increasing the ligand field strength).[12] In both cases, however, the excited state lifetime was compromised. By contrast, caging increases both the photochemical stability and the excited state lifetime.

Acknowledgment. Work supported by Progetto Strategico CNR "Reazioni di Trasferimento Monoelettronico", Ministero della Pubblica Istruzione (Italy), Swiss National Science Foundation, and Deutsche Forschungsgemeinschaft.

Stereochemical Assignment of Neocarzinostatin Chromophore. Structures of Neocarzinostatin Chromophore–Methyl Thioglycolate Adducts†

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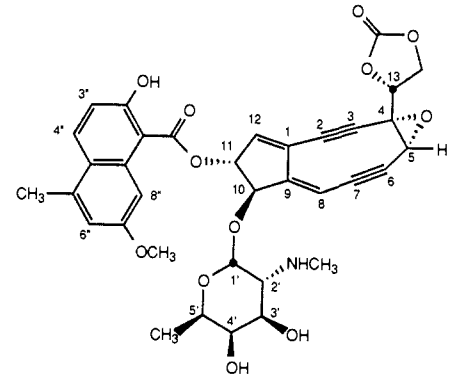
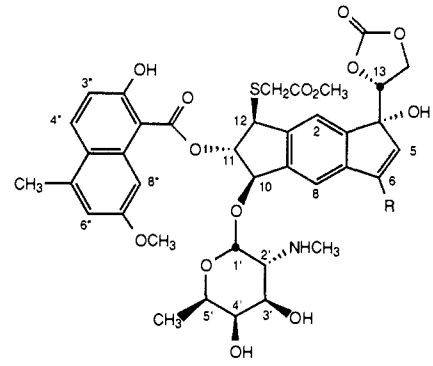
The nonprotein component of the antitumor antibiotic neocarzinostatin[1] (neocarzinostatin chromophore, **1**)[2,3] undergoes rapid and irreversible reaction with thiols to produce a species which is capable of cleaving DNA upon aerobic incubation.[4] Goldberg and co-workers first demonstrated that reaction of **1** with methyl thioglycolate produces a 1:1 adduct with the added incorporation of two hydrogen atoms.[5] Subsequently, we proposed structure **2** (planar form) for this adduct and presented the mechanism outlined in Scheme I to account for its formation.[6] We describe herein the isolation and complete characterization of **2** and a new product, the bisthiol adduct **3**. The full stereochemical assignment of **2** and, by induction, of **1** is also reported.

Dissociation of **1** from its protein complex was achieved with >85% efficiency by a modification of the procedure of Goldberg et al.[5,7] A freshly prepared solution of **1** ($6.6 \cdot 10^{-4}$ M) in 0.5 M methanolic acetic acid was deoxygenated at −78 °C and treated with excess distilled, deoxygenated methyl thioglycolate (**4**, 300 equiv) with reaction at −78 °C for 2 h followed by slow warming to 0 °C (10 °C/h). Volatiles were removed at 0 °C, 0.05 mm to provide a mixture containing **2** and **3** (ca. 1:1) as the major products. Preparative thin-layer chromatography (48:48:4 ethanol:benzene:acetic acid, R_f values 0.31 and 0.43 for **2** and **3**, respectively) with subsequent and final purification over Sephadex LH-20 (dichloromethane) provided **2** and **3** as amorphous films.[8]



