VOL. 57 NO. 4, APR. 2004 THE JOURNAL OF ANTIBIOTICS pp. 291 — 297

COMMUNICATIONS TO THE EDITOR

The First Total Synthesis of a Pyranonaphthoquinone Antitumor, BE-54238B

Sir:

A pyranonaphthoquinone, BE-54238B (**1**), was isolated by the Banyu group from the culture broth of *Streptomyces* sp. A54238 to show antitumor activities.[1)] The absolute structure of **1** was determined by NMR studies[1)] and X-ray analysis[2)] to be a nanaomycin analog fused with a pyrrolidine ring, and, therefore, to belong to a family of pyranonaphthoquinone antibiotics (Chart 1).

We have already reported the first total syntheses of related antibiotics such as nanaomycin D (**2**),[3,4)] kalafungin (**3**)[3,4)] and medermycin (**4**),[5,6)] and developed synthetic strategies for the stereoselective construction of densely-functionalized pyranonaphthoquinones from carbohydrates. Very recently, our total synthesis of medermycin was confirmed to be reasonable by the WILLIAMSON group,[7)] although the synthesis was once questioned.[8)]

We now wish to demonstrate both the utility and the versatility of our method in the first total synthesis of BE-54238B (**1**) to confirm its absolute structure.

Figure 1 illustrates our retrosynthetic analysis of **1**. On the basis of this methodology, the hexacyclic framework of **1** was retrosynthetically broken down into the tricyclic

Chart 1.

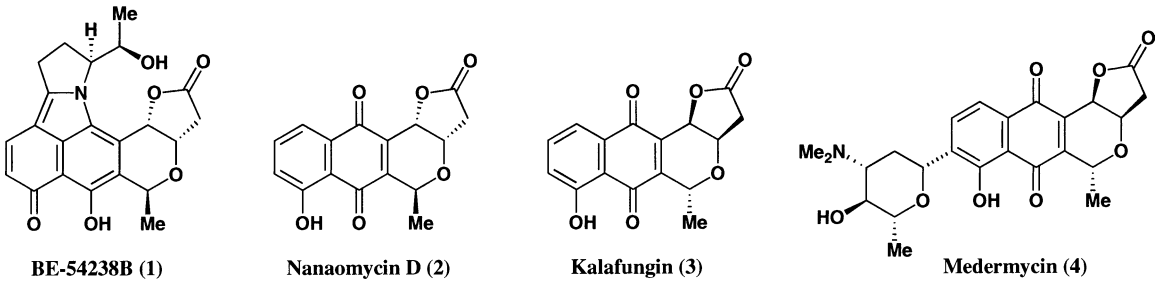
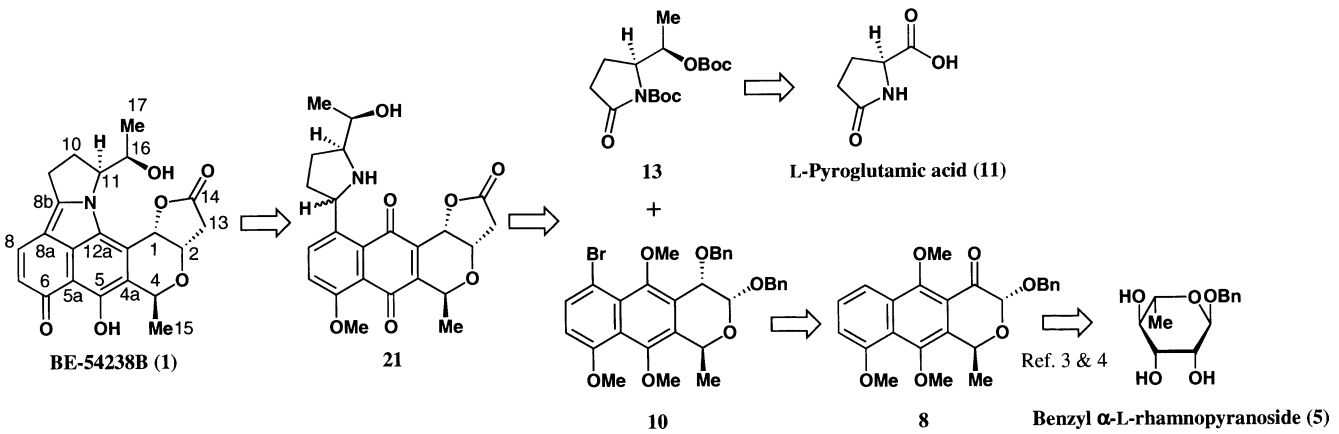
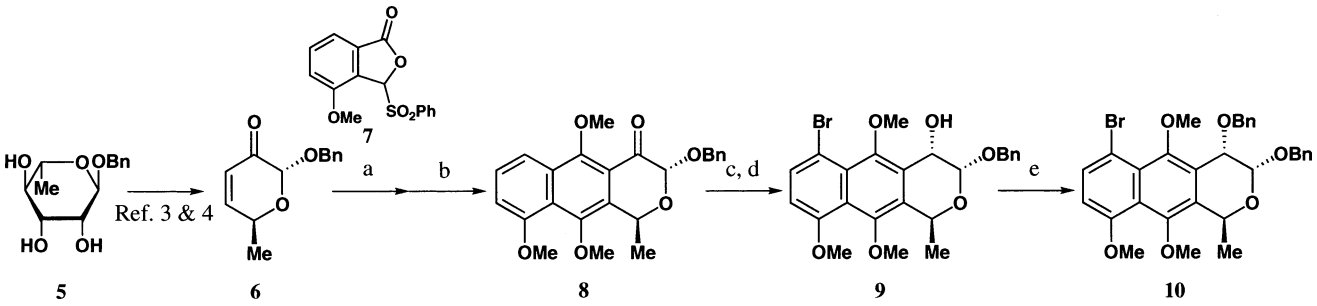


