

BDG SYNTHESIS

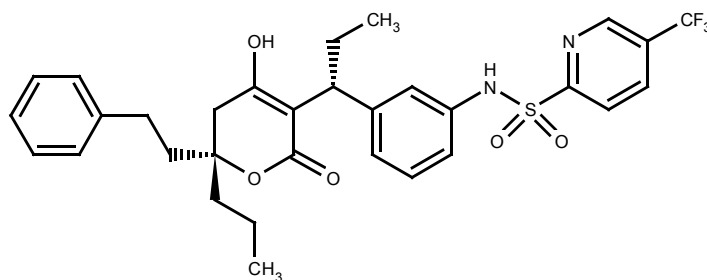
Certificate of Analysis

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

Barry Dent

Barry Dent, PhD, Director
1 April 2009

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: 98.3 %
Expiry Date: 1 April 2010
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 acid.

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 CF₃ carbon which is obscured by the methine carbons around 125 ppm.

High-resolution mass spectrum (ESI+): Found m/z 625.1970. C₃₁H₃₃F₃N₂NaO₅S [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.

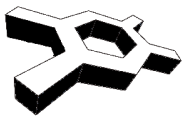
HPLC: A sharp peak is observed (98.3 %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

Elemental analysis:	Found:	C 61.50, H 5.71, N 4.59 %
C ₃₁ H ₃₃ F ₃ N ₂ O ₅ S	requires:	C 61.78, H 5.52, N 4.65 %

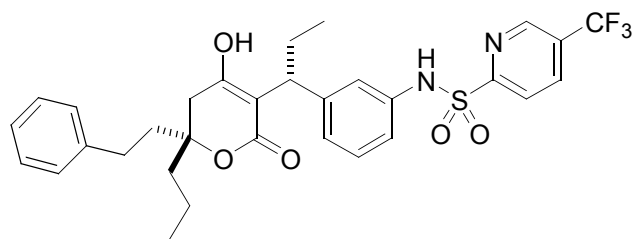
The elemental analyses fall within generally accepted limits for establishing the molecular formula given. The results may also be taken to imply the absence of significant quantities of water or inorganic salts (which have not been elsewhere tested for because of sample size limitations).

The quantity of this material available is limited, 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 research-grade materials. Research 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.

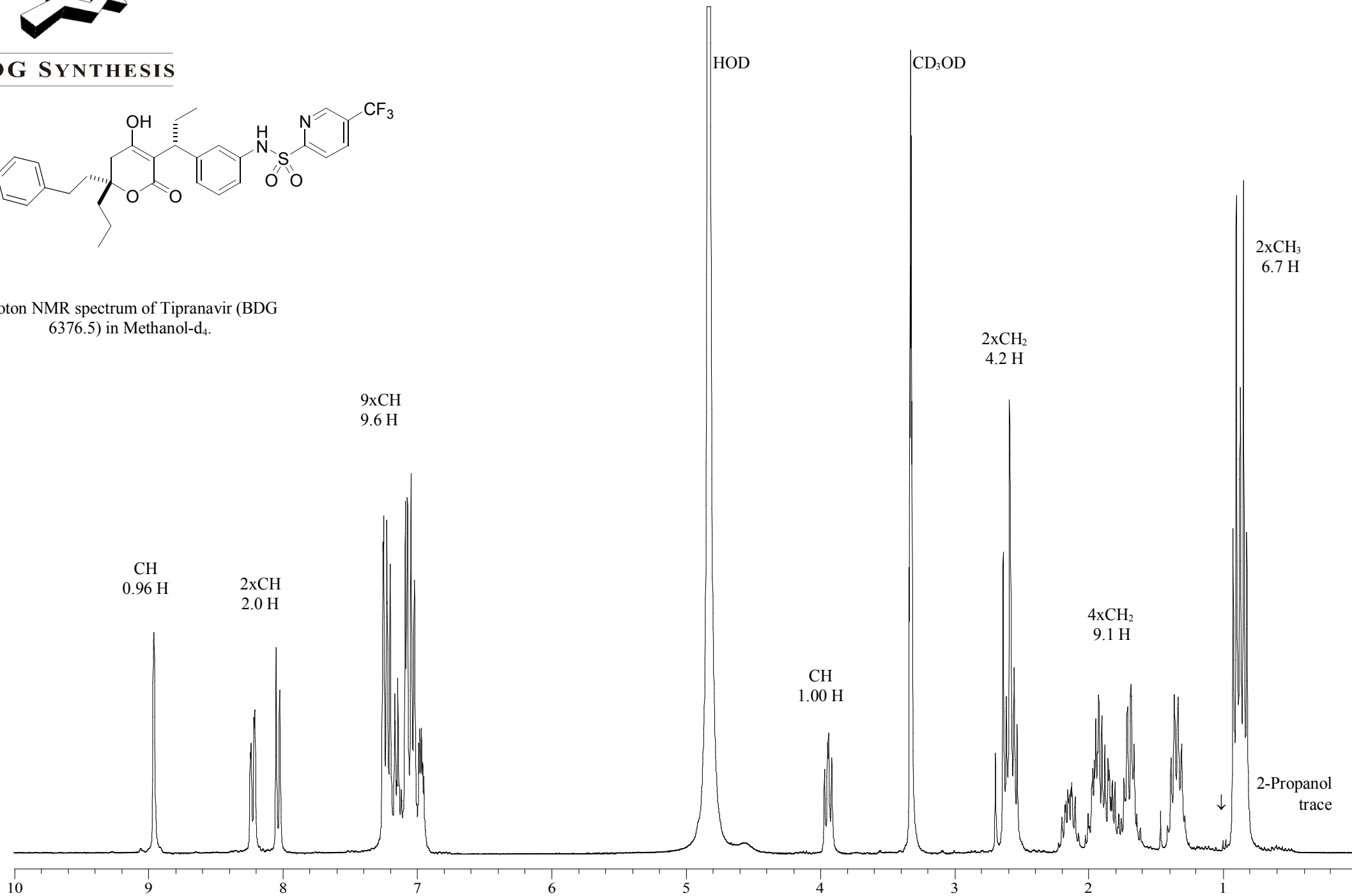
The expiry date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.

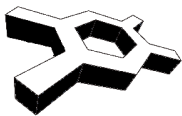


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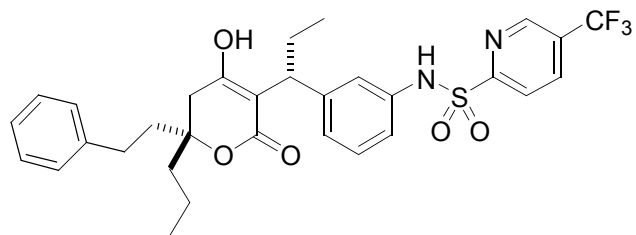


Proton NMR spectrum of Tipranavir (BDG 6376.5) in Methanol-d₄.





BDG SYNTHESIS



Carbon-13 NMR of Tipranavir (BDG 6376.5)
in Methanol-d₄.

