

Calciobetafite (new mineral of the pyrochlore group) and related minerals from Campi Flegrei, Italy; crystal structures of polymignyte and zirkelite: comparison with pyrochlore and zirconolite

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Abstract

Polymignyte, zirkelite and zirconolite have often been considered the same mineral. Their occurrence, with calciobetafite (a new member of the pyrochlore group), in a "sanidinite" from Campi Flegrei has allowed their crystal-chemical study and identification as three distinct phases. The three minerals are polymorphs of the compound: $(\text{Ca,Na,REE,Th} \dots)_2 \text{Zr}_2^{VI}(\text{Ti,Nb} \dots)_3^{VI}(\text{Fe,Ti}^{V,IV})\text{O}_{14}$. The crystal structure of zirconolite (space group $C2/c$) has been previously determined (Gatehouse *et al.*, 1981) on synthetic crystals; those of polymignyte (*e.g.*, *Acam*) and zirkelite (*e.g.*, *P3,2*) are described in the present paper. The crystal structures of polymignyte, zirkelite and zirconolite may be derived from that of pyrochlore. Chains, formed by distorted $(\text{Ca,REE} \dots)$ cubes alternating with $(\text{Ti,Nb} \dots)$ octahedra in pyrochlore, are replaced in the remaining minerals by either chains of Zr polyhedra with seven vertices or chains in which $(\text{Ti,Nb} \dots)$ octahedra alternate with distorted $(\text{Fe,Ti} \dots)$ tetrahedra or trigonal bipyramids. The different arrangement of these chains gives rise to the different symmetries of the three phases. The crystal structures of zirkelite and zirconolite are very similar, as they differ only in the stacking of identical pairs of layers of polyhedra. The difficulties in distinguishing the three polymorphs by X-ray powder diagrams are discussed.

Introduction

Calciobetafite, a new species of the pyrochlore group, and related minerals have been found at Monte di Procida (Campi Flegrei, Campania, Italy) in a rock known as "sanidinite". This subvolcanic rock is present in a phreatomagmatic explosion breccia. The age of this pyroclastic formation, determined on an alkali-trachitic obsidian by the K/Ar method (Gillot, pers. comm.) is 0.035 m.y., whereas the absolute age of sanidinite, determined with the same method on the enriched feldspathic fraction, is 0.084 (± 0.008) m.y. (Civetta, pers. comm.). The sanidinite is composed of 75% sanidine (Or_{65}), 16% plagioclase (An_{35}), Mg-hastingsitic amphibole (occasionally with a core of clinopyroxene), biotite, magnetite, apatite and sphene. Minor interstitial glass is also present. Occasional small colored crystals were found scattered throughout the rock: three possible species were tentatively recog-

nized on the basis of their morphology and some optical features: (1) small (0.1–0.2 mm) octahedral crystals, reddish brown in color and isotropic; (2) elongated prisms (maximum 0.4 mm) enclosed in sanidine and sometimes in the interstitial glass bordering the K-feldspar, dark red in color, showing parallel extinction and very weak pleochroism; (3) platy crystals with roughly hexagonal outline; luster resinous, brittle with splintery fracture; black in color and reddish brown in very thin splinters, showing birefringence with extinction parallel to an edge.

By X-ray analyses the three species were identified as a member of the pyrochlore group, polymignyte and zirkelite respectively; the last mineral is always the dominant phase in intergrowths of zirkelite, zirconolite and pyrochlore. Conflicting data have been reported in the literature concerning polymignyte, zirkelite and zirconolite, which have

Table 5. Calciobetafite: thermal vibration ellipsoid parameters;
electrostatic charge balance.

	rmsd (\AA)	U^a	U^b	U^c ($^\circ$)
Me8	0.091(3)	54.74	54.74	54.74
	0.102(3)	--	--	--
	0.102	--	--	--
Me6	0.076(3)	54.74	54.74	54.74
	0.122(3)	--	--	--
	0.122	--	--	--
O	0.068(12)	90	45	135
	0.117(7)	0	90	90
	0.121(8)	90	135	135
(O,F)	0.15(2)	isotropic		

Anions	Bonded cations	Bond strengths §
O	Me8, Me8, Me6, Me6	1.97
(O,F)	Me8, Me8, Me8, Me8	1.75

§ after Donnay and Allmann (1970) .

Estimated standard deviations (in parentheses) refer to the last digit.

Table 6. Calcibetafite: observed and calculated structure amplitudes. (* "unobserved" reflections)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
4	0	0	465.6	463.7	7	7	1	30.7	31.1	11	5	3*	10.9	1.9	8	8	8	235.6	232.8
8	0	0	546.5	546.6	9	7	1	15.1	9.7	13	5	3	20.3	-19.6	8	8	8	355.2	10.9
12	0	0	283.9	233.3	11	7	1	18.4	-16.2	7	7	3	70.9	70.6	8	8	8	52.4	-58.9
2	2	0*	9.9	10.1	9	9	1*	16.5	14.9	9	7	3	32.9	-29.4	7	7	3	23.2	-19.1
6	2	0	69.3	66.2	11	9	1	15.9	11.2	11	7	3	15.7	-9.3	7	7	3	45.3	47.7
10	2	0	41.8	-42.9	2	2	2	656.9	659.5	9	9	3*	21.2	-21.3	10	8	6*	32.1	32.5
14	2	0*	13.1	17.8	4	2	2	62.0	-61.7	4	4	4	477.6	473.9	8	8	6	26.0	-20.6
4	4	0	686.5	639.0	6	2	2	542.8	546.3	6	4	4*	5.9	8.2	6	6	5*	10.7	0.6
8	4	0	403.4	411.9	8	2	2	78.0	81.7	8	4	4	409.0	411.8	6	6	4*	13.7	-6.8
12	4	0	523.9	531.9	10	2	2	381.9	384.4	10	4	4*	5.0	0.4	11	7	5*	14.8	-1.6
6	6	0	76.4	-77.2	12	2	2	42.7	-43.3	12	4	4	285.7	287.1	8	8	5*	16.0	10.9
10	6	0	77.4	83.6	14	2	2	262.9	263.8	6	6	4	59.9	61.9	8	8	4	14.9	9.7
8	6	0	340.8	346.8	4	4	2*	8.5	6.3	8	6	4*	6.4	7.2	6	6	4	35.0	-32.0
12	6	0	258.5	261.9	6	4	2	12.3	-10.0	10	6	4	14.9	9.7	10	6	4	14.9	9.7
10	10	0	47.1	-43.7	6	4	2	13.4	9.9	12	6	4	35.0	-32.0	12	6	4	14.9	9.7
1	1	1	40.4	44.8	10	4	2	26.1	27.3	8	8	4	16.0	10.9	10	8	4	353.1	355.2
3	1	1	47.9	-51.7	12	4	2	16.2	10.7	8	8	4	16.0	10.9	8	8	4	16.0	10.9
5	1	1	53.0	-51.8	6	6	2	449.3	449.2	5	5	5	52.4	-58.9	5	5	5	52.4	-58.9
7	1	1	17.3	17.0	6	6	2	22.1	-22.7	7	5	5	23.2	-19.1	7	5	5	23.2	-19.1
9	1	1	18.4	15.2	9	6	2	334.7	338.5	9	5	5	45.3	47.7	9	5	5	45.3	47.7
11	1	1	16.1	11.0	12	6	2	22.2	17.8	11	5	5	32.1	32.5	11	5	5	32.1	32.5
13	1	1*	17.6	15.0	8	6	2*	13.0	16.8	7	7	5*	26.0	-20.6	7	7	5*	26.0	-20.6
3	3	1	110.1	104.4	10	6	2	14.9	12.4	9	7	5*	10.7	0.6	9	7	5*	10.7	0.6
5	3	3	13.5	-2.4	10	10	2	261.7	261.5	11	7	7	13.7	-6.8	11	7	7	13.7	-6.8
7	3	3	51.6	-62.0	3	3	3	163.2	161.2	9	9	5*	14.8	-1.6	9	9	5*	14.8	-1.6
9	3	3	23.2	-19.3	5	3	3	47.6	-46.1	6	6	6	390.0	391.3	6	6	6	390.0	391.3
11	3	3	5.1	4.3	7	3	3	72.7	-77.8	8	6	6*	2.8	1.4	8	6	6*	2.8	1.4
13	3	3	21.7	20.5	9	3	3	41.1	42.0	10	6	6	295.3	292.5	10	6	6	295.3	292.5
7	5	5	73.0	72.4	11	3	3	39.2	40.5	8	8	6*	14.8	13.6	8	8	6*	14.8	13.6
9	5	5	11.6	5.8	13	3	3	42.7	-40.0	10	8	6	26.7	27.3	10	8	6	26.7	27.3
11	5	5	15.9	-12.8	5	5	3*	20.9	18.4	7	7	7*	30.9	-25.6	7	7	7*	30.9	-25.6
13	5	5	27.1	-25.1	7	5	3*	26.1	25.1	9	7	7	29.8	-25.6	9	7	7	29.8	-25.6
1	5	1*	10.5	11.1	9	5	3*	9.8	-1.2	8	8	8	235.6	232.8	8	8	8	235.6	232.8

Table 8. Polymignyte: thermal vibration ellipsoid parameters.