Fig. 1

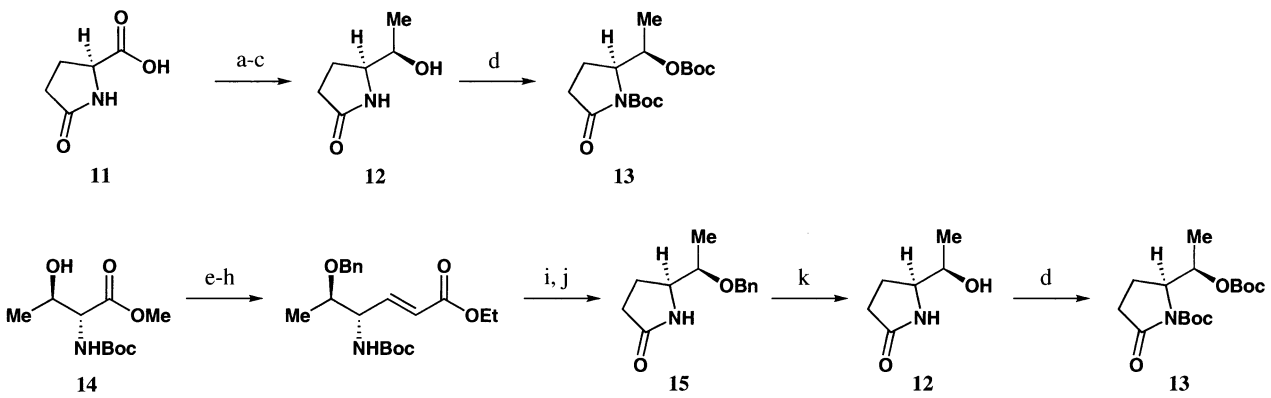


Scheme 1.



Conditions; (a) *t*-BuOLi/THF, –78°C to 40°C, 2 hours (b) Me_2SO_4, K_2CO_3/acetone, 40°C, 2 days; 83% in 2 steps (c) NBS/DMF, 0°C, 2 hours; 87% (d) $NaBH_4$/MeOH, 0°C, 1.5 hours; 93% (e) BnBr, NaH/DMF, rt, 1 hour; 89%.

Scheme 2.



