

The Measurement of Uncertainty in Grain Size Distribution

Interlaboratory Exercise Part 2 - Metallographic Images

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ABSTRACT

An investigation of the grain size distribution by the intercept method has been carried out through an interlaboratory exercise, ILE, on representative engineering metallic materials, (two Al alloys and two Ni alloys). The ILE was performed in two parts. In the first part reference images generated at NPL from optical micrographs were measured. In the second part, a set of optical micrographs was measured. This report is concerned with the second part of the ILE. The ILE was supported by measurements of intercept distributions obtained from a 3-D model shape, a tetrakaidecahedron. Comparisons were made between the model distribution data in the two Al alloys (AA 1050 and AA 5182) and the two Ni alloys (Waspaloy and IN 909) and the distribution data from the material microstructures. A particular aspect of the study was an evaluation of the uncertainty associated with the measurements.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
Head, Centre for Materials Measurement and Technology

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1 INTRODUCTION

NPL has collaborated with a number of partners from industry and academia [1] to examine the uncertainties associated with measuring grain size distributions in representative engineering alloys through the use of structured interlaboratory exercises (ILEs). The method used was based on the line intercept technique.

The exercise was performed in two parts:

- Part I - Measurement of Reference Images
- Part II - Measurement of Material Microstructures (Metallographic Images)

This report, CMMT(A)230, is concerned with Part II. Report CMMT(A)154 provides information on Part I [1]. A discussion of grain size distribution measurement methods was given in Part I. For the ILE in Part II sets of reference images and instructions were circulated to an inner circle of direct participants (APPENDIX A). Seven organisations provided measurement data for analysis. The measurement exercise was supported by modelling studies using 2-D sections taken from a 3-D distribution of grains. A tetrakaidecahedron was used as the model shape [1].

Background

There is a generic requirement for improved guidelines for the measurement of grain size distribution in a wide range of industrial materials. The properties of metallic products are frequently controlled by the grain size and size distribution of the underlying microstructures. UK industry faces international competition and steady improvements in consistency and performance are needed each year to maintain and enhance competitiveness. Standardised methods of measurement of structure are required to ensure that materials with repeatable properties are produced and a link between processing and structure reinforced.

It is widely recognised by industry that uniformity or otherwise of grain size distribution is an important feature of the microstructure that controls properties. In both single and multiphase materials the measurement of an average value of the grain size is, in itself, vital and there are existing and widely used standards for this purpose, such as ASTM E112, E1181 and E1382. However, there are no widely agreed procedures within the UK for measuring the equally important grain size distribution. Some routines are included in ASTM E1181 but they are seen to be inadequate by UK industry and there is a need to improve measurement practice. Background information on the different standards was given in the Part I report [1].

2 MATERIALS

Metallographic images were prepared (Figs 1-4) from four materials provided by the ILE participants.

Al alloy	AA1050	Alcan International Nominally equiaxed, commercially pure Al
Al alloy	AA5182	Alcan International Nominally equiaxed, Al alloy
Ni alloy	Waspaloy	Incotest Wide grained alloy
Ni alloy	IN909	Incotest Wide grained alloy

3 ILE PARTICIPATION AND PLAN

The following organisations contributed to the Part 2 ILE on metallographic images:

Organisation	Contact	Abbreviated name
Alcan International London & Scandinavian Metallurgical Co Ltd*	J Brown	ALCAN
National Physical Laboratory	E Birch	LSM/A-C
Rolls Royce	E Bennett	NPL
Sheffield Testing Laboratories	G Baxter	ROLLS ROYCE
Sheffield Hallam University	G Black	STL
Alstom	J Cawley	Sheffield Hallam
	G McColvin	ALSTOM

*Three sets of measurements were provided by different operators at LSM, but only on the first task in the ILE (see below).

The exercise consisted of two tasks

- Participants made measurements of intercept lengths within each grain on a diagonal reference line from corner to corner on one micrograph from each material.
- Participants made measurements of intercept lengths within each grain along lines on each image that they drew themselves. Five images were provided for each material.

Magnifications were calculated at NPL using images of a graticule of a given length taken under the same conditions as the reference images.

An abbreviated instruction set is repeated in APPENDIX B.

4 ILE RESULTS AND ANALYSIS

Participants provided the following results

- A calculated value of arithmetic mean linear intercept from the reference line.
- A calculated value of arithmetic mean linear intercept from their own set of lines on each image.
- A spreadsheet of individual intercept values for subsequent analysis.

The results were used to plot comparative figures of mean values and standard deviations together with analysis of different types of distribution data. An experimental investigation of the use of running average data values was also explored as a contribution towards answering the question - “how many intercept values need to be measured to give a specific value of uncertainty?”.

The distribution data were also compared with data from modelling studies using the tetrakaidecahedron.

4.1 MEAN VALUES

Mean values were obtained from 3 sets of measurements on the four materials.

- Arithmetic mean linear intercept from the reference line.
- Arithmetic mean linear intercept from the participants own lines.
- Mean value of estimated area from squaring the intercept data on participants lines.

The arithmetic mean linear intercepts and normalised values of these parameters are given in Table 1 and Figs 5-6. Arithmetic mean linear intercepts, arithmetic estimated areas, and normalised values of these parameters from measurements on participants own lines are given in Table 2 and Figs 7-10. Values for the parameters and their coefficients of variation are summarised in Tables 3 and 4.

Table 1
Diagonal Reference Line

AA 1050	Arithmetic mean linear intercept, μm^*	Normalised intercept
NPL	45.0	1.06
Alcan	42.8	1.00
Rolls Royce	39.1	0.92
LSM/A	47.2	1.11
LSM/B	45.7	1.07
LSM/C	47.3	1.11
Alstom	45.0	1.06
Sheffield Hallam University	30.0	0.70
Sheffield Testing Laboratories	41.6	0.98

AA5182	Arithmetic mean linear intercept, μm^*	Normalised intercept
NPL	39.9	1.04
Alcan	39.3	1.03
Rolls Royce	42.0	1.10
LSM/A	42.0	1.10
LSM/B	42.2	1.10
LSM/C	37.7	0.98
Alstom	36.9	0.96
Sheffield Hallam University	27.8	0.73
Sheffield Testing Laboratories	39.3	1.03

Waspaloy	Arithmetic mean linear intercept, μm^*	Normalised intercept
NPL	22.1	1.01
Alcan	22.4	1.02
Rolls Royce	18.5	0.84
	22.4	1.02
LSM/B	22.2	1.01
LSM/C	23.8	1.09
Alstom	23.9	1.09
Sheffield Hallam University	16.0	0.73
Sheffield Testing Laboratories	25.6	1.16

IN909	Arithmetic mean linear intercept, μm^*	Normalised intercept
NPL	11.1	1.1
Alcan	11.4	1.0
Rolls Royce	10.5	0.9
LSM/A	10.9	1.1
LSM/B	11.1	1.1
LSM/C	10.3	1.1
Alstom	12.5	1.1
Sheffield Hallam University	10.2	0.7
Sheffield Testing Laboratories	13.2	1.0

* About 20 intercepts were measured along the reference line

Table 2
Metallographic Image - Participants Lines

AA1050	Arithmetic mean estimated area, μm^2	Arithmetic mean linear intercept, μm	Normalised area	Normalised intercept	Number of intercepts measured
NPL	4622	56.8	1.30	1.14	208
Alcan	3986	53.5	1.12	1.08	496
Rolls Royce	3393	49.2	0.96	0.99	234
Alstom	3164	48.0	0.89	0.97	390
Sheffield Hallam University	2987	44.1	0.84	0.89	206
Sheffield Testing Laboratories	3157	46.5	0.89	0.94	288

AA5182	Arithmetic mean estimated area, μm^2	Arithmetic mean linear intercept, μm	Normalised area	Normalised intercept	Number of intercepts measured
NPL	1391	31.5	1.11	1.03	204
Alcan	1333	31.7	1.06	1.04	403
Rolls Royce	1142	29.6	0.91	0.97	235
Alstom	1258	31.0	1.00	1.02	241
Sheffield Hallam University	1231	30.1	0.98	0.99	208
Sheffield Testing Laboratories	1190	28.9	0.95	0.95	231

Waspaloy	Arithmetic mean estimated area, μm^2	Arithmetic mean linear intercept, μm	Normalised area	Normalised intercept	Number of intercepts measured
NPL	550	19.2	0.83	0.91	420
Alcan	642	20.9	0.97	0.99	346
Rolls Royce	648	20.2	0.98	0.96	233
Alstom	850	25.1	1.28	1.20	535
Sheffield Hallam University	616	19.7	0.93	0.94	194
Sheffield Testing Laboratories	680	20.6	1.02	0.98	264

IN909	Arithmetic mean estimated area, μm^2	Arithmetic mean linear intercept, μm	Normalised area	Normalised intercept	Number of intercepts measured
NPL	175	10.2	0.86	0.94	509
Alcan	201	11.3	0.99	1.04	643
Rolls Royce	202	10.3	0.99	0.94	262
Alstom	285	13.8	1.40	1.27	530
Sheffield Hallam University	152	8.8	0.75	0.81	239
Sheffield Testing Laboratories	209	11.1	1.02	1.02	289

Table 3 - Mean Values

	Magnification**	Mean linear intercept (reference line) μm	Mean linear intercept (from participants line) μm	Mean area* (estimated) μm^2
AA1050	240	42.6	49.7	3552
AA5182	473	38.3	30.5	1258
Waspalloy	500	21.9	21.0	664
IN909	500	11.2	10.9	204

* Estimated from squaring intercept values.

** Provided by NPL.

**Table 4 - Summary of Measurements
Coefficient of Variation, %**

	Magnification**	Mean linear intercept, μm , (from reference line)	Mean linear intercept, μm , (from participants line)	Mean area* (estimated) μm^2
AA1050	240	12.7	9.4	17.7
AA5182	473	11.2	3.7	7.3
Waspalloy	500	13.3	10.0	15.2
IN909	500	8.9	15.2	22.1

* Estimated from squaring intercept values

** Provided by NPL.

4.2 DISTRIBUTION PARAMETERS

The data obtained from the ILE were plotted using the following methods from the different distribution plotting options described in the part I report [1]:

Set A Number probability against Intercept

Fig 11

Set B Area (E)* probability against Normalised Intercept**

Fig 12

* Area (E) is estimated area from squaring intercept values.

** Intercept values normalised by the arithmetic mean.

Figures 11 and 12 comprise sets of graphs from the individual laboratory participants, with the materials plotted on the same graph for comparison. All the data were plotted as cumulative distributions - this avoids arbitrary decisions about classifying intercept values in bins.

Set A - The number probability plots against intercept value all have a characteristic Γ shape, with a steep initial portion and large tail. The length of the tail corresponds to the larger grains and is more extensive in those alloys with a wider distribution of

grains. As in Part I [1], the plot is not helpful in comparing distribution widths because each alloy has a different mean value of sizes.

Set B - The use of area probability (estimated) plots converts the Γ shape of the number probability plots to a fan-shape with the material with the more uniform distribution having a distribution plot closer to the vertical than the plots of materials with wider size distributions. This is consistent with the modelling predictions (see Part I report [1]).

4.3 RUNNING AVERAGE ANALYSIS

In order to provide information to address the question “how many intercepts need to be counted to give a specified degree of accuracy”, the intercept data were plotted from each participant as a running average mean. This was performed for both intercept and estimated area (intercept squared) values:

Set C Running average intercept against number (plotted by organisation and by material)

Set D Running average estimated area (E) against number (plotted by organisation and by material)

The results are shown in Figs 13-16.

In most cases it can be seen that for both types of graph the running average eventually settles down to a steady value. This happened more quickly on the intercept data than on the estimated area data and most quickly on AA 5182 Al alloy. The two wide grained Ni alloys and the commercial purity Al alloy required additional numbers of intercepts to be measured. It was concluded that about 200 intercepts resulted in a representative mean value of mean linear intercept, but for estimated areas 400 intercepts were required, especially for the wide grained alloys.

4.4 COMPARISON WITH MODELS

Size probability distributions were generated from the ILE intercept data measured at NPL and compared with the tetrakaidecahedron models, following the conclusions outlined in Part I regarding the discriminability of size probability plots. The Alcan data set is shown in Fig 17.

It is clear that the size distribution data from the Al alloys best fit the model (tetrakaidecahedron) data for the lognormal distribution with a σ of 0.3 while the size distribution data for the Ni alloys fit more closely to tetrakaidecahedron model data with wider distribution parameters. The data for the Ni alloys clearly do not fit a simple single parameter (for the width) distribution and appear to be a mix of distributions. Distribution data are commonly examined for their fit with mathematical models, such as for example the Lognormal, Weibull or other functions [2,3]. These are usually expressed as number probability curves. Figure 18 shows the Alcan data set plotted with a normal probability (by number) ordinate against a log intercept abscissa. The plots deviate at either end of the

distribution demonstrating non-compliance with the lognormal plot. This can be seen more clearly in Fig 19 where the estimated area is plotted against log normalised intercept together with the model tetrakaidecahedron data. It can be clearly seen that a lognormal distribution of grains in 3-D does not produce a lognormal plot of intercepts or areas.

4.5 UNCERTAINTY

Uncertainty is reasonably straightforward to specify in the measurement of a mean linear intercept grain size, because it can be quantified by the standard deviation (coefficient of variation) obtained by making several sets of measurements, either by one operator or by a number of operators. For a distribution this is more complicated because it is characterised by more than one parameter. This is why distribution data are frequently fitted to mathematical models. If the fit is reasonable (and this goodness of fit in itself requires quantification), then values for the parameters that describe the model distribution can be used to quantify the measured distribution. For example, μ_g (geometric mean) and σ (distribution width parameter) characterise a lognormal distribution.

$$f(x) = \frac{1}{x\sigma\sqrt{2\pi}} \exp \frac{-\left(\ln(x) - \ln(\mu_g)\right)^2}{2\sigma^2} \quad (1)$$

Even when a fit may be good over most of the data range the distribution can have “tails” that deviate at the ends. These tails may contain information that is particularly relevant to properties. Consequently there is no substitute for also plotting the full data set, either as a histogram or as a cumulative distribution. The cumulative plot has been chosen for this work because it avoids taking arbitrary decisions on bin sizes (for histograms) and is perceived to provide visual plots that makes discrimination between different materials easier.

The question thus arises; how best to quantify uncertainty when evaluating data as a cumulative distribution plot where the data do not easily fit a mathematical expression that can be represented by a few parameters? Cumulative plots for all the participants for each material are shown in Fig 20. The spread of the data in fans is clearly a visual expression of uncertainty. The data are most tightly spread for the AA 5182 equiaxed Al alloy, and fan out for the materials with a wider grain size. One way of quantifying this uncertainty could be to calculate the standard deviation associated with the normalised intercept values for specified values of cumulative area probability (say 20, 50, 70 and 90%). These are shown in Fig 21. It can be seen that there is a progressive increase in coefficient of variation (CV) as the cumulative area probability increases - consistent with the fanning out of the data. Also the CV is generally higher for the wider grained Ni alloys. There remains the question - how might these values change for data sets with higher or lower numbers of intercepts? Clearly this would be sensible to investigate through the use of the tetrakaidecahedron model approach and future work on this topic is recommended.

The coefficients of variation of the mean values of the exercise (Table 4) are plotted in Fig 22 in comparison with the coefficients of variation obtained in Part I of the exercise. The most significant difference was found from a comparison of the CVs from the reference lines. Clearly the use of reference images with a defined line gave much smaller uncertainties in measurement. However, for the measurements where participants placed their own lines the

results were less clear, sometimes surprisingly so, with slightly lower CVs for the measurements on metallographic images compared with the reference micrographs. The most likely cause of this discrepancy was due to sampling and further work is needed to investigate this issue.

5 SUMMARY AND CONCLUSIONS

Grain size distribution data have been obtained, through an interlaboratory intercomparison, on metallographic images prepared at NPL from four materials:

- CP Al* - AA 1050
- Al alloy - AA5182
- Ni alloy - Waspaloy
- Ni alloy - IN 909

* commercial purity (CP)

The Al alloys contained nominally uniform grain size distributions while the two Ni alloys contained mixed grain sizes.

Data from the reference images were compared with data generated from a 3-D model shape, a tetrakaidecahedron. Analysis of the model data indicated that the use of size distributions rather than number distributions enabled different structures to be discriminated more easily with respect to estimating the width of the distribution.

Uncertainties were calculated for the arithmetic mean values of intercept from the different materials and the wider grained materials generally had higher values of uncertainty. However, it was found difficult to define an uncertainty for the distribution widths. Further work is needed to define a suitable method, especially in those cases where the distribution data do not conveniently fit a mathematical expression.

6 REFERENCES

1. B Roebuck, E G Bennett and A Dickson. NPL Report CMMT(A)154, March 1999. The Measurement for Uncertainty in Grain Size Distribution Interlaboratory Exercise Part 1 - Reference Images.
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7 ACKNOWLEDGEMENTS

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8 FOOTNOTE

One of the graphs in Fig 20 of the report on Part I of the ILE was misplotted. The wrong data was used for the running average area plots for Waspaloy and AA 5182 in the Sheffield Testing Laboratory data set. A corrected graph is included as APPENDIX C in this report.

LIST OF CAPTIONS

	Al alloy - AA1050.
	Al alloy - AA5182.
Fig 3	Ni alloy - Waspaloy.
	Ni alloy - IN909.
Fig 5	Arithmetic mean linear intercepts from the diagonal reference line.
	Normalised values of mean linear intercept from the diagonal reference line.
	Arithmetic mean linear intercepts from participants lines.
	Normalised values of mean linear intercept from participants lines.
	Mean values of estimated areas from participants lines.
Fig10	Normalised values of mean estimated area from participants lines.
	Number probability plotted against intercept.
Fig 12	Area (E) probability plotted against normalised intercept.
	Running average intercept plotted against number (by organisation).
	Running average area (E) plotted against number (by organisation).
Fig 15	Running average intercept plotted against number (by material).
	Running average area (E) plotted against number (by material).
Fig 17	Comparison of distributions from the Alcan data sets and the tetrakaidecahedron model.
Fig 18	Number probability plotted against log intercepts for the Alcan data set.
	Estimated area probability plotted against log normalised intercept and compared with the model tetrakaidecahedron data
Fig 20	Area (E) probability plotted against normalised intercept - all participants for each material.
Fig 21	Coefficient of variation of the normalised intercept at fixed values (20, 50, 70 and 90%) of cumulative area probability.
Fig 22	Coefficient of variation of the mean values obtained by the measurement exercise in parts I and II.

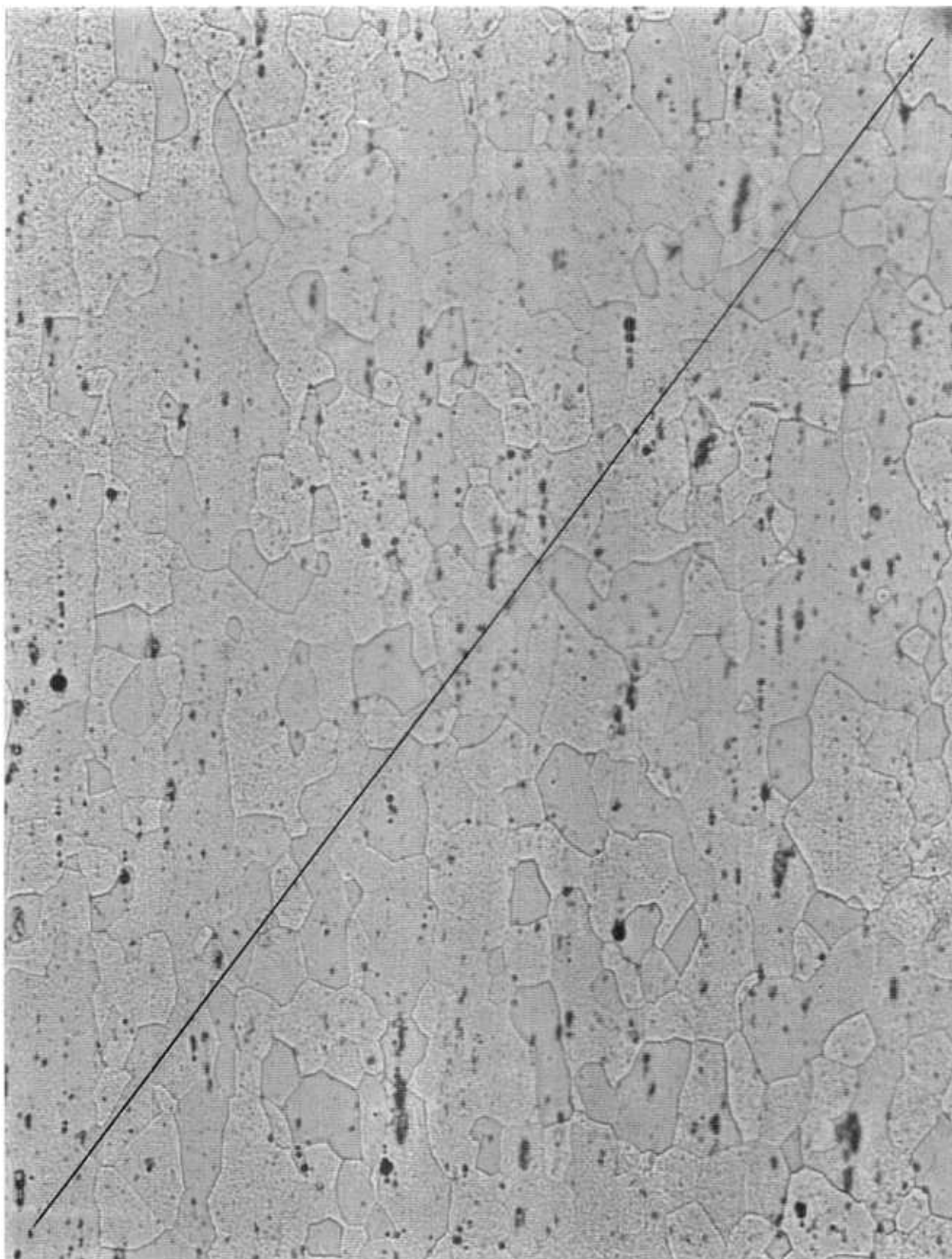


Fig 1 Al alloy - AA1050.



Fig 2 Al alloy - AA5182



Fig 3 Ni alloy - Waspaloy.

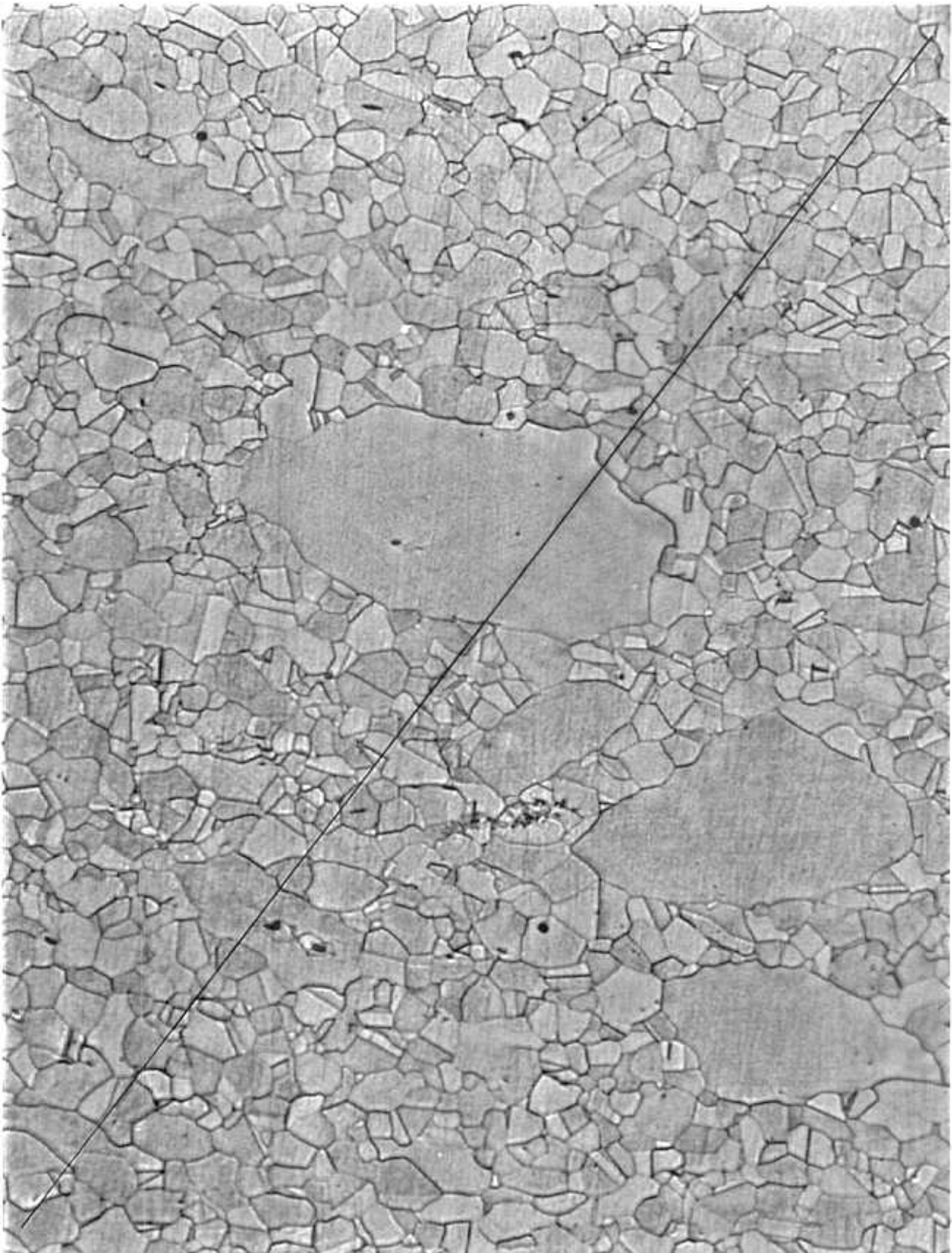
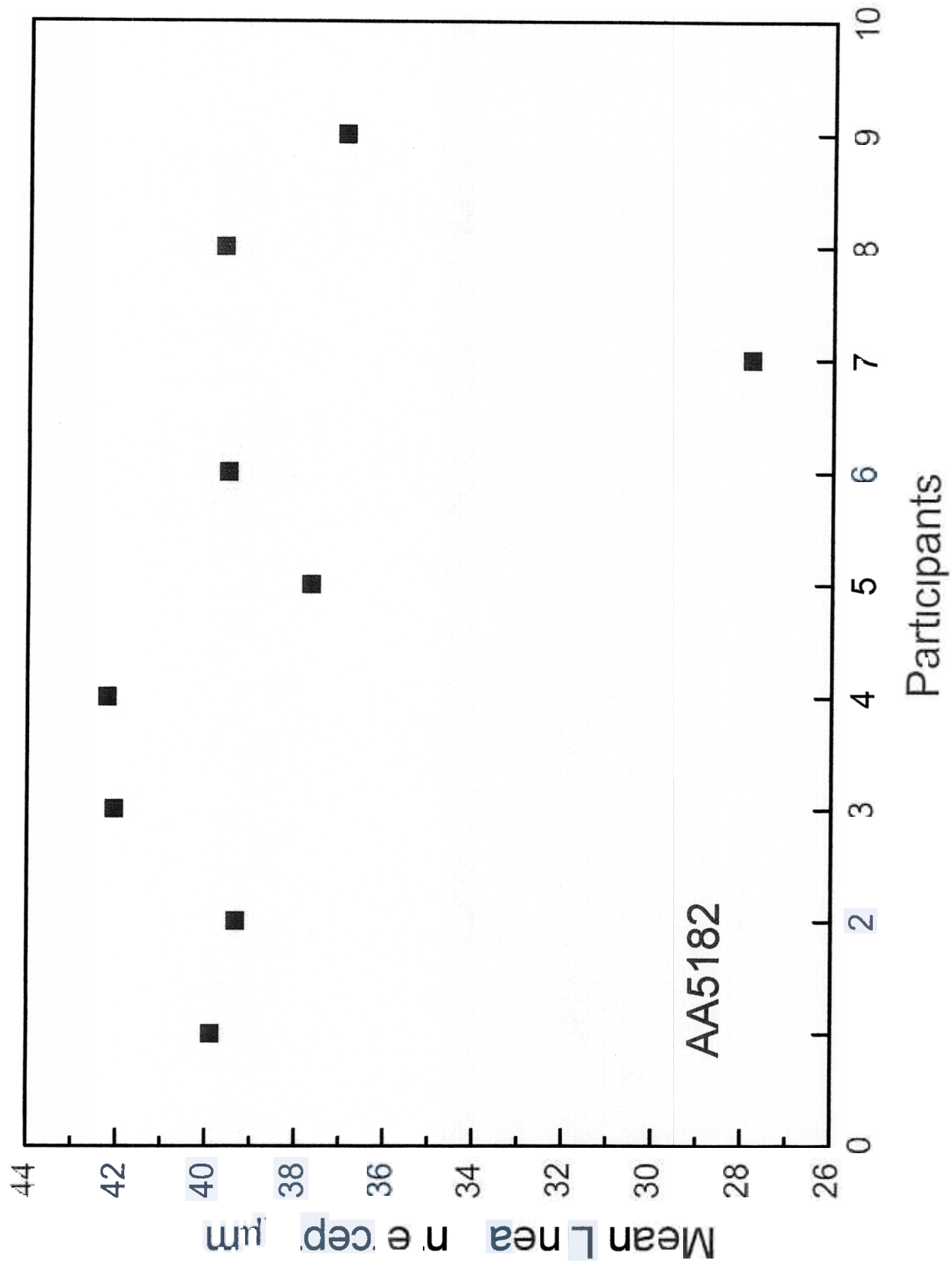
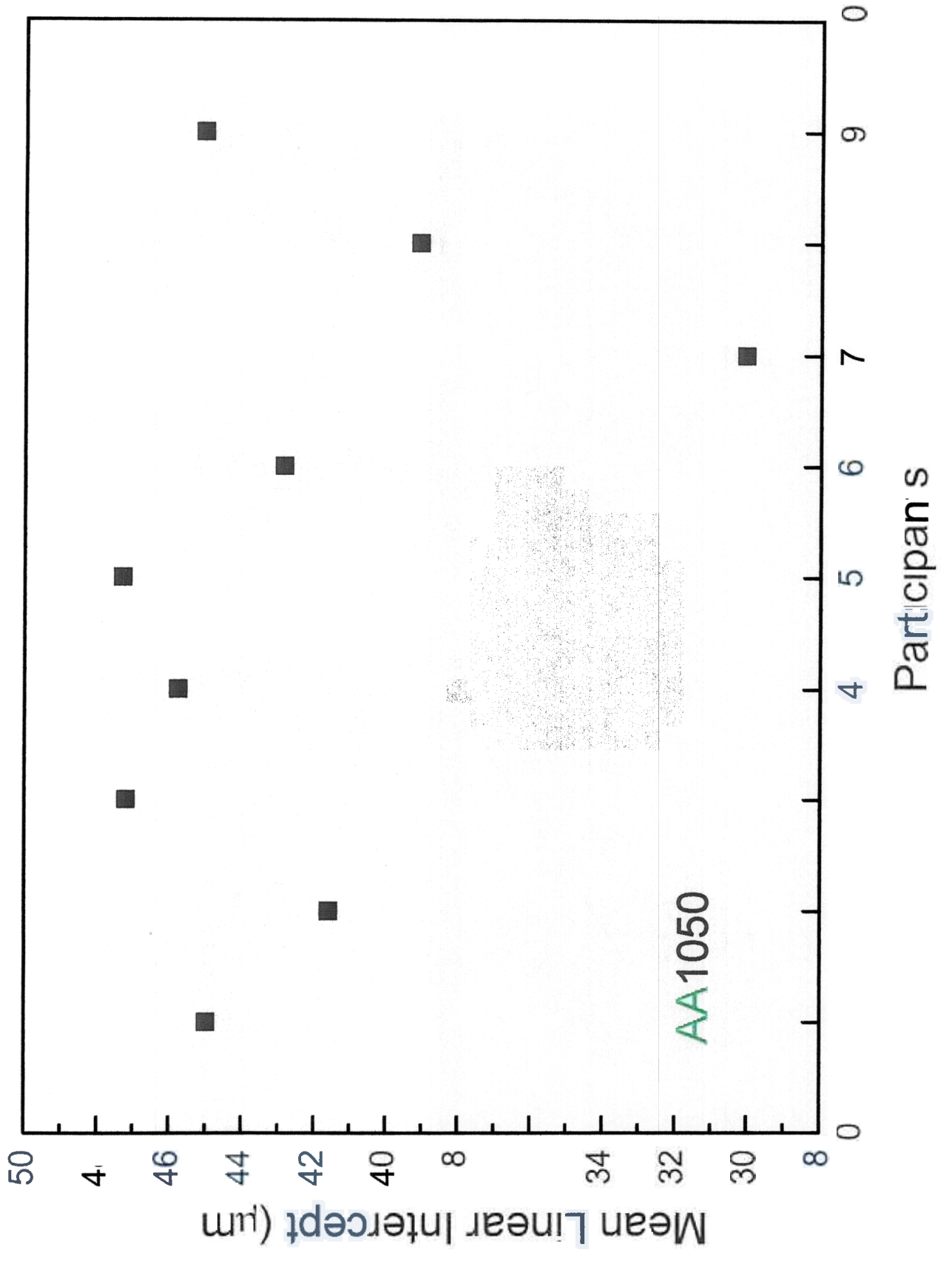
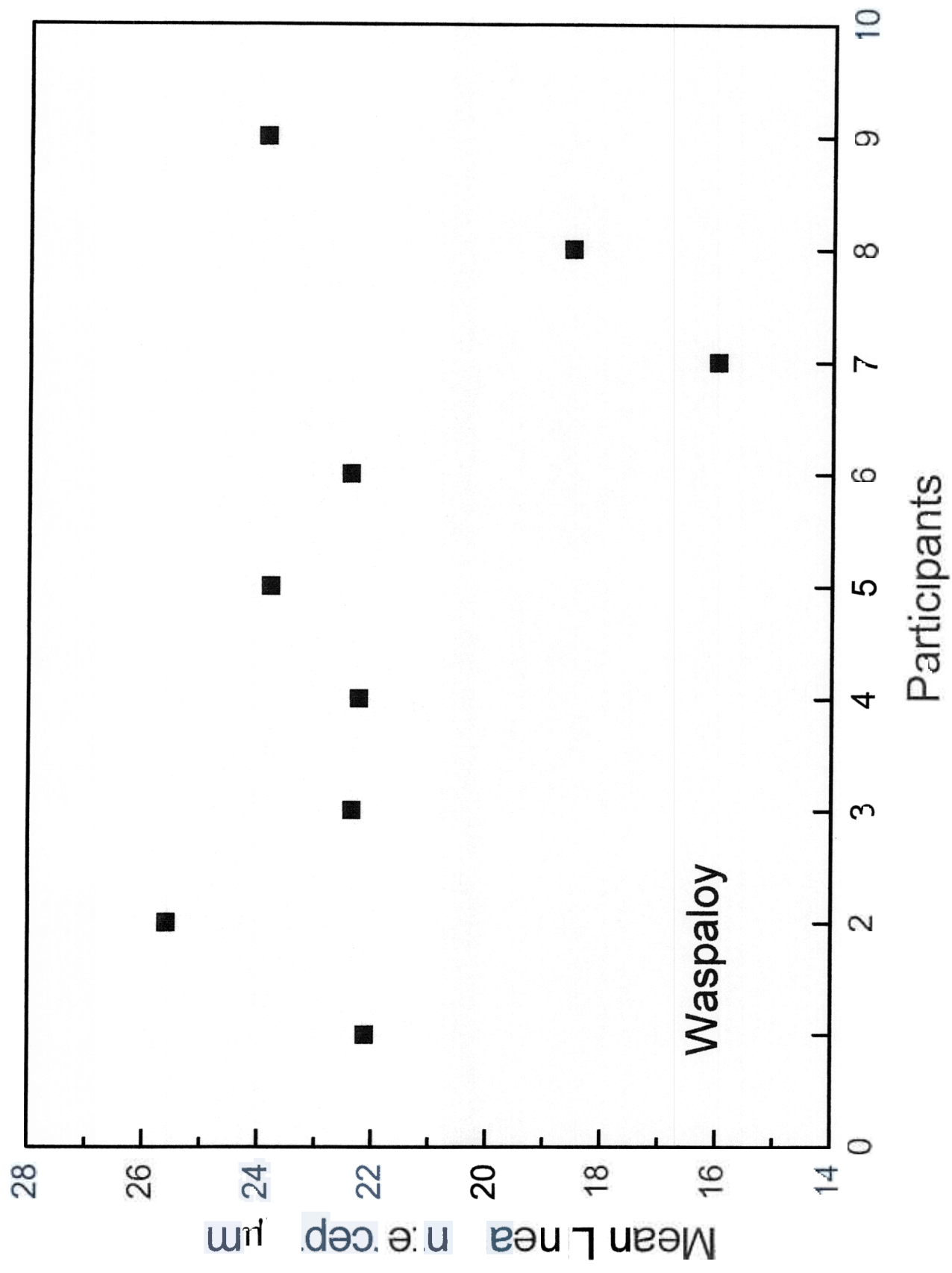


Fig 4 Ni alloy - IN909.