1



2 R = H

3 R = $SCH_2CO_2CH_3$

High resolution FABMS (calcd for $[M + H]^+$, $C_{38}H_{42}NO_{14}S$: 768.2326; found: 768.2429) of **2** confirmed its formulation as **1** + $HSCH_2CO_2CH_3$ + H_2. The FTIR spectrum (neat film) showed that the carbonate and naphthoate groups had been preserved (1807 and 1644 cm^{-1}, respectively) and indicated incorporation of **4** (1738 cm^{-1}). All carbon-bound proton resonances were well-resolved in the 400 MHz ^1H NMR spectrum ($CDCl_3$). In addition to readily discernible signals for the *N*-methylfucosamine, naphthoate, methyl thioglycolate, and carbonate appendages, seven resonances attributable to the rearranged core were visible: two aromatic and three nonaromatic singlets (δ 7.77, 7.23, 5.78, 5.24, 4.63) and two coupled olefinic doublets (δ 6.94, 6.30, J = 5.6 Hz). 2D-COSY revealed a coupling pathway linking the five singlets (C2–C12–C11–C10–C8–C2).[9] Whereas vanishingly small positive NOEs were observed in $CDCl_3$ ($\omega\tau_c \approx 1$), large negative NOEs could be obtained in dimethyl-d_6 sulfoxide (DMSO-d_6):D_2O (2:1, 23 °C, Figure 1).[10] Qualitatively similar information was obtained in a two-dimensional version of the CAMELSPIN experiment ($CDCl_3$).[11,12] The NOE studies corroborate the linkage established in the 2D-COSY experiment and further define the substitution pattern along the entire periphery of the nucleus, confirming the two-dimensional structure of **2** previously set forth.[6] The data also allow complete determination of the stereochemistry of the left-hand portion of the molecule. The trans,trans-arrangement of the substituents at C10, C11, and C12 was apparent from proton–proton coupling constants ($J_{10,11}$ and $J_{11,12} \leq 1$ Hz)[13] and multiple confirming NOEs (Figure

† Dedicated to Professor E. J. Corey on the occasion of his 60th birthday.

(1) Isolation of neocarzinostatin: Ishida, N.; Miyazaki, K.; Kumagai, K.; Rikimaru, M. *J. Antibiot.* **1965**, *18*, 68.

(2) Isolation of **1**: (a) Napier, M. A.; Holmquist, B.; Strydom, D. J.; Goldberg, I. H. *Biochem. Biophys. Res. Commun.* **1979**, *89*, 635. (b) Koide, Y.; Ishii, F.; Hasuda, K.; Koyama, Y.; Edo, K.; Katamine, S.; Kitame, F.; Ishida, N. *J. Antibiot.* **1980**, *33*, 342.

(3) (a) In determination of structure **1**, the C10 and C11 substituents were shown to be trans. The stereochemistry at C4, C5, C10, C11, and C13 was not assigned: Edo, K.; Mizugaki, M.; Koide, Y.; Seto, H.; Furihata, K.; Otake, N.; Ishida, N. *Tetrahedron Lett.* **1985**, *26*, 331. (b) The *N*-methylfucosamine appendage has been shown unambiguously to be a D-sugar: Edo, K.; Akiyama, Y.; Saito, K.; Mizugaki, M.; Koide, Y.; Ishida, N. *J. Antibiot.* **1986**, *39*, 1615. (c) The stereochemistry at C10 and C11 was recently suggested to be *R,R* based on a model of the binding of **1** to DNA: Schreiber, S. L.; Kiessling, L. L. *J. Am. Chem. Soc.* **1988**, *110*, 631.

(4) (a) Beerman, T. A.; Goldberg, I. H. *Biochem. Biophys. Res. Commun.* **1974**, *59*, 1254. (b) Beerman, T. A.; Poon, R.; Goldberg, I. H. *Biochim. Biophys. Acta* **1977**, *475*, 294. (c) Kappen, L. S.; Goldberg, I. H. *Nucleic Acids Res.* **1978**, *5*, 2959.

(5) Hensens, O. D.; Dewey, R. S.; Liesch, J. M.; Napier, M. A.; Reamer, R. A.; Smith, J. L.; Albers-Schönberg, G.; Goldberg, I. H. *Biochem. Biophys. Res. Commun.* **1983**, *113*, 538.

(6) Myers, A. G. *Tetrahedron Lett.* **1987**, *28*, 4493.

(7) Neocarzinostatin powder (0.500 g) was suspended in ice-cold 0.5 M methanolic acetic acid solution (20 mL), and the mixture was gently agitated for 2 h. The suspension was centrifuged and filtered, and the clear filtrate was stored on ice in the dark. The extraction was repeated by resuspending the protein pellet in fresh cold solvent. The filtrates were combined, and the content of **1** was determined by UV absorbance at 340 nm.[2a] We are indebted to Dr. Matthew Suffness, the National Cancer Institute, and Kayaku Co., Ltd. for generous supplies of neocarzinostatin powder.

