

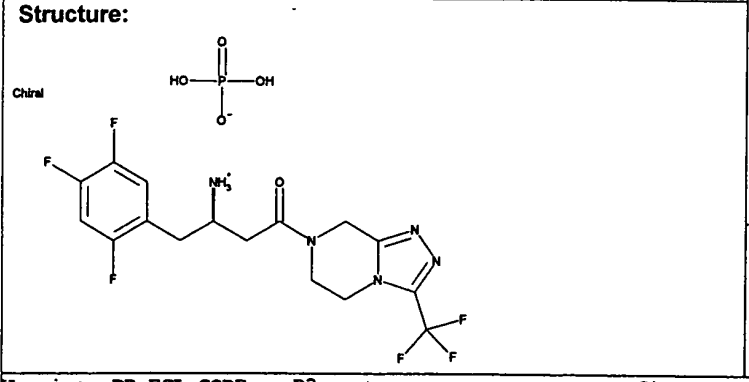
Empirical Formula: C16H16F6N5O 408.329 H2O4P 96.987
 Molecular Weight: 505.317
 Chemical name: (2R)-4-OXO-4-[3-(TRIFLUOROMETHYL)-5,6-DIHYDRO[1,2,4]TRIAZOLO[4,3-A]PYRAZIN-7(8H)-YL]-1-(2,4,5-TRIFLUOROPHENYL)BUTAN-2-AMINE PHOSPHORIC ACID SALT

L-224,715-006F007 18-APR-2002
 Project Code: 0726

Prepared By: K. HANSEN
 Amount Made: 588 G Reference: 72471-017
 Date Of Manufacture: 25-MAR-2002
 Sample ID(s): 200004568

Date(s) Tested: 25-MAR-2002
 Nature Of Change: FIRST DELIVERY
 Milled: N Process Change: N
 Restricted Release By: J. ARMSTRONG AND R. TILLYER

Send TO: Bob Martell WP78-110 520g Phi Form
 J. Dorwart RY818-C208 (already sent) 10 g (stability)
 Reserve Sample RY28 1 gram
 19-APR-2002



Warning: PB-ECL CODE P3 Storage Conditions: REFRIGERATED

500MG FREEZER - 19-APR-2002

Appearance	CLEAN	MICROSCOPIC EXAM	Observations	ANISOTROPIC, IRREGULAR SHAPED PARTICLES, SOME AGGLOMERATION	IDENTITY BY FTIR	Conforms YES
Color	WHITE				Reference Std.	L-224715-006F006 NB# 72471-011 PPCL# 0213276
Form	POWDER					
X-RAY		SURFACE AREA	Surface area	7.77 sq. m/g	SPECIFIC ROTATION (SOLID)	
Observations	CRYSTALLINE				Solvent	DI WATER
Reference Std.	1ST GMP DELIVERY	PARTICLE SIZE	Technique	MICRO TRAC	Concentration	1.0 (w/v)%
IMPURITY PROFILE			Distribution type	VOLUME	Wavelength	365 nm
RRT 0.95	0.13 %		Average size	63. um	Specific Rotation	-74.4 degrees*
RRT 0.97	0.14 %		S.D. of distribution	38. um	DSC CHARACTERIZATION	
Total impurities	0.32 %		% < size	143. um	T onset 1	216.9 degrees -C
# of quantifiable impurities <0.10%	1		Size	17. %	T max 1	218.7 degrees -C
No other impurity =>	0.05 %			25 um	Delta H 1	113.2 J/g
TITRATION - PERCHLORIC ACID		BULK DENSITY			TG	
Analyte	L-224715-006F	Loose density	0.46 g/mL		Weight Loss 1	N.D. w/w%
Result	100.0 (w/w)%*	Tapped density	0.60 g/mL		Final Temp	224.2 degrees -C
TITRATION - SODIUM HYDROXIDE		COLOR (SOLID)			HEAVY METALS BY ICP/MS	
Analyte	PHOSPHATE	Solvent	DI WATER		USP<231> and EPA regulated elements	<10. ppm
Result	19.9 (w/w)%*	Concentration	1.0 (w/v)%		POTENCY AS-IS	
ABSORBANCE - 1ST WAVELENGTH		Wavelength	440 nm		Potency	99.6 %
Solvent	DEIONIZED WATER	A# x 1000	5.		As	L-224715 PHOSPHATE SALT
Wavelength 1	267.0 nm	NEPHELOS			Rationale	{100-KF-GC} X 0.01{100 - (HPLC AREA% IMP + HPLC CHIRAL)}
A# @ wavelength 1	37.3 A#*	Solvent	DI WATER		PURITY	
KARL FISCHER (KF)		Concentration	1.0 (w/v)%		Purity	99.7 %
Water	<0.1 w/w%	Result	1.1 ntu		Rationale	BASED ON HPLC AREA%
RESIDUAL SOLVENTS		PH				
EtoH	N.D. (w/w)%	Solvent	DI WATER			
RESIDUE ON IGNITION BY ICP		Concentration	1.0 (w/v)%			
Wt% of residue	<0.05 (w/w)%	Sample pH	4.7 pH			
CHIRAL PURITY		DISC				
L-224,715*	99.99 %	Solvent	DI WATER			
L-400,224	N.D. %	Net wt. (sample)	2.2 grams			
		Visual Comparison to Std	< STD 1			

* Dry basis

COPIES

NaOH titration for phosphate (theory = 19.4%). Pd < 3 ppm, Ru < 3 ppm, Cu < 3ppm

Dept. Approval: [Signature] 18 Apr 2002 Del. Sheet By: SCOTT THOMAS 18-APR-2002
 Quality Approval: Jean M. Wyvrat 18 Apr 2002 Reviewed By: Beverly M. Eisele 18-Apr-2002
 Transferred By: P. Lopez 07-MAY-2002 Run ID: 103263798

CC: Egan RY818-B205, Eiserle RY818-C221, Thomas RY818-C208, Wyvrat RY818-B201, Hansen RY800-B269, Armstrong RY800-D371, Palucki RY800-B363, Kumke RY819-405

