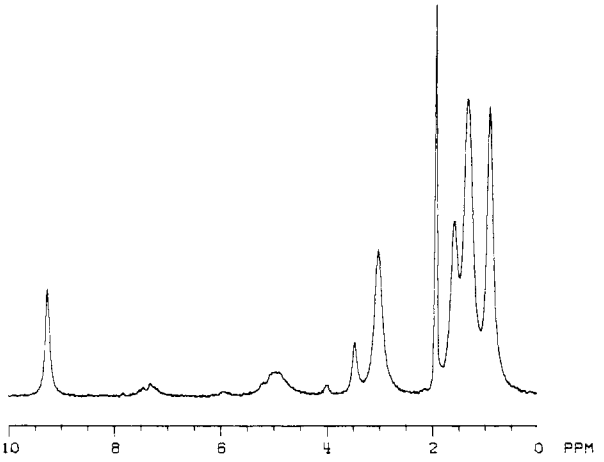

Figure 2. ^1H NMR spectra at 300 MHz in CD_3CN of solid isolated from a reaction mixture similar to that described in Figure 1 but containing 0.1 *M* nPe_4NBr. No additional resonances were seen in the range −60 to +60 ppm.[14]

by Reynolds and Holm to trap the dimer by introduction of the quaternary ammonium halide prior to addition of sulfur.[3] Yields of 50–55% of the $n = 2$ cluster are obtained with much of the remainder being the more soluble $n = 4$ cluster. When this strategy was followed in the aqueous Triton reaction mixture described above by making the buffer 0.1 M in tetra-*n*-pentylammonium bromide (*n*-Pe_4NBr) a bluish-black emulsion resulted upon introduction of the Triton/CH_3CN slurry.[12] After addition of solid sulfur, the emulsion slowly changed to a purplish-red color. Over the course of 2 days, the emulsion gradually broke down to a dark solid plus a faintly colored solution. Filtration and thorough washing with ether, water, and ether followed by drying in vacuo resulted in a solid which, when dissolved in CD_3CN, gives the ^1H NMR spectrum shown in Figure 2 in the 0–10 ppm range. The isotropically shifted resonances at 9.3 (meta H), 4.9 (ortho H), and 3.4 (para H) ppm are identical with those reported for $[Fe_2S_2(SPh)_4]^{2-}$ in this solvent.[3,14] Analysis of the solid is consistent with that calculated for $(n\text{-}Pe_4N)_2[Fe_2S_2(SPh)_4]$. Anal. Calcd for $C_{64}H_{108}N_2S_8Fe_4$: C, 63.55; H, 9.00; N, 2.32; Fe, 9.23; S, 15.90. Found: C, 63.27; H, 9.13; N, 2.27; Fe, 9.11; S, 15.75. On the basis of the starting amount of iron the yield is 96%. Substitution of increasingly shorter chain tetraalkylammonium salts for $n\text{-}Pe_4N^+$ in the above reaction results in solids contaminated by increasing proportions of $[Fe_4S_4(SPh)_4]^{2-}$. A possible explanation for this selectivity is that ion pairing with the relatively hydrophobic $n\text{-}Pe_4N^+$ inhibits exposure of $[Fe_2S_2(SPh)_4]^{2-}$ to water. Protic solvents including aqueous Triton are known to accelerate conversion of $[Fe_2S_2(SPh)_4]^{2-}$ to $[Fe_4S_4(SPh)_4]^{2-}$.[1,3,8,15] In the presence of Triton, $n\text{-}Pe_4N^+$ could thus function as a "phase-transfer catalyst" by increasing the solubility of $[Fe_2S_2(SPh)_4]^{2-}$ in hydrophobic regions of the Triton micelles or aggregates.[16] Decreased solubility of $[Fe_2S_2(SPh)_4]^{2-}$ in the presence of $n\text{-}Pe_4NBr$ may also play a role. Omission of Triton from the above reaction results in essentially complete precipitation within ~2 h. The solid consists of $(n\text{-}Pe_4N)_2[Fe_2S_2(SPh)_4]$ contaminated by 20–25% of $(n\text{-}Pe_4N)_2[Fe_4S_4(SPh)_4]$ based on relative areas of the meta H ^1H NMR resonances of the solid dissolved in CD_3CN.[17]

In these aqueous reaction mixtures, Triton X-100 appears to increase the solubility of FeSPh species resulting in (i) greater contact both among these species and between these species and solid sulfur, which may increase reaction rates, and (ii) greater homogeneity of reaction solutions or dilutions thereof (Figure 1) permitting the use of solution spectroscopies to characterize reaction progress. In this regard ^{19}F NMR could help us to delineate the assembly pathway(s) in aqueous Triton.[18]

In these aqueous-based media, the title clusters can be prepared in yields equivalent to or higher than those obtained in organic solvents. The primary objective of this work is to develop a system that could be used to address synthetic questions stemming from the likely presence of water during at least some stages of biological FeS and MoFeS cluster assembly. The aqueous-based reaction systems described here appear to be sufficiently well-behaved to fulfill this objective.

