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

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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, $\beta = 106° 23' \pm 5'$; $V = 799.7$ 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

[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
0	0	2	243.4*	-283.0
		4	8.3*	- 5.3
		6	18.3	18.5
0	1	1	113.8	114.6
		2	85.4	85.7
		3	78.1	-82.3
		4	50.0	-53.1
		5	17.3	-17.8
		6	63.4	62.2
		7	25.6	24.2
0	2	0	51.2	49.3
		1	66.2	-67.6
		2	99.1	-104.7
		3	14.8*	-13.4
		4	46.7	50.0
		5	18.9	19.8
		6	32.6	-34.5
		7	29.7	30.2
0	3	1	57.9	-59.9
		2	53.6	-55.8
		3	36.0	35.9
		4	49.6	-51.0
		5	5.0*	0.4
		6	39.0	38.1
		7	34.9	-34.5
0	4	0	19.9	-21.4
		1	135.1	147.6
		2	26.6	29.8
		3	97.6	-97.8
		4	62.1	61.5
		5	4.6*	4.0
		6	71.7	-68.5
		7	12.1*	-12.8
0	5	1	17.1	-17.6
		2	8.5*	6.7
		3	105.3	106.0
		4	41.6	42.5
		5	48.2	-44.6
		6	46.5	-43.7
		7	10.9*	-10.2
0	6	0	213.9	232.6
		1	37.8	39.9
		2	143.8	-150.1
		3	28.5	-29.9
		4	41.5	44.1
		5	59.9	57.9
		6	18.9	-19.8
		7	16.2	-16.1
0	7	1	56.1	56.3
		2	31.5	-31.8
		3	101.0	-97.8
		4	9.2*	- 3.1
		5	28.3	-28.0
		6	33.7	31.4
0	8	0	180.7	183.9
		1	28.8	-30.1
		2	103.2	-98.1
		3	48.9	-45.7
		4	33.2	-33.0
		5	12.1*	11.5
		6	40.4	36.5
0	9	1	34.3	27.9
		2	35.6	-32.7
		3	21.9	-20.7
		4	48.6	-48.3
		5	37.9	35.3
		6	45.0	42.3
0	10	0	38.7	-33.9
		1	82.9	84.4
		2	29.2	27.3
		3	110.2	-103.6
		4	28.0	26.6
		5	65.1	58.9
		6	18.5	-18.1
0	11	1	47.7	-47.2
		2	8.0*	7.9
		3	58.1	58.3
		4	8.3*	5.8
		5	27.6	-27.2
0	12	0	33.7	-32.1
		1	94.5	95.7
		2	30.7	-30.8
		3	43.7	-44.2
		4	39.1	42.0
		5	4.3*	4.2
0	13	1	29.8	-28.0
		2	7.6*	-10.1
		3	47.7	48.7
		4	16.8	-17.6
0	14	0	56.2	56.0
		1	28.5	-27.5
		2	2.9*	- 2.5
		3	20.1	-20.6
		4	21.1	-23.3
0	15	1	30.0	30.5
		2	14.4	-14.6
		3	36.0	-37.3
0	16	0	19.4	-19.6
		1	25.4	25.6
		2	3.6*	0.4
1	0	1	55.1	48.0

