Studies on Aromatic Sesquiterpenes. XIII.[1] Synthesis of Lacinilene A and Its Structural Isomer

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Synopsis. Lacinilene A (6-norcadalen-7-ol) was synthesized from anisole through 6-norcalamenen-7-ol. 6-Nordaucalen-7-ol was also synthesized.

Lacinilene A was isolated from the heartwood[2] and the sapwood[3] of Ohyonire *Ulmus laciniata* Mayr along with cadalene-type sesquiterpenes. The structure was proposed as 5-isopropyl-8-methyl-2-naphthol (6-norcadalen-7-ol) (**1**) on the basis of comprehensive spectral studies and some chemical derivation.[2,4]

To the best of our knowledge, however, the confirmation of lacinilene A by its total synthesis has not been reported.

In this paper we report the synthesis of **1**, a norsesquiterpenoid corresponding to naturally occurring 7-cadalenol,[2,3,5] starting from anisole through 6-norcalamenen-7-ol (**3**). The structural isomer of **1**, 6-nordaucalen-7-ol (**5**), was also synthesized via 6-norisocalamenen-7-ol (**7**) for an additional comparison with compound **1**.

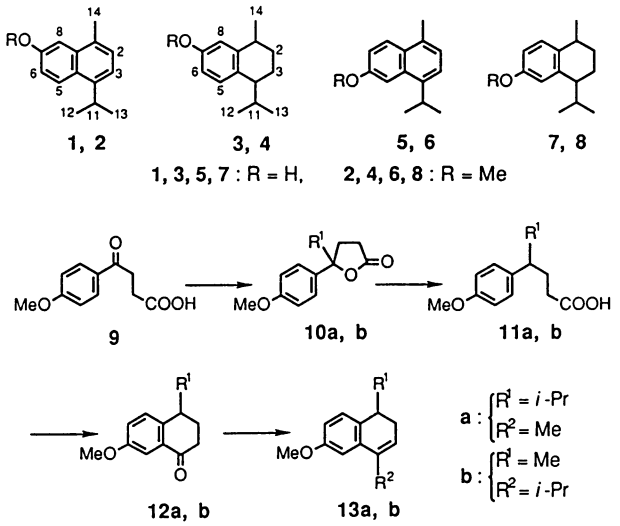
4-(4-Methoxyphenyl)-5-methyl-4-hexanolide (**10a**) derived from the keto acid[6] (**9**) and isopropylmagnesium bromide was reduced to 4-(4-methoxyphenyl)-5-methylhexanoic acid (**11a**) by the Clemmensen reduction, followed by cyclization of the corresponding acid chloride by anhydrous $SnCl_4$ to give 3,4-dihydro-4-isopropyl-7-methoxy-1(2*H*)-naphthalenone (**12a**). Reaction with methylmagnesium iodide followed by dehydration of the resulting alcohol with *p*-toluenesulfonic acid afforded 1,2-dihydro-1-isopropyl-6-methoxy-4-methylnaphthalene (**13a**).

Catalytic hydrogenation of **13a** in the presence of Pd–C in ethanol gave (±)-*cis*-7-methoxy-6-norcalamenene (**4**) as the sole product in good yield, whose stereochemistry was assigned by comparing its ^{1}H NMR spectrum with those of calamenene[7] and calamenen-7-ol,[5] showing that this hydrogenation favored a *cis*-isomer exclusively.

De-*O*-methylation of **4** with BBr_3 in dichloromethane at −10 °C yielded (±)-*cis*-**3** without formation of the *trans*-isomer. However, demethylation of **4** with 48% HBr in refluxing acetic acid gave an 82:18 mixture of *cis*-**3** and *trans*-**3**. The diastereomeric ratio was determined by ^{1}H NMR spectrum (especially, the different chemical shifts for the isopropyl proton signals).

Dehydrogenation of **3** by heating with Pd–C afforded the desired 2-naphthol **1**, whose ^{1}H NMR data and mp were identical with those of the natural lacinilene A.[2,3] Furthermore, lacinilene A methyl ether **2** was synthesized by the dehydrogenation of **4** or **13a**.

On the other hand, the isomer **5** was also prepared from the keto acid **9** in a similar synthetic fashion to that of **1**. Its physical properties (mp, IR, ^{1}H and ^{13}C NMR, and GC) were wholly different from those of **1**.



