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Solid-phase organic synthesis of 2-tridecanyl-1,4-naphthoquinone and 2-tridecanyl-1,4-naphthodiol that form redox-active micelles and vesicles

Alejandro Bugarin [a,*], Luis E. Martinez [b], Peter Cooke [c], Tadiqul Islam [d], Juan C. Noveron [d]

[a] Department of Chemistry and Biochemistry, University of Texas at Arlington, Arlington, TX 76019, USA
[b] Department of Chemistry and Biochemistry, Trinity University, San Antonio, TX 78212, USA
[c] CURRL, New Mexico State University, 945 College Dr., Las Cruces, NM 88003, USA
[d] Department of Chemistry, University of Texas at El Paso, El Paso, TX 79968, USA

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ABSTRACT

The solid-phase synthesis of new amphiphilic compounds is reported. It is based on a newly designed 1,4-naphthoquinone derivative that contains polar and nonpolar groups and self-assembles into micelles or vesicles in water depending on the concentration. They also display redox-active properties.

1. Introduction

Naphthoquinones and their derivatives continue to attract the attention of the chemical community and have been the subject of several studies [1]. This interest stems from the fact that many naturally occurring compounds contain the naphthoquinone skeleton and possess a variety of biological and pharmacological activities that include antibacterial, antifungal and anticancer, among others [2]. Their biological activity arises primarily from the redox active quinone that interferes with biochemical oxidation processes [3], particularly microsomal lipid peroxidation. In view of their potential use in biology and medicine, a myriad of derivatives of naphthoquinones have been reported and recently reviewed [4]. However, many derivatives of these compounds are insoluble in water and require the addition of co-solvents such as DMSO to generate aqueous solutions suitable for biological applications.

Another common strategy for their aqueous administration is to introduce hydrophilic groups into the core structure, where this approach has a significant impact on the bio-distribution and activity of such derivatives. Herein, we report the solid-phase organic synthesis (SPOS) [5] of novel amphiphilic naphthoquinones that posses a single hydrocarbon chain at C-2 and a chemically switchable head-group. In addition, these compounds are able to emulsify into micelles or vesicles in water depending on the concentration. Our solid-phase strategy serves as a key method for the incorporation of functional groups on naphthoquinones. It is well known that 1,4-naphthoquinones derivatives have a broad spectrum of biological activities [6] such as; antimycobacterial [7], antiparasitic [8], and cytotoxic activities [9]. This strategy has the potential to generate large libraries with varied biological activity by small changes on the naphthoquinone backbone or at the hydrocarbon chain [10].

The previously reported Fischer carbene complex approach [11], using solid-phase organic synthesis (FCC-SPOS), was employed to synthesize our naphthoquinones [12]. This method is based on immobilized Fischer carbene complexes, which undergo regioselective Dötz benzannulation [13] to afford naphthoquinones with predetermined substitutions [12]. This approach also allows for the formation of single benzannulation adducts from alkynes, which is an added advantage since in the absence of the solid support, the reaction could generate several regioisomeric products and would require extensive and difficult purification.

2. Results and discussion

2.1. Synthesis

The synthesis of amphiphilic naphthoquinone **8** started from commercially available phenyl lithium and chromium hexacarbonyl

* Corresponding author. Fax: +1 817 272 3808.
E-mail address: bugarin@uta.edu (A. Bugarin).

