

THIRD DECLARATION OF LEONARD J. CHYALL, PH.D.

I, the undersigned, Dr. Leonard J. Chyall, U.S. Passport No. 432624896, with a business address of Chyall Pharmaceutical Consulting LLC, 3000 Kent Avenue, Suite D1-105, West Lafayette, Indiana 47906, USA, having been warned that I must state the truth and that I shall be liable to the penalties prescribed by law should I fail to do so, hereby declare in writing as follows:

1. I am the same Leonard J. Chyall who submitted a declaration dated August 3, 2010 (the "First Chyall Declaration") and a declaration dated March 7, 2012 (the "Second Chyall Declaration"), in support of the position of Teva Pharmaceutical Industries Ltd. ("Teva") in the proceedings before the Honorable Deputy Registrar of Patents regarding Israel Patent Application No. 172563, filed by Merck & Co. Inc., U.S.A ("Merck").
2. This declaration was prepared in response to the Affidavit of Prof. Jerry L. Atwood submitted on behalf of Merck regarding an experiment that Prof. Atwood conducted in August 2012 (the "Second Atwood Affidavit"). I was advised by Teva's counsel that Merck does not rely on paragraphs 3, 6, 7, 8 and 9 of the Second Atwood Affidavit.
3. The fact that I have not commented on any particular point in the Second Atwood Affidavit does not mean that I accept or agree with that point. There is nothing in the Second Atwood Affidavit that causes me to change the views that I expressed in the First and Second Chyall Declarations.

byproducts and/or unreacted sitagliptin base dissolved in the reaction solvent and trapped inside the recovered solids, would make it more difficult to remove those now solidified impurities, byproducts and/or unreacted sitagliptin base through subsequent washing.

- F. Prof. Atwood states that he used 3 x 3 mL of isopropanol solvent to wash and filter the solids that he recovered. *See* Atwood Exhibit HH. This is a very small amount of solvent for washing Prof. Atwood's recovered solids when using a Büchner funnel with a diameter of approximately 7 cm. Using too little solvent for washing would result in ineffective removal of impurities, byproducts and unreacted starting materials.
9. Prof. Atwood's laboratory notebook, Exhibit HH of his Second Affidavit, by itself, does not provide enough detail to determine whether one or more of the above steps rendered Prof. Atwood's washing steps inadequate. Therefore, unlike my criticism of Prof. Atwood's previous experiments – which did not include any filtration and washing and therefore did not require that I conduct experiments to conclude that Prof. Atwood's assertions regarding the solids that he recovered were unreliable and without scientific merit – I could only prove the misleading nature of Prof. Atwood's New Experiment, which included a "filtration and washing" step, by conducting experiments.

My Experiments Prove That Prof. Atwood's "Filtration And Washing" Was Ineffective

10. I received from Teva a sample container labeled lot no. D6655070112, which I understand to contain Sitagliptin Free Base. The sample was assigned LIMS No. 308390, and I characterized the material using XPRD (see Exhibit A). The XRPD

pattern obtained for the material confirmed that the material was crystalline sitagliptin base as disclosed in PCT Publication No. WO 2009/070314 A2.

11. I first replicated as closely as possible the New Experiment described in the Second Atwood Affidavit. I conducted this replication to ensure that it was possible, based on the procedure described in Prof. Atwood's laboratory notebook, Exhibit HH, to obtain crystalline solids with the characteristic XRPD pattern of Prof. Atwood's alleged "2:1 salt".
12. However, my solution did not solidify overnight like Prof. Atwood's solution. In order to precipitate the reaction product, I cooled the reaction mixture using an ice bath with stirring. I filtered and washed my recovered solids using the same "filtration and washing" protocol that Prof. Atwood used to filter and wash his recovered solids. I analyzed the recovered solids by XRPD. The recovered solids had the same characteristic XRPD pattern as that of Prof. Atwood's solids (Exhibit B), which demonstrates that my use of an ice bath to precipitate solids did not affect the final product and was not a material deviation from Prof. Atwood's procedure. I refer to the solids that I recovered from this experiment as "the Replicated Atwood Wash Solids". A detailed description of how I obtained the Replicated Atwood Wash Solids is set forth in my laboratory notebooks, (Exhibit C).
13. I next conducted an experiment to see the effect of progressively more thorough washings than employed by Prof. Atwood's "filtration and washing" protocol. To do so, I again replicated Prof. Atwood's New Experiment, except at double the scale, so as to obtain a sufficient amount of material. This time the solution solidified overnight, like Prof. Atwood's solution. I analyzed the recovered solids by XRPD. The solids recovered on the Büchner funnel after washing once with isopropanol had