Depsidone Synthesis. Part 20.[1] Lecideoidin and Dechlorolecideoidin

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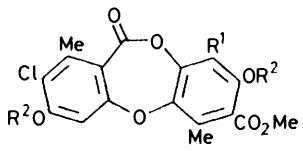
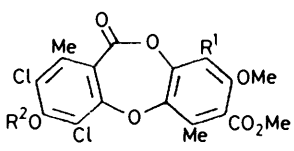
The synthesis of methyl 2,9-dichloro-3,8-dihydroxy-1,6-dimethyl-11-oxo-11*H*-dibenzo[*b*,*e*][1,4]dioxepin-7-carboxylate (lecideoidin) (1) and methyl 2-chloro-3,8-dihydroxy-1,6-dimethyl-11-oxo-11*H*-dibenzo[*b*,*e*][1,4]-dioxepin-7-carboxylate (dechlorolecideoidin) (2) by oxidation of benzophenones and subsequent steps is described.

Two new lichen depsidones, lecideoidin (1) and dechlorolecideoidin (2), both close relatives of gangaleoidin (4),[2] were recently isolated by Elix and his co-workers[3] from an unnamed *Lecidea* species. The structures of the *Lecidea* depsidones followed chiefly from spectroscopic studies but additional evidence was provided by the conversion by chlorination of both di-*O*-methyllecideoidin (3) and *O*-methylgangaleoidin (5) into the same depsidone (6). We sought to confirm the structures of lecideoidin (1) and dechlorolecideoidin (2) by unambiguous total synthesis and we adopted for this purpose a route based on oxidative coupling of suitably substituted benzophenones.[4]

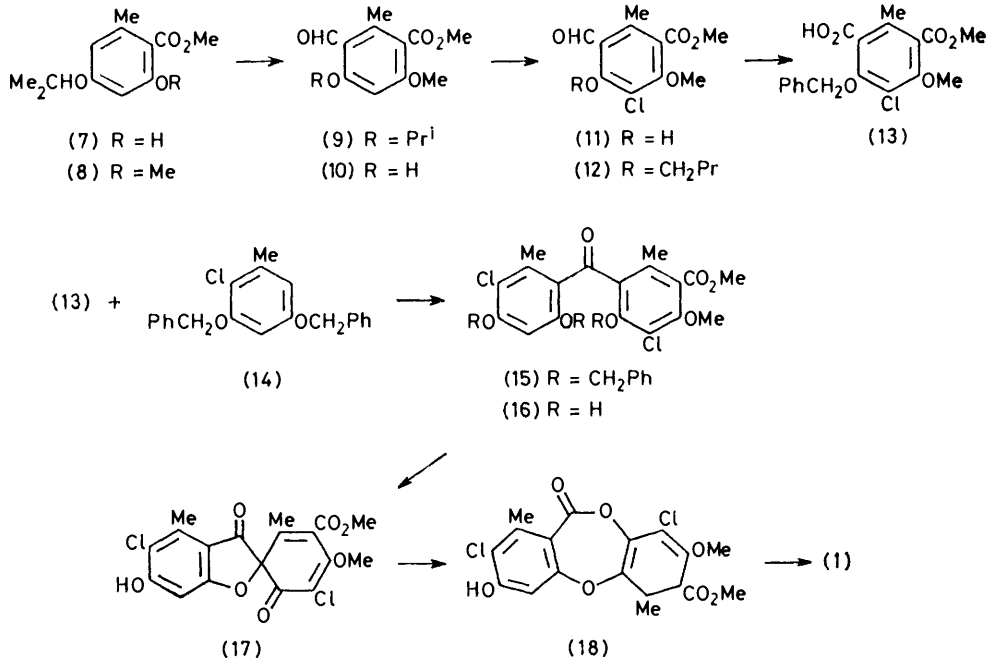


(1) R^1 = Cl, R^2 = H
(2) R^1 = R^2 = H
(3) R^1 = Cl, R^2 = Me



(4) R^1 = R^2 = H
(5) R^1 = H, R^2 = Me
(6) R^1 = Cl, R^2 = Me

For the synthesis of the required benzophenones (16) and (22) the acids (13) and (20) were required. The known aldehyde (10)[5] appeared to be a suitable precursor but its synthesis from methyl orsellinate in four steps proceeds in only *ca.* 20% overall yield. The key step in this sequence is the formylation of methyl 4-hydroxy-2-methoxy-6-methylbenzoate with dichloromethyl ether and titanium(IV) chloride. This reaction gives only a low yield of the desired aldehyde (10) the major product being the *O*-formate of the starting material which is inert to *C*-formylation. Consequently an alternative synthesis of the aldehyde (10) was devised in which the hydroxy-group was protected as an isopropyl ether during formylation.[6]



