ture-factor amplitudes calculated. The independent reflections thus calculated yielded $R = 17\%$ when compared with those for 8-azaguanine ($R = \sum ||F_A| - |F_M|| / \sum |F_A| \times 100$, where F_A = structure-factor amplitudes for 8-azaguanine [Sletten *et al.*, 1968], and F_M = structure-factor amplitudes for the mixed crystals).

Table 1. *Unit-cell parameters for* 8-*azaguanine* (*Sletten et al.*, 1968), *guanine* (*Thewalt et al.*, 1971) *and for the mixed crystal*

	Mixed crystal	8-Azaguanine	Guanine
a	3·57 (3) Å	3·5629 (5) Å	3·645 (5) Å
b	11·37 (11)	11·4404 (9)	11·277 (8)
c	16·32 (16)	16·4685 (16)	16·510 (8)
β	95·33 (9)°	95·13 (1)°	96·8 (1)°
Space group	$P2_1/c$	$P2_1/c$	$P2_1/n$
Z	4	4	4
D_x	~1·68 g cm^{-3}	1·691 g cm^{-3}	1·67 g cm^{-3}
D_m	1·65	1·687 (8)	—

The presence of both guanine and 8-azaguanine in these crystals was confirmed by dissolving exhaustively washed crystals and separating the compounds by column chromatography on Sephadex G-10 (Sweetman & Nyhan, 1968). Optical density measurements on the separated components indicated a two-to-one ratio, azaguanine to guanine, of the compounds in the mixed crystals.

A comparison of the crystal structures of 8-azaguanine monohydrate and of guanine monohydrate (Thewalt, Bugg & Marsh, 1971) has shown that guanine can substitute in the azaguanine lattice by a single change in the hydrogen-bonding scheme at C(8) [N(8) for 8-azaguanine], as noted by Thewalt *et al.*, 1971. Such a change can occur by adjusting the position of the water molecule in the asymmetric unit, and this is presumably the mechanism for the formation of the mixed crystal. I therefore propose that in the mixed crystal, molecules of guanine and 8-azaguanine occupy isomorphous positions, and that the difference between an asymmetric unit containing guanine and one containing 8-azaguanine lies in the position of the water molecule in the asymmetric unit and in the hydrogen-bonding scheme between position 8 of the base and the water molecule. No further work on the three-dimensional, X-ray analysis of the structure is contemplated.

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The N–S bond lengths in the isomers $S_6(NH)_2$. Refinement of the crystal structure of $S_6(NH)_2$–II.

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Refinement of the crystal structure of $S_6(NH)_2$–II has shown that the average length of the N–S bonds in $S_6(NH)_2$–II, 1·680 Å, is approximately equal to the length found in $S_6(NH)_2$–III, 1·672 Å, but smaller than the value observed in $S_6(NH)_2$–I, 1·724 Å. As suggested in an earlier paper the large value observed for the latter compound may be due to disorder.

In a previous paper (Postma, van Bolhuis & Vos, 1971; hereafter referred to as PBV) the average elongation of 0·052 Å of the N–S bonds in $S_6(NH)_2$–I relative to those in $S_6(NH)_2$–III has tentatively been ascribed to disorder in $S_6(NH)_2$–I. The N–S values in $S_6(NH)_2$–II determined by Weiss (1960) were not sufficiently accurate to be compared

Table 1. *Crystallographic data and experimental details*

Data	Method
Space group *Pnma*, $Z = 4$	Weissenberg photographs.
$a = 7·873$ (1) Å	Weissenberg photographs of zero-layer lines;
$b = 12·858$ (2)	$\lambda(\text{Cu } K\alpha) = 1·5418$, $\lambda(\text{Cu } K\alpha_1) = 1·54051$,
$c = 7·390$ (1)	$\lambda(\text{Cu } K\alpha_2) = 1·54433$ Å. Calibration with NaCl reflexion spots. Least-squares adjustment of 17 $0kl$ and 22 $hk0$ reflexions.
Crystal size	$0·46 \times 0·44 \times 0·55$ mm.
1682* reliable intensities hkl	Computer automated Nonius four-circle diffractometer, Mo-radiation, Zr-filtered, θ–2θ scan, $\sin \theta / \lambda \le 0·995$ Å^{-1}.
1682 reliable $F(hkl)$ values	Correction for L.P. and for absorption ($\mu = 16·5$ cm^{-1})†

* Not including the strong reflexion 020, which was omitted as its intensity was badly influenced by streaks.

† According to the Busing & Levy (1957) scheme.

with those in the other isomers. Therefore the crystal structure of this compound has been refined. The determination of the cell dimensions, the intensity measurements and the refinement of the structure were done in essentially the same way as described for $S_6(NH)_2$–III in PBV. Some experimental details are given in Table 1. In the final stages of the refinement only reflexions with $|F_c| > 1$ and $F_o > 3\sigma$ (where σ is the standard deviation in F, based on counting statistics) were considered. An extinction correction according to Zachariasen (1968) was applied, amounting to 20% in F for the strongest reflexions 040 and 210. The weighting scheme was $w = (w_c^{-1} + p|F|^2)^{-1}$ with $p = 0.0007$. The final coordinates and anisotropic thermal parameters are given in Tables 2 and 3. The N–H bonds were assumed to lie in the S–N–S planes. Their lengths were kept fixed at 0.99 Å. The hydrogen temperature factors were assumed to be isotropic and equal to exp $(-3.7 \sin^2 \theta/\lambda^2)$.

