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Performance of OFDA4000 instruments in three mill laboratories

By

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SUMMARY

In Biella in November 2005, the Commercial Regulations Committee approved the issue of Draft Test Method 62 covering the use of the OFDA4000 for the measurement of length and diameter properties of sliver and tops. The chairman of the Committee reminded delegates that during the DTM period, users were expected to accumulate experience with the method, so that performance data could be presented subsequently in support of a request to upgrade the DTM to a full test method.

This report details the results of a trial of three OFDA4000 instruments in three mill laboratories in Europe. The mills were requested to measure 9 top samples in blind replicate, presented as 18 individual samples. The top samples covered the MFD range 16 to 36 μm and H range 56 to 104 mm.

The 9 top samples were selected from the 40 that had been used in the 2005 round trial, and it was therefore possible to compare the results for both accuracy (with respect to the reference values obtained in 2004/05), and precision.

The results confirm that under commercial conditions the instruments performed in accordance with the statistics shown in DTM 62.

INTRODUCTION

The development of the OFDA4000 has been described in papers previously submitted to this group (Brims 2002, 2003, 2004, Brims & Baxter 2004, Baxter 2005). The instrument is designed to measure both length and diameter distributions, as well as the length-diameter profile, on wool sliver samples. The instrument can operate in 'native' mode, where it measures sliver for both length and diameter distribution, and in '70 mm' or 'snippet' mode, where it emulates an OFDA100 and measures 2 mm snippets spread on 70 mm glass slides, for diameter distribution only. The results of this trial are only concerned with measurements undertaken in the 'native' or sliver mode

The Almeter is described in IWTO-17. Additional data on its performance in measuring certain length distribution parameters was reported by Couchman & Holmes (2003). For the purposes of this trial, the Almeter is regarded as the reference instrument for the measurement of length distribution.

The OFDA100 and Laserscan are described in IWTO-47 and IWTO-12 respectively. For the purposes of this round trial, these instruments are regarded as reference instruments for the measurement of diameter distribution.

In Biella in November 2005, the Commercial Regulations Committee approved the issue of Draft Test Method 62 covering the use of the OFDA4000 for the measurement of length and diameter properties of sliver and tops. The chairman of the Committee reminded delegates that during the DTM period, users were expected to accumulate experience with the method, so that performance data could be presented subsequently in support of a request to upgrade the DTM to a full test method.

This report details the results of a trial of three OFDA4000 instruments in three mill laboratories in Europe. Two of the instruments had been installed for 4 years. None of these laboratories were IWTO-licensed and therefore the trial, similar to the 2004/05 trial, can be considered a reasonable representation of normal commercial usage of the instruments.