SCHEME 1

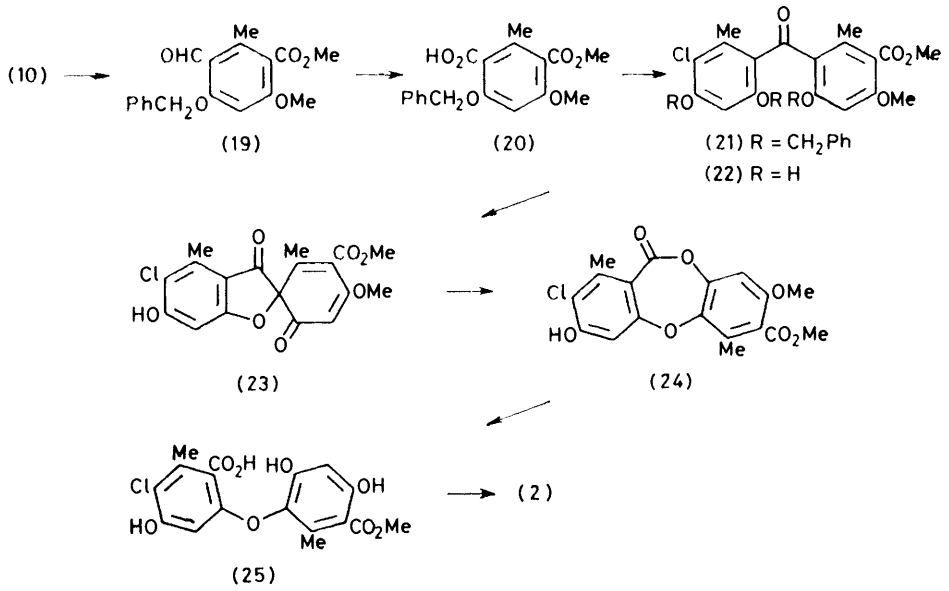
Isopropylation of methyl orsellinate[2] with one molar equivalent each of potassium carbonate and 2-bromopropane in *NN*-dimethylformamide at 80 °C gave the isopropyl ether (7) (75%) (Scheme 1). The derived

methyl ether (8) now underwent smooth formylation with dichloromethyl methyl ether and tin(IV) chloride in dichloromethane at 0 °C. The resultant aldehyde (9) then gave the desired product (10) in 53% overall yield on deprotection with titanium(IV) chloride in dichloromethane at 0 °C. Chlorination of compound (10) with chlorine in acetic acid gave the chlorophenol (11). The derived benzyl ether (12) on oxidation with sodium chlorite in the presence of an excess of sulphamic acid as a chlorine scavenger [7] gave the acid (13).

Friedel–Crafts reaction of the acid (13) and the orcinol (14) [4] gave the tri-*O*-benzylbenzophenone (15) which on hydrogenolytic debenzylation furnished the trihydroxybenzophenone (16). Oxidative coupling of this benzophenone was predicted, in the view of previous results,[4] to furnish only the grisadienedione (17), resulting from preferential formation of a phenoxy-radical on the ring of lower oxidation potential, which would undergo rearrangement *via* a keten [4] to the depsidone (18). The depsidone (18) resulting from this reaction was subjected to demethylation with boron trichloride and thus furnished synthetic lecideoidin (1) identical in all respects with an authentic sample.

