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Refinement of the crystal structures of realgar, AsS and orpiment, As_2S_3*

By **D. J. E. Mullen** and **W. Nowacki**

Universität Bern, Abteilung für Kristallographie und Strukturlehre**

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Auszug

Die Kristallstrukturen von Realgar, AsS und Auripigment, As_2S_3 wurden mittels Diffraktometerdaten verfeinert. Die Zelldimensionen von Realgar sind: $a = 9{,}325 \pm 0{,}003$ Å, $b = 13{,}571 \pm 0{,}005$ Å, $c = 6{,}587 \pm 0{,}003$ Å, $\beta = 106°\,23' \pm 5'$ und diejenigen für Auripigment sind: $a = 11{,}475 \pm 0{,}005$ Å, $b = 9{,}577 \pm 0{,}004$ Å, $c = 4{,}256 \pm 0{,}002$ Å, $\beta = 90°\,41' \pm 5'$. Die Raumgruppe ist in beiden Fällen $P2_1/n—C_{2h}^5$. Für Realgar wurden 1525 unabhängige Reflexe gemessen und der R-Wert ergab sich zu 0,045 (für 1205 Reflexe); für Auripigment 836 Reflexe und $R = 0{,}064$ (für 586 Reflexe).

Die Strukturen bestätigen die ursprünglichen Bestimmungen[1, 2]. Realgar weist in der Einheitszelle vier getrennte As_4S_4-Moleküle mit wiegenförmiger Konfiguration auf, welche durch van-der-Waalssche Kräfte zusammengehalten werden. Die Struktur von Auripigment besteht aus Schichten von kovalent gebundenen Schwefel- und Arsenatomen, die normal zu (010) liegen. Van-der-Waals-Kräfte sind für die Kohäsion zwischen den Schichten verantwortlich.

Bindungslängen und -winkel sind in beiden Strukturen sehr ähnlich; der mittlere (As—S)-Abstand beträgt 2,237(2) Å in Realgar und 2,283(5) Å in Auripigment.

Abstract

The crystal structures of realgar, AsS and orpiment, As_2S_3 were refined using diffractometer data. The cell dimensions of realgar are: $a = 9.325 \pm .003$ Å, $b = 13.571 \pm .005$ Å, $c = 6.587 \pm .003$ Å, $\beta = 106°\,23' \pm 5'$, and those of orpiment are: $a = 11.475 \pm .005$ Å, $b = 9.577 \pm .004$ Å, $c = 4.256 \pm .002$ Å, $\beta = 90°\,41' \pm 5'$. In both cases the space group is $P2_1/n$ (C_{2h}^5). A total of 1525 independent reflections were measured for realgar, and the final R factor was

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* Contribution no. 224, part 65 on sulfides and sulfosalts.

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[1] T. Ito, N. Morimoto and R. Sadanaga, The crystal structure of realgar. Acta Crystallogr. 5 (1952) 775—782.

[2] N. Morimoto, The crystal structure of orpiment (As_2S_3) refined. Min. Journal (Japan) 1 (1954) 160—169.

0.045 (based on 1205 reflections). For orpiment 836 independent reflections were obtained, and the final R factor was 0.064 (based on 586 reflections).

The structures are essentially the same as in the original determinations[1, 2]. Realgar has four separate As_4S_4 molecules per unit cell, with cradle-like molecular configurations, van der Waals forces being operative between the molecules. The structure of orpiment consists of layers of covalently bonded sulfur and arsenic atoms, lying normal to the (010) direction. Van der Waals forces are responsible for inter-layer cohesion.

Bond distances and angles are closely similar in the two structures, the mean As—S bond distances being 2.237(2) Å in realgar and 2.283(5) Å in orpiment.

Introduction

The structure of realgar was originally determined by ITO and coworkers[1] in 1952 by means of Harker-Kasper relationships and Fourier syntheses of $hk0$ and $0kl$ projections.

The structure of orpiment was originally determined by MORIMOTO[3] and subsequently refined by him[2] by means of Fourier syntheses.

The present refinements of the structures of realgar and orpiment were undertaken to obtain better As—S bond distances, and thus to afford a more precise comparison between the two sulfides.

I. Realgar, AsS

Experimental

Crystal data: $a = 9.325 \pm .003$ Å, $b = 13.571 \pm .005$ Å, $c = 6.587 \pm .003$ Å, $\beta = 106^\circ 23' \pm 5'$; $V = 799.7$ Å^3; $F(000) = 784.0$; $Z = 4$; λ (Cu$K\alpha$) $= 1.54718$ Å; linear absorption coefficient, μ (Cu$K\alpha$) $= 303$ cm^{-1}.

The material used for the collection of data came from the Lengenbach quarry (Binnatal, Switzerland).

A spherical sample, 0.186 mm in diameter, was made by polishing a crystal of realgar under the microscope. Cell dimensions were determined by the application of a least-squares method to back-reflection Weissenberg measurements, to obtain a "best fit" for cell parameters.

The space group is $P2_1/n$, with $h0l$ absent when $h + l = 2n + 1$ and $0k0$ absent when $k = 2n + 1$.

Intensity data were collected using a Supper-Pace two-circle diffractometer with Weissenberg geometry. A continous ω-scan procedure was used to collect a total of 1525 independent reflections

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[3] N. MORIMOTO, The crystal structure of orpiment. X-rays 5 (1949) 115—120.

Table 1. *Comparison of F_o and F_c values for realgar*
* denotes unobserved reflections ($F_o < 3\sigma$)

h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c
0	0	2	243.4*	-283.0	-1	0	1	133.2	-148.6	-1	7	3	55.8	-56.0	-2	0	8	18.8	18.6	2	7	6	35.6	34.3
		4	8.3*	- 5.3	1	0	3	38.9	42.6	1	7	4	105.9	105.6	2	1	0	42.5	-45.9	-2	7	6	28.1	-25.9
		6	18.3	18.5	-1	0	3	207.8*	224.3	-1	7	4	29.5	29.7	2	1	1	48.6	50.9	-2	7	7	6.2*	3.6
0	1	1	113.8	114.6	1	0	5	5.8*	- 2.6	1	7	5	9.9*	-10.3	-2	1	1	105.6	-108.8	2	8	0	55.0	-53.3
		2	85.4	85.7	-1	0	5	55.6	-54.5	-1	7	5	29.6	27.8	2	1	2	22.3	-19.7	2	8	1	141.8	-141.2
		3	78.1	-82.3	1	0	7	42.1	-44.0	1	7	6	56.2	-54.0	-2	1	2	129.4	-134.7	-2	8	1	22.8	-17.4
		4	50.0	-53.1	-1	0	7	12.0*	12.2	-1	7	6	19.0	-18.8	2	1	3	67.8	-70.4	2	8	2	51.9	49.1
		5	17.3	-17.8	1	1	0	34.2	37.0	-1	7	7	11.5*	- 9.5	-2	1	3	151.6	158.1	-2	8	2	12.5*	10.9
		6	63.4	62.2	1	1	1	8.4*	- 1.0	1	8	0	80.3	-76.8	2	1	4	62.1	64.6	2	8	3	74.5	70.8
		7	25.6	24.2	-1	1	1	86.1	-90.7	1	8	1	31.3	-29.3	-2	1	4	40.6	-39.8	-2	8	3	27.9	-25.3
0	2	0	51.2	49.3	1	1	2	135.5	-139.6	-1	8	1	51.9	-48.2	2	1	5	28.5	29.6	2	8	4	37.6	-37.2
		1	66.2	-67.6	-1	1	2	42.8	38.6	1	8	2	62.8	59.9	-2	1	5	78.1	-73.7	-2	8	4	39.9	35.6
		2	99.1	-104.7	1	1	3	62.8	62.0	-1	8	2	37.4	37.0	2	1	6	7.9*	5.5	2	8	5	15.2	-15.1
		3	14.8*	-13.4	-1	1	3	116.8	119.0	1	8	3	22.2	20.2	-2	1	6	94.8	86.8	-2	8	5	35.8	32.2
		4	46.7	50.0	1	1	4	108.4	114.5	-1	8	3	46.4	40.7	2	1	7	4.8*	3.1	2	8	6	28.6	26.7
		5	18.9	19.8	-1	1	4	54.0	56.7	1	8	4	69.9	-66.3	-2	1	7	25.3	23.7	-2	8	6	68.0	-59.3
		6	32.6	-34.5	1	1	5	6.5*	2.8	-1	8	4	68.0	-62.8	-2	1	8	25.5	-25.0	2	9	0	153.4	-147.6
		7	29.7	30.2	-1	1	5	38.3	-39.3	1	8	5	32.0	29.8	2	2	0	65.1	-70.5	2	9	1	18.4*	15.0
0	3	1	57.9	-59.9	1	1	6	56.1	-54.8	-1	8	5	14.1*	-11.8	2	2	1	216.2	-240.1	-2	9	1	21.6	19.4
		2	53.6	-55.8	-1	1	6	51.9	-50.1	1	8	6	40.7	36.8	-2	2	1	80.4	-82.9	2	9	2	27.6	25.1
		3	36.0	35.9	1	1	7	3.3*	- 1.6	-1	8	6	5.4*	- 2.5	2	2	2	77.6	80.0	-2	9	2	88.0	-86.3
