

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.

We thank Dr J. C. van de Grampel for his interest in this investigation and Drs P. A. Kroon for help during the intensity measurements. The calculations were done at the Computing Centre of the University of Groningen.

Part of the research was supported by the Netherlands Organization for the Advancement of Pure Research (Z.W.O.).

References

- BUSING, W. R. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 180–182.
 GRAMPSEL, J. C. VAN DE & VOS, A. (1969). *Acta Cryst.* **B25**, 611–617.
 PAULING, L. (1960). *The Nature of the Chemical Bond*. 3rd ed. Ithaca: Cornell Univ. Press.
 POSTMA, H. J., BOLHUIS, F. VAN & VOS, A. (1971). *Acta Cryst.* **B27**, 2480–2486.
 SASS, R. L. & DONOHUE, J. (1958). *Acta Cryst.* **11**, 497–504.
 WEISS, J. (1960). *Z. Anorg. Chem.* **305**, 190–197.
 ZACHARIASEN, W. H. (1968). *Acta Cryst.* **A24**, 421–424.

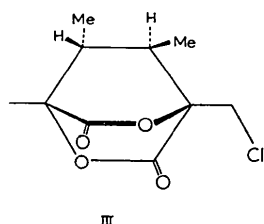
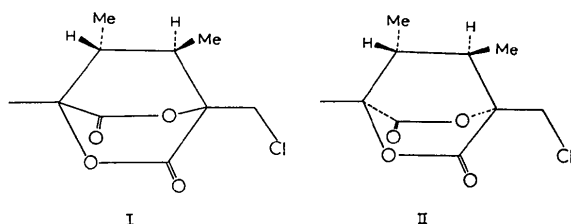
Acta Cryst. (1973). **B29**, 917

The crystal and molecular structure of scleratinic acid. By J. COETZER, *Chemical Physics Group, National Physical Research Laboratory, Council for Scientific and Industrial Research, P. O. Box 395, Pretoria, South Africa* and A. WIECHERS, *Department of Organic Chemistry, University of Pretoria, Pretoria, South Africa*

(Received 19 December 1972; accepted 20 December 1972)

The structure of scleratinic 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.

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



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-

firm the proposed structure and hence assign the absolute configuration.

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

The other crystal data are.

- a 7.099(1) Å
 c 19.087(2)
 $D_x = 1.39$ 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.

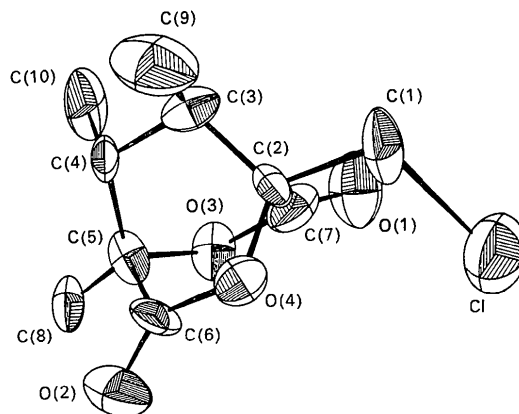


Fig. 1. Molecular geometry and atomic numbering used in the X-ray analysis.

A crystal (≈ 0.01 cm edges) was chosen from which were collected a total of 1013 independent reflexions with $\theta \leq 25^\circ$ using a Hilger and Watts four-circle automatic diffractometer with Mo $K\alpha$ (Zr, β -filtered) radiation. Of these intensities 864 were greater than $3\sigma(I)$, where $\sigma(I)$ is given by $(I_o + I_b)^{1/2}$. I_o is the total number of counts during the peak scan and I_b the number of counts for background intensity.

The intensities were corrected for Lorentz and polarization factors, but not for absorption. The structure was solved by the heavy-atom method.

