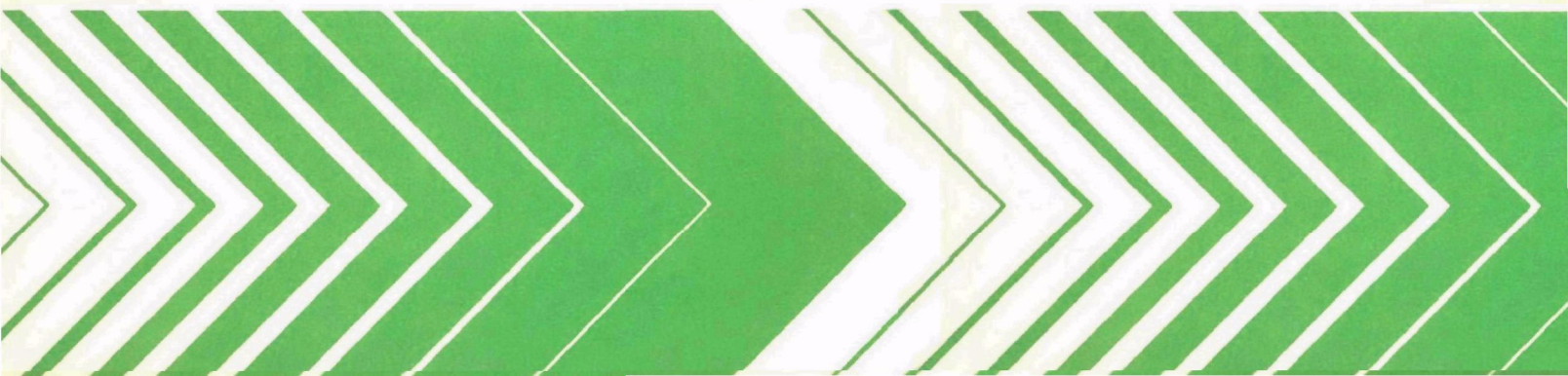


Research and Development



# Commercial Feasibility of Recovering Tomato Processing Residuals for Food Use



## **RESEARCH REPORTING SERIES**

Research reports of the Office of Research and Development, U.S. Environmental Protection Agency, have been grouped into nine series. These nine broad categories were established to facilitate further development and application of environmental technology. Elimination of traditional grouping was consciously planned to foster technology transfer and a maximum interface in related fields. The nine series are:

1. Environmental Health Effects Research
2. Environmental Protection Technology
3. Ecological Research
4. Environmental Monitoring
5. Socioeconomic Environmental Studies
6. Scientific and Technical Assessment Reports (STAR)
7. Interagency Energy-Environment Research and Development
8. "Special" Reports
9. Miscellaneous Reports

This report has been assigned to the ENVIRONMENTAL PROTECTION TECHNOLOGY series. This series describes research performed to develop and demonstrate instrumentation, equipment, and methodology to repair or prevent environmental degradation from point and non-point sources of pollution. This work provides the new or improved technology required for the control and treatment of pollution sources to meet environmental quality standards.

EPA-600/2-78-202  
September 1978

COMMERCIAL FEASIBILITY OF RECOVERING TOMATO PROCESSING  
RESIDUALS FOR FOOD USE

by

W. G. Schultz, H. J. Neumann , J. E. Schade  
J. P. Morgan, P. F. Hanni  
Western Regional Research Center, Science and Education Administration  
U.S. Department of Agriculture, Albany, California 94710

and

A. M. Katsuyama, H. J. Maagdenberg  
National Food Processors Association, Western Research Laboratory  
Berkeley, California 94710

Interagency Agreement EPA-IAG-D5-0795

Project Officer

H. W. Thompson  
Food and Wood Products Branch  
Industrial Environmental Research Laboratory  
Corvallis Field Station  
Corvallis, Oregon 97330

INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY  
OFFICE OF RESEARCH AND DEVELOPMENT  
U. S. ENVIRONMENTAL PROTECTION AGENCY  
CINCINNATI, OHIO 45268