Benzylation of the phenol (10) and subsequent oxidation of the product (19) yielded the acid (20) (Scheme 2). This was allowed to react with the orcinol (14) and thus gave the protected benzophenone (21) and thence the trihydroxybenzophenone (22). This benzophenone was predicted to yield the grisadienedione (23) on oxidation, rather than that derived from oxidative entry into the ring bearing two hydroxy-groups and of lower oxidation potential. The grisadienedione (23) could rearrange *via* a keten intermediate [4] to the depsidone (24). In the event this reaction gave only the grisadienedione (23) presumably the cyclohexadienone ring, lacking the chloro-substituent of the previous example (17), is not a sufficiently powerful leaving group for rearrangement to occur. The structure (23) of the grisadienedione was confirmed in the following way. The ^{1}H n.m.r. spectrum of the grisadienedione exhibited singlets at δ 5.52 assigned to an olefinic proton and at δ 6.78 assigned to an aromatic proton. Saturation of the methoxy signal at δ 3.88 gave a 30% enhancement in the integral of the olefinic proton and no nuclear Overhauser effect at the aromatic proton.

Thermolysis of the grisadienedione (23) in boiling phenetole gave the depsidone (24).[4] Unexpectedly this depsidone on treatment with boron trichloride gave the acid (25), which also resulted from similar treatment of the grisadienedione (23). However lactonization of the acid (25) was achieved by treatment with trifluoroacetic anhydride and the product was identical with dechlorolecideoidin (2) by the usual criteria.



SCHEME 2

EXPERIMENTAL

General directions have been given previously.[8]

Methyl 2-Hydroxy-4-isopropyloxy-6-methylbenzoate (7).—Methyl orsellinate (22.0 g), 2-bromopropane (15.0 g), dry potassium carbonate (16.7 g), and dry *NN*-dimethylformamide (220 ml) were stirred under dry nitrogen at 80 °C (bath) for 26 h. The mixture was poured into water and extracted with ethyl acetate. The crude product was chromatographed over silica gel with 0—2.5% ethyl acetate–light petroleum as eluant. The *product* (7) crystallized from pentane as prisms (20.5 g), m.p. 55—56.5 °C (Found: C, 64.1; H, 7.0%; M^+, 224. $C_{12}H_{16}O_4$ requires C, 64.25; H, 7.2%; *M*, 224), δ(CDCl$_3$, 90 MHz) 1.32 (6 H, d, Me), 2.47 (3 H, s, Me), 3.90 (3 H, s, OMe), 4.55 (1 H, s, septet, CH), 6.29 (2 H, s, ArH), and 11.74 (1 H, s, D$_2$O exchangeable OH).

Methyl 3-Formyl-4-hydroxy-6-methoxy-2-methylbenzoate (10).—The phenol (7) (14.4 g), dry potassium carbonate (15.0 g), dimethyl sulphate (1.5 g), and acetone (200 ml) were

stirred and boiled under reflux for 20 h. The usual work-up gave the methyl ether (8) as an oil (14.6 g), δ($CDCl_3$, 60 MHz), 1.32 (6 H, d, Me), 2.21 (3 H, s, Me), 3.67 and 3.76 (each 3 H, s, OMe), 4.55 (1 H, septet, CH), and 6.12 (2 H, s, ArH). Tin(IV) chloride (5.7 g) in dry dichloromethane (25 ml) was added dropwise at 0 °C over 0.5 h to a stirred solution of the methyl ether (14.6 g) and dichloromethyl methyl ether (9.7 g) in dry dichloromethane (75 ml). The mixture was stirred at 0 °C for 4 h and then work-up in the usual way gave the crude oily aldehyde (9) (15.0 g), δ($CDCl_3$, 60 MHz) 1.37 (6 H, d, Me), 2.43 (3 H, s, Me), 3.77 (6 H, s, OMe), 4.60 (1 H, septet, CH), 6.19 (1 H, s, ArH), and 10.32 (1 H, s, CHO). The aldehyde (9) (14.2 g) in dry dichloromethane (120 ml) was stirred at 0 °C and treated dropwise with titanium(IV) chloride (17.8 g) in dry dichloromethane (70 ml). The mixture was stirred at 0 °C for 4.5 h and then poured onto ice. The crude product was passed down a column of silica gel with 5—15% ethyl acetate–light petroleum as eluant. The aldehyde (10) formed prisms (9.3 g) (from dichloromethane–light petroleum), m.p. 133—134.5 °C (lit.,[5] 134—134.5 °C).

