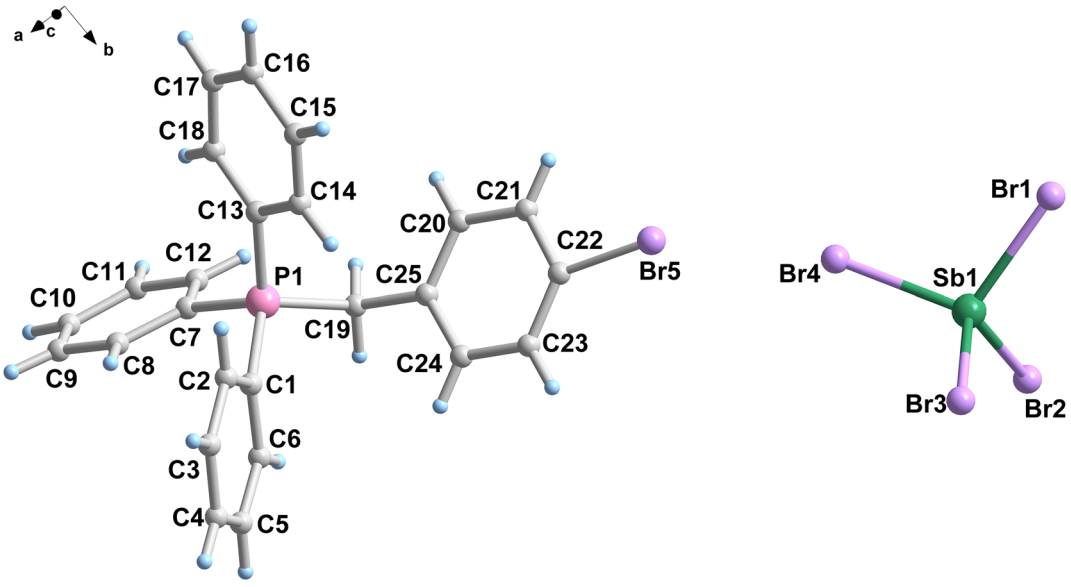
Xue Li and Guangyao Wang*

Crystal structure of [(4-bromobenzyl) triphenylphosphonium] tetrabromoantimony(III), $[C_{25}H_{21}BrP]^{+}[SbBr_4]^{-}$



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Abstract

$C_{25}H_{21}Br_5PSb$, monoclinic, $P2_1/c$ (no. 14), a = 10.5893(1) Å, b = 14.7743(2) Å, c = 18.9737(3) Å, β = 102.279(1)°, V = 2900.52(7) Å^3, Z = 4, $R_{gt}(F)$ = 0.0481, w$R_{ref}(F^2)$ = 0.1362, T = 293(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Clear light colourless block
Size:	0.12 × 0.10 × 0.08 mm
Wavelength:	CuKα radiation (1.54184 Å)
μ:	16.2 mm^{-1}
Diffractometer, scan mode:	Rigaku Synergy, ω scans
θ_{max}, completeness:	73.9°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	30343, 5811, 0.076
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4,967
$N(param)_{refined}$:	300
Programs:	Rigaku,[1] Olex2,[2] SHELX[3,4]

1 Source of materials

1 mmol of (4-bromobenzyl)triphenylphosphonium bromide and 1 mmol of $SbBr_3$ were dissolved in a 14 mL mixture of ethanol/dichloromethane in a 2:5 (v/v) ratio, and 0.5 mL of HBr solution was added. The resulting mixture was subjected to ultrasonic treatment for 10 min, ensuring the formation of a clear, homogeneous solution. The solution was kept at room temperature for several days, block-shaped single crystals were obtained.

***Corresponding author: Guangyao Wang**, School of Agriculture, Jilin Agricultural Science and Technology University, 132101 Jilin, Jilin, China, E-mail: wanggy_jlnku@163.com. https://orcid.org/0009-0006-4029-6143
Xue Li, College of Biology and Pharmaceutical Engineering, Jilin Agricultural Science and Technology University, 132101 Jilin, Jilin, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

