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Some Effects of Nitrogen Fertilizer on the Chemical Composition of Pearl Millet Grain

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Five pearl millet [*Pennisetum americanum* (L.) Leake] hybrids grown at two fertilizer levels during crop years 1976 and 1977 at Tifton, GA, were studied to determine the effect of nitrogen fertilizer on the millet grain. The millets studied were inbred hybrids Tift 18DB, Tift 23DB, and Tift 383 and the F_1 hybrids of Tift 23DA × Tift 18DB and of Tift 23DA × Tift 383. Nitrogen fertilizer levels were 12 lb/acre and 120 lb/acre. Protein content was increased by 19 to 55% in these hybrids by the increased use of fertilizer, with only minimal effect on the quality of the protein. Protein content varied from 8.8 to 14.1% for the hybrids grown at the low level of fertilizer and from 11.6 to 20.5% for those grown at the high level. Starch content was inversely related with protein content. Crude fiber showed little variance with fertilizer level. Mineral content was variable in the samples.

Pearl millet is a major world food grain, the staple of many Africans and Asians. It has better protein quality than most other cereals and a protein content ranging from 8 to 20.9% (Burton et al., 1972; Bailey et al., 1979). Pearl millet also contains significant quantities of the minerals necessary for good nutrition in people and animals (Casey and Lorenz, 1977). Comparative studies of protein quality and mineral constituents of some Indian varieties have shown that the high-protein varieties contain plentiful amounts of essential amino acids and calcium, phosphorus, and potassium. The influence of nitrogen fertilizer levels on some Indian varieties has also been investigated with respect to protein quality (Deosthale et al., 1972) and iron and magnesium uptake (Skukla and Bhatia, 1971). In the present report, the effect of nitrogen fertilizer was observed on the protein and starch content, amino acid composition, and the mineral uptake of five dwarf pearl millet inbreds and hybrids over two crop years.

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MATERIALS AND METHODS

Grain samples of five pearl millet inbreds and hybrids grown in the 1976 and 1977 seasons were obtained from the Georgia Coastal Plain Experiment Station, Tifton, GA. The millets were inbreds Tift 18DB, Tift 23DB, and Tift 383 and the F_1 hybrids of Tift 23DA × Tift 18DB and of Tift 23DA × Tift 383. They were grown on moderately fertile loamy sand at two levels of nitrogen fertilizer (12 lb/acre and 120 lb/acre), on two replicate plots. The base fertilizer (12 lb of N/acre) was 5–10–15 applied to all the plots with the additional nitrogen applied in the form of ammonium nitrate.

Clean pearl millet grain, free of glumes and broken kernels, was evaluated as received, without being dried, dehulled, or milled. Lipid-free meal was obtained by homogenizing and extracting the whole grain with petroleum ether in a laboratory tissue homogenizer and separating the supernatant and flour by centrifugation (3000*g* for 10 min). The flour was air-dried and ground with a mortar and pestle until it passed through a 60-mesh screen. The protein content of the lipid-free whole meal was estimated (dry weight basis) by the macro-Kjeldahl procedure (N × 6.25). The starch content was determined by polarimetric method 76-20 (AACC, 1971) on the lipid-free meal. The mineral content was determined on the whole millet pressed at 20 000 lb/in. for 10–15 s in a Sonar cap

Table I. Protein, Starch, and Amino Acid Composition of Fertilized Pearl Millet, 1976[a]

