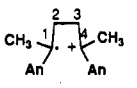
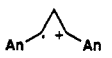
propane in 1,3-dichloroethane.[2a]



C, λmax 520 nm



D, λmax 580 nm

The absorption maximum of D was 580 nm and was quite different from that of the corresponding 1,4-analogue A, λ_{max} 505 nm. This means that both terminal sites of the acyclic radical cation are electronically interacting with each other and the degree of the interaction seems to depend on the chain length.

Introduction of two methyl substituents at C2 and C3 positions of A induces the destabilization, and the 1,4-radical cation B is no longer detectable. This also indicates that the mutual interaction of the terminal sites is reduced by the methyl substituents probably due to the electronic and steric effects. The effects induce the destabilization of the species. On the contrary, the two methyl substituents enhance the stability of the corresponding cyclic structure by reducing an electron deficiency of the C1–C4 bond of $5^{\bullet+}$, and the monomer radical cation $4^{\bullet+}$ is also stabilized by the methyl substituent. Thus, the lifetime of B becomes very short, if formed, which supports the previous observation that the cyclodimerization and cycloreversion of this series may proceed by a quasi-concerted mechanism.

Acknowledgment. This work was supported in part by a grant from the research program on "Creation of New Materials through Intelligient Design" of ISIR, Osaka University. We are grateful to the members of the Radiation Laboratory of ISIR for running the linear accelerator.

Registry No. 1, 637-69-4; $1^{\bullet+}$, 135639-41-7; 1 dimer cation, 135658-80-9; 2, 52498-14-3; $2^{\bullet+}$, 135684-17-2; 3, 52498-15-4; $3^{\bullet+}$, 135684-18-3; 4, 4180-23-8; $4^{\bullet+}$, 117467-10-4; 5, 19043-23-3; $5^{\bullet+}$, 112246-69-2.

Synthesis of Naphtho[*f*]ninhydrin

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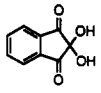
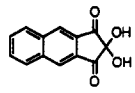
In 1910, Ruhemann discovered ninhydrin (1) and recognized its reaction with α-amino acids to form a purple-blue product known as Ruhemann's Purple.[1] Ninhydrin soon became widely used as a universal reagent for the analysis of α-amino acids in biochemical studies. In 1954, Oden and von Hofsten reported the use of ninhydrin for the development of latent fingerprints via reaction with the α-amino acids of palmar sweat residue, which comprises latent prints.[2] Since the 1960s, ninhydrin has become the most widely used reagent for the development of latent prints on porous surfaces (mainly paper). However, contrast and visualization of weak fingerprints, particularly on some surfaces such as paper and cardboard, are often unsatisfactory. More recently it was found that treatment of ninhydrin-developed prints with zinc chloride forms a coordination compound that is highly fluorescent under blue-green excitation, typically from an argon-ion laser.[3-5]

Since the early 1980s, ninhydrin analogues have been investigated as alternatives to ninhydrin, both in the conventional fingerprint detection mode and for laser detection after zinc chloride treatment.[7-9] Benzo[*f*]ninhydrin (2) was found to offer several advantages over ninhydrin for the fluorescence detection of latent fingerprints[8] and a convenient synthesis of benzo[*f*]ninhydrin has appeared.[10]

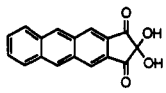
Another ninhydrin analogue with excellent potential as a fingerprint reagent[11] is the unknown compound naphtho[*f*]ninhydrin (3). Previous attempts to prepare 3 have been unsuccessful.[12,13] We now report the synthesis of this elusive compound.



