

Reactions of Bismuth Iodide with Ammonium, Phosphonium, and Bismuthonium Salts

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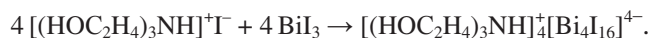
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Abstract—The reactions of ammonium, phosphonium, and bismuthonium salts with bismuth iodide were used to synthesize a series of complex compounds with bismuth-containing anions: $[(\text{HOC}_2\text{H}_4)_3\text{NH}]_4^+[\text{Bi}_4\text{I}_{16}]^{4-}$, $[\text{Ph}_3\text{EtP}]_3^+[\text{Bi}_2\text{I}_9]^{3-}$, and $[\text{Ph}_4\text{Bi}]_3^+[\text{Bi}_5\text{I}_{18}]^{3-}$. X-ray diffraction data show that the nitrogen atoms in the two types of crystallographically independent cations of the nitrogen-containing complex possess a distorted tetrahedral coordination [the CNC angles are $110.3(9)^\circ$ – $113.2(9)^\circ$]. In the tetranuclear centrosymmetric $[\text{Bi}_4\text{I}_{16}]^{4-}$ anion, the bismuth atoms have an octahedral coordination: Two types of groups, BiI_2 and BiI_3 , are bound with one another by four μ_2 - and two μ_3 -iodine bridges (the $\text{Bi}-\text{I}-\mu_2$, $\text{Bi}-\text{I}-\mu_3$, and $\text{Bi}-\text{I}-\mu_1$ distances are 3.1296(10), 3.2808(8); 3.3210(8) and 2.8670(8)–2.9108(9) Å, respectively). The coordination of the phosphorus atom in the $[\text{Ph}_3\text{EtP}]^+$ cations of the phosphorus-containing complex is close to tetrahedral (the CPC angles are 107.5° – 114.1°). In the binuclear $[\text{Bi}_2\text{I}_9]^{3-}$ anions, the bismuth atoms have an octahedral coordination. The axial $\text{I}-\text{Bi}-\text{I}$ angles are $167.52(2)^\circ$, $169.84(2)^\circ$, and $174.97(2)^\circ$. The terminal BiI_3 fragments $[\text{Bi}^2-\text{I}]^{7,8,9}$ 2.9238(7), 2.9236(7), and 2.9522(7) Å] are in the eclipsed conformation.

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Proceeding with our systematic research on the chemistry and structure of halogenated Bi(III) compounds, we have prepared a large series of bismuth complexes and studied their crystal structure [1–5]. The present work deals with the specific features of the synthesis of complexes with bismuth-containing anions prepared from ammonium, phosphonium, and bismuthonium salts and bismuth iodide. Triethanolammonium bis(μ_3 -iodo)tetrakis(μ_2 -iodo)deca-iodotetrabismuthate(III) $[(\text{HOC}_2\text{H}_4)_3\text{NH}]_4^+[\text{Bi}_4\text{I}_{16}]^{4-}$ (**I**) was prepared from equimolar amounts of triethanolammonium iodide and bismuth iodide at 20°C in acetone.

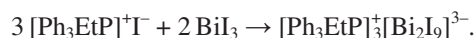


I

The nitrogen atoms in the two types of crystallographically independent cations in complex **I** exhibit a distorted tetrahedral coordination [CNC angles $110.3(9)^\circ$ – $113.2(9)^\circ$]. The Bi atoms in the tetranuclear centrosymmetric $[\text{Bi}_4\text{I}_{16}]^{4-}$ anion have an octahedral coordination. The axial angles at the Bi atoms vary in the range $169.90(3)^\circ$ – $176.28(2)^\circ$. Two

types of groups, BiI_2 , and BiI_3 , are bound with one another by four μ_2 - and two μ_3 -iodine bridges. The $\text{Bi}-\text{I}-\mu_2$, $\text{Bi}-\text{I}-\mu_3$, and $\text{Bi}-\text{I}-\mu_1$ distances are 3.1296(10), 3.2808(8); 3.3210(8) and 2.8670(8)–2.9108(9) Å. Note that with triethanolammonium bromide instead of triethanolammonium iodide, too, complex **I** was obtained. Probably, in the latter case, the first step in the synthesis of triethanolammonium iodide by ligand exchange between triethanolammonium bromide and bismuth iodide, and then complex **I** is formed.