(8) We obtained 1.5–2.5 mg (5–10%) each of pure **2** and **3**. These yields are for scrupulously purified samples and, as such, represent lower limits on the actual values.

(9) For clarity, we have numbered **2** and **3** to correspond with **1**.

(10) Fesik, S. W.; Olejniczak, E. T. *Magn. Reson. Chem.* **1987**, *25*, 1046.

(11) (a) Bothner-By, A. A.; Stephens, R. L.; Lee, J.; Warren, C. D.; Jeanloz, R. W. *J. Am. Chem. Soc.* **1984**, *106*, 811. (b) Rance, M. *J. Magn. Reson.* **1987**, *74*, 557.

(12) We would like to express our sincere gratitude to Dr. Mark Rance for his assistance in obtaining 2-D CAMELSPIN spectra.

(13) Jackman, L. M.; Sternhell, S. *Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry*; Pergamon Press: New York, 1969; pp 286–288, and references therein.

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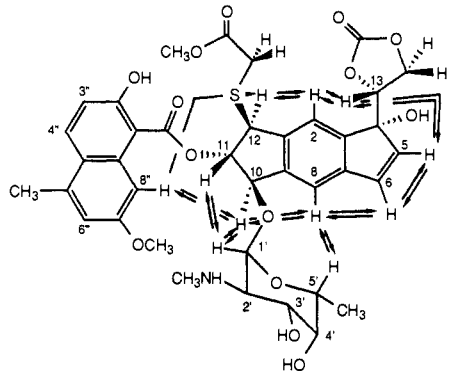
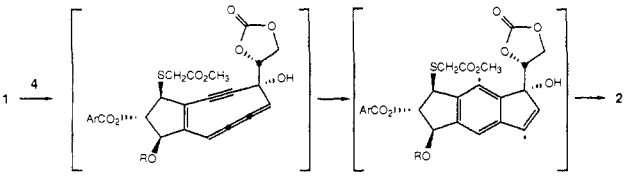


hν	NOE	%
2	12	7
2	13	4
5	6	19
5	13	10
6	5	12
6	8	8
8	6	14
8	10	6
8	5'	5
10	8	7
10	1'	14
10	8"	3
11	1'	14
12	2	9
13	2	4
13	5	5
1'	10	18
1'	11	10
5'	8	10
8"	10	2
8"	12	3

Figure 1. Summary of DNOE experiments in DMSO-d_6:D_2O (2:1, 23 °C).

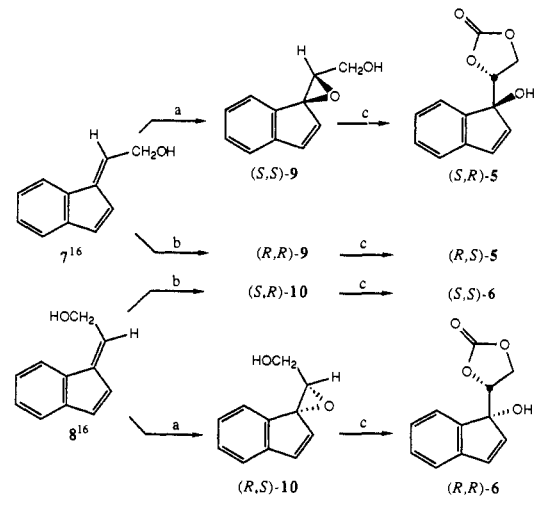
Scheme I. Proposed Mechanism for the Formation of **2**.



1). The absolute stereochemistry at these centers was revealed by three reciprocal NOEs between proton pairs H5–H8, H1′–H10, and H1′–H11, allowing correlation of the stereochemistry at C10 and C11 with the D-sugar appendage.[3b] Analysis of coupling constants ($J_{1',2'}$ and $J_{3',4'}$ = 3.2 Hz, $J_{2',3'}$ = 10.5 Hz, $J_{4',5'}$ < 1 Hz) shows that the fucosamine ring occupies a chair conformation with an axially oriented C1′ alkoxy substituent, as anticipated from the anomeric effect.[14] Imposing the exo-anomeric effect,[14] that is, a gauche orientation of O–C1′–O–C10 with dihedral angle +60°, then reveals simultaneous proximity of the three interacting proton pairs in the (10*R*, 11*S*, 12*S*)-diastereomer but not the (10*S*, 11*R*, 12*R*)-diastereomer (proximal proton pairs: H1′–H8, H1′–H10, and H5′–H11).[15] The latter diastereomer is further excluded upon examination of molecular models; we find no reasonable conformer which places either of the H5′–H8 or H1′–H11 pairs in proximity.