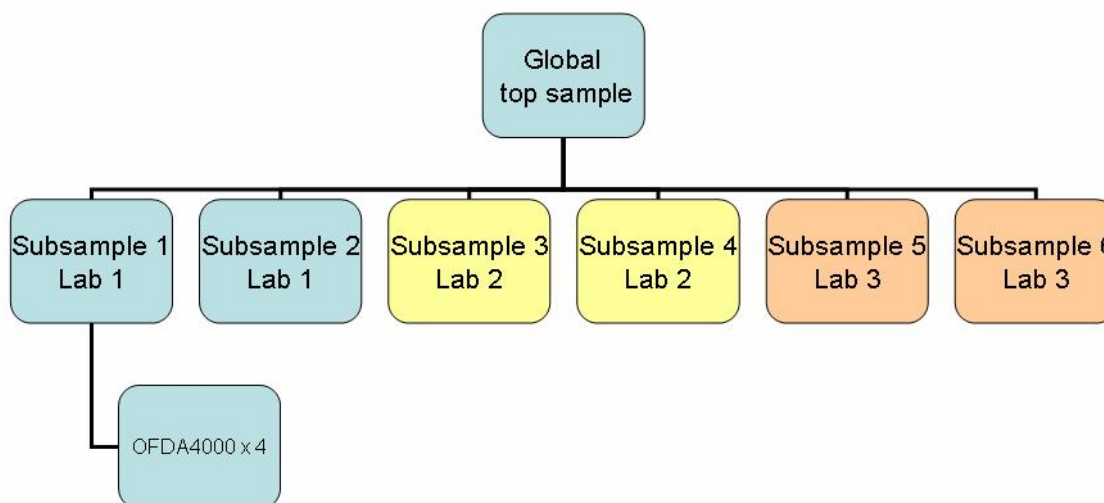
METHOD

Nine top samples were selected from across the range of the 40 that had been used in the 2004/05 round trial, so as to be able to compare the results for both accuracy (with respect to the reference values obtained in 2005), and precision.

The protocol for this trial closely followed that of the 2004/05 trial, but was simplified to include only OFDA4000 measurements on sliver. The protocol was discussed in general terms with representatives of the Federation of Australian Wool Organisations and the IWTO Technology & Standards Committee. The protocol is reproduced as Appendix 1.

The mills were requested to measure 18 individual top samples, which comprised 9 blind replicates. The top samples covered the MFD range 16 to 36 μm and H range 56 to 104 mm.

The schema is illustrated below:



The range of samples included tops from New Zealand, Australia, Germany and Italy. The range of properties are summarised in Table 1.

Table 1: Range of samples used in the trial

Property	Minimum	Maximum	Average
Mean fibre diameter μm	16.4	36.5	23.5
Standard deviation μm	3.3	9.4	5.2
Hauteur mm	56.6	104.6	75.6
Coefficient of variation CvH %	31.9	52.9	43.1
Barbe mm	68.3	130.8	90.1
K25H	1.0	10.5	6.1

ANALYSES

The OFDA4000 produces an enormous amount of information. The report on the 2004/05 trial considers most of the parameters, and since this latest project was intended to simply to confirm or otherwise the overall performance of the test method in commercial use, it was considered unnecessary to repeat the extensive analyses considered in that earlier report. The analyses are therefore limited to the following parameters which were considered of commercial importance and also capable of indicating whether the performance differed significantly from that documented earlier and covered by the statistics shown in the draft test method:

- Mean fibre diameter (MFD) and Standard deviation of diameter (SDD)
- Mean hauteur (H), coefficient of variation of hauteur (CvH), mean barbe (B), percentage of fibres below 25 mm hauteur (k25H), and mean hauteur of longest 1% of fibres (L1H)

Two sets of analyses were undertaken:

- Comparison of the grand mean values with the reference values obtained in the 2004/05 trial
- One-way analyses of variance undertaken using the following model:

Source of variation	Sum of squares	Degrees of freedom	Mean square	Expected mean square
Amongst laboratories	A	2	A/2	$\sigma_{\omega}^2 + 2 \sigma_L^2$
Within laboratories	B	3	B/3	σ_{ω}^2
Total	C	5	-	-

Where: σ_{ω}^2 is the within-laboratory component of variance (between "hanks")

And σ_L^2 is the between-laboratory component of variance

It will be noted that this model varies slightly from that used in the analysis of the 2004/05 trial. In that trial, the results of individual "tests" were analysed for the purposes of defining the precision statistics of the method. In this trial, the measurement regime established in DTM-62 has been used, in which the average of 2 individual measurements (each comprising a "test" as defined in DTM-62) on a hank has been used as the "test result".

As with convention, calculated negative values of between-laboratory components of variance are set to zero. Individual analyses were carried out for each sample using Unistat (2002)

95% confidence limits were calculated for the minimum case defined in DTM-62: a test result in a single laboratory produced from 2 individual test specimens as defined in the *Definitions* section of the method.

$$95\%CL = 1.96 * \text{sqrt}(\sigma_{\omega}^2 + \sigma_L^2)$$

No data were judged sufficiently outlying to be omitted from the analyses.

RESULTS

ACCURACY

The grand mean values for each of the 9 tops are shown in Table 2. Differences from the grand mean reference values obtained in the 2004/05 trial are shown in table 3.

