^{1}H NMR Analysis Report

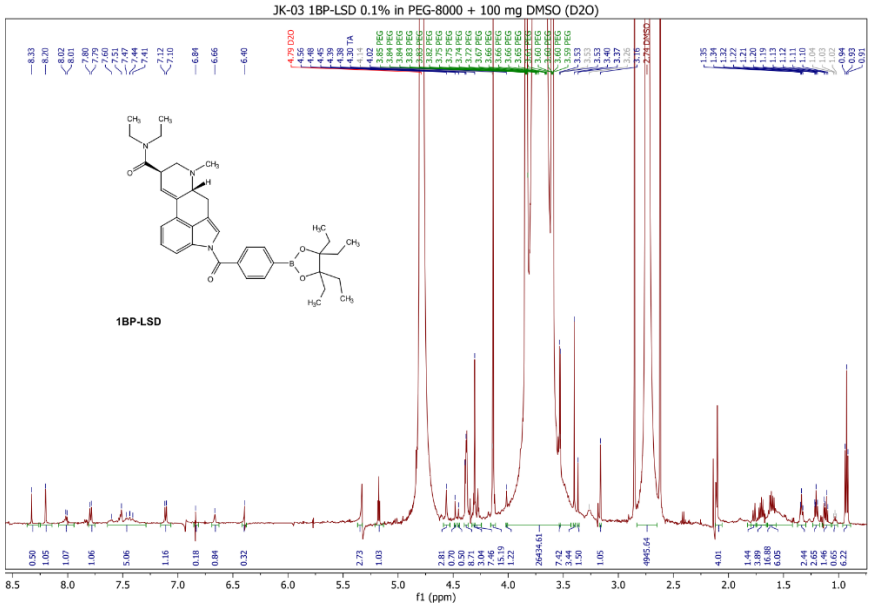
Sample name: JK-03 5-10-2026

Spectrometer: 600 Oxford (600 MHz) **Solvent**: D_2O

Expected compound: 1BP-LSD hemitartrate ~0.1% in PEG-8000

Identified compound: 1BP-LSD hemitartrate ~0.1% in PEG-8000

(6*a*R,9R)-*N*,*N*-Diethyl-7-methyl-4-(4-(4,4,5,5-tetraethyl-1,3,2-dioxaborolan-2-yl)benzoyl)-4,6,6a,7,8,9-hexahydroindolo[4,3-fg]quinoline-9-carboxamide





Summarized Results

The analysis was performed to determine whether the PEG-8000/DMSO formulation successfully incorporated 1BP-LSD hemitartrate and enabled its transfer into aqueous solution at approximately the intended concentration. This was particularly relevant because the active compound had previously been found to be very poorly soluble in water and alcohols. Despite the very low concentration of active compound relative to the PEG matrix, 1BP-LSD signals were clearly detectable in the ^{1}H NMR spectrum of the D_2O solution. Their estimated concentration of approximately 0.1% was close to the expected value, supporting successful transfer of the active compound into aqueous solution at approximately the intended concentration.

No signals attributable to hydrolysis products of 1BP-LSD were detected. An additional UV fluorescence test (see picture above) also provided no indication of significant formation of LSD, which would be expected to exhibit strong blue fluorescence. Taken together, the results support both successful aqueous delivery of 1BP-LSD from the PEG/DMSO formulation and its chemical stability under the conditions investigated.

Detailed Analysis

The product 1BP-LSD hemitartrate had previously been analyzed by ^{1}H NMR in DMSO-d_6. During sample preparation, it was observed to be very poorly soluble in protic solvents such as water and alcohols. Since sufficient solubility in these solvents was desired for the product's intended applications, a formulation containing 5 mg 1BP-LSD hemitartrate, 5 mL molten PEG-8000 (≈ 5.432 g), and two drops of DMSO (likely ~100 mg) was conceived as a carrier intended to facilitate incorporation and subsequent dissolution of the active compound in water or alcohol. The goal of the present ^{1}H NMR analysis was therefore to determine whether the active compound had been successfully incorporated into the PEG/DMSO formulation, transferred into aqueous solution at approximately the intended concentration, and remained hydrolytically stable under the conditions used.

Because the ratio of active compound to matrix was approximately 1:1000, the 1BP-LSD signals were expected to be very close to the detection limit, even at the relatively high concentration of the formulation. The number of scans was therefore increased eight-fold, from the 16 scans typically used for routine analyses to 128 scans.

At first inspection, the acquired spectrum showed mainly the water signal at 4.79 ppm, a large singlet around 3.7 ppm assigned to the PEG protons, and a surprisingly large DMSO signal. Instead of the expected PEG-to-DMSO ratio of approximately 50:1, the observed ratio was closer to 9:1.

