Inorganica Chimica Acta, *186* (1991) 95–101

Formation of tetrabenzoporphine skeleton by the reactions of phthalimide with zinc carbonates

Kunihiro Ichimura, Masako Sakuragi, Hisayuki Morii
Research Institute for Polymers and Textiles, 1-1-4 Higashi, Tsukuba, Ibaraki 305 (Japan)

Madoka Yasuike*, Yasumasa Toba, Masaru Fukui
Tsukuba Research Laboratories, TOYO INK Mfg. Co. Ltd., 27 Wadai, Tsukuba, Ibaraki 300-42 (Japan)

and **Osamu Ohno**
Department of Chemistry, Faculty of Science, Tokyo Institute of Technology, 2-12-1 Ookayama, Meguro-ku, Tokyo 152 (Japan)

(Received November 22, 1990; revised March 12, 1991)

Abstract

The high temperature reactions of phthalimide potassium salt (PIK) with various zinc carbonates were carried out. The reaction of PIK with zinc acetate yielded tetrabenzoporphinato zinc (ZnTBP). Tetra-tert-butyltetrabenzoporphinato zinc (ZntBu$_4$TBP), whose four tert-butyl substituents were positioned on the TBP periphery, was also prepared in a similar way using the 4-tert-butylphthalimide potassium salt (4-tBuPIK). These results were explained on the basis of the function of zinc acetate as the *meso*-methine carbon source of the TBP structure. The use of zinc phenylacetate instead of zinc acetate in the reaction of PIK led to the production of *meso*-phenyl substituted ZnTBPs, although they were different in the number of *meso*-phenyl substituent(s). The attempt to introduce alkyl residues at the *meso*-position(s) by the reaction of PIK in the presence of zinc alkylacetate having a long alkyl chain was not successful, except for the substitution of the methyl group using zinc propionate.

Introduction

Recently, we have reported that the reaction procedures studied for the preparation of a *meso*-tetraphenyltetrabenzoporphinato zinc complex (ZnP$_4$TBP) using zinc acetatc as a chelating agent in the reaction of various reactant combinations consisting of the sources for the isoindole unit of the TBP backbone and those for the *meso*-phenyl groups [1] lead to a complicated mixture of ZnTBPs with a different number of *meso*-phenyl substituents [2]. On the other hand, our method using zinc benzoate as a central metal source has succeeded in the unequivocal synthesis of ZnP$_4$TBP [3]. This situation is demonstrated in Scheme 1. The contribution of zinc acetate has been proposed for the formation of the TBP skeleton. This assumption has prompted us to examine the reactions of PIKs with some zinc carbonates having various groups at the carbonyl α-position, because it is expected that this reaction procedure conveniently forms the TBP framework and simultaneously introduces their residues at the *meso*-position or on the periphery of the TBP, as shown in Scheme 2. In this paper, we attempt the reactions of PIK in the presence of the zinc carbonates, such as zinc alkylacetate and zinc arylacetate, and discuss the structural elucidation of the reaction products.

Experimental

Preparation of ZnTBP

A mixture of PIK (3.7 g, 20 mmol) and zinc acetate dihydrate (8.8 g, 40 mmol) was well ground and put into a 200 ml round-bottomed flask fitted with an air condenser and a N$_2$ inlet and then heated at 360 °C for 1 h. After cooling to room temperature, 150 ml of pyridine was poured into the reaction product, followed by heating of the solution under reflux for 6 h to extract the green product. After the filtration of this extract with suction, the solvent was evaporated *in vacuo* to give 0.77 g of a deep-purple residue which was purified by column chro-

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*Author to whom correspondence should be addressed.

