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LINSEED OIL YIELD AND IODINE VALUE— REFRACTIVE INDEX CORRELATION FOR QUEENSLAND-GROWN LINSEED

By M. J. PRICE, B.Sc., Dip. Ind. Chem., Dip. Sug. Chem., A.R.A.C.I.

SUMMARY

Percentage oil (mean of eight varieties) varied at Hermitage in south-eastern Queensland from 40.5% in 1964 to 36.7% in 1965 and from 38.8% at Walkamin in northern Queensland in 1964 to 39.8% in 1965.

District and seasonal variations in iodine value (Wijs) and refractive index (25°C) were large in all varieties except Marine.

I. INTRODUCTION

In a previous communication (Price 1964), an iodine value—refractive index correlation coefficient of 0.95 was reported for solvent-extracted linseed oil obtained from various Queensland Department of Primary Industries field trials. During this study it was observed that the linseed varieties Bolley Golden and Marine produced oil of a consistently higher iodine value than that produced by other varieties such as Walsh and Hazeldean. As most of the samples originated from trials on the Darling Downs in south-eastern Queensland, it was felt necessary to determine if the varietal difference in oil quality would persist on the Atherton Tablelands, in northern Queensland.

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The results presented here were obtained from eight linseed varieties grown in replicated field trials at Hermitage Research Station (latitude 28° 13′S.) and Walkamin Research Station (latitude 17° 8′ S.) in 1964 and 1965. The seed used to plant both these areas was obtained from seed-increase trials conducted at Hermitage in 1963. In addition, a correlation coefficient and regression equation for iodine values and refractive indices of 30 miscellaneous samples of coldpressed linseed oil obtained from the 1962 harvest are presented.

II. MATERIALS AND METHODS

Varieties.—The following varieties of linseed were grown in randomized block design trials of four replicates: Walsh, Bonnydoon, Hazeldean, Marine, Viking, Dakota, Newlands and Bolley Golden. Each harvested sample was cleaned to remove all foreign material and the replicate samples were analysed for oil content, iodine value (Wijs) and refractive index (25°C).

Oil content.—Duplicate samples of seed were disintegrated, using a Braun high-speed mill. The meal was placed in a double-thickness Whatman extraction thimble in a Soxhlet apparatus and extracted with anhydrous ethyl ether (peroxide and alcohol-free) for 5 hr. The meal was removed, reground and re-extracted for a further 4 hr. Excess solvent was removed and the residual oil weighed.

Cold-pressed oil.—Forty-gram samples of seed were held between filter-paper discs and pressed at 5,000 lb/sq in in a 5·5-cm stainless steel die, using a hand operated hydraulic press. The expressed oil was decanted into glass phials, stoppered and stored in the dark until required for iodine value and refractive index determinations.

Refractive index.—Refractive indices of the cold-pressed oil samples were determined at $25^{\circ} \pm 0.1^{\circ}\text{C}$, using an Abbe refractometer with water-jacketed prisms (range 1.3-1.7 refractive index). The calibration of the instrument was checked within the operating range as suggested by Weissberger (1949, p. 1141). The value of $\frac{dn}{dt} = -3.6 \times 10^{-4}$ (where n = refractive index; t = temperature °C) determined for a composite sample of the linseed oil varieties examined agrees well with the value $\frac{dn}{dt} = -3.57 \times 10^{-4}$ quoted by Jamieson (1943, p. 462). Since the refractometer used was readable to four decimal places, the above temperature control was adequate to realise the potential of the instrument.

Iodine value (Wijs).—Wijs solution was prepared from iodine monochloride as described by Mehlenbacher (1960, p. 311); the halogen ratio was determined for each batch of iodine monochloride used. The glacial acetic acid used was free of reducing substances. Carbon tetrachloride was used as the sample solvent and was purified by the method described by Mehlenbacher (1960, p. 311).

Sodium thiosulphate solution was prepared and standardized as described in the Official Methods of Analysis of the Association of Official Agricultural Chemists (1960, p. 698).

III. RESULTS

Table 1 lists the analytical results for 11 varieties of linseed received from miscellaneous field trials in 1962. The regression equation relating iodine value to refractive index for these samples is almost identical with that given in Table 2. Also listed in Table 1 are the analytical results from the seed-increase trial at Hermitage in 1963. These results, although not subjected to statistical analysis, are included to demonstrate the quality of the parent seed samples used for planting the Hermitage and Walkamin trials in 1964.

