^{1}H NMR, Table I; ^{13}C NMR, Table II.

Bastadin-8 (7): 65 mg; white film; $C_{34}H_{27}O_9N_4Br_5$; UV (MeOH) λ_{max} 208 (log ϵ 5.1), 280 nm (log ϵ 3.9); IR (film on NaCl plate) 3340, 1700, 1660, 1590, 1530, 1490, 1450, 1420, 1360, 1280, 1240, 990 cm^{-1}; low-resolution MS (EI$^+$, 12 eV) m/z (relative intensity) 504 (7.0), 502 (18.7), 500 (18.7), 498 (5.8), 342 (15.8), 340 (21.1), 199 (7.6), 199 (7.0); ^{1}H NMR (DMSO-d_6, 300 MHz), Table I; ^{13}C NMR (DMSO-d_6, 75.4 MHz), Table II.

Bastadin-8 Tetramethyl Ether (11). Bastadin-8 (7) (3.3 mg) was stirred at room temperature in dimethylformamide (1.5 mL), with potassium carbonate (100 mg) and methyl iodide (180 μL) for 18 h. The reaction mixture was diluted with dichloromethane and filtered. Evaporation of the solvent gave the crude methylated derivative, which was purified by HPLC on silica gel (acetone–hexane, 4:6) to give the product as a white powder (2.3 mg, 66% yield): ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.69 (H-8, d, 2.0), 7.52 (H-27, H-31, s), 7.28 (H-12, dd, 8.0, 2.0), 7.16 (H-36, d, 2.0), 7.14 (H-17, d, 2.0), 6.97 (H-11, d, 8.0), 6.87 (H-4 (NH), t, 6.0), 6.76 (H-22, t, 6.0), 6.65 (H-19, d, 2.0), 6.28 (H-38, d, 2.0), 4.88 (H-6, m), 3.79 (H-1, bs), 3.78 (H-25, bs), 3.72, 3.38 (H-5), 3.46, 3.55 (H-21), 2.75 (H-20, m), 4.02 (3 H, s), 4.01 (3 H, s), 3.97 (3 H, s), 3.70 (3 H, s); LRMS (FAB$^+$, p-nitrobenzyl alcohol/magic bullet (dithiothreitol/dithioerythritol) matrix m/z (relative intensity) 1076.9 (33.2), 1074.9 (52.5), 1072.8 (24.7), 1070.8 (19.9); HR FAB m/z (formula) 1110.8194 ($C_{38}H_{35}N_4O_9{}^{79}Br_4{}^{81}BrNa$ requires 1110.8198), 1112.8194 ($C_{38}H_{35}N_4O_9{}^{79}Br_3{}^{81}Br_2Na$ requires 1112.8178), 1114.8035 ($C_{38}H_{35}N_4O_9{}^{79}Br_2{}^{81}Br_3Na$ requires 1114.8157), 1116.7491 ($C_{38}H_{35}N_4O_9{}^{79}Br^{81}Br_4Na$ requires 1116.8137).

Bastadin-10 (8): 2.7 mg; colorless oil; $C_{34}H_{28}O_9N_4Br_4$; UV (MeOH) λ_{max} 210 (log ϵ 4.7), 277 nm (log ϵ 3.7); IR (film on NaCl plate) 3340 br, 1698, 1662, 1590, 1545, 1483, 1421, 1290, 1240 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz), Table I. ^{13}C NMR (DMSO-d_6, 75.4 MHz), Table II.

Bastadin-9 (9): 45 mg; white powder from methanol–water; $C_{34}H_{28}O_8N_4Br_4$; UV (MeOH) λ_{max} 208 (log ϵ 5.1), 280 nm (log ϵ 4.0); IR (film on NaCl plate) 3420–3200, 1710, 1660, 1620, 1585, 1450, 1420, 1360, 1230 cm^{-1}; low-resolution MS (FAB$^+$, magic bullet matrix) m/z (relative intensity) 942 (1), 940 (2), 938 (2), 936 (1, M$^+$); ^{1}H NMR (DMSO-d_6, 300 MHz), Table I; ^{13}C NMR (DMSO-d_6, 75.4 MHz), Table II.

