NITROGEN INVESTIGATIONS

Methods and Materials: Four locations were selected in west-central Minnesota for nitrogen correlation-calibration studies in 1977. One experiment was established in Grant, Swift, Chippewa and Renville counties. The representative soils were Rolliss loam in Grant, Tara silty clay loam in Chippewa, and Winger silty clay loam in Swift and Renville counties.

The experimental design consisted of nitrogen rates of 0, 50, 100, 150 and 200 pounds per acre arranged in a 5 x 5 Latin square design. Nitrogen treatments, applied as ammonium nitrate, were broadcast and incorporated in the soil in late October. In addition, phosphorus and potassium were broadcast according to soil test recommendations.

Fifty soil cores were taken to a depth of 5 feet at each of the four locations in late October, 1976. Increments of 0-6", 6-12", 12-24", 24-36", 36-48", and 48-60" were analyzed for nitrate-nitrogen. In addition, soil analyses were made on a composite sample of the 0-6" depth for available phosphorus, exchangeable potassium and zinc.

Young-mature petioles were sampled 5 times in 2-week intervals beginning on July 19 to monitor nitrate-nitrogen in the tissue. Emission spectrographic analyses were made to ascertain nutrient levels in the plant tissue.

All plots were hand-harvested the second week of October. Representative samples from each plot were transported to North Dakota State University where the sugarbeets were processed through the tare laboratory.

Results and Discussion: In contrast to 1976[1/], improved soil moisture and early season temperatures allowed for above average sugar yields at all experimental locations. In the 1977 trials, the use of nitrogen fertilizer increased yield at the Renville and Swift county locations (Tables 2 and 3), which had medium soil nitrate levels, and decreased sugar yield on the Chippewa and Grant county locations (Tables 4 and 5), which tested very high in soil nitrates. It is of interest to note that in all cases excess use of nitrogen fertilizer increased the amino nitrogen in the sugarbeet brei. Since amino nitrogen interferes with crystallization in processing, a drastic reduction in the percent recoverable sugar is created.

Nitrate concentrations in the young-mature petioles at various times in the growing season was strongly related to the soil nitrate level and the amount of nitrogen fertilizer used (Figure 1). It is of special interest to note the nitrate concentration in the petioles at the August 30 sampling, which is approximately 6 weeks prior to harvest. At this time, the Grant and Chippewa locations, which had very high soil nitrate levels, had petiole concentrations well in excess of the desired 1,000 ppm level.

[1/] See "1976 Sugarbeet Research and Extension Reports", pp. 190-199.

Table 1. Soil nitrate-nitrogen levels at the experimental locations in the fall of 1976.

Soil Depth	Soil Nitrate-Nitrogen Levels			
	Grant Co.	Swift Co.	Chippewa Co.	Renville Co.
inches	lbs. NO_3-N/A			
0-6	123	40	38	37
6-12	20	23	38	24
12-24	46	15	164	34
24-36	50	12	40	34
36-48	27	13	13	19
48-60	19	13	12	17
Past crop	wheat	wheat	sweet corn	soybeans

Table 2. Effect of various rates of nitrogen on sugarbeet root yield, percentage sugar, gross sugar per acre, amino nitrogen and percent recoverable sugar. Renville County, Minnesota - 1977.

Nitrogen Rate	Roots	Sugar	Gross Sugar	Amino Nitrogen	Recoverable Sugar
lbs/A	T/A	%	lbs/A	ppm	%
0	26.1	16.2	8418	451	90
50	27.4	15.8	8620	538	89
100	28.3	14.9	8442	663	87
150	29.9	14.6	8716	729	85
200	29.8	14.0	8352	881	83
Significance	**	**	ns	**	
BLSD (.05)	2.1	.7	-	127	

Soil Nitrate Test (0-2') - 95 lbs/A

Table 3. Effect of various rates of nitrogen on sugarbeet root yield, percentage sugar, gross sugar per acre, amino nitrogen and percent recoverable sugar. Swift County, Minnesota - 1977.

Nitrogen Rate	Roots	Sugar	Gross Sugar	Amino Nitrogen	Recoverable Sugar
lbs/A	T/A	%	lbs/A	ppm	%
0	19.0	17.3	6578	447	91
50	21.4	16.6	7136	512	90
100	21.7	16.5	7178	491	90
150	22.8	15.8	7188	681	88
200	23.5	15.6	7316	692	87
Significance	*	**	ns	**	
BLSD (.05)	3.0	.7	-	102	

Soil Nitrate Test (0-2') - 78 lbs/A

Table 4. Effect of various rates of nitrogen on sugarbeet root yield, percentage sugar, gross sugar per acre, amino nitrogen and percent recoverable sugar. Chippewa County, Minnesota - 1977.

Nitrogen Rate	Roots	Sugar	Gross Sugar	Amino Nitrogen	Recoverable Sugar
lbs/A	T/A	%	lbs/A	ppm	%
0	21.6	15.9	8182	763	87
50	21.0	16.1	8116	799	87
100	22.3	15.4	7766	912	85
150	20.6	15.2	7584	957	84
200	21.3	14.6	6688	986	83
Significance	ns	**	**	*	
BLSD (.05)	-	.7	702	142	

Soil Nitrate Test (0-2') - 240 lbs/A

Table 5. Effect of various rates of nitrogen on sugarbeet root yield, percentage sugar, gross sugar per acre, amino nitrogen and percent recoverable sugar. Grant County, Minnesota - 1977.

Nitrogen Rate	Roots	Sugar	Gross Sugar	Amino Nitrogen	Recoverable Sugar
lbs/A	T/A	%	lbs/A	ppm	%
0	20.3	14.3	5804	945	81
50	21.8	14.1	6118	960	81
100	20.7	13.7	5756	1046	79
150	22.0	13.9	5976	1050	78
200	21.3	14.1	6146	1127	77
Significance	ns	ns	ns	ns	
BLSD (.05)	-	-	-	-	

Soil Nitrate Test (0-2') - 189 lbs/A

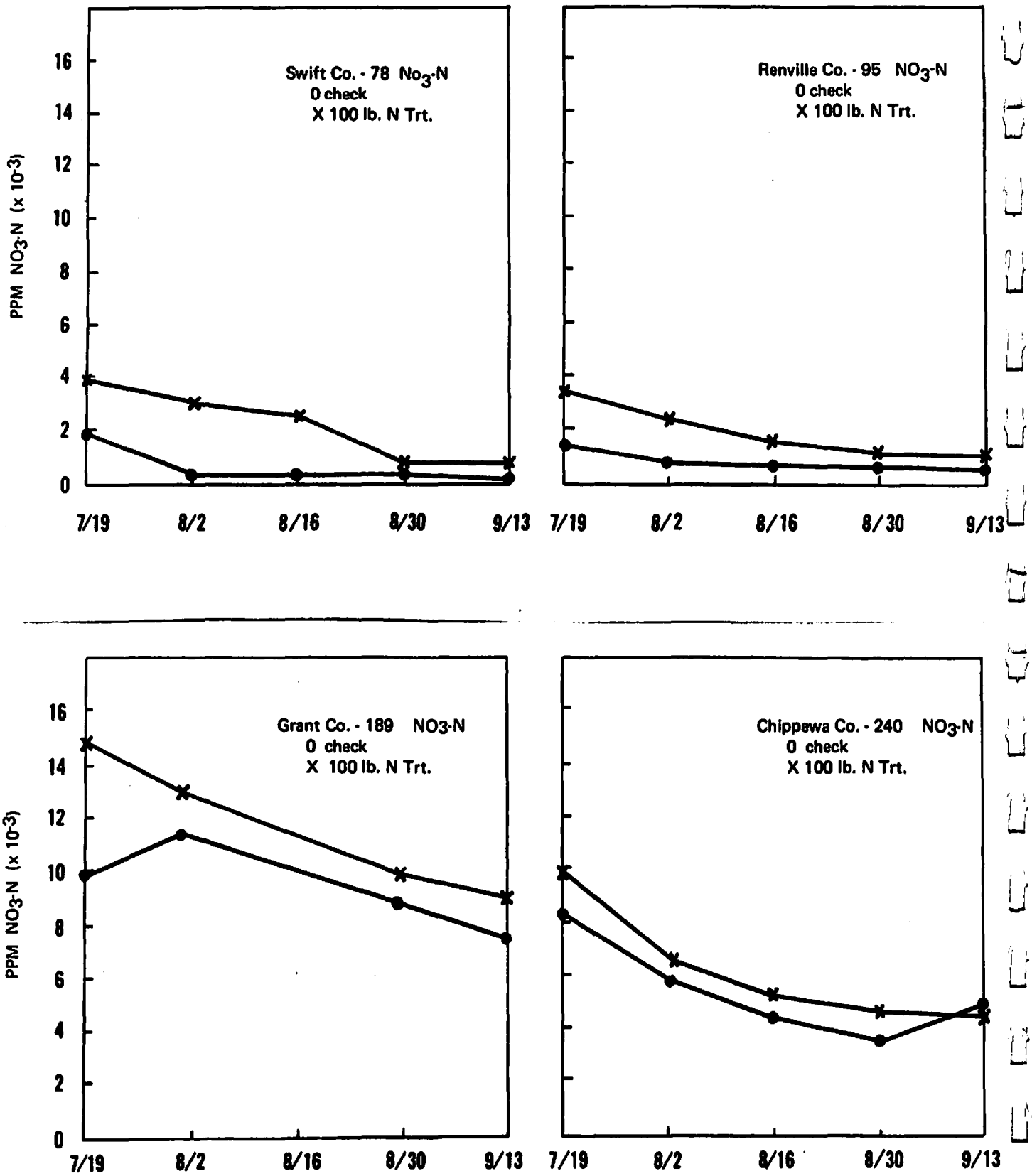

Figure 1. Effect of soil nitrates and fertilizer nitrogen on the NO_3-N concentration in sugarbeet petioles at various sampling dates and locations.

PHOSPHORUS AND POTASSIUM INVESTIGATIONS

Methods and Materials: One phosphorus and one potassium experiment was established on a Tara silty clay loam soil in Chippewa County.

Soil analyses indicated that phosphorus and potassium levels were medium and very high, respectively.

The experimental design consisted of potassium rates of 0, 100, 200, 300 and 400 pounds per acre and phosphorus rates of 0, 25, 50, 75 and 100 pounds per acre arranged in a 5 x 5 Latin square design. In addition, a starter-no starter split plot design was superimposed over the Latin square in the phosphorus experiment. Phosphorus and potassium treatments were broadcast in late October.

Young-mature petioles were sampled on August 16 to check the effect of fertilizer treatments on the concentration of phosphorus and potassium in the tissue.

All plots were hand-harvested the second week of October and representative samples from each plot were processed through the tare laboratory at North Dakota State University.

Results and Discussion: In the 1977 trials, no response to either broadcast phosphorus or potassium was noted (Tables 6 and 7). The lack of response to the addition of fertilizer potassium was predicted due to the very high soil test level, which is typical of most of the west-central Minnesota soils. However, in the case of fertilizer phosphorus, a response to 100 pounds P_2O_5 per acre was predicted based on Minnesota's present recommendations. Further calibration tests will be necessary in order to contemplate any adjustments in the present recommendation.

The percent P in the young-mature petioles was significantly influenced by broadcast phosphorus, but showed little response to the use of starter fertilizer (Table 6). In the potassium trial, the percent K in the petiole was increased over an already sufficient level in the check treatment (Table 7).

ACKNOWLEDGEMENTS

Sincere appreciation is extended to the Sugarbeet Research and Education Board and to the University of Minnesota Agricultural Extension Service for financing and supporting this research project.

Special recognition should be given to our farmer-cooperators, whose interest and aid made these experiments a success. They include Earl Davison in Grant County, Vernon Arnold in Swift County, Wayne Schwitter in Chippewa County and LeRoy Stamer in Renville County.

Special recognition should also be given to Jerome Lensing, Tim Wager, Mike O'Leary and George Nelson in the Soil Science Department, University of Minnesota, for their untiring efforts and attention to the field phase of this project.

Table 6. Effect of various rates of phosphorus on petiole phosphorus level, root yield, percentage sugar, gross sugar yield, impurity index and percent recoverable sugar. Chippewa County, Minnesota - 1977.

Phosphorus Rate	Petiole Phosphorus	Roots	Sugar	Gross Sugar	Impurity	Recoverable Sugar
lbs/A	%	T/A	%	lbs/A	Index	%
0	0.14	23.5	14.2	6670	1044	84
25	0.15	23.4	14.1	6570	1093	84
50	0.16	22.9	14.0	6416	1086	84
75	0.17	23.2	14.3	6614	1025	85
100	0.18	23.3	14.1	6580	1013	85
Significance	**	ns	ns	ns	ns	ns
BLSD (.05)	0.02	-	-	-	-	-
No Starter	0.15	23.5	14.3	6714	1029	85
Starter	0.16	23.0	14.0	6436	1076	84
Significance	ns	ns	*	ns	ns	ns
BLSD (.05)	-	-	0.29	-	-	-

Soil Test (available P) - 19 lbs/A

Table 7. Effect of various rates of potassium on petiole potassium level, root yield, percentage sugar, gross sugar yield, impurity index and percent recoverable sugar. Chippewa County, Minnesota - 1977.

Potassium Rate	Petiole Potassium	Roots	Sugar	Gross Sugar	Impurity	Recoverable Sugar
lbs/A	%	T/A	%	lbs/A	Index	%
0	2.65	25.7	14.5	7464	1143	83
100	2.88	25.1	14.7	7354	1146	83
200	2.99	25.3	14.6	7396	1162	83
300	2.96	25.1	14.7	7378	1123	83
400	3.37	22.1	15.0	6662	1109	83
Significance	+	ns	ns	ns	ns	
BLSD (.05)	-	-	-	-	-	

Soil Test (exchangeable K) - 396 lbs/A

WILD RICE FERTILIZATION RESEARCH - 1977

A PROGRESS REPORT
January 6, 1978

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Research was continued during 1977 on fertilization, nutrient requirement and water quality. Soil, water and air temperatures, and quality of paddy water were monitored during growing season to obtain information on the environment in which wild rice grows. Nitrogen rate studies were conducted on mineral soil at Grand Rapids with two varieties in first production year, and with three varieties in second production year. Three fertilization experiments were established on peat with a third year stand in Aitkin county to study N, P, K rates and to explore effectiveness of foliar fertilization. Plant samples were collected and analyzed to learn more about nutrient requirement of the plant.