Methyl 5-Chloro-3-formyl-4-hydroxy-6-methoxy-2-methylbenzoate (11).—The aldehyde (10) (5.2 g) and chlorine (1.82 g) were dissolved in acetic acid (120 ml) and the mixture was allowed to stand in a darkened vessel for 22 h. The mixture was then poured into water and worked up as usual. The *chloroaldehyde* (11) crystallized from ether–light petroleum as prisms (4.2 g), m.p. 95—96 °C (Found: C, 51.1; H, 4.35; Cl, 13.55%; M^+, 258, 260. $C_{11}H_{11}ClO_5$ requires C, 51.1; H, 4.3; Cl, 13.7%; *M*, 258, 260), δ($CDCl_3$, 90 MHz), 2.52 (3 H, s, Me), 3.93 and 3.98 (each 3 H, s, OMe), 10.23 (1 H, s, CHO), and 12.93 (1 H, s, OH).

Methyl 4-Benzyloxy-5-chloro-3-formyl-6-methoxy-2-methylbenzoate (12).—The substrate (11) (4.2 g), benzyl bromide (1.74 g), dry potassium carbonate (4.6 g), and dry *NN*-dimethylformamide (40 ml) were stirred together at room temperature under dry nitrogen for 2 h. The usual work-up gave the *aldehyde* (12) as prisms (4.1 g) (from ether–light petroleum), m.p. 81—83 °C (Found: C, 62.2; H, 5.0; Cl, 10.25%; M^+ 348, 350. $C_{18}H_{17}ClO_5$ requires C, 62.0; H, 4.9; Cl, 10.15%; *M*, 348, 350), δ($CDCl_3$, 90 MHz) 2.46 (3 H, s, Me), 3.95 and 3.98 (each 3 H, s, OMe), 5.10 (2 H, s, CH_2), 7.41 (5 H, m, Ph), and 10.29 (1 H, s, CHO).

6-Benzyloxy-5-chloro-4-methoxy-3-methoxycarbonyl-2-methylbenzoic Acid (13).—Sodium chlorite (80%, technical, 1.3 g) in water (13 ml) was added dropwise to a stirred solution of the aldehyde (12) (3.1 g) and sulphamic acid (3.1 g) in dioxan (100 ml) and water (30 ml). The mixture was stirred for 0.5 h and then poured into water and extracted with ethyl acetate. The *acid* (13) crystallized from ether–light petroleum as prisms (3.0 g), m.p. 123—126 °C (Found: C, 59.6; H, 4.9; Cl, 9.7%; M^+, 364, 366. $C_{18}H_{17}ClO_6$ requires C, 59.25; H, 4.7; Cl, 9.7%; *M*, 364, 366), δ($CDCl_3$, 90 MHz), 2.28 (3 H, s, Me), 3.92 and 3.94 (each 3 H, s, OMe), 5.09 (2 H, s, CH_2), 6.75br (1 H, OH), and 7.34 (5 H, m, Ph).

Methyl 4-Benzyloxy-3-(4,6-bisbenzyloxy-3-chloro-2-methylbenzoyl)-5-chloro-6-methoxy-2-methylbenzoate (15).—Trifluoroacetic anhydride (7 ml) in dry dichloroethane (50 ml) was added in a thin stream at 0 °C to a stirred solution of the acid (13) (1.4 g) in dry dichloroethane (70 ml). The orcinol (14)[4] (5.2 g) in dry dichloroethane (70 ml) was then added dropwise at 0 °C over 15 min The mixture was then stirred at room temperature for 20 h and then boiled under reflux for 5 h. The cooled solution was then diluted with ether and washed in turn with aqueous ammonia, water, and finally saturated brine. The crude product was chromatographed over silica gel with 2.5—10% ethyl acetate–light petroleum as eluant. The *benzophenone* (15) crystallized from dichloromethane–light petroleum as needles (900 mg), m.p. 159—160 °C (Found: Cl, 9.85%; M^+, 684, 686, 688. $C_{39}H_{34}Cl_2O_7$ requires Cl, 10.35%; *M*, 684, 686, 688), δ($CDCl_3$, 90 MHz), 2.10 and 2.23 (each 3 H, s, Me), 3.90 and 3.91 (each 3 H, s, OMe), 4.30br (2 H, CH_2), 4.75 and 5.13 (each 2 H, s, CH_2), 6.42 (1 H, s, ArH), and 7.24 (15 H, m, Ph).