Atom	rmsd(\AA)	Angle with respect to		
		<u>a</u> ($^{\circ}$)	<u>b</u> ($^{\circ}$)	<u>c</u> ($^{\circ}$)
Me8	0.083(2)	149(9)	90	59(9)
	0.090(2)	59(9)	90	31(9)
	0.094(1)	90	180	90
Me7	0.072(2)	107(8)	163(8)	90
	0.083(2)	17(8)	107(8)	90
	0.094(1)	90	90	180
Me6(1)	0.088(3)	157(14)	113(14)	90
	0.096(2)	113(14)	23(14)	90
	0.114(2)	90	90	180
Me6(2)	0.079(3)	142(14)	90	52(14)
	0.087(3)	52(14)	90	38(14)
	0.087(3)	90	180	90
Me4	0.071(9)	55(2)	145(2)	90
	0.180(7)	90	90	0
	0.194(7)	145(2)	125(2)	90
O(1)	0.081(9)	155(8)	78(16)	111(7)
	0.105(7)	82(15)	15(15)	78(11)
	0.137(7)	67(6)	82(10)	155(7)
O(2)	0.072(10)	45(9)	133(9)	80(6)
	0.110(7)	46(10)	44(10)	91(14)
	0.132(7)	83(11)	98(11)	169(6)
O(3)	0.086(13)	114(24)	156(24)	90
	0.108(11)	24(24)	114(24)	90
	0.134(10)	90	90	180
O(4)	0.092(12)	104(23)	166(23)	90
	0.102(11)	90	90	0
	0.114(10)	166(23)	76(23)	90
O(5)	0.085(12)	90	90	180
	0.096(12)	35(11)	125(11)	90
	0.139(10)	125(11)	145(11)	90

Estimated standard errors (in parentheses) refer to the last digit.

Table 9. Polymignyte: electrostatic charge balance.

Anions	Bonded cations	Bond strengths§
O(1)	Me8, Me6(1), Me6(2), (Me4,Me5)	1.95
O(2)	Me8, Me7, Me7, Me6(2)	1.97
O(3)	Me8, Me8, Me7, (Me4,Me5)	2.07
O(4)	Me8, Me8, Me7, Me6(1)	1.99
O(5)	Me7, Me6(2), Me6(2), Me5	1.99

§ after Donnay and Allmann (1970)

Table 10. Polymignite: observed and calculated structure amplitudes. (* "unobserved" reflections)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
0	0	2	40.7	39.4	0	14	0*	3.4	4.8	1	11	3	150.0	-151.1
0	0	4	441.4	445.9	0	14	2	99.5	101.8	1	11	5	178.2	-176.8
0	0	6	60.7	-54.6	0	14	4	63.3	65.6	1	11	7	111.8	-111.7
0	0	8	510.8	502.9	0	14	6	110.6	108.7	1	11	9	65.4	-34.0
0	0	10*	16.6	-15.1	0	14	8*	18.2	-13.5	1	11	11	68.7	-68.7
0	0	12	245.7	248.4	0	14	10	73.3	74.5	1	13	1	105.2	107.1
0	0	14	93.5	-102.6	0	16	0	155.7	155.6	1	13	3	94.5	95.6
0	2	0	54.1	-50.5	0	16	2	51.8	-49.4	1	13	5	93.3	93.8
0	2	2	57.6	-55.7	0	16	4	177.4	179.4	1	13	7	63.6	63.0
0	2	4	24.8	-22.0	0	16	6	52.7	-52.2	1	13	9	32.2	31.3
0	2	6	24.4	-22.7	0	16	8	116.1	117.2	1	15	1	144.4	-142.8
0	2	8*	0.	5.4	0	18	0	120.1	117.0	1	15	3	146.5	-148.0
0	2	10*	14.4	-10.8	0	18	2	66.6	64.2	1	15	5	102.8	-102.4
0	2	12	26.8	22.6	0	18	4	127.2	125.2	1	15	7	81.4	-80.3
0	2	14	32.0	32.8	0	18	6	36.7	32.9	1	15	9	80.5	-83.5
0	4	0	35.6	36.2	1	1	1*	13.2	-15.5	1	17	1	84.0	82.4
0	4	2	640.9	615.7	1	1	3*	18.5	-16.7	1	17	3	75.8	77.5
0	4	4	55.4	-52.8	1	1	5	20.1	20.9	1	17	5	54.5	54.7
0	4	6	522.2	509.9	1	1	7	29.0	-26.9	1	17	7	53.9	52.9
0	4	8	36.7	36.2	1	1	9	35.5	-32.5	1	19	1	82.5	-83.2
0	4	10	311.0	309.9	1	1	11*	12.9	-9.5	1	19	3	101.7	-103.7
0	4	12	119.4	-119.9	1	1	13	28.9	-29.4	2	0	0*	15.4	-11.1
0	4	14	206.0	213.3	1	3	1	123.1	-124.6	2	0	2	606.5	615.5
0	6	0	76.8	-74.8	1	3	3	37.8	-40.8	2	0	4	42.6	38.6
0	6	2*	4.8	1.6	1	3	5	107.5	-104.7	2	0	6	495.9	487.7
0	6	4*	10.2	11.6	1	3	7	86.5	-85.4	2	0	8	128.6	-129.5
0	6	6	48.5	51.8	1	3	9	52.9	-53.1	2	0	10	356.8	351.3
0	6	8	37.4	-41.1	1	3	11	47.9	-47.7	2	0	12*	23.1	-20.5
0	6	10*	22.5	25.7	1	3	13*	22.4	-25.8	2	0	14	182.5	196.0
0	6	12*	16.3	19.8	1	5	1	43.2	47.2	2	2	0	69.6	-69.2
0	8	0	601.6	589.8	1	5	3	105.5	108.0	2	2	2	47.7	-45.9
0	8	2*	12.4	-6.8	1	5	5	68.2	69.8	2	2	4	83.4	-81.2
0	8	4	395.2	392.5	1	5	7*	17.2	19.2	2	2	6	50.4	-45.2
0	8	6	56.2	-54.7	1	5	9*	9.6	-0.3	2	2	8*	16.4	-16.6
0	8	8	341.4	338.1	1	5	11*	8.1	-2.8	2	2	10*	12.3	12.2
0	8	10	29.0	-26.4	1	5	13*	5.9	-1.1	2	2	12*	16.5	1.4
0	8	12	219.1	222.9	1	7	1	156.2	-157.2	2	4	0	418.7	425.8
0	10	0*	19.8	23.4	1	7	3	172.2	-174.3	2	4	2	27.5	-24.5
0	10	2*	9.6	15.7	1	7	5	95.2	-93.4	2	4	4	651.5	630.7
0	10	4	99.5	104.7	1	7	7	91.6	-91.0	2	4	6*	18.7	-10.4
0	10	6*	18.0	-16.0	1	7	9	92.0	-91.4	2	4	8	355.1	350.3
0	10	8	48.7	48.4	1	7	11	58.3	-61.1	2	4	10	96.3	-97.9
0	10	10*	9.6	9.8	1	7	13	55.1	-55.8	2	4	12	257.3	258.5
0	10	12	71.0	71.6	1	9	1	120.0	122.8	2	6	0	33.3	34.3
0	12	0	90.8	-91.7	1	9	3	102.4	100.2	2	6	2	68.1	-69.4
0	12	2	306.7	309.8	1	9	5	73.6	74.7	2	6	4*	19.2	-16.5
0	12	4*	8.7	-5.7	1	9	7	59.5	59.9	2	6	6	29.8	-25.6
0	12	6	273.3	273.3	1	9	9	40.6	39.7	2	6	8*	22.2	21.7
0	12	8	49.5	-52.8	1	9	11*	16.9	15.8	2	6	10*	0.	-8.5
0	12	10	182.4	137.3	1	11	1	134.8	-133.8	2	6	12*	22.2	23.9

