Table 4 (cont.)

| $\mathrm{C}(5)-\mathrm{C}(4), \mathrm{C}(6)$ | 118.4 | -C(13), O(20) | $102 \cdot 3$ |
| :---: | :---: | :---: | :---: |
| -C(4), C(10) | $112 \cdot 3$ | $\mathrm{C}(13)-\mathrm{C}(12), \mathrm{C}(14)$ | 98.7 |
| -C(4), O(5) | $118 \cdot 3$ | -C(12), C(16) | 111.0 |
| -C(6), C(10) | $122 \cdot 1$ | -C(14), C(16) | $104 \cdot 6$ |
| -C(6), O(5) | $61 \cdot 3$ | $\mathrm{C}(14)-\mathrm{C}(8), \mathrm{C}(13)$ | $101 \cdot 6$ |
| -C(10), C(5) | $115 \cdot 3$ | -C(8), C(20) | 108.8 |
| $\mathrm{C}(6)-\mathrm{C}(5), \mathrm{C}(7)$ | 117.9 | -C(13), C(20) | $102 \cdot 2$ |
| -C(5), O(5) | 58.8 | $\mathrm{C}(15)-\mathrm{C}(8), \mathrm{C}(16)$ | $104 \cdot 8$ |
| -C(7), O(5) | $118 \cdot 1$ | $\mathrm{C}(16)-\mathrm{C}(13), \mathrm{C}(15)$ | 108.9 |
| $\mathrm{C}(7)-\mathrm{C}(6), \mathrm{C}(8)$ | 112.3 | -C(13), C(17) | $122 \cdot 4$ |
| $\mathrm{C}(8)-\mathrm{C}(7), \mathrm{C}(9)$ | 109.9 | -C(14), C(17) | $128 \cdot 6$ |
| -C(7), C(14) | $116 \cdot 8$ | $\mathrm{C}(19)-\mathrm{C}(4), \mathrm{N}(1)$ | 114.0 |
| -C(7), C(15) | 108.7 | C(20)-C(10), C(14) | $102 \cdot 6$ |
| -C(9), C(14) | 99.1 | $-\mathrm{C}(10), \mathrm{O}(20)$ | 108.2 |
| -C(9), C(15) | $116 \cdot 0$ | -C(10), N(1) | $113 \cdot 1$ |
| -C(14), C(15) | $106 \cdot 3$ | -C(14), O(20) | $106 \cdot 1$ |
| $\mathrm{C}(9)-\mathrm{C}(8), \mathrm{C}(10)$ | 99.7 | -C(14), N(1) | $121 \cdot 8$ |
| -C(8), C(11) | $105 \cdot 2$ | -O(20), N(1) | $104 \cdot 4$ |
| -C(10), C(11) | 113.3 | $\mathrm{O}(5)-\mathrm{C}(5), \mathrm{C}(6)$ | 59.9 |
|  |  | $\mathrm{O}(20)-\mathrm{C}(12), \mathrm{C}(20)$ | $103 \cdot 6$ |
|  |  | $\mathrm{N}(1)-\mathrm{C}(19), \mathrm{C}(20)$ | 114.1 |
|  |  | -C(19), C(21) | $108 \cdot 9$ |
|  |  | -C(20), C(21) | $113 \cdot 3$ |

Table 5. Intramolecular hydrogen-bond contacts $\leq 3 \cdot 30 \AA$

|  | $\mathrm{H} \cdots X$ | $A-\mathrm{H}^{\prime} \cdots X$ |
| :--- | :---: | :---: |
| $\mathrm{O}(2)-\mathrm{H}(\mathrm{O} 2) \cdots \mathrm{N}(1)$ | $1.94 \AA$ | $136^{\circ}$ |
| $\mathrm{O}(2)-\mathrm{H}(\mathrm{O} 2) \cdots \mathrm{O}(20)$ | 2.57 | 128 |
| $\mathrm{O}(11)-\mathrm{H}(\mathrm{O} 11) \cdots \mathrm{O}(5)$ | 2.90 | 80 |

Molecular packing in the crystal appears to be governed by van der Waals interactions. The relevant contact distances and angles for the two H atoms capable of acting as hydrogen-bond donors are listed in Table 5. There is a weak intramolecular hydrogen bond between $\mathrm{H}(\mathrm{O} 2)$ and $\mathrm{N}(1)$. The $\mathrm{H}(\mathrm{O} 11)$ atom does not bond at all as the only contact within $3.3 \AA$ which is suitable as an acceptor is poorly placed with respect to angle.

