

Exchange of Halogens in the 3a,6a-Diaza-1,4-Diphosphapentalene Derivatives: Crystal Structures of Iodides

A. N. Kornev*, V. E. Galperin, Yu. S. Panova, V. V. Sushev, A. V. Arapova,
G. K. Fukin, E. V. Baranov, and G. A. Abakumov

Razuvaev Institute of Organometallic Chemistry, Russian Academy of Sciences,
ul. Tropinina 49, Nizhny Novgorod, 603600 Russia

*e-mail: akornev@iomc.ras.ru

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Abstract—1,4-Dichloro-3a,6a-diaza-1,4-diphosphapentalene (**II**) easily exchanges halogen with methyl iodide to form the corresponding 1,4-diiodo derivative (**V**) in a quantitative yield. The reaction of compound **II** with diiodine (1 equiv) affords compound **III**, the crystal structure of which contains 55% **II** and 45% **V**. Under the conditions of iodine excess (1 : 3), a ionic compound (**IV**) is formed, the crystal of which contains alternating layers consisting of planar networks $[I_2I_3]^-$ and heterocyclic cations $[DDP-Cl]^+$. For the crystallographic information for compounds **III**–**V**, see CIF files CCDC no. 1560410 (**V**), 1560411 (**III**), and 1560412 (**IV**).

Keywords: heterophospholes, heteropentalenes, phosphorus halides

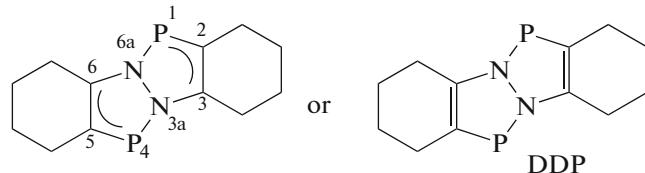
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INTRODUCTION

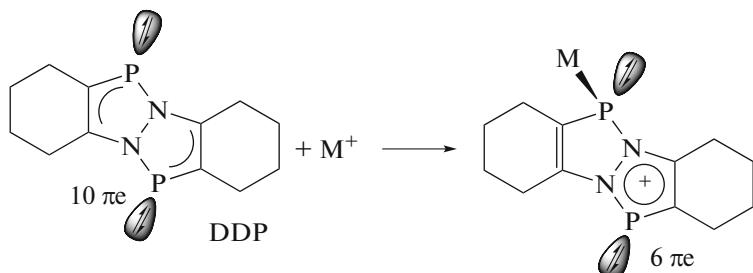
Phosphorus-containing heterocycles are universal building blocks for the preparation of organic π -conjugated polymeric materials with a high application potential in electronic and optoelectronic devices [1].

The most attention is presently given to functional heterophospholes, five-membered heterocycles containing heteroatoms along with phosphorus atoms [2]. 3a,6a-Diaza-1,4-diphosphapentalene (**I**, further DDP) synthesized by us for the first time [3] and belonging to the class of annelated diazaphospholes [4] demonstrates the chemical properties quite differ-

ent from the properties of aromatic azaphospholes and diazaphospholes.

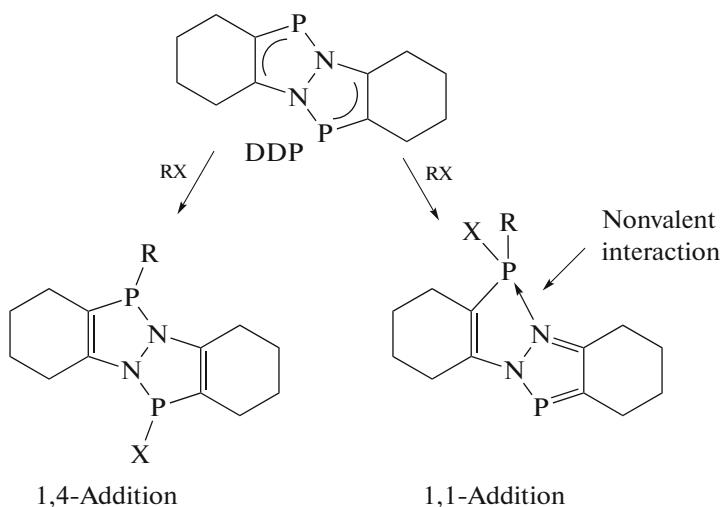


It has been revealed by us that DDP participates in complex formation with metal ions via a new mechanism in which the unpaired electron pair at the phosphorus atoms is not involved [3, 5].