(table 10. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
2	8	7*	13.4	6.8	3	5	7*	17.6	19.9	4	4	4	48.8	-50.0
2	8	2	453.7	451.4	3	5	9*	9.8	8.5	4	4	6	393.5	387.5
2	8	4	47.3	-47.0	3	5	11	33.2	-31.1	4	4	8	44.5	-45.3
2	8	6	352.0	350.9	3	5	13*	24.7	-26.0	4	4	10	249.9	248.4
2	8	8	76.6	-76.9	3	7	1	190.6	-192.5	4	4	12	110.2	-105.4
2	8	10	267.0	269.7	3	7	3	166.6	-169.4	4	6	0	105.2	-109.5
2	8	12	36.8	-40.2	3	7	5	97.7	-95.3	4	6	2	49.5	-52.8
2	10	0*	14.9	13.8	3	7	7	114.3	-113.9	4	6	4	52.1	-54.5
2	10	2	67.8	69.1	3	7	9	115.7	-113.7	4	6	6*	5.0	-4.5
2	10	4	53.3	-53.4	3	7	11	63.8	-65.9	4	6	8	66.9	-64.8
2	10	5	34.4	33.6	3	9	1	91.1	92.0	4	6	10*	4.2	1.2
2	10	8	27.6	28.0	3	9	3	57.6	55.7	4	6	12*	12.4	-2.0
2	10	11	57.2	53.7	3	9	5	31.2	31.0	4	8	0	433.5	430.5
2	10	12*	9.2	-13.6	3	9	7	40.0	39.1	4	8	2	61.2	-62.2
2	12	0	277.2	275.7	3	9	9	32.5	32.2	4	8	4	298.2	299.4
2	12	2	58.7	-56.9	3	9	11*	14.8	5.0	4	8	6	83.2	-83.2
2	12	4	271.8	276.1	3	11	1	189.3	-192.7	4	8	8	260.2	260.5
2	12	6	43.4	-41.6	3	11	3	124.6	-122.6	4	8	10	52.9	-52.3
2	12	8	226.6	225.8	3	11	5	144.4	-145.2	4	10	0*	4.1	3.2
2	12	10	66.8	-64.4	3	11	7	150.9	-149.1	4	10	2	35.0	-36.3
2	14	0	123.1	123.7	3	11	9	103.1	-103.1	4	10	4	28.8	24.5
2	14	2*	6.4	8.8	3	11	11	54.6	-55.0	4	10	6	45.6	-46.8
2	14	4	76.4	75.7	3	13	1	72.6	74.7	4	10	8	32.3	31.9
2	14	6	29.2	31.3	3	13	3	77.3	75.2	4	10	10*	17.0	-15.0
2	14	8	78.0	80.3	3	13	5	65.2	69.9	4	12	0	95.7	-96.0
2	16	0	27.2	-31.0	3	13	7	34.4	34.3	4	12	2	228.0	229.8
2	16	2	158.0	153.7	3	13	9*	19.5	19.9	4	12	4	56.4	-61.4
2	16	4	82.7	-83.7	3	15	1	188.0	-188.4	4	12	6	211.6	212.1
2	16	6	129.4	130.2	3	15	3	116.9	-119.1	4	12	8	57.8	-59.9
2	16	8	33.5	-35.5	3	15	5	76.4	-75.4	4	14	0*	11.9	-4.4
2	18	0	49.8	50.5	3	15	7	115.3	-118.1	4	14	2	57.1	56.6
2	18	2	119.4	120.7	3	17	1	59.5	59.9	4	14	4*	13.6	3.8
2	18	4	33.5	34.1	3	17	3	66.0	63.6	4	14	6	74.6	72.0
3	1	1	22.5	19.9	3	17	5	38.1	38.8	4	14	8*	21.6	-16.1
3	1	3	121.4	-121.0	4	0	0	560.4	627.2	4	16	0	127.7	133.2
3	1	5	69.0	-68.8	4	0	2	41.5	-48.9	4	16	2	72.9	-71.9
3	1	7*	9.2	-7.8	4	0	4	406.4	405.9	4	16	4	115.6	115.3
3	1	9*	24.2	-20.5	4	0	6	87.4	-89.8	4	16	6	70.7	-70.1
3	1	11	47.8	-47.0	4	0	8	354.0	350.6	4	18	0	90.5	90.7
3	1	13	52.2	-54.7	4	0	10	46.6	-45.3	4	18	2	32.5	33.8
3	3	1	104.2	-106.9	4	0	12	227.9	232.9	5	1	1	95.5	-98.4
3	3	3	114.2	-115.8	4	2	0	125.8	-128.8	5	1	3	51.5	-54.2
3	3	5	146.9	-146.1	4	2	2	104.4	-107.0	5	1	5	26.5	-26.0
3	3	7	76.7	-76.5	4	2	4	57.6	-56.8	5	1	7	75.3	-74.9
3	3	9	53.8	-54.9	4	2	6	77.3	-74.5	5	1	9	67.7	-69.6
3	3	11	74.5	-74.4	4	2	8	46.3	-45.5	5	1	11	28.0	-24.0
3	3	13	39.4	-40.3	4	2	10	32.6	-29.6	5	3	1	147.5	-150.3
3	5	1	58.3	57.3	4	2	12*	10.4	9.8	5	3	3	90.2	-90.8
3	5	3*	11.2	10.8	4	4	0	104.8	-108.3	5	3	5	119.3	-119.8
3	5	5*	11.2	-8.5	4	4	2	465.9	469.5	5	3	7	111.8	-111.4

(table 10. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
5	3	9	76.3	-77.5	6	6	0	41.1	-41.6	7	11	3	113.0	-110.9
5	3	11	56.9	-57.2	6	6	2	98.5	-97.2	7	11	5	119.4	-121.7
5	5	1	41.4	-42.0	6	6	4	44.8	-45.2	7	13	1	43.3	44.4
5	5	3	28.6	27.9	6	6	6	53.8	-56.6	7	13	3*	27.4	26.6
5	5	5*	19.5	14.4	6	6	8*	16.2	-18.6	8	0	0	324.8	322.7
5	5	7	33.2	-33.4	6	6	10	34.0	-32.5	8	0	2	53.6	-51.6
5	5	9	41.8	-42.4	6	8	0	41.7	-41.2	8	0	4	302.0	301.6
5	5	11*	18.6	-17.7	6	8	2	285.8	284.8	8	0	6	68.2	-67.1
5	7	1	156.7	-157.7	6	8	4	85.4	-87.7	8	0	8	202.8	199.8
5	7	3	132.9	-134.1	6	8	6	232.0	232.8	8	2	0	82.9	-82.5
5	7	5	127.8	-130.0	6	8	8	85.8	-82.7	8	2	2	75.8	-76.2
5	7	7	102.7	-104.7	6	8	10	196.8	196.5	8	2	4	47.9	-48.8
5	7	9	96.2	-94.8	6	10	0	63.1	-66.5	8	2	6	59.2	-61.0
5	7	11	73.0	-73.8	6	10	2*	16.2	10.8	8	2	8	38.6	-38.0
5	9	1	31.4	31.1	6	10	4	49.9	-47.4	8	4	0	112.1	-110.1
5	9	3	39.9	36.9	6	10	6*	7.3	-3.9	8	4	2	287.2	287.5
5	9	5	27.7	29.2	6	10	8*	24.6	-26.2	8	4	4*	22.9	-19.1
5	9	7*	16.5	10.0	6	12	0	156.4	157.5	8	4	6	258.0	253.7
5	9	9*	0.	-2.3	6	12	2	80.8	-79.7	8	4	8	74.7	-74.3
5	11	1	129.2	-130.7	6	12	4	198.8	200.9	8	6	0	72.5	-67.7
5	11	3	173.6	-176.1	6	12	6	64.4	-66.3	8	6	2	42.7	-40.5
5	11	5	191.8	-192.6	6	14	0	36.8	38.7	8	6	4	59.2	-59.0
5	11	7	110.0	-109.1	6	14	2*	25.2	-21.9	8	6	6*	13.8	-14.7
5	11	9	58.9	-61.3	6	14	4	58.3	57.2	8	8	0	256.7	257.2
5	13	1	45.4	46.3	6	16	0	82.6	-79.2	8	8	2	60.1	-58.9
5	13	3	43.2	44.2	7	1	1	38.5	-41.7	8	8	4	203.2	207.5
5	13	5	46.3	47.6	7	1	3	79.4	-77.8	8	8	6	69.9	-68.5
5	13	7*	19.8	25.2	7	1	5	59.0	-57.6	8	10	0*	9.9	11.1
5	15	1	127.7	-128.6	7	1	7	36.2	-38.0	8	10	2	36.1	-35.7
5	15	3	165.7	-166.5	7	1	9	35.5	-36.4	8	10	4*	4.0	-7.5
5	15	5	132.6	-130.0	7	3	1	108.5	-105.7	8	12	0	72.9	-69.5
5	17	1	40.4	39.4	7	3	3	91.1	-89.8	8	12	2	154.8	153.9
6	0	0	22.2	15.6	7	3	5	108.2	-107.0	9	1	1	30.1	-29.7
6	0	2	389.9	390.7	7	3	7	82.4	-81.5	9	1	3	45.1	-44.9
6	0	4	113.3	-113.5	7	3	9	60.6	-59.4	9	1	5*	22.8	-26.8
6	0	6	320.1	17.4	7	5	1*	6.0	1.8	9	3	1	57.8	-55.5
6	0	8	77.1	72.8	7	5	3*	17.9	-14.4	9	3	3	59.9	-63.7
6	0	10	253.1	252.0	7	5	5	27.1	-25.5	9	3	5	78.4	-78.3
6	2	0	37.7	-89.0	7	5	7*	2.7	-11.1	9	5	1*	12.6	-3.9
6	2	2	37.7	-39.2	7	5	9*	21.1	-8.5	9	5	3*	0.	8.6
6	2	4	113.2	-115.1	7	7	1	149.3	-149.8	9	5	5*	9.6	0.8
6	2	6	66.2	-64.4	7	7	3	133.7	-132.4	9	7	1	93.7	-92.4
6	2	8	36.7	-35.7	7	7	5	90.0	-87.2	9	7	3	106.9	-105.8
6	2	10*	22.5	-22.7	7	7	7	100.9	-100.3	9	9	1*	22.7	21.5
6	4	0	328.6	332.2	7	7	9	102.7	-99.0	10	0	0*	18.5	15.3
6	4	2	70.7	-70.0	7	9	1	42.3	40.2	10	0	2	249.6	247.9
6	4	4	352.4	350.0	7	9	3*	17.9	12.3	10	2	0	51.3	-54.0
6	4	6	55.3	-59.3	7	9	5*	14.0	-1.0	10	2	2	39.9	-40.5
6	4	8	278.8	278.2	7	9	7*	20.5	15.6	10	4	0	232.0	229.0
6	4	10	105.2	-100.2	7	11	1	154.5	-152.2	0	2	1	45.1	43.0

