20.3 ppm (CH_3). Mass spectrum: The low-resolution spectrum shows a weak molecular ion cluster and an intense cluster at molecular ion $- C_3H_7$ (loss of isopropyl group). The high-resolution spectrum did not read out the molecular ion but did identify the $M^+ - C_3H_7$ cluster. Calcd for $^{12}C_9{}^1H_9{}^{14}N_6{}^{11}B^{35}Cl_2{}^{37}Cl^{90}Zr$, $^{12}C_9{}^1H_9{}^{14}N_6{}^{11}B^{35}Cl_3{}^{90}Zr$, $^{12}C_9{}^1H_9{}^{14}N_6{}^{10}B^{35}Cl_3{}^{90}Zr$, $^{12}C_9{}^1H_9{}^{14}N_6{}^{11}B^{35}Cl_2{}^{37}Cl^{92}Zr$: 408.9098, 406.9094, 405.9133, 410.9066. Found: 408.9083, 406.9097, 405.9134, 410.9076.

Acknowledgment. We wish to thank the Research and Productive Scholarship Committee of the University of South Carolina for support of this research. We also acknowledge the National Science Foundation Regional Mass Spectrometry facility at the University of Nebraska.

Registry No. [HB(3,5-Me_2pz)$_3$]$ZrCl_3$, 80041-67-4; [HB(3,5-Me_2pz)$_3$]Zr(OMe)Cl_2, 80041-68-5; [*n*-BuB(pz)$_3$]$ZrCl_3$, 80041-69-6; [*i*-PrB(pz)$_3$]$ZrCl_3$, 80041-70-9; Na[*i*-PrB(pz)$_3$], 80041-71-0; Na[*n*-BuB(pz)$_3$], 80041-72-1; isopropylboronic acid, 80041-89-0; *n*-butylboronic acid, 4426-47-5; pyrazole, 288-13-1.

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Oxochromium(IV) Porphyrins and Their Relationship to Heme Proteins

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Recent evidence indicates the occurrence of ferryl porphyrin systems (oxoiron(IV) porphyrins) and oxoiron(V) porphyrins at the active sites of peroxidases[1] and cytochrome P 450,[2] respectively. As free oxoiron(IV) porphyrins themselves are unstable above −30 °C[3] and hence are only studied in solution,[4,5] other oxometal tetrapyrroles such as those of chromium[6-9] and manganese[10-13] are under investigation with a view to mimicking reactions of the active heme proteins mentioned. The model complexes can be obtained from their reduced counterparts by autoxidation or by oxidation with hypochlorite or iodosylbenzene. Thus, a chromium(V) corrole, CrO(OAC),[9,14] and a chromium(IV) phthalocyanine, CrO(Pc), have each been obtained as pure solids while a chromium(V) porphyrin of the presumed composition CrO(Cl)(TPP) has been investigated in solution.[6] For complexes with manganese, MnO(X)(TPP)·PhI (X unspecified, complex containing iodobenzene) has been obtained as a solid at −40 °C[12] and MnO(TPP) has been investigated by field ion desorption mass spectroscopy.[13]

We report here the preparation, isolation, and spectral characterization of the oxochromium(IV) porphyrins CrO(TTP) and CrO(OEP).[14] This work was undertaken as a continuation of our studies of oxo transition-metal porphyrins containing Ti^{IV}, V^{IV}, Mo^{IV}, Mo^{V}, W^{V}, Re^{V}, and Os^{VI}.[15] The reactivity of the oxochromium(IV) complexes toward alcohols and alkanes as typical substrates of peroxidases and cytochrome P 450,[1,16] respectively, has also been examined. First results are presented as follows.

While this note was in preparation, J.W.B. was informed that CrO(TPP) and CrO(TTP) with congruent properties have been obtained independently by Groves and co-workers[17a] during their current work on CrO(Cl)(TPP). Shortly thereafter, a paper of West and co-workers appeared describing a different synthesis and the X-ray structure determination of CrO(TPP).[17b]

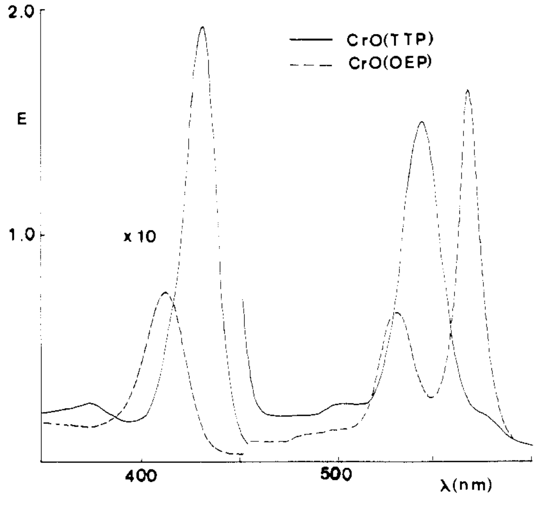

Figure 1. Optical absorption spectra of CrO(TTP) (—) and CrO(OEP) (- - -) in toluene. (The Soret bands are reduced by factors of 0.1.)

