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IN THE COURT OF ARBITRATION FOR SPORT

IN THE MATTER OF FLOYD LANDIS,

CAS 2007/A/1394

FLOYD LANDIS V. UNITED STATES ANTI-DOPING AGENCY

BRIEF SUBMITTED BY FLOYD LANDIS

I.

INTRODUCTION

This appeal comes at a troubled time for professional cycling. Efforts to combat doping in cycling are being brought with unprecedented vigor. Appellant Floyd Landis fully supports these efforts and condemns the impact of doping on professional cycling. However, to wrongly strip a champion of his victory due to a flawed test result is much worse than to have an athlete cheat his way to victory. To ensure a fair process and to protect against the travesty of wrongfully convicting a person for an act he or she did not commit, the anti-doping system must strike an adequate balance between the need for accuracy and reliability of laboratory test results and fairness in sport. The rules of the Union Cycliste International ("UCI") and the rules of the International Standard for Laboratories ("ISL") and related technical documents have been developed to create this balance. But, any balance created by these rules is thrown off when the meaning of the rules are tortured to satisfy a pre-determined result. Simply put, to protect the integrity of the adjudicative process, the panel must not mold the ISL and other rules in attempt to avoid addressing evidentiary concerns; rather, the panel must apply the fair, clear, and common sense meaning of the rules to the facts as they are presented. When these rules are so applied, it is clear that the Labaratorie National de Depistage et du Dopage ("LNDD") committed so many critical rule violations and errors in rendering its alleged adverse analytical finding ("AAF") that the results are inaccurate, unreliable and offensive to proper scientific analysis.

This case is staked on the accuracy and reliability of the Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry test results ("GC/C/IRMS" or "IRMS") for

appellant's sample taken after Stage 17 (Sample 995474) of the 2006 Tour de France.[1] While the GC/C/IRMS is the test hailed as the gold standard for the detection of synthetic testosterone, it is also a complex test that must be performed with precision in order to obtain reliable and accurate results. LNDD's testing methods failed at every step of the way. These errors are not technical – they have resulted in inaccurate and unreliable test results that are an offense to proper laboratory procedure and accurate results. The failures of LNDD's testing procedure are readily apparent in its results. The positive GC/C/IRMS test results of LNDD are inconsistent with the known science and peer-reviewed articles detailing the breakdown of testosterone and its metabolites. This appeal will address each of these errors in detail, but the following is by way of introduction.

First, LNDD failed in a critical and basic step in GC/C/IRMS – ensuring that the test is being performed on testosterone and not something else. The GC/C/IRMS test uses instruments that (1) identify the metabolized compounds of testosterone and then (2) measure the isotopic value of each of those compounds. The body metabolizes testosterone into four different compounds -- Androsterone ("Andro"), Etiocholanolone ("Etio"), 5α-Androstanediol ("5 Alpha") and 5β-Androstanediol ("5 Beta"). These compounds are interchangeably described as "metabolites" or "analytes." The isotopic value is a calculation that identifies whether the source of the testosterone is synthetic or exogenous (pharmaceutical) or endogenous (from the body).

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[1] During the arbitration below, the United States Anti-Doping Agency ("USADA") recognized the many failures of the testing by the Labaratorie National de Depistage et du Dopage ("LNDD") in the screening test, known as the Testosterone-Epitestosterone ("T/E") test. Even the Arbitration Association of America's panel below found that the T/E test results were not accurate or reliable and violated the relevant International Standards for Laboratories ("ISL"). *See* Majority Award. The appeal summarizes the grievous errors committed by LNDD that resulted in the rejected test results at VII. The Results of the GC/MS Test are Flawed, *infra*.

The reason why the GC/C/IRMS measures the isotopic value of testosterone's metabolites as opposed to testosterone is that testosterone is metabolized quickly into its component compounds.

LNDD's own laboratory documentation proves that in this case, testosterone's metabolites were not properly identified, all in violation of the ISL. Because of this failure, it is impossible to know what compounds LNDD actually measured. Given the magnitude of the violation of the particular ISL in this case, it is unknown what LNDD was actually measuring.

Urine is a dirty matrix – a waste matrix that contains many different compounds aside from testosterone. As a result, in order to test compounds in urine – like testosterone's metabolites – those compounds must be separated from all other compounds in the urine with precision. Once separated, they must be identified. The process of identification is made complicated by the particular way the GC/C/IRMS testing process works. At LNDD, it actually consists of the use of two separate instruments, a Gas Chromatography-Mass Spectrometer ("GC/MS") and a GC/C/IRMS. The GC/MS instrument identifies the compounds. The GC/C/IRMS instrument measures the isotopic value, or delta-delta value of those compounds. The GC/MS can not measure isotopic value. The GC/C/IRMS can not identify the compounds. The only common factor between the two tests is that the same compounds should elute at the same time as each other ("retention time") or elute at the same time as each other relative to an injected internal standard ("relative retention time"). In order to "match up" the two test results, a comparison must be made of the "retention time" or "relative retention time" between the two tests – that is how the technician knows that what substance is being measured for isotopic value. WADA TD20034IDCR requires that this occur within a specific and narrow time frame. In actuality, the relative retention time ranged between five and six times the permissible

percentage allowed by the ISL – a 500% to 600% error rate. A closer examination of LNDD's procedures demonstrates clearly why the retention time and relative retention time were so far off – a combination of running the two different instruments under different conditions and different equipment has made it impossible to compare retention times or relative retention times. Again, this is not a "technical" failure that does not impact the test results. Given the ISL violation, if LNDD can not show that it measured testosterone's compounds instead of some other compound, its test results are worse than unreliable, they are a farce.

Second, the failure to properly identify testosterone is just one of many, interrelated errors, all of which are either caused by or are necessitated by each other. The reasons for the failure in identification directly stem from LNDD's incompetence and lack of familiarity with proper GC/C/IRMS technique. LNDD demonstrated its incompetence in every other critical area related to GC/C/IRMS testing. These areas included the failure or absence of quality controls, which render other critical components of the chromatographic and laboratory technique unreliable. This incompetence is related to the poor chromatography produced by the LNDD technicians while operating the GC/C/IRMS instrument, which makes the final delta-delta values completely unreliable. This competence is demonstrated by LNDD's failure to maintain its GC/C/IRMS instrument within its linearity specifications and numerous other instances or examples of situations where LNDD's technicians simply failed to understand the errors they were making or the importance of those errors.

All of these examples of incompetence relate to the improper calculation of the delta-delta value in this case – the calculation that results in the adverse analytic finding in this case. More specifically, the delta-delta value is calculation of the ratio of the carbon 13 ("^{13}C") to carbon 12 ("^{12}C") ratio, as compared to an endogenous reference compound ("ERC"). Pursuant

to LNDD's own internal guidelines, this delta-delta value, if it exceeds -3.0 for any one testosterone metabolite, will establish a positive finding for synthetic testosterone. In greater detail, these examples of incompetence are as follows:

- LNDD failed its own quality control. LNDD injected a substance with a known isotopic value into appellant's samples so as to be able to determine whether its GC/C/IRMS instrument could properly measure its isotopic value. This substance, 5 Alpha AC, also called an internal standard, was measured outside of its target isotopic value in many of the sample fractions. This means, simply, that LNDD's GC/C/IRMS instrument failed to measure isotopic value of a known substance within the known parameters – that it simply was not accurate. This is strong evidence that LNDD could not measure other targets isotopes – including appellant's sample – with any accuracy.

- LNDD had no positive control. A positive control involves the running of a sample of the same matrix as the unknown sample (a urine sample in this case) that is known to contain the metabolites of synthetic testosterone. A positive control thus allows a laboratory the ability to test its own ability to properly measure isotopic value to a target value. However, in this case, LNDD's so called "positive control," Mix Cal Acetate, did not contain three of the four target analytes: 5 Alpha, Pdiol and Andro. Without these three key target analytes, there are no assurances that the IRMS instrument can accurately measure these substances. Accordingly, the target analytes that established the alleged positive finding are not in the so-called positive control. In combination with the failure of its quality control, LNDD created a situation whereby it could not test whether it was correctly measuring isotopic value for a known compound while being completely ignorant of knowing whether it could measure the target compounds with any precision at all.

- LNDD's GC/C/IRMS instrument was not linear. Linearity is the ability of an IRMS instrument to accurately quantify the isotopic ratio of each testosterone metabolite and endogenous reference compound in different samples regardless of their concentration. LNDD failed to keep its instrument linear.

- LNDD produced extremely poor chromatograms in its GC/C/IRMS testing. Good chromatography is the key to reliable IRMS test results and is a requirement of the ISL. LNDD did not comply with the ISL because LLNDD failed to generate chromatograms that avoided interference in the detection of the prohibited substances or their metabolites and markers by components of the sample matrix. Matrix interference was common and clear in the critical chromatograms in this case. Matrix interference and poor chromatography can and will result in dramatic swings in isotopic values. Again, in combination with the poor identification techniques described earlier, LNDD created another completely scientifically intolerable situation. Because it could not identify target compounds using retention time or relative retention time, LNDD simply selected peaks based upon visual inspection. But here, poor chromatography resulted in peaks overlapping on top of peaks with poor separation. As detailed below, using visual inspection to identify compounds from GC/MS to GC/C/IRMS is impossible, but attempting to do so across chromatograms that have matrix interference makes the impossible simply, again, a farce.

- LNDD failed to comply with ISL 3.2 and WADA TD2003LCOC (Laboratory Internal Chain of Custody), which sets forth the requirements of internal chain of custody within a laboratory.

- LNDD did not comply with WADA TD2003LCOC and ISO 17025.4.3.3.3, which prohibit the cross-outs, interlineations and other changes made to the laboratory documents supporting the alleged AAF in this case.

- LNDD's incompetence is also obvious from its lack of familiarity with its own GC/C/IRMS instruments, whereby its staff mischaracterized technical features of the instrument and failed to prepare its GC/C/IRMS instruments for use following receipt by failing to detach packing devices that effect instrument accuracy.

Third, the many examples of incompetence described above forced LNDD technicians to breach their own internal guidelines and rules, to delete and alter data in violation of the ISL and to lie and produce fraudulent documents when confronted with these failures. Simply put, once LNDD committed to mistake, error and incompetence, it had to hide it. Examples of this misconduct includes:

- LNDD failed to run GC/C/IRMS runs in sequence, and instead stopped and restarted them to obtain desired results;

- LNDD violated several ISL provisions that require data entry and record keeping be retained for each analyzed sample when its technicians conducted manual processing. Manual processing is the process by which LNDD's technicians manually adjusted the start and end points of the peaks in the chromatograms and otherwise personally manipulated the GC/C/IRMS instrument with no record of that manipulation. This widespread use of manual processing in this case was necessitated by the poor chromatography in this case.

- LNDD technicians violated the ISL when they deleted relevant data that was obtained during the testing process. LNDD technicians deleted test results they found to be "incorrect" or that "did not correspond" to what they expected to find. In particular, LNDD

technicians deleted test results related to LNDD's quality control steps, including, but not limited to, results from the Mix Cal Acetate and blank urine runs. •

- Lastly, LNDD and USADA, as part of the litigation below, produced numerous false statements and at least one fraudulent document to conceal the errors and omissions described above.

The results of all of these ISL errors and breaches of laboratory protocol are evident in the GC/C/IRMS test results. LNDD's GC/C/IRMS results show a breakdown of testosterone that is inconsistent with both the peer-reviewed literature and the science of testosterone metabolism. In summary, the human body metabolizes testosterone into the four target istopes Androsterone ("Andro"), Etiocholanolone ("Etio"), 5α-Androstanediol ("5 Alpha") and 5β-Androstanediol ("5 Beta") in relatively equal quantities. More importantly, the ratio of ^{13}C to ^{12}C in testosterone should be reflected in these analytes. In other words, if the ^{13}C to ^{12}C ratio indicates that the testosterone was of synthetic origin, then the ^{13}C to ^{12}C ratio of the target analytes should similarly, and in approximately the proportion, show that same origin. Thus, when the delta-delta values of the target isotopes should be consistent with each other, and their values should rise and fall with each other.

In this case, LNDD has declared a positive finding for exogenous testosterone using its GC/C/IRMS test when the delta-delta values were grossly inconsistent with each other. For example, in the B Sample, the 5β-Adiol - 5β-Pdiol value was -2.65‰. The 5α-Adiol - 5β-Pdiol was -6.39‰. The Etiocholanolone – 11-Ketoetio was -2.02‰. The Androsterone – 11-Ketoetio was -3.51‰ (failing to account for measurement of error, which if accounted for, would have meant this was less negative than the -3.0 positivity criteria). The IRMS test results for Sample B show a difference of -3.74 per mil between the 5 Alpha – Pdiol and the 5 Beta – Pdiol value.

Id. These differences are inconsistent with the known metabolism of testosterone – the difference in the delta-delta values is simply too great. Similar inconsistent results appear in LNDD's GC/C/IRMS test results for the testing of other stages from the 2006 Tour de France that LNDD tested in preparation for trial. These other test results had no positive findings in the T/E tests, but nonetheless, LNDD managed to find positive findings for the same samples for the GC/C/IRMS test results. Moreover, the test results were inconsistent with known science because only one testosterone metabolite of four tested outside of the -3.0 range in the B Sample, again which is inconsistent with the known science about the breakdown of testosterone. Other laboratories, such as the U.S. Olympic laboratory have required that at least two metabolites test outside of the -3.0 limit.

From a technical and legal standpoint, this case is about science, and the rules of the ISL and the UCI. Appellant will prove, as he did below, critical scientific errors that directly dispute the accuracy and reliability of LNDD's test results. However, more fundamentally, this case is one of conscience and of the integrity of a adjudicative system. To allow these myriad errors to stand is to validate gross laboratory errors and unreliable results, all with the effect of stripping a champion of his title and destroying a reputation and career. This would be a simple miscarriage of justice.

A. Statement Of Jurisdiction

On September 20, 2007, a three-arbitrator panel of the North American Court of Arbitration of Sport of the American Arbitration Association ("AAA") issued its decision in *USADA v. Floyd Landis*, Case No. AAA No. 30 190 00847 06 (the "Appealed Case"). The panel's decision consisted of a majority opinion finding that the alleged anti-doping rule violation had been established by a comfortable satisfaction (Brunet, P. and McLaren, R.) and a

contemporaneously filed dissenting opinion (Campbell, C.). On October 8 and 11, 2007, Mr. Landis filed his notice of intent to appeal. The Court of Arbitration for Sport ("CAS") has jurisdiction over this appeal pursuant to Arts. 280 and 242 of the UCI Cycling Regulations and R47 of the Code of Sports-related Arbitration ("CAS Code"). This brief is timely filed pursuant to R51 of the CAS Code.

B. Prayer For Relief

1. That the decision of the majority panel in *USADA v. Floyd Landis*, Case No. AAA No. 30 190 00847 06 be reversed;

2. That all allegations of any anti-doping rule violation, and related proceedings, against Appellant be dismissed with prejudice;

3. That Appellant be reinstated as the winner of the 2006 Tour de France and that (1) the classifications and records of the UCI and ASO reflect that Appellant is the winner of the 2006 Tour de France, (2) all UCI ProTour points and standing shall be restored to Appellant and (3) he shall be entitled to collect, retain and receive all other prizes and premiums associated with winning the 2006 Tour de France;

4. That any suspicion, restriction, or prohibition on Appellant's ability to race in any UCI, Olympic, or other associated organization be immediately voided and/or removed;

5. That Appellee shall bear all costs of the arbitration and all the legal fees and costs of Appellant in bringing this appeal; and

6. Such further relief as this Panel may deem necessary to effect the relief sought above.

C. Standard of Review

CAS Art. R57 provides that this is a *de novo* hearing, and that CAS shall review all of the facts and the law. As such, neither the Panel nor the parties are constrained in any way by the evidence that was previously presented; to the contrary, the Panel is entitled to consider new evidence. See H v. FIM (CAS 2000/A/281).

D. The Record From The Appealed Case, Its Previously Filed Exhibits And Testimony

Appellant respectfully requests that the entire record from the Appealed Case be made part of the record in this case. The record includes:

1. The pretrial motions, responses and briefs of the Parties, inclusive of exhibits to those filings;

2. The Interim Awards and Procedural Orders, and accompanying dissents;

3. All briefing filed in conjunction with the arbitration hearing held on May 14 – 23, 2007;

4. Appellant's trial exhibits;

5. Appellee's trial exhibits;

6. The DVD of the hearing;

7. Appellant's Proposed Findings of Fact; and

8. The Final Award and accompanying dissent.

II.

STATEMENT OF FACTS

This case involves the single issue of whether Floyd Landis violated an anti-doping rule based on the testing of Sample 995474 provided after Stage 17 of the 2006 Tour de France.[2] The Adverse Analytical Finding on Sample 995474 rested on the results from two testing methods: the GC/MS test[3] and the GC/C/IRMS test.

A. The Testing Of Sample 995474

The 2006 Tour de France (the "Tour") began on July 1, 2006, and ended on July 23, 2006. On July 23, 2006, Mr. Landis was declared the winner of the 2006 Tour, having won the general classification by 57 seconds.

On July 20, 2006, immediately after Stage 17, Mr. Landis provided a urine sample, Sample 995474, to the Union Cycliste International ("UCI"). Ex. 41, USADA0447. As set forth more fully below, this was one of eight samples Mr. Landis provided during the Tour. Sample 995474 was tested at the Labaratorie National de Depistage et du Dopage ("LNDD").

On July 25, 2006, after receiving allegedly positive test results on both the GC/MS and IRMS tests on the A Sample from Sample 995474, LNDD notified the Conseil de Prevention et du Lutte Contre le Dopage ("CPLD") and the UCI that the A Sample from Sample 995474 displayed an Adverse Analytical Finding ("AAF"). *See* Ex. 24, USADA0188-0199.

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[2] There will be references to testing completed on other Samples provided by Floyd Landis during the 2006 Tour; however, as will described below, these tests cannot provide the basis for any anti-doping rule violation and were performed by Appellee several months after the 2006 Tour as a means of gaining further evidence to be used during the arbitration.

[3] WADA rules require that a GC/MS test for testosterone, also known as the Testosterone/Epitestosterone test or "T/E" be corroborated by other testing method in order to establish an Adverse Analytic finding. *See* Exhibit 49, WADA0011-0021.