h	k	l	$\lvert F_o \rvert$	F_c
-1	0	1	133.2	-148.6
1	0	3	38.9	42.6
-1	0	3	207.8*	224.3
1	0	5	5.8*	- 2.6
-1	0	5	55.6	-54.5
1	0	7	42.1	-44.0
-1	0	7	12.0*	12.2
1	1	0	34.2	37.0
1	1	1	8.4*	- 1.0
-1	1	1	86.1	-90.7
1	1	2	135.5	-139.6
-1	1	2	42.8	38.6
1	1	3	62.8	62.0
-1	1	3	116.8	119.0
1	1	4	108.4	114.5
-1	1	4	54.0	56.7
1	1	5	6.5*	2.8
-1	1	5	38.3	-39.3
1	1	6	56.1	-54.8
-1	1	6	51.9	-50.1
1	1	7	3.3*	- 1.6
-1	1	7	0.0*	- 2.4
1	2	0	155.8	182.5
1	2	1	49.5	49.2
-1	2	1	23.8	-23.4
1	2	2	36.8	36.7
-1	2	2	143.8	159.2
1	2	3	146.7	-156.8
-1	2	3	14.5*	10.2
1	2	4	66.5	-70.2
-1	2	4	6.8*	4.5
1	2	5	92.0	96.0
-1	2	5	31.0	-32.2
1	2	6	52.6	53.9
-1	2	6	38.5	-37.6
1	2	7	8.0*	- 7.2
-1	2	7	25.5	-25.3
1	3	0	40.1	40.0
1	3	1	45.9	45.8
-1	3	1	19.8	-18.8
1	3	2	13.0*	- 5.1
-1	3	2	19.8	-18.4
1	3	3	87.8	-87.8
-1	3	3	71.0	75.6
1	3	4	77.5	-81.7
-1	3	4	25.5	-25.3
1	3	5	8.7*	7.4
-1	3	5	32.0	-32.2
1	3	6	47.6	48.0
-1	3	6	25.7	-25.3
1	3	7	3.0*	2.5
-1	3	7	41.5	-39.4
1	4	0	149.4	-159.2
1	4	1	219.4	236.4
-1	4	1	9.3*	- 7.6
1	4	2	25.4	-24.0
-1	4	2	101.0	-106.9
1	4	3	205.8*	-210.1
-1	4	3	36.6	-41.7
1	4	4	17.4*	18.0
-1	4	4	9.9*	9.8
1	4	5	48.7	47.7
-1	4	5	54.0	54.1
1	4	6	5.5*	- 4.8
-1	4	6	24.9	-25.1
1	4	7	10.8*	8.9
-1	4	7	33.0	-32.8
1	5	0	147.2	157.2
1	5	1	10.7*	1.1
-1	5	1	146.5	159.5
1	5	2	97.2	-95.0
-1	5	2	6.4*	-12.4
1	5	3	3.9*	- 1.6
-1	5	3	189.3	-206.2
1	5	4	7.8*	- 8.9
-1	5	4	51.0	50.4
1	5	5	57.1	55.8
-1	5	5	68.8	65.3
1	5	6	7.9*	7.3
-1	5	6	32.0	-30.7
1	5	7	17.1	-16.9
-1	5	7	27.1	25.6
1	6	0	132.5	-140.7
1	6	1	37.1	37.8
-1	6	1	91.3	-98.1
1	6	2	76.7	79.8
-1	6	2	116.5	-121.3
1	6	3	28.4	-28.2
-1	6	3	106.5	110.2
1	6	4	11.8*	11.9
-1	6	4	6.5*	1.9
1	6	5	18.6	18.5
-1	6	5	54.3	-50.5
1	6	6	16.5	17.7
-1	6	6	56.3	53.8
1	7	0	49.2	-45.1
1	7	1	37.7	-36.5
-1	7	1	35.6	32.4
1	7	2	48.3	-47.9
-1	7	2	46.6	-48.2
1	7	3	42.1	42.5

