was shown to be benzyl nitrate. Three minor peaks had retention times identical with those of α-nitrotoluene, benzaldehyde and benzyl alcohol. Unfortunately, pure benzyl nitrate partially decomposed on the vpc column to give peaks with retention times identical with those of benzaldehyde and benzyl alcohol. Thus it is unclear whether or not these latter compounds are produced in the photochemical reaction. Small amounts (<3%) of phenolic material,[6] possibly nitrophenols (ir and solubility behavior), could be isolated from the original photolysis mixture by extraction with sodium hydroxide.

The photochemical reaction herein reported constitutes another method for the functionalization of alkyl residues. It must also be kept in mind if nitric oxide is employed to scavenge photochemically generated alkyl radicals in solution.

Experimental Section

Reagent grade toluene was fractionally distilled while reagent grade cyclohexane was fractionally distilled from concentrated sulfuric acid prior to use. Nitric oxide (98.5% pure) was purchased from the Matheson Co. Irradiations were conducted in a RPR-100 Rayonet photochemical apparatus employing the 3500-Å range lamps. Vapor phase chromatographic analyses were performed on a 3% Dow 710 on Chromosorb W column at the temperatures indicated below. Cyclohexyl nitrate,[11] 1-nitrocyclohexene,[12] cyclohexyl nitrite,[13] benzyl nitrate,[14] and α-nitrotoluene[15] were prepared by published procedures. Nitrocyclohexane was obtained from the Aldrich Chemical Co. Products isolated by preparative vpc were identified by comparison of their vpc retention times and nmr and ir spectra with those of authentic samples. Relative yields were estimated by cutting out the vpc trace and weighing it. Detector response was assumed to be identical for all compounds. Yields are reported as percentages of the oily reaction product remaining after evaporation of excess solvent.

General Procedure.—A cylindrical Pyrex vessel equipped with a sintered glass gas inlet tube which extended to the bottom of the vessel was charged with 150 ml of solvent. Nitrogen was bubbled through the solvent for 4 hr and then likewise nitric oxide for 15 min prior to irradiation. While a slow stream of nitric oxide was allowed to bubble through the solvent, the initially colorless solution was irradiated for 20 hr and then nitrogen was passed through the resulting yellow solution for 15 min to remove nitrogen oxides. Excess solvent was removed under reduced pressure and shown by vpc and nmr to be essentially free of photolysis products. The remaining oil was used in the experiments below.

Cyclohexane Product Analysis.—A portion of the 1.81 g of yellow-green oil was analyzed by vpc at a column temperature of 104°. Retention times of 3.2, 5.1, and 9.4 min corresponded to cyclohexyl nitrate (20%), nitrocyclohexane (6%), and 1-nitrocyclohexene (3%), respectively. *Ca.* 25% of the yellow-green oil remained nonvolatile at 150° (0.01 mm).

Toluene Product Analysis.—A portion of the 0.567 g of residual brown oil was analyzed by vpc at a column temperature of 123°. A retention time of 4.6 min corresponded to benzyl nitrate (9%).

Registry No.—Nitric oxide, 10102-43-9; cyclohexane, 110-82-7; toluene, 108-88-3.

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[11] N. Kornblum and C. Teitelbaum, *J. Amer. Chem. Soc.*, **74**, 3076 (1952).

[12] A. T. Nielsen, *J. Org. Chem.*, **27**, 2001 (1962).

[13] A. Chrétien and Y. Longi, *C. R. Acad. Sci., Paris*, **220**, 746 (1945).

[14] F. L. M. Pattison and G. M. Brown, *Can. J. Chem.*, **34**, 879 (1956).

[15] N. Kornblum, *Org. Reactions*, **12**, 101 (1962).