(table 10. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
0	2	3	35.3	-35.8	0	18	5*	28.9	-27.5	1	19	2*	2.1	-7.6
0	2	5	114.1	-109.4	1	1	2	47.3	-48.0	1	19	4*	25.3	-26.5
0	2	7	128.5	-129.7	1	1	4	52.4	-55.1	2	2	1	41.1	-42.5
0	2	9*	15.4	3.1	1	1	6	131.6	-132.6	2	2	3	184.0	177.7
0	2	11	73.0	-74.6	1	1	8	109.9	-109.1	2	2	5	80.1	-81.5
0	2	13	41.1	33.3	1	1	10	77.3	-73.9	2	2	7	40.6	-37.9
0	4	1	93.4	94.0	1	1	12	94.6	-97.0	2	2	9	81.1	-79.7
0	4	3	64.6	-66.9	1	1	14	87.3	-90.5	2	2	11	43.9	44.6
0	4	5	109.6	110.5	1	3	2	32.0	32.2	2	2	13	54.7	-54.5
0	4	7*	10.9	-14.5	1	3	4	34.4	83.8	2	4	1	37.3	-38.2
0	4	9	64.4	63.5	1	3	6	70.0	69.3	2	4	3	104.0	105.8
0	4	11*	12.3	-4.0	1	3	8	37.5	85.1	2	4	5	49.7	-51.3
0	4	13	79.9	32.4	1	3	10	105.3	103.1	2	4	7	88.0	87.4
0	6	1	135.3	-131.6	1	3	12	82.2	83.9	2	4	9*	0.	-7.7
0	6	3	122.7	-121.9	1	3	14	76.0	75.0	2	4	11	78.9	78.5
0	6	5	80.5	-80.3	1	5	2	58.2	54.3	2	4	13*	8.7	-1.1
0	6	7	35.1	89.7	1	5	4	31.0	-81.9	2	6	1	105.9	103.8
0	6	9	128.7	-130.4	1	5	6	98.5	-99.8	2	6	3	100.5	-102.9
0	6	11	52.4	-56.1	1	5	8	93.4	-93.0	2	6	5	54.8	-49.7
0	6	13	80.1	-79.9	1	5	10	90.2	-91.7	2	6	7	102.7	-102.4
0	8	1	155.3	-159.1	1	5	12	69.4	-71.0	2	6	9*	17.9	21.4
0	8	3	137.6	138.6	1	7	2	20.5	22.9	2	6	11	78.4	-81.1
0	8	5	41.7	-41.1	1	7	4	31.2	30.3	2	6	13	35.8	-36.8
0	8	7	146.5	148.4	1	7	6	50.2	53.8	2	8	1	110.2	111.2
0	8	9*	19.9	-25.7	1	7	8	68.5	69.2	2	8	3	93.0	-92.8
0	8	11	127.3	126.7	1	7	10	59.2	57.8	2	8	5	150.6	152.0
0	8	13*	0.	-4.1	1	7	12	48.6	49.0	2	8	7	36.5	-36.3
0	10	1*	19.6	-18.5	1	9	2	39.9	-41.0	2	8	9	155.8	155.7
0	10	3	115.3	-116.0	1	9	4	37.3	-37.7	2	8	11*	20.0	-16.7
0	10	5	31.0	32.9	1	9	6	74.1	-73.8	2	10	1*	17.7	-12.0
0	10	7	99.6	-104.9	1	9	8	47.1	-49.0	2	10	3	75.4	80.8
0	10	9	79.6	-82.0	1	9	10	31.4	-29.2	2	10	5	123.9	-124.4
0	10	11	135.1	-136.5	1	9	12	63.8	-66.8	2	10	7	92.4	-92.1
0	12	1	181.9	182.4	1	11	2	25.1	26.6	2	10	9	103.5	-103.1
0	12	3	94.0	-86.2	1	11	4*	2.2	10.2	2	10	11*	17.1	-13.6
0	12	5	157.6	158.2	1	11	6*	4.0	5.7	2	12	1	115.2	-114.3
0	12	7	69.2	-65.7	1	11	8*	10.0	13.7	2	12	3	152.9	154.8
0	12	9	159.9	155.2	1	11	10	27.3	27.8	2	12	5	79.4	-76.4
0	12	11*	17.2	-9.7	1	13	2	30.7	29.6	2	12	7	185.0	181.5
0	14	1	77.4	-77.6	1	13	4*	14.0	-13.9	2	12	9	43.8	-44.3
0	14	3	53.5	-54.5	1	13	6	27.7	-29.3	2	12	11	144.0	147.4
0	14	5	75.8	-77.7	1	13	8	37.5	-35.2	2	14	1	30.1	32.3
0	14	7*	0.	-11.4	1	13	10	32.2	-32.4	2	14	3	59.9	-60.2
0	14	9	104.7	-107.2	1	15	2*	16.4	-15.5	2	14	5	51.8	-50.2
0	16	1	119.0	-120.2	1	15	4*	7.5	-4.3	2	14	7	103.5	-102.5
0	16	3	173.6	173.5	1	15	6*	7.6	-11.0	2	14	9	32.4	-33.5
0	16	5	97.4	-97.2	1	15	8*	18.2	-10.4	2	16	1	109.9	109.0
0	16	7	138.4	140.1	1	17	2*	22.7	-29.2	2	16	3	123.9	-124.7
0	18	1*	0.	3.4	1	17	4*	2.0	-6.0	2	16	5	180.1	180.2
0	18	3	46.7	-47.6	1	17	6*	13.5	12.1	2	16	7	49.2	-48.5

(table 10. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
2	18	1*	26.0	-14.6	4	2	3	25.0	-22.7	5	3	4	52.2	54.7
2	18	3*	9.1	-0.8	4	2	5	69.3	68.5	5	3	5	71.5	68.9
2	18	5	57.1	-58.3	4	2	7	114.3	-115.2	5	3	8	76.0	74.5
3	1	2	132.4	-130.7	4	2	9*	19.8	19.6	5	3	10	80.7	81.9
3	1	4	53.6	-54.6	4	2	11	54.2	-56.6	5	3	12	65.4	65.7
3	1	6	60.6	-59.8	4	2	13	29.2	28.3	5	5	2	65.7	67.2
3	1	8	91.2	-89.7	4	4	1	90.6	91.0	5	5	4	46.9	-48.7
3	1	10	102.1	-102.1	4	4	3	30.7	-31.2	5	5	6	102.3	-101.3
3	1	12	92.2	-95.3	4	4	5	70.8	73.0	5	5	8	72.4	-70.2
3	3	2	37.5	36.8	4	4	7*	14.9	-17.3	5	5	10	49.0	-47.9
3	3	4	80.7	82.1	4	4	9	70.5	73.8	5	7	2*	20.7	22.5
3	3	6	60.1	58.8	4	4	11*	15.0	4.4	5	7	4	29.3	29.8
3	3	8	71.7	72.9	4	6	1	89.3	-86.0	5	7	6	44.0	45.5
3	3	10	101.3	98.4	4	6	3	32.0	-28.1	5	7	8	65.4	65.2
3	3	12	92.3	90.5	4	6	5	99.2	-100.2	5	7	10	54.9	54.9
3	5	2	75.3	-78.1	4	6	7	49.4	50.2	5	9	2*	4.8	4.2
3	5	4	71.7	-71.2	4	6	9	84.3	-85.1	5	9	4*	20.5	-21.4
3	5	6*	10.8	-5.6	4	6	11	30.5	-25.1	5	9	6	71.8	-71.0
3	5	8	32.2	-30.5	4	8	1	102.3	-103.8	5	9	8	35.1	-34.1
3	5	10	129.5	-129.1	4	8	3	130.0	133.7	5	9	10*	12.9	3.0
3	5	12	66.5	-67.9	4	8	5	45.2	-46.8	5	11	2*	25.3	26.4
3	7	2*	11.0	-7.4	4	8	7	124.1	127.6	5	11	4*	2.5	14.4
3	7	4	31.7	36.0	4	8	9*	9.4	-2.4	5	11	6*	14.2	12.3
3	7	6	76.8	76.0	4	8	11	121.4	124.1	5	11	8*	20.1	20.7
3	7	8	58.0	59.6	4	10	1	24.8	25.3	5	13	2	31.9	33.3
3	7	10	46.0	45.0	4	10	3	62.6	-63.2	5	13	4*	8.2	-4.3
3	7	12	53.6	55.0	4	10	5*	12.8	8.3	5	13	6*	22.9	-21.8
3	9	2	104.0	-107.4	4	10	7	105.8	-106.3	5	13	8*	15.4	-17.6
3	9	4	31.1	-29.6	4	10	9	46.3	-43.1	5	15	2*	0.	-7.7
3	9	6*	1.7	-7.5	4	10	11	98.8	-96.2	5	15	4*	11.9	5.3
3	9	8	40.4	-39.8	4	12	1	157.4	157.8	5	15	6*	10.4	-1.1
3	9	10	59.8	-62.4	4	12	3	73.4	-71.6	5	17	2	30.5	-26.6
3	9	12	56.4	-57.4	4	12	5	142.4	144.0	6	2	1	61.5	-63.7
3	11	2*	8.0	4.4	4	12	7	46.5	-48.3	6	2	3	81.4	80.2
3	11	4*	14.5	15.1	4	12	9	153.1	151.5	6	2	5	39.0	-43.9
3	11	6	26.0	26.3	4	14	1	55.7	-52.9	6	2	7*	2.4	2.9
3	11	8*	17.0	18.4	4	14	3	30.0	-27.4	6	2	9	71.3	-70.3
3	11	10*	22.0	18.2	4	14	5	69.2	-70.2	6	2	11	28.0	26.5
3	13	2*	5.4	9.6	4	14	7*	13.5	-12.1	6	4	1	47.4	-47.2
3	13	4*	17.1	-8.8	4	16	1	113.8	-116.5	6	4	3	56.7	55.2
3	13	6*	2.6	-5.2	4	16	3	149.0	149.7	6	4	5*	20.5	-13.6
3	13	8	32.1	-28.6	4	16	5	69.9	-71.4	6	4	7	81.1	78.0
3	15	2*	11.1	-12.7	4	18	1*	4.0	3.1	6	4	9*	17.8	-16.8
3	15	4*	10.8	-2.4	5	1	2*	7.7	6.1	6	4	11	59.3	59.9
3	15	6*	4.9	-4.0	5	1	4	38.8	-38.5	6	6	1	30.2	26.7
3	15	8*	0.	-2.0	5	1	6	117.6	-116.2	6	6	3	105.0	-109.0
3	17	2*	4.5	-9.0	5	1	8	88.8	-89.0	6	6	5*	16.2	16.8
3	17	4*	7.6	-0.7	5	1	10	42.4	-41.1	6	6	7	44.8	-43.0
3	17	6*	14.9	1.9	5	1	12	75.6	-75.9	6	6	9*	11.8	-4.5
4	2	1	65.5	65.3	5	3	2*	11.0	9.3	6	8	1	71.4	68.2

