The Measurement and Control of Cotton Fibre Properties

Reference Methods for Cotton Fibre Maturity

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The largest impediment in the development of a fast, accurate and reproducible method for fibre maturity is the lack of a suitable reference measurement for calibration and validation purposes. Without an accepted reference adapting technologies such as near infrared reflectance (NIR) spectroscopy to measure fibre maturity will never be properly accomplished.

The following is a summarised version of a paper written on research into two potential reference methods for a NIR fibre maturity and fineness instrument being developed at the United States Department of Agriculture's (USDA) Southern Regional Research Center (SRRC). The objective of the research was to compare values of perimeter and wall thickness derived from the values given by two instruments; the Shirley Micromat and the Zellweger Uster Advanced Fibre Information Systems Fineness and Maturity Module (AFIS F&M). The aim was to choose one of the instruments as a reference method for calibrating NIR spectroscopy. Comparisons were made on the level of error associated with each method and the degree to which data from each method conformed with theoretical relationships.

INTRODUCTION

The advantages of NIR spectroscopy over other instruments currently used to analyse cotton fibres are the simplicity of sample preparation and the rapid, non-destructive nature of the test. The main disadvantage is that laboratory data used to calibrate the spectrophotometer are adversely affected by random error and drift thereby reducing the uniqueness of the relationship with corresponding sample spectra.

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It has been shown [1-3] that cotton fibre fineness and maturity, defined as perimeter (P) and cross-sectional wall thickness (t) or area (WA) respectively, are fundamental properties sensed by NIR.

In 1991 a NIR spectrophotometer, calibrated to measure fibre fineness, maturity and sugar, was incorporated into high volume instrument (HVI) lines used to class cotton. A recent evaluation found poor correlations between the HVI NIR estimates of maturity, fineness and sugar, and laboratory determined values [4,5]. Using data collected on over 2000 samples from the 1992 U.S. crop year the following coefficients of determination (R^2) between laboratory data and HVI NIR predicted values were found; maturity (0.303), fineness (0.144) and sugar (0.454). These numbers suggest that as currently implemented the HVI NIR does not give effective estimates of the above properties.

Given that fibre fineness and maturity are fundamental properties sensed by NIR, the poor correlations between the laboratory data and NIR estimates were assumed to be the result of significant errors in one or all of the following; sampling, NIR spectra or the laboratory reference method. Changes to the method of presenting NIR specimens for analysis so that cotton spectra could be measured with greater precision were reported recently [6]. Among the changes were increases in the specimen area scanned and the application of a minimum force to the specimen during measurement. Improvements have also been gained by using a diode-array instrument that allows a greater number of replicates to be measured [7]. If it is assumed that sampling error can be reduced by increasing sample size and thorough blending, then the remaining obstacle to achieving successful calibration of NIR is the lack of an accurate and precise laboratory reference method.

Data from a wide range of laboratory methods have been used to evaluate the relationship between NIR spectra and fibre fineness and maturity. Methods have included the Causticaire method [8,9], the Arealometer [3,9], the SDL Fineness and Maturity Tester (FMT) [8,10] and image analysis [3,9,10]. Of these, the best correlations have been found using mean values based on many replicates from airflow instruments such as the SDL FMT. This is due, in part, to the

comparatively smaller random error in the data from these methods compared to image analysis methods.

Random and Systematic Error

To obtain an indication of the level of random and systematic error affecting laboratory data and, hence, the amount of error affecting a NIR calibration, a new test statistic based on an internal correlation of paired replicate means was used. The statistic is able to give information on the amount and type of error, e.g. drift, affecting a calibration set. Ideally, the statistic, denoted M_{SPLIT}, will approach 1 for data affected by a small amount of error and be significantly less than 1 for data affected by a large amount of error. Further detail on the theory and properties of this statistic are given in [11].

Perimeter and Wall Thickness Values From Micromat and AFIS F&M Data

The various expressions of fineness and maturity given by the Micromat and AFIS F&M are theoretically related. In this study the fundamental expressions of fineness and maturity, i.e., perimeter and wall thickness, were derived to allow direct comparisons between Micromat and AFIS F&M data. The values measured by each instrument are presented in Table 1.