	Tift 18DB		Tift 23DB		Tift 383		Tift 23DA × 18DB		Tift 23DA × 383	
	fertilizer level		fertilizer level		fertilizer level		fertilizer level		fertilizer level	
	low	high	low	high	low	high	low	high	low	high
protein, %	14.7	20.1	12.3	17.7	11.6	16.4	11.0	15.9	12.5	15.9
	13.4	20.9	11.7	17.6	14.3	16.5	9.4	15.8	14.4	16.2
starch, %	60.2	55.6	61.6	57.9	64.7	60.3	65.4	63.6	62.7	62.6
	60.2	53.4	63.5	57.3	63.5	59.9	66.8	59.9	62.8	61.4
alanine	8.6	8.5	8.6	9.0	8.1	8.7	9.9	8.9	9.1	8.9
	8.0	8.5	8.9	8.3	8.6	8.5	8.3	8.3	8.2	8.9
valine	4.3	5.2	4.9	4.7	5.1	4.6	5.9	5.5	5.0	5.3
	5.0	4.8	5.0	5.5	4.8	5.2	5.8	5.1	5.9	4.5
glycine	3.1	2.6	3.1	2.7	3.8	2.7	3.2	2.3	3.4	2.5
	2.8	2.7	3.5	3.6	3.1	3.1	3.4	2.5	3.3	3.1
isoleucine	3.5	4.3	3.7	3.9	4.1	3.7	4.6	4.3	3.8	4.2
	4.1	4.2	3.8	4.7	4.0	4.3	4.0	4.1	4.5	3.7
leucine	10.2	11.1	10.5	11.2	10.9	11.2	10.3	11.5	10.4	11.2
	10.8	11.2	10.4	11.1	10.7	11.0	10.2	11.3	10.0	11.2
proline	6.7	7.3	7.0	7.3	7.1	7.1	6.5	7.1	7.1	7.0
	6.9	7.1	6.9	6.5	7.0	7.2	6.7	7.1	6.7	7.2
threonine	3.9	3.9	4.4	3.9	4.0	4.0	3.8	3.7	3.8	4.0
	4.0	4.0	4.1	4.0	4.2	4.1	4.3	4.0	3.8	4.0
serine	5.6	5.2	5.4	5.5	5.2	5.7	5.4	5.2	5.7	5.7
	5.6	5.7	5.5	5.3	5.8	5.7	5.5	5.4	5.6	6.2
methionine	2.4	1.8	2.3	2.1	2.6	2.4	2.6	2.2	2.7	2.4
	2.1	1.9	2.4	2.2	2.3	2.4	2.7	2.3	2.4	2.1
phenylalanine	4.9	5.3	5.1	5.3	5.3	5.4	5.0	5.5	4.7	5.2
	5.4	5.4	5.4	5.2	5.4	5.5	5.0	5.6	4.8	5.2
aspartic acid	8.5	8.3	8.5	8.3	8.2	8.6	8.1	8.2	8.2	8.4
	8.5	8.5	8.4	8.0	8.5	8.3	8.5	8.4	8.1	8.6
glutamic acid	22.0	22.0	20.8	22.4	21.0	22.0	19.7	21.6	20.9	21.0
	21.3	22.4	20.7	20.9	21.3	21.4	19.8	22.6	19.8	21.1
tyrosine	3.0	2.5	2.8	3.0	2.4	2.3	2.4	2.6	2.4	2.3
	2.5	2.6	2.8	2.8	2.6	2.7	2.7	2.7	2.8	2.8
lysine	3.0	2.7	2.9	2.2	3.0	2.4	3.1	2.2	2.7	2.4
	2.8	2.4	3.1	2.6	3.1	2.7	3.3	2.6	2.8	2.3
histidine	3.4	2.5	2.6	2.6	2.5	2.3	2.6	2.6	3.2	3.0
	2.7	2.3	2.3	2.4	2.8	2.5	3.4	2.5	3.5	2.3
arginine	4.9	5.2	5.4	4.4	4.5	4.7	4.9	4.9	5.1	4.7
	5.3	4.3	4.4	5.0	4.5	4.1	6.4	4.2	5.1	5.8
cysteine/2	1.9	1.6	1.9	1.8	1.9	1.9	1.9	1.7	2.1	1.9
	1.8	1.6	2.2	1.8	1.8	1.8	2.0	1.8	2.0	2.1
recoveries, %	86	89	86	87	97	87	95	90	89	94
	85	86	83	99	88	90	94	86	92	82

[a] Protein and starch reported on dry weight basis as percent of lipid-free meal; amino acids reported as g/16 g of nitrogen recovered. Samples from two replicate plots were analyzed.

no. 330 to give a smoothly surfaced disk. Mineral assays were made with an X-ray fluorescence spectrometer (General Electric XRD-6) according to the published procedure of Piccolo et al. (1968). Amino acid analyses were made by ABC Laboratories, Inc., Columbia, MO, by gas chromatography by the procedure of Kaiser et al. (1974). Crude fiber was determined by AACC method 32-15 (1971).