Acknowledgment. This research was supported in part by NSF Grant PCM-8216447. The NMR spectra were obtained on a NIC-300 spectrometer purchased with funds from NSF equipment Grant CHE-8209709.

(14) Peaks upfield of 3.4 ppm are due either to nPe_4N^+ or CH_3CN. Multiplet at ~7.5 ppm is due to PhSSPh. The UV–vis spectrum of the solid dissolved in DMF is identical to that published for $(Et_4N)_2[Fe_2S_2(SPh)_4]$ in the same solvent.[11]

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(17) Solid isolated after six days from a mixture of $(Et_4N)_2[Fe_4S_4(SPh)_4]$ and 0.1 *M* nPe_4NBr in buffered aqueous Triton gave isotropically shifted ^1H NMR resonances in CD_3CN due only to $[Fe_4S_4(SPh)_4]^{2-}$.

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Eudistomins C, E, K, and L, Potent Antiviral Compounds Containing a Novel Oxathiazepine Ring from the Caribbean Tunicate *Eudistoma olivaceum*[1]

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Of the 650 marine species assayed during the 1978 Alpha Helix Caribbean Expedition (AHCE 1978)[3] the colonial tunicate *Eudistoma olivaceum*[4] was the most active against *Herpes simplex* virus, type 1 (HSV-1). In the present report we assign structures **1**, **2**, **5**, and **6** (Table I), containing the previously unreported condensed oxathiazepine ring system, to eudistomins C, E, K, and L, respectively, which include the most active antiviral components of *E. olivaceum*. In the following communication[5] we assign the structures of eudistomins A, D, G, H, I, J, M, N, O, P, and Q, additional bioactive components isolated from *E. olivaceum*.

The methanol–toluene (3:1) extract[3] of *E. olivaceum* (IRCE 1-VII-81-3-1, IFE 21-V-82-1-3) was partitioned with toluene and water. The toluene phase yielded eudistomins G, H, and I (see following communication),[5] while extraction of the aqueous phase with chloroform yielded an oil, which was subjected to C_{18} reversed-phase medium-pressure liquid chromatography (MPLC) with methanol–water (7:3) then to silica gel MPLC

(1) Taken in part from: Harbour, G. C. Ph.D. Thesis, University of Illinois, Urbana, IL, 1983.

(2) (a) The University of Illinois. (b) Roswell Park Memorial Institute. (c) The Upjohn Co.

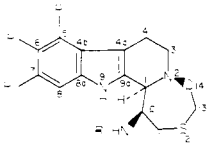
(3) Rinehart, K. L., Jr.; Shaw, P. D.; Shield, L. S.; Gloer, J. B.; Harbour, G. C.; Koker, M. E. S.; Samain, D.; Schwartz, R. E.; Tymiak, A. A.; Weller, D. L.; Carter, G. T.; Munro, M. H. G.; Hughes, R. G., Jr.; Renis, H. E.; Swynenberg, E. B.; Stringfellow, D. A.; Vavra, J. J.; Coats, J. H.; Zurenko, G. E.; Kuentzel, S. L.; Li, L. H.; Bakus, G. J.; Brusca, R. C.; Craft, L. L.; Young, D. N.; Connor, J. L. *Pure Appl. Chem.* **1981**, *53*, 795–817. Cf. especially Table VIII.

(4) The tunicate was identified by Dr. F. Lafargue, Laboratoire Arago, Université de Paris VI, France.

(5) Kobayashi, J.; Harbour, G. C.; Gilmore, J.; Rinehart, K. L., Jr. *J. Am. Chem. Soc.*, following paper in this issue.

0002-7863/84/1506-1524$01.50/0

Table I



	R	R′	R″	R‴
1 (C)[a]	H	OH	Br	H
2 (E)	Br	OH	H	H
3	H	OAc	Br	Ac
4	Br	OAc	H	Ac
5 (K)	H	H	Br	H
6 (L)	H	Br	H	H
7 (F)	H	OH	Br	$C_2H_3O_2$

[a] Letters refer to eudistomin components.

