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Facile iodination of the vinyl groups in protoporphyrin IX dimethyl ester and subsequent transformation of the iodinated moieties

Kota Miyata [a], Satoru Yasuda [a], Takuto Masuya [a], Satoshi Ito [a], Yusuke Kinoshita [b], Hitoshi Tamiaki [b], Toru Oba [a, *]

[a] Department of Material and Environmental Chemistry, Graduate School of Engineering, Utsunomiya University, Utsunomiya, Tochigi 321-8585, Japan
[b] Graduate School of Life Sciences, Ritsumeikan University, Kusatsu, Shiga 525-8577, Japan

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ABSTRACT

Iodination of protoporphyrin IX dimethyl ester using phenyliodine bis(trifluoroacetate) (PIFA) and I_2 was studied. Iodine added to both the C3- and C8-vinyl groups equally to afford the iodohydrin or iodoether in the presence of water or alcohol, respectively. Any *meso*-hydrogen atom was not substituted by an iodine atom under these conditions, although both the vinyl group and one of the *meso* positions of methyl pyropheophorbide-*a* bearing a chlorin π-system, a chlorophyll-*a* derivative, was modified with PIFA and I_2. The reaction intermediates derived from the porphyrin were more reactive than those from the chlorin and liable to form intermolecular linkages. The obtained 2-iodo-1-hydroxyethyl group was transformed into a formyl group by a mild treatment. The corresponding iodoether moiety was readily converted into the acetyl group under basic conditions. These transformations were also applicable to smaller olefins such as styrene.

1. Introduction

Iodination is a key reaction in organic synthesis and medicinal chemistry. Organoiodine compounds are employed in various reactions, including substitution and cross-coupling reactions.[1–3] Furthermore, ioversol, which contains a triiodophenyl group, is used as an X-ray contrast agent for computed tomography. Thyroid hormones thyroxine and triiodothyronine also contain iodophenyl groups. Consequently, new iodination methods with improved applicability, efficiency, and green chemistry are still extensively researched.[4–13]

Natural tetrapyrroles such as chlorophylls (Chls), protoporphyrin IX (PP-IX), and their derivatives have been studied as photosensitizers for photodynamic therapy,[14–16] artificial photosynthesis, and dye-sensitized solar cells.[17–21] Alteration of the C3-vinyl group in Chls is of considerable interest because this group has great influence on the photophysical properties of Chls.[22–28] However, there are few reports on the iodination of the vinyl groups of these tetrapyrroles. Although introduction of ^{131}I to the $C3^2$ and $C8^2$ positions of PP-IX and the pharmacokinetics of this iodinated porphyrin in mice were reported in 1960,[29] details of the pigment were not provided.

Wang et al. reported that the C20 position (one of the *meso* positions) of Chl derivative **1** (Scheme 1) was iodinated using phenyliodine bis(trifluoroacetate) (PIFA) and I_2 while leaving the C3-vinyl group unreacted.[30] Furthermore, there are no reports on the transformation of the peripheral iodinated groups derived from natural tetrapyrroles except for our late observation.

We have recently published some reports on the iodination of Chl derivative **1** (Scheme 1).[31–33] It was demonstrated that an iodine atom was selectively introduced at the C3-vinyl group or the C20 position of **1** by simply changing the reaction stoichiometry of PIFA and I_2. Furthermore, iodohydrin **4** was transformed to the epoxide **5** by treatment with ethylenediamine, and **5** was reacted with an acid and $NaIO_4$ to afford 3-formyl-chlorin **6**. Interestingly, the treatment of iodoether **7** with a base converted to **8** bearing the C3-acetyl group, which had, to the best of our knowledge, not been previously reported. These are useful alternatives to previous synthetic routes. For examples, hazardous OsO_4 was previously required to prepare **6** from **1**[34]; direct epoxidation of **1** to **5** with mCPBA is impossible, so the synthesis of **5** was accomplished using the Corey-Chaykovsky reaction of **6**[35]; and tetrapropylammonium perruthenate (TPAP) was required for oxidation of the 1-

* Corresponding author.
E-mail address: tob_p206@cc.utsunomiya-u.ac.jp (T. Oba).