Experimental Section

Oxidation of Hydroxochromium(III) Porphyrins with Sodium Hypochlorite. (a) Preparation of Oxo(tetra-*p*-tolylporphinato)chromium(IV), CrO(TTP).[14] Cr(OH)(TPP)·$2H_2O$[18-20] (152 mg, 0.2 mmol) in 100 mL of CH_2Cl_2/EtOH (98:2) was stirred with 1 mL of 2 M aqueous NaOH and 5 mL of aqueous NaOCl (13% active chlorine) until the color changed from violet-green to red. After the solution was washed with 2 M NaOH and taken to dryness in vacuo,

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(14) Abbreviations used: $(OAC)^{3-}$, octaalkylcorrole trianion; $(Pc)^{2-}$, phthalocyaninate dianion; $(TPP)^{2-}$ *meso*-tetraphenylporphinate dianion; $(TTP)^{2-}$, *meso*-tetra-*p*-tolylporphinate dianion; $(OEP)^{2-}$, octaethylporphinate dianion; Ph, C_6H_5; Et, C_2H_5.

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(18) Cr(OH)(TTP)·$2H_2O$ (according to elemental analysis) was obtained from $CrCl_3$ in benzonitrile,[15,19] chromatography on alumina, grade III with CH_2Cl_2/MeOH, and crystallization in presence of NaOH. This compound is similar to Fleischer's Cr(OH)(TPP)·$2H_2O$.[20]

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the residue was chromatographed at basic alumina (grade IV) with CH_2Cl_2 free from HCl and EtOH. Evaporation of the first red fraction, recrystallization from benzene, and washing with acetone yielded red crystals of CrO(TTP)[14] (101 mg, 68%): mol wt 736.8; field desorption mass spectrum A = 736 (11%), 720 (100%); optical spectrum (toluene) (Figure 1) λ_{max} (log ϵ) 580 (4.16), 546 (4.32), 520 (4.20), 433 (5.31) nm; IR (KBr) 1020 cm^{-1} (ν_{CrO}); ^{1}H NMR [C_6D_6, δ values vs. internal Me_4Si (multiplicity, assignments)] δ 2.37 (s, p-CH_3), 7.22 (d, m-H), 7.97 (d, o-H), 9.21 (s, pyrrole H). Anal. Calcd for $C_{48}H_{36}N_4CrO$: C, 78.24; H, 4.92; N, 7.60; O, 2.17. Found: C, 78.76; H, 5.25; N, 7.33; O, 2.20.

(b) Preparation of (Octaethylporphinato)oxochromium(IV). The analogous hypochlorite oxidation of the octaethylporphyrin complex CrOH(OEP)·$0.5H_2O$[21] gives the chromium(IV) complex CrO(OEP), in reasonable yield (39%); mol wt 600.8. Anal. Calcd for $C_{36}H_{44}N_4CrO$: C, 71.98; H, 7.38; N, 9.33; O, 2.66. Found: C, 72.14; H, 7.41; N, 9.34; O, 2.69. Field desorption mass spectrum: A = 600. The reaction is more difficult to follow optically because both substrate and product are red, but they have reversed intensity orders of the α and β bands [λ_{max} (log ϵ) (CH_2Cl_2) for CrOH(OEP)·$0.5H_2O$: 646 (2.67), 572 (3.77), 540 (3.89), 421 (5.06), 382 (4.40) nm. λ_{max} (log ϵ) (toluene) for CrO(OEP): 570 (4.50), 532 (4.09), 413 (5.15) nm]. CrO(OEP) is more reactive than CrO(TTP). It must be chromatographed with benzene instead of dichloromethane. IR (KBr): 1015 cm^{-1} (ν_{CrO}). ^{1}H NMR [C_6D_6, δ vs. internal Me_4Si (multiplicity, assignment)]: 1.86 (t, CH_3), 3.96 (q, CH_2), 10.56 (s, CH).