A. WEATHER AND PLANT DEVELOPMENT

The growing season of 1977 was unusual in that it started quite early and higher than normal temperature advanced plant development ahead of the usual pattern. At Grand Rapids, for example, the average air temperature (see table 1) was above normal in April (+8.3^{o}), May (+11.1^{o}), June (+2.0^{o}) and July (+1.8^{o}). Then in August cool weather (at Grand Rapids average air temperature -4.9^{o} below normal) slowed down plant development and, in some locations, affected filling of grain.

Soil, water and air temperatures were measured at three locations by 3-point automatic sensing and recording thermographs (see Fig. 1, 2, 3). Temperature measurements at the Grand Rapids and Aitkin county locations were discontinued in July because of instrument malfunction.

The air and water temperatures fluctuated much more than the soil temperatures. The mineral soil at Grand Rapids also showed greater temperature fluctuation than the two organic soils. The mean water temperatures were 2 to 10°F above those of mean soil temperatures. The daily air and water temperatures fluctuated by as much as 30 or 40°F.

In 1977, the peat soil in Aitkin county warmed up earlier than in 1976. Soil temperature on May 5 of 1977, for example, was 54° compared to 42°F on the same date in 1976. This earlier soil warm-up hastened germination and plant development. Plant development, generally, was 7 to 10 days ahead of 1976 up until early grain formation when cooler temperatures slowed it down. Wild rice in Aitkin county reached maturity in mid-August (Fig. 4).

At Grand Rapids, when compared with the normal (for a period 1931-60), all months from April through July in 1977 were warmer and consequently had a greater number of Growing-Degree-Days (see Fig. 5).

Table 1. Average air temperature as measured at three U.S. weather stations.[1)]

Station Year	Month April	May	June	July	August	5 Month Average	GDD T_b=40
	average air temperature, °F						
Fosston, Polk Co.							
Normal[2)]	41.0	54.6	63.6	69.4	67.5	59.2	2955
1974	41.0	50.5	63.4	71.6	62.8	57.9	2744
1975	34.8	55.7	61.9	70.5	64.6	57.5	2852
1976	46.6	54.9	66.8	68.8	70.9	61.6	3315
1977	49.1	66.4	64.6	70.3	60.6	62.2	3446
Grand Rapids, N.C. School							
Normal	39.9	52.7	62.0	67.4	65.1	57.4	2681
1974	41.6	49.4	62.7	70.7	62.8	57.4	2670
1975	34.7	57.0	62.2	71.5	65.2	58.1	2951
1976	47.1	54.4	66.1	68.2	67.4	60.6	3166
1977	48.2	63.8	64.0	69.2	60.2	61.1	3284
Aitkin							
1974	42.9	49.8	63.1	71.1	63.3	58.0	2770
1975	39.0M	59.4M	64.4M	72.1	66.2M	60.2	3141
1976	47.5	54.8	66.8	69.3M	68.1	61.3	3267
1977	48.3M	64.4M	65.4M	70.3M	61.0	61.9	3446

1) Source: Climatological Data, Minnesota, Vol. 80, 81, 82, 83, (1974-77), U.S. Dept. of Commerce.

2) Normals for the period 1931-60.

3) M = less than 10 days record missing.