Experimental

All melting and boiling points are uncorrected. IR spectra were measured on a Shimadzu Infrared Spectrometer IR-430, either as neat (liquids) or KBr disk (solids); the absorption is reported in cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL spectrometer JNM-FX90Q (90 MHz for ^{1}H and 22.5 MHz for ^{13}C) for $CDCl_3$ solutions with TMS as an internal standard; signals are reported in δ (ppm). Elemental analysis data (C, H, and N) agreed within ±0.3% with the calculated values.

4-(4-Methoxyphenyl)-5-methyl-4-hexanolide (10a). To dry anisole (50 ml) containing isopropylmagnesium bromide, prepared from isopropyl bromide (17.3 g) and Mg (3.4 g) in ether (20 ml), a powdered 4-(4-methoxyphenyl)-4-oxobutyric acid[6] (**9**) (10.4 g) was added with stirring at 5 °C. After stirring for 7 h at 40 °C, the reaction mixture was decomposed by adding 15% aqueous acetic acid solution under cooling. Distillation of the resulting product afforded **10a** as an oil (5.4 g, 46.2%), bp 162—163 °C/3 mmHg (1 mmHg=133.322 Pa). IR 1770. ^{1}H NMR 0.86 (3H, d, *J*=7 Hz), 0.89 (3H, d, *J*=7 Hz), 2.12 (1H, m, *J*=7 Hz), 2.46 (4H, broad s), 3.81 (3H, s), 6.88 (2H, d, *J*=9 Hz), 7.26 (2H, d, *J*=9 Hz).

4-(4-Methoxyphenyl)-5-methylhexanoic Acid (11a). A mixture of **10a** (11.7 g), toluene (18 ml), conc HCl (31 ml), and amalgamated zinc prepared from Zn (21.0 g) and $HgCl_2$ (2.1 g) was stirred under reflux for 6 h. Distillation of the resulting product obtained from the organic layer by the usual work-up afforded **11a** as an oil (10.0 g, 84.7%), bp 169—172 °C/3 mmHg. IR 1700. ^{1}H NMR 0.72 (3H, d, *J*=7 Hz), 0.96 (3H, d, *J*=7 Hz), 2.1 (6H, m), 3.78 (3H, s), 6.81 (2H, d, *J*=9 Hz), 7.02 (2H, d, *J*=9 Hz), 10.38 (1H, broad).

3,4-Dihydro-4-isopropyl-7-methoxy-1(2*H*)-naphthalenone (12a). A mixture of **11a** (11.8 g) and $SOCl_2$ (7.5 g) in benzene (40 ml) was refluxed for 2 h. Distillation of the reaction mixture afforded the acid chloride as an oil (11.8 g, 92.9%), bp 141—143 °C/4 mmHg; IR 1790.

To a solution of the acid chloride (11.8 g) in benzene (40 ml) was added anhydrous $SnCl_4$ (14.4 g) dropwise with stirring at 5 °C. After stirring for 2 h at 5 °C, the mixture was poured into ice water. Distillation of the resulting product afforded **12a** as an oil (9.6 g, 95.0%), bp 146—147 °C/3 mmHg. IR 1670. ^{1}H NMR 0.95 (6H, d, J=7 Hz), 1.9—2.2 (3H, m), 2.4—2.8 (3H, m), 3.82 (3H, s), 7.03 (1H, dd, J=9, 3 Hz), 7.20 (1H, d, J=9 Hz), 7.51 (1H, d, J=3 Hz). 2,4-Dinitrophenylhydrazone; orange leaves (from benzene), mp 173.0—174.0 °C.

The cyclization of **11a** with $POCl_3$ was also investigated. A solution of **11a** (4.8 g) and $POCl_3$ (3.8 g) in 1,1,2,2-tetrachloroethane (50 ml) was refluxed for 2 h with stirring; the mixture was poured into ice water. Distillation of the resulting product afforded **12a** (2.3 g, 52.3%).

(±)-*cis*-7-Methoxy-6-norcalamenene (4). A solution of **12a** (10.9 g) in ether (10 ml) was added to a Grignard reagent prepared from methyl iodide (16.7 g) and Mg (2.6 g) in ether (40 ml) with cooling in an ice bath. The reaction mixture was stirred at 5 °C for 7 h and then at 30 °C for 7 h; the mixture was then decomposed by adding ice and NH_4Cl. Evaporation of the solvent afforded the crude alcohol as an oil (10.7 g; IR 3400). This material was used directly for the next step without purification.