Conditions; (a) MeNH(OMe)•HCl, $(PyS)_2$, Ph_3P/THF, rt, 12 hours (b) MeLi/THF, –78°C, 4 hours (c) L-Selectride/THF, –78°C, 1 hour; 42% in 3 steps (d) $(Boc)_2O$, NaH/THF, rt, 1 hour; 84% (e) $BnOC(=NH)CCl_3$, TfOH/CH_2Cl_2-cyclohexane, –30°C, 12 hours; 73% (f) $LiBH_4$/THF, 45°C, 4 hours; 75% (g) $(COCl)_2$, DMSO, Et_3N/CH_2Cl_2, –78°C to rt, 1 hour (h) $Ph_3P{=}CHCO_2Et$/PhMe, rt, 1 hour; 94% in 2 steps (i) H_2, Pd-C/EtOH, rt, 4 hours; quant. (j) NaH/THF, rt, 4 hours; 85% (k) H_2, $Pd(OH)_2$-C/EtOH, rt, 1 hour; quant.

segment **10** and the pyrrolidine segment **13** *via* the key intermediate **21**. We expected that the pyrrolidine-fused structure **1** would be constructed by cyclization of **21** at C12a† to give the intermediary iminium ion followed by proton tautomerization. An additional advantage of this plan was the expectation that **10** could be derived from our nanaomycin precursor **8**.[3,4] The chirality would originate in benzyl α-L-rhamnopyranoside (**5**),[3,4] while that of **13** would be traced to L-pyroglutamic acid (**11**).

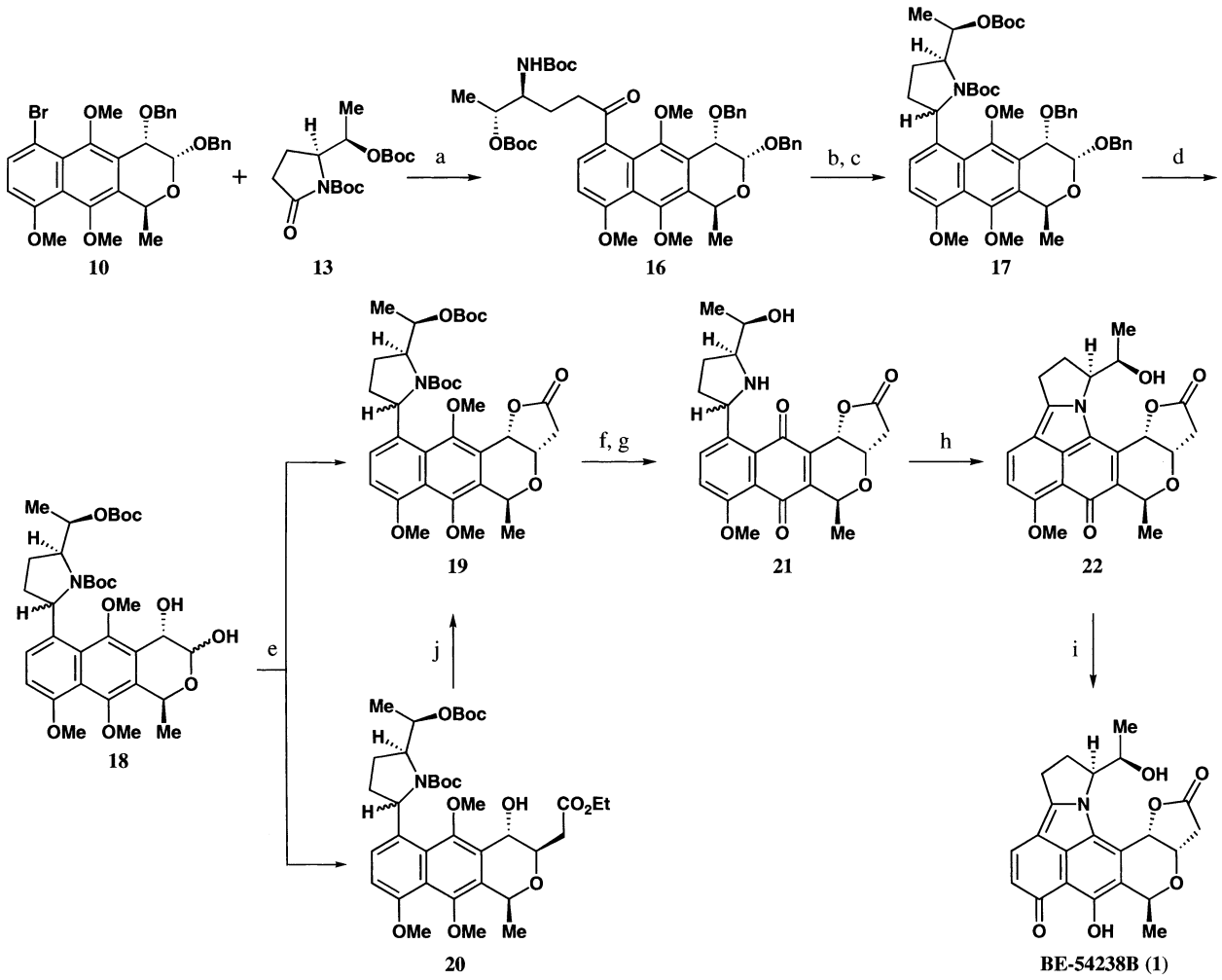
The *O*-benzyl precursor **8** was prepared according to our reported procedures[3,4] from benzyl 3,4,6-trideoxy-α-L-*glycero*-hex-3-enopyranosid-2-ulose (**6**) and 4-methoxy-3-(phenylsulfonyl)-1(3*H*)-isobenzofuranone (**7**), which were derived from L-rhamnoside **5** and *m*-methoxybenzoyl chloride, respectively (Scheme 1).