The most likely explanation is an inhomogeneous distribution of DMSO within the formulation caused by diffusion away from the centre of the wax during solidification. PEG-8000 undergoes substantial crystallization upon cooling from the molten state, and the growing crystalline PEG domains are expected to incorporate DMSO poorly. This can result in segregation of DMSO into the remaining less-crystalline regions. Some loss of DMSO through evaporation may further contribute to its redistribution toward air-exposed parts of the material.

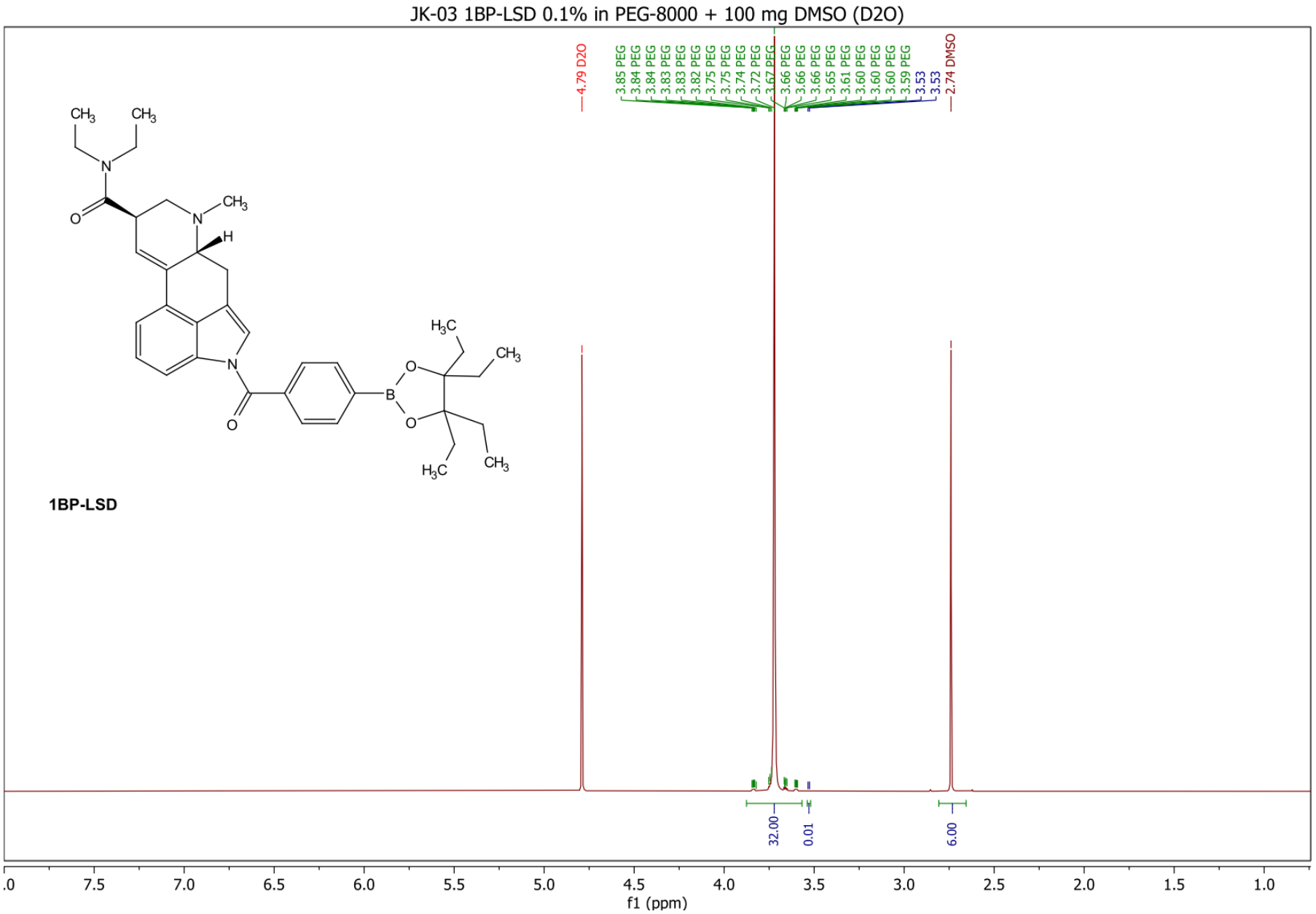
Under strong magnification, signals attributable to 1BP-LSD became visible. As these signals were close to the detection limit, their quantitative evaluation was affected by baseline shape, phase-correction-related distortions, and overlap with signals from minor impurities. Nevertheless, the resulting estimate of approximately 0.1% 1BP-LSD was close to the expected concentration.

Importantly, the NMR sample was prepared in D_2O, providing an aqueous environment closely representative of water. The observation of recognizable 1BP-LSD signals at approximately the expected concentration therefore indicates that the PEG/DMSO formulation successfully enabled transfer of the active compound into aqueous solution. This also argues against the compound being present predominantly as undissolved particles or strongly associated PEG/1BP-LSD aggregates: such species would be expected to exhibit substantially broadened resonances due to restricted molecular mobility and could consequently be largely undetectable by ^1H NMR. The observed signals are therefore consistent with the active compound being present predominantly, if not essentially completely, as NMR-visible dissolved species in the aqueous sample.

