by double-resonance-decoupling and 2-D NMR.[13] The structures of **1-3** follow directly from that of **4**. The chemical shifts of the carbons of **3** and **4** (supplementary material) are quite similar to those of tetrahydro-β-carboline skeletons found in known terrestrial alkaloids.[8,17]

The stereochemistry of the oxathiazepine ring is assigned tentatively as shown from the chemical shifts of C-1 (46.8 ppm) and H-1 (4.33 ppm), which argue a cis-fused ring,[8] the H-1, H-10 coupling constant (3.0 Hz), which suggests a cis relationship, and the CD spectra (MeOH) of **3** and **4**, both of which show a positive Cotton effect in the 250–300 nm region, indicating an α-configuration for H-1.[18]

Eudistomins K and L, two other eudistomins with the same ring system, were obtained by similar procedures and have been assigned structures **5** [$[\alpha]^{25}_D$ −102° (*c* 0.2, MeOH)] and **6** [$[\alpha]^{25}_D$ −77° (*c* 0.2, MeOH)], respectively ($C_{14}H_{17}BrN_3OS$, HRFABMS Δ 0.4 mmu for **5**, Δ 0.6 mmu for **6**). Both have indole UV chromophores like those of **1** and **2**. Their ^1H NMR spectra (Table II) agree completely with those of eudistomins C and E in the tetrahydropyridine and oxathiazepine ring regions and assign the substitution patterns shown for the benzene ring when compared with other reported bromoindoles.[11,12] Eudistomin K inhibits HSV-1 growth at 250 ng/disk and eudistomin L at 100 ng/disk.

Eudistomins C, E, K, and L can be considered to be biosynthetically derived from tryptophan (N-2–C-9a) and cysteine (C-1, C-10, C-11, and S-12).[19]

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Registry No. **1**, 88704-50-1; **2**, 88704-51-2; **3**, 88704-54-5; **4**, 88704-53-4; **5**, 88704-52-3; **6**, 88704-55-6.

Supplementary Material Available: ^{13}C NMR chemical shifts of **3** and **4** (1 page). Ordering information is given on any current masthead page.

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Eudistomins A, D, G, H, I, J, M, N, O, P, and Q, Bromo-, Hydroxy-, Pyrrolyl-, and 1-Pyrrolinyl-β-carbolines from the Antiviral Caribbean Tunicate *Eudistoma olivaceum*[1]

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Eudistomins C, E, K, and L, containing an oxathiazepinotetrahydro-β-carboline ring system and isolated from the colonial Caribbean tunicate *Eudistoma olivaceum*, the most active antiviral species assayed during the Alpha Helix Caribbean Expedition 1978,[2] are reported in the preceding communication.[3] In the present Communication we describe the isolation of eudistomins A, D, G, H, I, J, M, N, O, P, and Q from the extract of *E. olivaceum*. We assign the structures of eudistomins A, D, J, M, N, and O as the substituted β-carbolines **1–6** (Table I), respectively, and assign eudistomins G, H, I, P, and Q the structures **11–13**, **16**, and **17** (Table I), respectively, containing the previously unreported 1-pyrrolinyl-β-carboline ring system. These eudistomins exhibit modest activity against Herpes simplex virus, type 1 (D, G, H, I, N, and O), *Saccharomyces cerevisiae*, a yeast (H, N, O, and P), and *Bacillus subtilis*, a gram-positive bacterium (D, I, N, O, P, and Q).

Use of reversed-phase MPLC,[3] silica gel MPLC with chloroform–methanol (95:5), and, finally, silica gel HPLC with chloroform–methanol (98:2 for **1**, **5**, and **6**, 95:5 for **2**) afforded eudistomins A, N, O, and D, respectively, as yellow oils. Eudistomins J and M (**3** and **4**) were isolated as their acetyl derivatives **8** and **9** by silica gel HPLC ($CHCl_3$) following acetylation of crude fractions from silica gel MPLC.

The UV spectrum of a mixture of **5** and **6**[4] was quite characteristic of β-carbolines,[5] while FABMS[6a] showed a single M + H ion ($C_{11}H_8BrN_2$, Δ 0.3 mmu).[6b] The ^1H NMR spectrum of the mixture was well resolved for the isomers (Table II), however, and was indicative of two 3,4-unsubstituted β-carbolines,[7] with a bromine assigned to C-6 for **5** and to C-7 for **6** by comparison to model indoles.[8] Eudistomin N (**5**) has now been synthesized in three steps from tryptamine and glyoxylic acid.

