SYNTHESIS OF OPC-15161

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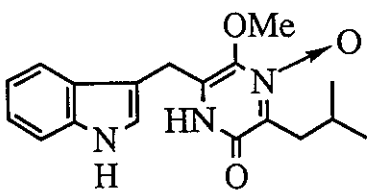
Abstract --- OPC-15161 (1), an inhibitor of superoxide anion generation by guinea pig macrophages, was synthesized from 5-chloro-3-isobutyl-6-methylpyrazine in several steps.

Some natural compounds, such as astechrome, Cypridina luciferin and OPC-15161 (1), consist of indole and pyrazine rings.[1-3] OPC-15161 (1), an inhibitor of superoxide anion generation by guinea pig macrophages, was recently obtained as a major degradation product of OPC-15160, which was isolated from the culture broth of the fungus Thielavia minor OFR-1561.[3] This substance had been previously synthesized by Kita et al.[4] from tryptophan methyl ester and by Ito et al.[5] from 2-(hydroxyimino)-4-methyl pentanoic acid and ethyl aminocyanoacetate.



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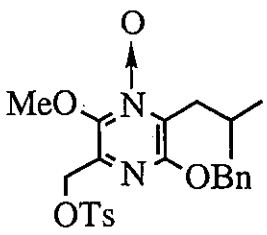

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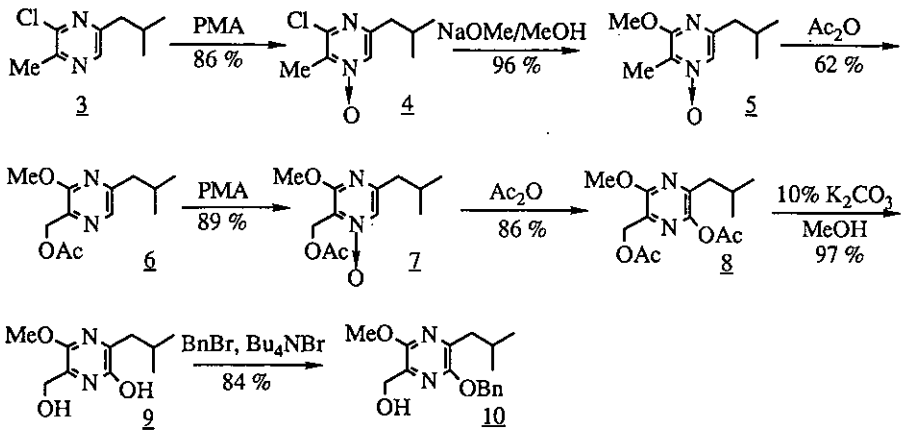
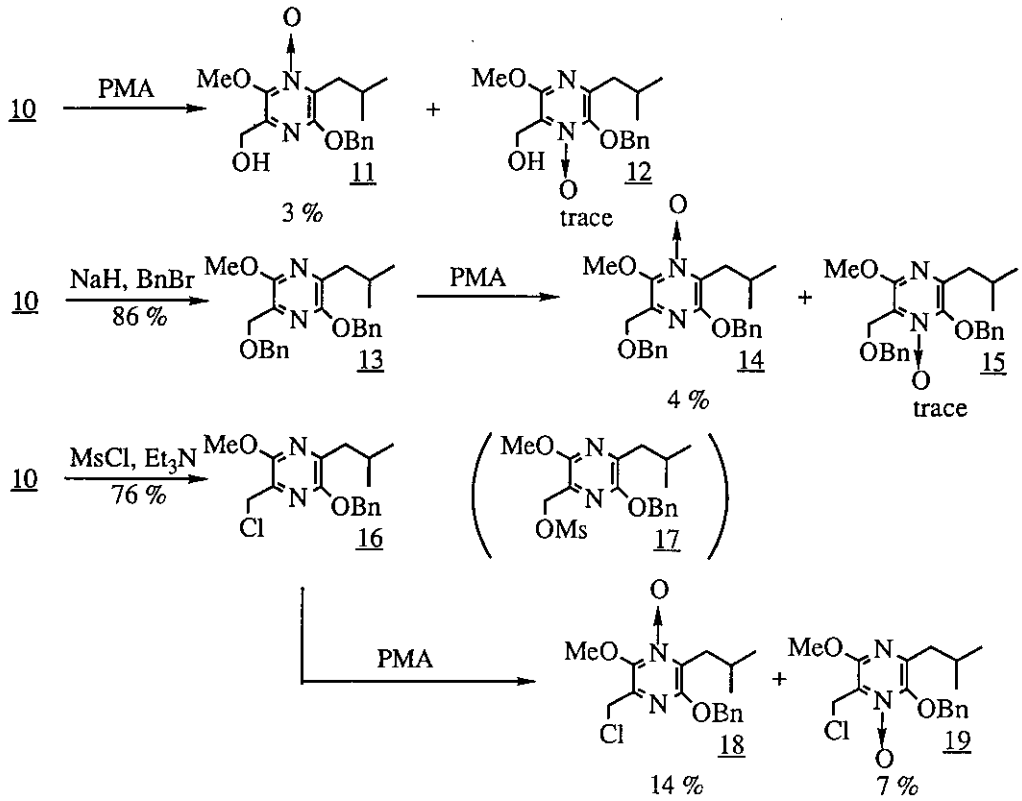
We were interested in the synthesis of these compounds and recently reported the coupling reaction[6] between indolylmagnesium bromide and tosyloxymethyl-pyrazines. To apply the reported method[6] to the synthesis of 1, the synthesis of 2-benzyloxy-3-isobutyl-5-methoxy-6-tosyloxymethylpyrazine 4-oxide (2) was attempted. However, the synthesis of 2 was unsuccessful. In this report, we describe a third method for synthesizing OPC-15161 from 5-chloro-3-isobutyl-6-methylpyrazine (3)[7] as the starting material via 2-benzyloxy-

