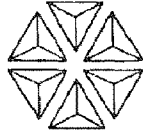


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NOTEBOOK No. 47583

Assigned to David Bete Benner

Subject _____

Department Name metabolic Diseases Chemistry

Department Number 8000102

Date Assigned July 5, 2000 Tom Karlton's

Date Completed 2-26-2001

Pages Completed from 001 to 200

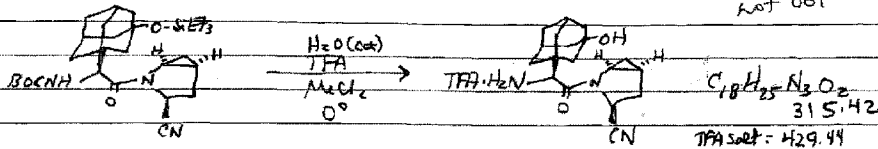
Continued from Notebook Number 45674

Continued in Notebook Number 48636

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DATE: 10-18-2000 PROJ. NO. DP4 08808 EXPT. NO. DAB 47583-081

SUBJECT: BMS-477118-01



	MW	Source	d	mmol	equiv	wt (vol)
1. SM	529.8	DAB 47583-080-15		0.1435	1	76 mg
2. Trifluoroacetic acid + 2 drops H ₂ O	114.02					1 mL
3. MeCl ₂ ; enclhyano		EM Science				1 mL

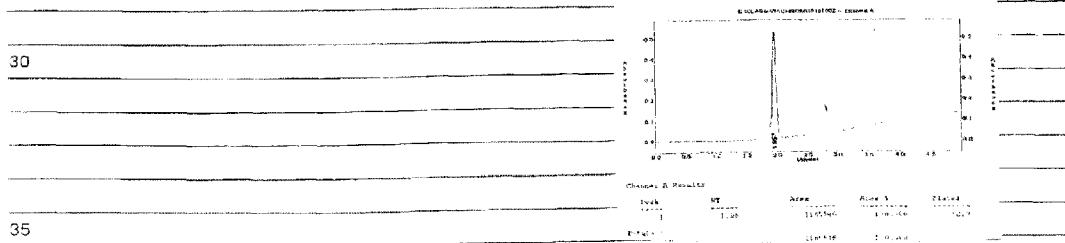
10 Dissolved SM in MeCl₂ & cooled to 0°C; added TFA and 2 drops of H₂O. Let stir at 0°C. TLC (SiO₂, 10% EtOAc / MeCl₂; HCl-MeOH/ninhydrin): no SM after 40 min. LC-MS (Phenomenex 4.5x50mm, 4 min gradient @ 4ml/min, > 220 nM; start % B = 2.0 → final % B = 100; A = 10: 90 MeOH/H₂O + 10 mM NH₄Ac, B = 90:10 MeOH/H₂O + 10 mM NH₄Ac): m/z 316 (m+H)⁺, 631 (2m+H)⁺ on (+)-ion @ RT = 1.190 min (97.7%); see no SM.

15 After 1.5 hr, removed solvents by rot. evapn. *in vacuo*, dried on hi-vac, then chased in enclhy. toluene. Dried on hi-vac, as white solid. Trichlorated solid with Et₂O (2x5 mL) & vacuum filtered. Collected white solid.

Weight 54 mg (0.1257 mmol, 88%)
 * NMR # DAB 47583-081-18 (CD₃OD, 500 MHz) ¹H & ¹³C.
 20 + LC-MS (Phenomenex 005 4.6x50mm, 4ml/min @ 4min gradient, > 220 nM; start % B = 0 → final % B = 100; A = 10:90 MeOH/H₂O + 0.1% TFA, B = 90:10 MeOH/H₂O + 0.1% TFA) m/z 631 (2m+H)⁺, 316 (m+H)⁺ @ RT = 2.029 min (75.7%)

25 * Analyt. HPLC:

Analytical HPLC Report
 File: C:\CLABS\VENUS\01047_002
 Sample ID: 0784-081-18
 Sample: 005 31, 2000 10140115
 Analyst: DEY-DAVID-000008
 Title Desc: Analyt. HPLC
 Acquisition: 08-11-2000 11:11:11
 Vial: 145
 Queue: 3 of 0
 Inlet: 3 of 100
 Equilibr. Time: 4 min
 Flow Rate: 1 ml/min
 Wavelength: 214
 Solvent A: 10% MeOH - 90% H₂O - 0.1% TFA
 Solvent B: 90% MeOH - 10% H₂O - 0.1% TFA
 Column 1: TAC 50 500 4.6 x 50 mm Ballistic (MSB01)



* Elemental: p. DAB 47583-088

SIGNED: David Patel DATE: 10-18-2000 WITNESSED AND UNDERSTOOD BY: Wu Xj DATE: 7/16/01

CROSS REFERENCES:

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