Bromination of **8** with NBS was successively followed by stereoselective hydride reduction[3,4] to give **9** and *O*-benzylation to provide the segment **10**.

The other segment **13** [$[\alpha]_D^{27}$ −69.6° (*c* 3.14, $CHCl_3$)] was synthesized from L-pyroglutamic acid (**11**) through **12** in four steps: 1) formation of the active ester, 2) reaction with MeLi,[9] 3) stereoselective reduction to give **12**

† The carbon-numbering protocol parallels that of the natural product **1**.[1]

Scheme 3.



Conditions; (a) *t*-BuLi/THF, –78°C, 6 hours; 83% (b) $LiBH_4$, $MgCl_2$/THF, 45°C, 4 hours; 84% (c) MsCl, Et_3N/CH_2Cl_2, –30°C, 1 hour; 80% (d) H_2, $Pd(OH)_2$-C/EtOH, rt, 40 minutes; quant. (e) $Ph_3P{=}CHCO_2Et$/PhMe, reflux, 2 days; **19**: 67%, **20**: 22% (f) TFA, rt, 10 minutes; quant. (g) CAN/aq. CH_3CN, 0°C, 10 minutes (h) MeOH, 60°C, 1 hour (i) BCl_3/CH_2Cl_2, rt, 4 hours; 60% in 3steps (j) $KHCO_3$, 18-crown-6/DMF, 90°C, 1 day; 80%.

$[[\alpha]_D^{28}$ +3.76° (*c* 1.01, $CHCl_3$)], 4) *N,O*-protection with Boc groups (Scheme 2). The enantiomeric purity of **13** was confirmed by identification with the authentic sample $[[\alpha]_D^{26}$ −72.4° (*c* 2.14, $CHCl_3$)], which was derived by an alternative route from *N*-*t*-butoxycarbonyl-D-*allo*-threonine methyl ester (**14**) through the intermediates **12** $[[\alpha]_D^{27}$ +4.23° (*c* 1.46, $CHCl_3$)] and **15** in 8 steps.

Coupling of **13** with the *t*-butyllithium generated anion of **10** smoothly proceeded to give the ketone **16**. This was reduced to an alcohol, which was cyclized through *O*-mesylation to give a 3 : 1 diastereomeric mixture of the pyrrolidine **17** (Scheme 3). The mixture was used for the following reactions, because the asymmetric center at C8b would disappear in the final stage. Hydrogenolysis of **17** gave the hemiacetal **18**, which was submitted to the Wittig reaction. The key reaction was carried out in refluxing toluene to give two products, **19** and **20**, in 67% and 22% yields as expected from our previous work.[3,4] The lactone **19** results from a two-step sequence including the intramolecular Michael cyclization of the intermediary Wittig α,β-unsaturated ester and concomitant lactonization of the resultant *cis* hydroxy ester. The lactone **19** was suitable for the synthesis of the natural product **1**, while the hydroxy ester **20** was recycled to **19** in high yield by heating with $KHCO_3$ and 18-crown-6 in DMF. Acidic removal of the *O*-Boc group in **19** was followed by

Table 1. Significant physico-chemical properties of compounds **1**~**22**.