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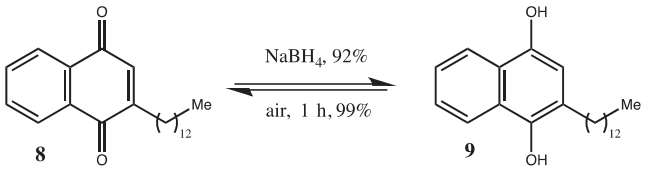
using a modified method originally developed by Connor et al. [14]. Thus, the polymer-supported Fischer carbene complex **5** was obtained in four steps by first reacting phenyl lithium and chromium hexacarbonyl, which produced adduct **2** that then underwent an ion exchange to the corresponding tetramethyl-ammonium-[(2-phenyl)oxidocarbene]pentacarbonyl-chromium **3** (Scheme 1). *O*-acylation of **3** with acetyl chloride was followed by reaction with the PL-Wang resin (Polymer Laboratories, 1% cross-linked 1.7 mmol/g) to produce resin-bound Fischer carbene complex **5** with 100% loading. The resin loading of the carbene complex can be easily monitored qualitatively by colorimetric analysis; the beads turn dark red in color, and appearance of characteristic Cr–CO stretches at 2060 and 1933 cm^{-1} in the IR spectrum of **5**, that correspond to the CO stretches found in analogous aryl carbene complexes, were observed [15]. With the polymer-supported Fischer carbene complex **5** on hand, the key Dötz benzannulation reaction was performed with 1-pentadecyne **6** under optimized conditions [12] (microwave-assisted) [16] and provided the amphiphilic naphthoquinone **8** after oxidative cleavage of the resulting resin-bound naphthol **7** in overall good yield (63%). It is noteworthy that no side products were observed, due to the employment of the solid support strategy. Finally, sodium borohydride reduction of **8** in methanol/THF produced the corresponding of 2-tridecanyl-1,4-naphthodiol **9** in 92% yield. (See the Section 4 for more details.)

Compound **9** is very sensitive to oxidation under air. It was observed by ^1H NMR and other characterization methods a promptly oxidation to compound **8** when adduct **9** is left open to the atmosphere (Scheme 2).



Scheme 2. Easy access to compounds **8** and **9**.

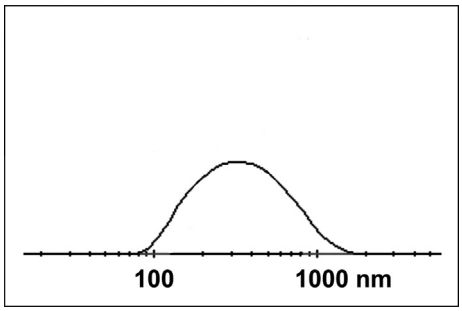


Fig. 1. Dynamic Light Scattering (DLS) of **8** at 1% wt.

2.2. Self-assembly in water

The self-assembly properties of the resulting amphiphilic naphthoquinones were studied in water at various concentrations. Aqueous solutions of compound **8**, at concentrations of 1%, wt, were sonicated at room temperature for 30 min, which formed stable emulsions. Dynamic Light Scattering (DLS) measurements reveled vesicles with hydrodynamic Z-average diameter of 292.5 nm and a polydispersity index (PDI) of 0.231, as depicted in (Fig. 1).

Atomic Force Microscopy (AFM) of the aqueous emulsions of **8** confirmed the presence of vesicles at concentration of 1% wt and revealed the spherical structure of the vesicles (Fig. 2).

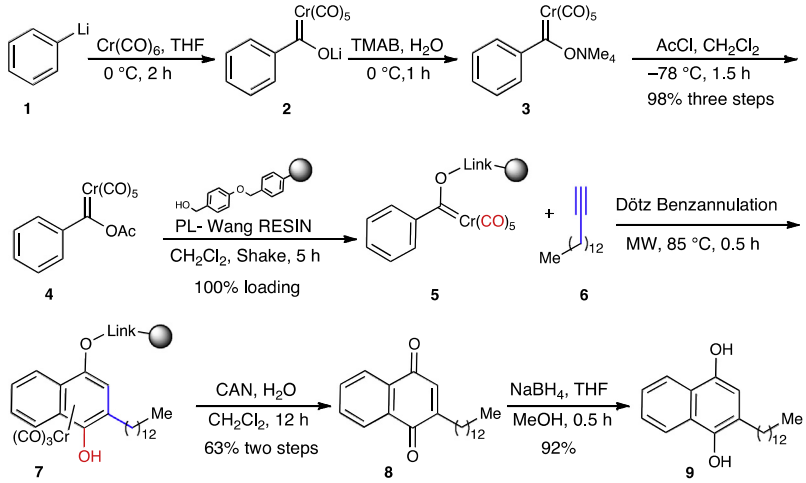
Upon dilution to <0.1% wt, emulsions of **8** formed micellar structures of 2 nm in diameter, according to AFM (Fig. 3). This behavior is consistent with earlier observations that amphiphiles with high-volume head groups have a preference to organize into micelles due to their conical shape [17]. At this concentration, the signal-to-noise ratio impeded DLS measurements of the micelle dispersion.

