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Trace Metals in Cottonseed Kernels[1]

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THE trace metals in cottonseed kernels have been determined spectrochemically as part of an investigation of the influence of environment and variety on the chemical composition. The results are of interest as the quantitative determination of the amounts present has received very little attention in the literature. McHargue (2) has reported the analysis of a single sample of kernels for copper, iron, manganese, and zinc by chemical methods. No spectrochemical examination of cottonseed kernels appears to have been attempted.

Twenty samples were analyzed, including those of eight varieties grown at Stoneville, Mississippi, and of one variety grown at 13 locations in the cotton-growing region of the country. They were obtained from experimental plots grown by the Bureau of Plant Industry, Soils, and Agricultural Engineering during the 1948 crop season. The varieties and locations are given in Table I.

[1] Report of a study made under the Research and Marketing Act of 1946.

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The Spectrochemical Method

The kernels were carefully removed from the hydrochloric acid fumed samples by hand, oven-dried overnight, and ground while warm in a mullite mortar. Throughout the separation of the kernels, the ashing of the samples, and the preparation of the spectrograms, elaborate care was taken to avoid contamination with metals. Ashing was accomplished by use of the magnesium nitrate method, following, in general, the procedure described earlier for the preparation of vegetable oils for spectrochemical analysis, with particular emphasis on the precautions described therein to avoid metallic contamination (3).

The spectrochemical technique made use of the modification of the line-width method of Coheur (1), described in an earlier paper (4). The magnesium oxide, resulting from ashing the sample with magnesium nitrate, is a suitable spectroscopic buffer and provides a major component line, Mg I 2942.11 A, for all analyses. Spectrograms were obtained on Eastman S. A. No. 1 plates[3] by use of a Littrow spectro-

[3] The mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

TABLE I

Ash Content and Spectrochemical Analysis of Cottonseed Kernels for Copper, Iron, and Manganese on Moisture-Free Basis

Variety and location	Ash	Copper			Iron			Manganese		
		Avg.	No. of detns.	Avg. deviation from mean	Avg.	No. of detns.	Avg. deviation from mean	Avg.	No. of detns.	Avg. deviation from mean
Stoneville, Miss.:	%	%		%	%		%	%		%
Acala 4-42	5.66	0.0017	3	13.0	0.0081	6	20.4	0.0013	6	9.2
Acala 1517	5.50	0.0016	3	12.5	0.0057	6	14.0	0.0013	3	8.0
Rowden 41B	5.47	0.0016	2	16.0	0.0073	4	10.2	0.0015	6	10.0
Mebane (Watson)	5.51	0.0017	2	6.1	0.0089	6	18.0	0.0015	6	11.3
Stoneville 2B	5.35	0.0014	4	8.8	0.0072	4	9.9	0.0010	5	11.0
Deltapine 15	5.35	0.0018	3	16.9	0.0073	3	13.7	0.0013	2	8.0
Coker 100 Wilt	5.43	0.0011	4	7.8	0.0057	8	22.8	0.0010	6	15.0
Wilds	5.57	0.0021	2	19.0	0.0072	2	9.7	0.0013	2	8.0
Average	5.48	0.0016			0.0072			0.0013		
Stoneville 2B:										
Statesville, N. C.	3.98	0.0016	4	17.2	0.0047	5	16.6	0.0012	5	13.3
Florence, S. C.	5.26	0.0012	4	12.5	0.0051	8	27.5	0.0011	5	12.7
Tifton, Ga.	4.79	0.0011	4	13.2	0.0053	7	11.1	0.0013	6	19.2
Auburn, Ala.	4.85	0.0010	4	2.8	0.0056	6	8.6	0.0014	4	14.3
Jackson, Tenn.	4.57	0.0015	5	8.0	0.0057	8	16.0	0.0014	8	12.9
Stoneville, Miss.	5.35	0.0014	4	8.8	0.0072	4	9.9	0.0010	5	11.0
St. Joseph, La.	5.31	0.0011	4	8.5	0.0073	4	6.0	0.0009	4	7.1
Chickasha, Okla.	4.81	0.0015	4	11.7	0.0063	6	15.3	0.0012	6	14.1
Greenville, Tex.	4.85	0.0017	2	0.0	0.0060	5	16.0	0.0012	4	25.0
College Station, Tex.	4.77	0.0017	2	18.0	0.0052	2	6.7	0.0013	2	3.9
State College, N. M.	4.32	0.0016	4	10.9	0.0057	7	17.0	0.0014	4	5.4
Sacaton, Ariz.	4.53	0.0016	3	14.2	0.0072	4	8.8	0.0012	4	18.8
Shafter, Calif.	5.24	0.0010	3	13.7	0.0057	7	28.8	0.0012	6	2.8
Average	4.82	0.0014			0.0059			0.0012		
All Samples:										
Highest	5.66	0.0021			0.0089			0.0015		
Lowest	3.98	0.0010			0.0047			0.0009		
Average	5.15	0.0015			0.0066			0.0013		

graph set to cover the region 2500-3500 A. A D.-C. arc, of 232 volts and 20 amperes, and a rotating sector adjusted to exclude all but 18.8% of the light from the excited samples were used.