RESULTS AND DISCUSSION

Protein Content. The protein content increased significantly at the high fertilizer level (Tables I and II). The increase ranged from 19 to 55% in the five cultivars (average of samples grown on two replicate plots). Tift 18DB had the highest protein content (14.1%) at the low fertilizer level and also the highest protein content (20.5%) at the high fertilizer level in 1976. The protein content of the millets differed considerably in the two crops, ranging in 1976 from 10.2 to 14.1% (dry weight basis) at the low fertilizer level and from 15.9 to 20.5% at the high fertilizer level and in 1977 from 8.8 to 11.6% at the low fertilizer level and from 11.6 to 16.5% at the high fertilizer level. This difference may be attributable to a later planting date and drier weather during the 1977 season. In spite of the poor performance of the 1977 crop, the effect of the high fertilizer level was demonstrated. Tift 18DB, which had a protein content of over a fifth less in 1977 than in 1976, exhibited increases of over 40% both seasons as a result of the high fertilizer level. Whereas the three inbred lines exhibited about the same level of increase in protein content for both seasons, the two F_1 hybrids were less influenced by level of fertilizer in the 1977 season.

Starch and Crude Fiber Content. The starch content ranged from 54.5% (dry weight basis) in Tift 18DB (20.5% protein, 1976 crop) to 66.4% in the 23DA × 18DB cross (8.8% protein, 1977 crop). On the basis of a seed count (Table III), the five millets can be divided into two groups: (a) Tift 18DB and Tift 23DB and (b) Tift 383, the F_1 hybrid of 23DA × 18DB, and the F_1 hybrid of 23DA × 383. This difference is also evident in the starch content of the two groups. Plots of the starch content vs. protein content were linear (Figure 1) with Tift 18DB and Tift 23DB averaging about 2.5% less starch than Tift 383 and the two F_1 hybrids. The data also show that the starch content of the millet is inversely related with the protein content. The regression line for Tift 18DB and Tift 23DB is defined by

$$y = -0.868x + 72.487$$

and the line for Tift 383 and the two F_1 hybrids by

$$y = -0.731x + 72.823$$

Table II. Protein, Starch, and Amino Acid Composition of Fertilized Pearl Millet, 1977[a]