Table 12. Zirkelite: electrostatic charge balance.

Anions	Bonded cations	Bond strengths§
O(1)	Me8(2), Me6(2), Me6(2), Me5	1.85
O(2)	Me8(1), Me8(2), Me7, Me5	2.18
O(3)	Me8(1), Me8(2), Me7, Me6(2)	2.02
O(4)	Me8(1), Me6(1), Me6(2), Me5	1.91
O(5)	Me8(2), Me7, Me7, Me6(2)	2.00
O(6)	Me8(1), Me7, Me7, Me6(1)	2.04
O(7)	Me7, Me6(1), Me6(2)	2.00

§ after Donnay and Allmann (1970)

Table 13. Zirkelite: observed and calculated structure amplitudes. (* "unobserved" reflections)

H	K	L	/F0/	/FC/	H	K	L	/F0/	/FC/	H	K	L	/F0/	/FC/
1	0	0	9.6	10.2	4	1	1*	3.0	11.5	2	1	2	49.8	72.7
2	0	0	12.2	11.5	4	1	-1	29.1	34.7	2	1	-2	11.9	9.2
3	0	0*	16.5	31.4	5	1	1*	6.8	11.3	3	1	2	13.6	15.4
4	0	0	12.3	13.8	5	1	-1*	0.	19.0	3	1	-2	31.5	29.1
5	0	0*	12.7	7.9	6	1	1*	0.	7.8	4	1	2	33.3	31.6
6	0	0	306.9	308.9	6	1	-1*	6.9	3.4	4	1	-2	43.5	48.0
7	0	0*	0.	28.6	7	1	1*	0.	9.6	5	1	2*	3.5	2.9
8	0	0*	1.7	9.1	7	1	-1*	0.	9.1	5	1	-2	18.1	17.7
1	1	0	13.8	14.9	8	1	1*	2.0	6.8	6	1	2*	10.8	10.7
2	1	0*	3.3	7.5	2	2	1	11.5	20.1	6	1	-2	19.2	24.6
3	1	0	19.0	13.3	3	2	1	12.5	13.8	7	1	2*	6.9	19.6
4	1	0	12.2	16.5	3	2	-1	15.9	23.8	7	1	-2*	0.	17.3
5	1	0	25.4	16.9	4	2	1*	9.1	10.4	8	1	2*	0.	6.1
6	1	0*	0.	11.6	4	2	-1	100.3	93.2	8	1	-2*	7.7	16.3
7	1	0	19.6	18.5	5	2	1	40.2	44.5	2	2	2	13.1	10.2
2	2	0	577.5	574.5	5	2	-1*	13.7	21.2	3	2	2	59.8	56.1
3	2	0	22.1	29.4	6	2	1	58.6	63.4	3	2	-2*	7.9	15.1
4	2	0*	2.6	3.5	6	2	-1*	3.1	5.0	4	2	2	331.4	328.4
5	2	0	16.8	20.8	7	2	1*	11.7	22.6	4	2	-2*	2.5	2.6
6	2	0*	4.4	4.1	7	2	-1*	10.5	9.9	5	2	2	47.8	50.3
3	3	0	18.1	18.7	3	3	1*	8.0	3.9	5	2	-2	40.7	41.3
4	3	0	18.8	18.0	4	3	1	11.1	11.4	6	2	2*	8.3	8.5
5	3	0*	0.	7.2	4	3	-1*	0.	21.4	6	2	-2	252.2	248.7
6	3	0*	3.4	17.0	5	3	1*	0.	13.0	7	2	2	15.8	15.3
7	3	0*	0.	4.6	5	3	-1*	6.5	9.2	7	2	-2*	15.6	10.9
4	4	0	259.3	259.1	6	3	1	23.4	23.3	3	3	2	25.1	17.9
5	4	0*	9.8	19.0	6	3	-1*	15.1	21.3	4	3	2*	12.5	21.8
6	4	0	11.9	9.1	4	4	1*	6.6	13.2	4	3	-2	17.3	20.6
5	5	0	24.1	23.0	5	4	1*	14.4	1.8	5	3	2	16.2	15.8
1	0	1*	0.	4.4	5	4	-1*	6.2	2.4	5	3	-2*	0.	18.3
1	0	-1*	4.4	2.0	6	4	1*	1.8	5.2	6	3	2	27.0	34.9
2	0	1*	8.6	17.1	6	4	-1	72.2	75.9	6	3	-2*	0.	28.3
2	0	-1	66.6	64.8	5	5	1*	0.	2.0	4	3	2*	8.0	6.1
3	0	1	65.7	85.2	1	0	2	13.7	9.0	5	4	2*	15.1	19.6
3	0	-1*	0.	11.0	1	0	-2	8.6	9.3	5	4	-2*	0.9	6.8
4	0	1	68.0	64.7	2	0	2	588.9	587.2	6	4	2	194.2	190.5
4	0	-1*	7.9	16.7	2	0	-2*	10.7	18.9	6	4	-2*	6.5	6.1
5	0	1*	3.8	13.4	3	0	2	69.3	85.4	5	5	2*	10.4	8.6
5	0	-1	15.3	22.1	3	0	-2	42.2	44.5	0	0	3	66.0	65.3
6	0	1*	6.8	15.0	4	0	2	14.1	9.9	1	0	3	40.5	38.0
6	0	-1*	2.8	15.0	4	0	-2	432.7	427.3	1	0	-3	18.7	25.4
7	0	1*	7.6	7.0	5	0	2*	13.6	15.9	2	0	3*	10.6	20.8
7	0	-1*	13.2	8.7	5	0	-2	40.8	48.8	2	0	-3*	10.6	21.4
8	0	1*	6.5	9.0	6	0	2*	10.0	12.1	3	0	3	24.2	24.4
8	0	-1	94.4	91.7	6	0	-2*	6.3	0.2	3	0	-3	15.3	21.1
1	1	1*	7.2	23.5	7	0	2	34.3	37.4	4	0	3	14.6	21.4
2	1	1	24.7	34.2	7	0	-2*	8.7	6.6	4	0	-3*	5.4	23.4
2	1	-1	17.6	18.2	8	0	2	212.3	210.7	5	0	3	35.4	33.2
3	1	1*	10.3	15.2	8	0	-2*	0.	1.0	5	0	-3	37.3	41.7
3	1	-1	15.6	11.1	1	1	2	40.5	48.8	6	0	3	90.2	93.1

(table 13. continued)