On July 27, 2006, USADA notified Mr. Landis of the AAF and commenced prosecution of the Appealed Case. *See* Exs. GDC00001-00003. In its communication to Mr. Landis, USADA indicated that he could either request testing of the B Sample of Sample 995474 or accept the AAF from the A Sample. Mr. Landis refused to accept the AAF and elected to have the B Sample tested. *See* Exs. GDC00004-00005.

Between August 3 and 5, 2006, LNDD tested the B Sample from Sample 995474. Ex. 25, USADA0365-0366.

The GC/MS and IRMS tests performed on the B Sample of Sample 995474 resulted in the alleged AAF at issue here. *See id.*

On August 5, 2006, the UCI notified Mr. Landis, USADA, the Agence Française de Lutte Contre le Dopage ("AFLD") and the media of its findings. *See* Ex. GDC00006.

On September 11, 2006, Mr. Landis filed pleadings before USADA's Anti-Doping Review Board to have this case dismissed. *See* Exs. GDC00007-00022. On September 18, 2006, the Anti-Doping Review Board rejected Mr. Landis's petition and the Appealed Case began. *See* Ex. GDC00023.

B. The Retesting Procedure

During the course of the 2006 Tour, Mr. Landis provided seven urine samples in addition to Sample 995474. Mr. Landis provided those samples at the conclusion of the following stages: Stage 2 (Sample 995642 on July 3), Stage 9 (Sample 994203 on July 11), Stage 11 (Sample 994277 on July 13), Stage 12 (Sample 994276 on July 14), Stage 15 (Sample 994075 on July 18), Stage 19 (Sample 994080 on July 22), and Stage 20 (Sample 994171 on July 23). *See* Ex. 41, USADA0412, 0419, 0426, 0433, 0440, 0447, 0458, 0465.

Each of these seven other samples was tested at LNDD. *See* Ex. 41, USADA0415, 0422, 0429, 0436, 0443, 0461, 0468. All of the A Samples from these other samples resulted in a negative finding on the GC/MS test and, thus, were not reported as an AAF. Accordingly, during the Tour: (1) Mr. Landis was not notified of any issue related to an anti-doping rule violation based on these other samples and (2) no further testing of the B Samples from these other samples was conducted.

Solely in preparation for arbitration, Appellee requested that LNDD test the B Samples of these other samples using the IRMS method. Mr. Landis strenuously objected to LNDD performing the testing of these B Samples because the same methods and procedures that Mr. Landis was challenging would be used, and LNDD had a conflict of interest. Following extensive briefing, the Panel in the Appealed Case permitted LNDD to perform these tests. The IRMS testing of the B Samples from the other samples provided by Mr. Landis during the Tour was commenced by LNDD on April 16, 2007.

The specific results of the retesting are summarized at Exhibit GDC01363. However, in sum, LNDD found that some of the B Samples were allegedly positive for testosterone. Notwithstanding the fact that these samples were previously reported negative for testosterone, at the arbitration, USADA sought to admit these test results as "corroborating evidence" for the alleged AAF in Sample 995474.

C. The Reprocessing Of The Electronic Data Files

As noted above, the AAF for Sample 995474 was based on two different testing methods: the GC/MS and the IRMS. During the IRMS testing process, several data files are created, which are known as Electronic Data Files ("EDFs"). The EDFs contain raw data, which is data before any analysis and interpretation.

Appellant requested that the EDF's be reprocessed because the IRMS test on Sample 995474 was performed on an older instrument, did not have audit capabilities, and, critically, allowed the operator to manually adjust, without any record, two important factors that affect the test results – the background points[4] and the integration of the peaks.[5]

Pursuant generally to the Panel's discovery rulings and, specifically, Procedural Order No. 2, on April 26, 2007, the EDFs from the IRMS test of Sample 995474 were supposed to be extracted from the instrument that performed the IRMS test, the IsoPrime1. Representatives of both Mr. Landis (Dr. Simon Davis and Dr. Will Price) and of USADA (Dr. Larry Bowers and Dr. Jeanine Jumeau), as well as by the Panel's expert, Dr. Francesco Botrè, arrived to observe this extraction; however, they were told that: (1) the EDFs from the IsoPrime1 (the instrument used to test Sample 995474) had already been copied to an archive CD and (2) the original information on the IsoPrime1 hard-drive had been erased.[6] This process involved a third party subcontractor removing the hard disk from the instrument control computer, copying the data and re-formatting the hard disk. This operation was carried out the morning we arrived to obtain the data. Further it did not appear that this followed the normal pattern of backing up the data.

On May 4, 2007, Dr. Botrè and representatives for both USADA and Mr. Landis arrived at LNDD to reprocess, or in other words re-analyze and interpret, the EDFs. Pursuant to

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4 Background is the isotopic value read by the detector when no substance (e.g., peak) is present.

5 Peak integration is accomplished by defining the start and end points of each peak.

6 Also on April 26, 2007, the log files from the IsoPrime2 were copied onto a separate CD. These log files are a record of the testing procedures performed in conjunction with the retesting of the other samples taken from Mr. Landis during the Tour. The log files are Exhibits GDC01056-01075.

directions provided by Mr. Landis's representatives, LNDD technicians performed a series of operations on the EDFs.[7]

The first operation occurred at Dr. Botrè's direction. This operation involved LNDD's attempt to reproduce the original test results using the same processes used, and the same instrument (IsoPrime1), to determine those results. In attempting to reproduce the original test results, LNDD's IRMS technicians used a manual processing technique, which they said they used during the original processing of Sample 995474. Manual Processing includes both: (1) manual adjustments to the background, and (2) manual integration of peaks. Tr. of R. at 1763:1-10. However, despite 22 attempts to do so, LNDD technicians were unable to reproduce the original IRMS test results on the A and B Samples of Sample 995474. The chart showing the number of reprocessing attempts is Exhibit GDC01365. The chart showing the results of the reprocessing is Exhibit GDC01350.

In addition, three other sets of values were obtained using three distinct processes: (1) delta-delta values[8] were calculated using the automatic background subtraction embedded within the software program, (2) delta-delta values were calculated with the automatic background subtraction disabled and (3) delta-delta values were calculated using the Masslynx software

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[7] Because LNDD technicians did not know how to transfer data from the CD onto the computer operating the IsoPrime1, Dr. Davis performed this part of the procedure.

[8] The delta-delta value equals the delta value of the target compound minus the delta value of the endogenous reference compound. The delta-delta value is the value used to determine an AAF and is expressed as the "per mil" value. This will be further explanation below.

loaded onto the IsoPrime2.[9] The chart showing the results of this reprocessing is Exhibit GDC01350.

III.

THE FUNDAMENTAL FAILURE OF THE GC/C/IRMS TEST: COMPLETELY UNRELIABLE IDENTIFICATION OF TESTOSTERONE METABOLITES

A. LNDD'S IRMS Test Results

LNDD's IRMS test results for Sample 995474 were unreliable and cannot be the basis for any anti-doping rule violation. The IRMS test has two different processes, a process to identify a particular substance in a complex solution with thousands of substances, and a process to measure the isotopic value of the substance previously identified. LNDD's IRMS test for Sample 995474 is subject to a fatal flaw: LNDD failed to properly identify the critical metabolites of testosterone as required by TD2003IDCR. Simply put, LNDD has no ability to establish that the substances measured in Sample 995474 were the critical metabolites of testosterone that were in fact supposed to be measured. Moreover, USADA can introduce no evidence to show that the failure to identify critical metabolites, as required by TD2003IDCR, did not cause the Adverse Analytic Finding.

B. The Theory Of The IRMS Test

The theory behind the IRMS test rests on the difference in the molecular structure of naturally produced (endogenous) or synthetically produced (exogenous) testosterone. Testosterone is composed of Carbon, Oxygen and Hydrogen atoms. However, there are several

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[9] LNDD IRMS technicians did not know how to convert the EDFs into data readable by Masslynx. Therefore, Dr. Davis performed this part of the operation. Tr. of R. at 1764:4-10.

isotopes of Carbon, including the stable isotopes ^{12}C and ^{13}C.[10] Testosterone and its metabolites are composed of a mixture of ^{13}C and ^{12}C. The ratio of ^{13}C and ^{12}C, however, in any individual will vary based on its source. For example, synthetically produced testosterone is produced from soy plants, which are particularly low in ^{13}C, also known as ^{13}C depleted, compared to natural testosterone. Thus, a person who uses synthetic testosterone will have testosterone with fewer ^{13}C atoms. In the context of anti-doping, the IRMS instrument measures the ratio of ^{13}C to ^{12}C, also known as the isotopic ratio or isotopic value, in specific metabolites of testosterone, as explained below.

The IRMS test does not measure the isotopic ratio of testosterone – it examines the metabolized products ("metabolites") of testosterone. The IRMS test measures the following four metabolites of testosterone: Androsterone ("Andro"), Etiocholanolone ("Etio"), 5α-Androstanediol ("5 Alpha") and 5β-Androstanediol ("5 Beta"). The following diagram is illustrative:

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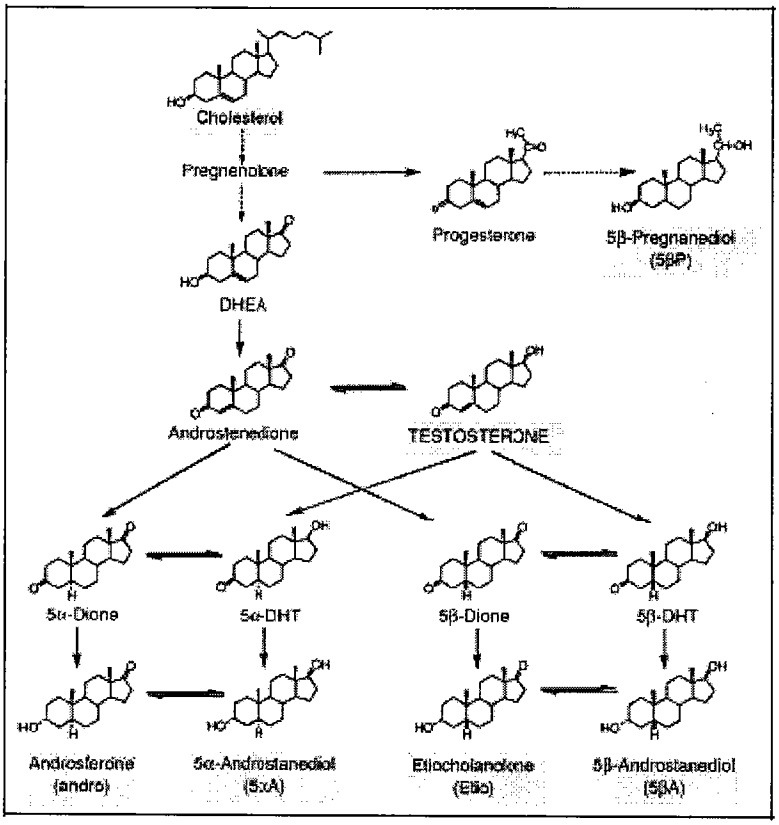
[10] The difference between ^{12}C and ^{13}C is that ^{13}C has one more neutron. Most all carbon on earth is ^{12}C, whereas, approximately 1.1% of all carbon is ^{13}C.



11 **Figure 131. Metabolic pathways of testosterone. From Maitre.** [24]

The carbon framework of the testosterone metabolites will maintain essentially the same isotopic value as the testosterone from which they originated. Therefore, measuring the isotopic ratio of the metabolites is tantamount to measuring the isotopic ratio of testosterone.

There are several individual variables that can cause endogenous testosterone and its metabolites to become ^{13}C depleted that are unrelated to using exogenous testosterone, such as diet. To account for these individual variables, the IRMS test compares the $^{13}C/^{12}C$ ratio of a testosterone metabolite to the $^{13}C/^{12}C$ ratio of an endogenous reference compound ("ERC"). By

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11 Maitre et al., Urinary Analysis of Four Testosterone Metabolites and Pregnanediol by Gas Chromatography–Combustion–Isotope Ratio Mass Spectrometry after Oral Administrations of Testosterone, Journal of Analytical Toxicology, Vol. 28, September 2004, USADA0799.

comparing the difference in the $^{13}C/^{12}C$ ratio between a testosterone metabolite and an ERC, if performed properly, indicate the likelihood of testosterone being from an exogenous source.

In theory, for any individual at any one time the $^{13}C/^{12}C$ ratio of an ERC should be close to that of a testosterone metabolite. If a person is using exogenous testosterone, however, there will be a detectable and significant difference between the $^{13}C/^{12}C$ ratio in a testosterone metabolite and an ERC. In other words, if a person is taking exogenous testosterone, his or her $^{13}C/^{12}C$ ratio for a testosterone metabolite will be different than the ratio for an ERC.[12]

That there is some detectable difference between the $^{13}C/^{12}C$ ratio between the metabolite and the ERC does not result in a positive test, however. Once the $^{13}C/^{12}C$ ratio for the ERC is subtracted from the testosterone metabolite, referred to as the $\delta^{13}C‰$ value or the delta-delta value, it must be compared to the positivity criteria mandated by WADA. The WADA positivity criteria for IRMS is as follows:

> The results will be reported as consistent with the administration of a steroid when the $^{13}C/^{12}C$ value measured for the **metabolite(s)** differs significantly i.e. by 3 delta units or more from that of the urinary reference steroid chosen. In some *Samples*, the measure of the $^{13}C/^{12}C$ value of the urinary reference steroid(s) may not be possible due to their low concentration. The results of such analysis will be reported as

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[12] A good summary of the IRMS theory is provided at Maitre et al., Urinary Analysis of Four Testosterone Metabolites and Pregandiol by Gas Chromotography-Combustion-Isotope Ratio Mass Spectrometry After Oral Administration of Testosterone, 28 Journal of Analytical Toxicology (Sept. 2004).

IRMS allows measurements of slight differences in the carbon isotope ratio ($^{13}C/^{12}C$) of the exogenous and endogenous testosterone. Synthetic testosterone is produced from precursors derived from plants with low ^{13}C content, whereas the ^{13}C and ^{12}C content in the natural endogenous form depends on the isotopic carbon composition of the food in a person's diet and is influenced by additional effects of human biological processing.

"inconclusive" unless the ratio measured for the metabolite(s) is below -28‰ based on non-derivatized steroid.[13]

See Exhibit WADA0011-0021, at 3.

As noted above, there are several metabolites whose isotopic values are measured by the IRMS instrument (Androsterone, Etiocholanolone, 5α-Androstanediol[14] and 5β-Androstanediol[15]), along with the isotopic value of two ERCs (11-Ketoetio and 5β-Pdiol). LNDD in theory identifies and measures all of these metabolites and ERCs. However, the relevant delta-delta numbers are calculated by subtracting the delta value of 11-Ketoetio (ERC) from the delta value of Etiocholanolone and Androsterone (metabolites) and from subtracting the delta value of 5β-Pdiol (ERC) from the delta value of 5β-Adiol and 5α-Adiol (metabolites).

On July 24, 2006, LNDD conducted the IRMS test on Mr. Landis's A Sample from Sample 995474. The delta-delta values were as follows:

Etiocholanolone – 11-Ketoetio	-2.58‰
Androsterone – 11-Ketoetio	-3.99‰
5β-Adiol - 5β-Pdiol	-2.15‰
5α-Adiol - 5β-Pdiol	-6.14‰

On August 3, 2006, LNDD began the IRMS test on the "B" sample. The delta-delta values were as follows:

[13] In the case of LNDD, it has already been conceded that, due to a measure of uncertainty of 0.8‰, the LNDD positivity criteria is a delta-delta value that is more negative than -3.8‰.

[14] Also referred to as 5α-Adiol.

[15] Also referred to as 5β-Adiol.

Etiocholanolone – 11-Ketoetio	-2.02‰
Androsterone – 11-Ketoetio	-3.51‰
5β-Adiol - 5β-Pdiol	-2.65‰
5α-Adiol - 5β-Pdiol	-6.39‰

C. How The IRMS Test Operates

The IRMS test consists of three main steps (1) sample preparation, (2) pre-IRMS compound identification by GC/MS and (3) IRMS analysis. Each one of these steps must be performed properly in order to obtain accurate delta-delta values.

1. Sample Preparation

The IRMS test begins with sample preparation. First, an aliquot is made from the sample; additionally, an aliquot made from blank urine,[16] which is taken from a pool of urine known not to contain synthetic testosterone (it is often the urine pooled from lab technicians). These aliquots are then cleaned through several physical, enzymatic and chemical treatments. The reason for this step is obvious – urine is a waste product, a "dirty" matrix, in which many other substances, in addition to testosterone and its metabolites, will be present. In order to ensure the accuracy of the IRMS results, the sample must be stripped of those other substances so that it is clear that the laboratory is not measuring/analyzing the wrong substances.

The aliquots are then separated into three fractions using further physical treatments. The three fractions created are as follows: (1) the F1 fraction, containing 11-Ketoetiocholanolone (11-Keto), (2) the F2 fraction, containing Etiocholanolone (Etio) and Androsterone (Andro) and

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[16] Additionally, the blank urine aliquot is used during the test as a "known negative" control.

(3) the F3 fraction, containing 5α-Androstanediol (5α-Adiol), 5ß-Androstanediol (5ß Adiol) and 5ß Pregnanediol (5ß Pdiol).

One of the last steps in sample preparation is the addition of an "internal standard." The internal standard, which in this case was 5 Alpha Androstanol Acetate, is a substance with a known isotopic value. It therefore serves as a quality control mechanism (to test whether the IRMS instrument is working properly) and also serves as an anchor to calculate relative retention times, which will be discussed in detail below.

2. The Instrument

As described below, the IRMS test uses two different instruments - the GC/MS instrument and the GC/C/IRMS instrument. Two instruments are needed because neither instrument can perform both the necessary functions to complete the test – identification and measurement. The GC/MS instrument cannot measure isotopic values, it can only identify substances; whereas, the GC/C/IRMS instrument can measure isotopic values, but it cannot identify substances. In some anti-doping laboratories, the GC/MS instrument is attached to, and part of, the IRMS instrument. However, at LNDD, two different and non-attached instruments were used.

3. The GC/MS Analysis: Compound Identification

Once the fractions are prepared, the first phase of IRMS testing – compound identification with the GC/MS instrument – begins.