Table 2: Grand mean values for each of the 9 tops

Grand means this trial							
ID	MFD	SDD	H	CvH	B	k25H	L1H
1	36.49	9.42	104.6	50.1	130.8	5.0	241.9
2	25.12	6.50	58.0	42.8	68.7	7.4	124.6
3	16.43	3.54	56.6	45.5	68.3	10.5	118.5
4	18.12	3.32	73.1	35.5	82.3	3.5	126.0
5	18.68	3.35	86.0	31.9	94.7	1.0	141.7
6	17.58	3.67	59.1	47.0	72.1	9.5	126.8
7	33.73	7.84	91.0	52.9	116.5	8.6	203.1
8	21.53	4.69	75.6	40.3	87.9	5.1	144.4
9	23.46	4.80	76.3	41.8	89.6	4.1	151.6
min	16.4	3.3	56.6	31.9	68.3	1.0	118.5
max	36.5	9.4	104.6	52.9	130.8	10.5	241.9
avg	23.5	5.2	75.6	43.1	90.1	6.1	153.2
sd	7.2	2.2	16.3	6.7	21.5	3.1	41.9

Table 3: Differences from reference values used in 2004/05 trial

New minus 2004/05 reference values							
ID	MFD	SDD	H	CvH	B	K25H	L1H
1	0.21	-0.15	1.1	0.2	1.7	0.3	5.5
2	-0.23	-0.17	-0.7	1.6	0.0	-0.3	1.1
3	0.00	0.02	-0.1	-0.2	-0.2	-0.4	-0.9
4	-0.04	0.01	-1.3	-0.2	-1.5	0.1	-0.7
5	0.06	0.06	0.5	-1.4	-0.2	-0.4	2.3
6	0.01	0.01	0.9	-1.0	0.6	-0.8	0.5
7	0.01	-0.18	2.0	-2.1	0.6	0.2	1.3
8	0.04	0.06	-0.3	-0.3	-0.5	0.7	0.8
9	-0.01	-0.07	-0.2	0.0	-0.2	0.2	0.1
Avg	0.01	-0.05	0.21	-0.40	0.05	-0.05	1.11
SD	0.116	0.098	1.014	1.048	0.87	0.469	1.908
count	9	9	9	9	9	9	9
t val	0.149	1.438	0.633	1.135	0.16	0.314	1.739
p val	0.885	0.188	0.545	0.289	0.877	0.762	0.120
signif	ns	ns	ns	ns	ns	ns	ns

It can be observed that none of the average differences for the parameters analysed are significantly different to zero at the 0.05 level. The individual grand mean differences on each top are all relatively small when the overall mean values are taken into account, especially considering that the tops samples have been in storage for 3 years. There are no obvious magnitude-related trends, as can be seen in figures 1 to 7. Since the aim of this trial was to confirm that commercial use of the draft test method produces results which comply with the statistics of the method, full GM and DVA analyses are not required.