I	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
6	0	-3	128.4	122.5	4	0	4	14.3	17.0	2	0	-5	14.7	20.5
7	0	3*	0.	20.6	5	0	4	16.6	12.3	3	0	5	17.5	12.9
7	0	-3	18.2	21.4	5	0	-4*	0.	2.1	3	0	-5*	7.8	4.3
8	0	3*	0.	19.9	6	0	4*	1.0	1.5	4	0	5*	7.0	19.9
8	0	-3*	3.5	13.0	6	0	-4*	5.7	5.6	4	0	-5	114.7	115.7
1	1	3	20.7	24.0	7	0	4*	5.7	11.7	5	0	5*	12.0	23.4
2	1	3	39.3	49.7	7	0	-4	71.2	67.1	5	0	-5	32.3	30.0
2	1	-3	26.6	35.3	8	0	4*	3.2	13.9	6	0	5*	1.1	19.8
3	1	3	37.2	40.0	8	0	-4	202.9	199.1	6	0	-5*	0.	24.3
3	1	-3*	10.6	18.3	1	1	4*	13.0	20.3	7	0	5	34.9	37.2
4	1	3	12.5	15.0	2	1	4*	7.5	10.2	7	0	-5*	0.	5.2
4	1	-3	14.1	14.3	2	1	-4*	0.	18.9	8	0	5	91.3	89.8
5	1	3	22.4	21.0	3	1	4	68.5	78.6	8	0	-5*	4.1	7.2
5	1	-3	37.9	47.2	3	1	-4	24.0	30.3	1	1	5	21.0	20.8
6	1	3	27.4	27.4	4	1	4	14.6	22.9	2	1	5	28.0	33.4
6	1	-3*	0.	18.8	4	1	-4	26.5	21.4	2	1	-5	27.1	30.5
7	1	3*	5.1	7.1	5	1	4	20.2	26.7	3	1	5	23.3	24.7
7	1	-3*	3.4	6.4	5	1	-4*	2.3	50.4	3	1	-5	67.0	81.9
8	1	3*	11.5	7.1	6	1	4	44.8	57.6	4	1	5*	0.	2.5
2	2	3	114.6	112.6	6	1	-4	18.4	20.4	4	1	-5*	2.6	10.3
3	2	3*	9.2	13.4	7	1	4*	0.	7.2	5	1	5	71.5	69.5
3	2	-3	21.2	23.4	7	1	-4*	0.	6.0	5	1	-5	13.8	11.0
4	2	3*	7.5	20.1	8	1	4	14.9	17.7	6	1	5*	7.7	7.0
4	2	-3*	9.5	15.3	2	2	4	11.1	8.4	6	1	-5	58.4	54.3
5	2	3*	9.8	7.5	3	2	4	17.5	17.2	7	1	5*	0.	6.1
5	2	-3*	8.7	15.0	3	2	-4	99.4	99.5	7	1	-5*	4.2	11.0
6	2	3*	7.0	8.2	4	2	4*	3.8	12.5	8	1	-5*	6.8	6.1
6	2	-3*	5.8	9.7	4	2	-4	287.5	282.5	2	2	5*	10.3	25.4
7	2	3*	19.4	28.8	5	2	4	27.2	31.5	3	2	5	46.8	53.3
7	2	-3*	5.8	10.2	5	2	-4	20.9	24.4	3	2	-5	20.5	14.2
3	3	3*	9.3	11.0	6	2	4	241.5	237.1	4	2	5	123.8	118.9
4	3	3*	5.1	14.4	6	2	-4*	9.9	2.4	4	2	-5*	7.3	12.2
4	3	-3	48.1	38.8	7	2	4*	20.2	16.7	5	2	5*	0.	4.6
5	3	3	40.2	40.5	7	2	-4	16.1	7.5	5	2	-5*	14.5	3.9
5	3	-3	26.6	32.0	3	3	4*	13.0	6.8	6	2	5*	5.0	12.2
6	3	3*	2.1	5.9	4	3	4	13.6	12.4	6	2	-5	58.9	66.8
6	3	-3*	7.6	2.2	4	3	-4	37.4	30.4	7	2	5*	2.8	12.5
4	4	3	91.8	96.0	5	3	4	38.5	51.4	7	2	-5	27.7	30.6
5	4	3*	0.	13.0	5	3	-4	19.8	27.3	3	3	5*	3.5	9.6
5	4	-3	25.3	20.0	6	3	4*	0.	9.8	4	3	5	43.1	39.8
6	4	3*	2.6	15.8	6	3	-4*	16.1	10.1	4	3	-5*	3.3	9.9
6	4	-3*	1.3	10.8	4	4	4*	2.4	1.3	5	3	5*	1.6	4.0
5	5	3*	2.7	11.3	5	4	4*	9.5	12.0	5	3	-5	77.4	70.9
1	0	4*	7.9	5.5	5	4	-4	44.7	48.6	6	3	5	18.1	7.8
1	0	-4	86.0	87.1	6	4	4*	1.7	8.0	6	3	-5*	12.5	9.1
2	0	4	10.6	14.9	6	4	-4	184.6	181.9	4	4	5*	5.8	20.5
2	0	-4	356.0	361.1	5	5	4*	3.3	6.5	5	4	5	27.4	31.1
3	0	4	57.6	53.6	1	0	5	40.5	43.7	5	4	-5*	3.0	6.7
3	0	-4	56.9	56.7	1	0	-5*	9.6	3.4	5	5	5*	0.	11.4
4	0	4	339.5	332.5	2	0	5	180.0	165.8	0	0	6	560.8	564.4

(table 13. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
1	0	6	23.6	19.8	5	4	-6*	1.8	10.1	5	4	-7*	0.	3.6
1	0	-6	23.8	29.9	1	0	7	36.5	40.0	1	0	8	86.8	96.8
2	0	6*	5.5	0.8	1	0	-7*	17.6	10.3	1	0	-8	28.1	37.6
2	0	-6	17.0	21.8	2	0	7	12.5	16.4	2	0	8	532.2	519.6
3	0	6	16.6	23.7	2	0	-7	94.5	83.9	2	0	-8	13.9	8.0
3	0	-6	22.2	5.6	3	0	7	95.8	100.8	3	0	8	74.5	73.8
4	0	6*	9.3	0.5	3	0	-7	60.9	65.1	3	0	-8	84.1	76.9
4	0	-6*	0.	2.9	4	0	7	80.5	71.2	4	0	8*	8.5	6.0
5	0	6	30.8	40.0	4	0	-7*	11.6	23.4	4	0	-8	366.2	366.3
5	0	-6	83.0	72.7	5	0	7	37.9	36.2	5	0	8*	10.9	14.8
6	0	6	252.8	21.1	5	0	-7*	9.6	6.1	5	0	-8	25.8	30.0
6	0	-6	232.4	140.4	6	0	7*	2.5	9.3	6	0	8*	10.7	14.2
7	0	6	20.2	18.2	6	0	-7*	0.9	10.7	6	0	-8*	11.1	7.2
7	0	-6*	0.	1.2	7	0	7*	9.0	15.6	7	0	8	31.8	28.1
8	0	6*	0.	11.6	7	0	-7	20.9	16.1	7	0	-8	26.6	29.3
8	0	-6*	1.8	5.1	8	0	7*	0.	15.3	8	0	8	144.0	153.2
1	1	6	24.9	26.8	8	0	-7	28.4	37.7	8	0	-8*	15.0	13.1
2	1	6	91.2	86.3	1	1	7	67.6	73.0	1	1	8	72.2	70.5
2	1	-6	54.1	63.9	2	1	7	19.5	18.2	2	1	8	65.7	68.8
3	1	6	20.1	25.0	2	1	-7	51.6	54.7	2	1	-8	17.7	15.4
3	1	-6	42.7	61.0	3	1	7*	10.2	5.3	3	1	8	22.0	21.9
4	1	6	23.5	26.1	3	1	-7	22.6	21.8	3	1	-8	46.6	47.5
4	1	-6	20.2	23.0	4	1	7	59.9	64.7	4	1	8	56.7	62.0
5	1	6	73.0	80.1	4	1	-7	68.7	69.3	4	1	-8	54.2	50.0
5	1	-6	15.8	24.3	5	1	7*	11.6	9.0	5	1	8	24.7	32.0
6	1	6*	10.6	13.9	5	1	-7*	12.2	16.8	5	1	-8	15.6	11.8
6	1	-6*	0.	33.0	6	1	7*	3.0	7.4	6	1	8	25.2	20.9
7	1	6	20.2	18.6	6	1	-7	13.6	12.4	6	1	-8	21.7	29.0
7	1	-6*	10.1	14.7	7	1	7	38.7	46.1	7	1	8	30.1	35.9
8	1	-6*	9.6	7.0	7	1	-7	39.3	41.2	7	1	-8*	0.	24.4
2	2	6	384.8	378.7	2	2	7	12.4	14.4	2	2	8	15.1	17.4
3	2	6*	4.1	14.4	3	2	7	21.7	26.3	3	2	8	42.6	52.6
3	2	-6*	8.9	4.4	3	2	-7	36.1	40.0	3	2	-8	31.1	43.1
4	2	6*	4.5	6.3	4	2	7*	1.8	18.5	4	2	8	260.2	266.9
4	2	-6	12.8	11.5	4	2	-7	59.6	57.0	4	2	-8*	6.1	9.5
5	2	6*	5.3	12.6	5	2	7	51.2	62.2	5	2	8	47.4	49.7
5	2	-6	19.1	23.9	5	2	-7	58.5	60.9	5	2	-8	67.4	65.0
6	2	6*	9.9	6.2	6	2	7	59.3	57.7	6	2	8*	8.0	20.9
6	2	-6*	5.7	5.3	6	2	-7*	0.	14.4	6	2	-8	207.3	206.5
7	2	6*	0.	8.8	7	2	7*	12.0	7.7	7	2	8	15.1	10.9
7	2	-6	62.7	57.6	7	2	-7*	2.1	4.0	7	2	-8	21.9	11.7
3	3	6	19.7	15.6	3	3	7	43.5	53.6	3	3	8	32.2	40.0
4	3	6	68.7	68.7	4	3	7*	6.8	6.3	4	3	8	31.0	28.6
4	3	-6*	6.5	21.6	4	3	-7*	2.4	13.7	4	3	-8	21.2	18.2
5	3	6*	5.0	14.9	5	3	7*	10.9	8.2	5	3	8	13.7	14.1
5	3	-6	43.7	55.3	5	3	-7*	13.4	8.9	5	3	-8	22.9	23.4
6	3	6	22.0	22.0	6	3	7	43.5	51.5	6	3	8	50.4	52.0
6	3	-6*	4.3	12.4	6	3	-7	41.7	39.2	6	3	-8	35.4	30.5
4	4	6	217.3	215.1	4	4	7*	5.0	9.3	4	4	8*	2.1	8.8
5	4	6	26.3	19.1	5	4	7	15.4	12.3	5	4	8	24.0	25.4