We have recently shown that DDP reacts with halohydrocarbons to form 1,1- or 1,4-addition products retaining the oxidation state of phosphorus [6, 7].

At the same time, similar addition reactions of phosphorus atoms of aromatic azaphospholes are unknown.



The use of 1,4-dihalogen derivatives of DDP as precursors via the substitution of halogens by bridging groups is assumed for the synthesis of oligomers and polymers containing the DDP block. We have previously synthesized 1,4-dichloro-3a,6a-diaza-1,4-diphosphapentalene (**II**) and 1,4-dibromo-3a,6a-diaza-1,4-diphosphapentalene [7, 8]. In this work, we describe the synthesis and crystal structure of the 1,4-diido derivative of DDP (**III**), as well as the crystal structures of compounds **IV** and **V** prepared by the reactions of compound **II** with molecular iodine taken in different molar ratios.

EXPERIMENTAL

Compounds **III**–**V** were synthesized in vacuo and under argon of high purity. The solvents (toluene, tetrahydrofuran (THF), diethyl ether, and benzene) were refluxed and distilled over sodium. Compound **II** was synthesized according to a described procedure [8].

Reaction of compound II with I₂ (1 : 1). A solution of diiodine (0.25 g, 1.0 mmol) in THF was added to a solution of compound **II** (0.32 g, 1.0 mmol) in THF, and the mixture was heated for 3 h at 60°C. The solution was concentrated in vacuo. The residue was kept at 0°C to precipitate red-brown crystals of product **III**. The yield was 0.24 g (60%).

For C₁₂H₁₆N₂P₂Cl_{1.0.9} (**III**) (FW = 403.4)

Anal. calcd., %	C, 35.73	H, 4.00	P, 15.36
Found, %	C, 35.68	H, 4.05	P, 15.40

IR (Nujol), ν , cm⁻¹: 1589 s, 1345 m, 1290 m, 1248 s, 1180 m, 1160 m, 1132 w, 1076 w, 1023 m, 961 m, 920 m, 855 w, 807 m, 587 m, 544 m.

Reaction of compound II with I₂ (1 : 3). A solution of diiodine (0.75 g, 3.0 mmol) in THF was added to a solution of compound **II** (0.32 g, 1.0 mmol) in THF, and the mixture was heated for 0.5 h at 60°C. The solution was concentrated in vacuo. The residue was kept at 0°C to precipitate dark red crystals of product **IV**. The yield was 0.53 g (65%).

For C₁₂H₁₆N₂P₂Cl_{0.75}I_{4.25} (**IV**) (FW = 816.12)

Anal. calcd., %	C, 17.69	H, 1.98	P, 7.60
Found, %	C, 17.73	H, 2.02	P, 7.57

IR (Nujol), ν , cm⁻¹: 1624 w, 1276 w, 1250 w, 1220 w, 1186 w, 1160 w, 1125 w, 1032 m, 958 w, 912 m, 802 m, 773 w, 592 w, 553 m, 465 m.

Reaction of compound II with CH₃I. A solution of diiodine (0.71 g, 5.0 mmol) in THF was added to a solution of compound **II** (0.32 g, 1.0 mmol) in THF, and the mixture was heated for 1 h at 60°C. The solution was concentrated in vacuo. The residue was kept at 0°C to precipitate dark red crystals of product **V**. The yield was 0.49 g (98%).

For C₁₂H₁₆N₂P₂I₂ (**V**) (FW = 504.01)

Anal. calcd., %	C, 28.60	H, 3.20	I, 50.36	P, 12.29
Found, %	C, 28.57	H, 3.25	I, 50.40	P, 12.25

¹H NMR (THF-d₈), δ , ppm: 1.6–3.2. ³¹P{¹H} NMR: 125.5 (br). IR (Nujol), ν , cm⁻¹: 1574 s, 1282 s, 1247 s, 1180 m, 1157 s, 1073 w, 1016 s, 964 m, 916 s, 852 m, 801 s, 675 w, 585 m, 543 m, 532 m, 479 s, 457 m.