Fig 5 Arithmetic mean linear intercepts from the diagonal reference line.







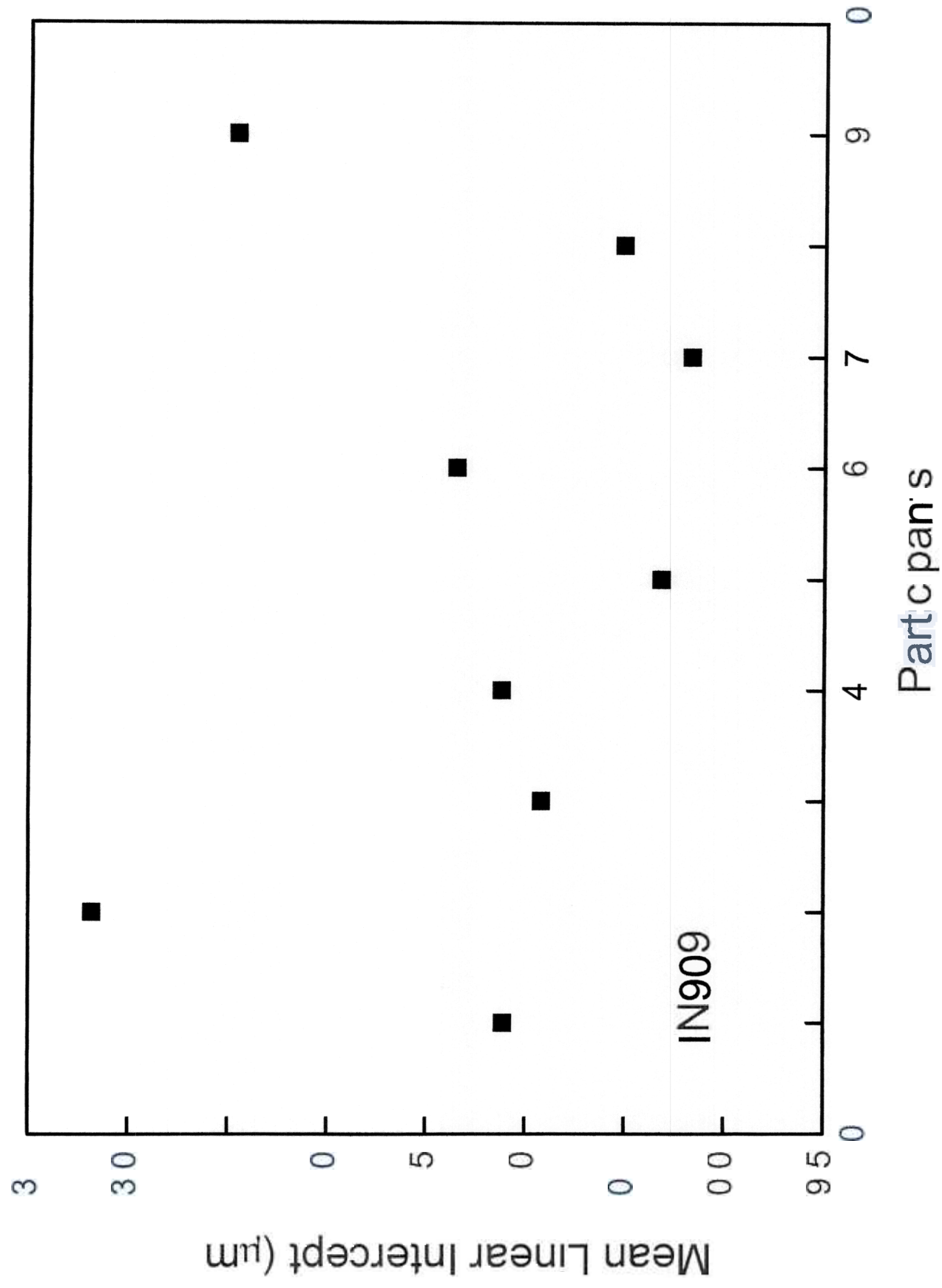
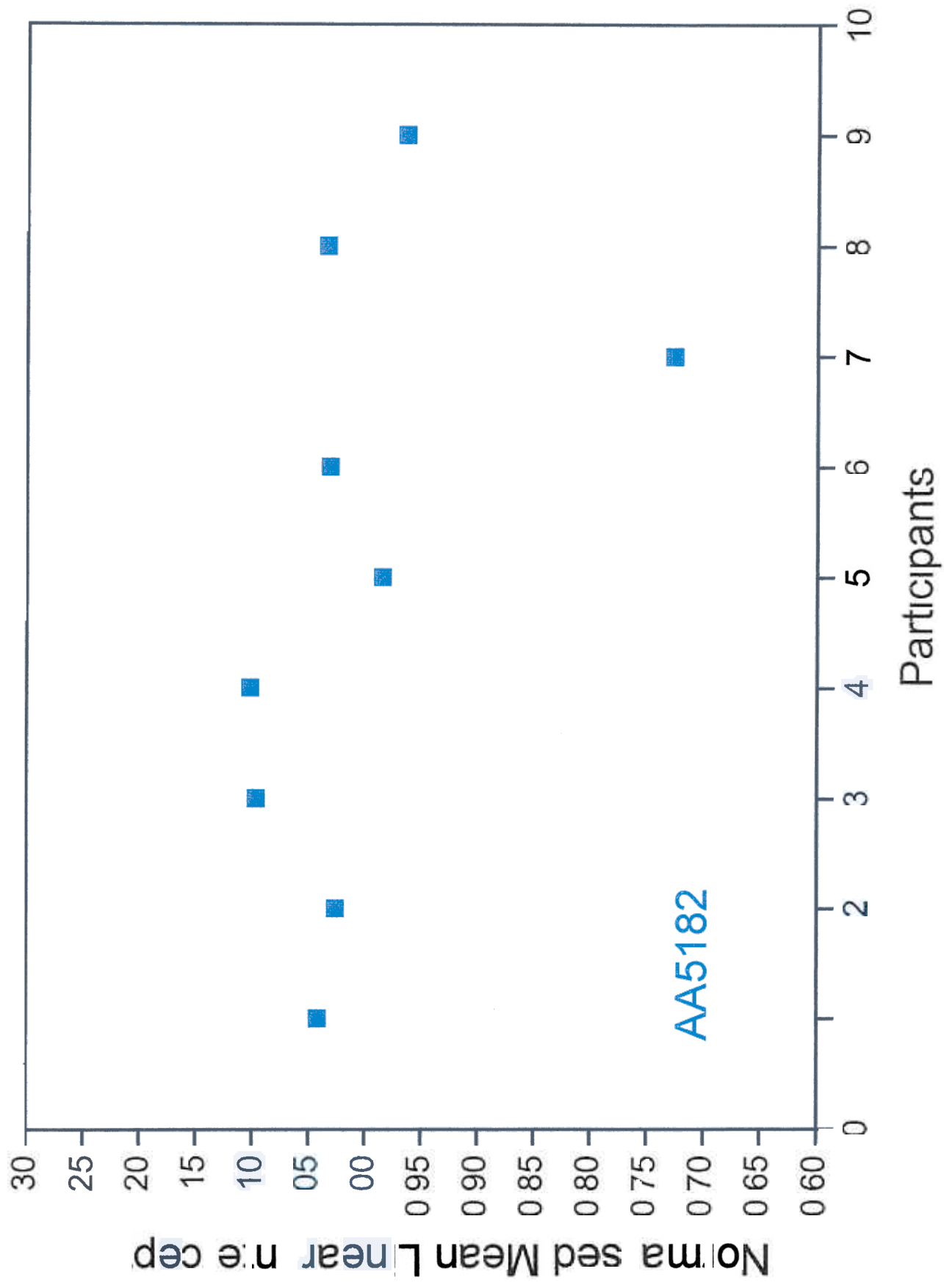
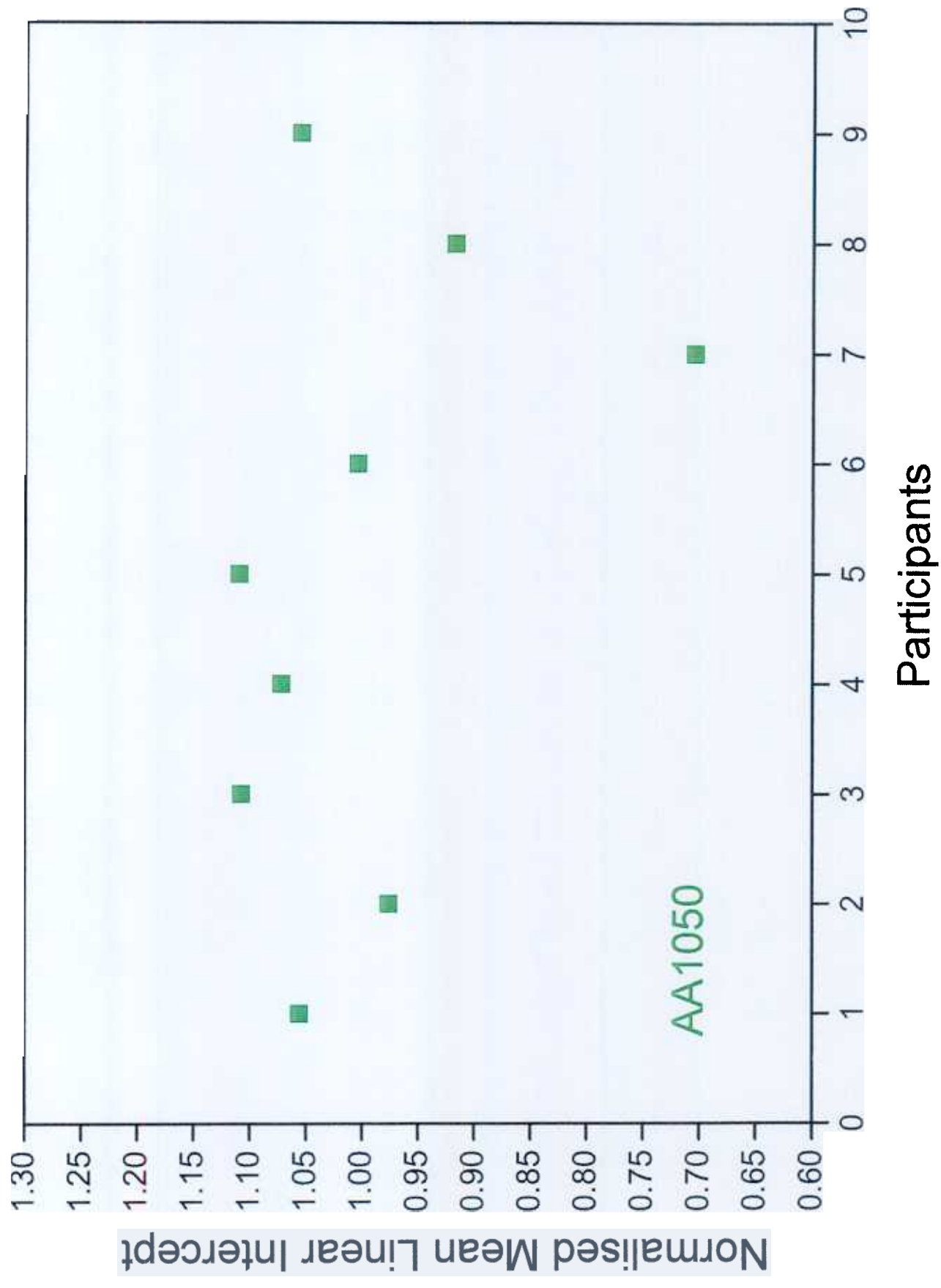
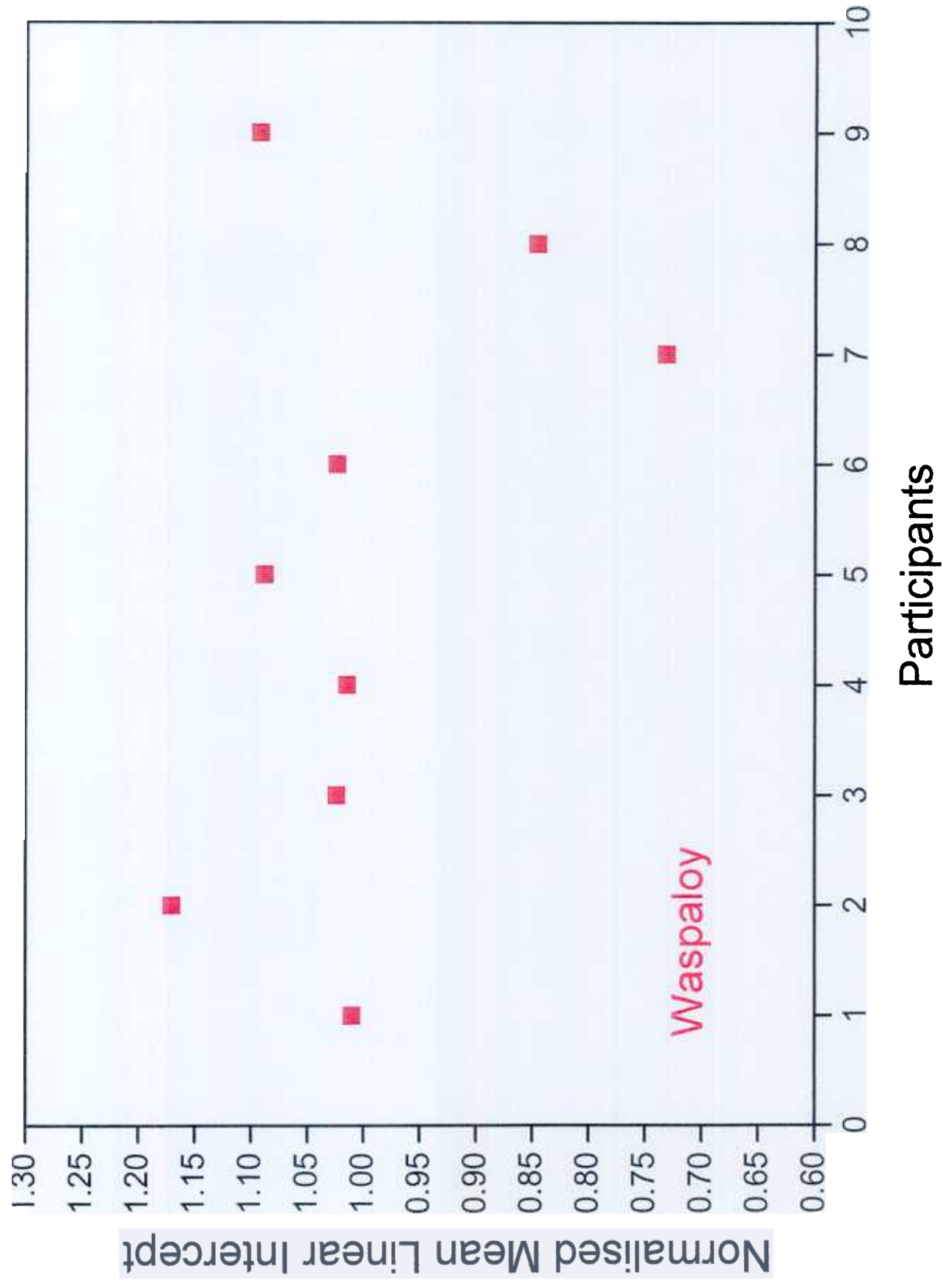


Fig 6 Normalised values of mean linear intercept from the diagonal reference line.







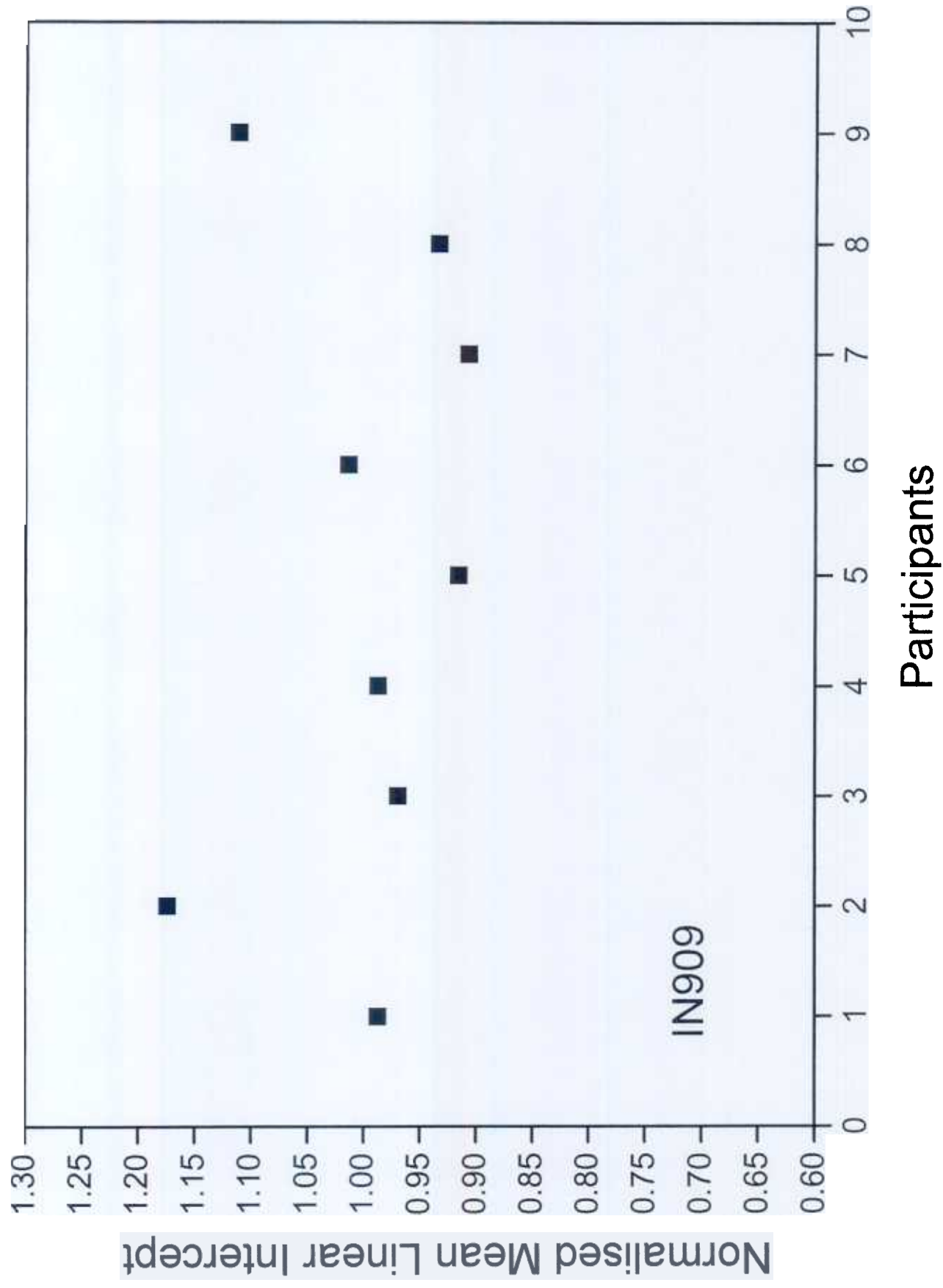
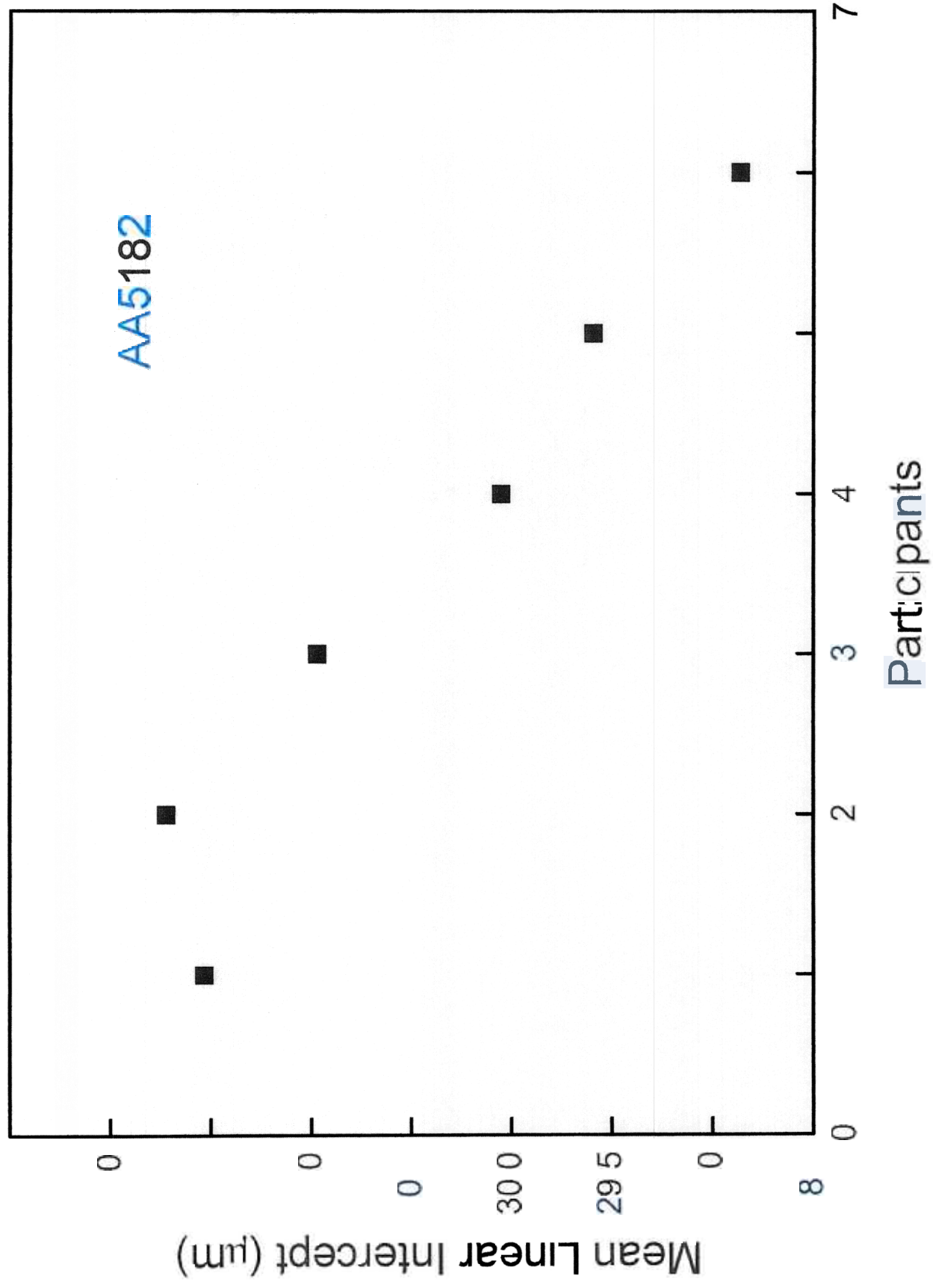
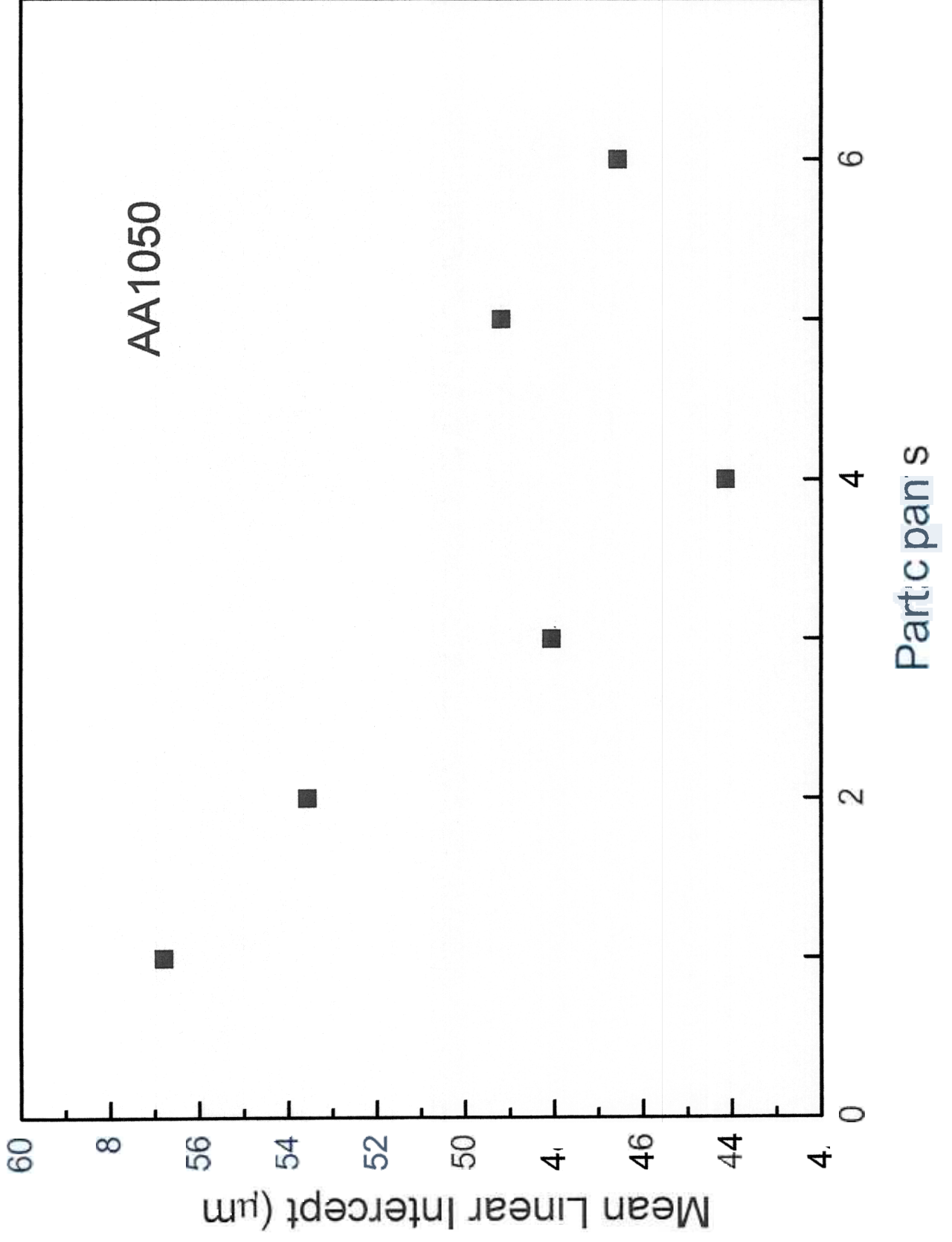
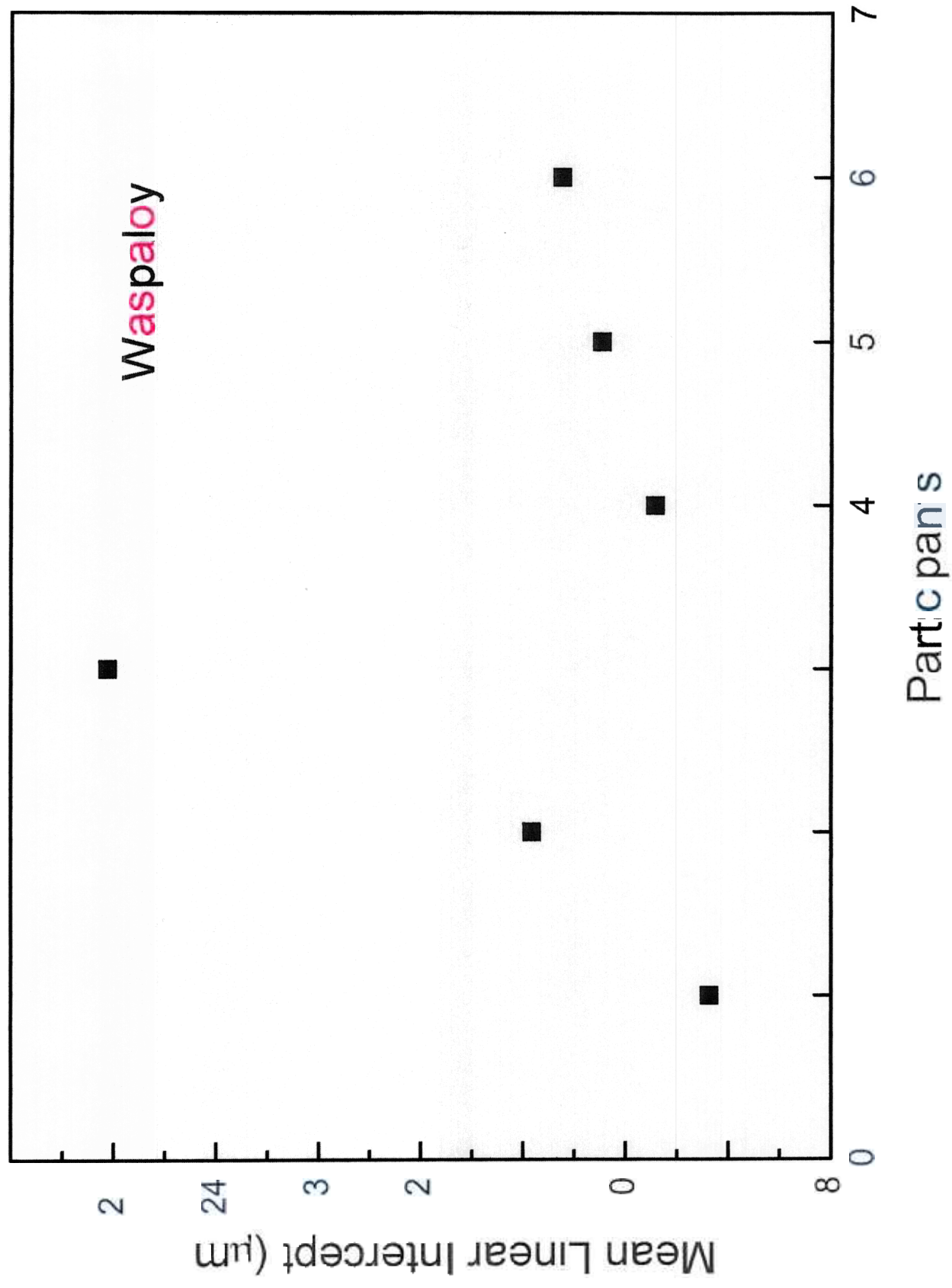


Fig 7 Arithmetic mean linear intercepts from participants lines.







