

# Synthesis and properties of 10-hydroxy-10-oxo-10*H*-10 $\lambda^5$ -phenoxaphosphine-2,8-dicarboxylic acid and related polybenzimidazoles

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Oxidation of 10-hydroxy-2,8-dimethyl-10*H*-10 $\lambda^5$ -phenoxaphosphine 10-oxide (**1**) with potassium permanganate in an alkaline medium afforded 10-hydroxy-10-oxo-10*H*-10 $\lambda^5$ -phenoxaphosphine-2,8-dicarboxylic acid (**2**). The latter exists as a stable crystal hydrate containing two water molecules. With the aim of examining the possibility of performing the synthesis of polybenzazoles based on acid **2**, the model reaction of the latter with *o*-phenylenediamine in polyphosphoric acid (PPA) was studied. New high-molecular-weight phosphorus-containing polybenzimidazoles were prepared by the reaction of **2** with various aromatic tetraamines in PPA and Eaton's reagent.

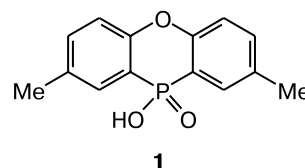
**Key words:** 10-hydroxy-2,8-dimethyl-10*H*-10 $\lambda^5$ -phenoxaphosphine 10-oxide, oxidation with potassium permanganate, 10-hydroxy-10-oxo-10*H*-10 $\lambda^5$ -phenoxaphosphine-2,8-dicarboxylic acid, polycondensation, *o*-phenylenediamine, polyphosphoric acid, Eaton's reagent, aromatic tetraamines, 1,2-diamino-4-(3,4-diaminophenoxy)-benzene, 1,2-diamino-(3,4-diaminophenylsulfonyl)benzene, phosphorus-containing polybenzimidazoles.

Polyheteroarylenes have attracted considerable attention as thermally stable and heat-, fire-, and chemically resistant systems, matrices for immobilization of catalysts, solid electrolytes for proton-conducting membranes,<sup>1–3</sup> etc. For example, polyheteroarylene-based film materials containing ionogenic acid groups (–SO<sub>3</sub>H, –COOH) were successfully tested in fuel elements. The chemical structures of such polymeric electrolytes can easily be modified as desired. These polymers are characterized by high moisture absorption and rather high proton conductivity at temperatures below 120 °C and at high humidity. In addition, they have satisfactory thermal stability and are chemically resistant under these conditions. Further progress in this field is expected to take place with the development of procedures for the synthesis of polymers possessing proton-conducting properties at temperatures higher than 120 °C under anhydrous conditions. Polymers containing phosphoryl groups can fulfill this criterion.<sup>1</sup>

Polybenzimidazoles (PBI) belong to a well studied class of polyheteroarylenes, which are used in fuel elements as polymer electrolyte complexes with orthophosphoric acid.<sup>4–7</sup> However, it appeared that H<sub>3</sub>PO<sub>4</sub> migrates from the membrane to the cathode compartment of a fuel cell during operation of the fuel element, resulting in a decrease in the performance of the latter. Hence, the development of procedures for the synthesis of PBI, in which ionogenic phosphorus-containing acid groups are

chemically bound to the polymer chain, is an urgent problem. The involvement of the phosphorus atom in the main polymer chain through P–C bonds is optimal for providing high chemical and thermal stability of such polymers.<sup>8</sup> The system can additionally be stabilized if the phosphorus atom is involved also in an aromatic fused heterocycle.

10-Hydroxy-2,8-dimethyl-10*H*-10 $\lambda^5$ -phenoxaphosphine 10-oxide (**1**) is a very interesting phosphorus-containing heterocyclic fused system, which is characterized by high thermal stability and is potentially suitable for the construction of PBI with hydroxyphosphoryl fragments.<sup>9</sup>



It seems most reasonable to use compound **1** as an intermediate for the preparation of the corresponding dicarboxylic acid. Oxidation of **1** with an excess of KMnO<sub>4</sub> in an aqueous KOH solution afforded 10-hydroxy-10-oxo-10*H*-10 $\lambda^5$ -phenoxaphosphine-2,8-dicarboxylic acid **2** (Scheme 1) in 46% yield.<sup>10</sup>