Oxidation of a Hydroxochromium(III) Porphyrin with Iodosylbenzene. CrOH(TTP)·$2H_2O$ (152 mg, 0.2 mmol) in 100 mL of CH_2Cl_2 (free from acid and alcohol) was treated with PhIO (ca. 88 mg, ca. 0.4 mmol) within 2 h at room temperature until the color changed from violet-green to red. The mixture was washed with water, filtered, and taken to dryness at about 40 °C in vacuo and finally dried in a high vacuum for 1 h. Workup by chromatography was performed as in the hypochlorite oxidation; however, the red fraction of CrO(TTP) was followed by a green, nonporphyrin product (no Soret band). Recrystallization of the red fraction from benzene yielded 72 mg (48%) of CrO(TTP).

Treatment of Hydrocarbons with CrO(TTP). The following compounds (μmol amounts) were treated with CrO(TTP) in CH_2Cl_2 (purified with CrO_2Cl_2) at 30 °C over several hours: naphthalene, cyclohexane, and *trans*-1-phenyl-2-methylethene. Neither a reduction of CrO(TTP) (which would be indicated by a color change from red to green) nor the formation of organic oxidation products (detectable by GLC) was observed.

A spectroscopic sample of CrO(TTP) (10^{-4}–10^{-5}M) in purified CH_2Cl_2 was stable for at least 1 day at 30 °C. On addition of PhIO (10^{-4} M), the optical spectrum due to the complex decayed rapidly within a few minutes. This rapid degradation did not occur with either PhI or $PhIO_2$.

Oxidation of Benzyl Alcohol with CrO(TTP). CrO(TTP) (5.77 μmol) and $PhCH_2OH$ (87 μmol) were dissolved in purified CH_2Cl_2 (0.3 mL). The formation of PhCHO as monitored by GLC was complete within 80 min at room temperature (2.81 μmol). The subsequent addition of PhIO (5.91 μmol) in one portion as a finely powdered solid led to the formation of further PhCHO (5.85 μmol, 100 min).

Results and Discussion

The oxidation of the hydroxochromium(III) porphyrins CrOH(TTP)·$2H_2O$ and CrOH(OEP)·$0.5H_2O$ by sodium hypochlorite furnished the corresponding oxochromium(IV) porphyrins CrO(TTP) and CrO(OEP) in good to reasonable yields.

As iodosylbenzene, $(PhIO)_n$,[22] served to oxidize CrCl(TPP) to CrO(TPP)Cl,[6] a Cr^V complex, we then applied this oxidant to CrOH(TTP)·$2H_2O$. The main product again was CrO(TTP), the administered excess of PhIO causing a degradative oxidation of the porphyrin system at CrO(TTP) which was detected by treating pure CrO(TTP) with PhIO independently. The metal oxidation of CrOH(TTP) by PhIO seems to be a more rapid process than this oxidative degradation, thus allowing the isolation of CrO(TTP). We did not obtain a chromium(V) porphyrin in these oxidations. Unlike CrCl(TPP),[6] Cr(OH)(TTP) does not seem to be a suitable starting material for the generation of an oxochromium(V) porphyrin. As a corollary, our CrO(TTP) does not catalyze the hydroxylation of alkanes or the epoxidation of alkenes by iodosylbenzene.

The optical spectra of CrO(TTP) and CrO(OEP) (Figure 1) are of the normal metalloporphyrin type, very slightly hypsochromically shifted. The ^{1}H NMR spectra display normal peak positions and line widths and thus indicate diamagnetism. The chemical shifts of the pyrrole protons in CrO(TTP) and the methine protons in CrO(OEP) are typical for a rather highly charged central metal.

After the solution was cooled to −60 °C (in toluene-d_8), the doublet of the o-tolyl protons splits into two doublets at δ 7.82 and 7.99 due to the freezing of the rotation of the p-tolyl groups about the porphyrin plane. This phenomenon indicates the axially unsymmetrical nature of the square-pyramidal oxo complex. Diamagnetism and a red color is likewise observed with the heavier metal homologue MoO(TTP);[23] this compound, however, has split o-tolyl proton signals already at room temperature, indicating a higher rotational barrier for the p-tolyl groups as compared with the chromium complex.