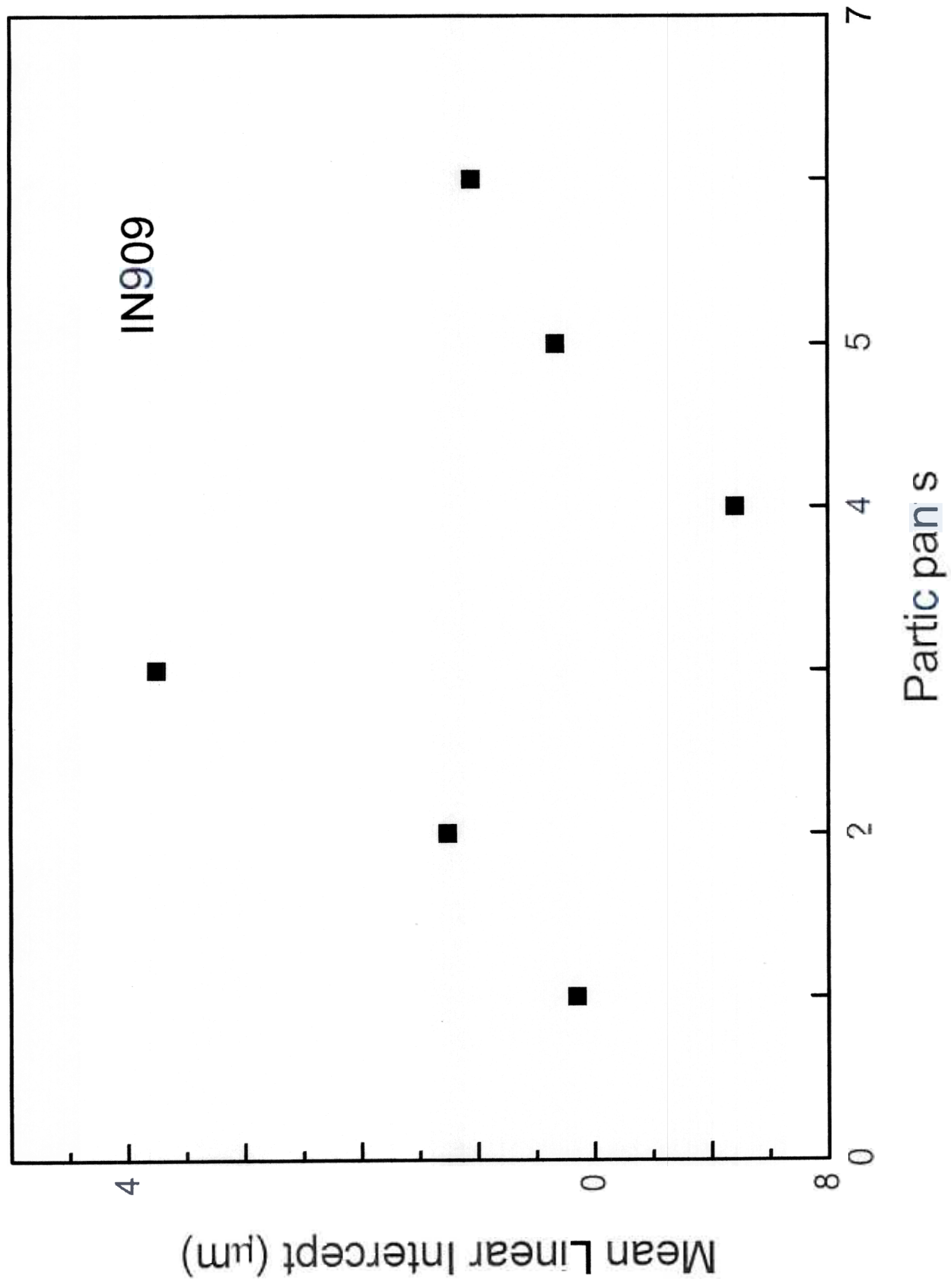
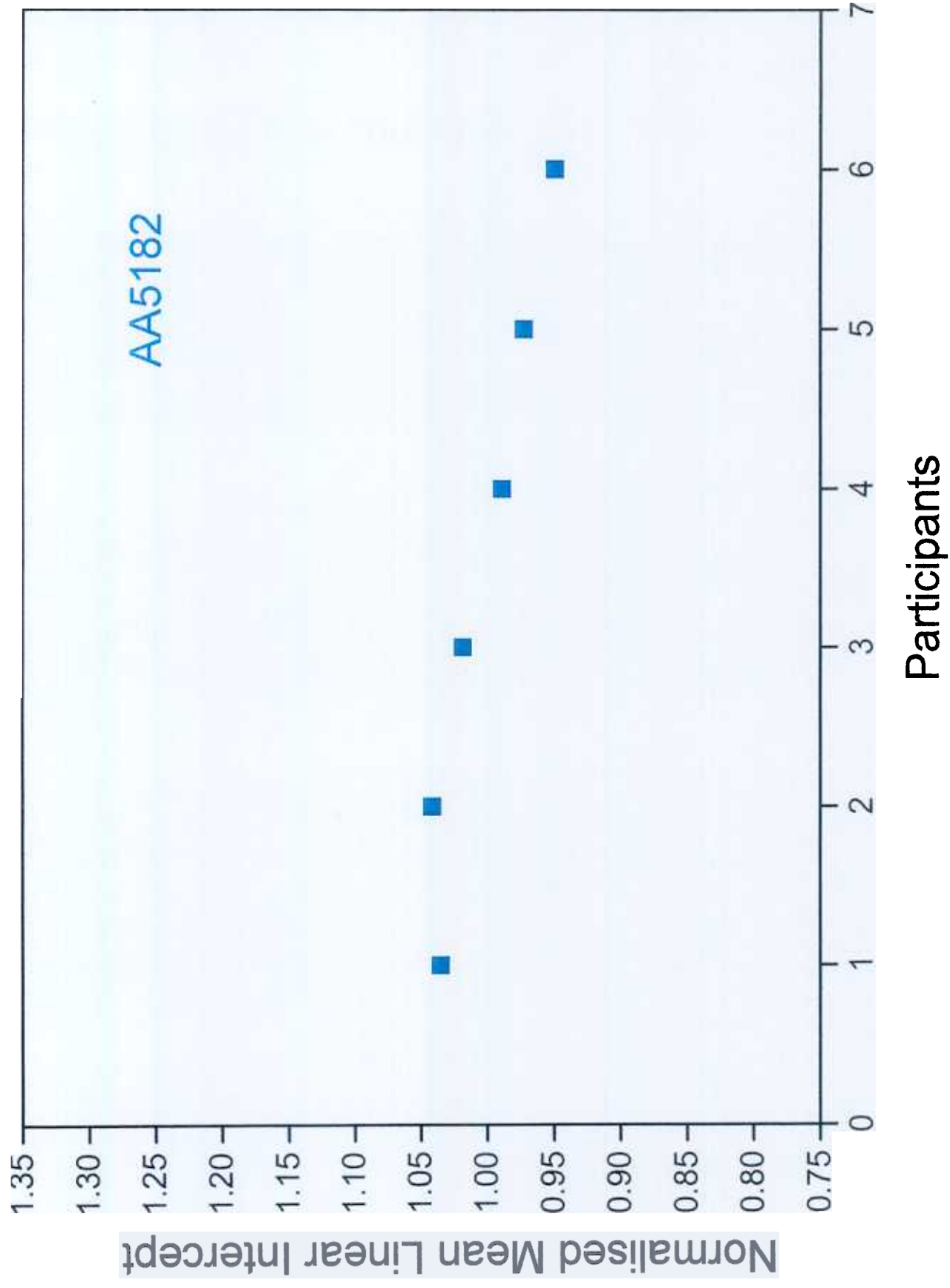
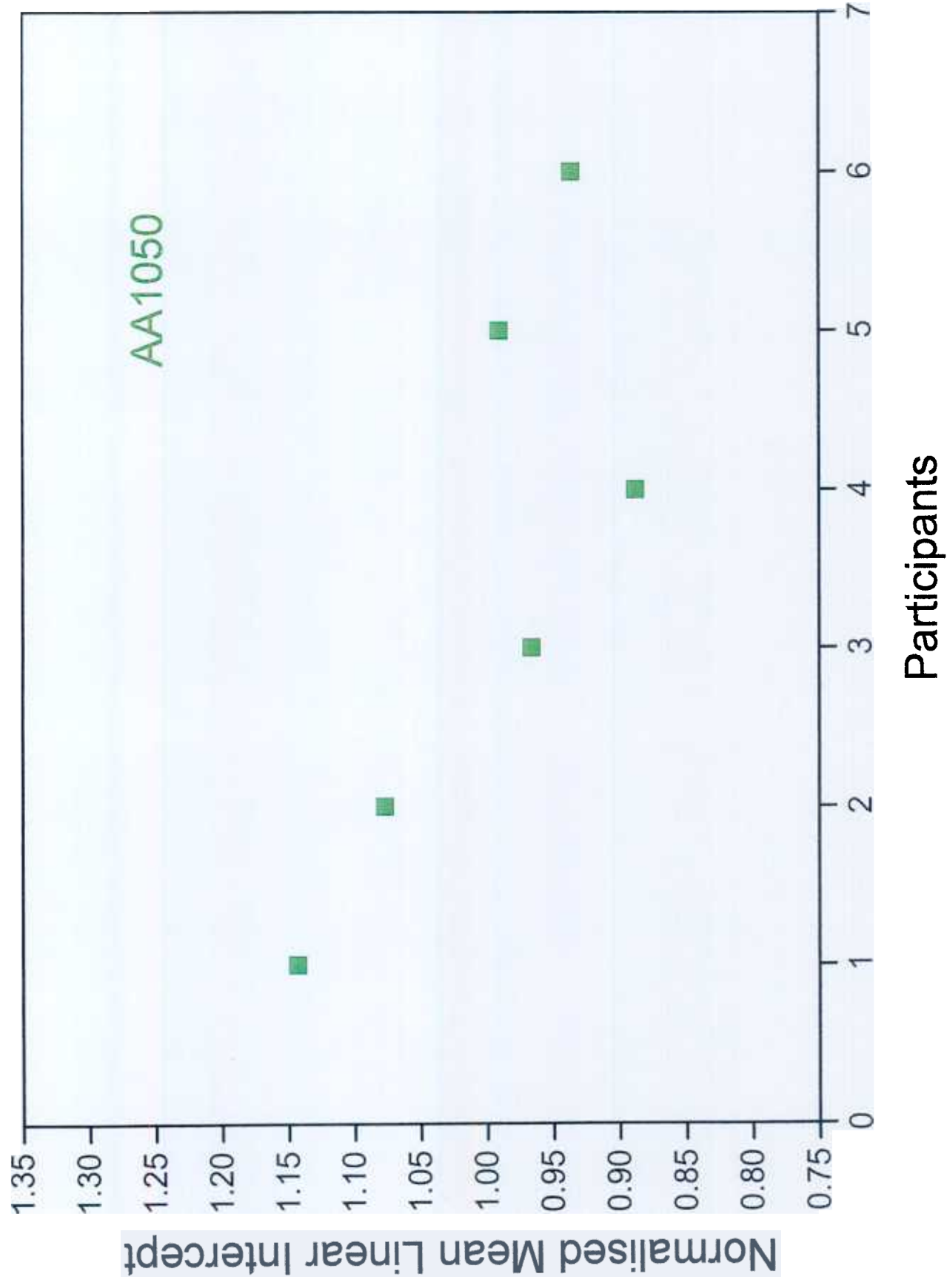
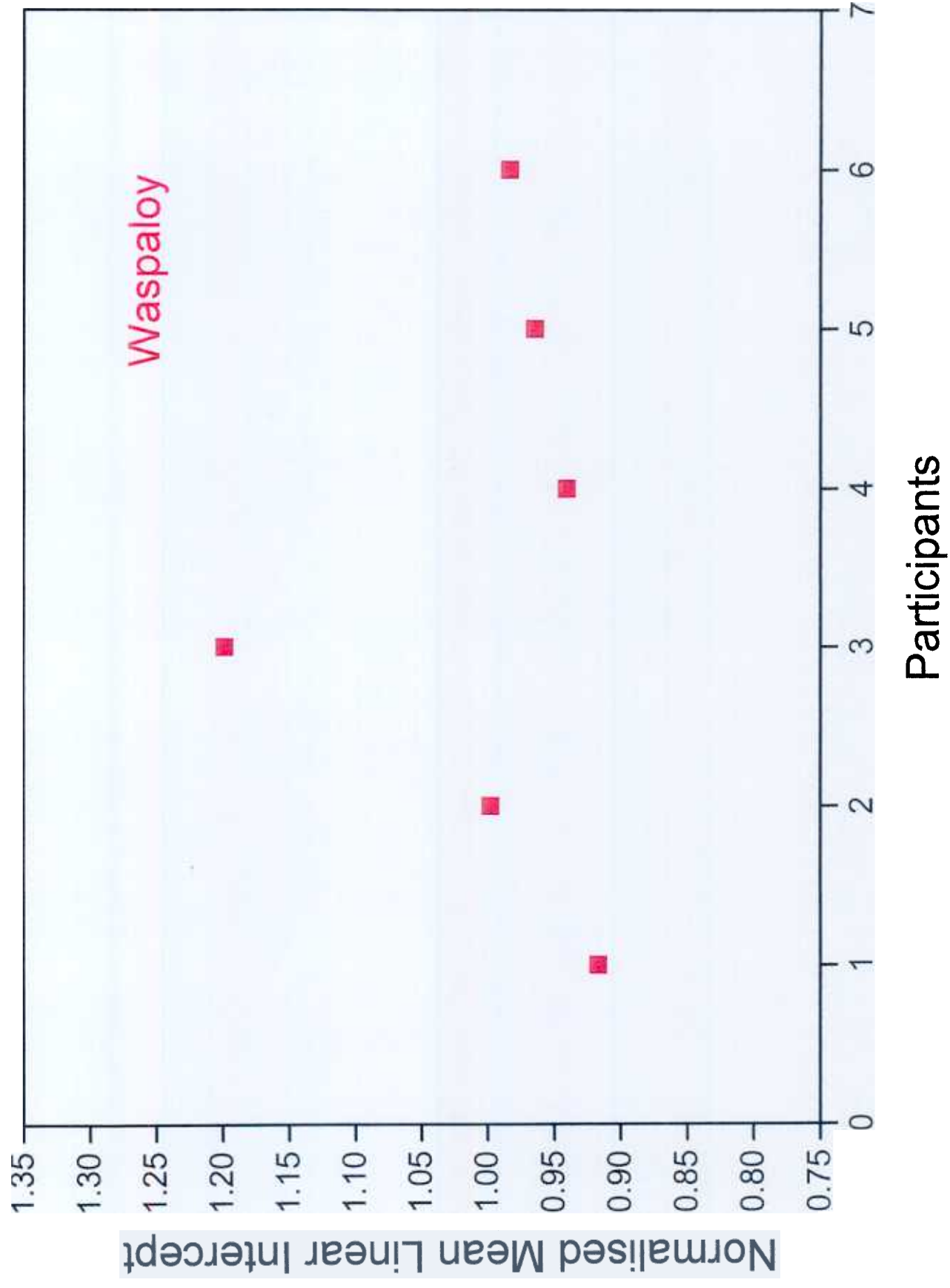


Fig 8 Normalised values of mean linear intercept from participants lines.







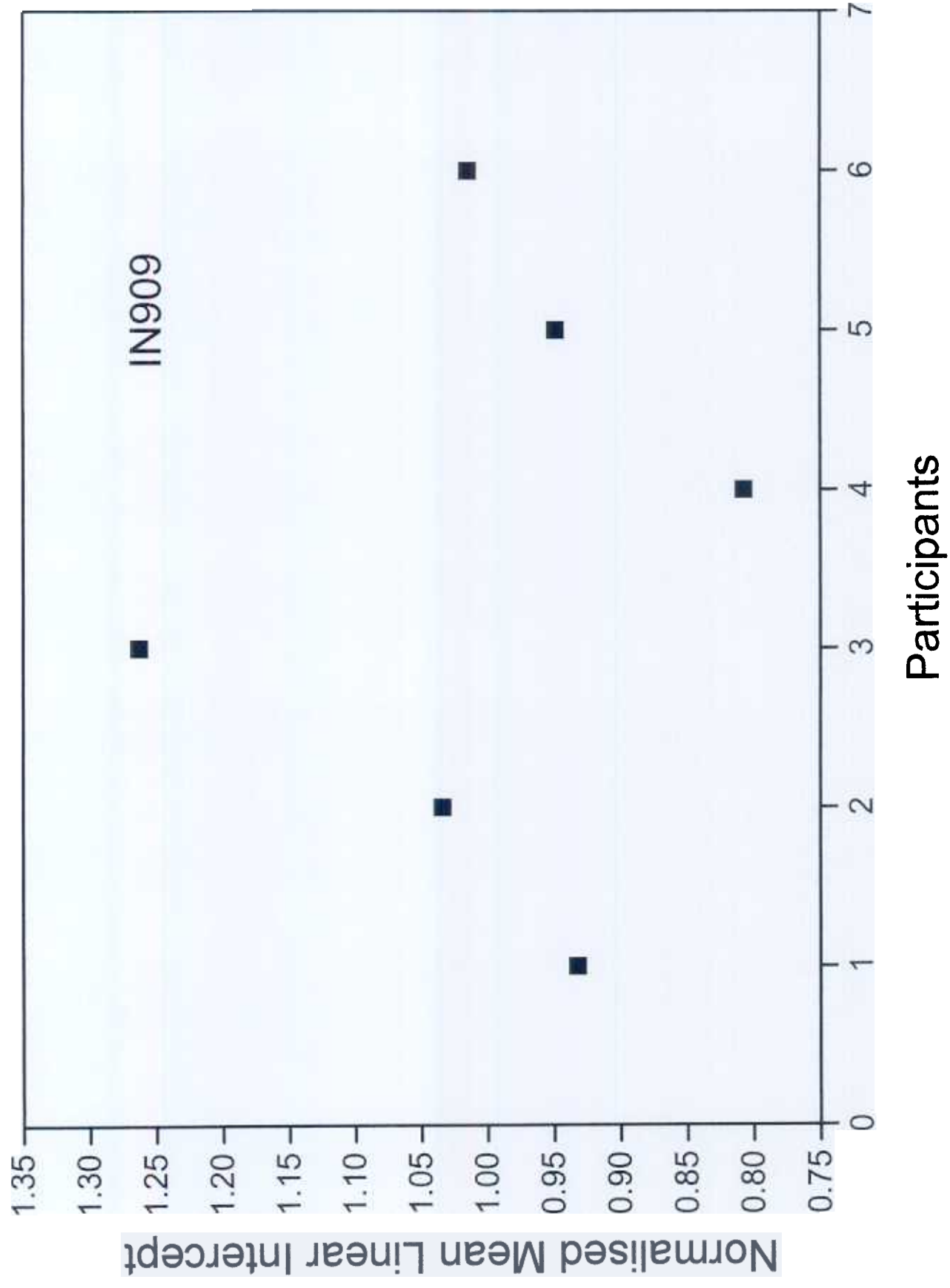
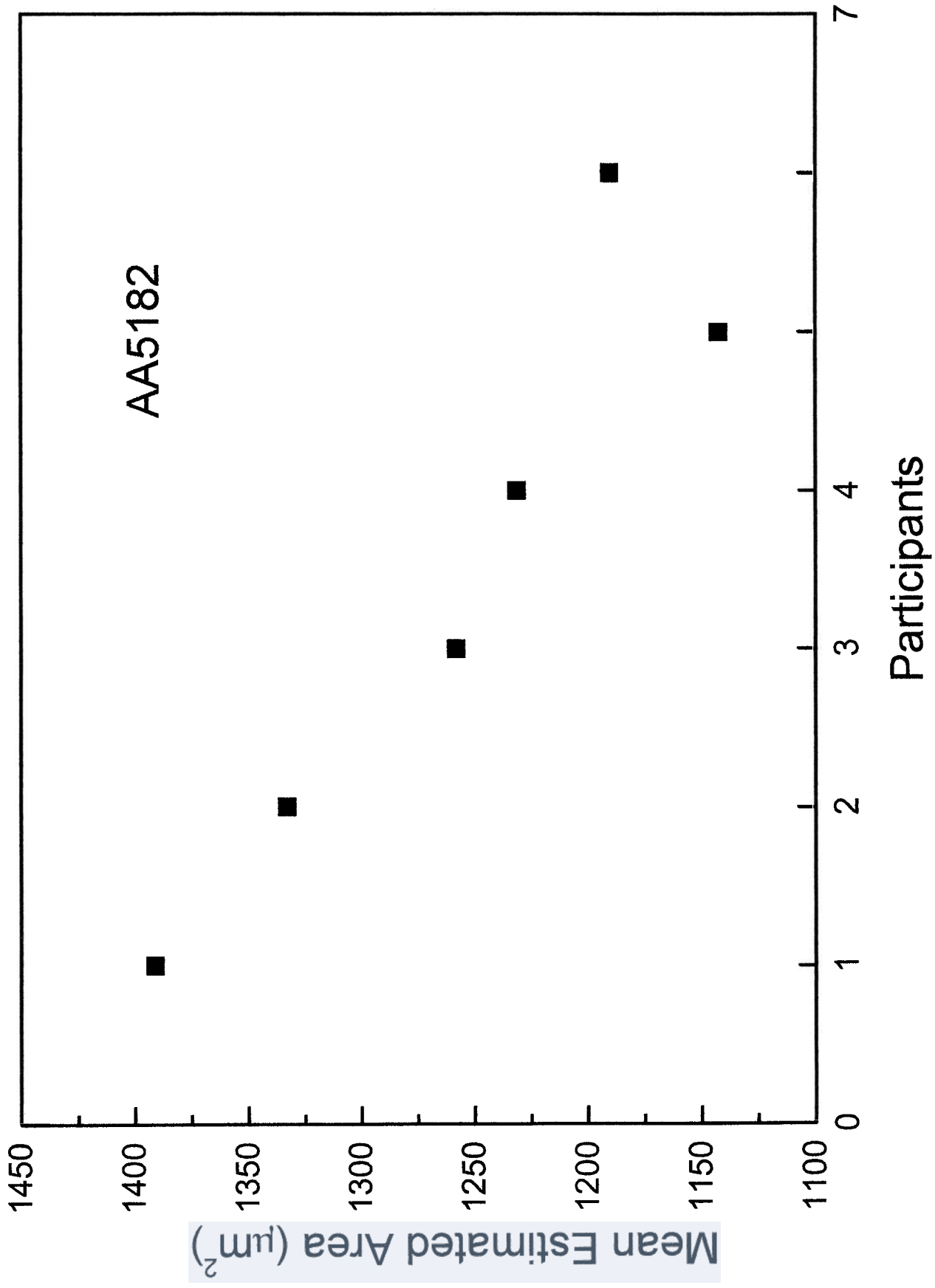
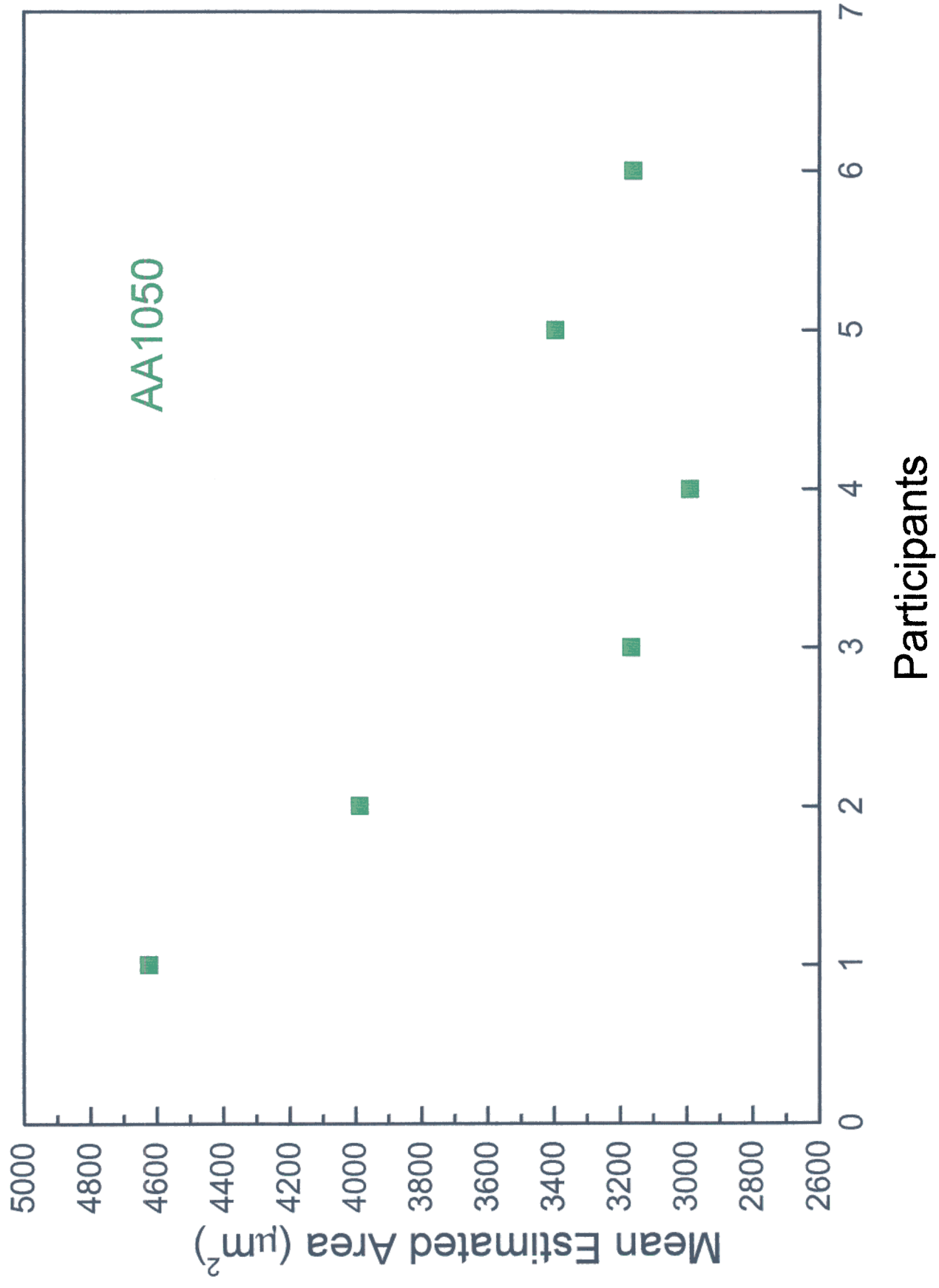
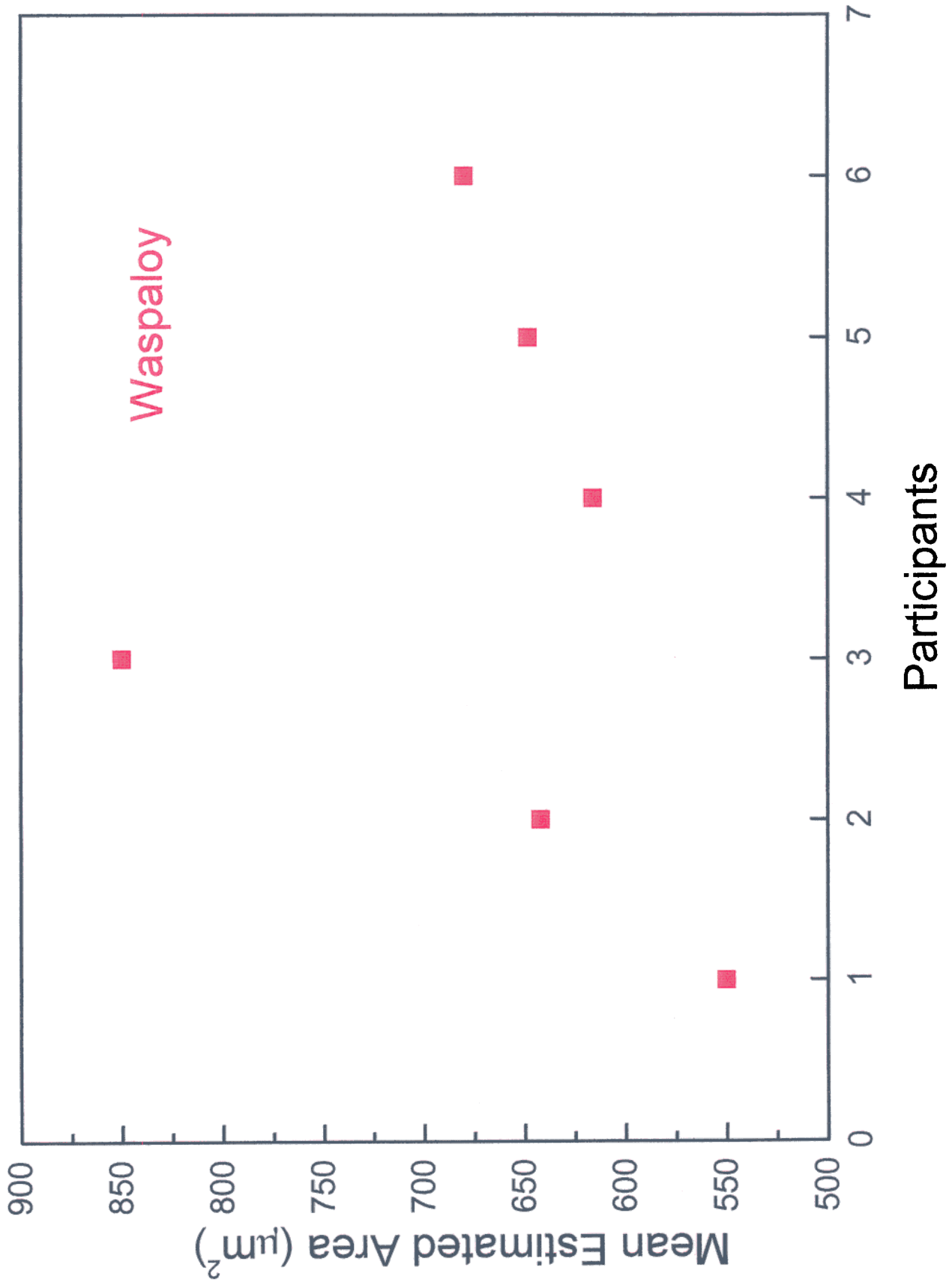