## DISCLAIMER

This report has been reviewed by the Industrial Environmental Research Laboratory, U.S. Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the U.S. Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

## FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report presents the results of a 2 year study on the technoeconomic feasibility of recovering a food grade material from the peel residue associated with the "dry" caustic peeling of tomatoes. The overall purpose of the project was to develop a beneficial use of a food processing residual which is currently considered a waste material. The results of this study should be of interest to tomato processors, regulatory agencies, manufacturers of specialty food chemicals and food researchers. For further information on this study, contact the Food and Wood Products Branch, Industrial Pollution Control Division, IERL-Ci.

David G. Stephan  
Director  
Industrial Environmental Research Laboratory  
Cincinnati

## ABSTRACT

In the United States tomatoes are typically peeled for commercial canning by first immersing them in a caustic bath to loosen the skin; then, the peel is removed either mechanically with rubber discs or with water sprays. When the peel is removed mechanically, the peel solids are not diluted and therefore, are similar to the pulp of whole tomatoes. Since this removed peel is at least 12% of the unpeeled weight and the peel is about 96% pulp, this peel pulp is a good source of potential food material. There are about 1.3 million tons\* of tomatoes peeled each year in the United States, resulting in at least 150,000 t/yr of recoverable pulp. Currently that pulp is discarded as peel at an expense of at least \$2.50/t or \$12.00/hr. Pulp recovery is attractive economically because it has a projected net worth of \$187/hr for a typical 40 t/hr peeling operation. The estimated overall net return per cannery is \$199/hr, including processing expenses. This is a \$7,500,000/yr potential gross return to the United States tomato processing industry and provides a corresponding ability to maintain lower product prices to the consumer.

A 2-year project was undertaken to determine the commercial feasibility of recovering pulp from the peelings of caustic peeled tomatoes. In 1975, peel from regular cannery operations was processed through a 20-gpm (5 t/hr) continuous-flow line. This processing consisted of acidifying the peel to pH 4.2 with food-grade hydrochloric acid, then separating the pulp from the skin with a paddle finisher (screen). Recovered peel pulp was found to be of food quality but contained high peeling-aid residues (150-450 ppm). Practically all tomato peeling operations use a peeling aid in the caustic bath to facilitate uniform peeling. Peeling aids in current use are approved for peeling but not as additives to the final product. In 1976, a 1-t/hr pilot peeling line was set up at a cannery to study modifications in the peeling process for the purpose of reducing peeling-aid residue in the recovered pulp. The principal modification was to pretreat the tomatoes by immersion in a 150°F aqueous bath (approximately pH 3.6) containing about 0.15% food-grade octanoic (caprylic) acid; subsequently, the tomatoes were immersed in caustic. The peel was removed with rubber-disc peelers. Recovered pulp met USDA Quality Grade A, and the octanoic acid levels were low (0 to 30 ppm). The proposed use of this recovered peel pulp is in combination with tomato pulp from regular sources for canned products such

---

\*This report follows the prevailing canning industry practice of using the International System of Units (SI) in the laboratory and U.S. units in the manufacturing operations. See TABLE OF CONVERSION FACTORS for U.S. to SI units.

as tomato sauce, catsup, paste, and fill juice for peeled tomatoes. Compositions of these products are governed by the FDA Standards of Identity, currently being revised; the current regulations allow the use of liquid from peels and cores but do not address the use of pulp from caustic peels.

This project was funded jointly by United States Department of Agriculture-Western Regional Research Center, National Food Processors Association-Western Laboratory, Environmental Protection Agency, and the California tomato processors. This report was submitted in fulfillment of Interagency Agreement EPA-IAG-D5-0795 under the partial sponsorship of the U.S. Environmental Protection Agency, and covers a period from May 1, 1975 to August 31, 1977. The experimental and analytical work was completed as of July 15, 1977.