	Tift 18DB		Tift 23DB		Tift 383		Tift 23DA × 18DB		Tift 23DA × 383	
	fertilizer level		fertilizer level		fertilizer level		fertilizer level		fertilizer level	
	low	high	low	high	low	high	low	high	low	high
protein, %	11.4	16.4	10.1	13.8	9.1	11.9	8.4	11.0	9.3	11.6
	11.7	16.5	10.6	15.5	10.6	14.6	9.2	12.2	ND[b]	11.8
starch, %	63.1	57.1	62.2	60.3	67.6	64.7	66.9	63.0	66.2	64.7
	61.7	58.7	64.3	59.2	65.1	61.8	65.9	60.9	ND	64.7
alanine	7.9	8.9	8.2	7.9	8.3	8.3	8.2	8.7	8.0	8.6
	8.6	8.5	7.7	8.3	8.0	8.6	8.4	8.7	ND	8.3
valine	6.5	6.0	6.2	6.2	5.5	5.8	6.3	6.2	6.0	6.4
	6.2	6.5	6.0	6.8	6.5	6.3	6.3	6.5	ND	6.2
glycine	2.6	2.5	3.6	2.7	3.1	3.5	2.0	2.9	3.3	3.0
	2.1	2.5	3.5	2.0	3.0	2.6	1.9	2.5	ND	3.1
isoleucine	5.2	5.3	4.5	5.0	4.3	4.5	4.8	4.8	4.5	5.3
	4.9	5.2	4.9	5.7	4.9	4.9	4.6	5.2	ND	4.9
leucine	11.8	12.6	10.5	11.4	10.5	10.7	10.7	11.2	10.2	12.1
	11.4	12.0	10.8	12.8	10.7	11.3	10.7	11.8	ND	11.2
proline	6.5	6.8	6.9	7.1	7.1	7.1	7.0	7.2	6.8	7.1
	6.9	6.5	6.9	6.8	6.8	7.2	7.0	6.8	ND	7.0
threonine	3.9	4.4	4.2	4.1	4.3	4.4	4.0	4.1	4.2	4.5
	4.2	4.0	4.4	4.0	4.3	4.0	4.1	4.2	ND	4.0
serine	4.6	6.4	4.7	5.2	5.6	5.3	4.6	4.9	4.5	5.2
	5.1	5.0	5.4	5.1	4.9	4.5	4.8	5.2	ND	4.8
methionine	2.3	2.0	2.4	2.3	2.6	2.8	2.5	2.6	2.4	2.2
	2.4	1.6	2.6	1.8	2.3	2.1	2.1	2.6	ND	2.3
phenylalanine	6.6	6.2	5.8	5.8	5.8	5.6	5.8	5.7	5.7	6.4
	5.9	6.8	6.7	6.8	6.1	5.9	5.5	6.0	ND	5.8
aspartic acid	9.3	9.4	8.3	8.3	8.6	8.3	8.2	8.3	11.7	8.5
	8.6	8.6	8.6	8.7	8.5	8.2	8.6	8.4	ND	8.1
glutamic acid	17.8	16.4	20.4	20.6	18.0	18.7	20.4	20.0	18.2	15.8
	18.9	19.6	17.8	18.4	16.2	21.7	19.5	17.8	ND	20.0
tyrosine	3.5	3.3	3.7	3.6	3.8	3.3	3.5	3.6	3.3	3.2
	3.0	3.8	4.3	3.8	4.0	3.4	3.6	3.3	ND	3.4
lysine	3.0	2.8	3.4	2.7	3.5	3.8	3.9	3.0	3.4	3.0
	3.4	2.7	3.6	2.7	3.6	2.9	3.7	3.1	ND	2.8
histidine	2.7	2.8	2.0	1.6	2.6	2.5	1.6	1.8	2.5	3.1
	2.2	2.0	2.2	1.8	5.0	1.7	2.6	2.6	ND	2.9
arginine	4.5	4.7	3.7	3.8	4.8	4.8	4.2	4.2	4.3	4.2
	4.6	3.5	2.8	3.2	4.6	3.4	5.1	4.1	ND	4.4
cysteine/2	1.3	1.4	1.8	1.8	1.8	1.2	1.5	1.7	1.5	1.8
	1.5	1.3	1.9	1.4	1.5	1.5	1.6	1.5	ND	1.8
recoveries, %	88	92	91	93	133	88	90	91	89	93
	96	95	94	87	92	92	91	94	ND	91

[a] Protein and starch reported on dry weight basis as percent of lipid-free meal; amino acids reported as g/16 g of nitrogen recovered. Samples from two replicate plots were analyzed. [b] ND = no data.

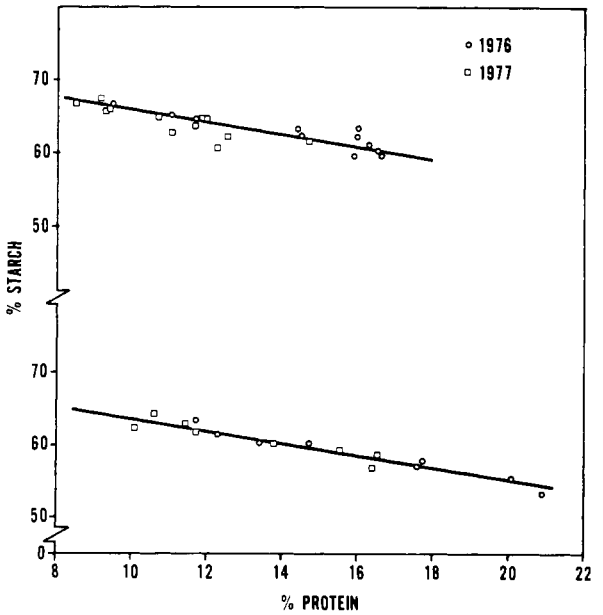

Figure 1. Starch content variation with protein content for pearl millet inbreds and hybrids. Upper line: data for Tift 383, Tift 23DA × 18DB, and Tift 23DA × 383. Lower line: data for Tift 18DB and Tift 23DB.