Compds.	$[\alpha]_D$ Mp (°C)	^{1}H-NMR (600 MHz; δ ppm; *J* Hz) FAB-MS (*m/z*)
1	−521°(*c* 0.29, DMSO) >209 (decomp.)	^{1}H-NMR($CDCl_3$): δ 1.30(3H, d, *J*=6.5), 1.65(3H, d, *J*=6.5), 2.70(1H, dd, *J*=8.5&13.0), 2.80(1H, d, *J*=18.0), 2.83(1H, dddd, *J*=8.5, 8.5, 8.5&13.0), 3.08(1H, dd, *J*=5.5&18.0), 3.16(1H, dd, *J*=8.5&17.0), 3.34(1H, ddd, *J*=8.5, 8.5&17.0), 4.30(1H, br q, *J*=6.5), 4.91(1H, br d, *J*=8.5), 4.98(1H, dd, *J*=3.0&5.5), 5.44(1H, q, *J*=6.5), 5.66(1H, d, *J*=3.0), 6.57(1H, d, *J*=9.0), 7.79(1H, d, *J*=9.0), 12.3(1H, br s) FAB-MS: 396$(M+H)^+$
6	−116°(*c* 3.39, $CHCl_3$) Oil	^{1}H-NMR($CDCl_3$): δ 1.38(3H, d, *J*=7.0), 4.66(1H, ddq, *J*=1.5, 2.5&7.0), 4.72(1H, d, *J*=12.0), 4.82(1H, d, *J*=12.0), 4.92(1H, br s), 6.08(1H, ddd, *J*=0.5, 2.5&10.5), 6.90(1H, dd, *J*=1.5&10.5), 7.28-7.37(5H, m) FAB-MS: 219$(M+H)^+$
8	−170°(c 2.37, MeOH) Oil	^{1}H-NMR(CD_3OD): δ 1.61(3H, d, *J*=6.5), 3.69(3H, s), 3.98(3H, s), 3.99(3H, s), 4.75(1H, d, *J*=12.0), 4.84(1H, d, *J*=12.0), 5.07(1H, s), 5.31(1H, q, *J*=6.5), 7.16(1H, dd, *J*=0.5&7.5), 7.25-7.34(5H, m), 7.51(1H, dd, *J*=7.5&8.0), 7.89(1H, dd, *J*=0.5&8.0) FAB-MS: 408(M^+)
9	−114°(c 2.27, $CHCl_3$) 51-52	^{1}H-NMR($CDCl_3$): δ 1.59(3H, d, *J*=6.5), 3.17(1H, d, *J*=4.0), 3.77(3H, s), 3.91(3H, s), 3.98(3H, s), 4.78(1H, d, *J*=12.0), 5.00(1H, d, *J*=12.0), 5.05(1H, d, *J*=2.0), 5.08(1H, dd, *J*=2.0&4.0), 5.47(1H, q, *J*=6.5), 6.69(1H, d, *J*=8.5), 7.28-7.44(5H, m), 7.68(1H, d, *J*=8.5) FAB-MS: 488, 490(M^+)