Acid **2** is a white crystalline powder, which does not melt up to 300 °C. The structure of **2** was confirmed

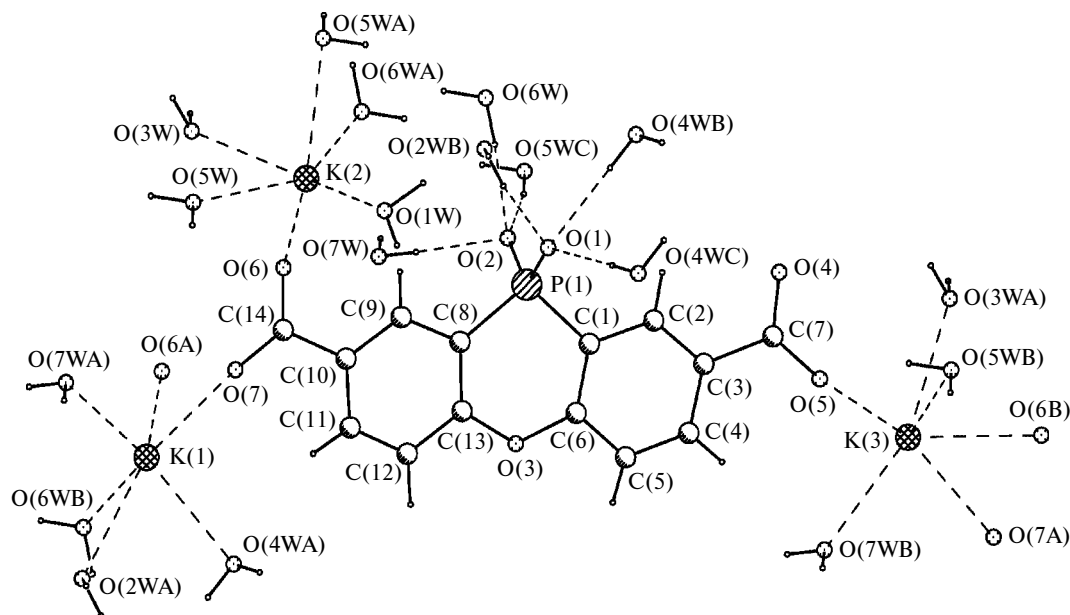
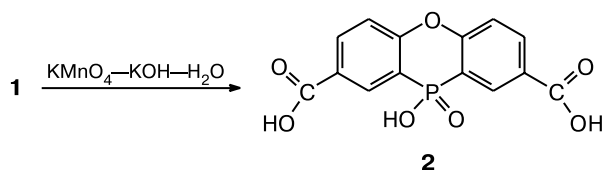


Fig. 1. Overall view of the potassium salt of **2** in the crystal. The coordination bonds and hydrogen bonds are indicated by dashed lines.

Scheme 1



by  $^1\text{H}$  and  $^{31}\text{P}\{^1\text{H}\}$  NMR spectroscopy and the results of X-ray diffraction study of its potassium salt (Fig. 1).

X-ray study demonstrated that the potassium salt of **2** crystallizes as a solvate containing seven water molecules (see Fig. 1).

The main bond lengths in **2** (Table 1) are similar to those in 10-[(2,8-dimethyl-10-oxo-10*H*-10 $\lambda^5$ -phenoxaphosphin-10-yl)oxy]-2,8-dimethyl-10*H*-10 $\lambda^5$ -phenoxaphosphine 10-oxide studied earlier.<sup>10</sup> The most substantial differences are observed in the central phosphorus-containing ring, which, unlike that in the compound studied earlier, is nonplanar with the P(1) atom deviating by 0.19 Å from the plane of the fused heterocyclic system.

The potassium cations are coordinated by water molecules and carboxy groups, whereas the  $\text{PO}_2^-$  group is involved only in hydrogen bonding with water molecules ( $\text{O}\cdots\text{O}$ , 2.754(2)–2.848(2) Å), resulting in equalization of the P–O bond lengths. The coordination number of the potassium atoms is 6. The number of water molecules involved in the coordination polyhedron varies from 4 (K(1) and K(2)) to 5 (K(3)). The K...O distances are in a range of 2.580(1)–3.100(1) Å. The contacts between  $\text{K}^+$  and the carboxy groups are shorter than those between  $\text{K}^+$  and the water molecules.