		4	49.6	-51.0	-1	1	7	0.0*	- 2.4	1	9	0	50.8	-48.3	-2	2	2	45.1	-44.5	2	9	3	28.1	-26.9
		5	5.0*	0.4	1	2	0	155.8	182.5	1	9	1	44.1	39.2	2	2	3	169.0	184.4	-2	9	3	18.0*	-14.9
		6	39.0	38.1	1	2	1	49.5	49.2	-1	9	1	33.2	31.7	-2	2	3	100.2	-106.0	2	9	4	26.6	27.5
		7	34.9	-34.5	-1	2	1	23.8	-23.4	1	9	2	7.8*	- 4.0	2	2	4	39.8	-43.6	-2	9	4	9.9*	10.2
0	4	0	19.9	-21.4	1	2	2	36.8	36.7	-1	9	2	127.2	123.7	-2	2	4	26.9	27.0	2	9	5	6.9*	0.3
		1	135.1	147.6	-1	2	2	143.8	159.2	1	9	3	44.5	-42.0	2	2	5	17.9	-19.1	-2	9	5	34.3	31.5
		2	26.6	29.8	1	2	3	146.7	-156.8	-1	9	3	29.0	25.2	-2	2	5	76.3	74.7	-2	9	6	10.2*	7.1
		3	97.6	-97.8	-1	2	3	14.5*	10.2	1	9	4	14.4*	11.7	2	2	6	49.1	52.1	2	10	0	59.0	-57.6
		4	62.1	61.5	1	2	4	66.5	-70.2	-1	9	4	38.0	-36.4	-2	2	6	42.8	-41.8	2	10	1	26.3	-22.1
		5	4.6*	4.0	-1	2	4	6.8*	4.5	1	9	5	10.6*	9.9	2	2	7	22.9	-23.6	-2	10	1	49.1	46.0
		6	71.7	-68.5	1	2	5	92.0	96.0	-1	9	5	30.5	-28.0	-2	2	7	13.5*	-13.1	2	10	2	69.1	67.4
		7	12.1*	-12.8	-1	2	5	31.0	-32.2	1	9	6	10.3*	- 9.2	2	3	0	159.1	-173.7	-2	10	2	14.6*	3.2
0	5	1	17.1	-17.6	1	2	6	52.6	53.9	-1	9	6	12.7	-11.7	2	3	1	28.4	-23.9	2	10	3	77.8	71.5
		2	8.5*	6.7	-1	2	6	38.5	-37.6	1	10	0	19.1*	19.2	-2	3	1	41.3	-40.9	-2	10	3	73.3	-69.8
		3	105.3	106.0	1	2	7	8.0*	- 7.2	1	10	1	51.2	49.2	2	3	2	33.0	32.9	2	10	4	38.6	-37.7
		4	41.6	42.5	-1	2	7	25.5	-25.3	-1	10	1	58.7	51.7	-2	3	2	45.7	-47.5	-2	10	4	52.4	46.4
		5	48.2	-44.6	1	3	0	40.1	40.0	1	10	2	26.9	25.5	2	3	3	14.8*	-14.1	2	10	5	7.1*	- 3.7
		6	46.5	-43.7	1	3	1	45.9	45.8	-1	10	2	8.9*	- 6.9	-2	3	3	40.7	-42.3	-2	10	5	36.2	32.8
		7	10.9*	-10.2	-1	3	1	19.8	-18.8	1	10	3	78.7	-72.2	2	3	4	25.4	25.9	-2	10	6	5.9*	- 4.9
0	6	0	213.9	232.6	1	3	2	13.0*	- 5.1	-1	10	3	33.7	-28.8	-2	3	4	8.0*	6.5	2	11	0	71.1	-66.7
		1	37.8	39.9	-1	3	2	19.8	-18.4	1	10	4	29.7	-28.9	2	3	5	94.5	98.1	2	11	1	17.5*	-17.0
		2	143.8	-150.1	1	3	3	87.8	-87.8	-1	10	4	65.1	58.8	-2	3	5	52.9	51.3	-2	11	1	14.5*	-17.3
		3	28.5	-29.9	-1	3	3	71.0	75.6	1	10	5	29.3	27.7	2	3	6	21.4	-21.7	2	11	2	69.0	68.7
		4	41.5	44.1	1	3	4	77.5	-81.7	-1	10	5	16.7	15.1	-2	3	6	7.1*	- 1.9	-2	11	2	81.2	-83.4
		5	59.9	57.9	-1	3	4	25.5	-25.3	-1	10	6	62.1	-55.1	2	3	7	55.2	-57.4	2	11	3	27.4	27.2
		6	18.9	-19.8	1	3	5	8.7*	7.4	1	11	0	54.0	51.6	-2	3	7	13.1*	-14.2	-2	11	3	10.6*	- 7.4
		7	16.2	-16.1	-1	3	5	32.0	-32.2	1	11	1	56.1	58.7	2	4	0	118.8	-118.6	2	11	4	6.1*	2.4
0	7	1	56.1	56.3	1	3	6	47.6	48.0	-1	11	1	56.1	54.4	2	4	1	50.5	53.2	-2	11	4	65.4	65.8
		2	31.5	-31.8	-1	3	6	25.7	-25.3	1	11	2	8.8*	- 6.5	-2	4	1	86.5	89.0	2	11	5	18.2	18.5
		3	101.0	-97.8	1	3	7	3.0*	2.5	-1	11	2	40.1	-41.2	2	4	2	51.7	50.9	-2	11	5	14.3	14.9
		4	9.2*	- 3.1	-1	3	7	41.5	-39.4	1	11	3	50.0	-50.0	-2	4	2	9.9*	7.5	2	12	0	42.0	41.7
		5	28.3	-28.0	1	4	0	149.4	-159.2	-1	11	3	42.1	-42.5	2	4	3	59.2	-58.9	2	12	1	35.7	37.6
		6	33.7	31.4	1	4	1	219.4	236.4	1	11	4	26.4	-25.9	-2	4	3	7.3*	3.2	-2	12	1	20.2	19.7
0	8	0	180.7	183.9	-1	4	1	9.3*	- 7.6	-1	11	4	20.7	-20.8	2	4	4	7.8*	- 3.8	2	12	2	28.7	-28.9
		1	28.8	-30.1	1	4	2	25.4	-24.0	1	11	5	63.6	66.1	-2	4	4	80.8	76.8	-2	12	2	38.3	-39.9
		2	103.2	-98.1	-1	4	2	101.0	-106.9	-1	11	5	18.6	17.8	2	4	5	9.5*	9.0	2	12	3	8.6*	-10.1
		3	48.9	-45.7	1	4	3	205.8*	-210.1	1	12	0	106.5	-102.7	-2	4	5	8.9*	2.2	-2	12	3	20.8	20.3
		4	33.2	-33.0	-1	4	3	36.6	-41.7	1	12	1	49.0	49.9	2	4	6	13.3*	13.7	2	12	4	31.5	32.7
		5	12.1*	11.5	1	4	4	17.4*	18.0	-1	12	1	49.0	-48.2	-2	4	6	7.9*	0.1	-2	12	4	18.1	-17.9
		6	40.4	36.5	-1	4	4	9.9*	9.8	1	12	2	7.0*	8.0	2	4	7	5.7*	5.1	-2	12	5	6.4*	6.7
0	9	1	34.3	27.9	1	4	5	48.7	47.7	-1	12	2	75.7	-76.0	-2	4	7	22.8	-21.0	2	13	0	6.7*	6.4
		2	35.6	-32.7	-1	4	5	54.0	54.1	1	12	3	37.0	-36.9	2	5	0	38.5	36.8	2	13	1	8.2*	- 6.4
		3	21.9	-20.7	1	4	6	5.5*	- 4.8	-1	12	3	4.4*	- 7.3	2	5	1	21.9	24.7	-2	13	1	43.4	42.5
		4	48.6	-48.3	-1	4	6	24.9	-25.1	1	12	4	16.9	18.4	-2	5	1	6.0*	- 4.1	2	13	2	42.4	43.2
		5	37.9	35.3	1	4	7	10.8*	8.9	-1	12	4	6.5*	- 6.5	2	5	2	71.2	73.5	-2	13	2	64.7	66.2
		6	45.0	42.3	-1	4	7	33.0	-32.8	-1	12	5	8.6*	- 7.4	-2	5	2	9.0*	4.3	2	13	3	13.5	-12.9
0	10	0	38.7	-33.9	1	5	0	147.2	157.2	1	13	0	15.8*	12.8	2	5	3	62.9	-63.7	-2	13	3	23.8	-25.4
		1	82.9	84.4	1	5	1	10.7*	1.1	1	13	1	49.8	-51.1	-2	5	3	43.6	44.8	2	13	4	53.3	-56.2
		2	29.2	27.3	-1	5	1	146.5	159.5	-1	13	1	25.8	24.4	2	5	4	64.6	-64.2	-2	13	4	8.4*	8.4
		3	110.2	-103.6	1	5	2	97.2	-95.0	1	13	2	20.2	-19.7	-2	5	4	93.5	90.7	2	14	0	14.1*	11.7
		4	28.0	26.6	-1	5	2	6.4*	-12.4	-1	13	2	20.0	-18.4	2	5	5	37.4	36.2	2	14	1	8.6*	9.9
		5	65.1	58.9	1	5	3	3.9*	- 1.6	1	13	3	7.7*	7.0	-2	5	5	3.8*	- 4.0	-2	14	1	14.6*	13.8
		6	18.5	-18.1	-1	5	3	189.3	-206.2	-1	13	3	64.0	-65.7	2	5	6	22.4	20.6	2	14	2	16.7	-16.1
0	11	1	47.7	-47.2	1	5	4	7.8*	- 8.9	1	13	4	20.9	20.7	-2	5	6	94.9	-89.7	-2	14	2	24.2	24.7
		2	8.0*	7.9	-1	5	4	51.0	50.4	-1	13	4	9.4*	8.9	-2	5	7	43.0	41.5	2	14	3	19.6	20.1
		3	58.1	58.3	1	5	5	57.1	55.8	1	14	0	58.7	-57.0	2	6	0	76.9	80.9	-2	14	3	30.1	31.7
		4	8.3*	5.8	-1	5	5	68.8	65.3	1	14	1	8.0*	8.7	2	6	1	179.3	190.4	-2	14	4	3.7*	- 1.7
		5	27.6	-27.2	1	5	6	7.9*	7.3	-1	14	1	28.3	-28.5	-2	6	1	49.9	51.3	2	15	0	11.4*	- 9.4
0	12	0	33.7	-32.1	-1	5	6	32.0	-30.7	1	14	2	40.7	43.2	2	6	2	71.9	-73.8	2	15	1	24.9	-24.1
		1	94.5	95.7	1	5	7	17.1	-16.9	-1	14	2	17.8	-18.1	-2	6	2	36.0	-36.3	-2	15	1	42.9	42.6
		2	30.7	-30.8	-1	5	7	27.1	25.6	1	14	3	32.6	32.6	2	6	3	24.4	-24.1	2	15	2	22.5	23.1
		3	43.7	-44.2	1	6	0	132.5	-140.7	-1	14	3	17.6	17.6	-2	6	3	83.1	83.6	-2	15	2	9.9*	- 9.3
		4	39.1	42.0	1	6	1	37.1	37.8	-1	14	4	25.8	-27.4	2	6	4	12.7*	10.3	-2	15	3	14.0	-15.0
		5	4.3*	4.2	-1	6	1	91.3	-98.1	1	15	0	65.9	-63.3	-2	6	4	24.5	21.6	2	16	0	16.9	-16.8
0	13	1	29.8	-28.0	1	6	2	76.7	79.8	1	15	1	11.5*	9.7	2	6	5	29.3	-28.5	2	16	1	16.9	-17.9
		2	7.6*	-10.1	-1	6	2	116.5	-121.3	-1	15	1	29.4	29.2	-2	6	5	65.2	-62.5	-2	16	1	5.3*	4.6
		3	47.7	48.7	1	6	3	28.4	-28.2	1	15	2	43.0	44.9	2	6	6	16.7	17.9	-2	16	2	3.6*	- 0.1
		4	16.8	-17.6	-1	6	3	106.5	110.2	-1	15	2	38.1	39.4	-2	6	6	22.4	-21.2	3	0	1	158.9	177.9
0	14	0	56.2	56.0	1	6	4	11.8*	11.9	-1	15	3	21.0	-22.0	-2	6	7	9.1*	- 9.5	-3	0	1	224.7*	-264.6
		1	28.5	-27.5	-1	6	4	6.5*	1.9	1	16	0	22.1	-20.9	2	7	0	10.2*	13.5	3	0	3	65.4	-70.1
		2	2.9*	- 2.5	1	6	5	18.6	18.5	1	16	1	27.4	-28.4	2	7	1	9.9*	3.3	-3	0	3	93.8	99.3
		3	20.1	-20.6	-1	6	5	54.3	-50.5	-1	16	1	16.2	16.0	-2	7	1	61.5	-63.1	3	0	5	17.4	-18.0
		4	21.1	-23.3	1	6	6	16.5	17.7	-1	16	2	5.2*	- 1.7	2	7	2	75.5	76.1	-3	0	5	4.8*	- 0.1
0	15	1	30.0	30.5	-1	6	6	56.3	53.8	2	0	0	23.0	-11.0	-2	7	2	76.7	79.1	-3	0	7	66.8	67.0
		2	14.4	-14.6	1	7	0	49.2	-45.1	2	0	2	67.4	-69.5	2	7	3	12.4*	-11.8	3	1	0	41.6	41.8
		3	36.0	-37.3	1	7	1	37.7	-36.5	-2	0	2	19.7	17.6	-2	7	3	83.1	84.7	3	1	1	87.0	86.5
0	16	0	19.4	-19.6	-1	7	1	35.6	32.4	2	0	4	10.4*	- 7.6	2	7	4	59.8	-59.3	-3	1	1	85.0	-88.7
		1	25.4	25.6	1	7	2	48.3	-47.9	-2	0	4	137.2	142.7	-2	7	4	22.0	-21.5	3	1	2	35.7	37.4
		2	3.6*	0.4	-1	7	2	46.6	-48.2	2	0	6	14.0	14.2	2	7	5	30.4	31.0	-3	1	2	140.8	-142.2
1	0	1	55.1	48.0	1	7	3	42.1	42.5	-2	0	6	72.1	-71.9	-2	7	5	86.7	-81.7	3	1	3	10.6*	8.5

