



BDG SYNTHESIS

Certificate of Analysis

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

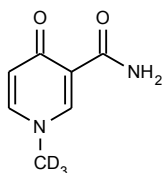
Neil Beare

Neil Beare, PhD, Director
2 June 2015

Name: *N*-Methyl-d₃-4-pyridone-3-carboxamide

CAS Number: 769-49-3 (unlabelled)

Structure:



Molecular Weight: C₇H₅D₃N₂O₂ = 155.17

Lot Number: BDG 7705.3

Appearance: White powder

Corrected Purity: 98.9 % (HPLC) - 1.2 % (water) = 97.8 %

Isotopic Purity: Under 0.5 % d₀

Re-test Date: 2 June 2020

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. The material is susceptible to static.

Identity and Purity

Proton NMR Spectrum

Identity: the signals are consistent with the proposed structure and in accord with literature where available.

Isotopic Labelling: signals at the sites of deuteration are absent, compared with the spectrum of unlabelled material, indicating clean deuteration.

Residual Solvents: no residual solvents are 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.

Isotopic Labelling: signals at the sites of deuteration have collapsed to small multiplets compared with the spectrum of unlabelled material, indicating clean deuteration.

High-resolution Mass Spectrum (ESI+)

Found m/z 178.0666. $C_7H_5D_3N_2NaO_2$ $[M+Na]^+$ requires m/z 178.0668. The deviation of 0.7 ppm is within normally accepted limits for the establishment of identity by HRMS. No signal for d_0 material was seen (detection limit about 0.5 %).

HPLC

A sharp, slightly tailing peak is observed (98.9 %). 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 53.42, H 3.35, D 4.02, N 17.75 %
$C_7H_5D_3N_2O_2 \cdot 0.1H_2O$	Requires:	C 53.56, H 3.34, D 3.85, N 17.85 %, H_2O 1.15 %
$C_7H_5D_3N_2O_2$	Requires:	C 54.18, H 3.25, D 3.89, N 18.05 %

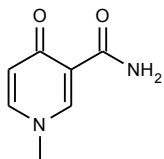
The elemental analyses fall somewhat outside those expected for anhydrous material; the presence of water is reasonably expected from the method of purification and/or the type of material, and the "best-fit" hydrated molecular formula is given. In the absence of a Karl-Fischer water analysis, we recommend that the "best-fit" water content be used when determining corrected purity.

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.

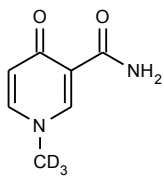
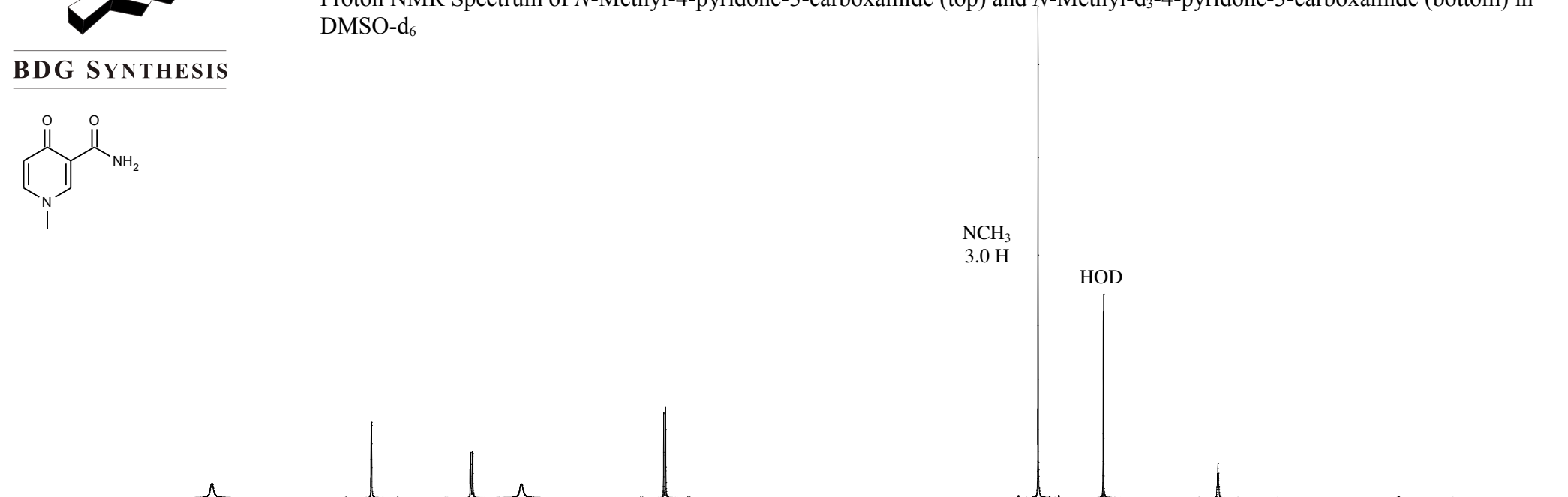
The re-test date is assigned from experience gained with the material in the laboratory and/or on storage. It is not possible to perform formal storage studies because of the small amount of material available.