6-iodomethyl-3-isobutyl-5-methoxypyrazine 4-oxide (**20**).



Scheme 1

Compound **3**[7] was oxidized with permaleic acid (PMA) to produce the corresponding 1-oxide (**4**). The position of the N-oxide group was determined on the basis of the ^{1}H-nmr spectra of the products.[8,9] Replacement of the chlorine atom of **4** with sodium methoxide in absolute methanol produced 3-isobutyl-5-methoxy-6-methylpyrazine 1-oxide (**5**), which was then converted to 6-acetoxymethyl-3-isobutyl-5-methoxypyrazine (**6**) by reflux in acetic anhydride. In order to introduce a hydroxyl group into the pyrazine ring, **6** was oxidized with PMA to give **7**, which was then converted to 2-acetoxy-6-acetoxymethyl-3-isobutyl-5-methoxypyrazine (**8**) by the reaction with acetic anhydride. After hydrolyzing **8** in 10% K_2CO_3, the product, 2-hydroxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine (**9**) was selectively O-benzylated with benzyl bromide under phase transfer catalysis conditions to give 2-benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine (**10**). The oxidation of **10** was then attempted. Direct oxidation with an oxidant, such as PMA or MCPBA, gave a mixture of the desired 2-benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine 4-oxide (**11**) and the isomeric 1-oxide (**12**). Compounds(**11**)and(**12**)were separated from each other by column chromatography, but their yields were very poor. Therefore, we attempted oxidation of the benzyl ether (**13**), after protecting the hydroxyl group of **10**, but with an even less satisfactory result (Scheme 2).



Scheme 2

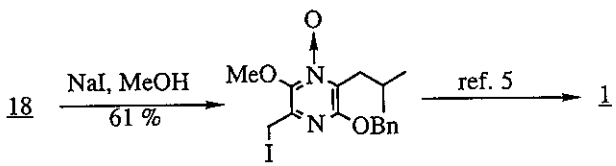
Compound(**10**)was treated with tosyl chloride in the presence of some bases, such as triethylamine, butyl lithium and sodium hydride. However, the starting material (**10**) was recovered in all cases. When **10** was treated with mesyl chloride in the presence of triethylamine, in the hope of producing the mesylated compound (**17**), the chlorinated substance (**16**) was obtained at a yield of 76%. Accordingly, the oxidation of 2-benzyloxy-6-chloromethyl-3-isobutyl-5-methoxypyrazine (**16**) was studied under various reaction conditions and the results are shown in Table 1. The products, 4-oxide (**18**) and 1-oxide (**19**), were separable from each other by medium pressure liquid chromatography.