Table 1. *(Continued)*

h	k	l	$\lvert F_o \rvert$	F_c
-3	1	3	52.0	55.7
3	1	4	21.0	-21.2
-3	1	4	23.6	20.1
3	1	5	77.3	-78.8
-3	1	5	8.6*	- 9.9
3	1	6	12.8*	11.8
-3	1	6	92.7	87.4
-3	1	7	26.4	-24.3
-3	1	8	45.2	-45.0
3	2	0	119.9	128.2
3	2	1	68.2	67.1
-3	2	1	114.0	-120.1
3	2	2	61.0	62.8
-3	2	2	115.5	125.9
3	2	3	39.4	40.4
-3	2	3	38.5	38.4
3	2	4	86.2	-91.8
-3	2	4	58.2	-59.9
3	2	5	13.3*	11.8
-3	2	5	34.4	-34.3
3	2	6	23.6	22.7
-3	2	6	16.3	15.6
-3	2	7	22.2	22.2
-3	2	8	7.0*	- 4.2
3	3	0	135.4	-145.6
3	3	1	17.4*	18.6
-3	3	1	55.4	58.5
3	3	2	73.3	76.5
-3	3	2	133.6	149.6
3	3	3	14.7*	14.9
-3	3	3	31.9	31.9
3	3	4	16.3*	-16.1
-3	3	4	51.1	-51.4
3	3	5	34.9	-35.4
-3	3	5	10.6*	- 8.5
3	3	6	41.3	42.8
-3	3	6	50.5	-48.4
-3	3	7	14.1*	11.6
3	4	0	95.0	-99.9
3	4	1	110.6	-108.4
-3	4	1	14.1*	10.2
3	4	2	35.9	37.4
-3	4	2	60.9	-64.8
3	4	3	14.8*	15.0
-3	4	3	123.5	130.7
3	4	4	11.0*	- 3.0
-3	4	4	97.8	97.9
3	4	5	19.8	19.3
-3	4	5	114.4	-109.4
3	4	6	9.8*	- 9.8
-3	4	6	43.0	-41.6
-3	4	7	43.7	42.7
3	5	0	73.1	-74.3
3	5	1	18.2*	-16.8
-3	5	1	27.0	-25.5
3	5	2	8.1*	4.4
-3	5	2	28.9	33.4
3	5	3	8.2*	3.1
-3	5	3	10.4*	- 6.6
3	5	4	8.1*	6.3
-3	5	4	8.3*	5.9
3	5	5	25.2	24.8
-3	5	5	7.7*	- 1.4
3	5	6	17.7	-17.2
-3	5	6	28.4	26.6
-3	5	7	0.0	3.6
3	6	0	71.8	-73.6
3	6	1	105.8	109.5
-3	6	1	175.4	-185.0
3	6	2	4.8*	- 2.2
-3	6	2	144.6	-152.0
3	6	3	18.6	18.5
-3	6	3	96.3	99.8
3	6	4	60.8	62.2
-3	6	4	52.9	52.1
3	6	5	29.6	-29.7
-3	6	5	39.3	-38.3
3	6	6	28.1	-30.0
-3	6	6	29.2	-28.0
-3	6	7	43.2	40.9
3	7	0	80.4	82.5
3	7	1	89.0	-89.8
-3	7	1	92.7	-92.1
3	7	2	14.8*	14.2
-3	7	2	105.1	-105.6
3	7	3	77.8	75.8
-3	7	3	35.0	34.4
3	7	4	10.1*	8.5
-3	7	4	6.8*	- 3.6
3	7	5	34.2	-33.2
-3	7	5	18.5	18.9
3	7	6	11.6*	12.8
-3	7	6	47.5	44.7
-3	7	7	21.3	-19.9
3	8	0	61.5	58.5
3	8	1	78.1	77.3
-3	8	1	62.1	-57.8
3	8	2	18.8	17.8
-3	8	2	61.8	61.3
3	8	3	13.7*	-13.7
-3	8	3	34.2	-31.1
3	8	4	55.9	-54.9
-3	8	4	72.9	66.2
3	8	5	2.3*	3.3
-3	8	5	32.0	28.3
-3	8	6	30.5	-28.3
-3	8	7	9.9*	5.4
3	9	0	13.5	-12.3
3	9	1	41.4	36.1
-3	9	1	16.8*	-15.3
3	9	2	34.9	32.4
-3	9	2	35.4	-34.4
3	9	3	7.8*	5.7
-3	9	3	85.6	83.4
3	9	4	48.6	-48.5
-3	9	4	21.5	20.8
3	9	5	60.3	-56.3
-3	9	5	9.2*	- 9.4
-3	9	6	32.2	30.6
3	10	0	34.5	-32.8
3	10	1	45.7	-44.1
-3	10	1	37.8	35.7
3	10	2	82.0	82.1
-3	10	2	41.2	-39.6
3	10	3	22.1	-18.6
-3	10	3	24.8	21.5
3	10	4	47.8	-45.9
-3	10	4	40.2	36.7
3	10	5	10.5*	9.9
-3	10	5	13.5*	-11.9
-3	10	6	36.7	-34.7
3	11	0	5.4*	- 1.4
3	11	1	54.5	55.9
-3	11	1	15.5*	14.1
3	11	2	49.6	-50.4
-3	11	2	71.1	73.6
3	11	3	0.0*	0.1
-3	11	3	0.0*	- 4.6
3	11	4	28.4	30.4
-3	11	4	29.4	-28.8
-3	11	5	20.2	-18.7
3	12	0	46.4	-45.1
3	12	1	3.6*	- 5.2
-3	12	1	28.2	-27.8
3	12	2	27.9	28.6
-3	12	2	47.1	-48.3
3	12	3	34.1	34.0
-3	12	3	64.8	67.3
3	12	4	5.8*	- 0.9
-3	12	4	50.4	53.3
-3	12	5	68.0	-71.3
3	13	0	18.8	17.3
3	13	1	71.3	-70.6
-3	13	1	18.0	-16.5
3	13	2	5.9*	- 8.6
-3	13	2	28.4	-28.6
3	13	3	47.7	47.5
-3	13	3	11.1*	12.1
-3	13	4	6.7*	7.4
3	14	0	16.5	-14.9
3	14	1	7.0*	5.1
-3	14	1	15.0	-14.4
3	14	2	7.7*	- 7.6
-3	14	2	19.9	-20.1
-3	14	3	5.3*	3.1
-3	14	4	22.7	24.8
3	15	0	24.9	23.1
3	15	1	3.4*	- 2.1
-3	15	1	50.8	-51.5
3	15	2	5.0*	- 3.1
-3	15	2	22.4	-23.6
-3	15	3	32.0	33.7
3	16	0	16.7	16.3
-3	16	1	20.2	20.6
4	0	0	86.8	-88.1
4	0	2	78.1	-82.3
-4	0	2	189.5*	209.9
4	0	4	46.7	45.2
-4	0	4	182.5	-198.3
4	0	6	20.7	-22.0
-4	0	6	69.3	70.4
-4	0	8	19.4	19.9
4	1	0	66.6	66.9
4	1	1	25.1	-25.9
-4	1	1	9.5*	- 5.1
4	1	2	19.6	-20.8
-4	1	2	90.1	95.9
4	1	3	13.1*	- 3.7
-4	1	3	59.0	63.7
4	1	4	9.7*	8.7
-4	1	4	103.5	-109.1
4	1	5	12.6*	13.0
-4	1	5	83.4	-85.7
4	1	6	10.1*	-10.2
-4	1	6	19.9	-18.6
-4	1	7	44.6	42.1
-4	1	8	27.5	26.2
4	2	0	97.0	-101.2
4	2	1	70.6	-73.3
-4	2	1	62.1	-61.3
4	2	2	132.9	140.5
-4	2	2	97.3	100.9
4	2	3	40.0	41.3
-4	2	3	17.5*	16.2
4	2	4	36.6	-37.3
-4	2	4	65.5	-68.1
4	2	5	13.0*	-14.3
-4	2	5	40.7	-41.9
4	2	6	9.9*	8.6
-4	2	6	20.5	20.5
-4	2	7	7.0*	3.7
-4	2	8	10.9*	-11.5
4	3	0	30.8	26.9
4	3	1	138.1	140.8
-4	3	1	23.3	-16.9
4	3	2	43.0	-42.6
-4	3	2	11.5*	12.9
4	3	3	7.2*	6.8
-4	3	3	120.2	-131.5
4	3	4	16.5*	16.9
-4	3	4	31.1	27.9
4	3	5	41.1	-44.2
-4	3	5	85.4	84.2
4	3	6	17.1	17.4
-4	3	6	2.5*	2.2
-4	3	7	16.2*	-15.8
4	4	0	48.8	-50.1
4	4	1	17.1*	14.4
-4	4	1	26.2	-26.6
4	4	2	169.7	178.0
-4	4	2	77.0	-77.5
4	4	3	19.4	-20.3
-4	4	3	75.4	79.6
4	4	4	129.2	-137.3
-4	4	4	24.8	-25.8
4	4	5	11.6*	11.8
-4	4	5	18.3	-16.0
4	4	6	20.8	20.7
-4	4	6	66.1	64.3
-4	4	7	28.0	25.8
4	5	0	36.2	-36.9
4	5	1	83.7	83.1
-4	5	1	127.5	-127.7
4	5	2	36.4	35.3
-4	5	2	102.8	-107.8
4	5	3	35.6	-35.1
-4	5	3	13.1*	12.2
4	5	4	31.6	-32.4
-4	5	4	81.6	80.7
4	5	5	22.7	-22.1
-4	5	5	32.4	30.6
4	5	6	17.2	-17.6
-4	5	6	20.0	-18.2
-4	5	7	6.7*	0.3
4	6	0	48.9	-47.7
4	6	1	33.9	-35.4
-4	6	1	115.8	-115.3
4	6	2	14.9*	-12.5
-4	6	2	96.1	98.0
4	6	3	10.4*	- 7.8
-4	6	3	10.7*	- 6.6
4	6	4	31.6	32.5
-4	6	4	72.1	-69.0
4	6	5	37.3	-37.5
-4	6	5	22.9	21.8
-4	6	6	29.6	28.4
-4	6	7	5.4*	3.7
4	7	0	26.8	23.5
4	7	1	10.8*	4.1
-4	7	1	37.6	37.1
4	7	2	20.5	-18.7
-4	7	2	20.8	-19.7
4	7	3	7.0*	- 6.6
-4	7	3	53.7	54.5
4	7	4	3.6*	- 4.1
-4	7	4	9.1*	- 6.8
4	7	5	25.1	24.6
-4	7	5	90.4	-85.5
-4	7	6	36.2	34.1
-4	7	7	23.3	22.4
4	8	0	55.8	-52.5
4	8	1	78.6	-77.6
-4	8	1	31.2	-30.3
4	8	2	12.7*	-10.4
-4	8	2	126.1	129.4
4	8	3	91.4	87.8
-4	8	3	47.2	45.6
4	8	4	24.9	25.1
-4	8	4	101.3	-89.2
4	8	5	45.7	-43.2
-4	8	5	15.9	-14.6
-4	8	6	4.3*	3.4
4	9	0	8.4*	1.6
4	9	1	69.9	-65.1
-4	9	1	36.8	33.8
4	9	2	9.3*	- 7.5
-4	9	2	22.9	24.4
4	9	3	14.6*	9.2
-4	9	3	32.0	-30.2
4	9	4	10.8*	9.0
-4	9	4	6.4*	- 6.3
-4	9	5	18.9	18.6
-4	9	6	3.8*	- 2.0
4	10	0	50.2	-45.6
4	10	1	37.6	-32.4
-4	10	1	92.3	-89.1
4	10	2	58.4	57.4
-4	10	2	11.4*	-11.3
4	10	3	24.6	-23.2
-4	10	3	53.2	51.4
4	10	4	48.1	-44.0
-4	10	4	6.1*	- 6.5
-4	10	5	49.1	-45.3
-4	10	6	26.6	24.7
4	11	0	11.8*	9.5
4	11	1	98.6	101.9
-4	11	1	48.3	-48.6
4	11	2	9.2*	- 9.2
-4	11	2	61.6	-64.6
4	11	3	28.0	-28.2
-4	11	3	34.1	-35.6
4	11	4	13.9	-14.9
-4	11	4	47.4	49.8
-4	11	5	44.1	46.1
4	12	0	25.3	23.6
4	12	1	8.8*	- 7.2
-4	12	1	62.9	-63.3
4	12	2	18.8	19.8
-4	12	2	27.0	-26.2
4	12	3	6.4*	- 5.4
-4	12	3	55.7	59.0
-4	12	4	11.3*	12.3
-4	12	5	15.4	-15.7
4	13	0	19.7	19.6
4	13	1	22.0	21.9
-4	13	1	43.5	-44.1
4	13	2	12.9	-13.4
-4	13	2	9.3*	-10.2
4	13	3	17.2	-17.3
-4	13	3	37.9	40.5
-4	13	4	30.7	32.4
4	14	0	17.4	-16.1
4	14	1	53.0	-54.3
-4	14	1	50.5	-51.2
4	14	2	35.1	-36.2
-4	14	2	27.2	28.3
-4	14	3	12.5*	13.9
4	15	0	16.0	-15.6
4	15	1	41.0	-42.5
-4	15	1	40.0	41.1
-4	15	2	7.1*	- 6.8
5	0	1	66.6	66.2
-5	0	1	100.2	-105.7
5	0	3	26.8	23.6
-5	0	3	23.0	-21.6
5	0	5	72.0	-77.9
-5	0	5	11.5*	- 4.8
-5	0	7	14.9	13.9
5	1	0	30.1	-31.4
5	1	1	123.5	-125.6
-5	1	1	89.3	-90.8
5	1	2	68.1	70.1
-5	1	2	25.1	25.5
5	1	3	23.9	23.7
-5	1	3	59.8	-64.3
5	1	4	55.7	-58.5
-5	1	4	46.0	-49.4
5	1	5	14.9	-13.1
-5	1	5	64.4	66.3
-5	1	6	17.3	17.7
-5	1	7	47.0	-45.5
5	2	0	79.5	82.2
5	2	1	79.8	-83.6
-5	2	1	176.4	183.1
5	2	2	113.7	-119.7
-5	2	2	45.1	-46.5
5	2	3	34.1	33.9
-5	2	3	40.1	-42.8
5	2	4	33.0	35.3
-5	2	4	33.2	32.0
5	2	5	21.4	-21.4
-5	2	5	10.9*	-10.7
-5	2	6	22.8	22.3
-5	2	7	8.4*	- 5.9
5	3	0	39.3	-40.4
5	3	1	48.7	-49.8
-5	3	1	89.5	-92.1
5	3	2	32.8	34.4
-5	3	2	100.8	104.3
5	3	3	16.9*	16.5
-5	3	3	33.5	34.1
5	3	4	35.3	-37.1
-5	3	4	17.1	-16.1
5	3	5	6.7*	5.3
-5	3	5	52.4	51.7
-5	3	6	25.1	-25.2
-5	3	7	17.4	-15.6
5	4	0	53.6	-53.5
5	4	1	142.8	-144.0
-5	4	1	151.6	152.9
5	4	2	23.4	23.3
-5	4	2	21.0	-22.5
5	4	3	28.9	29.7
-5	4	3	85.1	-89.6
5	4	4	23.7	24.5
-5	4	4	5.0*	2.3
5	4	5	13.1	12.5
-5	4	5	13.4*	12.9
-5	4	6	12.7*	11.6
-5	4	7	5.0*	- 0.8
5	5	0	19.8	-19.8
5	5	1	22.2*	20.3
-5	5	1	62.3	61.2
5	5	2	39.7	-38.8
-5	5	2	54.5	55.7
5	5	3	9.6*	- 7.9
-5	5	3	12.8*	10.6
5	5	4	9.1*	-10.1
-5	5	4	77.5	-78.3
5	5	5	10.6*	7.8
-5	5	5	117.7	-113.4
-5	5	6	17.3	16.9
-5	5	7	76.1	73.9
5	6	0	25.6	24.3
5	6	1	26.8	25.0
-5	6	1	22.4	15.6
5	6	2	9.0*	- 1.4
-5	6	2	82.6	82.4
5	6	3	12.2*	-12.7
-5	6	3	10.3*	- 7.6
5	6	4	20.7	-21.9
-5	6	4	89.0	-89.2
5	6	5	18.6	-17.5
-5	6	5	23.4	-23.1
-5	6	6	24.7	-23.8
-5	6	7	21.9	20.7
5	7	0	16.4*	15.5
5	7	1	20.2*	-21.4
-5	7	1	23.7	-23.0
5	7	2	78.4	79.7
-5	7	2	15.2*	-14.3
5	7	3	12.1*	9.6
-5	7	3	14.3*	16.2
5	7	4	37.3	-38.3
-5	7	4	3.4*	- 0.7
5	7	5	20.4	-21.1
-5	7	5	38.0	-37.7
-5	7	6	29.0	27.9
-5	7	7	20.6	19.9
5	8	0	80.7	76.6
5	8	1	19.1*	15.4
-5	8	1	9.9*	7.2
5	8	2	92.3	-87.7
-5	8	2	32.1	30.7
5	8	3	37.6	36.7
-5	8	3	25.4	-23.9
5	8	4	49.6	48.2
-5	8	4	29.8	-29.5
-5	8	5	16.7	16.7
-5	8	6	54.6	49.1
5	9	0	59.8	-53.2
5	9	1	94.6	-88.3
-5	9	1	104.8	-101.2
5	9	2	43.2	42.1
-5	9	2	4.5*	5.0
5	9	3	22.4	21.5
-5	9	3	20.3*	16.0
5	9	4	47.6	-46.2
-5	9	4	16.4	15.4
-5	9	5	46.9	44.3
-5	9	6	48.0	-44.4
5	10	0	10.7*	7.7
5	10	1	85.4	-80.5
-5	10	1	49.0	46.6
5	10	2	57.4	-54.7
-5	10	2	59.5	-57.7
5	10	3	20.8	19.8
-5	10	3	27.2	-25.4
5	10	4	17.2	17.4
-5	10	4	39.0	56.6
-5	10	5	7.0*	3.6
-5	10	6	5.3*	- 3.3
5	11	0	14.5*	-14.3
5	11	1	48.3	-49.1
-5	11	1	34.5	-36.0
5	11	2	8.7*	- 7.0
-5	11	2	23.8	23.2
5	11	3	5.3*	- 0.1
-5	11	3	0.0*	2.0
-5	11	4	7.0*	10.3
-5	11	5	35.4	-36.7
5	12	0	19.6	-18.6
5	12	1	5.4*	2.7
-5	12	1	28.0	28.7
5	12	2	6.2*	- 6.7
-5	12	2	10.4*	9.5
5	12	3	17.3	-17.6
-5	12	3	12.2*	12.7
-5	12	4	41.5	-44.1
5	13	0	0.0*	30.0
5	13	1	6.6*	- 3.5
-5	13	1	11.1*	10.5
5	13	2	24.1	-23.7
-5	13	2	7.6*	- 7.5
-5	13	3	16.5	16.3
-5	13	4	17.6	-18.6
5	14	0	57.3	54.9
5	14	1	10.1*	9.5
-5	14	1	20.1	-19.5
-5	14	2	64.2	66.7