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# 1-Phthalimido-1-deoxy-2,3:4,6-di- $O$-isopropylidene- $\alpha$-L-sorbofuranose 

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#### Abstract

C}_{20} \mathrm{H}_{23} \mathrm{NO}_{7}\), orthorhombic, $P 2_{1} 2_{1} 2_{1} ; a=$ 11.776 (1), $b=18 \cdot 162$ (2), $c=9.143$ (1) $\AA ; Z=4, D_{c}=$ $1 \cdot 32, D_{m}=1.33 \mathrm{~g} \mathrm{~cm}^{-3}$. Full-matrix least-squares refinement led to an $R$ of 0.051 . The molecular conformation in the crystal is the same as the preferred conformation in solution. Potential-energy calculations show that attractive London dispersion forces favor this conformation.


Introduction. NMR studies (Glass \& Williams, 1972) of the title compound (I) demonstrated the importance of the conformation illustrated in Fig. 1. Especially revealing was the intramolecular shielding of the hydrogens of the methyl group placed above the phthalimide

[^0]ring. It was suggested that the preference for this conformation may be due to a stabilizing interaction between the O atoms of the 1,3 -dioxane ring and the carbonyl carbons of the phthalimide moiety. Analogous interactions are the subject of considerable interest. Furthermore, such interactions may be of biological significance, for example in the binding of barbiturates (Voet, 1972) and the teratogenic properties of thalidomide (Jönsson, 1972). Thus an X-ray crystallographic structural analysis was performed on (I) to determine the significance of the interaction.
Preliminary X-ray photographs showed orthorhombic symmetry and the following systematic absences were observed: $h 00$ when $h=2 n+1,0 k 0$ when $k=$ $2 n+1$, and $00 l$ when $l=2 n+1$. The space group is thus determined to be $P 2_{1} 2_{1} 2_{1}$. Twelve reflections were centered on a Picker four-angle diffractometer and a least-
squares analysis of these values yielded the cell constants.

Data were collected on a Picker FACS-1 equipped with a monochromator to remove $\beta$ radiation. The radiation used was $\mathrm{Cu} K \alpha$ and the scan was $\theta-2 \theta$ to a maximum $2 \theta$ of $120^{\circ}$. The rate of scan was $1^{\circ} \mathrm{min}^{-1}$ over a range of $2.0^{\circ}$ base width. A dispersion correction was added to this base width to allow for $\alpha_{1}-\alpha_{2}$ splitting. Three reference reflections were measured after every 50 data to determine if the crystal had decomposed or moved. No correction was made for variation of the intensity of the reference reflections; the maximum variation was $4 \%$. 1580 reflections were measured, of which 1312 were considered above a threshold value, their intensity being greater than three times the standard deviation.

The structure was solved with MULTAN (Germain, Main \& Woolfson, 1971). The variance of $F_{o}^{2}$ was calculated from $\sigma^{2}\left(F_{o}^{2}\right)=\sigma_{c}^{2}\left(F_{o}^{2}\right)+\left(0.04 F_{o}^{2}\right)^{2}$, where $\sigma_{c}^{2}\left(F_{o}^{2}\right)$ is determined from the counting statistics and 0.04 is an added factor deduced from the $4 \%$ maximum variation in the intensities of the reference reflections. All non-hydrogen atoms were present on the first $E$ map examined. Refinement was begun and all H atoms were located from difference maps. The largest feature on a final difference map was 0.31 e $\AA^{-3}$. A full-matrix least-squares computer program was used with the temperature factors of the non-hydrogen atoms varying anisotropically while the H atoms had isotropic temperature factors. The final $R$ value ( $R=\Sigma\left|F_{o}\right|-\left|F_{\mathrm{c}}\right| /$ $\left.\Sigma\left|F_{o}\right|\right)$ was 0.051 . The scattering factors used for C, O and N were those of Hanson, Herman, Lea \& Skillman (1964), while the scattering factors for H were obtained from Stewart, Davidson \& Simpson (1965).