A mixture of the crude alcohol (10.7 g) and *p*-toluenesulfonic acid (0.1 g) in benzene (100 ml) was refluxed with stirring for 3 h. Alumina column chromatography (CCl_4) of the resulting product afforded **13a** as an oil (9.7 g, 89.8%), bp 124—126 °C/4 mmHg. IR 1600, 1570, 1485, 1285, 1240, 1050. ^{1}H NMR 0.78 (3H, d, J=7 Hz), 0.88 (3H, d, J=7 Hz), 1.82 (1H, m, J=7 Hz), 2.01 (3H, d, J=1.5 Hz), 2.35 (3H, m), 3.80 (3H, s), 5.75 (1H, broad), 6.68 (1H, dd, J=8, 3 Hz), 6.80 (1H, d, J=3 Hz), 7.02 (1H, d, J=8 Hz).

The dihydronaphthalene **13a** (8.6 g) was hydrogenated over 10% Pd–C (0.4 g) in ethanol (50 ml) at room temperature. After removing the catalyst by filtration, the solution was evaporated, and distillation of the residue afforded (±)-*cis*-**4** as an oil (8.2 g, 94.3%), bp 118—120 °C/3 mmHg. IR 1610, 1500, 1270, 1240, 1040. ^{1}H NMR 0.75 (3H, d, J=7 Hz), 1.00 (3H, d, J=7 Hz), 1.25 (3H, d, J=7 Hz), 1.67 (4H, broad m), 2.19 (1H, m, J=7 Hz), 2.5 (1H, m), 2.8 (1H, m), 3.73 (3H, s), 6.66 (1H, dd, J=9, 3 Hz), 6.67 (1H, d, J=3 Hz), 7.10 (1H, d, J=9 Hz). ^{13}C NMR 17.5 (q, *i*-Pr), 19.8 (C3), 21.3 (q, *i*-Pr), 23.2 (C14), 28.7 (C2), 31.2 (C1), 33.2 (C11), 43.1 (C4), 54.9 (OMe), 111.4 (C8), 113.3 (C6), 129.0 (C5), 131.9 (C10), 144.1 (C9), 157.4 (C7).

(±)-*cis*-6-Norcalamenene-7-ol (3). To a solution of *cis*-**4** (1.9 g) in dichloromethane (30 ml), a solution of BBr_3 (4.4 g) in dichloromethane (5 ml) was added with stirring at −10 °C. After 5 h the reaction mixture was poured into ice water. Distillation of the resulting product obtained from the organic layer by the usual work-up afforded (±)-*cis*-**3** as an oil (1.7 g, 94.4%), bp 137 °C/4 mmHg. IR 3300, 1605, 1495, 1460, 1260, 1230. ^{1}H NMR 0.75 (3H, d, J=7 Hz), 1.00 (3H, d, J=7 Hz), 1.21 (3H, d, J=7 Hz), 1.68 (4H, m), 2.17 (1H, m, J=7 Hz), 2.5 (1H, m), 2.8 (1H, m), 5.37 (broad, 1H), 6.61 (1H, dd, J=9, 3 Hz), 6.63 (1H, d, J=3 Hz), 7.05 (1H, d, J=9 Hz). ^{13}C NMR 17.5 (q, *i*-Pr), 19.9 (C3), 21.3 (q, *i*-Pr), 23.1 (C14), 28.6 (C2), 31.1 (C1), 33.0 (C11), 43.1 (C4), 112.8 (C8), 114.8 (C6), 129.3 (C5), 132.3 (C10), 144.5 (C9), 153.0 (C7).

A mixture of *cis*-**4** (2.0 g) and 48% HBr (20 ml) in acetic acid (20 ml) was refluxed with stirring for 10 h. The reaction mixture was extracted with hexane and evaporated. Distillation of the residue afforded a mixture of *cis*- and *trans*-**3** in the ratio of 80 : 20 (1.8 g, 94.7%). *trans*-**3**: ^{1}H NMR 0.70 (d, J=7 Hz), 0.97 (d, J=7 Hz). ^{13}C NMR 17.3, 21.2, 21.6, 22.1, 30.6, 31.8, 43.3, 112.7, 113.6.