Table 1. *(Continued)*

h	k	l	|F_o|	F_c
-5	14	3	31.3	-32.7
6	0	0	29.0	-28.8
6	0	2	74.5	-76.9
-6	0	2	15.8	15.9
6	0	4	59.1	61.5
-6	0	4	7.6*	- 5.5
-6	0	6	19.9	19.3
6	1	0	16.0*	13.8
6	1	1	36.2	-35.5
-6	1	1	13.9*	- 0.4
6	1	2	10.4*	- 7.4
-6	1	2	105.2	105.0
6	1	3	11.6*	8.3
-6	1	3	18.0*	16.9
6	1	4	9.3*	- 9.2
-6	1	4	90.4	-92.3
6	1	5	5.3*	- 5.0
-6	1	5	23.9	24.1
-6	1	6	11.8*	8.9
-6	1	7	47.0	-45.7
6	2	0	52.9	51.5
6	2	1	41.3	40.5
-6	2	1	145.5	147.4
6	2	2	25.3	-25.7
-6	2	2	22.0	20.7
6	2	3	51.1	52.3
-6	2	3	49.7	-53.6
6	2	4	17.3*	-15.5
-6	2	4	6.8*	- 1.8
6	2	5	52.4	-55.7
-6	2	5	43.3	-44.8
-6	2	6	26.4	26.3
-6	2	7	11.8*	12.0
6	3	0	7.0*	- 3.3
6	3	1	106.8	-106.6
-6	3	1	138.3	140.1
6	3	2	59.3	59.3
-6	3	2	84.8	84.6
6	3	3	60.1	60.1
-6	3	3	37.7	-39.7
6	3	4	4.3*	5.5
-6	3	4	79.9	-83.4
6	3	5	40.5	-42.6
-6	3	5	9.3*	9.6
-6	3	6	15.8	16.3
-6	3	7	3.3*	1.7
6	4	0	17.6*	-14.0
6	4	1	9.8*	6.8
-6	4	1	42.6	-42.0
6	4	2	20.3	-12.8
-6	4	2	31.7	31.9
6	4	3	21.1	-21.6
-6	4	3	22.4	24.5
6	4	4	5.9*	6.2
-6	4	4	37.0	-36.9
6	4	5	34.2	36.8
-6	4	5	9.1*	- 8.8
-6	4	6	11.6*	-12.3
-6	4	7	31.1	31.6
6	5	0	13.7*	- 2.1
6	5	1	38.4	-37.0
-6	5	1	11.9*	10.3
6	5	2	74.7	-73.9
-6	5	2	84.6	-86.0
6	5	3	28.3	27.5
-6	5	3	15.7*	-14.2
6	5	4	26.2	26.6
-6	5	4	66.4	67.8
-6	5	5	36.1	-35.2
-6	5	6	3.6*	- 1.4
-6	5	7	6.9*	4.6
6	6	0	5.5*	0.5
6	6	1	59.7	-57.6
-6	6	1	53.6	-52.0
6	6	2	41.5	-41.7
-6	6	2	36.2	35.5
6	6	3	35.1	-34.6
-6	6	3	32.3	31.0
6	6	4	31.1	31.4
-6	6	4	26.4	-27.5
-6	6	5	12.6*	-13.6
-6	6	6	38.6	36.0
-6	6	7	20.9	-20.3
6	7	0	62.2	61.0
6	7	1	19.3*	-18.3
-6	7	1	19.6*	-21.1
6	7	2	74.8	-74.5
-6	7	2	53.1	53.1
6	7	3	6.1*	6.7
-6	7	3	80.3	83.1
6	7	4	49.4	49.7
-6	7	4	30.7	-31.8
-6	7	5	21.0	20.9
-6	7	6	8.7*	- 7.3
6	8	0	21.2	17.8
6	8	1	11.7*	9.0
-6	8	1	26.4	24.1
6	8	2	22.2	-21.8
-6	8	2	12.7*	- 8.5
6	8	3	33.5	31.4
-6	8	3	15.1*	-15.3
6	8	4	5.2*	5.7
-6	8	4	35.4	34.7
-6	8	5	5.7*	- 5.2
-6	8	6	2.2*	1.3
6	9	0	14.6*	12.1
6	9	1	23.0*	-19.3
-6	9	1	39.8	36.9
6	9	2	22.9	22.6
-6	9	2	96.3	93.0
6	9	3	13.4	12.1
-6	9	3	67.7	-66.3
-6	9	4	88.5	-81.6
-6	9	5	13.4*	14.5
-6	9	6	15.7	16.1
6	10	0	24.7	23.1
6	10	1	30.9	27.6
-6	10	1	89.8	85.1
6	10	2	4.9*	- 1.5
-6	10	2	32.6	-31.9
6	10	3	11.2*	10.5
-6	10	3	7.0*	0.7
-6	10	4	19.0	-19.6
-6	10	5	83.1	-74.9
6	11	0	29.3	-28.1
6	11	1	5.6*	- 3.5
-6	11	1	4.4*	5.0
6	11	2	7.8*	- 3.5
-6	11	2	7.5*	5.4
-6	11	3	15.3*	15.8
-6	11	4	29.7	-31.4
-6	11	5	18.6	-19.9
6	12	0	8.9*	8.3
6	12	1	15.2*	-14.0
-6	12	1	31.4	-31.3
6	12	2	6.1*	- 5.4
-6	12	2	30.3	30.8
-6	12	3	3.3*	0.0
-6	12	4	24.1	-25.7
6	13	0	49.6	46.3
6	13	1	16.9	17.0
-6	13	1	31.7	-31.7
-6	13	2	7.3*	4.9
-6	13	3	34.3	35.6
-6	14	1	8.1*	- 9.0
-6	14	2	7.2*	- 7.9
7	0	1	8.2*	- 8.4
-7	0	1	77.7	78.3
7	0	3	37.2	-38.1
-7	0	3	76.6	-80.8
-7	0	5	18.2	-17.9
-7	0	7	0.9*	1.8
7	1	0	19.7	17.8
7	1	1	12.4*	-11.4
-7	1	1	9.9*	- 3.0
7	1	2	58.3	-56.3
-7	1	2	35.0	34.1
7	1	3	30.8	-29.7
-7	1	3	11.2*	12.2
7	1	4	47.8	48.6
-7	1	4	67.1	-66.7
-7	1	5	31.7	32.3
-7	1	6	38.9	37.6
-7	1	7	34.6	-34.1
7	2	0	77.4	75.9
7	2	1	52.8	-51.7
-7	2	1	21.2*	19.2
7	2	2	94.0	-94.7
-7	2	2	36.0	36.8
7	2	3	31.8	31.3
-7	2	3	30.9	-29.1
7	2	4	25.5	26.7
-7	2	4	7.4*	- 7.0
-7	2	5	95.7	100.3
-7	2	6	6.4*	5.1
-7	2	7	35.1	-36.4
7	3	0	40.2	-38.6
7	3	1	7.6*	6.0
-7	3	1	28.2	-27.3
7	3	2	14.6*	14.0
-7	3	2	41.0	-40.3
7	3	3	16.0	-15.0
-7	3	3	12.1*	-10.4
7	3	4	5.7*	3.8
-7	3	4	117.3	124.3
-7	3	5	6.3*	1.8
-7	3	6	38.8	-39.0
-7	3	7	9.4*	8.2
7	4	0	9.4*	6.3
7	4	1	35.6	-33.4
-7	4	1	23.2	-21.6
7	4	2	51.2	50.3
-7	4	2	13.4*	11.7
7	4	3	45.0	43.3
-7	4	3	11.4*	-11.8
7	4	4	15.2	-15.1
-7	4	4	8.5*	- 3.4
-7	4	5	74.5	74.3
-7	4	6	2.7*	- 1.7
-7	4	7	79.1	-79.5
7	5	0	40.7	-37.2
7	5	1	51.2	48.6
-7	5	1	52.4	49.6
7	5	2	24.0	23.4
-7	5	2	31.8	-30.2
7	5	3	5.1*	0.4
-7	5	3	45.1	47.0
7	5	4	23.2	24.5
-7	5	4	27.8	27.5
-7	5	5	42.3	-42.6
-7	5	6	9.0*	- 9.2
-7	5	7	0.0*	1.3
7	6	0	53.9	-51.3
7	6	1	30.4	28.2
-7	6	1	15.8*	-14.9
7	6	2	34.9	34.3
-7	6	2	49.6	48.7
7	6	3	26.5	-26.0
-7	6	3	21.4*	-19.6
7	6	4	3.1*	4.2
-7	6	4	38.7	-38.5
-7	6	5	33.9	34.6
-7	6	6	16.9	16.6
7	7	0	35.7	33.7
7	7	1	14.3*	13.4
-7	7	1	60.2	56.7
7	7	2	63.5	-62.7
-7	7	2	65.4	65.3
7	7	3	18.1	16.9
-7	7	3	44.0	-44.7
-7	7	4	74.4	-72.9
-7	7	5	23.8	24.3
-7	7	6	43.3	42.7
7	8	0	80.2	75.5
7	8	1	38.4	-35.3
-7	8	1	86.7	80.5
7	8	2	32.0	-30.6
-7	8	2	42.7	42.7
7	8	3	8.8*	- 8.6
-7	8	3	48.6	-47.1
-7	8	4	27.2	-27.7
-7	8	5	2.9*	5.5
-7	8	6	21.4	20.0
7	9	0	11.6*	- 8.5
7	9	1	18.6*	14.7
-7	9	1	20.5*	-18.7
7	9	2	7.5*	5.7
-7	9	2	10.4*	- 9.9
-7	9	3	7.7*	1.9
-7	9	4	5.4*	1.8
-7	9	5	14.6	14.6
7	10	0	7.6*	7.9
7	10	1	27.3	-24.4
-7	10	1	8.0*	- 1.6
7	10	2	21.0	-19.5
-7	10	2	22.5	22.6
-7	10	3	16.9*	-16.3
-7	10	4	9.9*	- 8.7
-7	10	5	23.5	23.5
7	11	0	39.2	-37.8
7	11	1	24.4	25.1
-7	11	1	14.6	14.6
-7	11	2	12.4	12.5
-7	11	3	47.6	49.9
-7	11	4	25.2	26.9
7	12	0	18.3	18.1
7	12	1	33.1	34.4
-7	12	1	46.2	-47.3
-7	12	2	47.7	48.6
-7	12	3	31.3	31.6
-7	13	1	33.0	32.4
-7	13	2	9.0	9.3
8	0	0	30.4	-29.0
8	0	2	56.0	57.0
-8	0	2	58.5	-58.1
-8	0	4	57.3	58.8
-8	0	6	26.2	-26.8
8	1	0	25.9	23.2
8	1	1	12.5*	9.8
-8	1	1	62.4	-57.5
8	1	2	16.7	-15.5
-8	1	2	58.9	58.6
8	1	3	33.4	32.9
-8	1	3	14.6*	14.5
-8	1	4	22.6	22.6
-8	1	5	30.5	29.9
-8	1	6	26.7	-25.5
-8	1	7	25.9	-26.0
8	2	0	29.6	27.0
8	2	1	13.3*	13.7
-8	2	1	16.6*	15.8
8	2	2	30.4	-29.6
-8	2	2	68.2	-67.9
8	2	3	6.9*	4.6
-8	2	3	2.0*	1.4
-8	2	4	69.6	72.2
-8	2	5	52.9	53.5
-8	2	6	28.9	-28.5
-8	2	7	52.4	-56.9
8	3	0	33.5	32.0
8	3	1	46.7	-44.9
-8	3	1	103.4	101.6
8	3	2	9.4*	- 9.7
-8	3	2	20.2	-21.0
8	3	3	6.1*	- 4.6
-8	3	3	96.9	-99.5
-8	3	4	21.9	22.0
-8	3	5	21.2	21.2
-8	3	6	5.0*	- 6.6
8	4	0	120.7	116.1
8	4	1	15.3*	12.8
-8	4	1	14.7*	-12.5
8	4	2	45.4	-44.0
-8	4	2	75.3	-75.2
8	4	3	9.2*	- 8.0
-8	4	3	13.3*	-12.9
-8	4	4	9.5*	- 6.9
-8	4	5	10.3*	-10.1
-8	4	6	2.5*	- 2.4
8	5	0	39.4	-36.7
8	5	1	0.4*	1.6
-8	5	1	41.3	40.3
8	5	2	32.6	31.5
-8	5	2	9.4*	- 6.9
8	5	3	5.8*	- 5.1
-8	5	3	50.7	-50.3
-8	5	4	23.8	-23.4
-8	5	5	10.4*	8.3
-8	5	6	39.1	38.5
8	6	0	18.8	-18.2
8	6	1	24.1	23.9
-8	6	1	34.9	33.2
8	6	2	5.4*	3.8
-8	6	2	22.0	-21.6
8	6	3	30.1	-31.3
-8	6	3	19.1*	-16.8
-8	6	4	28.2	27.9
-8	6	5	30.0	30.6
-8	6	6	4.9*	- 2.6
8	7	0	44.3	41.9
8	7	1	11.9*	7.6
-8	7	1	66.5	-64.0
8	7	2	16.8	-15.9
-8	7	2	39.3	38.4
-8	7	3	21.3	20.9
-8	7	4	14.0*	-14.0
-8	7	5	38.2	37.3
-8	7	6	12.9	12.8
8	8	0	25.5	-23.0
8	8	1	59.5	58.0
-8	8	1	41.6	39.1
8	8	2	24.6	23.9
-8	8	2	40.7	-39.7
-8	8	3	37.0	-35.1
-8	8	4	81.4	74.1
-8	8	5	25.0	24.5
8	9	0	48.1	43.9
8	9	1	38.9	-35.9
-8	9	1	23.1	22.8
8	9	2	5.6*	3.3
-8	9	2	21.0	22.5
-8	9	3	7.4*	4.7
-8	9	4	8.8*	8.9
8	10	0	37.0	33.7
8	10	1	10.6*	11.2
-8	10	1	11.0	-10.4
-8	10	2	21.6	-21.0
-8	10	3	16.9	16.3
-8	10	4	5.6*	5.0
8	11	0	21.6	-20.6
-8	11	1	47.4	47.4
-8	11	2	8.5*	- 8.7
-8	11	3	44.9	-47.4
9	0	1	85.6	87.2
-9	0	1	17.8	-18.5
9	0	3	46.8	-53.9
-9	0	3	7.4*	8.1
-9	0	5	11.3	-12.0
9	1	0	70.4	67.5
9	1	1	7.6*	- 7.2
-9	1	1	70.0	65.9
9	1	2	30.7	-30.0
-9	1	2	24.8	-24.0
9	1	3	20.2	21.2
-9	1	3	83.0	-82.6
-9	1	4	16.3	-16.3
-9	1	5	47.7	47.8
-9	1	6	4.4*	- 2.7
9	2	0	32.6	-32.3
9	2	1	11.7*	11.0
-9	2	1	19.6	-18.7
9	2	2	5.0*	4.3
-9	2	2	5.0*	- 3.0
-9	2	3	8.1*	3.9
-9	2	4	37.9	-37.0
-9	2	5	28.0	-28.8
-9	2	6	40.7	40.2
9	3	0	25.5	24.4
9	3	1	12.4*	10.2
-9	3	1	30.3	28.5
9	3	2	9.2*	- 9.5
-9	3	2	78.8	-79.1
-9	3	3	52.1	-52.6
-9	3	4	56.3	55.5
-9	3	5	8.4*	7.2
-9	3	6	17.9	-17.7
9	4	0	16.3	-14.8
9	4	1	7.5*	7.3
-9	4	1	35.3	-34.1
9	4	2	16.4	15.9
-9	4	2	35.9	-35.4
-9	4	3	31.5	31.8
-9	4	4	0.0*	- 0.0
-9	4	5	12.6	-13.4
-9	4	6	0.0*	- 0.3
9	5	0	26.5	25.4
9	5	1	9.6*	9.2
-9	5	1	44.7	-42.7
9	5	2	5.0*	- 3.9
-9	5	2	7.0*	5.9
-9	5	3	49.7	50.0
-9	5	4	8.0*	- 8.9
-9	5	5	2.7*	- 0.4
9	6	0	30.3	29.6
9	6	1	34.8	33.8
-9	6	1	12.4*	-13.8
9	6	2	5.4*	- 1.8
-9	6	2	5.9*	2.9
-9	6	3	13.7*	13.9
-9	6	4	69.6	69.7
-9	6	5	30.4	-30.4
9	7	0	25.8	25.2
9	7	1	22.9	22.1
-9	7	1	10.7*	10.8
-9	7	2	16.4	-15.9
-9	7	3	17.4	-16.1
-9	7	4	19.0	-18.8
-9	7	5	34.6	33.6
9	8	0	14.5	-14.3
9	8	1	19.6	18.9
-9	8	1	6.5*	- 4.3
-9	8	2	39.5	-38.3
-9	8	3	7.9*	- 3.1
-9	8	4	19.3	-18.1
9	9	0	59.8	55.1
-9	9	1	36.0	35.0
-9	9	2	36.9	-35.7
-9	9	3	83.7	-81.8
-9	10	1	8.1*	- 8.1
-9	10	2	22.1	-21.6
10	0	0	10.7*	- 8.2
-10	0	2	48.9	48.5
-10	0	4	25.3	-26.1
10	1	0	59.4	57.5
10	1	1	39.1	39.0
-10	1	1	11.7*	-11.1
-10	1	2	16.8	15.5
-10	1	3	4.5*	0.5
-10	1	4	48.0	46.4
-10	1	5	5.2*	0.1
10	2	0	8.9*	8.4
10	2	1	40.6	40.9
-10	2	1	6.1*	1.7
-10	2	2	13.1*	-12.7
-10	2	3	26.9	26.1
-10	2	4	6.2*	- 4.6
-10	2	5	6.3*	6.1
10	3	0	10.5*	10.1
10	3	1	7.6*	- 5.6
-10	3	1	11.3*	- 9.2
-10	3	2	12.9*	12.9
-10	3	3	37.2	37.5
-10	3	4	4.1*	0.6
-10	3	5	41.4	-42.4
10	4	0	4.3*	2.0
10	4	1	27.6	-28.0
-10	4	1	16.0	-15.6
-10	4	2	20.5	21.4
-10	4	3	5.9*	- 3.5
-10	4	4	36.3	36.0
-10	4	5	17.5	-18.2
10	5	0	66.7	-65.8
-10	5	1	11.3*	-10.0
-10	5	2	48.0	-47.6
-10	5	3	23.3	23.7
-10	5	4	16.2	-17.0
10	6	0	12.1	-11.9
-10	6	1	24.3	23.7
-10	6	2	14.3	13.5
-10	6	3	43.0	-44.1
-10	6	4	22.7	-23.4
-10	7	1	24.9	-24.5
-10	7	2	28.0	-27.3
-10	7	3	15.1	-15.0
-10	8	2	5.5*	- 5.0
-11	0	1	4.4*	- 1.8
-11	0	3	71.5	73.4
-11	1	1	18.1	17.6
-11	1	2	45.1	44.6
-11	1	3	21.0	-20.5
-11	1	4	15.5	15.5
-11	2	1	21.8	-22.2
-11	2	2	18.6	-18.9
-11	2	3	16.9	16.9
-11	2	4	7.6*	6.5
-11	3	1	5.9*	6.3
-11	3	2	47.3	-48.6
-11	3	3	12.5*	-12.9
-11	4	2	4.8*	3.3
-11	4	3	12.6	-13.3

($\sin\theta < 0.707$, $\mu < 45°$) about both b and c axes, the radiation used being Cu$K\alpha$.