TABLE 1
Analytical Results 1962 and 1963

	Miscell	aneous Samp	les 1962	Seed-increase Trial, Hermitage 1963				
Variety	Oil Percentage	Iodine Value	Refractive Index	Oil Percentage	Iodine Value	Refractive Index		
Walsh	. 35.7	159.8	1.4763	40.7	175 8	1.4780		
Bonny Doon	. 38.9	169.8	1.4773	40.0	179.6	1.4785		
Hazeldean	. 40.5	174-4	1.4780	41.0	184.9	1.4792		
Marine	. 38.1	189.8	1.4799	39.2	193.9	1.4802		
Viking	. 38.4	185.4	1.4792	39.3	195.1	1.4803		
Dakota	. 37.9	182.9	1.4790	38.3	190.7	1.4797		
Newlands	. 38.4	185-5	1.4793	39.4	-193.5	1.4802		
Bolley Golden	. 38.7	191-1	1.4799	40.0	197.3	1.4805		
Plate	. 37.4	160 8	1.4764					
Cheyenne	. 36.8	180.0	1.4788					
Calar	. 39.4	168-1	1.4774					
Mean	. 38.2	177.0	1.4783	39.7	188.8	1.4796		

Iodine value = 8398.5 (refractive index -1.4572)

r = 0.998

Standard error of estimate $= \pm 0.64$

TABLE 2

Analytical Results—Hermitage 1964, 1965 and Walkamin 1964, 1965

	Hermitage Research Station						Walkamin Research Station					
	1964			1965			1964			1965		
	Oil Percentage	Iodine Value	Refractive Index	Oil Percentage	Iodine Value	Refractive Index	Oil Percentage	Iodine Value	Refractive Index	Oil Percentage	Iodine Value	Refractive Index
Walsh	41.2	185-5	1.4791	39.2	164-8	1.4768	38.2	164-3	1.4770	40.6	171.9	1.4777
Bonny Doon	40.9	187-1	1.4793	36.5	166.3	1.4767	38.4	171.0	1.4772	40.2	176.5	1.4780
Hazeldean	41.9	185-3	1.4791	37.0	165-4	1.4767	40.0	174-4	1.4780	41-2	180-6	1.4786
Marine	40.0	197.7	1.4807	37.5	189-1	1-4799	37.6	184.5	1.4791	38-8	194-1	1.4801
Viking	40.6	198-1	1.4807	35-6	175-1	1.4781	39-1	180-5	1.4789	39.9	189.9	1.4798
Dakota	39.1	197-2	1.4806	36.5	178-9	1.4785	37.4	184-1	1.4789	38-5	187-1	1.4793
Newlands	39.8	196.5	1.4805	35.7	178-3	1.4786	39.6	182.0	1.4791	38.8	184-4	1.4791
Bolley Golden	41.0	201-2	1.4810	36.0	178-2	1.4784	39.9	185-1	1.4790	40.3	194.6	1.4802
Mean	40.6	193-6	1.4801	36.7	174.5	1.4780	38.8	178-2	1.4784	39.8	184.9	1.4791

Iodine value = 8396.23 (refractive index -1.4571)

r = 0.990

Standard error of estimate $= \pm 0.72$

Table 2 lists the analytical results (mean of 4) obtained from eight varieties grown in both districts in both years. The yearly averages for all varieties in each district are included, together with an overall regression equation for the complete trial period.

IV. DISCUSSION

Percentage oil.—The average oil content of all varieties grown in 1963 at Hermitage for seed-increase purposes was 39.7% (minimum 38.3%, Dakota; maximum 41.0%, Hazeldean). The same varieties grown at Hermitage in 1964 yielded an average oil content of 40.6% (minimum 39.1%, Dakota; maximum 41.9%, Hazeldean). In 1965 the mean oil content for all varieties at Hermitage fell to 36.7% (minimum 35.6%, Viking; maximum 39.2%, Walsh).

At Walkamin in 1964 the average oil content for all varieties was 38.8% (minimum 37.4%, Dakota; maximum 40.0%, Hazeldean). In the following year the mean oil content for all varieties at Walkamin was 39.8% (minimum 38.5%, Dakota; maximum 41.2%, Hazeldean).

There was less seasonal variation in oil content at Walkamin than at Hermitage. At Hermitage, Walsh was the only variety to maintain a reasonably constant oil content. Dakota produced the lowest yield of oil on four occasions and Hazeldean was the maximum yielder on four occasions.

Iodine value.—Seasonal variation in the mean iodine value of varieties grown at Hermitage was large—188·8 in 1963 to 193·6 in 1964 followed by 174·5 in 1965. Marine was the most consistent variety with respect to iodine value, registering 193·9 in 1963, 197·7 in 1964 and 189·1 in 1965. The Queensland commercial variety Walsh recorded 175·8 in 1963, 185·5 in 1964 and 164·8 in 1965.

At Walkamin in 1964 the mean iodine value for all varieties was 178·2 and in 1965 the mean had increased to 184·9. The variety Marine produced oil of the most consistent quality regardless of district or seasonal influence. Dakota, Newlands and Bolley Golden in both districts in both years produced oil above the minimum acceptable iodine value of 177 specified by the Australian Commonwealth Engineering Standards Association (1928, p. 9).