The reaction of triphenylethylphosphonium iodide with bismuth iodide in acetone gave complex **II**. According to X-ray diffraction data, it comprises three types of crystallographically independent triphenylethylphosphonium cations and binuclear $[\text{Bi}_2\text{I}_9]^{3-}$ anions in which two bismuth atoms are bound with one another by three iodine bridges.



II

The coordination of the P atoms in the $[\text{Ph}_3\text{EtP}]^+$ cations comes close to tetrahedral [CPC angles 107.5° – 114.1° , P–C distances 1.785(8)–1.801(7) Å]. In the

binuclear $[\text{Bi}_2\text{I}_9]^{3-}$ anions, the Bi atoms have an octahedral coordination like in complex **I**. The axial BiI angles are $167.59(2)$, $169.84(2)$, and $174.97(2)^\circ$, respectively. the terminal BiI_3 fragments $[\text{Bi}^2-\text{I}(7,8,9)$ $2.9238(7)$, $2.9236(7)$, and $2.9522(7)$ Å] are in the eclipsed conformation. The pairs of distances between the bismuth and bridging iodine atoms $[\text{Bi}^1-\text{I}^4$ $3.360(1)$ Å and Bi^2-I^4 $3.290(1)$ Å, Bi^1-I^5 $3.189(1)$ Å and Bi^2-I^5 $3.282(1)$ Å, Bi^1-I^6 $3.322(1)$ Å and Bi^2-I^6 $3.224(1)$ Å] are nonequivalent.

The reaction of tetraphenylbismuth 3-carboxy-4-hydroxybenzenesulfonate with bismuth iodide in a 1:2 molar ratio gives complex **III**.



III



According to preliminary X-ray diffraction data, complex **III** has three crystallographically independent tetraphenylbismuthonium cations located on mirror symmetry planes, and the linear anions, in symmetry centers. In the centrosymmetric five-nuclear $[\text{Bi}_5\text{I}_{18}]^{3-}$ anion, the octahedrally coordinated Bi atoms are coupled by triple iodine bridges. The BiI angles in the central fragments span a narrower range (77.99° – 79.21°) than in the terminal ones (78.07° – 80.60°).

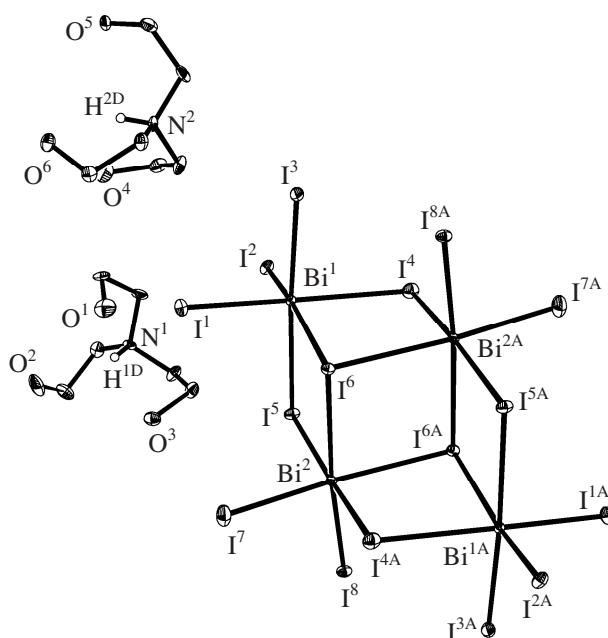


Fig. 1. Structure of the two types of cations and the anion in complex **I**.

Hence, the structure of adducts of bismuth iodide with ammonium, phosphonium, and bismuthonium salts is determined by the nature of the starting ammonium, phosphonium, and bismuthonium salt.

