 \\ 1 & 18\end{array}$ | $\begin{array}{llll}0 & 0 & 3\end{array}$ | 2 29 29 <br> 3 9 9 | ${ }^{1}{ }^{\text {4* }}$ | 0 27  <br> 1 15 27 | $1{ }^{1} 1514$ |
| $\begin{array}{lll}4 & 46 & 49 \\ 5 & 35 & 36\end{array}$ |  | $\begin{array}{lll}4 & 5 & 11\end{array}$ | 16 | $\begin{array}{lll}36 \\ 21 & 48 \\ \end{array}$ | 278 | $10^{4 *}$ 5 | $\begin{array}{ccc}1 & 19 . & 18 \\ 2 & 5 & 5\end{array}$ | $12 \quad 12$ | $\begin{array}{ccc}2 & 8 & 7 \\ 3 & 13 & 70\end{array}$ | ${ }_{2}^{2} \quad 14.14$ |  |
| 76 | 4139 | $4 \times$ | $39 \quad 10$ | $30 \quad 20$ |  |  |  | $15 \quad 15$ |  | 3 50 6 |  |

of such bonds on crystallization is the more probable cause of the insolubility of crystals in the chloroform in which they grew from gerrardine generated from its salt. This is in contrast to the proposal of Wright \& Warren (1967) that this is due to zwitterion formation. No hydrogen bonds exist between planes of stacks.

All intermolecular approaches of less than $4 \AA$ are listed in Table 3. None of these, excluding the $\mathrm{O}(1) \cdots \mathrm{H}-\mathrm{O}(2)$ hydrogen bond, is shorter than the expected van der Waals separation. The only atoms not participating in such approaches are $\mathrm{C}(3), \mathrm{C}(7)$, $\mathrm{C}(8)$ and N which are all sited towards the centre of the molecule and are thus shielded.

Table 3. Intermolecular contacts shorter than $4 \AA$

| Atom in parent molecule | Atom in symmetry related molecule | Symmetry operator | Interatomic distance |
| :---: | :---: | :---: | :---: |
| S(1) | $\mathrm{C}(1)$ | I | 3.58 Å |
| S(1) | S(1) | I | 3.98 |
| S(2) | C(11) | II | 3.76 |
| S(2) | $\mathrm{C}(10)$ | III | 3.92 |
| S(2) | C(1) | IV | 3.96 |
| S(3) | C(5) | II | 3.74 |
| S(4) | $\mathrm{C}(5)$ | V | $3 \cdot 80$ |
| S(4) | C(10) | VI | $3 \cdot 88$ |
| S(4) | C(2) | VII | 3.91 |
| S(4) | C(5) | II | 3.98 |
| C(1) | C(11) | VIII | $3 \cdot 88$ |
| C(2) | C(6) | IX | 3.75 |
| C(2) | $\mathrm{O}(2)$ | IX | $3 \cdot 92$ |
| C(4) | O (2) | X | $3 \cdot 42$ |
| $\mathrm{C}(5)$ | $\mathrm{C}(10)$ | III | 3.72 |
| C(5) | $\mathrm{O}(2)$ | X | $3 \cdot 89$ |
| C(6) | O(1) | IV | $3 \cdot 72$ |
| C(9) | O(1) | IV | $3 \cdot 41$ |
| C(11) | $\mathrm{O}(2)$ | X | 3.59 |
| $\mathrm{O}(1)$ | $\mathrm{O}(2)$ | IX | $2 \cdot 75$ |
| Symmetry operator | Translation elements |  |  |
| I | $\frac{1}{2}-x$ | $-y$ | $\frac{1}{2}+z$ |
| II | $-x$ | $y-\frac{1}{2}$ | $\frac{1}{2}-z$ |
| III | $\frac{1}{2}+x$ | $\frac{1}{2}-y$ | $1-z$ |
| IV | $x$ | $y$ | $1+z$ |
| V | $x-\frac{1}{2}$ | $\frac{1}{2}-y$ | $1-z$ |
| VI | $-x-\frac{1}{2}$ | -y | $z-\frac{1}{2}$ |
| VII | $x-\frac{1}{2}$ | $\frac{1}{2}-y$ | - |
| VIII | $-x$ | $y-\frac{1}{2}$ | $-z-\frac{1}{2}$ |
| ${ }_{\text {IX }}$ | $x$ $-x$ | $y$ $\frac{1}{2}+y$ | $z-1$ |



Fig. 2. Projection of the structure down [010].

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