hornblendes of this study the octahedral Al^{3+} contents do not show any such relation with increasing grade of metamorphism and their octahedral Fe^{2+} content is actually higher in the higher grades. But this difference in iron content may very well be due to differences in the original bulk compositions of the amphibolites swamping less conspicuous chemical differences induced by varying conditions of metamorphism.

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THE AMERICAN MINERALOGIST, VOL. 49, JANUARY-FEBRUARY, 1964

LATTICE PARAMETERS AND EXPANSION COEFFICIENTS OF FeS_2 (NATURAL AND SYNTHETIC), AND OF CoS_2

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Some Previous Investigations

Lattice parameter determinations of various pyrites were made by many investigators, *e.g.* Peacock and Smith (1941), Wasserstein (1949), Kerr *et al.* (1945), Gordon (1951), Swanson *et al.* (1955), Lepp (1956), and Kullerud and Yoder (1959). Only in some cases the measurements were made at known sample temperatures. The thermal expansion of pyrite has been estimated to be 0.00005 Å per °C. (Wasserstein, 1949). To obtain more exact values for the lattice constants a precise knowledge of the thermal expansion of pyrite is necessary.

X-ray studies of CoS_2 have been made by Lundqvist and Westgren (1938), Kerr (1945) and Klemm (1962). From these studies a lattice parameter between 5.5346 and 5.537 Å was obtained. The thermal expansion coefficient of CoS_2 is unknown.

Disulfides Used

The powder obtained from cubes and pyritohedrons of natural pyrite from Peru (Ombla Manto, Morococha, samples A and C) and from Missouri (Bellefontaine Quarry, near St. Louis, sample B), was used for lattice parameter determinations at various temperatures. The synthetic FeS_2 was prepared according to Wöhler's (1836) dry method using as starting reagents Fe_2O_3, S and NH_4Cl. Beautiful cubes, cube-octahedra and probably pyritohedra, all in one run, were obtained. They were ground to a fine powder for the *x*-ray studies. No SO_2 odor could be detected during the grinding.

Cobalt sulfide was prepared by precipitation by ammonium polysulfide from hot and slightly acidified cobaltous chloride aqueous solutions. To the washed and dried precipitate S was added in excess, the mixture sealed in silica tubes under hydrogen, the whole heated to 750° C. for 24 hours, and air-quenched. The synthesis was similar to that used by Swanson *et al.* (1955) to obtain FeS_2 and by Rosenqvist (1954) to obtain CoS_2. The dry disulfide displayed a powder pattern of the FeS_2 type.

The Powder Patterns

The patterns were obtained in precision cameras 64 mm in diameter at a constant temperature of camera and sample ($\pm 0.05°$ C.). The exposures were made at 10° intervals between 10 and 65° C. The asymmetric patterns obtained were measured with a comparator. No shrinkage correction was necessary (Straumanis and Ievins, 1959). As the powder mounts were thin (0.12–0.2 mm in diameter), absorption correction could be disregarded. The refraction correction was added to the constants calculated.

Chromium radiation ($\lambda K\alpha_1 = 2.28503$; $\lambda K\beta = 2.08059$ kX) was used throughout, yielding strong α_1-lines in case of FeS_2 ($332\alpha_1$, under $\theta = 82.40°$ at 25° C.) and a strong β line with CoS_2 ($333\beta_1$, under $\theta = 78.15°$ at 25° C.). To convert kX into Å, the factor of 1.00202 was used.

The expansion coefficient α was calculated from the expression

$$\alpha = \Delta a/a\Delta t \qquad (1)$$

t being the temperature (in ° C.) and a the lattice parameter.

Experimental Results

The three samples A, B and C of natural pyrite had lattice parameters which agreed closely with each other (Table 1, col. 1–3). The averages (in Å) of Table 1, col. 3 are plotted against temperature in Fig. 1, and the expansion coefficient of natural pyrite was calculated from eq. (1): $\alpha = 9.25 \times 10^{-6}$ deg.$^{-1}$.

The expansion coefficient obtained permitted reduction of the lattice parameters to values corresponding to 25° C. (Table 1 col. 4).

The synthetic preparations did not give sufficiently sharp powder pat-

Table 1. Lattice Parameters a in kX of Natural Pyrites Reduced to Parameters Corresponding to 25° C. ($\alpha = 9.25 \times 10^{-6}$)

Cr K α_1, radiation; 332 α_1, lines measured. Each a_t constant—average of two or three determinations.