Optical microscopy of the emulsion of compound **8** showed several bilayer structures, characteristic of vesicles (Fig. 4, left). The aqueous emulsion of **8** was treated with the phenoxazine dye Nile red indicator, a fluorescent indicator used to localize lipid phases within membranes, and optical fluorescent spectroscopy revealed bilayer structures typical of vesicles (Fig. 4, right) [18].

2.3. Redox-activity

The electrochemical properties of the amphiphilic naphthoquinone **8** were then studied by cyclic voltammetry [19]. Measurements of **8** in dichloromethane revealed a quasi-reversible redox potential of 0.504 V (Fig. 5), suggesting that the redox active properties are retained in the molecule.

In order to investigate if the redox-activity of the amphiphilic naphthoquinone is retained while they exist as emulsions, we prepared emulsions of **9** and react them with an aqueous iodine



Scheme 1. Solid-phase synthesis of 2-tridecanyl-1,4-naphthoquinone **8** and its reduction to 2-tridecanyl-1,4-naphthadiol **9**.

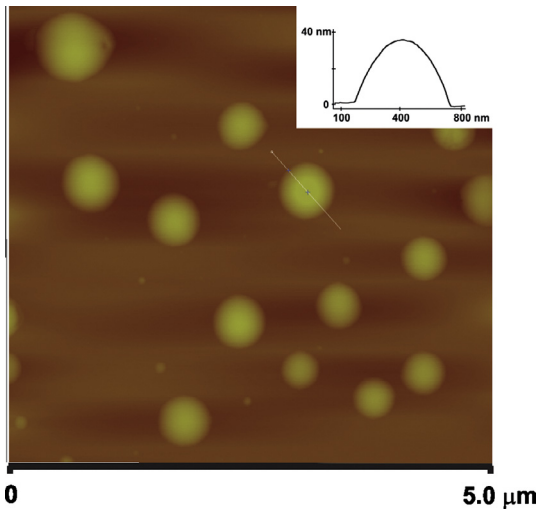


Fig. 2. AFM of vesicles of **8** on mica. Inset: vertical analysis of selected vesicles.

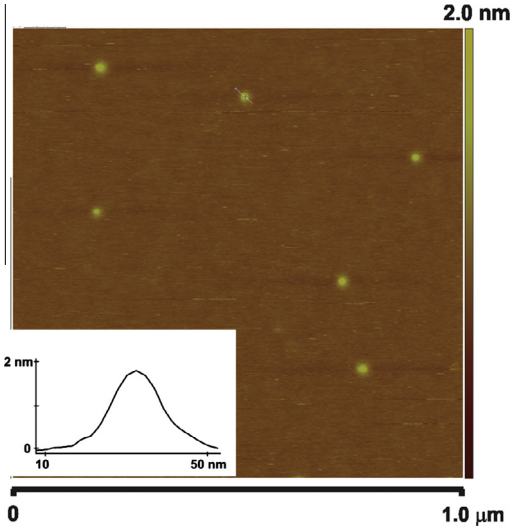


Fig. 3. ATM of micelles of **8** on mica. Inset: vertical analysis of selected micelles.

solution (484 mM). Using UV–Vis spectroscopy, we observed the immediate decrease in absorbance of the iodine band (λ_{max} = 350 nm), which has been used to follow iodine chemistry in water [20]. This clearly indicates that the vesicles are redox active in water and are capable of reducing iodine molecules (Fig. 6). In total, 6.5 equivalents of compound **9** vesicles were required to completely quench the iodine solution band. This is

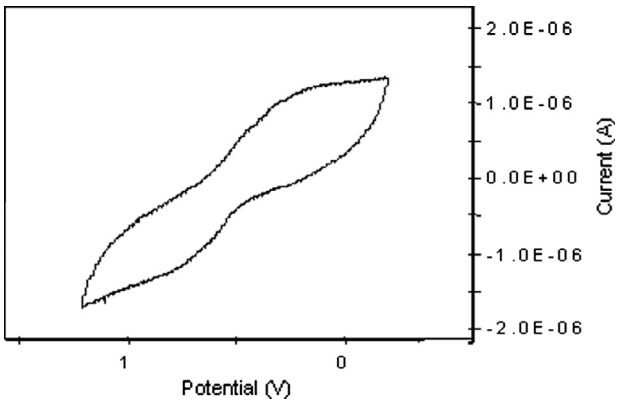


Fig. 5. Cyclic voltammetry of **8**.