The remaining portions of **1** and **2** ($C_6H_{12}N_2OS$, $C_8H_{14}N_2O_2S$ for **3** and **4**) were assigned from the ^1H NMR spectra of **1**–**4**. Extensive spin decoupling (Table II) of the 500-MHz spectrum of **4** revealed the isolated units $CHCH(NHCOCH_3)CH_2$ (H-1, H-10, and H-11),[10] CH_2 (H-13), and CH_2CH_2 (H-3 and H-4), to which must only be added a nitrogen, an oxygen, and a sulfur.[13] The chemical shifts (4.84 and 4.96 ppm) for H-13 correlated very well with those of H-2 of 4,6-dimethyl-1,3-thioxane (4.67 and 4.72 ppm),[14] and the chemical shift (71.0 ppm) of C-13 with that of C-2 of 1,3-thioxane (71.2 ppm),[15] thus assigning the unit SCH_2O; no other chemical shift was appropriate for a CHO proton (Table II).

The unit can be extended to $OCH_2SCH_2CH(NHAc)CH$ by the loss of C_5H_9NOS (HREIMS Δ 1.1 mmu), by the correspondence of C-11 (32.1 ppm) to the CH_2S carbons of pyochelin[16] and by the small coupling between H-11 and H-13, established by 2-D NMR, which locates these protons in the same ring. The oxygen of the unit is not attached to carbon (from the ^{13}C NMR spectrum, supplementary material) and must be attached to the unassigned nitrogen, as must the terminal CH group (H-1; 4.13 ppm). The chemical shifts of the CH_2CH_2 group (H-3, H-4) indicate bonding to aryl and nitrogen, as in $ArCH_2CH_2NOC$-$H_2SCH_2CH(NHAc)CH$, requiring the final bond (C-1, C-9a) be formed as in **4**. In agreement with this ring system, small long-range coupling between H-1 and H-3 (or H-4) is established

Table II. ^1H NMR Signals for Eudistomins C, E, K, L and Acetyl Derivatives

	δ, ppm, multiplicity (*J*, Hz)					
proton	**1**[a,b]	**3**[a,c]	**2**[a,b]	**4**[c,d]	**6**[a,b]	**5**[a,b]
H-1	4.00, s br	4.12, s br	4.00, s br	4.13, dd (3.0, 1.5, 1.5)	4.06, s br	4.05, s br
H-3	2.70, m	2.89, m	3.52, m	3.38, m (11.3, 2.5, 1.5)	2.77, m	2.78, m
	2.70, m	2.79, m	2.99, m	3.14, m (12.0, 11.3, 4.0)	2.77, m	2.78, m
H-4	3.01, m	3.12, m	3.25, m	3.10, m (12.0, 9.0, 2.5)	3.04, m	3.05, m
	3.54, m	3.60, m	3.52, m	3.60, m (9.0, 4.0, 1.5)	3.54, m	3.55, m
H-5	6.93, s	7.16, s			7.60, d (1.3)	7.36, d (8.4)
H-6						7.16, dd (8.4, 1.5)
H-7			6.73, d (8.5)	6.82, d (8.6)	7.20, dd (8.5, 1.3)	
H-8	7.45, s	7.56, s	7.14, d (8.5)	7.14, d (8.6)	7.28, d (8.5)	7.52, d (1.5)
H-9	8.97, s br	8.76, s br	9.08, s br	9.11, s br	9.30, s br	9.23, s br
H-10	3.54, m	5.00, m (10.1)	3.52, m	5.05, m (10.0, 5.8, 3.0, 1.5)	3.54, m	3.55, m
H-11	3.25, d (14.6)	3.34, d (14.6)	3.25, m (14.6)	3.33, d (14.6, 1.5)	3.29, m	3.29, m
	2.70, m (14.6)	2.79, m (14.6)	2.73, m (14.6)	2.79, dd (14.6, 5.8)	2.77, m	2.78, m
H-13	4.73, d (9.1)	4.83, d (9.0)	4.72, d (9.1)	4.84, d (9.1)	4.77, d (9.1)	4.77, d (9.2)
	4.87, d (9.1)	4.96, d (9.0)	4.86, d (9.1)	4.96, d (9.1)	4.90, d (9.1)	4.91, d (9.2)
10-NH		7.66, d (10.1)		6.65, d (10.0)		
10-NAc		1.80, s		1.81, s		
O-Ac		2.37, s		2.36, s		

[a] 360 MHz. [b] CD_3CN. [c] $CDCl_3$. [d] 500 MHz.

($CHCl_3$:MeOH, 9:1) and silica gel HPLC ($CHCl_3$:MeOH, 96.5:3.5 for **1** and 95:5 for **2**) to give eudistomins C [0.0017%, wet weight; $[\alpha]^{25}_D$ −52° (*c* 0.4%, MeOH); $C_{14}H_{17}BrN_3O_2S$, HRFABMS[6] Δ 2.6 mmu] and E [0.0015%; $[\alpha]^{25}_D$ −18° (*c* 0.1%, MeOH); $C_{14}H_{17}BrN_3O_2S$, HRFABMS Δ 1.8 mmu] as pale yellow oils. In preliminary antiviral assays, eudistomins C and E showed very strong inhibition of HSV-1 at 50 ng/12.5 mm disk.