Iodine value-refractive index relationship.—The following regression equation was derived for the 11 varieties harvested in 1962 and listed in Table 1:

Iodine value = 8398.5 (refractive index—1.4572).

On inserting the mean, minimum and maximum refractive index values for the linseed oil samples examined in 1962, 1963, 1964 and 1965 in the above equation, the calculated iodine values listed in Table 3 result.

TABLE 3

Comparative Calculated* and Determined Iodine Values

Item	1962	1963	1964 Walkamin	1964 Hermitage	1965 Walkamin	1965 Hermitage	
Mean (determined)	177.0	188-8	178.2	193.6	184.9	174.5	
Mean (calculated)	177-2	188-1	178.0	192.3	183.9	174.7	
Minimum (determined)	159.8	175.8	164.3	185-3	171.9	164.8	
Minimum (calculated)	160.4	174.7	166∙3	183.9	172.2	164.6	
Maximum (determined)	191.1	197.3	185.1	201.2	194.6	189-1	
Maximum (calculated)	190.6	195.7	183.0	199.9	193.2	190.6	

^{*} Calculated using equation: Iodine value = 8398.5 (refractive index -1.4572)

It is apparent therefore that the regression equation derived in 1962 can be successfully used to estimate the iodine values of samples grown in different areas in later years. If the regression equation derived from the 1964 and 1965 samples from Walkamin and Hermitage and listed below Table 2 was used to calculate the iodine values listed in Table 3, the results would all be 0.79 greater. On any one determination it would be possible for an error of \pm 0.0001 refractive units to occur, which in either of the regression equations results in an iodine value error of \pm 0.83. These errors are not significant when oil samples are being examined for plant-breeding purposes.

Table 4 compares the determined iodine values (mean of 4) with the calculated iodine values (mean of 4), using the following regression equation: Iodine value = $8396 \cdot 23$ (refractive index $-1 \cdot 4571$).

TABLE 4

Comparative Calculated* and Determined Iodine Values: Hermitage and Walkamin

Research Stations 1964 and 1965

Variety	Hermita	ge 1964	Hermitage 1965		Walkam	in 1964	Walkamin 1965	
	Determ. I.V.	Calc. I.V.	Determ. I.V.	Calc. I.V.	Determ. I.V.	Calc. I.V.	Determ. I.V.	Calc. I.V.
Walsh	185.5	184.9	164.8	165.2	164.3	166.9	171.9	173.0
Bonny Doon	187-1	186.6	166.3	164.8	171.0	168-8	176.5	175.9
Hazeldean	185.3	185-1	165.4	165.0	174.4	175.9	180.6	180.9
Marine	197.7	198.2	189-1	191.2	184.5	184.7	194.1	193.6
Viking	198-1	198.1	175.1	176.5	180.5	183.0	189.9	190.7
Dakota	197-2	196.9	178.9	179.9	184-1	183.0	187-1	187.0
Newlands	196.5	196.5	178.3	180.5	182.0	184.5	184.4	184.7
Bolley Golden	200.7	200.5	178-2	178.6	185.1	183.9	194.6	194.0
Average	193.6	193.3	174.5	175-2	178-2	178.8	184-9	185.0

^{*} Calculated using equation: Iodine value = 8396.23 (refractive index - 1.4571)

The largest variation between calculated and determined iodine values was 2.6 at Walkamin in 1964 for the variety Walsh. In 1965 the largest variation between calculated and determined iodine values was 2.2 for the variety Newlands grown at Hermitage.

The above results demonstrate that the regression equation is a rapid, reliable and cheap method of determining iodine values of oil samples and consequently the quality of oil. The use of cold-pressed oil for refractive index measurements eliminates any heat degradation of the oil sample and allows larger numbers of samples to be processed in a given time.

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REFERENCES

Association of Official Agricultural Chemists (1960).—"Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists" 9th Ed. (Association of Official Agricultural Chemists: Washington).

Australian Commonwealth Engineering Standards Association (1928).—Standards Nos. K2 to 6.

Jamieson, G. S. (1943).—"Vegetable Fats and Oils", 2nd Ed. (Reinhold:New York).

MEHLENBACHER, V. C. (1960).—"Analysis of Fats and Oils". (Garrard:Danville).

PRICE, M. J. (1964).—Proc. News Aust. Sect. Oil Colour Chem. Ass. 1 (11):15.

WEISSBERGER, A. (1949).—"Techniques of Organic Chemistry, Vol. 1, Part 2". (Interscience: New York).

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The author is an officer of the Agricultural Chemical Laboratory Branch, Division of Plant Industry, Department of Primary Industries, and is stationed at Brisbane.