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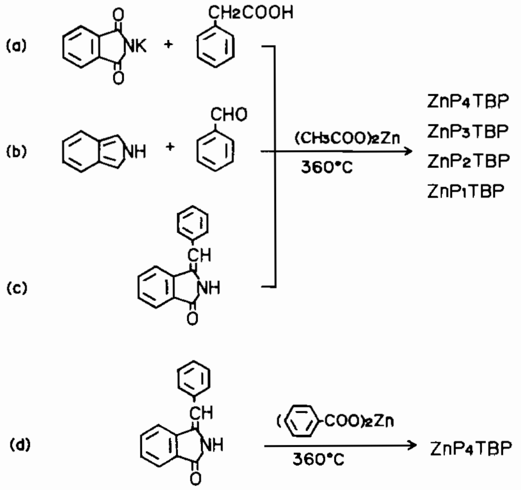
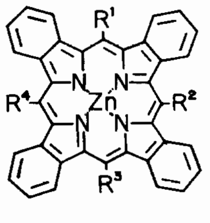
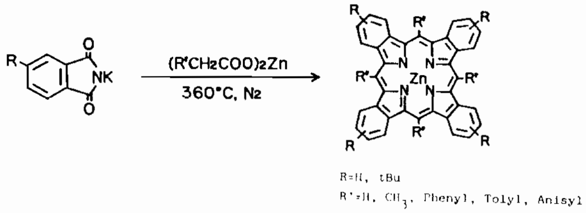


Scheme 1. Reaction scheme for the previously reported procedures of *meso*-tetraphenyltetrabenzoporphinato zinc complex (ZnP_4TBP).



	R¹	R²	R³	R⁴
ZnP₄TBP	Ph	Ph	Ph	Ph
ZnP₃TBP	Ph	Ph	Ph	H
ZnP₂TBP	Ph	Ph	H	H
ZnP₂TBP	Ph	H	Ph	H
ZnP₁TBP	Ph	H	H	H
ZnTBP	H	H	H	H

Structure of *meso*-phenyl substituted ZnTBP



Scheme 2. Schematic representation for the synthesis of ZnTBP derivatives by using of PIK with zinc carbonates.

matography on silica gel (Wakogel C-200) with benzene/THF (8/2) as the eluent. The evaporation of the solvent yielded 0.11 g of residue. The purity of this residue was *c*. 33% from the reported extinction coefficient of ZnTBP [4].

UV–Vis in pyridine: Soret band 409 (0.15) and 433 (1.0) nm; Q band 582 (0.045) and 629 (0.33) nm (absorption ratio). FD-MS: $m/z = 572$. ^1H NMR in DMF-d_7: *meso*-Hs, 11.35 (s, 4H); benzene ring-Hs, 8.26 and 9.97 (double-d, 16H) ppm.

Preparation of tetra-tert-butyl ZnTBP (ZntBu₄TBP)

A mixture of 4.8 g (20 mmol) of 4-tert-butylphthalimide potassium salt (4-tBuPIK) and an excess of zinc acetate (13.2 g, 60 mmol) was heated at 360 °C for 1 h in the same manner as previously described. The resulting green product was extracted with chloroform under reflux. The green solution was then evaporated *in vacuo*. The deep-purple residue was column chromatographed on aluminum oxide (Merck 1097) with benzene/hexane/THF (10/10/1). Further purification was carried out by preparative TLC (PLC) on aluminum oxide (Merck 5756) with the same solvent mixture to give 0.12 g of $ZntBu_4TBP$ (3% yield).

UV–Vis in dichloromethane: Soret band 400 (0.14) and 424 (1.0) nm; Q band 578 (0.034) and 628 (0.32) nm (absorption ratio). FD-MS: $m/z = 796$. ^1H NMR in $CDCl_3$: *meso*-Hs, 10.37 (s, 4H); benzene ring-Hs, 9.36 (d, 8H) and 8.26 (s, 4H); tert-butyl Hs, 1.93 (s, 36H) ppm.