The GC/MS instrument is composed of two major components: the gas chromatograph and the mass spectrometer. The gas chromatograph is used to separate molecules by sending these molecules through a column, which is essentially a tube coated with complex hydrocarbons. This coating is called the "stationary phase." Based on the interaction of each

individual molecule with the stationary phase, each compound moves through the column at different speeds. The amount of time each molecule takes to move though the column is the molecule's retention time. The fastest moving molecules reach the end of the column first, thus corresponding with the first peak in the chromatogram (Figure 1-3c). The next fastest molecule follow and create another peak in the chromatogram. This process continues until all of the remaining compounds have left the column (*See* Figure 1-3d).

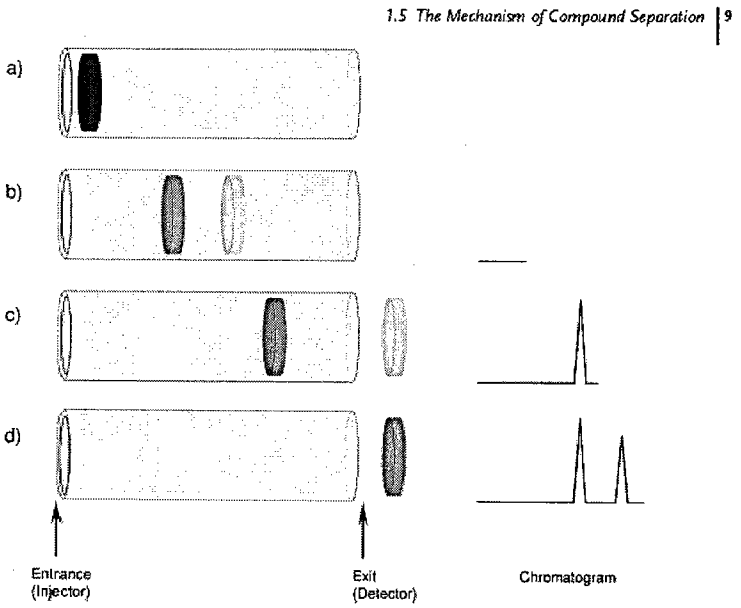
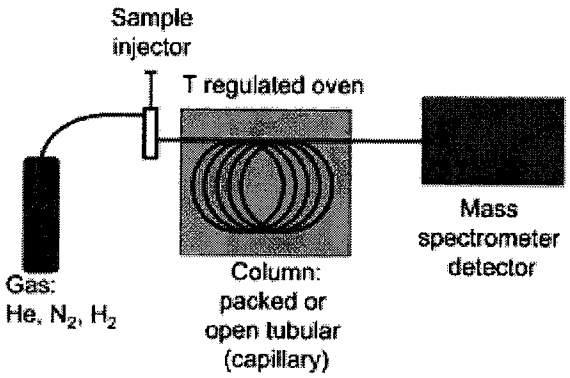


Figure 1-3 Separation of the sample in the column.

Different molecules can have the same retention times, however. Therefore, after each molecule's retention time is measured, they are passed to the mass spectrometer. The molecules are passed through a stream of electrons. Electrons passing near to, or contacting, the analyte result in one or more electrons being knocked off the molecule in question. This process, known as ionization, results in the molecule becoming "charged". A charged molecule is known as an ion. There are typically a number of different ions creates in this process, the parent ion and fragment ions. Parent ions are intact molecules that have simply lost one or more electrons

during ionization. Fragment ions are "fragments" of the parent ion broken off during the process of ionization. Once ionized, the mass spectrometer measures the abundance of the different ions, also called a response, using each ionized mass to charge (m/z) ratio. This is akin to a molecular fingerprint, and is recorded by the mass spectrometer.



The GC/MS test produces a series of documents called chromatograms. The chromatogram will show all molecules within a designated m/z ratio. The chromatogram is simply a graph with time on the X-axis and response, or quantity, on the Y-axis. On the chromatogram, there are several peaks, each of which should correspond to a single compound in the sample. In sum, the GC/MS chromatogram identifies compounds by their retention times and m/z ratio.[17]

4. Step 3: IRMS Analysis

After the identification of all of the target metabolites pursuant to the GC/MS analysis, the fractions are then injected into the GC/C/IRMS instrument. Once the fraction is injected into

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[17] Also as part of the GC/MS phase, LNDD ran a Mix Cal Acetate analysis. Mix Cal Acetate is a solvent containing 5 Alpha Androstanol Acetate ("5 Alpha AC"), 5 Beta Androstanol diAcetate ("5 Beta diAC"), Etiocholanolone AC ("Etio AC") and 11-ketoetio AC. The Mix Cal Acetate serves as a means to check the sensitivity of the GC/MS instrument and is also a control because retention times for the Mix Cal Acetate are known and should match with the sample.

the GC/C/IRMS instrument, the compounds in the fraction are separated by gas chromatography. Similar to the GC/MS test, these molecules have to travel through a column and their retention times are recorded. However, unlike in the GC/MS instrument, after the molecules reach the end of the column, the molecules are combusted in the combustion furnace. Only carbon dioxide remains after this step and there is no longer any means to measure the m/z ratio of the substance. However, the carbon dioxide is then analyzed by the isotope ratio mass spectrometer, which determines the different masses of carbon dioxide. This analysis then determines the compound's isotopic value.

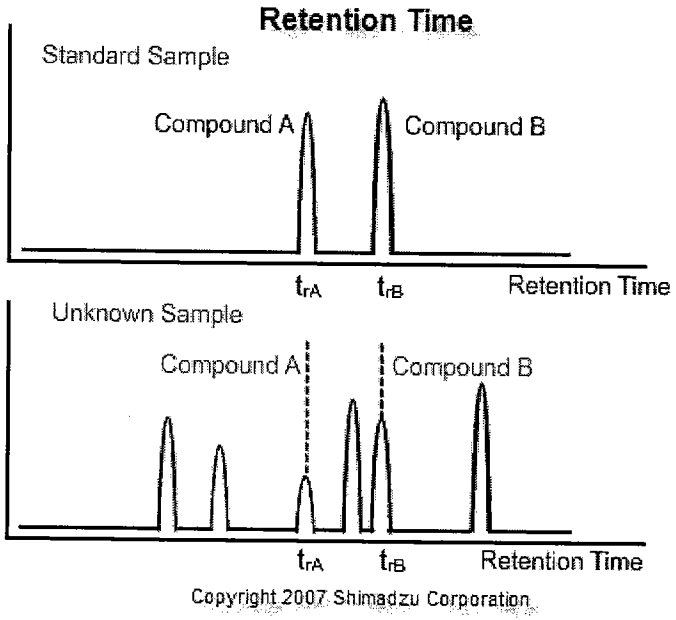
Although the only matrix that is injected into the GC/C/IRMS instrument described above is the fractions, there are several other samples introduced into the IRMS machine during the testing process. These include stability samples, Mix Cal IRMS samples, and Mix Cal Acetate samples. Indeed, to ensure accurate and reliable results, these samples are required to be in a specific order, which is as follows: (1) Stability run 1, (2) Stability run 2, (3) Stability run 3, (4) Mix Cal IRMS 003-1, (5) Mix Cal IRMS 003-2, (6) Mix Cal IRMS 003-3, (7) Mix Cal Acetate, (8) Blank Urine fraction F3, (9) Sample F3 fraction, (10) Blank Urine F1 fraction, (11) Sample F1 fraction, (12) Blank Urine F2 fraction, (13) Sample F2 fraction and (14) Mix Cal Acetate.

The stability runs serve to test the reproducibility of the IRMS detector. The Mix Cal IRMS is a mixture of four reference standard alkanes: decane, undecane, dodecane and methyldecanoate. The Mix Cal IRMS serves to show specificity, which means that the IRMS instrument will be able to reproduce the IRMS results over the range of values covered by the Mix Cal IRMS run. The Mix Cal IRMS injections are also run to check the precision of the instrument. The Mix Cal Acetate (Mix Cal AC) injection is run to calibrate the GC/C/IRMS

instrument. The Mix Cal Acetate contains four standard reference steroids with defined delta values. This sample also serves to test the accuracy of the IRMS instrument over a wide range of known delta values.

D. The Critical Importance Of Retention Time And Relative Retention Time: WADA TD2003IDCR

Retention time is the amount of time it takes a molecule to travel through the GC column. The reason that retention time can be used to identify compounds in the two phases of the IRMS test is that, because under constant chromatographic conditions, the retention time of a compound is reproducible. Thus, assuming that identical chromatographic conditions exist in both phases, the absolute retention times should be the same. The following example is illustrative:



The major problem of the use of retention time to identify compounds is the necessity of maintaining "exactly identical chromatographic conditions." A subtle temperature difference of

1 °C, a slightly increased carrier gas pressure, a larger column, or a few seconds of delay when starting the acquisition may cause retention time deviations.

Relative retention time was the approach used to overcome these limitations. It is calculated by dividing the retention time of the target analytes (in this case, 5alpha, 5beta, Andro, Etio, 11-ketioetio and Pdiol) by the retention time of a known internal standard (in this case, 5αAndrostanol Acetate). In other words, the compounds retention time is anchored by the internal standard. Relative retention time thus helps normalize the variations between systems because any change in the retention time will have an equal effect on both) and relative retention times can be used to compare between different systems.

The reason we have two separate testing processes is that one process, or instrument has the sole purpose of identifying the target analyte by several characteristics, including retention time, while the other testing process has the purpose of measuring the isotopic ratio and the retention time of the analyte being measured. Thus, the importance of the retention time and relative retention time are critical to accurate results is due to the fact that it is the only constant between the two instruments and is the only means by which one can tie the results from one instrument to the other.

Specifically, the GC/MS phase can only identify the testosterone metabolites. The GC/C/IRMS phase can only calculate isotopic ratios. In order to ensure that isotopic ratio for the correct metabolites in question are being measured, a comparison must be made of the chromatograms that have resulted from the GC/MS phase and the GC/C/IRMS phase. This comparison must compare the peaks in the chromatograms resulting from the GC/MS phase (that identify the substances) and the peaks in the GC/C/IRMS phase. There is only one way to make this comparison – by the amount of time that the molecules have taken to exit the GC column to

the mass detector. Time is the only constant between the GC/MS phase and the GC/C/IRMS phase of the test.

This requirement is so critical that a specific WADA technical document requires that retention times or relative retention times are consistent between the GC/MS and GC/C/IRMS phases. In order to be certain that the laboratory staff are calculating the isotopic values of the correct peak, TD2003IDCR requires that the retention time of the peaks from the GC/MS process fall within specified time periods of each other: plus or minus .2 minutes or 1%, whichever is smaller. Without conforming to this requirement, there is no way to be certain that the peaks selected by the technician in the IRMS chromatographs are in fact the peaks that were previously identified as the target compounds (e.g., 5 Alpha, 5 Beta, Andro, Etiocholanolone ("Etio"), 11-ketoetio and Pdiol). *See* Tr. of R. at 1400:1-1419:3. Specifically, WADA TD2003IDCR states that:

> For capillary gas chromatography, the retention time (RT) of the analyte shall not differ by more than one percent or ± 0.2 minutes (whichever is smaller) from that of the same substance in a spiked urine sample, Reference Collection sample, or Reference Material analyzed contemporaneously.

Exs. GDC00396-00400.

Overwhelming evidence introduced at the AAA hearing supported the fact that retention time and relative retention time was essential to properly identify testosterone's metabolites – and retention time and relative retention time was in fact supposed to be used by LNDD for precisely this purpose. Further, substantial evidence was introduced at the AAA hearing that LNDD did not ensure that the retention time and relative retention time for the target analytes in the GC/MS and GC/C/IRMS tests did not fall within the standard allowed in TD2003IDCR.

1. Appellant's Witnesses

At the AAA hearing, Dr. Meier-Augenstein testified that the variances in the relative retention times are so great that LNDD cannot identify its own internal standard or the other peaks associated with the target compounds. Tr. of R. at 1517:13–1520:15. He was not cross-examined on this central point.

2. Appellee's Witnesses

USADA's witnesses corroborated Dr. Meier-Augenstein's assertion that LNDD used relative retention time to properly identify the metabolites of testosterone in the GC/C/IRMS test for Sample 995474. At the AAA hearing, Cynthia Mongongu, an LNDD lab technician, testified that LNDD added an internal standard to the blank urine and to the athlete's sample "to calculate the relative retention time of the molecules analyzed." Tr. of R. at 653:8-10. Ms. Mongongu was asked whether the purpose of relative retention time was "to make sure that you're looking at the right peaks." *Id.* at 653:11-13. To which she replied, "Absolutely, Yes." *Id.*

Dr. J. Thomas Brenna's testimony at the AAA hearing also supports the importance of retention time and relative retention time. In describing the identification method for compounds in GC/C/IRMS, Brenna testified that LNDD's GC/C/IRMS chromatograms "have retention times that match . . . the previous GC/MS, and the GC/MS delivers structural information, like aliquots and so forth, that tell us which is which." *Id.* at 255:18-22.

> . . . 171 is a GC/MS run which was shown 22 this morning, before lunch, and it is of Sample 995474, Fraction 3, so it's exactly the sample that is of interest here. And there are three peaks of particular interest. There is the 5-beta, the 5-alpha and the pdiol, which is the ERC --
>
> Q. Okay. And then, could you tell me what 173 is?
>
> A. -- 173, which I think is also here somewhere -- but in any case, 173 is the GC combustion version of that same chromatogram, that same sample. Sorry, the GC combustion -- IRMS. Sorry. We've been calling it the IRMS. I apologize. The IRMS version of that.

Q. And what are the three peaks of interest there?

A. Same three.

Q. And how would I know --

A. 5-alpha, 5-beta --

Q. And how would I know which is which, because they just have numbers at the top.

A. Well, they have retention times that match on the previous -- with the previous GC/MS, and the GC/MS delivers structural information, like aliquots and so forth, that tell us which is which.[18]

Last, Montreal WADA Lab expert witness, Dr. Christiane Ayotte, confirmed the importance of the use of relative retention time in identification. She testified:

Q. Did you hear Ms. Mongongu testify yesterday that the Paris lab runs an internal standard -- I think it's 5 alpha andro-stenediol --

A. Androstanol.

Q. Thank you -- to -- for the purpose of identifying retention times?

A. Yes, I heard her.

Q. Okay. And does the Montreal laboratory have an internal standard that you run for that same purpose?

A. It's good practice to add in each assay a standard to determine the relative retention time of your analytes, the substance that you will -- that you will wish to measure after. It's common and very good practice, so we have the same -- as a matter of fact, we have the same substance as a standard for that purpose.[19]

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[18] HT 255:16-22.

[19] HT 811:23-812:18.

Dr. Ayotte continued:

> A. But I'd say on the contrary, it's -- it's necessary to establish the relative retention time. It's a necessity; otherwise, you don't know what you are measuring, so . . . .[20]
> (...)
>
> Q. So just so I can be clear as to what your testimony is: **In this case, the IRMS analysis, what is the purpose of the internal standard, in your opinion?**
>
> A. In that -- **in their procedure, that standard, that, as a matter of fact, is added** after several steps of the preparation, **is used to establish the relative retention times.**[21]

3. USADA's Pre-Hearing Brief

USADA's brief also specifically asserted that LNDD used retention time and relative retention time to properly identify the metabolites of testosterone in the IRMS test for Sample 995474. USADA's brief states, in relevant part:

> The second of the three steps in the LNDD test is pre-IRMS compound identification by GC/MS, the gold standard for compound identification in analytical chemistry applications. GC separates the compounds present in a mixture and MS identifies them. The first element of compound identification is the GC "retention time (RT)" and the second one is the molecular fingerprint recorded by the MS, which fragments the molecule into ions. Compound identification is achieved by matching GC retention times and MS ion patterns (Ion ratios) between the compound in the sample and a reference standard. . . .
>
> A parameter that is even better than the retention time is the relative retention time (RRT). It relies on the internal standard that was added to each tube during sample preparation. The internal standard has its own characteristic retention time. The relative retention time of any other compound is simply (RT of other compound)/(RT of internal standard). This makes comparisons of retention times easier because it normalizes them.

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[20] HT 813:3-6.

[21] HT 849:20-850:2.

See USADA's Pre-Hearing Brief ¶¶ 41-42.

Therefore, based on its admission (and the admission of its witnesses) that retention time and relative retention time is necessary to identify properly the target analytes being measured, USADA cannot now legitimately argue that: (1) retention times do not matter or (2) relative retention times were not used to identify the testosterone isotopes in this case.

4. The AAA Panel

Even though the AAA majority panel adopted a flawed analysis of the retention time/relative retention time issue, it also recognized the fundamental use of the WADA TD2003IDCR. Paragraph 179 of the Majority Award states:

> What [WADA TD2003IDCR] does is to ensure that the technician is calculating the isotopic values of the correct peak. The Technical Document requires that the retention time of the peaks from the GC/MS part of the CIR test process falls within specified time periods of each other: plus or minus .2 minutes or 1%, whichever is smaller. Without this requirement, there is no way to be certain that the peaks selected by the technician in the IRMS chromatographs are in fact the peaks that were previously identified as the target compounds (e.g. 5 Alpha, 5 Beta, Andro, Etiocholanolone ("Etio"), 11-ketoetio and Pdiol).

E. WADA TD2003IDCR Was Violated

The differences in the retention time and relative retention time of the target analytes in GC/MS phase and the GC/C/IRMS phase of the IRMS test of appellant's Sample A and Sample B from Sample 995474 were well in excess of the differences permitted by WADA TD2003IDCR. In some cases, the difference in the relative retention time was nearly nine times the permitted difference. Presentation of Dr. Meier-Augenstein ("Meier-Augenstein Presentation") at Slide 24; Closing Presentation at Slide 26. These violations are not a mere technicality, but rather directly affected whether LNDD properly identified the target analytes of testosterone. Simply put, LNDD cannot establish that the isotopic values used to support the AAF were indeed from a testosterone metabolite – the isotopic values could be from a substance

that bears no relation to any of the target anayltes (and therefore no relation to testosterone). The failure to properly identify these target analytes renders LNDD's IRMS test results unreliable and inaccurate.

F. Possible Reasons Why The Retention Time/Relative Retention Time Were So Far Off

Once appellant has established the violation of WADA TD2003IDCR – as he has – it becomes appellee's burden to demonstrate to a comfortable satisfaction that the violation did not cause the AAF. Although it is appellee's burden to explain why the relative retention time issue do not comply with the WADA TD2003IDCR, appellant will now explain why the retention time and relative retention times were substantially different.