Absolute and relative stereochemistry at C4 and C13 of **2** was determined as follows. Each of the four diastereomers of model compounds **5** and **6** was synthesized in nonracemic form as outlined in Scheme II and separately converted into its (*R*)- and (*S*)-α-methoxy-α-(trifluoromethyl)phenyl acetic acid ester (MTPA derivative, Mosher ester)[18] with excess MTPA chloride and 4-(dimethylamino)pyridine (0.6 M each) in methylene chloride at 23 °C for 1.5 h. Subjection of **2** to identical reaction conditions with (*R*)- and (*S*)-MTPA chloride produced, in each case, a triester derivative in which the C3′ equatorial alcohol, phenol, and C4 tertiary hydroxyl groups had been acylated (FABMS: 1416). The 400 MHz ^{1}H NMR spectrum of each Mosher ester derivative of **2**, **5**, and **6** was recorded in both $CDCl_3$ and C_6D_6 under conditions of high dilution ($2\cdot10^{-3}$ M).[21] Chemical shifts of the

Scheme II. Asymmetric Synthesis of Diastereomeric Model Compounds **5** and **6**[a]



[a] (a) stoichiometric (+)-diethyl tartrate, Ti(O*i*Pr)$_4$, *t*-BuOOH, CH_2Cl_2, −20 °C, 11 h;[17] (b) as in (a) with (−)-diethyl tartrate (ee **9** = 84%, ee **10** = 88%);[18,19] (c) CO_2, Cs_2CO_3, 3-Å molecular sieves, DMF, 23–40 °C.[20]

three carbonate protons and two cis-coupled olefinic resonances in each derivative of the four diastereomeric model compounds were then tabulated, providing 20 points for comparison with the corresponding derivatives of **2** (five proton resonances, two Mosher ester derivatives, two solvents). Quantitative assessment of the data was achieved by computing the χ_{20}^2 function[22] taking the reasonable value of 0.10 ppm for σ. We find χ_{20}^2 = 15.9, 69.0, 71.2, and 81.6 for the (*R*,*R*)-, (*S*,*S*)-, (*S*,*R*)- and (*R*,*S*)-diastereomers, respectively. The latter three diastereomers can therefore be rejected with >99.5% confidence (χ_{20}^2 (0.995) = 40.0), and the complete stereochemical assignment of **2** is then 4*R*, 10*R*, 11*S*, 12*S*, 13*R*. It follows that the corresponding (previously unassigned) stereocenters of **1** are 4*S*, 5*R*, 10*R*, 11*R*, 13*R*, and the stereochemistry of thiol attack on **1** is defined as anti to both the C11 naphthoate appendage and the epoxide oxygen.

Product **3** was shown to be a 2:1 adduct of **4** and **1** by HRFABMS (calcd for $[M + H]^+$, $C_{41}H_{46}NO_{16}S_2$ 872.2258; found: 872.2366). The FTIR spectrum (neat film) revealed a close correspondence with **2**: 1815 and 1792 cm^{-1} (carbonate), 1647 cm^{-1} (hydroxynaphthoate), and 1734 cm^{-1} (methyl ester). The 400 MHz ^{1}H NMR spectrum ($CDCl_3$) was nearly superimposable with that of **2** with the following distinguishing features: (1) resonances for a second methyl thioglycolate substituent were apparent; and (2) the doublet pair corresponding to H5 and H6 was absent, while a new, sharp singlet appeared at δ 5.90. Irradiation of this singlet (DMSO-d_6:D_2O 2:1, 23 °C) led to NOE of the carbonate methine and the more downfield $SCH_2CO_2CH_3$ protons, while irradiation of the latter led to NOE of the δ 5.90 singlet and H8. These experiments establish the point of attachment of the second methyl thioglycolate as C6; the assignment of structure **3** is otherwise identical with **2**.[23]

(14) Deslongchamps, P. *Stereoelectronic Effects in Organic Chemistry*; Pergamon Press: New York, 1983; Chapter 2.

