The total amount of nitrosopyrrolidine produced from heated pork was estimated by analyzing the combined cooked pork, cooked-out fat, and condensate. Recovery of nitrosopyrrolidine from the combined fractions was 30%, which is similar to the recoveries reported by others using steam distillation (Crosby et al., 1972; Telling et al., 1971). Nitrosopyrrolidine was not detected in uncooked samples of pork containing nitrite and putrescine. When corrected for recovery, three samples containing 0.02% sodium nitrite produced an average of 109 ppb of nitrosopyrrolidine. Three samples cooked with 0.02% sodium nitrite and 0.40% putrescine averaged 321 ppb. When relating the total amount of nitrosopyrrolidine produced to that determined in only the condensate of similar samples (Table I), it appears that approximately 20–40% of the nitrosopyrrolidine formed was given off as a volatile. Thus, it is likely that during the heat-induced formation of nitrosopyrrolidine in bacon, a significant portion of the nitrosamine may be volatilized from the product and not detected in the cooked bacon or rendered fat.

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Rapid Analysis of Potassium Sorbate in Dried Prunes by Ultraviolet or Colorimetric Procedures

A rapid analysis procedure for the determination of potassium sorbate in dried prunes was developed. It utilizes a rapid double extraction procedure which gives high extraction efficiency for potassium sorbate in dried prunes. This procedure also eliminates most of the interfering compounds which are found in dried prunes and cause high blank values with many present potassium sorbate analysis procedures. The extract can be read directly by ultraviolet (uv) instrumentation or by a colorimetric procedure. The colorimetric procedure allows small laboratories with limited resources to run potassium sorbate analysis by this extraction procedure.

It has been previously determined at this laboratory that from 200 to 600 ppm of potassium sorbate will preserve processed dried prunes, depending on their moisture content. Recently, concern in the United States and other countries over the increased use of food additives and their safety has led many countries to place rigid controls on their use, including bans on certain additives. This development has led to the increased need for new rapid and accurate methods of analysis. In the United States potassium sorbate is classified as GRAS (generally recognized as safe). In other countries the tolerance ranges from 0 to 1000 ppm (Dada, 1975).

The analysis of potassium sorbate in dried prunes is complicated by the large quantities of sugars, acids, and other naturally occurring compounds. These compounds and those formed during dehydration cause high blank values in many currently used ultraviolet or colorimetric procedures. This inaccuracy can be reduced by determining a blank value on untreated prunes from the same lot, but these are not always available and, in any event, lead to an increase in the analysis time. Several analytical procedures are currently used by prune processors (Stafford and Nury, 1969); one is rapid with a high blank value, and the others are slow and time consuming. Because of these factors and the increased necessity for accurate and rapid potassium sorbate analysis, a new analytical procedure was developed. It utilizes a rapid double extraction procedure which gives high extraction efficiency for potassium sorbate and eliminates most of the interfering compounds in prunes. The extract can be read directly by uv instrumentation or analyzed by the slightly modified colorimetric procedure of Nury and Bolin (1962). The colorimetric method allows small laboratories with a minimum of equipment to run potassium sorbate analysis of prunes by this procedure.

EXPERIMENTAL SECTION

Apparatus. A Bausch and Lomb Spectronic 20 colorimeter was used for the colorimetric procedure. A Beckman DB spectrophotometer was used for the uv

Table I. Comparison of Ultraviolet and Colorimetric Analysis

Potassium sorbate added, ppm	Uv anal.,[a] % recovery	Colorimetric anal.,[a] % recovery
250	98.6	96.4
500	98.2	97.8
750	99.0	97.6
1000	99.4	97.6
Av	98.8	97.3
SD	0.47	0.68

[a] Average of two runs.

procedure.

Reagents. All reagents and solvents used were analytical grade unless otherwise specified. Chloroform was extracted twice with 0.5 N $NaHCO_3$ (2:1), daily as needed, to remove the ethanol present which would interfere with the analysis. The chloroform was dried with anhydrous sodium sulfate. Eastman 2-thiobarbituric acid (0.5 g) was placed in a 100-ml volumetric flask with approximately 80 ml of distilled water. The thiobarbituric acid was dissolved by heating the flask in a boiling water bath; the flask was cooled and the solution made up to volume with distilled water. This solution should be prepared daily. The potassium dichromate–sulfuric acid solution was prepared by dissolving 0.49 g of $K_2Cr_2O_7$ in a 1-l. volumetric flask with approximately 500 ml of distilled water and then adding 8 ml of concentrated H_2SO_4. The solution was made up to volume with distilled water.

