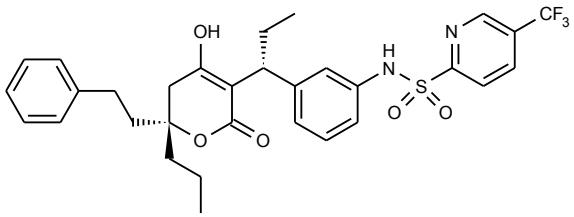
BDG SYNTHESIS

Certificate of Analysis

BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Barry Dent

Barry R. Dent, PhD, Director
2 December 2010

Name:	**Tipranavir**
CAS Number:	174484-41-4
Structure:	
Molecular Weight:	$C_{31}H_{33}F_3N_2O_5S$ = 602.66
Lot Number:	BDG 6376.5
Appearance:	White powder
Purity By HPLC:	98.7 %
Re-test Date:	2 December 2015

Storage and Handling:	Temperature:	refrigerate for prolonged storage; may be handled and shipped at ambient temperature.
	Humidity:	not believed to be hygroscopic; may be handled in normal laboratory atmosphere.
	Light:	protect from strong sunlight.
	Caution:	only experienced laboratory personnel should handle the material. Avoid contact of compound with acids.

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Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.
Residual Solvents: a trace (under 0.1 % *w/w*) of 2-propanol is observed.
Impurities: no significant impurities are evident in the spectrum.

Carbon-13 NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available. All carbon resonances are observed with the exception of the CF_3 carbon which is obscured by the methine carbons around 125 ppm.

High-resolution Mass Spectrum (ESI+)

Found *m/z* 625.1970. $C_{31}H_{33}F_3N_2NaO_5S$ $[M+Na]^+$ requires *m/z* 625.1954. The deviation of 2.5 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

A sharp, symmetrical peak is observed (98.7 %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental Analysis

	Found:	C 61.50, H 5.71, N 4.59 %
$C_{31}H_{33}F_3N_2O_5S$	Requires:	C 61.78, H 5.52, N 4.65 %

The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

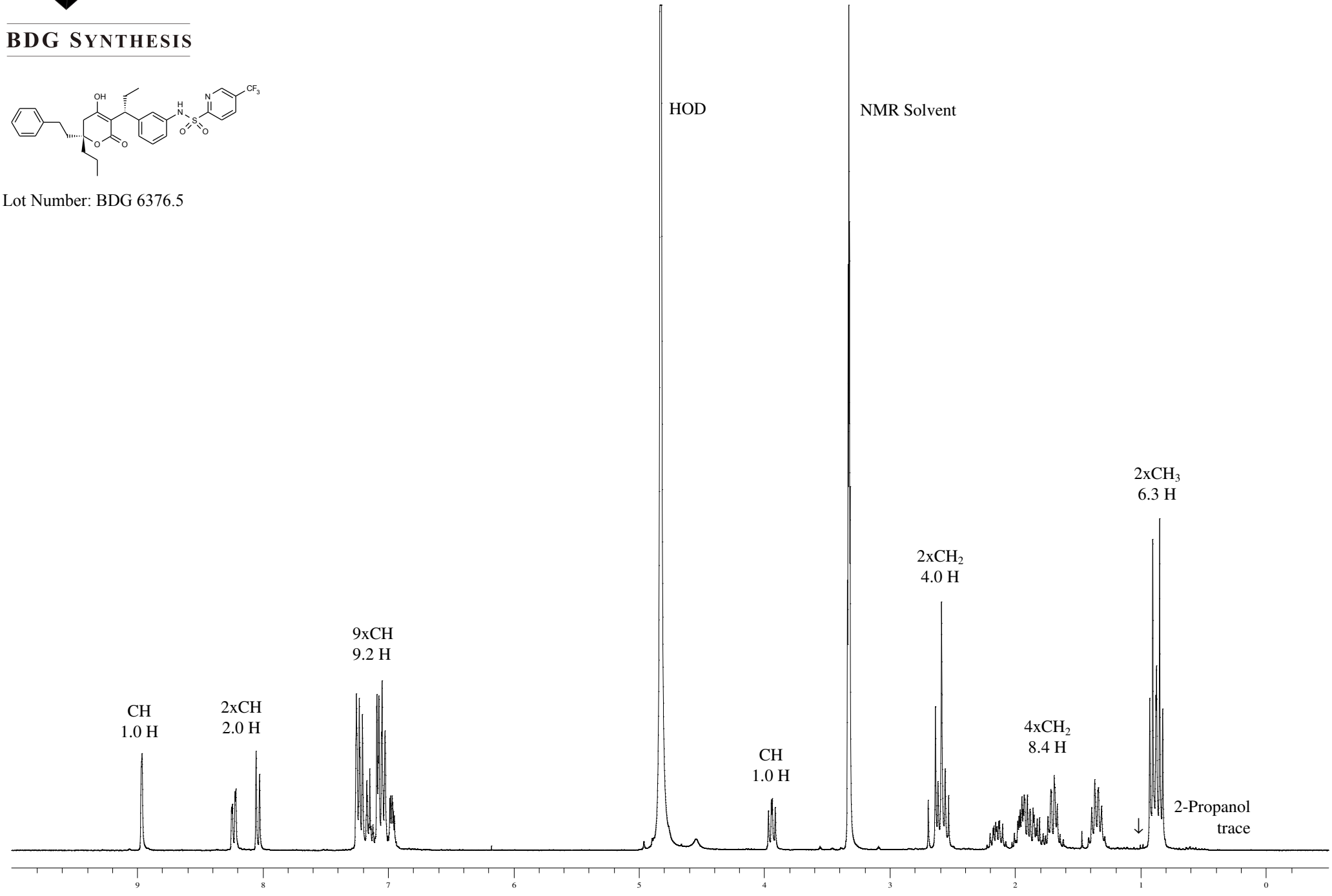
The available quantity of custom-synthesised material is always small, and this limits the extent and type of analytical data which can be obtained. This Certificate is presented in descriptive format for use by analytical chemists who are trained in the use of custom-synthesised materials. Custom materials often contain higher levels of residual solvents and/or water, and we urge you to use the corrected purity where needed rather than the raw HPLC purity. This compound is intended for use as an analytical reference material and it is not for human administration. Structures are shown with relative stereochemistry unless otherwise specified.