The H positions were idealized by means of the following scheme. Methylene hydrogens were assumed to be in a plane bisecting the $\mathrm{X}-\mathrm{C}-\mathrm{Y}$ plane and spread by a tetrahedral angle. For the methyl hydrogens, an ideal methyl group was rotated about the $\mathrm{C}-\mathrm{C}(\beta)$ bond until the residual with the observed methyl hydrogens was minimized. In both cases the ideal bond length was taken as $1.05 \AA$ and the $\mathrm{H}-\mathrm{C}-\mathrm{H}$ angle as $109.5^{\circ}$. Final atomic parameters are in Tables 1 and 2.*

Discussion. The conformation of (I) in the solid state determined by X -ray methods is the same as the preferred conformation in solution as determined by NMR studies. An ORTEP (Johnson, 1965) drawing of the molecule is shown in Fig. 1. Bond length and bond angle data are presented in Fig. 2.

The phthalimide ring is nearly planar. The r.m.s. deviation from the least-squares plane of $\mathrm{N}, \mathrm{C}(7)-\mathrm{C}(14)$ is $0.018 \AA$. The $O$ atoms $[O(7), O(14)]$ appear slightly

[^1]displaced from the plane of the five-membered imide ring by $0.08 \AA$ in the same direction. A similar displacement has been noted for the carbonyl O atoms of the phthalimide ring in $N$-( $\alpha$-glutarimide)-4-bromophthalimide (4-bromothalidomide) (Peterson, 1969). The distance of $\mathrm{C}(19)$, which is the C atom of the shielded methyl group above the phthalimide, from the least-squares plane of the phthalimide ring is $3.8 \AA$. This is close to the sum of the van der Waals radius of a methyl group and the half-thickness of an aromatic


Fig. 1. ORTEP (Johnson, 1965) drawing of (I) showing numbering scheme. Thermal ellipsoids for the non-hydrogen atoms correspond to a $50 \%$ probability level.


Fig. 2. Bond length ( $\AA$ ) and bond angle ( ${ }^{\circ}$ ) data for (I) with standard deviations in parentheses.
molecule ( $3.7 \AA$ ) (Pauling, 1960). The bond lengths and bond angles are very similar for the phthalimide ring in (I) and that in $N$-( $\alpha$-glutarimido)phthalimide (thalidomide) (Allen \& Trotter, 1971). The N atom in (I) is not quite planar, being $0.059 \AA$ out of the plane of the three atoms $[\mathrm{C}(7), \mathrm{C}(14), \mathrm{C}(1)]$ attached to it, but the sum of the angles $\mathrm{C}(7)-\mathrm{N}-\mathrm{C}(14), \mathrm{C}(7)-\mathrm{N}-\mathrm{C}(1)$

Table 1. Atomic positional parameters
The parameters and standard deviations (in parentheses) for the least significant digit of the non-hydrogen atoms are $\times 10^{4}$.