NMR spectra were recorded on Bruker DPX-200 and AV-400 spectrometers. IR spectra were measured on a PerkinElmer 577 instrument in the range from

4000 to 400 cm^{-1} in Nujol or on a PerkinElmer FT-IR 2000 instrument.

X-ray Crystallography. Crystallographic data were collected on Agilent Xcalibur E (for compounds **IV** and **V**) and Bruker AXS SMART APEX (for compound **III**) automated diffractometers (MoK_{α} radiation, $\lambda = 0.71073 \text{ \AA}$). Experimental arrays of intensities were integrated using the CrysAlisPro [9] (for **IV** and **V**) and SAINTPlus [10] (for **III**) programs. The structures were solved by a direct method followed by the full-matrix least-squares refinement for F^2 using the SHELXTL program [11]. An absorption correction was applied using the SCALE3 ABSPACK [12] (for **IV** and **V**) and SADABS [13] (for **III**) programs. All non-hydrogen atoms were refined in the anisotropic approximation. Hydrogen atoms were placed in the geometrically calculated positions and refined in the riding model. In compounds **III** and **IV**, the I and Cl halogen atoms are disordered over two positions with population ratios of 0.45 : 0.55 and 0.25 : 0.75, respectively. The crystallographic characteristics and the main refinement parameters are presented in Table 1. The crystallographic information for compounds **III**–

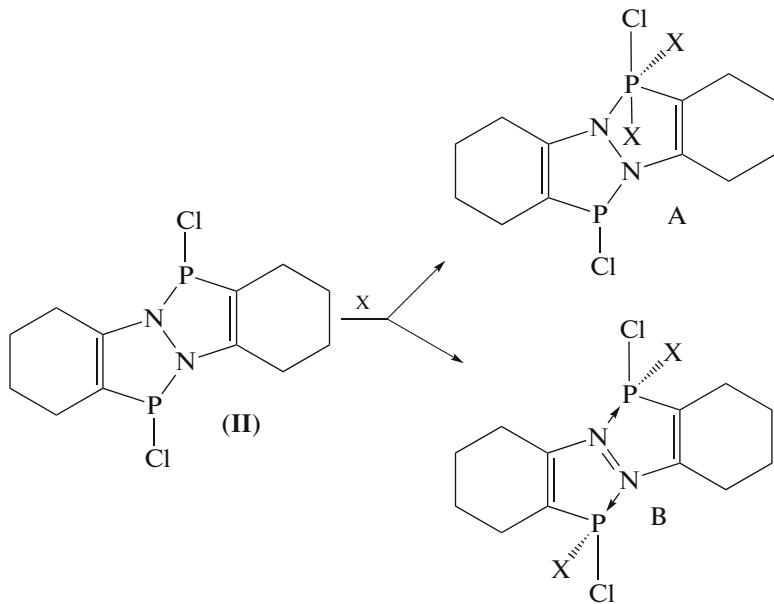
V was deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC no. 1560410 (**V**), 1560411 (**III**), and 1560412 (**IV**); deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

RESULTS AND DISCUSSION

It is known that tertiary phosphines reversibly react with halogens to form, as a rule, phosphonium salts [14].



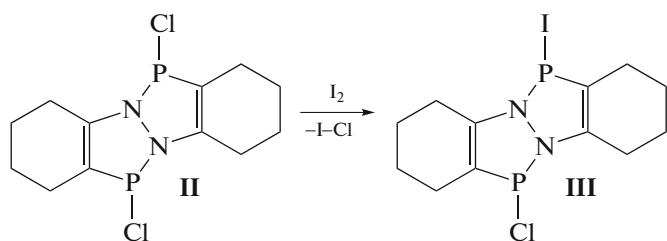
The properties of these compounds differ strongly depending on the nature of the organic substituent and halogen. Compound **II** is a more complicated representative of tertiary phosphines, and its reaction with iodine can proceed at several reaction centers. In particular, since DDP contains C=C multiple bonds, there is a principal possibility of halogen addition with the formation of $\text{Hal}-\text{C}=\text{C}-\text{Hal}$ fragments. In addition, there is an alternative of halogen addition either to one phosphorus atom, or to different phosphorus atoms to form compounds of pentavalent (A) or hypervalent (B) phosphorus atoms.