FIG. 1 GRAND RAPIDS - MEAN AIR, WATER, AND SOIL TEMPERATURES - 1977

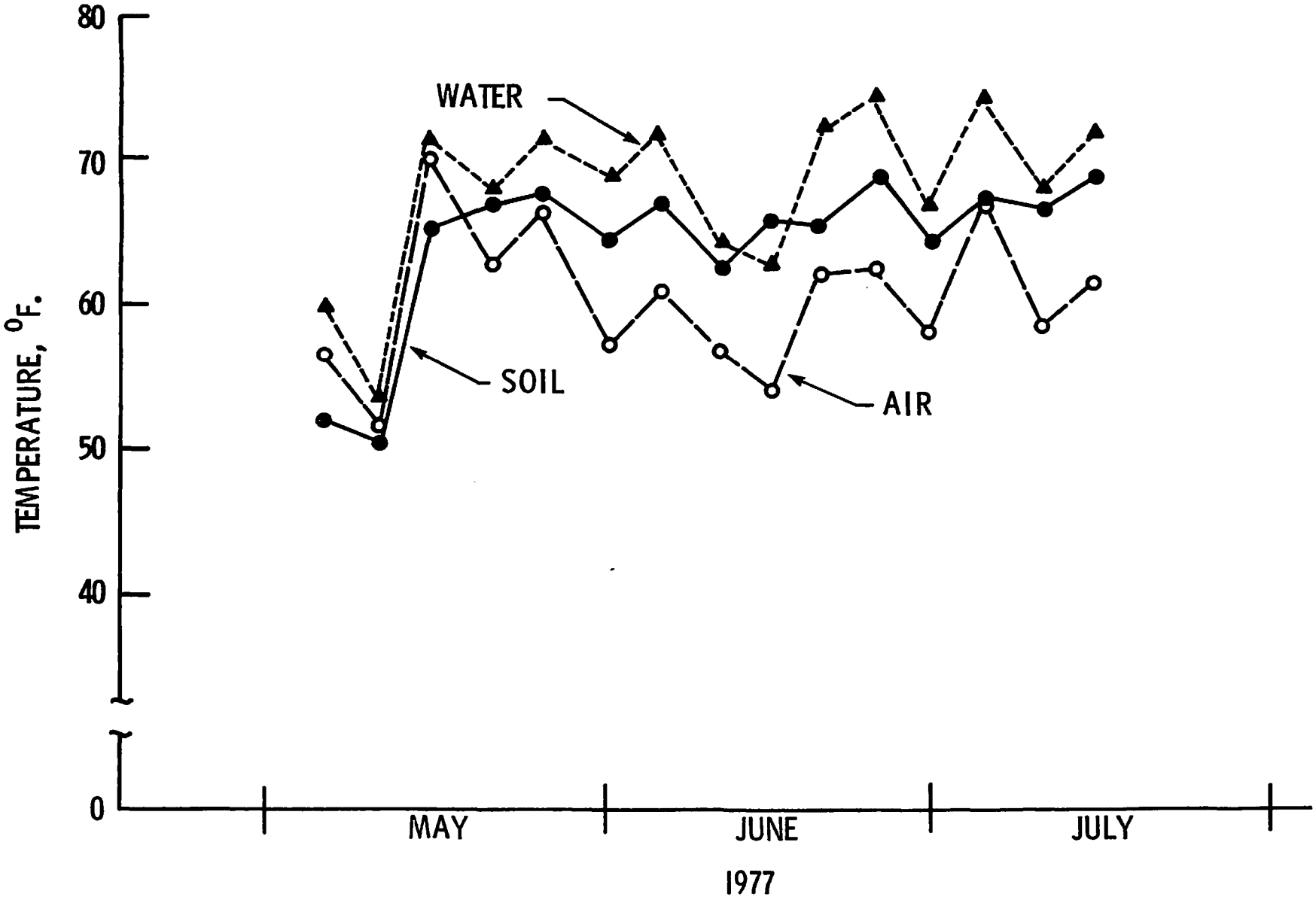


FIG. 2 KOSBAU BROS., AITKIN CO. - MEAN AIR, WATER, AND SOIL TEMPERATURES - 1977

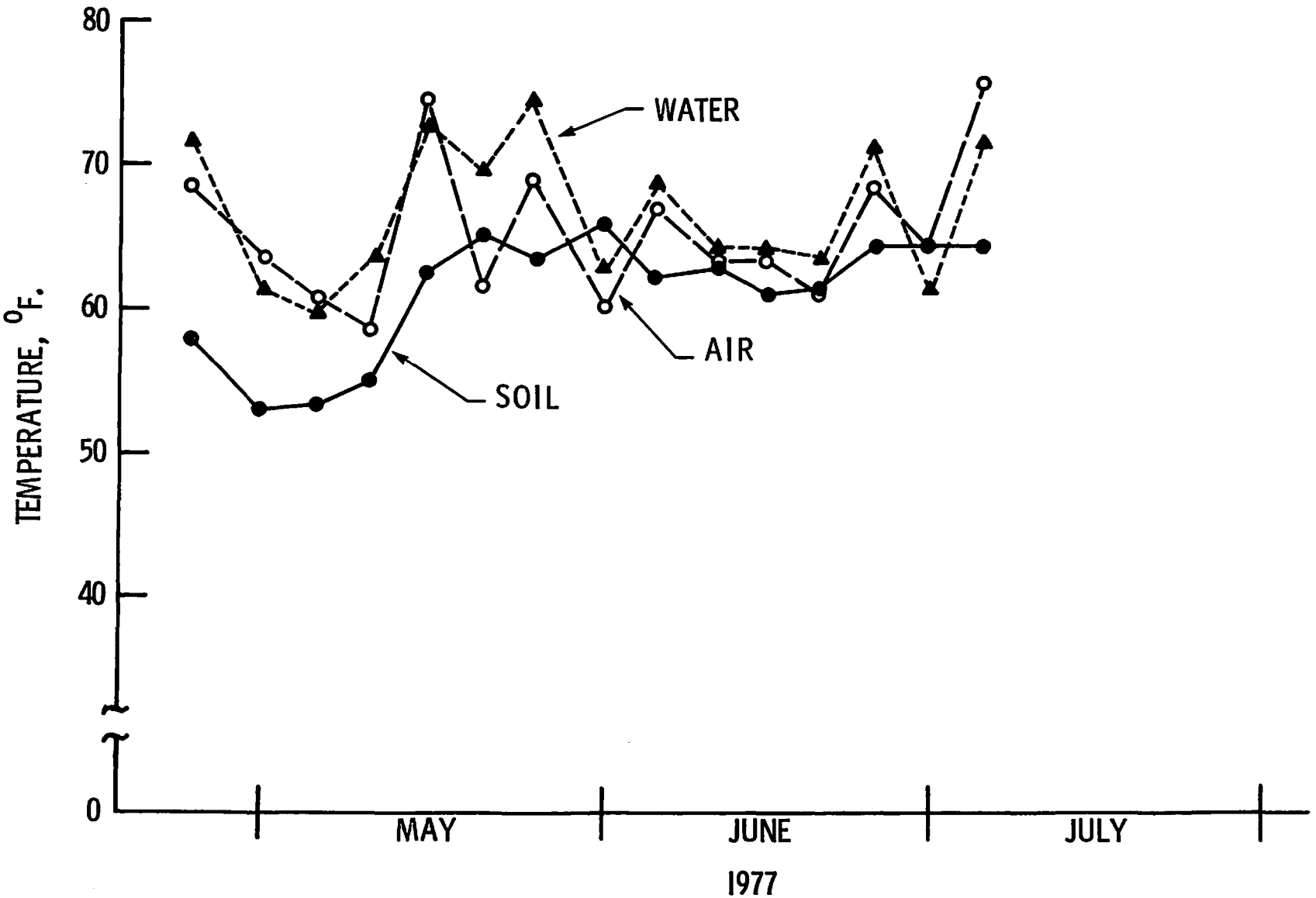


FIG. 3 IMLE AND GUNVALSON, GULLY, MINN. - MEAN AIR, WATER AND SOIL TEMPERATURES - 1977

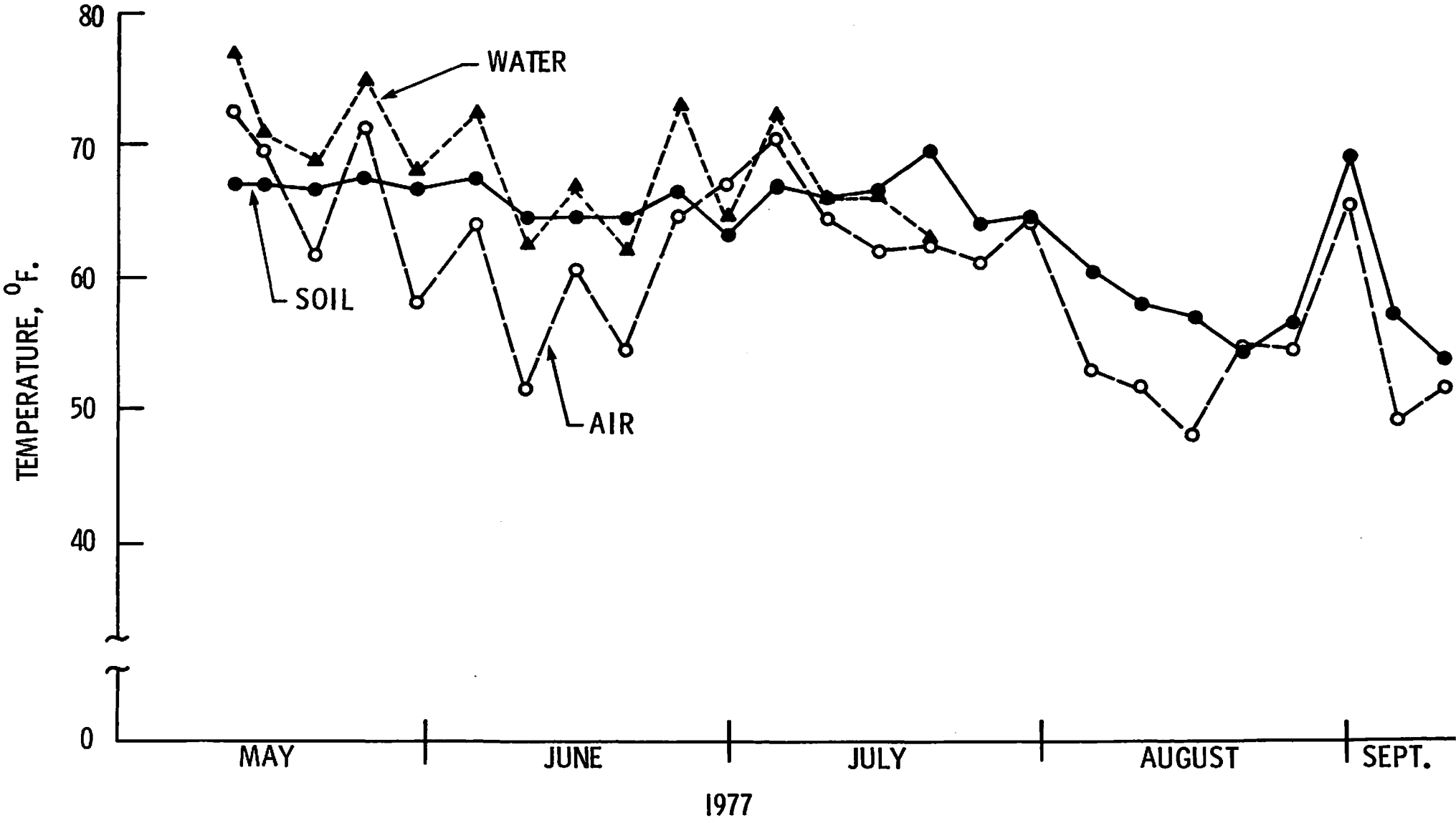
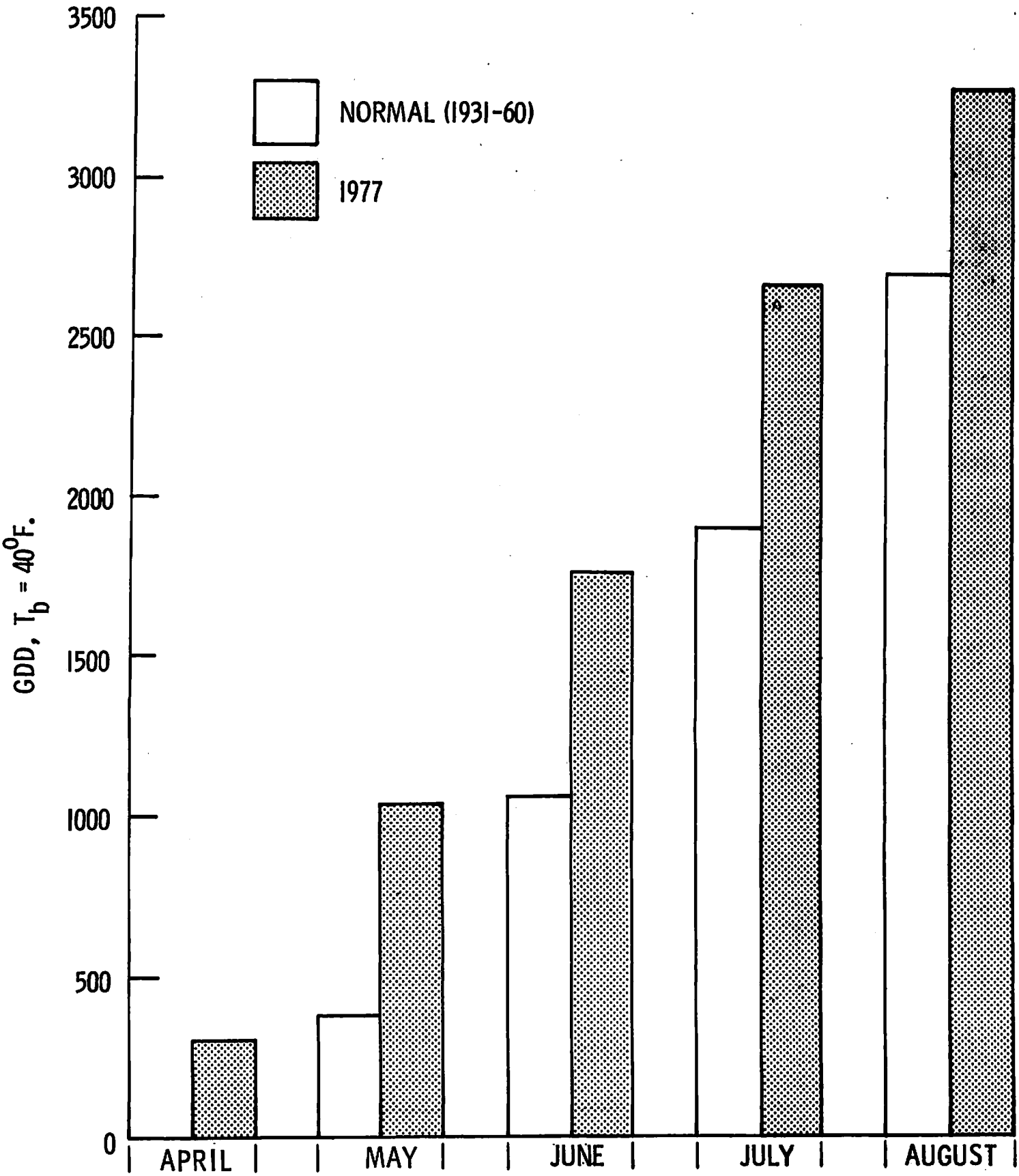


FIG. 4

WILD RICE DEVELOPMENT
K2 VARIETY, KOSBAU BROS., AITKIN CO. - 1977

	STAGES OF DEVELOPMENT	DAYS	DATE
VEGETATIVE GROWTH PHASE	0 EMERGENCE	0	APRIL
	1 FLOATING LEAF	18	MAY
	2 2ND AERIAL LEAF	31	
	3 EARLY TILLERING	42	
	4 MID TILLERING	49	JUNE
REPRODUCTIVE GROWTH PHASE	5 JOINTING	55	
	6 BOOT	62	
	7 EARLY FLOWERING	69	
	8 MID FLOWERING	79	JULY
	9 LATE FLOWERING	89	
	10 EARLY GRAIN FORMATION	99	
	11 MATURITY	118	AUGUST

FIG. 5 CUMULATIVE GROWING DEGREE DAYS, GRAND RAPIDS



B. CHEMICAL CHARACTERISTICS OF PADDY WATER

The quality of paddy water is of interest to the grower as well as to state and federal agencies. The grower may be concerned, for example, with the concentration of sulfate in the water. Plant response to fertilization may be related to nutrient levels found in the water. Public control agencies may want to know levels of nitrogen and phosphorus present in the water when it is released during draining of paddies.

Water samples were collected from several sites at three different paddy locations during 1977 growing season. At Grand Rapids, samples were collected from two experimental paddies used for N rate studies on a mineral soil. Source of water is the Prairie River. Water was also sampled at several sites within Kosbau Bros. paddies in Aitkin county. Paddies, at this location, derived water from the Little Willow River via a diversion ditch. One sampling site was located at the beginning of the diversion ditch, near the river, the other site was near a water intake pump at the far end of the diversion ditch. Four other sampling sites were located within two paddies, one a production paddy and the other paddy in which three fertilization trials were conducted. Water passed through two other production paddies before entering the research paddy. Wild rice was thinned on May 17 and it reached the jointing stage on June 14. The third sampling location was in the Gunvalson and Imle paddies near Gully. These paddies derived water from the Clearwater River.

Water samples were collected and stored in 250 ml polyethylene bottles to which a preservative had been added (2 ml of mercuric chloride solution, made up by dissolving 40 mg $HgCl_2$/L, to 250 ml of sample). Chemical analyses were made by the Research Analytical Laboratory, University of Minnesota. Information on location, sampling dates and the chemical composition of water are given in tables 2, 3, and 4.

The <u>alkalinity</u> of water is its capacity to neutralize acid. The results, in this report, indicate alkalinity obtained by titration with a combination of indicators having an endpoint of pH 5.0. In most natural waters, the alkalinity is practically all produced by dissolved carbonate and bicarbonate ions. Some organic acids also may contribute to titratable alkalinity. The values for alkalinity follow closely to those of hardness.

The <u>hardness</u> is calculated by multiplying calcium and magnesium concentrations by factors, adding up and is expressed as $CaCO_3$, mg/liter. Water from the Clearwater River is very hard (329 mg/L). The water from the Prairie River and the Little Willow River is moderately hard, with values of 118 and 101 mg/L, respectively. At Gully and at Grand Rapids, the values for alkalinity and hardness of paddy water were nearly the same as those found in the river water. In Aitkin county on peat, however, the paddy water showed 50% lower Ca and Mg contents than the river water, and a 3- to 7-fold decrease in alkalinity and hardness. Consequently, the Kosbau paddies had soft water with hardness values ranging from 28 to 46 mg/liter.

At Grand Rapids (table 2), concentrations of total N, P and K in paddy and river water were nearly the same, and remained relatively constant during the season. Total N concentration in the water ranged from 0.3 to 1.7 ppm, total P from 0.02 to 0.12 ppm, and K concentration did not exceed 2.3 ppm.

At Gully (table 4), the nutrient concentrations in the river and the paddy water were very similar. Total N and P contents were slightly higher than those measured at Grand Rapids. Potassium concentration of the water at Gully ranged from 1.6 to 8.8 ppm.

Paddy water at the Aitkin county location had considerably higher concentrations of total N, P and K than found in the river water (table 3). Total N concentration of paddy water ranged from 3.1 to 6.4 ppm compared to 0.8- 2.6 ppm found in samples collected from the diversion ditch.

Total P concentration in paddy water also was considerably higher than that of river water. Phosphorus levels were especially high at thinning time and towards the end of season. (Experimental Paddy thinned on 5/17, 7:30 AM; 3rd Production Paddy was thinned on 5/17, 8:00 PM). These increases probably resulted from agitation of water and soil during the thinning operation, and from water evaporation during the later part of growing season. Total P concentration of the water from diversion ditch ranged from less than 0.01 to 0.15 ppm while levels of 0.4 to 3.3 ppm were found in the paddy water. Potassium concentration of paddy water was nearly 7 times higher than that of river water and gradually decreased towards the end of growing season. Potassium levels of 20 ppm in the paddy water measured in mid-April, had decreased to 6- 10 ppm at the end of June.

The water in paddies at Grand Rapids and in Aitkin county did not contain appreciable amounts of sulfate. The sulfate concentration in the water at Gully, however, was relatively high, ranging from 85 to 342 ppm.

Table 2. Chemical composition of water collected from wild rice paddies during 1977 growing season. Location: Grand Rapids

Sample No.	Sampling Date	Location	Alkalinity as $CaCO_3$ mg/L	Hardness $CaCO_3$ mg/L	Total Kjeldahl N ppm	Ammonium N ppm	Nitrate & nitrite N ppm	Total P ppm	Soluble P ppm	Sulfate S ppm	Ca ppm	Mg ppm	K ppm
6.	5/3	PR	139	118	0.4	< 0.1	< 0.01	0.08	0.08	11.7*	27.7	11.9	1.5
10.	5/13	EP #1			0.3			0.06					2.3
11.	5/13	EP #4			0.6			0.07					1.5
12.	5/13	PR			0.4			0.05					1.5
30.	5/19	EP #1			0.7			0.09					1.8
31.	5/19	EP #4			1.0			0.08					1.6
38.	6/1	EP #1			.7			.03					1.2
39.	6/1	EP #4			.7			.03					1.2
40.	6/1	PR			1.0			.01					2.0
46.	6/8	EP #1			.8			.06					1.5
47.	6/8	EP #4			.3			.02					1.5
48.	6/8	PR			.2			.01					1.6
58.	6/14	EP #1			.9			.02					1.2
59.	6/14	EP #4			.5			.01					1.5
60.	6/14	PR			.4			.02					1.4
69.	6/28	EP #1	76	82	1.7	0.16	< 0.01	0.12	0.05	2	22.2	6.5	1.6
68.	6/28	EP #4	90	88	0.7	0.13	< 0.01	0.09	0.04	< 2	23.5	7.0	1.3
67.	6/28	PR	93	99	0.8	0.07	0.01	0.04	0.01	< 2	27.6	7.2	1.4
75.	7/12	EP #1			1.2			< 0.01					1.7
76.	7/12	EP #4			0.9			< 0.01					1.3
74.	7/12	PR			0.6			< 0.01					1.1

* ppm sulfate - S x 3 = ppm sulfate (SO_4)

Abbreviations and description of sampling sites at Grand Rapids

PR	Prairie River
EP #1	Experimental paddy No. 1, 2nd year stand
EP #4	Experimental paddy No. 4, 1st year stand

Table 3. Chemical composition of water collected from wild rice paddies during 1977 growing season. Location: Kosbau Bros., Aitkin County

Sample No.	Sampling Date	Location	Alkalinity as $CaCO_3$ mg/L	Hardness $CaCO_3$ mg/L	Total Kjeldahl N ppm	Ammonium N ppm	Nitrate & nitrite N ppm	Total P ppm	Soluble P ppm	Sulfate S ppm	Ca ppm	Mg ppm	K ppm
1.	4/12	DD	96	101	0.86	0.16	< 0.1	< 0.01	< 0.01	4.9*	26.6	8.3	2.5
2.	4/12	WI	97	106	1.33	0.22	< 0.1	< 0.01	< 0.01	4.9	27.8	8.9	2.4
3.	4/12	PP #3	10	28	5.66	1.08	0.4	1.72	1.10	5.4	6.6	2.8	17.9
4.	4/12	EP-D	26	44	3.87	0.75	< 0.1	0.41	0.23	1.9	11.0	4.1	18.6
5.	4/12	EP-1	27	46	4.02	0.74	< 0.1	0.42	0.19	2.6	11.5	4.2	20.3
7.	5/3	DD			1.80			0.04					2.0
8.	5/3	EP-D			3.40			0.57					14.7
9.	5/3	EP-1			4.20			0.61					14.7
16.	5/13	DD			4.60			0.04					1.3
17.	5/13	WI			4.60			0.09					1.4
18.	5/13	PP #3			5.3			2.14					15.4
19.	5/13	EP-D			3.9			0.78					14.9
20.	5/13	EP-1			4.2			0.82					17.2
21.	5/18	DD 5:30 PM			2.6			0.08					1.8
22.	5/18	WI 5:00 PM			1.7			0.15					1.4
23.	5/18	EP-1 4:00 PM			4.6			0.92					18.1
24.	5/18	EP-2 4:00 PM			4.9			0.94					18.9
25.	5/18	EP-D 4:30 PM			5.0			1.00					20.6
26.	5/18	PP #3 4:45 PM			5.0			2.91					17.5
27.	5/19	EP-1 5:00 PM			4.4			1.10					20.6
28.	5/19	EP-2 5:00 PM			4.9			1.02					20.6
29.	5/19	EP-D 5:15 PM			4.6			0.80					20.6
32.	5/24	EP-1			5.28			1.30					19.4
33.	5/24	EP-2			4.98			1.02					21.1
34.	5/24	EP-D			4.91			1.18					20.3
41.	6/1	EP-1			4.0			.92					19.3
42.	6/1	EP-D			3.9			1.26					17.2
43.	6/1	DD			2.4			.07					.5
44.	6/1	WI			1.7			.05					1.0
45.	6/1	PP #3			4.3			2.91					19.3
49.	6/8	EP-1			3.1			1.14					9.8
50.	6/8	EP-D			2.9			.95					9.2
51.	6/8	DD			1.1			.11					.4
52.	6/8	WI			1.3			.07					.3
53.	6/8	PP #3			2.6			2.00					8.4
54.	6/14	EP-1			3.4			1.32					15.2
55.	6/14	EP-2			3.5			1.41					17.2
56.	6/14	EP-D			3.3			1.15					9.2
57.	6/14	DD			0.9			0.02					0.1
61.	6/28	DD	358	112	1.5	0.16	0.03	0.01	0.02	< 2	29.3	9.5	0.1
62.	6/28	WI	114	120	1.2	0.16	< 0.01	0.08	0.03	< 2	31.7	9.9	0.2
63.	6/28	PP #3	15	32	6.4	3.65	< 0.01	2.77	2.34	< 2	7.7	3.2	6.0
64.	6/28	EP-D	23	44	4.1	0.70	< 0.01	1.55	1.19	< 2	10.3	4.5	9.2
65.	6/28	EP-1	15	41	9.3	2.80	0.01	3.30	2.32	< 2	9.7	4.1	10.5
66.	6/28	EP-2	20	38	12.5	4.32	0.01	12.10	3.56	< 2	9.4	3.6	19.3
72.	7/12	DD			1.06			<0.01					0.1
73.	7/12	EP-D			9.16			1.40					12.8

* ppm sulfate - S x 3 = ppm sulfate, SO_4, (4.9 x 3 = 14.7 ppm sulfate).