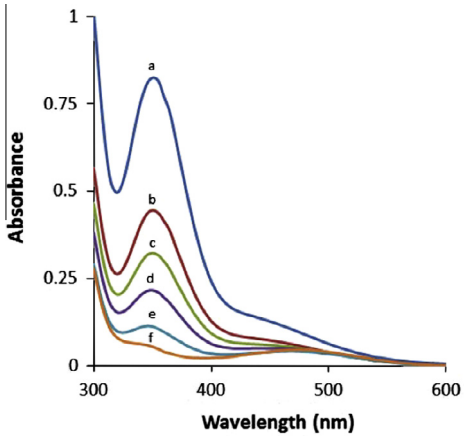


Fig. 6. (a) UV–Vis spectrum of 484 mM iodine in water, titrated with (b) 1.3, (c) 2.6, (d) 4.0, (e) 5.2, (f) 6.5 equiv. of vesicles of compound **9**.

more than the theoretical 1:1 molar ratio. Presumably, this excess results from the known aerobic oxidation of compound **9** to compound **8** (Scheme 2). The acquired reduction data suggest an iodine-water redox process associated with our amphiphilic molecules.

3. Conclusions

A concise solid-support synthesis and characterization of 2-tridecanyl-1,4-naphthoquinone (**8**) and 2-tridecanyl-1,4-naphthadiol (**9**) are reported. These molecules self-assemble into micelles or vesicles in water depending on the concentration. They also exhibit redox-active properties in water. The approach used for the

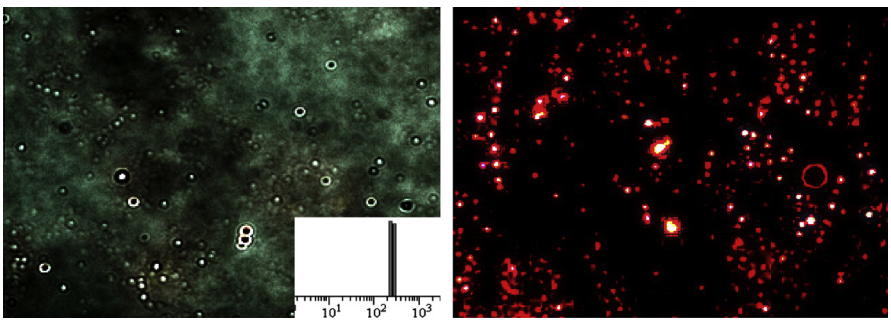


Fig. 4. Optical microcopy of vesicles of **8** before (a) and after (b) staining with Nile red indicator. Inset: DLS distribution of **8**.

synthesis of these amphiphilic molecules can be easily applied to the synthesis of naphthoquinone-derivative libraries that present new functional groups on the surface of vesicles. These outstanding properties (vesicles formation together with their redox potential) of the new synthesized adducts **8** and **9**, will aid as a platform for an easier drug delivery strategy *via* vesicles under aqueous media. Likewise, studies of naphthoquinones bioactivities might be performed using our strategy.

4. Experimental section

4.1. General considerations

All reactions were carried out under an oxygen free atmosphere in either oven-dried glassware with magnetic stirring or microwave resistant vials. All commercially obtained reagents were used as received. Solvents were obtained from a solvent purification system. Heating was accomplished by either; a heating mantle, silicone oil bath, or a microwave apparatus. Cooling was performed using ice bath or acetone/dry-ice bath. Purification of reaction products was carried out by flash column chromatography using silica gel if necessary. TLC visualization was accompanied with UV light and/or potassium permanganate staining. ^{1}H NMR spectra were recorded at 300 MHz, and are reported relative to $CDCl_3$ (δ 7.27). ^{1}H NMR coupling constants (J) are reported in Hertz (Hz) and multiplicities are indicated as follows: s (singlet), d (doublet), t (triplet), m (multiplet). Proton-decoupled ^{13}C NMR spectra were recorded at 75 MHz and reported relative to $CDCl_3$ (δ 77). UV–Vis spectra were recorded using the Agilent Cary 50 Conc. UV–Vis spectrophotometer, whereas for DLS analysis the Zetasizer Nano ZS90 instrument was used.