where x is the protein content and y is the starch content. The results also suggest an increase in protein density with increased protein content by comparing g/100 mL columns under high nitrogen and low nitrogen in Table III. There was a decrease in the number of kernels per gram and a weight increase of the kernels per 100 mL, indicative of a slight increase in kernel size as a result of enhanced use of nitrogen fertilizer. Crude fiber ranged between 1.0 and 1.4% in all of these samples and showed no substantial difference with the fertilizer level.

Amino Acid Composition. Amino acid composition of the millet protein is also presented in Tables I and II. The amount of each amino acid in the grain was substantially increased at the high fertilizer level. The amino acid concentration in the protein moiety was less markedly affected. Only leucine and glutamic acid showed any appreciable increase, in general, with an increased protein content. Lysine and methionine decreased as the protein concentration increased. However, the increased amount of protein in the millets grown at the high fertilizer level resulted in the presence of 20% or more of total lysine and methionine in most of the samples produced at the high fertilizer level.

The decrease in lysine content with increasing amounts of protein is not linear over the protein range being considered. Lysine decreases at a faster rate at the low protein

Table III. Pearl Millet Seed Count, 1977 Crop

hybrid	high nitrogen			low nitrogen		
	seeds/g	g/100 mL	seeds/100 mL	seeds/g	g/100 mL	seeds/100 mL
Tift 23DB	194	85.932	16 671	213	83.002	17 679
Tift 18DB	184	84.382	15 526	191	82.255	15 711
Tift 383	156	85.686	13 367	190	86.022	16 344
Tift 23DA × 18DB	147	87.433	12 853	172	84.882	14 600
Tift 23DA × 383	134	88.110	11 807	171	86.605	14 809

Table IV. Mineral Content (ppm) of Pearl Millet at Two Levels of Fertilizer, 1976[a]

sample description														
sample	fertilizer level	K	P	Ca	Mg	Mo	Pb	Sr	Br	Ti	Cu	Se	Zn	Fe	Mn
Tift 18DB	low	900	480	105	83	29	37	47	12	15	3.8	5.1	4.6	4.8	3.5
		839	620	95	94	28	36	48	13	22	3.6	4.3	4.8	5.1	3.0
	high	831	450	128	128	42	41	66	15	8	5.3	6.8	4.3	6.2	2.7
		879	480	119	125	41	41	63	16	33	4.2	4.5	4.9	6.5	4.4
Tift 23DB	low	985	570	99	64	32	37	49	13	8	4.2	5.8	4.5	2.9	2.4
		890	530	77	ND[b]	26	33	53	14	10	4.4	4.2	4.6	3.1	2.9
	high	1230	670	143	86	13	29	22	10	18	2.8	2.3	4.6	5.6	3.0
		1180	540	110	70	16	30	16	7	8	4.9	2.0	4.5	2.9	2.8
Tift 383	low	833	600	75	64	97	27	21	7	5	2.3	ND	4.9	3.7	3.5
		878	460	78	83	20	36	47	13	5	1.9	3.3	4.7	4.7	2.0
	high	765	580	85	81	37	42	61	17	6	6.0	5.8	5.1	6.5	4.1
		776	460	78	70	27	39	47	14	10	4.7	4.3	4.9	6.3	3.7
Tift 23DA × 18DB	low	908	460	83	77	24	36	37	11	6	2.4	3.1	4.8	3.0	2.7
		872	530	78	55	15	32	39	12	7	3.6	1.8	4.5	1.0	2.6
	high	980	470	90	53	26	36	49	13	6	4.9	3.4	4.4	3.7	2.7
		1027	450	93	77	28	37	44	13	6	4.2	3.6	4.6	3.9	3.3
Tift 23DA × 383	low	770	550	74	74	30	39	44	14	6	3.8	3.2	5.1	4.6	5.1
		805	510	81	75	19	35	27	10	ND	5.4	3.3	5.2	3.5	4.4
	high	899	390	108	79	27	36	41	12	6	3.3	3.4	4.9	5.0	4.1
		780	550	94	67	28	40	46	13	10	5.3	4.2	4.9	3.0	3.6