The carboxy groups in the salt of **2** differ both in the angle, which they form with the aryl fragment (C(7)–O(4)–O(5) and C(14)–O(7)–O(6) are 16.7 and 1.4°, respectively) and in that the C(10)–C(14) bond is elongated (1.517(2) Å) compared to the C(3)–C(7) bond (1.507(2) Å). The observed differences are, apparently, attributable to the influence of coordination to the potassium cations, because the K...O contacts with the C(14)O(7)O(6) group are shorter (2.580(1)–2.902(1) Å) than the analogous contacts with the C(7)O(4)O(5) group (2.771(1) Å).

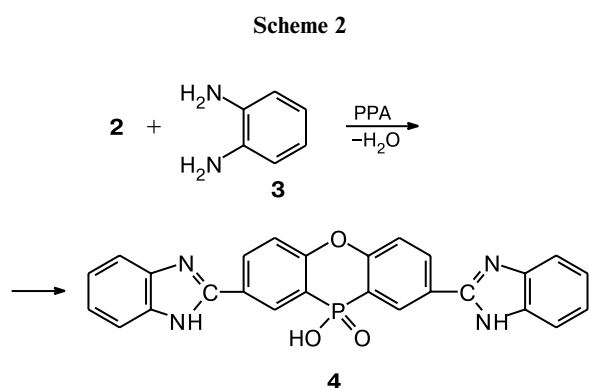
Table 1. Selected bond lengths ( $d$ ) and bonds angles ( $\omega$ ) in compound **2**

Bond	$d/\text{Å}$	Bond	$d/\text{Å}$	Angle	$\omega/\text{deg}$	Angle	$\omega/\text{deg}$
P(1)–O(1)	1.508(1)	O(4)–C(7)	1.257(2)	O(1)–P(1)–O(2)	114.53(8)	C(6)–O(3)–C(13)	123.38(13)
P(1)–O(2)	1.510(1)	O(5)–C(7)	1.258(2)	O(1)–P(1)–C(8)	111.47(8)	O(4)–C(7)–O(5)	125.02(17)
P(1)–C(8)	1.785(2)	O(6)–C(14)	1.257(2)	O(2)–P(1)–C(8)	108.40(8)	O(7)–C(14)–O(6)	125.25(16)
P(1)–C(1)	1.787(2)	O(7)–C(14)	1.246(2)	O(1)–P(1)–C(1)	109.92(8)		
O(3)–C(6)	1.373(2)	C(3)–C(7)	1.507(2)	O(2)–P(1)–C(1)	110.83(8)		
O(3)–C(13)	1.374(2)	C(10)–C(14)	1.516(2)	C(8)–P(1)–C(1)	100.84(8)		

The water molecules are linked to form dimers (O(2w)...O(6w)) and trimers O(1w)...O(3w)...O(4w)); the O...O distances are in a range of 2.807(2)–2.835(2) Å.

According to the results of elemental analysis, the resulting acid exists as a stable crystal hydrate containing two water molecules. The presence of these crystallization water molecules was also confirmed by investigation of polycondensation of **2**.

With the aim of examining the possibility of the formation of a polymer based on acid **2**, we carried out the model reaction of the latter with 1,2-diaminobenzene (**3**) in polyphosphoric acid (PPA). This reaction afforded 2,8-bis(1*H*-1,3-benzimidazol-2-yl)-10-hydroxy-10*H*-10λ<sup>5</sup>-phenoxaphosphine 10-oxide (**4**) (Scheme 2) in nearly quantitative yield.



The structure of compound **4** was confirmed by elemental analysis and <sup>1</sup>H and <sup>31</sup>P{<sup>1</sup>H} NMR spectroscopy.