1. LNDD Used Completely Different Method Files For GC/MS And IRMS Testing

To use retention time/relative retention time to identify compounds in a separate GC/MS instrument and IRMS instrument, as LNDD did, it is critical that the conditions under which both GCs operate are the same. These conditions include a number of factors, but most importantly, temperature. Temperature, and to a lesser extent, flow rate, govern the rate at which compounds will elute, in other words, pass through the column. Simply put, temperature is the primary variable that determines how long a given compound stays in the stationary phase. For any one compound, as a general rule, the higher the temperature the less time a compound spends in the stationary phase.

The temperature and flow rate are conditions that are set in the GC method file. The method file is an electronic program that instructs the GC on all aspects of its operation.

Therefore, in order to ensure proper identification in this case, the method files in the GC/MS and the GC/C/IRMS should have been identical.

They were not. The method files for the GC/MS and the GC/C/IRMS runs that tested Sample 995474 show dramatically different conditions. For the GC/MS, the GC method files show the following:

- The column is conditioned at 70 C for one minute;
- The temperature is then ramped up to 270 C, increasing 30 C every minute; and
- The temperature is then ramped up to 300 C, increasing 10 C every minute.

This dramatically differs from the method file for the GC/C/IRMS. For the GC/C/IRMS, the GC method file is as follows:

- The column is conditioned at 70 C for one minute;
- The temperature is then ramped up to 270 C, increasing 30 C every minute;
- The temperature is then ramped up to 280 C, increasing 0.6 C every minute;
- The temperature is then held at 280 C for three minutes;
- The temperature is then ramped up to 300 C, increasing 5 C every minute; and
- It is then held at 300 C for 5 minutes.

Notably, these programs are the same up until the temperature of each system reaches 270 C. After that, they differ dramatically. The result of this difference is that the RT and RRT (but not the order) of each elutant, or target analyte, is not comparable between the two systems. Again, the failure of LNDD to properly use its instruments has resulted in inaccurate and unreliable test results.

2. LNDD Used Different Columns In Its GC/MS And IRMS Phases

a. The Use of Two Different Columns Makes Calculation of Accurate Relative Retention Times Impossible

The column is the piece of equipment in the gas chromatograph that performs the critical function of separating the compounds. Columns are manufactured by various makers, and are replaceable. When using a separate GC/MS and GC/C/IRMS, as was the case here, the columns must be identical. The reason is simple – unless the columns are the same, the amount of time it takes for a compound to elute between the GC/MS and GC/C/IRMS will be so different that the retention times and relative retention times will not be comparable. Furthermore, different columns can even change the order in which compounds leave the column. *See* Skogsberg, U. et al., Investigation of the Retention Behavior of Steroids with Calixarene-based Stationary Phases by Modern NMR Spectroscopy, *Journal of Separation of Science,* vol. 26, pl 1119-24 (2003)

Indeed, the Majority Panel apparently recognized the importance of using the same column in the GC/MS and GC/C/IRMS phases, because in attempting to support its conclusions, the Majority Panel explicitly stated, "The GC Column is, of course, the same in both instruments." Majority Award, at para. 186.

The evidence at the AAA hearing reflects that this assertion is simply not true. In this case, LNDD used two different columns, with different characteristics. The column used in the GC/MS phase was Agilent 19091s-433. *See* USADA 0124, 0303.

Part Number	Description
19091S-433	HP-5MS, 0.25mm * 30m * 0.25um

Part number 19091s-433, as documented on USADA0124 and USADA0303, is the HP-5ms column, as documented at the Agilent website.[22]

Agilent describes its 19091s-433 column as a **non-polar** column with stationary phases comprised of 5% phenyl, 95% methyl-polysiloxane. However, the column used in the GC/C/IRMS phase was an Agilent DB-17ms column. *See* USADA 0153. The manufacturer classifies this column as a **midpolarity** column with stationary phases comprised of (50% phenyl)-methyl-polysiloxane.

Changes in polarity in the stationary phase of the column can effect changes in (1) compound retention time and (2) the order in which compounds elute from the column itself. *See* Skogsberg, U. et al., Investigation of the Retention Behavior of Steroids with Calixarene-based Stationary Phases by Modern NMR Spectroscopy, *Journal of Separation of Science,* vol. 26, p. 1119-24 (2003). Therefore, the use of the different columns renders the IRMS test results inaccurate and unreliable and contributed to the impermissible differences in RT and RRT in this case.

Further, USADA's attempt to overcome the retention time and relative retention time deficiencies described above by asserting that the Mix Cal Acetate can be used to calculate retention time or relative retention time is specious, at best. USADA attempted to make this argument during the Appealed Case by calling Dr. Brenna on rebuttal on May 23, 2007. At that

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22 http://www.chem.agilent.com/ecommerce/product/Product_Catalog_3.aspx?prod_search=19091S-433&Pid=32486. Accessed Oct. 14, 2007.

time, Dr. Brenna suggested that retention times could be calculated from the Mix Cal Acetate. Specifically, he stated:

> Q: I'm asking whether or not you can calculate the relative retention time off the mix cal acetate in this case. The mix cal acetate formulation used in this case. Yes or No?
>
> A. Yes.

Tr. of R. 1957:13-19.

However, Dr. Brenna was later forced to admit on cross-examination that his previous testimony was misleading because it is not possible to calculate the relative retention time in this case from the Mix Cal Acetate. The reason is simple – the following metabolites – 5 Alpha, Pdiol, and Andro (which are the key metabolites) – are not in the Mix Cal Acetate. *Id.* at 1958:1-3. When this was pointed out to him, he admitted "you cannot calculate a relative retention time from the mix cal acetate . . . I'm sorry."[23] Therefore, it is undisputed that the Mix Cal Acetate cannot be used to identify 5 Alpha, Andro and 5 Beta by relative retention time.

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[23] This was one of many material inconsistencies between Dr. Brenna's direct testimony and what was revealed on cross-examination, all of which directly impacts his credibility. Dr. Brenna's testimony on May 23, 2007 was inconsistent with his earlier testimony given on May 14, 2007. Before the testimony of Dr. Meier-Augenstein, Dr. Brenna testified that retention times and relative retention times of the target analytes on GC/MS were essential to the identification of the same compounds on IRMS. Tr. of R. 255:16-22. This directly contradicted his later testimony that the retention time and relative retention times from GC/MS are not expected to match within .2 minutes or 1% of the retention times and relative retention times on the IRMS. *Id.* at 1962:7–1965:25.

In addition, Dr. Brenna testified on direct examination that the retention times and relative retention times for the target analytes would not match if performed on different instruments. *See id.* at 1933:12-16. However, Dr. Brenna later admitted on cross-examination that he would not expect to observe differences between retention times and relative retention times of the magnitude seen in this case. Tr. of R. 1969:6-1970:23.

b. The Use of Two Different Columns is a Separate and Additional Violation of LNDD's Own Standard Operating Procedure

The use of two different columns violates the LNDD's own Standard Operating Procedure ("SOP") governing GC/MS testing. LNDD's SOP governing GC/MS testing is the LNDD SOP M-AN-52, Analyse GC/MS—Confirmation Qualitative des Metabolites de Testosterone et de les Precurseurs, LNDD 00664. It clearly indicates that the DB-17ms column be used. Indeed, this makes perfect sense because LNDD's accreditation documents require that for GC/C/IRMS analysis, the DB17ms column be used. *See* LNDD0086 and LNDD 0098. These accreditation documents are from the months before and after the testing of Sample 995474:

				(2,1x150-5µm)		
EC31	Urine	Détermination de l'origine des métabolites et précurseurs de la testostérone par CG/C/IRMS CD : $\delta^{13}C$	Extraction SPE Dérivation Ac_2O/Pyridine Analyse IRMS	Isoprime Micromass Extractions : Cartouche C18/ 1-MeOH 2- CH_2CN 3-H_2O/ CH_2CN (F1-F2) CH_2CN (F3) Colonne : DB17MS (35m-0.25mm-0.25µm)	EC31-VA1 I-CONF-31 : M-Ex-24 M-An-41	Variation maximale admissible = 2‰
EC32A	Urine	Analyse qualitative de glucocorticoïdes CD : Trr, Abondances relatives des ions caractéristiques	Extraction SPE Analyse CLHP/SM/SM	ThermoFinnigan LCQ DECA Ionisation: ESI Extraction : Cartouche C18/ CH_2Cl_2 Colonne : ZORBAX RX-C8 (2,1x150-5µm)	EC32 V.A.2 I-CONF-32A : - M-Ex-29 - M-An-45	Liste des composés répertoriés dans le dossier de validation EC32 (V.A.2)
EC32B	Urine	Analyse qualitative de glucocorticoïdes CD : Trr, Abondances relatives des ions caractéristiques	Extraction SPE Analyse CLHP/SM/SM/SM	ThermoFinnigan LCQ DECA Ionisation: ESI Extraction : Cartouche C18/ CH_2Cl_2 Colonne : ZORBAX RX-C8 (2,1x150-5µm)	EC32 V.A.2 I-CONF-32B : - M-Ex-29 - M-An-45	Liste des composés répertoriés dans le dossier de validation EC32 (V.A.2)

LNDD0086

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EC31	Urine	Détermination de l'origine des métabolites et précurseurs de la testostérone par CG/C/IRMS CD : $\delta^{13}C$	Extraction SPE Dérivation Ac_2O/Pyridine Analyse IRMS	Isoprime Micromass Extractions : Cartouche C18/ 1-MeOH 2- CH_2CN 3-H_2O/ CH_2CN (F1-F2) CH_2CN (F3) Colonne : DB17MS (35m-0.25mm-0.25µm)	EC31-VA1 I-CONF-31 : M-Ex-24 M-An-41	Variation maximale admissible = 0,8‰
EC32A	Urine	Analyse qualitative de glucocorticoïdes CD : Trr, Abondances relatives des ions caractéristiques	Extraction SPE Analyse CLHP/SM/SM	ThermoFinnigan LCQ DECA Ionisation: ESI Extraction : Cartouche C18/ CH_2Cl_2 Colonne : ZORBAX RX-C8 (2,1x150-5µm)	EC32 V.A.2 I-CONF-32A : - M-Ex-29 - M-An-45	Liste des composés répertoriés dans le dossier de validation EC32 (V.A.2)
EC32B	Urine	Analyse qualitative de glucocorticoïdes CD : Trr, Abondances relatives des ions caractéristiques	Extraction SPE Analyse CLHP/SM/SM/SM	ThermoFinnigan LCQ DECA Ionisation: ESI Extraction : Cartouche C18/ CH_2Cl_2 Colonne : ZORBAX RX-C8 (2,1x150-5µm)	EC32 V.A.2 I-CONF-32B : - M-Ex-29 - M-An-45	Liste des composés répertoriés dans le dossier de validation EC32 (V.A.2)

LNDD0098

Date de prise d'effet : 15/12/2006

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As made clear from LNDD's SOP and accreditation documents, LNDD itself recognizes that the same columns must be used in both the GC/MS and IRMS tests.

G. The Majority Panel Erred In Its Analysis Of Retention Time And Relative Retention Time

1. WADA TD2003IDCR Does Not Apply To Only One Instrument

The Majority Panel, in finding that no ISL violation occurred with respect to retention time and relative retention time, found that the WADA TD2003IDCR does not apply to retention times from two instruments:

> However, it must be noted, **that TD2003IDCR does not apply to RRTs between two different and separate instruments that are not of the same type.** Therefore, Dr. Meier-Augenstein misdirected himself in his testimony before the Panel by comparing RRTs not between two GC/MS or two GC/C/IRMS instruments, but instead between one GC/MS and one GC/C/IRMS.

Majority Award, para. 182. Again, this is incorrect and without any support in the evidence produced at the arbitration, even appellee's own witnesses. First and foremost, the Panel's position that Dr. Meier-Augenstein's analysis was incorrect is contradicted by the (1) the Panel's own statement at paragraph 179, (2) appellee's witnesses, (3) appellant's witnesses, and (4) USADA's briefs. And, the fundamental reasoning of the Majority Panel is incorrect. TD2003IDCR must apply to two different separate instruments in order to ensure proper identification of the target analytes. Here, compliance with TD2003IDCR is of critical importance because of the fact that two different instruments are used by LNDD.

In finding that the TD2003IDCR does not apply, the Majority Panel stated that "two different instruments" could not have comparable retention times/relative retention times due to the length of "plumbing" in the GC/C/IRMS instrument. Indeed that panel stated that:

> After the sample passes through the GC portion of the GC/C/IRMS system there is an additional length of plumbing in the GC/C/IRMS machine adding a significant amount of time to the total RT of the substance.

Majority Award, at para. 184. Indeed, they provide the following hypothetical to illustrate their point:

> The additional time added to the RT of the analyte or standard in the IRMS will always be a constant time, regardless of the individual substances or compounds being measured. Consequently, the retention times of the compounds emerging from the GC/MS system cannot be the same as those coming from the GC/C/IRMS. Likewise, the RRTs will also be different. Taking the example used above, **if the RT from the GC/MS is 10 min for the target analyte and 5 min for the internal standard, in the case of IRMS, we may be adding an additional 1 minute for the combustion of those compounds to take place.** The reason that the additional time is the same for each substance/compound is that the substance or compound is no longer in its original form; they have been combusted completely to form CO2. As such, the RT for the target analyte at the end of the IRMS would be 11 min and the RT for the internal standard is 6 min. This results in a RRT of 11/6. Arithmetically speaking it is not possible for the RTs and the RRTs to be identical in the GC/MS and GC/IRMS systems nor can it be ensured that it will be within TD2003IDCR.

Majority Award, at para. 185.

The "plumbing" referred to by the Majority Panel is the tubing that connects the GC to the combustion chamber to the IRMS and does not contain any stationary phase. Because there is no stationary phase, all substances pass through this "plumbing" at the same rate.

The Majority Panel is incorrect that this will add a "significant" amount of time (certainly not the amount of time seen in the retention times in this case). Leaving the issue of whether a significant amount of time would added aside, however, the Majority Panel's reasoning is fallacious. As noted above, there is no reason to use the GC/MS instrument to identify the target analytes if it is expected that the retention time or relative retention time of the target analytes would not match or otherwise tie together. Such reasoning makes the use of the GC/MS test by LNDD in the IRMS process superfluous. Further, there is a well-established procedure to account for the plumbing in the GC/C/IRMS instrument which, because the additional time is a

constant, is to simply subtract the period of time that the compounds travel through the additional length of plumbing from the retention times of the compounds. Simply put, the retention time of the GC/C/IRMS phase is determined by subtracting the time the compounds spend in the additional length of plumbing. This function is performed automatically by the OS2 software. By default the software is set to subtract 30 seconds, but this can be changed by the operator to reflect the actual amount of time that is added. Dr. Davis indicated that he checked this figure and that it was set to the proper amount. This procedure, known as building the "adjusted retention time," resolves the issues raised by the Majority Panel.

Moreover, the Majority Panel's hypothetical does not support the conclusion that TD2003IDCR does not apply to two different instruments. It simply means that proper procedure must be followed with respect to the calculation of relative retention time. It is well-accepted that the "hold-up time" (called "delay time" in the OS2 software) – the time that is used by the compound traveling through the "plumbing" – is a constant time that is subtracted from the retention times when calculating relative retention time. Thus, in the hypothetical above, the 1 minute would have been subtracted, thereby allowing a comparison of 10 minutes to 10 minutes for the target analyte and 5 minutes to 5 minutes for the internal standard. The Majority Panel's Award reflects a fundamentally misunderstanding of proper procedure as it related to relative retention time.

2. Visual Comparison Of Peaks Between The GC/MS And IRMS Is Useless

The most critical error that the Majority Panel made in connection with the identification issue in GC/C/IRMS testing is that somehow, visual inspection of peak heights alone would

allow a laboratory technician to make the necessary identification. Paragraph 186 of the Majority Award states:

> Instead, the lab compares the peaks and the sequence of the peaks from the GC/MS and GC/C/IRMS to identify the metabolites and the endogenous reference compounds. Specifically, to identify the substances in question, one would compare the pattern of peak heights and retention times in the GC/C/IRMS chromatograms, anchored by the internal standard with a known RT, with the pattern of peaks heights and RTs in the GC/MS chromatograms obtained from the same aliquot of the sample.

Majority Award, at Para. 186.

First, such an argument again discards that fact that LNDD uses the GC/MS instrument as the means for identifying the target analytes. Second, because there is no retention time analysis in this case, the Majority Panel is clearly referring to visually comparing peak heights to establish identification. Apart from the fact that no ISL permits this "eyeballing" identification method (and WADA TD2003IDCR sets forth the proper identification method), comparison of peak heights from the GC/MS to GC/C/IRMS phase is without any support in the evidence or any recognized standards. Indeed, "eyeballing" the peak heights to try to identify the substances in the GC/MS phase with the substances in the GC/C/IRMS phase is illogical because the peak heights do not represent the same thing. In the GC/MS phase, peak heights are a function of ion current, whereas in the GC/C/IRMS phase, the peaks are proportional to the amount of carbon (in the form of CO_2) that has entered the ion source of the IRMS. These two measurements bear no relation to each other. Simply put, a technician cannot simply conclude that a "big" GC/MS peak is the same substance as a "big" GC/C/IRMS peak. Equally, a "little" GC/MS peak is not necessarily the same substance as a "little" GC/C/IRMS peak. As an example, a nitrogenous compound would not even appear on the IRMS because it lacks carbon.

The Majority Panel's analysis rests on assumptions that have no basis in the evidence at the arbitration, the testimony of USADA's own witnesses, in WADA standards, or in any other recognized method of identification procedure. Therefore, the conclusions based on these flawed assumptions – that the differences in the RT and RRT were acceptable – must be rejected.

IV.

OTHER FAILURES IN THE IRMS TEST

A. Failed Quality Control

LNDD's quality control methods provide no assurance that the IRMS instrument or the associated testing processes were precise, accurate, or reliable. Because LNDD's quality control measures were ineffective — and in some cases, by their own admission, deliberately manipulated — it is entirely possible, indeed likely, that the foregoing ISL violations caused the alleged AAF. Furthermore, the failure of LNDD's quality control measures eliminates any "safety net" that might otherwise suggest that the ISL violations and other improper laboratory practices did not cause the AAF.