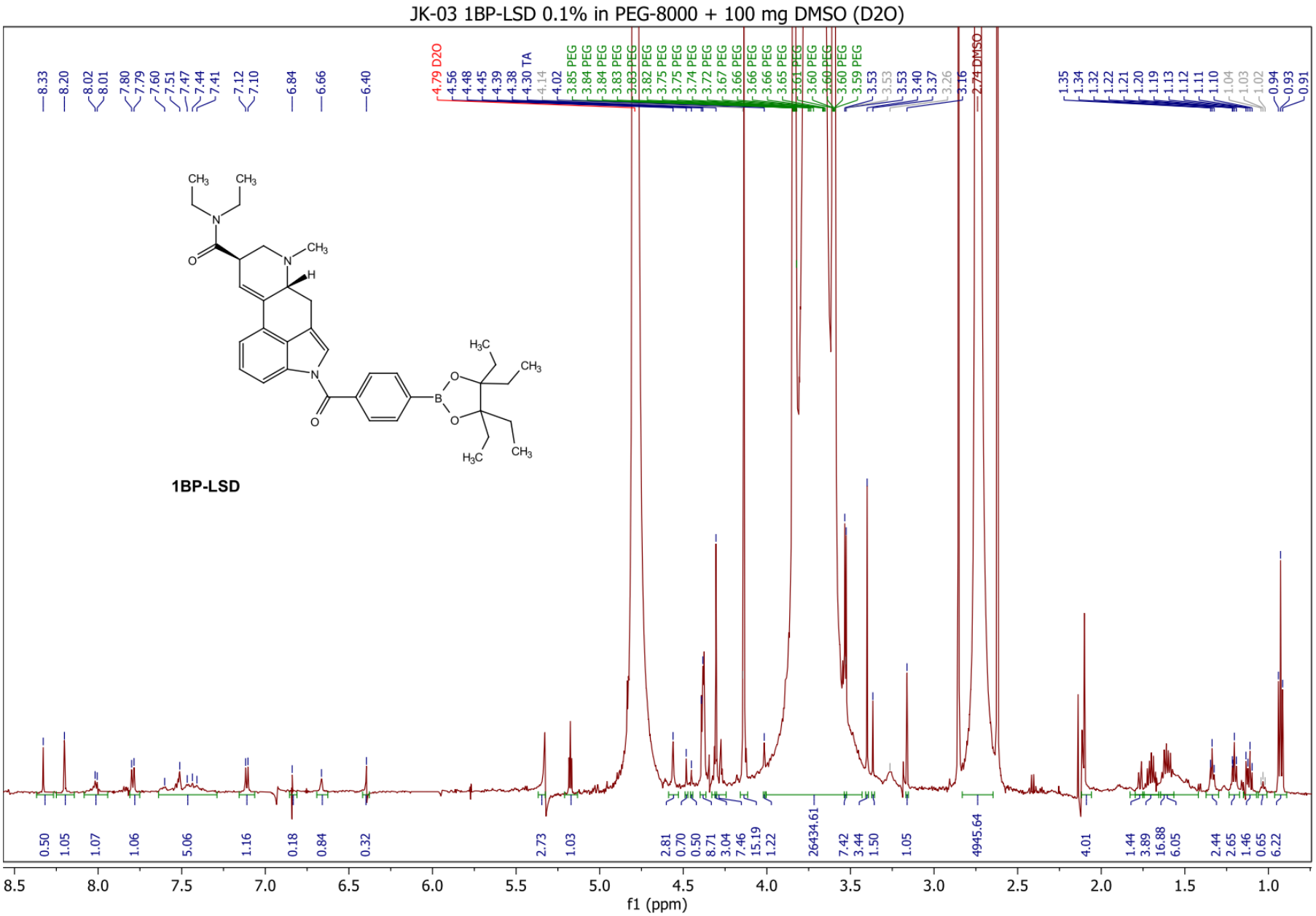
This result demonstrates successful aqueous delivery under the conditions tested. It does not, by itself, establish identical solubility behavior in other solvents such as alcohols, although it demonstrates that the formulation can overcome the poor direct water solubility of the starting material under the conditions investigated.

As an additional test for possible trace formation of LSD through hydrolysis, the sample solution was examined under UV light at 366 nm. LSD is known to exhibit strong deep-blue fluorescence in solution, which was not observed. Instead, only weak greenish fluorescence was observed (see picture on page 1). This fluorescence may be intrinsic to 1BP-LSD or could be associated with degradation products of lysergamides, such as Lumi-LSD. The observation therefore does not provide evidence for significant LSD formation.

^{1}H NMR: full non-empty spectrum



^{1}H NMR: full non-empty spectrum, strong zoom



^{1}H NMR: cut and zoomed spectrum