4.2. Synthesis of O-acylated Fischer carbene complex (**4**)

Chromium hexacarbonyl (3.00 g, 13.6 mmol) and dry THF (20.0 mL) were placed in a 100 mL two-necked round bottom flask under N_2 atmosphere. The flask was cooled to 0 °C and a solution of phenyllithium **1** (20.5 mmol, 1.9 M in cyclohexane-ether, 10.8 mL) was slowly added over a period of 20 min and allowed to stir for 2 h. The solvent was removed under vacuum and the resulting orange red residue (**2**) was added to a solution of tetramethyl ammonium bromide (4.19 g, 27.2 mmol) in 20.0 mL of oxygen-free water. The reaction mixture was allowed to stir at 0 °C for 2 h. The product was extracted twice with CH_2Cl_2 (50 mL) and the combined organic phases were dried over anhydrous $MgSO_4$. The solvent was concentrated under reduce pressure to afford **3** as a red solid. To a stirred solution of crude red solid **3** (4.64 g, 12.5 mmol) in 10.0 mL of CH_2Cl_2 at −78 °C, acetyl chloride (1.27 g, 16.3 mmol) was added. After stirring at 0 °C for 1.5 h, the reaction mixture was allowed to warm to room temperature. The solvent and the unreacted acetyl chloride were removed under reduced pressure to yield (4.54 g, 13.4 mmol) of the *O*-acetylated adduct **4** in 98% yield as a bright red solid.

4.3. Synthesis of resin-bound Fischer carbene complex (**5**)

All the bright red solid **4** was diluted with 20.0 mL of CH_2Cl_2 and this solution was transferred via cannula to a 50.0 mL fritted funnel (solid-phase peptide synthesizer) containing poly-styrene PL-Wang resin (1.47 g, 2.5 mmol–1.7 mmol/g specified by manufacturer). The reaction mixture was shaken on a wrist shaker at room temperature for 3 h, after which the mixture was filtered. Then, the resin was washed sequentially with CH_2Cl_2 (50 mL), THF (25 mL), and CH_2Cl_2 (25 mL) and dried under vacuum to constant weight to give 2.28 g (3.88 mmol) of the resin-bound complex **5** as a red solid. IR (KBr) ν 2060, 1933 cm^{-1}. (Found: Cr, 5.99%. 100% loading @ 1.7 mmol/g requires Cr, 5.99%.)

4.4. Synthesis of 2-tridecylnaphthalene-1,4-dione (**8**)

To a microwave process vial (10.0 mL), resin **5** (500 mg, 0.575 mmol) was added and the vial was sealed with an aluminum/Teflon crimp top. Then, a solution of 1-pentadecyne **6** (2.875 mmol) in dry CH_2Cl_2 (5.0 mL) was added under Nitrogen atmosphere. The reaction mixture was subjected to microwave irradiation (Biotage EmrysTM Optimizer—300 W maximum power) at 85 °C for 30 min. The reaction mixture was filtered and the resin was washed sequentially with CH_2Cl_2 (50 mL), THF (25 mL), and CH_2Cl_2 (25 mL) and dried under reduce pressure to afford resin-bound naphthol **7**. Resin-bound naphthol **7** (0.575 mmol) was suspended in a mixture of 9.0 mL of CH_2Cl_2 and ceric ammonium nitrate (1.575 g, 2.875 mmol) in 3.0 mL of water. The resulting suspension was stirred for 12 h and then filtered through a fritted glass funnel. The resin was washed with water (15.0 mL) and CH_2Cl_2 (15.0 mL). The resulting clear solution was washed with 10% NaOH (15 mL) and water (10.0 mL). The organic layer was dried over $MgSO_4$ and filtered. The solvent was removed under vacuum to afford 63% of pure 1,4-naphthoquinone **8** (112 mg, 0.329 mmol) as pale yellow solid; IR (thin film) ν 2956, 2918, 2852, 1663, 1623, 1595, 1337, 1247, 788 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.06 (m, 2H), 7.72 (m, 2H), 6.78 (s, 1H), 2.56 (t, *J* = 7.01 Hz, 2H), 1.57 (quintet, *J* = 6.90 Hz, 2H), 1.25 (s, 20H), 0.87 (t, *J* = 6.51 Hz, 3H). 13C NMR ($CDCl_3$, 75 MHz) δ 185.2, 151.9, 134.6, 133.6, 133.5, 126.5, 125.9, 31.8, 30.02 29.8, 29.63, 29.6, 29.58, 29.54, 29.48, 29.34, 29.32, 29.07, 28.94, 28.9, 27.9, 22.6, 14.1. HRMS (ESI−) calcd for $C_{23}H_{31}O_2$ requires *m*/*z* 339.2330, found 339.2326.