Dicarboxylic acid **2** was used for the synthesis (Scheme 3) of polybenzimidazoles **7** and **8** based on 1,2-diamino-4-(3,4-diaminophenyl)sulfonylbenzene (**5**) and 1,2-diamino-4-(3,4-diaminophenoxy)benzene (**6**), respectively. These reactions can be performed both in a

**Table 2.** Data from dynamic thermogravimetric (TGA) analysis of polymers **7** and **8** in air (the rate of temperature rise was 5 K min<sup>-1</sup>)

Polymer	Solvent	<i>T</i> /°C*
<b>7</b>	PPA	500
	Eaton's reagent	525
<b>8</b>	PPA	475
	Eaton's reagent	450

\* The 5% weight-loss temperature in air.

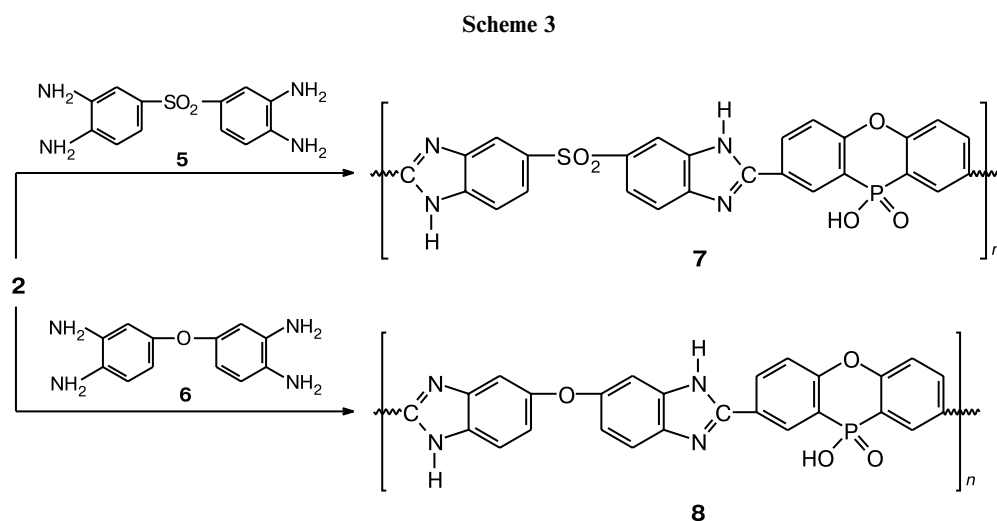
PPA medium and in Eaton's reagent (a solution of P<sub>2</sub>O<sub>5</sub> in methanesulfonic acid<sup>11</sup>).

According to the results of viscosimetry, polycondensation under the conditions used afforded relatively high-molecular-weight PBI. In concentrated sulfuric acid, the intrinsic viscosities [η] of polymers **7** and **8**, which were prepared in PPA, were 2.35 and 4.72 dL g<sup>-1</sup>, respectively (at 25 °C). Transparent, stable, and elastic films were cast from 5% solutions of polymers **7** and **8** in formic acid. Studies of the proton-conducting properties of these films are presently underway.

Under the conditions of dynamic thermogravimetric analysis (TGA) in air (the rate of temperature rise was 5 K min<sup>-1</sup>), the polymers based on **5** exhibit higher thermooxidative stability than the polymers based on **6**. The 5% weight-loss temperatures of polymer samples, which have similar molecular weights and were prepared according to different procedures (in a PPA medium and in Eaton's reagent), are somewhat different (Table 2).

Intensive oxidative thermal decomposition of phosphorus-containing PBI **7** and **8** in air starts at temperatures above 600 °C.

Therefore, the phosphorus-containing polymers synthesized in the present study are among the best known PBI in thermooxidative stability. However, it should be



noted that, unlike purely organic PBI,<sup>2–3</sup> phosphorus-containing PBI **7** and **8** retain up to 23% of the initial weight at 700–800 °C in air to form solid noncombustible coke at temperatures up to 1000 °C.

