1974). In general there were great varietal differences with respect to the concentrations of the minerals (Mg, Ca, Zn, and Fe) determined in this study. It should be pointed out, however, that the choice of agricultural practices might prove to be much more effective in decreasing the phytate content of the grain than the choice of variety. There was no significant difference in phytate P concentrations among the varieties in 1972, when average phytate P concentration was high, most probably due to higher availability of P to the grains. In fact average phytate P concentration of the wheat varieties in 1972 (284 mg/100 g) was about one and a half times that in 1973 (177 mg/100 g).

Reinhold (1971) has attributed the difference in phytate concentrations of Iranian breads in Fars area mainly to the method of baking. The present study indicates that the differences might also be attributed to the environmental conditions under which wheat is grown. Thus, it can be expected to find significant yearly variations of phytate concentration in the village breads.

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Atomic Absorption Spectrometric Determination of Eight Trace Metals in Orange Juice following Hydrolytic Preparation

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The further application of acid hydrolysis as a preparative procedure for orange juice is described. The hydrolysate is a useful matrix for the flame atomic absorption determination of eight metals, Ca, Cu, Fe, K, Mg, Mn, Na, and Zn. Precision studies are described, and the contents of these eight elements in eight different orange juice samples from Florida are given.

A recent publication by the authors (McHard et al., 1976) describes a hydrolysis procedure for the preparation of orange juice for the atomic absorption spectrometric determination of calcium. It was predicted that the procedure would be useful for the determination of several other elements as well. This prediction has been verified by the work presented in this paper.

Of the principal elements found in the analysis of plants, only eight—calcium, copper, iron, magnesium, manganese, potassium, sodium, and zinc—are readily determinable at the levels encountered in orange juice by common flame atomic absorption or flame emission methods. A recent

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Table I. Concentrations (in Micrograms per Milliliter) of Eight Metals in Eight Brands of Florida Orange Juice[a]

Element	Orange juice brand								Range	Mean
	1	2	3	4	5	6	7	8		
Calcium	91	87	88	85	88	97	97	105	85-105	92.3
Copper	0.31	0.26	0.29	0.32	0.28	0.29	0.33	0.25	0.25-0.33	0.29
Iron	0.74	0.73	0.91	1.67	2.45	1.13	0.92	1.18	0.73-2.45	1.22
Magnesium	109	109	100	100	98	108	115	109	98-115	106
Manganese	0.12	0.12	0.11	0.13	0.12	0.11	0.13	0.11	0.11-0.13	0.12
Potassium	1900	1775	1800	1900	1800	1900	1925	1900	1775-1925	1863
Sodium	3.45	2.95	2.10	2.65	5.0	8.95	5.25	4.05	2.10-8.95	4.30
Zinc	0.36	0.37	0.37	0.38	0.33	0.38	0.37	0.36	0.33-0.38	0.37

[a] Values are based on single strength orange juice.

publication (Isaac and Johnson, 1975) describes the determination of seven of these elements as a collaborative study involving a number of interested laboratories. Dry ashing and wet ashing were compared and no significant differences were found in the results.

Dry ashing and wet ashing are time consuming. Hydrolysis of orange juice by strong acid, which is suitable for preparation of many samples in a short time, was the method of choice of sample preparation for the metal analyses reported in the present work.

EXPERIMENTAL SECTION

Apparatus. The single beam spectrometer cited in the previous study (McHard et al., 1976) was used in this work. Instrument parameters and wavelengths specified in the manufacturer's guide (Instrumentation Laboratory, 1972) were followed. Intensity readings were taken from the digital readout system supplied with the instrument. Three multielement lamps were used, i.e., a Ca, Mg, and Al hollow cathode lamp for Ca and Mg, a Fe, Cu, Co, Cr, Mn, Ni, and Zn multielement hollow cathode lamp for Fe, Cu, Mn, and Zn, and a Na and K hollow cathode lamp for Na and K. All three lamps were purchased from Varian Techtron, Palo Alto, Calif.

Reagents. Analytical grade reagents were used in preparing all standard solutions. Magnesium, iron, copper, and zinc as metals were dissolved in acid and diluted to volume. Sodium, potassium, and calcium standard solutions were prepared from the dry carbonates. The manganese standard was prepared from potassium permanganate and then reduced to Mn^{II} with oxalic acid before diluting to final volume. A mixed standard was made from the above reagents to correspond to the following concentrations: 10 000 ppm of K, 1000 ppm each of Ca and Mg; 100 ppm of Na; 4 ppm each of Cu, Mn, and Zn; and 10 ppm of Fe. Other reagents were the same as described in the prior publication (McHard et al., 1976).