As described thus far, the oxochromium(IV) complex CrO(TTP) is closely analogous to MoO(TTP). However, we were puzzled that the optical spectrum of CrO(TTP) is virtually identical with that assigned in the literature to the oxochromium(V) complex "red CrO(Cl)(TPP)" (λ_{max} 582, 544, 427 nm).[6] Such a similarity would not be expected since all other oxometal(V) porphyrins of the type MO(X)(TPP) (M = Mo, W, Re; X = OMe, Cl, F, etc.) have so-called "hyper" spectra,[15a] i.e., extra bands in the visible. This puzzle was solved when we learned that the published spectrum of CrO(Cl)(TPP) is in error and is in fact the spectrum of CrO(TPP) which was formed from CrO(Cl)(TPP) by inadvertent reduction of the spectral sample.[17] Solutions of CrO(Cl)(TPP) are also red but show weak, broad bands throughout the 500–600-nm region.[17,24]

In the pure, crystalline state, both oxochromium(IV) complexes are stable for months. The reactivity of CrO(TTP) toward some typical substrates of cytochrome P 450 dependent monooxygenases was studied. No reduction of the complex and no organic oxidation products were observed with the following compounds: naphthalene, cyclohexane, cyclohexene, and *trans*-1-phenyl-2-methylethene. Therefore, no alkane, alkene, or aromatic oxidizing activity is found with an oxochromium(IV) porphyrin.

However, the quantitative oxidation of 1 mol of benzyl alcohol by 2 mol of CrO(TTP) is a typical example of alcohol oxidation. CrO(TTP) probably acts as a single hydrogen atom abstractor, yielding CrOH(TTP), and so could resemble peroxidase compound II. From CrOH(TTP), CrO(TTP) may be regenerated with PhIO. Therefore, a quantitative oxidation of the alcohol by PhIO in the presence of the reduction product of CrO(TTP) occurs. As a whole, CrO(TTP) could be regarded as a model of peroxidase compound II and not as a model of the active oxygen species generated at the cytochrome P 450 catalytic centre.

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[22] According to vibrational spectroscopy, iodosylbenzene is in fact a polymer with I–O–I–O chains, no doubly bound oxygen being present (H. Siebert, personal communication). Field ion desorption mass spectra show peaks for PhI and PhIO at gentle filament heating; stronger heating causes decomposition of the sample without production of peaks attributable to $(PhIO)_n$. A mass spectrum is not obtained in an electron bombardment ion source (J. J. Veith, personal communication).

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In the first-row oxometal(IV) porphyrins, MO(Por),[14] the reactivity toward organic substrates increases in the series TiO(Por) ≈ VO(Por) ≪ CrO(Por) < MnO(Por) < FeO(Por). Examples of the latter two species have yet to be obtained in a pure state. In moving along this series, the increasing number of d electrons cause an increasing repulsion of the p electron pairs at the terminal oxo group, thus destabilizing and activating the oxygen atom. The instability of the manganese systems is not due to the central metal alone since very stable, red, diamagnetic nitridomanganese(V) porphyrins have been isolated, e.g., MnN(TTP) and MnN(OEP).[25] However, the paramagnetic oxochromium(V) porphyrins appear to be very reactive.[6]

Acknowledgment. We thank Dr. J. J. Veith for the mass spectra and Professor J. T. Groves for communication of his results prior to publication. Financial support by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged.

Registry No. CrO(TPP), 78833-34-8; CrO(OEP), 79919-63-4; Cr(OH)(TTP), 33519-59-4; Cr(OH)(OEP), 50733-41-0; PhCH$_2$OH, 100-51-6.

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Preparation of a Spirocyclic Phosphorane with a P^{V}–P^{V} Bond

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In recent years there has been much interest in compounds containing a phosphorus–phosphorus bond with highly coordinated phosphorus atoms in these laboratories[1,2] and in others.[3] A primary thrust of the work in these laboratories has been to demonstrate that compounds with P^{V}–P^{V} bonds are stable. In fact, it was shown that when the two phosphorus atoms belong to cyclenphosphorane, such a compound could be isolated.[3] In this study it has been shown that a compound containing P^{V}–P^{V} bond can be prepared, where one phosphorus atom does not belong to a cyclic ring system and where the phosphorus atoms have coordination numbers of 5 and 4.

Experimental Section

Materials and General Procedures. The starting materials [(C$_6$H$_5$N)CH$_3$NCO]$_2$PCl[4] and NaP(O)(OC$_2$H$_5$)$_2$[5] were prepared according to published methods. Dry nitrogen was used to prevent the effects of moist air. All solvents were dried and purified by conventional methods and handled under a dry nitrogen atmosphere.