## CONTENTS

Foreword . . . . .	iii
Abstract . . . . .	iv
Figures . . . . .	viii
Tables . . . . .	viii
Abbreviations and Symbols . . . . .	ix
Conversion Factors for Units . . . . .	x
Acknowledgments . . . . .	xi
1. Introduction . . . . .	1
2. Conclusions . . . . .	4
3. Recommendations . . . . .	5
4. Materials and Control Instruments . . . . .	6
5. Experimental Procedures . . . . .	9
6. Results and Discussion, General . . . . .	26
Washing and Sorting . . . . .	28
Peeling Aids . . . . .	28
Carboxylic Acid Peeling . . . . .	30
Peeling-Aid Pretreatment . . . . .	30
Modified-Caustic Peeling . . . . .	31
Peel Removal . . . . .	31
Peel Flow . . . . .	32
Peel Alkalinity . . . . .	32
Acidification . . . . .	34
Salt (Sodium Chloride) . . . . .	34
Pulp Extraction . . . . .	35
Vacuum Evaporation . . . . .	38
Unit Operation Times . . . . .	39
Product Color, Flavor, and Vitamins . . . . .	40
Consistency, Viscosity, and Pectin . . . . .	41
Amino Acids . . . . .	45
Product Mold, Insect Fragments, and Bacterial Counts . . . . .	45
Pesticides . . . . .	46
U.S. Standards of Identity . . . . .	47
Effluents and Wastes . . . . .	49
Economics . . . . .	51
References . . . . .	52
Bibliography . . . . .	55
Appendices	
A. Vitamin A Activity Method . . . . .	56
B. Photometric Determination of Anionic Peeling Aids . . . . .	59
C. Determination of Fatty Acids in Processed Tomato Products . . . . .	62



## FIGURES

<u>Number</u>	<u>Page</u>
1 Tomato peel component diagram . . . . .	2
2 1975 process flow diagram . . . . .	10
3 1975 equipment profile . . . . .	11
4 Peel tank, 1975 . . . . .	12
5 Hot break vessel, 1975. . . . .	13
6 Tramp metal trap, 1975. . . . .	14
7 Modified caustic peeling, 1976. . . . .	17
8 Acid required to reduce pH of caustic peel and pulp . . . . .	33

## TABLES

<u>Number</u>	<u>Page</u>
1 Summary of Typical 1975-1976 Recovered Peel Pulps . . . . .	27
2 Acidification Data, 1975. . . . .	35
3 Summary of 1975 Processing Conditions and Solids Recovery . . .	37
4 Unit Operations Times, 1975 . . . . .	40
5 Storage Stability of Recovered Pulp, 1976 . . . . .	42
6 1976 Canned Samples of Recovered Pulps, 1976. . . . .	43
7 Pesticide Residues in Recovered Peel Pulp . . . . .	48
8 Projected Economics of a Commercial Installation. . . . .	51

## ABBREVIATIONS AND SYMBOLS\*

ACS	--	American Chemical Society
C	--	degrees Celsius (also, carbon in chemical formulas)
ca	--	about
COD	--	chemical oxygen demand
F	--	degrees Fahrenheit
ft	--	foot
g	--	gram
gal/t	--	gallons per ton
GLC	--	gas-liquid chromatography
gpm	--	gallons per minute
HCl	--	hydrochloric acid
hp	--	horsepower
in.	--	inch
IU	--	International Units
lb/gal	--	pounds per gallon
l/min	--	liters per minute
mg	--	milligram
ml	--	milliliter
mM	--	millimoles
mμ	--	millimicrons
N	--	solution normality
NaCl	--	sodium chloride
NF	--	National Formulary
NTSS	--	natural tomato soluble solids
NTTS	--	natural tomato total solids
pH	--	hydrogen ion concentration, $\log 1/(H^+)$
ppm	--	parts per million
psig	--	pounds per square inch, gauge pressure
QC	--	quality control
rpm	--	revolutions per minute
t/hr	--	tons per hour
t/yr	--	tons per year
TS	--	total solids
w/v	--	weight per volume
w/w	--	weight per weight

\*This report follows the prevailing canning industry practice of using U.S. (English) units in the manufacturing operations and Metric (SI) units in the laboratories. See CONVERSION FACTORS FOR UNITS on page ix.

