IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE PATENT TRIAL AND APPEAL BOARD

HARVEST TRADING GROUP, INC.,
Petition

v.

VIREO SYSTEMS, INC. AND
UNEMED CORPORATION,

Patent Owners

U.S. PATENT NO. 8,354,450
IPR 2016-00945

U.S. PATENT NO. 8,962,685
IPR 2016-00947

DECLARATION OF JASON WIGGERS

Exhibit 2012

DECLARATION

I, Jason Wiggers, hereby declare and say:

1. I am over the age of eighteen (18) and have personal knowledge of the matters set forth herein.

2. I am the Operations Manager of Vireo Systems, Inc. ("Vireo"). I have held this position since November 2007. My job responsibilities include overseeing of operations, which include but are not limited to research and development, manufacturing, and product development. I have 9 years of laboratory and commercial scale supplement and drug manufacturing experience. I have a bachelor's degree in Biology with a minor in Chemistry at David Lipscomb University from 2004.

3. Vireo is one of the co-owners of U.S. Patent Nos. 8,354,450 (the " '450 Patent"), 8,962,685 (the " '685 Patent"), 8,026,385 (the " '385 Patent"), and 7,608,641 (the " '641 Patent).

4. I understand that the '450 Patent is subject to a petition for inter partes review in Proceeding No. IPR2016-00945, and that the '685 Patent is subject to a petition for inter partes review in Proceeding No. IPR2016-00947. I further understand that the '450 Patent is identified as a continuation in part of the '385 Patent, and the '385 Patent is a continuation of the '641. I further understand and that the '685 Patent is a divisional of the '450 Patent.

5. I was requested by Vireo's counsel to prepare samples of creatine hydrochloride according to the methods described in the '641 Patent. I have reviewed the '641 Patent and am familiar with the procedures that it describes.

6. To conduct the procedures, I supervised the work performed by Vireo-trained personnel. I specified the procedures used and am personally familiar with the testing, and I can testify to such if called to do so. The procedures performed under my supervision for purposes of the requested creatine hydrochloride preparation are attached hereto as **Exhibit A**.

Method 1: Acetyl Chloride Mixture.

7. The first procedure for preparing creatine hydrochloride is described in lines 4-14 of column 3 of the '641 Patent, using the following steps. First, creatine monohydrate is blended with acetyl chloride in a vessel. Ethanol is added to the vessel to dissolve the blended mixture. The temperature of the solution is raised to be between 24° C and 50° C. The '641 Patent specifically discloses that 25° C is preferred. At these conditions, creatine hydrochloride salt precipitates in a granular form. These granules may then be collected and packaged for consumption.

8. In order to conduct this procedure, I directed that 8.89 grams (0.059 mol) of creatine monohydrate be weighed out and placed into an Erlenmeyer flask. 6 ml (0.084 mol) of acetyl chloride were measured in a 10 ml graduated cylinder.

These weights equal the molar ratio of 1:1.4 described in the ‘641 Patent at column 2, lines 53-56. In addition, 10 ml of SDA-35A ethanol were measured out in a 10 ml graduated cylinder for dissolving, as instructed in the ‘641 Patent at column 2, lines 60-61. **See Exhibit A, at p. 3.**

9. The reaction is exothermic. The ‘641 Patent, at column 2, lines 63-67, instructs that temperature should be maintained below 50° C to reduce the amount of creatinine hydrochloride and creatine ethyl ester hydrochloride contaminants produced by the reaction. Therefore, in order to stay within the instructed temperature range and minimize the impact of the heat energy associated with the reaction, I directed that the vessel containing the reactants be placed in an ice bath to manage or regulate the temperature of the reaction. This is a common and standard laboratory technique. The initial temperature was measured to be 14° C. Next, the acetyl chloride was poured into the flask and stirred to ensure a complete mixture. Due to the exothermic reaction, the temperature was then measured to be 41° C, that is, within the directed temperature range of the ‘641 Patent. The ethanol was then added to dissolve the mixture. As the reaction continued, a slurry formed from the resulting precipitated salt. **See Exhibit A, at p. 3.**

10. Once the temperature reached a steady 25° C, the slurry was emptied onto a sterile filter paper in accordance with standard procedures for isolating a precipitated compound. Additional ethanol was used to wash the precipitate. The

excess liquid was removed, and then the entire composition was set to air dry at room temperature for 3 hours. The final sample product measured 11 grams. **See Exhibit A, at p. 3.**

11. I directed that 100 mg of the dried sample be isolated in sterile packaging for pick up and transport by Samir Saleh for purity testing, and that 5 g of the dried sample be used for testing solubility as described further below.

Method 2: HCl Gas Infusion

12. The second procedure for preparing creatine hydrochloride is described in lines 15-28 of column 3 of the '641 Patent, using the following steps. A diethyl ether solvent is provided, into which gaseous hydrochloride is bubbled. Creatine monohydrate is then stirred into the diethyl ether solvent. This results in a creatine hydrochloride precipitate. The precipitate is filtered and washed using fresh diethyl ether. The precipitate is then dried to isolate the creatine hydrochloride and collect it for consumption. The '641 Patent teaches that there can be a range of concentrations of gaseous hydrochloride, so long as it exceeds the molar equivalent of the creatine monohydrate to be added to the solvent.

13. In order to conduct this procedure, I directed that 5 ml of diethyl ether be poured into an Erlenmeyer flask, and 4.45 g of creatine monohydrate be measured out. Tubing for bubbling in gaseous hydrochloride was threaded into the flask and submerged in the ether. The entire apparatus was placed in the fume

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