Bastadin-9 Tetramethyl Ether (12). Methylation of bastadin-9 (9) (5.2 mg) was carried out using the same procedure described for the methylation of bastadin-8 (7). Purification by HPLC on silica gel gave 3.5 mg of 12 (64% yield): ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.53 (H-27, H-31, s), 7.44 (H-8, d, 2.0), 7.19 (H-36, d, 2.0), 7.03 (H-12, dd, 8.3, 2.0), 6.97 (H-16, d, 8.3), 6.91 (H-17, dd, 8.3, 2.0), 6.85 (H-11, d, 8.0), 6.79 (H-4 (NH), t, 6.0), 6.66 (H-19, d, 2.0), 6.61 (NH, t, 6.0), 6.27 (H-38, d, 2.0), 3.67 (H-1, bs), 3.86 (H-25, bs), 3.53 (2 H, q, 6.3), 3.46 (2 H, q), 2.75 (2 H, t), 2.73 (2 H, t), 4.05 (3 H, s), 4.00 (3 H, s), 3.90 [OCH_3, (C-15), s], 3.49 (s H, s); low-resolution MS (FAB$^+$) m/e (relative intensity) 1014 [7.1 (M + Na)$^+$], 1016 (21.1), 1018 (28.3), 1020 (26.3), 1022 (1.2); high-resolution MS (EI$^+$) m/e (relative intensity) 511.8354 [$C_{17}H_{11}O_2N_2{}^{79}Br_3$, calcd 511.8371, –3.3 ppm (34.4)], 513.8460 (95.8), 515.8416 (100), 517.8291 (37.5).

Bastadin-11 (10): 4.3 mg; whitish film; $C_{34}H_{26}O_8N_4Br_4$; UV (MeOH) λ_{max} 208 (log ϵ 4.3), 285 (log ϵ 3.4), 331 nm (log ϵ 3.5); IR (film on NaCl plate), 3300 br, 1718, 1662, 1646, 1544, 1495, 1479, 1447, 1418, 1284, 1243 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz), Table I; ^{13}C NMR (DMSO-d_6, 75.4 MHz), Table II.

Bastadin-11 Tetramethyl Ether (13). To bastadin-11 (10) (2.0 mg) in dimethylformamide was added excess ethereal diazomethane, and the mixture was allowed to stand for 3 h. The solvent was evaporated, and the residue was purified by HPLC on silica gel to give the tetramethyl derivative (<1 mg): ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.30 (H-4 (NH), d, 11.4), 7.54 (H-8, d, 2.7), 7.46 (H-27, H-31, s), 7.38 (H-12, dd), 7.34 (H-5, dd), 7.12 (1 H, d, 2.0), 6.96 (H-16, d, 8.4), 6.90 (H-17, dd), 6.90 (H-11, d, 8.4), 6.71 (H-19, d, 2.1), 6.58 (NH, t, 5.4), 6.25 (H-38, d, 2.0), 6.17 (H-6, d, 14.7), 3.73 (2 H, bs), 3.65 (2 H, bs), 3.58 (2 H, q, 5.4), 2.82 (2 H, t, 5.4), 4.01 (6 H, s), 3.93 (3 H, s), 3.92 (3 H, s).

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Registry No. 3, 79067-76-8; 4, 75513-47-2; 5, 79067-75-7; 6, 79067-74-6; 7, 127709-45-9; 8, 127687-08-5; 9, 127687-07-4; 10, 127687-09-6.

Acid-Catalyzed Rearrangement of Arenerol

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Upon treatment with p-toluenesulfonic acid in benzene, the sesquiterpene quinol arenerol (1) underwent skeletal rearrangement and intramolecular ether formation. The structure of the rearranged and cyclized product 4 was determined by X-ray analysis.

Some time ago we reported the structure of the sesquiterpene quinol arenerol (1), which had been isolated from the Pacific sponge *Dysidea areneria*.[1] In the course of trying to establish the structure of 1 (Chart I) by spectral and chemical methods, prior to obtaining crystals of 1 diacetate for X-ray analysis, we had treated 1 with p-toluenesulfonic acid in benzene to see if it would readily form a cyclic ether as would have been expected if the exocyclic methylene group were at position 8,13; cf. cyclization of zonarol.[2] Treatment of 1 in this acid solution overnight at room temperature followed by 1/2 h at reflux temperature resulted in the formation of a new product isomeric with 1 (MS analysis) in approximately 60% isolated yield. This rearranged product possessed one secondary and three quaternary methyl groups, but no exocyclic double bond. It formed only a monoacetate (acetic anhydride/pyridine) and hence was considered to contain a new ether ring. The proton NMR spectral data did not conform to that reported for some possible rearrangement