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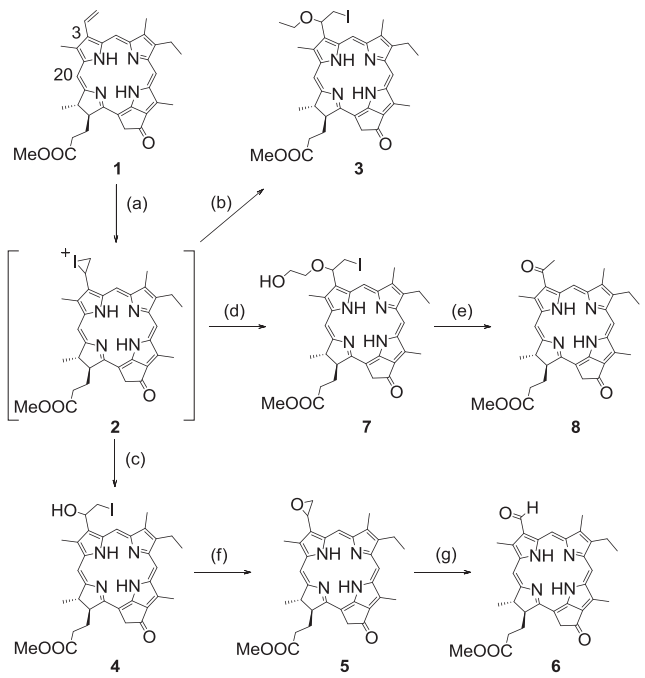


Scheme 1. Conversions of the vinyl group in chlorophyll derivative **1**. (a) PIFA (0.5 eq.), I_2 (1 eq.), 1,2-dichloroethane, r.t.; (b) EtOH, r.t.; (c) morpholine, r.t.; (d) ethylene glycol, r.t.; (e) (i) NaOH, THF/H_2O, r.t., (ii) H_2SO_4/MeOH, r.t.; (f) ethylenediamine, 1,2-dichloroethane, r.t.; (g) TsOH, $NaIO_4$, THF/H_2O, r.t.

hydroxyethyl derivative obtained by hydrobromination and subsequent hydration of **1** to prepare **8**.[36]

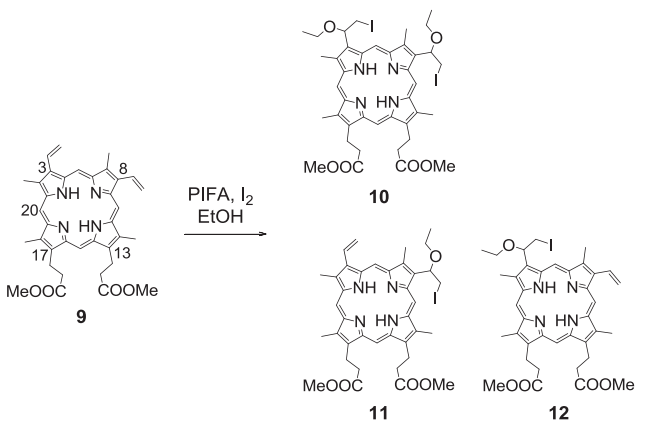
Despite the fact that the iodination of the two vinyl groups in PP-IX would potentially widen the application of cyclic tetrapyrroles and their related compounds, very little has been published on such a transformation. Furthermore, derivatization of the resultant peripheral iodinated groups has not been explored. Based on their structural similarities, we expect that PP-IX would undergo the same reactions that Chl derivatives did. Although their reactivities are not the same. Consequently, herein we report our findings on the iodination of a PP-IX derivative **9** (Scheme 2) as well as the subsequent epoxidation, formylation, and acetylation of the product. Visible absorption and fluorescence spectra of the iodinated porphyrin are presented. Styrene was employed to investigate the scope of these reactions, and a possible mechanism of the acetylation *via* the iodoether is also discussed.