(table 13. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
5	4	-8	18.6	23.3	5	4	9*	7.8	5.5	1	0	11	65.2	67.1
0	0	9	125.5	112.1	5	4	-9	50.9	51.5	1	0	-11	24.9	25.6
1	0	9	52.3	60.0	1	0	10*	13.7	19.5	2	0	11	57.9	46.6
1	0	-9	73.1	79.7	1	0	-10	80.8	80.2	2	0	-11	16.4	16.7
2	0	9	15.6	11.7	2	0	10	13.0	10.8	3	0	11*	12.5	5.6
2	0	-9	18.9	19.2	2	0	-10	366.3	358.4	3	0	-11	53.6	59.1
3	0	9*	5.5	0.8	3	0	10*	11.1	13.8	4	0	11	17.2	15.3
3	0	-9	29.2	29.3	3	0	-10	111.5	112.2	4	0	-11*	5.1	3.3
4	0	9*	1.9	19.0	4	0	10	282.8	283.7	5	0	11*	13.6	6.7
4	0	-9	16.3	13.9	4	0	-10	16.5	11.6	5	0	-11	40.4	42.1
5	0	9	69.8	69.5	5	0	10	56.6	61.9	6	0	11*	0.	18.3
5	0	-9*	13.7	19.1	5	0	-10	19.3	18.8	6	0	-11*	0.	17.6
6	0	9	17.0	16.5	6	0	10*	3.3	15.3	7	0	11	55.3	58.5
6	0	-9	53.4	46.5	6	0	-10*	2.7	7.9	7	0	-11*	18.1	23.2
7	0	9*	13.6	6.4	7	0	10	16.9	23.3	1	1	11	33.0	36.2
7	0	-9	53.4	59.5	7	0	-10	59.2	60.4	2	1	11	50.4	49.6
8	0	9*	3.9	9.7	8	0	10*	14.1	14.5	2	1	-11	20.2	16.7
8	0	-9*	14.3	7.7	8	0	-10	157.4	160.1	3	1	11	23.7	24.9
1	1	9	25.1	26.2	1	1	10	84.5	87.0	3	1	-11	81.5	82.0
2	1	9	24.9	27.6	2	1	10	20.5	21.2	4	1	11	26.3	30.8
2	1	-9	68.3	69.1	2	1	-10	58.0	60.3	4	1	-11*	0.	7.3
3	1	9	52.8	56.2	3	1	10	51.7	55.1	5	1	11	61.9	63.5
3	1	-9	16.6	20.7	3	1	-10	25.7	25.4	5	1	-11	15.2	18.6
4	1	9	21.8	21.8	4	1	10	72.6	72.0	6	1	11	19.2	17.7
4	1	-9*	10.9	9.8	4	1	-10	28.3	28.6	6	1	-11	67.2	67.2
5	1	9*	0.	4.7	5	1	10	22.8	22.1	7	1	11*	0.	9.3
5	1	-9	41.6	45.0	5	1	-10	41.5	40.4	7	1	-11*	0.	10.8
6	1	9	46.4	48.9	6	1	10	45.2	46.1	2	2	11*	1.5	21.7
6	1	-9*	4.8	8.3	6	1	-10	24.0	23.2	3	2	11	72.6	74.1
7	1	9*	0.	19.0	7	1	10	38.7	40.2	3	2	-11	21.1	20.1
7	1	-9*	14.5	20.7	7	1	-10	28.9	35.1	4	2	11	22.9	22.3
2	2	9	21.7	19.1	2	2	10*	12.1	1.8	4	2	-11	12.6	9.9
3	2	9*	13.4	18.2	3	2	10	24.8	25.4	5	2	11	21.0	17.1
3	2	-9	72.9	75.4	3	2	-10	83.9	81.7	5	2	-11	31.1	31.9
4	2	9	14.1	12.2	4	2	10	12.9	13.5	6	2	11*	1.2	10.9
4	2	-9*	2.5	12.6	4	2	-10	236.9	239.3	6	2	-11	29.5	30.3
5	2	9*	2.7	2.3	5	2	10	19.3	10.1	3	3	11*	4.5	15.0
5	2	-9*	10.9	16.1	5	2	-10	54.6	53.1	4	3	11	45.5	44.4
6	2	9*	13.2	8.8	6	2	10	177.5	180.7	4	3	-11*	8.4	8.4
6	2	-9*	3.5	7.1	6	2	-10*	1.5	2.9	5	3	11	26.4	24.5
7	2	9	48.1	45.5	3	3	10	52.8	52.8	5	3	-11	68.5	68.1
7	2	-9*	5.1	15.2	4	3	10	18.4	18.9	4	4	11*	1.8	15.2
3	3	9	24.1	24.7	4	3	-10	55.6	54.5	5	4	11	50.1	52.4
4	3	9	16.5	17.3	5	3	10*	8.4	28.0	5	4	-11*	10.2	19.4
4	3	-9	58.3	59.5	5	3	-10	21.9	20.2	0	0	12	271.1	266.0
5	3	9	33.5	34.5	6	3	10	33.1	30.9	1	0	12	28.9	28.1
5	3	-9*	1.4	10.1	6	3	-10	31.0	27.4	1	0	-12	89.4	90.9
6	3	9*	4.4	12.5	4	4	10	13.0	15.3	2	0	12*	6.8	0.0
6	3	-9*	2.1	12.6	5	4	10*	13.0	21.3	2	0	-12*	10.7	15.8
4	4	9	38.5	36.7	5	4	-10	53.6	49.0	3	0	12	39.0	40.8