The UV spectra of eudistomins C and E [e.g., **1**, λ_{max}^{MeOH} 226 nm (ε 23 400), and 287 nm (8000)] argue the presence of an indole chromophore,[7] the infrared spectra show a broad band at 3250 cm^{-1} which can be assigned to NH or OH (but no carbonyl band), and in the ^1H NMR spectra (Table II) the D_2O-exchangeable proton at 8.97 ppm for **1** (9.08 for **2**) can be assigned to the indole NH.[8] The coupling constants (Table II)[9] and numbers of aromatic protons assign 2,3,5,6- and 2,3,4,5-tetrasubstituted (indole numbering) indole nuclei for **1** and **2**, respectively, with two additional rings required.

Compounds **1** and **2** were converted to their diacetyl derivatives **3** [$[\alpha]^{25}_D$ −43° (*c* 0.8, $CHCl_3$); $C_{18}H_{20}BrN_3O_4S$, HREIMS Δ 0.8 mmu] and **4** [$[\alpha]^{25}_D$ +18° (*c* 0.5, $CHCl_3$); HREIMS Δ 0.4 mmu] [UV spectra[7] like those of **1** and **2**, IR ($CHCl_3$) 1760 and 1655 cm^{-1}, establishing a phenol and an aliphatic amine].

(6) Rinehart, K. L., Jr. *Science* (*Washington, D. C.*) **1982**, *218*, 254–260. HRFABMS data refer to the M + H ion and HREIMS to the M ion.
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(9) The lower field aromatic proton is H-8,[10] since it shifts downfield (7.45 → 7.69 for **1**, 7.14 → 7.41 ppm for **2**) in acetone-d_6[11,12] while the other aromatic proton has nearly the same chemical shift (6.93 → 6.99 for **1**, 6.73 → 6.78 ppm for **2**) in acetone-d_6.
(10) The numbering system employed follows that for β-carbolines (Patterson, A. M.; Capell, L. T.; Walker, D. F. "The Ring Index", 2nd ed.; American Chemical Society: Washington, DC, 1960; p 372). For the present fused ring system a different numbering system would be employed (present C-10, C-11, C-3 = Patterson C-1, C-2, C-7, etc.), but to facilitate ^1H NMR comparisons with other eudistomins[5] we have retained the β-carboline numbering.
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(13) The 500-MHz ^1H NMR spectrum of **4** allows one to estimate coupling constants of almost all protons, as shown in Table II. The smaller coupling constants ($J_{10,11}$ and $J_{1,3(4')}$, all 1–2 Hz), including long-range couplings, were observed by double-resonance decoupling, while the presence of tiny couplings ($J_{11(11'),13(13')}$ and $J_{1,3'(4)}$, all <1 Hz) was revealed by a 2-D ^1H NMR study of **4**.
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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]

Acknowledgment. The Alpha Helix Expedition 1978 was supported by a grant from the National Science Foundation (PCM 77-12584) and post-expedition studies in Urbana by the National Institute of Allergy and Infectious Diseases (AI 04769). Mass spectrometry was provided in part by grants from the National Institute of General Medical Sciences (GM 27029) and the National Science Foundation (PCM-8121494) and NMR spectroscopy in part by the University of Illinois NSF Regional Instrumentation Facility (NSF CHE 79-16100). We thank J. C. Cook and M. K. Cochran for mass spectra, Dr. N. S. Scott and D. W. Phillipson for NMR spectra, T. G. Holt for antiviral assays, and L. S. Shield for editorial assistance, all at the University of Illinois, and Dr. W. C. Krueger and M. D. Prairie, The Upjohn Company, for CD spectra. We also thank the governments of Mexico and Belize for permission to carry out scientific research in their territorial waters and Dr. M. E. Rice and her associates at the Smithsonian Tropical Research Center, Fort Pierce, Florida for their help and the use of their facilities.

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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(19) A fifth compound, eudistomin F [$C_{16}H_{18}BrN_3O_4S$, HREIMS Δ 0.2 mmu], also belongs to the same oxathiazepine group, with a UV spectrum nearly identical with that of eudistomin C and ^1H NMR signals like those for **1** in Table II. Mass spectral losses of $C_4H_7NO_2$ and $C_5H_9NO_2S$ [HREIMS] locate the additional $C_2H_2O_2$ unit on C-10, C-11, or 10-N of eudistomin C and allow the assignment of partial structure **7**.

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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Received September 29, 1983

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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