LNDD identifies four quality control measures: (1) internal standard 5α-androstanol acetate, (2) negative control "blank urine," (3) positive control "mix acetate" and (4) an instrument performance check. *See* Ex. B to USADA's Response to Second Request for Production of Documents ¶ 4 at 8. USADA likewise identifies these same four measures as the appropriate quality control measures and uses these quality control measures as a basis for arguing that the IRMS test results for Sample 995474 are reliable. *See* USADA's Pre-Hearing Brief ¶¶ 53-58. But, as shown below, none of these quality control measures were effective or reliable and, therefore, they do not show that LNDD's test results can be accepted as accurate or reliable.

1. Internal Standard 5 Alpha AC Was Not Reliable Or Effective

The failure of LNDD to measure properly the internal standard, 5α-androstanol acetate ("5 Alpha AC"), establishes that the IRMS instrument was not accurate and cannot provide this panel with any quality control assurances. 5 Alpha AC is a substance that has a known isotopic quantity and is added to the Mix Cal Acetate, as well as to every Sample Fraction ("F1, F2, F3") and Blank Urine Fraction (Blank Urine 1, Blank Urine 2, Blank Urine 3 ("hereinafter "BLU 1, BLU2, BLU3"). Because there is a known isotopic value for this substance, if LNDD's testing process were accurate, LNDD should have identified 5 Alpha AC at a theoretic delta value of -30.46, within a measurement of error of .5 delta units. *See* Ex. 24, USADA0175. During the IRMS test for Sample 995474, however, LNDD measured 5 Alpha AC outside of its known isotopic value range in several of the fractions. Indeed, Dr. Meier-Augenstein demonstrated at the arbitration that 5 Alpha AC was measured outside of its acceptable isotopic values in Sample Fraction B3, the Sample Fraction LNDD states shows an AAF. *See* Meier-Augenstein Presentation at Slides 52, 54; Closing Presentation at Slides 39, 40, 134, 136. This means that LNDD's IRMS instrument could not accurately measure the known isotopic value of a compound.

The importance of accurately measuring the isotopic value of 5 Alpha AC is undisputed, and was stated by USADA's own expert, Dr. Brenna. Dr. Brenna identified 5 Alpha AC as an important quality control measure. Dr. Brenna stated, "It also has . . . [5 Alpha AC] that has been added to every sample that elutes early, and that standard is further checked to determine that the instrument is running properly during analysis of every particular sample. And then there were standards run after the sets of analytes. So there were standards at each level." Tr. of R. at 237:13-19. Put differently, one of the indicia of reliability for the IRMS test results is

whether the IRMS instrument was capable of accurately measuring known isotopic values in the quality controls. Here the IRMS test results are not reliable because of LNDD's failure to measure properly the internal standard during the testing of Sample 995474.

2. The Sample Blank Urine Control Fails

The Sample Blank Urines do not provide any quality control assurance. First and foremost, the blank urines do not contain known quantities of any substance – it is simply urine pooled from unknown persons. It therefore does not provide any ability to test or measure the GC/C/IRMS instrument's accuracy or precision. Further, as previously described, the internal standard 5 Alpha AC was determined to be outside of the measurement of uncertainty for the Sample B F3 fraction – the same fraction USADA relied upon to establish the AAF.

Furthermore, when the Blank Urine Samples were reprocessed on May 4–5, 2007 pursuant to this Panel's discovery order, the variability in the results was incredibly broad – to broad for it to be of any analytic use. Specifically, the B Sample 5 Alpha, when measured with automatic subtraction as opposed to the method used by LNDD, went from -1.6 delta-delta to -3.45 delta-delta, and the A Sample 5 Alpha went from -1.59 delta-delta to -3.65 delta-delta. The delta-delta variances between manual processing and automatic processing are too great (more than a 2 per mil difference) to provide any assurance that the blank urine provided effective quality control. This is especially important given that these blank urine fractions are the same fractions USADA relied upon to establish the AAF.

3. The Mix Cal Acetate Control Was Flawed

The Mix Cal Acetate is neither a positive control, as USADA claims, nor an effective quality control. A positive control must include known positives of all the target analytes to be effective. Here, the Mix Cal Acetate cannot serve as a positive control because it does not

contain three target analytes: 5 Alpha, Pdiol and Andro. Without these three key target analytes, there are no assurances that the IRMS instrument can accurately measure these substances. Accordingly, only one of the four delta-delta values, Etio – 11-ketoetio, can be measured with any confidence. The Etio – 11-ketoetio delta-delta value, for both the A Sample and the B Sample from Sample 995474, however, was never an issue in this case because its delta-delta values were negative under the WADA positivity criteria and LNDD positivity criteria.

Furthermore, the Mix Cal Acetate cannot serve as an effective quality control measure because the Mix Cal Acetate is a "clean matrix." As a clean matrix, the Mix Cal Acetate contains only 5 Alpha AC, Etiocholanolone AC, 5 Beta Androstanediol diAC, 11-ketoetio AC and a solvent. In short, there are no other unidentified substances in the Mix Cal Acetate that could create interference. In contrast, urine is an exceptionally complex matrix, which means that it contains a number of unidentified compounds. As a result, even though the IRMS instrument correctly measured the isotopic value of the known compounds in the Mix Cal Acetate, this, standing alone, does not establish that the IRMS instrument can accurately identify and measure these compounds in a dirty, or complex, matrix with substantial interference. The difference between the Mix Cal Acetate's clean matrix and urine's dirty matrix is evident simply by looking at the chromatograms. A chromatogram for the Mix Cal Acetate shows no interference and has easily identifiable and defined peaks, whereas, the chromatograms for the Sample and Blank urine fractions shows substantial interference and undefined peaks. *Compare* Ex. 24, USADA0184 *with* Ex. 24, USADA0173; Ex. 25, USADA0349. The testimony of Dr. Meier-Augenstein is compelling in this regard. Dr. Meier-Augenstein describes the chromatographic analysis of the Mix Cal Acetate as similar to "shooting fish in a barrel," unlike the related analysis of human samples. Tr. of R. at 1452:8-13.

4. Instrument-Performance Checks Show A Lack of Linearity

LNDD's assertion that it conducts instrument checks is meaningless because despite these checks, the evidence establishes the machine was not working properly at time Sample 995474 was tested. In particular, the evidence establishes that LNDD's IRMS instrument was not linear. Linearity is the ability of an IRMS instrument to accurately quantify the isotopic ratio of each testosterone metabolite and endogenous reference compound in different samples regardless of their concentration. In other words, linearity is the ability of the instrument to accurately measure isotopic ratios in different samples that have various concentrations of the target analyte. Linearity is critically important to the accuracy and reliability of an IRMS instrument because it ensures that the instrument will measure the isotopic ratio of a target analyte accurately in samples whether there is a large amount of the analyte present or a small amount.

In this case, the linearity tests were not done pursuant to LNDD's Standard Operating Procedures ("SOP"). LNDD's SOP dictates that linearity runs must be performed once per month. *See* Ex. 26, LNDD0161-0187. They were not. LNDD's linearity testing dates were: (1) June 26, 2006, roughly one month before the Stage 17 A Sample was tested (Ex. 26, LNDD0313, 0315, 0317), (2) July 31, 2006, roughly one week after Mr. Landis's A Sample was tested (Ex. 26, LNDD0320, 0322, 0324) and (3) September 25, 2006, roughly a month-and-one-half after Mr. Landis's B Sample was tested (Ex. 26, LNDD0327, 0329, 0331) (Ex. GDC00522, IsoPrime Manual Section 6, Page 31, "Checking the System") (describing how to perform the linearity tests).

Further, this is not a mere technical deficiency, the testimony of Dr. Simon Davis shows that LNDD's IRMS instrument was not linear. Dr. Davis explains that the IsoPrime1 instrument "drifted in and out of linearity, and . . . there was also a degree of uncertainty as to how unlinear

it was, because they [LNDD] did not do the tests properly over the full range." Tr. of R. at 1782:11-15.

Most importantly, the IsoPrime1 instrument was not linear under the specifications provided by GVI. Dr. Davis testified that the linearity on the IsoPrime1 instrument must be "equal or less of .3 [per mil]" to be within specification. Tr. of R. at 1986:9-10. Further, Dr. Davis explained that the instrument must be linear over the full range in the spectrometer from 1E minus 8 amps down to 1E minus 9 amps such that the isotopic value for the same compound should not deviate by more than .3 per mil. But LNDD's IsoPrime instrument ***did vary*** by more than .3 per mil for the linearity runs done on June 26, 2006, just before the testing of the A Sample in this case. *See* Ex. GDC01367. The IsoPrime1 that was used to perform the IRMS test for Sample 995474 did not meet the linearity specifications required by the manufacturer and, thus, the AAF based on test results from this machine are unreliable.

5. LNDD's Quality Controls Are Meaningless Because They Were Not Performed Properly

The IRMS testing process, and the effective use of quality controls, requires that the Mix Cal Acetate and other quality controls be run in a specific sequence and without manual interruption. In other words, the temporal and sequential relationship between the quality controls and the testing of the sample and blank urine fractions is critical. Failure to adhere to these temporal and sequential relationships renders the quality controls meaningless. Indeed, USADA acknowledged the paramount importance of the temporal relationship between the quality controls and sample and urine fractions when it asserted repeatedly that the quality controls were run "immediately before and immediately after" or "minutes before and minutes after" Mr. Landis's A and B Samples. *See* USADA Pre-Hearing Brief ¶ 79 ("The Mix Cal

Acetate results from the controls run immediately before and immediately after Respondent's A and B samples . . . ."); USADA Response Brief ¶ 27 ("In its Pre-Hearing Brief, USADA went into considerable detail to explain how the Mix Cal Acetate, Blank Urine and Mix Cal IRMS controls run in the same sequence minutes before, during, and minutes after Respondent's sample. . . ."). USADA's assertions, however, are patently false. There is substantial, and uncontroverted, evidence that the quality controls here were not run within minutes of testing Sample 995474.

LNDD's own document package shows that ***there was a five-hour fourteen-minute gap*** between the running of the Sample A F2 fraction of Sample 995474, *see* Ex. 24, USADA0166, and the running of the Mix Cal Acetate. Ex. 24, USADA0183. The expected time gap between the initiations of these two injections is 45 minutes - the time it takes to process the Sample A F2 fraction. Such a significant gap eliminates the effectiveness of the quality controls because the quality controls were not run under the same condition as the sample run. The summary chart can be seen at Closing Presentation at Slide 42. In testing the B Sample of Sample 995474, LNDD again failed to run quality controls within the proper time frame. LNDD's document package again shows that there was a ***four-hour forty-minute gap between the running of the first Mix Cal Acetate***, Ex. 25, USADA0362, and the running of the Sample B F3 Blank Urine of Sample 995474. Ex. 25, USADA0347. The summary chart can be seen at Closing Presentation at Slide 45. Accordingly, the quality controls provide no assurances.

Incredibly, Ms. Mongongu, when pressed to explain these gaps, testified that she forgot to add the Mix Cal Acetate to the A Sample and had to rerun the Samples – yet, there are no documents to reflect these runs. *See* Tr of R. at 600:20-601:3. Ms. Mongongu also testified that

she could not remember what happened during the gap in the testing of the B Sample. Tr. of R. at 608:5-8.

In totality, the failure of LNDD's quality control measures should give the Panel no assurance in the accuracy or reliability of LNDD's test results.

B. Poor Chromatography

Good chromatography is the key to reliable IRMS test results. LNDD's chromatography is not sufficient and is in violation of ISL 5.4.4.2.1 because LNDD failed to properly generate chromatograms that avoided interference in the detection of the prohibited substances or their metabolites and markers by components of the sample matrix. USADA has no compelling evidence to carry its burden that LNDD's failure to comply with ISL 5.4.4.2.1 did not cause the AAF. LNDD's poor chromatography should give this Panel no confidence in the accuracy or reliability of LNDD's GC/MS or IRMS findings because the quality of chromatography directly impacts the test results.

There is overwhelming scientific support for the principle that good chromatography is critical to accurate results. Such support can be seen in the peer-reviewed literature referenced during Dr. Meier-Augenstein's testimony. *See* Meier-Augenstein Presentation at Slide 5; Ex. GDC01297.

ISL 5.4.4.2.1 requires that:

> Confirmation methods for Non-threshold Substances must be validated. Examples of factors relevant to determining if the method is fit for the purpose are: Matrix interferences. The method should avoid interference in the detection of Prohibited Substances or their Metabolites or Markers by components of the Sample Matrix.

This ISL applies to the determination of the AAF in this case.

In support of, but independent of, the violation of ISL 5.4.4.2.1, poor chromatography has a direct effect on the accurate, or inaccurate, determination of isotopic values (for the IRMS test) and the quantification of testosterone, epitestosterone and the T/E ratio (for the T/E test).

Matrix interference and poor chromatography can result in dramatic swings in isotopic values, as shown in the study of marine organisms described. *See* Meier-Augenstein Presentation at Slides 28-30. Further, even small coeluting peaks, peaks representing compounds that have the same retention times, can have a substantial isotopic effect on larger peaks. An example of this was Exhibit 120 (a demonstrative exhibit that USADA's counsel asked Dr. Meier-Augenstein to prepare). This exhibit proved that even a small coeluting peak could have more than a -2 per mil effect on the target peak, where the isotopic value of the smaller peak was a hypothetical -70 per mil.

Dr. Meier-Augenstein's testimony furthers explains that IRMS peaks could have been incompletely combusted and the isotopic values of those peaks could be as low as -700 per mil. Tr. of R. at 1488:14-1489:23. Indeed, as Dr. Meier-Augenstein pointed out, the isotopic values for the background were more negative than -120 per mil in several of Respondent's samples. Tr. of R. at 1489:19-23.

Poor chromatography, especially of the kind present in this case, substantially affects the accuracy and reliability of LNDD's GC/MS and IRMS findings, and USADA has introduced no credible evidence to rebut this presumption. In fact, USADA cannot introduce such evidence because LNDD's poor chromatography did in fact contribute to the inaccurate results in this case. Dr. Meier-Augenstein provided evidence showing that the following chromatograms were so poor that they resulted in inaccurate and unreliable IRMS results for Sample 995474:

The chromatogram at Exhibit 24, USADA0173 (Sample A, Fraction 3). *See* Tr. of R. at 1433:18-1434:9.

The chromatogram at Exhibit 25, USADA0349 (Sample B, Fraction 3). *See* Tr. of R. at 1416:9-1417:10.

Likewise, Dr. Davis concluded that the following chromatograms were so poor that they resulted in inaccurate and unreliable IRMS results for all of the other alleged AAF from other Tour stages:

Stage 11: The chromatogram at Exhibit 88, LNDD1110 (Sample B, Fraction 3). *See* Tr. of R. at 1848:7-1849:9.

Stage 15: The chromatogram at Exhibit 86, LNDD0894 (Sample B, Fraction 3). *See* Tr. of R. at 1850:23-1851:10.

Stage 19: The chromatogram at Exhibit 87, LNDD0991 (Sample B, Fraction 3). *See* Tr. of R. at 1851:11-1852:10.

Stage 20: The chromatogram at Exhibit 84, LNDD0704 (Sample B, Fraction 3). *See* Tr. of R. at 1852:11-1853:8.

It was not just appellant's witnesses that support this argument, LNDD's own witnesses demonstrated the poor quality of the chromatography in the IRMS testing. LNDD witness Ms. Mongongu admitted that there was matrix interference around the internal standard. *See* Tr. of R. at 615:10-17. Worse still, the evidence shows even more matrix interference surrounding the target analytes. *See* Ex. 24, USADA0173; Ex. 25, USADA0349; Ex. 84, LNDD0704; Ex. 86, LNDD0894; Ex. 87, LNDD0991; Ex. 88, LNDD1110.

Dr. Catlin's testimony also confirms LNDD's poor chromatography in its IRMS testing. On cross-examination, Dr. Catlin agreed that the chromatography in some of the tests supporting

the adverse analytical findings were "not good." Tr. of R. at 1213:8-13; *see also id.* at Tr. of R. at 1213:9-13 (conceding that some of the chromatograms were poor); Tr. of R. at 1229:1-1230:25 (described some of those chromatograms as having a grade of C or lower), Ex.-86, LNDD0894; Ex. 88, LNDD1110.

Further, the witnesses called by USADA did not provide probative evidence to support the assertion that the chromatography is sufficient because the testimony of the WADA laboratory directors is inherently conflicted, the testimony lacked specificity as to the issues raised by Mr. Landis, and the testimony was largely inconsistent. *See* discussion below.

In response to testimony about poor chromatography, USADA called Dr. Brenna to testify that, notwithstanding the interference shown by a visual examination of the F3 B Sample chromatogram, the two-over-one trace graph for the F3 B Sample showed good peak separation and a flat background. *See* Tr. of R. at 268:2-269:9. This testimony was offered for the purpose of assuring that there was no effect from matrix interference. However, it is unpersuasive because Dr. Meier-Augenstein showed that the two-over-one trace cannot provide the assurance that Dr. Brenna described. This is because to understand the effect of matrix interference, the technician must also account for the actual change (i.e., rise) in background from the measurement of the internal standard — in this case, 5 Alpha Androstanol AC — to the Pdiol peak in the F3 Sample. Dr. Meier-Augenstein illustrated this point with two summary charts; *see* Meier-Augenstein Presentation at Slides 17-18, showing that the background ***was not flat,*** as it changed by more than four per mil between the internal standard and the pregnandiol.

The summary charts prepared, and testified to, by Dr. Meier-Augenstein are slides 17 and 18 from his presentation. Each summary chart shows that, for both Sample A and Sample B, the fraction on which USADA focused – the F3 fraction – had a high downward-sloping baseline,

which directly effects peak integration, and makes comparisons of peaks impossible (especially through "eyeballing," as was done here). These summary charts are reliable because they do not rely upon a subjective evaluation of the quality of the chromatograms, but rather constitute a representation of background points over the retention times shown in each of the relevant chromatograms.

Notably, on cross-examination Dr. Shackleton conceded that he could not prove "that the matrix interference . . . did not affect the adverse analytical result." Tr. of R. at 216:12–217:23. Thus, USADA's evidence fails to show that poor chromatography did not cause the AAF.