### Experimental

The <sup>1</sup>H and <sup>31</sup>P NMR spectra were recorded on a Bruker AMX-400 instrument (400.13 and 161.98 MHz, respectively) in DMSO-d<sub>6</sub>. The chemical shifts δ were calculated using the residual signals for the protons of the deuterated solvent as the internal standard (<sup>1</sup>H) and 85% H<sub>3</sub>PO<sub>4</sub> as the external standard (<sup>31</sup>P). The melting points were measured on a Boetius hot-stage apparatus and are uncorrected. The course of the reactions and the purity of the reaction products were monitored by TLC on Silufol UV-254 plates. Dynamic TGA was carried out on a MOM Q-1000 derivatograph. The rate of temperature rise was 5 K min<sup>-1</sup>. The weight of the sample was 40–60 mg. The reduced viscosities were measured on a Ubbelohde capillary viscosimeter at 25 °C. The intrinsic viscosities [η] were calculated by extrapolation of the linear dependence η<sub>int</sub>/c to the zero concentration. The starting concentration of the solutions of the polymers was 0.5 g dL<sup>-1</sup>. Before measurements, the solutions and solvents were kept under temperature-controlled conditions at a temperature of measurements for 1 h. Tetraamines **5** (m.p. 182–183 °C) and **6** (m.p. 150–151 °C) were prepared and purified according to known procedures.<sup>12</sup> 1,2-Phenylenediamine (**3**) (Aldrich, m.p. 101–103 °C) was used without additional purification. Eaton's reagent was prepared by dissolution of P<sub>2</sub>O<sub>5</sub> (1 g) in methanesulfonic acid (10 g) at room temperature.<sup>11</sup>

**10-Hydroxy-10-oxo-10H-10λ<sup>5</sup>-phenoxaphosphine-2,8-dicarboxylic acid (2).** 10-Hydroxy-2,8-dimethyl-10H-10λ<sup>5</sup>-phenoxaphosphine 10-oxide (**1**) (35.79 g, 0.138 mol) was dissolved in a hot (70–80 °C) 10% aqueous KOH solution (200 mL) with stirring using a magnetic stirrer. At this temperature, KMnO<sub>4</sub> (136 g, 1.5 mol) was added portionwise (~10 g) as the color of the solution disappeared until oxide **1** was completely oxidized. The hot mixture was filtered. The filtrate was cooled to ~20 °C and acidified with 1 M HCl to pH 2–3. The precipitate that formed was separated, washed with distilled water until the pH became neutral and the chloride ions completely disappeared, and dried in a vacuum desiccator at 100 °C for 10 h. The yield of acid **2** was 22.41 g (46%), m.p. (with decomp.) > 300 °C. Found (%): C, 47.12; H, 3.52; P, 8.34. C<sub>14</sub>H<sub>9</sub>O<sub>7</sub>P·2 H<sub>2</sub>O. Calculated (%): C, 47.20; H, 3.68; P, 8.69. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>), δ: 7.25 (dd, 2 H, H(4), H(6), <sup>3</sup>J<sub>H,H</sub> = 8.6 Hz, <sup>4</sup>J<sub>H,P</sub> = 5.0 Hz); 7.92 (dd, 2 H, H(3), H(7), <sup>3</sup>J<sub>H,H</sub> = 8.8 Hz, <sup>4</sup>J<sub>H,H</sub> = 2.0 Hz); 8.34 (dd, 2 H, H(1), H(9), <sup>3</sup>J<sub>H,P</sub> = 12.0 Hz, <sup>4</sup>J<sub>H,H</sub> = 2.0 Hz); 13.34 (br.s, 3 H, OH). <sup>31</sup>P{<sup>1</sup>H} NMR (DMSO-d<sub>6</sub>), δ: -10.82 s.

**Tripotassium salt 2** was prepared in quantitative yield by treating acid **2** with a calculated amount of a 1 M aqueous K<sub>2</sub>CO<sub>3</sub> solution, m.p. (with decomp.) > 300 °C (water-acetone). <sup>1</sup>H NMR (D<sub>2</sub>O), δ: 7.31 (dd, 2 H, H(4), H(6), <sup>3</sup>J<sub>H,H</sub> = 8.6 Hz, <sup>4</sup>J<sub>H,P</sub> = 6.2 Hz); 8.00 (dd, 2 H, H(3), H(7), <sup>3</sup>J<sub>H,H</sub> = 8.8 Hz, <sup>4</sup>J<sub>H,H</sub> = 2.0 Hz); 8.28 (dd, 2 H, H(1), H(9), <sup>3</sup>J<sub>H,P</sub> = 13.0 Hz, <sup>4</sup>J<sub>H,H</sub> = 2.2 Hz). <sup>31</sup>P{<sup>1</sup>H} NMR (D<sub>2</sub>O), δ: 0.49 s.

