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# Copper(I) Complexes with Phenylphosphonous Acid Diamide

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**Abstract**—Copper(I) halide complexes with phenylphosphonous diamide ligand (L), $C_{56}H_{100}Br_4Cu_4N_8P_4$ (**II**) and $C_{56}H_{100}Cu_4I_4N_8P_4$ (**III**), were synthesized and studied by X-ray diffraction to determine their molecular and crystal structures (CIF files CCDC nos. 1426622 (**II**), 1426623 (**III**)). The prepared coordination compounds are cubane-like tetramers like $L_4Cu_4X_4$. During the reaction, copper(I) is coordinated to the phosphorus atom of ligand L. The stability of the complexes depends considerably on the halogen atom. The iodide complexes are most stable during storage.

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## INTRODUCTION

Monovalent copper halide complexes with phosphorous acid esters were obtained for the first time by A.E. Arbuzov et al. [1]. Copper(I) halide salts show high complexing ability towards ligands containing a trivalent phosphorus atom and form stable coordination compounds $CuX \cdot P(OR)_3$.

Copper(I) complexes with chemically different P(III)-organic ligands containing P–O [1–3], P–C [4, 5], or P–N [6–8] bonds have now been synthesized. These coordination compounds have found use for identification of phosphites and phosphines, in fine organic synthesis, e.g., in hydrogenation [9, 10], cyclopropanation [11, 12], and hydrogen transfer [12] reactions. The coordination of Cu(I) halides to the synthesized bi- and oligodentate arenephenylphosphamide ligands has been studied [12]. However, according to the literature, Cu(I) complexes with simple tetraethylphenylphosphonous diamide, $Ph{-}P(NEt_2)_2$, have been barely studied. Furthermore, one paper [13] briefly reports that the reaction of copper monoiodide with $Ph{-}P(NEt_2)_2$ gives, in the authors' opinion, a mixture of products, $\{[PhP(NEt_2)_2]CuI\}_4$ and $[PhP(NEt_2)_2]_2CuI$, which has not been investigated (in particular, for the crystal structure). Therefore, we prepared these metal complexes and proved their structures by X-ray diffraction.

## EXPERIMENTAL

Copper(I) halides used in this study were washed with a dilute 18% aqueous HCl solution and then with anhydrous diethyl ether and dried in vacuum (1 mm Hg) at 50°C for 2 h.

**Synthesis of tetraethylphosphonous diamide (L)** [14]. Dichlorophenylphosphine (18.0 g, 0.1 mol) in 20 mL of anhydrous benzene was added with stirring to a solution of diethylamine (29.3 g, 0.4 mol) in 30 mL of anhydrous benzene at a temperature of 5°C. The mixture was stirred for 2 h at room temperature. Diethylamine hydrochloride was filtered off and the benzene solution was concentrated. The residue was distilled in vacuum. The yield of the transparent colorless mobile liquid was 21.1 g (84%), bp = 115–118°C (1.5 mm Hg).

$^{31}$P NMR (benzene; δ, ppm): 98.08; $^{1}$H NMR ($CDCl_3$; δ, ppm): 1.18 (t, 12H, $CH_3$), 3.16 (m, 8H, $CH_2$), 7.51 (m, 2H, *o*-CH), 7.40 (m, 2H, *m*-CH), 7.29 (m, 1H, *p*-CH). Literature data: bp = 92–94°C (1 mm Hg), $^{31}$P NMR (δ, ppm): 98 [15].

**Synthesis of chloro(tetraethyldiamidophenylphosphonito)copper(I) (I)**. Copper(I) chloride (0.38 g, 3.8 mmol) was added with stirring to ligand L (0.95 g, 3.8 mmol) in 20 mL of absolute benzene. The mixture was stirred for 2 h at room temperature and the solvent was removed in vacuum. The resulting semicrystalline product was dissolved in hexane with heating. The solution was filtered and hexane was evaporated to give white crystals. The yield of **I** was 1.03 g (77%). mp = 97–98°C. $R_f$ 0.43 (B).

$^{31}$P NMR (benzene; δ, ppm): 86.26; $^{1}$H NMR ($CDCl_3$; δ, ppm): 1.15 (t, 12H, $CH_3$), 3.10 (m, 8H,

$CH_2$), 7.78 (m, 2H, *o*-CH), 7.49 (m, 2H, *m*-CH), 7.41 (m, 1H, *p*-CH).