Eudistomin D (**2**), like N and O, contains a β-carboline UV chromophore,[4] but the two maxima at longest wavelength are shifted bathochromically (347 → 373 nm and 335 → 361 nm), and eudistomin D contains an oxygen not found in N and O

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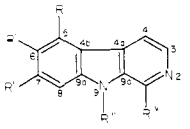
Table I



	R	R′	R″	R‴	R^{iv}
1 (A)[a]	H	OH	Br	H	*b*
2 (D)	Br	OH	H	H	H
3 (J)	H	OH	Br	H	H
4 (M)	H	OH	H	H	*b*
5 (N)	H	Br	H	H	H
6 (O)	H	H	Br	H	H
7	Br	OAc	H	Ac	H
8	H	OAc	Br	Ac	H
9	H	OAc	H	H	*b*
10	H	OAc	Br	H	*b*
11 (G)[a]	H	H	Br	H	*c*
12 (H)	H	Br	H	H	*c*
13 (I)	H	H	H	H	*c*
14	H	H	Br	H	*d*
15	H	H	Br	H	*e*
16 (P)	H	OH	Br	H	*c*
17 (Q)	H	OH	H	H	*c*

[a] Letters refer to eudistomin components. [b] 2-Pyrrolyl. [c] 1-Pyrridin-2-yl. [d] 2-Pyrrolidinyl. [e] *N*-Acetylpyrrolidin-2-yl.

($C_{11}H_8BrN_2O$, HRFABMS Δ 1.1 mmu). An aromatic hydroxyl was confirmed by conversion of **2** to its diacetyl derivative **7** ($C_{15}H_{12}BrN_2O_3$, HRFABMS Δ 0.7 mmu; IR 1780 cm^{-1}) and located at C-6 by the characteristic coupling for H-7 and H-8. Synthetic **2** has now been prepared in five steps from 5-methoxytryptamine and glyoxylic acid. The diacetyl derivative ($C_{15}H_{12}BrN_2O_3$, HRFABMS Δ 0.2 mmu) of eudistomin J (**3**) was assigned as **8** from its ^1H NMR spectrum relative to (Table II).

Eudistomins A and M contain the β-carboline ring system substituted by a 2-pyrrolyl group at C-1. Eudistomin M (**4**) was isolated as its acetate (**9**, $C_{17}H_{14}N_3O_2$, HRFABMS Δ 0.9 mmu), and eudistomin A (**1**, $C_{15}H_{11}BrN_3O$, HRFABMS Δ 1.8 mmu) was converted to its acetate (**10**, $C_{17}H_{13}BrN_3O_2$, HRFABMS Δ 2.0 mmu). Both acetates had aryl ester bands at 1760 cm^{-1} and UV spectra[4] with the β-carboline chromophore's[5] longer wavelength bands shifted bathochromically. The ^1H NMR spectra of **9**, **1**, and **10** (Table II) retain H-3 and H-4 but lack H-1, while the oxygen is located at C-6 and the bromine at C-7 by comparison to eudistomin J and its acetate. The remaining C_4N group is identified as a 2-substituted pyrrole (H-1′, 8.77 s br; H-3′, 6.94 m, J = 2.4 Hz; H-4′, 6.49 m, J = 2.4 Hz; H-5′, 7.11 m).[9] Additional support for the structures assigned derives from the ^{13}C NMR spectrum of **1**,[4] compared to those of β-carbolines[10] and 2-substituted pyrroles.[11,12]

After the methanol–toluene extract of *E. olivaceum* (IRCE 1-VII-81-3-1) was diluted with sodium nitrate,[3] the toluene-soluble layer was subjected to silica gel column chromatography ($CHCl_3$) to give a mixture of eudistomins G, H, and I (**11**–**13**) from which