[a] Dry weight basis; samples from two replicate plots were analyzed. [b] ND = no data.

Table V. Mineral Content (ppm) of Pearl Millet at Two Levels of Fertilizer, 1977[a]

sample description														
sample	fertilizer level	K	P	Ca	Mg	Mo	Pb	Sr	Br	Ti	Cu	Se	Zn	Fe	Mn
Tift 18DB	low	1013	520	79	67	17	33	18	8	4	4.1	2.1	4.3	2.5	2.1
		1030	600	76	67	14	31	26	10	2	4.6	1.4	4.4	2.2	2.1
	high	1160	430	113	90	29	37	45	13	4	4.5	4.0	4.7	3.1	4.1
		879	440	82	60	12	30	25	6	5	4.5	0.9	4.7	3.6	2.9
Tift 23DB	low	1013	630	78	49	18	32	17	9	4	4.1	1.8	4.8	2.9	4.8
		1013	630	78	49	17	33	42	11	ND[b]	3.9	1.7	4.6	2.5	5.8
	high	1157	600	138	91	15	29	23	8	3	2.0	1.1	4.9	3.1	5.7
		900	570	80	ND	23	35	37	11	5	3.2	2.6	4.3	0.8	3.3
Tift 383	low	800	530	55	20	28	37	36	12	3	5.6	4.0	5.5	5.1	3.9
		747	420	55	69	69	28	13	ND	5	4.2	ND	5.0	4.9	3.3
	high	740	500	59	59	63	28	20	6	1	4.7	ND	4.7	4.1	2.5
		547	410	52	ND	15	32	22	8	3	4.2	1.5	5.2	4.9	4.2
Tift 23DA × 18DB	low	910	450	74	84	21	39	61	16	3	5.3	3.2	5.0	4.7	4.5
		938	450	88	82	28	40	53	15	5	5.3	4.2	4.6	1.1	3.1
	high	707	530	63	ND	27	37	45	11	2	4.0	3.7	5.7	4.6	3.9
		850	520	63	68	28	40	45	14	6	4.9	4.1	4.9	3.2	4.0
Tift 23DA × 383	low	850	520	63	68	30	41	52	14	9	5.2	4.6	5.1	2.9	3.4
		ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	high	792	480	75	56	20	37	51	9	ND	3.8	3.2	5.1	4.6	5.1
		770	550	74	74	23	37	30	11	3	5.4	3.3	5.2	3.5	4.4

[a] Dry weight basis; samples from two replicate plots were analyzed. [b] ND = no data.

levels, between 8 and 12%, than at the high protein levels, between 14 and 21% (see Figure 2). Regression analyses of the 1976–1977 combined data show that the lysine–protein relationship at the low protein level can be expressed by

$$y = -0.220x + 5.39$$

and at the high protein level by

$$y = -0.055x + 3.23$$

where y is the protein content and x is the amino acid content. The slope of the line for the high protein content is very close to zero, indicating practically no change in lysine with increasing protein. The methionine–protein relationship, on the other hand, can be expressed by

$$y = -0.061x + 2.91$$

over the entire protein range.