Figure 1

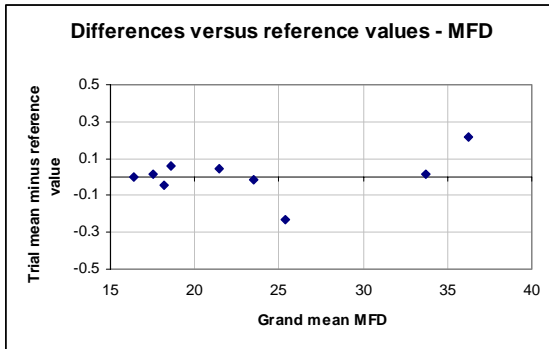


Figure 2

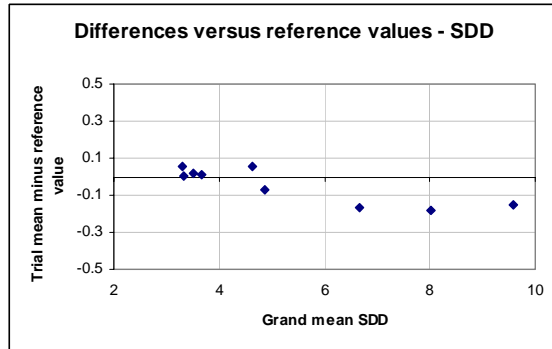


Figure 3

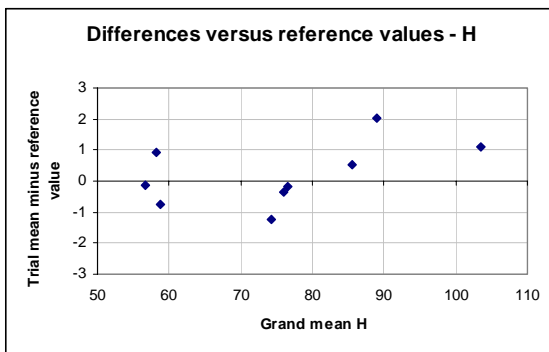


Figure 4

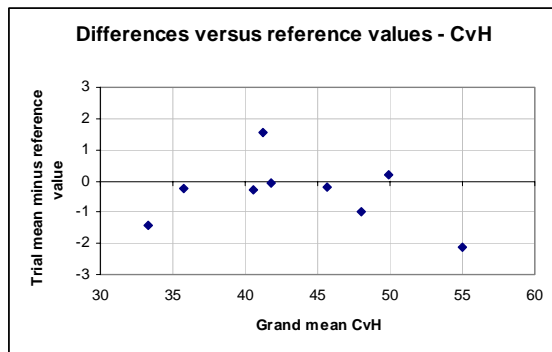


Figure 5

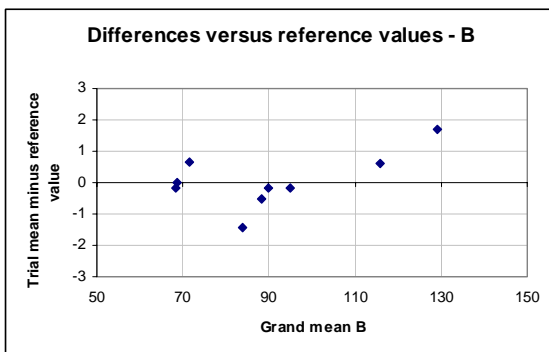


Figure 6

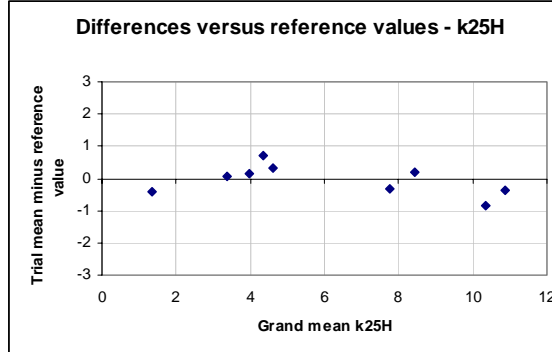
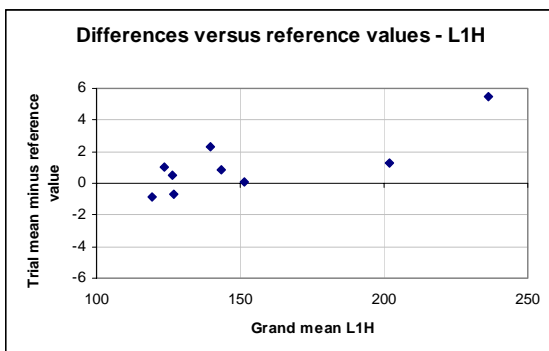