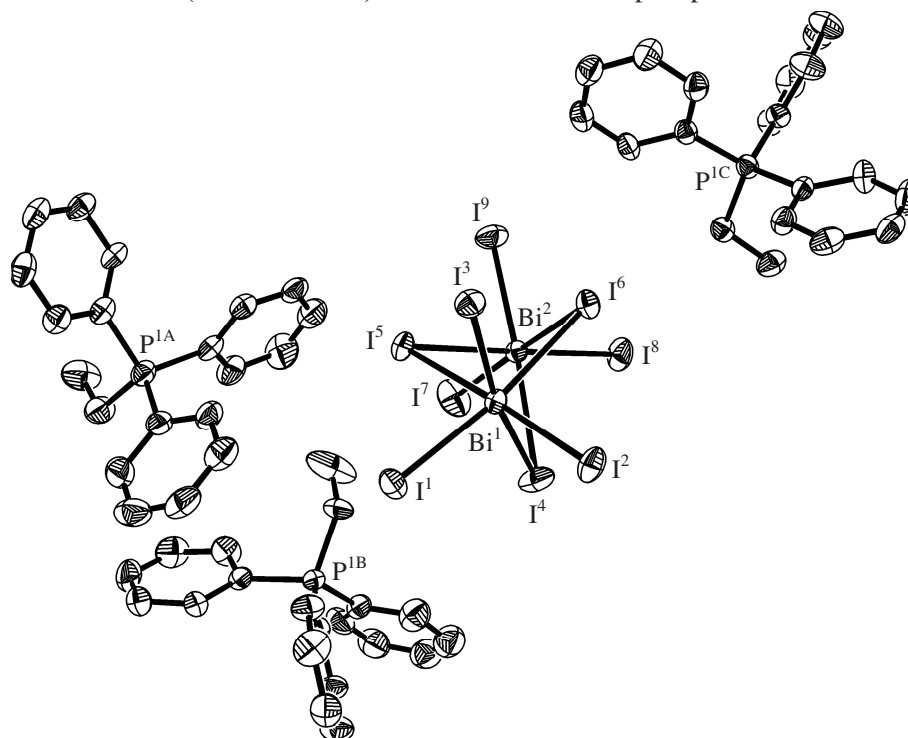


Fig. 2. Structure of complex **II**.

Table 1. Crystallographic data and parameters of experiment and refinement of structures **I** and **II**

Parameter	Value	
	I	II
<i>M</i>	3463.08	2434.05
<i>T</i> , K	100(2)	100(2)
Syngony	Orthorhombic	Monoclinic
Space group	<i>Pbca</i>	<i>P2₁/n</i>
Unit cell parameters	<i>a</i> 13.3862(7) Å	<i>a</i> 12.602(2) Å
	<i>b</i> 22.0695(11) Å	<i>b</i> 27.125(4) Å
	<i>c</i> 22.9034(11) Å	<i>c</i> 20.924(3) Å
	α 90°	α 90°
	β 90°	β 101.024(3)°
	γ 90°	γ 90°
<i>V</i> , Å ³	6766.3(6)	7020.5(17)
<i>Z</i>	4	4
ρ_{calc} , g cm ⁻³	3.400	2.303
μ , mm ⁻¹	17.715	9.066
<i>F</i> (000)	6032	4432
Crystal shape	Prysm	Prysm
(dimensions, mm)	(0.33×0.13×0.11)	(0.86×0.56×0.49)
θ range, deg	1.78–26.00	1.81–27.00
<i>h</i> , <i>k</i> , and <i>l</i> ranges	–16 ≤ <i>h</i> ≤ 16,	–16 ≤ <i>h</i> ≤ 16,
	–21 ≤ <i>k</i> ≤ 27,	–34 ≤ <i>k</i> ≤ 34,
	–28 ≤ <i>l</i> ≤ 26	–26 ≤ <i>l</i> ≤ 26
Number of reflections measured	38923	64825
Number of independent reflections	6651 (<i>R</i> _{int} 0.0583)	15322 (<i>R</i> _{int} 0.0542)
Refinement variables	271	667
<i>GOOF</i>	1.048	1.064
<i>R</i> factors on <i>F</i> ² > 2σ(<i>F</i> ²)	<i>R</i> ₁ 0.0381, <i>wR</i> ₂ 0.0950	<i>R</i> ₁ 0.0419, <i>wR</i> ₂ 0.0984
<i>R</i> factors on all reflections	<i>R</i> ₁ 0.0507, <i>wR</i> ₂ 0.0991	<i>R</i> ₁ 0.0672, <i>wR</i> ₂ 0.1052
Residual electron density (min/max), e Å ⁻³	–2.989/2.586	–1.823/3.020

EXPERIMENTAL

The X-ray diffraction patterns of complexes **I** and **II** were obtained on a Bruker AXS Smart Apex automatic diffractometer (MoK α radiation, λ 0.71073 Å, graphite monochromator). The structures were solved by the direct method using the SIR program [6] and

refined by the least-squares method anisotropically for non-hydrogen atoms using SHELXL-97 program [7]. All calculations were carried out using the SHELXL-97 [7] and Bruker SHELXTL [8] programs. The principal crystallographic data and results of structure refinement are listed in Table 1, atomic coordinates and thermal parameters, in Table 2, and principal interatomic distances and bond angles, in Table 3.