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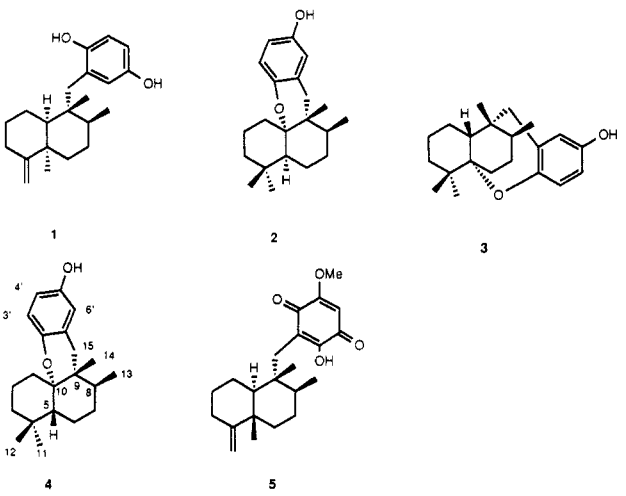
0022-3263/90/1955-4709$02.50/0

Chart I



products such as aureol (**2**),[3] chromoazonarol,[4] or 8-epi-chromazonarol.[3] Based on proton NMR spectral data, we speculated earlier[1] that the cyclization product of **1** might be **3**. Eventually we were able to prepare crystals suitable for X-ray analysis of the rearrangement product. The particular form of the crystal presented some problems for the X-ray analysis, but the structure was confirmed to be that shown by formula **4**. Ether **4** with its trans decalin ring fusion is stereoisomeric with aureol (**2**), which has the cis ring fusion. Compound **4** apparently does not form via a concerted migration of methyl and hydrogen groups since that would lead to a cis relationship between H-5 and C-15, whereas these groups are trans in the product **4**. Instead **4** presumably arises via protonation at C-11, methyl migration and loss of the C-10 proton to give a $\Delta^{5,10}$ olefin. This then undergoes a trans addition of the phenolic group to give **4**. The putative olefinic intermediate would have the same structure as the olefin obtained by treating aureol (**2**) with boron trifluoride etherate in acetic acid.[3] Heating ilimaquinone (**5**)[5] under acid conditions yields an analogous $\Delta^{5,10}$ olefin.[6]

$$1 \xrightarrow{p\text{-TsOH, } C_6H_6, \Delta} 4$$

Although the absolute configuration of arenerol (**1**) has not been determined, it seems most likely that it is the same as that of averol, aureol (**2**), and ilimaquinone (**5**). Although ilimaquinone had initially been reported[5] to be enantiomeric to averol[7] and aureol, Capon and MacLeod[6] have recently demonstrated that these compounds all have the same absolute configuration at the two chiral centers that they have in common, i.e., all are 8*S*,9*R*. Capon and MacLeod's work suggests therefore that arenerol (**1**) and the rearrangement product **4** both have 5*R*,8*S*,9*R*,10*S* configurations.[8]

There are four crystallographically independent molecules of compound **4**, A, B, C, and D in the asymmetric unit. Figure 1[9] is a perspective drawing for molecule A