2. Results and discussion

First, we investigated the iodoetherification of the vinyl groups in PP-IX dimethyl ester (**9**) using the homogeneous conditions under which chlorin **1** was found to be iodinated efficiently (Table S1). Accordingly, compound **9** was stirred with PIFA (0.5 eq.) and I_2 (1 eq.) in 1,2-dichloroethane for 1 h at room temperature. Ethanol (EtOH) was then added, and the mixture was additionally stirred for 2 h at room temperature. Unexpectedly, whereas chlorin **1** was successfully iodinated under these conditions, we obtained very little of the iodinated porphyrins **10**, **11**, or **12**. The ^{1}H NMR signals of the products were too broad to be resolved, suggesting oligomerization of **9**. Matrix-assisted laser desorption ionization-time of flight mass spectrometry (MALDI-TOF-MS) analysis indicated the presence of some iodinated porphyrins ($m/z = 763$ and 935) as well as iodinated dimeric and trimeric components (*e.g.* $m/z = 1526$, 1698, 2288, and 2461; Fig. S1). Interestingly, the reaction of **1** did not give dimers and trimers under the same conditions.

We assumed that the iodonium intermediates derived from porphyrin **9** are more reactive than those from chlorin **1**. Consequently, we reacted **9** in the presence of EtOH to immediately trap the unstable intermediates and suppress the oligomerization. The reaction mixture was purified using silica gel column chromatography. The MS spectrum of the black products clearly showed ion peaks with m/z increments of 172 and 344 units, indicating addition of 1 and 2 eq. of EtOI to **9**, respectively. Reaction with double the amount of reagents (1 eq. of PIFA and 2 eq. of I_2) and EtOH afforded the 3,8-di(1-ethoxy-2-iodoethyl) derivative **10** in 80% isolated yield after purification with chromatography.

Fig. 1 shows the ^{1}H NMR spectra of these samples, thus obtained di-iodinated compound **10** and the mixture of **10** and the mono-iodinated porphyrins **11** and **12**. The spectrum for **10** lacks the vinyl proton signals observed for **9**, around 6.15–6.40 ($C3^2$ and $C8^2$) and 8.20–8.30 ppm ($C3^1$ and $C8^1$), while the broadened proton signal at 6.13 ppm (2H) is assignable to the $C3^1$ and $C8^1$ protons in 1-ethoxy-2-iodoethyl group. The two multiplets at 4.10 and



Scheme 2. Iodoetherification of protoporphyrin-IX dimethyl ester (**9**). PIFA (0.5 eq.), I_2 (1.0 eq.), EtOH/1,2-dichloroethane.

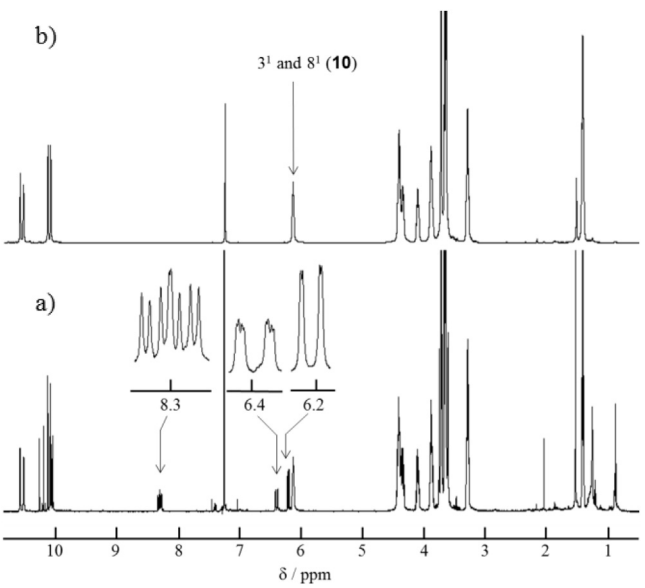


Fig. 1. ^{1}H NMR spectra of (a) **10** and (b) a mixture of **10**, **11** and **12**. The expanded portions show the vinyl proton signals for **11** and **12** in $CDCl_3$.