Since the addition of halogen atoms to phosphines of the $\text{R}_2\text{P}\text{Hal}$ type is an equilibrium process [14], the exchange of halogens is also possible, which was observed indeed.

The storage of a mixture of solutions of equivalent amounts of dichloro-DDP (**II**) and diiodine in chlo-

roform for 24 h followed by solvent removal results in brown crystals. The X-ray diffraction analysis of the obtained product **II** shows that the crystal consists of an approximately equimolar mixture of DDP dichloride and diiodide; i.e., in this case, no complete replacement of chlorine by iodine occurs.

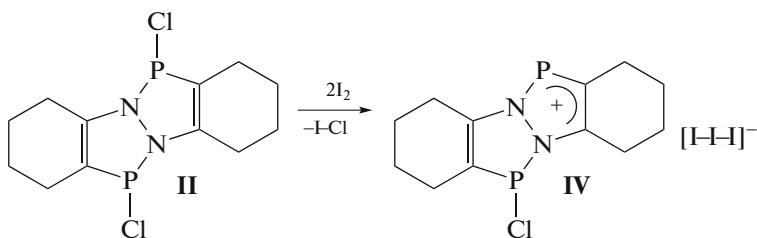


The main crystallographic characteristics for compound **III** are presented in Table 1. The molecular structure of compound **III** is shown in Fig. 1. Selected bond lengths and bond angles are given in Table 2.

In the crystal structure of compound **III**, the halogen atoms (I and Cl) are disordered over two positions with a population ratio of 0.45 : 0.55, respectively. The geometry of compound **III** resembles that of compound **II**, which was characterized by us earlier [8]. A molecule of compound **III** has the symmetry center at the center of the nitrogen–nitrogen bond. The conformation of the five-membered heterocycles in compound **III** is close to planar: the average deviation of the atoms from the plane is 0.05 Å. The sum of the

angles at the nitrogen atoms is 358.5°. The C–C distance in the heterocycles (1.348(2) Å) corresponds to the C(sp²)–C(sp²) multiple bonds. The phosphorus atom has a pyramidal geometry with a sum of bond angles of 290.8°. An analysis of the crystal packing revealed numerous short contacts between the atoms of the adjacent molecules: H···I 2.95, C···H 2.85, and H···H 2.35 Å.

The reaction of compound **II** with diiodine carried out under the same conditions but in a diiodine excess (1 : 3) affords product **IV** of the ionic structure containing the organophosphorus cation and triiodide anion.



The main crystallographic characteristics and refinement parameters for compound **IV** are given in Table 1. The molecular structure of compound **IV** is shown in Fig. 2a. Selected bond lengths and bond angles are listed in Table 2.

The crystal structure of compound **IV** additionally contains a diiodine molecule forming short contacts with the triiodide anions. As a result, a planar polyanionic network is formed of alternating I₂ and I₃⁻ fragments (Fig. 3). The I···I distances between the diiodine molecule and triiodide anion (3.309 Å) range between the values of the doubled covalent (2.80 Å) and van der Waals (3.96 Å) radii. The distances between the triiodide-anionic fragments of the network are 3.474 Å. The I–I bonds in the triiodide anions are not equivalent (2.822 and 3.070 Å) because of a distinction in the coordination of the terminal iodine atoms.

Note that iodine tends to form oligomeric and polymeric zigzag chains, whose shape depends on the counterion. The salts with superconducting properties are formed with some organoelement counterions [15].

The cationic moiety of compound **IV** is a monohalogen-substituted DDP block and is formed due to the partial exchange of chlorine by iodine in compound **II** and the subsequent elimination of the iodide anion. The halogen atoms (I and Cl) are disordered with a population ratio of 0.25 : 0.75, respectively. The geometry of the heteropentalene framework in the cation of compound **IV** is close to planar, the maximum deviation of the atoms from the plane is 0.047(1) Å (for the tricoordinated phosphorus atom), and the average deviation of 0.021 Å. The vector of the Cl(1)–P(1) bond forms an angle of 78.1° with the heterocycle plane. This angle is somewhat smaller (71.3°) in the case of the I(1)–P(1) vector. In the cation of compound **IV**, the five-membered heterocycles are non-equivalent, which is seen from the difference in bond lengths. The C(7)–C(12) bond (1.399(6) Å) is aromatic, whereas the C(1)–C(6) bond (1.319(6) Å) partially shifts from conjugation. At the same time, the migration of the positive charge to the N(1)N(2)C(7)C(12)P(2) heterocycle results in the enhancement of P(2)–N(1) bonding and in the shortening of the bond to 1.658(3) Å compared to P(1)–N(2) (1.760(4) Å) in the adjacent heterocycle. The

