Triclinic Labs

Scientific Report

^{1}H NMR Analyses of RD286 and RD500 Treated Samples of Fentanyl

Aseptic Health, LLC

Prepared for Autumn Ryan

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This is a non-cGMP report

Introduction

Aseptic Health, LLC is developing products to neutralize potent compounds such as fentanyl. Two products are entitled RD286 and RD500, otherwise known as Sanitize IT. A protocol was developed to test the effectiveness of the products.

For the effectiveness testing, separate samples of fentanyl (~1 mg) were treated with each RD286 and RD500 at room temperature without sonication or vortexing to mimic the spraying of the products over fentanyl powder spilled on a surface (see the experimental section). Aliquots of the treated samples were analyzed by ^{1}H nuclear magnetic resonance (NMR) to determine whether the treatment of each product was effective in degrading fentanyl. Reference ^{1}H NMR spectra of fentanyl and the two products were acquired for comparison. This report summarizes the results of the investigation. **Table 1** summarizes the samples received, prepared, and the corresponding NMR files.

Table 1. Summary of Samples and Data Acquired.

Sample Description	Lot Number of Triclinic Labs Identifier	NMR Filename
RD286	09012021Lab1 Sanitize IT TCL16043	N/A
RD500	03212022LAB1 TCL16898	N/A
Fentanyl reference standard	TCL16846	NMR1-1968
RD286 treated fentanyl sample (one-minute incubation)	1129-47-1	NMR1-2267
RD286 treated fentanyl sample (ten-minute incubation)[1]	1129-20-1	NMR1-1970
RD500 treated fentanyl sample (ten-minute incubation)	1129-47-2	NMR1-2266
Intact RD286	1129-33-1	NMR1-2103
Intact RD500	1129-47-3	NMR1-2265

Results

The ^{1}H NMR spectra of fentanyl treated with RD286 (about 1 mg of fentanyl in 1 mL of RD286) are compared to reference spectra (RD286 and fentanyl) in **Figures 1 - 3**. The ^{1}H NMR spectra of the RD286 treated fentanyl samples exhibit distinctive peaks at ~ 7.15, ~7.08, ~2.40, ~2.05, and ~1.85 ppm when compared to the spectrum of RD286 (**Figures 2 - 3**). These distinctive signals in the RD286 treated sample are likely resulted from a trace amount of degraded fentanyl in the solution, which displayed shifted peak positions compared to those from the fentanyl reference standard. This suggests that the fentanyl was degraded by treatment with RD286. However, not all of

[1] Triclinic Labs non-GMP report for Aseptic Health, LLC, "^{1}H NMR Analyses of RD286 and RD520 Treated Samples of Fentanyl", R2022021.02, dated March 10, 2022.

the fentanyl dissolved in RD286 at 1 mg: 1 mL ratio, and therefore the total effectiveness at 1 mg:1 mL ratio has not been confirmed.

The ^{1}H NMR spectrum of fentanyl treated RD500 (about 1 mg of fentanyl in 1 mL of RD500) is compared to reference spectra (RD500 and fentanyl) in **Figures 4 - 6**. However, no ^{1}H NMR signals corresponding to degraded fentanyl were detected in the fentanyl treated RD500, indicating that fentanyl was not degraded by RD500 after a 10-minute incubation time. It was noted that undissolved fentanyl remained after the 10-minute incubation.

Conclusion

The ^{1}H NMR analysis on a fentanyl reference standard and a series of RD solution treated fentanyl samples showed an evidence that fentanyl was degraded by treatment of RD286 after one- and ten-minute incubations, but RD500 did not show any evidence of fentanyl degradation after a ten-minute incubation time.

Experimental

^{1}H NMR Spectroscopy

The reference standards for fentanyl (Cayman Chemical Co.) were prepared for ^{1}H NMR analysis by dissolving 0.674 mg of material into 0.75 mL of D_2O**Error! Bookmark not defined.**. The intact RD286 and RD500 samples were prepared by adding each sample directly into coaxial tubes. The RD286 and RD500 treated samples of fentanyl were initially exposed by separately weighing 0.924 and 0.999 mg of fentanyl into a container. To the separate containers, 1 mL of RD286 and RD500 were added and allowed to incubate at room temperature for one minute and ten minutes, respectively. The fentanyl was not fully dissolved upon treatment by RD286 and RD500. The supernatant was then added to a 5-mm NMR tube and analyzed.