Preparation of meso-phenyl substituted Zn-TBP

A mixture of 0.92 g (5 mmol) of PIK and 3.35 g (10 mmol) of zinc phenylacetate was reacted at 360 °C for 1 h in the same manner as the previous reaction. The reaction product was extracted with hot dioxane and then purified by the combination of column chromatography (Merck 1097) and PLC on aluminum oxide (Merck 5756) with a solvent mixture of benzene/hexane (1/1). This purification gave 0.1 g of green product. The further purification on the alumina using benzene/hexane/THF (10/10/1) led to the successful isolation of at least three components: 33 mg (3% yield) of *meso*-tetraphenyltetrabenzoporphinato zinc (ZnP_4TBP) in fraction 1 (fr. 1), 60 mg (5.5% yield) of *meso*-triphenyltetrabenzoporphinato zinc (ZnP_3TBP) in fr. 2, 18 mg (2% yield) of *meso*-diphenyltetrabenzoporphinato zinc (ZnP_2TBP) in fr. 3.

UV–Vis in CH_2Cl_2: fr. 1, Soret band 427 (0.14) and 455 (1.0) nm, Q band 603 (0.048) and 650 (0.25) nm; fr. 2, Soret band 412 (0.19) and 442 (1.0) nm, Q band 588 (0.039) and 640 (0.27) nm; fr. 3, Soret band 408 (0.12) and 432 (1.0) nm, Q band 584 (0.036) and 633 (0.55) nm (absorption ratio).

FD-MS: fr. 1: $m/z = 876$, 966, 1056; fr. 2: $m/z = 800$, 890, 980; fr. 3: $m/z = 724$, 814, 904.

^1H NMR: fr. 1 in $CDCl_3$: *meso*-phenyl Hs: 8.52 (*o*-H, 8H), 8.27 (*p*-H, 4H), 8.19 (*m*-H, 8H) ppm; benzene ring Hs; 7.50 and 7.43 (16H) ppm; fr. 2 in CD_2Cl_2: *meso*-H: 10.60 (1H) ppm, *meso*-phenyl Hs; 8.22 (*o*-Hs, 6H), 8.01 (*p*-Hs, 3H), 7.92 (*m*-Hs, 6H) ppm; benzene ring Hs: 9.38 (2H), 7.90 (2H), 7.55 (2H), 7.30 (4H), 7.25 (2H), 7.12 (4H) ppm.

Physical measurements

Ultraviolet and visible (UV–Vis) absorption spectra were obtained using a JASCO Ubest-30 spectrophotometer. Field desorption mass spectra (FD-MS) were carried out with a Hitachi M-80 mass spectrometer at 10–20 mA. ^{1}H NMR and ^{1}H–^{1}H COSY spectra were measured with a NICOLET NT-360NB nuclear magnetic resonance spectrometer. Three-dimensional high pressure liquid chromatography (3D-HPLC) was taken on a system containing a JASCO model 880-PU HPLC pump equipped with a MERCK Lichrospher RP-18 Hiber column (particle size 5 μm; 125 × 4 mm ID) and a Ohtsuka Denshi MCPD-350PC spectro multichannel photodetector.

Results and discussion

Formation of TBP skeleton

According to our knowledge concerning the TBP synthesis [2, 3], it has been assumed that the production of a complicated mixture of ZnTBPs with a different number of *meso*-phenyl substituents by the reaction of 3-BPI with zinc acetate is probably caused by the functioning of this zinc carbonate as a chelating agent for the tetramerization of 3-BPI, that is, the participation of zinc acetate is proposed for the formation of the TBP skeleton. This assumption is readily substantiated by the fact that the reaction of PIK in the presence of zinc acetate at 360 °C provides ZnTBP, which is characterized by the UV–Vis absorption spectrum in pyridine [4]: Soret band 409 (0.15) and 433 (1.0) nm, Q band 582 (0.045) and 629 (0.33) nm (absorption ratio). The production of ZnTBP is also confirmed from the ^{1}H NMR spectrum, where the *meso*-proton signal is observed at 11.35 ppm as a singlet and the benzene ring Hs as two double doublets at 8.26 and 9.97 ppm in DMF-d_7. The chemical shifts to low magnetic field are caused by the deshielding effect of the porphyrin current [5]. The detection of the $m/z = 572$ ion peak in its FD-MS, which corresponds to the molecular weight of ZnTPP, supports this assignment. The set of these spectra are presented in Fig. 1.