Lacinilene A (1). The tetralin **3** (2.8 g) was dehydrogenated by heating with 5% Pd–C (0.3 g) for 4 h at 230 °C. The reaction mixture was diluted with benzene, and the catalyst was filtered off. After removing the solvent, the residue was recrystallized from petroleum ether to give **1** as woolly crystals (1.6 g, 59.3%), mp 102.0—103.0 °C (lit,[2] mp 101—102 °C). ^{1}H NMR data were identical with those of the natural lacinilene A.[2] IR 3350, 1620, 1440, 1380, 1200, 860, 820. ^{13}C NMR 19.4 (C14), 23.6 (q, *i*-Pr), 23.6 (q, *i*-Pr), 28.5 (C11), 107.5 (C8), 116.7 (C6), 119.3 (C3), 126.0 (C5), 126.9 (C10), 127.2 (C2), 130.5 (C1), 134.4 (C9), 142.9 (C4), 152.5 (C7). 1,3,5-Trinitrobenzene complex: orange microcrystals (from ethanol), mp 127.0—128.0 °C. Acetate: leaves (from petroleum ether), mp 94.0—95.0 °C (lit,[2] mp 94—96 °C). ^{1}H NMR data were identical with those of the acetate derived from natural lacinilene A.[2] ^{13}C NMR 19.4 (C14), 21.1 (q, Ac), 23.5 (q, *i*-Pr), 23.5 (q, *i*-Pr), 28.5 (C11), 115.8 (C8), 120.2 (C6), 121.2 (C3), 125.4 (C5), 127.2 (C2), 129.5 (C10), 131.7 (C1), 133.7 (C9), 142.8 (C4), 147.9 (C7), 169.6 (C=O).

Concentration of the filtrates followed by chromatography on silica gel with benzene afforded 6-norcadalene (0.1 g).[8]

Lacinilene A Methyl Ether (2). The tetralin **4** (5.5 g) was dehydrogenated by heating with 5% Pd–C (0.5 g) for 4 h at 230 °C. The resulting products were crystallized from petroleum ether to give **2** as prisms (3.8 g, 70.4%), mp 62.0—63.0 °C. IR 1630, 1430, 1260, 1230, 1040, 850, 830, 820. ^{1}H NMR 1.35 (6H, d, J=7 Hz), 2.59 (3H, s), 3.65 (1H, m, J=7 Hz), 3.90 (3H, s), 7.16 (1H, dd, J=9, 3 Hz), 7.23 (3H, broad s), 8.04 (1H, d, J=9 Hz). ^{13}C NMR 19.6 (C14), 23.6 (q, *i*-Pr), 23.6 (q, *i*-Pr), 28.5 (C11), 55.2 (OMe), 103.8 (C8), 117.2 (C6), 119.2 (C3), 125.5 (C5), 126.8 (C10), 127.1 (C2), 130.7 (C1), 134.2 (C9), 142.8 (C4), 157.0 (C7). Picrate: orange microcrystals (from methanol), mp 106.0—107.0 °C. 1,3,5-Trinitrobenzene complex: orange needles (from methanol), mp 92.0—93.0 °C.

The dehydrogenation of **13a** (4.0 g) with 5% Pd–C (1.2 g) for 4 h at 200 °C afforded **2** (1.6 g, 40.0%).

4-(4-Methoxyphenyl)pentanoic Acid (11b). The Grignard reaction of **9** (20.8 g) with methylmagnesium iodide (0.25 mol) in ether (60 ml) and anisole (100 ml) for 8 h at 5 °C gave 4-(4-methoxyphenyl)-γ-valerolactone (**10b**) (16.2 g, 78.6%), bp 153—155 °C/3 mmHg, needles (from petroleum ether), mp 35.0—36.0 °C. IR 1770. ^{1}H NMR 1.67 (3H, s), 2.50 (4H, m), 3.77 (3H, s), 6.87 (2H, d, J=9 Hz), 7.29 (2H, d, J=9 Hz).