Abbreviations and description of sampling sites at Kosbau Bros.

DD	Diversion Ditch at bridge near Little Willow River
WI	Water intake near new shed
PP #3	Third production paddy from shed
EP-D	Ditch on east side of experimental paddy
EP-1	Experimental paddy, sample 1
EP-2	Experimental paddy, sample 2

Table 4. Chemical composition of water collected from wild rice paddies during 1977 growing season. Location: Gunvalson and Imle, Gully

Sample No.	Sampling Date	Location	Alkalinity as $CaCO_3$ mg/L	Hardness $CaCO_3$ mg/L	Total Kjeldahl N ppm	Ammonium N ppm	Nitrate & nitrite N ppm	Total P ppm	Soluble P ppm	Sulfate S ppm	Ca ppm	Mg ppm	K ppm
13.	5/12	PP-T	203	495	2.5	0.11	< 0.1	0.06	< 0.01	114*	118.0	48.7	8.8
14.	5/12	PP-I	235	362	1.1	0.06	< 0.1	0.03	< 0.01	39.4	87.8	34.6	5.5
15.	5/12	CR	233	329	4.6	0.13	< 0.1	0.08	0.04	29.6	80.1	31.3	5.4
36.	5/31	PP-T			2.6			0.06					7.5
37.	5/31	PP-I			2.3			0.04					7.2
35.	5/31	CR			1.1			0.09					2.9
70.	6/29	PP-T	218	560	4.0	0.28	< 0.01	0.27	0.03	80.5	138.0	52.4	5.5
71.	6/29	PP-I	216	302	1.2	0.13	< 0.01	0.06	0.01	28.2	74.0	28.2	1.6

* ppm sulfate - S x 3 = ppm sulfate (SO_4)

Abbreviations and description of sampling sites at Gully

CR	Clearwater River
PP-T	Production paddy near thermograph
PP-I	Production paddy near inlet

Note: Heavy rain on 5/30

C. NITROGEN RATE AND VARIETY STUDIES ON MINERAL SOIL

Two experiments were conducted on a mineral soil at the North Central Experiment Station, Grand Rapids. Experiment 1 (First Year Stand) was established in spring of 1977 with two early maturing varieties: Experimental 1 and K2. Experiment 2 (second year stand) has been established in spring of 1976 with K2, M3 and Johnson wild rice varieties.

FIRST YEAR STAND[1)]

The experimental paddy was in fallow during 1976 and was fumigated with methyl bromide in fall of 1976. Four rates of nitrogen were used in this experiment: 0, 20, 40 and 80 pounds per acre. Urea (46-0-0) was applied with a 3-foot Gandy spreader on April 29 and incorporated into the soil by rototilling. Phosphorus and potassium were not applied because relatively high availability of these nutrient elements was indicated by soil tests (pH 5.6, P 64, K 220). Two early maturing varieties, Experimental 1 and K2, were grown. A split-plot design was used in this experiment with the N rates as main plots and varieties as subplots. Each plot, containing one N rate and both varieties, occupied a 14 x 16 ft. area. Each N treatment was replicated 5 times. The wild rice was planted in rows one foot apart on May 3. Plant population in this experiment was 2-3 plants per square foot. Water level was maintained at about 8-12 inches. Ten plants were selected at random from each plot at jointing stage (Exp. 1 on 6/20; K2 on 6/27), and five plants at late flowering (Exp. 1 on 7/21; K2 on 7/28) for weight measurements and plant analysis. Copper sulfate (15 pounds per acre) was applied to control algae. The plants were seriously damaged by hail on June 27 when Exp. 1 was in boot with some panicles emerging, and K2 was in the jointing stage. A 4 x 10 foot square area from each plot was hand-harvested for yield determination.

The K2 variety outyielded the Exp. 1 by 410 pounds per acre (table 5). It should be pointed out, however, that Exp. 1 was more advanced in development than K2 when the hail storm occurred and may have received more serious damage. Highest yield was produced with 40 pounds per acre of nitrogen. Generally, 20 pounds per acre of N have been sufficient during first production year. In this case, however, additional N may have been required for plant recovery from hail.

At jointing, individual plants of K2 had accumulated more dry matter than those of Exp. 1 (table 6). At the late flowering stage, the K2 variety had accumulated about 4 grams more dry matter per plant than the Exp. 1. The application of nitrogen resulted in greater production of dry matter.

[1)] This experiment was conducted in cooperation with Dr. W. Anson Elliott, Department of Agronomy and Plant Genetics.

Table 5. Effect of nitrogen application on the yield of the two wild rice varieties, Grand Rapids, 1st year stand, 1977

Variety	N Rate, LB/Acre				Average (variety)
	0	20	40	80	
	Grain Yield, LB/Acre				
K2	807[1)]	920	952	887	892
Exp. 1	416	492	551	470	482
Average (Rate)	612	706	752	679	

1) 7% moisture

(a)

Variety	Grain yield, LB/Acre
K2	892
Exp. 1	482
Significance	**
BLSD (0.05)	59

(b)

N rate, LB/Acre	
0	612
20	706
40	752
80	679
Significance	+
BLSD (0.1)	98

Table 6. Effect of nitrogen application on plant weight of two varieties at jointing and late flowering, Grand Rapids, 1st year stand,1977

Development stage and variety	N Rate, LB/Acre 0	20	40	80	Average (variety)
	Dry matter, grams per plant				
(a) Jointing					
K2	4.42	4.27	4.51	3.98	4.30
Exp. 1	3.51	3.08	3.09	3.42	3.28
Average (Rate)	3.96	3.67	3.80	3.70	
Significance (variety)	**				
BLSD (.05)	0.55				
(b) Late Flowering					
K2	13.63	15.84	18.32	16.20	16.00
Exp. 1	11.25	11.36	11.58	13.38	11.89
Average (Rate)	12.44	13.60	14.95	14.74	
Significance (variety)	**				
BLSD (.05)	1.70				

Table 7. Effect of nitrogen application on N concentration in 2nd leaf at jointing, Grand Rapids, 1st year stand, 1977

Variable		N % in dry matter
(a) N rate, LB/Acre		
0		4.13[1)]
20		4.21
40		4.34
80		4.80
Significance		NS
(b) Variety		
K2		4.14
Exp. 1		4.60
Significance		*
BLSD (.05)		0.34
(c) Interaction		
N Rate x	Variety	
0	K2	3.73
0	Exp. 1	4.53
20	K2	4.03
20	Exp. 1	4.39
40	K2	4.09
40	Exp. 1	4.59
80	K2	4.71
80	Exp. 1	4.89
Significance		NS
CV%		6

1) Average of 20 plants per variety.

SECOND YEAR STAND

The nitrogen-variety experiment, established in spring of 1976, was continued in 1977. Three wild rice varieties were grown: the early maturing K2 and M3, and the late maturing Johnson wild rice. Three N rates were used: 0, 40, 80 pounds per acre. To minimize cross-pollination, the Johnson wild rice was placed between the two earlier maturing varieties and each variety was grown in a 48 x 72 foot area separated by a 10 feet wide alley from the adjoining variety. Individual plots were 12 x 14 feet in size. Each N treatment was replicated five times. Straw was disked and rototilled into the soil. Urea was applied on April 29 and rototilled into the soil. The stand was not thinned and an excessive plant population resulted for the M3 variety (12 plants per square foot). Plant population for the K2 and Johnson varieties was only 5 plants per square foot. Phosphorus and potassium were not applied because of relatively high soil test levels (pH 5.6, P 64, K 240). Water level was maintained at about 8-12 inches. Ten plants were selected from each plot at jointing, and five plants at late flowering for weight measurements and plant analysis. Copper sulfate (15 pounds per acre) was applied to control algae. Pesticides were used to control disease and rice worm. At the jointing stage, plants were seriously damaged by hail which occurred on June 27. The plants, generally, were shorter than previous years at this location, probably due to hail damage and high temperatures.

Wild rice responded well to nitrogen treatments and exhibited height and color differences, particularly the K2 and Johnson varieties. Plants in N0 plots were shorter and lighter in color than those receiving either N40 or N80 treatments. The M3 lodged quite badly because of excessive plant density. There was moderate lodging in the N80 treatment plots of the Johnson and K2 varieties.

Grain yields of all three varieties, were about 50% below those of the previous year probably due to hail damage. Johnson wild rice out yielded K2 and M3 varieties due to later harvest date which resulted in more mature grain and better filled grain. Highest yield of K2 variety was obtained with 80 pounds per acre of N (table 8). Overall, 80 pounds of N per acre increased the grain yield by 100 pounds per acre over the N0 treatment.

Nitrogen concentration in second leaf at jointing was increased in all varieties by N applications (table 9). Total N uptake by the plants of K2 and Johnson varieties, at the late flowering stage, was increased by the N80 treatment (table 10).

Table 8. Effect of nitrogen application on the yield of three wild rice varieties, Grand Rapids, 2nd year stand, 1977

N Rate LB/Acre	Variety K2	M3	Johnson	Average (Rate)
	Grain Yield, LB/Acre			
0	290[1)]	331	311	311
40	295	344	475	371
80	386	377	473	412
Average (Variety)	324	351	420	
[1)] 7% moisture significance	+	NS	NS	
BLSD (0.10)	80			
CV%	19	16	29	

Table 9. Effect of nitrogen application on N concentration in 2nd leaf at jointing, Grand Rapids, 2nd year stand, 1977

N Rate LB/Acre	Variety K2	M3	Johnson	Average (Rate)
	N% in dry matter			
0	3.60[1)]	2.77	3.28	3.22
40	4.13	3.32	4.06	3.84
80	4.51	3.66	4.30	4.16
Average (Variety)	4.08	3.25	3.88	

[1)] Average of 50 plants

Significance	**	**	**
BLSD (0.05)	0.39	0.47	0.48
CV%	7	10	9

Table 10. Effect of nitrogen application on total uptake of N by the wild rice plant at late flowering, Grand Rapids, 2nd year stand, 1977

N Rate LB/Acre	Variety K2	M3	Johnson	Average (Rate)
	— N in milligrams per plant —			
0	69[1)]	71	78	73
40	84	103	117	102
80	102	115	163	127
Average (Variety)	85	96	120	

1) Average of 25 plants

Significance	+	NS	+
BLSD (0.10)	24		59
CV%	22	39	39

D. FERTILIZATION STUDIES ON PEAT

Three fertilizer experiments, carried out in a Kosbau Bros. paddy in Aitkin county during 1976, were continued in 1977. A third year stand of K2 was used. Fertilizer was applied to 12 x 12 foot plot areas in fall of 1976 and was incorporated into the soil by rototilling. The paddy was flooded on April 12. Rice was thinned once by airboat on May 17 when most plants were in floating leaf stage. Plant population at early tillering was 4-5 plants per square foot. Copper sulfate (20 pounds per acre) was applied on June 8 to control algae. Paddy was sprayed with chemicals to control blight and rice worm. There was no lodging and no damage was caused by rice worm or blackbirds, but there were some bare spots in some plot areas. A 4 x 4 foot area was hand-harvested from each plot for yield measurement on August 16. There was a general lack of response to fertilizer treatments at this location in 1977. Perhaps this was related to the relatively high concentrations of nutrient elements found in the paddy water (table 3). Total N ranged from 3.1 to 6.4 ppm, total P from 0.4 to 3.3 ppm, and potassium concentrations as high as 20 ppm were found in the paddy water early in the season.

Table 11. Depth of peat and soil test values of experimental areas, Kosbau Bros., Aitkin County[1)]

Area	Depth of peat inches	Soil test results for 0-6 inch depth		
		pH	extractable P pp2M	exchangeable K pp2M
NP Trial	18-21-11	4.4	15-18	100-115
NK Trial	8-11	4.6	29-40	93-137
Foliar - Fertilization	7-11-18	4.6	20	100

1) Samples collected on 10/7/76.

NP RATE TRIAL

The NP experiment was located in an area within the paddy where the peat layer was thickest, ranging from 18-21 inches on the north side and gradually decreasing to 11 inches on the south side. Water depth in this portion of paddy was greatest and grain yields were higher than in the other experimental areas. Three nitrogen rates were used: 0, 40, 80 pounds per acre, with urea as the N source. Concentrated superphosphate (0-46-0) was used to supply phosphorus, applied at three rates: 0, 40, 80 pounds P_2O_5 per acre. All plots received 60 pounds K_2O per acre. Fertilizer treatments were replicated six times.

Phosphorus concentration in second leaf at jointing ranged from 0.55-0.64%, and P uptake at late flowering ranged from 63-83 mg per plant. Neither of these two chemical characteristics within the plant were significantly affected by phosphorus treatments. Little or no visual differences in plants, related to either N or P treatments, were observed during the growing season.