Fig 9 **Mean values of estimated areas from participants lines.**







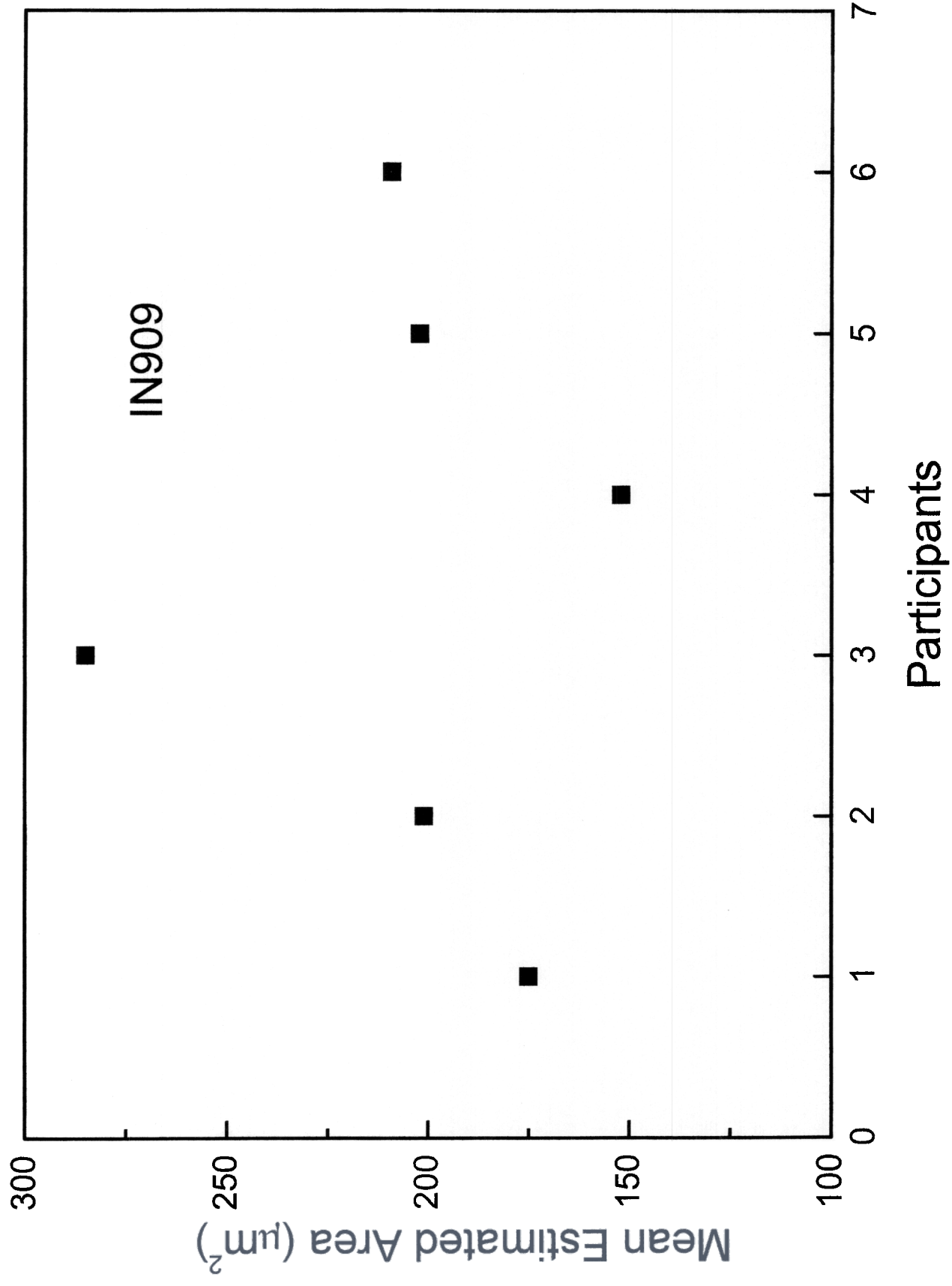
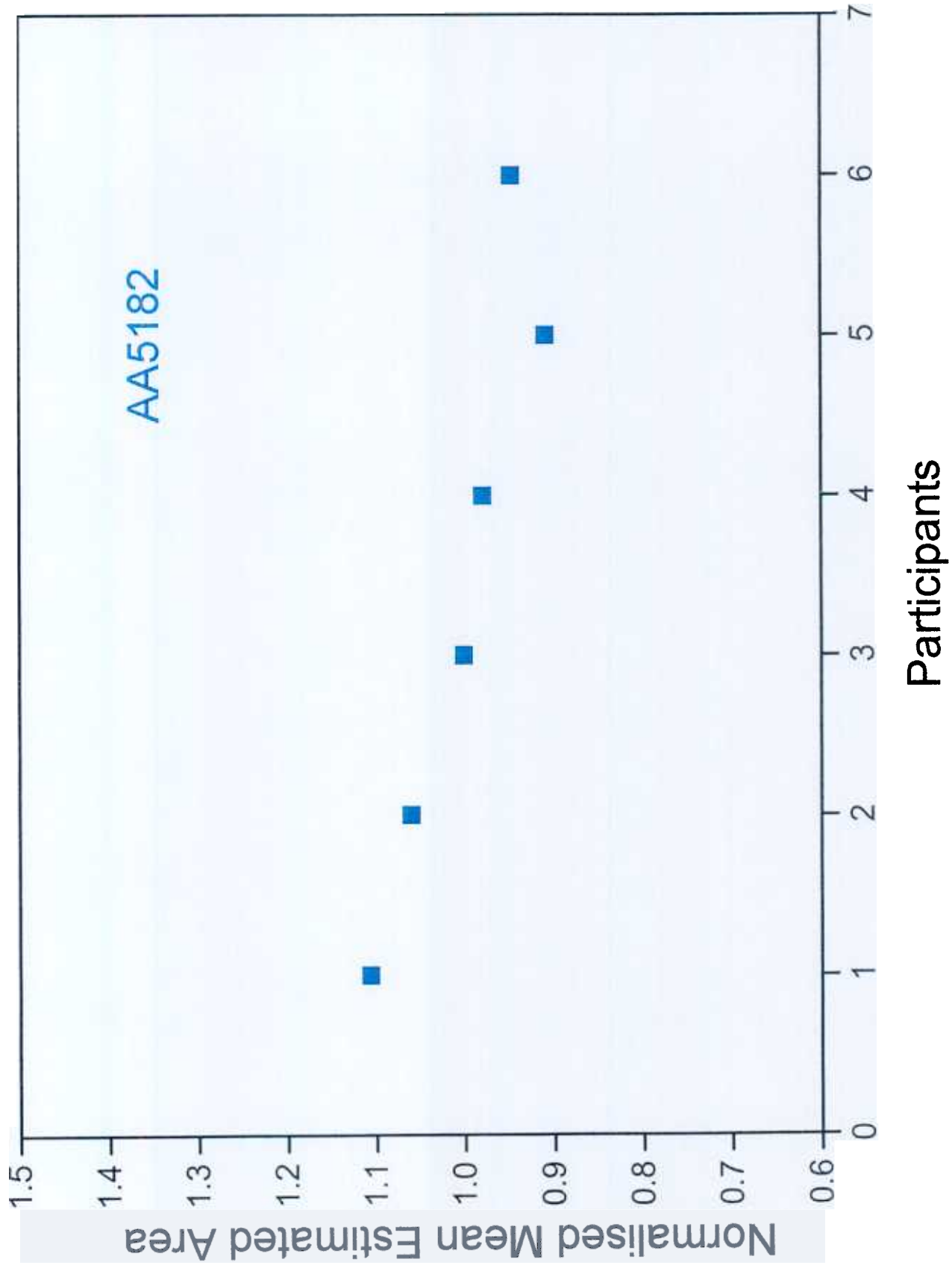
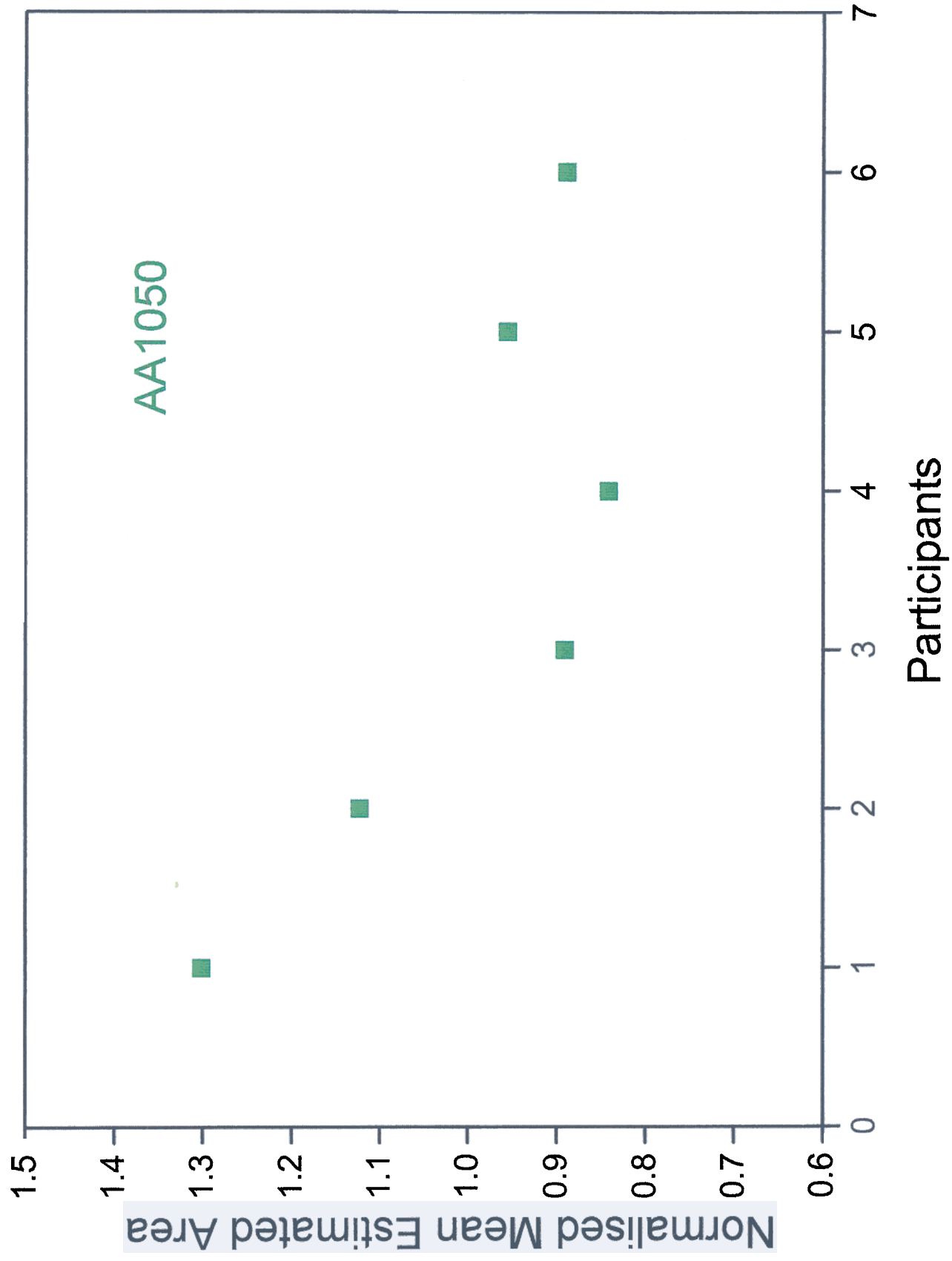
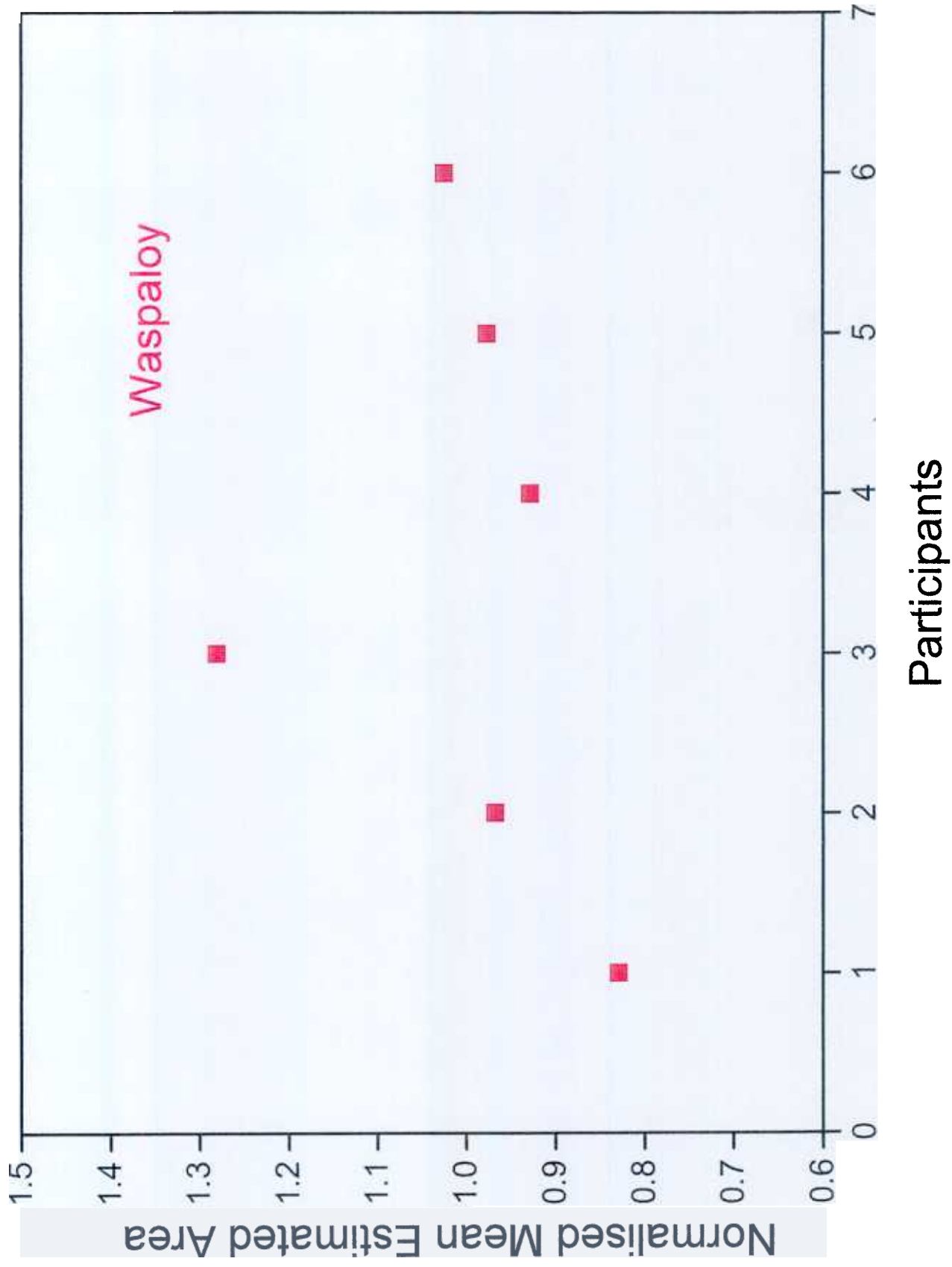


Fig10 **Normalised values of mean estimated area from participants lines.**







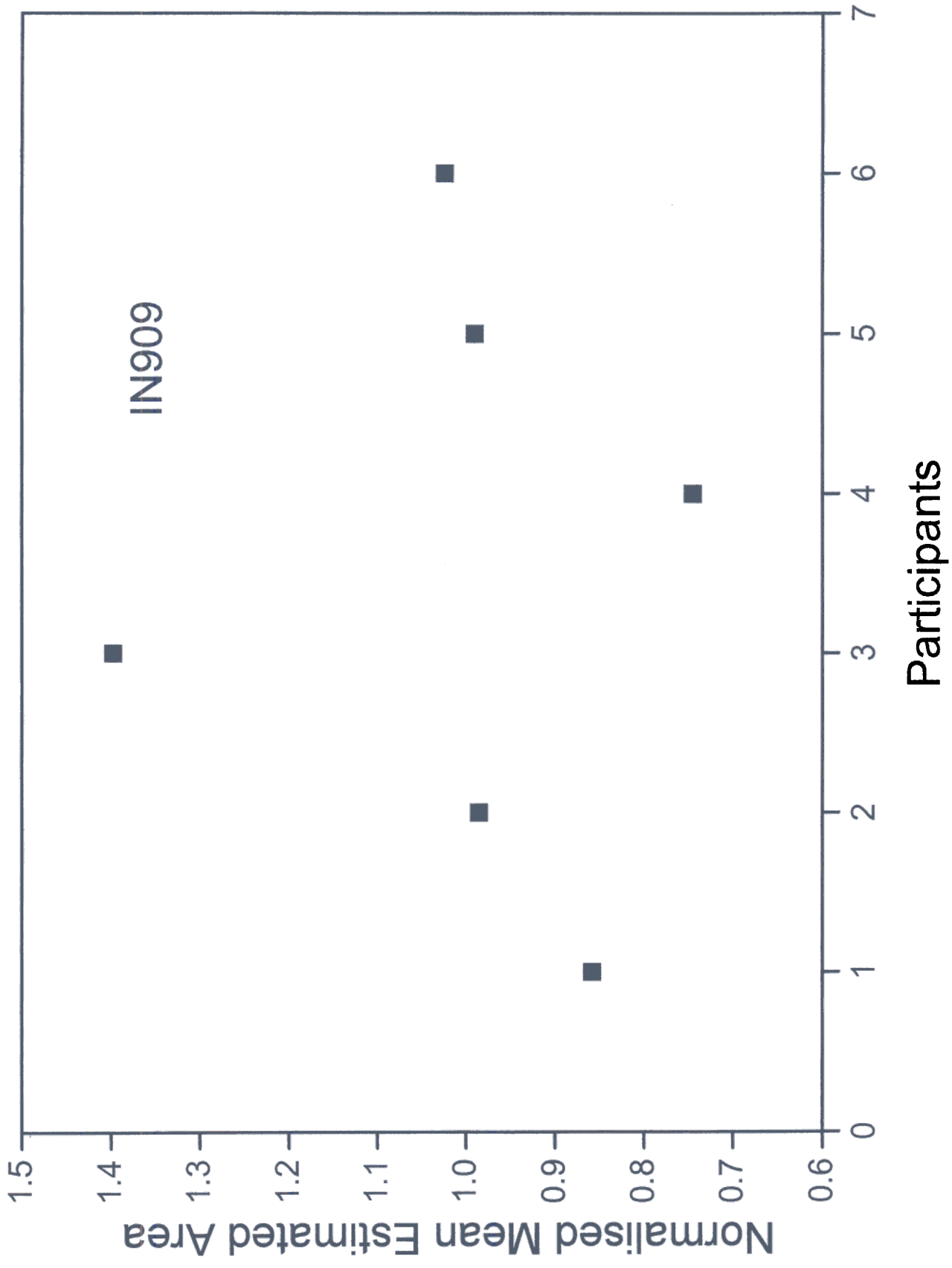
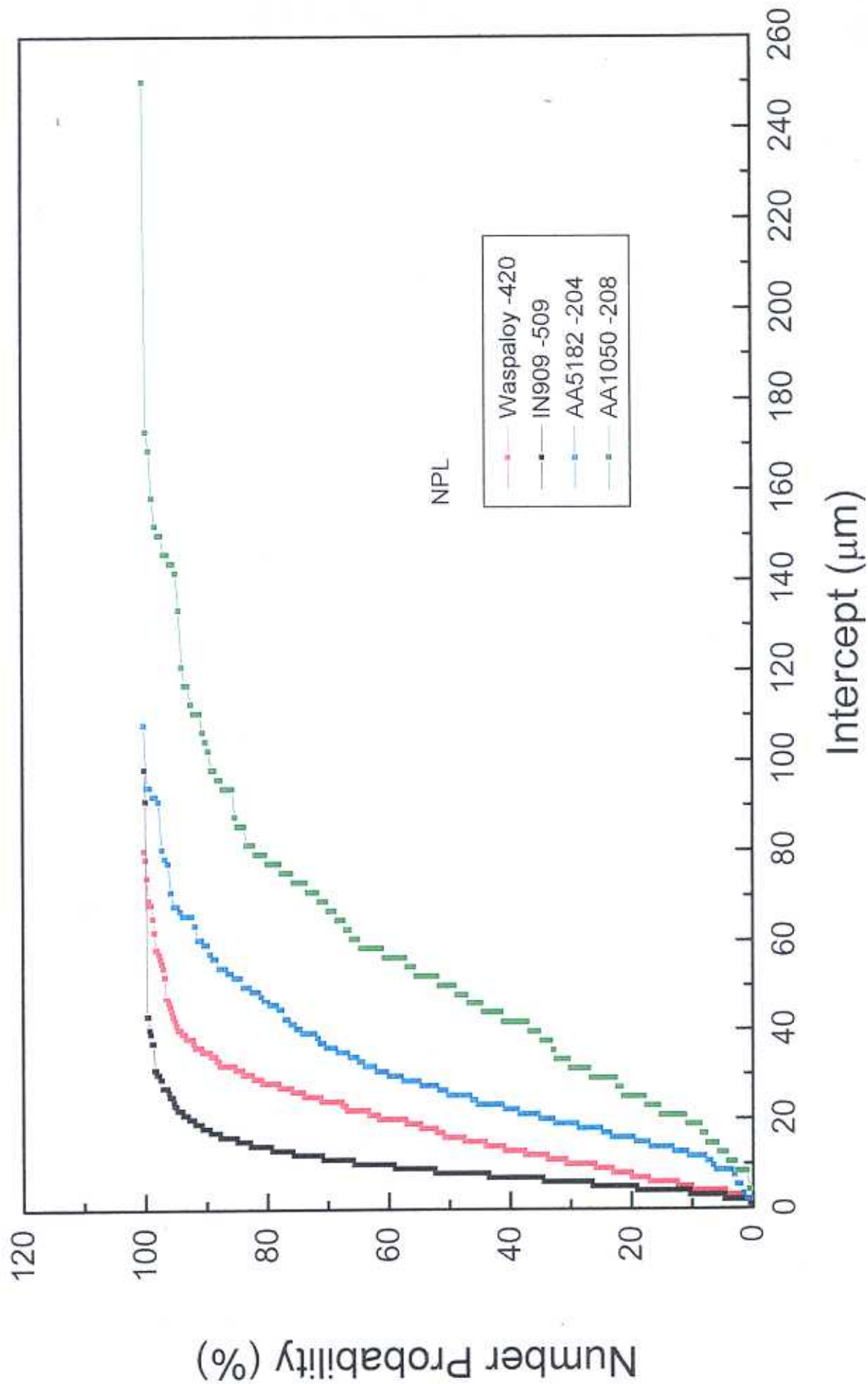
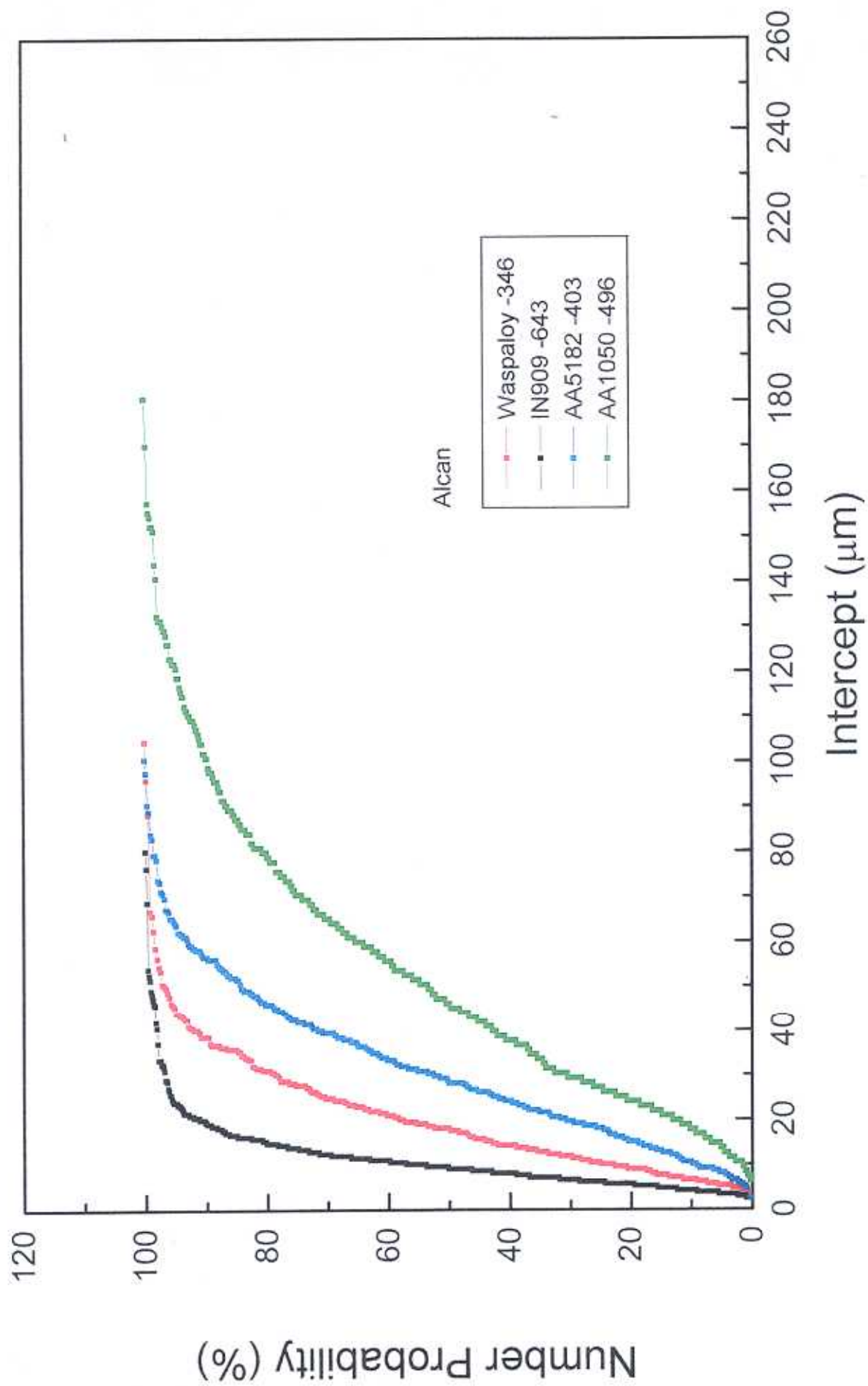
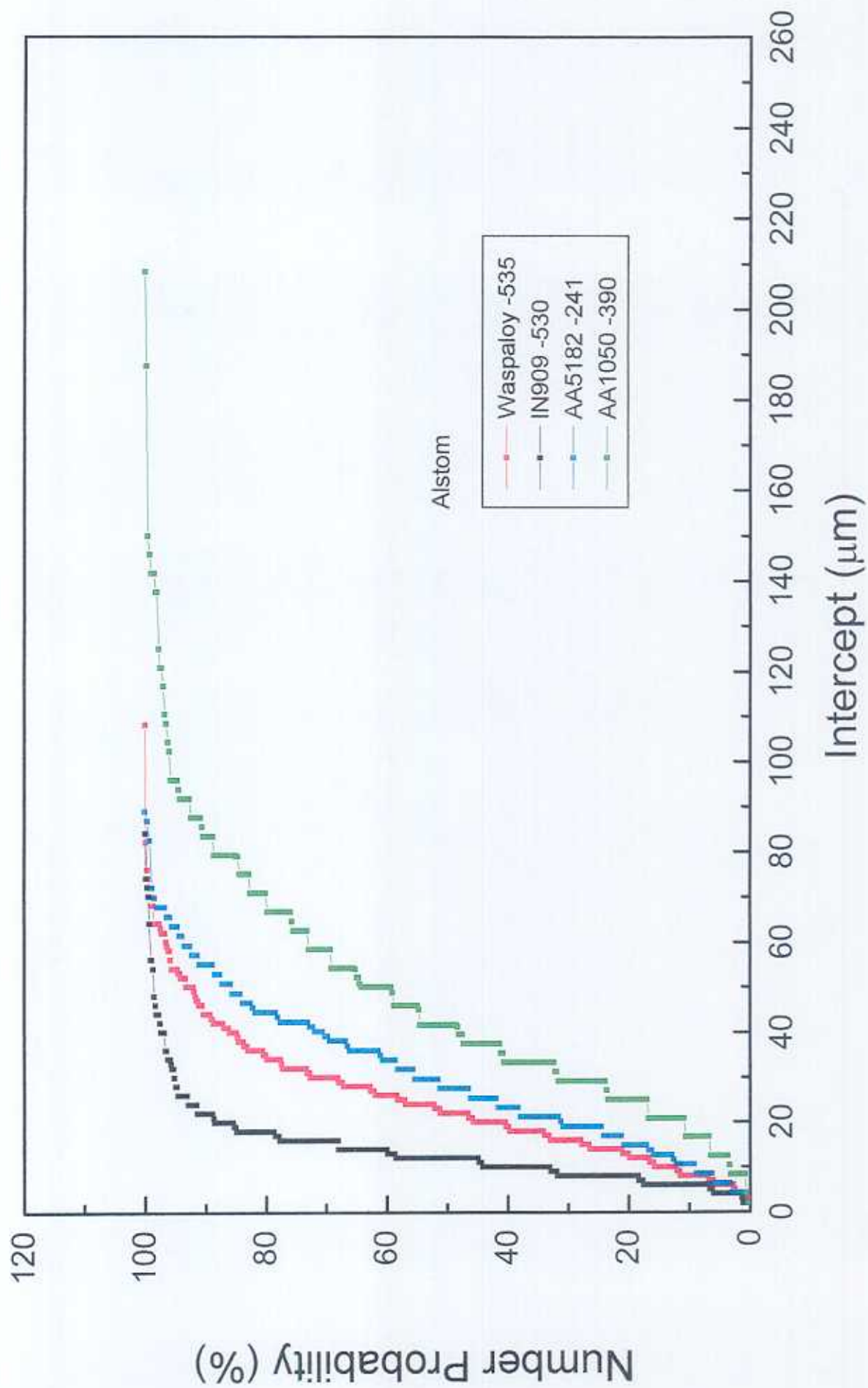
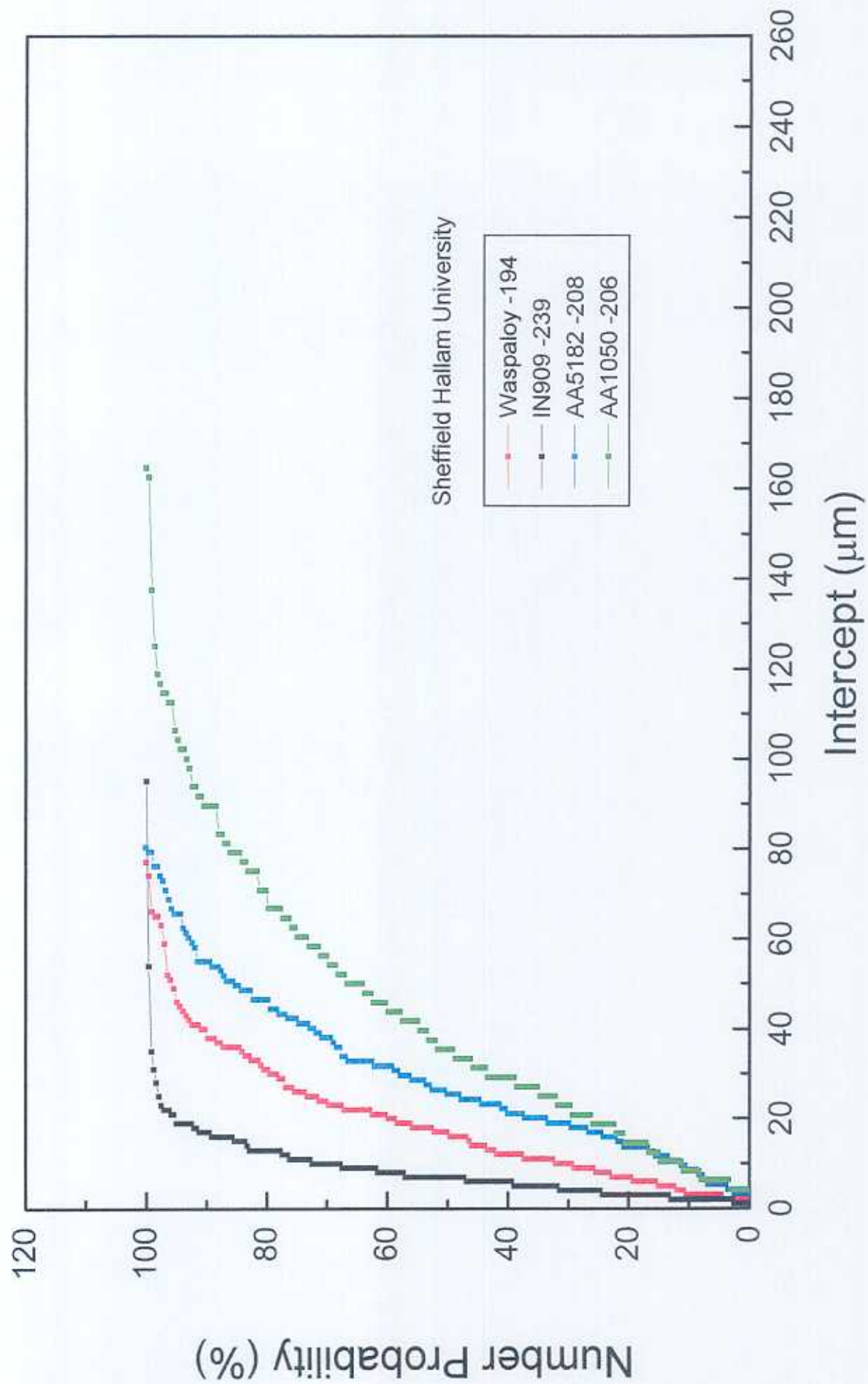


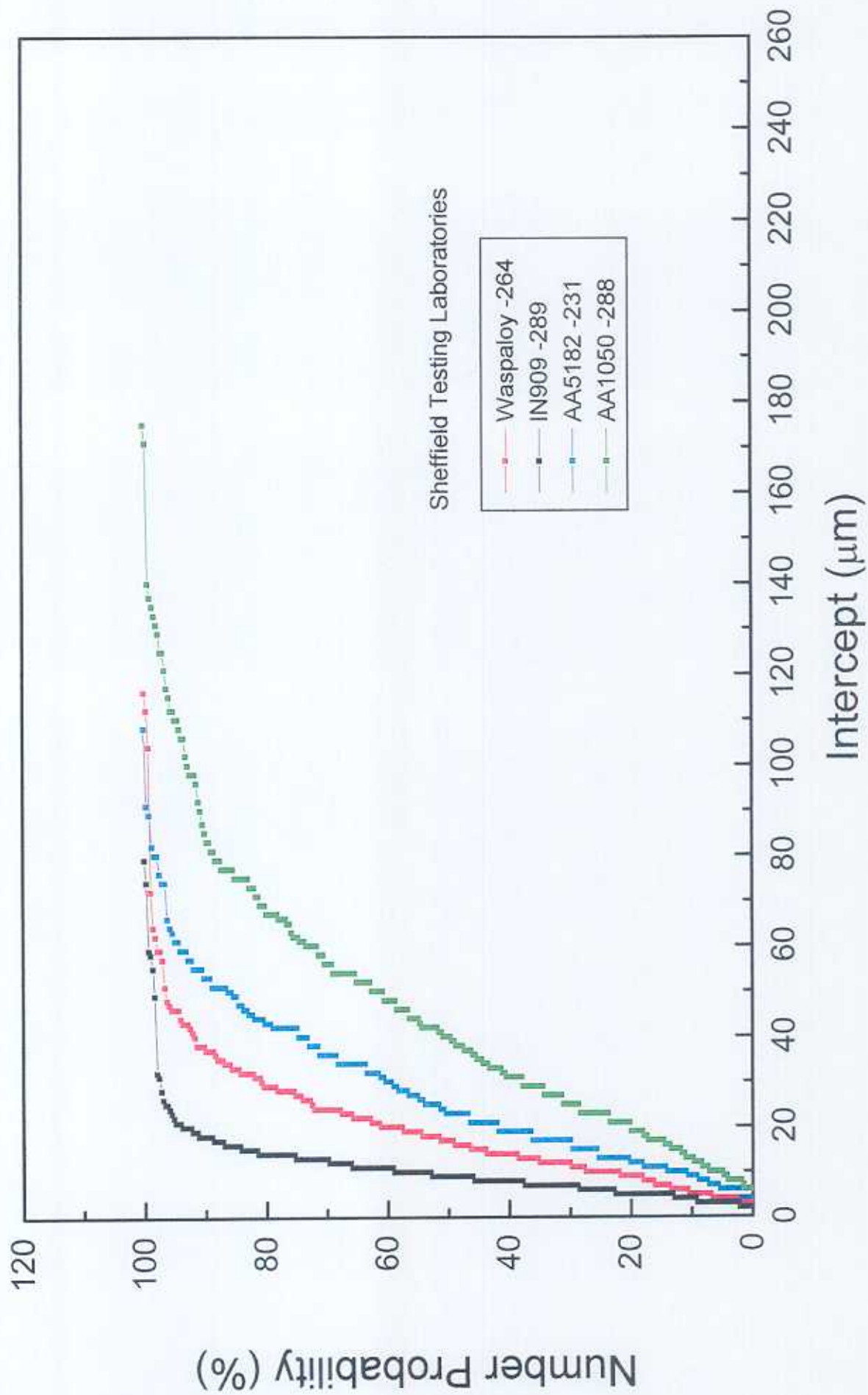
Fig 11 Number probability plotted against intercept.