Table 1. Crystallographic data and parameters of X-ray diffraction experiments for compounds **III–V**

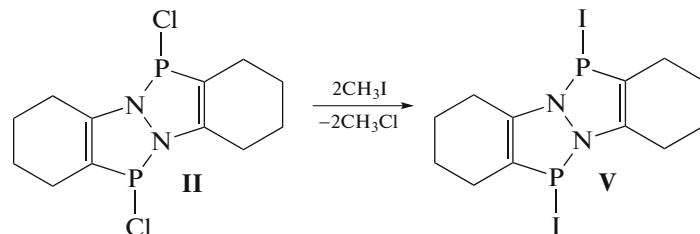
Parameter	Value		
	V	III	IV
Empirical formula	$C_{12}H_{16}I_2N_2P_2$	$C_{12}H_{16}Cl_{1.10}I_{0.90}N_2P_2$	$C_{12}H_{16}Cl_{0.75}I_{4.25}N_2P_2$
<i>FW</i>	504.01	403.41	816.12
Temperature, K	100(2)	100(2)	100(2)
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	$P2_1/n$	$P\bar{1}$	$P2_1/n$
<i>a</i> , Å	7.5107(6)	5.1570(4)	9.35331(9)
<i>b</i> , Å	7.1272(5)	8.1788(7)	14.15418(15)
<i>c</i> , Å	15.1283(9)	8.8592(7)	15.95965(16)
α , deg	90	95.848(1)	90
β , deg	101.837(6)	93.371(1)	97.2926(10)
γ , deg	90	95.540(1)	90
<i>V</i> , Å ³	792.60(9)	369.09(5)	2095.78(4)
<i>Z</i>	2	1	4
ρ , mg m ⁻³	2.112	1.815	2.587
μ , mm ⁻¹	4.155	2.360	6.559
<i>F</i> (000)	476	198	1480
Crystal size, mm	0.40 × 0.20 × 0.03	0.39 × 0.28 × 0.09	0.20 × 0.10 × 0.10
Date collection over θ range, deg	3.17–28.00	2.52–27.00	2.95–26.00
Ranges of reflection indices	$-9 \leq h \leq 9$, $-9 \leq k \leq 9$, $-19 \leq l \leq 19$	$-6 \leq h \leq 6$, $-10 \leq k \leq 10$, $-11 \leq l \leq 11$	$-11 \leq h \leq 11$, $-17 \leq k \leq 17$, $-19 \leq l \leq 19$
Measured reflections	12389	3256	30977
Independent reflections	1885	1561	4107
Reflections with $I > 2\sigma(I)$	1629	1516	3896
R_{int}	0.0380	0.0107	0.0238
Number of refined parameters	82	82	187
Goodness-of-fit (F^2)	1.021	1.000	1.050
R_1 , wR_2 ($I > 2\sigma(I)$)	0.0509, 0.1479	0.0216, 0.0548	0.0276, 0.0604
R_1 , wR_2 (for F^2 for all reflections)	0.0579, 0.1529	0.0228, 0.0555	0.0299, 0.0614
Residual electron density ($\rho_{\text{min}}/\rho_{\text{max}}$), $e \text{ \AA}^{-3}$	1.135/–0.676	0.387/–0.280	2.783/–1.320

same shortening is also observed for the P(2)–C(12) bond (1.745(4) Å) compared to P(1)–C(1) (1.828(4) Å) in the adjacent heterocycle.

Multiple short contacts between the iodine atoms of the polyanionic network with the phosphorus atoms

of the cationic moiety (3.62–3.70 Å) are observed in the crystal structure of compound **IV**.