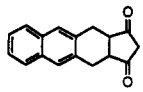
1



2



3



4

5

Results and Discussion

Our initial attempt to prepare naphtho[*f*]ninhydrin (3) involved an adaptation of the method published by Heffner, Sarafyn, and Joullié[10] for the synthesis of benzo[*f*]ninhydrin (2). In their method, the three-ring skeleton of 2 was conveniently constructed by ultrasonication of 1,2-bis(bromomethyl)benzene with activated zinc metal and 1,4-cyclopentadiene in dioxane.[10] Although we were able to repeat the reported cyclization, even extended ultrasonication of 2,3-bis(bromomethyl)naphthalene[14] under the same conditions failed to produce the desired tetracyclic Diels–Alder adduct 4. For another Diels–Alder cyclization, a much lower adduct yield was reported when an *o*-xylylene intermediate was replaced with the annulated analogue 5.[15]

Attention was then shifted to the preparation of unknown 2,3-trimethyleneanthracene (8), a potential precursor to 3 by oxidation. Friedel–Crafts acylation of indan with phthalic anhydride gave 75–90% yields of 6 (Scheme I), which was cyclized with fuming sulfuric acid to provide 55–60% yields of the substituted anthraquinone 7. Reduction of 7 to substituted anthracene 8 was accomplished in 72% yield with aluminum cyclohexoxide in cyclohexanol. Unfortunately, hydrocarbon 8 was unaffected by selenium dioxide in refluxing dioxane[16] and was transformed back into the substituted anthraquinone precursor

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0022-3263/91/1956-6243$02.50/0

7 by the action of pyridinium chlorochromate (PCC). Reaction of 8 with 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ), a reagent that has been shown to give good yields of ketones from cycloalkyl-substituted aromatic polycyclic hydrocarbons,[17] gave an inseparable mixture of oxidation products.

The successful preparation of naphtho[*f*]ninhydrin (3) (Scheme II) is patterned after the procedure of Jones and Wife[18] in which dimsyl anion was condensed with dimethyl 2,3-naphthalenedicarboxylate followed by a Pummerer rearrangement to give a chloro thioether that was hydrolyzed to give benzo[*f*]ninhydrin (2). Although 2,3-naphthalenedicarboxylic acid (12) and dimethyl 2,3-naphthalenedicarboxylate (13) are known compounds, the reported preparative methods[19-23] are impractical[21-23] or contain steps that have been questioned.[19,20,24]

Friedel–Crafts acylation of 1,2,4-trimethylbenzene with benzoyl chloride and $AlCl_3$ in CH_2Cl_2 gave the diaryl ketone 9[25] in 75% yield.[26] For the oxidation of all three methyl groups in 9 to carboxylic acid functions, a two-stage oxidation first with HNO_3 and then with alkaline $KMnO_4$ was utilized to afford a 75% yield of tricarboxylic acid 10.[27] In concentrated H_2SO_4,[28] 10 cyclized to form the anthraquinone dicarboxylic acid 11 in 75% yield.[30] Reduction of 11 with zinc and ammonium hydroxide gave a 75% yield of 2,3-naphthalenedicarboxylic acid (12), which was esterified with HCl in MeOH to produce an 85% yield of diester 13.

For the condensation of diester 13 with dimsyl anion, NaH was utilized as the base rather than NaOMe, which was employed by Jones and Wife in the benzo[*f*]ninhydrin-forming sequence.[18] Due to the low solubility of 13 in DMSO, THF was employed as a cosolvent. Upon acidification with HCl, 30–35% yields of the yellow-orange, chloro thioether 14 were obtained.[32] The chloro thioether 14 was hydrolyzed in refluxing aqueous dioxane to provide naphtho[*f*]ninhydrin (3) as an orange-red solid in 80% yield.[34] Interestingly, the hydrolysis of 14 was found to be considerably slower than that of the chloro thioether precursor to benzo[*f*]ninhydrin (2). The behavior of 3 as a reagent of the detection of latent fingerprints will be reported separately.

All new compounds were characterized by ^1H NMR and IR spectra and by elemental analyses.