The Clemmensen reduction of **10b** (20.6 g) for 6 h gave **11b** (17.9 g, 86.1%), bp 162—164 °C/3 mmHg, needles (from benzene), mp 42.0—43.0 °C. IR 1710. ^{1}H NMR 1.24 (3H, d, J=7 Hz), 1.91 (2H, t, J=7 Hz), 2.22 (2H, t, J=7 Hz), 2.69 (1H, m, J=7 Hz), 3.77 (3H, s), 6.82 (2H, d, J=9 Hz), 7.09 (2H, J=9 Hz), 11.51 (1H, broad).

7-Methoxy-6-norisocalamenene (8). The acid chloride (11.0 g; bp 124—125 °C/3 mmHg; IR 1790) obtained from **11b** was cyclized by $SnCl_4$ (25.3 g) to give 3,4-dihydro-7-methoxy-4-methyl-1(2*H*)-naphthalenone (**12b**) (8.5 g, 92.4%), bp 128—129 °C/3 mmHg. IR 1680. ^{1}H NMR 1.31 (3H, d, J=7 Hz), 1.9 (2H, m), 2.5 (2H, m), 2.9 (1H, m), 3.77 (3H, s), 6.95 (1H, dd, J=8, 2 Hz), 7.18 (1H, d, J=8 Hz), 7.40 (1H, d, J=2 Hz). 2,4-Dinitrophenylhydrazone: reddish orange leaves (from xylene), mp 221.0—221.5 °C.

The Grignard reaction of **12b** (10.0 g) with isopropylmagnesium bromide (0.156 mmol) in ether (30 ml) gave the crude alcohol (12.1 g; IR 3400) as an oil. The dehydration of the crude alcohol (12.1 g) with *p*-toluenesulfonic acid (1.0 g) in refluxing benzene (100 ml) afforded 1,2-dihydro-4-isopropyl-6-methoxy-1-methylnaphthalene (**13b**) (11.1 g) as an oil. IR 1600, 1565, 1490, 1240. ^{1}H NMR 1.14 (9H, d, J=7 Hz), 2.18 (2H, m), 2.80 (2H, m, J=7 Hz), 3.73 (3H, s), 5.75 (1H, t, J=5 Hz), 6.57 (1H, dd, J=8, 2 Hz), 6.78 (1H, d, J=2 Hz), 6.98 (1H, d, J=8 Hz).

The catalytic hydrogenation of **13b** (8.3 g) over 10% Pd–C (0.2 g) in ethanol (50 ml) afforded a mixture of *cis*- and *trans*-**8** in the ratio of 77 : 23 as an oil (6.5 g), bp 112—116 °C/4

mmHg. IR 1610, 1495, 1245, 1050. *cis*-**8**: ^{1}H NMR 0.77 (3H, d, *J*=7 Hz), 1.04 (3H, d, *J*=7 Hz), 1.23 (3H, d, *J*=7 Hz), 1.7 (4H, m), 2.28 (1H, m, *J*=7 Hz), 2.6 (1H, m), 2.8 (1H, m), 3.77 (3H, s), 6.69 (1H, dd, *J*=8, 2 Hz), 6.75 (1H, broad s), 7.06 (1H, d, *J*=8 Hz). ^{13}C NMR 17.5 (q), 19.5 (t), 21.4 (q), 23.4 (q), 28.8 (t), 31.2 (d), 32.1 (d), 43.9 (d), 55.2 (q), 111.3 (d), 113.2 (d), 129.4 (d), 135.4 (s), 141.1 (s), 157.3 (s). *trans*-**8**: ^{1}H NMR 0.71 (d, *J*=7 Hz), 1.00 (d, *J*=7 Hz), 1.25 (d, *J*=7 Hz). ^{13}C NMR 17.4, 21.3, 21.6, 22.3, 32.0, 32.3, 44.2, 55.3, 111.1, 113.4, 127.7, 135.5, 141.4.