The Reaction of Active Methylene Compounds with Carbon Disulfide in the Presence of Ammonia. III. The Reaction of Cyclopentanone and Cycloheptanone[1a]

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It was previously shown[2,3] that acetone and cyclohexanone, when allowed to react with carbon disulfide and ammonia, afforded thiazine derivatives. 2-Thio-1,3-thiazines were obtained from acetone, and 4-thiono-3,1-thiazine from cyclohexanone. Methyl ethyl ketone proved to give both types of compounds in this reaction.[3] Thus the structure of reactant ketone affected the course of reaction.

The present work was designed to test the behavior of cyclopentanone and cycloheptanone in the reaction concerned. Our finding is that cyclopentanone assumed the reaction pattern of cyclohexanone; however, the intermediate, 2-iminocyclopentanedithiocarboxylic acid (I), was isolated rather than the expected thiazine IIa. A number of thiazines were prepared by the coupling of I with ketone in the presence of morpholine: 4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine-2-spirocyclopentane (IIa), 2,2-dimethyl-4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine (IIb), 4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine-2-spirocyclohexane (IIc), 4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine-2-spirocycloheptane (IId), 2,2-diethyl-4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine (IIe), and 2-isopropyl-2-methyl-4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine (IIf). The reaction of I with methyl ethyl ketone was repeated several times, but it proved to be abortive. On the basis of infrared (ir), ultraviolet (uv), and nuclear magnetic resonance (nmr) spectra and the fact that it led to IIa, IIb, IIc, IId, IIe, and IIf, the structure I was assigned. In addition, when allowed to react with 2,4-dinitrophenylhydrazine, I gave cyclopentanone 2,4-dinitrophenylhydrazone accompanying dedithiocarboxylation. Comparison of ir and uv spectra of IIa, IIb, IIc, IId, IIe, and IIf with those of 4(1H)-thiono-5,6,7,8-tetrahydro-4H-3,1-benzothiazine-2-spirocyclohexane and 2-ethyl-2,5,6-trimethyl-4-(1H)-thiono-4H-3,1-thiazine in turn provided evidence for the assignments of the structures of IIa, IIb, IIc, IId, IIe, and IIf.

It may be noteworthy that I is much more sensitive to nickel ion than is dimethyl glyoxime and may be used for analytical purpose.[4]

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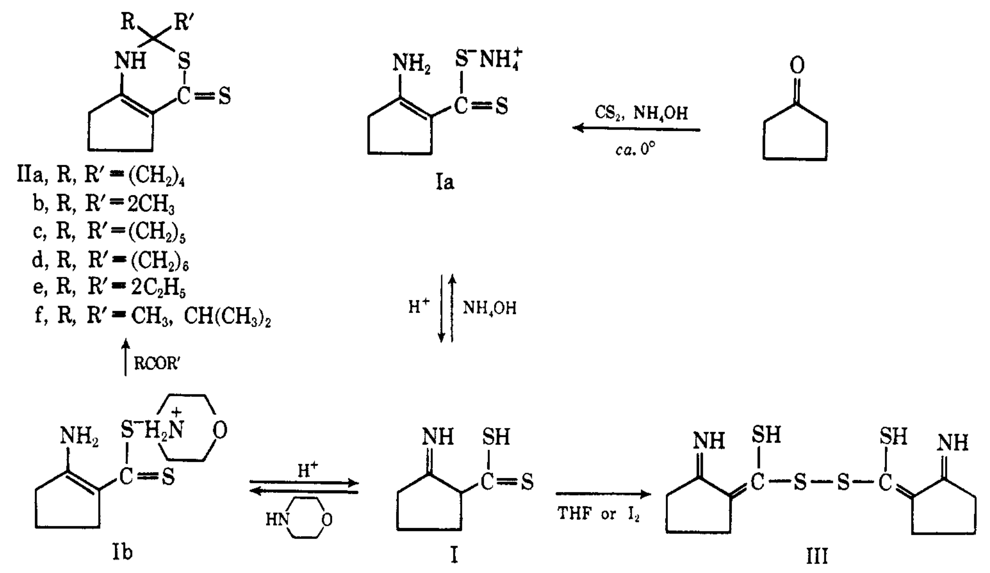
[1] (a) A part of this paper was read at the 21st Annual Meeting of the Chemical Society of Japan, Suita, April 1968. (b) Department of Chemistry, Faculty of Science, Chiba University. (c) Department of Chemistry, Faculty of Science, Shizuoka University. (d) The Institute of Physical and Chemical Research.