Figure 7



PRECISION

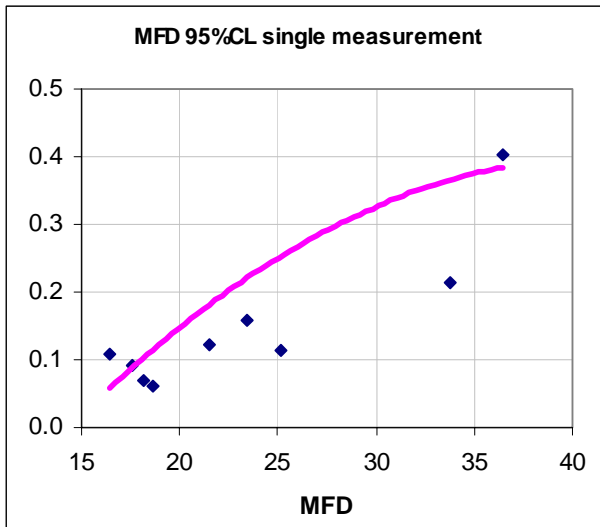
The results of the analyses of variance for the primary parameters shown in DTM-62 (table 8.1) are shown below. In addition to plotting the estimated 95% confidence limit values against the precision relationships shown in the DTM, the total variance values for each top sample have been compared against calculated values taken from table 8.1 of the DTM using an appropriate F-test.

Mean fibre diameter

Table 4 – Comparison of MFD components of variance estimates

MFD				In DTM 62			
Top	within	between	95%CL	MFD	within	between	95%CL
1	0.042	0.000	0.40	36.5	0.009	0.030	0.39
2	0.001	0.002	0.11	25.1	0.005	0.012	0.25
3	0.002	0.001	0.11	16.4	0.001	0.000	0.07
4	0.000	0.001	0.07	18.1	0.002	0.000	0.10
5	0.001	0.000	0.06	18.7	0.002	0.001	0.12
6	0.002	0.000	0.09	17.6	0.002	0.000	0.07
7	0.006	0.006	0.21	33.7	0.008	0.025	0.36
8	0.004	0.000	0.12	21.5	0.003	0.006	0.19
9	0.006	0.000	0.16	23.5	0.004	0.009	0.22

Figure 8 – comparison of estimated 95% confidence limits for MFD against calculated values from DTM-62



F-Test

	Cases	Mean	Standard Deviation	Standard Error
C1	9	0.0083	0.0131	0.0044
C2	9	0.0134	0.0142	0.0047
Total	18	0.0108	0.0136	0.0032

F(8,8) = 1.1820
 Right-Tail Probability = 0.4094 **NS**

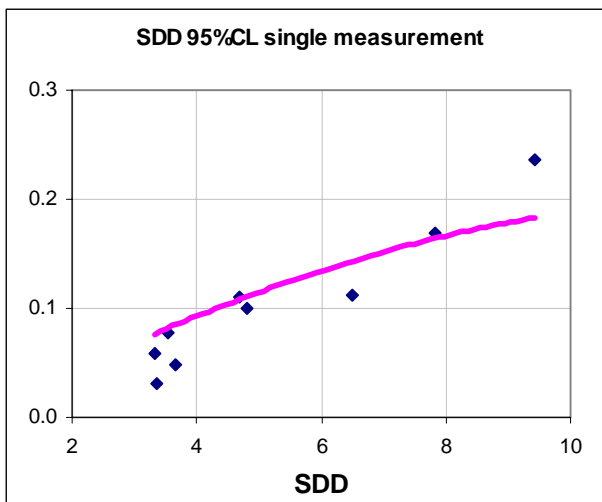
95% Confidence Interval = 0.2666 5.2401

Standard deviation of diameter

Table 5: Comparison of SDD components of variance estimates

SDD				In DTM 62			
Top	within	between	95%CL	SDD	within	between	95%CL
1	0.010	0.005	0.24	9.4	0.003	0.006	0.18
2	0.003	0.000	0.11	6.5	0.002	0.003	0.14
3	0.002	0.000	0.08	3.5	0.001	0.001	0.08
4	0.000	0.001	0.06	3.3	0.001	0.001	0.08
5	0.000	0.000	0.03	3.4	0.001	0.001	0.08
6	0.000	0.000	0.05	3.7	0.001	0.001	0.09
7	0.001	0.007	0.17	7.8	0.002	0.004	0.16
8	0.003	0.000	0.11	4.7	0.001	0.002	0.11
9	0.000	0.002	0.10	4.8	0.001	0.002	0.11