The chromatography supporting the AAF for Stage 17, as well as the chromatography for Stages 11, 12, 15 and 20, was poor and, therefore, the IRMS test results are inaccurate and unreliable.

C. Breakdown Of Testosterone Is Inconsistent With Known Science

LNDD's IRMS results show a breakdown of testosterone that is inconsistent with both the peer-reviewed literature and the science of testosterone metabolism. The testosterone isotopes 5 Alpha and 5 Beta share the same carbon skeleton and, therefore, their isotopic values should be consistent. In particular, when influenced by of the administration of exogenous testosterone, their values should rise and fall together.

Here, LNDD's IRMS results for Sample A report that there is a difference of -3.99 per mil between the 5 Alpha – Pdiol value and the 5 Beta – Pdiol value. *See* Ex. 107. Likewise, the IRMS test results for Sample B show a difference of -3.74 per mil between the 5 Alpha – Pdiol and the 5 Beta – Pdiol value. *Id.* (A summary chart detailing this information was made part of Dr. Meier-Augenstein's Presentation at Slide 82.). These differences are ***far greater*** than the differences between the testosterone metabolites found in ***any*** peer reviewed study. Indeed, the

Shackleton study (Exhibit 40, USADA1245) shows the greatest difference for test subjects between 5 Alpha and 5 Beta is -2.5 per mil delta-delta. Likewise, the Aguilera study (Exhibit 40, USADA1229), shows that the greatest difference between 5 Alpha and 5 Beta for control subjects was -1.39 per mil. Even the non-peer reviewed Cologne Study, relied on by USADA, shows no differences as large as those reported in this case. *See* Ex. 34. LNDD's IRMS test results for Sample 995474 are anomalous, unreliable, and inconsistent with the known science of testosterone metabolism and raises substantial questions as to the reliability of these results.

Moreover, LNDD's IRMS test results for the retesting of Sample 994075 (Stage 15), Sample 994080 (Stage 19) and Sample 994171 (Stage 20) are similarly inconsistent with the known science regarding the metabolism of testosterone and, therefore, are unreliable. These results are inconsistent with the metabolism of testosterone because they each exhibit a difference between the 5 Alpha and 5 Beta values of -1.5, -3.13 and -3.54, respectively. Again, these differences are far greater than the maximum difference seen in many peer-reviewed studies. A summary chart of these values is at Exhibit GDC01363.

The IRMS test results for Sample 995474 and the other samples tested are also inconsistent with known science because the overall pattern shown by the IRMS test results and the T/E test results for the corresponding Sample inconsistent with both the peer-reviewed literature, and the known effect of testosterone. In fact, Dr. Amory testified that the T/E results do not "look like anything we've seen in studies of men who have been administered exogenous testosterone." Tr. of R. at 1586:11-13. USADA has no evidence to contradict this fact; indeed, at the arbitration, USADA only introduced the anecdotal testimony of Joe Papp, a known performance enhancing drug user who used testosterone while also using several other

substances. USADA has no credible or relevant evidence to contradict the fact that the IRMS test results here are not consistent with known science.

The IRMS test results are also inconsistent with known science because, as explained by Dr. Amory, Mr. Landis's leutenizing hormone ("LH") values, as shown before and after July 23 (Stage 20), are inconsistent with the chronic use of testosterone. *See* Tr. of R. at 1550:1-1552:13; see also Ex. GDC00620. Dr. Amory's testimony with respect to LH has never been contested.

The IRMS test results are also anomalous, and therefore unreliable, because the alleged doping would have been inconsistent with common sense. LNDD's last-stage test results (Sample 994171) suggest that Mr. Landis took testosterone on the final Tour de France stage into Paris — a stage that is not typically contested for a Tour leader, and was not contested in 2006 when Landis had a 1-minute lead.

The anomalous results produced by these tests, in total, corroborate the lack of accuracy and reliability in LNDD's testing processes.

V.

SYSTEMATIC MANIPULATION, DESTRUCTION, AND MANUFACTURING OF EVIDENCE ALSO COMPELS DISMISSAL OF ALL DOPING ALLEGATIONS

A. LNDD'S Manual Processing Of Data Violated The ISL And Affected The Result

As noted above, there is undisputed evidence that LNDD personnel manually processed the IRMS results for Sample 995474. Such manual processing violated several ISL provisions, including: (a) ISL 5.4.4.4.1.4, which requires that data entry be recorded with an audit trail, and

(b) ISL 5.2.6.1, which requires that the laboratory document procedures to ensure a coordinated record related to each analyzed sample. There is no evidence that the violation of ISL 5.4.4.4.1.4 and ISL 5.2.6.1 did not cause the AAF in this case.

Manual processing is the process by which LNDD's technicians manually adjusted the start and end points of the peaks and added and deleted background points in the chromatograms associated with Sample 995474. The widespread use of manual processing in this case was necessitated by the poor chromatography. *See, e.g.*, Tr. of R. at 743:15-744:5.

The technician's ability to pick and choose where to begin and end each peak has a tremendous – and determinative – effect on the final delta-delta values, such that it can cause an isotopic value to vary from a negative to a positive finding. As noted by Dr. Davis at the arbitration, using the same software as the software by LNDD – OS2 – one can make a negative sample into a positive sample rather easily. For this reason ISL 5.4.4.4.1.4. requires that:

> All data entry, recording of reporting processes and all changes to reported data shall be recorded with an audit trial. This shall include the date and time, the information that was changed, and the individual performing the task.

It is undisputed that LNDD applied manual processing to achieve the IRMS test results that constitute the AAF associated with Sample 995474. *See* Tr. of R. at 724:11-17. However, LNDD violated ISL 5.4.4.4.1.4 by failing to record, at any point, the calculations or data entry associated with the samples in question in this case.

Further, as made clear in Dr. Davis's testimony, the OS2 software on the IsoPrime1 was able to print and record data and results. *See* Tr. of R. at 1882:9-22; 1874:23-1875:13. Thus, had LNDD wanted to comply with the ISL, it could have.

Although it is now undisputed that LNDD manually processed the IRMS test results, USADA during pretrial proceedings provided contrary evidence. For example, in his Second

Request for Production of Documents at Request No. 10, Mr. Landis requested: "All DOCUMENTS that relate to the creation and accuracy of the background subtraction method used by LNDD in the IRMS test." In response, LNDD provided the following answer: "Background Subtraction is embedded in the instrument software, which is proprietary to the instrument manufacturer. LNDD has no separate documentation." *See* Ex. B to USADA's Response to Respondent's Second Request for Production of Documents ¶10 at 10. This is simply not true because LNDD does in fact have separate documentation relating to the removal of background subtraction; i.e., LNDD's SOP for the IRMS test. Additionally, Mr. Landis also served the following interrogatory: "Please explain, with mathematical formulas, how LNDD performed and applied background subtraction to sample 995474 and related controls." First Request for Production of Documents § II ¶ 8. In response, LNDD answered: "See response to second request, C10." *See* Ex. C to USADA's Response to Respondent's Second Request for Production of Documents ¶ 8 at 2. The response cited by LNDD reads: "Background Subtraction is embedded in the instrument software, which is proprietary to the instrument manufacturer. LNDD has no separate documentation." *See* Ex. B to USADA's Response to Respondent's Second Request for Production of Documents ¶ 10 at 10. This answer misleadingly indicated that background subtraction was not done manually, but rather by the instrument software. This response was not true, because LNDD used a ***manual*** background subtraction method in obtaining the reported results for Sample 995474. *See* Tr. of R. at 724:15-17.

This manual processing had a dramatic effect on the final isotopic values in this case, further rendering those results inaccurate and unreliable. Indeed, during the May 4-5, 2007 reprocessing attempt, LNDD was unable to reproduce its original results using "manual

processing," even though the same technician attempted to achieve these results by working on the same machine that ran the original processing. These technicians tried more than 20 times to do so. A summary of LNDD's failed attempts to achieve the same initial results is shown at Exhibit GDC01365. A summary of the different results from the attempt to reproduce the original results is shown at Exhibit GDC01350.

The variation between these different methods is significant, often times greater than 2 per mil. *See* Ex. GDC01350. LNDD's failure to reproduce its original results, likely because the technicians cannot replicate the manual processing they performed on the original test, belies any reliability in the IRMS test results for Sample 995474. Indeed, even USADA's expert, Dr. Brenna, testified that the variation in the reprocessing results would cause him concern. Tr. of R. 359:17-24.

LNDD's manual processing and background subtraction techniques were inaccurate and unreliable here because many factors, including: (1) the inexperience of the LNDD technicians; (2) the evidence of other errors committed by LNDD technicians as proven during the arbitration; and (3) the great variation in the results achieved by LNDD technicians in this case. In particular, the lack of accuracy and reliability of the final isotopic data from LNDD's manual processing and background subtraction techniques highlights the need for compliance with ISL 5.4.4.4.1.4 and ISL 5.2.6.1. Such compliance is necessary to eliminate confusion about the methods which were used to achieve the IRMS values that constituted the alleged AAF.

The importance of complying with ISL 5.4.4.4.1.4 and ISL 5.2.6.1 is particularly evident in this case, given that LNDD technicians repeatedly discarded results that they felt were unacceptable. For example, on cross-examination:

- Cynthia Mongongu admitted that she re-ran and saved a sample with the same number – ***thereby deleting the initial run*** – because the initial run "was not correct." *See* Tr. of R. at 595:22.
- Claire Frelat admitted that, ***because she deleted over sample runs***, the only way to know that she had not done so for improper purposes was to take her word for it. *See* Tr. of R. at 714:17-24.

The fact that LNDD repeatedly deleted over these sample runs shows that they were having problems with equipment and/or problems with their quality controls. Simply put, the deletion of data is indicative of LNDD concealing the fact that its instrument was not operating as expected.

The importance of complying with ISL 5.4.4.4.1.4 and ISL 5.2.6.1 is particularly evident in this case given that LNDD had no formal training program for its technicians, thereby allowing individual technicians to employ different techniques and standards. Critically, they also failed to retain any data that would reflect the decisions they made, precluding any supervision by a more experienced IRMS technician. For example, on cross-examination:

Dr. Buisson stated that she was in charge of the chemistry department and supervised IRMS technicians, Tr. of R. 922:21-926:5, and had a PhD in IRMS. Tr. of R. 915:24-916:8.

Regarding the limited extent of her training of Claire Frelat, Buisson stated: "if she had any questions, I was there to answer them." Tr. of R. at 929:19-930:1.

Dr. Davis testified that, when he asked Cynthia Mongongu how she chose data points during manual reprocessing, she replied, "I'm using my experience." Tr. of R. 1841:14-15. But according to Claire Buisson, that experience was not a product of training. *See* Tr. of R. at 929:19-930:1.

Even the AAA Majority below recognized that the "practices of the Lab in training is employees appears to lack the vigor the Panel would expect in the circumstances given the enormous consequences to athletes of an AAF," and that the evidence in this case gave them "some cause for concern." Majority Opinion, ¶ 311.

Dr. Brenna testified that LNDD's technicians' manual processing technique as being very "mechanical and identical from run to run to run." Tr. of R. at 275:10-11. This testimony is simply inconsistent with the fact that the Laboratory technicians could not reproduce the original test results and is also inconsistent with the fact that LNDD repeatedly re-ran and deleted sample runs. *See, e.g.*, Tr. of R. at 1843:13-22; Ex. GDC01350.

B. Deletion Of Data

LNDD laboratory technicians deleted relevant data that was obtained during the testing process. LNDD technicians deleted test results they found to be "incorrect" or that "did not correspond." *See* Tr. of R. 712:14-714:11. In particular, LNDD technicians deleted test results related to LNDD's quality control steps, including, but not limited to, results from the Mix Cal Acetate and blank urine runs. When its technicians deleted data during the testing of Sample 995474 and during the retesting process, LNDD failed to comply with ISL 5.4.4.4.1.4, which requires that data entry be recorded with an audit trail. Likewise, when its technicians deleted data during the testing of Sample 995474 and during the retesting process, LNDD failed to comply with ISL 5.2.6.1, which requires that the laboratory have documented procedures to ensure a coordinated record related to each analyzed sample. Indeed, the fact that the destruction and deletion of data involved quality control measures further demonstrates that the IRMS test results for Sample 995474 are not reliable. Moreover, USADA cannot present evidence that the failure to comply with ISL 5.4.4.4.1.4 and ISL 5.2.6.1 did not cause the AAF in this case.

Admittedly, destruction of data does not always constitute an ISL violation. For example, there might not be a violation if sequence files were deleted, but the sequence was rerun in its entirety and the deletion was properly recorded. But that is not the case here. LNDD deleted data that was part of or related to the IRMS test results which were reported as an AAF.

The first example of data destruction occurred in Sample 995474. For both the A and B Samples, there was a summary page entitled "Batch Data Processing Results." This summary page contained values reflecting the individual test results from each of the tests conducted in the Sample A and Sample B sequences. For Sample A, the summary page is Exhibit 24, USADA0155. For Sample B, the summary page is Exhibit 25, USADA0359. Because the individual test results on the "Batch Data Processing Results" page ***do not match*** the results on the individual test pages that were included in the document package, it is clear that LNDD cherry-picked the results they wanted for both the Sample A and the Sample B sequences.

For Sample A, the results of the Mix Cal IRMS 003-2, Exhibit 24, USADA0179, ***do not match*** the results shown on the "Batch Data Processing Results" page. Ex. 24, USADA0155.

For Sample B, the results of the Mix Cal IRMS 003-3, Exhibit 25, USADA0359, ***do not match*** the results shown on the "Batch Data Processing Results" page. Ex. 25, USADA0331.

Also for Sample B, the results of the Mix Cal IRMS 003-2, Exhibit 25, USADA0358, ***do not match the results*** shown on the "Batch Data Processing Results" page. Ex. 25, USADA0331.

The second example of data destruction also occurred in conjunction with the testing of Sample 995474. For the Sample A and Sample B sequences, there are time gaps: 5 hours and 14 minutes and 4 hours and 40 minutes, respectively, which as noted above, should not have occurred because the injections are supposed to be run without any interruption. On cross-

examination, Claire Frelat testified that controls were rerun because the results were "not correct." *See* Tr. of R. at 595:14-22.

The third example of data destruction occurred in conjunction with the retesting process, during which the B Samples taken on July 3 (Sample 995642), July 11 (Sample 994203), July 13 (Sample 994277), July 14 (Sample 994276), July 18 (Sample 994075), July 22 (Sample 994080) and July 23 (Sample 994171) were tested. The IRMS testing of these samples was conducted on LNDD's IsoPrime2 instrument. The IsoPrime2 is able to retrieve a record of all operations performed in connection with the testing of a particular sample. These files, called "log files," were recovered for Sample 995642, Sample 994203, Sample 994277, Sample 994276, Sample 994075, Sample 994080 and Sample 994171. *See* Exs. GDC01056-01075.

The log files from the IsoPrime2 show numerous instances where LNDD technicians deleted data. The deletion of data occurred when an LNDD technician, either Cynthia Mongongu or Claire Frelat, saved over a file with an identical file name, thereby deleting the original data. Such deletions occurred on multiple occasions for the same file. Absent production of these log files, there would have been no indication that this data manipulation occurred. USADA's representative, Dr. Larry Bowers, resisted production of the log files reflecting these deletions, however, they were eventually produced only at the insistence of Dr. Botrè.

Additional examples of the destruction of data are set forth below:

- A sample run was saved at 11:48:08 and then saved over with the same file name at 12:05:22. This sample run at 12:05:22 was later saved over by a sample that was run at 12:32:50. Ex. GDC01056.

- A sample run was saved at 12:16:25 and then saved over with the same file name at 12:48:27. Exs. GDC01056-01057.
- A sample run was saved at 14:05:03 and then saved over with the same file name at 15:26:12. Ex. GDC01057.
- A sample run was saved at 20:17:24 and then saved over with the same file name at 21:08:36. Ex. GDC01058
- A sample run was saved at 08:45:36 and then saved over with the same file name at 08:48:14. The sample run saved at 08:48:14 was later saved over with the same file name at 8:59:07. Ex. GDC01069.
- A sample run was saved at 09:40:44 and then saved over with the same file name at 10:31:27. Ex. GDC01070.
- A sample run was saved at 09:56:19 and then saved over with the same file name at 10:47:05. Ex. GDC01070.
- A sample run was saved at 10:11:56 and then saved over with the same file name at 11:00:53. Ex. GDC01070.
- A sample run was saved at 13:53:00 and then saved over with the same file name at 13:55:43. Ex. GDC01073. The sample run saved at 13:55:43 was later saved over with the same name at 14:41:39. Ex. GDC01073

Indeed, USADA's own expert witnesses stated that, in their practice, they would not delete relevant data once it had been acquired. Dr. Catlin testified that the UCLA lab ***never*** deleted data after it was obtained, and that if he discovered that data had been deleted, he would investigate to determine why such a deletion occurred. Tr. of R. at 1237:7-1238:19. Dr. Ayotte, after cross-examination on the subject, also admitted that her laboratory deleted only control

samples that led to the IRMS instrument being corrected and were not part of any testing process involving a sample. Tr. of R. at 864:15-867:20; 907:21-25.

Despite the fact that their own expert witnesses testified to the importance of retaining all testing data, USADA and LNDD attempted to hide their destruction and deletion of data in both their repeated refusal to produce the log files and USADA's repeated statement that the quality controls for the A and B Samples were run either "immediate before and immediately after" or "minutes before and minutes" after the testing of the samples in the IRMS sequence. *See, e.g.*, Tr. of R. at 719:10-12. As shown above, this is simply not true.

The deletion of data and overwriting of files render the test results inaccurate and unreliable, and these techniques, as employed by LNDD, constitute a substantial deviation from proper laboratory practices and belie any reliability in the IRMS test results. *See* Tr. of R. 1818:15-17.

The destruction and deletion of data in both the testing of Sample 995474 and the B Samples from the other Tour Stages constitutes a violation of ISL 5.4.4.4.1.4 and ISL 5.2.6.1, and those deletions remove any assurance as to the accuracy or reliability of the final test results. Although USADA's witnesses, Claire Frelat and Cynthia Mongongu, offered explanations for a few of these deletions, their explanations are based solely on selective memory from weeks or months earlier, with no corroboration. Indeed, at several points during the testimony of both witnesses, their memories were shown to be selective, inconsistent or wrong. For example, Cynthia Mongongu remembered with precision all of the events, minute by minute, relating to the chain of custody from 10 months earlier, but testified on cross-examination that she could not remember the last ***year*** that the IRMS instrument needed servicing. Tr. of R. 509:8–516:20. With the future of the Tour de France winner on the line, no weight should be given to the

uncorroborated and inconsistent memories of these witnesses. The need for data, rather than memory, is precisely why following the ISL rules against data destruction are so important.