[2] T. Takeshima, T. Hayashi, M. Muraoka, and T. Matsuoka, *J. Org. Chem.*, **32**, 980 (1967).

[3] T. Takeshima, T. Imamoto, M. Yokoyama, K. Yamamoto, and M. Akano, *ibid.*, **33**, 2877 (1968).

[4] M. Yokoyama and T. Takeshima, *Anal. Chem.*, **40**, 1344 (1968).

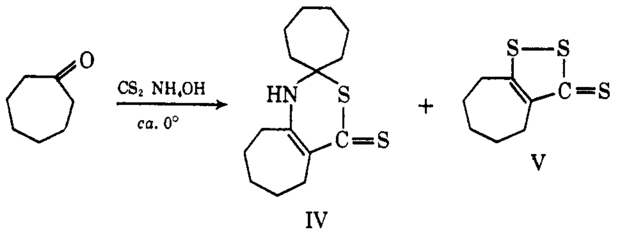
SCHEME I



When I was refluxed with tetrahydrofuran or oxidized with iodine, di(2-iminocyclopentylidenemercaptomethyl) disulfide (III) was produced (Scheme I). The structure of III was tentatively assigned by ir, nmr, and mass spectra.

Cycloheptanone reacted in the same pattern as that of cyclohexanone to give 4(1H)-thiono-5,6,7,8-tetrahydro-4H-3,1-cycloheptathiazine-2-spirocycloheptane (IV) (Scheme II). In addition, a small amount of 4,5,6,7-tetrahydro-1,2-cycloheptadithiole-3-thione (V)[5] was isolated; the same applied to the case of methyl ethyl ketone.[3] The structure IV was assigned in the same way as above (consult Experimental Section).

SCHEME II



Experimental Section

Ammonium 2-Amino-1-cyclopentene-1-dithiocarboxylate (Ia).—A mixture of cyclopentanone (25 g, 0.30 mol), carbon disulfide (30 g, 0.39 mol), and 300 ml of aqueous ammonia (28%) was stirred below 0° for *ca.* 8 hr. The yellow solid product was collected, washed with ether, and dried: yield *ca.* 70 g. When the crude product was recrystallized from ethanol, *ca.* 40 g of yellow plates (Ia) was obtained: yield *ca.* 80%; mp 135–137° dec; uv max (99% EtOH) 301, 388 mμ (log ϵ 3.95, 4.35); ir (KBr) 3390 (m, ν_{NH_2}), 3820 (m, ν_{NH_2}), 3080 (m, $\nu_{NH_4^+}$), 1615 (s, δ_{NH_2}), 1595 (s, $\nu_{conjd\ C=C}$), 1455 (vs, ν_{ring}), 1430 (m, ν_{ring}), 1418 cm^{-1} (s, ν_{ring}). *Anal.* Calcd for $C_6H_{12}N_2S_2$: C, 40.90; H, 6.87; N, 15.90; S, 36.33. Found: C, 41.11; H, 6.69; N, 15.65; S, 36.27. Compound Ia was easily soluble in water and decolorized bromine water. It produced various coloration toward metal ions though much less sensitive than I (see below).

