Crystal structure of caesium tetramethyldithioimidodiphosphinate

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In the title crystal, the salt [CsMe2P(S)NP(S)Me2] is self-assembled as an undulating supramolecular two-dimensional polymeric structure, poly[(μ₄-tetramethyldithioimidodiphosphinato)caesium], [Cs(C₄H₁₂NP₂S₂)]ₙ, which is parallel to the bc plane. The Cs cations are hexacoordinated, being chelated by two thioimidophosphinate groups and two sulfur atoms from neighboring ligands. The anions are linked to the Cs cations by Cs⋅⋅⋅S and Cs⋅⋅⋅N electrostatic interactions.

1. Chemical context

Dichalcogenoimidodiphosphinate anions [R₂P(E)NP(E)R₂]⁻ (E = O, S, Se, Te) are versatile complexing reagents with a strong tendency to form inorganic (carbon-free) chelate rings (Haiduc & Silaghi-Dumitrescu, 1986; Cea-Olivares & Muñoz, 1993; Hernández-Arganís et al., 2004; Slawin et al., 1994). The monoanionic ligands have been investigated as ligands for both main-group elements (Silvestru & Drake, 2001; Woollins, 1996) and transition metals (Rudler et al., 1997). The widespread interest in dichalcogenoimidodiphosphinates stems from their potential uses as lanthanide shift reagents (Rudler et al., 1997), industrial catalysts (Leung et al., 2000; Yamazaki et al., 2020), luminescent materials (Ma et al., 2019) as well as in metal extraction processes (du Preez et al., 1992). As part of our ongoing research on dichalcogenoimidodiphosphinate anions, we report herein the synthesis and crystallographic study of the title compound (I).

2. Structural commentary

In the asymmetric unit of the title compound (I) (Fig. 1), the tetramethyldithioimidodiphosphinate anion is bent with a P—N—P angle of 132.16 (6)°, and chelates the Cs cation through...
S⋯Cs⋯N electrostatic interactions [S⋯Cs⋯N = 53.074 (17)°; S⋯Cs = 3.4377 (3) Å; N⋯Cs = 3.2054 (9) Å]. The bond distances of 2.0003 (4), 1.6075 (10), 1.6179 (10) and 1.9869 (4) Å for S1—P1, P1—N1, N1—P2 and P2—S2, respectively, suggest that the anion is a delocalized system (Cea-Olivares & Nöth, 1987; Churchill et al., 1971). The phosphorus atoms are in an approximately tetrahedral environment, the average bond angles being S—P—N = 113.9°, S—P—C = 109.4°, and C—P—C = 103.4°.

3. Supramolecular features

In the crystal, the salt [CsMe₂P(S)NP(S)Me₂] (I) is self-assembled as an undulating supramolecular 2D polymeric structure, which is parallel to the bc plane (Figs. 2 and 3). The Cs cations are hexacoordinated and linked to four different anions by Cs⋯S and Cs⋯N electrostatic interactions (Fig. 4). Analysis of this CsS₆N₂ polyhedron with the SHAPE 2.1 program (Llunell et al., 2013) gave CShM values of 9.50434 and 8.43874 for a regular octahedron and a trigonal prism,
Table 1
Experimental details.

| Property                      | Value                      |
|-------------------------------|----------------------------|
| Chemical formula              | [Cs(C₄H₁₂NP₂S₂)]           |
| Mₜ                           | 333.12                     |
| Crystal system, space group   | Monoclinic, P2₁/c          |
| Temperature (K)               | 100                        |
| a, b, c (Å)                   | 11.0320 (2), 12.4326 (3), 8.2173 (2) |
| β (°)                         | 93.8752 (4)                |
| V (Å³)                        | 1124.48 (4)                |
| Z                             | 4                          |
| Radiation type                | Mo Kα                      |
| μ (mm⁻¹)                      | 3.89                       |
| Crystal size (mm)             | 0.18 × 0.15 × 0.12         |
| Data collection               | Bruker SMART APEXII DUO    |
| Absorption correction         | Multi-scan (SADABS, Bruker, 2016) |
| T max                         | 1.00, 0.147                |
| No. of measured, independent  | 15582, 5097, 4791           |
| observed [I > 2σ(I)] reflections |                           |
| R_m                          | 0.017                      |
| (sin θ)₃₃₃_max (Å⁻¹)           | 0.833                      |
| Refinement                    |                            |
| R(F² > 2σ(F²)), wR(F²), S    | 0.017, 0.035, 1.08         |
| No. of reflections            | 5097                       |
| No. of parameters             | 95                         |
| H-atom treatment             | H-atom parameters constrained |                        |
| Δρ_max, Δρ_min (e Å⁻³)        | 0.58, −0.76                |