Relative layer scale factors were calculated from cross-level data. Lorentz, polarization and absorption corrections were made, and also corrections for background, but no correction was applied for secondary extinction effects. The data were placed on an approximate absolute scale by means of a Wilson plot.

Table 2a. (1) *Fractional positional coordinates*
(standard deviations in brackets)

Atom	x	y	z
As(1)	0.12199 (10)	0.02060 (6)	0.76392 (15)
As(2)	0.42373 (10)	0.86090 (6)	0.85582 (15)
As(3)	0.32051 (10)	0.87334 (7)	0.17716 (14)
As(4)	0.04014 (10)	0.83917 (7)	0.71491 (14)
S(1)	0.34521 (22)	0.00604 (14)	0.70138 (32)
S(2)	0.21388 (22)	0.02299 (14)	0.11566 (32)
S(3)	0.23875 (24)	0.77413 (14)	0.63924 (34)
S(4)	0.10702 (24)	0.78976 (15)	0.05105 (34)

(2) *Anisotropic temperature factors* (estimated standard deviations in brackets)
Temperature factors given by:

$$T = \exp\left[-\left(h^2B_{11} + k^2B_{22} + l^2B_{33} + 2hkB_{12} + 2hlB_{13} + 2klB_{23}\right)\right]$$

Atom	B_{11}	B_{12}	B_{13}
As(1)	0.00903 (21)	0.00127 (11)	0.00720 (27)
As(2)	0.00767 (21)	0.00036 (10)	0.01062 (28)
As(3)	0.00828 (21)	0.00053 (11)	0.00684 (27)
As(4)	0.00820 (21)	— .00171 (11)	0.00634 (28)
S(1)	0.00881 (30)	— .00096 (25)	0.01136 (56)
S(2)	0.00842 (29)	0.00043 (24)	0.00829 (53)
S(3)	0.00999 (31)	— .00138 (25)	0.01049 (60)
S(4)	0.00901 (31)	— .00233 (26)	0.01176 (61)

Atom	B_{22}	B_{23}	B_{33}
As(1)	0.00301 (09)	0.00144 (15)	0.01723 (41)
As(2)	0.00315 (09)	— .00069 (15)	0.01989 (42)
As(3)	0.00364 (09)	0.00149 (16)	0.01522 (41)
As(4)	0.00369 (09)	— .00130 (17)	0.01828 (42)
S(1)	0.00284 (11)	0.00163 (37)	0.01781 (59)
S(2)	0.00316 (12)	— .00209 (33)	0.01570 (57)
S(3)	0.00299 (12)	— .00382 (37)	0.01826 (62)
S(4)	0.00368 (13)	0.00225 (38)	0.01856 (62)

Table 2b. *Fractional positional coordinates* (ITO *et al.*[1])

	x	y	z
As(1)	0.118	0.024	0.759
As(2)	0.425	0.860	0.858
As(3)	0.318	0.873	0.181
As(4)	0.038	0.839	0.710
S(1)	0.346	0.008	0.705
S(2)	0.213	0.024	0.120
S(3)	0.245	0.775	0.637
S(4)	0.115	0.785	0.048

Refinement of the structure

Attempts to solve the structure using a symbolic-addition programme were not successful, and the E maps produced contained many spurious peaks.

The coordinates of ITO and co-workers[1] were taken as input coordinates, and a refinement was carried out on these. The initial R factor for all the data with ITO's coordinates was 0.144. Five rounds of isotropic least-squares refinement, using a block-diagonal approximation, led to a R factor of 0.073. Reflections were given weights proportional to $1/\sigma^2$ where σ is the estimated standard deviation of the observed structure amplitude (F_o). Weights were highest for medium-value intensities, with a fall-off for strong and weak values. A minimum value of $F_o \gg 12.0$ was used as a cut-off for low-value data, and only reflections with values greater than this minimum (1205 reflections) were used in the refinement. 320 reflections were omitted: of these all except 24 had values of $F_o < 2\sigma$, and 24 had values of $F_o < 3\sigma$.

Refinement was then continued anisotropically for five more rounds (1205 reflections), and the R factor dropped to 0.045. At this stage the shifts indicated for the positional coordinates and the anisotropic temperature factors were negligible compared with their standard deviations.

The final R factor for all reflections (1525), both observed and unobserved, was 0.055. A listing of structure factors is given in Table 1. The final coordinates and temperature factors are given in Table 2a. Coordinates obtained by ITO *et al.*[1] are given in Table 2b for comparison.

Bond distances and angles, together with their estimated standard deviations, were calculated by means of a programme, and these appear in Table 3. In Table 4 are listed the parameters defining the temperature ellipsoids of the atoms.

Table 3. *Bond distances and angles*
(standard deviations in brackets)

a) Intramolecular bond distances

As(1)—S(1)	2.242 (2) Å	As(3)—S(4)	2.238 (2) Å
As(1)—S(2)	2.232 (2)	As(4)—S(3)	2.231 (2)
As(2)—S(1)	2.243 (2)	As(4)—S(4)	2.228 (2)
As(2)—S(3)	2.238 (2)	As(1)—As(4)	2.571 (1)
As(3)—S(2)	2.247 (2)	As(2)—As(3)	2.566 (1)

b) Intramolecular bond angles

S(1)—As(1)—S(2)	95.03 (7)°	As(2)—As(3)—S(2)	99.24 (6)°
S(1)—As(2)—S(3)	94.51 (8)	As(2)—As(3)—S(4)	99.32 (6)
S(2)—As(3)—S(4)	95.11 (7)	As(1)—As(4)—S(3)	99.85 (6)
S(3)—As(4)—S(4)	94.92 (8)	As(1)—As(4)—S(4)	99.99 (7)
As(4)—As(1)—S(1)	98.67 (6)	As(1)—S(1)—As(2)	101.26 (8)
As(4)—As(1)—S(2)	99.07 (6)	As(1)—S(2)—As(3)	101.23 (8)
As(3)—As(2)—S(1)	99.55 (6)	As(2)—S(3)—As(4)	100.87 (8)
As(3)—As(2)—S(3)	99.17 (6)	As(3)—S(4)—As(4)	100.76 (8)

c) Intramolecular non-bonded distances

As(1)—As(2)	3.467 (1) Å	As(1)—S(3)	3.681 (2) Å
As(1)—As(3)	3.462 (1)	As(1)—S(4)	3.682 (2)
As(2)—As(4)	3.445 (1)	As(2)—S(2)	3.673 (2)
As(3)—As(4)	3.439 (1)	As(2)—S(4)	3.668 (2)
S(1) —S(2)	3.299 (3)	As(3)—S(1)	3.678 (2)
S(1) —S(3)	3.291 (3)	As(3)—S(3)	3.664 (2)
S(2) —S(4)	3.309 (3)	As(4)—S(1)	3.657 (2)
S(3) —S(4)	3.285 (3)	As(4)—S(2)	3.661 (2)

d) Intermolecular distances

S(4)—As(3)II	3.793 (2) Å	S(1)—As(2)III	3.587 (2) Å
As(4)—As(3)II	3.505 (1)	S(2)—As(2)III	3.685 (2)
As(4)—As(2)II	3.563 (1)	As(1)—S(3)IV	3.669 (2)
S(3)—As(2)II	3.518 (2)	As(4)—As(1)III	3.628 (1)
S(4)—As(2)II	3.615 (2)	As(1)—As(1)III	3.621 (1)
S(4)—S(3)II	3.743 (2)	S(2)—As(4)III	3.442 (2)
S(1)—As(3)III	3.410 (2)	S(2)—As(1)III	3.492 (2)

(I, II, III, IV refer to symmetry related general positions: x, y, z; $\frac{1}{2} + x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$; $-x$, $-y$, $-z$; $\frac{1}{2} - x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$ respectively, or their cell-shifted equivalents).

Table 4. *Parameters defining temperature ellipsoids of the atoms*
(Isotropic temperature factors B; u = thermal displacement parameters)

Atom	$<B>$	Axis	B	u	Direction cosines		
					cos(1)	cos(2)	cos(3)
As(1)	2.62 Å^2	1	3.02 Å^2	.196	.795	.388	.466
		2	2.06	.162	−.263	.913	−.312
		3	2.76	.187	−.547	.125	.828
As(2)	2.57	1	2.03	.160	.835	−.474	−.279
		2	2.40	.174	.477	.877	−.061
		3	3.28	.204	.274	−.082	.958
As(3)	2.58	1	2.59	.181	.925	−.373	−.073
		2	2.88	.191	.355	.778	.518
		3	2.27	.169	−.136	−.505	.853
As(4)	2.77	1	2.24	.169	.667	.668	.330
		2	3.12	.199	−.440	.710	−.550
		3	2.94	.193	−.602	.222	.767
S(1)	2.49	1	2.55	.180	.613	−.679	−.404
		2	1.70	.147	.539	.734	−.414
		3	3.21	.202	.577	.036	.816
S(2)	2.47	1	2.69	.185	.791	.597	−.134
		2	1.90	.155	−.427	.696	.577
		3	2.82	.189	.438	−.399	.805
S(3)	2.71	1	2.72	.186	.820	.336	−.463
		2	1.81	.151	−.054	.851	.522
		3	3.59	.213	.569	−.403	.716
S(4)	2.75	1	1.63	.144	.677	.595	−.433
		2	3.38	.207	−.735	.535	−.415
		3	3.24	.203	−.015	.599	.800

Results and discussion

The structure obtained by refinement is essentially that of Ito *et al.*[1]. This may be described as consisting of cradle-like, covalently-bonded As_4S_4 molecules (Fig. 2), held together by van der Waals forces. The unit cell contains four such molecules.

Ito *et al.* give the following description of the As_4S_4 unit: "four sulphur and four arsenic atoms, covalently bonded, form a square and a tetrahedron respectively. The sulphur square cuts through the arsenic tetrahedron in the middle." This is clearly seen in the [001] projection

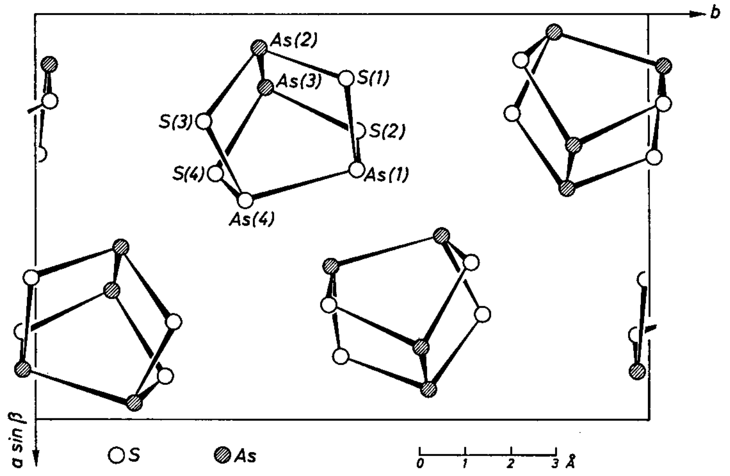

Fig. 1. Realgar. [001] projection

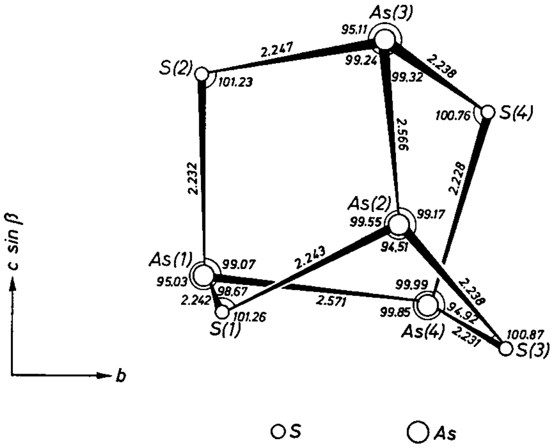

Fig. 2. As_4S_4 molecule showing cradle shape. [100] projection. (Bond distances in Å, angles in degrees)

in Fig. 1. This figure also shows that planes of weak van der Waals forces exist normal to the *b* axis giving rise to a fair cleavage on (010).

The As_4S_4 molecule has eight independent As—S bond distances, ranging from 2.228(2) Å to 2.247(2) Å and two As—As bond distances of 2.566(1) Å and 2.571(1) Å. The S—As—S angles range from 94.5° to 95.1°, the As—As—S angles from 98.7° to 100.0°, and the As—S—As angles from 100.8° to 101.3°.

Intermolecular contacts are all greater than 3.4 Å, the shortest being S(1)—As(3)III of 3.410(2) Å, S(2)—As(4)III of 3.442(2) Å and S(2)—As(1)III of 3.492(2) Å.

The sum of the van der Waals radii for sulfur and arsenic[4] is of the order of 3.85 Å, and therefore intermolecular contacts in excess of this value are not given in Table 4, being considered as non-bonding distances.

The shape of the molecule in the solid state is similar to its gaseous configuration, as described by LU and DONOHUE[5], and ALLEN and SUTTON[6]. The mean values of the intramolecular bond distances As—S and As—As are 2.237 Å and 2.569 Å respectively, while in the gas the corresponding distances are 2.23 Å and 2.49 Å. The mean bond angles in solid and gas also show close agreement, being as follows for the crystal and gas respectively: As—S—As, 101.03° and 101°; As—As—S, 99.36° and 100°; and S—As—S, 94.89° and 93°.