Table 1. The oxidation of **16** (Reaction Time : 8 h ; Reaction Temperature: reflux)

Entry	Oxidant	Solvent	Oxidant/**16** (molar ratio)	Yield(%) **18**	Yield(%) **19**
1	PMA	$CHCl_3$	2.5	13	9
2	PMA	$CHCl_3$	4	14	7
3	PMA	$CHCl_3$	10	9	7
4	PMA	$ClCH_2CH_2Cl$	5	14	8
5	MCPBA	$CHCl_3$	5	0	0

The coupling reaction between indole and **18** was then conducted by two methods, using indolylmagnesium bromide and indolylzinc chloride. However, both methods caused the formation of many products which could not be separated.

Consequently, **18** was converted to 2-benzyloxy-6-iodomethyl-3-isobutyl-5-methoxypyrazine 4-oxide (**20**) by treatment with sodium iodide under heating in methanol. Compound **20** is an intermediate for the synthesis of **1**.[5] Thus the formal synthesis of **1** was accomplished (Scheme 3).



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Scheme 3

EXPERIMENTAL

The melting and boiling points are uncorrected. Distillation of the liquid products was conducted using a micro boiling apparatus (Sibata, Model G70-250RS). Medium-pressure column chromatography was conducted using a UVILOG ALPC-100 as the pump, UVILOG 5IIIa as the UV detector (Oyo Bunko Kiki Co. Ltd., Tokyo) and Kiesel gel 60 (Merck AG, Darmstadt) as the packing material.

^{1}H-Nmr spectral data were obtained with a Varian Gemini-300 in $CDCl_3$ using TMS as the internal standard. Other spectral data were obtained using the following instruments. Ir spetra: Japan Spectroscopic Co. A-100; Ms: Hitachi M-80 spectrometer.

Oxidation of 5-Chloro-3-isobutyl-6-methylpyrazine (3): A solution of 3 (5.0 g, 27.2 mmol), 60% H_2O_2 (2.0 g, 35.1 mmol) and maleic anhydride (4.0 g, 40.8 mmol) in $CHCl_3$ (100 ml) was stirred overnight at room temperature. The reaction mixture was then washed successively with H_2O, 10% $KHCO_3$ and H_2O. The organic layer was worked up as usual to give a mixture, which was purified by silica gel column chromatography with hexane containing an increasing amount of AcOEt to produce 4 as colorless needles (from MeOH-H_2O); mp 62-63°C; yield: 4.7 g (86%); ms: m/z 200 (M^+); ^{1}H-nmr: 0.98 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.15 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.55 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 2.59 (s, 3H, pyrazine CH_3), 7.95 (s, 1H, pyrazine H) ppm; Anal. Calcd for $C_9H_{13}N_2OCl$: C, 53.87; H, 6.53; N, 13.96. Found: C, 53.84; H, 6.52; N, 13.92.

Synthesis of 3-Isobutyl-5-methoxy-6-methylpyrazine 1-Oxide (5): In a NaOMe-MeOH solution, prepared from abs. MeOH (115 ml) and sodium (2.5 g, 108.7 mg atom), 4 (4.4 g, 21.8 mmol) was heated under reflux for 2 h. After the solvent was removed by distillation in vacuo, the residue was triturated with water and extracted with Et_2O. The extract was worked up as usual to give a crystalline mass, which was recrystallized from MeOH-H_2O to produce 4.1 g (96%) of 5 as colorless needles; mp 60-62°C; ms: m/z 196 (M^+); ^{1}H-nmr: 0.94 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$); 2.10 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.38 (s, 3H, pyrazine CH_3), 2.43 (d, J = 6 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.98 (s, 3H, OCH_3), 7.67 (s, 1H, pyrazine H) ppm; Anal. Calcd for $C_{10}H_{16}N_2O_2$: C, 61.20; H, 8.22; N, 14.28. Found: C, 61.10; H, 8.17; N, 14.27.

Reaction of 3-Isobutyl-5-methoxy-6-methylpyrazine 1-Oxide (5) with Acetic Anhydride: A solution of 5 (1.9 g, 9.7 mmol) in Ac_2O (60 ml) was refluxed for 1 h and poured into ice-water. The solution was made alkaline with powdered K_2CO_3 and extracted with Et_2O. A standard work-up of the extract produced a dark-brown oil, which was purified by distillation to give 1.4 g (62%) of 6 as a colorless oil; bp 145-7°C/3 torr; ms: m/z 238 (M^+); ir (neat): 1750 (C=O) cm^{-1}; ^{1}H-nmr: 0.96 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.14 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.16 (s, 3H, $CH_2OCOC\underline{H}_3$), 2.55 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.98 (s, 3H, CH_3O), 5.22 (s, 2H, $C\underline{H}_2OCOCH_3$), 7.93 (s, 1H,

pyrazine H) ppm; Anal. Calcd for $C_{12}H_{18}N_2O_3$: C, 60.49; H, 7.61; N, 11.76. Found: C, 60.45; H, 7.62; N, 11.71.

