# Tris(*N*,*N*-diethylcarbamato-$\kappa^2 S$,*S'*)arsenic(III)

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## Key indicators

Single-crystal X-ray study

$T$ = 298 K

Mean $\sigma$(C–C) = 0.006 Å

$R$ factor = 0.033

$wR$ factor = 0.088

Data-to-parameter ratio = 18.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $[As\{S_2CN(C_2H_5)_2\}_3]$, the coordination environment of the As atom can be described as a distorted trigonal antiprism.

## Comment

The chemistry of arsenic(III)–1,1-dithiolates has been the subject of considerable research in recent decades (Raston *et al.*, 1975). It has been stimulated by either the potential applications (*e.g.* fungicides, herbicides, desiccants and wood preservatives) or their interesting structural behaviour that results from the various coordination patterns of the 1,1-dithiolate ligands (Cea-Olivares *et al.*, 1995). The inhibition action of arsenic(III) on the activity of enzymes is a strong field of biochemical investigation. Arsenic is cytotoxic and the mechanism of toxicity is believed to be due to the ability of arsenite (As$^{III}$) to bind protein thiols. It can also induce chromosomal damage and inhibit DNA repair (Garje *et al.*, 1997). As a continuation of our interest in sulfur-containing ligands, we report here the synthesis and structure of the title compound, (I).



(I)
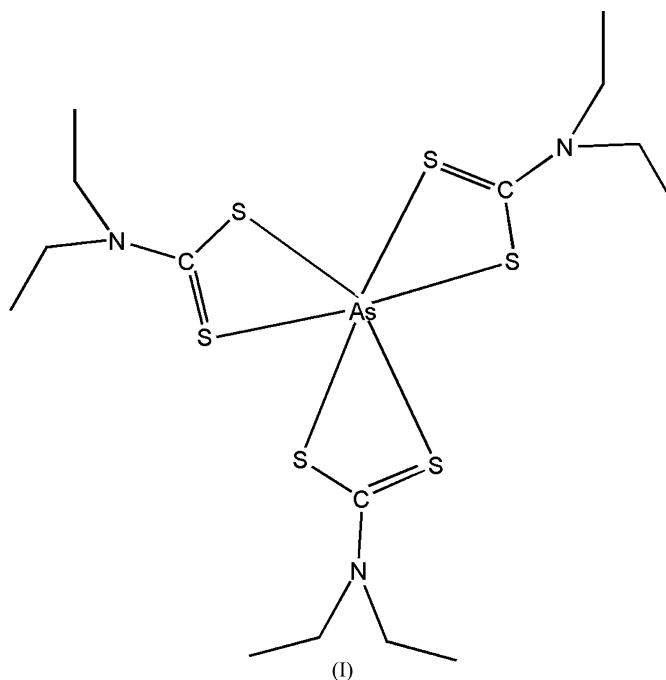
The molecular structure of (I) is shown in Fig. 1. The bidentate ligands are chelated to the As atom, forming a polyhedron which can be described as a distorted trigonal antiprism. There are three short As—S bonds and three long
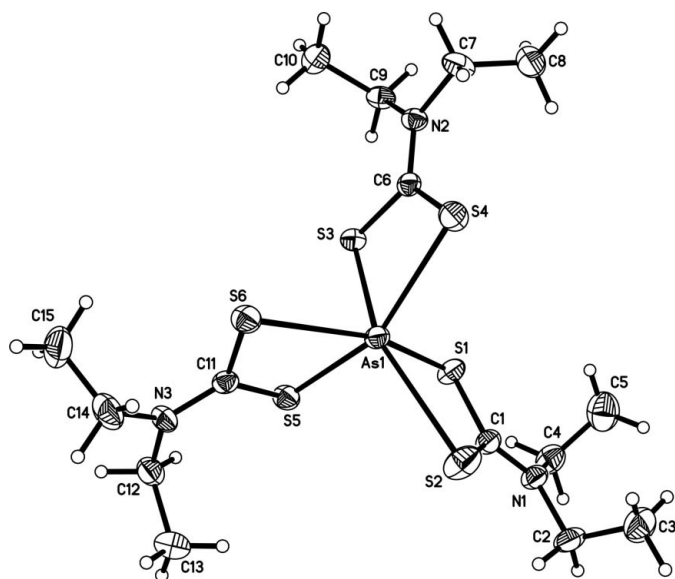
**Figure 1**
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering schemes.
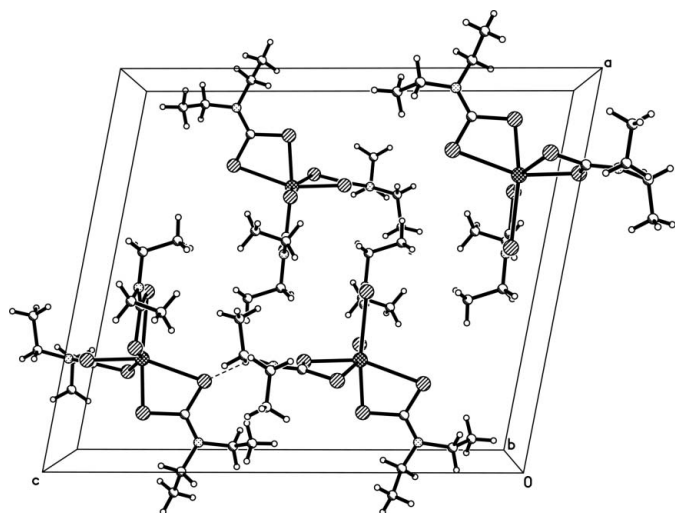


**Figure 2**
The packing of the title complex (I).

As—S bonds. All the three short bonds (mean As—S = 2.35 Å) indicate a strong 'S' coordination, while the other set corresponds to a weak As—S interaction (mean As—S = 2.84 Å). It is noticeable that, in all ligands, the C—S bond associated with the strong As—S bond is significantly longer (mean 1.762 Å) than that (mean = 1.683 Å) associated with the weak As—S bond, showing clearly the localization of the double bonds.