Using the program, *ORFLS*, of Busing, Martin & Levy (1962) the trial parameters were refined to an R [$R = (\sum ||F_o| - |F_c||) / \sum |F_o|$] of 0.108, with anisotropic thermal factors, and unit weights assigned to all the reflexions. Table 1

Table 2. *Interatomic distances* (Å) *and bond angles* ($^\circ$)

Standard deviations in parentheses.			
C(1)—C(1)	1.84 (2)	C(1)—C(2)	1.54 (2)
		C(2)—C(3)	1.50 (2)
		C(3)—C(4)	1.56 (2)
C(6)—O(2)	1.16 (2)	C(3)—C(9)	1.58 (2)
C(7)—O(3)	1.16 (2)	C(4)—C(5)	1.54 (2)
C(6)—O(4)	1.37 (2)	C(4)—C(10)	1.59 (2)
C(7)—O(3)	1.39 (2)	C(5)—C(8)	1.48 (2)
C(2)—O(4)	1.43 (2)		
C(5)—O(3)	1.48 (2)	C(5)—C(6)	1.52 (2)
		C(2)—C(7)	1.53 (2)
Cl—C(1)—C(2)	109.3 (1.2)	C(4)—C(5)—C(6)	105.2 (1.1)
C(1)—C(2)—O(4)	105.7 (1.2)	C(4)—C(5)—C(8)	120.7 (1.4)
C(1)—C(2)—C(3)	111.4 (1.2)	O(3)—C(5)—C(6)	103.0 (1.2)
C(1)—C(2)—C(7)	110.9 (1.2)	O(3)—C(5)—C(8)	106.1 (1.1)
O(4)—C(2)—C(3)	112.4 (1.1)	C(6)—C(5)—C(8)	113.3 (1.4)
O(4)—C(2)—C(7)	109.2 (1.1)	C(5)—C(6)—C(2)	127.4 (1.6)
C(3)—C(2)—C(7)	107.4 (1.2)	C(5)—C(6)—C(4)	114.3 (1.1)
C(2)—C(3)—C(4)	106.3 (1.1)	O(2)—C(6)—O(4)	118.3 (1.6)
C(2)—C(3)—C(9)	112.5 (1.3)	C(2)—O(4)—C(4)	112.0 (1.0)
C(4)—C(3)—C(9)	110.1 (1.4)	C(5)—O(3)—C(7)	117.0 (1.0)
C(3)—C(4)—C(5)	110.9 (1.1)	O(3)—C(7)—O(1)	120.5 (1.5)
C(3)—C(4)—C(10)	110.7 (1.2)	O(3)—C(7)—C(2)	107.2 (1.4)
C(5)—C(4)—C(10)	110.0 (1.4)	O(1)—C(7)—C(2)	132.2 (1.6)
C(4)—C(5)—O(3)	107.2 (1.1)		

