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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.

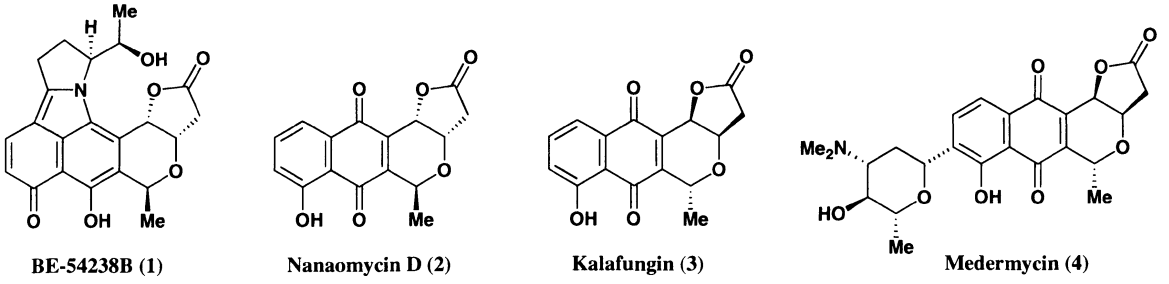
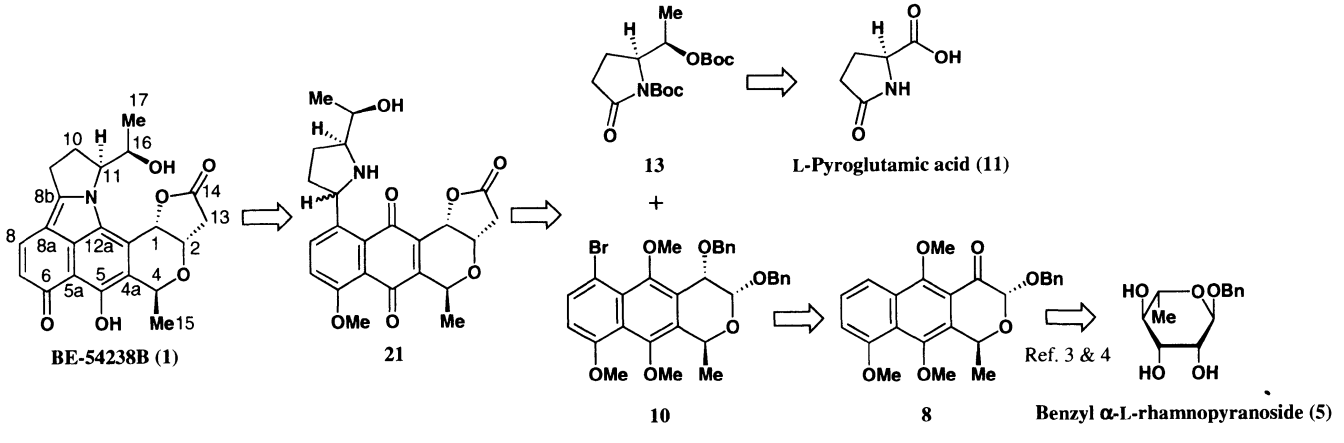
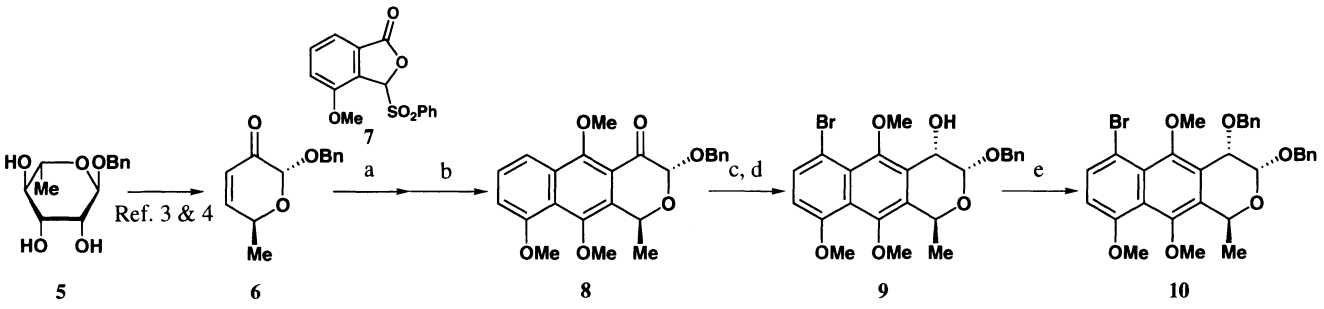