The IR spectra were recorded on a FMS-1201 Fourier IR spectrometer in KBr pellets.

Triethanolammonium bis(μ₃-iodo)tetrakis(μ₂-iodo)decaiodotetrabismuthate(III) (I). A mixture of 0.27 g of triethanolammonium iodide, 0.59 g of bismuth triiodide, and 20 ml of acetone was stirred for 1 h at 20°C, and the resulting solution was evaporated for 24 h. Yield 0.78 g (91%), red cherry crystals, mp 202°C. IR spectrum, ν , cm⁻¹: 524 m, 845 m, 906 s, 995 s, 1023 s, 1050 s, 1083 s, 1252 m, 1446 s, 2920 w. Found, %: C 8.01, H 1.33. C₂₄H₆₀Bi₄I₁₀N₄O₁₂. Calculated, %: C 8.31, H 1.73.

Triphenylethylphosphonium tris(μ₂-iodo)hexaiododibismuthate(III) (II). A mixture of 0.70 g of bismuth iodide, 0.70 g of triphenylethylphosphonium iodide, and 50 ml of acetone was stirred for 1 h. The solvent was removed, and the residue was crystallized from DMSO. Yield 1.41 g (90%), dark red crystals, mp 194°C. IR spectrum, ν , cm⁻¹: 535 s, 574 m, 688 m, 731 s, 944 s, 987 s, 1019 m, 1117 s, 1440 s, 1637 s, 2857 w, 2924 w. Found, %: C 29.12, H 2.56. C₆₀H₉₀Bi₂I₉P₃. Calculated, %: C 29.58, H 2.46.

Tetraphenylbismuthonium dodeca(μ₂-iodo)hexaiodopentabismuthate(III) (III). A mixture of 0.23 g of tetraphenylbismuth 3-carboxy-4-hydroxybenzenesulfonate, 0.36 g of bismuth triiodide, and 30 ml of acetone was kept at 20°C for 24 h. Yield 0.50 g (98%), dark red crystals, mp 182°C. IR spectrum, ν , cm⁻¹: 431 m, 443 m, 650 m, 679 s, 714 s, 723 s, 836 w, 989 s, 1011 m, 1051 m, 1158 w, 1182 w, 1325 m, 1433 s, 1469 m, 1562 m, 1633 w, 1696 w, 2855 w, 2923 w, 2990 w, 3045 w, 3069 w, 3443 m. Found, %: C 17.50, H 1.15, I 46.58. C₇₂H₆₉Bi₈I₁₈. Calculated, %: C 17.70, H 1.23, I 46.82.

