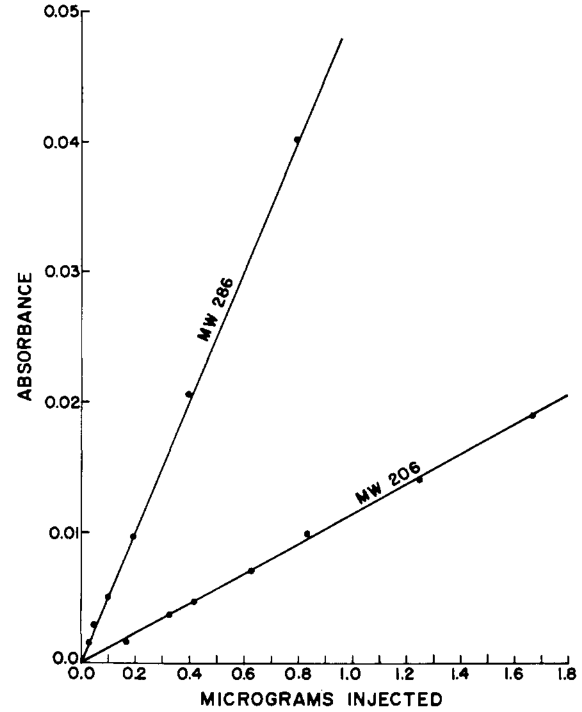

Figure 5. HPLC calibration curves for mol wt 286 and 206 compounds.

hr, indicated that the mol wt 206 compound gradually increased to 26% (8 hr), whereas aflatoxin D_1 reached a maximum of 23% (4 hr) and then gradually decreased to 3% (8 hr). The quantitative values reported here for aflatoxin D_1, mol wt 286, are not in good agreement with data obtained by Lee et al. (1974), who reported about 30% of D_1 in the crude product from 1-hr ammoniation of aflatoxin B_1. However, their data, obtained by absorptivity measurements on the chloroform-soluble fraction of the crude product, were undoubtedly subject to interferences.

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Rapid Screening Determination of Nitrate in Baby Food Using the Nitrate-Selective Electrode

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A nitrate-selective electrode was used to rapidly determine the nitrate nitrogen content of baby food. Interferences were eliminated by the use of two cation resins (Al and Ag). Of 53 samples analyzed, five (green beans, garden vegetables, spinach, squash, and beets) were over 20 ppm of nitrate nitrogen. The method is fast (15–20 min) but appears to read a few parts per million high for samples having a nitrate nitrogen content less than about 10 ppm.

The determination of nitrate in baby foods is important because infants are particularly susceptible to nitrate poisoning (Kamm et al., 1965). The current method (Kamm et al., 1965) is a colorimetric method requiring 2 to 3 days to do both nitrate and nitrite. The paper discusses the application of the nitrate-selective electrode method of Paul and Carlson (1968) to the analysis of baby food. The method reported here takes only about 10–20 min and was designed for use as a rapid-scanning method to identify samples over about 20 ppm of nitrate nitrogen. Such samples could then be analyzed by the more accurate method of Kamm et al. (1965).

EXPERIMENTAL SECTION

Apparatus used included: nitrate ion electrode Model 92-07, Orion Research, Cambridge, Mass.; a pH meter with an expanded millivolt scale.

Samples. All baby food samples were Gerber's brand (an arbitrary choice) purchased at a commercial grocery. Samples were taken directly from the jar.

Reagents; Nitrate Standards. Knowns of 1, 4, 9, 18, and 45 ppm of nitrate nitrogen were made in 0.01 *N* KH_2PO_4 using sodium nitrate.

Resins. Both resins were Dowex 50W-X8 cation exchange resin. Conversion to the appropriate form was

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Table I. Nitrate Nitrogen Content of Baby Food[a]