**2,8-Bis(1H-1,3-benzimidazol-2-yl)-10-hydroxy-10H-10λ<sup>5</sup>-phenoxaphosphine 10-oxide (4).** A mixture of **2** (1.782 g, 0.005 mol), **3** (1.081 g, 0.01 mol), and PPA (10.8 g) was stirred

under argon at 80 °C until the mixture became homogeneous. Then the reaction mixture was heated at 100 °C for 2 h, at 120 °C for 2 h, at 140 °C for 2 h, at 160 °C for 2 h, at 180 °C for 2 h, and at 200 °C for 16 h, cooled to 140 °C, and poured into water. The precipitate that formed was filtered off, washed with distilled water to neutral pH, and dried *in vacuo* at 100 °C for 10 h. Compound **4** was prepared in a yield of 2.13 g (92%). M.p. > 300 °C. (100% HCOOH). Found (%): C, 68.03; H, 3.52; N, 12.11; P, 6.87. C<sub>26</sub>H<sub>17</sub>N<sub>4</sub>O<sub>3</sub>P. Calculated (%): C, 67.24; H, 3.69; N, 12.06; P, 6.67. <sup>1</sup>H NMR (DMSO-d<sub>6</sub> + 10% KOH solution in MeOH, 4 : 1), δ: 6.81–6.83 (m, 4 H, C<sub>6</sub>H<sub>4</sub>); 7.16 (dd, 2 H, H(4), H(6), <sup>3</sup>J<sub>H,H</sub> = 8.6 Hz, <sup>4</sup>J<sub>H,P</sub> = 5.8 Hz); 7.40–7.42 (m, 4 H, C<sub>6</sub>H<sub>4</sub>); 8.25 (dd, 2 H, H(3), H(7), <sup>3</sup>J<sub>H,H</sub> = 8.6 Hz, <sup>4</sup>J<sub>H,H</sub> = 2.2 Hz); 8.50 (br.s, 2 H, NH); 8.62 (dd, 2 H, H(1), H(9), <sup>3</sup>J<sub>H,P</sub> = 12.8 Hz, <sup>4</sup>J<sub>H,H</sub> = 2.4 Hz). <sup>31</sup>P{<sup>1</sup>H} NMR (DMSO-d<sub>6</sub> + 10% KOH solution in MeOH, 4 : 1), δ: -4.77 s.

**Poly-2-[6-(1H-1,3-benzimidazol-5-ylsulfonyl)-1H-1,3-benzimidazol-2-yl]-10-hydroxy-10H-10λ<sup>5</sup>-phenoxaphosphine 10-oxide (7).** *Method A (in Eaton's reagent).* A mixture of **5** (0.557 g, 0.002 mol), **2** (0.713 g, 0.002 mol), and freshly prepared Eaton's reagent (2.8 mL) was vigorously stirred under argon at 80 °C for 2 h. Then the reaction mixture was heated at 100 °C for 1 h, at 120 °C for 1 h, and at 140 °C for 15 h, diluted with an equal amount of 85% H<sub>3</sub>PO<sub>4</sub>, poured into water, and dispersed. The precipitate that formed was separated, washed with distilled water to neutral pH, extracted with MeOH in a Soxhlet apparatus, and dried *in vacuo* at 100 °C for 10 h. The yield of polymer **7** was 1.25 g (quantitative yield). The polymer is soluble in H<sub>2</sub>SO<sub>4</sub> ([η] = 0.85 dL g<sup>-1</sup>), HCOOH, and CF<sub>3</sub>COOH.

