

## ASCORBIC ACID DETERMINATION IN PROCESSED POTATOES

M.J. Egoville<sup>1</sup>, J.F. Sullivan<sup>2</sup>, M.F. Kozempel<sup>1</sup>, W.J. Jones<sup>1</sup>**Abstract**

A method to determine ascorbic acid at any stage of potato processing was developed. Potato samples are frozen in dry ice, lyophilized and analyzed using a modified spectrophotometric method. This method is simple, accurate, inexpensive and has a detection limit of 1.2 mg ascorbic acid/100 g dry weight potatoes.

**Compendio**

Se desarrolló un método para determinar el ácido ascórbico en cualquiera de las etapas del procesamiento de papa. Muestras de papa son congeladas en hielo seco, liofilizadas y analizadas utilizando un método modificado de espectrofotometría. Este método es sencillo, preciso, y de bajo costo y tiene un límite de detección de 1,2 mg de ácido ascórbico/100 g de peso seco de papas.

**Introduction**

Potato ranks first among all vegetable crops consumed by Americans. The per capita consumption of potatoes is 110-120 pounds (5), supplying approximately  $\frac{1}{4}$  of the daily requirement of ascorbic acid (AA) (6). Potatoes are the leading processed vegetable crop (4) and as such, the effects of processing on the nutrient content of potatoes are of great concern to the process industry.

Ascorbic acid (Vitamin C), in particular, is of interest due to reports (1, 2, 8, 15) concerning the loss of this important nutrient during various processing steps. Difficulties in monitoring AA levels during process stages are encountered because of the susceptibility of AA to atmospheric oxidation (3). This further complicates sample handling and is a hindrance to accurate and reliable determinations of AA containing foods. It was therefore our purpose to develop a simple, rapid and accurate procedure which could be used to monitor AA concentrations throughout all phases of a potato processing operation.

Potatoes are reported to contain very low indigenous levels of dehydroascorbic acid (DHA) (14), which is further reported to be quantitatively lost

during processing/cooking (9). Since this procedure is intended for use in the process industry, our efforts were focused solely on AA determinations and not DHA.

In this report data are presented regarding sampling, storage and modification of existing methodology related to the determination of AA in potato and potato products.

### Materials and Methods

*Potatoes*—Maine Russet Burbank and Maine Kennebec potatoes were used in this study.

*Apparatus*—Perkin—Elmer Coleman Model 55 UV—Visible Spectrophotometer with a sipper cell having a path length of 1 cm was used.

*Reagents*—Reagent grade chemicals were used for analysis. The extracting solution was 0.4% oxalic acid containing 20% acetone (11). To a solution of 4.0 gm oxalic acid dissolved in 500 ml H<sub>2</sub>O, 200 ml acetone was added and diluted to 1 L with H<sub>2</sub>O. The pH was adjusted to 1.1 with concentrated H<sub>2</sub>SO<sub>4</sub> (12). A stock solution of 2,6 Dichloroindophenol (DCIP) was prepared by dissolving 100 mg of dye in 100 ml warm H<sub>2</sub>O, and adding 84 mg NaHCO<sub>3</sub>. This was diluted to 500 ml with H<sub>2</sub>O and filtered. The DCIP stock solution was further diluted with H<sub>2</sub>O such that when 1 ml of extracting solution and 9 ml of DCIP solution were reacted, an absorbance value (at 520 nM) of 0.300 to 0.350 was obtained (10). Ascorbic acid (AA) standard stock solution consisted of 100 mg AA per 100 ml of extracting solution.

*Ascorbic Acid Standard Curve*—Appropriate aliquots of stock AA solution (1.0 ml to 5.0 ml), pipetted into 100 ml volumetric flasks containing extracting solution, yielded concentrations of 10.0 to 50.0 µg/ml. One ml aliquots of these dilutions were analyzed using the modified colorimetric method detailed below.

*Sampling Procedure*—To develop the sampling procedure, our study was performed using mashed potatoes from a pilot plant potato flake process (15).

The samples of mashed potatoes were immediately immersed in a bed of powdered dry ice to quench ascorbic acid oxidation (8). After 30-60 minutes a portion of the frozen mass was placed in lyophilizing jars and dried under vacuum (<100 µ pressure). After drying (3 days) the lyophilized samples were ground in a Wiley Mill to pass through a 20-mesh screen and stored in a -16 C freezer until analyzed. Another portion of the frozen mass was stored, w/o lyophilizing in a freezer (-16 C) until analyzed.

*Analytical*—Moisture content was determined by drying the samples to constant weight in a vacuum oven at 70 C, for 16-18 hours (13).