The ^{1}H NMR spectra were acquired on a Bruker NEO 400 MHz (9.4 T) spectrometer using TopSpin v4.1.1 software. Each spectrum was processed using TopSpin v4.1.1 and referenced to the chemical shift of the residual D_2O (4.79 ppm) peak. Detailed acquisition parameters are listed in **Table 2**.

Table 2. NMR Acquisition Parameters.

Parameter Name	Parameter Value
Transmitter Frequency	400.15 MHz
Acquisition Time	4 - 6 sec
Spectral Width	6250 Hz
Number of Scans	64 - 256
Sequence	ZG
P1 (pulse width)	12.76 µsec
PLW1 (pulse power)	13.68 W
D1 (relaxation delay)	4 sec
Line Broadening	0.5 Hz

Figure 1. Comparative analysis of the ^{1}H NMR spectra of the fentanyl reference standard, the intact RD286, and the RD286 treated fentanyl samples.

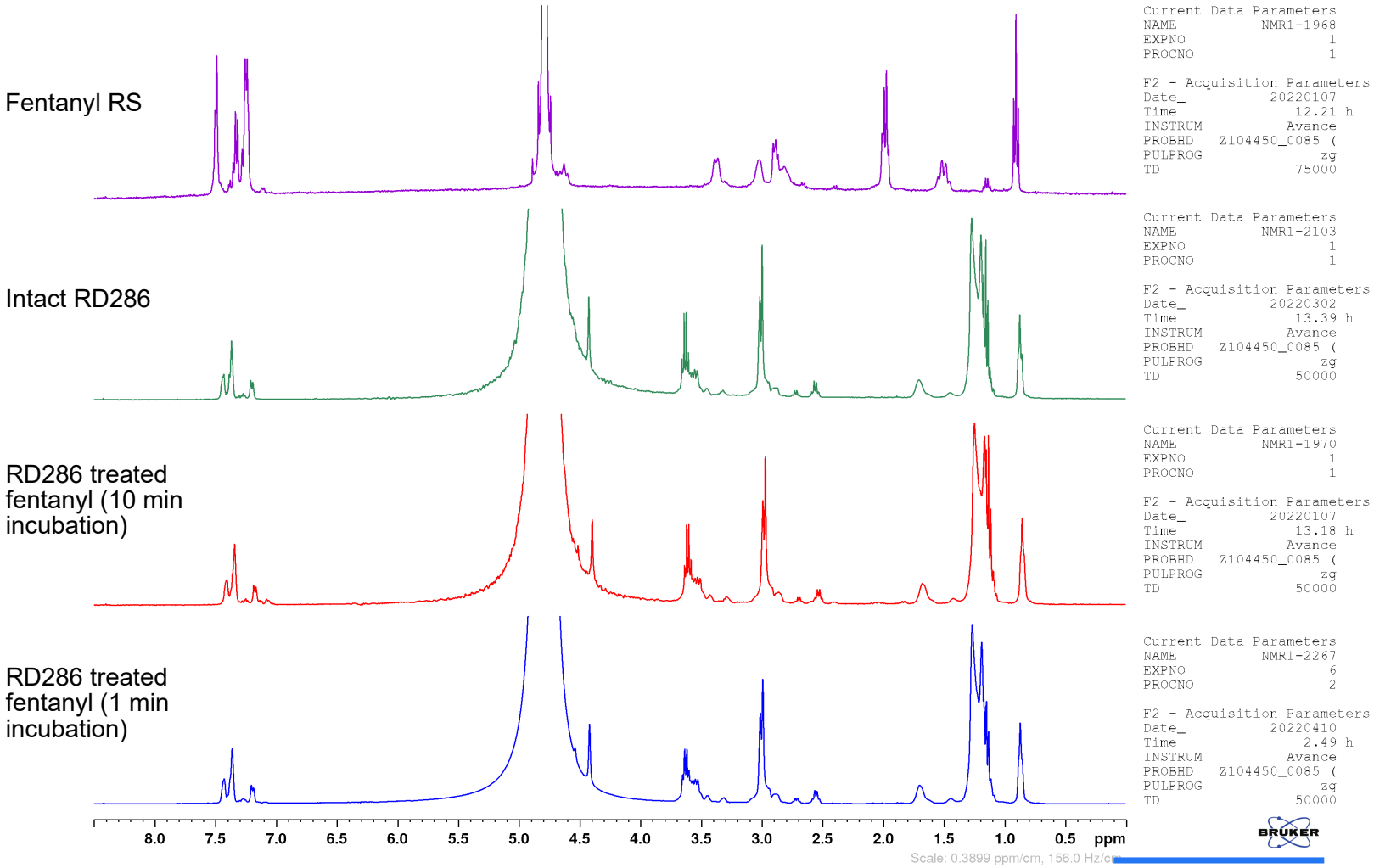

Figure 2. Comparative analysis of the ^{1}H NMR spectra (7.8 - 6.7 ppm) of the fentanyl reference standard, the intact RD286, and the RD286 treated fentanyl samples.

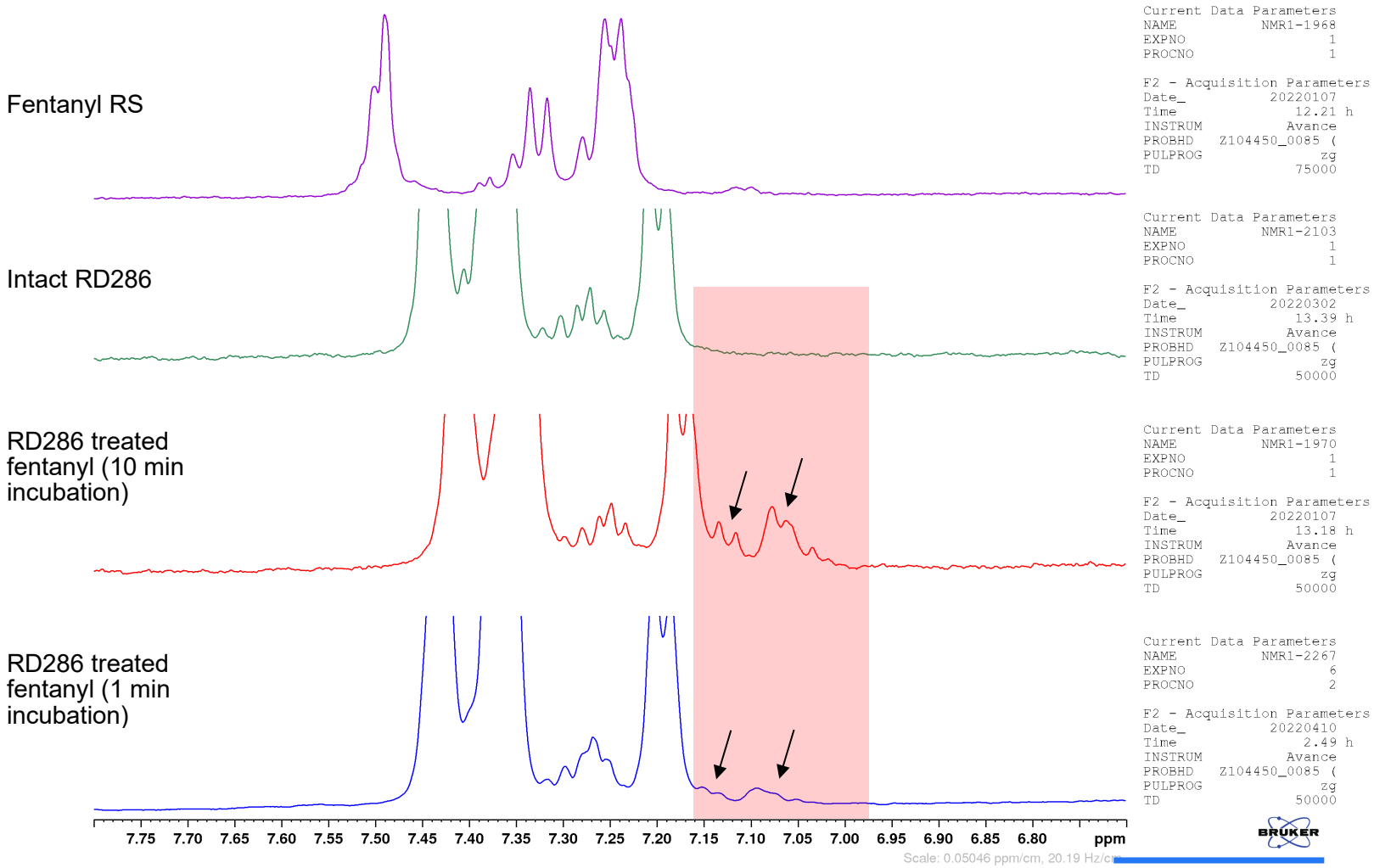

Figure 3. Comparative analysis of the ^{1}H NMR spectra (3.2 – 1.2 ppm) of the fentanyl reference standard, the intact RD286, and the RD286 treated fentanyl samples.