Conversion of Ia into 2-Iminocyclopentanedithiocarboxylic Acid (I).—Compound Ia (5-g) was dissolved in 50 ml of acetic acid at 60–70°. Ammonia gas evolved. The resulting solution was cooled and to this was added water until the solution became turbid. The yellow solid material which separated from the solution was collected and recrystallized from methanol to give *ca.* 1.8 g of yellow plates (I): yield *ca.* 38%; mp 101–102° dec (rapid heating) and 96° dec (slow heating); uv max (*n*-heptane) 309.5, 389 mμ (log ϵ 3.39, 4.10); uv max (99% EtOH) 302, 388 mμ (log ϵ 4.05, 4.38); ir ($CHCl_3$) 3450 (m, ν_{NH}), 1618 (sh, δ_{NH}), 1605 (vs, $\nu_{C=N}$), 1470 (vs, ν_{ring}), 1450 (m, ν_{ring}), 1425 cm^{-1} (m, ν_{ring}); nmr (CD_3SOCD_3) δ 10.70 (m, 1, **NH**), 9.00 (s, 1, **SH**), 3.40 (t, 1, C-1 **H**), 2.95 (t, 2, C-5 **H**$_2$), 2.72 (t, 2, C-3 **H**$_2$), 1.85 (m, 2, C-4 **H**$_2$). *Anal.* Calcd for $C_6H_9NS_2$: C, 45.28; H, 5.70; N, 8.80; S, 40.22. Found: C, 45.29; H, 5.51; N, 9.02; S, 40.14. Compound I was easily soluble in hot water, methanol, ethanol, acetone, and ether and hardly soluble in hydrochloric acid. Dissolved in methanol, I instantly decolorized bromine water. Sodium nitroprusside test showed bluish green. It was remarkably sensitive to Ni(II) ion producing pink-red precipitates or coloration (sensitivity, *ca.* 20 ppb). Moreover, I produced various coloration with other metal ions: Fe(II), dark green; Fe(III), yellowish green; Mn(II), yellowish green; Zn(II), yellowish green; Cu(II), brown; Co(II), brown. These metal ions were much less sensitive than was the nickel ion. When warmed with 2,4-dinitrophenylhydrazine in methanol containing sulfuric acid, I afforded yellow cyclopentanone 2,4-dinitrophenylhydrazone, mp 144–145°, undepressed by admixture with an authentic sample.

Morpholinium 2-Amino-1-cyclopentene-1-dithiocarboxylate (Ib).—Compound I (5 g) was dissolved in 50 ml of morpholine and heated until evolution of ammonia gas subsided. The yellow crystals of I changed to an orange material. This was washed with ether and recrystallized from methanol to give *ca.* 6 g of short orange prisms (Ib): yield *ca.* 80%; mp 173–183° dec (rapid heating) and 157–162° dec (slow heating); uv max (99% EtOH) 303, 393 mμ (log ϵ 3.40, 3.94); ir ($CHCl_3$) 3455 (s, ν_{NH_2}), 3338 (m, ν_{NH_2}), 2850 (m, $\nu_{NH_2^+}$), 1605 (vs, $\nu_{conjd\ C=C}$), 1465 cm^{-1} (s, ν_{ring}); nmr (CD_3COCD_3) δ 3.60 (m, 4, **NH**$_2$, **NH**$_2^+$), 2.75 (m, 8, C-2',3',5',6' **H**$_2$), 2.10 (t, 4, C-3,5 **H**$_2$), 1.60 (m, 2, C-4 **H**$_2$). *Anal.* Calcd for $C_{10}H_{18}S_2O$: C, 48.77; H, 7.37; N, 11.38; S, 26.00. Found: C, 48.68; H, 7.07; N, 11.18; S, 25.71.

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[5] A. Thuillier and J. Vialle, *Bull. Soc. Chim. Fr.*, 2194 (1962).