Computer programs: APEX3 (Bruker, 2016), SAINT (Bruker, 2016), SHELXTL (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), and OLEX2 (Dolomanov et al., 2009).

respectively, meaning that the coordination environment of the cesium atom is highly irregular. These polyhedra interconnect either by sharing vertices or an edge. The Cs⁻⋅S ionic bond distances vary from 3.4377 (3) to 3.4726 (3) Å, which are close to the value of 3.51 Å predicted from the ionic radii (Shannon, 1976). Regarding the N−Cs bond distances, two different distances were determined. One of them is 3.2054 (9) Å, which is close to the value of 3.13 Å predicted from the ionic radii, and the other is 3.651 Å, which is less than the value of 4.4 Å predicted from the van der Waals radii (Batsanov, 2001). Furthermore, five methyl groups are located in a close vicinity of the Cs⁺ cation with the Cs⁻⋅H distance shorter than 4 Å, but only the shortest Cs₁⋅H₂C(1−x, 1−y, 1−z) distance of 3.269 Å is similar to those observed in [LiCs(HMDS)₂]⁺ and can be labeled as an agostic interaction (Ojeda-Amador et al., 2016). The cyclic motifs Cs₂S₂, Cs₂N₂, Cs₂N₂P₂S₂ in this arrangement possess crystallographic inversion symmetry.

4. Database survey

The current version of the Cambridge Structural Database (Version 2021.1, updated August 2021; Groom et al., 2016) contains only three cseum dichalcogenoimidodiphosphinates, (18Ce)Cs₂P₂(EP)NH(C₄H₁₂)(P₂)₂ (BENSAP, BENSET and BENSIX for E = O, S and Se; Hernández-Arganis et al., 2004). Furthermore, only five compounds each containing two [Me₂P(S)NP(S)Me]⁻ ligands and one $M^2⁺$ cation ($M = Fe, Ni, Pd, Cd, Co$) are included in the database: IMSPFE10, IMSPNI10, OCANEL, TASSAXN and ZACZAE (Churchill & Wormald, 1971; Churchill et al., 1971; Blic et al., 2000; Ghesner et al., 2005 and Silvestru et al., 1995). The dinuclear species MIWYUM with two [Mn(CO)₃]$^+$ cations (Zuniga-Villarreal et al., 2001) is also noteworthy. No compound with [Me₂P(E)NP(E)Me]²⁻ ($E = O$ or Se) is included in the database.

5. Synthesis and crystallization

Cs[Me₂P(S)]₂N (I) was obtained by the reaction of [Me₂P(S)NH(S)(Me)] with Cs₂CO₃, according to a method previously described (Schmidpeter & Ebeling, 1968) and isolated solvent-free. The salt Cs[Me₂P(S)]₂N was recrystallized by slow evaporation from methanol. The spectroscopic data of the received sample (vide infra) coincided with the published ones and are therefore not reported; however, they can be consulted in the above-mentioned reference.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were positioned geometrically (C−H = 0.98 Å) and constrained using the riding-model approximation with $U_{iso}(H) = 1.5 U_{eq}(C)$.

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Computing details

Data collection: APEX3 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

Poly[([μ₄-tetramethyldithioimidodiphosphinato)caesium]

Crystal data

\[\text{[Cs(C}_4\text{H}_12\text{NP}_2\text{S}_2)]}\]

\[M_r = 333.12\]

Monoclinic, \( P2_1/c \)

\[a = 11.0320 (2) \text{ Å}\]

\[b = 12.4326 (3) \text{ Å}\]

\[c = 8.2173 (2) \text{ Å}\]

\[β = 93.8752 (4)°\]

\[V = 1124.48 (4) \text{ Å}^3\]

\[Z = 4\]

\(D_x = 1.968 \text{ Mg m}^{-3}\)

\(\lambda = 0.71073 \text{ Å}\)

\(\theta = 2.4–25.2°\)

\(\mu = 3.89 \text{ mm}^{-1}\)

\(T = 100 \text{ K}\)