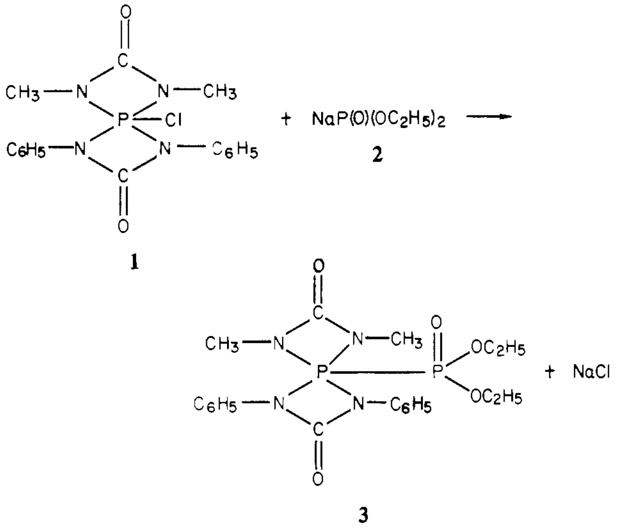
Preparation of 1,3-Dimethyl-5,7-diphenyl-4-(diethoxyphosphinyl)-1,3,5,7-tetraaza-4λ^5-phosphaspiro[3.3]heptane-2,6-dione (3). A solution of 1.32 g (8.2 mmol) of (C$_2$H$_5$O)$_2$P(O)Na in 30 mL of benzene was added dropwise to a stirred solution of 3 g (8.2 mmol) of [O═C(NCH$_3$)NC$_6$H$_5$]$_2$PCl in 100 mL of benzene. The reaction was conducted in a 250-mL two-necked flask, which was equipped with a reflux condenser, dropping funnel, and provision for the ingress and egress of dry nitrogen. After the addition was complete, the reaction mixture was refluxed for 4 h. Filtration of NaCl and removal of solvent afforded an oily residue, which was purified by recrystallization from THF to produce a white powder in 42% yield (1.64 g).

Anal. Calcd for $C_{20}H_{26}N_4O_5P_2$: C, 51.7; H, 5.6; N, 12.0; P, 13.5. Found: C, 51.7; H, 5.7; N, 11.8; P, 13.5. IR data (Nujol): 1780 s, 1610 w, 1510 s, 1485 s, 1395 w, 1365 s, 1350 sh, 1320 w, 1270 s, 1250 w, 1165 w, 1100 w, 1030 s, 930 w, 800 w, 780 w, 715 w, 660 w cm^{-1}. The mass spectrum exhibited a molecular ion (*m/e* 464, 1% relative intensity) and peaks corresponding to the following: $C_9H_{10}N_2O_2P_2$ (240, 7%); $C_7H_5N_2OP_2$ (195, 100%); C_7H_5NO (119, 87%); C_7H_5N (91, 54%). ^{1}H NMR (CH$_2$Cl$_2$): δ 1.21 (CH$_3$–CH$_2$O); 2.6 (CH$_3$–N); 4.21 (CH$_2$); 7.46 (C$_6$H$_5$). ^{31}P NMR (CH$_2$Cl$_2$): $\delta(P_O)$ 12.0; $\delta(P_N)$ −80.9; $J_{P_O-P_N}$ = 708.7 Hz.

Spectroscopic Measurements. The infrared spectrum of compound 3 was recorded as a Nujol mull by a Perkin-Elmer 735 B spectrophotometer with NaCl plates. Its mass spectrum was recorded by a Varian CH5 instrument operating at an ionizing voltage of 70 eV. ^{1}H and ^{31}P NMR spectra were recorded on a Bruker WH-90 instrument at spectrometer frequencies of 60 and 36.43 MHz, respectively. ^{1}H and ^{31}P chemical shifts (negative direction upfield) were measured with respect to internal Me$_4$Si and external 85% H$_3$PO$_4$, respectively.

Results and Discussion

The reaction of the spirocyclic compound 1 with NaP(O)(OC$_2$H$_5$)$_2$ (2) in benzene as solvent results in a phosphorus–phosphorus-bonded product, the plausible structure for which is 3. On the basis of an X-ray analysis[4] it was shown that the chlorine atom in 1 is in an equatorial position of a trigonal-bipyramidal configuration together with two nitrogen atoms. The same structure is adopted for 3 with the exocyclic phosphorus in an equatorial position. A proof for the P–P bond is given by the ^{31}P NMR. The spin–spin coupling constant between the two phosphorus atoms falls in the range[2] of directly bonded phosphorus atoms ($^1J_{PP}$ = 708.9 Hz). It is inconceivable for J_{PP} to be over 700 without P–P direct bonding. For comparison so far coupling constants with directly bonded phosphorus atoms of coordination numbers 5 and 4 are not known. In $Me_3P \rightarrow PF_5$ a $^1J_{PP}$ of 715 Hz was found.[6] The stability of 3 is surprising: it can be transferred into the gas phase to get a good mass spectrum. The mass



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