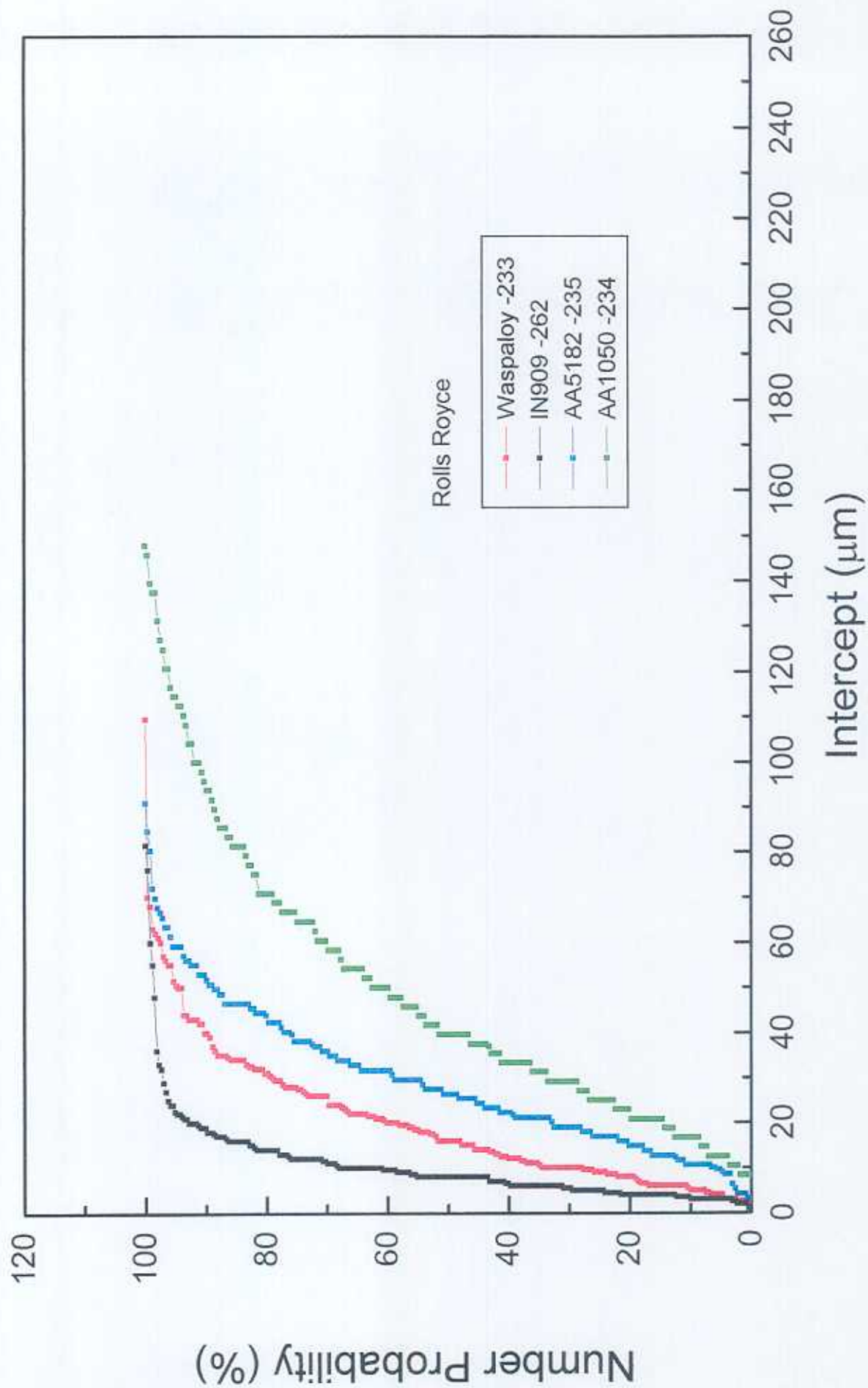
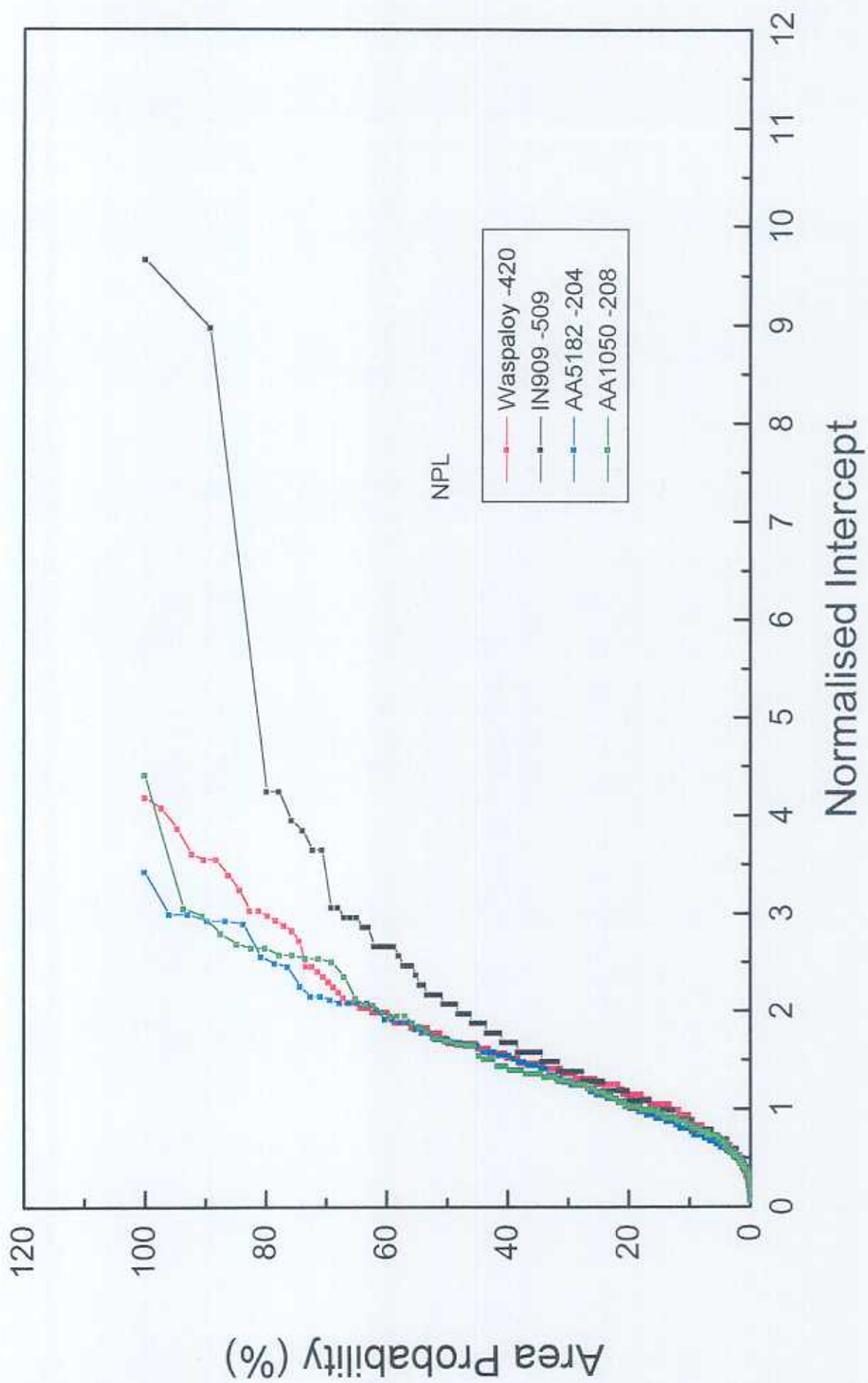
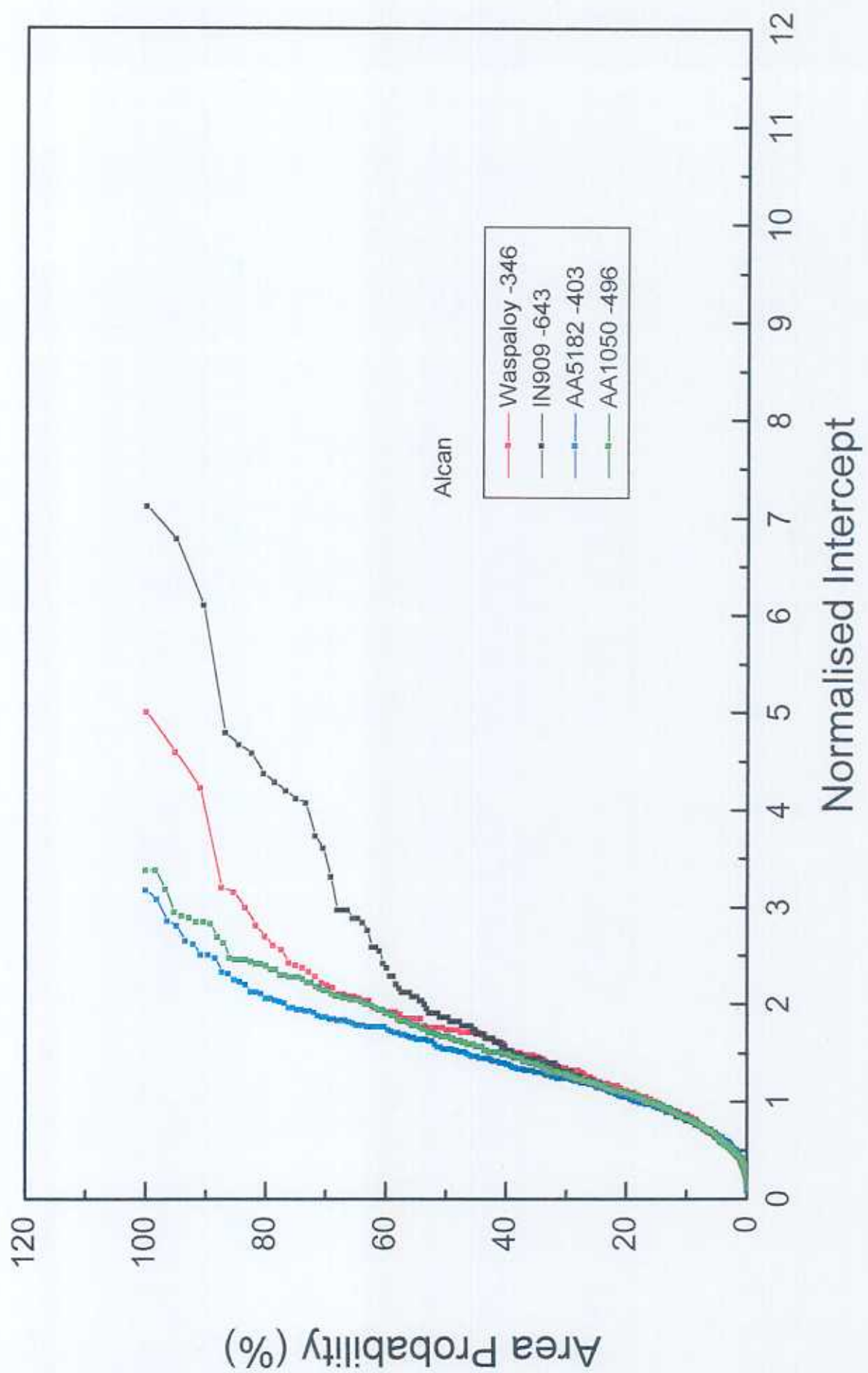
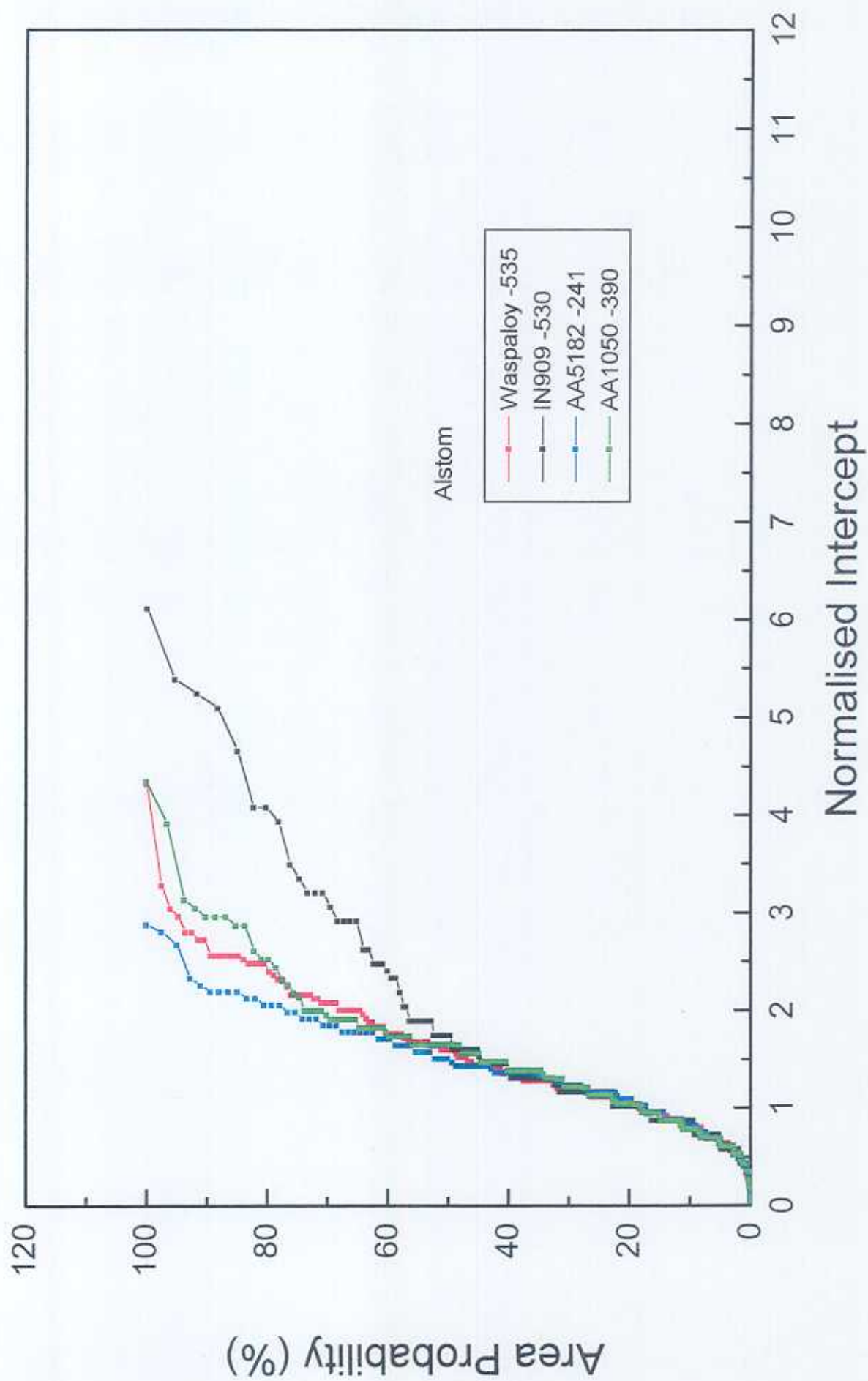
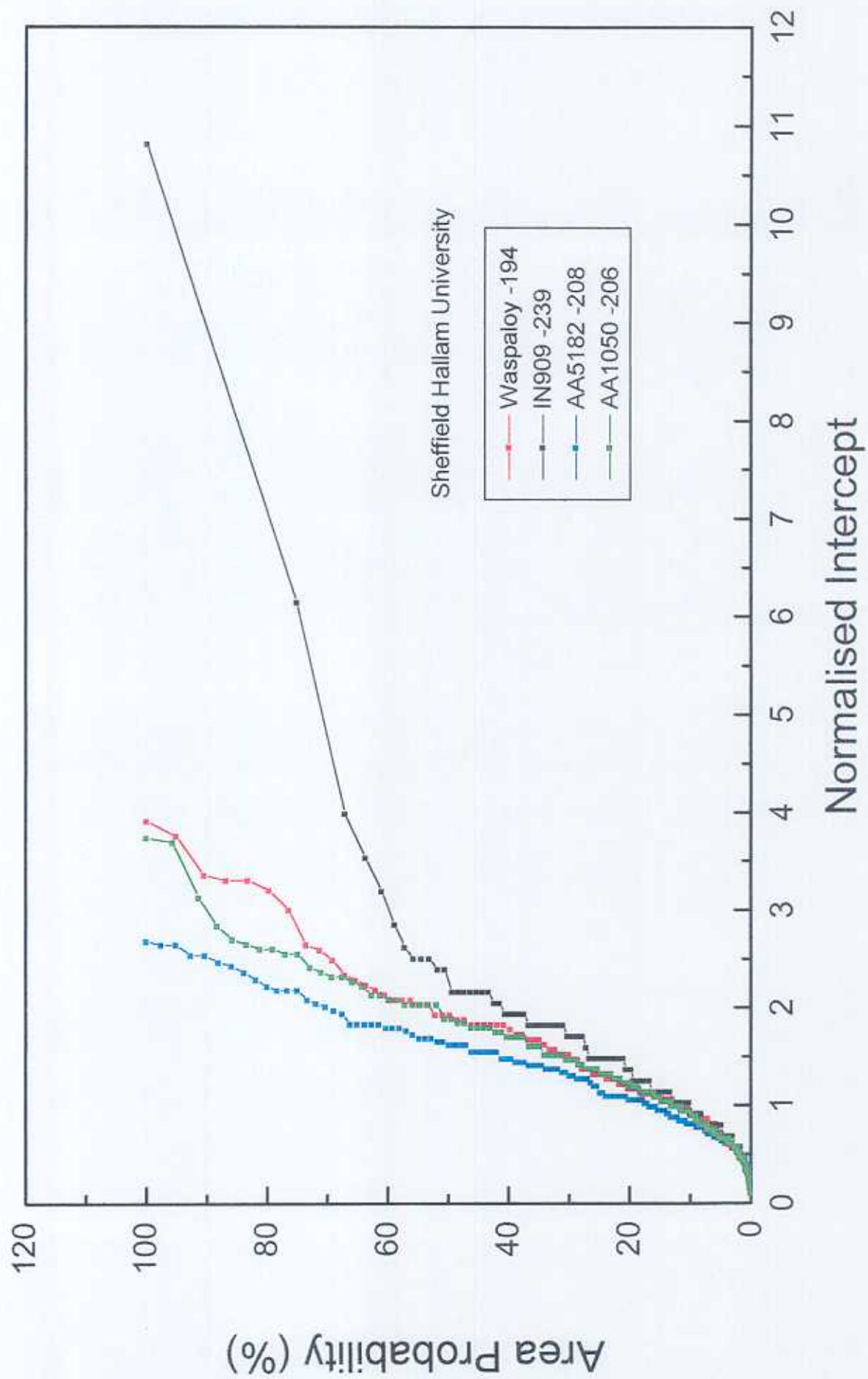


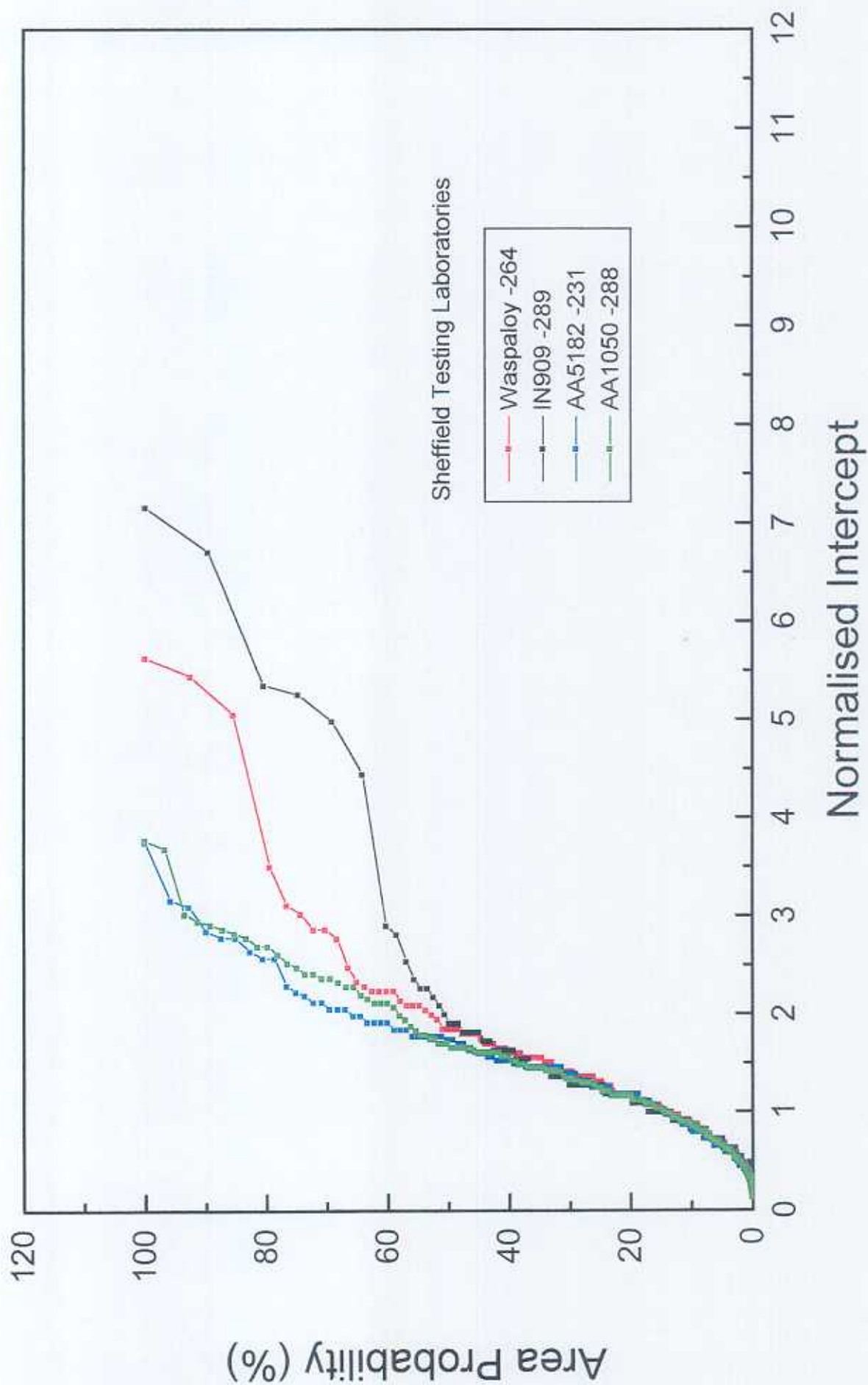
Fig 12 Area (E) probability plotted against normalised intercept.











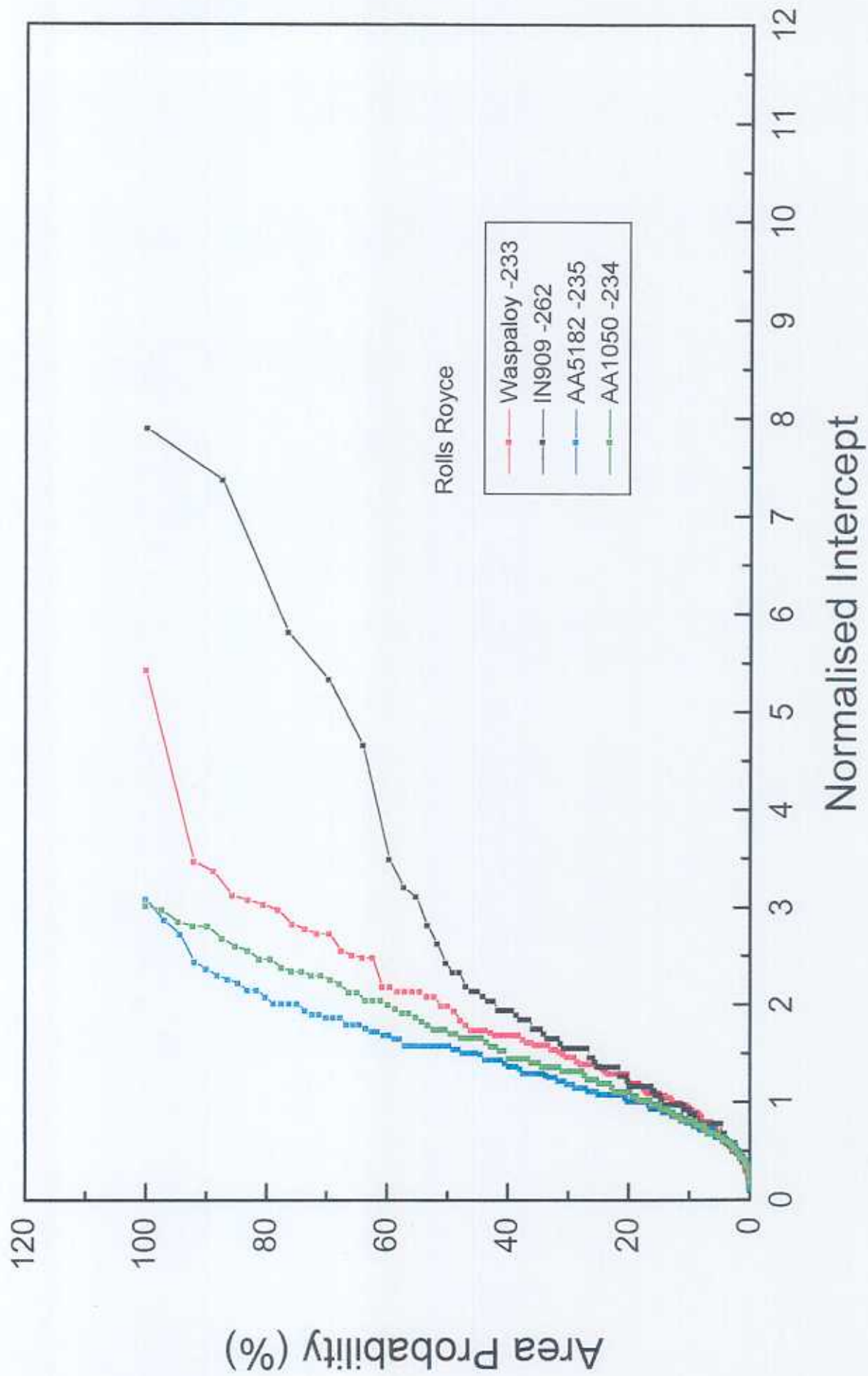
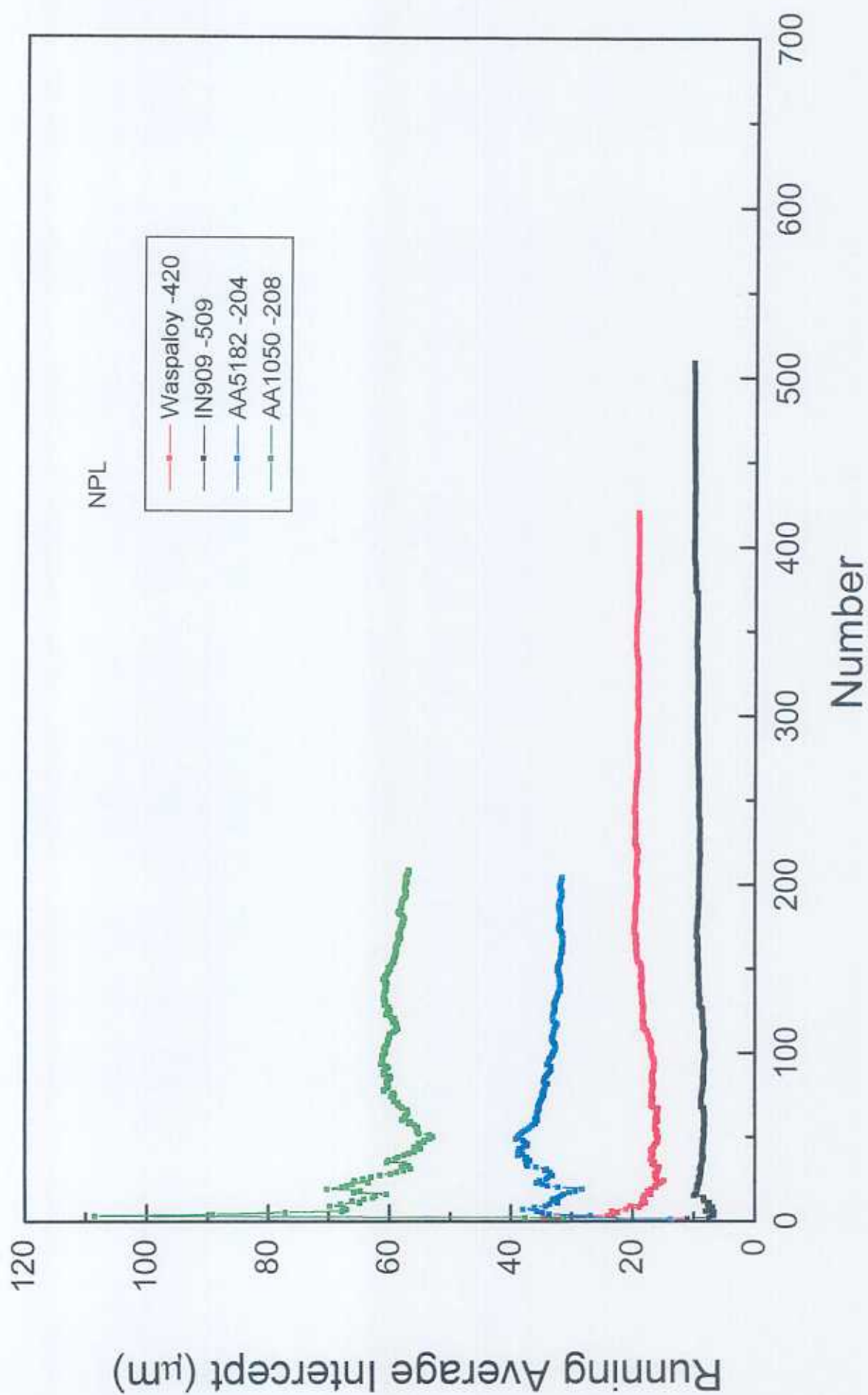
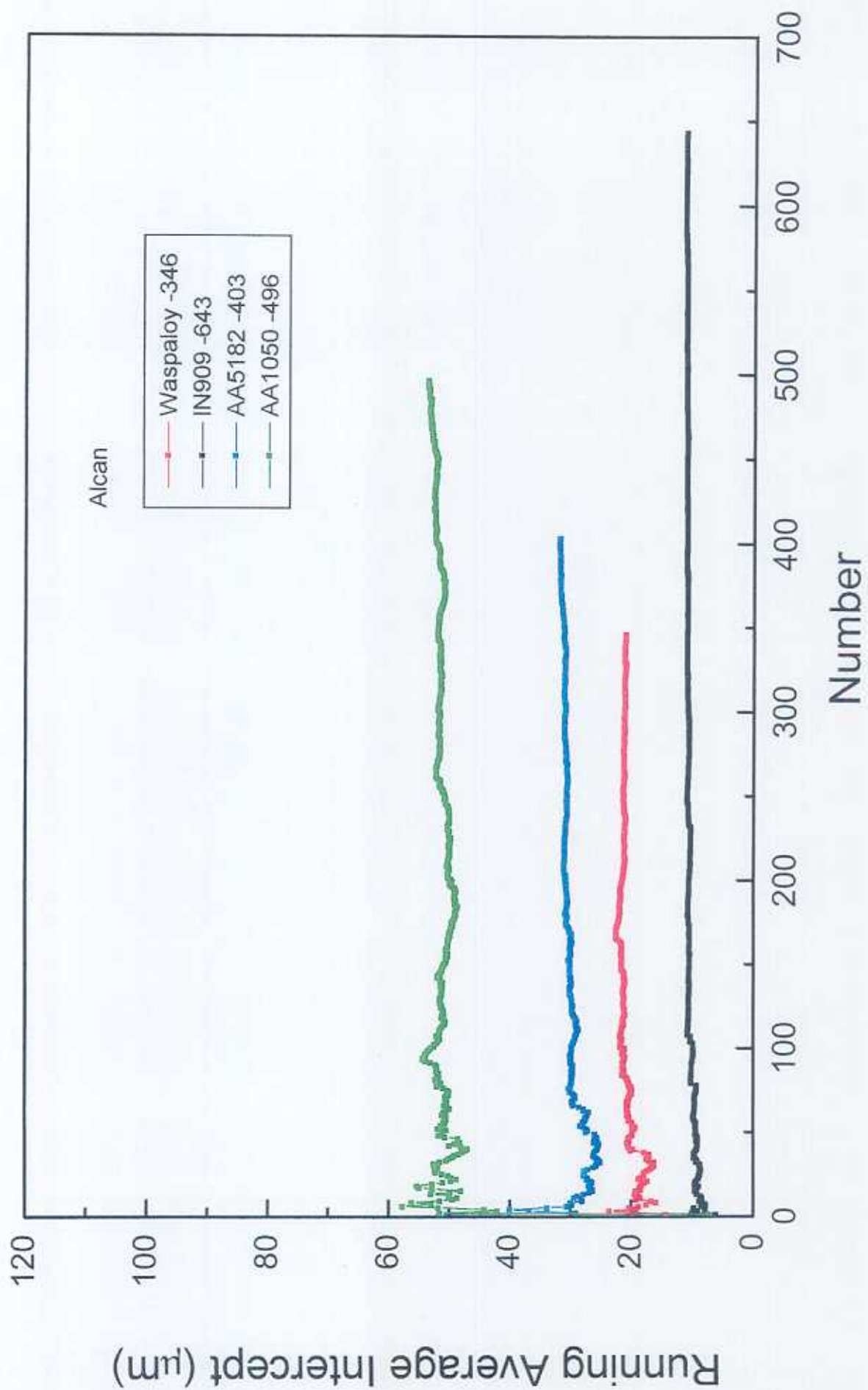
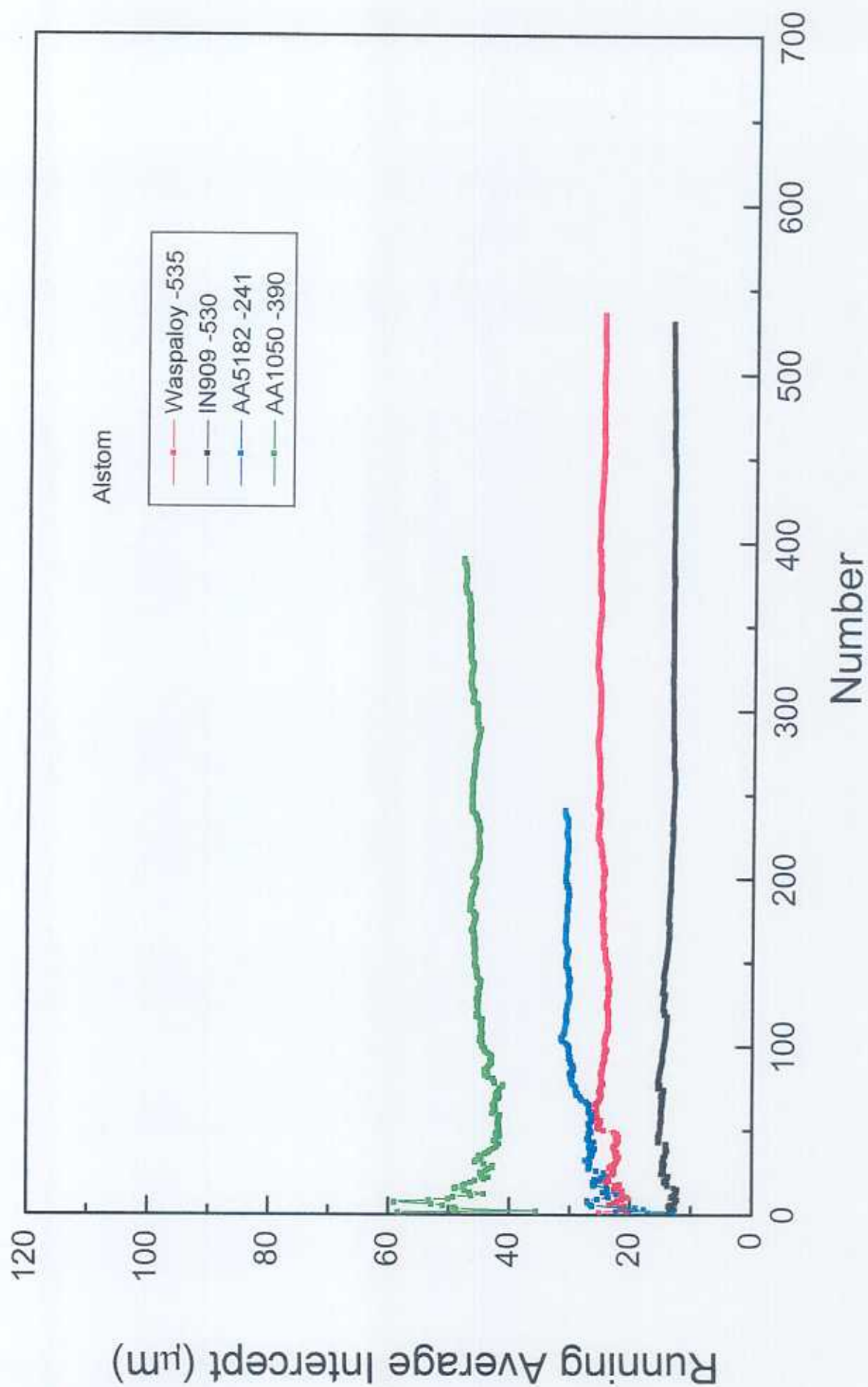
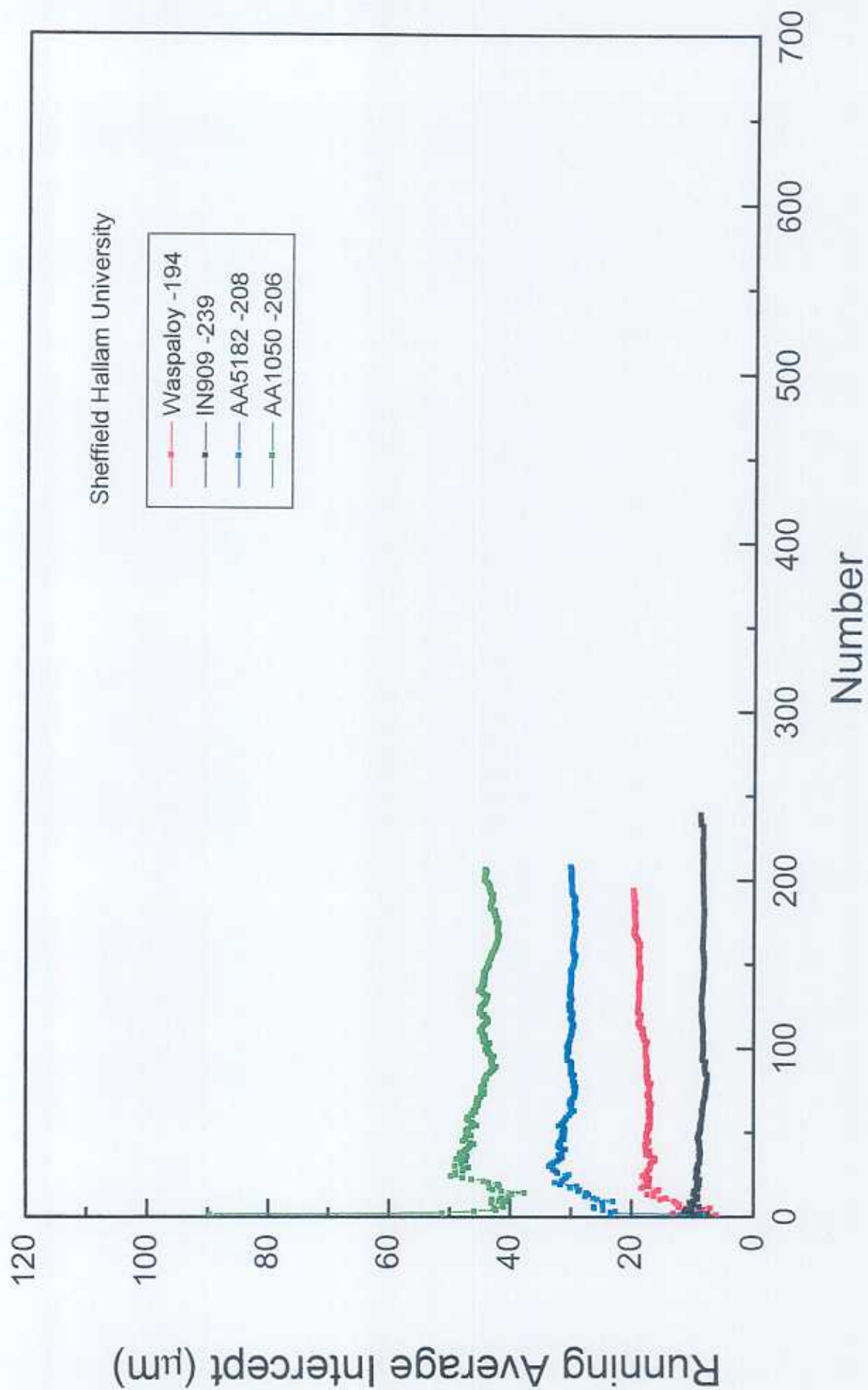