II. Orpiment, As_2S_3

Experimental

Crystal data: $a = 11.475 \pm 0.005$ Å, $b = 9.577 \pm 0.004$ Å, $c = 4.256 \pm 0.002$ Å, $\beta = 90°41' \pm 5'$; $V = 467.7$ Å^3; $F(000) = 456.0$; $Z = 4$; $\lambda(\mathrm{Cu}K\alpha) = 1.54718$; linear absorption coefficient, $\mu(\mathrm{Cu}K\alpha) = 298.8\ \mathrm{cm}^{-1}$.

The material from which a single crystal was chosen for data collection came also from Binnatal. Orpiment is a pale yellow mineral with perfect (010) cleavage. This cleavage led to difficulties in obtaining a suitable crystal, giving rise to streaking of spots in many cases.

The crystal used for data collection was a small, approximately cylindrical needle with a diameter of 0.014 mm and a length of 0.127 mm. Cell dimensions were obtained from measurements on back-reflection Weissenberg photographs, and a least-squares programme was used to obtain the best set of cell parameters from the measurements.

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[4] L. PAULING, *The nature of the chemical bond.* Ithaca: Cornell Univ. Press, p. 260 (1960).

[5] C. S. LU and J. DONOHUE, An electron diffraction investigation of sulfur nitride, arsenic disulfide (realgar), arsenic trisulfide (orpiment) and sulfur. J. Amer. chem. Soc. **66** (1944) 818—827.

[6] P. W. ALLEN and L. E. SUTTON, Tables of interatomic distances and molecular configurations obtained by electron diffraction in the gas phase. Acta Crystallogr. **3** (1950) 46—72, p. 67.

The space group is $P2_1/n$, with $h0l$ absent when $h + l = 2n + 1$ and $0k0$ absent when $k = 2n + 1$.

Intensity data were collected on a Supper-Pace two-circle diffractometer, Cu$K\alpha$ radiation being used. A total of 836 independent reflections ($\sin\theta < 0.707$, $\mu < 48°$) were measured about the a (needle) axis (layers $0kl$—$11\,k\,l$).

The intensities were adjusted to take account of background, and Lorentz and polarization corrections were made. Absorption corrections appropriate for a cylindrical crystal were applied, but no correction was made for secondary extinction effects.

Refinement of the structure

A preliminary comparison of the structure amplitudes (F_0) with those of MORIMOTO[2] showed no agreement whatever. A closer inspection revealed that the data were related by the transformation $h = H$, $k = K$, $l = -L$, where H,K,L refer to MORIMOTO's indices. Using MORIMOTO's coordinates as input for a structure-factor calculation, an initial R factor of 0.81 was obtained. The index transformation above indicates mis-indexing of the data by MORIMOTO, using (unintentionally) an unconventional cell with an acute angle β, although the cell dimensions are quoted conventionally[2]. This error probably arises because the angle β is measured as $90.5° \pm 0.5°$ and is very close to $90°$. Since $\beta \sim 90°$, the errors in calculated structure factors and in final positional parameters, as given by MORIMOTO, will be negligible.

Applying the above transformation ($x = X$, $y = Y$, $z = -Z$) to MORIMOTO's coordinates (X, Y, Z), and using the coordinates x,y,z as input for a structure-factor calculation, an initial and acceptable R factor of 0.23 was obtained.

Five rounds of isotropic least-squares refinement (block-diagonal approximation) using 586 reflections (and omitting the remainder, of which 224 had values of $F_0 < \sigma$ and 26 had values of $F_0 < 2\sigma$) reduced the R factor to 0.100. At this point, layer scales were adjusted with reference to calculated structure factors, since data about one axis only were available and no cross-level scaling could be applied. One further round of isotropic least-squares refinement reduced R to 0.094, since small adjustments only were required for the layer scale factors. Refinement was then continued anisotropically for a further six rounds, and the final R factor was 0.064. At this stage, all parameter shifts were negligible compared with the corresponding standard deviations.

Table 5. *Comparison of F_o and F_c values for orpiment*
* denotes unobserved reflections ($F_o < 3\sigma$)

h	k	l	$\lvert F_o \rvert$	F_c	h	k	l	$\lvert F_o \rvert$	F_c	h	k	l	$\lvert F_o \rvert$	F_c	h	k	l	$\lvert F_o \rvert$	F_c	h	k	l	$\lvert F_o \rvert$	F_c
0	0	2	72.6	67.7	1	5	3	17.2*	-21.8	-2	6	1	35.8	-42.2	3	6	4	29.2	-29.4	-4	8	1	8.5*	5.1
		4	45.0	-43.6	-1	5	3	3.0*	2.9	2	6	2	24.4*	-17.0	-3	6	4	0.2*	- 2.5	4	8	2	7.5*	- 2.4
0	1	1	11.0*	- 9.5	1	5	4	24.1*	-21.3	-2	6	2	20.1*	19.1	3	7	0	20.8*	-15.1	-4	8	2	0.2*	- 0.5
		2	121.8	126.5	-1	5	4	24.8*	-20.4	2	6	3	1.8*	3.8	3	7	1	72.8	74.3	4	8	3	14.6*	-17.7
		3	79.4	-76.7	1	6	0	32.9	-31.5	-2	6	3	23.0*	-21.0	-3	7	1	21.2*	19.1	-4	8	3	5.3*	4.7
		4	51.7	-48.4	1	6	1	66.7	68.0	2	6	4	7.2*	- 7.6	3	7	2	9.5*	9.9	4	9	0	21.5*	16.8
		5	24.1*	-27.2	-1	6	1	13.9*	14.8	-2	6	4	6.9*	- 4.9	-3	7	2	15.6*	-15.7	4	9	1	5.9*	- 1.6
0	2	0	177.8	-190.0	1	6	2	49.3	48.1	2	7	0	26.2*	-29.9	3	7	3	24.1*	-20.7	-4	9	1	12.5*	-11.5
		1	109.9	111.3	-1	6	2	12.8*	10.9	2	7	1	20.1*	22.3	-3	7	3	58.7	57.4	4	9	2	43.9	-46.8
		2	12.5*	7.0	1	6	3	18.4*	-11.4	-2	7	1	3.7*	0.6	3	7	4	7.5*	- 5.8	-4	9	2	63.1	-61.0
		3	39.9	39.2	-1	6	3	36.7	-37.7	2	7	2	32.1	-32.7	-3	7	4	18.2*	18.2	4	10	0	9.0*	5.4
		4	64.4	57.5	1	6	4	30.7	30.1	-2	7	2	48.1	46.9	3	8	0	18.9*	-17.3	4	10	1	44.0	-41.3
		5	39.8	-37.0	-1	6	4	21.1*	-19.0	2	7	3	4.0*	- 5.6	3	8	1	0.2*	- 0.6	-4	10	1	19.6*	-20.1
0	3	1	95.5	-99.6	1	7	0	8.4*	3.5	-2	7	3	33.1	-34.4	-3	8	1	71.5	-72.0	4	10	2	18.3*	-17.6
		2	40.1	-43.6	1	7	1	27.0*	27.1	2	7	4	26.0	-29.4	3	8	2	10.0*	7.4	-4	10	2	29.1	-25.8
		3	111.4	106.7	-1	7	1	48.4	55.5	-2	7	4	24.1	23.5	-3	8	2	29.2*	26.3	4	11	0	8.2*	- 7.4
		4	14.3*	12.0	1	7	2	38.8	-38.1	2	8	0	9.4*	5.7	3	8	3	45.4	45.7	5	0	1	38.9	39.4
		5	31.7	32.1	-1	7	2	32.1*	30.8	2	8	1	52.5	58.5	-3	8	3	20.0*	14.9	-5	0	1	0.2*	-13.3
0	4	0	66.2	-65.2	1	7	3	52.0	50.0	-2	8	1	14.9*	-13.5	3	9	0	35.9	-34.2	5	0	3	32.2	-31.9
		1	112.0	-114.1	-1	7	3	0.2*	- 7.2	2	8	2	22.8*	25.8	3	9	1	44.9	-44.3	-5	0	3	42.8	-42.0
		2	42.3	-38.3	1	7	4	11.6*	-12.7	-2	8	2	23.5*	-19.2	-3	9	1	18.9*	2.0	5	1	0	56.3	56.7
		3	35.0	-31.6	-1	7	4	0.2*	- 1.2	2	8	3	28.6	30.3	3	9	2	12.8*	15.8	5	1	1	7.1*	- 5.9
		4	24.9*	-24.2	1	8	0	10.8*	-12.2	-2	8	3	5.1*	- 7.2	-3	9	2	19.5*	-10.9	-5	1	1	36.5	42.2
0	5	1	165.3	166.4	1	8	1	35.6	-39.7	2	9	0	5.3*	4.0	3	9	3	24.8	25.2	5	1	2	26.6*	25.6
		2	49.2	-53.3	-1	8	1	26.7*	29.0	2	9	1	22.0*	-14.2	-3	9	3	35.4	-34.6	-5	1	2	22.3*	18.5
		3	33.0	-26.3	1	8	2	12.6*	- 6.9	-2	9	1	19.0*	-14.5	3	10	0	35.1	-32.9	5	1	3	30.0	26.3
		4	9.1*	11.0	-1	8	2	32.5*	29.7	2	9	2	10.9*	21.2	3	10	1	6.2*	12.4	-5	1	3	27.1*	-27.2
0	6	0	75.3	78.1	1	8	3	9.0*	13.1	-2	9	2	0.2*	- 0.9	-3	10	1	45.4	44.4	5	1	4	0.2*	- 4.0
		1	48.7	48.3	-1	8	3	53.4	52.0	2	9	3	8.1*	7.2	3	10	2	36.7	-35.9	-5	1	4	15.5*	11.8
		2	35.0	-34.6	1	9	0	41.1	-42.9	-2	9	3	22.5*	21.8	-3	10	2	29.0	-24.9	5	2	0	66.5	-69.9
		3	4.4*	7.5	1	9	1	6.9*	6.2	2	10	0	28.9	-23.6	3	11	0	21.5*	21.3	5	2	1	54.0	-52.3
		4	55.9	-54.9	-1	9	1	42.8	-47.0	2	10	1	36.4	-38.8	3	11	1	19.8*	21.1	-5	2	1	7.5*	-15.0
0	7	1	46.3	-50.1	1	9	2	0.2*	- 2.2	-2	10	1	9.7*	15.0	-3	11	1	28.9	-29.2	5	2	2	6.6*	-15.0
		2	86.9	83.5	-1	9	2	7.2*	- 5.4	2	10	2	14.5*	-11.8	4	0	2	96.1	-93.7	-5	2	2	42.0	43.2
		3	5.4*	1.6	1	9	3	31.3	-31.2	-2	10	2	2.2*	10.6	-4	0	2	92.8	-90.4	5	2	3	0.2*	3.3
		4	17.0*	-16.2	-1	9	3	0.2*	- 0.9	2	11	0	13.4*	9.6	4	0	4	102.3	-102.5	-5	2	3	2.1*	3.3
0	8	0	71.1	-73.1	1	10	0	7.5*	10.5	2	11	1	0.2*	2.5	-4	0	4	96.2	-90.1	5	2	4	18.4*	-22.4
		1	13.6*	12.6	1	10	1	11.8*	12.8	-2	11	1	12.9*	15.4	4	1	0	12.2*	- 5.3	-5	2	4	11.9*	-10.6
		2	32.2	31.5	-1	10	1	30.7	-33.0	3	0	1	65.3	60.8	4	1	1	80.0	78.3	5	3	0	74.2	-78.2
		3	10.8*	7.8	1	10	2	29.8	-28.8	-3	0	1	199.5*	214.1	-4	1	1	77.4	78.3	5	3	1	61.7	63.9
0	9	1	44.6	-43.2	-1	10	2	23.9	-17.6	3	0	3	60.2	-58.4	4	1	2	149.5	150.2	-5	3	1	46.2	-48.5
		2	44.6	-45.4	1	11	0	39.0	37.5	-3	0	3	23.9*	-22.1	-4	1	2	144.2	142.4	5	3	2	34.5	-36.7
		3	7.4*	6.3	1	11	1	10.0*	- 7.1	3	0	5	33.0	36.6	4	1	3	10.6*	-11.3	-5	3	2	6.6*	- 6.4
0	10	0	69.9	69.1	-1	11	1	0.2*	1.3	-3	0	5	45.6	-46.7	-4	1	3	12.5*	-10.6	5	3	3	17.8*	23.4
		1	35.6	-37.5	2	0	2	48.1	-46.4	3	1	0	35.8	37.0	4	1	4	0.2*	1.0	-5	3	3	8.3*	2.7
		2	22.1*	20.2	-2	0	2	36.9	42.3	3	1	1	193.9	197.3	-4	1	4	0.2*	- 5.4	5	3	4	17.9*	18.8
0	11	1	27.8	26.9	2	0	4	12.5*	- 5.4	-3	1	1	49.0	-49.2	4	2	0	86.9	-96.1	-5	3	4	7.5*	- 7.5
1	0	1	128.0	132.2	-2	0	4	8.1*	6.5	3	1	2	51.0	-53.5	4	2	1	91.4	94.8	5	4	0	33.8	37.9
-1	0	1	44.4	-46.8	2	1	0	46.2	52.0	-3	1	2	12.3*	11.8	-4	2	1	89.5	93.8	5	4	1	35.6	33.9
1	0	3	8.4*	- 6.3	2	1	1	9.7*	-13.3	3	1	3	28.4	-19.1	4	2	2	22.6*	21.7	-5	4	1	22.6*	22.8
-1	0	3	100.2	-92.2	-2	1	1	20.9*	26.3	-3	1	3	51.7	50.0	-4	2	2	25.2*	28.3	5	4	2	23.4*	-20.6
1	0	5	21.8	-18.7	2	1	2	8.3*	8.7	3	1	4	23.1*	-21.6	4	2	3	42.9	41.7	-5	4	2	6.7*	- 9.2
-1	0	5	19.0*	14.7	-2	1	2	17.1*	-15.3	-3	1	4	37.2	-33.9	-4	2	3	41.9	41.8	5	4	3	33.8	30.6
1	1	0	29.7*	30.5	2	1	3	26.8*	-20.0	3	1	5	44.9	-48.0	4	2	4	48.0	46.2	-5	4	3	37.8	34.8
1	1	1	47.0	46.8	-2	1	3	14.1*	12.0	-3	1	5	26.7	-29.0	-4	2	4	40.5	40.4	5	4	4	6.6*	- 7.3
-1	1	1	106.9	114.0	2	1	4	16.3*	5.2	3	2	0	65.4	70.1	4	3	0	3.4*	4.9	-5	4	4	34.5	32.4
1	1	2	21.8*	17.0	-2	1	4	9.7*	- 9.0	3	2	1	11.0*	- 8.4	4	3	1	91.7	-93.4	5	5	0	14.9*	15.3
-1	1	2	45.6	-48.2	2	1	5	5.4*	- 4.7	-3	2	1	105.3	-111.8	-4	3	1	91.7	-92.9	5	5	1	25.8*	-30.2
1	1	3	92.0	89.6	-2	1	5	3.4*	4.0	3	2	2	40.5	41.8	4	3	2	53.6	-54.2	-5	5	1	11.9*	-14.5
-1	1	3	0.2*	-10.7	2	2	0	12.3*	6.8	-3	2	2	86.6	87.3	-4	3	2	42.7	-45.7	5	5	2	13.6*	12.3
1	1	4	21.5*	-18.4	2	2	1	76.8	-78.4	3	2	3	56.1	51.3	4	3	3	69.4	68.9	-5	5	2	28.4	-30.7
-1	1	4	22.8*	-19.3	-2	2	1	89.3	94.8	-3	2	3	21.5*	24.3	-4	3	3	71.8	61.7	5	5	3	8.8*	-16.2
1	1	5	11.1*	- 7.1	2	2	2	30.0	34.9	3	2	4	56.6	-61.0	4	3	4	4.0*	- 5.4	-5	5	3	2.9*	- 8.3
-1	1	5	22.3	-20.8	-2	2	2	41.7	-40.5	-3	2	4	16.6*	26.9	-4	3	4	0.2*	3.6	5	5	4	28.2	-29.3
1	2	0	34.6	-38.3	2	2	3	58.8	-60.3	3	2	5	17.6*	-17.2	4	4	0	21.4*	22.4	-5	5	4	8.2*	- 9.2
1	2	1	76.5	-79.7	-2	2	3	62.5	61.0	-3	2	5	37.9	37.9	4	4	1	99.1	-102.3	5	6	0	22.3*	-29.0
-1	2	1	51.3	48.1	2	2	4	6.9*	7.7	3	3	0	116.0	-123.3	-4	4	1	119.3	-123.7	5	6	1	27.6	28.0
1	2	2	88.7	88.8	-2	2	4	20.9*	-18.7	3	3	1	43.8	-46.8	4	4	2	32.9	35.4	-5	6	1	22.3*	18.9
-1	2	2	41.8	43.5	2	2	5	24.4	-25.7	-3	3	1	13.8*	9.0	-4	4	2	17.0*	22.6	5	6	2	0.2*	2.6
1	2	3	0.2*	- 3.1	-2	2	5	14.5*	17.5	3	3	2	74.8	76.7	4	4	3	59.9	-59.5	-5	6	2	19.4*	-12.3
-1	2	3	65.1	66.9	2	3	0	82.3	88.9	-3	3	2	54.3	-56.2	-4	4	3	68.7	-63.1	5	6	3	15.9*	-17.1
1	2	4	23.8*	23.2	2	3	1	32.3	33.6	3	3	3	26.4*	22.6	4	4	4	19.9*	20.6	-5	6	3	17.5*	-14.0
-1	2	4	28.5*	-28.6	-2	3	1	38.5	-36.1	-3	3	3	34.8	-30.6	-4	4	4	25.5*	22.4	5	6	4	13.0*	15.3
1	2	5	13.3*	11.7	2	3	2	79.7	82.6	3	3	4	26.4*	24.3	4	5	0	22.2*	-17.7	-5	6	4	20.5	-25.2
-1	2	5	10.7*	- 7.2	-2	3	2	46.5	-46.6	-3	3	4	67.1	60.9	4	5	1	14.9*	11.7	5	7	0	8.2*	8.7
1	3	0	46.4	-50.9	2	3	3	36.1	35.4	3	4	0	112.2	-119.1	-4	5	1	3.4*	4.9	5	7	1	21.0*	-17.7
1	3	1	26.6	-24.5	-2	3	3	26.7*	-19.8	3	4	1	16.1*	-16.6	4	5	2	50.9	-50.7	-5	7	1	46.2	47.9
-1	3	1	16.7*	-15.8	2	3	4	28.1*	31.6	-3	4	1	18.2*	-16.0	-4	5	2	30.0	-34.3	5	7	2	11.4*	-13.6
1	3	2	33.9	-36.5	-2	3	4	35.5	-32.6	3	4	2	65.4	-63.6	4	5	3	93.5	-95.0	-5	7	2	16.3*	12.0
-1	3	2	103.4	104.1	-2	3	5	0.2*	- 1.7	-3	4	2	63.6	-64.2	-4	5	3	94.0	-88.8	5	7	3	8.5*	- 2.8
1	3	3	49.0	-48.3	2	4	0	28.4	-28.5	3	4	3	0.2*	2.7	4	5	4	9.0*	10.8	-5	7	3	6.7*	6.2
-1	3	3	13.9*	15.9	2	4	1	2.1*	-10.4	-3	4	3	0.2*	1.0	-4	5	4	11.4*	12.8	5	8	0	42.8	39.2
1	3	4	38.8	40.4	-2	4	1	9.7*	10.5	3	4	4	70.7	67.9	4	6	0	98.1	98.5	5	8	1	48.9	-47.9
-1	3	4	40.0	37.5	2	4	2	11.5*	- 8.7	-3	4	4	10.5*	-14.0	4	6	1	57.3	57.7	-5	8	1	21.8*	-23.6
1	3	5	3.9*	- 8.3	-2	4	2	16.1*	13.8	3	5	0	110.1	116.1	-4	6	1	64.4	61.0	5	8	2	42.2	41.0
-1	3	5	8.4*	4.7	2	4	3	11.3*	12.1	3	5	1	65.1	-66.8	4	6	2	1.8*	- 0.8	-5	8	2	0.2*	5.9
1	4	0	70.0	72.5	-2	4	3	9.7*	- 8.9	-3	5	1	7.4*	- 6.5	-4	6	2	5.0*	4.2	5	8	3	8.2*	- 6.5
1	4	1	23.3*	-22.4	2	4	4	8.1*	- 4.0	3	5	2	31.8	-31.8	4	6	3	48.5	51.6	-5	8	3	0.2*	- 4.1
-1	4	1	44.6	-45.4	-2	4	4	29.9	22.7	-3	5	2	67.4	66.7	-4	6	3	37.3	32.4	5	9	0	5.6*	0.6
1	4	2	84.3	-84.5	2	5	0	36.9	-38.9	3	5	3	3.8*	- 8.9	4	6	4	25.4	-24.9	5	9	1	16.2	15.2
-1	4	2	59.2	-60.0	2	5	1	33.4	-34.3	-3	5	3	25.3*	-21.4	-4	6	4	29.6	-27.0	-5	9	1	15.7*	-15.2
1	4	3	2.6*	9.8	-2	5	1	16.8*	14.4	3	5	4	5.9*	2.0	4	7	0	8.7*	4.1	5	9	2	26.4	23.2
-1	4	3	10.0*	-12.3	2	5	2	39.6	-40.1	-3	5	4	42.8	-42.7	4	7	1	0.2*	- 0.6	-5	9	2	21.3*	19.8
1	4	4	33.4	-26.6	-2	5	2	0.2*	- 1.6	3	6	0	92.3	95.1	-4	7	1	11.4*	13.9	5	10	0	0.2*	- 5.1
-1	4	4	32.3*	26.5	2	5	3	19.3*	-14.5	3	6	1	10.5*	-10.4	4	7	2	76.6	77.8	5	10	1	15.7*	13.9
1	5	0	28.4*	35.1	-2	5	3	24.9*	23.2	-3	6	1	57.9	59.2	-4	7	2	70.4	70.2	-5	10	1	10.9*	-11.0
1	5	1	28.2*	-27.5	2	5	4	0.2*	- 2.4	3	6	2	42.3	45.4	4	7	3	21.2*	21.6	-5	10	2	13.3*	-13.4
-1	5	1	32.6	-36.5	-2	5	4	10.2*	11.6	-3	6	2	0.2*	- 1.6	-4	7	3	30.0	27.5	6	0	2	48.6	-50.5
1	5	2	48.2	46.3	2	6	0	19.6*	26.5	3	6	3	60.0	-62.9	4	8	0	84.8	-87.6	-6	0	2	44.4	41.1
-1	5	2	81.0	-80.6	2	6	1	5.4*	8.6	-3	6	3	38.2	-31.9	4	8	1	4.5*	1.7	6	0	4	22.7*	26,1