Grain yields ranged from 589- 790 pounds per acre (table 12). Although the N40 and N80 treatments, combined with either the P0 or the P40 treatments, tended to be related with higher grain yield, the differences were not significant.

Table 12. Effect of nitrogen and phosphorus application on grain yield of wild rice, Kosbau Bros.,1977

P Rate P_2O_5, LB/Acre	N Rate LB/Acre 0	40	80	Average (P Rate)
	Grain yield, LB/Acre			
0	589[1)]	710	790	696
40	663	640	738	680
80	696	746	666	703
Average (N Rate)	649	699	731	

1) 7% moisture

Significance NS

NK RATE TRIAL

Exchangeable potassium in the experimental area ranged from 93-137 pp2m, considered to be relatively low to medium (table 11). Depth of peat ranged from 8-11 inches. Nitrogen treatments consisted of the following rates: 0, 40, 120 pounds per acre. Potassium was applied at rates of 0, 60 and 200 pounds K_2O per acre. Urea and muriate of potash were the fertilizer materials used in this experiment. All plots received 40 pounds P_2O_5 per acre. Fertilizer was applied by hand during fall of 1976 and incorporated into the soil by rototilling.

At jointing, no visual plant responses to fertilizer treatments were evident. The N concentration in second leaf at jointing, however, was significantly higher in plants from N120 treatment than in plants from N0 treatment (table 13). At the boot stage, there were typical color and height differences observed associated with N treatments. Plants in N0 treatment plots were shorter and lighter in color than those receiving N treatments. Nitrogen uptake at late flowering ranged from 117-189 mg per plant, but the differences were not significantly different. Response of wild rice to K treatments was not detected by visual observation. Potassium concentration in second leaf at jointing was relatively high and, generally, not affected by the application of K (table 14). Potassium uptake, at late flowering, ranged from 317 to 489 mg per plant. The yield of wild rice, in this experiment, ranged from 372 to 454 pounds per acre. Neither nitrogen nor potassium treatments had any effect on the yield (table 15).

Table 13. Effect of NK application on N concentration in 2nd leaf at jointing, Kosbau Bros., 1977

K Rate K_2O, LB/Acre	N Rate, LB/Acre 0	40	120	Average (K Rate)
	N% in dry matter			
0	4.14[1)]	4.76	4.54	4.48
60	4.07	3.86	4.39	4.11
200	4.17	4.34	4.67	4.39
Average (N Rate)	4.13	4.32	4.53	

1) Average of 30 plants

Significance +

BLSD (0.10) 0.44

Table 14. Effect of NK application on K concentration in 2nd leaf at jointing, Kosbau Bros., 1977

K Rate K_2O, LB/Acre	N Rate, LB/Acre 0	40	120	Average (K Rate)
	K% in dry matter			
0	3.48[1)]	3.26	3.41	3.38
60	3.54	3.67	3.58	3.60
200	3.52	3.48	3.47	3.49
Average (N Rate)	3.51	3.47	3.49	

1) Average of 30 plants

Significance +

BLSD (0.10) 0.24

Table 15. Effect of nitrogen and potassium application on grain yield of wild rice, Kosbau Bros., 1977

K Rate K_2O, LB/Acre	N Rate, LB/Acre 0	40	120	Average
	Grain yield, LB/Acre			
0	438[1)]	438	454	443
60	398	402	381	394
200	372	448	448	423
Average	403	429	428	

1) 7% moisture

Significance NS

FOLIAR FERTILIZATION TRIAL

The foliar fertilization experiment, established in 1976, was continued in 1977. It was expanded to six replications per treatment. Soil application of fertilizer (40+40+60) was made during fall of 1976 and incorporated into the soil. The same production practices were followed as used in NK and NP trials. Fertilizer solution was sprayed on 12 x 12 foot plot areas with a backpack sprayer at a rate of 330 ml per plot (equivalent to 26.4 gallons per acre or 250 pounds per acre rate). Spraying pressure was maintained at 30 psi by using a carbon dioxide cylinder. The formulation, fertilizer materials used and costs were discussed, in detail, in the 1976 Wild Rice Research Progress Report. In 1977, foliar fertilizer applications were made on the following dates:

1st spraying, 6/21 -- at boot stage,
2nd spraying, 7/8 -- mid-flowering,
3rd spraying, 7/22 -- early grain formation.

No "leaf burn" damage was observed in this trial during 1977.

Although the total amounts of N, P and K contained by wild rice at late flowering were higher in plants receiving two or three sprayings than those with no foliar application, the differences were not significant. Wild rice yield was increased slightly by foliar fertilization (table 16). The most effective treatment consisted of soil application (40+40+60), and two foliar applications (30+12+18+2.6 S) made at mid-flowering and early grain formation.

Foliar fertilization of wild rice is not a recommended practice at the present time. With the varieties and production know-how that are available today, nutrient requirements of the plant can be satisfied effectively and more economically by conventional soil applications of NPK and N topdressing. But there is a potential that should be explored by continued investigation of this new practice. First, the positive trends in wild rice yield obtained in the 1976 and 1977 trials look promising. Secondly, growers already are applying pesticides and topdress-N by airplane or helicopter. So there is a possibility of combining these operations, thus, making foliar fertilization more economical than it is today.

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Table 16. Effect of foliar and soil application of fertilizer on grain yield of wild rice, Kosbau Bros., 1977

Foliar Application									Soil Application		
	Total Plant Nutrients Applied, LB/Acre										
Number	N		P_2O_5		K_2O		S		None	40+40+60	Average (Foliar)
									Grain yield, LB/Acre		
None			None						530[1)]	560	545
1 x	15	+	6	+	9	+	1.3		-	548	548
2 x	30	+	12	+	18	+	2.6		-	628	628
3 x	45	+	18	+	27	+	3.9		556	495	526
Average (soil)									543	558	

1) 7% moisture;

Significance NS

COMPARISON OF PROMESOL 30 AND CALCIUM CARBONATE

C. J. Overdahl

County Extension Directors and farmers frequently ask about the performance of Promesol 30 as a liming material. This is a liquid product composed primarily of trihydroxy glutaric acid with approximately 9 percent calcium and 7 percent nitrogen.

To observe what effect the product has on soil pH, an experiment was initiated comparing Promesol 30 to analytical grade calcium carbonate on two Minnesota soils. The pH of the Promesol 30 used in this trial was 8.0.

Soils were a Hubbard loamy sand from the Becker Irrigation Farm and a LeSueur silty clay loam from the Waseca Experiment Station. After treatment, soils were stored with moisture held at field capacity for six weeks in small pots. They were then air dried and the pH was determined. The results are shown in tables 1 and 2.

Results

The tables show pH values from each of four replications on two acid soils. The calcium carbonate powder changed the soil pH in direct relationship to the treatment rate. For an approximate comparison between analytical grade calcium carbonate and agricultural lime, which is coarser, the rates of calcium carbonate shown in the tables should be doubled, a half ton analytical grade equals about one ton of ag lime.

The Promesol 30 had no effect on soil pH, even at 127 gallons per acre. This was a theoretical value needed to equal one ton of lime.

These results are the same as was obtained by Dr. T. R. Peck at the University of Illinois and Dr. Roy Flannery at Rutgers University.

These latter two researchers also related the product to crop yield and some experiments measured the effect on cation exchange capacity and calcium content.

Table 1. Promesol 30 effect on soil pH compared to calcium carbonate analytical grade.

LeSueur silty clay loam from Waseca, MN.

Reps	Calcium Carbonate tons/acre							
	0	1/2	1½	3	4½	6	12	24
	soil pH							
1	5.1	5.3	5.7	6.3	6.9	6.8	7.3	7.4
2	5.2	5.5	5.8	6.3	6.7	6.9	7.0	-
3	5.2	5.4	5.7	6.3	6.2	6.9	7.1	-
4	5.2	5.4	5.6	6.3	6.6	6.9	7.1	-
	Promesol 30 gallons/acre							
	0	2½	5	7½	10	20	40	127
	soil pH							
1	5.1	5.1	5.1	5.1	5.1	5.1	5.1	5.0
2	5.2	5.0	5.1	5.1	5.0	5.1	5.1	5.0
3	5.2	5.2	5.1	5.1	5.1	5.1	5.1	5.1
4	5.2	5.1	5.1	5.2	5.2	5.2	5.2	5.2

Table 2. Promesol 30 effect on soil pH compared to calcium carbonate analytical grade.

Hubbard loamy sand from Becker Irrigation Farm.

Reps	Calcium Carbonate tons/acre							
	0	1/2	1½	3	4½	6	12	24
	soil pH							
1	5.3	5.7	6.4	7.0	7.2	6.9	7.4	7.7
2	5.4	5.8	6.5	7.0	7.2	7.5	7.5	-
3	5.4	5.8	6.5	6.9	7.2	7.4	7.5	-
4	5.5	5.9	6.3	6.9	7.2	7.4	7.6	-
	Promesol 30 gallons/acre							
	0	2½	5	7½	10	20	40	127
	soil pH							
1	5.3	5.4	5.2	5.3	5.2	5.3	5.2	5.2
2	5.4	5.3	5.3	5.3	5.2	5.2	5.2	5.2
3	5.4	5.3	5.2	5.2	5.2	5.3	5.3	5.2
4	5.5	5.3	5.2	5.3	5.2	5.4	5.2	5.0

Field Experiments with Gypsum

Crop Yields
1977

G.R. Blake and Jerome Lensing

Gypsum	Corn Dodge	Corn Steele	Corn Goodhue	Corn Blue Earth	Soybeans Dodge
T/A		Bu/A			Bu/A
0	127.1	91.1	143.4	144.5	28.9
1	114.9	93.2	136.0	165.9	30.7
4	117.5	97.5	140.7	152.1	29.2
Signif.	*	NS	NS	NS	NS
L.S.D. 0.05	8.8				

Modulus of Rupture
Fayette Silt Loam
1976

G.R. Blake

The following data result from three separate methods of soil packing. Gypsum was thoroughly mixed with the soil, previous to preparing briquets at the rates indicated.

In one method the soil was dried and put into frames in a loose condition. Briquets were saturated in a tray by capillarity for one hour. They were then placed in an oven at 50°D overnight and dessicated for one hour.

In each of the remaining methods the soil was moistened and packed into frames at 25 and 100 psi saturated by capillarity for one hour, dried in an oven at 50°D overnight and dessicated one hour.

Soil Condition	Loose Soil	Packed, 25 p.s.i.		Packed, 100 p.s.i.		
Gypsum, T/A	1	0	1	0	1	10
	0.119	1.668	1.167	2.468	1.309	2.065
	0.082	1.399	1.221	2.456	1.432	1.777
	0.193	1.396	1.043	2.527	1.681	1.951
	0.216	1.504	1.108	2.513	1.468	1.814
	0.196		1.264	2.412	1.546	
				2.708		
Sums	0.806	5.967	5.803	15.085	7.436	7.607
Means	0.161	1.492	1.161	2.514	1.487	1.902

In another experiment Fayette silt loam in a plastic condition was worked with a spatula to destroy all aggregates and was then put into molds and dried. Mean moduli of rupture for 29 samples without and 37 samples with 16 T/A gypsum were 33.43 and 33.44 respectively (Data Book 51:19).

Modulus of Rupture*

Fayette Silt Loam
Gypsum Rate, T/A
1976

	Modulus of Rupture at Gypsum Rates Shown			
	0	2	8	16
	1.166	1.318	1.230	1.201
	0.985	1.519	1.111	1.060
	0.987	1.450	1.307	1.284
	1.196	1.446	1.222	1.336
	1.188	1.351	0.947	1.291
	1.313	1.454	0.930	1.313
	1.233	1.396	0.840	1.199
	0.645	0.892	1.345	0.732
	0.596	0.907	1.599	0.917
	0.781	0.885	1.242	0.794
	0.722	0.951	1.424	1.054
	1.283	1.262	1.125	0.932
	1.136	1.167	1.100	0.841
	0.982	1.016	1.035	1.014
	1.110	1.062	1.106	1.106
	1.017	1.251	1.077	1.125
	0.951	1.304		1.108
	1.103	1.212		0.973
	0.861	1.251		1.026
	0.960	1.050		0.628
	0.754	1.260		1.014
	0.835			
Totals	21.804	25.404	18.640	21.948
Means	0.991	1.210	1.165	1.045

*Soil placed in molds in loose condition. See data book 51:11

Plant Tissue Analysis
ICP Optical Emission Spectroscopy

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Plant tissue and grain samples submitted to the Research Analytical Laboratory in 1977 were analyzed by either of two emission spectrometers. In September 1977 the Jarrell-Ash 1.5 meter solution-rotating disk optical emission spectrometer (SRD-OES) was replaced by a radio frequency inductively coupled argon plasma optical emission spectrometer (ICP-OES). Both instruments were used on plant material throughout the summer of 1977. The Jarrell-Ash spectrometer was put into service in 1966 by Gordon Pryor under the direction of Dr. Larry Smith, Department of Agronomy, and an interdepartmental committee which included Dr. Dick Rust of our department. In 1972 the administration of the spectrometer was transferred to Soil Science and integrated into this laboratory. Table 1 shows a summary of sample volume that was handled by the Jarrell-Ash spectrometer during its period of use.

In November 1976 we initiated a request through our department to Director Keith Huston of the Agricultural Experiment Station to purchase an ICP spectrometer, because the older unit was becoming out-dated, and the greater sensitivity of the ICP would provide the additional capability of multielement analysis on a wide variety of material including soil extracts and water. An ICP spectrometer was purchased from Applied Research Laboratories of Sunland, California, with $70,000 funding from the Experiment Station. The spectrometer was installed in early 1977, and we spent the next several months quantifying spectral line interference, establishing technique, and analyzing customer soil extracts. On July 1, 1977 we began to accept plant tissue samples for ICP analysis. The responsibility for the operation of the ICP was given to Robert Grande.

The new spectrometer is giving us more reliable results than the older one, but since the change from one to another of different design occurred in the midst of some research projects, data comparability has been of concern. The data is generally comparable. The agreement on the NBS orchard leaves SRM is very good with the exception of Fe and Al. The differences for Fe and Al appear to be largely due to the different approach taken in calibration. The older spectrometer was initially calibrated against plant material supplied by Dr. A.L. Kenworthy of Michigan State and later included the reference material supplied by Dr. J.B. Jones of Ohio State (material in Table 5). In 1971 the NBS Orchard SRM 1571 was added to the calibration set. The new ICP spectrometer is calibrated against "pure" chemical standard solutions obtained commercially because the ICP has fewer interferences and is capable of handling a wider variety of materials. If any element in the plant material ash is not completely released by 2N HCl, low recovery will result. This appears to be the situation with our ICP analysis for Fe and Al which shows recoveries of between 50-90% for the four currently available National Bureau of Standards botanical reference materials. These results are not unlike those reported by other laboratories. Higher recoveries of Fe and Al can be obtained by ashing at higher temperatures or by wet acid digestion of the sample or ash but loss of certain other elements will occur.