Standards simulating orange juice were prepared by making a series of dilutions of 1, 2.5, 5, and 7.5 ml of the above mixture of elements plus dextrose and phosphoric acid in amounts approximately equivalent to those expected in a 10-g sample of frozen concentrated orange juice (FCOJ). In most instances, these dilutions corresponded to the useful working range for the elements under consideration. They were processed through the hydrolysis procedure exactly like the orange juice samples.

Procedure. Individual 10-g samples of FCOJ were weighed into 100-ml volumetric flasks. Ten-milliliter portions of 10 M nitric acid were added, one portion to each flask. The mixtures of sample and acid were agitated slowly by rotating the flasks by hand for a few seconds. The flasks were then allowed to stand overnight in a bath of room temperature water. The cooling effect of the water bath was necessary to prevent excessive foaming during the initial stages of the hydrolysis procedure. The following morning, the bath with flasks was heated to 80 °C for 5 h. At the end of the heating period, the flasks and contents were allowed to cool to room temperature and diluted to volume (100 ml) with deionized water. The solids remaining were removed by filtering the flask's contents through rapid filtration paper into clean, dry plastic bottles. Dilutions of the filtered matrices were made 1/1 with water for the determination of Cu, Fe, Mn, and Zn. For the measurement of Ca, Mg, Na, and K, dilutions were made 40/1. All dilutions were made with deionized water. All glassware and containers were acid washed prior to use.

RESULTS AND DISCUSSION

The procedure described here follows that described in the authors' prior paper (McHard et al., 1975). Ten molar HNO_3 was found to be an effective hydrolysis agent and was easier to use than the previously recommended concentrated HNO_3. In order that the number of dilutions could be minimized, the working ranges for calcium and magnesium were adopted as 1–2 ppm. Because it was desired to determine sodium and potassium in this same solution, working ranges of 0.02–0.15 ppm and 4–30 ppm, respectively, were decided on. A 40/1 dilution of the filtered, prediluted matrix was required to reach these working ranges as mentioned in the Procedure Section. For copper, iron, manganese, and zinc, the dilution of the filtered matrix was 1/1, and the working ranges were 0.1–0.75 ppm for Cu, Mn, and Zn while that for Fe was 0.25–2.5 ppm.

Figure 1 shows the plots of the analytical curves for the eight metals. The graphs have the coordinates labeled so that the arbitrary intensity values convert directly to concentrations based on single strength orange juice (SSOJ) without any necessity for dilution corrections. It will be noted that the graphs for sodium, manganese, and zinc are all straight lines. Copper deviates slightly from linearity at the higher concentrations, but there is noticeable deviation from linearity in the plots for iron, magnesium, and calcium. The analytical graph for potassium at low concentrations was curved, but in the analytical range used for analysis, the graph was linear as shown in Figure 1.

The data in Table I show the concentrations measured for the eight elements in eight different consumer brand frozen concentrates. These eight juices are labeled 1 through 8. In all instances, the range of values is rather narrow. This is especially true for magnesium, manganese, and zinc.

The precision of measurement (see Table II) using the procedure described here was determined. Ten separate samples of one of the orange juices brands were carried through the hydrolysis procedure and, using the variation of the signal response, coefficient of variance (CV) figures were obtained. The signal response values given represent the average of three consecutive digital readings. To obtain these consecutive readings, the aspirator tube to the ne-