(1) Temp. ° C.	(2) Sample	(3) a_t kX	(4) a_{25} kX
10.0	A	5.40552	5.40629
25.0	A	5.40630	
	B	5.40634	5.40630
	C	5.40627	
35.0	A	5.40677	5.40628
45.0	A	5.40726	
	B	5.40702	5.40622
	C	5.40738	
55.0	A	5.40799	5.40649
65.0	A	5.40828	
	B	5.40882	5.40619
	C	5.40807	
		Average	5.40630
		Refr. corr.	+0.00019
			$a_{25} = 5.40649 \pm 0.00008$ kX
			or 5.41741 Å

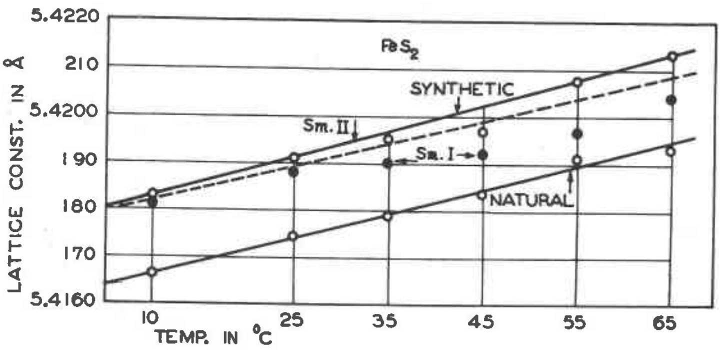

FIG. 1. Lattice parameter *a* in Å (with the refraction correction added) of natural pyrite (samples A, B and C) and of synthetic pyrite (Sm. I and Sm. II) *vs.* temperature.

terns. The last lines were frequently broad and weak, although still separated into α_1 and α_2. Figure 1 shows the expansion of the lattice of a FeS_2 sample (Sm. I) delivering sharper lines. The lines obtained from Sm. II were still sharper, and it can be seen that the expansion coefficient of this sample is nearly equal to that of natural pyrite: $\alpha = 9.65 \times 10^{-6}$. By averaging the constants from both samples, an $\alpha = 8.3 \times 10^{-6}$ is obtained (Fig. 1, dashed line). The average lattice parameter of the synthetic pyrite is then:

$$\begin{array}{rl} & 5.40778 \text{ kX at } 25° \text{ C.} \\ \text{refr. corr.} & 0.00019 \\ \hline & 5.40797 \text{ kX or } 5.4080 \pm 0.0004 \text{ kX or } 5.4189 \text{ Å.} \end{array}$$

It also clearly follows from Fig. 1 that the lattice constants of these synthetic pyrites are larger than those of the natural ones used in the present measurements.

The change of the dimensions of the CoS_2 lattice with temperature is shown in Fig. 2, from which an expansion coefficient of $\alpha = 12.89 \times 10^{-6}$ deg.$^{-1}$ is derived. By means of this coefficient, the lattice parameters of CoS_2 were reduced to values corresponding to 25° C. (Table 2).

The results obtained with FeS_2-CoS_2 solid solutions are published elsewhere (Straumanis *et al.*, 1964)

CONCLUSIONS

As all the precautions were taken to secure high precision lattice constant measurements (*i.e.*, precision camera, high angles of reflection—θ between 78 to 82°— constant temperature of the samples, minimum absorption) it is believed that the constants obtained represent reliable measurements. Indeed, they agree with the best determinations of other

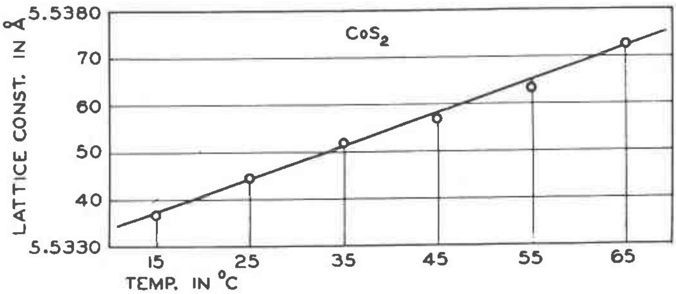

FIG. 2. Lattice parameter a of synthetic CoS_2 (in Å) *vs.* temperature. Refraction correction is added.