Oxidation of 6-Acetoxymethyl-3-isobutyl-5-methoxypyrazine (6): A solution of 6 (800 mg, 3.4 mmol), 60% H_2O_2 (200 mg, 4.3 mmol), and maleic anhydride (600 mg, 6.6 mmol) in $CHCl_3$ (15 ml) was stirred overnight at room temperature. The reaction mixture was treated similarly to the oxidation of 3, and the product was purified by distillation to give 800 mg (89%) of 7 as a colorless oil; bp 150-160°C/1 torr; ms: m/z 254 (M^+); ir (neat): 1740 (C=O) cm^{-1}; ^{1}H-nmr: 0.96 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.09 (s, 3H, $CH_2COC\underline{H}_3$), 2.17 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.47 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 4.00 (s, 3H, CH_3O), 5.33 (s, 2H, $C\underline{H}_2OCOCH_3$), 7.88 (s, 1H, pyrazine H) ppm; Anal. Calcd for $C_{12}H_{18}N_2O_4$: C, 56.68; H, 7.14; N, 11.02. Found: C, 56.70; H, 7.17; N, 11.01.

Reaction of 6-Acetoxymethyl-3-isobutyl-5-methoxypyrazine 1-Oxide (7) with Acetic Anhydride: A solution of 7 (100 mg, 0.4 mmol) in Ac_2O (2.5 ml) was treated similarly to the reaction of 5 with Ac_2O. The product was purified by distillation to produce 100 mg (86%) of 8 as a colorless oil; bp 155-165°C/1 torr; ms: m/z 296 (M^+); ir (neat): 1750 (C=O), 1780 (C=O) cm^{-1}; ^{1}H-nmr: 0.96 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.12 (s, 3H, $CH_2OCOC\underline{H}_3$), 2.16 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.34 (s, 3H, $OCOCH_3$), 2.49 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.98 (s, 3H, OCH_3), 5.15 (s, 2H, $C\underline{H}_2OCOCH_3$) ppm; Anal. Calcd for $C_{14}H_{20}N_2O_5$: C, 56.75; H. 6.80; N, 9.45. Found: C, 56.59; H, 6.80; N, 9.33.

Hydrolysis of 2-Acetoxy-6-acetoxymethyl-3-isobutyl-5-methoxypyrazine (8): A solution of 8 (11.4 g, 38.5 mmol) in a mixture of 10% K_2CO_3 (115 ml) and MeOH (115 ml) was stirred for 0.5 h at room temperature, followed by removal of the solvent by distillation in vacuo. Water was added to the residue and the solution was extracted with Et_2O. After a standard work-up, the crude products were recrystallized from hexane to produce 8.0 g (97%) of 9 as colorless needles; mp 110-113°C; ms: m/z 212 (M^+); ir (KBr): 3100 (OH) cm^{-1}; ^{1}H-nmr: 0.94 (d, J = 6 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.17 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.62 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.92 (s, 3H, OCH_3), 4.68 (s, 2H, $C\underline{H}_2OH$) ppm; Anal. Calcd for $C_{10}H_{16}N_2O_3$: C, 56.59; H, 7.60; N, 13.20. Found: C, 56.30; H, 7.52; N, 13.12.

Benzylation of 2-Hydroxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine (9): Ten percent NaOH (120 ml) was added at room temperature to a mixture of 9 (3.0 g, 14.2 mmol), BnBr (1.9 ml, 15.7 mmol), and a catalytic amount of

tetrabutylammonium bromide in CH_2Cl_2 (120 ml). After ultrasound was applied to the reaction mixture for 3 h, the mixture was made acidic with 5% HCl and extracted with CH_2Cl_2. The organic layer received a standard work-up followed by silica gel column chromatography with hexane containing an increasing amount of AcOEt, to give 3.6 g (84%) of **10**; cololess prisms (from hexane); mp 55-57°C; ms: m/z 302 (M^+); ir (KBr): 3475 (OH) cm^{-1}; ^{1}H-nmr: 0.96 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.19 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.65 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.93 (s, 3H, OCH_3), 4.66 (s, 2H, $C\underline{H}_2OH$), 5.36 (s, 2H, $OC\underline{H}_2C_6H_5$), 7.26-7.45 (m, 5H, $OCH_2C_6\underline{H}_5$) ppm; Anal. Calcd for $C_{17}H_{22}N_2O_3$: C, 67.53; H, 7.33; N, 9.29. Found: C, 67.37; H, 7.33; N, 9.19.