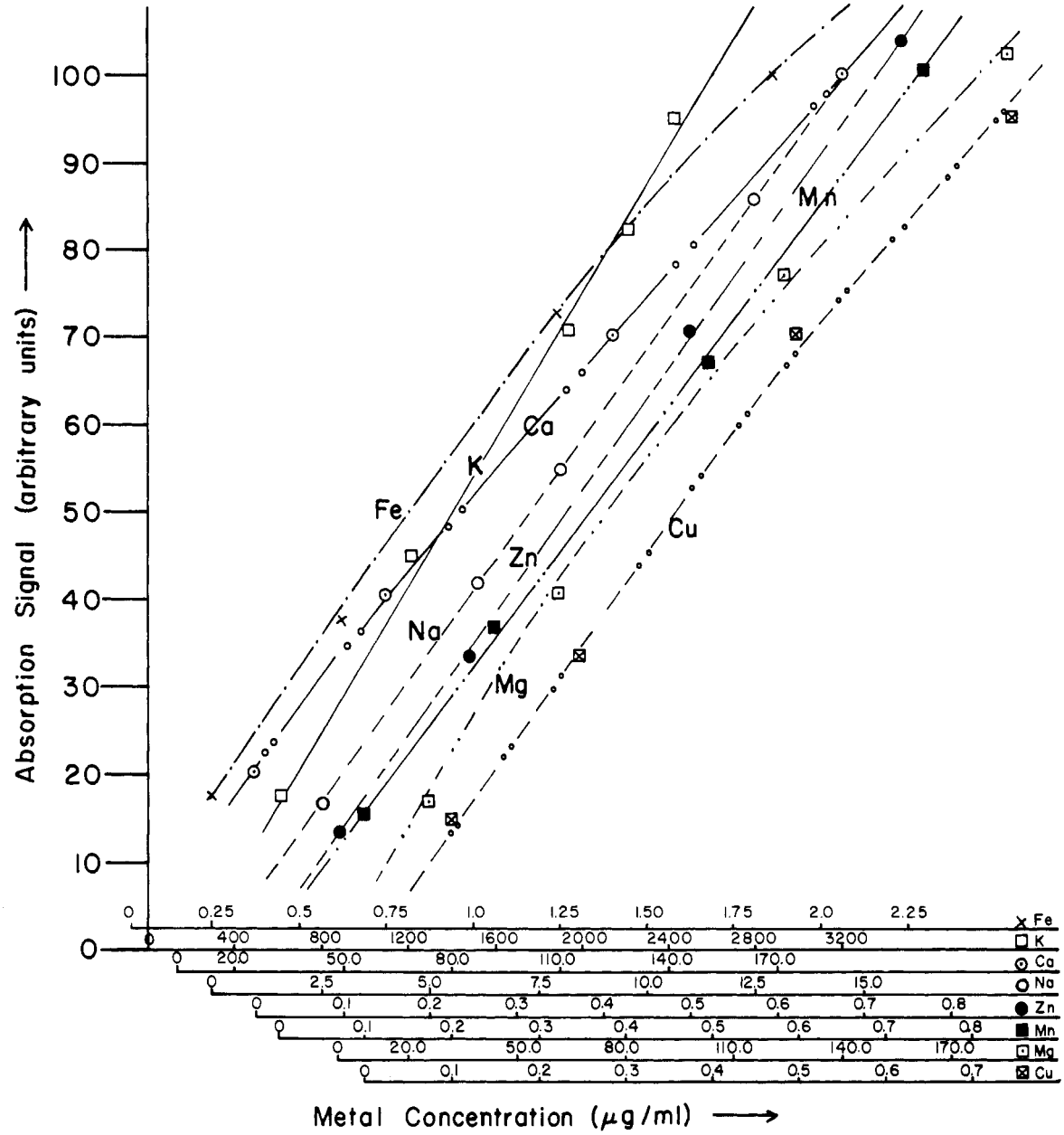

Figure 1. Analytical curves for Fe, K, Ca, Na, Zn, Mn, Mg, and Cu orange juice matrix. Abscissa reads directly in concentration units of single strength orange juice.

Table II. Precision of Measurements for Eight Metals in Orange Juice[a]

Replicate	Calcium	Copper	Iron	Magnesium	Manganese	Potassium	Sodium	Zinc
1	100	76	177	601	87	101	137	101
2	100	79	169	601	78	96	136	99
3	98	78	173	600	78	97	133	98
4	98	78	169	588	84	98	136	100
5	99	79	174	588	83	101	140	100
6	97	74	169	580	85	97	132	102
7	97	79	171	584	83	99	132	101
8	94	77	168	584	86	101	130	102
9	102	80	175	593	86	103	136	108
10	93	76	169	580	85	102	133	103
$\bar{X}$	97.8	77.6	171.4	590	83.5	99.5	134.5	101.4
CV	2.80	2.37	1.83	1.42	3.80	2.43	2.22	2.72

[a] $\bar{X}$ = average value of 10 replicate samples; CV = coefficient of variance. Note: Intensity readings are on an arbitrary scale and have not been converted to units of concentration.

bulizer of the burner assembly on the atomic absorption spectrometer was dipped into the sample, a reading taken and, following a water rinse in each case, two more readings were taken in the same manner. An average of these three readings was taken as the intensity value for that replicate.