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Table 2. Atomic coordinates ($\times 10^4$) and isotropic equivalent thermal parameters ($\times 10^3$) in structures **I** and **II**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}, \text{\AA}^2$	Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}, \text{\AA}^2$
Complex I					Complex II				
Bi ¹	1873(1)	1289(1)	5568(1)	11(1)	I ⁷	872(1)	832(1)	4627(1)	109(1)
I ¹	2041(1)	1354(1)	6834(1)	32(1)	I ⁸	1988(1)	2280(1)	4454(1)	86(1)
N ¹	304(6)	4325(5)	6556(4)	18(2)	I ⁹	4180(1)	1105(1)	4877(1)	87(1)
O ¹	1480(7)	3501(5)	7060(5)	46(3)	P ^{1C}	5962(2)	3202(1)	4226(1)	48(1)
C ¹	1387(9)	4268(7)	6385(6)	31(3)	C ^{1C}	6773(6)	3514(3)	4898(4)	55(2)
Bi ²	–1306(1)	363(1)	5613(1)	12(1)	C ^{2C}	7778(7)	3705(4)	4828(5)	82(3)
I ²	2127(1)	2563(1)	5337(1)	21(1)	C ^{3C}	8465(8)	3905(4)	5350(5)	90(3)
O ²	–292(9)	4513(5)	7723(4)	48(3)	C ^{4C}	8151(9)	3940(4)	5935(5)	86(3)
N ²	5302(7)	2360(4)	6645(4)	17(2)	C ^{5C}	7185(10)	3754(4)	6014(5)	86(3)
C ²	1964(8)	4041(7)	6911(6)	31(3)	C ^{6C}	6488(7)	3537(3)	5495(4)	62(2)
I ³	3951(1)	978(1)	5528(1)	22(1)	C ^{7C}	6624(6)	2636(3)	4087(3)	49(2)
O ³	–886(6)	3312(4)	6663(4)	29(2)	C ^{8C}	6264(6)	2183(3)	4263(4)	57(2)
C ³	71(10)	4948(6)	6793(5)	27(3)	C ^{9C}	6834(8)	1767(3)	4206(4)	72(2)
I ⁴	1801(1)	980(1)	4235(1)	27(1)	C ^{10C}	7754(8)	1786(3)	3971(5)	76(2)
O ⁴	4230(7)	3354(4)	6957(4)	36(2)	C ^{11C}	8127(8)	2233(3)	3774(5)	79(3)
C ⁴	–696(10)	4889(6)	7275(6)	34(3)	C ^{12C}	7561(7)	2656(3)	3827(4)	66(2)
I ⁵	–510(1)	1635(1)	5522(1)	20(1)	C ^{13C}	5807(6)	3577(2)	3503(3)	46(2)
O ⁵	7224(6)	2865(4)	6932(3)	24(2)	C ^{14C}	6057(7)	4077(3)	3529(4)	64(2)
C ⁵	–386(10)	4141(6)	6075(6)	28(3)	C ^{15C}	5883(8)	4343(3)	2957(5)	80(3)
I ⁶	1044(1)	–116(1)	5760(1)	16(1)	C ^{16C}	5493(7)	4130(4)	2374(5)	69(2)
O ⁶	5184(7)	2388(4)	7857(4)	28(2)	C ^{17C}	5232(7)	3638(4)	2356(4)	71(2)
C ⁶	–507(10)	3460(6)	6087(5)	28(3)	C ^{18C}	5386(7)	3360(3)	2916(4)	61(2)
I ⁷	–1627(1)	475(1)	6850(1)	38(1)	C ^{19C}	4650(6)	3069(2)	4395(4)	55(2)
C ⁷	4394(10)	2544(6)	6307(6)	28(3)	C ^{20C}	3973(7)	3531(3)	4447(5)	75(3)
I ⁸	–3337(1)	715(1)	5394(1)	20(1)	P ^{1B}	–1919(2)	–367(1)	2588(1)	51(1)
C ⁸	4280(10)	3222(6)	6342(6)	28(3)	C ^{1B}	–2340(6)	–308(3)	1726(4)	50(2)
C ⁹	6188(9)	2324(6)	6251(5)	28(3)	C ^{2B}	–3387(6)	–176(3)	1450(4)	64(2)
C ¹⁰	7166(9)	2338(7)	6597(6)	31(3)	C ^{3B}	–3703(8)	–132(4)	787(4)	79(3)
C ¹¹	5118(10)	1784(5)	6983(5)	25(3)	C ^{4B}	–2986(9)	–220(3)	388(4)	79(3)
C ¹²	4591(9)	1947(5)	7551(5)	24(3)	C ^{5B}	–1943(9)	–342(4)	654(5)	94(3)
Complex II					C ^{6B}	–1613(7)	–394(4)	1325(4)	72(2)
Bi ¹	2268(1)	1064(1)	1843(1)	44(1)	C ^{7B}	–2751(7)	28(3)	2970(4)	62(2)
I ¹	1053(1)	259(1)	1088(1)	67(1)	C ^{8B}	–2796(9)	513(4)	2801(5)	96(3)
Bi ²	2270(1)	1325(1)	3861(1)	45(1)	C ^{9B}	–3540(10)	834(5)	3031(6)	110(4)
I ²	1696(1)	1769(1)	761(1)	78(1)	C ^{10B}	–4135(9)	648(5)	3421(6)	105(3)
I ³	4288(1)	802(1)	1425(1)	62(1)	C ^{11B}	–4085(9)	175(5)	3643(6)	103(3)
I ⁴	311(1)	1482(1)	2592(1)	82(1)	C ^{12B}	–3381(7)	–134(4)	3382(6)	94(4)
I ⁵	2697(1)	329(1)	3055(1)	55(1)	C ^{13B}	–2074(6)	–1003(6)	2808(4)	54(2)
I ⁶	3696(1)	1830(1)	2914(1)	59(1)	C ^{14B}	–2308(6)	–1357(3)	2331(5)	66(2)