Consequently, the structural elucidation of the reaction product of PIK with zinc acetate unequivocally proves the formation of ZnTBP. On the other hand, the reaction of PIK with zinc benzoate does not yield TBP based on no observations of the characteristic absorptions of Soret and Q bands in UV–Vis, although in the case of the reaction of 3-BPI with zinc benzoate, ZnP$_4$TBP is produced [3]. Of course, heating PIK in the absence of any other reactants does not lead to the production of TBP. These experimental results allow us to presume that zinc acetate acts not only as the source for the central metal but also as the source for the methine carbon to tetramerize PIK, resulting in the formation of ZnTBP.

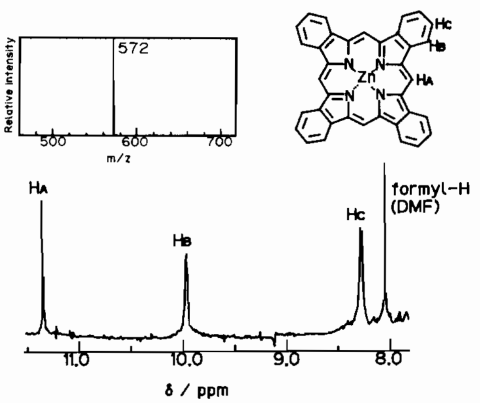


Fig. 1. 360 MHz ^{1}H NMR spectrum of ZnTBP prepared by the reaction of PIK in the presence of zinc acetate at 360 °C and its assignment. DMF-d_6 is used as a solvent. The inset figure shows the FD-MS of this product. The ion peak of $m/z = 572$ corresponds to the molecular weight of ZnTBP.

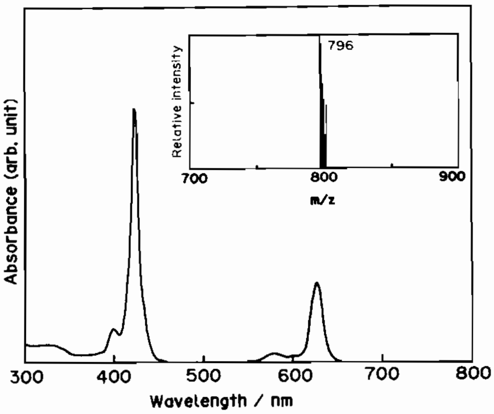


Fig. 2. UV–Vis absorption spectrum of tetra-tert-butyl substituted ZnTBP (ZntBu$_4$TBP) in CH_2Cl_2 obtained by the reaction of 4-tert-butyl PIK with zinc acetate. The FD-MS, where $m/z = 796$ ion peak corresponding to the molecular weight of the ZnTBP is detected, is also shown in the inset.

These results allow us to examine the reaction of the 4-tert-butylphthalimide potassium salt (4-tBuPIK) with zinc acetate for the preparation of tetra-tert-butyltetrabenzoporphinato zinc (ZntBu$_4$TBP), because it is expected that such bulky substituents like tert-butyl attached at the TBP periphery overcome the solubility problem of ZnTBP in common organic solvents due to inhibiting the stacking of this macrocyclic plane. The formation of the TBP structure is evidently confirmed by the observation of the characteristic Soret absorption bands (400 and 424 nm) and Q bands (578 and 628 nm) in dichloromethane solution, as shown in Fig. 2. Figure 3 shows the ^{1}H NMR spectrum in $CDCl_3$, where the proton signal of the four *meso*-Hs (4H) is located at 10.37 ppm as a singlet, those of both the inner 8Hs and the outer 4Hs on the benzene