The Relationship Between Perimeter and Wall Thickness

An inspection of plots of wall thickness versus perimeter from experimental data provides an indication of how much random error affects the experimental data. The large smear of points, which is the main feature of the relationship between these properties, would seem to indicate that the relationship is not a close one. However, for a set containing a wide range of cottons a regression line with a positive slope would be expected. Alternately, if only one variety is represented a regression line would be expected to show wall thickness largely independent of perimeter. Of interest in this study is the clarity with which these relationships are born out in the experimental data. Any error in the data will result in the distortion of the expected theoretical relationships.

MATERIALS AND METHODS

Cotton Samples

Two sets of cotton were examined in this study. The first comprised four major U.S. varieties, Acala 1517-88, DP-50, DP-90 and Paymaster HS26, grown at 20 locations (a total of 80 samples) across the cotton belt as part of the National Variety Cotton Test (NCVT) Program in 1993. All cottons in this set were ginned under the same conditions at the USDA Ginning Laboratory in Stoneville, Mississippi.

The second set represents a collection of 41 cottons from Africa, Central and South America and Asia. These cottons had a wider range of fibre properties than the first and had also been subject to a wider range of preparations. The purpose of testing this second set was to validate, on cottons other than U.S. growths, the relationships found between AFIS F&M and Micromat measurements.

Instrument Methods

The Micromat and AFIS F&M both measure fibre fineness and maturity indirectly. This means that the instruments measure some fibre or fibre bundle property that varies concomitantly with fineness and maturity and a mathematical equation is used to convert instrument readings to expressions of fineness and maturity.

The Micromat

The SDL Micromat Tester is a double compression airflow instrument designed to measure fibre fineness and maturity. Micronaire is also measured.

In this study amendments were made to the prescribed SDL method in an attempt to increase the precision of the data. They included increasing checks of pressure and flow gauges in order to avoid drift in values. Early in the study P_L values were found to drift over half a day when only an initial adjustment was made to pressure and flow meters. The drift was halted when checks were increased to one every half hour during testing. As well, the SDL method calls for a blended specimen weighing between 3.8 and 4.2 g. However, Montalvo *et al.* [12] found that P_L and P_H values increased as specimen weight was increased

within this prescribed range. In view of this specimens were weighed consistently to 4 ±0.005 g to increase the precision of data.

The AFIS F & M Module

The AFIS is an instrument that has been developed to rapidly measure a range of single fibre properties. Measurement is based on detecting the amount of light that is scattered and occluded by an object, i.e., a fibre or nep, as it is transported by air through a beam of near infrared light.

Preparing an AFIS specimen involves hand-drawing a 0.2 g tuft of fibers into a 25 cm length. Up to 10,000 fibres can be analysed per specimen, though this number does not represent the total number of fibres fed into the system. Many more fibres are rejected from the analysis because they are tangled or have hooked ends as they pass through the light beam. Discrimination of fibres on this basis is aimed at avoiding the inclusion of erroneous measurements and ensuring the accurate reporting of fibre distributions. Specimen preparation had a considerable effect on the accuracy and precision of AFIS data. Better precision in fibre property means was found when specimens were prepared from mechanically drawn fibres rather than hand-drawn fibres. This was attributed to the greater evenness and parallelisation of fibres in the mechanically drawn specimen which allowed fibres to be separated more easily for measurement. Means were also affected when the density of the specimen was outside the prescribed weight to length ratio.

Order of Testing

Experiments I and II - NCVT93 Cottons

Two experiments (I and II) were conducted using the NCVT93 cottons. In Experiment I, 25 g of raw fibre from each of the 80 samples were cleaned mechanically using a fibre blender and divided into six 4 ±0.005 g specimens for Micromat analysis. The order of testing was random though each of the six specimens was measured before the next sample was tested. The specimens from a sample were then collected together and re-divided equally into four sub-samples of six grams. From each sub-sample a set of five AFIS specimens was prepared by hand. The specimens in each set were measured consecutively with 5000 fibres

being counted for each measurement. One set of each sample (one to 80) was analysed before a second set was tested.

In Experiment II, 100 g of raw cotton from each of the 80 samples was blended and cleaned. The cleaned fibre mass was divided into 20 4 ±0.005 g specimens for Micromat analysis. One set of five specimens was analysed for each of the 80 samples before another set of specimens prepared from the same sample was tested. The order of testing was random.

Experiment III - International Cottons

The same approach of splitting replicates into sets and testing the first set of all samples, in random order, before testing the second set was used.