h	k	l	$\lvert F_o \rvert$	F_c
-1	7	3	55.8	-56.0
1	7	4	105.9	105.6
-1	7	4	29.5	29.7
1	7	5	9.9*	-10.3
-1	7	5	29.6	27.8
1	7	6	56.2	-54.0
-1	7	6	19.0	-18.8
-1	7	7	11.5*	- 9.5
1	8	0	80.3	-76.8
1	8	1	31.3	-29.3
-1	8	1	51.9	-48.2
1	8	2	62.8	59.9
-1	8	2	37.4	37.0
1	8	3	22.2	20.2
-1	8	3	46.4	40.7
1	8	4	69.9	-66.3
-1	8	4	68.0	-62.8
1	8	5	32.0	29.8
-1	8	5	14.1*	-11.8
1	8	6	40.7	36.8
-1	8	6	5.4*	- 2.5
1	9	0	50.8	-48.3
1	9	1	44.1	39.2
-1	9	1	33.2	31.7
1	9	2	7.8*	- 4.0
-1	9	2	127.2	123.7
1	9	3	44.5	-42.0
-1	9	3	29.0	25.2
1	9	4	14.4*	11.7
-1	9	4	38.0	-36.4
1	9	5	10.6*	9.9
-1	9	5	30.5	-28.0
1	9	6	10.3*	- 9.2
-1	9	6	12.7	-11.7
1	10	0	19.1*	19.2
1	10	1	51.2	49.2
-1	10	1	58.7	51.7
1	10	2	26.9	25.5
-1	10	2	8.9*	- 6.9
1	10	3	78.7	-72.2
-1	10	3	33.7	-28.8
1	10	4	29.7	-28.9
-1	10	4	65.1	58.8
1	10	5	29.3	27.7
-1	10	5	16.7	15.1
-1	10	6	62.1	-55.1
1	11	0	54.0	51.6
1	11	1	56.1	58.7
-1	11	1	56.1	54.4
1	11	2	8.8*	- 6.5
-1	11	2	40.1	-41.2
1	11	3	50.0	-50.0
-1	11	3	42.1	-42.5
1	11	4	26.4	-25.9
-1	11	4	20.7	-20.8
1	11	5	63.6	66.1
-1	11	5	18.6	17.8
1	12	0	106.5	-102.7
1	12	1	49.0	49.9
-1	12	1	49.0	-48.2
1	12	2	7.0*	8.0
-1	12	2	75.7	-76.0
1	12	3	37.0	-36.9
-1	12	3	4.4*	- 7.3
1	12	4	16.9	18.4
-1	12	4	6.5*	- 6.5
-1	12	5	8.6*	- 7.4
1	13	0	15.8*	12.8
1	13	1	49.8	-51.1
-1	13	1	25.8	24.4
1	13	2	20.2	-19.7
-1	13	2	20.0	-18.4
1	13	3	7.7*	7.0
-1	13	3	64.0	-65.7
1	13	4	20.9	20.7
-1	13	4	9.4*	8.9
1	14	0	58.7	-57.0
1	14	1	8.0*	- 8.7
-1	14	1	28.3	-28.5
1	14	2	40.7	43.2
-1	14	2	17.8	-18.1
1	14	3	32.6	32.6
-1	14	3	17.6	17.6
-1	14	4	25.8	-27.4
1	15	0	65.9	-63.3
1	15	1	11.5*	9.7
-1	15	1	29.4	29.2
1	15	2	43.0	44.9
-1	15	2	38.1	39.4
-1	15	3	21.0	-22.0
1	16	0	22.1	-20.9
1	16	1	27.4	-28.4
-1	16	1	16.2	16.0
-1	16	2	5.2*	- 1.7
2	0	0	23.0	-11.0
2	0	2	67.4	-69.5
-2	0	2	19.7	17.6
2	0	4	10.4*	- 7.6
-2	0	4	137.2	142.7
2	0	6	14.0	14.2
-2	0	6	72.1	-71.9