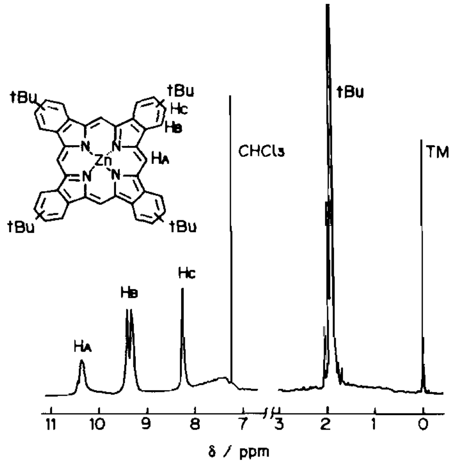


Fig. 3. 360 MHz ^1H NMR spectrum of $ZntBu_4TBP$ in $CDCl_3$ and its assignment.

rings at 9.36 ppm as a doublet and at 8.26 ppm as a singlet, respectively, and that of 36H of the t-butyl group on the benzene rings at 1.93 ppm as a singlet. The ion peak of $m/z = 796$ on FD-MS in Fig. 2, which is consistent with the molecular weight of $ZntBu_4TBP$, is evidence for the above assignment. These spectral data show the production of $ZntBu_4TBP$. This result suggests the feasibility that the high-temperature reaction of a commercially available phthalimide potassium salt having an appropriate substituent with zinc acetate conveniently leads to the ZnTBP possessed of the corresponding group at its periphery.

Formation of meso-substituted ZnTBP derivatives

The other possibility to enhance the solubility of ZnTBP in common organic solvents is the introduction of the substituents at its *meso*-positions, as in the case of porphyrin compounds. At first, our purpose was focussed on the alkyl substitution, because it was expected that the reactions of PIK with various zinc alkylacetates could lead to the preparation of the ZnTBPs with the corresponding alkyl residue at the *meso*-positions. The reaction of PIK in the presence of zinc propionate at 360 °C yields a trace amount of the TBP-like complex from its absorption spectrum in pyridine (Soret band 411 and 435 nm, Q band 584 and 630 nm). The FD-MS of this reaction product supports the existence of *meso*-methyl substituted ZnTBPs from the detection of $m/z = 628$, 614, 600, 586 and 572 ion peaks, which correspond to the molecular weight of *meso*-tetramethyltetrabenzoporphinato zinc ($ZnMe_4TBP$), *meso*-trimethyltetrabenzoporphinato zinc ($ZnMe_3TBP$), *meso*-dimethyltetrabenzoporphinato zinc ($ZnMe_2TBP$), *meso*-monomethyltetrabenzoporphinato zinc ($ZnMe_1TBP$) and ZnTBP, respectively. The set of these spectra is shown in Fig. 4. Development of this reaction procedure to other zinc alkylacetates having long alkyl residues, such as zinc peralgonate, has failed in the formation of the TBP skeleton, probably owing to the lack of reaction temperature resulting from the low melting point of such a zinc carbonate with a long alkyl chain. For the formation of these *meso*-alkyl substituted ZnTBPs, the reaction procedure using 3-alkylidenephthalimidine with zinc acetate, reported by Russian workers [6], may be preferable.

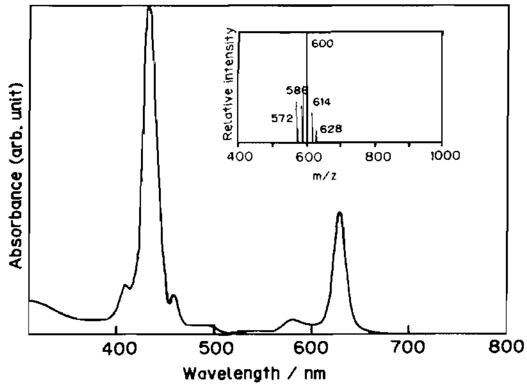


Fig. 4. UV–Vis absorption spectrum in pyridine and FD-MS of the reaction product of PIK with zinc propionate at 360 °C.