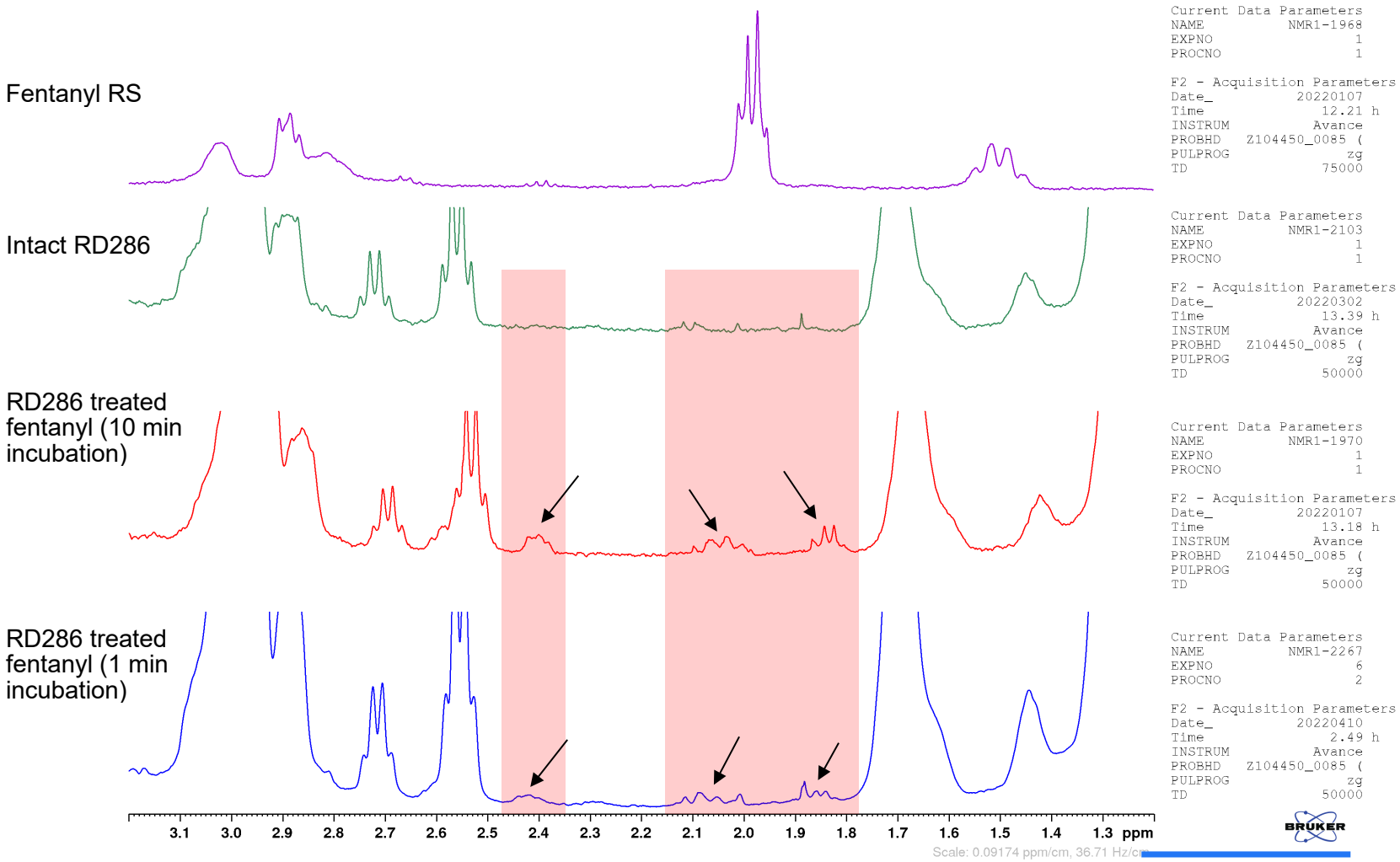

Figure 4. Comparative analysis of the ^{1}H NMR spectra of the fentanyl reference standard, the intact RD500, and the RD500 treated fentanyl.