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4.35 ppm originate from the $C3^2$–H_2 and $C8^2$–H_2 protons, respectively, and the latter overlaps the 13/17-CH_2 proton signals as demonstrated by H,H–COSY analysis. The ^{1}H NMR signals for the ethoxy groups (1.41 and 3.88 ppm) appear somewhat broadened and have complicated fine structures suggesting the sample being a mixture of the $C3^1$ and $C8^1$ epimers. Fig. 1b shows the characteristic resonances from the vinyl groups in the range of 6.10–8.40 ppm accompanied by the proton signals for the 1-ethoxy-2-iodoethyl groups at 4.10, 4.35, and 6.13 ppm. Their peak areas indicate virtually equal yields of regioisomers **11** and **12**. It is also demonstrated that the ratio of the di-iodinated (**10**) to mono-iodinated products (**11** and **12**) in this mixture is roughly 1:1. These results indicate that the reactivities of the C3- and C8-vinyl groups are roughly equal.

Interestingly, the conditions examined in this study afforded no *meso*-iodinated product from **9**. Even though compound **9** was reacted with 3 eq. of PIFA and I_2, the *meso*-positions remained intact. This is in clear contrast to chlorin **1**, which underwent iodination at the C20 position with 1 eq. of PIFA and 2 eq. of I_2. This different reactivity may arise from steric hindrance from the C18-methyl groups as well as electronic effects at the C20 position.[37,38] It is also noted that neither compound **10** nor hematoporphyrin dimethyl ester lacking vinyl groups oligomerized when they were reacted with PIFA and I_2. These results indicate that the oligomerization of **9** occurs primarily through the C3- and C8-vinyl moieties. Fig. S2 shows examples of possible dimeric and trimeric structures assignable to the aforementioned MALDI-TOF-MS peaks. The analysis indicates the reaction of an unreacted vinyl group and an iodonium ion at the early stage of the reaction, to form intermolecular linkages between the C3/8 moieties. The fact that porphyrin **9** afforded oligomers but chlorin **1** did not indicates a difference in the reactivities of the vinyl groups and/or iodonium ions of these tetrapyrroles. These presumed dimers and trimers may have photodynamic activity based on their structural similarity to Photofrin.

Fig. 2 shows the UV–Vis absorption spectrum of the di-iodinated porphyrin **10**. The Soret absorption of **10** presents a broad band peaking at 405 nm, while those of the substrate **9** and hematoporphyrin, a 3,8-devinyl derivative of **9**, are at 407 and 405 nm, respectively. The Q absorption bands are observed at 502, 536, 571, and 624 nm, similar to those of hematoporphyrin (503, 537, 573 and 626 nm), and exhibit blue-shifts of 4–5 nm from those of **9**. The relative intensities of the Q bands are similar to those of **9** and hematoporphyrin.

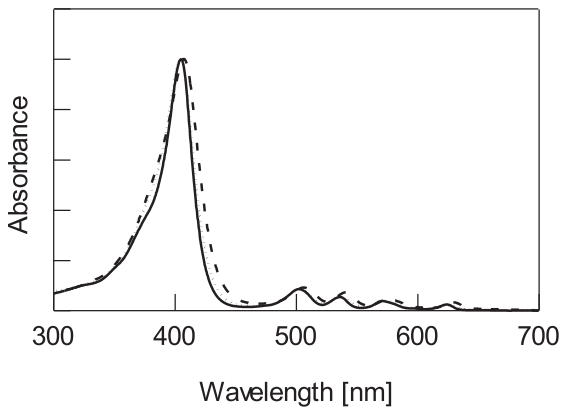


Fig. 2. UV–Vis absorption spectra of hematoporphyrin dimethyl ester (dotted line), **9** (broken line) and **10** (solid line) recorded in $CHCl_3$ at room temperature: normalized at the Soret peaks.

