

## Tris(ethylenediamine)cobalt(III) tetrathioarsenate(V)

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### Key indicators

Single-crystal X-ray study  
 T = 293 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
 R factor = 0.027  
 wR factor = 0.047  
 Data-to-parameter ratio = 24.4

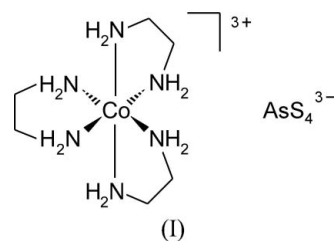
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $[\text{Co}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{AsS}_4$ , was synthesized under solvothermal conditions in ethylenediamine (en) at 413 K. The compound consists of octahedral  $[\text{Co}(\text{en})_3]^{3+}$  cations and tetrahedral  $\text{AsS}_4^{3-}$  anions.

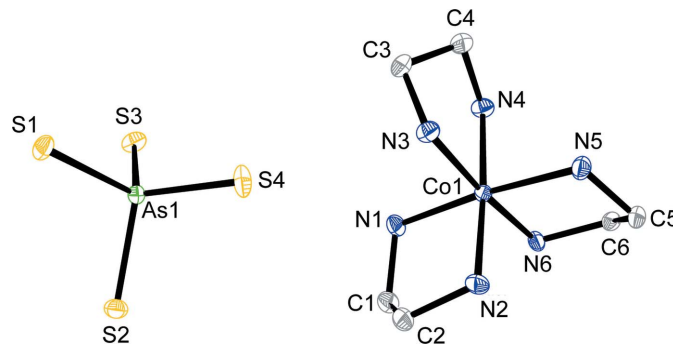
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### Comment

Polychalcogenidometalates possess diverse and interesting structural chemistry, and exhibit useful physical and chemical properties which are promising for application in modern technologies (Manos *et al.*, 2005). Thioarsenates have been prepared using molten alkali-metal polychalcogenide flux techniques and high-temperature solid-state techniques (Iyer & Kanatzidis, 2002, 2004), and some have been synthesized in lower-temperature solvothermal/hydrothermal reactions (Chou & Kanatzidis, 1994*a,b*, 1995; Jia *et al.*, 2006; Fu *et al.*, 2005; Kanatzidis & Chou, 1996). The title compound, (I), was synthesized under solvothermal conditions using ethylenediamine as the solvent.



Compound (I) (Fig. 1) consists of octahedral  $[\text{Co}(\text{en})_3]^{3+}$  cations and tetrahedral  $\text{AsS}_4^{3-}$  anions. The coordination geometry of Co is slightly distorted from octahedral, reflected in the *trans* N–Co–N angles of 173.79 (11)–175.77 (11)°



**Figure 1**  
 The asymmetric unit of (I), showing displacement ellipsoids at the 50% probability level. H atoms have been omitted.

(Table 1). In the  $\text{AsS}_4^{3-}$  anions, the As—S distances and S—As—S angles (Table 1) demonstrate a significant distortion from an ideal tetrahedral geometry.

## Experimental

The title compound was obtained by a typical solvothermal synthetic procedure.  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (0.027 g), arsenic (0.014 g) and sulfur powder (0.022 g) were placed in a glass tube with 2 ml of ethylenediamine and stirred for 5 min. The mixture was sealed in a Teflon-lined stainless steel bomb and heated at 413 K for 5 d. After cooling slowly to ambient temperature, the products were washed with ethanol then water, and orange block crystals of (I) were obtained.

### Crystal data

$[\text{Co}(\text{C}_2\text{H}_8\text{N}_2)_3](\text{AsS}_4)$   
 $M_r = 442.40$   
 Tetragonal,  $P4_2bc$   
 $a = 15.2781$  (19) Å  
 $c = 13.559$  (4) Å  
 $V = 3165.1$  (10) Å<sup>3</sup>  
 $Z = 8$

$D_x = 1.857$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 3.68$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, orange  
 $0.30 \times 0.18 \times 0.15$  mm

### Data collection

Bruker SMART APEXII CCD  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2001)  
 $T_{\min} = 0.384$ ,  $T_{\max} = 0.576$

18629 measured reflections  
 3981 independent reflections  
 3343 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$   
 $\theta_{\text{max}} = 29.1^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.047$   
 $S = 0.97$   
 3981 reflections  
 163 parameters  
 H-atom parameters constrained

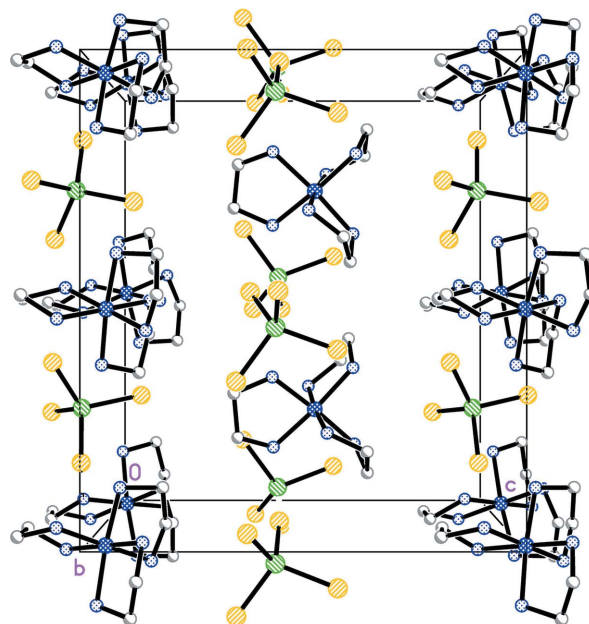
$w = 1/[\sigma^2(F_o^2) + (0.0093P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.007$   
 $\Delta\rho_{\text{max}} = 0.32$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 1845 Friedel pairs  
 Flack parameter: 0.002 (7)

**Table 1**

Selected geometric parameters (Å, °).

As1—S1	2.1687 (8)	Co1—N2	1.968 (2)
As1—S2	2.1761 (10)	Co1—N3	1.960 (3)
As1—S3	2.1692 (9)	Co1—N4	1.970 (3)
As1—S4	2.1520 (8)	Co1—N5	1.969 (2)
Co1—N1	1.963 (2)	Co1—N6	1.960 (3)
S1—As1—S2	106.70 (4)	S3—As1—S4	110.93 (4)
S1—As1—S3	106.00 (4)	N3—Co1—N6	174.89 (11)
S1—As1—S4	111.97 (3)	N2—Co1—N4	175.77 (11)
S2—As1—S3	113.85 (4)	N1—Co1—N5	173.79 (11)
S2—As1—S4	107.38 (4)		

H atoms were positioned geometrically with C—H = 0.97 Å and N—H = 0.90 Å and allowed to ride during subsequent refinement with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$ .



**Figure 2**

Perspective view of the unit-cell contents of (I). H atoms have been omitted.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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