Table 5. *(Continued)*

h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c
-6	0	4	27.0	-23.5	6	9	1	0.2*	1.4	7	8	0	3.3*	4.0	-8	8	1	3.6*	-7.7	10	2	1	32.7	31.0
6	1	0	72.5	83.0	-6	9	1	27.0	-27.0	7	8	1	0.2*	-1.1	8	8	2	2.9*	0.7	-10	2	1	18.0*	-23.1
6	1	1	20.0	-19.1	6	9	2	7.3*	5.1	-7	8	1	68.8	-68.3	-8	8	2	9.7*	8.3	10	2	2	48.0	46.4
-6	1	1	11.9*	18.8	-6	9	2	17.7*	10.5	7	8	2	1.7*	2.0	8	9	0	3.3*	-5.4	-10	2	2	48.2	-50.0
6	1	2	6.9*	-6.9	6	10	0	2.2*	-6.6	-7	8	2	6.8*	6.0	8	9	1	3.3*	-1.3	10	2	3	36.9	-32.2
-6	1	2	9.5*	8.7	6	10	1	14.2*	-10.6	7	9	0	19.4*	-13.2	-8	9	1	18.7*	-17.0	-10	2	3	27.8*	26.6
6	1	3	8.6*	-7.9	-6	10	1	10.6*	-7.9	7	9	1	50.2	-46.9	9	0	1	16.6*	16.1	10	3	0	15.7*	-13.5
-6	1	3	0.2*	-1.1	7	0	1	71.7	-67.8	-7	9	1	4.0*	-7.9	-9	0	1	14.6*	9.7	10	3	1	57.3	52.2
6	1	4	37.8	-40.5	-7	0	1	82.8	89.2	-7	9	2	0.2*	0.6	9	0	3	24.8	23.4	-10	3	1	49.5	-47.6
-6	1	4	35.5	35.4	7	0	3	140.9	-139.4	7	10	0	15.7*	-14.9	-9	0	3	10.3*	9.8	10	3	2	14.7*	15.7
6	2	0	14.2*	15.6	-7	0	3	31.2	-31.7	8	0	2	19.2*	18.6	9	1	0	19.1*	-26.5	-10	3	2	3.9*	-1.6
6	2	1	85.7	85.4	7	1	0	58.4	63.3	-8	0	2	13.6*	5.3	9	1	1	0.1*	-4.2	10	3	3	37.5	38.2
-6	2	1	68.5	-68.2	7	1	1	84.4	85.9	8	0	4	32.0	-36.0	-9	1	1	12.7*	11.5	-10	3	3	15.8*	-19.2
6	2	2	35.8	33.4	-7	1	1	61.1	60.9	-8	0	4	28.7	-33.4	9	1	2	22.7*	-22.4	10	4	0	13.3	-9.4
-6	2	2	12.0*	-19.1	7	1	2	15.3*	-16.6	8	1	0	0.2*	-3.6	-9	1	2	23.5*	-24.1	10	4	1	29.4	-26.2
6	2	3	2.0*	-2.4	-7	1	2	42.5	40.8	8	1	1	16.3*	20.4	9	1	3	4.5*	-0.8	-10	4	1	19.7*	22.7
-6	2	3	10.2*	7.6	7	1	3	25.8*	-28.0	-8	1	1	20.3*	21.4	-9	1	3	8.1*	14.0	10	4	2	9.5*	0.7
6	2	4	19.1*	-22.1	-7	1	3	114.8	102.5	8	1	2	59.9	59.1	9	2	0	34.2	39.2	-10	4	2	9.9*	-1.1
-6	2	4	24.4*	17.8	7	1	4	5.7*	1.9	-8	1	2	49.3	53.6	9	2	1	14.6	15.0	-10	4	3	18.6*	-35.3
6	3	0	68.6	-69.8	-7	1	4	14.9*	-9.6	8	1	3	26.0*	-26.4	-9	2	1	27.9	27.9	10	5	0	28.1	-25.3
6	3	1	31.9	32.0	7	2	0	86.8	89.4	-8	1	3	23.9*	-22.4	9	2	2	35.5	39.8	10	5	1	48.0	-46.4
-6	3	1	47.7	-47.7	7	2	1	15.6*	18.0	8	1	4	25.5	-26.3	-9	2	2	4.5*	-14.9	-10	5	1	38.5	39.6
6	3	2	22.7*	-22.6	-7	2	1	80.4	-82.6	-8	1	4	23.1*	-22.2	9	2	3	4.9*	-4.1	10	5	2	4.7*	-3.8
-6	3	2	42.5	37.0	7	2	2	76.2	74.2	8	2	0	74.7	-80.6	-9	2	3	16.6*	13.3	-10	5	2	14.9*	-11.4
6	3	3	5.6*	7.5	-7	2	2	43.6	41.5	8	2	1	47.7	46.2	9	3	0	23.3	25.1	10	6	0	22.8*	16.1
-6	3	3	6.4*	-7.7	7	2	3	72.0	70.5	-8	2	1	36.0	39.1	9	3	1	26.1	-24.5	10	6	1	4.2*	-2.3
6	3	4	8.3*	-7.5	-7	2	3	4.9*	4.2	8	2	2	0.2*	-10.7	-9	3	1	24.7	23.9	-10	6	1	12.3*	-9.1
-6	3	4	0.2*	0.2	7	2	4	22.4	-23.5	-8	2	2	9.2*	4.0	9	3	2	15.5*	17.0	10	6	2	30.9	-28.9
6	4	0	11.3*	-16.9	-7	2	4	0.2*	-0.8	8	2	3	7.6*	12.7	-9	3	2	26.1	23.9	-10	6	2	29.3	34.8
6	4	1	50.5	-52.2	7	3	0	84.1	-88.1	-8	2	3	9.8*	10.7	9	3	3	30.5	-33.0	10	7	0	36.6	39.7
-6	4	1	51.1	48.2	7	3	1	55.8	-56.2	8	2	4	28.2	30.4	-9	3	3	11.1*	14.4	10	7	1	7.8*	7.8
6	4	2	0.2*	14.6	-7	3	1	1.7*	10.1	-8	2	4	26.5*	25.3	9	4	0	1.6*	-1.9	-10	7	1	14.4*	-18.0
-6	4	2	6.7*	-5.5	7	3	2	79.3	80.1	8	3	0	5.5*	11.5	9	4	1	26.6	-27.9	11	0	1	32.5	35.0
6	4	3	41.4	36.9	-7	3	2	60.6	-59.9	8	3	1	63.1	-63.4	-9	4	1	42.3	-47.0	-11	0	1	85.4	97.7
-6	4	3	31.6	-31.7	7	3	3	18.0*	-10.7	-8	3	1	56.3	-57.8	9	4	2	8.8*	-5.8	-11	0	3	0.2*	-8.5
6	4	4	17.0*	16.8	-7	3	3	14.6*	-13.9	8	3	2	11.3*	-14.3	-9	4	2	10.3*	-5.8	11	1	0	12.1*	10.3
-6	4	4	0.2*	4.2	7	3	4	25.6	26.0	-8	3	2	0.2*	-14.5	9	4	3	14.2*	-5.4	11	1	1	95.6	97.3
6	5	0	11.7*	-13.4	-7	3	4	34.8	33.6	8	3	3	41.4	42.8	-9	4	3	19.1*	-21.8	-11	1	1	23.2	-19.8
6	5	1	34.1	-32.0	7	4	0	51.0	-53.8	-8	3	3	44.5	41.8	9	5	0	9.1*	3.1	11	1	2	34.6	-34.4
-6	5	1	23.9*	26.3	7	4	1	18.8*	17.1	-8	3	4	15.1*	14.0	9	5	1	4.6*	-3.7	-11	1	2	11.2*	1.6
6	5	2	0.2*	-1.2	-7	4	1	17.3*	14.6	8	4	0	3.3*	-2.1	-9	5	1	5.5*	-9.4	-11	1	3	12.9*	23.8
-6	5	2	26.7*	-28.8	7	4	2	49.6	-51.0	8	4	1	61.0	-59.7	9	5	2	15.2*	20.7	11	2	0	23.7	24.0
6	5	3	5.9*	2.2	-7	4	2	100.5	-96.2	-8	4	1	40.9	-40.9	-9	5	2	0.1*	2.6	11	2	1	11.4*	-8.7
-6	5	3	10.0*	9.4	7	4	3	17.8*	18.8	8	4	2	11.7*	12.0	9	5	3	16.2*	13.1	-11	2	1	45.5	-50.4
6	5	4	16.9*	20.5	-7	4	3	20.8*	19.9	-8	4	2	11.3*	-7.9	-9	5	3	16.2*	-13.3	11	2	2	14.1*	13.9
-6	5	4	10.5*	-13.2	7	4	4	39.9	46.3	8	4	3	22.5*	-23.9	9	6	0	19.5*	-18.1	-11	2	2	48.7	49.1
6	6	0	3.8*	4.0	-7	4	4	27.5	-30.4	-8	4	3	8.7*	-5.5	9	6	1	10.8*	-10.9	11	3	0	47.5	-47.8
6	6	1	15.8*	12.1	7	5	0	29.4	26.6	8	5	0	2.9*	1.6	-9	6	1	8.1*	-0.5	11	3	1	15.8*	-14.0
-6	6	1	27.7*	-30.7	7	5	1	35.9	-34.6	8	5	1	74.7	73.3	9	6	2	13.9*	-10.1	-11	3	1	11.1*	-12.7
6	6	2	42.5	-41.3	-7	5	1	18.8*	-18.2	-8	5	1	55.6	55.6	-9	6	2	4.6*	5.1	11	3	2	53.5	48.5
-6	6	2	19.7*	16.2	7	5	2	104.6	-106.1	8	5	2	21.4*	-17.5	9	7	0	5.2*	19.3	-11	3	2	21.3	-19.5
6	6	3	28.3	-28.0	-7	5	2	26.2*	25.0	-8	5	2	35.8	-36.6	9	7	1	26.7	27.8	11	4	0	66.3	-65.6
-6	6	3	21.7*	18.9	7	5	3	12.9*	14.4	8	5	3	19.2*	-19.8	-9	7	1	17.0*	-15.6	11	4	1	13.5*	-10.0
6	7	0	66.4	66.1	-7	5	3	29.3	-32.5	-8	5	3	28.7	-27.2	9	7	2	15.5*	-13.5	-11	4	1	19.9*	-17.8
6	7	1	26.7	29.5	7	6	0	6.5*	1.5	8	6	0	44.5	44.1	-9	7	2	10.8*	15.0	11	4	2	51.5	-48.3
-6	7	1	18.4*	23.7	7	6	1	11.3*	10.3	8	6	1	16.0*	13.1	9	8	0	3.3*	-1.3	-11	4	2	34.2*	-28.3
6	7	2	21.7*	20.7	-7	6	1	74.4	73.9	-8	6	1	36.6	33.4	9	8	1	16.8*	16.1	11	5	0	67.0	65.5
-6	7	2	7.2*	-5.7	7	6	2	6.7*	7.5	8	6	2	13.5*	-19.0	-9	8	1	23.0	22.0	11	5	1	30.5	-29.4
6	7	3	0.2*	-8.7	-7	6	2	77.6	73.2	-8	6	2	0.2*	-4.6	10	0	2	81.3	-77.9	-11	5	1	0.2*	0.1
-6	7	3	0.2*	3.6	7	6	3	36.1	-36.5	8	6	3	5.4*	7.9	-10	0	2	68.1	70.5	11	5	2	11.2*	-10.9
6	8	0	4.8*	-6.6	-7	6	3	10.2*	-7.0	-8	6	3	9.0*	2.0	10	1	0	40.6	44.4	-11	5	2	34.2*	31.9
6	8	1	2.3*	-6.1	7	7	0	3.3*	-2.0	8	7	0	0.2*	-0.6	10	1	1	19.2*	-18.6	11	6	0	46.2	47.3
-6	8	1	34.6	33.5	7	7	1	90.8	89.7	8	7	1	36.9	-34.4	-10	1	1	24.9	25.0	11	6	1	2.6*	-7.9
6	8	2	19.7*	17.2	-7	7	1	4.9*	-6.2	-8	7	1	15.4*	-13.0	10	1	2	28.1	-26.6	-11	6	1	32.9	35.6
-6	8	2	29.9	-28.6	7	7	2	32.0	32.5	8	7	2	40.1	37.6	-10	1	2	24.4	21.4					
6	8	3	6.4*	-11.3	-7	7	2	11.9*	-13.0	-8	7	2	39.8	38.5	10	1	3	18.1*	-13.2					
-6	8	3	14.9*	18.9	7	7	3	4.1*	3.8	8	8	0	50.9	-50.8	-10	1	3	13.6*	8.2					
6	9	0	38.8	-36.8	-7	7	3	23.4	30.8	8	8	1	24.6*	24.3	10	2	0	0.2*	-5.4					