	x	*y*	*z*	U_{iso}*/U_{eq}
Sb1	−0.29059 (3)	0.84883 (2)	0.61408 (2)	0.04832 (15)
Br1	−0.52310 (6)	0.80475 (6)	0.57658 (4)	0.0773 (2)
Br2	−0.23436 (8)	0.95849 (5)	0.52650 (4)	0.0758 (2)
Br3	−0.25289 (8)	0.91717 (5)	0.73554 (3)	0.0752 (2)
Br4	−0.16391 (8)	0.70512 (6)	0.62217 (5)	0.0826 (3)
P1	0.70586 (10)	0.33212 (7)	0.64386 (6)	0.0360 (2)
C1	0.7686 (4)	0.3940 (3)	0.7248 (3)	0.0422 (9)
C2	0.7511 (6)	0.3662 (4)	0.7919 (3)	0.0573 (13)
H2	0.697104	0.317688	0.795848	0.069*
C3	0.8154 (8)	0.4119 (5)	0.8532 (3)	0.0737 (18)
H3	0.803794	0.393974	0.898415	0.088*
C4	0.8956 (7)	0.4829 (6)	0.8475 (4)	0.081 (2)
H4	0.939077	0.512660	0.888774	0.097*
C5	0.9116 (8)	0.5102 (6)	0.7811 (5)	0.089 (3)
H5	0.966613	0.558230	0.777518	0.106*
C6	0.8473 (7)	0.4673 (5)	0.7195 (4)	0.0678 (16)
H6	0.856627	0.487497	0.674471	0.081*
C7	0.8430 (4)	0.2854 (3)	0.6157 (3)	0.0412 (9)
C8	0.9525 (6)	0.2650 (5)	0.6677 (4)	0.0722 (19)
H8	0.953366	0.274859	0.716199	0.087*
C9	1.0595 (7)	0.2302 (6)	0.6476 (5)	0.093 (3)
H9	1.131290	0.214138	0.682814	0.112*
C10	1.0622 (6)	0.2187 (4)	0.5761 (4)	0.0693 (17)
H10	1.137125	0.198598	0.562927	0.083*
C11	0.9548 (7)	0.2368 (5)	0.5247 (4)	0.0707 (17)
H11	0.954814	0.225696	0.476488	0.085*
C12	0.8446 (6)	0.2718 (5)	0.5437 (3)	0.0633 (15)
H12	0.772379	0.286030	0.508178	0.076*
C13	0.5970 (4)	0.2456 (3)	0.6587 (3)	0.0407 (9)
C14	0.4996 (5)	0.2658 (4)	0.6955 (3)	0.0514 (11)
H14	0.491927	0.324137	0.712575	0.062*
C15	0.4146 (6)	0.1989 (5)	0.7067 (4)	0.0676 (16)
H15	0.351912	0.211398	0.733004	0.081*
C16	0.4230 (6)	0.1136 (5)	0.6787 (5)	0.0746 (19)
H16	0.365881	0.068520	0.686095	0.089*
C17	0.5161 (6)	0.0949 (4)	0.6395 (5)	0.0741 (19)
H17	0.519059	0.037831	0.619165	0.089*
C18	0.6045 (6)	0.1598 (4)	0.6303 (4)	0.0551 (13)
H18	0.668634	0.146268	0.605310	0.066*
C19	0.6244 (5)	0.4067 (4)	0.5723 (3)	0.0510 (12)
H19A	0.682592	0.455704	0.567025	0.061*
H19B	0.606327	0.373060	0.527426	0.061*
C20	0.3836 (6)	0.4053 (4)	0.5547 (3)	0.0578 (13)
H20	0.383660	0.351870	0.528786	0.069*
C21	0.2663 (6)	0.4425 (5)	0.5632 (4)	0.0696 (18)
H21	0.188224	0.414163	0.543748	0.084*
C22	0.2697 (6)	0.5218 (5)	0.6009 (4)	0.0677 (19)
C23	0.3810 (7)	0.5637 (5)	0.6302 (4)	0.0699 (18)
H23	0.380220	0.617203	0.655916	0.084*
C24	0.4978 (6)	0.5261 (4)	0.6218 (3)	0.0562 (13)
H24	0.575179	0.555042	0.641764	0.067*
C25	0.4993 (5)	0.4470 (3)	0.5843 (2)	0.0420 (10)
Br5	0.0966 (3)	0.5578 (3)	0.6001 (2)	0.0779 (7)
Br5A	0.1234 (3)	0.5898 (3)	0.6211 (2)	0.0784 (7)[a]

[a]Occupancy: 0.534 (7).

2 Experimental details

The crystal data were collected on a Rigaku diffractometer.[1] The structure was determined using the SHELXT program within the Olex2 and subsequently refined with SHELXL.[2–4] The structural model was visualized using Diamond.[5]

3 Comment

This study reports a antimony(III)-based salt compound, comprising a (4-bromobenzyl)triphenylphosphonium (**4BBTP**) cation, and a [$SbBr_4$]$^-$ anion. The **4BBTP** cation carries a positive charge, with the tetrahedron [$SbBr_4$]$^-$ anion serving as the counterion. The bond lengths of Sb–Br within the anion as well as the geometric parameters of the phosphonium cation are in agreement with values reported in the literatures.[6–9]

Interionic Br···Br distances < 3.5 Å are observed beween one bromido ligand of the tetrabromido antimonate anion and the bromo substituent of the cation **indicating** weak halogen–halogen interactions.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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