Methyl 5-Chloro-3-(3-chloro-4,6-dihydroxy-2-methylbenzoyl)-4-hydroxy-6-methoxy-2-methylbenzoate (16).—The benzophenone (15) (890 mg) in ethyl acetate (100 ml) containing concentrated hydrochloric acid (4 drops) was stirred with 10% palladium charcoal (400 mg) under hydrogen until absorption ceased. The usual work-up gave the *benzophenone* (16) as a viscous oil (540 mg) (Found: M^+, 414.0232. $^{12}C_{18}{}^1H_{16}{}^{35}Cl_2{}^{16}O_7$ requires *M*, 414.0273); δ($CDCl_3$, 60 MHz) 1.96 (6 H, s, 2 × Me), 3.83 (6 H, s, 2 × OMe), 6.33br (1 H, OH), 6.58 (1 H, s, ArH), and 7.08 and 11.68 (each 1 H, s, OH).

Methyl 2,9-Dichloro-3-hydroxy-8-methoxy-1,6-dimethyl-11-oxo-11H-dibenzo[b,e][1,4]*dioxepin-7-carboxylate* (18)—A stirred solution of the benzophenone (16) (400 mg) and potassium carbonate (4.7 g) in water (100 ml) was treated dropwise with a solution of potassium hexacyanoferrate(III) (810 mg) in water (50 ml). The solution was stirred for 10 min and then acidified with dilute hydrochloric acid and extracted with ethyl acetate. The crude product was filtered through a column of silica gel with 10% ethyl acetate–light petroleum as eluant. The *depsidone* (18) crystallized from dichloromethane–light petroleum as prisms (240 mg), m.p. 213—216 °C (Found: C, 52.55; H, 3.45%; M^+, 412, 414, 416. $C_{18}H_{14}Cl_2O_7$ requires C, 52.3; H, 3.4%; *M*, 412, 414, 416), δ($CDCl_3$, 80 MHz) 2.37 and 2.56 (each 3 H, s, Me), 3.86 and 3.93 (each 3 H, s, OMe), 6.16 (1 H, s, OH), and 6.82 (1 H, s, ArH).

Methyl 2,9-Dichloro-3,8-dihydroxy-1,6-dimethyl-11-oxo-11H-dibenzo[b,e][1,4]*dioxepin-7-carboxylate* (*Lecideoidin*) (1).—Boron trichloride (100 mg) in dry dichloromethane (20 ml) was added at −10 °C to a stirred solution of the depsidone (18) (44 mg) in dry dichloromethane (20 ml). The mixture was stirred at −10 °C for 15 min and at room temperature for 30 min. The mixture was poured into water and extracted with ethyl acetate. The *depsidone* (1) crystallized from toluene as needles (35 mg), m.p. 246—248 °C (lit.,[3] 247 °C) identical (mixed m.p., mass and ^1H n.m.r. spectra, and R_F values in three solvent systems) with an authentic sample (Found: C, 51.25; H, 3.1%; M^+, 398, 400, 402. $C_{17}H_{12}Cl_2O_7$ requires C, 51.15; H, 3.05%; *M*, 398, 400, 402), δ($CDCl_3$, 80 MHz) 2.56 and 2.66 (each 3 H, s, Me), 3.99 (3 H, s, OMe), 5.40 (1 H, s, OH), 6.83 (1 H, s, ArH), and 11.81 (1 H, s, OH).

Methyl 4-Benzyloxy-3-formyl-6-methoxy-2-methylbenzoate (19).—Benzylation of the phenol (10) as above gave the aldehyde (19) as prisms (from dichloromethane–light petroleum), m.p. 128—129.5 °C (lit.,[5] 128—129.5 °C).