Plate, clear light white

\[0.18 \times 0.15 \times 0.12 \text{ mm}\]

Data collection

Bruker SMART APEXII DUO diffractometer

Radiation source: Incoatec ImuS with multilayer mirrors

Detector resolution: 8.333 pixels mm\(^{-1}\)

\(\omega\) scans

Absorption correction: multi-scan (SADABS; Bruker, 2016)

\(R_{\text{int}} = 0.017\)

\(\theta_{\text{max}} = 36.3°, \theta_{\text{min}} = 2.5°\)

\(h = -17\rightarrow 17\)

\(k = -19\rightarrow 20\)

\(l = -13\rightarrow 13\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\[R(F^2 > 2\sigma(F^2)) = 0.017\]

\[wR(F^2) = 0.035\]

\[S = 1.08\]

5097 reflections

95 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

\(w = 1/[(\sigma(F_c^2) + (0.0129P)^2 + 0.2359P)/P]^3\)

where \(P = (F_c^2 + 2F_e^2)/3\)

\((\Delta/σ)_{\text{max}} = 0.004\)

\(Δρ_{\text{max}} = 0.58 \text{ e Å}^{-3}\)

\(Δρ_{\text{min}} = -0.76 \text{ e Å}^{-3}\)
**Special details**

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

|    | x     | y     | z     | U(eq) |
|----|-------|-------|-------|-------|
| Cs1| 0.56629 (2) | 0.59017 (2) | 0.27106 (2) | 0.01538 (2) |
| S1 | 0.40180 (2)  | 0.36434 (2)  | 0.15753 (4)  | 0.01505 (5)  |
| S2 | 0.16545 (3)  | 0.55491 (2)  | 0.67865 (3)  | 0.01535 (5)  |
| P1 | 0.25673 (2)  | 0.42906 (2)  | 0.25349 (3)  | 0.01072 (5)  |
| P2 | 0.21826 (2)  | 0.61338 (2)  | 0.46982 (3)  | 0.01115 (5)  |
| N1 | 0.14280 (11) | 0.14599 (10) | 0.09220 (14) | 0.0180 (2)   |
| H1A| 0.1713  | 0.5193  | 0.0261  | 0.027*     |
| H1B| 0.0672  | 0.4811  | 0.1397  | 0.027*     |
| H1C| 0.1280  | 0.3964  | 0.0232  | 0.027*     |
| C2 | 0.18262 (11) | 0.32802 (10) | 0.37023 (15) | 0.0181 (2) |
| H2A| 0.1574  | 0.2678  | 0.2988  | 0.027*     |
| H2B| 0.1111  | 0.3595  | 0.4165  | 0.027*     |
| H2C| 0.2392  | 0.3021  | 0.4586  | 0.027*     |
| C3 | 0.08798 (10) | 0.57113 (10) | 0.35577 (14) | 0.0165 (2) |
| H3A| 0.1128  | 0.6988  | 0.2514  | 0.025*     |
| H3B| 0.0551  | 0.7301  | 0.4185  | 0.025*     |
| H3C| 0.0255  | 0.6158  | 0.3356  | 0.025*     |
| C4 | 0.31966 (11) | 0.72634 (9)  | 0.50889 (15) | 0.0169 (2) |
| H4A| 0.3379  | 0.7596  | 0.4052  | 0.025*     |
| H4B| 0.3952  | 0.7012  | 0.5661  | 0.025*     |
| H4C| 0.2807  | 0.7794  | 0.5766  | 0.025*     |

