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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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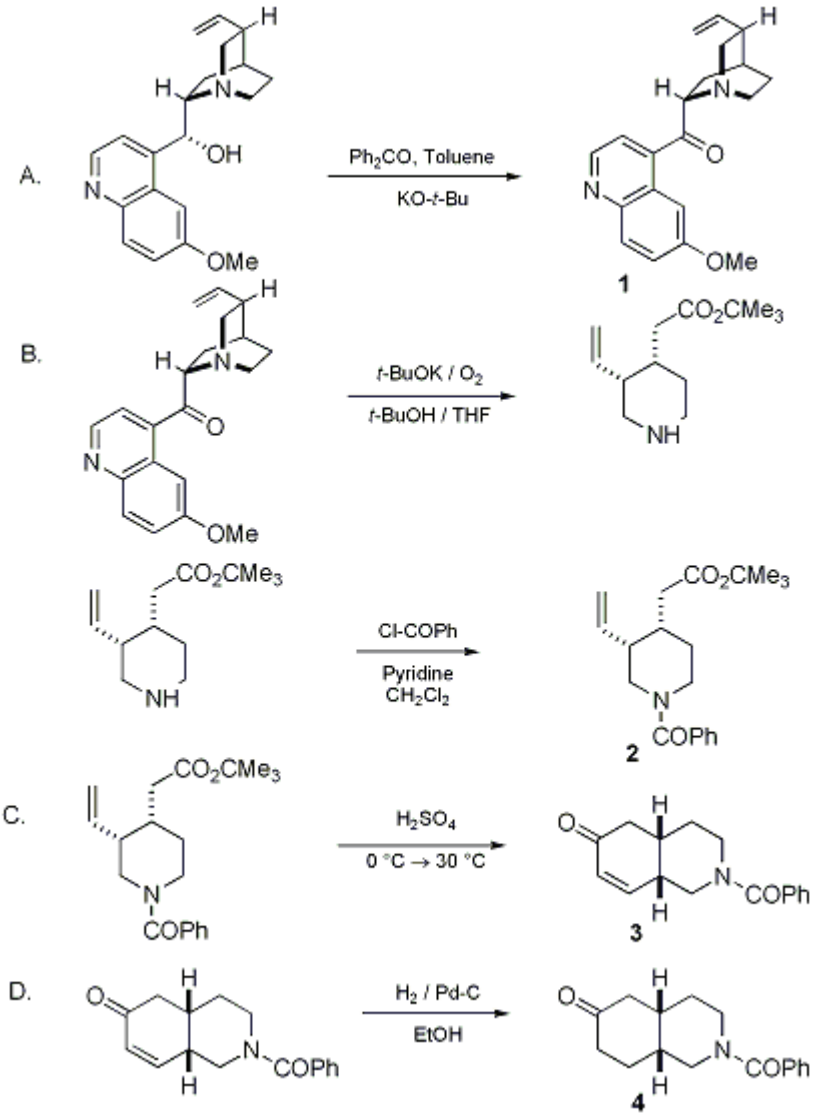
SYNTHESIS OF cis-4a(S),8a(R)-PERHYDRO-6(2H)-ISOQUINOLINONES FROM QUININE: 4a(S), 8a(R)-2-BENZOYLOCTAHYDRO-6(2H)-ISOQUINOLINONE

[6(2H)-Isoquinolinone, 2-benzoyloctahydro-, (4aS-cis)-]



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1. Procedure

A. Quininone **(1)**. A 2-L, three-necked, round-bottomed flask (Note 1) equipped with a mechanical stirrer, reflux condenser and a thermocouple is charged with 112.2 g (0.61 mol) of benzophenone (Note 2) and 600 mL of toluene (Note 3) under a positive pressure of nitrogen (N_2). Quinine, 111.11 g (0.31 mol) (Note 4), is then added in one portion (Note 5), followed by 87.1 g (0.78 mol) of potassium tert-butoxide (Note 6) added in one portion. The slurry is heated to reflux with an electric mantle for 8 hr (Note 7). The reaction mixture is allowed to cool overnight to room temperature and then to 5-10°C