For $C_{56}H_{100}N_8P_4Cl_4Cu_4$

| | | |
|---|---|---|
| anal. calcd., %: | P, 8.82; | Cu 18.09. |
| Found, %: | P, 8.84; | Cu, 18.10. |

**Synthesis of bromo(tetraethyldiamidophenylphosphonito)copper(I) (II)**. Copper(I) bromide (0.55 g, 3.8 mmol) was added with stirring to ligand L (0.95 g, 3.8 mmol) in 20 mL of absolute benzene. The mixture was stirred for 3 h at room temperature and the solvent was removed in vacuum. The resulting semicrystalline product was dissolved in hexane with heating. The solution was filtered and hexane was evaporated to give transparent white crystals. The yield of **II** was 1.02 g (68%). mp = 136–138°C. $R_f$ 0.58 (A) and 0.27 (B).

$^{31}P$ NMR (benzene; δ, ppm): 84.77; $^1H$ NMR ($CDCl_3$; δ, ppm): 1.13 (t, 12H, $CH_3$), 3.10 (m, 8H, $CH_2$), 7.79 (m, 2H, *o*-CH), 7.51 (m, 2H, *m*-CH), 7.38 (m, 1H, *p*-CH).

For $C_{56}H_{100}N_8P_4Br_4Cu_4$

| | | |
|---|---|---|
| anal. calcd., %: | P, 7.82; | Cu, 16.05. |
| Found, %: | P, 7.82; | Cu, 16.14. |

**Synthesis of iodo(tetraethyldiamidophenylphosphonito)copper(I) (III)**. Copper(I) iodide (0.36 g, 1.9 mmol) was added with stirring to ligand L (0.48 g, 1.9 mmol) in 15 mL of absolute benzene. The mixture was stirred for 3 h at room temperature and the solvent was removed in vacuum. The resulting semicrystalline product was dissolved in hexane with heating. The solution was filtered and hexane was evaporated to give white crystals. The yield of **III** was 0.66 g (79%). mp = 185–187°C. $R_f$ 0.22 (B).

$^{31}P$ NMR (benzene; δ, ppm): 78.15; $^1H$ NMR ($CDCl_3$; δ, ppm): 1.10 (t, 12H, $CH_3$), 3.13 (m, 8H, $CH_2$), 7.80 (m, 2H, *o*-CH), 7.51 (m, 2H, *m*-CH), 7.36 (m, 1H, *p*-CH).

For $C_{56}H_{100}N_8P_4I_4Cu_4$

| | | |
|---|---|---|
| anal. calcd., %: | P, 7.00; | Cu, 14.35. |
| Found, %: | P, 6.81; | Cu, 14.23. |

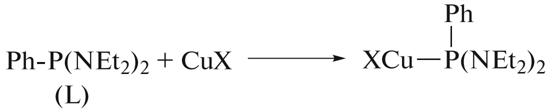
$^{31}P$ NMR spectra were recorded in benzene on a Bruker AV 300 spectrometer operating at 121.49 MHz and referred to 85% phosphoric acid; $^1H$NMR spectra were recorded in $CDCl_3$ on a Bruker AV 400 instrument (400 MHz) and referred to TMS. Mass spectra were run on a Ultraflex TOF/TOF (Bruker Daltonics) using a nitrogen laser (λ = 337) in the positive ion mode. TLC analysis was carried out on Silufol plates with benzene (A) and 3 : 1 hexane–dioxane (B) solvent systems. The spots were visualized by UV irradiation or by calcination of the plates at 200–250°C.

**X-ray diffraction** study of compounds **II** and **III** was carried out on an Apex DUO automated diffractometer (graphite monochromator, $\lambda(\mathrm{Mo}K_\alpha)$ = 0.71073 Å, ω-scan mode). The empirical absorption and systematic error corrections were applied using the SADABS software. The structures were solved by the direct method and refined by the full-matrix least squares method on $F^2_{hkl}$ with anisotropic thermal parameters for all hydrogen atoms. Hydrogen atom positions were calculated geometrically and refined in the riding model. The structures of **II** and **III** were solved and refined using the SHELX software, version 2009-9.13 [16], and the molecular geometry was analyzed using the PLATON program [17]. The principal crystallographic data and structure refinement details for **II** and **III** are summarized in Table 1, while the bond lengths and bond angles are in Table 2.