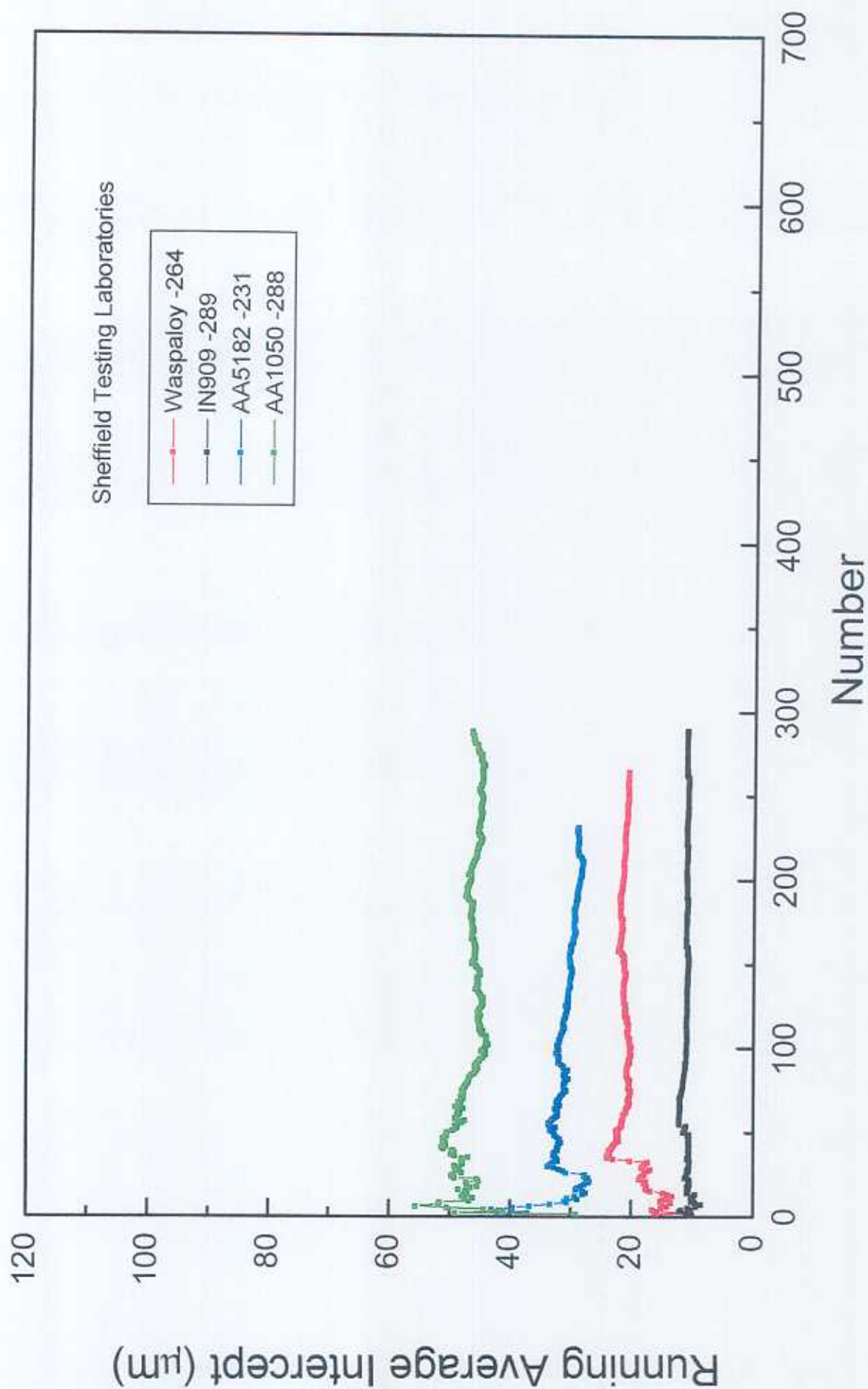
Fig 13 Running average intercept plotted against number (by organisation).











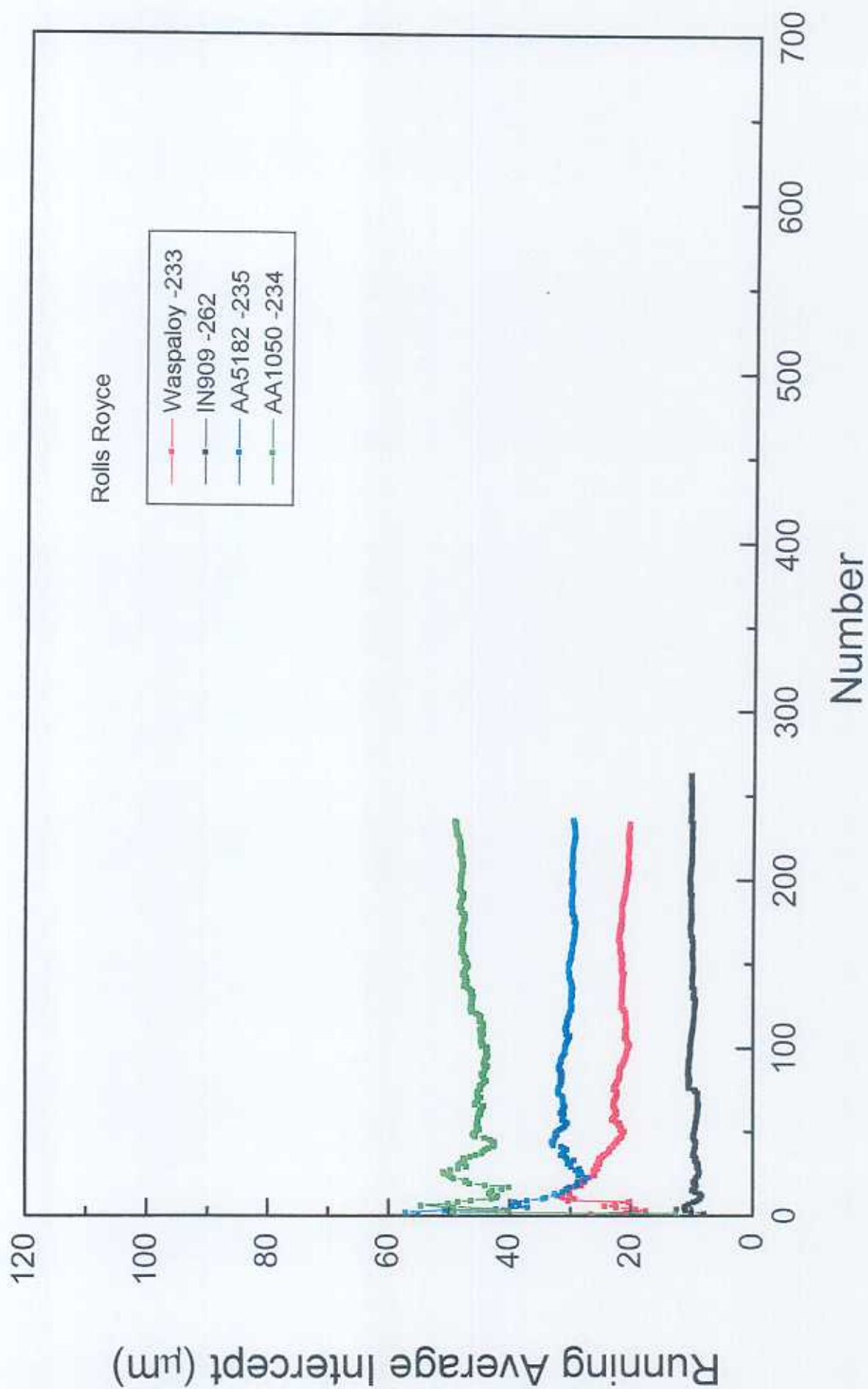
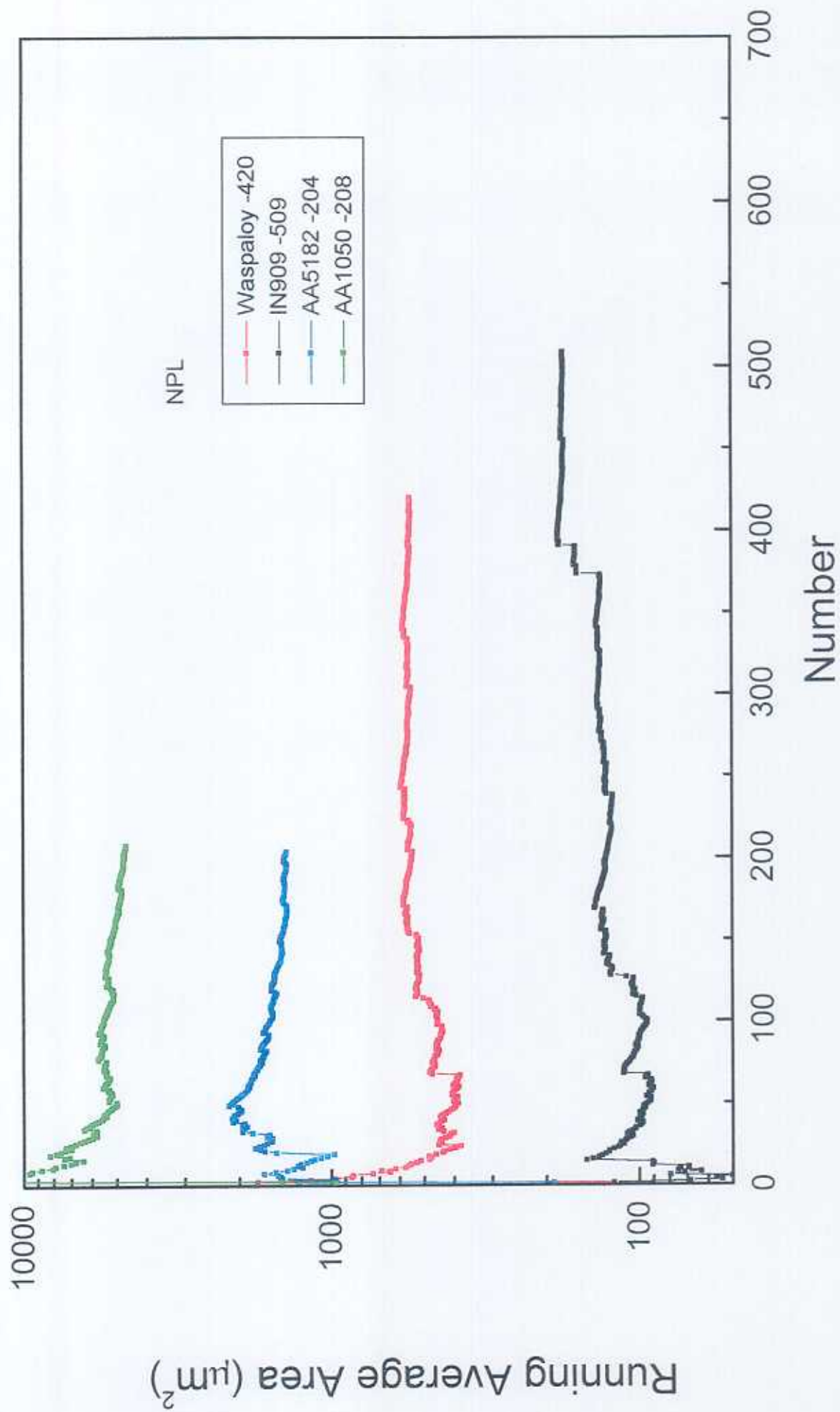
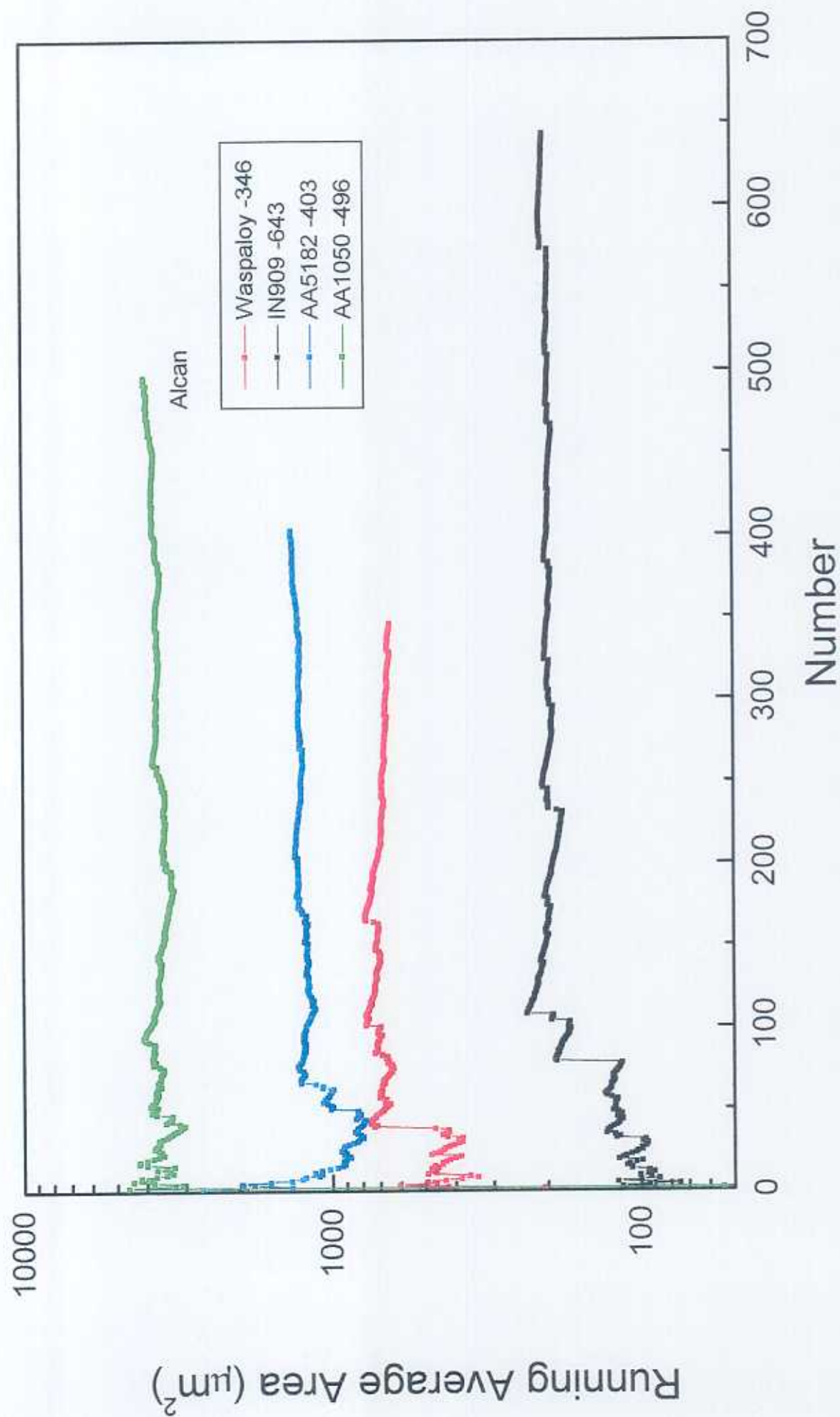
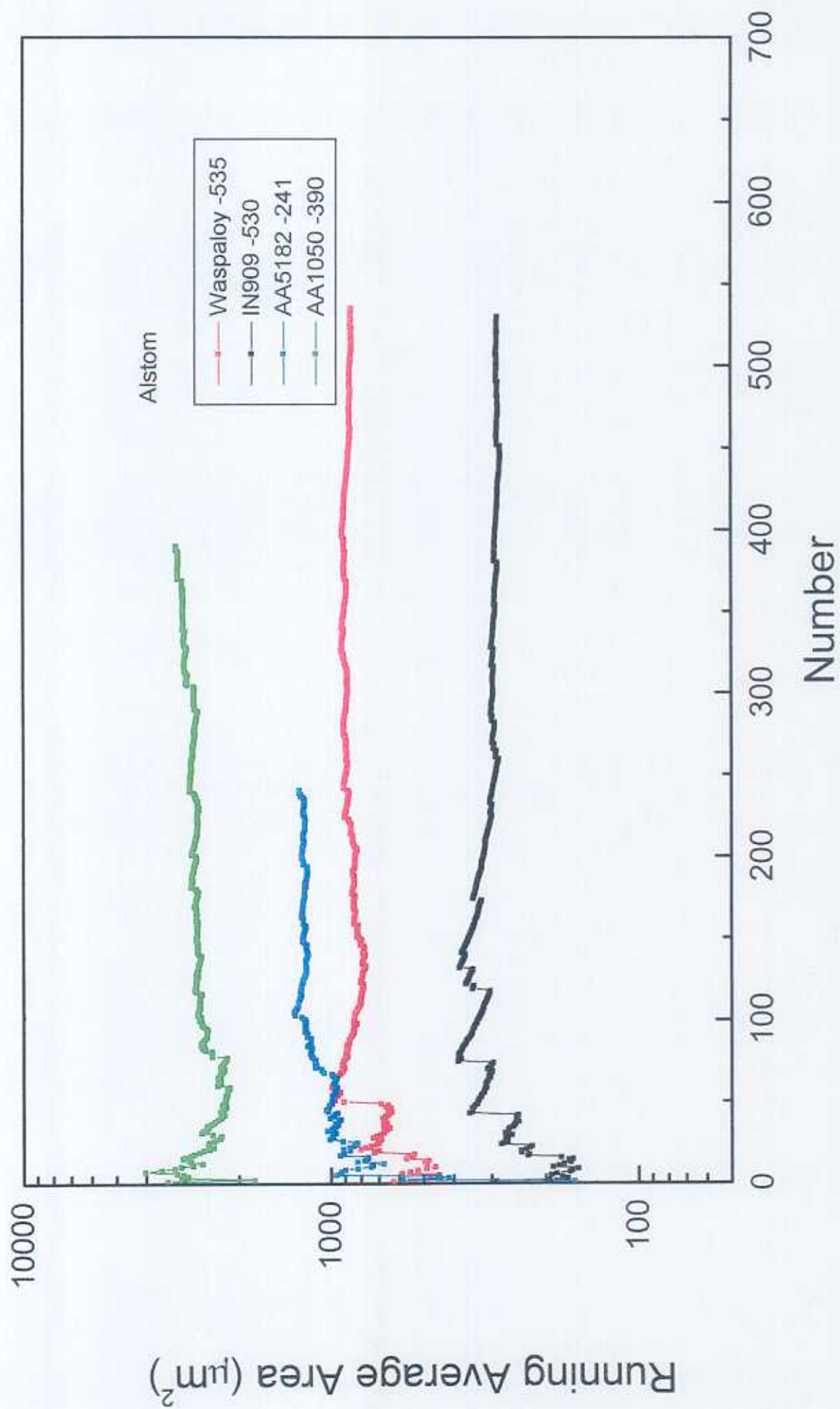
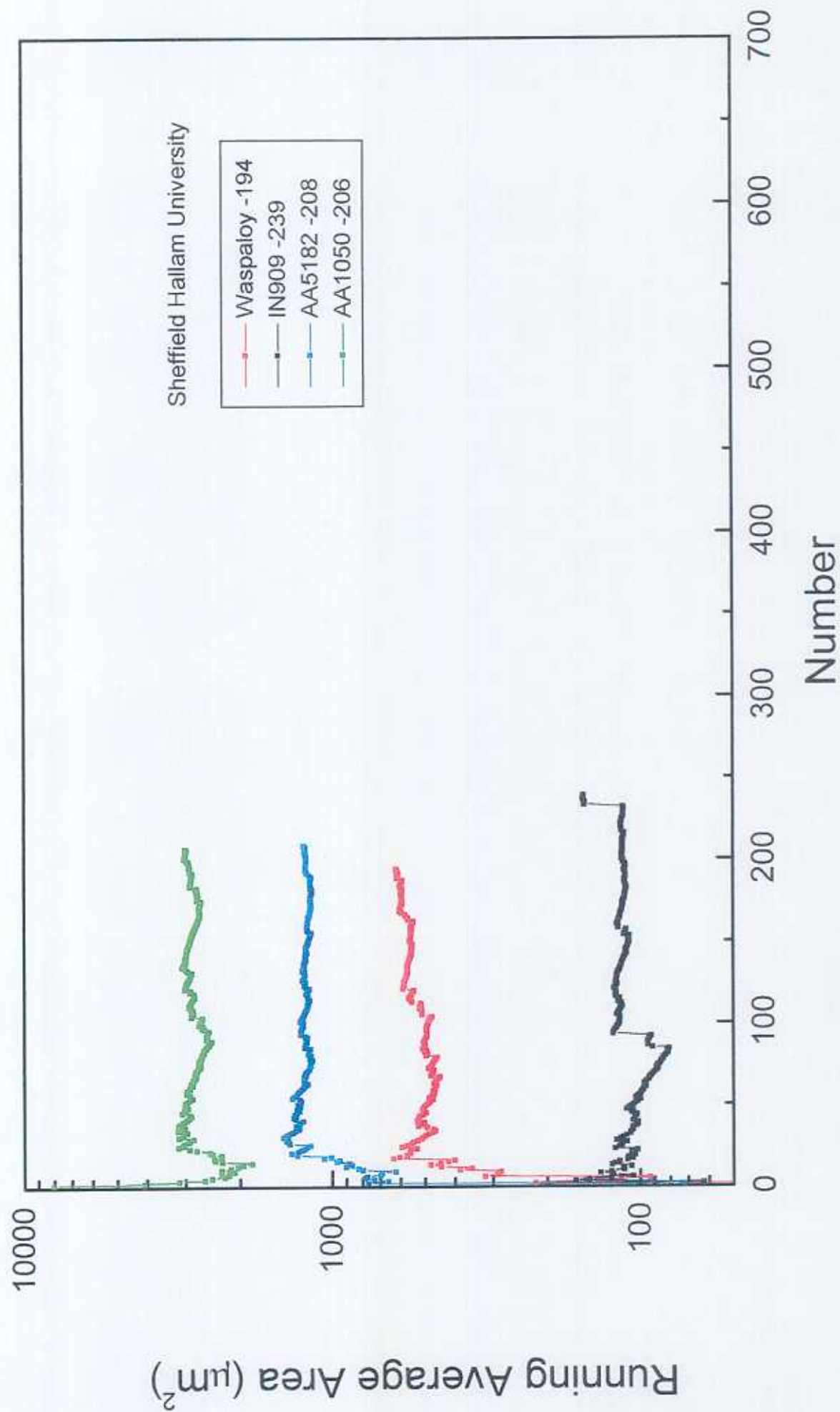


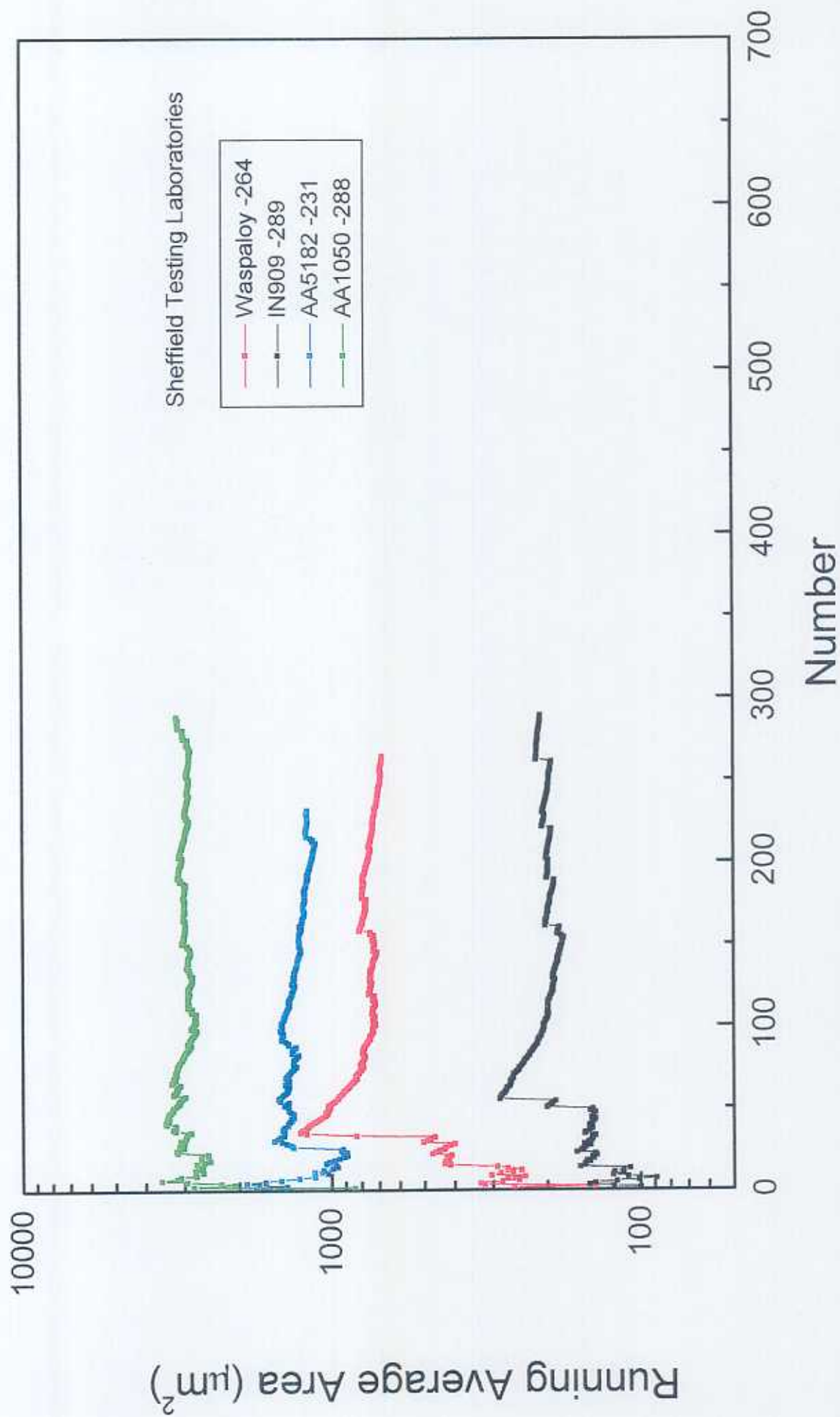
Fig 14 **Running average area (E) plotted against number (by organisation).**











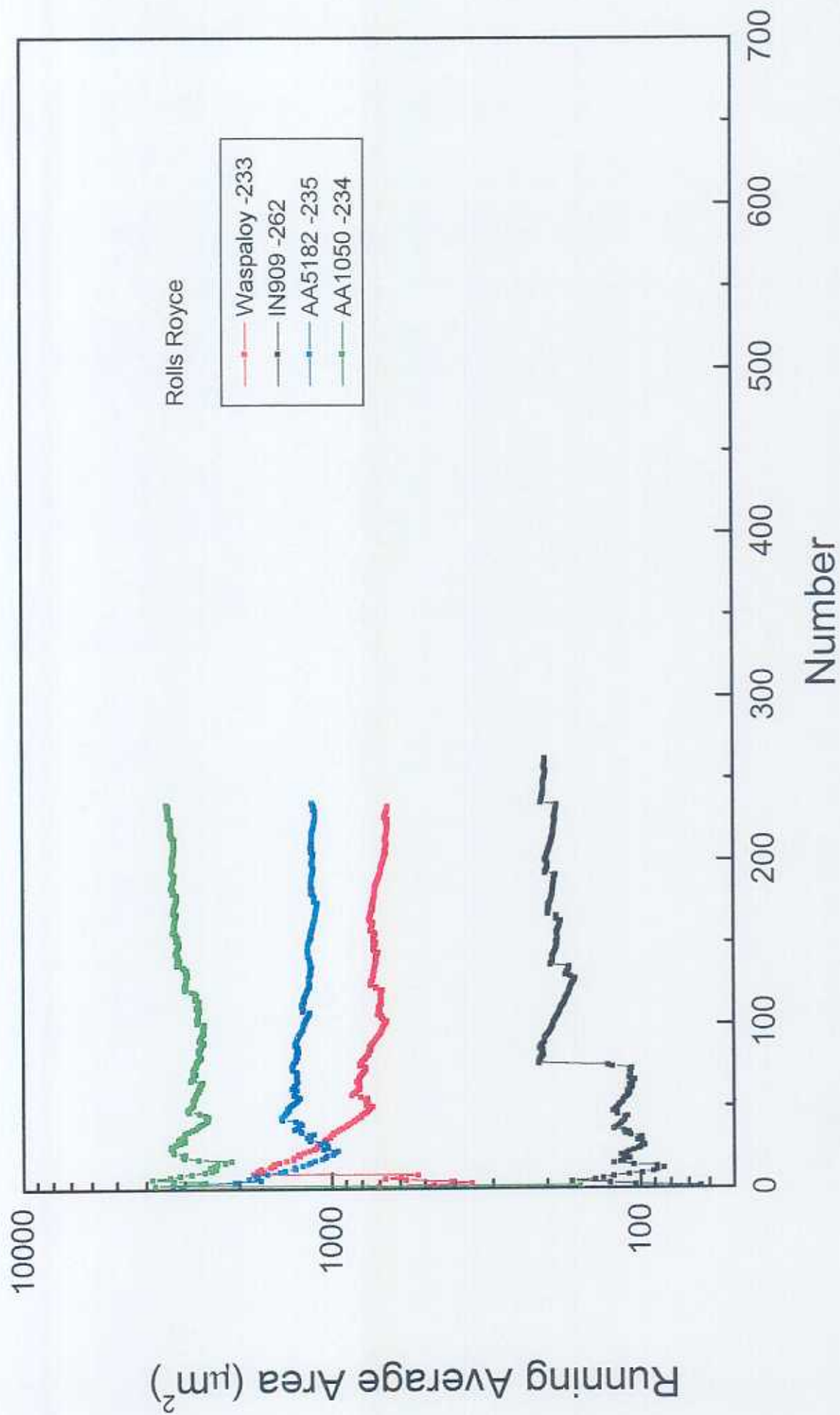
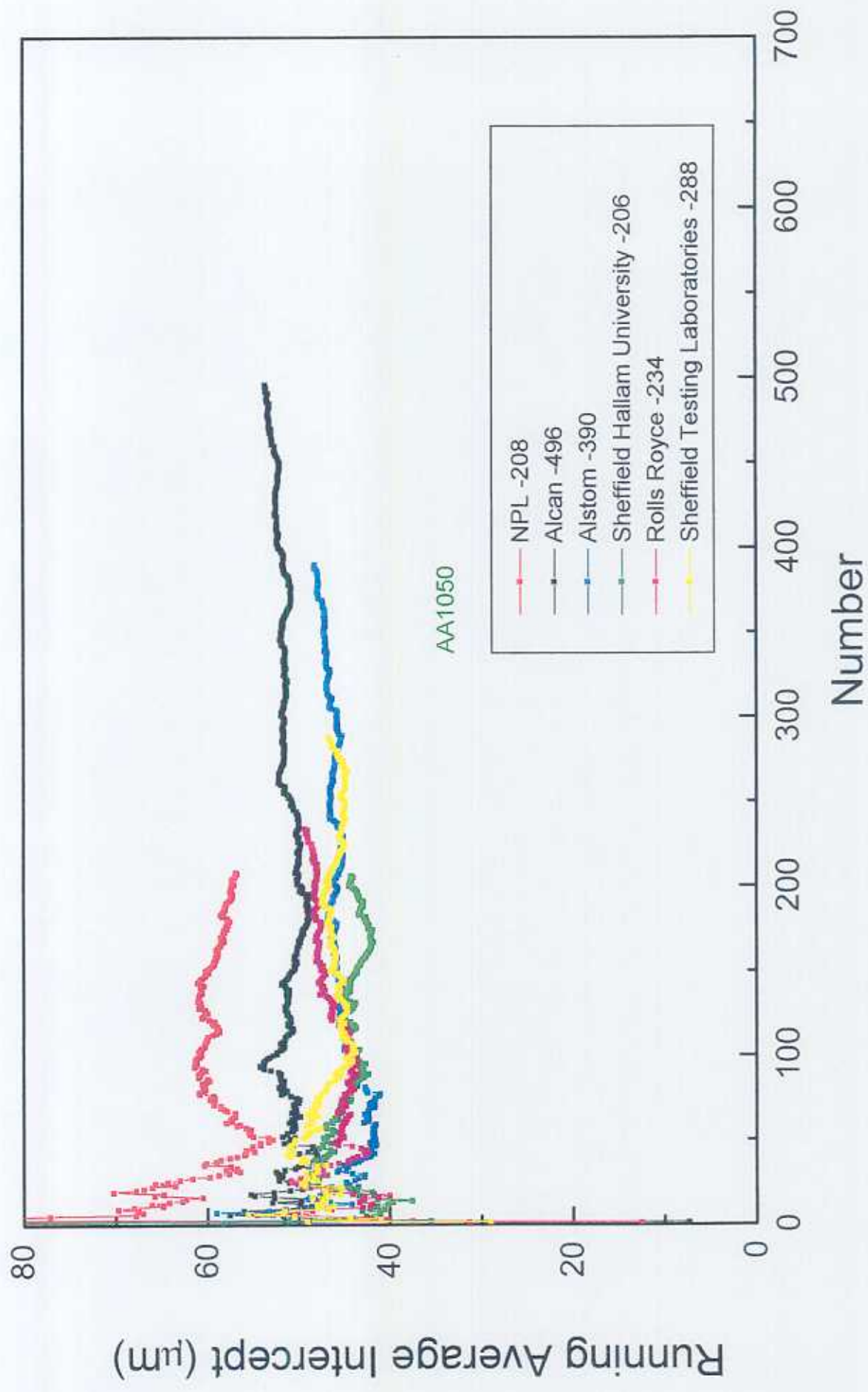
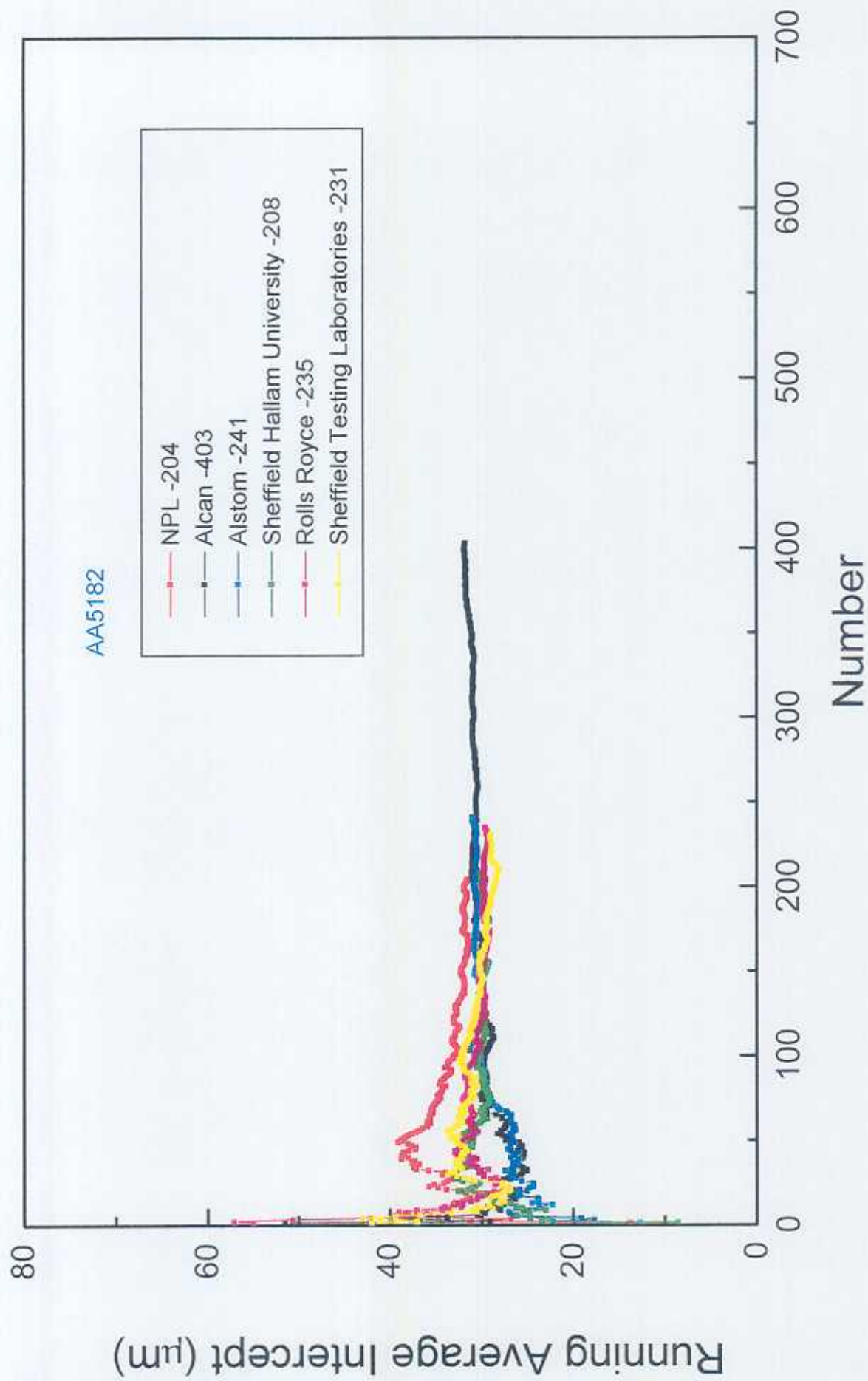
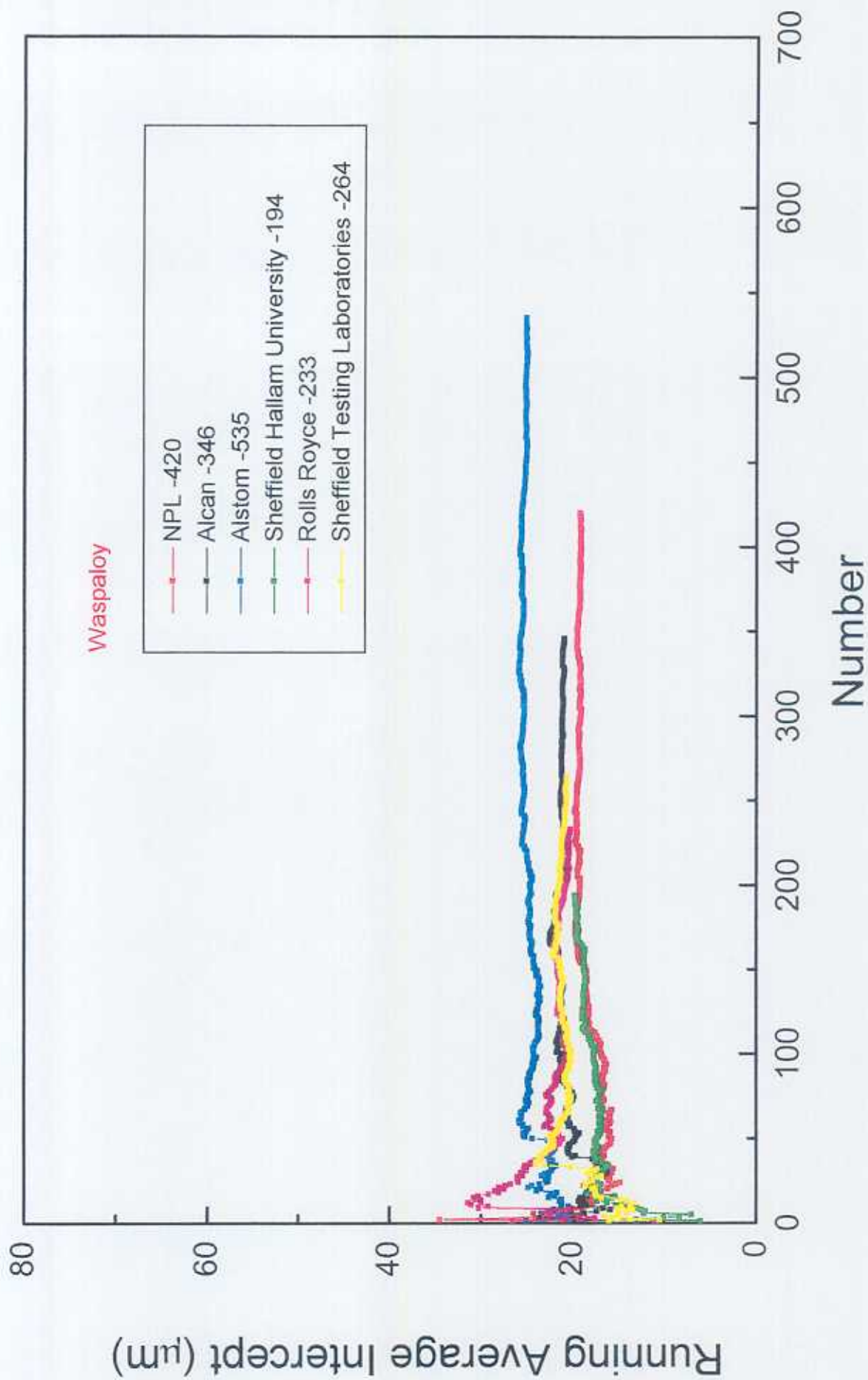


