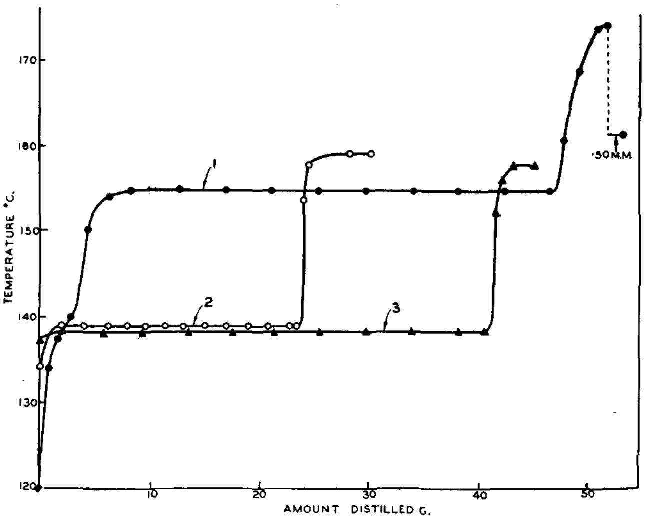

FIG. 8. Fractionation of the fatty acid methyl esters of Terminalia Kaernbachiana at 1.00 mm.

The vigorous action of the wipers on a reflux of low viscosity, formed by the test mixture, may generate a spray which is easily entrained by the fast moving vapors.

Comparison of this column with spinning band columns described in the literature is only possible at atmospheric pressure. Birch *et al.* reported an H.E.T.P. of 5.5 cm. for a column of 36-mm. internal diameter. For columns of 5 to 6 mm. internal diameter H.E.T.P. values of 7.8 and 7.1 (1), 3.12 (7), and 2.50 cm. (8) have been reported. The present column with rotor A had a plate equivalence of 70 under total reflux and therefore an H.E.T.P. of 2.07 cm. A semimicro column 75 cm. in length and 6-mm. diameter with a type A rotor has recently been constructed in the Dairy Research Section of this organization, for which an H.E.T.P. of only 1.17 cm. was found (9). This improvement in efficiency of these two columns over earlier columns is attributed mainly to the centering of the rotors by bearings.

The spinning band column has several desirable features: a low hold-up, a low pressure drop, does not need preflooding, and is not subject, as are packed columns, to channelling. One disadvantage not eliminated in the present column is the low take-off rate (3-10 g./hr.), but with pressure control and automatic take-off this is compensated by the small attention needed, apart from changing receivers. The present work shows that for most efficient operation different types of rotors should be used at atmospheric and low pressures.

The column described has lately been reconstructed and the length of the rotor extended to 366 cm. Rotor E was remade, eliminating the use of silver solder. This is attacked by fatty acids and the copper salts formed would catalyze the polymerization of unsaturated fatty acids. No mechanical difficulty resulted from this extension, but it was found that the rotor speed could be reduced to 700 r.p.m. without decrease of efficiency.

Acknowledgments

This work formed part of the research program of the Organic Section of the Division of Industrial Chemistry, Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia. The writer gratefully acknowledges the practical assistance and suggestions contributed at times by members of this Division, in particular W. G. Jones, S. E. Powell, J. Mills, R. B. Morrow, and R. Schoenfeld.

Summary

A spinning band fractionating column is described for operation at pressures down to 0.5 mm. of mercury. A study has been made of various designs of rotor. One type consisting of flat blades of stainless steel interspaced with bearings at regular intervals, which centered it in the column, gave an H.E.T.P. of 2.07 cm. at atmospheric pressure. At low pressures (0.5 mm.) its efficiency was found to be small, and a more suitable rotor has been developed in which strips of stainless steel gauze are attached to the blades. These wipe the walls of the column and provide an even film of reflux. Sharp separations of fatty acid methyl esters, fatty alcohols, and acetates have been made, using this rotor at pressures of 0.5-2.0 mm. On account of the low pressure drop (0.4 mm. mercury) it is possible to fractionate compounds with 30 or 32 carbon atoms without decomposition.