Table 2. *Final relative coordinates*

The standard deviations in parentheses were calculated by the least-squares program and are given in units of the last decimal place. For numbering of the atoms see Fig. 1.

	x	y	z
S(1)	0.12273 (5)	0.13499 (4)	0.23294 (7)
S(2)	0.20291 (6)	0.09317 (4)	0.48710 (7)
S(3)	0.01944 (7)	0.13554 (4)	0.67245 (8)
N(1)	0.2091 (3)	0.25*	0.1749 (3)
N(2)	0.0737 (3)	0.25	0.7645 (3)
H(1)	0.32	0.25	0.10
H(2)	0.14	0.25	0.88

* Located on mirror plane.

For the 1575 reflexions used in the last stages of the refinement we obtained $R = (\sum |\Delta F|^2/\sum |F_o|^2)^{1/2} = 0.043$, $R_w = (\sum w|\Delta F|^2/w|F_o|^2)^{1/2} = 0.064$. A list of observed and calculated structure factors is available on request. In the final difference map maxima and minima with 'heights' varying from -0.42 to 0.66 eÅ^{-3} (e.s.d. 0.10 eÅ^{-3}) occur at about 0.8 Å from the sulphur atoms. This may be due to the difference in weighting scheme with the least-squares refinement and possibly also to inadequacies in the model used for the atomic vibrations. Analysis of the anisotropic thermal motion of the sulphur and nitrogen atoms (for method see PBV) showed that the molecules of $S_6(NH)_2$–II cannot be considered as rigid bodies. Therefore no corrections for thermal motion have been made to the bond lengths and angles.

The structure in [100] projection is shown in Fig. 1. In contrast to the structure of $S_6(NH)_2$–III where strong intermolecular interactions were observed, the molecules of $S_6(NH)_2$–II are separated by almost normal van der Waals

distances. Only the distances $S(2,x,y,z) \ldots S(3,\bar{x},\bar{y},1-z) = 3.62$ Å and $S(2,x,y,z) \ldots S(3,\frac{1}{2}+x,y,1\frac{1}{2}-z) = 3.58$ Å are slightly shorter than the sum of the relevant van der Waals radii $[2r(S) = 3.70$ Å, Pauling, 1960]. As observed for the other compounds with formula $S_{8-x}(NH)_x$ the molecules in $S_6(NH)_2$–II have the crown conformation. The dihedral angle between the best planes through the upper and lower four atoms is $0.36°$, the best height (for definition see PBV) is 1.018 Å.

The bond lengths and angles are given in Table 4. The S–S bonds, 2.058$_5$ Å on average, may be compared with those in $S_6(NH)_2$–III, average value 2.055 Å, and in $S_6(NH)_2$–I, average value 2.048 Å. The average value of the N–S bonds, 1.680 Å, lies close to the average values ob-

Table 4. *Molecular geometry*

The standard deviations were calculated from the standard deviations in Table 2 after multiplication by 2.

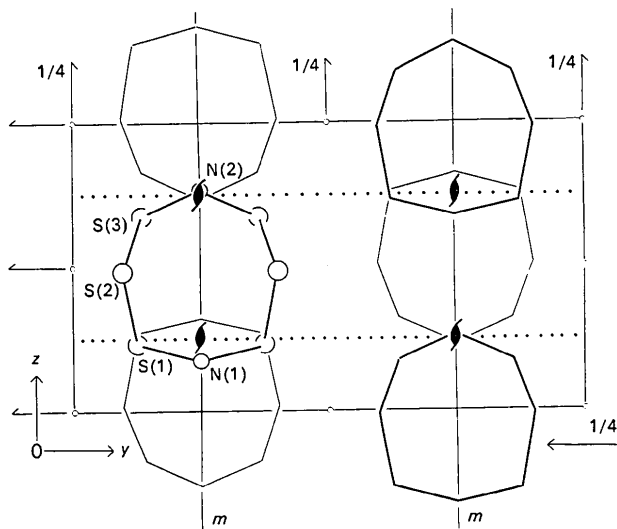| Bond lengths		Valence angles	
N(1)–S(1)	1.683 (3) Å	S(1′)–N(1)–S(1)	122.9 (2)°
S(1)–S(2)	2.053 (2)	N(1)–S(1)–S(2)	109.8 (2)
S(2)–S(3)	2.064 (2)	S(1)–S(2)–S(3)	108.8 (1)
S(3)–N(2)	1.677 (3)	S(2)–S(3)–N(2)	108.8 (2)
		S(3)–N(2)–S(3′)	122.7 (2)

Fig. 1. The crystal structure of $S_6(NH)_2$–II in [100] projection. The (positive) x axis points upwards. Intermolecular distances, smaller than the sum of the relevant van der Waals radii are $S(2,x,y,z)\cdots S(3,\bar{x},\bar{y},1-z) = 3.62$ Å and $S(2,x,y,z)\cdots S(3,\frac{1}{2}+x,y,1\frac{1}{2}-z) = 3.58$ Å.