(table 13. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
3	0	-12*	14.7	10.3	7	0	-13*	16.5	3.8	6	1	14	33.6	30.8
4	0	12	13.2	15.5	1	1	13	78.6	78.1	6	1	-14	40.9	36.9
4	0	-12*	10.8	3.1	2	1	13	29.9	29.6	2	2	14	14.3	18.1
5	0	12	30.0	26.2	2	1	-13	16.1	19.3	3	2	14	66.5	67.9
5	0	-12	61.3	61.9	3	1	13*	10.4	12.9	3	2	-14	35.2	37.8
6	0	12	187.0	182.6	3	1	-13	25.7	24.3	4	2	14	136.3	140.4
6	0	-12	145.1	149.9	4	1	13	62.3	61.7	4	2	-14*	7.8	10.5
7	0	12	32.2	35.1	4	1	-13	94.5	94.9	5	2	14	24.8	29.7
7	0	-12	45.9	46.2	5	1	13*	6.9	13.6	5	2	-14	46.1	51.2
1	1	12	27.4	28.7	5	1	-13*	12.4	12.9	3	3	14	59.4	59.1
2	1	12	58.2	63.3	6	1	13*	17.4	3.4	4	3	14	41.9	41.1
2	1	-12	54.5	50.7	6	1	-13*	8.7	11.2	4	3	-14	31.6	28.3
3	1	12	68.9	70.4	2	2	13	14.1	13.2	5	3	14	25.5	22.8
3	1	-12	55.6	57.1	3	2	13	26.2	28.6	5	3	-14	27.4	26.3
4	1	12	24.6	23.4	3	2	-13*	9.1	8.2	4	4	14*	12.7	11.1
4	1	-12	34.5	36.7	4	2	13*	0.	22.8	0	0	15	29.6	22.6
5	1	12	72.6	66.4	4	2	-13	16.8	15.2	1	0	15*	17.8	31.4
5	1	-12	43.3	46.9	5	2	13	73.9	77.4	1	0	-15	75.6	79.0
6	1	12	44.4	45.3	5	2	-13	43.3	43.3	2	0	15*	4.0	7.4
6	1	-12	44.6	45.4	6	2	13	18.8	15.9	2	0	-15*	0.	16.9
7	1	12	24.7	25.4	6	2	-13*	0.	21.3	3	0	15	35.3	31.7
7	1	-12	21.0	23.9	3	3	13	89.4	85.8	3	0	-15	26.7	32.6
2	2	12	218.6	217.1	4	3	13	20.8	19.9	4	0	15*	0.	18.5
3	2	12	31.5	36.8	4	3	-13*	18.5	6.6	4	0	-15*	3.9	6.4
3	2	-12	68.2	66.8	5	3	13*	6.0	9.0	5	0	15	84.1	79.3
4	2	12*	7.7	8.3	5	3	-13*	9.5	7.5	5	0	-15	36.5	29.8
4	2	-12*	8.9	10.9	4	4	13*	4.3	9.1	6	0	15	16.1	12.1
5	2	12	32.8	30.3	1	0	14	87.8	92.2	6	0	-15*	12.4	5.4
5	2	-12	18.1	13.8	1	0	-14	36.9	34.8	1	1	15	31.5	36.7
6	2	12*	6.4	10.0	2	0	14	226.8	222.5	2	1	15	25.1	19.1
6	2	-12*	3.0	9.5	2	0	-14*	6.1	1.4	2	1	-15	79.4	82.1
3	3	12	31.8	30.1	3	0	14	25.8	34.6	3	1	15	73.0	72.8
4	3	12	56.0	53.7	3	0	-14	43.3	52.7	3	1	-15*	3.2	9.3
4	3	-12	31.2	28.5	4	0	14*	7.2	8.2	4	1	15	20.8	21.4
5	3	12	39.4	44.0	4	0	-14	168.0	171.2	4	1	-15	23.4	24.6
5	3	-12	74.9	55.1	5	0	14	25.8	26.2	5	1	15	23.1	18.4
4	4	12	150.9	149.6	5	0	-14	43.9	45.0	5	1	-15	65.0	62.1
1	0	13	45.1	41.3	6	0	14*	8.7	13.6	6	1	15	64.2	63.6
1	0	-13*	12.0	11.5	6	0	-14*	3.2	14.2	6	1	-15*	0.	12.1
2	0	13*	0.	14.6	7	0	14	38.1	43.0	2	2	15	16.4	13.7
2	0	-13	43.1	39.8	7	0	-14	28.1	25.4	3	2	15*	14.3	24.8
3	0	13	100.8	106.9	1	1	14	41.7	47.7	3	2	-15	75.1	78.2
3	0	-13	41.5	39.7	2	1	14	65.9	67.5	4	2	15*	11.2	8.8
4	0	13	36.3	32.5	2	1	-14	30.5	34.4	4	2	-15*	0.	19.8
4	0	-13*	0.	23.8	3	1	14	34.4	35.8	5	2	15	20.1	22.4
5	0	13*	0.	7.7	3	1	-14	55.5	56.9	5	2	-15	25.2	18.0
5	0	-13*	15.4	24.0	4	1	14	60.2	59.3	3	3	15	20.5	18.8
6	0	13*	10.2	10.4	4	1	-14	38.0	41.1	4	3	15	21.6	17.1
6	0	-13*	6.5	6.4	5	1	14	35.3	37.6	4	3	-15	67.7	67.1
7	0	13*	17.9	19.5	5	1	-14	29.7	27.9	1	0	16	21.2	25.8

(table 13. continued)

H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/	H	K	L	/FO/	/FC/
1	0	-16	53.9	59.3	4	1	-17*	0.	6.9	3	1	19*	12.5	9.6
2	0	16*	11.7	10.7	5	1	17	54.7	54.6	3	1	-19	27.3	27.2
2	0	-16	195.6	195.1	5	1	-17	30.2	26.7	4	1	19	45.2	40.7
3	0	16	64.1	59.9	2	2	17	30.2	19.8	4	1	-19	62.9	63.5
3	0	-16	76.2	77.3	3	2	17	59.7	53.7	2	2	19*	4.6	7.6
4	0	16	135.5	147.6	3	2	-17	19.0	18.7	3	2	19	26.2	23.1
4	0	-16*	2.3	13.3	4	2	17	45.0	42.8	3	2	-19*	3.7	4.2
5	0	16*	0.	34.0	4	2	-17*	5.1	4.4	4	2	19	21.8	21.4
5	0	-16	40.7	41.3	3	3	17	18.7	14.0	4	2	-19	52.0	52.6
6	0	16*	13.1	17.8	4	3	17	62.9	59.2	3	3	19	58.2	56.8
6	0	-16	18.8	12.8	4	3	-17	20.4	17.5	1	0	20	66.1	67.6
1	1	16	76.8	79.2	0	0	18	171.6	175.5	1	0	-20	31.4	28.1
2	1	16	40.9	41.0	1	0	18*	17.2	19.8	2	0	20	127.9	130.6
2	1	-16	35.1	38.4	1	0	-18	87.6	84.6	2	0	-20*	4.0	0.4
3	1	16	42.8	44.5	2	0	18*	9.9	0.4	3	0	20*	11.2	16.0
3	1	-16	32.9	31.0	2	0	-18	15.0	10.1	3	0	-20*	15.5	11.2
4	1	16	53.4	54.6	3	0	18	39.8	35.3	4	0	20*	0.	7.4
4	1	-16	53.5	48.3	3	0	-18	27.8	26.2	4	0	-20	87.8	94.9
5	1	16	30.6	30.6	4	0	18*	10.6	12.1	1	1	20*	5.4	12.0
5	1	-16	27.0	27.6	4	0	-18*	6.5	6.2	2	1	20	68.2	66.4
6	1	16	44.1	41.2	5	0	18	21.6	26.7	2	1	-20	40.8	38.9
6	1	-16	26.0	27.3	5	0	-18	47.5	50.2	3	1	20	32.8	28.7
2	2	16*	6.3	4.1	1	1	18	31.1	30.6	3	1	-20	55.3	54.2
3	2	16	26.0	23.0	2	1	18	36.7	43.4	4	1	20*	13.0	20.9
3	2	-16	65.7	67.5	2	1	-18	55.6	55.0	4	1	-20*	11.1	23.8
4	2	16*	9.5	15.1	3	1	18	72.7	70.7	2	2	20	13.0	14.2
4	2	-16	131.4	137.6	3	1	-18*	11.2	26.2	3	2	20	48.2	43.7
5	2	16	35.7	40.5	4	1	18	30.5	28.1	3	2	-20	33.1	24.1
5	2	-16	43.3	38.4	4	1	-18	33.1	33.2	0	0	21	46.6	50.9
3	3	16	46.3	44.1	5	1	18	24.8	29.0	1	0	21*	0.	1.9
4	3	16	32.0	34.2	5	1	-18	53.6	53.3	1	0	-21	37.1	35.3
4	3	-16	32.7	31.1	2	2	18	136.3	142.6	2	0	21*	2.8	4.8
1	0	17	55.5	56.8	3	2	18*	20.4	26.2	2	0	-21	15.3	13.3
1	0	-17	33.8	30.3	3	2	-18	64.0	60.9	3	0	21	30.3	30.1
2	0	17	126.7	115.2	4	2	18*	3.9	12.2	3	0	-21	36.4	34.6
2	0	-17*	2.6	8.1	4	2	-18*	7.9	8.4	4	0	21	24.6	17.6
3	0	17*	16.7	2.5	3	3	18	36.5	28.4	4	0	-21*	15.1	1.2
3	0	-17*	9.7	0.7	1	0	19	31.3	32.4	1	1	21	33.1	33.9
4	0	17*	4.8	7.0	1	0	-19*	14.4	0.3	2	1	21	23.6	21.2
4	0	-17	67.6	64.9	2	0	19	15.3	13.8	2	1	-21	64.2	63.0
5	0	17	19.7	18.4	2	0	-19	105.4	97.3	3	1	21	48.1	44.2
5	0	-17	72.1	70.4	3	0	19	76.9	65.3	3	1	-21	28.7	18.5
5	0	17	19.4	17.7	3	0	-19	26.0	28.6	2	2	21	45.1	46.7
6	0	-17*	0.	16.9	4	0	19	64.9	64.2	1	0	22	27.6	27.0
1	1	17*	12.2	5.5	4	0	-19	22.1	18.9	1	0	-22*	3.0	12.0
2	1	17	67.4	66.3	5	0	19	38.6	28.4	2	0	22*	5.6	10.2
2	1	-17	31.1	26.0	5	0	-19	21.1	21.4	2	0	-22	76.1	81.8
3	1	17	29.9	28.9	1	1	19	49.2	48.1	3	0	22	49.9	44.8
3	1	-17	57.8	58.8	2	1	19	30.0	26.8	3	0	-22	58.6	59.1
4	1	17*	6.8	7.3	2	1	-19	18.7	14.2	1	1	22	54.3	53.7