The structures of **II** and **III** are deposited with the Cambridge Crystallographic Data Centre (CCDC) (nos. 1426622 (**II**) and 1426623 (**III**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

## RESULTS AND DISCUSSION

The reaction of monovalent copper halides (CuX) with the ligand L was conducted, according to [6], in absolute benzene at room temperature according to the equation:



$$\underset{\text{(L)}}{\text{Ph-P(NEt}_2)_2} + \text{CuX} \longrightarrow \text{XCu}-\overset{\text{Ph}}{\overset{|}{\text{P}}}(\text{NEt}_2)_2$$

X = Cl (**I**), Br (**II**), I (**III**)

The pure complexes **I**–**III** obtained for the first time were isolated upon recrystallization as white crystalline solids. Complexes **I** and **II** are unstable on storage in air and gradually decompose to give mainly oxidation products. Complex **I** is less stable than **II** and after 12 h, it decomposes with color change to green and then to black. The $^{31}P$ NMR spectrum of **I** after a 12 h storage shows signals of decomposition products at 25–28 ppm. Compound **II** is relatively more stable but after 7–10 days, it also decomposes to give a green-brown pulp whose $^{31}P$ NMR spectrum exhibits signals in the same range (25–26 ppm). The composition of iodine compound **III** remains unchanged for a month. Hence, the complexes XCu–$PPh(NEt_2)_2$ differ considerably in the stability depending on the halogen atom X, the stability decreasing in the series X = I > Br > Cl.

The formation of complexes **I**–**III** is confirmed by the absence of the $^{31}P$ NMR signal at 98.00 ppm,

**Table 1.** Principal crystallographic data and structure refinement parameters for **II** and **III**

| Parameter | Value | |
|---|---|---|
| | **II** | **III** |
| $M$ | 1583.11 | 1771.07 |
| $T$, K | 100(2) | 100(2) |
| System | Triclinic | Triclinic |
| Space group | $P\bar{1}$ | $P\bar{1}$ |
| $Z/Z'$ | 2/1 | 2/1 |
| $a$, Å | 12.9755(5) | 13.0183(5) |
| $b$, Å | 13.4562(5) | 13.5169(5) |
| $c$, Å | 21.2524(8) | 21.3307(8) |
| α, deg | 86.4020(10) | 86.3503(8) |
| β, deg | 89.7150(10) | 89.8461(8) |
| γ, deg | 67.9000(10) | 68.0782(8) |
| $V$, Å$^3$ | 3430.6(2) | 3474.1(2) |
| ρ(calcd.), g cm$^{-3}$ | 1.533 | 1.693 |
| μ, cm$^{-1}$ | 36.86 | 31.17 |
| $F$(000) | 1616 | 1760 |
| $2\theta_{max}$, deg | 58 | 58 |
| Number of measured reflections | 43180 | 43723 |
| Number of unique reflections | 18246 | 18483 |
| Number of reflections with $I > 2\sigma(I)$ | 11645 | 13993 |
| Number of refined parameters | 701 | 701 |
| $R_1$ | 0.0487 | 0.0394 |
| $wR_2$ | 0.0844 | 0.0814 |
| GOOF | 0.955 | 0.990 |
| $\Delta\rho_{max}/\Delta\rho_{min}$, $e$ Å$^{-3}$ | 0.814/–0.591 | 1.689/–0.721 |

which is characteristic of uncoordinated ligand L [15], and by the appearance of broadened signals at 86.26 (**I**), 84.77 (**II**), and 78.15 ppm (**III**), which is consistent with the coordination-induced upfield shifts ($-\Delta\delta_p$) of 11.82, 13.23, and 19.85 ppm for **I**–**III**, respectively. These $-\Delta\delta_p$ values are indicative of the formation of the Cu–P bond, i.e., the Cu(I) atom in complexes **I**–**III** is coordinated to phosphorus. The invariability of the ligand skeleton upon complex formation is confirmed by $^1$H NMR spectra, which show the signals for all hydrogen atoms at theoretical ligand intensity ratio.