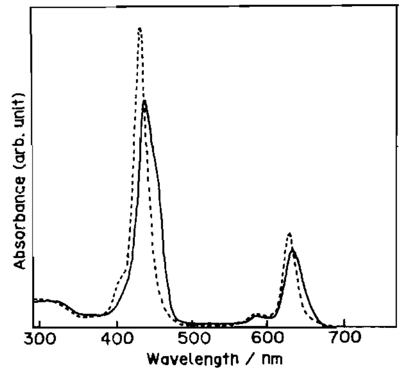


Fig. 5. UV–Vis absorption spectra of *meso*-phenyl substituted ZnTBPs in CH_2Cl_2 prepared by the two methods: (——) by the reaction of PIK with zinc phenylacetate; (– – –) by the reaction of 3-BPI in the presence of zinc acetate.

Another of our interests for *meso*-substitution has been directed toward the synthesis of *meso*-aryl substituted ZnTBPs. Since the structural elucidation of *meso*-phenyl substituted ZnTBPs has been well established [2, 3], the reaction of PIK with zinc phenylacetate has been employed for the production of the *meso*-phenyl substituted ZnTBP (ZnPTBP). The formation of the TBP skeleton and the introduction of the phenyl residue into the *meso*-position is confirmed by the measurements of the visible absorption spectrum in Fig. 5 and the FD-MS in

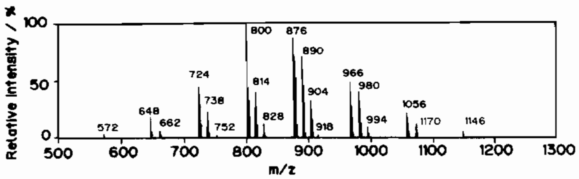


Fig. 6. FD-MS of the reaction product of PIK with zinc phenylacetate.

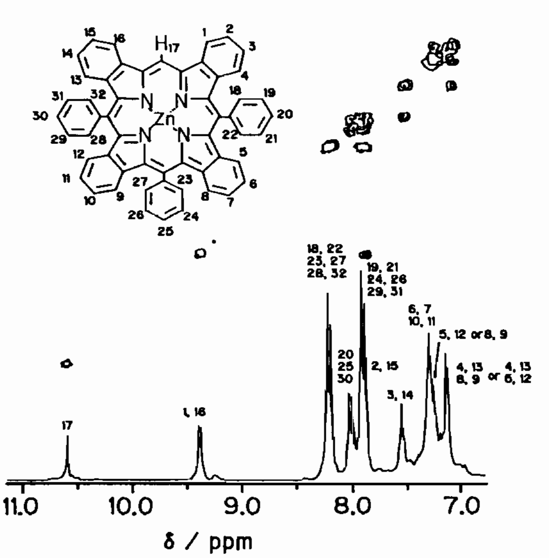


Fig. 7. ^1H NMR and ^1H–^1H COSY spectra of ZnP_3TBP in CD_2Cl_2 and its assignment.

Fig. 6, although the product comprises a complicated mixture of ZnTBPs with a different number of *meso*-phenyl substituent(s) and benzyl adducts, as well as in the case of the reaction of 3-BPI with zinc acetate [2].

The careful purification of this reaction product by PLC succeeds in the separation of at least three components. From the previously reported visible absorption data of the ZnPTBPs in dichloromethane [2], the product is composed of ZnP_4TBP in 3% yield from fr. 1, ZnP_3TBP in 5.5% yield from fr. 2, and ZnP_2TBP in 2% yield from fr. 3. By the reaction of 3-BPI with zinc acetate, ZnP_4TBP, ZnP_3TBP and ZnP_2TBP were obtained in 0.4, 4.9, and 5.4% yield, respectively [2]. The difference in the component ratio of each ZnPTBP prepared by these two methods gives rise to the difference in the UV–Vis as shown in Fig. 5. Figure 7 shows the *J*-correlated ^1H–^1H COSY and ^1H NMR spectra of the ZnPTBP in fr. 2, where the *meso*-triphenyl substituted structure is demonstrated. The positional numbers of the Hs on ZnP_3TBP structure correspond to the numbers on the ^1H NMR spectrum. There are probably two isomers of ZnP_2TBP in fr. 3, which possess two phenyl groups at either the neighboring or opposite *meso*-positions. Table 1 summarizes the results obtained on the products by the reaction of PIK with zinc phenylacetate.