h	k	l	$\lvert F_o \rvert$	F_c
-2	0	8	18.8	18.6
2	1	0	42.5	-45.9
2	1	1	48.6	50.9
-2	1	1	105.6	-108.8
2	1	2	22.3	-19.7
-2	1	2	129.4	-134.7
2	1	3	67.8	-70.4
-2	1	3	151.6	158.1
2	1	4	62.1	64.6
-2	1	4	40.6	-39.8
2	1	5	28.5	29.6
-2	1	5	78.1	-73.7
2	1	6	7.9*	5.5
-2	1	6	94.8	86.8
2	1	7	4.8*	3.1
-2	1	7	25.3	23.7
-2	1	8	25.5	-25.0
2	2	0	65.1	-70.5
2	2	1	216.2	-240.1
-2	2	1	80.4	-82.9
2	2	2	77.6	80.0
-2	2	2	45.1	-44.5
2	2	3	169.0	184.4
-2	2	3	100.2	-106.0
2	2	4	39.8	-43.6
-2	2	4	26.9	27.0
2	2	5	17.9	-19.1
-2	2	5	76.3	74.7
2	2	6	49.1	52.1
-2	2	6	42.8	-41.8
2	2	7	22.9	-23.6
-2	2	7	13.5*	-13.1
2	3	0	159.1	-173.7
2	3	1	28.4	-23.9
-2	3	1	41.3	-40.9
2	3	2	33.0	32.9
-2	3	2	45.7	-47.5
2	3	3	14.8*	-14.1
-2	3	3	40.7	-42.3
2	3	4	25.4	25.9
-2	3	4	8.0*	6.5
2	3	5	94.5	98.1
-2	3	5	52.9	51.3
2	3	6	21.4	-21.7
-2	3	6	7.1*	- 1.9
2	3	7	55.2	-57.4
-2	3	7	13.1*	-14.2
2	4	0	118.8	-118.6
2	4	1	50.5	53.2
-2	4	1	86.5	89.0
2	4	2	51.7	50.9
-2	4	2	9.9*	7.5
2	4	3	59.2	-58.9
-2	4	3	7.3*	3.2
2	4	4	7.8*	- 3.8
-2	4	4	80.8	76.8
2	4	5	9.5*	9.0
-2	4	5	8.9*	2.2
2	4	6	13.3*	13.7
-2	4	6	7.9*	0.1
2	4	7	5.7*	5.1
-2	4	7	22.8	-21.0
2	5	0	38.5	36.8
2	5	1	21.9	24.7
-2	5	1	6.0*	- 4.1
2	5	2	71.2	73.5
-2	5	2	9.0*	4.3
2	5	3	62.9	-63.7
-2	5	3	43.6	44.8
2	5	4	64.6	-64.2
-2	5	4	93.5	90.7
2	5	5	37.4	36.2
-2	5	5	3.8*	- 4.0
2	5	6	22.4	20.6
-2	5	6	94.9	-89.7
-2	5	7	43.0	41.5
2	6	0	76.9	80.9
2	6	1	179.3	190.4
-2	6	1	49.9	51.3
2	6	2	71.9	-73.8
-2	6	2	36.0	-36.3
2	6	3	24.4	-24.1
-2	6	3	83.1	83.6
2	6	4	12.7*	10.3
-2	6	4	24.5	21.6
2	6	5	29.3	-28.5
-2	6	5	65.2	-62.5
2	6	6	16.7	17.9
-2	6	6	22.4	-21.2
-2	6	7	9.1*	- 9.5
2	7	0	10.2*	13.5
2	7	1	9.9*	3.3
-2	7	1	61.5	-63.1
2	7	2	75.5	76.1
-2	7	2	76.7	79.1
2	7	3	12.4*	-11.8
-2	7	3	83.1	84.7
2	7	4	59.8	-59.3
-2	7	4	22.0	-21.5
2	7	5	30.4	31.0
-2	7	5	86.7	-81.7