|  | $x$ | $y$ | $z$ |
| :---: | :---: | :---: | :---: |
| N | -779 (4) | 4519 (2) | 5133 (4) |
| $\mathrm{O}(2)$ | -3545 (3) | 4281 (2) | 3275 (4) |
| $\mathrm{O}(3)$ | -3366 (3) | 3052 (2) | 3166 (5) |
| $\mathrm{O}(4)$ | -323 (3) | 3113 (2) | 2820 (4) |
| $\mathrm{O}(5)$ | -1759 (3) | 4399 (2) | 2205 (4) |
| O(6) | 412 (4) | 3827 (2) | 964 (4) |
| $\mathrm{O}(7)$ | -960 (4) | 3507 (2) | 6587 (4) |
| $\mathrm{O}(14)$ | -69 (4) | 5573 (2) | 4072 (5) |
| C(1) | -1957 (5) | 4621 (3) | 4728 (6) |
| C(2) | -2348 (4) | 4154 (3) | 3463 (6) |
| C(3) | -2224 (4) | 3316 (3) | 3495 (6) |
| C(4) | -1484 (5) | 3135 (3) | 2234 (6) |
| C(5) | -1616 (5) | 3775 (3) | 1238 (6) |
| C(6) | -631 (5) | 3911 (3) | 229 (6) |
| C(7) | -385 (5) | 3977 (3) | 6057 (6) |
| C(8) | 846 (5) | 4129 (3) | 6284 (6) |
| C(9) | 1665 (5) | 3740 (4) | 7047 (7) |
| $\mathrm{C}(10)$ | 2711 (7) | 4022 (4) | 7112 (8) |
| $\mathrm{C}(11)$ | 3016 (6) | 4664 (4) | 6344 (8) |
| $\mathrm{C}(12)$ | 2207 (5) | 5055 (3) | 5572 (7) |
| C(13) | 1107 (5) | 4757 (3) | 5515 (6) |
| C(14) | 88 (5) | 5024 (3) | 4790 (6) |
| $\mathrm{C}(15)$ | -4114 (5) | 3605 (3) | 3588 (6) |
| C(16) | -4340 (6) | 3548 (4) | 5244 (8) |
| C(17) | -5181 (6) | 3574 (4) | 2712 (9) |
| C(18) | 545 (5) | 3167 (3) | 1732 (6) |
| C(19) | 1644 (6) | 3233 (5) | 2608 (9) |
| C(20) | 534 (6) | 2500 (3) | 734 (8) |

Table 2. Idealized hydrogen-atom positional ( $\times 10^{3}$ ) and thermal parameters

|  | $x$ | $y$ | $z$ | Atomic $B$ |
| :---: | :---: | :---: | :---: | :---: |
| H(1) | 147 | 324 | 756 | $10 \cdot 0$ |
| H(2) | 333 | 376 | 776 | 12•1 |
| H(3) | 386 | 485 | 635 | $6 \cdot 2$ |
| H(4) | 241 | 555 | 505 | $4 \cdot 8$ |
| H(5) | -207 | 518 | 444 | $4 \cdot 8$ |
| H(6) | -246 | 449 | 564 | $10 \cdot 0$ |
| H(7) | -493 | 395 | 556 | $10 \cdot 6$ |
| H(8) | -358 | 363 | 582 | $5 \cdot 5$ |
| H(9) | -467 | 302 | 549 | $4 \cdot 5$ |
| H(10) | -499 | 366 | 160 | $9 \cdot 2$ |
| H(11) | -556 | 305 | 284 | $9 \cdot 8$ |
| H(12) | -574 | 398 | 308 | $6 \cdot 1$ |
| H(13) | -191 | 311 | 448 | $2 \cdot 4$ |
| H(14) | -170 | 264 | 172 | $6 \cdot 1$ |
| H(15) | -234 | 370 | 59 | $5 \cdot 9$ |
| H(16) | -67 | 353 | -64 | $6 \cdot 1$ |
| H(17) | -69 | 445 | -18 | $7 \cdot 5$ |
| H(18) | 180 | 274 | 317 | $7 \cdot 9$ |
| H(19) | 157 | 367 | 336 | $4 \cdot 7$ |
| H(20) | 232 | 334 | 189 | $7 \cdot 9$ |
| H(21) | 72 | 203 | 134 | $5 \cdot 8$ |
| H(22) | -27 | 245 | 25 | $9 \cdot 7$ |
| H(23) | 115 | 257 | -9 | $7 \cdot 2$ |