Procedure. The prune samples were prepared for extraction by blending 50.0 g of prune flesh in a 1-quart blender jar with 450 ml of distilled water for 5 min. A 10.0-g aliquot of the resultant slurry was weighed into a 50-ml volumetric flask and made up to volume with distilled water. Ten milliliters of the solution was pipetted into a 250-ml separatory funnel and the sorbic acid was extracted by shaking with 100 ml of chloroform for 1 min. Approximately 70 ml of the chloroform layer was drained off into a 125-ml Erlenmeyer flask containing 5 g of anhydrous sodium sulfate. Fifty milliliters of the chloroform extract is used for either the colorimetric or uv analysis.

Colorimetric Analysis. The 50 ml of initial chloroform extract was placed in a 125-ml separatory funnel and the sorbic acid present was extracted into 15 ml of 0.5 N $NaHCO_3$ by shaking for 1 min. The chloroform layer was carefully drained off and discarded. The remaining $NaHCO_3$ layer was quantitatively transferred into a 25-ml volumetric flask. Hydrochloric acid (6 N) was slowly added with agitation until the solution was acid to pH paper; it was then made up to volume with distilled water. Two milliliters of the solution was pipetted into a test tube containing 2 ml of the potassium dichromate–sulfuric acid solution. Two milliliters of distilled water was similarly pipetted for use as a reagent blank. The test tubes were lightly covered with polyethylene caps and then placed into a boiling water bath for 5 min. The test tubes were removed from heating and 2 ml of thiobarbituric acid solution was added immediately. Heating was then resumed for an additional 10 min. The test tubes were removed and cooled. The absorbances of the solutions were read at 530 nm.

The standard curve was prepared by making up solutions containing 1, 2, 3, and 4 μg/ml potassium sorbate in distilled water. Two-milliliter aliquots were pipetted into test tubes containing 2 ml of the potassium dichromate–sulfuric acid solution and the analysis proceeded exactly as with the previous sample solution. The net absorbance ($A_{STD} - A_{reagent\ blank}$) vs. micrograms/milliliter was plotted for each solution.

Uv Analysis. In this analysis the 50 ml of initial chloroform extract was placed in a 125-ml separatory funnel and the sorbic acid present was extracted by shaking with 25 ml of 0.3 N $NaHCO_3$ solution for 1 min. The chloroform layer was carefully drained off and discarded. The absorbance of the remaining $NaHCO_3$ layer was read at 254 nm.

The standard curve for the uv procedure was obtained by making up 0.3 N $NaHCO_3$ solutions containing 1, 2, 3, and 4 μg/ml potassium sorbate. The absorbances of the solutions were read at 254 nm. The absorbance of each solution vs. micrograms per milliliter potassium sorbate was plotted.

RESULTS AND DISCUSSION

The extraction efficiencies of the two analysis procedures at four concentrations of potassium sorbate which cover the range that occur on processed prunes are shown in Table I along with the standard deviation of the analysis. These analyses were made using blank values obtained from untreated prunes of the same lot. The quantity of potassium sorbate present in parts per million was obtained by multiplying micrograms per milliliter potassium sorbate times 250.

The average blank value for eight samples of untreated prunes, received from the three main growing areas for the colorimetric procedure, was 10 ppm with a standard deviation of 2 ppm. The average value for the uv procedure was 18 ppm with a standard deviation of 2 ppm. Because of the small blank value and its standard deviation it is possible to obtain accurate sorbate analysis without running untreated prunes from the same lot by subtracting the average blank value from the sorbate value.

The recovery of potassium sorbate is slightly less in the colorimetric procedure because of difficulty in obtaining quantitative recovery of the 0.5 N $NaHCO_3$ extract from the separatory funnel.

The results of these experiments show that either procedure will produce accurate results throughout the normal range of use of potassium sorbate to preserve processed dried prunes. It is also possible to absolutely determine the presence or absence of potassium sorbate in prunes. This is necessary for prunes intended for export to countries which have a zero tolerance. For this purpose, the basic solution used for uv analysis is acidified; a shift will occur in the uv absorption maxima from 254 to 263 nm if sorbic acid is present. The acidified extract may also be extracted with chloroform and concentrated in a rotary evaporator. The resultant solution is then spotted, along with a known sorbic acid solution, on a thin-layer plate (Höyem, 1962).

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