Table 3. *Observed and calculated structure factors*

The columns are k , l , $10F_{\text{obs}}$ and $10F_{\text{calc}}$

k	l	$10F_{\text{obs}}$	$10F_{\text{calc}}$
0	0	1000	1000
0	1	1000	1000
0	2	1000	1000
0	3	1000	1000
0	4	1000	1000
0	5	1000	1000
0	6	1000	1000
0	7	1000	1000
0	8	1000	1000
0	9	1000	1000
0	10	1000	1000
0	11	1000	1000
0	12	1000	1000
0	13	1000	1000
0	14	1000	1000
0	15	1000	1000
0	16	1000	1000
0	17	1000	1000
0	18	1000	1000
0	19	1000	1000
0	20	1000	1000
0	21	1000	1000
0	22	1000	1000
0	23	1000	1000
0	24	1000	1000
0	25	1000	1000
0	26	1000	1000
0	27	1000	1000
0	28	1000	1000
0	29	1000	1000
0	30	1000	1000
0	31	1000	1000
0	32	1000	1000
0	33	1000	1000
0	34	1000	1000
0	35	1000	1000
0	36	1000	1000
0	37	1000	1000
0	38	1000	1000
0	39	1000	1000
0	40	1000	1000
0	41	1000	1000
0	42	1000	1000
0	43	1000	1000
0	44	1000	1000
0	45	1000	1000
0	46	1000	1000
0	47	1000	1000
0	48	1000	1000
0	49	1000	1000
0	50	1000	1000
0	51	1000	1000
0	52	1000	1000
0	53	1000	1000
0	54	1000	1000
0	55	1000	1000
0	56	1000	1000
0	57	1000	1000
0	58	1000	1000
0	59	1000	1000
0	60	1000	1000
0	61	1000	1000
0	62	1000	1000
0	63	1000	1000
0	64	1000	1000
0	65	1000	1000
0	66	1000	1000
0	67	1000	1000
0	68	1000	1000
0	69	1000	1000
0	70	1000	1000
0	71	1000	1000
0	72	1000	1000
0	73	1000	1000
0	74	1000	1000
0	75	1000	1000
0	76	1000	1000
0	77	1000	1000
0	78	1000	1000
0	79	1000	1000
0	80	1000	1000
0	81	1000	1000
0	82	1000	1000
0	83	1000	1000
0	84	1000	1000
0	85	1000	1000
0	86	1000	1000
0	87	1000	1000
0	88	1000	1000
0	89	1000	1000
0	90	1000	1000
0	91	1000	1000
0	92	1000	1000
0	93	1000	1000
0	94	1000	1000
0	95	1000	1000
0	96	1000	1000
0	97	1000	1000
0	98	1000	1000
0	99	1000	1000
0	100	1000	1000

Table 1. Refined atomic parameters ($\times 10^4$)

Thermal parameters are of the form $T = \exp [-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl) \times 10^{-4}]$.
Standard deviations are given in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cl	689 (8)	7918 (9)	0	354 (17)	511 (21)	48 (2)	229 (16)	8 (5)	-55 (5)
O(1)	10649 (20)	9526 (25)	1554 (7)	278 (39)	665 (66)	36 (4)	283 (43)	-32 (10)	-17 (14)
O(2)	3087 (23)	5155 (21)	675 (10)	368 (47)	236 (39)	78 (7)	44 (36)	43 (16)	5 (14)
O(3)	7128 (19)	8160 (20)	1771 (6)	323 (36)	443 (43)	19 (3)	260 (34)	16 (8)	37 (9)
O(4)	6419 (16)	7478 (15)	386 (6)	270 (31)	229 (29)	25 (3)	127 (26)	0 (8)	-21 (8)
C(1)	10023 (27)	10109 (31)	71 (9)	218 (46)	469 (66)	29 (5)	186 (48)	24 (13)	-23 (15)
C(2)	8221 (19)	9477 (20)	626 (8)	111 (30)	186 (38)	21 (4)	45 (28)	9 (9)	12 (10)
C(3)	7649 (27)	11234 (22)	714 (8)	342 (54)	188 (42)	25 (4)	144 (40)	-13 (13)	-12 (12)
C(4)	5742 (19)	10351 (22)	1250 (8)	127 (32)	240 (41)	26 (4)	124 (32)	11 (9)	7 (11)
C(5)	5127 (23)	8043 (26)	1498 (8)	189 (39)	345 (51)	17 (4)	125 (38)	-6 (10)	22 (12)
C(6)	4687 (23)	6718 (21)	832 (9)	173 (41)	129 (37)	37 (6)	1 (33)	13 (12)	26 (12)
C(7)	8957 (31)	9112 (27)	1339 (9)	408 (64)	285 (51)	27 (5)	235 (48)	25 (15)	18 (13)
C(8)	3451 (28)	6927 (34)	2041 (9)	281 (53)	510 (75)	33 (6)	277 (57)	32 (14)	31 (17)
C(9)	6941 (36)	11839 (33)	0 (11)	454 (79)	366 (70)	43 (8)	107 (59)	-18 (20)	54 (19)
C(10)	6364 (32)	11945 (37)	1906 (10)	306 (60)	634 (91)	38 (7)	255 (63)	12 (16)	-73 (21)

lists the final atomic parameters. Bond angles and lengths (Table 2) were calculated with *ORFFE* (Busing, Martin & Levy, 1964). Observed and calculated structure factors are given in Table 3.