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Tables for the Conversion of Spectrophotometer Readings to A.O.C.S. Photometric Color Values

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ALTHOUGH the spectrophotometric method for determining oil colors possesses obvious advantages over the old method employing Lovibond color glasses used in the Wesson oil colorimeter, one disadvantage which is being felt by some laboratories results from the added work and time required to obtain color readings, compared to the older method. While the manufacturer of the spectrophotometer currently being most widely used in determining oil colors is supplying scales in terms of the A.O.C.S. color components corresponding to the readings at the four wavelengths, it is understood that only two components will be covered on one scale strip, thus requiring changing the scale on the instrument during the course of determining the color of an oil. When a large number of oils are being read however, this becomes less objectionable because it is no doubt customary to make all readings on a group of oils at

CONVERSION: TRANSMITTANCE TO PHOTOMETRIC COLOR COMPONENTS

WAVE LENGTH 460 mµ $PC_{460} = 1.29D_{460} = 1.29\ (-\log T_{460})$							
T.	P.C.	T	P.C.	T	P.C.	T	P.C.
.25	3.36	.5	.86	.5	.41	.5	.172
.5	2.97	22	.85	48	.41	74	.169
.75	2.74	.5	.83	.5	.40	.5	.165
1.0	2.58	23	.82	49	.40	75	.161
1.25	2.48	.5	.81	.5	.39	.5	.157
1.5	2.39	24	.80	50	.39	76	.154
1.75	2.29	.5	.79	.5	.38	.5	.150
2.0	2.19	25	.78	51	.38	77	.146
2.25	2.13	.5	.76	.5	.37	.5	.142
2.5	2.08	26	.75	52	.37	78	.139
2.75	2.02	.5	.74	.5	.36	.5	.135
3.0	1.96	27	.73	53	.36	79	.132
3.25	1.92	.5	.72	.5	.35	.5	.128
3.5	1.89	28	.71	54	.35	80	.125
3.75	1.85	.5	.70	.5	.34	.5	.121
4.0	1.81	29	.69	55	.33	81	.118
4.25	1.78	.5	.68	.5	.32	.5	.114
4.5	1.75	30	.67	56	.32	82	.111
4.75	1.71	.5	.66	.5	.31	.5	.107
5.	1.68	31	.66	57	.31	83	.104
.5	1.63	.5	.65	.5	.307	.5	.101
6	1.58	32	.64	58	.305	84	.098
.5	1.53	.5	.63	.5	.300	.5	.094
7	1.49	33	.62	59	.295	85	.091
.5	1.45	.5	.61	.5	.289	.5	.087
8	1.42	34	.60	60	.286	86	.084
.5	1.38	.5	.59	.5	.281	.5	.081
9	1.35	35	.59	61	.277	87	.078
.5	1.32	.5	.58	.5	.272	.5	.075
10	1.29	36	.57	62	.268	88	.072
.5	1.26	.5	.56	.5	.263	.5	.068
11	1.24	37	.56	63	.259	89	.065
.5	1.21	.5	.55	.5	.254	.5	.062
12	1.19	38	.54	64	.250	90	.059
.5	1.16	.5	.53	.5	.245	.5	.056
13	1.14	39	.53	65	.241	91	.053
.5	1.12	.5	.52	.5	.237	.5	.050
14	1.10	40	.51	66	.233	92	.047
.5	1.08	.5	.50	.5	.228	.5	.044
15	1.06	41	.50	67	.224	93	.041
.5	1.04	.5	.49	.5	.220	.5	.038
16	1.03	42	.49	68	.216	94	.035
.5	1.01	.5	.48	.5	.212	.5	.032
17	.99	43	.47	69	.208	95	.029
.5	.97	.5	.46	.5	.204	.5	.026
18	.96	44	.46	70	.200	96	.023
.5	.94	.5	.45	.5	.196	.5	.020
19	.93	45	.45	71	.192	97	.017
.5	.91	.5	.44	.5	.188	.5	.014
20	.90	46	.43	72	.184	98	.011
.5	.88	.5	.42	.5	.180	.5	.008
21	.87	47	.42	73	.176	99	.006
						.5	.003