**Atomic displacement parameters (Å²)**

|    | U₁₁   | U₂₂   | U₃₃   | U₁₂   | U₁₃   | U₂₃   |
|----|-------|-------|-------|-------|-------|-------|
| Cs1| 0.01350 (3) | 0.01249 (3) | 0.02027 (4) | −0.00100 (2) | 0.00205 (2) | −0.00067 (2) |
| S1 | 0.01356 (11) | 0.01225 (11) | 0.01975 (12) | 0.00045 (9) | 0.00416 (9) | −0.00285 (9) |
| S2 | 0.01700 (12) | 0.01704 (12) | 0.01254 (11) | 0.00079 (10) | 0.00488 (9) | 0.00192 (9) |
| P1 | 0.01031 (10) | 0.01168 (11) | 0.01021 (11) | −0.00022 (9) | 0.00089 (8) | 0.00053 (8) |
| P2 | 0.01079 (11) | 0.01129 (11) | 0.01158 (11) | 0.00056 (9) | 0.00243 (9) | 0.00048 (9) |
| N1 | 0.0121 (4)  | 0.0131 (4)  | 0.0136 (4)  | −0.0001 (3)  | 0.0023 (3)  | −0.0024 (3)  |
| C1 | 0.0179 (5)  | 0.0217 (5)  | 0.0138 (5)  | 0.0031 (4)   | −0.0035 (4) | −0.0002 (4)  |
| C2 | 0.0184 (5)  | 0.0178 (5)  | 0.0182 (5)  | −0.0051 (4)  | 0.0030 (4)  | 0.0026 (4)   |
| C3 | 0.0139 (4)  | 0.0177 (5)  | 0.0180 (5)  | 0.0024 (4)   | 0.0024 (4)  | 0.0046 (4)   |
| C4 | 0.0167 (5)  | 0.0149 (5)  | 0.0193 (5)  | −0.0021 (4)  | 0.0040 (4)  | −0.0031 (4)  |
### Geometric parameters (Å, °)

| Bond          | Length (Å)   | Angle (°)  |
|---------------|--------------|------------|
| Cs1—S1        | 3.4377 (3)   |            |
| Cs1—S1'       | 3.4726 (3)   |            |
| Cs1—S1''      | 3.6077 (3)   |            |
| Cs1—S2'''     | 3.4667 (3)   |            |
| Cs1—N1'''     | 3.6506 (9)   |            |
| Cs1—N1''''    | 3.2054 (9)   |            |
| Cs1—H2A'      | 3.8388       |            |
| Cs1—H2C''''   | 3.2692       |            |
| Cs1—H4A       | 3.5176       |            |
| Cs1—H4B''''   | 3.5628       |            |
| Cs1—H4C''''   | 3.4588       |            |
| Cs1—H4D''''   | 3.7984       |            |
| S1—Cs1''      | 3.4726 (3)   |            |
| S1—Cs1''''    | 3.6077 (3)   |            |
| S1—P1         | 2.0003 (4)   |            |
| S2—Cs1'''     | 3.4667 (3)   |            |
| S2—P2         | 1.9869 (4)   |            |
| P1—N1         | 1.6075 (10)  |            |
| P1—C1         | 1.8052 (11)  |            |

| Bond          | Length (Å)   | Angle (°)  |
|---------------|--------------|------------|
| S1—Cs1—S1'   | 153.276 (4)  |            |
| S1—Cs1—S1''  | 86.999 (7)   |            |
| S1—Cs1—S1''  | 89.749 (6)   |            |
| S1—Cs1—S2''' | 92.185 (7)   |            |
| S1—Cs1—N1''' | 144.629 (15) |            |
| S1—Cs1—N1''''| 104.024 (15) |            |
| S1—Cs1—H2A'  | 52.3         |            |
| S1—Cs1—H2A'  | 147.1        |            |
| S1—Cs1—H2A'  | 68.5         |            |
| S1—Cs1—H4A   | 101.4        |            |
| S1—Cs1—H4A   | 55.0         |            |
| S1—Cs1—H4B   | 102.3        |            |
| S1—Cs1—H4B   | 102.3        |            |
| S1—Cs1—H4B   | 53.8         |            |
| S1—Cs1—H4C   | 69.8         |            |
| S1—Cs1—H4C   | 74.0         |            |
| S1—Cs1—H4C   | 80.0         |            |
| S2—Cs1—S1'   | 114.494 (7)  |            |
| S2—Cs1—S1''  | 93.387 (7)   |            |
| S2—Cs1—N1''' | 51.243 (15)  |            |
| S2—Cs1—H2A'  | 68.7         |            |
| S2—Cs1—H4A   | 153.9        |            |
| S2—Cs1—H4B   | 148.8        |            |
| S2—Cs1—H4C   | 161.7        |            |
| N1—Cs1—S1    | 53.074 (17)  |            |