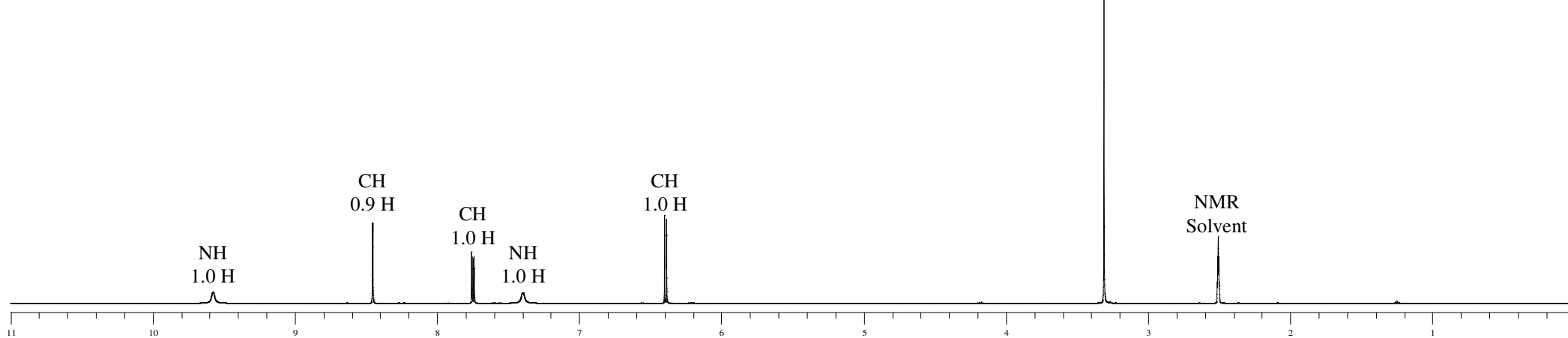
BDG SYNTHESIS



Proton NMR Spectrum of *N*-Methyl-4-pyridone-3-carboxamide (top) and *N*-Methyl-*d*₃-4-pyridone-3-carboxamide (bottom) in DMSO-*d*₆