Category	Food	Av ppm of nitrate nitrogen
Fruits	Prunes with tapioca (3)	4 ± 2
	Pears* (2)	6 ± 0
	Pears and pineapple* (2)	6 ± 0
	Apricots with tapioca* (2)	6 ± 0
	Bananas with pineapple and tapioca (3)	7 ± 1
	Plums with tapioca (2)	7 ± 0
	Applesauce (2)	8 ± 1
	Applesauce and apricots (2)	8 ± 1
	Peaches (2)	12 ± 0
	Bananas with tapioca (2)	13 ± 2
Desserts	Blueberry buckle (2)	3 ± 1
	Raspberry cobbler* (2)	4 ± 1
	Dutch apple dessert (2)	6 ± 0
	Fruit dessert with tapioca (4)	6 ± 2
	Peach cobbler (2)	6 ± 1
Puddings	Cherry vanilla (4)	7 ± 2
	Vanilla custard (3)	9 ± 1
	Butterscotch (3)	10 ± 1
	Chocolate custard (3)	13 ± 1
Cereals	High protein cereal with applesauce and bananas (2)	8 ± 0
	Oatmeal with applesauce and bananas (2)	8 ± 0
	Rice cereal with applesauce and bananas (2)	11 ± 1
	Mixed cereal with applesauce and bananas (2)	12 ± 1
Egg and dairy	Egg yolks* (2)	5 ± 0
	Cottage cheese (4)	8 ± 2
	Egg yolks and ham (2)	17 ± 1
Meats	Beef liver (2)	8 ± 0
	Veal (3)	8 ± 3
	Ham (2)	9 ± 1
	Beef (2)	10 ± 1
	Lamb (2)	10 ± 0
	Pork (2)	10 ± 1
	Chicken (2)	11 ± 1
Dinners	Cream of chicken soup (3)	7 ± 1
	Vegetables and chicken (2)	8 ± 0
	Vegetables and turkey (2)	8 ± 0
	High meat (2)	9 ± 0
	Macaroni-tomato with beef (3)	9 ± 3
	Turkey rice with vegetables (2)	10 ± 0
	Chicken noodle dinner (3)	11 ± 1
	Vegetables and liver (2)	11 ± 1
	Vegetables and beef (3)	13 ± 4
	Vegetables and lamb (2)	14 ± 1
Vegetables	Creamed corn* (2)	12 ± 1
	Mixed vegetables* (3)	12 ± 1
	Sweet potatoes* (2)	13 ± 0
	Peas* (3)	14 ± 2
	Carrots* (5)	15 ± 1
	Green beans (4)	41 ± 3
	Garden vegetables (4)	51 ± 3
	Spinach (3)	117 ± 4
	Squash (2)	215 ± 5
	Beets (3)	483 ± 22

[a] Asterisk denotes samples requiring extra Al resin. Numbers in parentheses are number of determinations. Plus or minus numbers are the average deviation from the average, always rounded upward.

achieved by equilibration with either $Al_2(SO_4)_3$ or $AgNO_3$. Excess salt was removed by washing with deionized water. The resin was air-dried for use.

Procedure. Weigh out 20 g of baby food into a 150-ml beaker, use a spatula, and add about 3 g of Al resin and 2 g of Ag resin (the exact amounts seem not to be critical). Add sufficient water to dilute the sample to read on scale, usually 20, 60, or 80 ml. A graduated cylinder was found to be sufficiently accurate. Stir well for 3–4 min and then measure standards and unknowns under constant stirring conditions. Electrode response appeared not to be disturbed by the fairly "thick" solutions of baby food. Samples marked with an asterisk in Table I required extra Al resin; an excess of Al resin was required. After an unknown had been measured, an additional spatula full of this resin was added until the meter needle came to its lowest value.

RESULTS

Nitrate Recovery. Green beans were used for recovery studies. The recovery of nitrate from green beans was 104% (47 ppm found, 45 ppm added) in three independent trials.

Reproducibility. Multiple determinations were made on all of the baby foods, many at different dilutions. Carrots, which were high in NO_3^- compared with the majority of the items and hence gave the largest spread were used for these studies. Five samples of carrots were run, giving values of 15, 15, 15, 15, and 14 ppm of nitrate nitrogen. In general, vegetables showed the greatest reproducibility, with fruits and desserts next, while meats and meat-containing products were the poorest.

Nitrate Levels. Values of baby food samples are given in Table I. These values are the average of at least two determinations, three or more being done if a difference of more than 2 ppm was found.

DISCUSSION

The averages of all samples and the average deviation from the average are reported in Table I. No samples were less than 3 ppm of nitrate nitrogen. Only 8 of the 53 samples had an average deviation of 3 or more ppm of nitrate nitrogen. However, 16 samples showed zero deviation which indicates that all determinations gave exactly the same value (since all rounding was done upward). From Table I and the reproducibility studies it would appear that the values obtained for any given sample are quite consistent. The greatest difficulty in obtaining reproducibility (±22 ppm of nitrate nitrogen) was found with beets which were also highest in nitrate nitrogen (483 ppm).