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N1—Cs1—S1\textsuperscript{i} 105.154 (17)  C3—P2—C4 104.09 (6)  
N1—Cs1—S1\textsuperscript{ii} 114.158 (17)  C4—P2—Cs1\textsuperscript{iii} 93.37 (4)  
N1—Cs1—S2\textsuperscript{iii} 131.503 (18)  C4—P2—S2 110.14 (4)  
N1—Cs1—N1\textsuperscript{iii} 93.76 (2)  Cs1—N1—Cs1\textsuperscript{iii} 86.24 (2)  
N1\textsuperscript{ii}—Cs1—H2A\textsuperscript{i} 94.3  P1—N1—Cs1\textsuperscript{ii} 100.76 (4)  
N1—Cs1—H2\textsuperscript{A} 157.3  P1—N1—Cs1 105.60 (4)  
N1—Cs1—H2C\textsuperscript{iii} 121.1  P1—N1—P2 132.16 (6)  
N1—Cs1—H4A 50.2  P2—N1—Cs1 121.68 (4)  
N1—Cs1—H4B\textsuperscript{iv} 78.0  P2—N1—Cs1\textsuperscript{ii} 89.80 (4)  
N1—Cs1—H4B 50.7  P1—C1—H1A 109.5  
N1\textsuperscript{ii}—Cs1—H4C\textsuperscript{iv} 145.1  P1—C1—H1B 109.5  
N1—Cs1—H4C\textsuperscript{v} 55.1  P1—C1—H1C 109.5  
H2C\textsuperscript{iii}—Cs1—S1\textsuperscript{i} 55.1  H1A—C1—H1B 109.5  
H2C\textsuperscript{iii}—Cs1—S1\textsuperscript{ii} 119.8  H1A—C1—H1C 109.5  
H2C\textsuperscript{iii}—Cs1—S1 146.0  H1B—C1—H1C 109.5  
H2C\textsuperscript{iii}—Cs1—S2\textsuperscript{iii} 67.4  P1—C2—H2A 109.5  
H2C\textsuperscript{iii}—Cs1—S2 52.3  P1—C2—H2B 109.5  
H2C\textsuperscript{iii}—Cs1—H2A\textsuperscript{i} 51.3  P1—C2—H2C 109.5  
H2C\textsuperscript{iii}—Cs1—H2A 89.6  H2A—C2—H2B 109.5  
H2C\textsuperscript{iii}—Cs1—H2B\textsuperscript{i} 74.0  H2A—C2—H2C 109.5  
H2C\textsuperscript{iii}—Cs1—H2B 109.0  H2B—C2—H2C 109.5  
H2C\textsuperscript{iii}—Cs1—H2C\textsuperscript{iv} 126.8  P2—C3—H3A 109.5  
H4A—Cs1—S1\textsuperscript{i} 109.3  P2—C3—H3B 109.5  
H4A—Cs1—S1\textsuperscript{ii} 105.2  P2—C3—H3C 109.5  
H4A—Cs1—H2\textsuperscript{A} 107.2  H3A—C3—H3B 109.5  
H4A—Cs1—H4B\textsuperscript{v} 49.1  H3A—C3—H3C 109.5  
H4A—Cs1—H4C\textsuperscript{v} 44.4  H3B—C3—H3C 109.5  
H4B—Cs1—S1\textsuperscript{iv} 135.7  Cs1—C4—H4A 63.3  
H4B—Cs1—S1\textsuperscript{i} 62.8  Cs1—C4—H4B 59.7  
H4B\textsuperscript{v}—Cs1—S1\textsuperscript{ii} 60.5  Cs1—C4—H4C 159.4  
H4B—Cs1—S2\textsuperscript{ii} 128.8  P2—C4—Cs1 91.05 (4)  
H4B—Cs1—N1\textsuperscript{ii} 78.7  P2—C4—H4A 109.5  
H4B\textsuperscript{v}—Cs1—N1\textsuperscript{iii} 151.7  P2—C4—H4B 109.5  
H4B—Cs1—H2\textsuperscript{A} 110.6  P2—C4—H4C 109.5  
H4B\textsuperscript{v}—Cs1—H2A\textsuperscript{i} 84.9  H4A—C4—H4B 109.5  
H4B—Cs1—H4\textsuperscript{A} 26.5  H4A—C4—H4C 109.5  
H4B—Cs1—H4\textsuperscript{B} 75.2  H4B—C4—H4C 109.5  
H4B\textsuperscript{v}—Cs1—H4C\textsuperscript{v} 24.8  

Symmetry codes: (i) −x+1, y+1/2, −z+1/2; (ii) −x+1, −y+1, −z; (iii) −x+1, −y+1, −z+1; (iv) x, −y+3/2, z−1/2; (v) −x+1, y−1/2, −z+1/2.