h	k	l	$\lvert F_o \rvert$	F_c
2	7	6	35.6	34.3
-2	7	6	28.1	-25.9
-2	7	7	6.2*	3.6
2	8	0	55.0	-53.3
2	8	1	141.8	-141.2
-2	8	1	22.8	-17.4
2	8	2	51.9	49.1
-2	8	2	12.5*	10.9
2	8	3	74.5	70.8
-2	8	3	27.9	-25.3
2	8	4	37.6	-37.2
-2	8	4	39.9	35.6
2	8	5	15.2	-15.1
-2	8	5	35.8	32.2
2	8	6	28.6	26.7
-2	8	6	68.0	-59.3
2	9	0	153.4	-147.6
2	9	1	18.4*	15.0
-2	9	1	21.6	19.4
2	9	2	27.6	25.1
-2	9	2	88.0	-86.3
2	9	3	28.1	-26.9
-2	9	3	18.0*	-14.9
2	9	4	26.6	27.5
-2	9	4	9.9*	10.2
2	9	5	6.9*	0.3
-2	9	5	34.3	31.5
-2	9	6	10.2*	7.1
2	10	0	59.0	-57.6
2	10	1	26.3	-22.1
-2	10	1	49.1	46.0
2	10	2	69.1	67.4
-2	10	2	14.6*	3.2
2	10	3	77.8	71.5
-2	10	3	73.3	-69.8
2	10	4	38.6	-37.7
-2	10	4	52.4	46.4
2	10	5	7.1*	- 3.7
-2	10	5	36.2	32.8
-2	10	6	5.9*	- 4.9
2	11	0	71.1	-66.7
2	11	1	17.5*	-17.0
-2	11	1	14.5*	-17.3
2	11	2	69.0	68.7
-2	11	2	81.2	-83.4
2	11	3	27.4	27.2
-2	11	3	10.6*	- 7.4
2	11	4	6.1*	2.4
-2	11	4	65.4	65.8
2	11	5	18.2	18.5
-2	11	5	14.3	14.9
2	12	0	42.0	41.7
2	12	1	35.7	37.6
-2	12	1	20.2	19.7
2	12	2	28.7	-28.9
-2	12	2	38.3	-39.9
2	12	3	8.6*	-10.1
-2	12	3	20.8	20.3
2	12	4	31.5	32.7
-2	12	4	18.1	-17.9
-2	12	5	6.4*	6.7
2	13	0	6.7*	6.4
2	13	1	8.2*	- 6.4
-2	13	1	43.4	42.5
2	13	2	42.4	43.2
-2	13	2	64.7	66.2
2	13	3	13.5	-12.9
-2	13	3	23.8	-25.4
2	13	4	53.3	-56.2
-2	13	4	8.4*	8.4
2	14	0	14.1*	11.7
2	14	1	8.6*	9.9
-2	14	1	14.6*	13.8
2	14	2	16.7	-16.1
-2	14	2	24.2	24.7
2	14	3	19.6	20.1
-2	14	3	30.1	31.7
-2	14	4	3.7*	- 1.7
2	15	0	11.4*	- 9.4
2	15	1	24.9	-24.1
-2	15	1	42.9	42.6
2	15	2	22.5	23.1
-2	15	2	9.9*	- 9.3
-2	15	3	14.0	-15.0
2	16	0	16.9	-16.8
2	16	1	16.9	-17.9
-2	16	1	5.3*	4.6
-2	16	2	3.6*	- 0.1
3	0	1	158.9	177.9
-3	0	1	224.7*	-264.6
3	0	3	65.4	-70.1
-3	0	3	93.8	99.3
3	0	5	17.4	-18.0
-3	0	5	4.8*	- 0.1
-3	0	7	66.8	67.0
3	1	0	41.6	41.8
3	1	1	87.0	86.5
-3	1	1	85.0	-88.7
3	1	2	35.7	37.4
-3	1	2	140.8	-142.2
3	1	3	10.6*	8.5

Table 1. *(Continued)*

h	k	l	$\|F_o\|$	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 Å²	1	3.02 Å²	.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

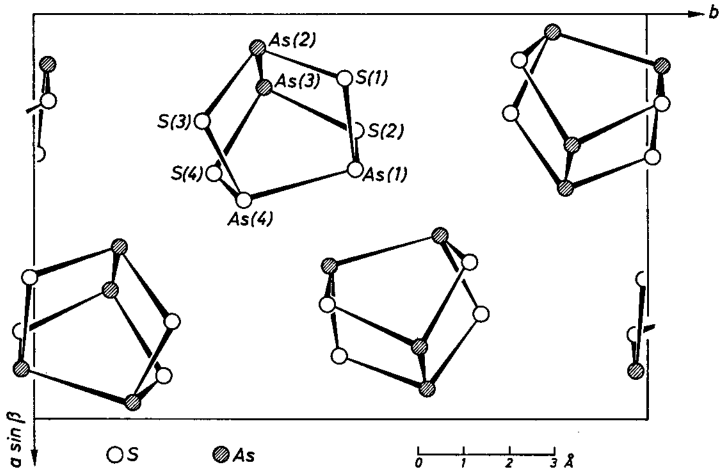

Fig. 1. Realgar. [001] projection

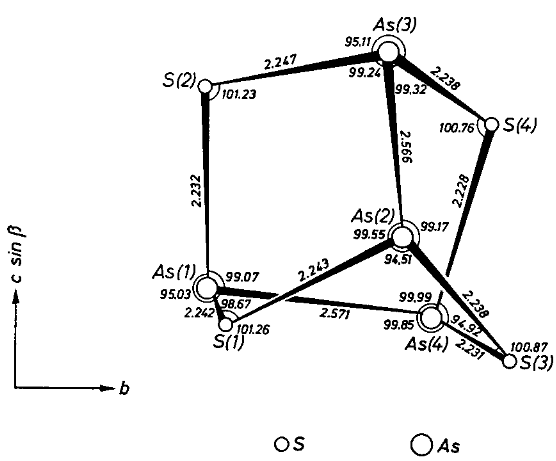

Fig. 2. As_4S_4 molecule showing cradle shape. [100] projection. (Bond distances in Å, angles in degrees)

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°.