Table 1

Departments serviced and numbers of samples analyzed
on the Jarrell-Ash 1.5 meter direct reading emission spectrometer by G.R. Pryor
Instrument in service 1966 to September 1977

	1966	1967	1968	1969	1970	1971	1972	1973	1974	1975	1976	1977*
Soil Science	2,230		6,947	5,273	5,196	7,163	4,129	4,601	3,593	6,416	5,796	8,809
Agronomy	9,269		10,143	7,431	7,947	3,130	348	617	1,096	1,016	1,395	970
Interdepartmental	(under Soil Science up to 1972)						1,200	1,245	1,985	1,273	2,329	1,020
College of Forestry	1,100		1,989	422	-	551	2,723	2,101	2,150	628	1,393	1,169
Animal Science	140		405	413	3,123	542	218	1,440	977	731	245	606
Horticulture	100	Not catagorized	353	962	212	539	632	723	164	962	2,049	722
Ag Engr.	-		-	-	-	608	665	244	303	144	-	-
ARS	-		-	-	-	-	-	2,402	1,609	2,194	1,716	1,070
U.S. Forest Service (NCFES)			-	-	-	-	110	1,365	2,591	545	463	2,100
Entomology	-		-	-	-	465	3,082	-	-	-	-	-
N.D. State U.	-		-	-	-	-	-	-	-	-	1,905	-
Other:	1,119		387	136	579	165	573	208	220	597	560	491
Food Science, Plant Pathology, Botany, Zoology, Department of Natural Resources, Medicine, Veterinary Medicine, Archeology, Geology, Limnology, Agricultural Engineering, Fisheries and Wildlife												
Yearly Totals	13,958	13,349	20,224	15,183	16,830	13,163	13,726	14,907	14,984	14,506	17,782	19,581

Total samples analyzed by emission spectroscopy during twelve year period----------188,193
Total samples analyzed on Jarrell-Ash spectrometer from January 1, 1966 to September, 1977----------179,964
(Instrument permanently shut down September, 1977. Replaced by ARL ICP spectrometer)

Total elemental determinations (12 elements per sample)----------2,159,568

Average labor, supplies and maintenance costs per sample----------$1.20
(Instrument operation costs only; does not include sample preparation)
Average labor, supplies and maintenance costs per element----------$ .10
Average depreciation of capital investment per sample----------$ .24
Average depreciation of capital investment per element----------$ .02

*includes 8,229 samples analyzed on the new ICP emission spectrometer

Following is a series of tables listing the results we obtained on the ICP spectrometer for a variety of plant tissue materials that have been analyzed by other refereed methods.

Table 2 gives the values obtained by ICP-OES analysis in this laboratory for the NBS tomato, spinach, pine, orchard leaves, and bovine liver standard reference materials. The low values obtained for Na on the orchard leaves are the same as those we obtain by atomic absorption.

Table 3 shows the long term precision for 21 NBS orchard leaves samples analyzed over a 10 week period. Two different dilutions (5 and 10x) of ash from 36 samples of NBS reference material were submitted as unknowns by Dr. Robert Dowdy and reported as short term since they were analyzed over a 2½ hour period. Data reported by ICP-OES analysis at Georgia and an AOAC spark emission study are shown for comparison. Short and long term precision as shown in this table includes any subsampling and sample preparation error. An example of instrumental error is shown in Table 4 where the high concentration calibration standard was analyzed 12 times as an unknown over a 3 hour period during the analysis of 41 plant tissue samples. Normalization and recalibration of the calibration curve took place during this run as shown.

Table 5 gives the values obtained by ICP-OES analysis in this laboratory for alfalfa, tomato, and wheat samples that were obtained from Ohio State in 1967.

Table 6 gives the value obtained by ICP-OES analysis in this laboratory for 12 plant tissue samples obtained from the Inter-Institute Committee (CII) Bondy, France. The assigned means are those obtained by a CII referee study which included several laboratories employing atomic absorption spectroscopy and other methods.

Table 7 lists the elements that have spectral interferences, the interfering element, and the quantitative amount of interference.

Table 8 compares the detection limits measured by the ICP spectrometer to the atomic absorption spectrometer limits expressed in commercial literature. Also listed are the more realistic lower limits expressed as LQD for the ICP spectrometer and Sensitivity and LOCR for atomic absorption spectrometry.

Summary

No conclusions are drawn from the raw data shown in the tables. All analyses were treated as customer unknowns so they would realistically represent our work. The areas of discrepancy will be explored to separate analytical differences from preparation method differences.

All concentrations reported in ppm unless shown otherwise.

Table 2 ICP-OES determinations on NBS botanical standard reference material[a]

element	Tomato Leaves (SRM 1573) assigned NBS value	Tomato Leaves found n=2 mean	Spinach Leaves (SRM 1570) assigned NBS value	Spinach Leaves found n=2 mean	Pine Needles (SRM 1575) assigned NBS value	Pine Needles found n=2 mean	Orchard Leaves (SRM 1571) assigned NBS value	Orchard Leaves found n=2 mean	Bovine Liver (SRM 1577) assigned NBS value	Bovine Liver found n=2 mean
P%	0.34 ± 0.02	0.32	0.55 ± 0.02	0.51	0.12 ± 0.02	0.117	0.21 ± 0.01	0.192	-	0.985
K%	4.46 ± 0.03	4.47	3.56 ± 0.03	3.58	0.37 ± 0.02	0.381	1.47 ± 0.03	1.51	0.97 ± 0.06	0.966
Ca%	3.00 ± 0.03	3.05	1.35 ± 0.03	1.40	0.41 ± 0.02	0.440	2.09 ± 0.03	2.10	(0.012)*	0.012
Mg%	(0.07)	0.691	-	0.916	-	0.123	0.62 ± 0.02	0.620	(0.060)	0.057
Al	(1,200)	356	870 ± 50	452	545 ± 30	488	-	132[b]	-	6.03
Fe	690 ± 25	438	550 ± 20	490	200 ± 10	124	300 ± 20	200	270 ± 20	230
Na	-	338	-	13,909	-	15.8	82 ± 6	34.2	2,430 ± 130	2,174
Mn	238 ± 7	218	165 ± 6	168	675 ± 15	705	91 ± 4	90.2	10.3 ± 1.0	8.31
Zn	62 ± 6	65.8	50 ± 2	54.2	-	77.0	25 ± 3	30.2	130 ± 10	138
Cu	11 ± 1	9.6	12 ± 2	11.6	3.0 ± 0.3	3.1	12 ± 1	13.5	193 ± 10	76
B	(30)	37.0	(30)	28.0	-	18.0	33 ± 3	33.5	-	1.0
Pb	6.3 ± 0.3	8.0	1.2 ± 0.2	3.0	10.8 ± 0.5	13.5	45 ± 3	50.0	0.34 ± 0.08	1.2
Ni	-	1.0	(6)	5.4	(3.5)	2.6	1.3 ± 0.2	1.2	-	0.09
Cr	4.5 ± 0.5	1.6	4.6 ± 0.3	2.2	2.6 ± 0.2	1.85	2.6 ± 0.3	1.2	-	0.44
Cd	(3)	2.6	(1.5)	1.5	(<0.5)	0.28	0.11 ± 0.01	0.15	0.27 ± 0.04	0.23
Co	(0.6)	0.58[b]	(1.5)	2.0	(0.1)	0.94	(0.2)	0.22[b]	(0.18)	1.6

[a]1.0 gm oven dried (85°C) and dry ashed at 485°C for 10 hrs. Ash dissolved in 10 ml 2N HCl. The bovine liver was treated the same except that it was stored in a desicator rather than oven dried. The ash from the liver was a consolidated mass of char.
[b]n=2 has greater than 10% difference.
*Brackets indicate not certified

Table 3

Short and Long Term Precision Study and Comparative Results in the Analysis of NBS Orchard Leaves Reference Material by Plasma Emission at Minnesota in Reference to Plasma Emission Values from Georgia and Spark Emission Values from an AOAC Collaborative Study

Concentration Means	Dilution used	P %	K %	Ca %	Mg %	Al ppm	Fe ppm	Na ppm	Mn ppm
Assigned Mean by NBS		.21	1.47	2.09	.62	-	300	82	91
Assigned Range by NBS		.20-.22	1.44-1.5	2.06-2.12	.60-.64	-	280-320	76-88	87-95
[1]ICP - Long Term U of M	10x	.194	1.53	2.15	.640	126	193	44	93
[2]ICP - Short Term U of M	5x	.192	1.49	2.09	.625	122	176	50	88
[2]ICP - Short Term U of M	10x	.195	1.55	2.26	.666	131	191	52	96
[3]ICP - Georgia	20x	.22	1.60	1.99	.62	234	183	140	101
[4]Spark - AOAC	5-10x	.19	1.38	1.97	.63	215	241	206	94
Standard Deviation									
ICP - Long Term	10x	.007	.056	.070	.023	14	22	2	5
ICP - Short Term	5x	.004	.032	.029	.020	7	6	2	2
ICP - Short Term	10x	.007	.041	.041	.011	7	7	2	2
ICP - Georgia	20x	.005	.07	.07	.02	79	12	50	2
Spark - AOAC	5-10x	.03	.17	.20	.04	49	62	183	27
Relative Standard Deviation (%C.V.)									
ICP - Long Term	10x	3.5	3.6	5.6	3.6	11	11	4	5
ICP - Short Term	5x	2.1	2.2	1.4	1.9	5	3	3	2
ICP - Short Term	10x	3.7	2.6	1.8	1.7	5	4	4	2
ICP - Georgia	20x	2.2	4.4	3.5	2.2	34	7	36	2
Spark - AOAC	5-10x	16.8	12.4	10.3	5.9	23	26	89	28
Spectra Interferences, Computer Corrected for "Long and Short Term" Data Above									
Interfering Elements		Cu	-	-	Ca	Ca	Mn/Cu/Al	-	Fe
Concentration Error (ppm) (in above ICP Long Term Means)		<10	0	0	<5	47-86	<0.2	0	0.56

Continued Next Page

Table 3 Continued

Concentration Means	Dilution used	Zn ppm	Cu ppm	B ppm	Pb ppm	Ni ppm	Cr ppm	Cd ppm	Co ppm
Assigned Means by NBS		25	12	33	45	1.3	(2.3)	0.11	(0.2)
Assigned Range by NBS		22-28	11-13	30-36	42-48	1.1-1.5	-	0.9-.13	-
[1]ICP - Long Term U of M	10x	28.3	12.7	33.8	51.7	1.66	1.3	0.32	0.00-3
[2]ICP - Short Term U of M	5x	26.9	11.5	33.2	44.7	1.54	1.6	0.24	-
[2]ICP - Short Term U of M	10x	29.2	11.8	35.0	47.7	1.60	1.8	0.25	-
[3]ICP - Georgia	20x	28	17	41	48	-	2.9	0.41	0.2
[4]Spark - AOAC	5-10x	28	15	32	-	-	-	-	-
Standard Deviation									
ICP - Long Term	10x	1.4	0.4	0.7	2.5	0.23	0.18	0.08	0.7
ICP - Short Term	5x	1.4	0.4	0.7	1.4	0.13	0.27	0.03	-
ICP - Short Term	10x	1.3	0.4	0.7	1.2	0.26	0.18	0.07	-
ICP - Georgia	20x	5	4	7	5	-	0.73	0.15	0.06
Spark - AOAC	5-10x	6	3	5	-	-	-	-	-
Relative Standard Deviation (%C.V.)									
ICP - Long Term	10x	5	3	2.2	4.8	14	14	25	24
ICP - Short Term	5x	5	4	2.0	3.1	8	17	14	-
ICP - Short Term	10x	5	3	2.0	2.6	16	10	27	-
ICP - Georgia	20x	18	24	1.7	10.4	-	26	37	26
Spark - AOAC	5-10x	22	17	15.6	-	-	-	-	-
Spectra Interferences, Computer Corrected for "Long and Short Term" Data Above									
Interfering elements		Cu/Mg	-	Fe/K	-	-	Mn	Cu/Fe	Fe
Concentration Error (ppm) (in above ICP Long Term Means)		.12-.16	0	.35-.8	0	0	.04-.28	.01-.15	.04-.1

[1]Long term, 21 samples analyzed over 10 week period, 11/9/77-1/25/78. 1 g ashed at 485° for 10 hrs.
[2]Short term, 36 samples analyzed over a 2½ hr period. Slow ashed at 450°C for 24 hrs. Submitted by R. Dowdy, ARS
[3]Georgia, J.B. Jones, Elemental Analysis----Plant Tissue Ash by Plasma Emission Spect., Comm. Soil Sci. and Plant Anal. (Vol. 8, No. 4, 1977)
[4]AOAC, J.B. Jones, Collaborative study by 11 spark emission spectrograph laboratories, Jour. of the AOAC (Vol 58, No. 4, 1975)

Table 4

Precision Evaluation

Analyses of high standard solution* as an unknown at 12 different times over a 3 hour period during the analysis of 41 plant tissue samples** (Concentration in µg/ml in 2N HCl)