Both Tables II and III are designed to compare the values of this paper to the only known reported values of nitrate nitrogen content of baby food (Kamm et al., 1965). In Table II samples are grouped by type of food and the two sets of values compared based on number of samples analyzed, range of values obtained, and average value. It should first be noted that Kamm et al. ran several samples. The range and average of values from this paper are generally higher than those of Kamm et al. This fact and the fact that no values less than 3 ppm of nitrate nitrogen were found strongly suggest that the values reported here run 3–5 ppm high. Unpublished data from other laboratories using this technique also tend to run slightly high. The recovery for this method (104%) was quite good and may account for the high values when comparing to the data of Kamm et al., since they report lower recoveries.

The major thrust of this work, however, was to find a rapid method of identifying samples over 20 ppm of nitrate nitrogen. Comparing samples this paper found over 20 ppm to those of Kamm et al. (Table III); it can be seen that the method appears accurate for this purpose. This paper found no samples over 20 ppm of nitrate nitrogen that were not also found that high by Kamm et al. In fact, two samples found that high by Kamm et al. were found to be lower.

Differences in samples do occur. Two jars of green beans

Table II. Comparison to Published Values of Infant Foods by Category

Food	This paper			Kamm et al. (1965)		
	No. of samples	Nitrate nitrogen, ppm		No. of samples	Nitrate nitrogen, ppm	
		Range	Av		Range	Av
Fruits	10	4-13	8	22	0-7	2
Desserts	5	3-6	5			
Puddings	2	7-10	9	5	0.8-11	4
Cereals	4	8-12	10	22	0.4-48	13
Egg products + custards	4	5-17	11	13	0.9-11	4
Dairy products	1		8	9	0-7	3
Meats	7	8-11	10	15	1-11	4
Meat dinners	10	8-14	10	20	0.9-16	7
Vegetables	10	12-483	97	61	0.2-492	68

Table III. Comparison to Published Values of Infant Foods over 20 ppm Nitrate Nitrogen

Food	This paper, nitrate nitrogen, ppm	No. of samples	Kamm et al. (1965) nitrate nitrogen, ppm	
			Range	Av
Green beans	41	3	16-71	37
Garden vegetables	51	5	19-62	41
Spinach	117	5	244-379	312
Squash	215	5	10-93	64
Beets	483	6	144-492	222
Mixed vegetables	(12)	2	21-24	22
Carrots	(15)	8	15-38	23
Graham crackers	Not tested	1		48
Wax beans	Not tested	2	73-129	101

(different lot numbers) were found to contain 41 and 80 ppm of nitrate nitrogen. This could be caused by a difference either in the amount of water added or the nitrate content of the beans themselves. While the cause of the difference is of interest, the real concern is with the nitrate nitrogen content of the food as it is consumed. It is the authors' experience that most baby food is consumed directly from the jar.

Vegetables are generally found to be highest in nitrate nitrogen. A survey of nitrate content of vegetables by Boswell et al. (1967) also showed fairly high values for some of the same vegetables reported here. Boswell et al. also note that the nitrate content of vegetables is influenced by such factors as plant variety, maturity at harvest, climate, geography, and fertilization. These factors could account for the large differences found in different samples of the same vegetable.

Since the development of this method, another method using the nitrate electrode has come to the attention of the authors. The method of Milham et al. (1970) employs a buffer to eliminate interferences and would appear to also be adaptable for a rapid-screening method for baby food.

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Preparation and Characterization of Some Long-Chain Ammonium Polyphosphates

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The preparation and characterization of four polymorphic forms of long-chain ammonium polyphosphates and an ammonium hydrogen polyphosphate are described. Single-crystal data are given for three of the polymorphs and for the new ammonium hydrogen polyphosphate salt.

Although crystalline long-chain ammonium polyphosphates are not produced as fertilizers, their high nutrient content and apparent complete availability as plant food make them interesting as potential fertilizer materials. Their reactions with the soils need further study.

The crystalline long-chain ammonium polyphosphates are similar in many respects to the well-known sodium and potassium Kurrol's salts. Thilo and Dostál (1959) showed that compounds with the general composition ABX_3 tend to exist in several polymorphic forms. Both the ammonium and alkali metal long-chain polyphosphates belong to this group and occur in several polymorphic modifications. Most of the earlier work was with the alkali metal polyphosphates. Four temperature-dependent polymorphs of KPO_3 (Von Jost and Schulze, 1971) and three forms of $NaPO_3$ (Inorganic Index to the Powder Diffraction File, 1972) are known to exist. Structural analysis of these and a series of other polyphosphates with metaphosphate composition have shown that the polyphosphate anion consists of interconnected PO_4 tetrahedra forming "infinite" linear polyphosphate chains. Altogether, six different structural arrangements of the polyphosphate chains have been determined (Thilo, 1965).

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