with an ice-water mixture. 2 N Hydrochloric acid (HCl), 400 mL , is added slowly keeping the temperature below 20°C. The contents of the flask are transferred to a 2-L separatory funnel with 300 mL of 2 N HCl . After two layers separate, the lower aqueous layer is collected in a 3-L Erlenmeyer flask, and the organic layer is washed with 2 N HCl (2 × 250 mL). The combined aqueous layers are cooled to 0-5°C and treated dropwise with 260 mL of 5 N sodium hydroxide (NaOH) with stirring to pH 9.5. An oil initially separates that becomes a yellow solid after vigorous stirring at 0-5°C. The solid is filtered using a Büchner funnel, rinsed with water (2 × 200 mL), and dried in an oven at 60°C for 48 hr to afford the ketone **1** as a light yellow solid weighing 97.9 g (98%, (Note 8)).

B. N-Benzoylmeroquinene tert-butyl ester **(2)**. A 2-L, three-necked, round-bottomed flask is equipped with a mechanical stirrer, subsurface glass-fritted gas tube for oxygen (O_2) addition (the gas tube is also connected to a bubbler containing silicone oil to monitor the flow of oxygen gas), and a thermocouple. The flask is charged with 800 mL of tetrahydrofuran (THF) and 200 mL of tert-butyl alcohol (tert-BuOH). The solution is purged with O_2 under stirring for 15 min and then treated with 78.4 g of potassium tert-butoxide (0.70 mol) in one portion (Note 9). The yellow reaction mixture is cooled (ice bath) while the O_2 addition is continued for another 15 min. Crude quininone **1**, 90.0 g, (0.28 mol) is then added portionwise over 10 min with continued O_2 addition, resulting in a blood red mixture, concomitant with an exotherm to 35°C (Note 10). After the solution is stirred for 1.5 hr at ambient temperature, the gas purge is stopped and 80 mL of glacial acetic acid (HOAc) is added carefully with vigorous stirring. Volatile material is removed under reduced pressure at ≤ 50°C from the resulting thick slurry that is then suspended in 400 mL of water. The pH is adjusted to 10 by the addition of 60 mL of concd ammonium hydroxide (NH_4OH) and the aqueous solution is extracted with ether (4 × 200 mL), washed with a saturated solution of brine (3 × 250 mL, (Note 11)), dried over sodium sulfate (Na_2SO_4), filtered and concentrated (Note 12) to furnish meroquinene tert-butyl ester as a viscous oil (40.5 g, 64% crude recovery, (Note 13)). A 1-L, three-necked, round-bottomed flask equipped with a mechanical stirrer, thermocouple, addition funnel, condenser, and N_2 inlet is charged with 40.5 g (0.18 mol) of crude meroquinene tert-butyl ester and 235 mL of dichloromethane (CH_2Cl_2) (Note 14). Pyridine, 16.0 mL, (0.19 mol), (Note 15) is added, followed by dropwise addition of 25.0 mL (0.21 mol) of benzoyl chloride (Note 15) at a rate to maintain a gentle reflux over 15 min. Upon complete addition, the reaction mixture is stirred at ambient temperature for 2 hr (Note 16) and washed successively with H_2O (200 mL), 1 N HCl (2 × 150 mL), 2 N NaOH (2 × 150 mL), and brine (300 mL). The organic phase is dried over Na_2SO_4, filtered and concentrated to give the thick brown oily benzamide **2** (61.2 g, 100%, (Note 17)).