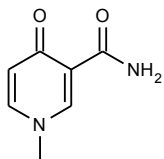


Lot Number: BDG 7705.3

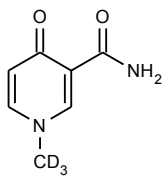
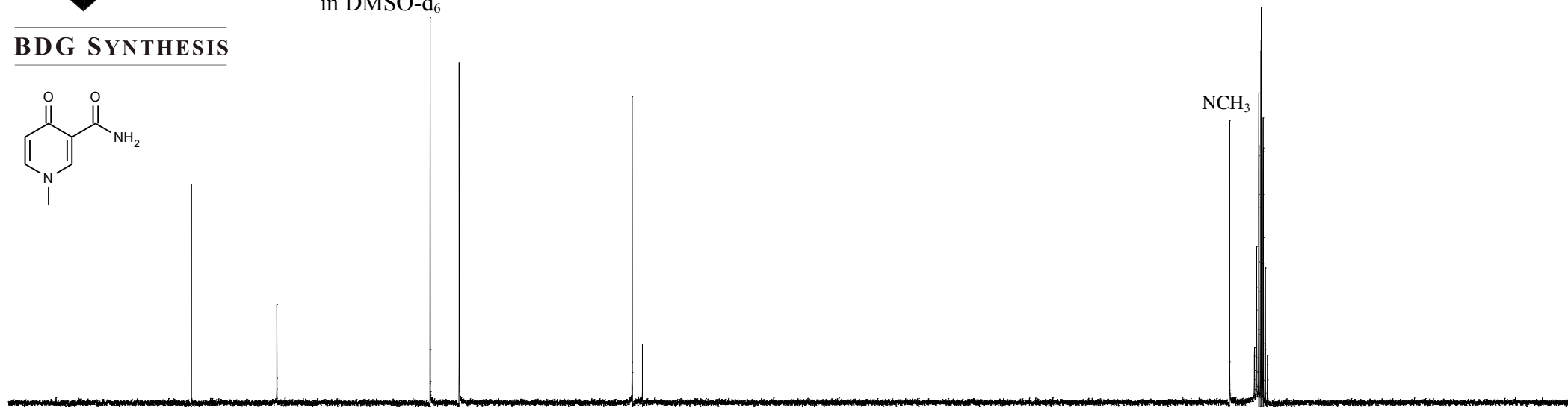




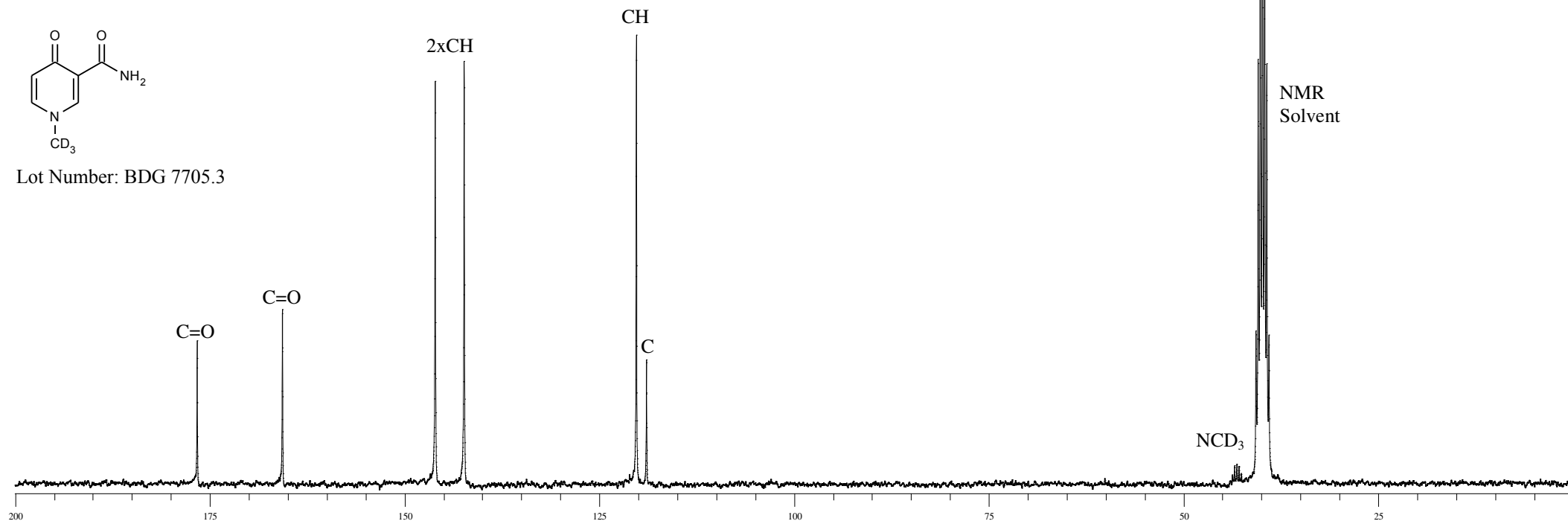
BDG SYNTHESIS



Carbon-13 NMR Spectrum of *N*-Methyl-4-pyridone-3-carboxamide (top) and *N*-Methyl-*d*₃-4-pyridone-3-carboxamide (bottom) in DMSO-*d*₆