(15) It is perhaps noteworthy that application of this same conformational analysis to **1** leads to a structure in which the amino nitrogen lies in close proximity to H11, in near alignment with the C11–H11 bond axis. This suggests an intriguing possibility for the extreme base-sensitivity of **1** ($t_{1/2}$ = 0.6 min at 0 °C, pH 8), i.e., an internal eliminative pathway for cumulene formation, rather than the substitutive pathway which operates in the formation of **2** and **3** [base-sensitivity of **1**: Povirk, L. F.; Goldberg, I. H. *Biochemistry* **1980**, *19*, 4773].

(16) Prepared from indene and 1-chloro-1,2-ethanediol diacetate [Nagasawa, J.-i.; Araki, Y.; Ishido, Y. *J. Org. Chem.* **1981**, *46*, 1734] after the following: Neuenschwander, M.; Vögeli, R.; Fahrni, H.-P.; Lehmann, H.; Ruder, J.-P. *Helv. Chim. Acta* **1977**, *60*, 1073.

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(21) Appropriate control experiments established that chemical shift values were essentially invariant over at least a tenfold range in concentration.

(22)

$$\chi_{20}^2 = \sum_{i=1}^{20} (\delta_{i\text{Adduct}} - \delta_{i\text{Model}})^2 / \sigma^2$$

(23) We presently consider two mechanisms as likely for the formation of **3**: (1) a hydrogen atom abstraction (by C-2)–radical recombination (at C-6) reaction of the biradical intermediate of Scheme I with **4** and (2) addition of thiol radical to the cumulene intermediate of Scheme I (at C-6), transannular ring closure, and hydrogen atom abstraction (by C-2). Experiments designed to distinghish between these mechanisms are in progress.

We find that incubation of **1** with 0.2 M $DSCH_2CO_2CH_3$ in both 9:1 CD_3OD:CD_3CO_2D and 9:1 CH_3OD:CH_3CO_2D, as described above, leads to >80% incorporation of deuterium at C-2 and C-6 in isolated **2**.[24] These observations provide strong support for the biradical intermediate of Scheme I. Its formation from the cumulene-enyne of Scheme I, as compared with the Bergman reaction[25] postulated to occur in the calichemicin[26] and esperamicin[27] antibiotics, represents a new molecular rearrangement and a distinct strategy for the spontaneous generation of carbon-centered free radicals at or below ambient temperature.

Acknowledgment. Generous financial assistance from the National Institutes of Health (CA-47148-01) and Merck & Co., Inc., a Dreyfus New Faculty Award (to A.G.M.), and a National Science Foundation predoctoral fellowship (to P.J.P.) are gratefully acknowledged. We thank David Wheeler, Scott Ross, Dr. James Yesinowski, and Dr. Eric Anslyn for their assistance in obtaining NMR spectra and Richard Barrans for assistance with statistical analyses. We are indebted to our colleagues Professor Peter Dervan, Professor Dennis Dougherty, Professor Robert Grubbs, and Professor John Roberts for many fruitful discussions.

Supplementary Material Available: A tabulation of complete and assigned ^1H NMR spectral data for **2** and **3** and the 2-D COSY spectrum of **2** and chemical shift comparisons for (*R*)- and (*S*)-Mosher ester derivatives of **2**, **5**, and **6** (3 pages). Ordering information is given on any current masthead page.