Table I. Bond Distances with Esd's in Parentheses

	A	B	C	D
C(1)–C(2)	1.528 (5)	1.528 (6)	1.512 (5)	1.532 (6)
C(1)–C(10)	1.540 (5)	1.537 (5)	1.530 (5)	1.540 (5)
C(2)–C(3)	1.508 (6)	1.519 (5)	1.524 (5)	1.510 (6)
C(3)–C(4)	1.536 (6)	1.514 (6)	1.526 (6)	1.541 (6)
C(4)–C(5)	1.555 (5)	1.551 (5)	1.560 (5)	1.560 (5)
C(4)–C(11)	1.529 (6)	1.545 (6)	1.534 (6)	1.540 (6)
C(4)–C(12)	1.536 (6)	1.542 (5)	1.544 (5)	1.525 (6)
C(5)–C(6)	1.532 (5)	1.520 (5)	1.530 (5)	1.539 (5)
C(5)–C(10)	1.558 (5)	1.548 (4)	1.538 (4)	1.542 (5)
C(6)–C(7)	1.518 (5)	1.521 (5)	1.527 (5)	1.520 (5)
C(7)–C(8)	1.546 (5)	1.525 (5)	1.532 (5)	1.535 (5)
C(8)–C(9)	1.561 (5)	1.563 (5)	1.552 (5)	1.564 (5)
C(8)–C(13)	1.524 (6)	1.534 (5)	1.527 (5)	1.525 (5)
C(9)–C(10)	1.556 (5)	1.570 (5)	1.572 (5)	1.564 (5)
C(9)–C(14)	1.530 (5)	1.534 (5)	1.531 (5)	1.528 (5)
C(9)–C(15)	1.554 (4)	1.549 (5)	1.542 (4)	1.542 (4)
C(10)–O(22)	1.452 (4)	1.453 (4)	1.460 (4)	1.454 (4)
C(15)–C(16)	1.506 (5)	1.510 (5)	1.509 (5)	1.509 (5)
C(16)–C(17)	1.388 (5)	1.386 (5)	1.388 (5)	1.391 (5)
C(16)–C(21)	1.402 (4)	1.394 (4)	1.400 (4)	1.397 (4)
C(17)–C(18)	1.383 (5)	1.386 (5)	1.395 (5)	1.389 (5)
C(17)–O(22)	1.384 (4)	1.383 (4)	1.372 (4)	1.378 (4)
C(18)–C(19)	1.398 (4)	1.401 (5)	1.391 (5)	1.388 (5)
C(19)–C(20)	1.391 (5)	1.384 (5)	1.374 (5)	1.388 (5)
C(20)–C(21)	1.383 (5)	1.386 (5)	1.393 (5)	1.381 (5)
C(20)–O(23)	1.386 (4)	1.386 (4)	1.399 (4)	1.385 (4)
	E	F		
O–C	1.471 (9)	1.485 (9)		
O′–C′	1.45 (2)	1.52 (2)		

Table II. Selected Torsional Angles with Esd's in Parentheses

	A	B	C	D
C(10)–C(9)–C(15)–C(16)	−42.2 (4)	−49.0 (4)	−45.8 (4)	−46.3 (4)
C(9)–C(15)–C(16)–C(17)	12.7 (5)	22.9 (4)	16.5 (5)	19.1 (4)
C(15)–C(16)–C(17)–O(22)	−0.3 (6)	−5.3 (5)	−1.6 (5)	−3.7 (5)
C(16)–C(17)–O(22)–C(10)	21.9 (4)	17.3 (4)	20.7 (4)	19.7 (4)
C(17)–O(22)–C(10)–C(9)	−53.2 (3)	−44.5 (3)	−51.3 (3)	−48.2 (3)
O(22)–C(10)–C(9)–C(15)	62.0 (3)	59.6 (3)	62.8 (3)	60.3 (3)
C(10)–C(1)–C(2)–C(3)	56.1 (5)	54.9 (5)	56.8 (4)	57.3 (5)
C(1)–C(2)–C(3)–C(4)	−60.5 (5)	−58.2 (5)	−59.5 (4)	−58.6 (5)
C(2)–C(3)–C(4)–C(5)	56.0 (4)	53.8 (4)	52.9 (4)	51.8 (5)
C(3)–C(4)–C(5)–C(10)	−48.7 (4)	−48.3 (4)	−46.3 (4)	−46.4 (5)
C(4)–C(5)–C(10)–C(1)	45.4 (4)	47.0 (4)	45.1 (4)	47.6 (4)
C(5)–C(10)–C(1)–C(2)	−48.3 (4)	−49.5 (4)	−49.7 (4)	−51.7 (4)
C(5)–C(6)–C(7)–C(8)	−56.1 (4)	−54.6 (4)	−54.1 (4)	−54.8 (4)
C(6)–C(7)–C(8)–C(9)	54.9 (4)	53.8 (4)	52.9 (4)	54.9 (4)
C(7)–C(8)–C(9)–C(10)	−54.2 (3)	−53.7 (3)	−53.4 (3)	−55.0 (3)
C(8)–C(9)–C(10)–C(5)	57.4 (3)	55.9 (4)	57.4 (3)	56.9 (4)
C(9)–C(10)–C(5)–C(6)	−58.6 (4)	−57.1 (4)	−58.6 (4)	−56.5 (4)
C(10)–C(5)–C(6)–C(7)	56.5 (3)	55.5 (3)	56.1 (3)	54.7 (4)