The widths of the Mg I 2942.11 A line, at the maximum densities of the selected lines of the metals to be quantitatively determined, were measured from profiles obtained with an automatic recording microphotometer (4). The lines used for the quantitative analyses were Cu I 3273.96 A, Fe I 3020.64 A, and Mn II 2576.10 A. The only likely interference is with iron, from the Cr I 3020.67 A line, but none of the samples were found to contain chromium.

The qualitative determinations of all elements except boron and zinc were made from the same spectrograms used for the quantitative determinations. Qualitative determinations for boron and zinc were made by setting the spectrograph to photograph the region 2100-2500 A and using Eastman 103 A-O plates.

Results

The concentrations of copper, iron, and manganese, determined by the quantitative spectrochemical analysis of each of the 20 samples of cottonseed kernels, are given in Table I. The overall precision is, with but a few exceptions, within ±20% of the average amount present. Results of recovery tests (Table II), wherein known amounts of each of the elements were added to previously analyzed samples and the samples reanalyzed, showed a deviation from the mean of somewhat less than twice that of the unknowns themselves. These and other tests made when the method was first modified (4) indicate that the accuracy of the procedure is about the same as the precision, within ±20% of the amount present.

TABLE II

Recovery Tests on Cottonseed Kernels

Element	Originally present	Total amount present[1]	Found	Deviation
	%	%	%	%
Cu	0.0016	0.0020	0.00176	−12.0
Cu	0.0011	0.0015	0.00188	+25.3
Cu	0.0017	0.0021	0.00223	+ 6.2
Cu	0.0010	0.0014	0.00155	+10.7
Fe	0.0072	0.0088	0.0101	+14.8
Fe	0.0073	0.0089	0.00975	+ 9.6
Fe	0.0052	0.0068	0.00872	+29.3
Fe	0.0056	0.0072	0.00755	+ 4.9
Mn	0.0012	0.0016	0.00153	− 4.4
Mn	0.0009	0.0013	0.00129	− 7.7
Mn	0.0013	0.0017	0.00126	−25.9
Mn	0.0014	0.0018	0.00132	−26.7

[1] In the recovery tests 0.0004% Cu, 0.0016% Fe, and 0.0004%Mn were added to each sample.

The data show a variation from 0.0010 to 0.0021% for copper, from 0.0047 to 0.0089% for iron, and from 0.0009 to 0.0015% for manganese, and average values of 0.0015, 0.0066, and 0.0013% for copper, iron, and manganese, respectively. McHargue (2) reported 0.0054, 0.0150, and 0.0013%, respectively, for copper, iron, and manganese in cottonseed kernels. The smaller quantities found in this investigation for copper and iron can probably be accounted for by the precautions taken in the preparation and analysis of the samples to avoid any metallic contamination. Contamination by iron and copper is particularly difficult to avoid.

Considering the diversity of variety and environment the ranges in concentrations of copper, iron, and manganese in the kernels are rather low. No significant variations were found in kernel content of trace elements associated with variety or location of growth.

Qualitative inspection of the spectrograms showed that boron and zinc were present in trace quantities in all samples. However no lines were found in any spectrogram to indicate the presence of aluminum, chromium, nickel, or tin.

As applications of the data presented on trace metals may be useful to problems of plant physiology, the total ash content of each sample has been given in Table I. These ash contents show a variation of from 3.98 to 5.66%, with an average of 5.15%. There is no apparent effect of variety for the samples grown at Stoneville, Mississippi. The sample from Statesville, North Carolina, with 3.98% ash, is significantly lower than the average, as are, to a lesser extent, samples from Jackson, Tennessee, 4.57% ash, and from State College, New Mexico, 4.32% ash. The last three samples listed, State College, New Mexico, Sacaton, Arizona, and Shafter, California, were raised on irrigated soil and show a range of ash content from 4.32 to 5.24%. In view of the variations found in ash content of kernels from the several irrigated locations it appears that there may not be any typical differences in this respect between those grown in irrigated locations and in rain-belt locations.

Acknowledgment

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Summary

Spectrochemical analysis of cottonseed kernels of 8 varieties of cotton grown at one location and 1 variety grown at 13 locations showed no typical differences in contents of copper, iron, and manganese associated with variety and location of growth. All samples contained traces of boron and zinc. They were not found to contain traces of aluminum, chromium, nickel, and tin.

REFERENCES

1. Coheur, Pierre, J. Optical Soc. Am., *36*, 498-500 (1946).
2. McHargue, J. S., J. Am. Soc. Agron., *18*, 1076-1083 (1926).
3. O'Connor, R. T., Heinzelman, D. C., and Jefferson, M. E., J. Am. Oil Chem. Soc., *24*, 185-189 (1947).
4. O'Connor, R. T., Heinzelman, D. C., and Jefferson, M. E., J. Am. Oil Chem. Soc., *25*, 408-414 (1948).

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