Figure 9 – comparison of estimated 95% confidence limits for SDD against calculated values from DTM-62



F-Test

	Cases	Mean	Standard Deviation	Standard Error
C1	9	0.0038	0.0046	0.0015
C2	9	0.0038	0.0026	0.0009
Total	18	0.0038	0.0037	0.0009

F(8,8) = 2.9609
 Right-Tail Probability = 0.0729 **NS**

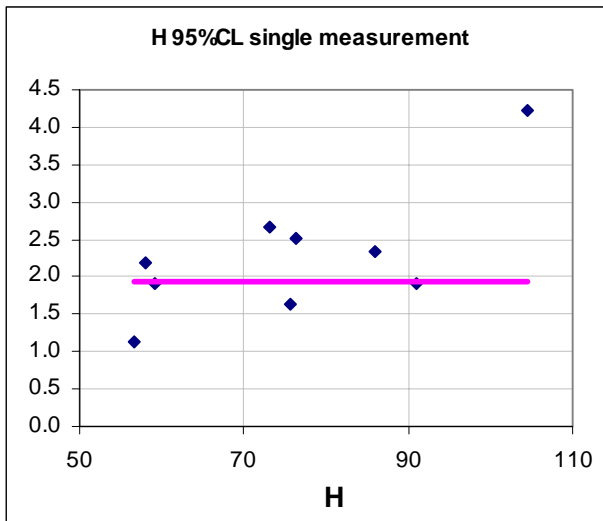
95% Confidence Interval = 0.6679 13.1265

Hauteur

Table 6: Comparison of Hauteur components of variance estimates

H				In DTM 62			
Top	within	between	95%CL	H	within	between	95%CL
1	2.278	2.390	4.23	104.6	0.631	0.339	1.93
2	1.253	0.000	2.19	58.0	0.631	0.339	1.93
3	0.104	0.229	1.13	56.6	0.631	0.339	1.93
4	1.337	0.500	2.66	73.1	0.631	0.339	1.93
5	1.049	0.369	2.33	86.0	0.631	0.339	1.93
6	0.941	0.014	1.92	59.1	0.631	0.339	1.93
7	0.941	0.000	1.90	91.0	0.631	0.339	1.93
8	0.660	0.044	1.65	75.6	0.631	0.339	1.93
9	0.714	0.917	2.50	76.3	0.631	0.339	1.93

Figure 10 – comparison of estimated 95% confidence limits for H against calculated values from DTM-62