C. 4a(S),8a(R)-2-Benzoyl-1,3,4,4a,5,8a-hexahydro-6(2H)-isoquinolinone **(3)**. A 1-L, three-necked, round-bottomed flask is equipped with a mechanical stirrer, Teflon-coated thermocouple, 500-mL addition funnel and a N_2 inlet. Concentrated sulfuric acid (H_2SO_4), 275 mL is added to the flask and the flask is cooled to 0°C under N_2. The addition funnel is charged with a solution of 56.0 g (0.17 mol) of crude benzamide **2** in 60 mL of CH_2Cl_2, that is added dropwise over 15 min, maintaining the internal temperature between 0-10°C. Upon complete addition, the cooling bath is removed and the reaction mixture is stirred vigorously for another 2 hr (Note 18) as the temperature rises to 30°C. The reaction mixture is poured onto 1 kg of crushed ice with stirring and when the ice has melted the two layers are separated. The aqueous phase is extracted further with CH_2Cl_2 (4 × 200 mL). The combined organic phases are washed with water (2 × 500 mL), brine (500 mL), dried over Na_2SO_4, filtered, and concentrated to a light brown semisolid (Note 19) that is dissolved in 100 mL of CH_2Cl_2 and precipitated with 250 mL of hexanes to furnish 34.7 g (80%) of enone **3** as light yellow crystals (Note 20).

D. 4a(S),8a(R)-2-Benzoyloctahydro-6(2H)-isoquinolinone **(4)**. Palladium (Pd), 10% on carbon, 4.0 g , (Note 21) is placed in a 500-mL Parr bottle under N_2 and carefully wetted with 50 mL of cold denatured ethanol (EtOH). A slurry of 34.7 g of enone **3** (0.14 mol) in denatured EtOH (250 mL) is added and the Parr shaker apparatus assembled. After the system is purged with nitrogen-hydrogen (N_2/H_2), the reaction is shaken at 50 psi H_2 and 50°C until H_2 uptake is complete (1 hr, (Note 22)). The catalyst is filtered over a Celite pad (Note 23) and rinsed with warm chloroform ($CHCl_3$) (4 × 75 mL). The filtrate is concentrated under reduced pressure, dissolved in 90 mL of CH_2Cl_2 and crystallized with 200 mL of hexanes. The crystalline solid is filtered, rinsed with hexanes and dried to afford 34.3 g (98%, (Note 24)) of the ketone **4**, representing a 51% yield over four steps.