6-Benzyloxy-4-methoxy-3-methoxycarbonyl-2-methylbenzoic Acid (20).—Oxidation of the aldehyde (19) (3.8 g) with sodium chlorite, as above, gave the *acid* (20) which crystallized from dichloromethane–light petroleum as prisms (3.7 g), m.p. 165—167 °C (Found: C, 65.75; H, 5.7%; M^+, 330. $C_{18}H_{18}O_6$ requires C, 65.45; H, 5.5%; *M*, 330), δ($CDCl_3$, 90 MHz) 2.37 (3 H, s, Me), 3.79 and 3.90

(each 3 H, s, OMe), 5.18br (1 H, OH), 5.19 (2 H, s, CH_2), 6.39 (1 H, s, ArH), and 7.36 (5 H, s, Ph).

Methyl 4-Benzyloxy-3-(4,6-dibenzyloxy-3-chloro-2-methylbenzoyl)-6-methoxy-2-methylbenzoate (21).—The acid (20) (2.2 g), the orcinol (14) (9.0 g), and trifluoroacetic anhydride (11 ml) were allowed to react as before. After 24 h at room temperature, work-up and chromatography over silica gel with 5—15% ethyl acetate–light petroleum as eluant gave the *benzophenone* (21) which crystallized from dichloromethane–light petroleum as prisms (1.8 g), m.p. 167—168 °C (Found: C, 72.0; H, 5.55; Cl, 5.4%; M^+, 650, 652. $C_{39}H_{35}ClO_7$ requires C, 71.95; H, 5.4; Cl, 5.45%; *M*, 650, 652), δ($CDCl_3$, 90 MHz) 2.10 and 2.14 (each 3 H, s, Me), 3.79 and 3.86 (each 3 H, s, OMe), 4.70 (4 H, s, 2 × CH_2), 5.08 (2 H, s, CH_2), 6.25 and 6.32 (each 1 H, s, ArH), and 7.24 (15 H, m, 3 × Ph).

Methyl 3-(3-Chloro-4,6-dihydroxy-2-methylbenzoyl)-4-hydroxy-6-methoxy-2-methylbenzoate (22).—Hydrogenolytic debenzylation of the *O*-benzylbenzophenone (21) (1.7 g), as before, gave the *benzophenone* (22) which crystallized from methanol–chloroform as prisms (740 mg), m.p. 193.5—194.5 °C (Found: C, 56.6; H, 4.6%; M^+, 380, 382. $C_{18}H_{17}ClO_7$ requires C, 56.8; H, 4.5%; *M*, 380, 382), δ($CDCl_3$, 80 MHz) 1.81 and 2.01 (each 3 H, s, Me), 3.85 and 3.87 (each 3 H, s, OMe), 6.02 (1 H, s, D_2O exchangeable OH), 6.44 and 6.58 (each 1 H, s, ArH), and 8.59 and 10.54 (each 1 H, s, D_2O exchangeable OH).

Methyl 5-Chloro-2,3-dihydro-6-hydroxy-4,6′-dimethyl-4′-methoxy-2′,3-dioxospiro[benzofuran-2,1′-cyclohexa-3′,5′-diene]-5′-carboxylate (23).—Oxidation of the benzophenone (22) (670 mg) as before, and work-up after 30 min gave the *grisadienedione* (23) which crystallized from acetone–ether as prisms (600 mg), m.p. 150—170 °C with resolidification to needles, m.p. 224—228 °C (Found: C, 57.2; H, 4.1; Cl, 9.4%; M^+, 378, 380. $C_{18}H_{15}ClO_7$ requires C, 57.1; H, 4.0; Cl, 9.35%; *M*, 378, 380), δ($CDCl_3$, 90 MHz) 1.79 (3 H, s, vinyl Me), 2.55 (3 H, s, ArMe), 3.88 (6 H, s, 2 × OMe), 5.52 (1 H, s, vinyl), and 6.78 (1 H, s, ArH), $\nu_{max.}$($CHCl_3$) 1 742 (coumaranone C=O), 1 671 (dienone C=O), and 1 610 (C=C) cm^{-1}; $\lambda_{max.}$(EtOH) 284 and 330 nm (ε 15 200 and 10 700).