C. No Evidence of a Continuous Chain Of Custody

LNDD failed to comply with ISL 3.2 and WADA TD2003LCOC (Laboratory Internal Chain of Custody), which set forth the requirements of internal chain of custody within a laboratory. Moreover, USADA cannot, and has not, introduced compelling evidence to carry its burden that the failure to comply with ISL 3.2 and WADA TD2003LCOC did not cause the AAF in this case.

ISL 3.2 defines Laboratory Internal Chain of Custody as:

> Documentation of the sequence of Persons in possession of the Sample and any portions of the Sample taken for Testing. [Comment: Laboratory Internal Chain of Custody is generally documented by a written record of the date, location, action taken, and the individual performing an action with a Sample or Aliquot.]

WADA ISL 5.2.2.2 requires that:

> The Laboratory shall have Laboratory Internal Chain of Custody procedures to maintain control of and accountability for *Samples* from receipt through final disposition of the *Samples*. The procedures must incorporate the concepts presented in the *WADA* Technical Document for Laboratory Internal Chain of Custody (Annex C).

WADA TD2003LCOC, states in pertinent part:

> The Laboratory Internal Chain of Custody is documentation (worksheets, logbooks, forms, etc.) that records the movement of *Samples* and *Sample* Aliquots during analysis. . . .
>
> Within the Laboratory, the Laboratory Internal Chain of Custody shall be a ***continuous*** record of individuals in possession of the samples or *Sample* Aliquots. . . .
>
> In the case of *Samples*, the Laboratory Internal Chain of Custody should record all movement from receipt in the Laboratory through storage and sampling to disposal. In the case of Aliquots, the Laboratory Internal Chain of Custody should record all movement from preparation through analysis.

There is a substantive difference between the term "continual" and "continuous." To have "continuous" possession means without interruption, whereas "continual" means repeated possession.

Chain of custody must document all intra-laboratory transfers. Exs. GDC00219-00232. An impeccable chain of custody is necessary "[t]o ensure that the urine tested suffered no contamination, tampering, or mislabeling." Ex. GDC00222.

LNDD's chain of custody documents, Exhibit 25, USADA0253-0254, are summary reports that show the date, time and location of the sample at a given moment, but do not show a continuous record of intra-laboratory transfers of the A and B Sample bottles as required by the ISL.

Further, the individual documents presented by USADA to support the summary report do not suffice to create a proper chain of custody. These documents simply indicate that a laboratory technician performed a task involving the sample bottle at the time recorded; they do not establish ***when*** the sample bottle was transferred to the laboratory technician and from whom it was transferred.

Moreover, the complete failure to record ***both*** people to the transfer is fatal to USADA's position that there is no break in the chain of custody of the sample bottles because it requires the Panel to assume that the person previously listed on the summary report retained the sample bottle for the entire time before transferring the bottle to the person listed next on the summary report. In other words, in order to sustain USADA's position, the Panel must assume that there was no unrecorded transfer that took place between the two technicians listed on the summary report. For example, LNDD asks the Panel to assume that because its documentation shows that person "A" completed a task with the sample bottle at 9:00 and that person "B" performed a task

at 11:00 with the sample bottle, that a person "C" could not have obtained the sample bottle at 10:00 but did not record it. The Panel may not make such an assumption.

The following show no fewer than ***nine*** breaks in the intra-laboratory transfers of the Sample A and Sample B bottles and aliquots:

- On July 21, 2006, LNDD failed to record who removed the Sample A bottle from the refrigerator and when he or she did so. Ex. 25, USADA0253.
- On July 21, 2006, LNDD failed to record how the Sample A bottle was transferred from Martin in Salle 107 to Garcia in Salle 106, when the sample was transferred and where it was transferred. *Id.*
- On July 22, 2006, LNDD failed to record who removed that Sample A bottle from the refrigerator and when it was removed. Ex. 25, USADA0253.
- On July 22, 2006, LNDD failed to record how the Sample A bottle was transferred from Cerpolini in S. 103 to Mongongu in S. 104, which occurred sometime between 10:50 and 11:20, where it was transferred and when it was transferred. *Id.*
- On July 22, 2006, LNDD failed to record how the Sample A bottle was transferred from Mongongu in S. 104 to Cerpolini, which occurred sometime between 11:20 and 12:45, where the transfer occurred and when it was transferred. *Id.* (Ms. Mongongu testified that she had the bottle between 11:20 a.m. and 11:25 a.m. and that she gave it to Operator 18 at 11:25 a.m. However, the chain of custody document shows that Operator 18 had the bottle at 12:45 p.m., so there is no documentation showing the location of the bottle from 11:25 a.m. to 12:45 p.m.)

- On July 23, 2006, LNDD failed to record who removed the Sample A bottle from the refrigerator and when the transfer occurred. *Id.*
- On July 28, 2006, LNDD failed to record who removed the Sample B bottle from the freezer and where the transfer occurred. Ex. 25, USADA0254.
- On August 3, 2006, LNDD failed to record how, where and when the Sample B bottle was removed from the freezer. And, LNDD failed to record how, when and where the B sample bottle was transferred from Cerpolini in an unknown location to Frelat in S. 004, which occurred between 9:12 and 11:03. *Id.*
- On August 3, 2006, LNDD failed to record the transfer of the Sample B bottle from Frelat in S. 004 to Barlagne in S. 103. *Id.*
- Particularly significant is the testimony of Claire Frelat, who conceded that there was no documentation of intra-laboratory transfers: "The transfer is not recorded, it is not written." Tr. of R. at 687:14-20; *see* Tr. of R. at 688:7-18.
- Other WADA-accredited laboratories have chain of custody procedures that comply with the ISL because their chain of custody forms require that the person who had the bottle previously and the person who received the bottle be marked, which is in direct contrast to the procedures used by LNDD in conjunction with Sample 995474.

Specifically, the Montreal laboratory chain of custody document establishes the time, date and location of the bottle, who had the sample bottle and to whom the sample bottle was given. This is in contrast to LNDD, which only provides evidence of who had the sample bottle for a particular operation. Exs. GDC00030-00031. With significant time gaps between

operations, it is crucial to determine who was responsible for, and maintained, the sample at all times. This is particularly true where, as here, there are undisputed leaks within the LNDD lab.

Similarly, the UCLA laboratory chain of custody documentation records both parties to the intra-laboratory transfer, which, unlike LNDD, creates a ***continuous*** chain of custody. Exs. GDC00032-00033.

These breaks in the intra-laboratory chain of custody are ***not*** simply technical in nature. Nor can these breaks simply be resolved by stating that the Sample bottle was in a secured area in the laboratory and, thus, in a control zone. The clear reading of the ISL requires that if the bottle is not in the possession of the technician, it should be marked that it was left in a controlled area. This did not occur here. This should cause great concern with regard to the reliability of the test results for Sample 995474 because of several instances when a person was said to have physical possession of the Sample bottle for unexplained periods of time. The following explains illustrate this point.

On July 21, 2006, the A sample bottle was removed from the refrigerator at 7:25 and was not returned until 9:25, two hours later, during which time the only documented task completed was the creation of aliquots, a task that should take no longer than ten minutes. *See* Ex. 25, USADA0253.

On July 22, 2006, the A sample bottle was removed from storage at 9:05 and not returned until 12:45, over three and a half hours later. During these three and a half hours, the operators who purportedly had possession of the A bottle were conducting chemistry for both the T/E and IRMS tests, tasks that do not require use of the sample bottle for such a significant amount of time. *See* Ex. 24, USADA0119-0120, 0200.

On July 23, 2006, the A sample bottle was removed from the refrigerator at 14:20 and not returned until 17:00, over two and a half hours later. During this time, the aliquot for the second confirmation T/E test, which was the only reason for removing the bottle from storage, was completed at 15:00. Despite the A sample bottle being removed for a task that takes only five minutes, the bottle was not replaced until two hours after it was removed. *See* Ex. 24, USADA0079; Ex. 25, USADA0253, 0256.

In the examples above, the only reasonable explanation USADA can provide for the location of the bottle was that it was somewhere within the laboratory, in a "controlled zone." This explanation is not sufficient, however. Accordingly, in addition to the failure to record intra-laboratory transfers, the chain of custody documents clearly did not record the location of the bottle during the time it was in the laboratory.

D. Errors In The Preparation Of Laboratory Documents

LNDD did not comply with WADA TD2003LCOC and ISO 17025.4.3.3.3, which prohibit the "corrections" made to the documentation supporting the alleged AAF for Sample 995474.

WADA TD2003LCOC states that:

> [a]ny forensic corrections that need to be made to the comment should be done with a single line through and the change should be initialed and dated by the individual making the change. . . . No white out or erasure that obliterates the original entry is acceptable.

The ISO 17025.4.3.3.3 states:

> If the laboratory's document control system allows for the amendment of documents by hand pending the re-issue of the documents, the procedures and authorities for such amendments shall be defined. Amendments shall be clearly marked, initialed and dated. A revised document shall be formally re-issued as soon as practicable.

There are numerous violations of WADA TD2003LCOC and ISO 17025.4.3.3.3 throughout the document package supporting the alleged AAF for Sample 995474. These violations consist of improper corrections or deletions to the rider number, sample number, time, values and to other critical data.

An example of those cross-outs is at Exhibit 24, USADA0200, where there are ***six improper corrections on one page***.

Another example is at Exhibit 24, USADA0009, where there is an improper change from an unknown number to what appears to be Sample 995474.

These improper correction procedures reflect sloppy and unprofessional laboratory techniques that can provide no assurance as to the accuracy and reliability of LNDD's test results. Indeed, even the AAA Majority opinion concedes that LNDD committed "sloppy practices." Majority Opinion, ¶ 290. Incredibly, the AAA Majority found that the practices here were so sloppy that "***in the future*** an error like this could result in a dismissal of an AAF finding by the Lab." *Id.* The future is irrelevant to Mr. Landis's case and provides no solace. As the Majority concedes, these errors are sufficient to result in a dismissal of an AAF and should result in the dismissal of the AAF finding in this case.

These errors constitute a substantial deviation from good laboratory practice and affect the reliability of the test results. Indeed, Dr. Goldberger testified that the mislabeling, misnumbering and correction technique of LNDD is of significant concern, and based on the totality of forensic corrections in this case, opined that "I can't trust [the reliability of the report and test results]. I think it's unreliable." *See* Tr. of R. at 1049:20-21.

E. False Evidence From LNDD And USADA

The credibility of LNDD's witnesses and test results, and of the credibility of USADA as well, must be seriously question in light of the fact that LNDD likely created a fraudulent document to support their claim, as shown at Exhibit 26, LNDD0440. This document was produced to Mr. Landis during discovery in March 2007. It purports to be a reference solution log maintained contemporaneously from January 19 to June 26, 2006. But the document shows cross-outs indicating that ***the date was changed*** in two of the entries from March 16, 2007 to March 6, 2006. It strains credulity to believe that the author of the document would have mistakenly placed a "2007" date in 2006, ***especially considering that this document was provided to Mr. Landis in the middle of March 2007***. Furthermore, the handwriting on this document — purportedly spanning five months — is identical. This manufacturing of evidence gives no assurance in the accuracy or reliability of LNDD's test results or in the integrity of its laboratory processes and personnel. Notably, USADA ***did not even contest the evidence that this document was a forgery***. *See* Dissent, ¶ 30.

Likewise, many of USADA's representations in its pre-hearing briefs and discovery responses were proven false at arbitration — a fact conceded by the AAA Majority, which was "trouble[d]" by these misstatements. Majority Opinion, ¶ 305. In addition to preventing Mr. Landis from presenting an adequate defense (since he relied on those representations), these inaccuracies show intentional misconduct by LNDD and USADA. Below are several examples of statements made by USADA to the Panel and to Appellant before the hearing that were later proven to be false. Many of these statements were proven false by USADA's own witnesses and by undisputed documentary evidence:

In its Pre-Hearing Brief, and again in its Pre-Trial Response Brief, USADA represented to the AAA Panel that the reliability of LNDD's IRMS test results – the gravamen of this case – was unquestionable because "the Mix Cal Acetate, Blank Urine and Mix Cal IRMS controls run in the same sequence **minutes before, during, and minutes after** Respondent's sample produced the expected analytical results." USADA's Pre-Trial Response Brief ¶ 27 (emphasis added); *see* USADA's Pre-Hearing Brief ¶ 79. But the document package prepared by LNDD irrefutably establishes that the Mix Cal Acetates on Respondent's A and B samples were not run minutes before and/or minutes after Respondent's sample. *See* discussion above. Quite the contrary, the Mix Cal Acetate on the A sample was run 5 hours 14 minutes after the F3 fraction. *See* Ex. 24, USADA0166, 0183. And, the Mix Cal Acetate on the B sample was run 4 hours 40 minutes after the F3 fraction. *See* Ex. 25, USADA0347, 0362. Thus, USADA's arguments were patently false based on the information in the document package.

In further support of its assertions that LNDD's IRMS test was reliable, USADA claimed that "[b]ecause the IRMS instrument was accurate in measuring all of the controls, the results for Respondent's samples, which were analyzed by the IRMS instrument at the same time, must also be accurate." USADA's Pre-Trial Response Brief ¶ 27 (emphasis added); *see* USADA's Pre-Hearing Brief ¶ 77. But this assertion also was proved incorrect because the IRMS machine ***did not*** accurately measure the internal standard, 5α-Androstanol, in several of the fractions associated with Respondent's A and B samples. *See* Meier-Augenstein Presentation at Slides 52, 54; Closing Presentation at Slides 39, 40, 134, 136.

Likewise, USADA's statement in its Pre-Hearing Brief that "Respondent's sample is positive by any criteria," is false. USADA's Pre-Hearing Brief, Heading L. USADA's witness, Dr. Catlin, testified that according to UCLA's positivity criteria, a sample is not classified as

adverse unless the delta-delta values for ***both*** 5-Alpha and 5-Beta are more negative than -3 per mil. *See* Tr. of R. at 1222:10-20. The delta-delta values of the 5-Alpha and 5-Beta in Respondent's samples were not more negative than -3 per mil; thus, ***Respondent's IRMS test was not positive under UCLA's positivity criteria***. *See* Exs. GDC00536-00537.

Dr. Catlin was not the only USADA expert to contradict a pre-trial representation by USADA. USADA asserted that "when WADA has established a positivity criteria, they [WADA laboratories] are not expected (let alone required) to conduct their own studies to validate that criteria." USADA's Pre-Trial Response Brief ¶ 6. Dr. Ayotte, however, testified to the contrary – WADA laboratories ***are*** required to validate their methods. *See* Tr. of R. at 856:13-857:18 ("That's exactly what they should do.").

In addition to suffering contradictions by its own witnesses, USADA had no evidence to defend its pre-trial statements when Respondent's witnesses challenged them. For instance, in its Pre-Trial Hearing Brief, USADA wrote that "[t]he co-eluting peak [on the screen test] was substantially eliminated during the . . . confirmation . . . ." Pre-Hearing Brief ¶ 144. Later, in its Pre-Trial Response Brief, USADA again stated "although considerable background is still visible, the confirmation chromatograms show a better (i.e., narrower) peak shape." Pre-Trial Response Brief ¶ 59. But Dr. Goldberger provided evidence that the co-eluting peak seen on the GC/MS screen ***was not*** eliminated and that the confirmation chromatograms were either of the same quality as the screening or worse. *See* Tr. of R. at 1075:19-1086:18. USADA did not dispute this testimony, and therefore concedes the issue.

Nor did USADA contest Dr. Davis's testimony showing that LNDD misunderstood the critical aspects (i.e., Penning pressure) as to the operation of the IsoPrime instrument. USADA claimed that the green light displayed on the IsoPrime instrument changes colors if the Penning

pressure of the machine rises too high. But Dr. Davis showed that the light in question was simply a power light, unrelated to the Penning pressure, and certainly did not change colors. *Compare* USADA's Pre-Hearing Brief ¶ 106 *with* Tr. of R. at 1788:10-1789:6. Where LNDD does not understand how to operate the machine at issue, the results coming from that machine cannot be taken seriously.

USADA's statements also conflicted with the plain meaning of relevant documents. In its Pre-Trial Response Brief, USADA claimed that "[t]here is no WADA requirement to document the location of a sample bottle." USADA Pre-Trial Response Brief ¶ 8 n.8. However, WADA TD2003LCOC specifically states that "[a] chain of custody is required for both 'A' and 'B' *Sample* bottles . . . prepared for a testing procedure." And, the same technical document further states that "[i]n the case of *Samples*, the Laboratory Internal Chain of Custody should record all movement from receipt in the Laboratory through storage and sampling to disposal." Ex. GDC00233.

Perhaps most troubling is LNDD and USADA's repeated attempts to hide evidence at every step of the proceedings. For example, USADA initially claimed that "no post acquisition corrections of the data have been performed by LNDD in relation to sample 995474 other than those shown in the laboratory documentation package." Ex. C to USADA's Response to Respondent's Second Request for Production of Documents ¶ 6 at 2. Yet during Ms. Mongongu's and Ms. Frelat's testimony, both stated that they in fact manually processed and corrected data after it was acquired. *See* discussion above.

Likewise, when Respondent asked for all documentation related to the creation and accuracy of the background subtraction method used by LNDD in the IRMS test, USADA responded that background subtraction was "embedded in the instrument software" and that

"***LNDD had no separate documentation***." Ex. B to USADA's Response to Respondent's Second Request for Production of Documents ¶ 10 at 10. When Respondent asked LNDD to explain how it performed and applied background subtraction to Sample 995474 and related controls, USADA again stated that background subtraction was embedded in the instrument software. Ex. C to USADA's Response to Respondent's Second Request for Production of Documents ¶ 8 at 2. These statements were proven false after both LNDD operators testified that they manually changed the background points and that LNDD ***had an SOP describing such a method***. *See* Tr. of R. at 455:10-456:8; 724:11-725:25. When combined with the fact that Mr. Landis had no opportunity to depose these witnesses before the trial, the ever-changing landscape of LNDD and USADA's story and document production prevented a meaningful assessment of the facts.