4(1H)-Thiono-5,6-dihydro-4H-3,1-cyclopentathiazine-2-spirocyclopentane (IIa).—A solution of Ib (5 g) and cyclopentanone (6 g) in 200 ml of ethanol was refluxed for 4 hr. The resulting colorless material was collected and recrystallized from methanol to give *ca.* 0.2 g of morpholinium morpholine-N-dithiocarboxylate, mp 233–234° (in sealed tube).[2,3] The filtrate was concentrated and water was added. The yellow material which separated from the solution was recrystallized from ethanol to give *ca.* 3 g of yellow plates (IIa): mp 189–190°; uv max (99% EtOH) 336, 407 mμ (log ϵ 3.89, 4.36); ir ($CHCl_3$) 3398 (s, ν_{NH}), 1555 (vs, $\nu_{\text{conjd C=C}}$), 1518 cm^{-1} (vs, ν_{ring}); nmr (CF_3COOH) δ 9.50 (br, 1, NH), 2.97 (t, 4, C-5,7 H_2), 2.30 (m, 6, C-6,2′,6′ H_2), 2.05 (m, 4, C-3′,4′ H_2). *Anal.* Calcd for $C_{11}H_{15}NS_2$: C, 58.65; H, 6.71; N, 6.22; S, 28.41. Found: C, 58.77; H, 6.68; N, 6.50; S, 28.18.

2,2-Dimethyl-4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine (IIb).—A mixture of Ib (5 g), acetone (5 g), and 200 ml of ethanol was refluxed for 4 hr and worked up as above to yield *ca.* 3.2 g of yellow plates (IIb): yield 80%, mp 172–173°; uv max (99% EtOH) 336, 406 mμ (log ϵ 3.67, 4.26); ir ($CHCl_3$) 3397 (s, ν_{NH}), 1548 (vs), 1520 (s), 1453 cm^{-1} (m); nmr (CF_3COOH) δ 9.50 (br, 1, NH), 2.95 (t, 4, C-5,7 H_2), 2.27 (m, 2, C-6 H_2), 1.87 (s, 6, $2CH_3$). *Anal.* Calcd for $C_9H_{13}NS_2$: C, 54.26; H, 6.58; N, 7.03; S, 32.13. Found: C, 54.53; H, 6.73; N, 7.11; S, 31.87.

4(1H)-Thiono-5,6-dihydro-4H-3,1-cyclopentathiazine-2-spirocyclohexane (IIc).—A mixture of Ib (5 g), cyclohexanone (6 g), and 200 ml of ethanol was refluxed for 4 hr and worked up as above to yield *ca.* 4 g of yellow plates (IIc): yield 85%; mp 186–187°; uv max (99% EtOH) 334, 406 mμ (log ϵ 3.91, 4.41); ir ($CHCl_3$) 3385 (s), 1550 (vs), 1520 (s), 1450 cm^{-1} (m); nmr (CF_3COOH) δ 9.40 (br, 1, NH), 2.93 (t, 4, C-5,7 H_2), 2.25 (m, 6, C-6,2′,6′ H_2), 1.80 (m, 6, C-3′,4′,5′ H_2). *Anal.* Calcd for $C_{12}H_{17}NS_2$: C, 60.24; H, 7.16; N, 5.85; S, 26.75. Found: C, 60.20; H, 7.09; N, 5.99; S, 26.79.

4(1H)-Thiono-5,6-dihydro-4H-3,1-cyclopentathiazine-2-spirocycloheptane (IId).—A mixture of Ib (5 g), cycloheptanone (4,3 g), and 200 ml of ethanol was refluxed for 4 hr and worked up as above to yield *ca.* 4.0 g of yellow plates (IId): yield 80%; mp 204–205°; uv max (99% EtOH) 338, 408 mμ (log ϵ 3.80, 4.32); nmr (CF_3COOH) δ 9.10 (br, 1, NH), 2.95 (t, 4, C-5,7 H_2), 2.33 (m, 6, C-6,2′,7′ H_2), 1.73 (s, 8, C-3′,4′,5′,6′ H_2). *Anal.* Calcd for $C_{13}H_{19}NS_2$: C, 61.64; H, 7.56; N, 5.53; S, 25.27. Found: C, 61.42; H, 7.40; N, 5.50; S, 25.45.