Table III. Hydrogen Bonds in the Crystal Structure of 4

X–H···Y	distance (Å) X–Y	angle (deg) X–H–Y
O(23)B–H(23)B···O(23)A	2.707 (4)	167 (3)
O(23)A–H(23)A···OE	2.625 (5)	160 (4)
OE···O(23)B(−*x*, 0.5 + *y*, 2 − *z*)	2.719 (5)	
O(23)D–H(23)D···O(23)C	2.704 (4)	170 (4)
O(23)C–H(23)C···OF	2.573 (6)	174 (1)
OF···O(23)D(1 − *x*, 0.5 + *y*, 2 − *z*)	2.686 (6)	

with atomic numbering system. The four molecules of **4** in the asymmetric unit have very similar bond distances (Table I) and bond angles. The largest differences in bond lengths and bond angles are 0.027 Å for C(3)–C(4) and 1.9° for C(2)–C(3)–C(4), respectively. The distance between $C(10)_{sp^3}$ and O(22) is larger than that between $C(17)_{sp^2}$ and O(22) as expected. Similarly, bond $C(9)_{sp^3}$–C(15) is longer than bond $C(16)_{sp^2}$–C(15) (see Table I). The four mole-

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(8) Professor Capon kindly attempted to make the appropriate chemical correlations with a small sample of the diacetate of **1**, which we were able to supply, but sample degradation during saponification precluded completion of the correlation.
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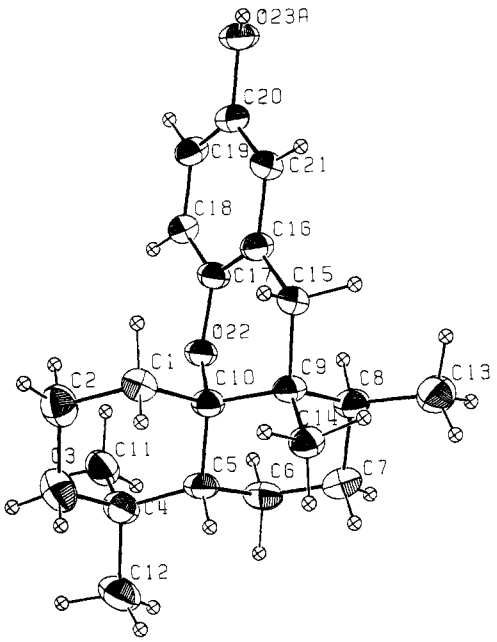

Figure 1. Perspective view for molecule A of 4 with numbering scheme.

cules have some conformational differences. The significant differences occur around the C(9)–C(15), C(15)–C(16), C(17)–O(22), and O(22)–C(10) bonds as shown in Table II. The stereochemistry is identical for molecules A, B, C, and D. The two cyclohexane rings are both in the chair conformation. Both the benzyl carbon C(15) and the methyl group bonded to C(8) are attached equatorially. The ring C(1) through C(10) is flattened around C(5) and C(10) as indicated by the conformational angles listed in Table II. This flattening is due to the steric interactions between C(1) and C(14) (distance = 3.09 Å, 3.06 Å, 3.09 Å, and 3.07 Å in A, B, C, and D, respectively) and that between O(22) and C(11) (3.01 Å, 2.99 Å, 3.05 Å, and 3.02 Å). The two cyclohexane rings in 4 are trans-fused. The packing of molecules in the crystal of 4 is dictated by hydrogen bonding, with six hydrogen bonds involved (Table III). It is interesting that the molecules form molecular pairs, and the pairs form chains in the solid state. The pairs are oriented along the crystallographic $a\cdots c$ diagonal and the chains are along the b axis.

Experimental Section

Cyclization of Arenerol (1) to 4.[10] Five milligrams of 1 in 15 mL of dry benzene containing ~50 mg of *p*-toluenesulfonic acid was stirred at room temperature overnight and finally the reaction mixture was heated under reflux for $^1/_2$ h. Toluenesulfonic acid was neutralized with a calculated amount of aqueous $NaHCO_3$. The neutral aqueous layer was extracted with chloroform (25 mL) four times. The combined organic layers were washed with water and dried (Na_2SO_4). The residue obtained after evaporation of the solvents was chromatographed over silica gel using hexane–chloroform as eluent to give 3 mg of 4: mp 115–116 °C; IR ($CHCl_3$) ν_{max} 3200 cm^{-1} (OH); UV (MeOH) λ_{max} (ϵ) 338 (886), 268 (1473), 260 (1578), 242 (5614) nm; ^1H NMR ($CDCl_3$, 270 MHz) 0.77 (3 H, d, 7.5, H-13), 0.92 (6 H, br s), 1.13 (3 H, s), 2.54 (2 H, s, H-15), 4.30 (1 H, br s, OH), 6.48 (1 H, d, 3, H-6′), 6.60 (1 H, dd, 9, 3, H-4), 6.69 (d, 9, H-3); LRMS (70 eV), m/z 314 (6 M^+), 281 (15), 207 (55), 191 (46), 95 (10), 83 (100).