These persistent false statements are not isolated instances but rather a consistent pattern of statements that support USADA's blanket assurances that LNDD performed its tests properly, that its technicians were knowledgeable and well-trained, and that the laboratory procedures occurred without error. These blanket assurances were proven false, and should be assigned no weight.

VI.

THE EVIDENCE OF THE WADA LABORATORY DIRECTORS CALLED BY USADA TO OPINE THAT THE LNDD LAB RESULTS WERE ACCURATE SHOULD BE VIEWED WITH MISTRUST

As noted by the AAA Majority decision, Mr. Landis objected to the AAA Panel's use of a WADA lab director as an expert to be consulted by the AAA Panel. Majority Opinion, ¶ 56. It is not possible for a WADA lab director to be impartial when reviewing the work of a WADA accredited lab because flaws in that lab's work reflect negatively on WADA and all WADA labs.

Moreover, the WADA Code of Ethics prohibits a WADA lab director from providing evidence in defense of an athlete in an anti-doping case. *See* Ex. 8, at Annex B, Sections 3.3 and 3.4, *see also*, Closing Presentation at Slide 159.

Indeed, as noted in the Dissenting Opinion, "the Laboratory Directors are bound by an Ethics Code of Conduct that has been interpreted to preclude them from disclosing the errors of one of their fellow laboratories on behalf of an athlete," which means that if Dr. Botrè knew of an error by LNDD, "and that error was causing" Mr. Landis "to be convicted of a doping offense," he may not provide evidence "on behalf of the athlete and disclose the error." Dissent, ¶ 14. That the AAA Majority Panel's expert witness was ethically barred from providing evidence in favor of Mr. Landis shows that "WADA's purported 'Code of Ethics' unnecessarily operates as an obstacle to the search for truth," and "may even, in practice, improperly lead to the withholding of evidence." *Id.* ¶ 15.

VII.

THE RESULTS OF THE GC/MS TEST ARE FLAWED

A. The GC/MS Test Was Fatally Flawed

The GC/MS test was unreliable and inaccurate due to the failure of LNDD to acquire three diagnostic ions (pursuant to TD2004EAAS and WADA TD2003IDCR). *See* Majority Opinion, ¶¶ 158-172. The GC/MS test was unreliable and inaccurate because (1) LNDD failed to properly identify testosterone and epitestosterone in the confirmation testing of the testosterone to epitestosterone ratio test ("T/E test") procedure using the Gas Chromatography-Mass Spectrometry ("GC/MS") test as required by WADA TD2003IDCR.

LNDD's initial screening test is called the T/E test. The theory behind the T/E test is that the urinary testosterone to epitestosterone ratio remains relatively constant and is not known to

be altered by exercise. Ex. GDC00234. The administration of exogenous testosterone results in an increase in the concentration of testosterone in the urine but does not change the concentration of epitestosterone. *Id.* Thus, the ratio of testosterone to epitestosterone ratio increases.

The T/E test is performed using a GC/MS instrument, which identifies different substances within a urine sample. The GC/MS instrument produces a series of documents called chromatograms. A chromatogram is simply a graph with retention time on the X-axis and response, or quantity, on the Y-axis. The chromatogram also displays peaks associated with testosterone and epitestosterone. The absolute amount of testosterone and epitestosterone is calculated by measuring the area under their respective peaks. The ratio of testosterone to epitestosterone, however, is measured using their response factors from the chromatograms. The reported concentrations of testosterone and epitestosterone are then corrected to a specific gravity.

The T/E test has two phases: the screen phase and the confirmation phase. WADA TD2004EAAS permits testing for an abnormal T/E ratio using a single aliquot and a single ion (m/z 432). *See id.* at WADA0011 ("The T/E value is given by the peak area or peak height ratio of testosterone and epitestosterone . . . obtained by measuring the ion at m/z 432 by GC/MS Analysis . . . [T]he Screening Procedure . . . is normally conducted on a single aliquot . . . .").

Pursuant to WADA TD2004EAAS, the confirmation of a purportedly elevated (1) concentration of testosterone, (2) concentration of epitestosterone or (3) T/E ratio must be conducted pursuant to WADA TD2003IDCR. *See* Ex. GDC00396-00400. WADA TD2004EAAS, which governs the testing and reporting of testosterone, epitestosterone, T/E ratios and other endogenous steroids, states:

> Confirmation of elevated T/E values, concentration of testosterone, epitestosterone or any other steroid metabolite under consideration is to be

> performed in triplicate. The confirmation of the identity of any steroid reported with abnormal properties must be made (refer to technical document TD2003IDCR). Appropriate calibration (e.g., calibration curve, deuterated standards, quality control samples) is to be included in the protocol of the Confirmation Procedure.

Ex. 9 at 2 (emphasis added).

WADA TD2003IDCR states:

> Selected Ion Monitoring[24] Mode. In some cases, it may be necessary to monitor selected ions in order to detect the substance at the Minimum Required Performance Limits. When selected ions are monitored, ***at least three diagnostic ions must be acquired***. The relative abundance of a diagnostic ion shall preferably be determined from the peak area or height of integrated selected ion chromatograms.

See Ex. GDC00397.

The requirements of WADA TD2003IDCR were not met on ***any*** confirmation testing.

The Data Analysis Parameters for the first A confirmation show the acquisition of a single diagnostic ion at m/z 432.40. Ex. 24, USADA0086.

The chromatogram for the first A confirmation shows the acquisition of a single diagnostic ion at m/z 432.40. Ex. 24, USADA0093.

The Data Analysis Parameters for the second A confirmation show the acquisition of a single diagnostic ion at m/z 432.40. Ex. 24, USADA0207.

The chromatograms for the second A confirmation show the acquisition of the same diagnostic ion at m/z 432.40. Ex. 24, USADA0213, 0215.

The Data Analysis Parameters for the B confirmation show the acquisition of a single diagnostic ion at m/z 432.40. Ex. 25, USADA0270.

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[24] "Selected Ion Monitoring" ("SIM") is defined in relevant part at TD2003IDCR: "Acquisition of ions of one or more pre-determined discrete m/z values for specified dwell times."

The chromatograms for the B confirmation show the acquisition of a single diagnostic ion at m/z 432.40. Ex. 25, USADA0277, 0280, 0282, 0284.

LNDD clearly violated TD2003IDCR by acquiring and analyzing only one diagnostic ion at m/z 432 in both the A and B confirmation T/E tests.

LNDD's failure to comply with TD2003IDCR renders the T/E test results inaccurate and unreliable. When LNDD's T/E chromatogram is compared with a proper T/E confirmation chromatogram, (i.e., a chromatogram showing the proper acquisition and analysis of three diagnostic ions) , the differences are apparent. Compare Ex. 25, USADA0282 with Exhibit GDC00524.

The purpose of acquiring and analyzing three diagnostic ions is to be certain that the measured substances are, in fact, testosterone and epitestosterone. When asked about the "significance of the fact that LNDD did not provide the chromatograms showing the analysis of the three diagnostic ions," Dr. Goldberger testified that LNDD's "T/E ratios are not supported by the chemistry that they conducted in their laboratory. It's unreliable." Tr. of R. at 1066:19-21. Dr. Goldberger testified that in his more than 20 year's experience with GC/MS in drug testing, he had never seen so many errors in a single sample. Tr. of R. at 1090:12

Dr. Bruce Goldberger's testimony highlighted the importance of acquiring three diagnostic ions. Specifically, Dr. Goldberger testified that, even when conducting only a cursory search, ***he found more than 10 other compounds***, including non-steroid-related compounds, at the same retention time and abundance as the diagnostic ion (m/z 432.10 to m/z 433.10) relied upon by LNDD in this case to characterize the substances as testosterone and epitestosterone. Tr. of R. at 1065:2-16. Therefore, there can be no assurance that the substances measured are actually testosterone and epitestosterone.

Further proof of LNDD's flawed testing methodology for testosterone and epitestosterone is shown by LNDD's identification of a substance that was ***not*** supposed to be present in the T/E test. LNDD's identification of deuterated androsterone – ***which should not have been present in the confirmation T/E test*** – renders the T/E test results inaccurate and unreliable. Ex. 24, USADA0054. Deuterated androsterone, which does not appear naturally in human urine, is an artificial marker that is sometimes used as an internal standard. LNDD's identification of deuterated androsterone in an aliquot to which no deuterated androsterone has been added further underscores the problems associated with failing to adhere to TD2003IDCR. Therefore, LNDD's identification of deuterated androsterone in the T/E testing process gives no confidence that the T/E test results were accurate. Ex. 24, USADA00057.

As further proof of LNDD's flawed testing methodology for testosterone and epitestosterone, LNDD proceeded with the B Sample T/E confirmation using a sample it knew was too degraded for analysis. Pursuant to WADA TD2004EAAS:

> To report an Adverse Analytical Finding of an elevated T/E value, testosterone or epitestosterone concentration or any other endogenous steroid parameters, the concentration of free testosterone and/or epitestosterone in the specimen is not to exceed 5% of the respective glucuroconjugates.

Ex. 49, WADA0012. In this case, the test for degradation on the B Sample showed that the ratio was 7.7% – much greater than the allowable 5% limit.

Because the T/E tests for Sample 995474 are unreliable and inaccurate, the longitudinal studies introduced by USADA (at Exhibit 30), are irrelevant and have no evidentiary weight.

The evidence from the T/E tests is of no evidentiary value and, therefore, should be entirely disregarded.

B. The GC/MS Test Should Also Be Rejected Because Of Poor Chromatography

The T/E test results associated with Sample 995474 are also inaccurate and unreliable because of poor chromatography. In the GC/MS chromatograms related to the T/E test, Dr. Goldberger showed that the chromatogram at Exhibits 24 and 25, USADA0093 (the Sample A confirmation) & USADA0277 (the Sample B confirmation), were so "horrible" as to be unreliable. Tr. of R. at 1059:18. Additionally, LNDD's widely varying results for testosterone and epitestosterone for Sample 995474 provide additional corroborating evidence that the T/E test results are unreliable and inaccurate. These variations resulted in T/E ratios ranging from a low of 4.9 (in the first screen) to a high of 11.4 (in the second B test confirmation). Ex. 24, USADA0054, 0057, 0101, 0223; Ex. 25, USADA0288. There is substantial evidence showing that poor chromatography contributed to inaccurate results, as shown by the greatly varying T/E ratios reported by LNDD for Sample 995474.

VIII.

CONCLUSION

For the foregoing reasons, LNDD has violated several ISL rules in the testing of Sample 995474 and there is no evidence that these violations did not cause the AAF. The Panel should find no comfortable satisfaction in the test results and should dismiss the allegations against Appellant.

APPELLANT'S WITNESS LIST

A. JOHN AMORY

Dr. John Amory is a medical doctor and a professor at the University of Washington. Dr. Amory is the attending Physician at the University of Washington Hospital and is Board Certified in Internal Medicine. Dr. Amory's research is primarily in the study of testosterone and testosterone replacement therapy. Dr. Amory will provide background information on testosterone such as the molecular make-up and testosterone metabolism. Also, Dr. Amory will discuss the various methods of administering synthetic testosterone, i.e., injection, oral and topical. The next area of testimony Dr. Amory will address is the effect of testosterone on the body. This includes information concerning how the administration of synthetic testosterone affects the production of endogenous testosterone production. In addition, Dr. Amory will address how synthetic testosterone affects a person's T/E ratio and will opine Floyd's T/E results. The next area of Dr. Amory's testimony is the physiological effects of testosterone, such as its effects on muscle, endurance, recovery, and time to see benefit. Dr Amory will also comment on the lab errors by LNDD, such as documentation errors and chain of custody breaks. Dr. Amory's general conclusion is that Mr. Landis' results are not reliable.

B. SIMON DAVIS

Simon Davis is the Technical Director of Mass Spec Solutions. Mass Spec Solutions is one of the only manufacturers of Isotope Ratio Mass Spectrometers in the world. He was previously a Stable Isotope Systems Engineer and a Project Manager at Micromass UK, Ltd. (the manufacturer of the IRMS equipment used by LNDD). He has a PhD in Stable Isotope Mass Spectrometry from Liverpool JMU, in Association with Cambridge University. He will testify regarding a brief summary of his experience and background; the principles and operation of an Isotope Ratio Mass Spectrometer; the principles and operation of the GC/C/IRMS test for exogenous testosterone; his review of the analytical reports of the testing conducted by LNDD on the urine sample(s) provided by Floyd Landis during the 2006 Tour de France, and the LNDD's interpretation of those results; his observation of the LNDD's GC/C/IRMS testing in April 2007; the re-processing of the GC/C/IRMS electronic data files of the urine sample(s) provided by Floyd Landis during the 2006 Tour de France; and his opinion regarding the accuracy and reliability of the analytical reports of the GC/C/IRMS testing conducted by LNDD on the urine sample(s) provided by Floyd Landis during the 2006 Tour de France. His curriculum vitae will be provided.

C. BRUCE GOLDBERGER

Dr. Goldberger is a Professor at the University of Florida, Departments of Pathology, Immunology and Laboratory Medicine and Department of Psychiatry, University of Florida. He has an M.S. Degree in Forensic Toxicology from the University of Maryland School of Medicine, and is the current President of the American Academy of Forensic Sciences. He will testify regarding a brief summary of his experience and background; the proper operation and management of a laboratory; and the principles of GC/MS testing. He will testify to the numerous errors committed by the laboratory, the principles and operation of an GC/MS testing;

his review of the analytical reports of the testing conducted by LNDD on the urine sample(s) provided by Floyd Landis during the 2006 Tour de France, and the LNDD's interpretation of those results; and his opinion regarding the accuracy and reliability of the analytical reports of the GC/MS testing conducted by LNDD on the urine sample(s) provided by Floyd Landis during the 2006 Tour de France. His curriculum vitae will be provided.

D. ALLEN LIM

Dr. Allen Lim has a PhD in exercise physiology and has extensive cycling coaching experience. Dr. Lim is an expert in the use of power data in training cyclists and assisted in the development of the PowerTap product. Dr. Lim will address how power data is commonly used in cycling training and why it is important. Dr. Lim will also testify about his experiences as Mr. Landis' coach and the use of power data in Mr. Landis' training. Dr. Lim will discuss his collection of power data from Mr. Landis' various rides over . Dr. Lim will then compare Mr. Landis' stage 17 performance to Mr. Landis' other performance. Dr. Lim will opine that Mr. Landis' performance during Stage 17 were not superhuman. Dr. Lim will also address Mr. Landis' strategy during Stage 17 and his strategy affected his performance.

E. WOLFRAM MEIER-AUGENSTEIN, CCHEM, MRSC

Dr. Wolfram Meier-Augenstein is a senior lecturer in Environmental Forensics at Queen's University in Belfast, Ireland. He is a well-recognized research scientist in the field of carbon isotope ratio testing and mass spectrometry. Dr. Meier-Augenstein will testify on the theory of carbon isotope ratio testing and the reasons why the LNDD's single isotope criteria does not make sense and is inconsistent with good science. He will testify that the results of LNDD's carbon isotope ratio testing in this case, both as to the Stage 17 results and the retesting results, are unreliable and inconsistent. He will testify about the following errors: (1) the poor application and use of standards; (2) poor chromatography; (3) the lack of the use of a reference population; (3) the inconsistencies in the results of the various IRMS tests; (4) the impact of poor linearity, (5) incorrect mass balance equation, and (6) LNDD's failure to identity the target analytes. Further, Dr. Meier-Augenstein will rebut contentions by USADA as it relates to carbon isotope ratio testing, in particular, as to the value and correctness of its use of its internal standards, laboratory procedures with respect to IRMS, and application of interpretation of its chromatograms.

F. FLOYD LANDIS

Mr. Landis will testify on his own behalf. Mr. Landis will deny the use of synthetic testosterone. Mr. Landis will also testify about his general character including his religious upbringing. Lastly, Mr. Landis will also discuss his strategy and capabilities.

G. DANIEL DUNN

Daniel Dunn submitted a declaration in connection with his observations during the further testing of Respondent's remaining B samples from the 2006 Tour de France. If necessary, Respondent intends on cross-examining Mr. Dunn on the statements made in this declaration and his conduct during the retesting process at LNDD.

H. TIMOTHY BROCKWELL

Timothy Brockwell is a GVI Development Scientist who has worked with MicroMass/GVI since 1996. Respondent designates Mr. Brockwell as an adverse witness. Mr. Brockwell submitted a declaration in connection with USADA's response to Respondent's Motion in Limine to Exclude the Restesting Evidence. If necessary, Respondent intends on cross-examining Mr. Brockwell on the statements made in this declaration and other various aspects of the Isoprime's software.

I. KEITH GOODMAN

Keith Goodman is the senior director of analytical chemistry at Xanthus Pharmaceuticals, Inc. He will be called to testify on the theory of carbon isotope ratio testing and the reasons why the LNDD's single isotope criteria does not make sense and is inconsistent with good science. He will testify that the results of LNDD's carbon isotope ratio testing in this case, both as to the Stage 17 results and the retesting results, are unreliable and inconsistent. He will testify about the following errors: (1) the poor application and use of standards; (2) poor chromatography; (3) the lack of the use of a reference population; (3) the inconsistencies in the results of the various IRMS tests; (4) the impact of poor linearity, (5) incorrect mass balance equation, and (6) LNDD's failure to identity the target analytes. Further, Dr. Goodman will rebut contentions by USADA as it relates to carbon isotope ratio testing, in particular, as to the value and correctness of its use of its internal standards, laboratory procedures with respect to IRMS, and application of interpretation of its chromatograms.

J. ALL DESIGNATED, INITIAL AND SUPPLEMENTAL, WITNESSES BY USADA

Respondent now designates witnesses USADA has previously designated as witnesses in the Appealed Case. These witnesses include:

- Dr. Cedric Shackleton
- J. Thomas Brenna, Ph.D.
- Dr. Rodrigo Aguilera
- Don H. Catlin, Ph.D.
- Janine Jumeau
- Cynthia Mongongu
- Claire Frelat
- Jacques de Ceaurriz
- Esther Cerpolini

- Ruddy Barlagne
- Dr. Corinne Buisson

DATED: November 20, 2007

Respectfully submitted,

By: 

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