2. Notes

1. All glass apparatus was dried thoroughly under a flow of dry N_2. All ground glass joints were tightly sealed with Teflon tape and then wrapped with Parafilm. All the preparations were performed in an efficient fume hood while wearing gloves and adequate eye protection.
2. Benzophenone purchased from Aldrich Chemical Company, Inc. was used as received.
3. Toluene, dichloromethane, acetic acid, ammonium hydroxide, concentrated H_2SO_4, 5 N NaOH and 37% HCl were purchased from Mallinckrodt Inc. ; tetrahydrofuran, tert-butyl alcohol, anhydrous Na_2SO_4, and NaCl were purchased from EM Science ; potassium tert-butoxide was purchased from Aldrich Chemical Company, Inc. ; hexanes was purchased from Baxter , and dry O_2 was purchased from Air Products . All these reagents were used as received.
4. Quinine (purity 90%) purchased from Aldrich Chemical Company, Inc. , was used as received.
5. A mild endotherm was noted: the temperature fell from 22°C to 19°C.
6. The color changed to yellowish brown immediately upon addition of potassium tert-butoxide and a mild exotherm was noted, as the temperature rose to 29°C.
7. The slurry became very thick as the temperature approached reflux, requiring vigorous stirring. The color of the mixture gradually changed to dark orange and at the end of the reaction the color was fluorescent orange.
8. The material was sufficiently pure as determined by HPLC (Zorbax C-8 column RX 25 cm; flow rate 2.5 mL/min; mobile phase acetonitrile/water (1:1); UV detection at 254 nm showed four peaks t_R(min.) at 0.9 (quinine), 1.3 (quininone), 4.4 (toluene), 5.4 (benzophenone). A part of this material (8.0 g) was dissolved in 250 mL of diethyl ether at room temperature, left in a freezer for 48 hr, and 6.9 g of light yellow solid was obtained upon filtration; mp 102-104°C.
9. The reaction mixture displayed an exotherm from ambient temperature to 35°C.
10. Quininone was added at a rate to keep the temperature below 25°C; otherwise the yield of this step was much lower. Oxygen uptake increased upon the addition of quininone, but slowed as the reaction proceeded (15 min).
11. If three phases result, add the minimum amount of water (100 mL) that affords two phases (some color in the aqueous phase was noted). Additional quantities of water should be avoided because of the high water solubility of the product.
12. The product was concentrated under reduced pressure at room temperature, but concentration at higher temperature resulted in a lower yield (35-40%).
13. Any attempts to purify the product by distillation resulted in lower yields because of pyrolysis. The undistilled product was sufficiently pure for most purposes. The yield range was 60-75%; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.32-1.63 (m, 11 H), 2.02-2.30 (m, 4 H), 2.63-3.06 (m, 4 H), 4.90-5.18 (m, 2 H), 6.01-6.10 (m, 1 H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ: 28.1, 28.9, 35.7, 39.4, 43.0, 46.1, 51.4, 80.1, 116.6, 137.2, 172.3 .
14. Six volumes of CH_2Cl_2 are used to ensure efficient stirring, since a solid separates after the addition of benzoyl chloride.
15. Reagent grade pyridine and benzoyl chloride were purchased from Aldrich Chemical Company, Inc. , and used as received.
16. The reaction appeared complete by HPLC. R_f for the product=0.54 (by TLC analysis on silica gel 60 F-254 precoated plates, hexanes:EtOAc, 1:1, freshly prepared).
17. The crude product was sufficiently pure and used as such for the next step (purification by column chromatography using hexanes:EtOAc, 4:6 as an eluant affords a 92-95% yield of the purified product). A part of the crude product (5.0 g) was crystallized from 50 mL of diethyl ether to furnish light yellow needles; mp 62-64°C (lit.[2] mp 65-67°C); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.39-1.52 (m, 11 H), 2.08-2.31 (m, 3 H), 2.33-2.65 (m, 1 H), 2.97-3.25 (m, 2 H), 3.65-3.75 (m, 1 H), 4.50-4.74 (m, 1 H), 5.08-5.12 (m, 2 H), 5.79-5.92 (m, 1 H), 7.31-7.41 (m, 5 H) ; ^{1}H NMR (CD_3SOCD_3, 25°C) δ: 1.39 (m, 11 H), 2.02-2.55 (m, 4 H), 2.90-3.50 (m, 3 H), 4.21-4.42 (m, 1 H), 4.90-5.12 (m, 2 H), 5.76-5.94 (m, 1 H), 7.31-7.41 (m, 5 H) ; ^{1}H NMR (CD_3SOCD_3, 90°C) δ: 1.39-1.51 (m, 11 H), 2.04-2.21 (m, 3 H), 2.41-2.49 (m, 1 H), 3.06 (ddd, 1 H, J = 2.7, 3.7 and 10.7), 3.24 (dd, 1 H, J = 3.2 and 13.2), 3.91-4.00 (m, 2 H), 5.02-5.13 (m, 2 H), 5.78-5.87 (m, 1 H), 7.31-7.41 (m, 5 H) .; ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 28.1, 35.8, 38.7, 42.2, 46.0, 47.5, 52.4, 80.4, 118.1, 127.0, 128.3, 129.4, 134.7, 136.2, 170.8, 171.8 .
18. The color of the reaction mixture changed from light yellow to dark brown at the end of the reaction with the formation of solid particles. The reaction appeared complete by TLC analysis (silica gel 60 F-