is $359^{\circ}$. The N atom of the phthalimide ring in 4 -bromothalidomide is planar (Peterson, 1969), but that in thalidomide itself is pyramidal (Allen \& Trotter, 1971).
The tetrahydrofuran ring approximates an envelope conformation; starting at $O(5) q=0.324 \AA$ and $\varphi=$ $138.06^{\circ}$ (Cremer \& Pople, 1975). The r.m.s. deviation of the least-squares plane of $\mathrm{O}(5), \mathrm{C}(2), \mathrm{C}(3)$ and $\mathrm{C}(4)$ is $0.015 \AA$ and $C(5)$ is displaced from this plane by $0.499 \AA$. The conformation of furanose rings usually involves puckering of $\mathrm{C}\left(2^{\prime}\right)$ or $\mathrm{C}\left(3^{\prime}\right)$ (Spencer, 1959; Sundaralingam, 1965, 1969; Sundaralingam \& Jensen, 1965) [although sometimes both $C\left(2^{\prime}\right)$ and $C\left(3^{\prime}\right)$ (Sundaralingam, 1965, 1969; Sundaralingam \& Jensen, 1965)]. However, if the furanose is fused to a six-membered ring, as in cyclic adenosine- $3^{\prime}, 5^{\prime}$-phosphate (Watenpaugh, Dow, Jensen \& Furberg, 1968) or cyclic uridine $-3^{\prime}, 5^{\prime}$-phosphate (Coulter, 1969), then $\mathrm{C}\left(4^{\prime}\right)$ is displaced from the plane of the other four atoms of the ribose ring. Such a conformation is analogous to the conformation of the furanose ring in (I). For the 1,3-dioxolane ring in (I) starting at $\mathrm{O}(5) q$ is $0.310 \AA$ and $\varphi$ is $46.56^{\circ}$. Displaced by $0 \cdot 356 \AA$ from the plane of $C(2), C(3)$ and $O(2)$ is $C(15)$, and $O(3)$ is removed by $0 \cdot 140 \AA$ from this plane on the opposite side.
The 1,3 -dioxane ring is in a chair conformation; $q=$ $0.493 \AA, \varphi=356 \cdot 72^{\circ}, \theta=11 \cdot 14^{\circ}$. Substantial puckering occurs in the $\mathrm{O}(6)-\mathrm{C}(18)-\mathrm{O}(4)$ moiety as expected (de Kok \& Romers, 1970; Nader, 1975), whereas the $\mathrm{C}(4)-\mathrm{C}(5)-\mathrm{C}(6)$ portion is flattened relative to cyclohexane, undoubtedly because of its fusion with the five-membered tetrahydrofuran ring. This is best seen by consideration of the torsional angles $\mathrm{O}(6)-\mathrm{C}(18)-$ $\mathrm{O}(4)-\mathrm{C}(4) \quad(58 \cdot 9), \quad \mathrm{O}(4)-\mathrm{C}(18)-\mathrm{O}(6)-\mathrm{C}(6) \quad(-60 \cdot 1)$, $\mathrm{C}(18)-\mathrm{O}(6)-\mathrm{C}(6)-\mathrm{C}(5) \quad(51 \cdot 9), \quad \mathrm{C}(5)-\mathrm{C}(4)-\mathrm{O}(4)-\mathrm{C}(18)$ $(-50 \cdot 5), \mathrm{O}(6)-\mathrm{C}(6)-\mathrm{C}(5)-\mathrm{C}(4)(-42 \cdot 5)$ and $\mathrm{C}(6)-\mathrm{C}(5)-$ $\mathrm{C}(4)-\mathrm{O}(4)\left(41 \cdot 7^{\circ}\right)$.