The molecular geometry and atomic numbering are shown in Fig. 1. With the stereochemistry of C(3) and C(4) known, the absolute configuration of scleratinic acid is shown to be that of structure III. The three six-membered rings incorporated in the bicyclic skeleton all assume the boat conformation. The C=O bond lengths of 1.16 [C(6)-O(2)] and 1.16 Å [C(7)-O(1)], the C(*sp*²)-O lengths of 1.37 [C(6)-O(4)] and 1.39 Å [C(7)-O(3)], and the C(*sp*³)-O distances of 1.43 [C(2)-O(4)] and 1.48 Å [C(5)-O(3)] respectively, found in the two lactone rings are typical for these atomic pairs (Coetzer & Pieterse, 1972). An average C(*sp*³)-C(*sp*³) interatomic distance of 1.54 Å is obtained, which shows good agreement with the value of 1.545 Å in diamond. The C-Cl bond length of 1.84 Å is slightly longer than normal, e.g. Gabe & Glusker (1971), 1.81 Å.

The shortest approach distance of 3.33 Å is observed between the atoms C(1) and O(2) in molecules which are separated by unit-cell translations in the *a* and *b* directions.

References

- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Report ORNL-TM-306, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- COETZER, J. PIETERSE, M. J. (1972). *Acta Cryst.* B28, 620-624.
- GABE, E. J. & GLUSKER, J. P. (1971). *Acta Cryst.* B27, 1925-1930.
- WAAL, H. L. DE, WIECHERS, A. & WARREN, F. L. (1963). *J. Chem. Soc.* pp. 953-956.
- WIECHERS, A. (1963). Ph.D. Thesis, Univ. of Pretoria, South Africa.

Acta Cryst. (1973). B29, 919

The crystal structure of β -BaSrFe₄O₈. By E. LUCCHINI, D. MINICHELLI and S. MERIANI, *Istituto di Chimica Applicata, Università di Trieste, via Valerio 2, Trieste, Italy*

(Received 2 October 1972; accepted 7 December 1972)

BaSrFe₄O₈, sintered and quenched from temperatures lower than 1180°C, reveals a new β -phase, hexagonal with $a=b=5.448$ and $c=8.091$ Å, space group *P6̄m2* with two formula units per unit cell. The structure of this β -phase was determined by means of isomorphism with the α -BaFe₂O₄ structure, the final *R* value being 0.17. The space group of β -BaSrFe₄O₈ is not that of α -BaFe₂O₄ because of the presence of a strontium atom.

An investigation of the alkaline earth ferrites led to the study of the structure and polymorphism of the reaction products obtained from BaCO₃, SrCO₃ and Fe₂O₃ powder mixtures. A reaction product of composition BaSrFe₄O₈ was reported in an earlier investigation (Meriani & Slocari, 1970). It was obtained by sintering pressed pellet mixtures of barium and strontium carbonates, in a 1:1 molecular ratio, with the calculated amount of Fe₂O₃ at 1200°C. The sintered product was quenched in air from 1200°C. It showed orthorhombic symmetry with the following lattice

parameters: $a=5.516$, $b=8.265$ and $c=9.188$ Å (± 0.001 Å); space group *Pnna*.

A further investigation revealed that when the above mixtures were sintered and quenched from temperatures lower than 1180°C a new phase could be obtained. The diffraction pattern of this new phase, hereafter called β -BaSrFe₄O₈, could be indexed according to hexagonal symmetry. The least-squares refined lattice parameters of this β -phase are $a=b=5.448$ and $c=8.091$ Å (± 0.001 Å) at room temperature. Assuming one formula unit per unit