F-Test

	Cases	Variance	Standard Error
C1	9	1.5260	0.4118
C2	9	0.9700	0.3283
Total	18		

F(8,8) = 1.5732
 Right-Tail Probability = 0.2681 **NS**

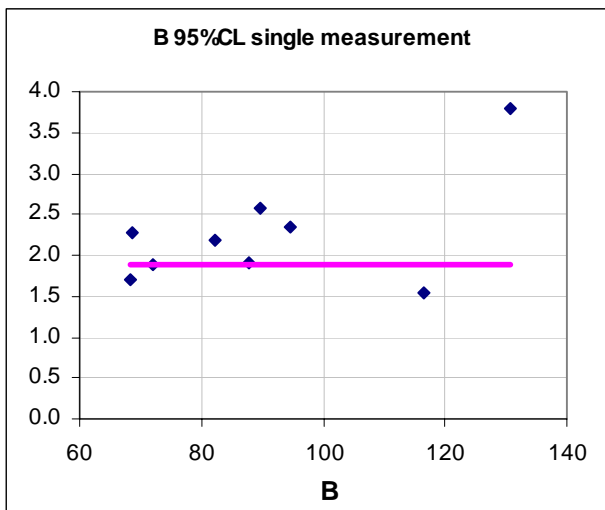
95% Confidence Interval = 0.3549 6.9744

Barbe

Table 7: Comparison of Barbe components of variance estimates

B				In DTM 62			
Top	within	between	95%CL	B	within	between	95%CL
1	2.986	0.781	3.80	130.8	0.439	0.475	1.87
2	1.322	0.013	2.27	68.7	0.439	0.475	1.87
3	0.037	0.720	1.71	68.3	0.439	0.475	1.87
4	0.707	0.534	2.18	82.3	0.439	0.475	1.87
5	0.939	0.496	2.35	94.7	0.439	0.475	1.87
6	0.574	0.341	1.87	72.1	0.439	0.475	1.87
7	0.329	0.286	1.54	116.5	0.439	0.475	1.87
8	0.789	0.149	1.90	87.9	0.439	0.475	1.87
9	0.250	1.484	2.58	89.6	0.439	0.475	1.87

Figure 11 – comparison of estimated 95% confidence limits for B against calculated values from DTM-62



F-Test

	Cases	Variance	Standard Error
C1	9	1.4160	0.3967
C2	9	0.9140	0.3187
Total	18		

F(8,8) = 1.5492
 Right-Tail Probability = 0.2750 **NS**

95% Confidence Interval = 0.3495 6.8682

CONCLUSIONS

Using blind replicated samples taken from 9 tops selected from the original 40 used in the 2004/05 round trial, and measured in three independent mill laboratories, this trial has demonstrated:

1. The grand mean values for the most important commercial parameters (MFD, SDD, H, CvH, B, k25H, L1) were all in good agreement with the original reference values obtained in 2004/05.
2. The total variances and calculated 95% confidence limits were, on average, not significantly different from the values which would be calculated using the precision statistics tabulated in DTM-62.

This trial has therefore demonstrated that in normal commercial use, the OFDA4000 instruments perform sliver measurements in accordance with the expected performance documented in IWTO DTM 62.

RECOMMENDATIONS

OFDA4000 instruments have now been in use in mill laboratories for 4 years. IWTO DTM-62 has been available for commercial use and evaluation for over 2 years, and given the satisfactory results of this trial, it should now be upgraded to a full test method.

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- IWTO-12-03: *Measurement of the Mean & Distribution of Fibre Diameter Using the Sirolan-Laserscan Fibre Diameter Analyser.*
- IWTO-17-04: *Determination of fibre length and distribution parameters.*
- IWTO-47-00: *Measurement of the mean and distribution of fibre diameter of wool using an Optical fibre Diameter Analyser (OFDA)*
- IWTO DTM-62-05: *Determination of the fibre length, length distribution, mean fibre diameter and fibre diameter distribution of wool top and slivers by the OFDA4000*
- Unistat© (2002) Unistat Statistical Package, version 5, Unistat Ltd, London

APPENDIX 1- PROTOCOL USED FOR THE TRIAL

Objectives

The main requirement is to examine the performance of OFDA4000 measurements in sliver mode between three instruments in three mill laboratories.

Principles

- 3 instruments
- In each laboratory, measure 2 blind replicates of a supplied set of 9 tops on OFDA4000
- BSC to keep adequate spare sample sets to allow additional or repeated participation
- Parameters to be examined include major commercial OFDA4000 length distribution data (H, CvH, B, K25H, L1H), MFD and SDD.