WAVE LENGTH 550 mµ $PC_{550} = 69.7D_{550} = 69.7\ (-\log T_{550})$							
T	P. C.	T	P. C.	T	P.C.	T	P.C.
60.0	15.46	70.0	10.80	80.0	6.75	90.0	3.19
.2	15.36	.2	10.71	.2	6.68	.2	3.12
.4	15.26	.4	10.62	.4	6.60	.4	3.05
.6	15.16	.6	10.54	.6	6.53	.6	2.99
.8	15.06	.8	10.45	.8	6.45	.8	2.92
61.0	14.96	71.0	10.36	81.0	6.38	91.0	2.86
.2	14.86	.2	10.28	.2	6.31	.2	2.79
.4	14.76	.4	10.20	.4	6.23	.4	2.72
.6	14.67	.6	10.11	.6	6.16	.6	2.65
.8	14.57	.8	10.03	.8	6.08	.8	2.58
62.0	14.47	72.0	9.95	82.0	6.01	92.0	2.52
.2	14.37	.2	9.87	.2	5.94	.2	2.45
.4	14.28	.4	9.78	.4	5.86	.4	2.38
.6	14.18	.6	9.70	.6	5.79	.6	2.32
.8	14.09	.8	9.61	.8	5.71	.8	2.25
63.0	13.99	73.0	9.53	83.0	5.64	93.0	2.19
.2	13.89	.2	9.45	.2	5.57	.2	2.13
.4	13.80	.4	9.37	.4	5.50	.4	2.07
.6	13.70	.6	9.28	.6	5.42	.6	2.00
.8	13.61	.8	9.20	.8	5.35	.8	1.94
64.0	13.51	74.0	9.12	84.0	5.28	94.0	1.87
.2	13.42	.2	9.04	.2	5.21	.2	1.81
.4	13.32	.4	8.96	.4	5.14	.4	1.75
.6	13.23	.6	8.87	.6	5.06	.6	1.68
.8	13.14	.8	8.79	.8	4.99	.8	1.62
65.0	13.04	75.0	8.71	85.0	4.92	95.0	1.55
.2	12.95	.2	8.63	.2	4.85	.2	1.49
.4	12.86	.4	8.55	.4	4.78	.4	1.43
.6	12.76	.6	8.47	.6	4.70	.6	1.36
.8	12.67	.8	8.39	.8	4.63	.8	1.30
66.0	12.58	76.0	8.31	86.0	4.56	96.0	1.23
.2	12.49	.2	8.23	.2	4.49	.2	1.17
.4	12.40	.4	8.15	.4	4.42	.4	1.11
.6	12.31	.6	8.07	.6	4.35	.6	1.04
.8	12.22	.8	7.99	.8	4.28	.8	.98
67.0	12.12	77.0	7.91	87.0	4.22	97.0	.92
.2	12.03	.2	7.83	.2	4.15	.2	.86
.4	11.94	.4	7.75	.4	4.08	.4	.80
.6	11.85	.6	7.68	.6	4.01	.6	.74
.8	11.76	.8	7.60	.8	3.94	.8	.67
68.0	11.67	78.0	7.52	88.0	3.87	98.0	.61
.2	11.58	.2	7.44	.2	3.80	.2	.56
.4	11.50	.4	7.37	.4	3.73	.4	.49
.6	11.42	.6	7.29	.6	3.66	.6	.43
.8	11.33	.8	7.22	.8	3.59	.8	.37
69.0	11.24	79.0	7.14	89.0	3.53	99.0	.31
.2	11.15	.2	7.06	.2	3.46	.2	.25
.4	11.06	.4	6.98	.4	3.39	.4	.19
.6	10.98	.6	6.91	.6	3.32	.6	.13
.8	10.89	.8	6.83	.8	3.25	.8	.06

WAVE LENGTH 620 mµ $PC_{620}=41.2(-\log T_{620})$			
T	P.C.	T	P.C.
80.0	3.99	90.0	1.89
.2	3.94	.2	1.85
.4	3.90	.4	1.81
.6	3.85	.6	1.77
.8	3.81	.8	1.73
81.0	3.77	91.0	1.69
.2	3.72	.2	1.65
.4	3.68	.4	1.61
.6	3.63	.6	1.57
.8	3.59	.8	1.53
82.0	3.55	92.0	1.49
.2	3.50	.2	1.45
.4	3.46	.4	1.42
.6	3.42	.6	1.38
.8	3.37	.8	1.34
83.0	3.33	93.0	1.30
.2	3.29	.2	1.26
.4	3.24	.4	1.23
.6	3.20	.6	1.19
.8	3.16	.8	1.15
84.0	3.12	94.0	1.11
.2	3.08	.2	1.07
.4	3.03	.4	1.04
.6	2.99	.6	1.00
.8	2.95	.8	.96
85.0	2.91	95.0	.92
..2	2.87	.2	.88
.4	2.82	.4	.85
.6	2.78	.6	.81
.8	2.74	.8	.77
86.0	2.70	96.0	.73
.2	2.66	.2	.69
.4	2.61	.4	.66
.6	2.57	.6	.62
.8	2.53	.8	.58
87.0	2.49	97.0	.54
.2	2.45	.2	.51
.4	2.41	.4	.47
.6	2.37	.6	.43
.8	2.33	.8	.40
88.0	2.29	98.0	..36
.2	2.24	.2	.33
.4	2.20	.4	.29
.6	2.16	.6	.26
.8	2.12	.8	.22
89.0	2.08	99.0	.18
.2	2.05	.2	.15
.4	2.01	.4	.11
.6	1.97	.6	.07
.8	1.93	.8	.03