The dilution effect of the lysine concentration with increasing protein content in cereals is generally considered to be reflective of an increase in the lysine-deficient prolamin fraction in the grain. This appears not to be entirely

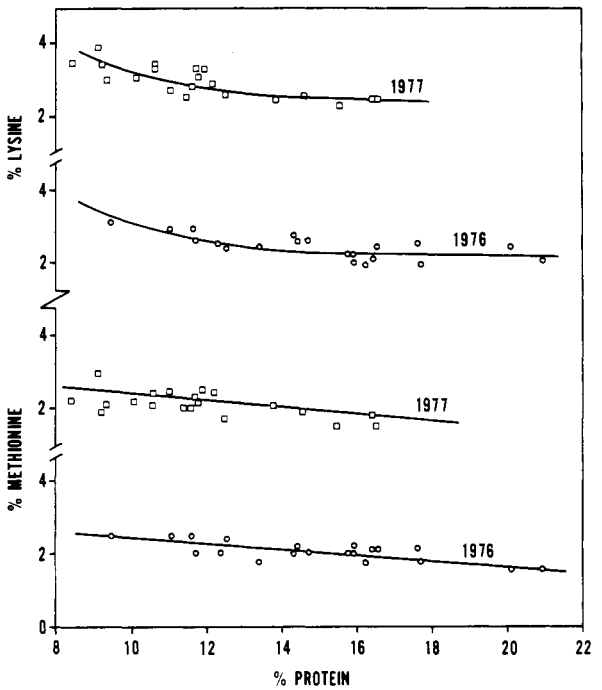

Figure 2. Lysine and methionine variation with protein content for pearl millet inbreds and hybrids.

the situation at the higher protein concentrations in pearl millet.

Mineral Content. Mineral assays are presented in Tables IV and V. The predominate mineral in all five millets was potassium, followed by phosphorus, and approximately equal amounts of calcium and magnesium. Increased nitrogen fertilizer had little effect on mineral concentrations. Concentrations of copper, iron, zinc, and manganese varied between about 2 and 6 ppm, and the amount of these minerals was little affected by fertilizer level, season, or millet type. Magnesium, reported to increase with increased fertilizer, varied between 20 and 128 ppm, a somewhat higher level than that reported by Shukla and Bhatia (1971), but showing no particular relationship to amount of fertilizer. The amount of bromine remained fairly constant at about 12 ppm, and the concentration of strontium varied between 13 and 66 ppm for all of the millets over the two seasons.

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Mass Spectral Characterization of 2,4-Disubstituted 1,3-Dioxolanes Found in Flavors

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A number of 2,4-disubstituted-1,3-dioxolanes, only a few of which are approved for food use, have been found in commercial flavors. These 2,4-disubstituted-1,3-dioxolanes can be readily characterized by mass spectroscopy. The mass spectra of the acetals of the more common flavoring aldehydes are presented here for the first time.

The presence of substituted 1,3-dioxolanes, which are frequently found in commercial flavors, can result from the condensation of flavoring aldehydes with the solvent propylene glycol. A gas chromatogram of a typical commercial flavor containing such condensation products is presented in Figure 1. Because many of these substituted dioxolanes are not permitted in food products nor characterized in the literature, a procedure using mass spectroscopy has been developed for their rapid positive identification. Their mass spectra and a procedure for structural elucidation are presented.

EXPERIMENTAL SECTION

Preparation of Substituted Dioxolanes. The parent 1,3-dioxolane was purchased from Phatz and Bauer (Stamford, CT). The remaining derivatives were synthesized according to the method of Lucas and Guthrie (1950) by reacting the aldehyde with 1,2-propylene glycol in the presence of a mineral acid.

Combined Gas Chromatography–Mass Spectroscopy. The substituted 1,3-dioxolanes from commercial flavors and from synthetic mixtures were separated and identified by using a Varian/Aerograph Series 1200 instrument fitted with a flame ionization detector and coupled to a Hitachi RMU-6L single focusing magnetic sector mass spectrometer, respectively. A small fraction of the column effluent was bypassed through a fine metering valve into a single stage glass jet separator (McFadden, 1973) leading into the ion source. Both the molecular separator and valve were maintained at 200 °C with a convection circulated air oven.

A 2 mm (i.d.) × 3 m glass column packed with 5% Carbowax 20M on 60–80 mesh acid-washed DMCS Chro-

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