2,2-Diethyl-4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine (IIe).—A mixture of Ib (5 g), diethyl ketone (10 g), and 200 ml of ethanol was refluxed for 4 hr and worked up as above to yield *ca.* 2.3 g of yellow plates (IIe): yield 50%; mp 183–184°; uv max (99% EtOH) 338, 411 mμ (log ϵ 3.76, 4.19); nmr (CF_3COOH) δ 9.56 (br, 1, NH), 2.95 (t, 4, C-5,7 H_2), 2.35 (m, 6, C-6 H_2, $-CH_2-CH_3$), 2.03 (t, 6, 2-CH_3). *Anal.* Calcd for $C_{11}H_{17}NS_2$: C, 58.13; H, 7.54; N, 6.16; S, 28.16. Found: C, 58.36; H, 7.45; N, 6.19; S, 27.90.

2-Isopropyl-2-methyl-4(1H)-thiono-5,6-dihydro-4H-3,1-cyclopentathiazine (IIf).—A mixture of Ib (5 g), methyl isopropyl ketone (10 g), and 200 ml of ethanol was refluxed for 4 hr and worked up as above to yield *ca.* 2.8 g of yellow plates (IIf): yield 60%; mp 184–185°; uv max (99% EtOH) 338, 409 mμ (log ϵ 3.79, 4.31); nmr (CF_3COOH) δ 9.56 (br, 1, NH), 2.95 (t, 4, C-5,7 H_2), 2.45 [m, 1, $-CH(CH_3)_2$], 2.33 (s, 3, CH_3), 2.15 [d, 6, $-CH(CH_3)_2$], 2.00 (m, 2, C-6 H_2). *Anal.* Calcd for $C_{11}H_{17}NS_2$: C, 58.13; H, 7.54; N, 6.16; S, 28.16. Found: C, 58.34; H, 7.35; N, 6.33; S, 28.09.

Compounds IIa–IIf were easily soluble in methanol, pyridine, and hydrochloric acid and hardly soluble in water; in methanol solution they readily decolorized bromine water.

Di(2-iminocyclopentylidenemercaptomethyl) Disulfide (III).—Compound I (5 g) and an equivalent amount of sodium hydroxide were dissolved in a requisite amount of water and oxidized with iodine solution in the usual way. The yellow material obtained was recrystallized from pyridine–water to yield 4.5 g of yellow plates (III): yield *ca.* 90%; mp 152–153°; uv max (99% EtOH) 308, 398 mμ (log ϵ 4.01, 4.64); ir ($CHCl_3$) 3450 (s, ν_{NH}), 1650 (sh, δ_{NH}), 1630 (sh, $\nu_{C=N}$), 1600 cm^{-1} (vs, $\nu_{\text{conjd C=C}}$); nmr (CD_3SOCD_3) δ 10.80 (m, 2, 2NH), 9.30 (s, 2, 2SH), 2.95 (t, 4, C-5,5′ H_2, J = 6 cps), 2.70 (t, 4, C-3,3′ H_2, J = 6 cps), 1.90 (m, 4, C-4,4′ H_2, J = 8 cps); mass spectrum (75 eV) m/e (rel intensity) 318 (0.04), 316 (0.2), 158 (20), 157 (100), 126 (65), 125 (20), 124 (71), 76 (98). *Anal.* Calcd for $C_{12}H_{16}N_2S_4$: C, 45.50; H, 5.10; N, 8.80; S, 40.50. Found: C, 45.75; H, 4.95; N, 8.90; S, 40.53.

Compound III was also obtained by refluxing I with tetrahydrofuran for 0.5 hr; the yield was 40%.