Fig 15 Running average intercept plotted against number (by material).







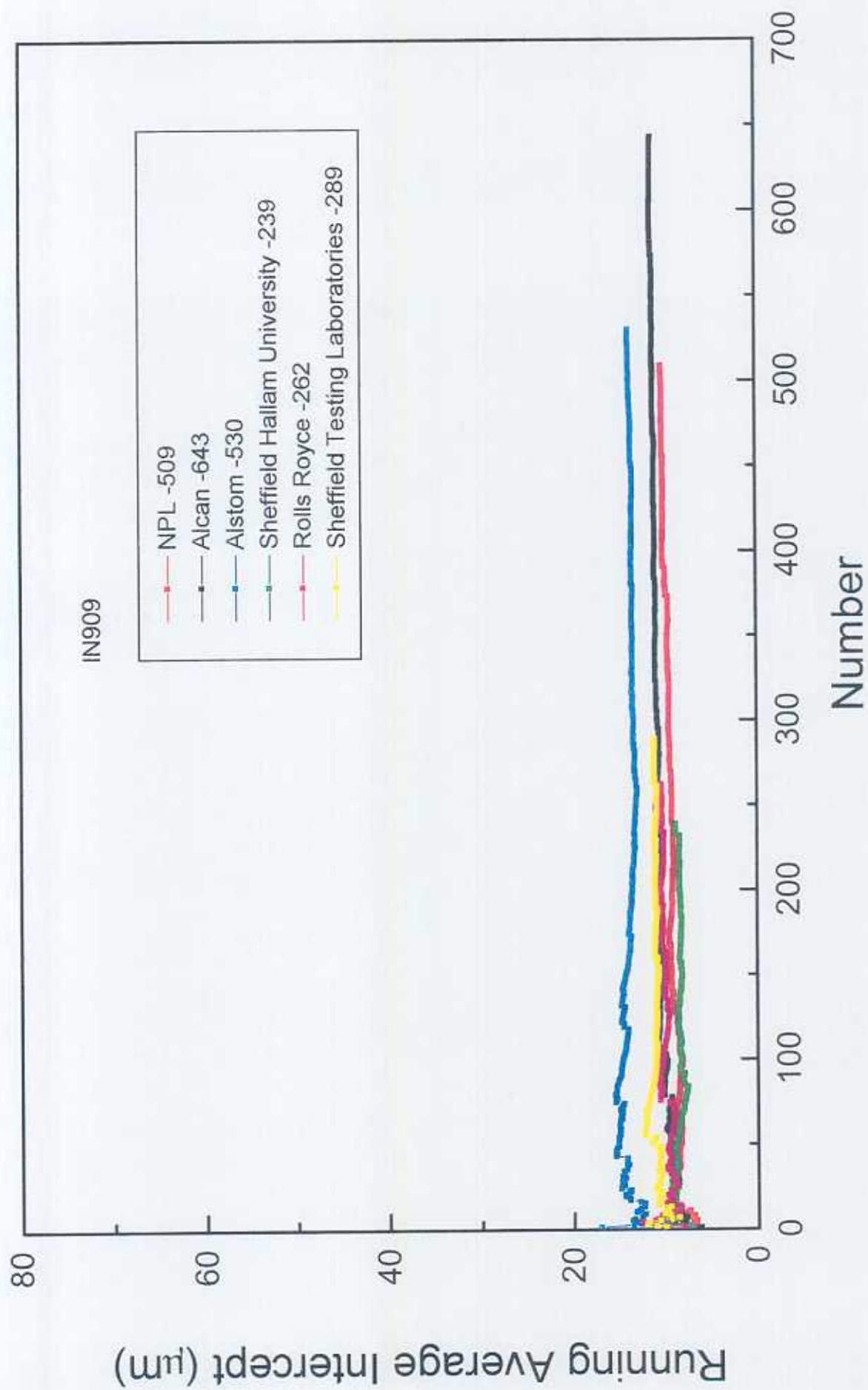
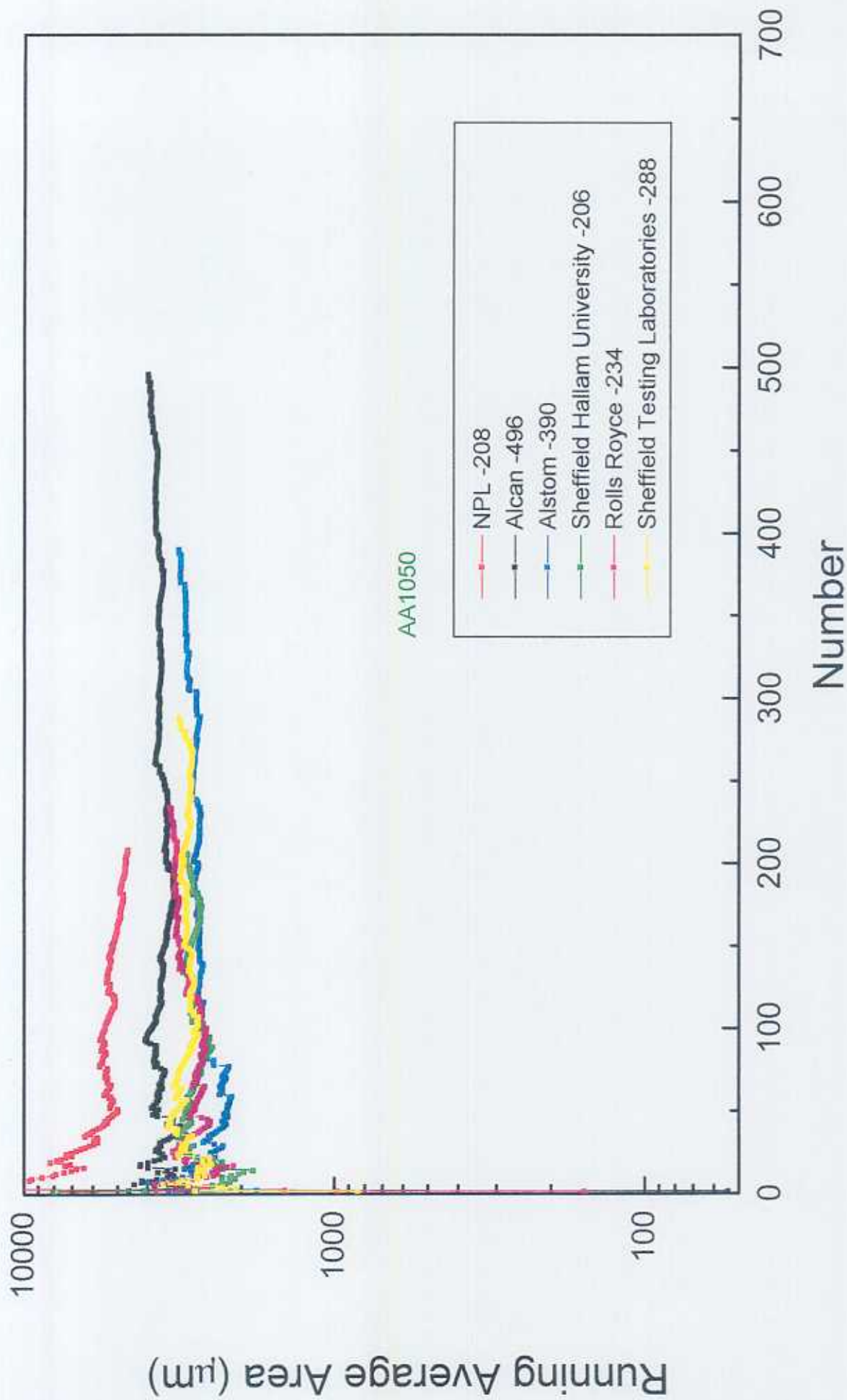
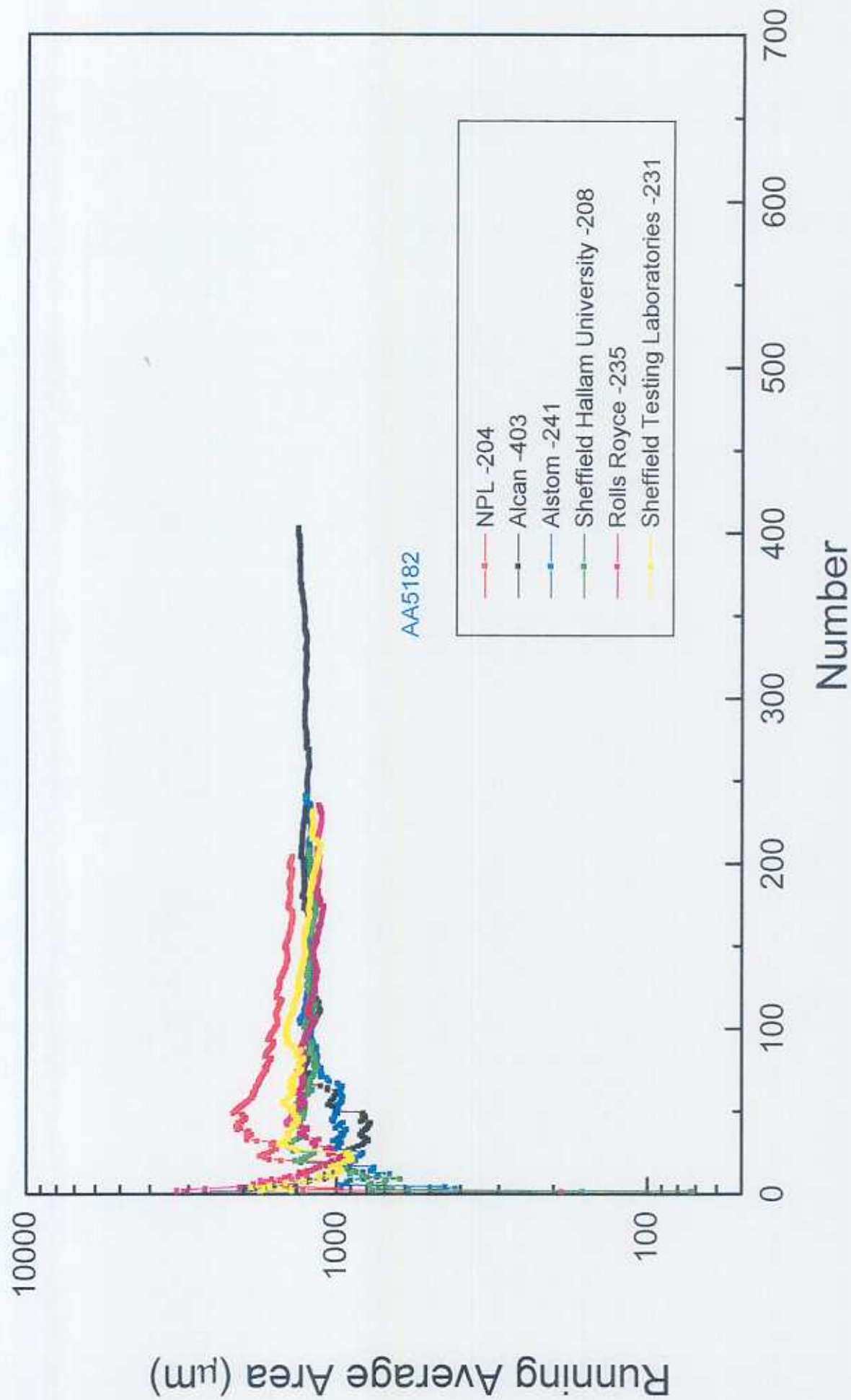
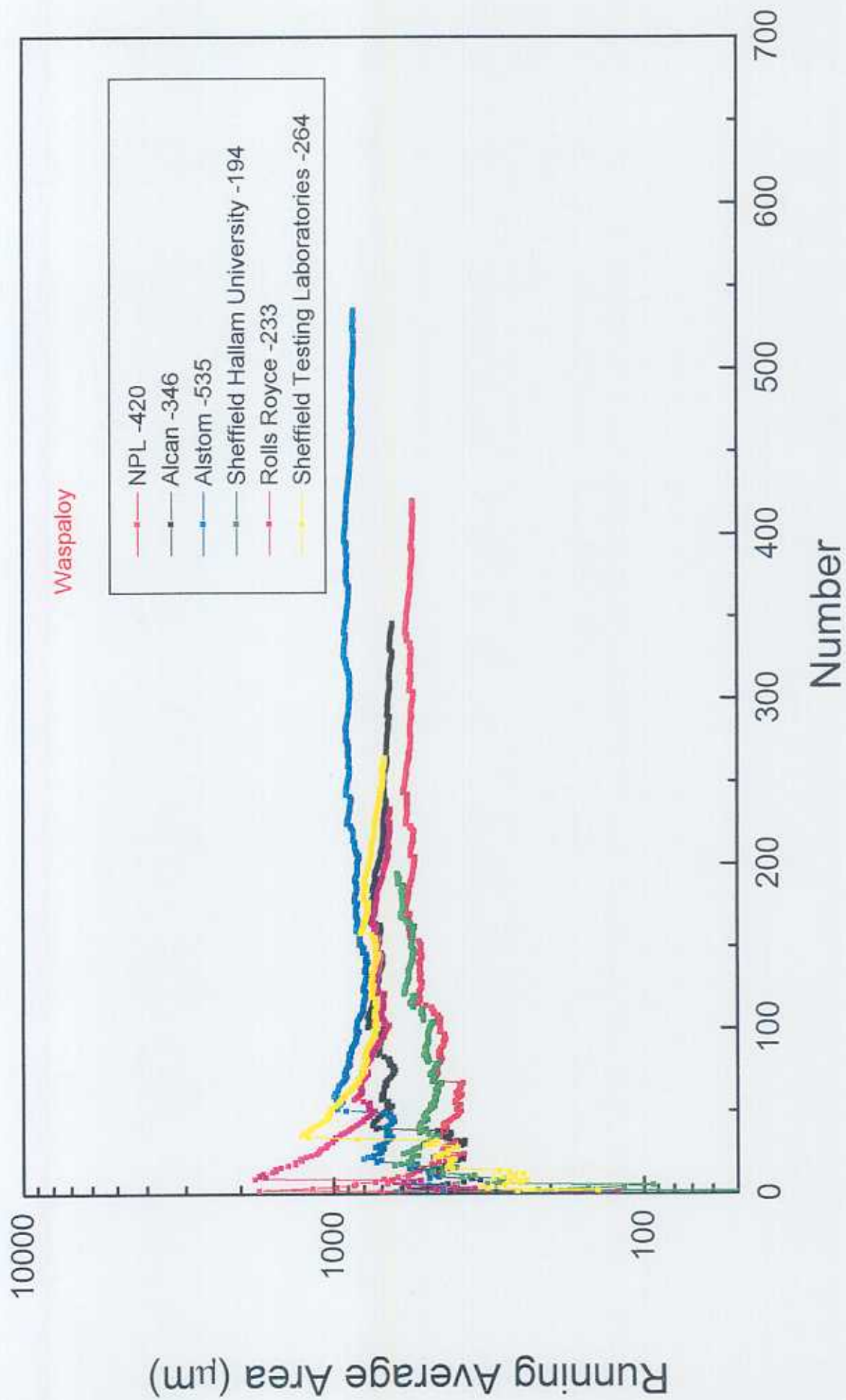
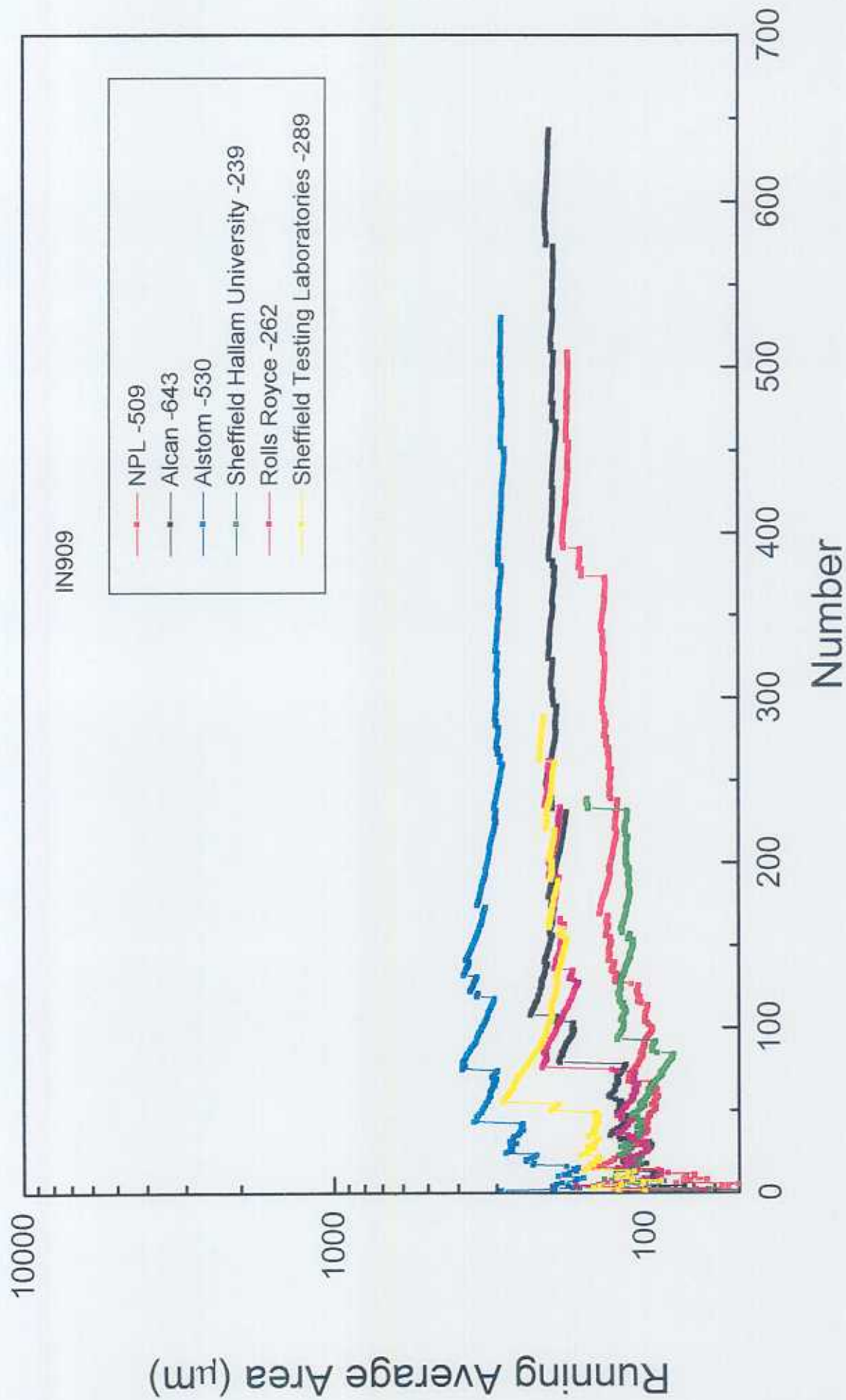


Fig 16 Running average area (E) plotted against number (by material).









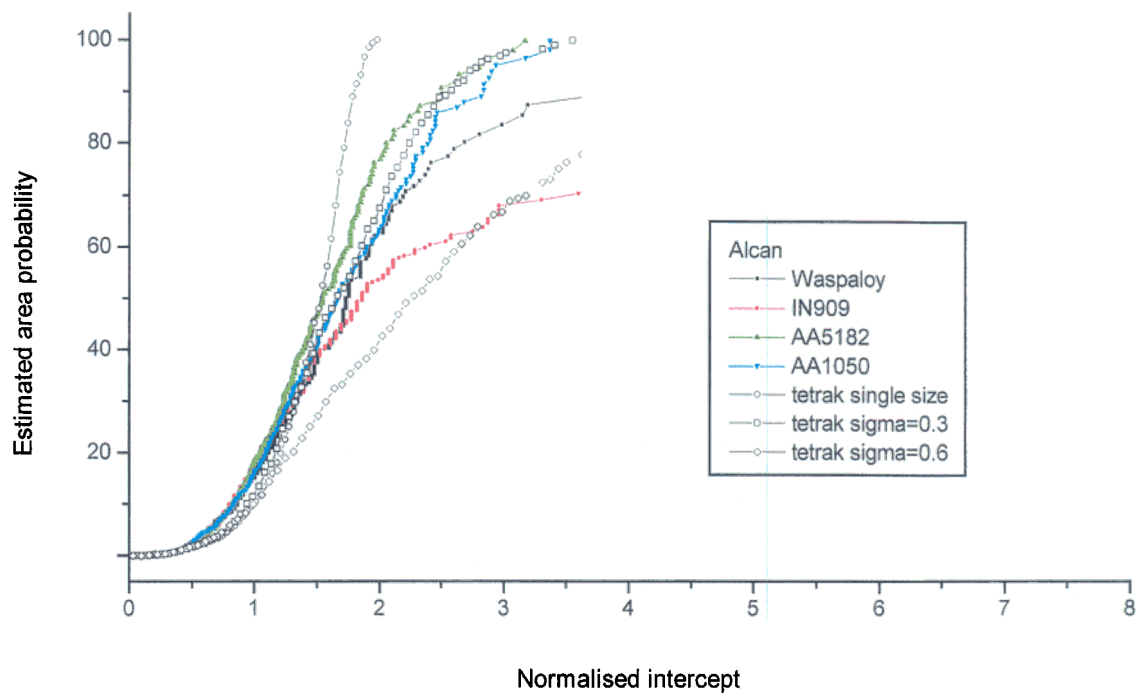


Fig 17 Comparison of distributions from the Alcan data sets and the tetrakaidecahedron model.

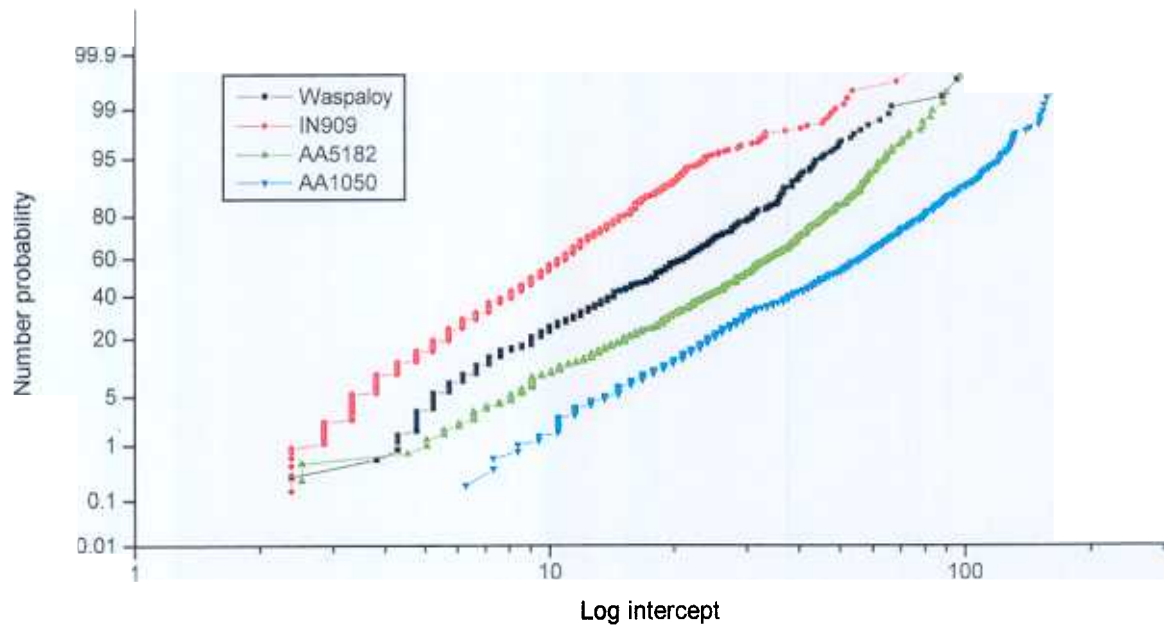


Fig 18 Number probability plotted against log intercepts for the Alcan data set.