Time lapse min.	Time diff. min.	K 2000	Ca 3000	Mg 1000	Al 100	Fe 100	Mn 100	Zn 100	Cu 10.0	B 10.0	Pb 10.0	Ni 10.0	Cd 10.0	Co 10.81
0		1947	2914	973	109	97.3	97.4	9.77	9.74	9.68	9.70	9.73	9.74	10.5
	2													
2		1980	2949	994	101	98.7	99.3	9.95	9.88	9.87	9.90	9.89	9.87	10.6
	13													
15		1950	2922	991	103	98.6	97.4	9.86	9.85	0.83	0.89	9.83	9.84	10.6
	10													
25		1953	2941	1002	103	99.3	99.8	9.92	9.94	9.92	9.90	9.91	9.92	10.6
	5													
30		1876	2844	975	100	96.5	98.3	9.67	9.65	9.60	9.66	9.64	9.65	10.2
	4													
34		1867	2843	974	99.8	96.8	99.0	9.68	9.70	9.65	9.61	9.61	9.60	10.3
	15	Calibration "normalized" by computer on blank and high standard												
49		2017	3030	1004	98.5	100.8	99.8	10.01	10.13	10.06	10.02	10.08	10.05	10.9
	3													
52		2001	3036	1013	104.9	101.7	97.4	10.10	10.17	10.12	10.07	10.12	10.10	11.02
	15													
67		1998	3046	1019	94.7	102.2	96.4	10.15	10.22	10.18	10.17	10.16	10.16	11.23
	3													
70		1958	2983	1004	95.3	100.3	96.4	9.96	10.03	9.98	9.96	9.96	9.97	10.92
	13	Calibration "normalized" by computer on blank and high standard												
83		2018	3018	996	89.2	100.3	100.6	9.93	10.03	10.05	9.98	10.01	9.99	10.93
	2													
85		2044	3072	1019	89.2	101.7	103.9	10.06	10.19	10.29	10.15	10.15	10.15	11.02
	26	Noon break - Recalibrated - New calibration curve fit by computer on blank and high standard												
111		1987	2984	986	102.6	98.9	97.9	9.83	9.94	9.90	9.82	9.89	9.87	10.55
	17													
128		1997	3010	1003	94.5	99.7	98.8	9.92	10.08	10.03	9.92	9.96	9.94	10.65
	28													
159		1975	2992	994	100.7	100.0	93.5	9.96	10.06	9.97	9.88	9.91	9.91	10.57
	26													
185		1942	2969	993	99.6	99.6	94.6	9.89	10.03	9.90	9.85	9.85	9.86	10.48
$\bar{x}$		1969	2972	996	99.1	99.5	98.2	9.92	9.98	9.94	9.90	9.92	9.91	10.69
s		47.9	67.1	14.5	5.37	1.70	2.44	0.13	0.17	0.19	0.16	0.17	0.16	0.28
RSD%		2.4	2.3	1.5	5.4	1.7	2.5	1.3	1.7	1.9	1.6	1.7	1.6	2.6

*It is standard practice to run the high standard as an unknown between every 10-20 samples. If any element concentration is off more than 5% of the calibrated value, a blank and high standard are run using the "normalizing" subroutine in the computer.

**The plant tissue samples run during this period are those in Tables 2 and 5.

Table 5 ICP-OES determinations on OARDC[a] referee plant tissue materials[b]

element	Alfalfa Assigned Mean	Alfalfa Assigned Range	Alfalfa ICP found Mean(n=2)	Tomato Assigned Mean	Tomato Assigned Range	Tomato ICP found Mean(n=2)	Wheat Assigned Mean	Wheat Assigned Range	Wheat ICP found Mean(n=2)
P%	[c]0.30	0.29-0.31	0.286	0.52	0.49-0.55	0.448	0.17	0.16-0.18	0.142
	[d]0.30	0.26-0.35		0.52	0.44-0.60		0.17	0.13-0.21	
K%	1.24	1.17-1.31	1.38	3.43	3.30-3.56	3.66	1.53	1.42-1.64	1.82
	1.25	1.07-1.43		3.66	2.64-4.68		1.53	1.19-1.87	
Ca%	1.61	1.53-1.69	1.68	4.14	3.88-4.40	4.84	0.17	0.16-0.18	0.197
	1.46	1.17-1.75		4.16	2.89-5.43		0.20	0.11-0.29	
Mg%	0.33	0.31-0.35	0.378	0.98	0.94-1.02	0.947	0.07	-	0.084
	0.32	0.26-0.38		0.90	0.74-1.06		0.07	0.04-0.10	
Al	81	80-82	43.4[e]	105	99-111	146	38	36-40	42.9
	81	55-107		109	50-168		38	27-49	
Fe	118	112-124	125	195	183-207	102	64	61-67	66.2
	122	89-155		188	105-271		70	46-94	
Na	630	626-634	522	2340	2251-2429	2270	200	196-204	43.2
	681	473-889		2811	2101-3521		224	61-387	
Mn	41	39-43	44.2	566	538-594	568	81	76-86	89.8
	42	31-53		580	488-672		82	70-94	
Zn	-	-	36.3	-	-	141	-	-	22.6
Cu	11	10-12	8.2	20	19-21	12	5	-	3.6
	11	6-16		21	12-30		6	2-10	
B	27	26-28	34	91	85-97	89	3	-	2.2
	30	20-40		94	68-120		4	0-9	

[a]OARDC - Ohio Agricultural Research and Development Center, Ohio State University. This material was distributed by J.B. Jones in 1967 for atomic emission analysis by 17 participating laboratories including this facility. These samples have been used as calibration standards on the Jarrell-Ash spectrometer.
[b]1.0 gm oven dried at (85°c) and dry ashed at 485°C for 10 hrs. Ash dissolved in 10 ml 2N HCl.
[c]Mean after removing outliners greater than 10% of the mean.
[d]Mean without removing outliners.
[e]n=2 has greater than 10% difference.

Table 6 Comparison of analyses of CII botanicals[a] by ICP-OES analysis at Minnesota[b] with ICP-OES at Applied Research Laboratories, Sunland California[c] against assigned French CII (Committee Inter-Institute) values obtained by other methods including atomic absorption spectrometry

element	Orange (Spain) CII assigned mean	Orange (Spain) CII assigned range[e]	Orange (Spain) ICP Calif n=2 mean	Orange (Spain) ICP Minn n=2 mean	Vigne (France CII assigned mean	Vigne (France CII assigned range	Vigne (France ICP Calif n=2 mean	Vigne (France ICP Minn n=2 mean	Codia discolor (N. Calidonia) CII assigned mean	Codia discolor (N. Calidonia) CII assigned range	Codia discolor (N. Calidonia) ICP Calif n=2 mean	Codia discolor (N. Calidonia) ICP Minn n=2 mean
P%	.15	.142-.166	.126	.148	.207	.197-.216	.218	.200	.025	.021-.029	.020	.020
K%	1.51	1.41-1.67	1.51	1.58	1.23	1.15-1.30	1.29	1.23	.37	.334-.399	.370	.381
Ca%	4.91	4.71-5.06	4.4	5.03	2.55	2.44-2.73	2.60	2.58	.83	.794-.868	.727	.834
Mg%	.32	.296-.341	.264	.311	.28	.263-.298	.278	.284	.28	.273-.298	.259	.292
Al	-	-	612	221	-	-	366	223	-	-	255	248
Fe	472	433-497	354	382	265	239-304	246	250	87	81.1-95.5	92.7	58.0
Na[f]	380	-	-	266	260	-	-	164	1400	-	-	1296
Mn	75	69.9-80.4	66.6	68.3	196	184-203	188	180	653	630-680	415	522
Zn	70	61.9-75.1	52.0	128	75.5	66.8-81.8	78.1	108	11.3	10.1-12.9	8.6	19.1
Cu	8.5	7.3-9.7	5.8	13.2	880	793-979	848	864	4.69	3.75-5.95	3.1	3.4
B	39.5	-	-	42	48	-	-	42	25	-	-	27
Pb	-	-	7.8	7.2	-	-	18	18	-	-	3.0	.18
Ni	-	-	1.6	2.1	-	-	1.6	1.4	-	-	>200	200
Cr	-	-	1.1	.68	-	-	.9	.44	-	-	>5	4.4
Cd	-	-	-	0.0	-	-	-	0.0	-	-	-	0.0
Co	-	-	0.5	1.8	-	-	.5	.02	-	-	>20	28

Continued--

Table 6 Continued

element	Eucalyptus (Spain) CII assigned mean	Eucalyptus (Spain) CII assigned range	Eucalyptus (Spain) ICP Calif n=2 mean	Eucalyptus (Spain) ICP Minn n=2 mean	Cotton (Africa) CII assigned mean	Cotton (Africa) CII assigned range	Cotton (Africa) ICP Calif n=2 mean	Cotton (Africa) ICP Minn n=2 mean	Olive (Spain) CII assigned mean	Olive (Spain) CII assigned range	Olive (Spain) ICP Calif n=2 mean	Olive (Spain) ICP Minn n=2 mean
P %	.085	.076-.093	.078	.084	.36	.343-.382	.365	.352	.083	.080-.089	.078	.084
K %	.68	.630-.759	.741	.739	1.84	1.76-1.89	1.84	1.81	.51	.44-.58	.56	.540
Ca %	1.56	1.46-1.68	1.31	1.68	3.1	2.88-3.27	3.2	3.04	2.59	2.47-2.74	2.61	2.81
Mg %	.21	.191-.224	.190	.218	.41	.396-.430	.429	.408	.13	.125-1.38	.131	.135
Al	-	-	246	65.4[d]	-	-	320	178	-	-	373	138
Fe	102	94.5-110.9	89.5	100	404	382-452	339	250	169	152-191	166	147
Na[f]	1700	-	-	1750	500	-	-	371	80	-	-	20.5
Mn	574	538-602	307	580	220	207-229	215	190	48	44.8-53.3	50.7	44.3
Zn	12.4	10.3-15.3	10.3	18.3	127	114-137	103	118	15	14.5-17	13.9	23[d]
Cu	8.4	6.8-9.4	6.3	6.4	15	136-16.5	14.4	12	37	33.5-38.5	37.8	42
B	35	-	-	36	25	-	-	25	18	-	-	17
Pb	-	-	5.9	1.3	-	-	6.7	2.6	-	-	7.4	7.9[d]
Ni	-	-	2.7	3.4	-	-	1.7	1.05[d]	-	-	.9	.9[d]
Cr	-	-	.4	.11	-	-	1.0	.56	-	-	.5	.18
Cd	-	-	-	.1[d]	-	-	-	.09[d]	-	-	-	0.0
Co	-	-	.3	0.0	-	-	.7	.43[d]	-	-	.2	.52

Continued--

Table 6 Continued

element	Palm (Africa) CII assigned mean	Palm (Africa) CII assigned range	Palm (Africa) ICP Calif n=2 mean	Palm (Africa) ICP Minn n=2 mean	Peach (France) CII assigned mean	Peach (France) CII assigned range	Peach (France) ICP Calif n=2 mean	Peach (France) ICP Minn n=2 mean	Corn (Spain) CII assigned mean	Corn (Spain) CII assigned range	Corn (Spain) ICP Calif n=2 mean	Corn (Spain) ICP Minn n=2 mean
P%	.165	.160-.172	.149	.158	.292	.271-.301	.276	.276	.219	.204-.231	.204	.206
K%	.885	.815-.940	.852	.907	2.19	2.06-2.31	2.25	2.24	1.94	1.79-2.01	1.86	1.92
Ca%	.80	.766-.834	.696	.815	2.15	2.05-2.27	1.98	2.26	1.12	1.07-1.18	.986	1.11
Mg%	.295	.283-.309	.269	.306	.475	.449-.497	.475	.486	.36	.351-.375	.344	.355
Al	-	-	182	166	-	-	263	175	-	-	-	158
Fe	197	187-212	156	183	302	264-334	223	244	259	242-276	227	214
Na[f]	170	-	-	126	260	-	-	134	1700	-	-	1640
Mn	631	602-658	117	604	81	77.6-88.1	84.3	76.3	66	61.5-70.7	63.6	55.2
Zn	23	19.9-26.2	19.0	47.2[d]	28	23.2-32.3	24.0	39.6	27	24.5-31.6	22.6	72.8[d]
Cu	8.9	7.1-10.5	5.9	9	15	131-17.5	14.3	14	11.4	10.2-13.4	6.5	10.5
B	15	-	-	14	38	-	-	40	23	-	-	23
Pb	-	-	3.4	2.3[d]	-	-	5.5	2.5	-	-	4.4	2.6
Ni	-	-	.5	.89	-	-	1.2	.86[d]	-	-	.6	.56[d]
Cr	-	-	.5	.19	-	-	.6	.33	-	-	.9	.28
Cd	-	-	-	0.0	-	-	-	.20[d]	-	-	-	0.0
Co	-	-	(.2)	0.0	-	-	.3	0.0	-	-	.3	0.0

Continued--

Table 6 Continued

element	Apple, Golden (Belgium) CII assigned mean	range	ICP Calif n=2 mean	ICP Minn n=2 mean	Apple, Cox's (France) CII assigned mean	range	ICP Calif n=2 mean	ICP Minn n=2 mean	Hevea (Ivory Coast) CII assigned mean	range	ICP Calif n=2 mean	ICP Minn n=2 mean
P%	.16	.157-.170	.148	.158	.177	.170-.183	.169	.165	.26	.246-.269	.252	.260
K%	1.45	1.37-1.52	1.46	1.51	1.04	.949-1.12	1.05	.987	.93	.886-.988	.978	1.00
Ca%	1.27	1.20-1.36	1.18	1.34	2.14	2.06-2.22	1.96	2.12	1.08	.987-1.148	.912	1.11
Mg%	.29	.269-.303	.278	.297	.29	.280-.311	.282	.282	.33	.307-.355	.331	.358
Al	-	-	162	113[d]	-	-	1045	672	-	-	109	86.9
Fe	308	277-345	216	188	1120	1028-1281	1047	726	160	135-183	143	150
Na[f]	160	-	-	90.7	570	-	-	228	100	-	-	51.7
Mn	47	41.9-51.5	46.1	45.2	136	130-142	136	122	162	153-168	147	162
Zn	29	26.0-34.5	26.1	38.8	79	69.1-84.6	79.1	92.6	37.7	34.4-37.4	36.3	56.3
Cu	8.2	6.9-9.9	7.1	7.7	173	146-208	175	171	14.8	131-17.8	16.5	14.8
B	29	-	-	30	33	-	-	32	58	-	-	60
Pb	-	-	11	12	-	-	22	24	-	-	6.0	1.8
Ni	-	-	4.9	5.3	-	-	4.3	4.0	-	-	1.6	1.8
Cr	-	-	.6	.24	-	-	2.3	1.1	-	-	.6	.20
Cd	-	-	.3	.13	-	-	1.0	.28	-	-	.6	0.0
Co	-	-	-	0.0	-	-	-	0.0	-	-	-	0.0

[a]CII materials provided by M. Pinta, Office Da La Recherche Scientifique et Technique Autre-Mer, Services Scientifiques Centraux Bondy, France. ref: "Applied Spectroscopy," vol. 32, no. 1, Jan/Feb 1978 pp1-30.
[b]1.0 gm oven dried (85°C) and dry ashed at 485°C for 10 hrs. Ash dissolved in 10 ml 1N HCl.
[c]1.0 gm dry ashed at 200°C for 8 hrs. followed by HNO_3 acid treatment and ashing again at 485°C for 20 min. Taken up in 1.25N HCl. As reported in "Applied Spectroscopy," vol. 32, no. 1, Jan/Feb 1978, pp1-30.
[d]n=2 has greater than 10% difference
[e]range=$\bar{x}\pm$Std. Dev.
[f]Assigned Na values by CII were from one determination and reported with reservation and for information only.