10	−143°(*c* 2.72, $CHCl_3$) 50-51	^{1}H-NMR($CDCl_3$): δ 1.60(3H, d, *J*=6.5), 3.73(3H, s), 3.78(3H, s), 3.97(3H, s), 4.72(1H, d, *J*=12.0), 4.92(1H, d, *J*=11.0), 4.94(1H, d, *J*=2.0), 5.05(1H, d, *J*=2.0), 5.10(1H, d, *J*=12.0), 5.20(1H, d, *J*=11.0), 5.58(1H, q, *J*=6.5), 6.67(1H, d, *J*=8.0), 7.19-7.46(10H, m), 7.65(1H, d, *J*=8.0) FAB-MS: 578, 580(M^+)
12	+4.23°(*c* 1.46, $CHCl_3$) Oil	^{1}H-NMR($CDCl_3$): δ 1.15(3H, d, *J*=6.5), 2.02(1H, dddd, *J*=5.0, 6.5, 10.0&12.5), 2.08(1H, dddd, *J*=6.0, 8.0, 9.5&12.5), 2.30(1H, ddd, *J*=6.5, 9.5&16.5), 2.37(1H, ddd, *J*=6.0, 10.0&16.5), 3.52(1H, d, *J*=3.0), 3.66(1H, ddd, *J*=3.0, 5.0&8.0), 3.87(1H, ddq, *J*=3.0, 3.0&6.5), 7.10(1H, br s) FAB-MS: 130$(M+H)^+$
13	−72.4°(*c* 2.14, $CHCl_3$) 87-88	^{1}H-NMR($CDCl_3$): δ 1.29(3H, d, *J*=6.5), 1.45(9H, s), 1.54(9H, s), 2.02(1H, dddd, *J*=10.0, 10.0, 10.0&13.0), 2.11(1H, dddd, *J*=1.5, 1.5, 10.0&13.0), 2.39(1H, ddd, *J*=1.5, 10.0&17.5), 2.71(1H, ddd, *J*=10.0, 10.0&17.5), 4.12(1H, ddd, *J*=1.5, 1.5&10.0), 5.30(1H, dq, *J*=1.5&6.5) FAB-MS: 396$(M+H)^+$
14	−26.3°(*c* 1.39, $CHCl_3$) Oil	^{1}H-NMR[$(CD_3)_2CO$]: δ 1.21(3H, d, *J*=6.0), 1.40(9H, s), 3.69(3H, s), 4.04(1H, ddq, *J*=6.0, 6.0&6.0), 4.09(1H, d, *J*=6.0), 4.15(1H, dd, *J*=6.0&8.5), 6.07(1H, br s) FAB-MS: 219$(M+H)^+$
15	−7.80°(*c* 0.59, $CHCl_3$) Oil	^{1}H-NMR($CDCl_3$): δ 1.18(3H, d, *J*=6.0), 1.93(1H, dddd, *J*=4.0, 6.0, 10.5&13.0), 2.18(1H, dddd, *J*=6.5, 8.0, 10.5&13.0), 2.28(1H, ddd, *J*=6.0, 10.5&16.5), 2.36(1H, ddd, *J*=6.5, 10.5&16.5), 3.49(1H, dq, *J*=4.0&6.0), 3.75(1H, ddd, *J*=4.0, 4.0&8.0), 4.46(1H, d, *J*=11.5), 4.62(1H, d, *J*=11.5), 5.75(1H, br s), 7.27-7.37(5H, m) FAB-MS: 220$(M+H)^+$