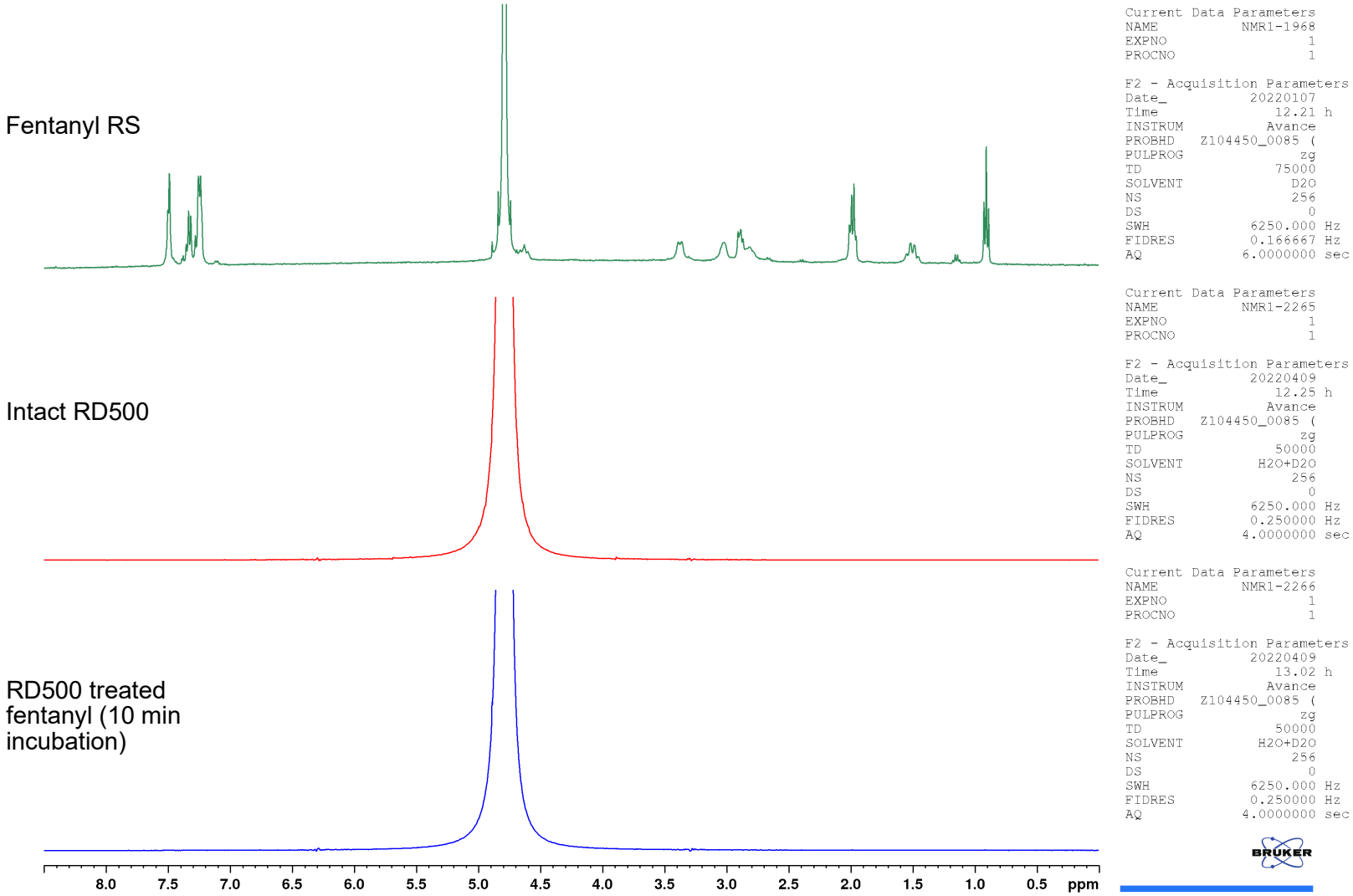

Figure 5. Comparative analysis of the ^{1}H NMR spectra (7.8 - 6.7 ppm) of the fentanyl reference standard, the intact RD500, and the RD500 treated fentanyl.