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(26) Clar[25] reports a nearly quantitative yield of 9 from the reaction of benzoyl chloride, 1,2,4-trimethylbenzene, and $AlCl_3$ in the less convenient solvent of CS_2.
(27) Our procedure is patterned after one reported by Clar.[25]
(28) Cyclization of a closely related compound was performed in polyphosphoric acid.[29]
(29) Gunderman, K.-D.; Klockenbring, G.; Röker, C.; Brinkmeyer, H. *Liebigs Ann. Chem.* **1976**, 1873.
(30) Impure 11 has been obtained in low yield in the ozonolysis of commercially available, albeit very expensive, naphthacene-5,12-quinone.[31]
(31) Copeland, P. G.; Dean, R. E.; McNeil, D. *J. Chem. Soc.* **1961**, 3858.
(32) Competitive attack of the dimsyl anion on the anthracene ring[33] in the first step may be responsible for the relatively low yield.
(33) Russell, G. A.; Weiner, S. A. *J. Org. Chem.* **1966**, *31*, 248.
(34) Attempts to scale-up the hydrolysis reaction led to reduced percentage yields due to incomplete reaction. The product and unconsumed reactant can be separated by chromatography on silica gel with CH_2Cl_2 and then CH_2Cl_2–EtOAc (1:1) as eluents.

Experimental Section

Melting points were determined with a Mel-Temp capillary apparatus and are uncorrected. IR spectra were obtained with Nicolet MS-X and Perkin-Elmer Model 1600 FT-IR instruments. ^1H NMR spectra were measured with an IBM AF-200 spectrometer and chemical shifts are reported in parts per million (δ) downfield from tetramethylsilane. Elemental analyses were performed by Desert Analytics of Tucson, AZ.

Materials. Unless specified otherwise, reagent-grade reactants were used as received from chemical suppliers. DMSO was distilled from calcium hydride. THF and dioxane were distilled from sodium.

Carboxylic Acid 6. To a suspension solution of $AlCl_3$ (24.50 g, 184 mmol) in 100 mL of CH_2Cl_2 at room temperature was added a solution of phthalic anhydride (12.35 g, 83.4 mmol) and indan (9.86 g, 83.4 mmol) in 100 mL of CH_2Cl during a 50-min period. The reaction mixture was stirred for 12 h and poured into an HCl–ice slurry. The organic layer was separated, dried over $MgSO_4$, and evaporated in vacuo to give 16.41 g (74%) of a pale yellow solid, which was used without further purification for the next step. A sample for elemental analysis was recrystallized from $CHCl_3$ to give 6 as a pale yellow solid with mp 178–180 °C: IR (deposit) 3063 (OH), 1693, 1667 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.09 (pent, 2 H), 2.91 (m, 4 H), 7.23 (d, 1 H), 7.29 (dd, 1 H), 7.47–7.68 (m, 4 H), 8.08 (dd, 1 H). Anal. Calcd for $C_{17}H_{14}O_3$: C, 76.68; H, 5.30. Found: C, 76.58; H, 5.34.

2,3-Trimethyleneanthraquinone (7). A solution of carboxylic acid 6 (2.48 g, 9.32 mmol) in 25 mL of concentrated H_2SO_4 was stirred at 110 °C for 3 h and poured onto ice. The resulting mixture was stirred and then allowed to stand for 48 h at room temperature[35] and filtered. The crude product was chromatographed on silica gel with CH_2Cl_2 as eluent to give 1.39 g (60%) of yellow solid with mp 185–186 °C: IR (deposit) 1671 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.18 (pent, 2 H), 3.05 (t, 4 H), 7.72–7.81 (m, 2 H), 8.10 (s, 2 H), 8.23–8.30 (m, 2 H). Anal. Calcd for $C_{17}H_{12}O_2$: C, 82.24; H, 4.87. Found: C, 81.91; H, 4.73.