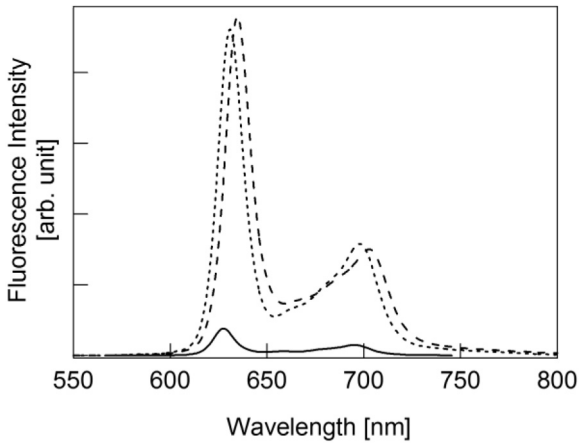
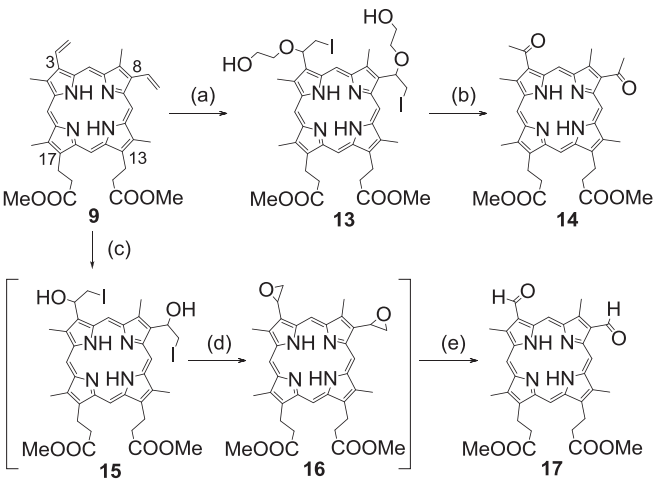


Fig. 3. Fluorescence emission spectra of hematoporphyrin dimethyl ester (dotted line), **9** (broken line) and **10** (solid line) $CHCl_3$ at room temperature (10 μM): excited at each Soret maximum.

The fluorescence spectrum of **10** is shown in Fig. 3. The fluorescence maximum for **10** is observed at 628 nm, while that for **9** and hematoporphyrin are at 635 and 631 nm, respectively. The Stokes shifts are 125 (**9**), 102 (**10**), and 127 cm^{-1} (hematoporphyrin). The band-widths (full width at half maximum) are 388 (**9**), 332 (**10**), and 389 cm^{-1} (hematoporphyrin). The fluorescence intensity of **10** is approximately one-tenth those of **9** and hematoporphyrin.

The iodoether-type porphyrin can be converted into the acetylporphyrin in the same way as chlorin **1** to **8** *via* **7** (Scheme 1). As **10** was obtained by the reaction with EtOH, **13** was afforded by the reaction with ethylene glycol (in 84% yield). Reaction of **13** with aqueous NaOH (Scheme 3) transformed both the iodoether moieties into acetyl groups with simultaneous hydrolysis of the propionic esters at the C13 and C17 positions to give 3,8-diacetyl-3,8-devinyl-protoporphyrin. Treatment of this acid with trimethylsilyldiazomethane affords **14** in 41% yield. It is well known that compound **14** can be prepared from **9** *via* hematoporphyrin.[39] The multi-step reaction for this preparation includes addition of HBr to the vinyl groups, hydrolysis of the bromoethyl groups, and Ley-



Scheme 3. Conversion of the vinyl groups of porphyrin **9**. (a) Ethylene glycol, PIFA (1 eq.), I_2 (2 eq.), 1,2-dichloroethane, r.t.; (b) (i) NaOH, THF/H_2O, r.t., (ii) TMS-CHN_2, MeOH, $CHCl_3$, r.t.; (c) PIFA (2 eq.), I_2 (1 eq.), THF/H_2O, r.t.; (d) ethylenediami-ne, 1,2-dichloroethane, 50 °C; (e) TsOH, $NaIO_4$, THF/H_2O, r.t.