RESULTS

M^2_{SPLIT} Values For Micromat and AFIS Paired Means

Table 2 lists M^2_{SPLIT} values calculated for internal correlations of Micromat and AFIS F&M replicates. Values are given on a per set basis to illustrate the effect of drift on correlations. The drift in Micromat values was associated with the frequency with which the pressure and airflow meters were checked. Initially, checks were only made prior to testing. However, following the observance of drift, checks were increased to each half hour. For sets three to five the drift in values was partially stabilised with the consequence that M^2_{SPLIT} values were higher. The overall consequence of drift in Micromat values was an M^2_{SPLIT} value of only 0.267 for perimeter paired means. Correlations between wall thickness paired means remained high despite the drift in values (M^2_{SPLIT} = 0.901), suggesting a degree of robustness in the measurement of this property.

M^2_{SPLIT} values for AFIS F&M data suggest there was a slight drift in both wall thickness or perimeter values. However, the lower M^2_{SPLIT} values were attributed to a change in the sliver preparation procedure that resulted in the linear density of specimens being increased over the time tests were carried out.

Overall, Micromat M^2_{SPLIT} values for perimeter replicates were smaller than AFIS F&M M^2_{SPLIT} values, indicating a greater level of error in the Micromat measurement of this property. The reverse was true for wall thickness measurements.

The variability in Micromat perimeter measurements can be partly explained by examination of the equations converting P_L and P_H values to fibre property values. It can be shown that a change, up or down, in both P_L and P_H produces a larger change in perimeter than in wall thickness. A similar examination of the AFIS equations shows that changes in V_0 and V_{40} values are translated proportionately to values of cross-sectional area and the degree of thickening.

The Relationship Between Wall Thickness and Perimeter

Figures 2 and 3 are plots of wall thickness and perimeter derived from Micromat and AFIS F&M values respectively. The regression line through the plot in Figure 2 shows that Micromat wall thickness and perimeter values (from Experiment II) increased together as expected for a set containing four varieties of cotton grown at 20 different locations. The line has a positive slope that is significantly different from zero and there is an even scatter of points on either side of the line, corresponding with the theoretical depiction of the relationship in Figure 1. Data from Experiment I was similarly related. Figure 3 shows the same plot using AFIS F&M wall thickness and perimeter values. The slope of the trend line is not different from zero and there is little to suggest that a relationship between wall thickness and perimeter actually exists.

CONCLUSION

Results from this work show that the Micromat is more robust in representing changes in wall thickness and perimeter and that the AFIS is more sensitive to the inherent fibre-to-fibre variability found in cotton. To some extent the larger error associated with AFIS F&M values should be expected since each mean is determined from 100,000 fibres as opposed to around 20 million fibres for Micromat values.

It remains that data from any indirect method such as NIR, airflow or light scattering instrument should be traceable to directly measured values. Direct measurement of fibre cross-section dimensions by image analysis is theoretically the most accurate way of determining fibre maturity. However, because it is practical to measure only small numbers of fibres by current applications, data are

affected by large random error and unusable for NIR calibration purposes. There is also, as yet, no standard approach to making these measurements.

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Table 1. Fibre Properties Measured

Shirley Micromat	**AFIS Fineness and Maturity Module**
Instrument measures	**Instrument measures**
P_L and P_H (mm H_2O), change in airflow across a plug of fibres compressed to two volumes during test. P_L is the pressure drop at low compression; P_H is the pressure drop at higher compression.	V_0 and V_{40}, voltage signals detected by photoelectric sensor when fibre impinges upon beam - V_0 is the signal representing the amount of light occluded by the fibre V_{40} is the amount of light scattered at a 40° angle by the fibre.
Calibrated fibre properties	**Calibrated fibre properties**
Micronaire	Diameter μm
Maturity Ratio	Degree of Thickening (θ) and CV
Percent Maturity	Immature Fibre Fraction IFF (% of fibres with values of θ less than 0.25)
Linear Density mtex	Cross-Sectional Area (WA) μm^2 and CV
	Fine Fibre Fraction FFF - (% of fibres with WA less than 60 μm^2)
	MicronAFIS
Fibre properties derived for this investigation	**Fibre properties derived for this investigation**
Perimeter μm	Perimeter μm
Wall Thickness μm	Wall Thickness μm

Table 2. M^2_{SPLIT} values for Micromat and AFIS data

		M^2_{SPLIT}	
set	**replicates per mean value**	**Perimeter**	**Wall Thickness**
	Micromat		
1-2	4	0.527	0.920
3-5	6	0.750	0.972
1-5	10	0.267	0.901
	AFIS		
1-2	5	0.875	0.846
3-4	5	0.766	0.883
1-4	10	0.834	0.830

Figure 1. Theoretical plot of wall thickness versus perimeter. Lines show relationship at constant degree of thickening.