2,3-Trimethyleneanthracene (8). A stirred solution of anthraquinone 7 (2.00 g, 8.06 mmol) in 30 mL of a 1.0 M solution of aluminum cyclohexoxide in cyclohexanol[36] was heated at 160 °C for 24 h and cooled to room temperature, and 40 mL of 10% HCl was added. The mixture was extracted with CH_2Cl_2 and the organic layer was washed with water, dried over $MgSO_4$, and evaporated in vacuo. Cyclohexanol was removed from the residue by a simple high vacuum distillation at 0.08 Torr. The crude product was chromatographed on silica gel with toluene as eluent to give 1.27 g (72%) of a white crystalline solid with mp 228–230 °C: ^1H NMR ($CDCl_3$) δ 2.16 (pent, 2 H), 3.09 (t, 4 H), 7.38–7.43 (m, 2 H), 7.80 (s, 2 H), 7.92–7.99 (m, 2 H), 8.31 (s, 2 H). Anal. Calcd for $C_{17}H_{14}$: C, 93.54; H, 6.46. Found: C, 93.69; H, 6.51.

2,4,5-Trimethylbenzophenone (9). To a mixture of 1,2,4-trimethylbenzene (10.00 g, 83.2 mmol) and $AlCl_3$ (11.70 g, 87.5 mmol) in 10 mL of CH_2Cl_2 at 0 °C was added 11.70 g (83.2 mmol) of benzoyl chloride dropwise during a 30-min period. The reaction mixture was stirred at room temperature for 6 h and poured into a mixture of 25 mL of concentrated HCl and 50 g of ice. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with 5% $NaHCO_3$, dried over $MgSO_4$, and evaporated in vacuo. Vacuum distillation through a Vigreaux column gave 13.62 g (73%) of product with bp 130 °C/0.15 Torr (lit.[25] bp 328–329 °C): IR (film) 1662 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.10–2.35 (s, 9 H), 7.06 (d, 2 H), 7.35–7.60 (m, 3 H), 7.75–7.85 (m, 2 H).

Benzophenone-2,4,5-tricarboxylic Acid (10). A mixture of ketone 9 (12.50 g, 55.8 mmol) in 75 mL of 20% HNO_3 was refluxed for 5 days and cooled. The aqueous layer was decanted from the very thick, yellow semisolid. The semisolid was rinsed with 75 mL of cold water and dissolved in 125 mL of 10% NaOH to give a very dark brown solution, which was transferred into a three-necked flask equipped with a mechanical stirrer and reflux condenser. To the stirred and refluxing solution was added 35.0 g

(35) The initially formed, very fine crystals are very difficult to separate by filtration.
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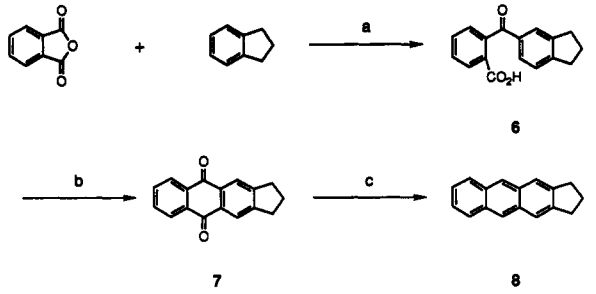
Scheme I[a]



[a] (a) $AlCl_3$, CH_2Cl_2; (b) fuming H_2SO_4; (c) aluminum cyclohexoxide, cyclohexanol.

(0.222 mol) of $KMnO_4$ in portions (*caution*-frothing may occur) during a 40-min period, and the reaction mixture was refluxed for 3 h. The reaction mixture was allowed to cool somewhat and was filtered. The collected solid (MnO_2) was refluxed in water for 6 h and filtered while hot. The combined filtrates were evaporated in vacuo to one-half volume, cooled, and acidified slowly with concentrated HCl. The resulting solid was filtered and air-dried to give 12.20 g (70%) of white crystals with mp 281–283 °C (lit.[25] mp 275 °C): IR (Nujol) 3312 (OH), 1743, 1707, 1687, 1655 (C=O) cm^{-1}; ^1H NMR (CD_3COCD_3) δ 7.45–7.85 (m, 6 H), 9.47 (s, 1 H).