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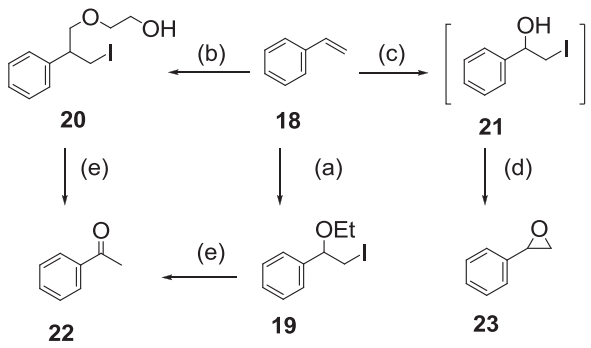


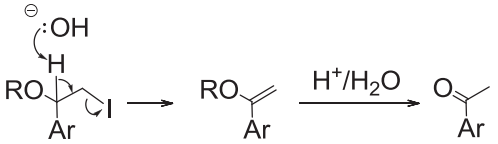
Scheme 4. Conversion of styrene through iodination of the vinyl group. (a) (i) PIFA (0.5 eq.), I_2 (1 eq.), 1,2-dichloroethane, r.t., (ii) EtOH; (b) PIFA (0.5 eq.), I_2 (1 eq.), 1,2-dichloroethane, r.t., (iii) ethylene glycol; (c) PIFA (0.5 eq.), I_2 (1 eq.), THF/H_2O, r.t.; (d) ethylenediamine, 1,2-dichloroethane, 50 °C; (e) NaOH, THF/H_2O, reflux or ^{t}BuOK, ^{t}BuOH, reflux.

Griffith oxidation using TPAP. Our new protocol is an inexpensive alternative to this popular sequence.

Like the transformation of chlorin **1** to **6** *via* **4** (Scheme 1), porphyrin **9** can be transformed into the 3,8-diformyl derivative **17** *via* iodination (Scheme 3). However, the procedure for the porphyrin required some tuning. Although the vinyl group of chlorin **1** was successfully converted into a 1-hydroxy-2-iodoethyl group by treatment of the iodonium intermediate with morpholine in 1,2-dichloroethane, iodohydrin **15** was not obtained from porphyrin **9** under the same conditions. Addition of water (20 vol %) to the system did not result in this conversion but gave oligomerization of **9**. However, when the solvent was replaced with aqueous THF, **15** was successfully produced, as confirmed from ^{1}H NMR and MS spectra. Mixing obtained **15** with ethylenediamine in 1,2-dichloroethane, followed by stirring at room temperature gave the di-epoxy derivative **16** accompanied by intermediates that contained both epoxy and 1-hydroxy-2-iodoethyl moieties. Raising the reaction temperature to 50 °C accelerated the reaction to afford **16** as the major product. Since compound **16** was not sufficiently stable to be isolated using column chromatography, crude **16** was used without purification in the reaction with *p*-toluenesulfonic acid (TsOH) and $NaIO_4$. Compound **17** is successfully obtained by this greener method without using OsO_4 previously reported.[40,41]

As shown in Scheme 4, the present vinyl group iodination protocol as well as the conversions *via* this iodination are also applicable to smaller olefins such as styrene (**18**).[6–11] Compound **18** was dissolved in 1,2-dichloroethane and stirred with 0.5 eq. of PIFA and 1.0 eq. of I_2 for 1 h at room temperature. To this system was added alcohol, and the mixture was stirred for additional 2 h. This procedure effected iodoetherification of **18** in 80% and 77% yields for EtOH (**19**) and ethylene glycol (**20**), respectively. Neither oligomerization nor substitution at the aromatic ring were observed. Essentially the same procedure in aqueous THF gave iodohydrin **21**, and subsequent treatment with ethylenediamine afforded styrene oxide (**23**, 72%). Benzaldehyde can be prepared from styrene oxide by reaction with TsOH and $NaIO_4$.[42] These reactions are also applicable to 1-allyl-4-methoxybenzene (data not shown).