Crystal Data. Compound 4 cocrystallized with methanol in a 2:1 ratio. Crystal system monoclinic; space group $P2_1$; cell dimensions a = 18.074 (4), b = 13.037 (1), and c = 16.255 (4) Å, α = 90.00 (0), β = 96.40 (3), and γ = 90.00 (0)°, and V = 3805.6 Å^3; Z = 8 for $C_{21}H_{30}O_2 \cdot {}^1/_2CH_3OH$; formula weight 330.49; calculated density 1.15 g/cm^3.

X-ray Crystal Structure Determination. Crystals of 4 were obtained from a chloroform solution equilibrated with methanol. The volume of the data crystal was 0.05 × 0.30 × 0.73 mm^3. The X-ray diffraction data were collected on a CAD4 diffractometer with Ni-filtered Cu Kα radiation (Cu Kα, λ = 1.54178 Å, μ = 4.99 cm^{-1}) at −110 (1) °C. Fifty-six reflections ($41° > \theta > 21°$) and Cu Kα_1 wavelength (1.54051 Å) were used for lattice constants. Systematic absences were $0k0$ ($k = 2n + 1$). All data with $1.0° \leq 2\theta \leq 150.0°$ in $-22 \leq h \leq 22$, $0 \leq k \leq 16$ and $0 \leq l \leq 20$ were recorded. The scan technique employed was ω–0.333θ obtained from the shape analysis of three reflections, which were evenly distributed in the reciprocal space. The theory of this technique was discussed by Werner[11] and Einstein.[12] The maximum scan time for a single reflection was 70 s. The scan angle was calculated as $(0.95 + 0.15 \tan \theta)°$. The receiving aperture, located 173 mm from the data crystal, had a constant width of 3.00 mm and a constant height of 6.00 mm. Three intensity control monitors were measured every 7200 s of X-ray exposure time and they showed a maximum difference of 0.031 and an esd of 0.010. Lorentz–polarization corrections were applied. No absorption correction was made. Among 8157 unique data, there were 6933 observed ones [$I \geq 2\sigma(I)$]. The crystal structure determination was difficult due to the fact that there were four molecules and two methanol molecules (96 non-hydrogen atoms) in the asymmetric unit and that the solvent molecules were disordered. The structure was solved by using the direct methods with the program SHELXS-86,[13] and all 92 non-hydrogen atoms of the four molecules were located. These atoms were refined anisotropically by the program SHELX76,[14] with 6933 observed data. The non-hydrogen atoms of the two disordered methanol molecules and 106 out of 128 hydrogen atoms were located from successive difference Fourier syntheses and were refined isotropically. Fourteen hydrogens were given calculated positions without any refinement. The eight hydrogen atoms for the two CH_3OH molecules were not found and not included. The weighting function used for the refinement was $w = 1/\sigma^2(F)$. A final R of 0.055 and R_w of 0.070 were obtained. The maximum shift/sd was 0.054 for non-hydrogen atoms and 0.091 for hydrogens. The largest and smallest peaks in the final difference map were +0.25 and −0.26 e/Å^3. The EOF = $[\sum w(F_o - F_c)^2/(N - NP)]^{1/2}$ = 2.1, where N was the number of reflections used (N = 6933), and NP was the number of parameters refined (NP = 1407). The absolute configuration of 4 was not determined.

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Registry No. 1, 87764-13-4; 4·Y_2CH_3OH, 127707-57-7.

Supplementary Material Available: Lists of bond angles, positional and (anisotropic and isotropic equivalent) thermal parameters for non-hydrogen atoms, and positional and isotropic thermal parameters for hydrogen atoms (12 pages). Ordering information is given on any current masthead page.

(10) Experimental conditions are as described in ref 1.

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