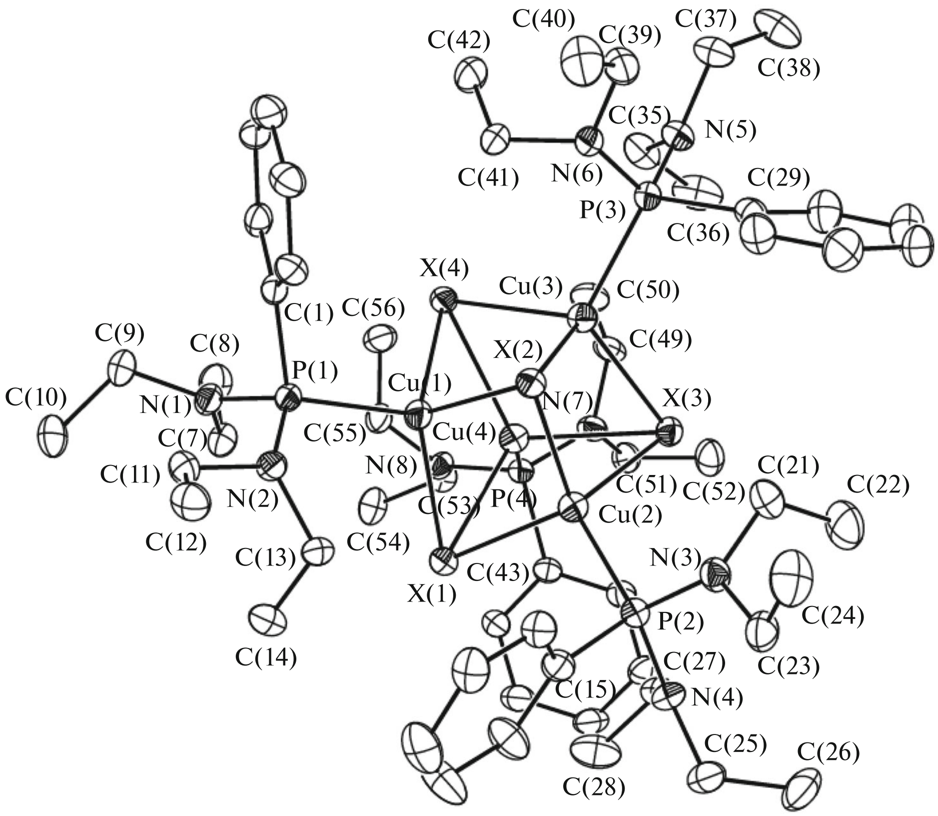
Metal complexes **II** and **III** were also studied by mass spectrometry to confirm the molecular masses. The MALDI mass spectrometry data indicate that these complexes are unstable and degrade during the investigation. The mass spectra exhibit masses for the degradation products: $[Cu_2Br_2 + L + H]^+ = 540.41$ (**II**), $[Cu + L + H]^+ = 316.02$ (**III**), which was shown previously for similar acyclic systems [18].

The structures of complexes **II** and **III** were studied by X-ray diffraction. The crystals of **II** and **III** were shown to be isostructural. They are cubane-shaped tetramers of the $L_4Cu_4X_4$ type, where L is the acyclic phenylphosphorous amide ligand containing P–C and P–N bonds, X = Br or I (figure).

The Cu and halogen X atoms form a highly distorted cube, the degree of distortion being dependent on the type of halogen atom (figure, Tables 1 and 2). The $Cu_4X_4$ cubane core can also be described as a combination of two interpenetrating tetrahedra with copper and halogen atoms at the vertices. The root-