4(1H)-Thiono-5,6,7,8-tetrahydro-4H-3,1-cycloheptathiazine-2-spirocycloheptane (IV).—A mixture of cycloheptanone (25 g, 0.26 mol), carbon disulfide (30 g, 0.39 mol), and 290 ml of aqueous ammonia (28%) was stirred below 0° for 8 hr. The reaction mixture was kept overnight in an icebox. The yellow solid product was collected, washed with water, and dried: yield *ca.* 20 g. The crude product was recrystallized from methanol to give *ca.* 18 g of yellow plates (IV): yield *ca.* 50%; mp 199–200°; color reaction toward cupric ion, deep red; uv max (99% EtOH) 335, 409 mμ (log ϵ 3.67, 4.32); ir ($CHCl_3$) 3392, (s, ν_{NH}), 1520 (vs, $\nu_{\text{conjd C=C}}$), 1460 (m, ν_{ring}), 1450 cm^{-1} (m, ν_{ring}). *Anal.* Calcd for $C_{15}H_{23}NS_2$: C, 64.03; H, 8.24; N, 4.98; S, 22.75. Found: C, 63.90; H, 7.95; N, 5.09; S, 22.45.

4,5,6,7-Tetrahydro-1,2-cycloheptadithiole-3-thione (V).—The preceding mother liquor from which IV was removed was concentrated under diminished pressure at 40° and allowed to stand at 0° for 3 hr. The yellow solid material was collected and recrystallized from carbon tetrachloride to give *ca.* 3.5 g of yellow needles: yield *ca.* 7%; mp 98–99° (lit.[5] mp 99°); uv max (99% EtOH) 229 244, 279, 312, 413 mμ (log ϵ 4.04, 3.81, 3.86, 3.73, 4.00); ir ($CHCl_3$) 2980 (s, ν_{CH}), 2930 (vs, ν_{CH}), 2855 (s, ν_{CH}), 1520 (m, $\nu_{\text{conjd C=C}}$), 1300 (vs, $\nu_{\text{heteroring}}$), 1150 (vs, $\nu_{C=S}$), 530 cm^{-1} (s, ν_{S-S}); nmr ($CDCl_3$) δ 2.98 (t, 4, C-1′,5′ H_2), 1.75 (m, 6, C-2′,3′,4′ H_2). *Anal.* Calcd for $C_8H_{10}S_3$: C, 47.53; H, 4.99; S, 47.49. Found: C, 47.47; H, 4.96; S, 47.25.

Registry No.—Ammonia, 7664-41-7; cyclopentanone, 120-92-3; cycloheptanone, 502-42-1; I, 18521-91-0; Ia, 18521-92-1; Ib, 18521-93-2; IIa, 18521-94-3; IIb, 18521-95-4; IIc, 18521-96-5; IId, 18521-97-6; IIe, 18521-98-7; IIf, 18521-99-8; III, 18522-00-4; IV, 18522-01-5; V, 6125-89-9.

Acknowledgment.—The authors wish to express their heartfelt thanks to Dr. Hiroshi Midorikawa for his helpful comments and suggestions received during the course of this work. Thanks are also due to Dr. Kiyoko Yamamoto, Dr. Heinosuke Yasuda, Dr. Toshio Hayashi, and Mr. Kenzo Miyairi for their kind help in many respects.

Synthesis of (−)-1,2β,3,4,4a,5,6,7,8,8aβ-Decahydro-4aα,8β-dimethyl-7-oxo-2-naphthoic Acid Methyl Ester

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In connection with studies directed toward the total synthesis of *cis-* and *trans-*fused bicyclic sesquiterpenes of the eudalene group having carbon skeleton I,[1,2] the

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[1] We thank the donors of the Petroleum Research Fund administered by the American Chemical Society for their initial support (Grant No. 283-G) of this program, and the National Institutes of Health for continued support (Grant No. 5-R01-GM-13441-01 to -03). Portions of this Note were abstracted from Y. Wang, M.S. Thesis, Washington University, 1966.

[2] The numbering system and nomenclature used are essentially those of W. Cocker and T. B. H. McMurry, *J. Chem. Soc.*, 4549 (1956).