Fig. 1

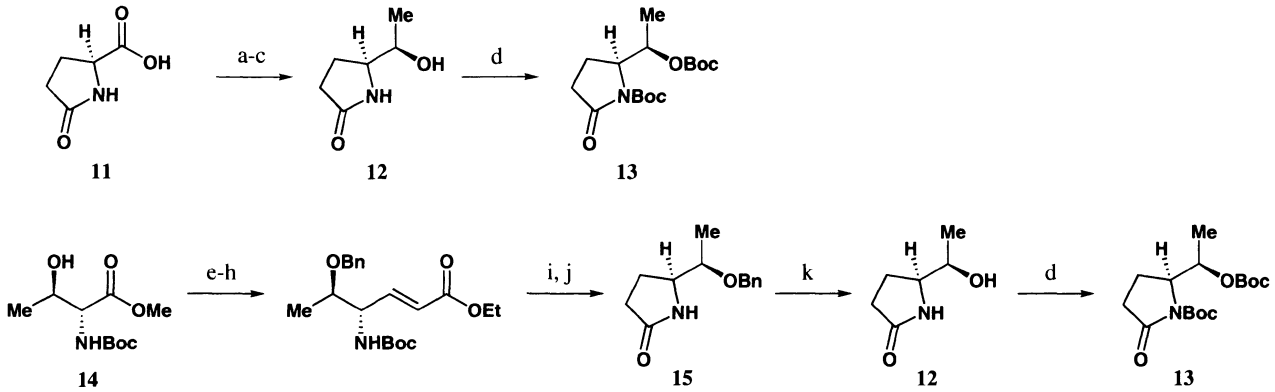


Scheme 1.



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**).

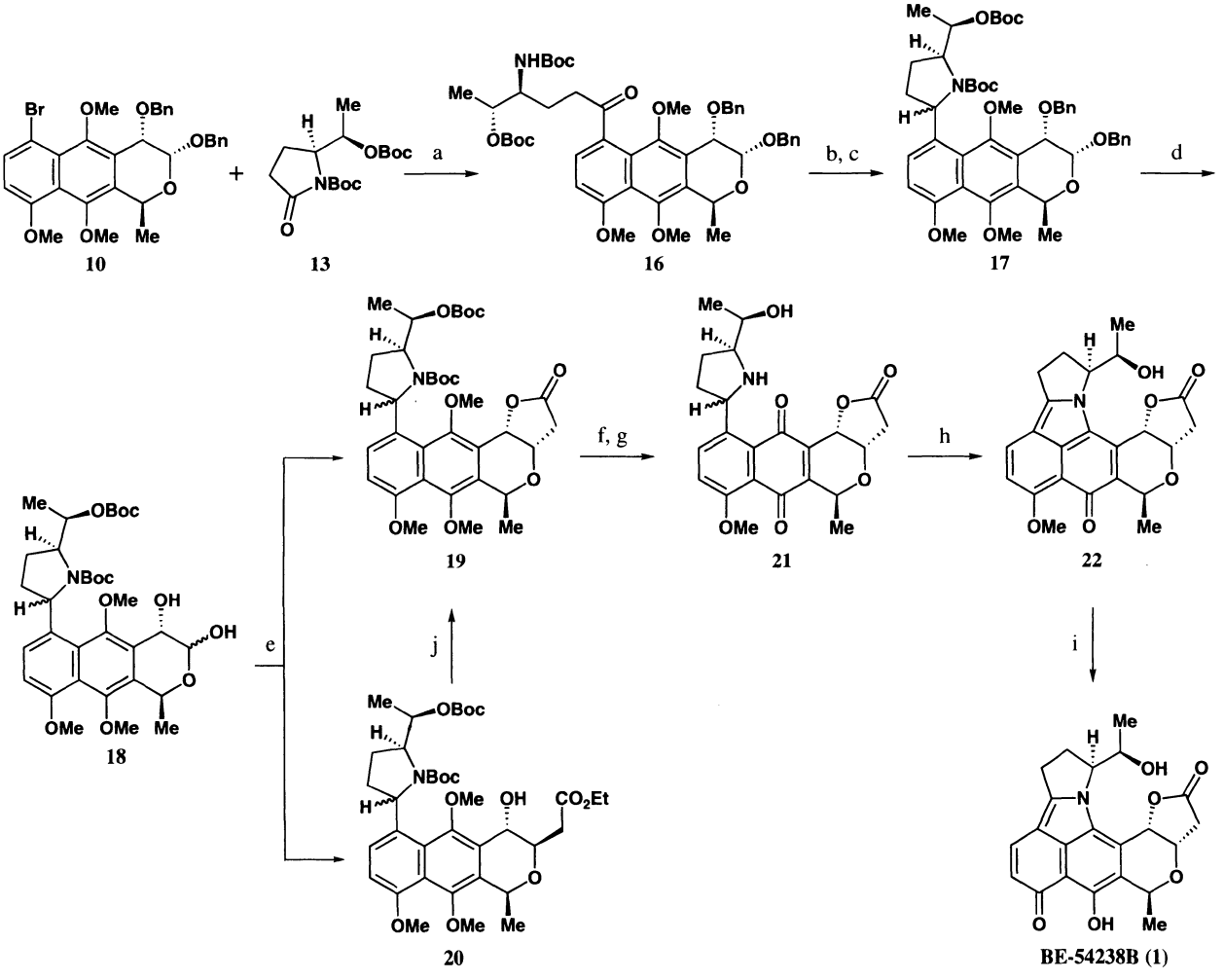
The *O*-benzyl precursor **8** was prepared according to our reported procedures[3,4] from benzyl 3,4,6-trideoxy-α-L-*glycero*-hex-3-enopyranosid-2-ulose (**6**) and 4-methoxy-3-(phenylsulfonyl)-1(3*H*)-isobenzofuranone (**7**), which were derived from L-rhamnoside **5** and *m*-methoxybenzoyl chloride,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

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