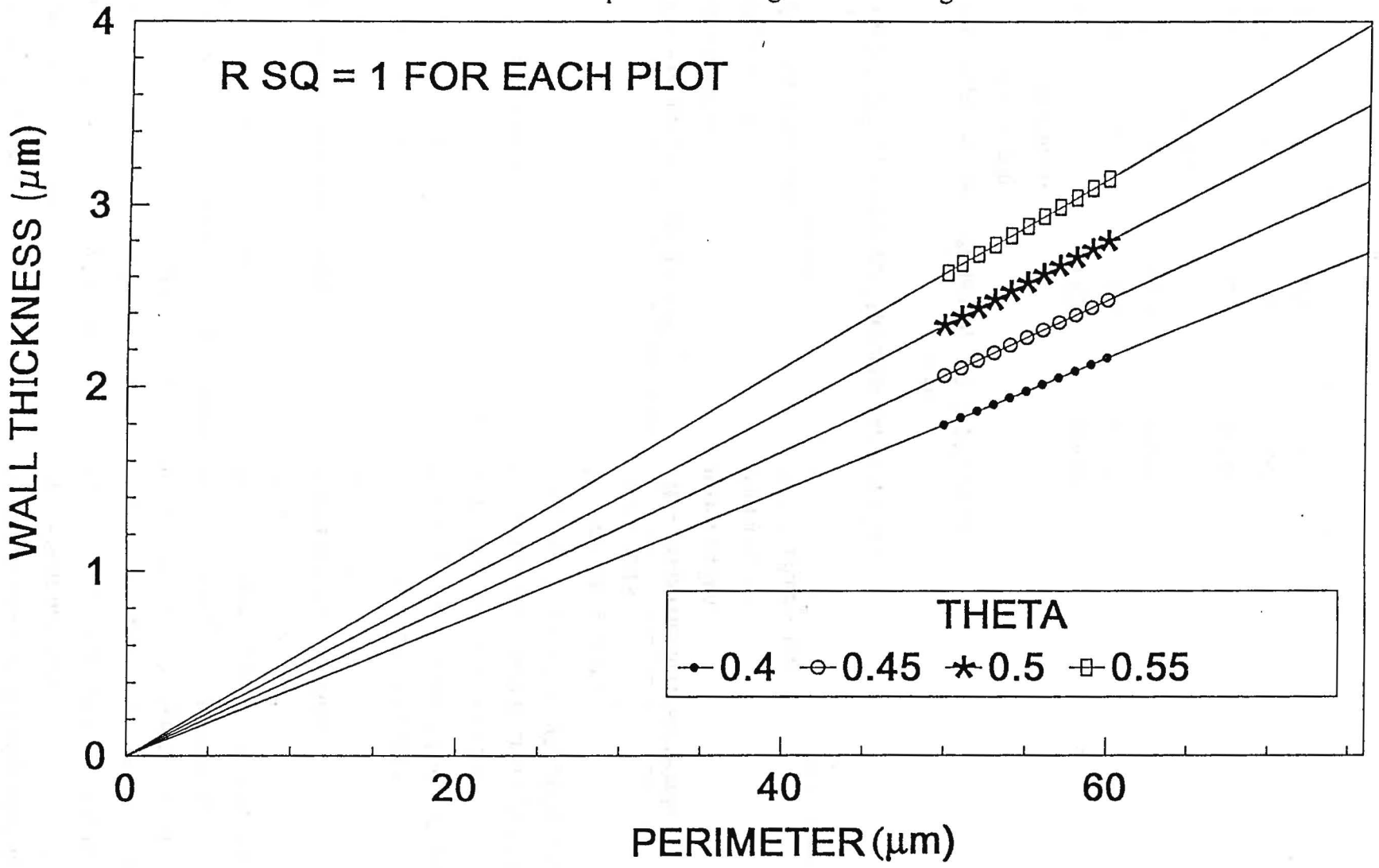

Figure 2. Wall thickness versus perimeter (values derived from Micromat data. 80 USA Cottons (NCVT)) 1993 Crop.