Oxidation of 2-Benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine (**10**): A solution of **10** (1.0 g, 3.3 mmol), 60% H_2O_2 (400 mg, 6.3 mmol) and maleic anhydride (600 mg, 6.3 mmol) in $CHCl_3$ (157 ml) was refluxed for 4 h, and then treated similarly to the oxidation of **3**. The reaction mixture was purified by silica gel chromatography. Compounds **12** (trace) and **11** (31.5 mg, 3%) were successively eluted with hexane containing an increasing amount of AcOEt.

2-Benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine 4-Oxide (**11**): colorless prisms (from hexane-Et_2O); mp 65-66°C (lit.,[5]: mp 68.0-69.2°C); The spectral data agreed with the previous report.[5]

2-Benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine 1-Oxide (**12**): a viscous oil; CI-ms: m/z 319 (M^++1); ^{1}H-nmr: 0.87 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.11 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.43 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.69 (s, 3H, OCH_3), 4.87 (s, 2H, $C\underline{H}_2OH$), 5.31 (s, 2H, $OC\underline{H}_2C_6H_5$), 7.36-7.40 (m, 5H, $OCH_2C_6\underline{H}_5$) ppm.

Benzylation of 2-Benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine (**10**): A solution of **10** (100 mg, 0.3 mmol) in dry THF (2 ml) was added to a suspension of NaH (15 mg, 0.4 mmol) in dry THF (1 ml). BnBr (0.04 ml, 0.4 mmol) was added after the solution had been stirred at room temperature for 1 h. The reaction mixture was stirred overnight at room temperature, washed with H_2O and dried over Na_2SO_4. After a standard work-up of the organic layer, the product was purified by silica gel column chromatography, eluting with hexane containing an increasing amount of AcOEt, to produce **13** (100 mg, 86%) as a colorless oil; ms: m/z 392 (M^+); ^{1}H-nmr: 0.93 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.18 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.64 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.91 (s, 3H, OCH_3), 4.47 (s, 2H, $C\underline{H}_2OCH_2C_6H_5$), 4.60 (s, 2H, $CH_2OC\underline{H}_2C_6H_5$),

5.37 (s, 2H, $C\underline{H}_2C_6H_5$), 7.25-7.46 (m, 10H, $CH_2OCH_2C_6\underline{H}_5$ and $OCH_2C_6\underline{H}_5$) ppm.

Oxidation of 2-Benzyloxy-6-benzyloxymethyl-3-isobutyl-5-methoxypyrazine (13): A solution of 13 (100 mg, 0.3 mmol), 60% H_2O_2 (32 mg, 0.6 mmol) and maleic anhydride (55.4 mg, 0.6 mmol) in $CHCl_3$ (6 ml) was refluxed for 4 h. The reaction mixture was then treated similarly to the oxidation of 3. The reaction mixture was applied to a silica gel column and 15 (trace) and 14 (4.9 mg, 4%) were successively eluted with hexane containing an increasing amount of AcOEt.

2-Benzyloxy-6-benzyloxymethyl-3-isobutyl-5-methoxypyrazine 4-Oxide (14): a viscous oil; CI-ms: m/z 409 (M^++1); ^{1}H-nmr: 0.94 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.23 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.83 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 4.02 (s, 3H, OCH_3), 4.59 (s, 2H, $CH_2OC\underline{H}_2C_6H_5$), 4.66 (s, 2H, $C\underline{H}_2OCH_2C_6H_5$), 5.40 (s, 2H, $OC\underline{H}_2C_6H_5$), 7.32-7.44 (m ,10H, $OCH_2C_6\underline{H}_5$ and $CH_2OCH_2C_6\underline{H}_5$) ppm.