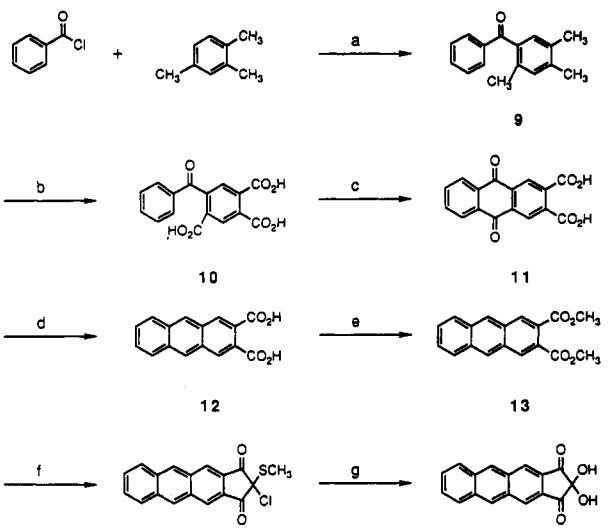
Anthraquinone-2,3-dicarboxylic Acid (11). A solution of triacid 10 (2.10 g, 6.69 mmol) in 21 g of concentrated H_2SO_4 was stirred and heated at 120 °C for 3 h and then poured onto 30 g of ice. The precipitate was filtered, washed with water, and air-dried to give 1.36 g (72%) of a pale yellow solid with mp >310 °C (lit.[31] mp 342 °C): IR (Nujol) 3166 (OH), 1638, 1618 (C=O) cm^{-1}; ^1H NMR (CD_3SOCD_3) δ 7.93–8.03 (m, 2 H), 8.20–8.30 (m, 2 H), 8.56 (s, 2 H).

Anthracene-2,3-dicarboxylic Acid (12). To 50 mL of 20% NH_4OH were added sequencially 1.00 g (3.38 mmol) of diacid 11 and then 3.75 g of activated zinc dust, and the blood-red mixture was refluxed. As soon as the color was discharged, the mixture was filtered while hot and the filtered material was refluxed with 50 mL of 20% NH_4OH for 2 h. This mixture was filtered while hot and the combined filtrates were cooled to 0 °C and acidified to pH 1 with 6 N HCl. The mixture was allowed to stand at room temperature for 1 day and then filtered to give 0.70 g (77%) of a bright yellow solid with mp >310 °C (lit.[19] mp 345 °C): IR (Nujol) 3135 (OH), 1701, 1674 (C=O) cm^{-1}; ^1H NMR (CD_3SOCD_3) δ 7.55–7.70 (m, 2 H), 8.06–8.25 (m, 2 H), 8.49 (s, 2 H), 8.79 (s, 2 H).

Dimethyl Anthracene-2,3-dicarboxylate (13). Into a refluxing mixture of diacid 12 (6.32 g, 23.8 mmol) in 80 mL of dry methanol was slowly passed HCl gas for 36 h. The mixture was cooled to 0 °C and filtered, and the collected solid was dissolved in CH_2Cl_2. The solution was washed with 5% $NaHCO_3$, dried over $MgSO_4$, and evaporated in vacuo to give 5.94 g (85%) of yellow solid with mp 149–151 °C (lit.[22] mp 151 °C): IR (deposit) 1717 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 3.99 (s, 6 H), 7.53–7.63 (m, 2 H), 8.00–8.10 (m, 2 H), 8.44 (s, 2 H), 8.51 (s, 2 H).