The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

BDG SYNTHESIS

Proton NMR Spectrum of Tipranavir in Methanol-d_4

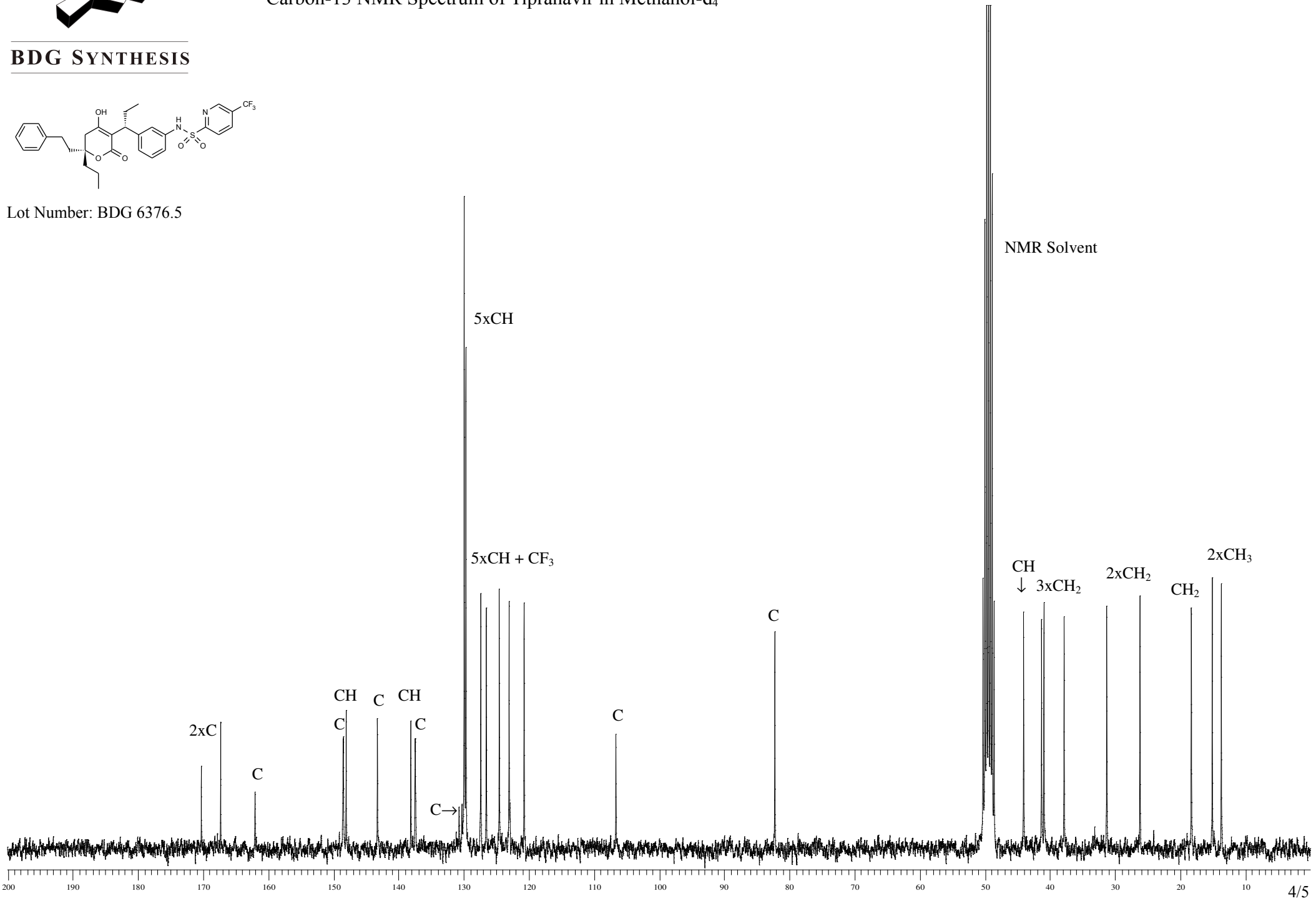
Lot Number: BDG 6376.5



BDG SYNTHESIS

Carbon-13 NMR Spectrum of Tipranavir in Methanol-d_4

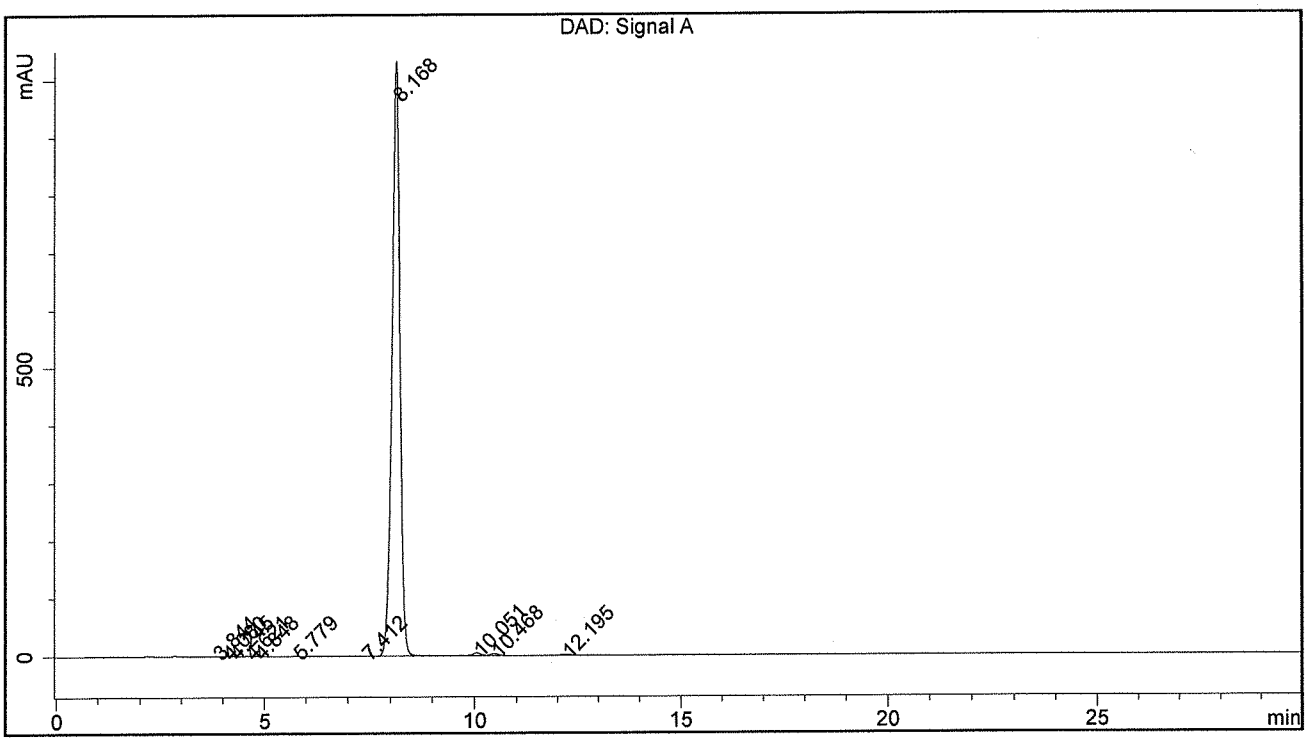
Lot Number: BDG 6376.5



BDG - Analysis of Tipranavir

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
Mobile Phase : 27:10:63 50mM Phosphate Buffer pH=5.9: Methanol : Acetonitrile
Flow Rate : 1.0 mL/min
Sample Solvent : Mobile Phase
Column Temperature : 20C
Injection Volume : 10 uL
Detection : UV at 205 nm

Sample Name	BDG 6376.5	**Instrument**	AnalyticalLC01
Acquisition	02/12/2010, 16:01:28	**Method (rev.)**	LC10023a (7)
Sequence	BDG_02Dec2010a - Reprocessed	**Vial Position**	1
Operator	solvation010\cerityadmin	**Injection**	1 of 1



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	3.84 min	0.6039	3.1739	0.0793 min	0.023 %
2	4.08 min	0.5033	3.5394	0.1020 min	0.026 %
3	4.25 min	0.8831	4.7498	0.0847 min	0.035 %
4	4.62 min	0.6806	5.0166	0.1140 min	0.037 %
5	4.85 min	1.1367	11.0824	0.1437 min	0.082 %
6	5.78 min	0.4215	4.7705	0.1620 min	0.035 %
7	7.41 min	0.2529	2.2347	0.1331 min	0.017 %
8	8.17 min	1032.1016	13337.7459	0.1908 min	98.711 %
9	10.05 min	4.6594	66.0613	0.2134 min	0.489 %
10	10.47 min	3.2367	44.9029	0.2119 min	0.332 %
11	12.19 min	1.6728	28.6913	0.2581 min	0.212 %