**Table 2.** Bond lengths (*d*, Å) and bond angles (ω, deg) in complexes **II** and **II**

| Bond | *d*, Å | | Bond | *d*, Å | |
|---|---|---|---|---|---|
| | **II** | **III** | | **II** | **III** |
| Cu(1)–P(1) | 2.2081(11) | 2.2574(11) | Cu(4)–X(4) | 2.5094(7) | 2.6605(6) |
| Cu(1)–X(2) | 2.5269(7) | 2.6899(6) | Cu(4)–X(3) | 2.5716(7) | 2.7151(6) |
| Cu(1)–X(1) | 2.5556(7) | 2.6950(6) | P(1)–N(1) | 1.680(3) | 1.678(4) |
| Cu(1)–X(4) | 2.5315(6) | 2.6989(5) | P(1)–N(2) | 1.683(3) | 1.691(3) |
| Cu(2)–P(2) | 2.2081(12) | 2.2577(11) | P(1)–C(1) | 1.830(4) | 1.826(4) |
| Cu(2)–X(1) | 2.5096(7) | 2.6830(6) | P(2)–N(4) | 1.672(3) | 1.673(3) |
| Cu(2)–X(2) | 2.5372(7) | 2.6868(6) | P(2)–N(3) | 1.677(3) | 1.685(3) |
| Cu(2)–X(3) | 2.6038(7) | 2.7258(6) | P(2)–C(15) | 1.837(4) | 1.834(4) |
| Cu(3)–P(3) | 2.2071(12) | 2.2616(11) | P(3)–N(5) | 1.682(3) | 1.688(3) |
| Cu(3)–X(3) | 2.4559(7) | 2.6393(6) | P(3)–N(6) | 1.685(3) | 1.681(3) |
| Cu(3)–X(2) | 2.6051(7) | 2.7294(6) | P(3)–C(29) | 1.819(4) | 1.826(4) |
| Cu(3)–X(4) | 2.6170(7) | 2.7199(6) | P(4)–N(7) | 1.675(3) | 1.678(3) |
| Cu(4)–P(4) | 2.1989(12) | 2.2529(12) | P(4)–N(8) | 1.686(3) | 1.682(3) |
| Cu(4)–X(1) | 2.5033(7) | 2.6683(6) | P(4)–C(43) | 1.823(4) | 1.825(4) |
| Angle | ω, deg | | Angle | ω, deg | |
| | **II** | **III** | | **II** | **III** |
| P(1)Cu(1)X(2) | 108.62(4) | 102.77(3) | N(2)P(1)C(1) | 102.38(17) | 102.26(17) |
| P(1)Cu(1)X(1) | 117.40(4) | 115.62(3) | N(1)P(1)Cu(1) | 114.88(12) | 115.28(12) |
| X(2)Cu(1)X(1) | 104.91(2) | 112.855(19) | N(2)P(1)Cu(1) | 111.92(12) | 111.55(12) |
| P(1)Cu(1)X(4) | 120.59(4) | 110.94(3) | C(1)P(1)Cu(1) | 111.52(13) | 111.90(13) |
| X(2)Cu(1)X(4) | 103.02(2) | 107.083(18) | N(4)P(2)N(3) | 112.96(18) | 112.68(18) |
| X(4)Cu(1)X(1) | 100.42(2) | 107.295(19) | N(4)P(2)C(15) | 104.30(18) | 102.69(18) |
| P(2)Cu(2)X(1) | 121.80(4) | 112.64(3) | N(3)P(2)C(15) | 99.92(18) | 100.57(18) |
| P(2)Cu(2)X(2) | 108.95(4) | 102.79(3) | N(4)P(2)Cu(2) | 113.14(13) | 114.56(13) |
| X(1)Cu(2)X(2) | 105.97(2) | 113.34(2) | N(3)P(2)Cu(2) | 111.81(13) | 112.39(12) |
| P(2)Cu(2)X(3) | 119.49(4) | 117.29(3) | C(15)P(2)Cu(2) | 113.80(14) | 112.69(14) |
| X(1)Cu(2)X(3) | 98.67(2) | 104.736(19) | N(5)P(3)N(6) | 113.24(17) | 112.70(17) |
| X(2)Cu(2)X(3) | 99.02(2) | 106.138(18) | N(5)P(3)C(29) | 100.52(17) | 101.04(18) |
| P(3)Cu(3)X(3) | 128.62(4) | 118.53(3) | N(6)P(3)C(29) | 102.86(18) | 102.48(18) |
| P(3)Cu(3)X(2) | 104.30(4) | 101.35(3) | N(6)P(3)Cu(3) | 110.19(12) | 109.62(12) |
| X(3)Cu(3)X(2) | 101.13(2) | 107.384(19) | N(5)P(3)Cu(3) | 115.95(12) | 117.15(13) |
| P(3)Cu(3)X(4) | 118.67(4) | 115.67(3) | C(29)P(3)Cu(3) | 112.99(13) | 112.64(13) |
| X(3)Cu(3)X(4) | 100.34(2) | 107.268(19) | N(7)P(4)N(8) | 112.21(17) | 112.05(17) |
| X(2)Cu(3)X(4) | 98.61(2) | 105.378(18) | N(7)P(4)C(43) | 100.14(17) | 100.48(18) |
| P(4)Cu(4)X(1) | 115.79(4) | 108.49(3) | N(8)P(4)C(43) | 102.91(17) | 102.43(18) |
| P(4)Cu(4)X(4) | 124.98(4) | 119.31(3) | N(7)P(4)Cu(4) | 112.65(12) | 112.81(13) |
| X(1)Cu(4)X(4) | 102.48(2) | 109.218(19) | N(8)P(4)Cu(4) | 115.83(12) | 116.72(12) |
| P(4)Cu(4)X(3) | 109.96(3) | 106.71(3) | C(43)P(4)Cu(4) | 111.52(13) | 110.54(13) |
| X(1)Cu(4)X(3) | 99.69(2) | 105.441(19) | C(9)N(1)P(1) | 125.4(3) | 124.6(3) |
| X(4)Cu(4)X(3) | 100.16(2) | 106.800(19) | C(7)N(1)P(1) | 118.8(3) | 119.4(3) |
| Cu(4)X(1)Cu(2) | 80.71(2) | 70.927(17) | C(11)N(2)P(1) | 121.8(3) | 121.3(3) |
| Cu(4)X(1)Cu(1) | 76.85(2) | 67.863(16) | C(13)N(2)P(1) | 116.2(3) | 116.2(3) |
| Cu(2)X(1)Cu(1) | 73.80(2) | 64.302(16) | C(23)N(3)P(2) | 123.1(3) | 123.2(3) |
| Cu(1)X(2)Cu(2) | 73.82(2) | 64.321(16) | C(21)N(3)P(2) | 117.8(3) | 116.5(3) |
| Cu(1)X(2)Cu(3) | 77.59(2) | 69.029(16) | C(25)N(4)P(2) | 124.5(3) | 123.6(3) |
| Cu(2)X(2)Cu(3) | 77.88(2) | 69.175(16) | C(27)N(4)P(2) | 120.0(3) | 120.7(3) |
| Cu(3)X(3)Cu(4) | 79.86(2) | 70.579(17) | C(35)N(5)P(3) | 118.8(3) | 119.3(3) |
| Cu(3)X(3)Cu(2) | 79.35(2) | 69.913(16) | C(37)N(5)P(3) | 121.6(3) | 120.6(3) |
| Cu(4)X(3)Cu(2) | 77.69(2) | 69.589(16) | C(39)N(6)P(3) | 124.1(3) | 124.9(3) |
| Cu(4)X(4)Cu(1) | 77.18(2) | 67.918(16) | C(41)N(6)P(3) | 115.6(3) | 115.4(3) |
| Cu(4)X(4)Cu(3) | 78.03(2) | 70.195(17) | C(51)N(7)P(4) | 124.7(3) | 124.8(3) |
| Cu(1)X(4)Cu(3) | 77.29(2) | 69.041(16) | C(49)N(7)P(4) | 118.4(3) | 118.3(3) |
| N(1)P(1)N(2) | 112.79(17) | 113.20(17) | C(55)N(8)P(4) | 117.1(3) | 118.2(3) |
| N(1)P(1)C(1) | 102.21(17) | 101.42(17) | C(53)N(8)P(4) | 120.2(3) | 120.7(3) |