WAVE LENGTH 670 mµ $PC_{670}=56.4(-\log T_{670})$			
T	P.C.	T	P.C.
80.0	5.47	90.0	2.58
.2	5.41	.2	2.52
.4	5.35	.4	2.47
.6	5.29	.6	2.42
.8	5.22	.8	2.36
81.0	5.16	91.0	2.31
.2	5.10	.2	2.26
.4	5.04	.4	2.21
.6	4.98	.6	2.15
.8	4.92	.8	2.10
82.0	4.86	92.0	2.04
.2	4.80	.2	1.98
.4	4.74	.4	1.93
.6	4.68	.6	1.88
.8	4.62	.8	1.83
83.0	4.56	93.0	1.78
.2	4.50	.2	1.72
.4	4.44	.4	1.67
.6	4.38	.6	1.62
.8	4.32	.8	1.57
84.0	4.27	94.0	1.52
.2	4.21	.2	1.46
.4	4.15	.4	1.41
.6	4.09	.6	1.36
.8	4.03	.8	1.31
85.0	3.98	95.0	1.26
.2	3.92	.2	1.20
.4	3.86	.4	1.15
.6	3.80	.6	1.10
.8	3.74	.8	1.05
86.0	3.69	96.0	1.00
.2	3.63	.2	.95
.4	3.58	.4	.90
.6	3.52	.6	.85
.8	3.47	.8	.80
87.0	3.41	97.0	.75
.2	3.35	.2	.70
.4	3.30	.4	.65
.6	3.24	.6	.60
.8	3.18	.8	.55
88.0	3.13	98.0	.50
.2	3.07	.2	.45
.4	3.02	.4	.40
.6	2.96	.6	.35
.8	2.91	.8	.30
89.0	2.85	99.0	.25
.2	2.80	.2	.20
.4	2.74	.4	.15
.6	2.69	.6	.10
.8	2.63	.8	.05

one wavelength at a time. In the absence of four scales either permanently mounted on the instrument or quickly interchangeable without the necessity of resetting the zero adjustment, two means occurred to us of speeding and simplifying the calculation of the A.O.C.S. photometric color values.

The first is a special slide rule. This would embody the four A.O.C.S. color component scales on a single strip flanked on either side by percentage transmittance scales in the correct special relationship to the others. (Two scales, in order to avoid any error due to parallax in positioning the index line.) The use of this device is obvious. The spectrophotometer readings would be taken and recorded in terms of percentage transmittance. Then the various A.O.C.S. photometric color components would be read from the appropriate scales by setting the index line engraved on a glass slide, or cursor, fastened to the device as on a slide rule, setting the index line over the percentage transmittance scale at the proper value, then reading off the corresponding A.O.C.S. color component value for that wavelength from the appropriate scale. It may be noted that parallax error could be avoided altogether by having a percentage transmittance scale adjacent to each color component scale. It would thus be unnecessary to check each time to be sure that the index line showed the same reading on both percentage transmittance scales outside of the four color component scales.

Since the construction of such a device involved equipment not readily available to us as well as a type of skill and qualifications to which we do not lay claim and because it seemed unlikely that one could be made up to our order without considerable correspondence, delay, and expense, we provided the alternative means of simplifying the calculation, that is, by making up tables of A.O.C.S. color components in terms of percentage transmittance, for readings at the four different wavelengths. Since these tables have proved useful and time-saving in our own laboratory and since several acquaintances to whom they have been given have also commented favorably on them, it was thought that other laboratories might find these useful, especially in the first months of operation under the new photometric color method, and they are accordingly given herewith.