A modified spectrophotometric method of Loeffler and Ponting (10) was used to measure reduced AA in potato samples. One gram of lyophilized

sample was extracted for 5 min with 20.0 ml of extracting solution. Since the cooked and mashed samples contained about 80% water, 5 grams of frozen, (unlyophilized) sample and 16.1 ml extracting solution were used for extraction. This gave approximately the same ratio of potato solids to extracting medium as used for the freeze-dried samples or flake samples.

The solutions were filtered through Whatman No. 2V filter paper or equivalent. To correct for any turbidity in the potato mixture a blank of 1 ml filtered sample and 9 ml H<sub>2</sub>O was prepared and the absorbance measured at 520 nM. Additional 1 ml samples were pipetted into test tubes (18 x 150 mm), 9 ml of DCIP solution was added and the solution gently mixed. A reaction time of 45 seconds was begun when the DCIP solution was added to the sample; afterward the resultant pink chromogen was measured. The absorbance of the sample was corrected for the sample blank. This value (L<sub>2</sub>) was subtracted from the absorbance value obtained when 1 ml of extracting solution was reacted with 9 ml of DCIP solution (L<sub>1</sub>). The  $\Delta L$  value was used to determine the concentration of ascorbic acid in the sample by referring to the standard curve.

*Spiking Experiments*—Varying amounts of AA were added to batches of potato mash and analyzed following the above procedure. To test the repeatability of the method, 45 kg batches of mashed potatoes were used. Each batch of potatoes was spiked with 3.655 g of AA. This was equal to spiking each 45 kg batch with 50 mg AA/100 g dry weight potatoes. The AA solution was prepared by addition of the minimum amount of water needed to dissolve the acid. The 3.655 g of AA was blended into each 45 kg batch using a Hobart mixer open to the atmosphere. Ascorbic acid content was determined before and after AA addition.

## Results and Discussion

Figure 1 shows linear regression data obtained from the AA standardization at concentration ranges from 0 to 50  $\mu\text{g}/\text{ml}$ . The high regression coefficient ( $r=0.9997$ ) indicates reliability of the method over the entire concentration range.

Table 1 presents AA concentrations for mashed potatoes using both the freezing and the freezing plus lyophilizing procedures. These samples represent two different cultivars used in the potato flake process and samples were taken from eight different process runs. Samples of mashed potatoes were frozen in dry ice, sampled [stored frozen (F) or lyophilized (F-L)], then analyzed following the modified analytical procedure. The pooled standard deviation in these data is 0.44 mg/100 gm and the means are 1.8 and 4.4 for the F and F-L procedures respectively. A null hypothesis indicated a highly significant difference between the two procedures. With a standard deviation of 0.5 mg/100 g for the F-L procedure, the limit of detection ( $\pm tS(x)$ ) is 1.2 mg/100 gm @ the 95% confidence level.

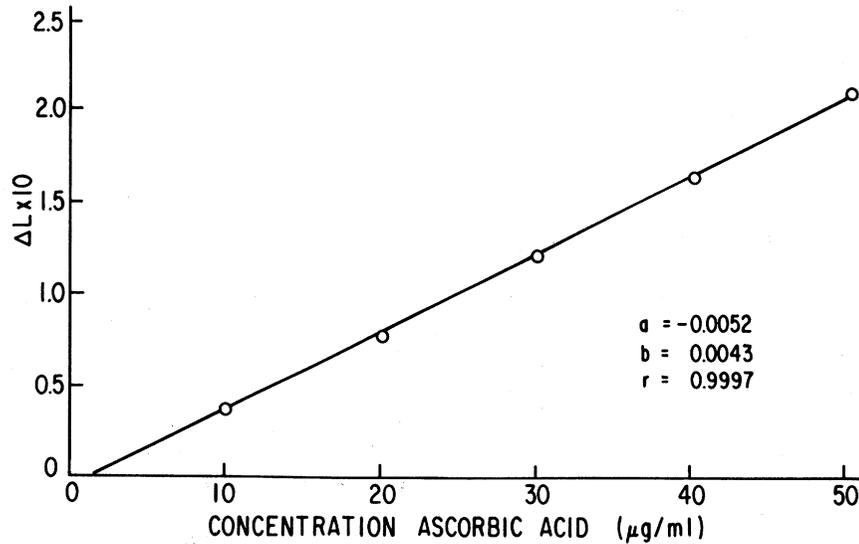


FIG. 1. Ascorbic acid standard calibration curve.

$$Y = bx + a$$

$$\text{Absorbance} = 0.0043 * \text{Conc.} - 0.0052$$

$r = 0.9997$  is the correlation coefficient.