254 precoated plates, hexanes:ethyl acetate, 2:8, freshly prepared); R_f for benzoyl derivative = 0.74, R_f for enone = 0.25.
19. HPLC of the crude product indicated the presence of only cis isomer; no trans isomer was detected in the crude product; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.52-1.82 (m, 2 H), 2.47-2.56 (m, 3 H), 2.82-2.96 (m, 1 H), 3.21-3.41 (m, 1 H), 3.50 (dd, 1 H, J = 13.5 and 4.1), 3.52-3.72 (m, 1 H), 4.35-4.45 (m, 1 H), 6.09 (d, 1 H, J = 9.7), 6.85-7.05 (m, 1 H), 7.27-7.42 (m, 5 H) ; ^{1}H NMR (CD_3SOCD_3, 25°C) δ: 1.35-1.70 (m, 2 H), 2.46-2.50 (m, 3 H), 2.82 (m, 1 H), 3.22-4.08 (m, 4 H), 5.98 (m, 1 H), 6.92 (m, 1 H), 7.34-7.44 (m, 5 H) ; ^{1}H NMR (CD_3SOCD_3, 90°C) δ: 1.46-1.59 (m, 2 H), 2.48-2.50 (m, 2 H), 2.80 (m, 1 H), 3.01 (m, 1 H), 3.22-3.31 (m, 1 H), 3.51 (dd, 1 H, J = 4.2 and 13.4), 3.60-3.64 (m, 1 H), 3.84-3.87 (m, 1 H), 5.9 (dd, 1 H, J = 2.2 and 10.1), 6.77 (dd, 1 H, J = 3.0 and 9.8), 7.33-7.45 (m, 5 H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ: 27.2, 33.8, 37.0, 41.8, 44.9, 46.2, 126.8, 128.6, 129.8, 131.0, 135.8, 150.6, 170.8, 198.1 .
20. If there was no crystallization, a few crystals of crude product were added to the flask to initiate crystallization. The mp was 148-150°C (lit.[2] mp 150-152°C).
21. Palladium on activated carbon (10%) was purchased from Aldrich Chemical Company, Inc. , and used as received.
22. After 75 min an aliquot was drawn and analyzed by ^{1}H NMR which indicated the presence of enone (< 5%); another 1.0 g of Pd was added and the mixture heated at 50°C/50 psi of H_2 for another 45 min.
23. The palladium on activated carbon (10%) was not allowed to become completely dry because of its flammable nature.
24. The data for the pure product is: mp 179-181°C (lit.[2] mp 182-183°C); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.49-1.60 (m, 2 H), 2.01-2.13 (m, 2 H), 2.25-2.60 (m, 6 H), 3.03-3.22 (m, 2 H), 3.61-3.81 (m, 1 H), 4.45-4.59 (m, 1 H), 7.28-7.41 (m, 5 H) ; ^{1}H NMR (CD_3SOCD_3, 25°C) δ: 1.30-1.62 (m, 2 H), 1.82 (m, 2 H), 2.18-2.65 (m, 6 H), 3.04 (m, 1 H), 3.20 (m, 1 H), 3.48 (m, 1 H), 4.22 (m, 1 H), 7.32-7.48 (m, 5 H) ; ^{1}H NMR (CD_3SOCD_3, 90°C) δ: 1.34-1.52 (m, 2 H), 1.66-1.78 (m, 1 H), 1.80-2.02 (m, 1 H), 2.20-2.31 (m, 2 H), 2.31-2.37 (m, 2 H), 2.48-2.57 (m, 2 H), 3.00-3.12 (m, 1 H), 3.27 (dd, 1 H, J = 3.6 and 13.2), 3.88 (m, 2 H), 7.30-7.50 (m, 5 H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ: 25.5, 26.6, 27.6, 35.0, 37.4, 39.8, 45.9, 47.2, 126.8, 128.5, 129.6, 136.1, 171.0, 210.5

Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

[3]

In this procedure, quinine is oxidatively degraded to meroquinene esters that are subsequently cyclized to N-acylated cis-decahydroisoquinolones in excellent overall yield, while maintaining the cis stereochemistry at the ring juncture. Furthermore, with the commercial availability of quinine, high overall yields, and ease of isolations, meroquinene and subsequent products are attractive members of a practical "chiral pool".

Oxidation of the quinine C-9 hydroxy substituent to the ketone is best accomplished using the Woodward[4] benzophenone/potassium t-butoxide method, now using toluene. The other oxidation methods investigated (Swern, Jones, ROCl variations) were less effective or limited because of the poor solubility of the substrate. Thermodynamic equilibration of these ketones has also been reported.[4]

Quininone, the most readily available member of the series, was used for the autoxidation studies. The Doering autoxidation procedure,[5] that employs only tert-BuOH, was modified to include a THF:tert-BuOH (4:1) mixture as the solvent. Likewise, the pressurized Parr bottle setup as described[5] was replaced with a simple subsurface gas addition; the solvent was presaturated with O_2 gas, (compressed air could also be used as the O_2 source) followed by t-BuOK addition and continued O_2 gas purge. The autoxidations could likewise be conducted in the presence of ethanol or methanol, thereby producing the corresponding ethyl or methyl esters. Formation of these esters could occur via the reactive intermediate bicyclic lactam.[5]