Table 2. (Contd.)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} , Å ²	Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} , Å ²
Complex I					Complex II				
C ^{15B}	–2388(8)	–1849(3)	2490(6)	81(3)	C ^{8A}	405(8)	–1969(4)	2327(6)	88(3)
C ^{16B}	–2274(8)	–1985(4)	3110(7)	95(3)	C ^{9A}	10(9)	–1815(5)	1684(6)	98(4)
C ^{17B}	–2071(9)	–1638(5)	3597(6)	99(4)	C ^{10A}	367(10)	–1401(5)	1462(6)	95(3)
C ^{18B}	–1924(8)	–1156(4)	3449(5)	81(3)	C ^{11A}	1116(9)	–1113(4)	1831(5)	88(3)
C ^{19B}	–534(6)	–168(4)	2799(4)	79(3)	C ^{12A}	1516(7)	–1244(3)	2476(5)	71(2)
C ^{20B}	–31(10)	–257(6)	3507(5)	161(7)	C ^{13A}	3030(6)	–2187(3)	3537(4)	59(2)
P ^{1A}	1778(2)	–1856(1)	3542(1)	60(1)	C ^{14A}	3917(6)	–2120(3)	4013(4)	56(2)
C ^{1A}	2097(6)	–1312(3)	4031(4)	57(2)	C ^{15A}	4871(7)	–2381(3)	3979(4)	68(2)
C ^{2A}	1433(8)	–1155(4)	4448(5)	81(3)	C ^{16A}	4903(8)	–2694(3)	3473(5)	75(3)
C ^{3A}	1695(10)	–721(5)	4797(6)	109(4)	C ^{17A}	4012(8)	–2766(3)	3016(5)	78(3)
C ^{4A}	2558(9)	–455(4)	4730(5)	91(3)	C ^{18A}	3063(7)	–2512(3)	3031(4)	71(2)
C ^{5A}	3207(7)	–604(3)	4324(4)	69(2)	C ^{19A}	829(7)	–2258(4)	3816(4)	83(3)
C ^{6A}	2994(6)	–1030(3)	3985(4)	60(2)	C ^{20A}	1248(9)	–2482(5)	4482(5)	113(4)
C ^{7A}	1198(6)	–1668(3)	2728(4)	62(2)					