Method

Sample preparation

1. 9 commercial tops to be selected from the original group of 40 tops used in the 2004/05 trial that had been sourced from Australia, New Zealand and Europe. Tops are to cover as wide a range as feasible of both diameter and length. Each top should be in the weight range 20 to 30 g/m.
2. Each lab is to receive blind duplicate samples of each top
3. Each top to be initially at least 20 m in length
4. Each top to be cut into 8 by 1.8m lengths
5. Each 1.8m length to be twisted under slight tension by 54 turns (ie. 30 turns/m), doubled and the ends secured by a rubber band (in general accordance with Appendix 5 of IWTO-17)
6. Each twisty to be placed in a re-sealable polythene bag
7. Each set of 8 twisties to be randomly divided into 2 sets of 4
8. Each subset of 4 twisted to be labelled with a random number selected from the range 1 to 18. A record must be kept of the sample number pairing.
9. From each subset of 4, select 3 for the trial to be allocated at random to the 3 laboratories.. Remainder to be stored in case the trial needs to be repeated in either 1 or all laboratories (in which case the sample numbers will be changed).
10. Once all tops have been sampled, the twisties for each laboratory (18 in total) will be boxed up and couriered to the laboratories, together with instructions.

Preparatory in each laboratory:

1. All measurements and sample preparation to be carried out in an atmosphere complying with IWTO-52.
2. Upon receipt of the trial samples, and prior to measurement, check that:
 - The needle bed is clean and contains no bent needles
 - The beard guide is clean and free of any neps
 - The optical path is clear of dust and fibres
 - The gripper jaws are clean and in good condition.
3. At the start of each measurement session, ensure that the instrument is set up according to the operating manual. At least one sample of known characteristics shall be measured. If the mean fibre diameter or mean fibre length varies from the known values by more than the tolerances shown in the table below, (or from the mean values determined by at least 10 measurements carried out

immediately after the instrument has been calibrated), the instrument shall be checked, adjusted, or re-calibrated until satisfactory performance is obtained.

Top MFD or MFL	Tolerance (μm or mm)
Up to 15.0 μm	0.3
15.1 to 20.0 μm	0.6
20.1 to 25.0 μm	0.8
25.1 to 30.0 μm	1.0
30.1 to 35.0 μm	1.2
35.1 μm or greater	1.4
Less than 60 mm	1.5 mm
60.1 mm or greater	2.0 mm

4. Check calibration of OFDA4000 in top mode in accordance with IWTO DTM 62.

Sample measurements

1. Each participant will receive 18 standard twisted hanks. Each hank is slightly longer than normal (twisted length approx 0.8m rather than 0.6m)
2. All hanks must be exposed in an atmosphere complying with IWTO-52 for at least 24 hours prior to testing.
3. To avoid any transcription errors, data will be stored in MES files, copies of which are to be sent to the analyst by email at the end of the measurement work.
4. Do not cut snippets from predrawn end of top since this will bias the diameter.
5. Length measurement - The sample is to be untwisted immediately before the start of testing. The sliver, held with one end in each hand, is then straightened by putting it under slight tension and subjecting it to gentle shaking. Follow procedure as described in DTM 62, set predraw length to 150mm. Carry out sufficient draws (usually 6 to 7) to allow the preset limit of 4000 counts to be achieved. Repeat this measurement. Each measurement comprises a single test result for the purposes of this trial.
6. Once the measurement is completed, do not instruct the instrument to clear the needle bed: instead remove remaining sample manually by pushing it down out of the needle bed then retwist it and place into bag that it came from. This can then be re-measured with a shorter predraw of 20mm if required.

Analyses

1. Receive from each laboratory all instrument validation data, all MES files from OFDA4000
2. Confirm satisfactory instrument validations.
3. Confirm all necessary data files
4. Extract all primary parameters on a single measurement basis
5. Check all datasets for individual outliers
6. Combine/average all specimen and subsample data for each sample in each laboratory for principal commercial parameter means (MFD, SDD, H, B, CvH, k25H and L1H)
7. Calculate grand means across all laboratories for supplied samples and compare parameter grand means against reference grand means obtained in 2004/05 trial
8. Carry out analyses of variance for each sample and each instrument type, with the effects being: laboratories (as appropriate), and residual for each parameter and each instrument/laboratory. Calculate precision values as 95% confidence limits for all parameters against precision values quoted in IWTO-DTM-62.