The previous result presents unambiguous evidence that the use of zinc phenylacetate as a chelating agent in the reaction of PIK succeeds in the introduction of its phenyl residue(s) into the *meso*-position(s) and the methylene carbon of the zinc phenylacetate acts as the source for *meso*-carbons to lead the TBP skeleton. The same situation is also caused by the reactions of either zinc *p*-tolylacetate or zinc *p*-anisylacetate with PIK resulting in the production of *meso*-*p*-tolyl or *meso*-*p*-anisyl substituted ZnTBPs, respectively. Without exception, these *meso*-aryl substituted ZnTBPs are comprised of a complicated mixture of ZnTBP derivatives having a different number of *meso*-aryl substituents and the additional complexes with either *p*-methylbenzyl or *p*-methoxybenzyl group(s), as well as in the case of the reaction of PIK with zinc phenylacetate. The FD-MS data and their assignments are summarized in Table 2. The differences of 104 or 120 in the mass ion peaks are consistent with the molecular weights of *p*-methylbenzyl or *p*-methoxybenzyl fragments.

The existence of the benzyl adducts of each ZnPTBP is substantiated by both 3D-HPLC analysis and FD-MS as summarized in Table 3, where the absorption and the mass data are collected. Figure 8 shows the 3D-HPLC result of the collected compound in fr. 2 as an example, demonstrating the contour plots of the HPLC and the UV–Vis absorption spectra of two fractions at retention times of 5.9 and 7.0 min. The FD-MS shows that this fraction consists of three components with the mass ion peaks of $m/z = 800$, 890 and 980, which correspond to the molecular weights of ZnP_3TBP and its benzyl adduct(s). Since the ion peak of $m/z = 800$ is the most intensively detected on FD-MS, the component of the first fraction at 5.9 min on 3D-HPLC is assignable to ZnP_3TBP and the second one to its benzyl adduct. Another component with two parts of the benzyl residue, $m/z = 980$ on FD-MS, is slightly detectable at 8.5 min on the 3D-HPLC. These absorption spectra have identical λ_{max} of both the Soret and Q bands, which is indicative of possessing the same backbone. This means that the spectra are little affected by the benzyl fragment(s), on account of the inconjugation of this group to the TBP π-electrons.

The formation of these benzyl adducts is probably brought about by the pyrolysis of zinc phenylacetate at a high temperature, resulting in the generation of active species such as the benzyl radical. Although these impurities may overcome several disadvantages in the optical evaluation of the ZnTBPs, both the

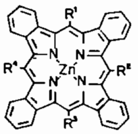
TABLE 1. Spectral data and yields of the products obtained by the reaction of PIK with zinc phenylacetate

Compound	Absorption data in dichloromethane		FD-MS data *m/z*	Yield (%)
	Soret band	Q band		
ZnP$_4$TBP	427(0.14), 455(1.0)	603(0.048), 650(0.25)	876, 966, 1056	3.0
ZnP$_3$TBP	412(0.19), 442(1.0)	588(0.039), 640(0.27)	800, 890, 980	5.5
ZnP$_2$TBP	408(0.12), 432(1.0)	584(0.036), 633(0.55)	724, 814, 904	2.0

Data in parentheses denote absorption ratio.

TABLE 2. FD-MS data of the reaction products of PIK with zinc *p*-tolylacetate (A) or zinc *p*-anisylacetate (B), and their assignments

Run	Complex	FD-MS data, *m/z*
A	Zntolyl$_4$TBP	932, 1036(932+104), 1140(932+104X2)
	Zntolyl$_3$TBP	842, 946(842+104), 1050(842+104X2), 1154(842+104X3)
	Zntolyl$_2$TBP	752, 856(752+104), 960(752+104X2), 1064(752+104X3)
	Zntolyl$_1$TBP	662, 766(662+104), 870(662+104X2), 974(662+104X3)
	ZnTBP	572, 676(572+104), 780(572+104X2), 884(572+104X3)
B	Znanisyl$_4$TBP	996
	Znanisyl$_3$TBP	890, 1010(890+120)
	Znanisyl$_2$TBP	784, 904(784+120), 1024(784+120X2)
	Znanisyl$_1$TBP	678, 798(678+120), 918(678+120X2)
	ZnTBP	572, 692(572+120), 812(572+120X2)