Table 3. Interatomic distances and bond angles structures I and II

Bond <i>d</i> , Å		Angle ω, deg		Bond <i>d</i> , Å		Angle ω, deg	
Complex I				Complex I			
Bi ¹ –I ³	2.8670(8)	I ³ Bi ¹ I ²	96.47(2)	C ³ –C ⁴	1.515(17)	I ⁸ Bi ² I ^{4A}	94.40(2)
Bi ¹ –I ²	2.8829(8)	I ³ Bi ¹ I ¹	88.19(3)	I ⁴ –Bi ^{2A}	3.0577(9)	I ⁷ Bi ² I ^{4A}	86.51(3)
Bi ¹ –I ¹	2.9108(9)	I ² Bi ¹ I ¹	97.26(3)	O ⁴ –C ⁸	1.439(16)	I ⁵ Bi ² I ^{4A}	171.46(3)
Bi ¹ –I ⁴	3.1296(10)	I ³ Bi ¹ I ⁴	86.92(2)	O ⁵ –C ¹⁰	1.394(16)	I ⁸ Bi ² I ^{6A}	88.77(2)
Bi ¹ –I ⁵	3.2808(8)	I ² Bi ¹ I ⁴	92.08(2)	C ⁵ –C ⁶	1.513(18)	I ⁷ Bi ² I ^{6A}	174.65(3)
Bi ¹ –I ⁶	3.3210(8)	I ¹ Bi ¹ I ⁴	169.90(3)	I ⁶ –Bi ^{2A}	3.2105(8)	I ⁵ Bi ² I ^{6A}	92.98(2)
N ¹ –C ⁵	1.494(15)	I ³ Bi ¹ I ⁵	176.28(2)	O ⁶ –C ¹²	1.438(14)	I ^{4A} Bi ² I ^{6A}	88.28(2)
N ¹ –C ¹	1.506(14)	I ² Bi ¹ I ⁵	83.21(2)	C ⁷ –C ⁸	1.506(17)	I ⁸ Bi ² I ⁶	175.01(2)
N ¹ –C ³	1.511(16)	I ¹ Bi ¹ I ⁵	95.53(2)	C ⁹ –C ¹⁰	1.530(18)	I ⁷ Bi ² I ⁶	93.95(3)
O ¹ –C ²	1.400(17)	I ⁴ Bi ¹ I ⁵	89.38(2)	C ¹¹ –C ¹²	1.524(17)	I ⁵ Bi ² I ⁶	88.19(2)
C ¹ –C ²	1.517(18)	I ³ Bi ¹ I ⁶	96.03(2)	Symmetry code A [– <i>x</i> , – <i>y</i> , – <i>z</i> + 1]			
Bi ² –I ⁸	2.8713(8)	I ² Bi ¹ I ⁶	167.03(2)	Complex II			
Bi ² –I ⁷	2.8762(10)	I ¹ Bi ¹ I ⁶	86.57(2)	Bi ¹ –I ³	2.9329(6)	I ³ Bi ¹ I ¹	93.01(2)
Bi ² –I ⁵	3.0099(8)	I ⁴ Bi ¹ I ⁶	85.16(2)	Bi ¹ –I ¹	2.9466(6)	I ³ Bi ¹ I ²	91.51(2)
Bi ² –I ^{4A}	3.0577(9)	I ⁵ Bi ¹ I ⁶	84.09(2)	Bi ¹ –I ²	2.9488(6)	I ¹ Bi ¹ I ²	92.47(2)
Bi ² –I ^{6A}	3.2105(8)	C ⁵ N ¹ C ¹	112.4(10)	Bi ¹ –I ⁵	3.1889(6)	I ³ Bi ¹ I ⁵	93.51(2)
Bi ² –I ⁶	3.3354(8)	C ⁵ N ¹ C ³	112.6(9)	Bi ¹ –I ⁶	3.3223(6)	I ¹ Bi ¹ I ⁵	87.45(2)
O ² –C ⁴	1.425(17)	C ¹ N ¹ C ³	111.6(10)	Bi ¹ –I ⁴	3.3603(7)	I ² Bi ¹ I ⁵	174.97(2)
N ² –C ⁹	1.492(14)	N ¹ C ¹ C ²	108.1(9)	Bi ² –I ⁸	2.9236(7)	I ³ Bi ¹ I ⁶	87.88(2)
N ² –C ⁷	1.497(15)	I ⁸ Bi ² I ⁷	90.41(3)	Bi ² –I ⁷	2.9238(7)	I ¹ Bi ¹ I ⁶	169.84(2)
N ² –C ¹¹	1.509(15)	I ⁸ Bi ² I ⁵	94.07(2)	Bi ² –I ⁹	2.9522(7)	I ² Bi ¹ I ⁶	97.63(2)
O ³ –C ⁶	1.449(15)	I ⁷ Bi ² I ⁵	92.35(3)	Bi ² –I ⁶	3.2237(6)	I ⁵ Bi ¹ I ⁶	82.39(2)

Table 3. (Contd.)

Bond d , Å		Angle ω , deg		Bond d , Å		Angle ω , deg	
Complex II				Complex II			
Bi ² –I ⁵	3.2822(6)	I ³ Bi ¹ I ⁴	167.59(2)	P ^{1C} –C ^{19C}	1.792(7)	C ^{1C} P ^{1C} C ^{19C}	110.6(4)
Bi ² –I ⁴	3.2896(7)	I ¹ Bi ¹ I ¹	98.51(2)	P ^{1C} –C ^{7C}	1.798(7)	C ^{1C} P ^{1C} C ^{7C}	108.6(3)
P ^{1C} –C ^{1C}	1.785(8)	I ² Bi ¹ I ⁴	92.59(2)	P ^{1C} –C ^{13C}	1.801(7)	C ^{1C} P ^{1C} C ^{13C}	110.3(4)

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