Cyclization of N-acyl meroquinenes with neat polyphosphoric acid (PPA, thick reaction mixture) required 5 days at ambient temperature and resulted in a 2.4:1 mixture of trans:cis-enones, respectively (55% yield, eq 1).[2] The diastereomeric mixture could easily be separated by column chromatography to provide pure samples of either substance. It was also shown that the pure trans- and cis-enones equilibrated under the PPA cyclization conditions to afford the same 2.4:1 (trans:cis) mixture of enones. Thermodynamic equilibration of either enone also occurred with p-toluenesulfonic acid (p-TsOH) in THF. Cyclization of N-acyl meroquinenes could be conducted in a mixture of PPA:H_2SO_4 (0°C → 20° C) with complete cis-stereocontrol in essentially quantitative yield (30 min) affording only the cis-product. Equilibration of the γ-position occurred at elevated temperatures (> 25°C). It was then determined that concentrated H_2SO_4 could replace the PPA:H_2SO_4 mixture, with identical (cis) product profile. Trifluoroacetic anhydride (TFAA) also effected the present cyclization albeit with poor efficiency, but alternative acids (H_3PO_4, $AlCl_3/CH_2Cl_2$, TFAA, HCl, CH_3SO_3H, HOAc, HNO_3) did not.[6] [7] [8] Either TFAA or mixtures of Ac_2O with catalytic H_2SO_4 have been employed for the cyclization of ω-olefinic acids, although the substrates did not contain stereogenic centers. Polyphosphoric acid has also been used extensively for the acylation of alkenes at high temperatures, usually at 100°C.[9] [10] [11] [12]

Furthermore, attempted cyclization of N-carboxymethyl meroquinene ethyl ester failed to afford any of the enone, suggesting that the carboxylic acid was an intermediate. The N-protecting groups, in addition to benzoyl, that are tolerated include CO_2Me, pivaloyl, acetyl, toluenesulfonyl, CBz, and alkyl.

Enones were readily reduced in EtOH solution with catalytic palladium (10% on carbon) under an atmosphere of H_2 and the perhydroisoquinolones were isolated by chromatography or crystallization.

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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

Quininone (8);
Cinchon-9-one, 6'-methoxy-, (8a)- (9); (84-31-1)

Benzophenone (8);
Methanone, diphenyl- (9); (119-61-9)

Toluene (8);

Benzene, methyl- (9); (108-88-3)

Quinine (8);
Cinchonan-9-ol, 6'-methoxy-, (8a, 9R)- (9); (130-95-0)

Potassium tert-butoxide:
tert-Butyl alcohol, potassium salt (8);
2-Propanol, 2-methyl-, potassium salt (9); (865-47-4)

N-Benzoyl meroquinene tert-butyl ester:
4-Piperidineacetic acid, 1-benzoyl-3-ethenyl-, 1,1-dimethylethyl ester, (3R-cis)- (9); (52346-13-1)

tert-Butyl alcohol (8);
2-Propanol, 2-methyl- (9); (75-65-0)

Acetic acid (8,9); (64-19-7)

Meroquinene tert-butyl ester:
4-Piperidineacetic acid, 3-ethenyl-, 1,1-dimethylethyl ester, (3R-cis)- (9); (52346-11-9)

Pyridine (8, 9); (110-86-1)

Benzoyl chloride (8, 9); (98-88-4)

4a(S), 8a(R)-2-Benzoyl-1,3,4,4a,5,8a-hexahydro-6(2H)-isoquinolinone:
6(2H)-Isoquinolone, 2-benzoyl-1,3,4,4a,5,8a-hexahydro-, (4aS-cis)- (9); (52346-14-2)

4a(S), 8a(R)-2-Benzoyloctahydro-6(2H)-isoquinolinone:
6(2H)-Isoquinolinone, 2-benzoyloctahydro-, (4aS-cis)- (9); (52390-26-8)