Chlorine cannot completely be replaced by iodine using the treatment of DDP dichloride with diiodine. For this purpose, we used the exchange reaction with methyl iodide



The reflux of compound **II** with a minor excess of MeI (1 : 5) in a THF solution provides a quantitative yield of diiodide **V**. The ^{31}P NMR spectrum of compound **V** exhibits a broad singlet at 125.5 ppm indicating dissociation processes that occur in the solution. The crystals of compound **V** suitable for X-ray diffrac-

tion analysis were obtained by keeping a THF solution of compound **V** at 0°C. The main crystallographic characteristics for crystals **V** are given in Table 1. The molecular structure of compound **V** is shown in Fig. 2b. Selected bond lengths and bond angles are given in Table 2.

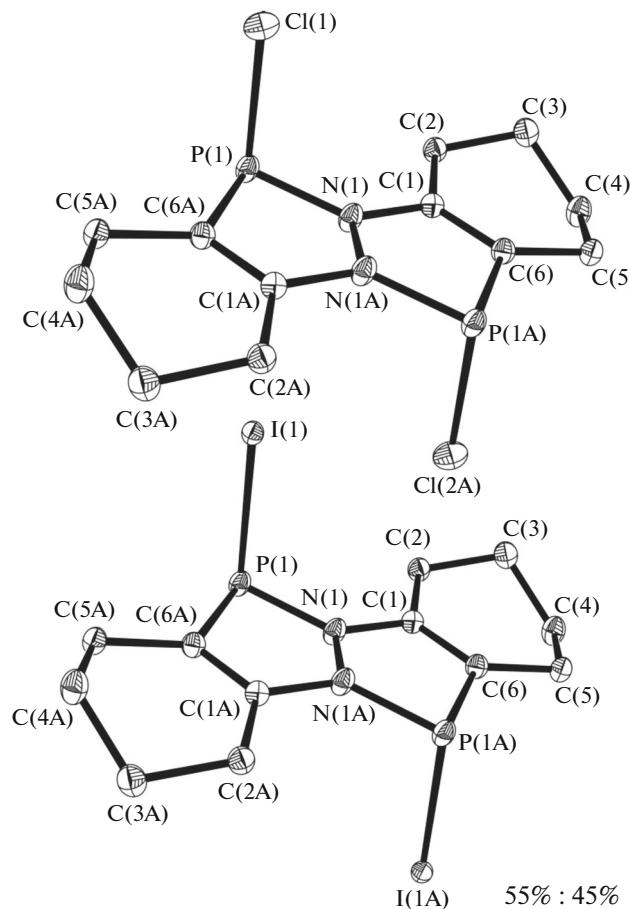


Fig. 1. Molecular structure of compound **III**. The halogen atoms (Cl and I) are disordered over two positions with a population ratio of 0.55 : 0.45, respectively. Hydrogen atoms are omitted for clarity.