A diagram of the coordination polyhedron showing the two trigonal faces of the antiprism is depicted in Fig. 3. The plane defined by S1, S3 and S5, which are the strongly coordinated S atoms, is essentially parallel to that defined by S2, S4 and S6 (the two planes are at an angle of 4.5 (2)° and As is displaced by 1.36 (2) and 1.09 (3) Å, respectively, from the planes.

## Experimental

To a stirred solution of $AsI_3$ (0.2 mmol) in acetonitrile (20 ml), $(C_2H_5)_2NCS_2Na$ (0.6 mmol) was added. The reaction mixture was stirred for 4 h at 298 K. An orange–red solution was obtained, which was then filtered. The solvent was gradually removed by evaporation under vacuum and a solid product was obtained. The solid was recrystallized from ethanol and orange–red crystals of (I) were formed (yield 79%; m.p. 402 K). Analysis calculated for $C_{15}H_{30}AsN_3S_6$: C 34.66, H 5.82, N 8.09%; found: C 34.28, H 5.53, N 8.22%.

### Crystal data

| | |
|---|---|
| $[As(C_5H_{10}NS_2)_3]$ | $Z = 4$ |
| $M_r = 519.70$ | $D_x = 1.424$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 15.868$ (5) Å | $\mu = 1.92$ mm$^{-1}$ |
| $b = 8.389$ (3) Å | $T = 298$ (2) K |
| $c = 18.548$ (6) Å | Block, orange-red |
| $\beta = 100.990$ (4)° | $0.33 \times 0.31 \times 0.30$ mm |
| $V = 2423.9$ (14) Å$^3$ | |

### Data collection

| | |
|---|---|
| Siemens SMART CCD diffractometer | 12134 measured reflections |
| $\varphi$ and $\omega$ scans | 4257 independent reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | 3141 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.569$, $T_{max} = 0.596$ | $R_{int} = 0.038$ |
| (expected range = 0.536–0.561) | $\theta_{max} = 25.0°$ |

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 1.0408P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.033$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.088$ | $(\Delta/\sigma)_{max} = 0.002$ |
| $S = 1.01$ | $\Delta\rho_{max} = 0.67$ e Å$^{-3}$ |
| 4257 reflections | $\Delta\rho_{min} = -0.46$ e Å$^{-3}$ |
| 232 parameters | |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| As1—S3 | 2.3417 (12) | S1—C1 | 1.759 (3) |
| As1—S1 | 2.3513 (12) | S2—C1 | 1.680 (4) |
| As1—S5 | 2.3573 (12) | S3—C6 | 1.765 (3) |
| As1—S6 | 2.8047 (13) | S4—C6 | 1.681 (4) |
| As1—S4 | 2.8221 (13) | S5—C11 | 1.761 (3) |
| As1—S2 | 2.9009 (15) | S6—C11 | 1.687 (3) |
| | | | |
| S3—As1—S1 | 88.29 (4) | S5—As1—S4 | 157.28 (4) |
| S3—As1—S5 | 91.29 (3) | S6—As1—S4 | 98.13 (3) |
| S1—As1—S5 | 90.64 (3) | S3—As1—S2 | 154.60 (4) |
| S3—As1—S6 | 90.78 (3) | S1—As1—S2 | 67.81 (3) |
| S1—As1—S6 | 159.97 (4) | S5—As1—S2 | 97.20 (4) |
| S5—As1—S6 | 69.37 (3) | S6—As1—S2 | 114.62 (3) |
| S3—As1—S4 | 69.37 (3) | S4—As1—S2 | 105.36 (4) |
| S1—As1—S4 | 100.24 (3) | | |

All H atoms were positioned geometrically and treated as riding on their parent atoms [C—H = 0.97 Å and $U_{iso} = 1.2U_{eq}(C)$ for CH$_2$ H atoms, and C—H = 0.96 Å and $U_{iso} = 1.5U_{eq}(C)$ for CH$_3$ H atoms].

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics:

*SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

## References

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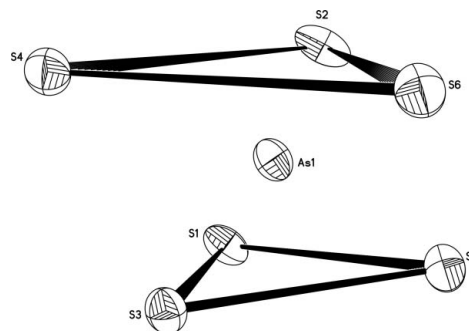
**Figure 3**
A schematic diagram of the coordination polyhedron in (I) showing the two trigonal faces of the antiprism.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.