TABLE 1. — *Ascorbic acid concentration of mashed potatoes mg AA/100 g dry weight potato.*

Run#	Cultivar <sup>a</sup>	Frozen		Frozen & Lyophilized	
		Det. 1	Det. 2	Det. 1	Det. 2
1	Russet Burbank	0.5	1.5	3.7	3.3
2	Russet Burbank	1.1	1.1	3.7	3.7
3	Russet Burbank	3.2	3.2	5.1	4.7
4	Kennebec	0	0	4.2	3.7
5	Kennebec	0	0	6.0	5.6
6	Kennebec	4.4	4.4	4.7	4.7
7	Kennebec	0	0	2.3	1.9
8	Kennebec	4.7	4.2	7.4	6.0

<sup>a</sup>Geographic Origin-Maine.

To further determine an optimum AA procedure, a series of experiments was conducted in which mashed potatoes (45 kg batches) were spiked with varying known amounts of AA, frozen in dry ice, sampled [stored frozen (F) or lyophilized (F-L)], then analyzed. Results of these experiments are shown

in Table 2. Mash potato samples in this table were taken from the same potato runs as reported in Table 1.

To determine the precision of the spiking procedure, the sampling step and the analytical procedure, four identical 45 kg batches of mashed potatoes were spiked with equal amounts (3.655 g) of AA. The assay results were 50.0, 53.7, 56.2, and 57.0 mg AA/100 g dry weight potato (naturally present plus added). Applying the "t" test at the 95% confidence level gave an error of  $\pm 10.0$  mg/100 gm. Although this error seems high for identical batches, relative when compared to the errors in the data obtained from standard solutions, this error includes both sampling and analytical errors. There are inherent errors associated with the preparation and sampling of raw materials containing solids, such as mashed potatoes (7).

TABLE 2. — *Ascorbic acid in potato mash with different amounts of AA spike mg AA/100 g dry weight potato.*

Run#	Cultivar <sup>a</sup>	AA Spike	Frozen		Frozen & Lyophilized	
			Det. 1	Det. 2	Det. 1	Det. 2
1	Kennebec	12.8	10.2	9.7	13.5	13.0
2	Kennebec	24.2	16.3	15.2	21.4	21.4
3	Kennebec	24.8	14.4	14.4	24.2	24.2
4	Kennebec	27.5	28.5	28.5	29.8	28.4
5	Kennebec	32.4	29.2	28.1	29.8	29.8
6	Russet Burbank	42.3	42.3	42.8	42.3	41.9
7	Russet Burbank	48.6	53.2	54.0	50.2	48.8
8	Russet Burbank	48.6	52.4	52.4	51.2	51.2

<sup>a</sup>Geographic Origin-Maine.

The data presented in Tables 1 and 2 are illustrative of the difference between the frozen (F) and the frozen and lyophilized (F-L) procedures with the F-L procedure generally yielding higher AA values. These results indicate that to ensure accurate AA determination care must be taken to avoid oxidative degradation prior to the actual analysis of samples. This is accomplished by the use of the dry ice snow into which potato samples are submerged, simultaneously freezing and replacing atmospheric oxygen with carbon dioxide. Subsequent lyophilization and storage at freezer temperature (-16 C) also appear to allow for longer pre-analysis storage periods of AA containing potato samples.

The procedures described herein were used successfully for AA determination on samples obtained from all stages of a pilot plant potato flake process, namely: raw (starting potatoes), peeled, blanched, cooked, riced and drum dried (15). Accurate determination of certain key potato components, such as initial AA concentration, are necessary for incorporation into

predictive models of processing operations (8). Use of the method of Loeffler, *et al.* (10) with our modification, yielded lower limits of detection for AA of 1.2 mg/100 g dry weight potato. Pre-process screening of fresh potatoes reflected the expected variation in AA levels, by cultivar as well as geographical origin of potatoes (Table 3).

TABLE 3. — *Ascorbic acid concentration<sup>a</sup> of raw potatoes mg AA/100 g dry weight potato.*

Cultivar	Location	Concentration <sup>b</sup>
Atlantic	New York	118
	North Carolina	136
	Florida	115
Kennebec	Maine	60
	Pennsylvania	82
Norchip	Maine	66
	North Dakota	54
	Pennsylvania	100
Russet Burbank	Maine	55
Superior	New York	135
	Maine	112
	Pennsylvania	114
BelRus	Maine	38
Gold Rus	Maine	45

<sup>a</sup>Sampling procedure was freezing and lyophilizing.

<sup>b</sup>Average of three determinations.

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