Table 2. Selected bond lengths (*d*) and angles (ω) in compounds **III–V**

Bond	<i>d</i> , Å	Angle	ω , deg
III			
I(1)–P(1)	2.5215(5)	N(1)P(1)C(6) ^{#1}	87.87(6)
Cl(1)–P(1)	2.2461(8)	N(1)P(1)Cl(1)	103.28(5)
I(1')–P(1)	2.5169(9)	C(6) ^{#1} P(1)Cl(1)	99.68(5)
P(1)–N(1)	1.698(1)	N(1)P(1)I(1')	103.83(5)
P(1)–C(6) ^{#1}	1.796(1)	C(6) ^{#1} P(1)I(1')	99.91(5)
N(1)–C(1)	1.390(2)	Cl(1)P(1)I(1')	0.60(4)
N(1)–N(1) ^{#1}	1.408(2)	N(1)P(1)I(1)	104.55(5)
C(1)–C(6)	1.348(2)	C(6) ^{#1} P(1)I(1)	99.88(5)
C(1)–C(2)	1.498(2)	Cl(1)P(1)I(1)	1.29(2)
C(2)–C(3)	1.531(2)	I(1')P(1)I(1)	0.72(3)
C(6)–P(1) ^{#1}	1.796(1)	C(1)N(1)N(1) ^{#1}	109.4(1)
IV			
Cl(1)–P(1)	2.195(2)	I(2)I(3)I(4)	176.83(1)
I(1)–P(1)	2.218(2)	N(2)P(1)C(1)	87.2(2)
I(2)–I(3)	2.8221(3)	N(2)P(1)Cl(1)	98.6(1)
I(3)–I(4)	3.0695(3)	C(1)P(1)Cl(1)	97.6(1)
I(1S)–I(1S) ^{#1}	2.7839(5)	N(2)P(1)I(1)	106.3(1)
P(1)–N(2)	1.760(4)	C(1)P(1)I(1)	100.7(2)
P(1)–C(1)	1.828(4)	Cl(1)P(1)I(1)	8.26(6)
P(2)–N(1)	1.658(3)	N(1)P(2)C(12)	89.5(2)
P(2)–C(12)	1.745(4)	N(2)N(1)C(6)	110.9(3)
N(1)–N(2)	1.368(5)	N(2)N(1)P(2)	115.8(2)
N(1)–C(6)	1.443(5)	C(6)N(1)P(2)	133.3(3)
N(2)–C(7)	1.341(5)	C(7)N(2)N(1)	111.0(3)
C(1)–C(6)	1.319(6)	C(7)N(2)P(1)	134.1(3)
C(1)–C(2)	1.485(6)	N(1)N(2)P(1)	114.8(2)
C(2)–C(3)	1.517(6)	C(6)C(1)C(2)	122.5(4)
C(7)–C(12)	1.399(6)	C(6)C(1)P(1)	112.7(3)
C(7)–C(8)	1.514(6)	C(2)C(1)P(1)	124.8(3)
V			
I(1)–P(1)	2.570(1)	N(1)P(1)C(1)	88.1(2)
P(1)–N(1)	1.696(3)	N(1)P(1)I(1)	103.9(1)
P(1)–C(1)	1.797(4)	C(1)P(1)I(1)	99.6 (1)
N(1)–C(2) ^{#1}	1.392(4)	C(2) ^{#1} N(1)N(1) ^{#1}	109.2(3)
N(1)–N(1) ^{#1}	1.406(6)	C(2) ^{#1} N(1)P(1)	134.2(3)
C(1)–C(2)	1.342(5)	N(1) ^{#1} N(1)P(1)	114.9(3)
C(1)–C(6)	1.507(5)	C(2)C(1)C(6)	123.4(3)
C(2)–N(1) ^{#1}	1.392(4)	C(2)C(1)P(1)	111.5(2)
C(2)–C(3)	1.502(5)	C(6)C(1)P(1)	125.0(3)
C(3)–C(4)	1.516(6)	C(1)C(2)N(1) ^{#1}	114.9(3)
C(4)–C(5)	1.442(9)	C(1)C(2)C(3)	125.4(3)
C(5)–C(6)	1.507(7)	N(1) ^{#1} C(2)C(3)	119.7(3)