Table 3. *Thermal parameters*

Temperature factor is given by exp $[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{23}klb^*c^* + 2U_{13}hla^*c^*)]$. Standard deviations as calculated by the least-squares program are given in parentheses. All values are multiplied by 10^4.

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
S(1)	370 (2)	390 (2)	359 (2)	−27 (3)	−163 (3)	−39 (3)
S(2)	399 (2)	404 (2)	465 (3)	192 (3)	163 (4)	4 (4)
S(3)	476 (2)	475 (2)	418 (3)	−47(4)	235 (4)	191 (4)
N(1)	321 (8)	464 (10)	299 (9)	0	0	97 (14)
N(2)	545 (13)	637 (15)	279 (10)	0	0	−25 (19)

served in $S_6(NH)_2$-III, 1·672 Å (PBV) and $S_4(NH)_4$, 1·675 Å (Sass & Donohue, 1958), but is shorter than the value found for $S_6(NH)_2$-I 1·724 Å (van de Grampel & Vos, 1969). As assumed in PBV the relatively large average value observed for $S_6(NH)_2$-I may be due to disorder.

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The crystal and molecular structure of sceleratinic acid.

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The structure of sceleratinic acid, $C_{10}H_{13}ClO_4$, has been determined by three-dimensional X-ray analysis. The crystals are trigonal with $a=7·099$ (1) and $c=19·087$ (2) Å, space group $P3_1$, $Z=3$. The molecule consists of a bicyclic framework which includes two δ-lactone rings.

Sceleratinic acid, $C_{10}H_{13}ClO_4$, occurs naturally as the necic acid of the alkaloid sceleratine (plant origin: *Senecio sceleratus*). By chemical and spectroscopic methods, the acid was shown to have the structure I (Wiechers, 1963; de Waal, Wiechers & Warren, 1963).

I   II
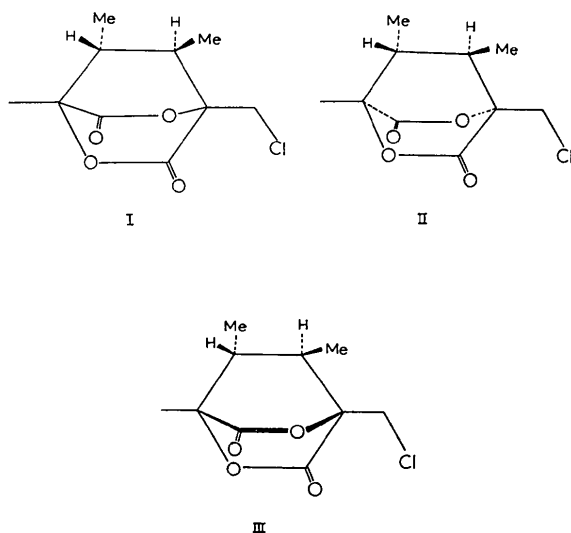
Chemical degradation of the molecule produced (+)-butane-2,3-dicarboxylic acid, thus establishing the stereochemistry of the two bridgehead carbon atoms carrying the methyl groups. Still lacking, however, were the orientations of the two lactone rings for the assignment of the absolute configuration of the molecule (structure II or III). A three-dimensional X-ray analysis was undertaken in order to con-

III

firm the proposed structure and hence assign the absolute configuration.

Colourless pyramidal crystals of sceleratinic acid were obtained by recrystallization from methanol. Oscillation, Weissenberg and precession photographs showed the crystals to be trigonal, with space group $P3_1$ (000*l* only for $l=3n$).

The other crystal data are.

a 7·099(1) Å
c 19·087(2)
$Dx=1·39$ g cm^{-3}
$D_m=1·38$ (measured by flotation in a mixture of ethanol and bromoform)
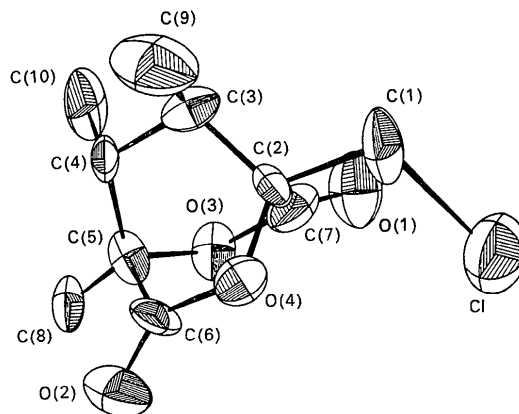M.W. 232·66
$Z=3$
$\lambda=0·70926$ Å.

Fig. 1. Molecular geometry and atomic numbering used in the X-ray analysis.