The iodoether moieties obtained from styrene can also be converted into acetyl groups. Iodoether **20** was dissolved in aqueous THF, followed by addition of aqueous NaOH. The reaction mixture was refluxed for 4 h to give acetophenone (**22**) in 86% yield, while **19** remains unreacted after stirring with aqueous NaOH overnight at room temperature. Both **19** and **20** were also converted into **22** by refluxing with ^{t}BuOK in ^{t}BuOH. These results demonstrate that this acetylation is not restricted to porphyrins and chlorins but is also applicable to the standard olefins. We speculate that the acetylation proceeds *via* the enolether (Scheme 5). Abstraction of the benzyl proton triggers elimination of an iodide ion, and hydrolysis of the resulting enolether gives an acetyl group.



Scheme 5. Possible mechanism for acetylation of the iodoether.

3. Conclusion

The vinyl groups of PP-IX dimethyl ester were iodinated with PIFA and I_2 to yield iodoethers and iodohydrins. The reactivities of the C3- and C8-vinyl groups were equal. The *meso*-hydrogen atoms of the porphyrin were not substituted by iodine atoms, while both the vinyl groups and the *meso* (C20) position of the chlorin reacted under similar conditions. The reaction intermediates of the porphyrin were more reactive than those of the chlorin, which afforded intermolecularly linked porphyrins. These results demonstrate the differences in the reactivities of the vinyl groups, the *meso* positions, and/or the iodination intermediates of these tetrapyrroles. The obtained iodohydrin and iodoether moieties of the porphyrin were readily converted into epoxy, formyl, or acetyl groups. Styrene was transformed into styrene oxide, benzaldehyde, or acetophenone without aromatic substitution nor oligomerization *via* similar procedures. This study demonstrates the uniqueness of porphyrins and the versatility of the reactions presented, and may facilitate the development of new strategies for the application of tetrapyrroles.

4. Experimental

4.1. General

Chl-*a* was extracted from *Spirulina* and used as a material to prepare compound **1** as described previously.[43,44] The other reagents were purchased from Kanto Chemical Co., Inc., Tokyo Chemical Industry Co., Ltd., or Wako Pure Chemical Industries Ltd., and used as provided. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were recorded on a Varian VNMR-500. Chemical shifts are reported in ppm with reference to tetramethylsilane. MALDI-TOF-MS and high-resolution mass spectrometry-atmospheric-pressure chemical ionization (HRMS-APCI) spectra were measured using a Bruker autoflex II and a Bruker micrOTOF II, respectively.

Synthesis of new compounds **10** and **13** were shown below and please see the preparation of known compounds **9**, **14**, **17**, **19**, **20**, **22** and **23** in supporting information.

4.2. Synthetic procedure

4.2.1. 3,8-Di(1-ethoxy-2-iodoethyl)deuteroporphyrin IX dimethyl ester (*10*)

Compound **9** (250 μmol, 148 mg) was dissolved in 1,2-dichloroethane (100 mL), followed by addition of EtOH (10 mL). PIFA (1 eq., 108 mg) and I_2 (2 eq., 127 mg) were added to the solution, and the mixture was stirred for 2 h at room temperature. The mixture was washed with 10 wt% $Na_2S_2O_3$ aq. and brine. The