Table 7

Measured Spectral Interferences in the ICP Spectrometer[a]

effect of (x)		on channel (y)	error in channel (y) of (x)
	(x)	(y)	ppm
1ppm	Ca	Al	0.00230 - 0.0040
"	Mg	Zn	0.00025
"	Na	Zn	0.00000 - 0.0002
"	Cu	Zn	0.00920
"	Fe	B	0.00140 - 0.0043
"	Fe	Cd	0.00068 - 0.0001
"	Cu	Cd	0.00090 - 0.0011
"	Fe	Co	0.00020 - 0.0005
"	Mn	Cr	0.00040 - 0.0031
"	Fe	Hg	0.00038 - 0.0067
"	Cu	P	0.43000 - 0.8800
"	Mn	Fe	0.00135
"	Al	Fe	0.00053

[a]All 20 elements were aspirated as single element 2N HCl solutions (x), several times over a 6 month period, and the effect of element (x) was measured in element channel (y). Only those interferences that are significant in biological material, soil extracts, and water samples are reported here.

On-line computer software corrections are made for these interferences.

Concentrations of interfering élements used were as follows: 500 ppm for Ca and Mg, 200 ppm for Fe, and 100 ppm for Cu, Mn, Al and sodium.

Table 8

Comparison of Detection Limits and Practical Lower Concentration Limits between ICP-OES, FAAS, and SRD-OES Methods

ICP Wave-length	Ele-ment	Detection Limits: ICP-OES[a] Range µg/ml	Detection Limits: Flame Atomic Absorption (FAAS) µg/ml	Practical Lower Limits: Flame Atomic Absorption: Sensi-tivity[c] µg/ml	Practical Lower Limits: Flame Atomic Absorption: LOCR[d] µg/ml	Practical Lower Limits: ICP LQD[e] µg/ml	Practical Lower Limits: SRD-OES LLR[f] µg/ml
2136.2	P	0.003-0.245	not applicable	-	-	1.23	20
7664.9	K	0.07-0.312	0.005	0.04	0.1-2	1.56	100
4226.7	Ca	0.006-0.094	0.003	0.08	0.2-20	0.470	20
2790.7x2	Mg	0.016-0.046	0.0005	0.007	0.02-2	0.230	10
3961.5x2	Al	0.005-0.058	0.1[g]	1	5-100	0.290	1
2599.4x2	Fe	0.003-0.008	0.02	0.12	0.3-10	0.040	2
5890.0x2	Na	0.01-0.221	0.002	0.015	0.03-1	1.11	30
2576.1x2	Mn	0.001-0.004	0.01	0.05	0.1-10	0.020	1
2025.5x2	Zn	0.003-0.010	0.005	0.02	0.05-2	0.050	0.1
3247.5x2	Cu	0.003-0.010	0.01	0.1	0.2-10	0.050	0.1
2497.7x2	B	0.003-0.006	6[g]	40	-	0.030	0.2
2203.5x2	Pb	0.02-0.067	0.05	0.5	1-20	0.335	4
2316.0x2	Ni	0.01-0.031	0.02	0.15	0.3-10	0.155	4
2677.2x2	Cr	0.001-0.005	0.02	0.1	0.2-10	0.025	4
2265.0x2	Cd	0.004-0.006	0.02	0.025	0.05-2	0.030	1
3453.0x2	Co	0.2	0.03	0.2	0.5-10	1.00	4
2536.5x2	Hg	0.02-0.057	0.5	7.5	20-300	0.285	-
1937.6x2	As	0.1-0.316	0.1[h]	0.8	5-50	1.58	4
1960.9x2	Se	0.1-0.879	0.1[h]	0.5	2-50	4.40	-

[a]Measured by this laboratory. In general, the lowest concentration shown is in single element solutions. The larger value was the latest data obtained 3-21-78 on multielement solutions in 2N HCl.
[b]Values taken from commercial literature for single element solutions in D.I. water.
[c]Sensitivity defined as ug/ml that gives a 1% absorption signal.
[d]LOCR is defined as the lower optimum concentration range (Manual of methods for chemical analysis of water and wastes, EPA-62516-74-003).
[e]LQD is defined in ICP literature as the lowest quantitatively determinable concentration or 5x the detection limit.
[f]Lowest limit reported, solution rotating disk optical emission spectrometer.
[gh]Nitrous oxide and argon-hydrogen flame respectively.

WATER SOFTENING AND LIME WASTES FROM THE MINNEAPOLIS WATER DEPARTMENT

C. P. Klint and C. J. Overdahl*

Large cities have lime disposal problems in the water softening process. From 10 to 12 truckloads of a limy material from the Minneapolis water treatment facility at Fridley need to be disposed of daily.

Several thousand tons of material are added to river water and are consumed yearly. This creates a waste product of calcium carbonate and magnesium hydroxide. The lime yield is about 1¼ tons of dry material per million treated gallons of water.

Representatives of the Fridley plant contacted the University of Minnesota Soil Science Department and the Anoka County Extension Office seeking help on what uses this product might have in agriculture.

The product used in the treatment process is brought in by the carload as calcium oxide. The softening process of the Mississippi River water is about as follows: calcium oxide + water yields calcium hydroxide. Calcium hydroxide reacts with the river water hardness (calcium bi-carbonate) and precipitates calcium carbonate plus water. There is also a reaction with the magnesium bi-carbonate in the water with calcium hydroxide to yield calcium carbonate plus magnesium carbonate and water. The magnesium carbonate will further react with calcium hydroxide to form magnesium hydroxide and calcium carbonate. The latter two compounds are the waste product to be disposed. Some other elements are also extracted from the river water.

These other elements vary with analysis. A report from the Serco Sanitary Engineering Laboratories, Inc. using standard methods for examination of wastewater and according to EPA methods for chemical analysis of water and wastes, 1974, gave the following analysis on dry weight basis for these other elements which appear to be in small quantity:

	ppm	%
Cyanide	3.3	.0003
Manganese	932.0	.0932
Iron	3163.0	.3163
Arsenic	6.3	.0006
Cadmium	13.0	.0013
Hexavalent chromium	6.	.0006
Copper	16.0	.0016
Lead	67.0	.0067
Zinc	50.0	.0050
Mercury	0.23	.000023
Sulfate	1998.0	.1998

pH = 9.6

* Area Extension Agent, Soils, Anoka, and Extension Specialist, Soils, respectively, University of Minnesota.

A separate report gave the average calcium carbonate content as 89.5% and magnesium hydroxide as 3.13%. The University of Minnesota Soil Testing Laboratory reported a 90.6% calcium carbonate equivalent.

Field Trials in Anoka County

Field application was first tried in the fall of 1974. The material contained 66% water and 33% solids. It spread quite easily with a manure spreader. The application rate was 10 tons per acre of waste or about 3.3 tons per acre of dry matter. The soil pH of a check area in the fall of 1975 was 6.3 and the treated area had a pH of 6.7. This initial effort revealed that the material would probably be a satisfactory liming product.

Further efforts were made in the greenhouse with additional field trials to check for possible harmful effects when higher rates were used. Plant tissue analysis was also made to get an opinion on whether some of the possible harmful ingredients were absorbed in any quantity by the plant.

Greenhouse and Second Field Trial

In the second series of trials, lime wastes were applied to the soil for greenhouse study in April 1976. Soybeans and rye were planted in May 1976. Soils were sampled to determine pH in October 1976. Soil treatment rates on a dry weight basis and the corresponding pH values were as follows:

Waste lime Rate/A	pH
Peat soil (greenhouse)	
Check	6.3
2 tons	6.8
4 "	7.0
12 "	7.6
Sandy loam soil (greenhouse)	
Check	6.3
2 tons	7.6
4 "	7.6
8 "	7.9
16 "	8.0
32 "	8.0
64 "	8.3
128 "	8.5
Sandy loam soil (field)	
Check	6.3
2 tons	6.4
4 "	6.8
8 "	7.0
16 "	7.4

Observations revealed no positive or negative effect generally on plant growth or appearance. At waste treatment above 64 tons per acre on the sandy loam soil, soybeans showed some abnormal coloring.

Rye and soybeans were planted in both the greenhouse pots and in the field trial. The plant analysis from the University of Minnesota Soil Science Department plant analysis laboratory is shown in the following tables.

Summary

In these limited experiments, there were several interesting relationships of lime rates to plant nutrient content, but in line with what is expected. Since no adverse effects were observed, a preliminary opinion would be that this lime waste would be a satisfactory liming material and disposal on farms having acid soils would be a reasonable approach. Research should be conducted from other lime waste sites where some heavy metals may be in higher concentrations.

RYE

Greenhouse

	%				ppm											
	P	K	Ca	Mg	Al	Fe	Na	Mn	Zn	Cu	B	PB	Nl	Cr	Cd	Co
Check	.25	2.2	1.4	.30	248	307	1025	376	21	6	27	4	4	1.0	.20	1.9
4 ton	.18	1.51	.75	.63	154	252	1298	323	22	6	24	4	3	.92	.23	1.5
16 "	.11	.88	1.8	.93	103	773	1092	59	15	7	23	6	6	1.4	.59	2.0

RYE

Field

Check	.15	1.16	.74	.18	399	1225	71	269	31	3	26	6	2	1.1	.47	1.3
4 ton	.29	1.53	.58	.31	274	1170	79	92	40	4	18	5	3	1.0	.35	1.7
16 "	.23	1.65	.56	.27	317	1099	94	125	31	4	14	6	2	1.2	.38	1.9
Standard levels for rye																
Suff.	.21	1.51	.21	.21	-	11	-	16	21	6	6	-	-	-	-	-
Excess	.81	5.50	-	-	400	550	-	350	151	71	56	-	-	-	-	-

SOYBEANS

Greenhouse

Check	.36	1.67	1.9	.67	200	315	813	74	34	11	45	4	8	.9	.22	2.6
4 ton	.19	.94	1.09	1.02	105	171	169	46	21	7	33	3	5	.7	.31	1.7
16 "	.20	.96	.48	1.27	121	191	1189	28	23	8	25	3	5	.7	.33	1.1

SOYBEANS

Field

Check	.20	1.57	.90	.58	175	562	58	75	34	6	26	5	3	.8	.3	1.3
4 ton	.30	1.60	.57	.320	194	903	63	88	43	5	21	4	3	.8	.4	1.4
16 "	.35	1.74	.49	.40	52	291	24	47	43	7	28	3	3	.5	.2	1.6
Standard levels for soybeans																
Suff.	.26	1.51	.36	.26	-	51	-	21	21	10	21	-	-	-	-	-
Excess	.81	5.50	3.00	2.00	-	750	-	550	150	70	100	-	-	-	-	-

EFFECT OF WEX ON CROP YIELDS

W. Fenster, G. Randall, W. Nelson, S. Evans and R. Schoper

The product Wex is a wetting agent which, when applied to the soil, is claimed to increase the yield of various field crops by aiding soil nutrient availability. Specifically, claims are made for Wex as an agent which helps crops achieve their genetic potential by releasing more soil nutrients, increasing trace mineral uptake, enhancing fertilizer activity, and aiding in superior root development.

With these claims in mind, experiments were conducted during 1976 and 1977 using Wex as a soil additive on corn, soybeans and wheat at the Waseca, Lamberton and Morris Experiment Stations.

EXPERIMENTAL PROCEDURE

The experiments were located on a Webster clay loam at Waseca, Nicollet silty clay loam at Lamberton and a Forman clay loam at Morris.

The treatments, arranged in a randomized, complete-block design, were replicated six times in each experiment at Waseca and Morris and five times at Lamberton. In 1976, the treatments consisted of a check, 8 and 16 oz. of Wex per acre at all locations on corn, soybeans and wheat. In 1977, the treatments consisted of a check, 16 oz. of Wex and 16 oz. per acre of an experimental surfactant named MGJ-A for corn and wheat and MGJ-B for soybeans. All treatments were applied preplant and incorporated with the soil at Lamberton and surface applied at Waseca and Morris. In all cases, adequate N, P and K treatments were applied and good weed and insect control were maintained.

RESULTS AND DISCUSSION

The yield data for wheat, soybeans and corn are presented in Tables 1-3. There were no significant yield differences among treatments for any of the crops, at any of the locations, for either 1976 or 1977.

Various other criteria were used to aid in determining whether Wex improved plant growth. The data from these measurements, which included stand counts, early plant growth, percentage of essential nutrients in the plant at various stages of growth and percent protein in the wheat grain, will be presented in the future in the form of a soil series.

Table 1. Effect of varying levels of Wex on wheat yields.

Treatment Material	Rate	Waseca 1976	Waseca 1977	Lamberton 1976	Lamberton 1977	Morris 1976	Morris 1977
	oz/A	bu/A					
Check		55	42	25	49	25	47
Wex	8	54		25		26	
Wex	16	53	43	26	49	26	48
MGJ-A	16		45		49		47
Significance (.05)		ns	ns	ns	ns	ns	ns

Table 2. Effect of various levels of Wex on soybean yields.

Treatment Material	Rate	Waseca 1976	Waseca 1977	Lamberton 1976	Lamberton 1977	Morris* 1977
	oz/A	bu/A				
Check		36	53	17	46	29
Wex	8	37		16		
Wex	16	37	53	17	49	30
MGJ-B	16		53		47	31
Significance (.05)		ns	ns	ns	ns	ns

* 1976 yields lost due to hail damage.

Table 3. Effect of varying levels of Wex on corn yields.

Treatment Material	Rate	Waseca 1976	Waseca 1977	Lamberton 1976	Lamberton 1977	Morris* 1977
	oz/A	bu/A				
Check		77	137	23	142	117
Wex	8	70		22		
Wex	16	71	137	25	139	107
MGJ-A	16		132		140	113
Significance (.05)		ns	ns	ns	ns	ns

* 1976 yield lost due to hail damage.