2-Benzyloxy-6-benzyloxymethyl-3-isobutyl-5-methoxypyrazine 1-Oxide (15): ^{1}H-nmr: 0.86 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.17 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.42 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.92 (s, 3H, OCH_3), 4.71 (s, 2H, $CH_2OC\underline{H}_2C_6H_5$), 4.84 (s, 2H, $C\underline{H}_2OCH_2C_6H_5$), 5.30 (s, 2H, $OC\underline{H}_2C_6H_5$), 7.33-7.47 (m, 10H, $OCH_2C_6\underline{H}_5$ and $CH_2OCH_2C_6\underline{H}_5$) ppm.

Mesylation of 2-Benzyloxy-6-hydroxymethyl-3-isobutyl-5-methoxypyrazine (10): A solution of 10 (1.0 g, 3.3 mmol), Et_3N (0.9 ml, 6.6 mmol) in dry CH_2Cl_2 (10 ml) was added to MsCl (0.3 ml, 3.4 mmol) at 0°C. After stirring at room temperature overnight, water was added to the reaction mixture. The organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined CH_2Cl_2 extract was worked up as usual to produce a crude product, which was purified by silica gel column chromatography. Compound(16)(810 mg, 76%) was obtained; colorless needles (from hexane); mp 52-53°C; ms: m/z 320 (M^+); ^{1}H-nmr: 0.95 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.19 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.66 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.97 (s, 3H, OCH_3), 4.65 (s, 2H, CH_2Cl), 5.36 (s, 2H, $OC\underline{H}_2C_6H_5$), 7.25-7.48 (m, 5H, $OCH_2C_6\underline{H}_5$) ppm; Anal. Calcd for $C_{17}H_{21}N_2O_2Cl$: C, 63.64; H, 6.60; N, 8.73. Found: C, 63.69; H, 6.58; N, 8.88.

Oxidation of 2-Benzyloxy-6-chloromethyl-3-isobutyl-5-methoxypyrazine (16): A solution of 16 (51 mg, 0.2 mmol), 60% H_2O_2 (44 mg, 0.8 mmol) and maleic anhydride (78 mg, 0.8 mmol) in $CHCl_3$ (2.5 ml) was refluxed for 8 h. The reaction mixture was then treated similarly to the oxidation of 3. The crude

products were purified by silica gel column chromatography. Compounds(19)(3.7 mg, 7%) and(18)(7.3 mg, 14%) were successively eluted with hexane containing an increasing amount of AcOEt.

2-Benzyloxy-6-chloromethyl-3-isobutyl-5-methoxypyrazine 4-Oxide (18): colorless prisms (from pentane); mp 61-63°C; CI-ms: m/z 337 (M^++1), ^{1}H-nmr: 0.94 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.23 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.83 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 4.12 (s, 3H, OCH_3), 4.60 (s, 2H, CH_2Cl), 5.40 ((s, 2H, $OC\underline{H}_2C_6H_5$), 7.32-7.48 (m, 5H, $OCH_2C_6\underline{H}_5$) ppm; Anal. Calcd for $C_{17}H_{21}N_2O_3Cl$: C, 60.62; H, 6.28; N, 8.32. Found: C, 60.64; H, 6.23; N, 8.38.

2-Benzyloxy-6-chloromethyl-3-isobutyl-5-methoxypyrazine 1-Oxide (19) : ms: a viscous oil; m/z 336 (M^+); ^{1}H-nmr: 0.87 (d, J = 7 Hz, 6H, $CH_2CH(C\underline{H}_3)_2$), 2.18 (m, 1H, $CH_2C\underline{H}(CH_3)_2$), 2.44 (d, J = 7 Hz, 2H, $C\underline{H}_2CH(CH_3)_2$), 3.98 (s, 3H, OCH_3), 4.86 (s, 2H, CH_2Cl), 5.35 (s, 2H, $OC\underline{H}_2C_6H_5$), 7.34-7.47 (m, 5H, $OCH_2C_6\underline{H}_5$) ppm.

Synthesis of 2-Benzyloxy-6-iodomethyl-3-isobutyl-5-methoxypyrazine 4-Oxide (20): A solution of 18 (14.7 mg, 0.04 mmol), NaI (13 mg, 0.09 mmol) in MeOH (0.5 ml) was refluxed for 4 h. After evaporation of the solvent, water was added, and the mixture was extracted with Et_2O. The extract was worked up as usual and the residue was subjected to preparative TLC on silica gel (hexane: AcOEt 7:3) to produce 20 (11.4 mg, 61%) as a colorless needles (from hexane-Et_2O); mp 75-77°C (lit.[5] 78.3-79.5°C). The spectral data agreed with the previous report.[5]

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