6-Norisocalamenen-7-ol (7). The demethylation of **8** (2.0 g) with BBr_3 (4.6 g) in CH_2Cl_2 (35 ml) at −10 °C afforded a mixture of *cis*- and *trans*-**7** in the ratio of 72 : 28 as an oil (1.6 g, 84.2%), bp 129—130 °C/3 mmHg. IR 3300, 1610, 1495, 1460, 1240. *cis*-**7**: ^{1}H NMR 0.73 (3H, d, *J*=7 Hz), 1.00 (3H, d, *J*=7 Hz), 1.21 (3H, d, *J*=7 Hz), 1.66 (4H, m), 2.2 (1H, m), 2.6 (1H, m), 2.8 (1H, m), 5.56 (1H, s), 6.61 (1H, dd, *J*=8, 2 Hz), 6.70 (1H, d, *J*=2 Hz), 6.99 (1H, d, *J*=8 Hz). ^{13}C NMR 17.3 (q), 19.3 (t), 21.3 (q), 23.3 (q), 28.9 (t), 31.0 (d), 32.2 (d), 43.6 (d), 113.1 (d), 114.4 (d), 129.6 (d), 135.5 (s), 141.4 (s), 152.7 (s). *trans*-**7**: ^{1}H NMR 0.68 (d, *J*=7 Hz), 0.97 (d, *J*=7 Hz), 1.23 (d, *J*=7 Hz), 7.08 (d, *J*=8 Hz). ^{13}C NMR 17.2, 21.2, 21.6, 22.3, 31.8, 32.3, 44.0, 112.9, 114.6, 127.9, 135.7, 141.7.

6-Nordaucalen-7-ol (5). The tetralin **7** (2.1 g) was dehydrogenated by heatig with 5% Pd–C (0.2 g) at 230 °C for 4 h to give 2-naphthol **5** (0.6 g, 28.6%), prisms (from petroleum ether), mp 94.0—95.0 °C. IR 3350, 1620, 1390, 1215, 830. ^{1}H NMR 1.30 (6H, d, *J*=7 Hz), 2.60 (3H, s), 3.46 (1H, m, *J*=7 Hz), 5.47 (1H, broad), 7.10 (1H, dd, *J*=9, 2.5 Hz), 7.12 (1H, d, *J*=7.5 Hz), 7.26 (1H, d, *J*=7.5 Hz), 7.43 (1H, d, *J*=2.5 Hz), 7.91 (1H, d, *J*=9 Hz). ^{13}C NMR 19.4 (C14), 23.4(q, *i*-Pr), 23.4 (q, *i*-Pr), 28.5 (C11), 106.5 (C5), 116.5 (C7), 122.1 (C3), 124.4 (C2), 127.0 (C8), 128.4 (C9), 132.0 (C1), 132.8 (C10), 141.3 (C4), 152.9 (C6). Acetate: oil. IR 1765. ^{1}H NMR 1.35 (6H, d, *J*=7 Hz), 2.32 (3H, s), 2.61 (3H, s), 3.59 (1H, m, *J*=7 Hz), 7.18—7.35 (3H, m), 7.81 (1H, d, *J*=2 Hz), 8.00 (1H, d, *J*=9 Hz). ^{13}C NMR 19.4 (C14), 21.2 (q, Ac), 23.5 (q, *i*-Pr), 23.5 (q, *i*-Pr), 28.6 (C11), 114.9 (C5), 120.1 (C7), 122.2 (C3), 126.3 (C2), 126.4 (C8), 131.0 (C9), 132.0 (C1), 132.1 (C10), 142.6 (C4), 148.3 (C6), 169.6 (C=O).

7-Methoxy-6-nordaucalene (6). The naphthalene **6** was prepared from **8** (2.0 g) by the same method as described above for **5**, oil (1.0 g), bp 116 °C/2 mmHg. IR 1620, 1430, 1260, 1220, 830. ^{1}H NMR 1.37 (6H, d, *J*=7 Hz), 2.60 (3H, s), 3.61 (1H, m, *J*=7 Hz), 3.89 (3H, s), 7. 10 (1H, d, *J*=7.5 Hz), 7.15 (1H, dd, *J*=9, 2.5 Hz), 7.26 (1H, d, *J*=7.5 Hz), 7.42 (1H, d, *J*=2.5 Hz), 7.90 (1H, d, *J*=9 Hz). ^{13}C NMR 19.4 (C14), 23.4 (q, *i*-Pr), 23.4 (q, *i*-Pr), 28.6 (C11), 55.2 (OMe), 103.2 (C5), 116.9 (C7), 122.0 (C3), 124.4 (C2), 126.5 (C8), 128.4 (C9), 132.0 (C1), 132.7 (C10), 141.5 (C4), 157.3 (C6). Picrate: brown needles (from ethanol), mp 107.0—108.0 °C. 1,3,5-Trinitrobenzene complex: yellow needles (from methanol), mp 98.0—99.0 °C.

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