BDG SYNTHESIS

Certificate of Analysis

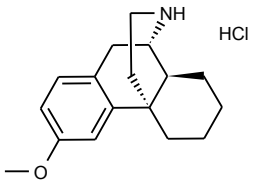
BDG Synthesis certifies that this reference material meets or exceeds the specifications stated herein.

Neil Beare, PhD, Director
11 April 2024

Name: **3-Methoxymorphinan HCl**

CAS Number: 1087-69-0

Structure:



Molecular Weight: $C_{17}H_{23}NO \cdot HCl = 293.83$

Lot Number: BDG 18266

Appearance: White, crystalline solid

Corrected Purity: 99.8 % (HPLC) - 0.5 % (ethyl acetate) - 0.2 % (2-propanol) = 99.1 %

Re-test Date: 11 April 2025

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.

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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: small amounts of 2-propanol (0.2 % *w/w*) and ethyl acetate (0.5 % *w/w*) 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.

High-resolution Mass Spectrum (TOF MS ES+)

Found *m/z* 258.1858. $C_{17}H_{24}NO$ $[M+H]^+$ requires *m/z* 258.1858. The deviation of 0.0 ppm is within normally accepted limits for the establishment of identity by HRMS.

HPLC

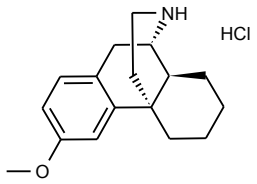
A somewhat broadened, tailing peak is observed (99.8 %). Note: in the absence of reference materials for preparing calibration curves, it is assumed that all peaks have the same detector response. Where possible, the conditions of analysis follow a pharmacopeial or literature method, or have been adapted from same.

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 3-Methoxymorphinan HCl in DMSO-d_6



Lot Number: BDG 18266

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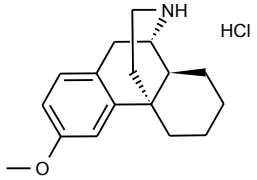
Carbon-13 NMR Spectrum of 3-Methoxymorphinan HCl in DMSO-d_6



Lot Number: BDG 18266

Analysis of BDG 18266: 3-Methoxymorphinan HCl

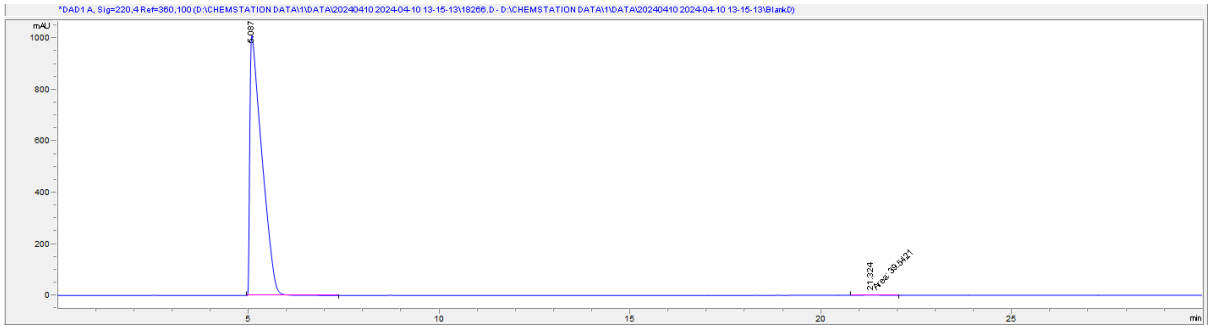
Date: 11/04/2024

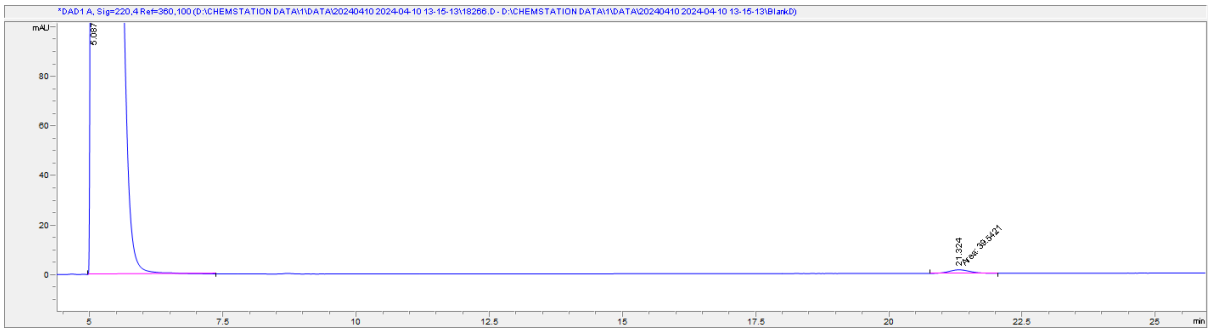
Technician: CJC

Column: Phenomenex Luna Phenyl Hexyl (2) 5 µm 250 x 4.6 mm
Guard: Phenomenex Security Guard Phenyl RP 4 x 3 mm
Mobile Phase: 50:20:30 10 mM KH_2PO_4 pH = 3 : MeOH : ACN
Gradient: Isocratic
Flow Rate: 1.0 mL/min
Sample solvent: Mobile Phase
Sample Concentration: ~1 mg/mL
Column Temperature: 40°C
Injection Volume: 10 µL
Detection: UV 220 nm

Notes:

Chromatogram of sample at 220 nm with blank subtracted





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2	21.324	MM	39.5	1.4	0.4596	0.178	0.869