The final R factor for all 836 reflections was 0.089. Table 5 is a listing of structure factors. The final coordinates and temperature factors are given in Table 6a, and **Morimoto**'s coordinates, for comparison, appear in Table 6b. Bond distances and angles are given in Table 8.

Results and discussion

The structure obtained by refinement is basically the same as that determined by **Morimoto**[2]. It is essentially a layer-like structure of covalently-bonded arsenic and sulfur atoms (Fig. 4). The layers are normal to the [010] direction and are held together by van der Waal's forces. Perfect cleavage thus occurs normal to the [010] direction. The

Table 6a. 1. *Fractional positional coordinates*
(standard deviations in brackets)

Atom	x	y	z
As(1)	0.26469 (15)	0.19171 (22)	0.86274 (41)
As(2)	0.48677 (15)	0.32122 (22)	0.36072 (41)
S(1)	0.40151 (39)	0.12128 (47)	0.50811 (98)
S(2)	0.34738 (39)	0.39723 (51)	0.01011 (105)
S(3)	0.12234 (37)	0.29354 (48)	0.55896 (96)

2. *Anisotropic temperature factors* (estimated standard deviations in brackets)
Temperature factors given by:

$$T = \exp\left[-\left(h^2B_{11} + k^2B_{22} + l^2B_{33} + 2hkB_{12} + 2hlB_{13} + 2klB_{23}\right)\right]$$

Atom	B_{11}	B_{12}	B_{13}
As(1)	0.00372 (25)	− 0.00025 (34)	− 0.00344 (56)
As(2)	0.00397 (25)	− 0.00029 (34)	− 0.00338 (54)
S(1)	0.00461 (41)	0.00167 (70)	0.00022 (145)
S(2)	0.00446 (41)	0.00109 (67)	− 0.00656 (155)
S(3)	0.00388 (40)	0.00073 (66)	− 0.00465 (141)

Atom	B_{22}	B_{23}	B_{33}
As(1)	0.00633 (36)	0.00187 (89)	0.02790 (170)
As(2)	0.00596 (34)	− 0.00169 (85)	0.02775 (171)
S(1)	0.00516 (53)	− 0.00085 (175)	0.02551 (255)
S(2)	0.00602 (60)	0.00055 (181)	0.03064 (262)
S(3)	0.00629 (62)	0.00547 (173)	0.02758 (256)

Table 6b. *Fractional positional coordinates* (Morimoto[2])

	x	y	z*
As(1)	0.267	0.190	0.857
As(2)	0.484	0.323	0.357
S(1)	0.395	0.120	0.500
S(2)	0.355	0.397	0.987
S(3)	0.125	0.293	0.590

* z coordinates converted from Morimoto's cell to cell defined in this study.

layers themselves are considered by Morimoto[2] to be composed of As—S—As—S chains with a sulfur bridge between arsenic atoms in parallel neighbouring chains. These chains lie parallel with the c direction (Fig. 4).

Six independent As—S distances occur in the As_2S_3 layers, ranging from 2.243(5) Å to 2.308(5) Å. The shortest non-bonded contacts

Table 7. *Parameters defining temperature ellipsoids of atoms*
(Isotropic temperature factors B; u = thermal displacement parameters)

Atom	$\langle B \rangle$	Axis	B	u	cos(1)	cos(2)	cos(3)
As(1)	2.10 Å^2	1	1.63 Å^2	.144	.716	−.093	.691
		2	2.19	.167	.518	.735	−.438
		3	2.49	.178	−.467	.672	.575
As(2)	2.10	1	2.42	.175	.704	.211	−.678
		2	2.22	.168	−.334	.941	−.054
		3	1.66	.145	.626	.265	.733
S(1)	2.06	1	2.62	.182	.889	.456	−.042
		2	1.68	.146	−.414	.840	.350
		3	1.87	.154	.195	−.294	.936
S(2)	2.26	1	2.99	.195	.748	.192	−.635
		2	2.24	.168	.053	.937	.346
		3	1.56	.141	.661	−.292	.691
S(3)	2.12	1	2.68	.184	.350	−.654	−.671
		2	2.33	.172	.744	.629	−.226
		3	1.35	.131	.570	−.420	.706

Table 8. *Bond distances and angles*
(estimated standard deviations in brackets)

a) Covalent-bond distances

As(1)—S(1)	2.292 (5) Å	As(2)—S(1)	2.243 (5) Å
As(1)—S(2)	2.270 (5)	As(2)—S(2)	2.293 (5)
As(1)—S(3)	2.289 (5)	As(2)—S(3)II	2.308 (5)

b) Bond angles

As(1)—S(1)—As(2)	103.7 (2)°	S(2)—As(1)—S(3)	94.6 (2)°
As(1)—S(2)—As(2)	101.0 (2)	S(3)—As(1)—S(1)	104.1 (2)
As(1)—S(3)—As(2)II	87.9 (2)	S(1)—As(2)—S(2)	98.6 (2)
S(1)—As(1)—S(2)	98.6 (2)	S(2)—As(2)—S(3)II	105.0 (2)
		S(3)II—As(2)—S(1)	92.8 (2)

c) Non-bonded distances

As(1)—As(2)II	3.191 (2) Å	S(2)—S(3)	3.641 (6) Å
As(1)—As(2)	3.568 (3)	S(3)—S(1)II	3.295 (6)
As(1)—As(2)	3.521 (3)	S(3)—S(1)II	3.527 (6)
S(1)—S(2)	3.440 (6)	As(1)—S(2)IV	3.475 (5)
S(1)—S(2)	3.459 (6)	S(1)—S(1)III	3.242 (6)
S(2)—S(3)	3.350 (6)		

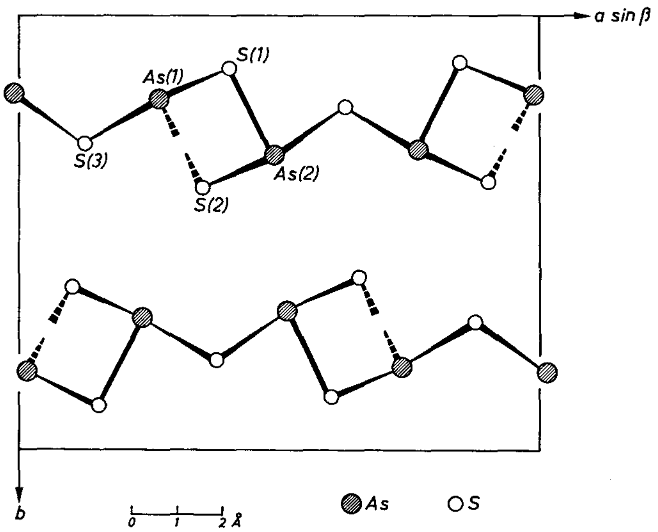

Fig. 3. Orpiment. [001] projection

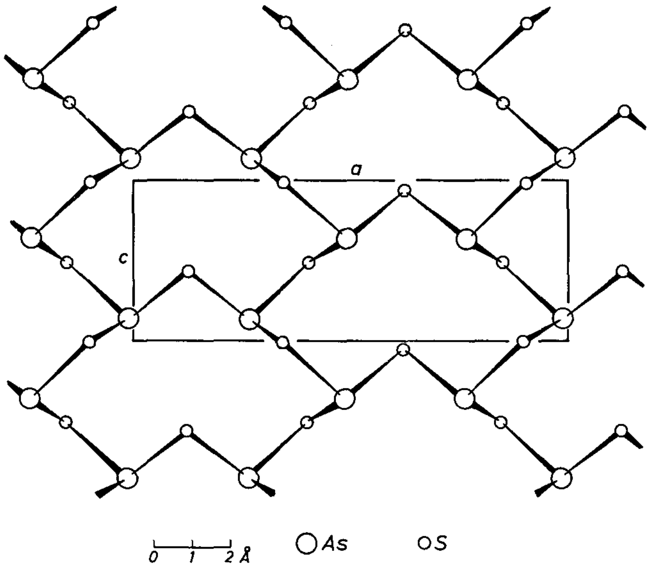

Fig. 4. Orpiment layer structure. [010] projection

between the layers are distances of 3.242(6) Å between S(1)—S(1)III and 3.475(5) Å between As(1)—S(2)IV.

An interesting feature of the structure is the large difference between the bond angle of the sulfur atom bridging the spiral chains

[S(3)] and the bond angles of the other two sulfur atoms. The angle As(1)—S(3)—As(2) is 87.9(2)°, while the angles As(1)—S(1)—As(2) and As(1)—S(2)—As(2) are 103.7(2)° and 101.0(2)° respectively.

Comparison of the structure of realgar and orpiment

Realgar shows certain striking structural similarities to orpiment, although these are not apparent from a cursory inspection of the two structures. Realgar consists of discrete As_4S_4 units, while orpiment is a layer-like structure. However, ITO *et al.* have shown[1] that both structures may be built from the same basic (As_2S_2) structural units, giving rise to similar bond distances and angles in both cases (as a comparison of Table 4 with Table 8 will show). In the case of orpiment an additional sulfur atom is added to each As_2S_2 unit to account for the required stoichiometry.

The sulfides of arsenic also bear comparison with arsenic oxides. Thus arsenolite, As_4O_6, also has a cradle-type structure, with additional oxygen atoms attached to the arsenic atoms, while the other modification of As_2O_3 (claudetite) has an infinite layer structure.

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