Chloro Thioether 14. Under nitrogen, a solution of diester 13 (0.85 g, 2.89 mmol) in 4 mL of dry DMSO and 5 mL of THF was added dropwise during a 15-min period to a stirred mixture of 0.35 g (8.75 mmol) of sodium hydride (60% dispersion in mineral oil) in 4 mL of dry DMSO and 2 mL of dry THF, and the mixture was stirred at room temperature for 12 h. The THF was removed in vacuo and the residual DMSO by a simple high vacuum distillation. The dark orange solid residue was dissolved in water (25 mL) and extracted with CH_2Cl_2 (25 mL). The aqueous layer was added dropwise to 25 mL of 6 N HCl during a 45-min period. The orange precipitate was filtered and stirred for 3 h in 200 mL of CH_2Cl_2. The mixture was filtered and the filtrate was washed with water (2 × 100 mL), dried over $MgSO_4$, and evaporated in vacuo. The residue was chromatographed on silica gel with CH_2Cl_2 as eluent to give 0.30 g (33%) of a yellow-orange solid with mp 212 °C (dec): IR (deposit) 1736, 1714 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.51 (s, 3 H), 7.61–7.74 (m, 2 H), 8.05–8.18 (m, 2 H), 8.76 (s, 2 H), 8.81 (s, 2 H). Anal. Calcd for $C_{18}H_{11}ClO_2S$: C, 66.16; H, 3.39. Found: C, 66.40; H, 3.14.

Scheme II[a]



[a] (a) $AlCl_3$, CH_2Cl_2; (b) HNO_3; then $KMnO_4$, aqueous NaOH; (c) concd H_2SO_4; (d) Zn, 20% NH_4OH; (e) CH_3OH, HCl; (f) NaH, DMSO, THF; then HCl, H_2O; (g) aqueous dioxane.

Naphtho[*f*]ninhydrin (3).[34] Chloro thioether 14 (0.100 g, 0.30 mmol) was added in small portions during a 2-h period to a stirred solution of 9 mL of peroxide-free dioxane and 4 mL of distilled water at 95 °C. Heating was continued for 36 h and the reaction mixture was filtered while hot. Dioxane was evaporated from the filtrate in vacuo, and 10 mL of water was added. The mixture was filtered and the collected solid was air-dried to give 0.068 g (80%) of orange-red solid with mp 245 °C (dec): IR (Nujol) 3331 (OH), 1741, 1718 (C=O) cm^{-1}; ^1H NMR (CD_3SOCD_3) δ 7.59 (s, 2 H, O*H*), 7.70–7.80 (m, 2 H), 8.20–8.30 (m, 2 H), 8.90 (s, 2 H), 9.05 (s, 2 H). Anal. Calcd[37] for $C_{17}H_8O_3 \cdot 0.4H_2O$: C, 76.34; H, 3.32. Found: C, 76.26; H, 3.66.

Acknowledgment. This research wss supported by United States Department of Justice, National Institute of Justice, grant number 87-IJ-CX-0030 to Professor E. Roland Menzel in the Department of Physics of Texas Tech University.

Registry No. 4, 135989-72-9; 6, 6321-58-0; 7, 53933-88-3; 8, 7258-46-0; 9, 52890-52-5; 10, 135989-69-4; 11, 27485-15-0; 12, 10210-28-3; 13, 56306-53-7; 14, 135989-70-7; 15, 135989-71-8; DMSO, 67-68-5; 1,4-cyclopentadienone, 13177-38-3; 2,3-bis(bromoethyl)naphthalene, 38998-33-3; indane, 496-11-7; phthalic anhydride, 85-44-9; benzoyl chloride, 98-88-4; 1,2,4-trimethylbenzene, 95-63-6.

(37) The formula $C_{17}H_8O_3$ is for the triketone formed by dehydration of 3. The elemental analysis sample was dried for an extended period with heating in vacuo before submission for analysis and dried again in vacuo just prior to analysis.

Conjugated Polyene Synthesis via Disilyl Derivatives: A Direct Access to Ostopanic Acid, a Plant Anticancer Agent, and to (6*E*)-LTB$_3$ Leukotriene

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Various classes of natural products such as alcohols,[1] aldehydes,[2] ketones,[3] and acids[4] contain a conjugated