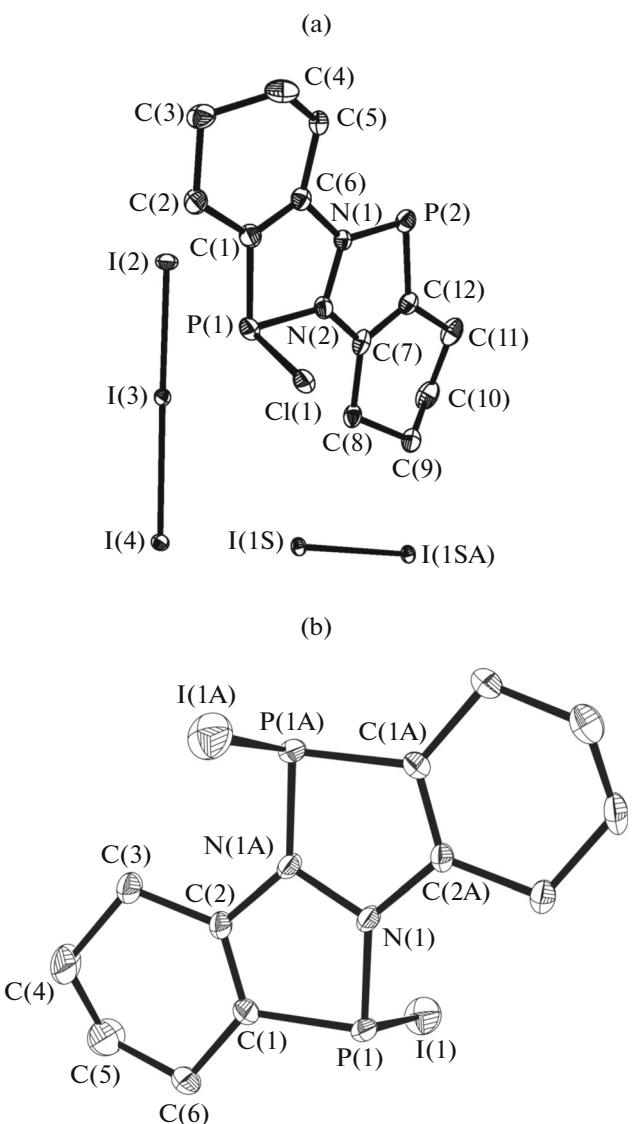


Fig. 2. Molecular structures of compound (a) **IV** and (b) **V**. Thermal ellipsoids are presented with 30% probability. Hydrogen atoms are omitted for clarity.

The geometry of compound **V** resembles that of the starting compound **II** [8]. A molecule of compound **V** is centrosymmetric (the center is situated at the middle of the nitrogen–nitrogen bond). The conformation of the five-membered heterocycles in compound **V** is close to planar: the average deviation of the atoms from the plane is 0.04 Å. The sum of the angles at the nitrogen atoms is 358.4°. The C–C bond lengths in the heterocycles (1.342(5) Å) correspond to the C(sp^2)–C(sp^2) multiple bonds. The phosphorus atom has a pyramidal geometry with a sum of bond angles of 291.6°. The P···P distances between the adjacent molecules in the crystal structure of compound **V** (3.364 Å) (Fig. 4) are considerably shorter than the doubled van der Waals radius of the phosphorus atoms (3.8 Å) [16]. Short intermolecular contacts are well

known for pnictogen compounds [17–19]. A model possible for nonvalence P···P interactions is the conjugation of the unpaired electron pair of the phosphorus atom with the antibonding orbital $\sigma^*(P-N)$ of the adjacent molecule [17]. This intermolecular P···P distance is 3.199(1) and 3.194(1) Å for the related molecules of DDP dichloride and dibromide, respectively [7, 8].

Thus, the conducted studies show that the chlorine exchange in 1,4-dichloro-3a,6a-diaza-1,4-diphosphaphentalene (**II**) by iodine under the action of MeI occurs quantitatively to form diiodide **V**, whereas this exchange with molecular iodine is incomplete. The crystal structures of products **III** and **IV** show disordering of the halogen atoms (iodine and remained chlorine). A remarkable feature of the packing of com-

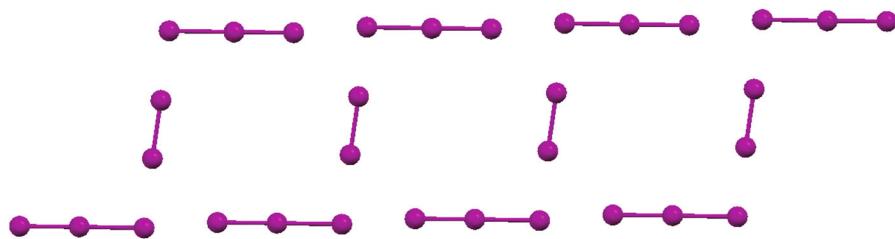


Fig. 3. Network of the ladder type of the alternating fragments I_2 and I_3^- in the crystal structure of compound **IV**.

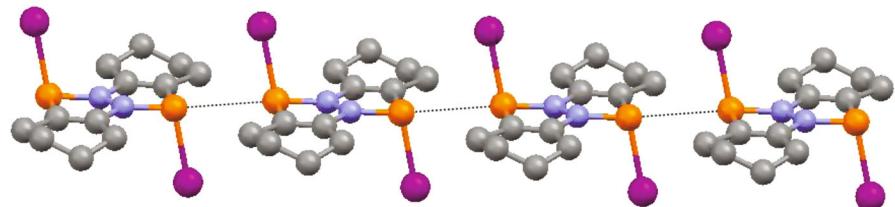


Fig. 4. Nonvalence interaction $P \cdots P$ between the adjacent molecules in the crystal structure of compound **V**.

ound **IV** is the polyanionic network of the ladder type consisting of the alternating I_2 and I_3^- fragments. Short intermolecular contacts $P \cdots P$ were found for the diiodo derivative of DDP (**V**) due to which the molecules in crystal form one-dimensional chains.

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