

TABLE I-continued

	K	Ca	Na	Mg	Li
Peach Leaves (0.070 mg/mL)					
C ( $\mu\text{mol/L}$ )	40.2 $\pm$ 0.5	7.9 $\pm$ 2.2	<sup>c</sup>	13.5 $\pm$ 0.7	
recovery <sup>b</sup> (%)	92.4 $\pm$ 9.4	29.0 $\pm$ 8.6		108.5 $\pm$ 12.4	
RSD <sup>a</sup> (%)	1.31	22.6		1.79	
Citrus Leaves (0.065 mg/mL)					
C ( $\mu\text{mol/L}$ )	28.9 $\pm$ 0.3	13.2 $\pm$ 0.5	<sup>c</sup>	14.3 $\pm$ 0.7	
recovery <sup>b</sup> (%)	95.5 $\pm$ 10.1	25.9 $\pm$ 2.9		92.2 $\pm$ 11.3	
RSD <sup>a</sup> (%)	0.75	1.44		0.72	

<sup>a</sup>Relative standard deviation.

<sup>b</sup>Apparent recovery<sup>17</sup> calculated as the ratio of the slope measured for the standard addition curve to the slope measured for the calibration curve with sample buffer.

<sup>c</sup>Apparent recovery<sup>17</sup> calculated as the ratio of the concentration measured using GEMBE to the expected concentration calculated from the SRM certificate and assuming complete dissolution of the analytes.

<sup>d</sup>Value calculated using the calibration curve obtained with sample buffer.

<sup>e</sup>Value below the LOD.

Sample buffer alone with known concentrations of analytes (excluding melamine) was used to characterize the experimental apparatus, optimize the separation parameters, and construct calibration curves. As shown in FIG. 15A, measurements were taken of sample buffer with various concentrations (3, 10, 30, 100, and 300  $\mu\text{mol/L}$ ) of each of the inorganic cations potassium, as indicated by "A", calcium, as indicated by "B", sodium, as indicated by "C", magnesium, as indicated by "D", and lithium, as indicated by "E". As shown in FIG. 15B, the data of FIG. 15A was converted to peaks by taking the derivative of the detector signal with respect to time. Quantitative data analysis was performed using Mathematica (Wolfram Research, Champaign, Ill.). Data was binned into  $\sim 0.2$  s increments for derivation and semi-automated peak finding. The original data, unbinned and undifferentiated was then fit to an error function and a linear offset to account for background signal over a span of time containing the step width on either side of the step. Potassium, lithium, and melamine steps were fit individually, while calcium, sodium, and magnesium steps were fit simultaneously to the sum of these error functions and a linear offset.

#### Whole Milk Results.

No effort was made to avoid pipetting the irregular particles present in the whole milk. A standard addition method was used to assess any matrix effects and provide quantitative measurement of the cation content of the milk. As shown in FIG. 16, depicted in graphs "a", measurements were taken at 10, 20, and 30  $\mu\text{mol/L}$  of each analyte (except lithium and melamine) added to the sample solution. The results are summarized in Table I. Apparent recoveries were calculated as the ratio of the slope measured for the standard addition curve for the milk to the slope measured for the calibration curve with sample buffer. Typical apparent recoveries were within 3 standard deviations of 100% (with the exception of magnesium) indicating minimal interference with diluted milk.

#### Dirt, Estuarine Sediment, and Coal Fly Ash Results.

As shown in FIG. 16, depicted in graph "b", measurements were taken at 0, 5, 10, and 15  $\mu\text{mol/L}$  of each analyte (except lithium and melamine) added to the dirt solution. The results are summarized in Table I. Potassium, calcium, sodium, and magnesium were present in the dirt at measurable concentrations. Apparent recoveries were calculated as the ratio of the slope measured for the standard addition curve for dirt to the slope measured for the calibration curve with sample buffer. Standard errors for the measured concentrations and recoveries were greater than those for milk, likely due to the greater heterogeneity of the dirt samples.

As shown in FIG. 16, depicted in graph "c", measurements were taken at 0, 5, 10, and 15  $\mu\text{mol/L}$  of each analyte (except lithium and melamine) added to the estuarine sediment solution. Additionally, as shown in FIG. 16, depicted in graph "d", measurements were taken at 0, 5, 10, and 15  $\mu\text{mol/L}$  of each analyte (except lithium and melamine) added to the coal fly ash solution. The results are summarized in Table I. The concentrations were determined using the mean step heights and the calibration curve measured for the sample buffer. Comparison between the measured values and the SRM certificates shows that GEMBE detected approximately  $\frac{1}{2}$  of the potassium and the calcium, approximately  $\frac{2}{3}$  of the sodium, and approximately  $\frac{1}{4}$  of the magnesium that constitute the estuarine sediment and approximately all of the calcium and sodium and approximately  $\frac{2}{3}$  of the magnesium content of the coal fly ash. These results suggest that much of the potassium, calcium, sodium, and magnesium in the estuarine sediment and coal fly ash remained undissolved or otherwise unavailable for detection using GEMBE.

#### Leaves Results.

The tomato, peach, and citrus leaves suspended in sample buffer were analyzed in triplicate for the presence of potassium, calcium, sodium, and magnesium. As shown in FIG. 16, depicted in graph "e", measurements were taken at 0, 5, 10, and 15  $\mu\text{mol/L}$  of each analyte (e.g. potassium, calcium, sodium, and magnesium) added to the tomato leaves in sample buffer. Additionally, as shown in FIG. 16, depicted in graph "f", measurements were taken at 0, 5, 10, and 15  $\mu\text{mol/L}$  of each analyte (e.g. potassium, calcium, sodium, and magnesium) added to the peach leaves in sample buffer. Also, as shown in FIG. 16, depicted in graph "g", measurements were taken at 0, 5, 10, and 15  $\mu\text{mol/L}$  of each analyte (e.g. potassium, calcium, sodium, and magnesium) added to the citrus leaves in sample buffer. The results are summarized in Table I.

Apparent recoveries were calculated as the ratio of the concentration measured using GEMBE and the expected concentration calculated from the SRM certificate, along with the assumption of complete dissolution of these elements in to the sample buffer. Comparison to the SRM certificates revealed that approximately all of the potassium and magnesium, and approximately  $\frac{1}{3}$  (peach and citrus) or approximately  $\frac{2}{3}$  (tomato) of the calcium present in the samples was dissolved and measurable using GEMBE.

It is noted that terms like "preferably," "generally," "commonly," and "typically" are not utilized herein to limit the scope of the claimed invention or to imply that certain features are critical, essential, or even important to the structure