(24) Goldberg et al. report that incubation of **1** with $DSCH_2CO_2CH_3$ (concentration not specified) in 0.1 M CD_3CO_2D in CH_3OD leads to production of a monoadduct displaying signals at δ 7.83 (s) and δ 7.01 (d) "at reduced intensity" with "incomplete deuterium incorporation from deuteriothioglycolate".[5]

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NMR Determination of Association Constants for Calixarene Complexes. Evidence for the Formation of a 1:2 Complex with Calix[8]arene

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Calixarenes are cyclic oligomers made up of benzene units just as cyclodextrins are made up of glucose units. A variety of calixarenes may now be synthesized in good yields,[1] and they are now useful as a basic skeleton in the design of new functionalized host molecules.[2-8] Beginning in 1984 we synthesized and studied sulfonatocalixarenes, the first example for water-soluble calixarenes.[9-14] However, the question on molecular recognition in solution remained unresolved. We report here the NMR method,[15] which is more complicated but applicable to a variety of guest molecules. We have determined the association constants (K), ΔH, and ΔS for the complexation of trimethylanilinium chloride (**1**) and *p*-sulfonatocalix[*n*]arene (n = 4, 6, and 8: $\mathbf{2}_n$). During this study we unexpectedly found unequivocal evidence for the formation of the 1:2 complex with $\mathbf{2}_8$.

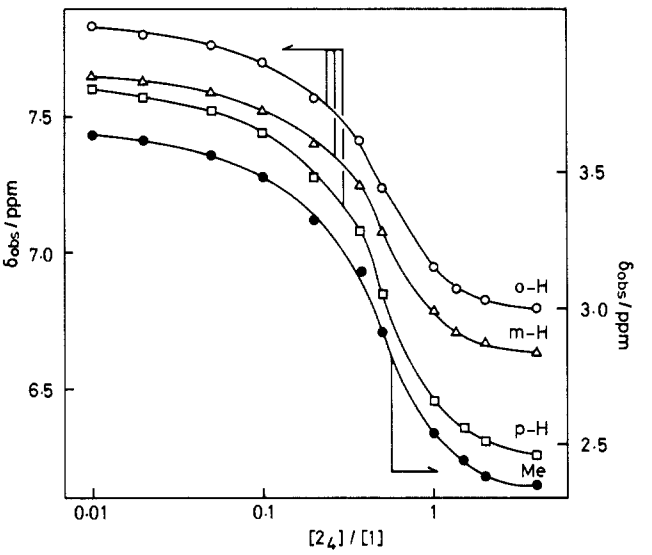
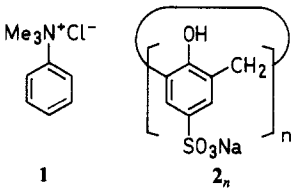


Figure 1. Plots of δ_{obsd} versus $[\mathbf{2}_4]/[\mathbf{1}]$. The concentration of $\mathbf{2}_4$ was maintained constant (1.00 mM) while that of **1** was varied (0.25-100 mM): D_2O, 25 °C, pD 7.3 with 0.1 M phosphate buffer, internal standard DSS.

Table I. Association Constants and Thermodynamic Parameters

parameter	$\mathbf{2}_4$	$\mathbf{2}_6$	$\mathbf{2}_8$ 1:1	$\mathbf{2}_8$ 1:2
$10^{-2}K$ (M^{-1}, at 25 °C)	56.0 ± 2.5	5.5 ± 0.4	52.0 ± 0.5	46.0 ± 0.5
ΔG (kcal mol^{-1}, at 25 °C)	−5.1 ± 0.5	−3.7 ± 0.2	−5.1 ± 0.2	−5.0 ± 0.1
ΔH (kcal mol^{-1})	−6.2 ± 0.3	−0.25 ± 0.10	0.0 ± 0.10	0.0 ± 0.10
ΔS (cal mol^{-1} deg^{-1})	−3.6 ± 0.8	11.7 ± 0.3	17.0 ± 0.3	16.7 ± 0.1



1 $\mathbf{2}_n$

Figure 1 shows the chemical shift of **1** in the presence of $\mathbf{2}_4$ in D_2O at 25 °C.[16] It is seen from Figure 1 that all peaks shift to

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(15) JEOL GX-400 NMR apparatus. Schürmann and Diederich used the similar NMR method for the determination of binding constants for cyclophanes, but the measurements were carried out only at the constant temperature: Schürmann, G.; Diederich, F. *Tetrahedron Lett.* **1986**, *27*, 4249.

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