Methyl 2-Chloro-3-hydroxy-8-methoxy-1,6-dimethyl-11-oxo-11H-dibenzo[b,e][1,4]dioxepin-7-carboxylate (24).—The grisadienedione (23) (500 mg) was heated under reflux in phenetole (37 ml) for 10 min under dry nitrogen. The solvent was removed by steam distillation and the residue was crystallized from dichloromethane–light petroleum forming needles (412 mg) of the *depsidone* (24), m.p. 226—228 °C (Found: C, 57.15; H, 4.1; Cl, 9.3%; M^+, 378, 380. $C_{18}H_{15}ClO_7$ requires C, 57.1; H, 4.0; Cl, 9.35%; *M*, 378, 380), δ($CDCl_3$, 80 MHz) 2.37 and 2.54 (each 3 H, s, Me), 3.78 and 3.90 (each 3 H, s, OMe), 6.12 (1 H, s, D_2O exchangeable OH), and 6.66 and 6.80 (each 1 H, s, ArH). The *methyl ether*, prepared in the usual way (iodomethane, potassium carbonate, *NN*-dimethylformamide) formed needles (from dichloromethane–light petroleum), m.p. 240.5—241 °C (Found: C, 57.8; H, 4.05; Cl, 9.3%; M^+, 392, 394. $C_{19}H_{17}ClO_7$ requires C, 58.1; H, 4.35; Cl, 9.05%; *M*, 392, 394), δ($CDCl_3$, 80 MHz) 2.40 and 2.54 (each 3 H, s, Me), 3.78, 3.90, and 3.94 (each 3 H, s, OMe), and 6.66 (2 H, s, ArH).

2-(4,6-Dihydroxy-3-methoxycarbonyl-2-methylphenoxy)-5-chloro-4-hydroxy-6-methylbenzoic Acid (25).—(*a*) Boron trichloride (100 mg) in dry dichloromethane (20 ml) was added at −10 °C to a stirred solution of the depsidone (24) (35 mg) in dry dichloromethane (35 ml). The mixture was stirred at −10 °C for 15 min and then worked up in the usual way. The *acid* (25) crystallized from methanol as prisms (23 mg), m.p. 212—215 °C (decomp.) (Found: C, 53.2; H, 4.0; Cl, 9.1%; M^+, 382, 384. $C_{17}H_{15}ClO_8$ requires C, 53.35; H, 3.9; Cl, 9.3%; *M*, 382, 384), δ(CD_3SOCD_3), 80 MHz), 2.07 and 2.28 (each 3 H, s, Me), 3.78 (3 H, s, OMe), and 6.04 and 6.35 (each 1 H, s, ArH).

(*b*) Boron trichloride (640 mg) in dry dichloromethane (125 ml) was added at −10 °C to a stirred solution of the grisadienedione (23) (280 mg) in dichloromethane (125 ml). The mixture was stirred at −10 °C for 15 min and at room temperature for 30 min. The usual work-up gave the acid (25) as prisms (115 mg) (from methanol), m.p. 212—215 °C (decomp.).

Methyl 2-Chloro-3,8-dihydroxy-1,6-dimethyl-11-oxo-11H-dibenzo[b,e][1,4]dioxepin-7-carboxylate (*Dechlorolecideoidin*) (2).—The acid (25) (65 mg), dry toluene (1.0 ml), and trifluoroacetic anhydride (0.16 ml) were stirred together at room temperature for 2 h. The solvents were removed under reduced pressure and the residue was subjected to preparative t.l.c. The major band gave *dechlorolecideoidin* (2) which formed needles (30 mg) (from dichloromethane–light petroleum), m.p. 237—238 °C (lit.,[3] 237 °C) identical (mixed m.p., mass and ^1H n.m.r. spectra, and R_F values in three solvent systems) with an authentic sample (Found: C, 56.1; H, 3.4; Cl, 9.8%; M^+, 346, 366. $C_{17}H_{13}ClO_7$ requires C, 56.0; H, 3.6; Cl, 9.7%; *M*, 346, 366), δ($CDCl_3$, 80 MHz) 2.54 and 2.67 (each 3 H, s, Me), 3.97 (3 H, s, OMe), and 6.72 and 6.84 (each 1 H, s, ArH).

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