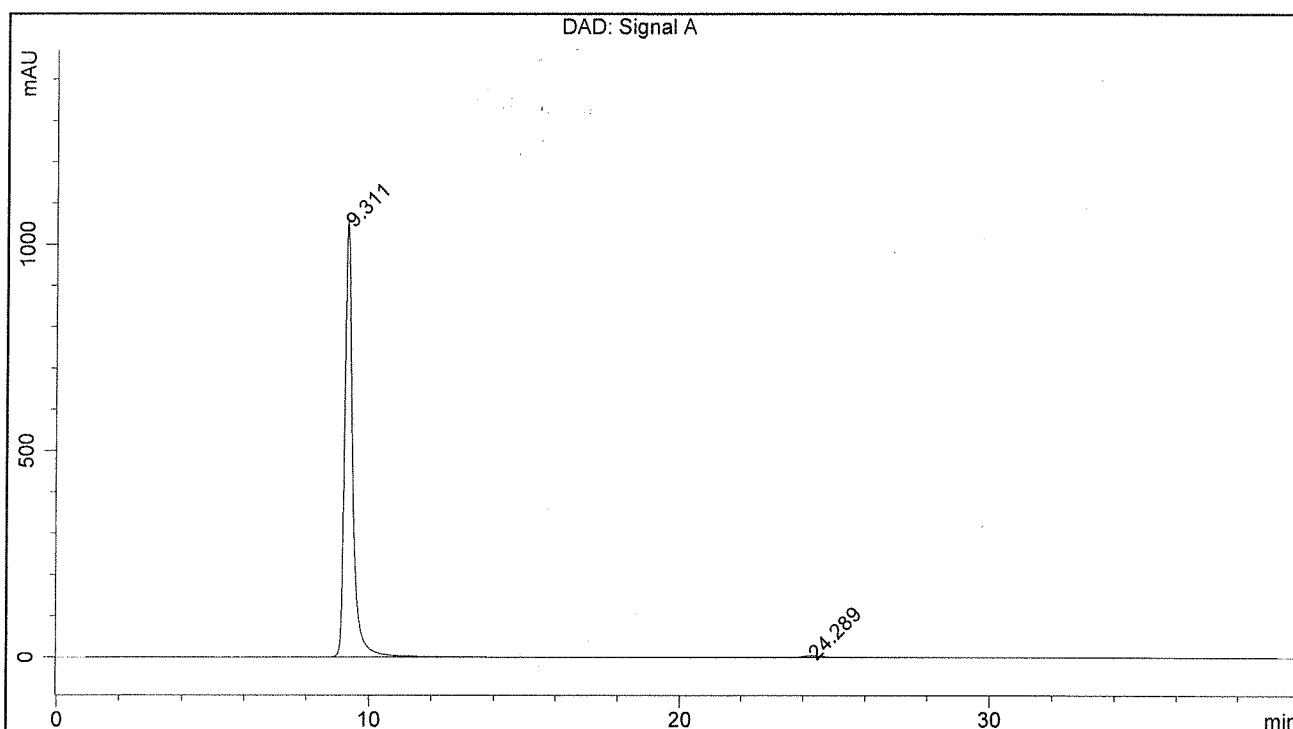
Lot Number: BDG 7705.3



BDG - Analysis of N-Methyl-d3-4-pyridone-3-carboxamide

Column : Phenomenex Luna C18(2) 5um 250 x 4.6 mm
 Guard : Phenomenex Security Guard C18 RP 4 x 3 mm
 Mobile Phase : 96:4:0.05 Water : Acetonitrile : Acetic Acid
 Flow Rate : 1.0 mL/min
 Sample Solvent : Mobile Phase
 Column Temperature : 20C
 Injection Volume : 10 uL
 Detection : UV at 254 nm

Sample Name	BDG 7705.3	Instrument	AnalyticalLC01
Acquisition	02/06/2015, 21:49:45	Method (rev.)	LC10284b (9)
Sequence	BDG_02Jun2015b - Reprocessed	Vial Position	31
Operator	solvation010\cerityadmin	Injection	2 of 2



Area Percent Report

Peak#	RT	Peak Height	Peak Area	Width	Area %
1	9.31 min	1051.4606	19007.6937	0.2749 min	98.874 %
2	24.29 min	4.3138	216.4731	0.7294 min	1.126 %