The color components were calculated from logarithm tables for each full percentage transmittance point in the ranges covered. Intermediate values were then interpolated, assuming a linear function, with the result that some may vary from the exact color component values by 0.01 A.O.C.S. color unit. Since the uncertainty in each of the four color component terms averages about .06 color units, corresponding to an actual uncertainty of 0.2% in the transmittance readings, it is apparent that the discrepancy of .01 color unit is without practical significance. Moreover when we realize that the standard deviation of A.O.C.S. photometric color readings amounts at this time to around 0.2 to 0.3, it did not seem worth the extra time and effort involved to calculate all the fractional values in the table to the third decimal place just to eliminate a discrepancy of .01 in some of them. It may be mentioned that we deliberately arranged the data so that all four tables come within the compass of standard 8½ x 11 in. office paper so that for protection against hard usage it can be slipped into a celluloid envelope designed for that size sheet. Indeed, by a slight photo-reduction the typed tables all go on one side of that size sheet.

A word might be added concerning our preference for reading the spectrophotometer in terms of percentage transmittance. In the first place, it seems more natural to read a horizontal scale from left to right, as in the case of the slide rule. Another advantage is in the fact that the transmittance scale is of uniform scale division size. Estimation to the nearest tenth is the same on all parts of the scale. Other users have expressed the same preference for reading in terms of transmittance.

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The Storage of Cottonseed. IX. Behavior of Cottonseed During Storage Under Mill Conditions[1]

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IN a series of experiments, carried out with the cooperation of industry during the years 1944 through 1950, an investigation has been made on the behavior of cottonseed of various moisture contents stored at commercial mills. Previous reports of observations on the heating of cottonseed and the chemical changes that take place in this commodity during storage have been made by Malowan (1, 2, 3) on relatively small quantities of seed and by Altschul *et al.* (4, 5, 6, 7) in connection with the chemical treatment of cottonseed to prevent deterioration. No detailed account has been found in the literature of the damage that occurs in piles of cottonseed (up to 40 tons) during storage. It is the purpose of this paper therefore to present the changes that occur as a result of storage: such as heating, the formation of free fatty acids, variations in chemical composition, and the increases in the refining loss and color of the refined oil. The experiments reported here have been made on 20- to 40-ton lots of seed drawn from the daily receipts of the various mills that cooperated in this investigation.

Experimental Procedure at the Mill

The following procedure was adhered to in each of the mills where special bins were erected to house the seed. The bins were built of wood and lined with building paper (15-pound asphalt impregnated felt). Each bin had a built-in false bottom approximately one foot from the floor which permitted aeration of the seed pile on installation of a suitable suction fan. Generally aeration was applied whenever the temperature of the seed exceeded 90° to 100°F. The temperature at various levels within a single bin was measured by thermocouples which were connected to a potentiometer *via* a multiple point handswitch. A record of the daily temperatures registered by each thermocouple inside the bin was forwarded to the Southern Regional Research Laboratory along with a record of the atmospheric temperature.

In a majority of the experiments, samples of seed were withdrawn with a screw auger from several spots in the bin at weekly intervals. The small samples were combined and thoroughly mixed before one-half of the composite was forwarded to the laboratory for analyses for moisture and free fatty acids contents.

Uniformity in the moisture content of the seed was desirable and was attained by selection of seed of one moisture content from the daily receipts. As a result, the experimental lots of seed were not necessarily similar to the regular daily collections of any one mill at any given time.

Two methods of collecting the experimental seed were followed. In one, receipts were set aside in a large pile in an empty storage house until a volume of approximately 60 to 75 tons had been collected. In the other, the stream of seed being unloaded from the trucks was diverted to the storage bin. In most cases this stream of seed was divided in two, and one portion fell into the storage bin while the other was directed to an empty spot in the storage house to be processed as soon as possible after the beginning of the experiment.

In those experiments where a large bulk of seed was collected first, one-half of this seed pile was

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[1] Presented at the 41st Annual Meeting of the American Oil Chemists' Society, Atlanta, Ga., May 1-3, 1950.

[2] Fellow of N.C.P.A. for the period of January 1, 1948 to April 16, 1948.

[3] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

[4] This work was conducted with these companies under an informal memorandum of understanding with the Bureau of Agricultural and Industrial Chemistry.