Table II. ^1H NMR Data for Eudistomins and Their Derivatives

	δ, ppm, multiplicity (J, Hz)[a]																
	5	**6**	**2**	**7**	**8**	**9**	**1**	**10**	**11**	**15**	**12**	**13**	**16**	**17**
H-1	8.94, s	8.94, s	8.98, s[b]	9.58, s	9.49, s									
H-3	8.40, d (5.2)	8.40, d (5.0)	8.55, d (5.4)	8.71, d (5.5)	8.63, d (5.3)	8.35, d (5.4)	8.29, d (5.1)[b]	8.37, d (5.1)[b]	8.49, d (5.1)	8.27, d (5.1)	8.50, d (5.1)	8.48, d (5.0)	8.42, d (5.4)	8.41, d (5.0)[b]
H-4	8.11, d (5.2)	8.07, d (5.0)	8.39, d (5.4)	8.67, d (5.5)	7.92, d (5.3)	7.81, d (5.4)	7.82, d (5.1)	7.92, d (5.1)	7.99, d (5.1)	7.70, d (5.1)	7.99, d (5.1)	8.03, d (5.0)	8.09, d (5.4)	8.09, d (5.0)
H-5	8.43, d (1.7)	8.18, d (8.4)	Br	Br	7.89, s	7.86, d (1.4)	7.79, s	8.11, s	8.04, d (8.3)	7.82, d (8.4)	8.31, d (1.4)	8.18, d (7.8)	7.84, s	7.67, d (1.0)
H-6	Br	7.40, dd (8.4, 1.2)	OH	OAc	OAc	OAc	OH	OAc	7.42, dd (8.3, 1.0)	7.28, dd (8.4, 1.6)	Br	7.31, t (7.8, 7.1)	OH	OH
H-7	7.66, dd (8.7, 1.7)	Br	7.36, d (8.7)	7.43, d (8.8)	Br	7.32, d (8.8, 1.4)	Br	Br	Br	Br	7.66, d (8.7, 1.4)	7.58, t (8.1, 7.1)	Br	7.20, dd (8.8, 1.0)
H-8	7.61, d (8.7)	7.84, d (1.2)	7.58, d (8.7)	8.40, d (8.8)	8.72, s	7.63, d (8.8)	7.91, s	8.02, s	7.77, d (1.0)	7.45, dd (1.6)	7.52, d (8.7)	7.62, d (8.1)	8.10, s	7.71, d (8.8)
H-9	10.87, s br	10.87, s br				10.40, s br	10.90, s br	10.92, s br						
H-5′									4.26, m	3.64, m	4.23, m	4.27, m	4.22, m	4.24, m
H-4′									2.08, m	2.12, m	2.09, m	2.08, m	2.08, m	2.10, m
H-3′									3.28, m	2.67, m (6.7)	3.30, m	3.30, m	3.25, m	3.25, m
H-2′										5.85, d (6.7)				
NH									10.93, s	11.23, s	11.01, s	10.91, s	11.08, s br	10.99, s br

[a] Nicolet NT-360; CD_2Cl_2, except as noted. [b] CD_3COCD_3.

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11 (0.0015% wet weight) crystallized from hexane–ethyl acetate (2:1) and was recrystallized from methylene chloride to yield colorless needles, mp 204–206 °C ($C_{15}H_{12}BrN_3$, HREIMS Δ 2.0 mmu). The mother liquid on C_{18} reversed-phase MPLC (MeOH:H_2O, 9:1) gave **12** (0.0011%, yellow powder, mp 140–142 °C, $C_{15}H_{12}BrN_3$, HREIMS Δ 0.7 mmu) and **13** (0.0010%, colorless powder, mp 153–155 °C, $C_{15}H_{13}N_3$, HREIMS Δ 1.5 mmu). The UV spectra of **11–13**[4] argue the presence of a β-carboline chromophore.[5] Signals at 176.3–176.8 ppm in the ^{13}C spectra of **11–13**[4] are assignable to an imino carbon (C=N)[13] and deuterium-exchangeable signals at 10.9–11.0 ppm to an NH proton (Table II). Reduction of **11** (FABMS, M + H, *m/z* 314, Br) with sodium borohydride in methanol gave amine **14** (FABMS, M + H, *m/z* 316, Br), which was acetylated to **15** (FABMS, M + H, *m/z* 358, Br; NCO, 1650 cm^{-1}). The UV spectrum of **15**[4] is nearly identical with that of the β-carboline harman.[5,14] The ^1H NMR spectra of **11–13** (Table III) establish the substitution pattern as a β-carboline skeleton,[7,10,14,15] in which the benzenoid ring is unsubstituted in **13** but substituted in **11** and **12** by bromine at C-7 and C-6, respectively.[8] The ^{13}C chemical shifts assignable to C-1 through C-9a of **11–13**[4] also agree well with those of known β-carbolines.[10]

The three coupled methylene groups of **11–13** near 4.2, 2.1, and 3.3 ppm (Table II) may be assigned to H-5′, H-4′, H-3′, and ^{13}C signals near 62.0, 34.8, and 21.7 ppm [4] to C-5′, C-4′, and C-3′, respectively. The three-carbon unit $CH_2CH_2CH_2$ must be attached to the imine nitrogen at one end (CH_2 near 4.2 and 62.0 ppm) and to the C=N group (C-2′) at the other [CHNAc of **15** (Table II) coupled ($J_{2',3'}$ = 6.7 Hz) to a terminal CH_2 group (near 3.3 and 21.7 ppm)], thus completing the assignments as **11–13**.