Table 1. Continued.

Compds.	$[\alpha]_D$ Mp (°C)	^{1}H-NMR (600 MHz; δ ppm; *J* Hz) FAB-MS (*m/z*)
16	−83.1°(*c* 1.90, $CHCl_3$) 56-57	^{1}H-NMR($CDCl_3$): δ 1.29(3H, d, *J*=6.5), 1.36(9H, s), 1.48(9H, s), 1.66(3H, d, *J*=6.5), 1.71(1H, br s), 2.02(1H, br s), 2.61(1H, br s), 2.78(1H, br s), 3.50(3H, s), 3.76-3.82(1H, m), 3.82(3H, s), 4.01(3H, s), 4.55(1H, br d, *J*=9.5), 4.72(1H, d, *J*=12.0), 4.76(1H, dq, *J*=4.0&6.5), 4.88(1H, d, *J*=10.5), 4.89(1H, s), 5.09(1H, d, *J*=12.0), 5.12(1H, br d, *J*=10.5), 5.14(1H, s), 5.58(1H, q, *J*=6.5), 6.80(1H, d, *J*=8.0), 7.16(1H, d, *J*=8.0), 7.20-7.46(10H, m) FAB-MS: 830$(M+H)^+$
17	−143°(*c* 1.45, $CHCl_3$) Form	^{1}H-NMR($CDCl_3$): δ 1.25, 1.25(1:3, 3H in total, each d, *J*=6.5), 1.29, 1.54(3:1, 9H in total, each s), 1.36-1.52(2H, m), 1.56, 1.59(3:1, 9H in total, each s), 1.60, 1.62(1:3, 3H in total, each d, *J*=6.5), 1.75-1.83(1H, m), 2.35-2.47(1H, m), 3.71, 3.78(3:1, 3H in total, each s), 3.82(3H, s), 3.87, 3.97(1:3, 1H in total, each br d, *J*=9.0), 3.95, 3.96(1:3, 3H in total, each s), 4.72, 4.73(3:1, 1H in total, each d, *J*=12.0), 4.92(1H, d, *J*=10.5), 5.00, 5.02(3:1, 1H in total, each d, *J*=1.0), 5.06, 5.08(1:3, 1H in total, each d, *J*=1.0), 5.09, 5.09(3:1, 1H in total, each d, *J*=12.0), 5.20, 5.26(1:3, 1H in total, each d, *J*=10.5), 5.53, 5.74(1:3, 1H in total, each dq, *J*=1.5&6.5), 5.61, 5.62(1:3, 1H in total, each q, *J*=6.5), 5.83, 5.96(3:1, 1H in total, each d, *J*=8.0), 6.73, 6.77(3:1, 1H in total, each d, *J*=8.0), 6.95, 6.99(1:3, 1H in total, each d, *J*=8.0), 7.17-7.47(10H, m) FAB-MS: 814$(M+H)^+$
18	Form	^{1}H-NMR($CDCl_3$): δ 1.26(3H, d, *J*=6.5), 1.30, 1.49(3:1, 9H in total, each s), 1.44-1.54(2H, m), 1.54, 1.56(1:3, 9H in total, each s), 1.64, 1.65(1:3, 3H in total, each d, *J*=6.5), 1.75-1.87(1H, m), 2.24, 2.26(1:3, 1H in total, each d, *J*=10.0), 2.40-2.50(1H, m), 3.78, 3.83(1:3, 3H in total, each s), 3.90, 3.97(3:1, 3H in total, each s), 3.91, 4.01(1:3, 1H in total, each br d, *J*=9.0), 3.98, 3.99(3:1, 3H in total, each s), 3.99, 4.03(3:1, 1H in total, each d, *J*=11.0), 4.94, 4.95(1:3, 1H in total, each dd, *J*=1.5&10.0), 5.22, 5.25(1:3, 1H in total, each dd, *J*=1.5&11.0), 5.49, 5.51(1:3, 1H in total, each q, *J*=6.5), 5.53, 5.75(1:3, 1H in total, each dq, *J*=1.5&6.5), 5.85, 5.96(3:1, 1H in total, each d, *J*=8.5), 6.77, 6.82(3:1, 1H in total, each d, *J*=8.5), 7.00, 7.05(1:3, 1H in total, each d, *J*=8.5) FAB-MS: 634$(M+H)^+$
19	−217°(*c* 0.84, $CHCl_3$) Form	^{1}H-NMR($CDCl_3$): δ 1.26(3H, d, *J*=6.5), 1.33, 1.54(3:1, 9H in total, each s), 1.38-1.52(2H, m), 1.53, 1.54(1:3, 3H in total, each d, *J*=6.5), 1.56, 1.57(3:1, 9H in total, each s), 1.79, 1.82(3:1, 1H in total, each dd, *J*=8.5&13.0), 2.37-2.47(1H, m), 2.72, 2.72(1:3, 1H in total, each d, *J*=17.5), 2.95, 2.97(1:3, 1H in total, each dd, *J*=4.0&17.5), 3.81, 3.86(1:3, 3H in total, each s), 3.87, 3.94(3:1, 3H in total, each s), 3.89, 3.99(1:3, 1H in total, each br d, *J*=9.0), 3.98, 3.99(1:3, 3H in total, each s), 4.71, 4.74(1:3, 1H in total, each dd, *J*=2.5&4.0), 5.39, 5.40(1:3, 1H in total, each q, *J*=6.5), 5.55, 5.77(1:3, 1H in total, each dq, *J*=1.5&6.5), 5.60, 5.62(1:3, 1H in total, each d, *J*=2.5), 5.83, 5.94(3:1, 1H in total, each d, *J*=8.0), 6.80, 6.84(3:1, 1H in total, each d, *J*=8.0), 7.01, 7.06(1:3, 1H in total, each d, *J*=8.0) FAB-MS: 658$(M+H)^+$

Table 1. Continued.