4.5. Synthesis of 2-tridecylnaphthalene-1,4-diol (**9**)

To a mixture of $NaBH_4$ (14 mg, 0.365 mmol) in dry MeOH (2.5 mL) was added salt a solution of 2-tridecylnaphthalene-1,4-dione **8** (50 mg, 0.147 mmol) in dry MeOH (2.5 mL) at room temperature under argon. The reaction mixture was stirred at room temperature until completion (0.5 h). Then, the reaction mixture was quenched using oxygen free water (15 mL) under argon. Extracted with degassed chloroform and dried with Na_2SO_4 under argon. The solution was concentrated under reduce pressure, to afford pure compound **9** (47 mg, 92% yield) as white solid; IR (thin film) ν 3247, 2920, 2850, 1386, 1204, 949, 756 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.09 (t, *J* = 9.08 Hz, 2H), 7.44 (quintet, *J* = 7.46 Hz, 2H), 6.62 (s, 1H), 2.67 (t, *J* = 7.61 Hz, 2H), 1.63 (quintet, *J* = 8.37 Hz, 2H), 1.24 (s, 20H), 0.87 (t, *J* = 6.85 Hz, 3H). 13C NMR ($CDCl_3$, 75 MHz) δ 145.5, 125.6, 124.5, 121.7, 121.1, 110.4, 31.8, 30.2, 30.1, 29.7, 29.62, 29.58, 29.57, 29.4, 29.3 (6C), 29.2, 22.6, 14.1. HRMS (ESI) calcd for $C_{23}H_{34}O_2$ requires *m*/*z* 342.2559, found 341.2557.

4.6. Procedures to micelles and vesicles formation

4.6.1. Preparation of micelles/vesicles

In a 50 mL round-bottom flask, 40.0 mg of compound **8** were treated with distilled water (20 mL) and sonicated to obtain a turbid vesicle dispersion. This dispersion was analyzed using normal conditions and standards for Dynamic Light Scattering. Different dilutions of compound **8** were performed to obtain vesicles or micelles. Upon dilution to <0.1% wt, emulsions of **8** formed micellar structures of 2 nm in diameter, according to AFM.

*4.6.2. Preparation of vesicles from compound (**9**)*

41.5 mg of **9** were treated with distilled water (20 mL) and sonicated to obtain a turbid vesicle dispersion. This dispersion was used to titrate iodine as describe below.

4.6.3. Preparation of stock iodine solution

First, a saturated iodine solution was prepared by sonication of excess iodine in water for 30 min. To a 1.0 mL of the freshly prepared saturated iodine solution were added 5 mL of distilled water to afford our stock iodine solution (484 mM). Once iodine is dissolved into water, it establishes the following equilibrium.

$$\mathbf{I_2 + H_2O \rightleftharpoons OI(\overset{\ominus}{aq}) + 2H(\overset{\oplus}{aq}) + I(\overset{\ominus}{aq})}$$

Since UV–Vis spectra cannot be accurate acquired on dispersions, the vesicles and the iodine mixture were filtered through 0.45 μm pore size PTFE syringe filter before the measurement and the data is presented in Fig. 6.

Acknowledgments

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.bioorg.2014.06.005.

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