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organic phase was dried over Na_2SO_4, followed by evaporation to dryness. The residue was purified by silica gel column chromatography ($CHCl_3$/ethyl acetate, 10:1) to isolate **10** in 80% yield (200 μmol, 187 mg): ^{1}H NMR (the C3^1, C8^1-epimeric mixture, $CDCl_3$, 500 MHz) δ 10.57, 10.52, 10.12, 10.07 (4s, 4H, *meso*), 6.15–6.09 (m, 2H, CH–3^1, 8^1), 4.44–4.35 (2 m, 4H, CH_2–13^1, 17^1), 4.40–4.30, 4.13–4.05 (2 m, 4H, CH_2–3^2, 8^2), 3.93–3.82 (m, 4H, OCH_2CH$_3$), 3.73–3.61 (6s, 18H, CH_3–2^1, 7^1, 12^1, 18^1, $COOCH_3$), 3.33–3.24 (2 m, 4H, CH_2–13^2, 17^2), 1.44–1.38 (m, 6H, OCH$_2$*CH$_3$*), −3.67 (br s, 2H, NH); ^{13}C NMR (the C3^1, C8^1-epimeric mixture, $CDCl_3$, 125 MHz) δ 173.67, 173.64, 98.92, 98.40, 97.39, 96.58, 78.00, 77.97, 65.82, 65.79, 51.90, 51.87, 37.01, 36.98, 21.96, 15.62, 15.59, 12.29, 12.27, 11.97, 11.84, 11.05, 10.97, 10.95, 10.94. (The alpha and beta carbons of the pyrrole units were not detected at 25 °C.[45] However, these signals are partially observrd in the range of 135–155 ppm by variable temperature NMR experiment and HMBC. See Fig. S6); HRMS (APCI) found: *m/z* 936.1763, calcd. for $C_{40}H_{48}I_2N_4O_6$: MH^+, 936.1769; Mp > 300 °C.

4.2.2. 3,8-Di[1-(2-hydroxyethyloxy)-2-iodo]ethyldeuteroporphyrin IX dimethyl ester (*13*)

Compound **9** (250 μmol, 148 mg) was dissolved in 1,2-dichloroethane (100 mL). Ethyleneglycol (10 mL), PIFA (1 eq., 108 mg) and I_2 (2 eq., 127 mg) were added to the solution. The mixture was stirred for 2 h at room temperature. The mixture was then washed with 10 wt% $Na_2S_2O_3$ aq. and brine. The organic phase was dried over Na_2SO_4, and the solvent was removed *in vacuo*. The residue was purified by silica gel column chromatography (1,2-dichloroethane/acetone, 10:1). Yield, 84% (210 μmol, 203 mg); ^{1}H NMR (the C3^1, C8^1-epimeric mixture, $CDCl_3$, 500 MHz) δ 10.48, 10.42, 10.10, 10.06 (4s, 4H, *meso*), 6.21–6.14 (m, 2H, CH–3^1, 8^1), 4.44–4.37 (2 m, 4H, CH_2–13^1, 17^1), 4.39–4.32, 4.10–4.03 (2 m, 4H, CH_2–3^2, 8^2), 4.00–3.86 (m, 8H, OCH_2CH_2OH), 3.73–3.61 (s, 18H, CH_3–2^1, 7^1, 12^1, 18^1, $COOCH_3$), 3.31–3.24 (2 m, 4H, CH_2–13^2, 17^2), −3.67 (br s, 2H, NH); ^{13}C NMR (the C3^1, C8^1-epimeric mixture, $CDCl_3$, 125 MHz) δ173.48, 173.46, 98.44, 97.83, 97.45, 96.70, 78.39, 78.37, 78.34, 78.32, 71.50, 62.17, 62.15, 51.79, 51.75, 36.81, 36.79, 21.79, 21.77, 12.17, 12.14, 12.04, 11.85, 11.68, 10.60, 10.51, 10.50. (The alpha and beta carbons of the pyrrole units were not detected at 25 °C.[45] However, these signals are partially observrd in the range of 135–155 ppm by variable temperature NMR experiment and HMBC. See Fig. S10); HRMS (APCI) found: *m/z* 967.1621, calcd. for $C_{40}H_{48}I_2N_4O_8$: MH^+, 967.1634; Mp > 300 °C.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.tet.2018.05.040.

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