A: R^1-R^4=H or tolyl
B: R^1-R^4=H or anisyl

TABLE 3. FD-MS data and HPLC results of the isolated compounds from the reaction product of PIK with zinc phenylacetate by PLC on aluminum oxide

Run	Retention time (min)	Absorption data (nm)		FD-MS *m/z*	Assignment
		Soret band	Q band		
Fr. 1	5.73	432sh, 459	606, 651	876	ZnP$_4$TBP
	6.93	432sh, 459	606, 651	966	benzyl adduct
	8.56	432sh, 460	607, 652	1056	benzyl adduct
Fr. 2	5.94	417, 442	592, 638	800	ZnP$_3$TBP
	6.94	418, 443	593, 638	890	benzyl adduct
				980	benzyl adduct
Fr. 3	6.47	409, 434	584, 630	724	ZnP$_2$TBP
	7.09	407, 432	582, 631	814	benzyl adduct
				904	benzyl adduct

Absorption data were obtained on the 3D-HPLC system equipped with a Merck Lichrospher RP-18 Hiber column and a Ohtsuka Denshi MCPD-350PC spectro multichannel photodetector, using a solvent mixture of methanol/acetone (8/2 vol. ratio).

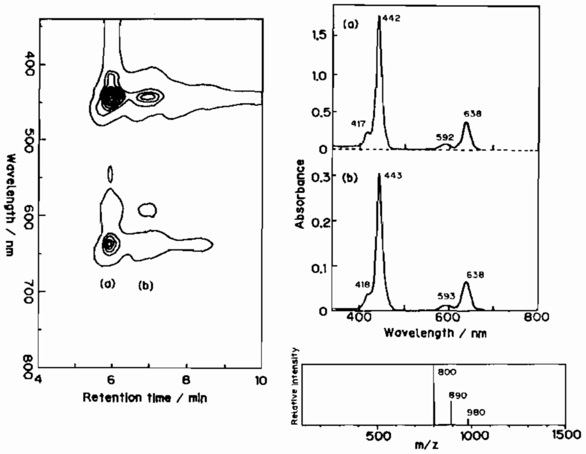


Fig. 8. 3D-HPLC contour plots, visible absorption spectra and FD-MS of a mixture of ZnP_3TBP and its benzyl adducts isolated from the reaction product of PIK with zinc phenylacetate by PLC on aluminum oxide, by use of benzene/hexane/THF (10/10/2 vol. ratio) as an eluent.

excellent solubility in common organic solvents and the satisfactory compatibility into polymeric materials stem from the existence of these impurities. This is favorable for using this complex as a photosensitizer in the photoimaging application field [8].

Conclusions

The reactions of either PIK or 4-tBuPIK with zinc acetate evidently give ZnTBP and $ZntBu_4TBP$, respectively. These results suggest that zinc acetate serves not only as the chelating agent but also as the source for the *meso*-methine carbons of the ZnTBP derivatives. This reaction procedure does not extend to the reactions of PIK with zinc carbonates having a long alkyl chain in order to obtain *meso*-alkyl substituted ZnTBPs, but to the reactions of PIK with zinc arylacetates, such as zinc phenylacetate, zinc tolylacetate and zinc anisylacetate, to yield *meso*-phenyl substituted ZnTBP, *meso*-tolyl substituted ZnTBP and *meso*-anisyl substituted ZnTBP, respectively, although these products consist of the ZnTBPs having a different number of *meso*-aryl substituent(s) and additional contaminations with the benzyl (or *p*-methylphenyl, *p*-methoxybenzyl) residue(s).

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