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.

	x	y	z	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 sceleratinic 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.

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*

$BaSrFe_4O_8$, sintered and quenched from temperatures lower than 1180°C, reveals a new β-phase, hexagonal with $a = b = 5\cdot448$ and $c = 8\cdot091$ Å, space group $P\bar{6}m2$ with two formula units per unit cell. The structure of this β-phase was determined by means of isomorphism with the α-$BaFe_2O_4$ structure, the final R value being 0·17. The space group of β-$BaSrFe_4O_8$ is not that of α-$BaFe_2O_4$ because of the presence of a strontium atom.

An investigation of the alkalinic earth ferrites led to the study of the structure and polymorphism of the reaction products obtained from $BaCO_3$, $SrCO_3$ and Fe_2O_3 powder mixtures. A reaction product of composition $BaSrFe_4O_8$ was reported in an earlier investigation (Meriani & Sloccari, 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 Fe_2O_3 at 1200°C. The sintered product was quenched in air from 1200°C. It showed orthorhombic symmetry with the following lattice

parameters: $a = 5\cdot516$, $b = 8\cdot265$ and $c = 9\cdot188$ Å ($\pm 0\cdot001$ 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-$BaSrFe_4O_8$, could be indexed according to hexagonal symmetry. The least-squares refined lattice parameters of this β-phase are $a = b = 5\cdot448$ and $c = 8\cdot091$ Å ($\pm 0\cdot001$ Å) at room temperature. Assuming one formula unit per unit

cell, the theoretical density is 4·60 g cm⁻³, which compares with the observed value of 4·62 g cm⁻³ measured by pycnometry.

Encouraged by these results, we investigated the possible presence of isomorphism with the high temperature hexagonal form, α-BaFe₂O₄, whose structure was previously reported by Meriani (1972). Intensities were recorded by means of a scintillation counter in conjunction with a pulse-height-analyser, using Co $K\alpha$ radiation and a Siemens diffractometer. The similarity of the diffraction powder pattern with that of α-BaFe₂O₄ suggested this hypothesis. No systematic extinctions were observed for the reflexions collected down to 90° in 2θ. The possible space groups were tested by fixing atoms in special positions according to the model proposed for α-BaFe₂O₄ structure, and comparing experimental and theoretical intensities by means of a computer program written by Smith (1967).

The best agreement was obtained in the space group $P\bar{6}m2$ (No. 187 of International Tables for X-ray Crystallography, 1952). This space group is not the same as that proposed for the α-BaFe₂O₄ model ($P6_322$), and the difference is probably due to the presence of a strontium atom, which reduces the symmetry: strontium atoms were supposed to have replaced alternate barium atoms in a regular fashion along the c axis. The presence of a strontium atom has the effect of generating $00l$ (with $l = 2n + 1$) reflexions, which were not present in the α-BaFe₂O₄ data.

The major discrepancy between calculated and experimental values was noted for the $00l$ reflexions. Furthermore this difference of intensity was not constant and depended on sample preparation. Prolonged grinding produced abnormally high intensities of $00l$ reflexions whereas coarse grained material, obtained by prolonged firing of the pressed pellet, enhanced the $h0l$ reflexions. To justify this phenomenon, preferred orientation is put forward. Great effort was undertaken to reduce orientation effects by sieving powders, which were previously ground and then reheated at about 800°C for several hours, through a 100 mesh sieve directly on the flat sample holder of the goniometer. This technique greatly reduced the abnormally high intensities of the $00l$ reflexions although it did not eliminate certain discrepancies between the calculated and observed intensities of the 111 and, to a smaller extent, the 101 reflexions. By means of this technique the final agreement index, $R = \sum||I_o| - |I_c||/\sum|I_o|$, was reduced to 0·17. Any further refinement of atomic positions was beyond the limit of the available experimental data.

On the basis of the reported crystal parameters and diffraction pattern, the structure of the β-BaSrFe₄O₈ form is considered to be of the same type as that of α-BaFe₂O₄.

In terms of the positions of the space group $P\bar{6}m2$ the proposed atomic positions are:

Ba   in 1(a) 0, 0, 0
Sr   in 1(b) 0, 0, ½
2 Fe(1) in 2(h) ⅓, ⅔, z_1 with $z_1 = 0.245$
2 Fe(2) in 2(i) ⅔, ⅓, z_2 with $z_2 = 0.255$
O(1)   in 1(c) ⅓, ⅔, 0
O(2)   in 1(f) ⅔, ⅓, ½
6 O(3) in 6(n) x, $\bar{x}$, z_3 with $x = 0.515$, $z_3 = 0.260$.

The meaningful interatomic distances, calculated with the computer program of Domenicano & Vaciago (1966) are: Ba–O and Sr–O = 3·14 Å, Fe–O = 1·96 Å, O–O = 2·80 Å and Ba–Fe = 3·69 Å.

The best final data for the observed and calculated intensities and interplanar spacings are reported in Table 1.

Table 1. Debye–Scherrer diagram of β-BaSrFe₄O₈ at 20°C

h k l	d_{obs}	d_{calc}	I_{obs}	I_{calc}
0 0 1	8·154	8·095	6	3
1 0 0	4·727	4·711	3·3	2·7
1 0 1	4·073	4·072	1·2 {	5
0 0 2	4·055	4·047		2·4
1 0 2	3·078	3·070	97	100
1 1 0	2·725	2·720	61	59
0 0 3	2·681	2·698	<1	<1
1 1 1	2·585	2·578	9·1	1
2 0 0	2·348	2·356	1	3
1 0 3	2·336	2·341	1	<1
2 0 1	2·258	{ 2·262	2	2
1 1 2		2·257		
2 0 2	2·037	2·036	21	12·5
0 0 4	2·028	2·024	13	11
1 1 3	1·915	1·915	1	<1
1 0 4	1·863	1·859	2	1
2 1 0	1·778	{ 1·781	1	1
2 0 3		1·774		<1
2 1 1	1·738	1·739	<1	1
2 1 2	1·627	1·630	22	25
1 1 4	1·620	1·624	24	19
0 0 5		1·619	0	0
3 0 0	1·577	1·570	11	8·8
3 0 1		1·542	<1	<1
2 0 4	1·534	1·535	1	2·5
1 0 5		1·531	<1	<1
2 1 3		1·486	<1	<1
3 0 2		1·464	<1	<1
1 1 5		1·391	<1	<1
2 2 0	1·360	1·360	7	7
3 0 3		1·357	<1	<1
0 0 6		1·349	0	0
2 2 1		1·341	<1	<1
2 1 4	1·337	1·337	4·5 {	1
2 0 5	1·334	1·334		1
3 1 0		1·307	<1	<1
1 0 6	1·299	1·297	7	5·2
3 1 1		1·290	<1	<1
2 2 2		1·289	0	0
3 1 2	1·242	1·243	12 {	9·5
3 0 4	1·241	1·241		7·2
2 2 3		1·214	<1	<1

References

Domenicano, A. & Vaciago, A. (1966). Private communication. Ist. superiore Sanità, Roma.
International Tables for X-ray Crystallography (1952). Vol. I. Birmingham: Kynoch Press.
Meriani, S. (1972). Acta Cryst. B28, 1241–3.
Meriani, S. & Sloccari, G. (1970). Acta Cryst. B26, 1184.
Smith, D. K. (1967). UCRL-50264. 12 June 1967. Lawrence Radiation Laboratory, Livermore, California.