General view of isotructural complexes **II** (X = Br) and **III** (X = I) with non-hydrogen atoms being represented as probability atomic displacement ellipsoids.

mean-square deviation of the core from $T_d$ local symmetry is 0.076 and 0.078 Å for complexes **II** and **III**, respectively. Copper(I) complexes with this type of core geometry have been reported in the literature, e.g., complexes with phosphorous amides [6] and with phosphine ligands such as $PPh_3$ [19].

The geometric parameters of the cubane core in **II** and **III** are typical of this type of complexes [20]. The Cu–Br bond lengths vary in the range of 2.4559(7)–2.6170(7) Å (average, 2.5(1) Å, according to analysis of the CCDC data), while Cu–I bond lengths are in the 2.6393(6)–2.7294(6) Å range (average, 2.69(5) Å). The intramolecular distances between the halogen and copper atoms in both structures (Br···Br 3.8790(1)–4.0299(1), Cu···Cu 3.0415(1)–3.2461(1) Å in **II**; and I···I 4.2836(2)–4.4866(1), Cu···Cu 2.8620(1)–3.1048(1) Å in **III**) are longer than twice the van der Waals radii of the elements (Br 1.85, I 1.98 and Cu 1.4 Å) and apparently these are not binding contacts. The P–C, 1.819(4)–1.837(4) Å, and P–N, 1.672(3)–1.691(3) Å, bond lengths as well as the bond lengths and bond angles in the peripheral phenyl and diethylamide groups do not differ, to within the determination error, from the average values for similar compounds present in the CCDC. The nitrogen atoms are slightly pyramidalized; the sums of angles at the N(2) atom are 352.7(9)° and 352.2(9)° for **II** and **III**, respectively.

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