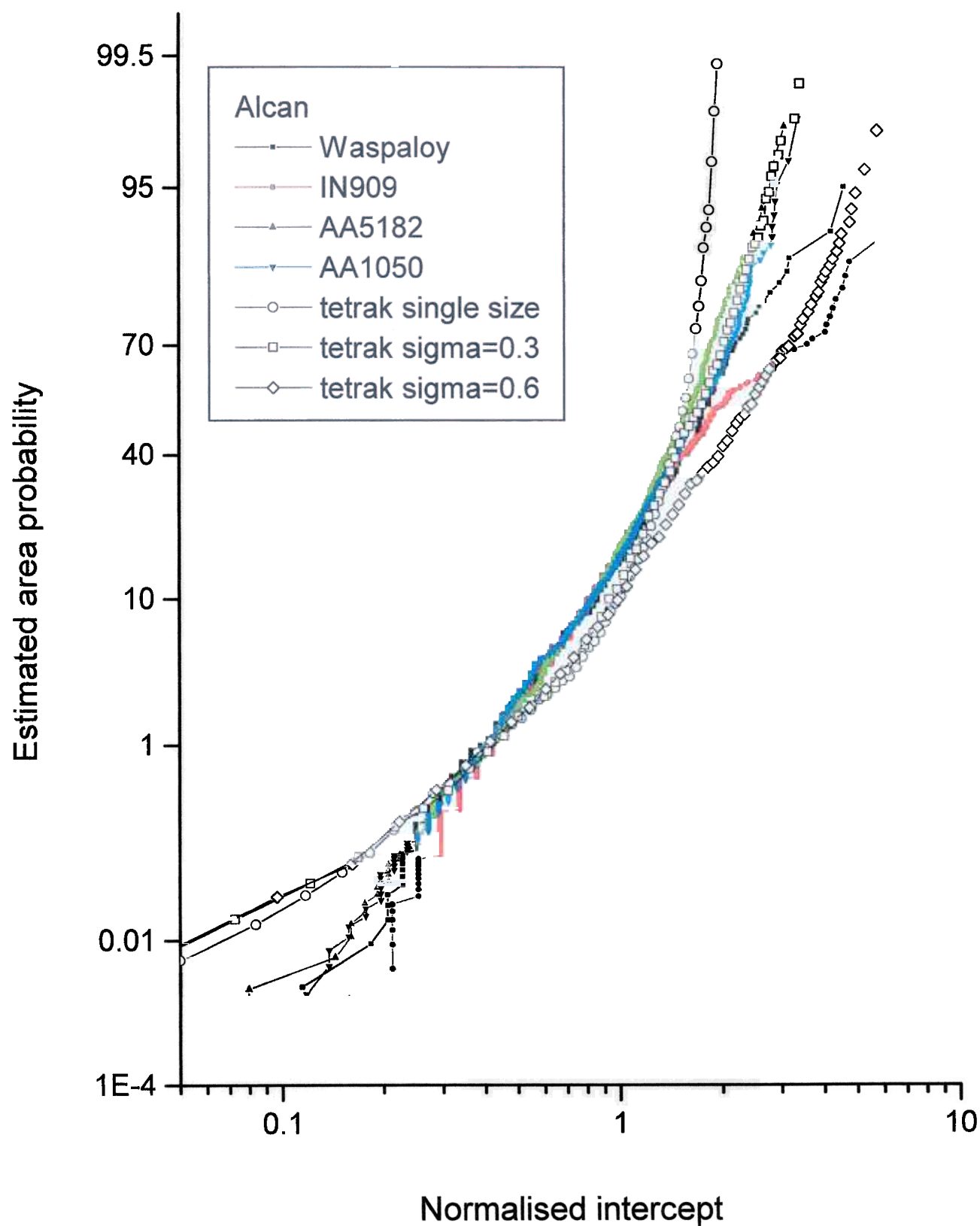
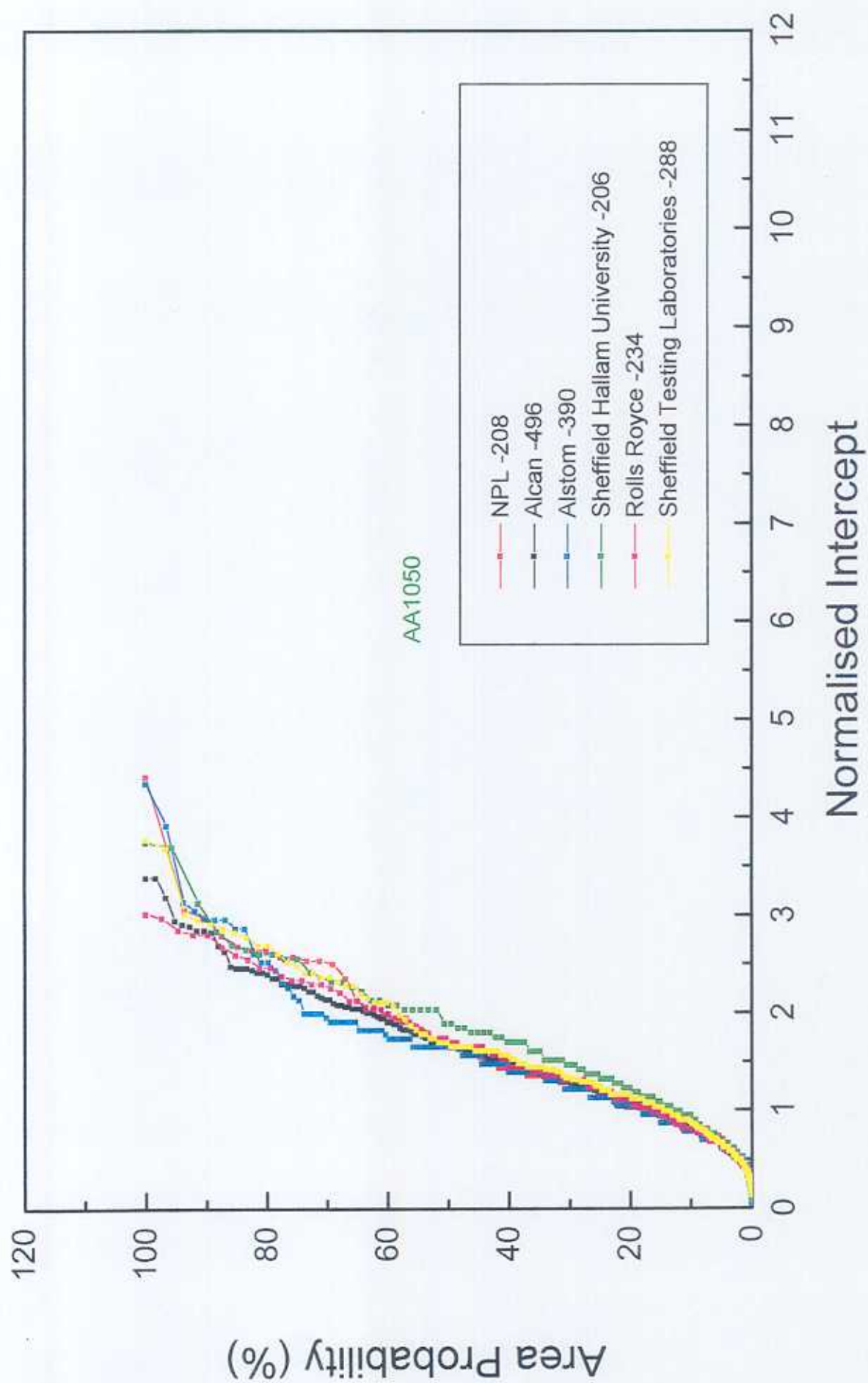
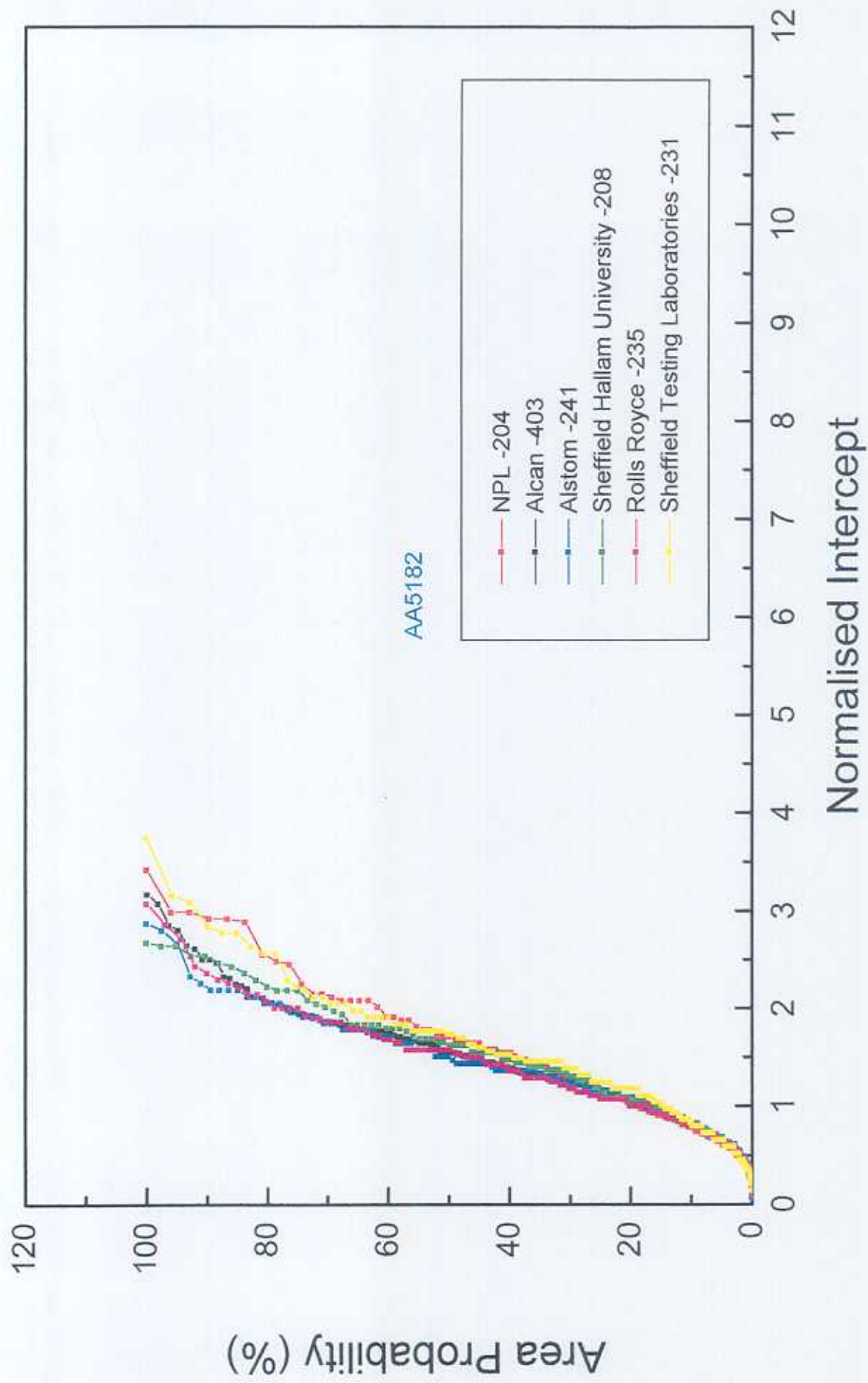
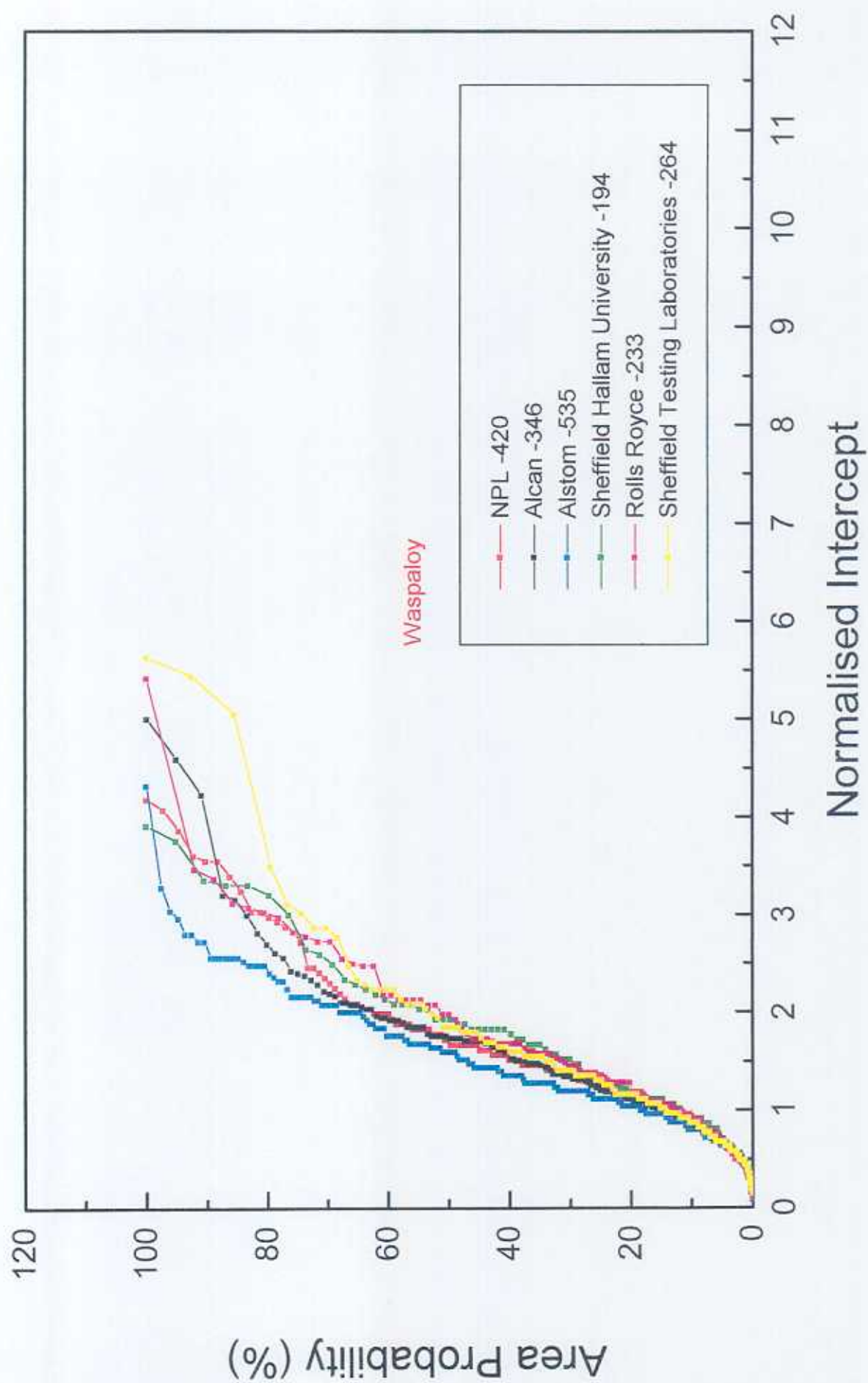


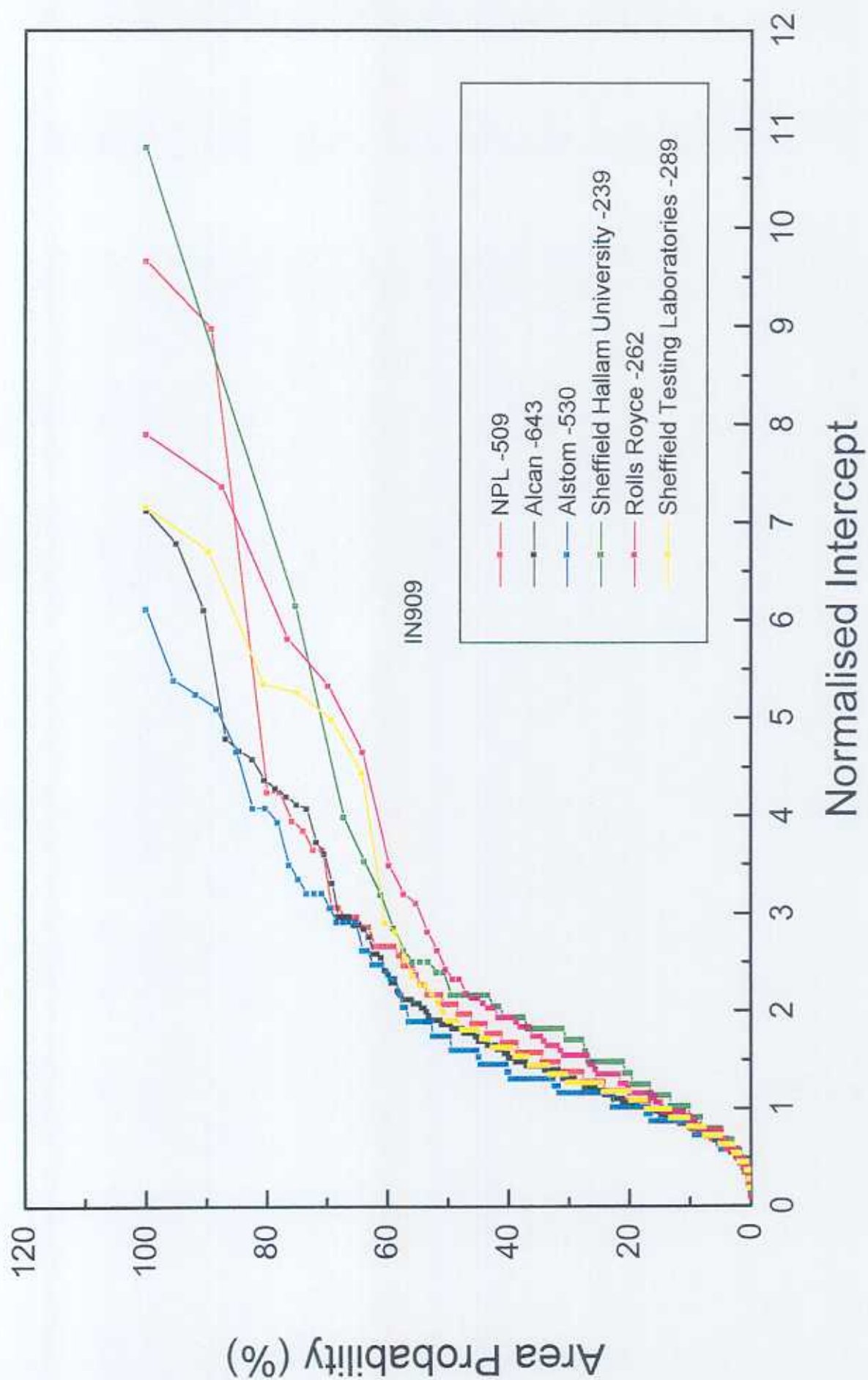
Fig 19 Estimated area probability plotted against log normalised intercept and compared with the model tetrakaidcahedron data

Fig 20 Area (E) probability plotted against normalised intercept all
participants for each material.









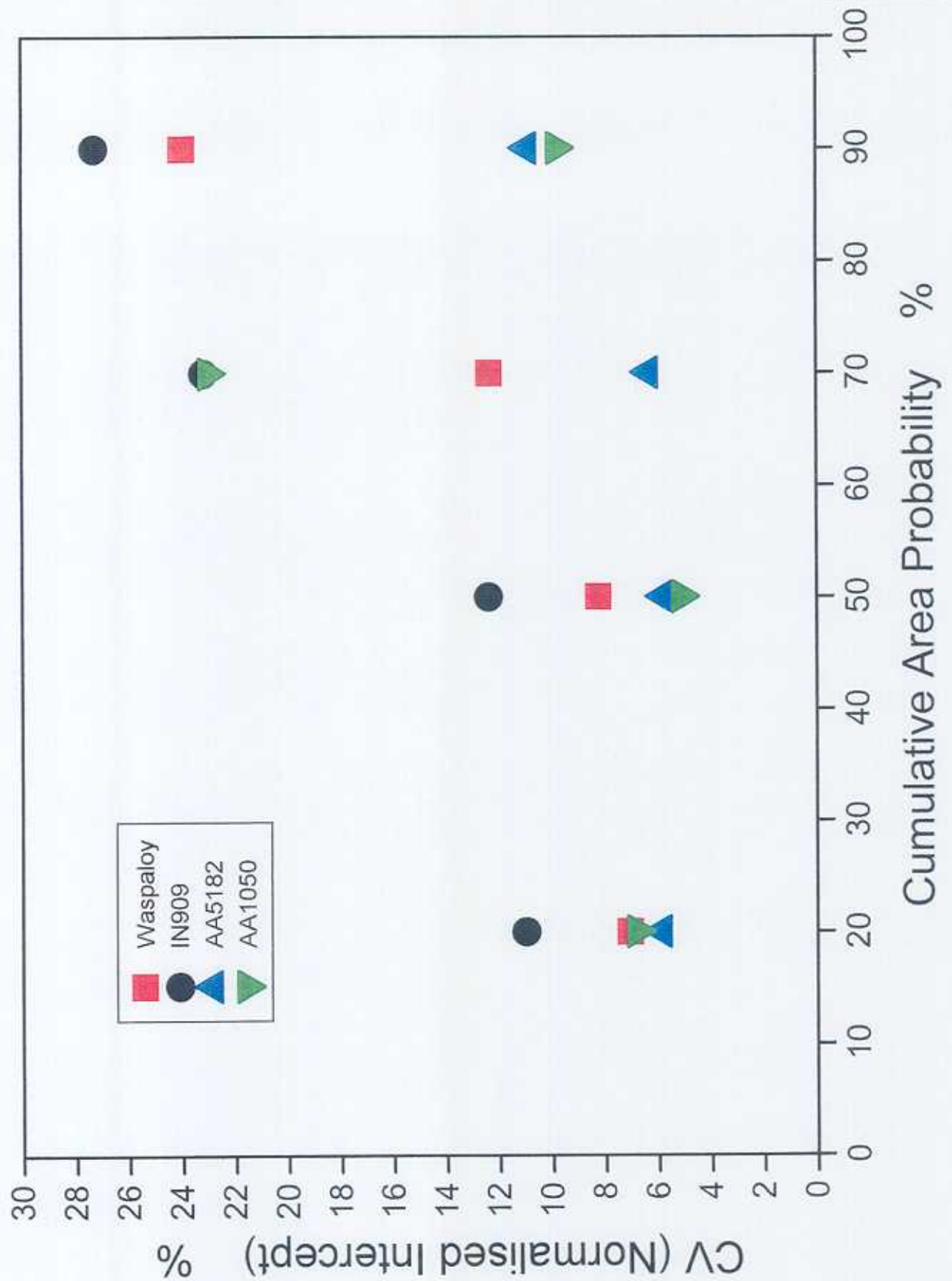


Fig 21

Coefficient of variation of the normalised intercept at fixed values(20, 50, 70 and 90%) of cumulative area probability.

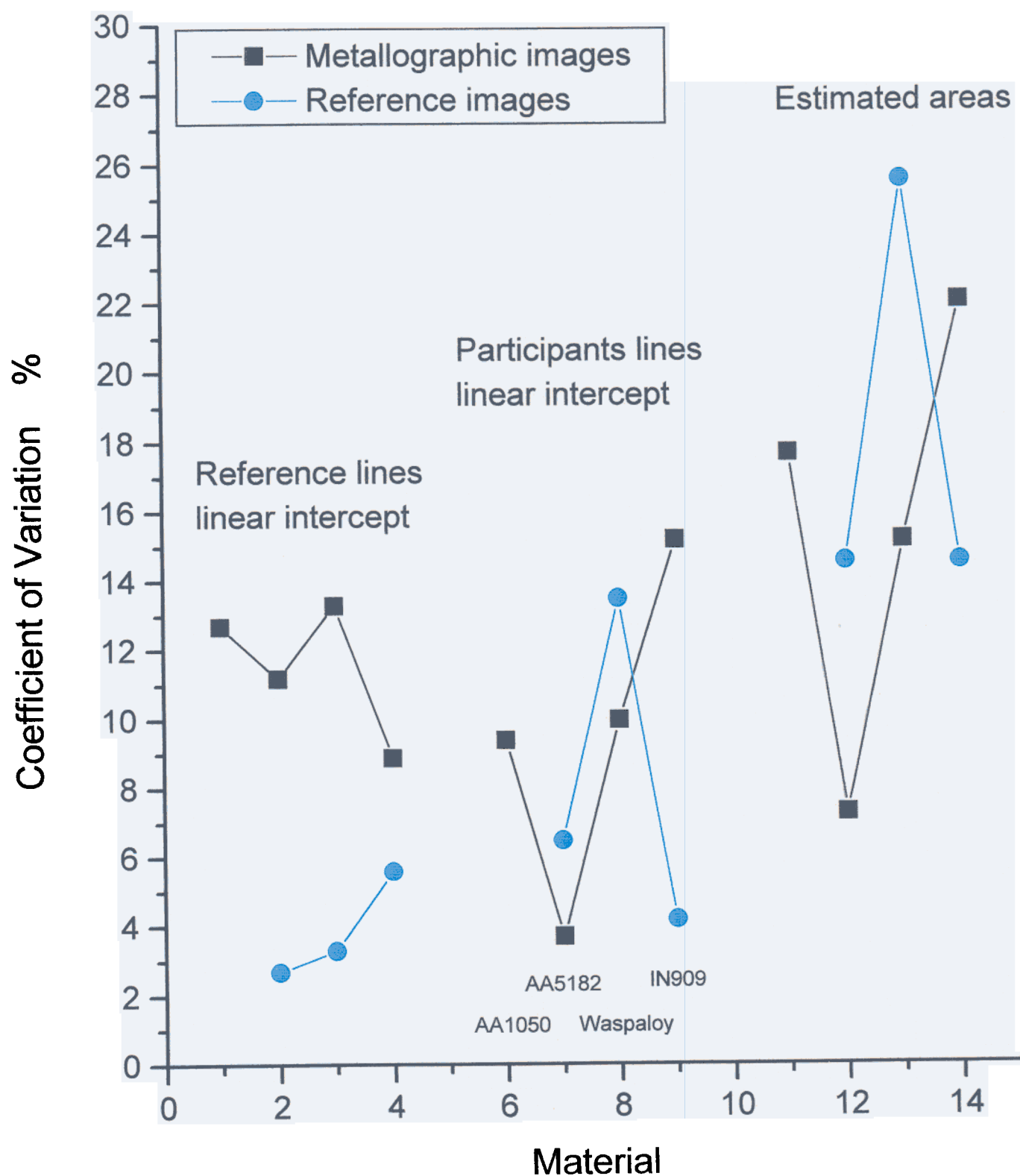


Fig 22 Coefficient of variation of the mean values obtained by the measurement exercise in parts I and II.

APPENDIX A

MMP14 PARTICIPANT ADDRESSES

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* Information pack sent - either data not received in time for inclusion in this report, or pack sent for information only.

APPENDIX B MMP14 GRAIN SIZE MEASUREMENT

ABBREVIATED INSTRUCTION SET ILE PART 2 METALLOGRAPHIC IMAGES

ILE II PACK CONTENTS

Please check that the contents of your Inter - Laboratory Exercise (ILE) stage II pack are as follows.

- 1) **Instruction Sheets**
- 2) **Data Sheets**
- 3) **Printed Images (20 images in total)**

Waspaloy			(5 Images)	Magnification X500
Image A	Image B	Image D		
Image F	Image I			
IN909			(5 Images)	Magnification X500
Image A	Image B	Image D		
Image E	Image F			
Aluminium Alloy AA5182			(5 Images)	Magnification X473
Image A	Image E	Image F		
Image I	Image J			
Commercial Purity Aluminium AA1050			(5 Images)	Magnification X240
Image E	Image F	Image G		
Image H	Image J			

- 4) **Floppy disks (20 disks in total) 1024 x 772 pixels**

Waspaloy images			0.477 $\mu\text{m}/\text{pixel}$
Set 1A,	waspa.tif	waspb.tif	waspd.tif
Set 1B	waspf.tif	waspi.tif	
IN909 images			0.477 $\mu\text{m}/\text{pixel}$
Set 2A	IN9aa.tif	IN9ea.tif	IN9fa.tif
Set 2B	IN9ba.tif	IN9da.tif	
Aluminium Alloy AA5182			0.505 $\mu\text{m}/\text{pixel}$
Set 3A	Alala.tif	Alale.tif	Alalf.tif
Set 3B	Alali.tif	Alalj.tif	
Commercial Purity Aluminium AA1050			1.05 $\mu\text{m}/\text{pixel}$
Set 4A	Alume.tif	Alumef.tif	Alumg.tif
Set 4B	Alumh.tif	Alumj.tif	

INSTRUCTION SHEETS, PRINTED IMAGES

- .0 The first part of the exercise is to be carried out by all participants, even those who will be using automatic or semi-automatic image analysis for the additional parts of the exercise. Linear intercept measurements by manual methods are to be carried out on the following images supplied using the magnifications quoted below. The intercept line is across the diagonal of the image as indicated by the markers (coloured circular discs) on the photo micrograph. We recommend using a steel rule.*

Waspaloy Image A	Magnification x500
IN909 Image A	Magnification x500
Aluminium Alloy AA5182 Image A	Magnification x473
Commercial Purity Aluminium AA1050 Image E	Magnification x240

- 1 For Waspaloy Image A, a diagonal line is drawn across the image as indicated by the markers. The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 1a. From the measurements on data sheet 1a, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 1a.
- 1.2 For IN909 Image A, a diagonal line is drawn across the image as indicated by the markers. The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 1b. From the measurements on data sheet 1b, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 1b.
- .3 For Aluminium Alloy AA5182 Image A, a diagonal line is drawn across the image as indicated by the markers. The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 1c. From the measurements on data sheet 1c, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 1c.
- 1.4 For Commercial Purity Aluminium AA1050 Image E, a diagonal line is drawn across the image as indicated by the markers. The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 1d. From the measurements on data sheet 1d, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 1d

The data provided from this exercise is to establish a control set of data and to highlight any problems which may occur from misinterpretation of the instructions.

* The steel rule should conform to BS 4372, 1968 (1996), Specification for Engineers Steel Measuring Rules

INSTRUCTION SHEETS, PRINTED IMAGES

Manual Linear Intercept Measurement, for those not using automatic or semi automatic image analysis system.

- 2 Measurement of linear intercepts on Waspaloy Images, Image A, Image B, Image D, Image F and Image I. Participants are to draw their own lines on the images for linear intercept measurements. These lines shall be parallel to the x axis (longest side) and spaced apart such that any grain is not intercepted by more than one line. For each of these lines, the intercept of the line with the underlying grain is measured. Grains which touch the border of the image are not to be measured.

At least one line shall be drawn on each of the images. The total number of lines drawn should ensure that at least 200 grains are intercepted. However, participants may draw as many lines as they wish providing that any grain is not intercepted by more than one line. The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 2a. From the measurements on data sheet 2a, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 2a.

- 2.2 Measurement of linear intercepts on IN909 Images, Image A, Image B, Image D, Image E and Image F. Repeat the exercise as per instructions 2.1.

The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 2b. From the measurements on data sheet 2b, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 2b.

- 2.3 Measurement of linear intercepts on Aluminium Alloy AA5182 Images, Image A, Image E, Image F, Image I and Image J. Repeat the exercise as per instructions 2.1.

The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 2c. From the measurements on data sheet 2c, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 2c.

Measurement of linear intercepts on commercial purity aluminium AA1050 Images, Image E, Image F, Image G, Image H and Image J. Repeat the exercise as per instructions 2.1.

The length of the line intercepting each grain is measured (in mm) and the calculated length in μm entered onto the data sheet 2d. From the measurements on data sheet 2d, calculate the arithmetic mean linear intercept grain size and the standard deviation. Enter the data on data sheet 2d.

If an "In - House" procedure exists for grain size measurement, please use it on each of the images . Please send details of the procedure and results on separate sheets of paper or spreadsheet.

INSTRUCTION SHEETS, DIGITAL IMAGES

3.0 Semi-Automatic or Automatic Image Analysis

3. Measurement of linear intercepts on TIF images, waspa.tif, waspb.tif, waspd.tif, waspf.tif and waspi.tif. **The calibration factor for these images is 0.477 $\mu\text{m}/\text{pixel}$.** There is no geometric distortion between the X and Y axis. Linear intercepts to be measured along the X axis, the intercept lines spaced apart such that any grain is not intercepted by more than one line. At least one intercept line shall be used on each of the Waspaloy images. The total number of intercept lines used should ensure that at least 200 grains are intercepted. However, participants may use as many intercept lines as they wish providing that any one grain is not intercepted by more than one line.

Data can be returned either as a computer printout or as a spread sheet.

- 3.2 Measurement of linear intercepts on TIF images, IN9aa.tif, IN9ea.tif, IN9fa.tif, IN9ba.tif and IN9da.tif. **The calibration factor for these images is 0.477 $\mu\text{m}/\text{pixel}$.** At least one intercept line shall be used on each of the IN909 images. Instructions for measurement the same as 3.1

Data can be returned either as a computer printout or as a spread sheet.

- 3.3 Data Measurement of linear intercepts on TIF images, Alala.tif, Alale.tif, Alalf.tif, Alali.tif and Alalj.tif. **The calibration factor for these images is 0.505 $\mu\text{m}/\text{pixel}$.** At least one intercept line shall be used on each of the aluminium alloy AA5182 images. Instructions for measurement the same as 3.1

Data can be returned either as a computer printout or as a spread sheet.

- 3.4 Measurement of linear intercepts on TIF images, Alume.tif, Alumf.tif, Alung.tif, Alumh.tif and Alumj.tif. **The calibration factor for these images is 1.05 $\mu\text{m}/\text{pixel}$.** At least one intercept line shall be used on each of the commercial purity aluminium AA1050 images. Instructions for measurement the same as 3.1

Data can be returned either as a computer printout or as a spread sheet.

If an "In - House" procedure exists for grain size measurement by semi automatic or automatic image analysis, please use it on each of the images . Please send details of the procedure and results on separate sheets of paper or spreadsheet.

DATA SHEETS

Instructions 1.1 to 1.4, all participants to complete the following data sheets

Data Sheet 1a	Waspaloy
Data Sheet 1b	IN909
Data Sheet 1c	Aluminium Alloy AA5182
Data Sheet 1d	Commercial Purity Aluminium AA1050

Alternatively, the data can be sent back on floppy discs

Instructions 2.1 to 2.4, participants using manual linear intercept measurements complete the following data sheets (minimum of 200 intercepts for each set). Results from linear intercept measurements (Instructions 2.1 to 2.4), at least one line to be drawn across each of the 5 images provided. Use spare data sheets as necessary.

Data Sheet 2a	Waspaloy
Data Sheet 2b	IN909
Data Sheet 2c	Aluminium Alloy AA5182
Data Sheet 2d	Commercial Purity Aluminium AA1050

Alternatively, the data can be sent back on floppy discs

Instructions 3.1 to 3.4, participants using automatic or semi-automatic image analysis are not provided with data sheets since the results can be provided as a computer printout.

Alternatively, the data can be sent back on floppy discs

APPENDIX C

CORRECTED FIG 20 (Part I Report) (SHEFFIELD TESTING LABORATORY DATA)

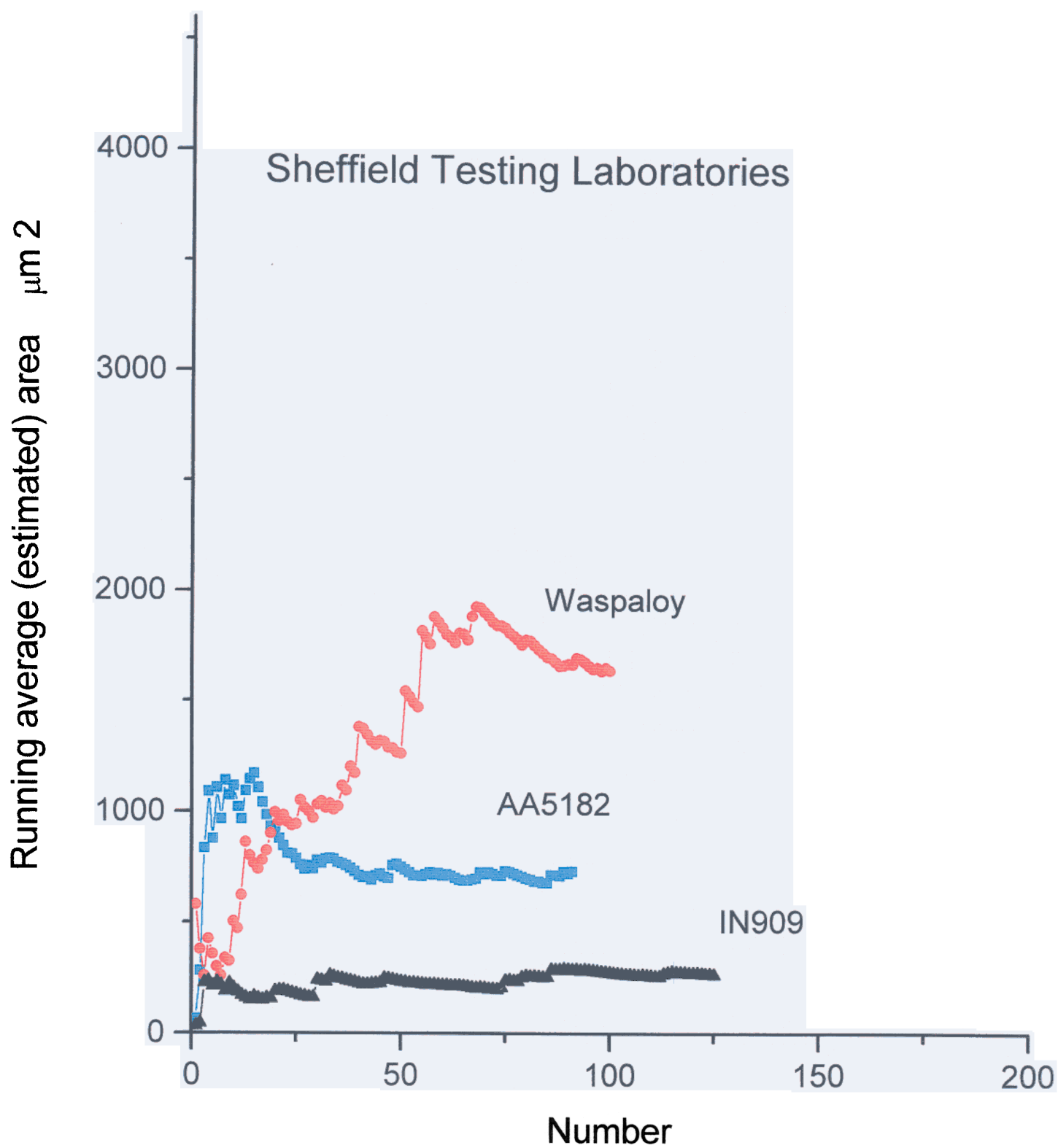


Fig 20 (Part I report) corrected