Two additional eudistomins belong to this 1-pyrrolinyl-β-carboline ring system. More polar, eudistomins P [**16**, mp 128–130 °C ($C_{15}H_{13}BrN_3O$, HRFABMS Δ 1.6 mmu)] and Q [**17**, mp 120–125 °C ($C_{15}H_{14}N_3O$, HRFABMS Δ 0.3 mmu)] were isolated as minor products from the chloroform layer which yielded eudistomins A, D, J, M, N, and O (cf. above) and C and E.[3] Their bromohydroxy-β-carboline ring system is assigned from their UV spectra (like eudistomins D and J), while their ^1H NMR spectra (Table II) assign benzene ring patterns like those of J (P) and M (Q) and their 1-pyrrolinyl and pyridine ring pattern like that of **11–13**.

The eudistomins in the present report are all considered to be biosynthetically derived from 1 mol of tryptophan (C-3–C-9a, N-2, N-9). Eudistomins A and M, as well as G, H, I, P, and Q, are presumed to contain, in addition, glutamate-derived units—C-1 and the pyrrole ring in A and M, C-1, and the pyrrolinyl ring in G, H, I, P, and Q.

Acknowledgement.[16] We thank Dr. R. G. Hughes, Jr., Roswell Park Memorial Institute, for advice on antiviral assays, and Dr. F. Lafargue, Université de Paris, for identification of *Eudistoma olivaceum*.

Registry No. 1, 88704-36-3; **2**, 88704-37-4; **3**, 88704-38-5; **4**, 88704-39-6; **5**, 59444-69-8; **6**, 88704-40-9; **7**, 88729-60-6; **8**, 88704-41-0; **9**, 88704-42-1; **10**, 88729-61-7; **11**, 88704-43-2; **12**, 88704-44-3; **13**, 88704-45-4; **14**, 88704-46-5; **15**, 88704-47-6; **16**, 88704-48-7; **17**, 88704-49-8.

Supplementary Material Available: UV data for eudistomins and their derivatives and ^{13}C NMR shifts of **1** and **11–13** (2 pages). Ordering information is given on any current masthead page.

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cis-Diamminedichloroplatinum(II) Induced Distortion in a Double-Helical DNA Fragment

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Since Rosenberg's discovery,[2] that *cis*-diamminedichloroplatinum(II) (cis-platinum or cis-Pt) displays antitumor activity, findings from several laboratories clearly indicate that the bifunctional cis-Pt reacts with DNA after hydrolysis inside the cells, resulting in *cis*-$Pt(NH_3)_2^{2+}$ binding preferentially to two neighboring guanine bases on the same strand of DNA.[3] This suggestion was originally made by Stone, Sinex, and Kelman[3a] and subsequently evidenced by Bauer, Lippard, Haseltine, and coworkers.[3b-d] Several authors have suggested that the thus induced double-helix distortion is quite severe, resulting in denaturation of the DNA up to several base pairs.[4] In order to study this proposal, we investigated the decamer double helix (III) (see abbreviations)[5] after binding of cis-Pt to the central G-G sequence.

Our results indicate that—at least below 28°C—all central base pairs remain intact after chelation of *cis*-$Pt(NH_3)_2^{2+}$ by the G-G sequence. However, structural changes are induced, and the melting temperature appears to be lowered with respect to the non-platinated duplex.

The deoxynucleotide decamers I and II were synthesised by using an improved phosphotriester approach.[6] Strand I, d(T-C-T-C-G-G-T-C-T-C), has the chelating G-G dimer situated in the center and no other reactive sites are present for Pt binding. The other strand has the complementary sequence d(G-A-G-A-C-C-G-A-G-A) (for numbering used, see abbreviations).[5]

The chelation of cis-Pt at both guanine N7 positions of the purified product, obtained after reaction of strand I with an equimolar amount of cis-Pt (I-Pt), was ascertained with the use of high-frequency proton NMR. We studied the pH dependency of the nonexchangeable base protons[7] (see Figure 1), and by the

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(5) Abbreviations: cis-Pt, *cis*-diamminedichloroplatinum(II); NOE, nuclear Overhauser enhancement; DSS, 4,4-dimethyl-4-silapentanesulfonic acid sodium salt. Decamers: I, d(T-C-T-C-G-G-T-C-T-C) (numbering, T(1), C(2)–C(10)); I-Pt, d(T-C-T-C-G-G-T-C-T-C)-cis-Pt (platinum bound at both guanine N7 atoms); II, d(G-A-G-A-C-C-G-A-G-A) (numbering, G(11), A(12)–A(20)); III, I + II; III-Pt, I-Pt + II.
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