A major objective of this structural study was to determine whether there is an intramolecular stabilizing electrostatic interaction between the $O$ atoms of the 1,3-dioxane ring and the carbonyl C atoms of the phthalimide ring. The results proffer no evidence for a strong interaction of this type. The relevant nonbonded distances $\operatorname{are} \mathrm{C}(7) \cdots \mathrm{O}(6) 4 \cdot 758, \mathrm{C}(7) \cdots \mathrm{O}(4) 3 \cdot 350, \mathrm{C}(14) \cdots \mathrm{O}(6)$ $4 \cdot 136$, and $\mathrm{C}(14) \cdots \mathrm{O}(4) 3 \cdot 940 \AA$. The sum of the van der Waals radii for O and C is $3 \cdot 1 \AA$ (Pauling, 1960). Thus, there is no evidence for substantial electrostatic attraction.
To gain more insight into the relative potential energies for the conformers of (I) obtained by rotation about the $\mathrm{C}(1)-\mathrm{C}(2)$ bond, calculations were made with the Lennard-Jones 6-12 potential function and the constants listed by Hopfinger (1973). The calculations show that the preferred conformation is near the lowest potential-energy minimum. Further analysis reveals that the preferred conformer is favored over the other minimum-energy conformations (by $3 \cdot 0-3 \cdot 6$ $\mathrm{kcal} \mathrm{mol}^{-1}$ ) principally due to favorable London dispersion forces in the preferred conformer. This result is reminiscent of the greater thermodynamic stability
of the axial over the equatorial isomer of thiacyclohexane l-oxide (Johnson \& McCants, 1964; Martin \& Uebel, 1964; Lambert \& Keske, 1966) because of attractive van der Waals interaction between the axial $O$ atom and the syn-axial hydrogens (Johnson \& McCants, 1964; Allinger, Hirsch, Miller \& Tyminski, 1969). Although it is reasonable to ascribe the conformational preference in (I) to attractive van der Waals interactions it should be emphasized that the calculations for (I) did not include energy minimization, e.g. by rotation about bonds other than $C(1)-C(2)$, or entropy considerations. Furthermore, no attempt was made to calculate electrostatic interactions.

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# $S$-Carboxymethyl-L-cysteine Sulfoxide (Configuration 2R:4R) 

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#### Abstract

C}_{5} \mathrm{H}_{9} \mathrm{SO}_{5} \mathrm{~N}\), orthorhombic, $P 2_{1} 2_{1} 2_{1} ; a=$ 4.786 (2), $b=8.312$ (1), $c=18.914$ (5) $\AA ; Z=4, D_{x}=$ $1.723, D_{m}=1.732(5) \mathrm{g} \mathrm{cm}^{-3}$ (flotation at $21^{\circ} \mathrm{C}$ ). The structure has been determined by standard Fourier techniques from X-ray diffractometer data and refined by least-squares methods to $R=0.035$ for 924 independent reflections. As found by X-ray analysis, the title compound exists as an 'apparent' zwitterion. The cysteine carboxyl and the methyl carboxyl groups of adjacent molecules are involved in a very strong hydrogen bond [O . . O $2 \cdot 449$ (3) $\AA$ ].


Introduction. The title compound and an epimeric sulfoxide were prepared by oxidation of $S$-carboxymethyl-L-cysteine (SCMC). Fractional crystallization from water was used to separate and purify the two epimers. The title compound was the first epimer to crystallize from solution (analysis, calculated for $\mathrm{C}_{5} \mathrm{H}_{9} \mathrm{SO}_{5} \mathrm{~N}$ : $30.77 \% \mathrm{C}, 7.17 \% \mathrm{~N}, 4.64 \% \mathrm{H}$; composition found: $30.96 \% \mathrm{C}, 6.37 \% \mathrm{~N}, 4.78 \% \mathrm{H})$. Precession photographs showed mmm symmetry. Systematic absences ( $h 00, h=2 n+1 ; 0 k 0, k=2 n+1 ; 00 l, \quad l=2 n+1$ ) observed on the films and verified on the diffractometer


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[^1]:    * A list of structure factors and the anisotropic thermal parameters for the heavy atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31892 ( 9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