Compds.	$[\alpha]_D$ Mp (°C)	^{1}H-NMR (600 MHz; δ ppm; *J* Hz) FAB-MS (*m/z*)
20	−72.4°(*c* 1.15, $CHCl_3$) Form	^{1}H-NMR($CDCl_3$): δ 0.92, 1.48(3:1, 9H in total, each s), 1.30, 1.31(1:3, 3H in total, each t, *J*=6.5), 1.30, 1.31(3:1, 3H in total, each d, *J*=6.5), 1.52, 1.54(1:3, 9H in total, each s), 1.60, 1.64(1:3, 3H in total, each d, *J*=6.5), 1.70, 1.91(1:3, 1H in total, each dd, *J*=8.0&12.0), 1.94-2.02, 2.05-2.18(1:3, 2H in total, each m), 2.61, 2.70(1:3, 1H in total, each dddd, *J*=8.5, 8.5, 12.0&12.0), 2.62, 2.63(1:3, 1H in total, each dd, *J*=9.0&16.0), 3.06, 3.07(1:3, 1H in total, each dd, *J*=3.0&16.0), 3.72, 3.74(3:1, 3H in total, each s), 3.84, 3.90(1:3, 1H in total, each ddd, *J*=3.0, 9.0&9.0), 3.88, 3.95(3:1, 3H in total, each s), 3.96, 3.98(3:1, 3H in total, each s), 3.99, 4.15(1:3, 1H in total, each ddd, *J*=1.5, 8.5&8.5), 4.22, 4.22(1:3, 2H in total, each q, *J*=6.5), 4.48(1H, br s), 4.85, 4.86(3:1, 1H in total, each br d, *J*=9.0), 5.18, 5.19(3:1, 1H in total, each q, *J*=6.5), 5.44, 5.58(1:3, 1H in total, each dq, *J*=1.5&6.5), 6.15, 6.30(3:1, 1H in total, each d, *J*=8.5), 6.77(1H, d, *J*=8.0), 6.99, 7.11(1:3, 1H in total, each d, *J*=8.0) FAB-MS: 704$(M+H)^+$
22	Form	^{1}H-NMR($CDCl_3$): δ 1.35(3H, d, *J*=6.5), 1.62(3H, d, *J*=6.5), 2.75-2.92(2H, m), 2.77(1H, d, *J*=17.5), 3.05(1H, dd, *J*=5.5&17.5), 3.22(1H, dd, *J*=8.5&17.0), 3.43(1H, ddd, *J*=8.5, 8.5&17.0), 4.15(3H, s), 4.43(1H, br q, *J*=6.5), 4.90(1H, dd, *J*=3.5&5.5), 5.05(1H, dd, *J*=1.0&9.0), 5.25(1H, q, *J*=6.5), 5.43(1H, d, *J*=3.5), 7.04(1H, d, *J*=8.5), 8.04(1H, d, *J*=8.5) FAB-MS: 410$(M+H)^+$

oxidative de-*O*-methylation to give the quinone **21**. This was effectively cyclized to **22** as expected above. However, both intermediates **21** and **22** were very unstable on purification.

Thus, without purification, **22** was de-*O*-methylated by BCl_3 to give the tautomerized compound **1** as a hydrochloride salt, which was identical in all respects with the salt of the natural BE-54238B (**1**),[1,2] completing the first total synthesis to establish the absolute structure.

Acknowledgment

The present work was financially supported by Grant-in-Aid for Specially Promoted Research and Scientific Research A from MEXT. We are grateful to 21COE "Practical Nano-Chemistry" from MEXT, Japan and Advanced Research Institute for Science and Engineering, Waseda University for the generous supports of our program. We also thank Drs. H. SUDA and M. TSUKAMOTO, Banyu Pharmaceutical Co., Ltd. for helpful discussions.

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(Received January 21, 2004)

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