authors, where some control of the sample temperature was made, within the limits of error, especially in case of natural pyrite (Table 3). The agreement is poorer with synthetic FeS_2. As the preparations were pure and as the extent of the FeS_2 phase is very narrow (FeS_2, partially decomposed in vacuum at 525° C., showed the same lattice parameter), there is only one explanation for the fluctuations of the constants: difficulties in the determination of the exact Bragg angle of the last lines because of their breadth. This, of course, suggests that the synthetic pyrite crystals were not as perfect as the natural ones. Nevertheless the present measurements show that crystals of the synthetic pyrite may have a slightly larger lattice constant than those of the natural mineral. There seems to be no relationship between the crystal habit and the lat-

TABLE 2. LATTICE PARAMETER OF SYNTHETIC CoS_2 REDUCED TO A PARAMETER CORRESPONDING TO 25° C. ($\alpha=12.89\times10^{-6}$)

$CrK\beta_1$, radiation; (333 β_1) lines were measured. Each constant-average of two determinations.

Temp. ° C.	a_t kX	a_{25} kX
15.0	5.52234	5.52305
25.0	310	310
35.0	383	312
45.0	433	293
55.0	500	286
65.0	590	305
		Average 5.52302±0.00008 kX
		Refraction corr.+0.00016
		$a_{25}=5.52318$ kX or 5.5343_4 Å

The refraction correction was calculated using the x-ray density $d_x=4.8206$ at 25° C.

TABLE 3. LATTICE PARAMETERS (AT 25° C.) AND EXPANSION COEFFICEINTS OF FeS_2 AND CoS_2

Sample	a_{25} Å	Temp. in ° C.	Reference	Expans. Coeff.
Pyrite, natural	5.4175 ± 0.0003	25	Peacock, and Smith 1941	—
	5.4176 ± 0.0001	25	Kerr, 1945	—
	5.4177 ± 0.0003	25	Gordon, 1951	—
	$5.4174_1 \pm 0.00008$	25	This paper	9.25×10^{-6}
Pyrite, synthetic	$5.4165 \pm ?$	25	Swanson *et al.*, 1955	—
	5.4175 ± 0.0003	?	Lepp, 1956	—
	5.419 ± 0.020	26?	Kullerud and Yoder, 1959	—
	5.4189 ± 0.0004	25	This paper	$(8.3\text{–}9.65) \times 10^{-6}$
Cattierite, natural	5.5346 ± 0.0005	?	Kerr, 1945	—
	$5.537 \pm ?$	?	Klemm, 1962	—
CoS_2	5.64	?	Kerr, 1945	—
	5.5343 ± 0.0001	25	This paper	12.89×10^{-6}

tice constant. The thermal expansion coefficient of CoS_2 is larger than that of FeS_2.

The authors are grateful to Prof. T. Rosenqvist, Technical University of Norway, Trondheim, for cobalt sulfide samples and discussion.

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THE AMERICAN MINERALOGIST, VOL. 49, JANUARY–FEBRUARY, 1964

X-RAY POWDER DATA FOR AMINOFFITE

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When aminoffite was first described by Hurlbut (1937), single-crystal x-ray data were included, but not powder data. The writer borrowed one of the type specimens (Harvard Museum No. 106917) and removed a few small crystals for a powder pattern. The measured data were indexed

TABLE 1. X-RAY POWDER DATA FOR AMINOFFITE

(Harvard Museum No. 106917)
CuKα Radiation
Camera Diameter 57.3 mm
R.O.M. Film No. 1770

hkl	$d_{calc.}$(Å)	$d_{obs.}$(Å)	$I_{obs.}$	hkl	$d_{calc.}$(Å)	$d_{obs.}$(Å)	$I_{obs.}$
110	9.758			213	2.887		
101	7.990			402	2.820	2.840	9
200	6.900	6.97	7	332	2.710	2.730	½
211	5.222			510	2.707		
002	4.900 }	4.90	4 diff	303	2.663		
220	4.879 }			431 }	2.657		
112	4.379 }	4.40	7	501 }			
310	4.364 }			422	2.611	2.614	10
301	4.164			323	2.484		
202	3.995	4.02	8	521	2.479		
321	3.564			004	2.450	2.455	2
222	3.456 }	3.48	7	440	2.440		
400	3.450 }			114	2.376	2.380	6
312	3.258 }	3.30	½	512	2.369		
330	3.253 }			530	2.367		
103	3.179			413	2.338		
411	3.167 }	3.11	7	204	2.309	2.315	1
420	3.036 }			600	2.300		
		2.96*	½	611	2.210		

* Magnetite impurity.