From the values for CV, one can observe that the precision is best for magnesium and poorest for manganese. This is to be expected because magnesium is present in

relatively large amounts (~100 ppm) and gives an intense signal, whereas manganese is present at the lowest concentration of all eight metals (see Table I). All the precision values are considered to be in an acceptable range.

SUMMARY

It has been demonstrated that the hydrolysis procedure proposed for the determination of calcium in orange juice can be used also for the determination of seven other metals by flame atomic absorption spectrometry. Concentration values are compared for eight commercial brands produced and sold in Florida.

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Identification of Some Volatile Compounds in Cooked Chicken

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A fraction isolated from boiling chicken broth by continuous steam distillation and pentane extraction was analyzed by gas–liquid chromatography/mass spectrometry (GLC/MS) with both packed and open tubular columns. Some of the compounds separated by gas–liquid chromatography (GLC) were trapped and identified by mass spectrometry and infrared analysis. In addition, headspace vapor above boiling chicken broth was analyzed for gaseous compounds by gas–solid (GSC) chromatography. Of the 53 compounds identified, 30 had not been previously identified in cooked chicken. The 53 included sulfur compounds, aldehydes, alcohols, amines, acids, 2-alkylfurans, ketones, hydrocarbons (cyclic and acyclic), alkylbenzenes, a terpene, and a nitrile.

A recent review by Wilson and Katz (1972) of compounds isolated from cooked chicken meat revealed that 136 volatile compounds had been identified up to the time of its publication. Janney and coworkers (1974) in a subsequent publication reported identification of an additional six volatile compounds from fried chicken. Also, Harkes and Begemann (1974) identified 11 previously unreported unsaturated aldehydes from chicken broth. In spite of the large number of compounds identified from cooked chicken, no single compound or mixture of compounds having a cooked chicken aroma has been found. However, no serious attempt has been made to determine the aroma of mixtures of these compounds at various concentrations. Apparently the volatiles of cooked chicken constitute an exceedingly complex mixture of organic compounds and require further study.

Since the yield of volatile material from cooked chicken meat is quite low, gas–liquid chromatography/mass spectrometry (GLC/MS) was chosen as the principal analytical technique to give the maximum amount of information for identification of the components.

EXPERIMENTAL SECTION

Isolation of Meat Volatiles. Processed ice-packed ready-to-cook frying chickens weighing 2.5–3 lb were obtained locally. They were immediately bagged in polyethylene and stored at 4 °C until used. Meat from the leg, breast, and thigh was separated from skin, fat, and bone and then cut into small pieces (about 1-cm^3 cubes). One chicken yielded about 400–450 g of meat. Meat (400 g) was placed in a 3-l. round-bottomed flask and 425 ml of distilled water added. The flask was connected to a Likens and Nickerson-type (1964) steam distillation, continuous pentane extraction apparatus. Pentane and chicken broth were boiled for 8 h. After the apparatus had cooled, the flask containing the pentane, 120 ml, was removed. The extract was maintained at 40 °C and was concentrated to about 0.5 ml by blowing a gentle stream of high purity nitrogen on its surface. The pentane extract was further concentrated to about 50 μl by allowing it to stand at room temperature. This procedure undoubtedly favored the concentration of higher boiling point compounds and the loss of lower boiling point compounds.

Broth for Headspace Analysis. One frying chicken was partially thawed and cut up as described in the section Isolation of Meat Volatiles. The meat and 425 ml of distilled water were placed in a 3-l. three-necked flask. The flask was equipped with a thermometer for measuring broth temperature, a water cooled condenser, and a silicone rubber septum for withdrawal of headspace samples. Meat was boiled for 2 h, then the temperature of the broth was reduced to 72 °C and held constant for a 2-h period, during which headspace samples were withdrawn for GSC analysis.

Analytical Methods. In this research, the approaches used in an attempt to identify the volatile compounds from cooked chicken meat were: analysis of headspace vapors of chicken broth by GLC; GLC separation of components of the pentane extract and collection of fractions for mass spectrometric and infrared analysis, and GLC/MS analysis by use of a 500 ft × 0.03 in. i.d. open tubular column, and a 500 ft × 0.02 in. open tubular column, each coated with a different stationary phase, and a 12 ft × $^1/_8$ in. packed

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