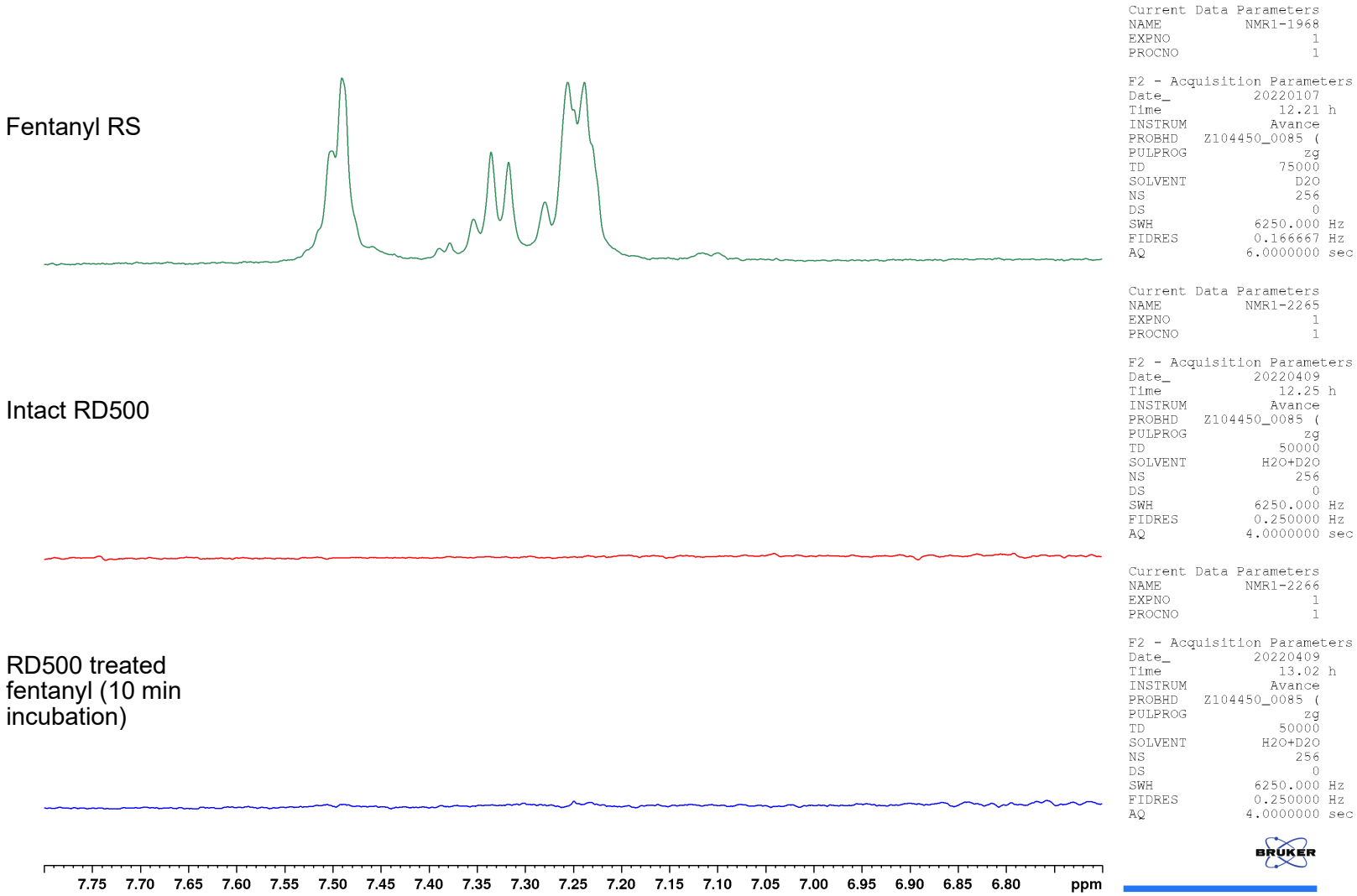

Figure 6. Comparative analysis of the ^{1}H NMR spectra (3.2 – 1.2 ppm) of the fentanyl reference standard, the intact RD500, and the RD500 treated fentanyl.