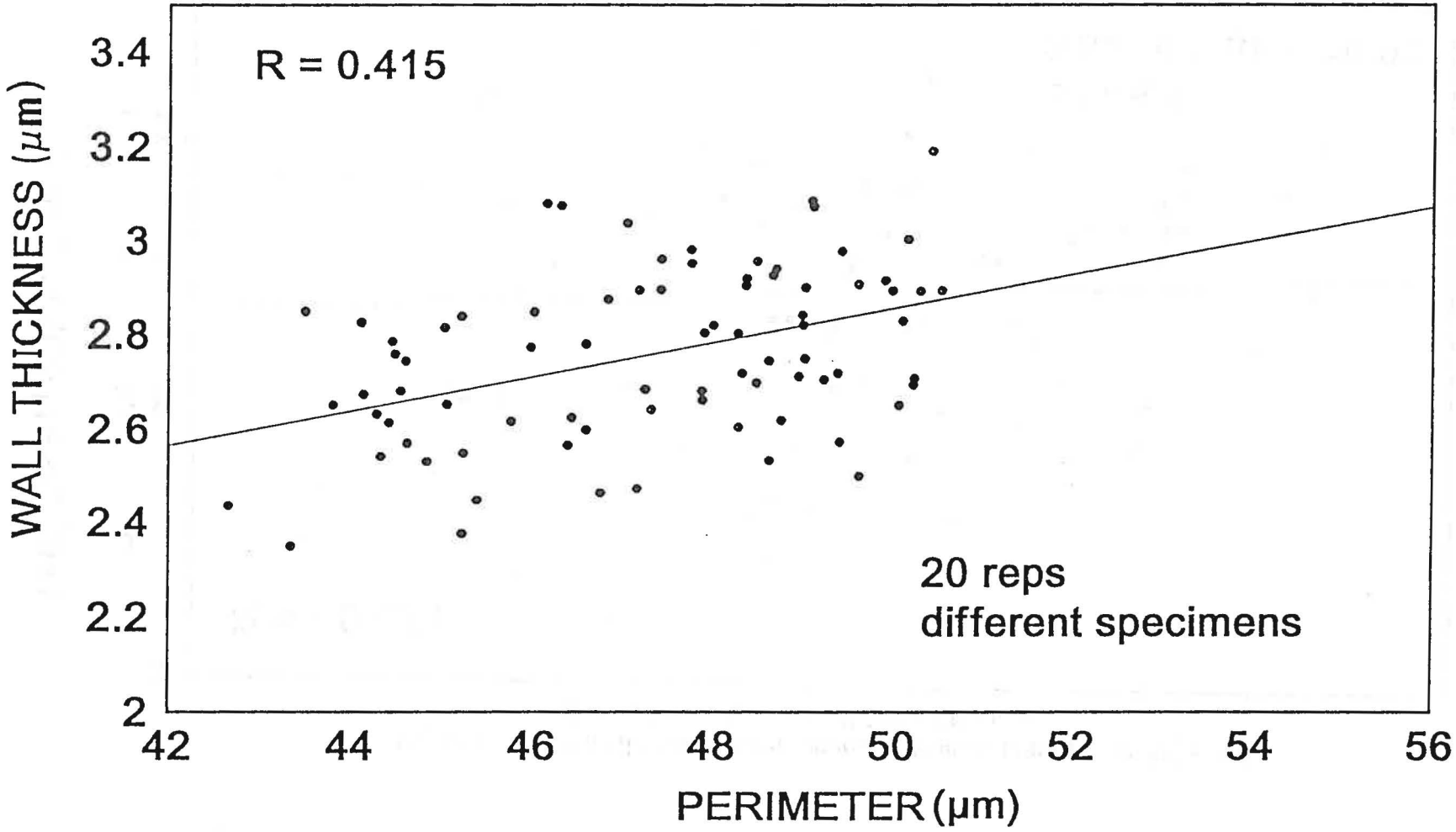

Figure 3. Wall thickness versus perimeter (values derived from AFIS F&M data). 80 USA Cottons (NCVT)) 1993 Crop.