*Method B (in PPA).* A mixture of **5** (0.557 g, 0.002 mol), **2** (0.713 g, 0.002 mol), and PPA (4.5 g) was vigorously stirred under argon at 100 °C for 1 h and heated at 120 °C for 1 h. Then P<sub>2</sub>O<sub>5</sub> (1.5 g) was added. The reaction mixture was heated at 140 °C for 1 h, at 160 °C for 2 h, and at 180 °C for 20 h, cooled to 140 °C, diluted with an equal volume of 85% H<sub>3</sub>PO<sub>4</sub>, poured into water, and dispersed. The precipitate that formed was separated, washed with distilled water to neutral pH, extracted with MeOH in a Soxhlet apparatus, and dried *in vacuo* at 100 °C for 10 h. The yield of polymer **7** was 1.14 g (quantitative yield). The polymer is soluble in H<sub>2</sub>SO<sub>4</sub> ([η] = 2.35 dL g<sup>-1</sup>), HCOOH, and CF<sub>3</sub>COOH.

**Poly-2-[6-(1H-1,3-benzimidazol-5-yloxy)-1H-1,3-benzimidazol-2-yl]-10-hydroxy-10H-10λ<sup>5</sup>-phenoxaphosphine 10-oxide (8).** *Method A (in Eaton's reagent).* Polymer **8** was synthesized in a yield of 1.65 g (quantitative yield) from **6** (0.691 g, 0.003 mol), **2** (1.069 g, 0.003 mol), and freshly prepared Eaton's reagent (3.9 mL) analogously to the synthesis of polymer **7**. The polymer is soluble in H<sub>2</sub>SO<sub>4</sub> ([η] = 2.9 dL g<sup>-1</sup>), HCOOH, and CF<sub>3</sub>COOH.

*Method B (in PPA).* Polymer **8** was prepared from **6** (4.837 g, 0.021 mol), **2** (7.483 g, 0.021 mol), and PPA (60.71 g) analogously to the synthesis of polymer **7**. The yield was 10.56 g

\* The synthesis of polymers **7** and **8** was monitored by measuring the reduced viscosities of the polymers isolated from samples, which were withdrawn from the reaction solutions. The samples were quenched with water, refluxed, and neutralized with aqueous ammonia. The polymers that formed were separated, washed with distilled water until the pH became neutral, and dried *in vacuo* at 100 °C for 1 h.

(quantitative yield). The polymer is soluble in H<sub>2</sub>SO<sub>4</sub> ( $[\eta] = 4.72 \text{ dL g}^{-1}$ ), HCOOH, and CF<sub>3</sub>COOH.

**X-ray diffraction study of 2 (C<sub>14</sub>H<sub>20</sub>K<sub>3</sub>O<sub>14</sub>P).** X-ray diffraction data for compound **2** were collected at  $-120 \text{ }^\circ\text{C}$  on an automated four-circle Syntex P2<sub>1</sub> diffractometer (Mo-K $\alpha$ , graphite monochromator,  $\theta/2\theta$  scanning technique,  $2\theta_{\text{max}} = 56^\circ$ ). At  $-120 \text{ }^\circ\text{C}$ , crystals are triclinic:  $a = 10.007(2)$ ,  $b = 11.354(2)$ ,  $c = 11.685(2)$ ,  $\alpha = 64.82(3)$ ,  $\beta = 70.36(3)$ ,  $\gamma = 76.68(3)^\circ$ ,  $V = 1125.7(4) \text{ cm}^3$ , space group  $P\bar{1}$ ,  $Z = 2$ ,  $M = 560.57$ ,  $d_{\text{calc}} = 1.654 \text{ g cm}^{-3}$ ,  $\mu = 7.44 \text{ mm}^{-1}$ ,  $F(000) = 576$ . Of a total of 5760 measured reflections, 5449 independent reflections were used in subsequent calculations and refinement. The structure of **2** was solved by direct methods and refined by the full-matrix least-squares method with anisotropic displacement parameters for nonhydrogen atoms. The hydrogen atoms were revealed from difference electron density maps and refined isotropically. The final reliability factors were as follows:  $R = 0.0386$  based on 4558 reflections with  $I > 2\sigma(I)$ ,  $wR_2 = 0.1124$ , GOOF = 1.043 based on all measured reflections. All calculations were carried out using the SHELXL program package (version 5).

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