# CONVERSION FACTORS FOR UNITS

<u>U.S. Units</u>			<u>Metric (SI) Units</u>
F	(F-32) 0.5556	=	C, degrees Celsius
ft	(ft) 0.3048	=	m, meter
gal	(gal) 3.785	=	l, liters
gpm	(gpm) 3.785	=	l/m, liters per min
gal/t	(gal/t) $4.172 \times 10^{-3}$	=	l/kg, liters per kilogram
hp (electric)	(hp) 746.0	=	W, watts
in.	(in.) 0.02540	=	m, meter
lb/gal	(lb/gal) 0.1198	=	g/cc, gram per cubic centimeter
psig	(psig + 14.7) x 6894	=	N/m <sup>2</sup> , newton per sq. meter (absolute)
t (short)	(t) 907.2	=	kg, kilogram

## ACKNOWLEDGMENTS

The authors appreciate the support given to this project by the tomato processing industry; not only did it ease the burden, but tasks and judgments were often shortened and made possible in a few months which otherwise might have required several years. In particular, we wish to thank Tillie Lewis Foods and Hunt-Wesson Foods at whose canneries the field work was performed. Their participation allowed a practical approach that could not be duplicated in a laboratory or pilot plant. The U.S. Environmental Protection Agency supported this experimentation in part, thus enabling the work and development to be carried out in the short period of 2 years.

## SECTION 1

### INTRODUCTION

In the United States tomatoes are normally peeled by loosening the skin with a hot-caustic bath and removing the peel (skin with adhering pulp, see Figure 1) either mechanically, such as with rubber discs, or with water sprays. The use of water sprays has declined because of the large amount of water needed (500-1,500 gal/t\*) and the subsequent problem of waste disposal of the dilute solution. Removal of the peel mechanically with rubber discs reduces the water consumption to a negligible amount so that the peel has about the same solids content as fresh tomatoes. This material is currently discarded as solid waste and constitutes at least 12% of the original tomato weight. Since this peel is about 96% pulp, it is a potential source of food material. This pulp has a value of up to \$50/t as recovered pulp, and from a typical 40 t/hr peeling operation, at least 5 t/hr of peel is available with a gross value of \$250/hr of operation. Currently that pulp is discarded as peel at an expense of at least \$2.50/t. Since there are about 1.3 million tons of tomatoes peeled each year in the United States resulting in at least 150,000 t/yr of recoverable pulp, there is a potential gross value of \$7,500,000/yr, and a corresponding opportunity to hold down the product price to consumers.

Despite the economic incentive, there were several technical obstacles such as insecticide residues, residuals of the caustic and surfactants from the caustic-peeling applicator, acidification of the alkaline peel, recovered-pulp quality, product labeling, etc. With these potentials and obstacles in mind, a two-year project, beginning in 1975, was undertaken jointly by the U.S. Department of Agriculture (USDA)-Western Regional Research Center, National Food Processors Association (NFPA)-Western Research Laboratory, and the tomato processing industry. The initial plans were described in April 1975 (1) and were based on trends in commercial practice and prior information on pulp recovery potential (2) (3) (4) (5).

Utilization of tomato peel was considered during pilot tests on the mechanical, rubber-disc peel removal system in 1973 (2). In a single, large-scale test conducted in 1974, pulp was prepared from tomato peel (3). Until recently tomato peel was removed with water sprays after caustic immersion, resulting in too dilute a peel for practical pulp recovery. As more of the rubber disc, mechanical peel removal units have been installed, more peel is available that retains the solids concentration of a fresh tomato; thus pulp recovery from the peel is now practical.

---

\*This report follows the prevailing canning industry practice of using U.S. (English) units in the manufacturing operations and Metric (SI) units in the laboratories. See CONVERSION FACTORS FOR UNITS on page ix.

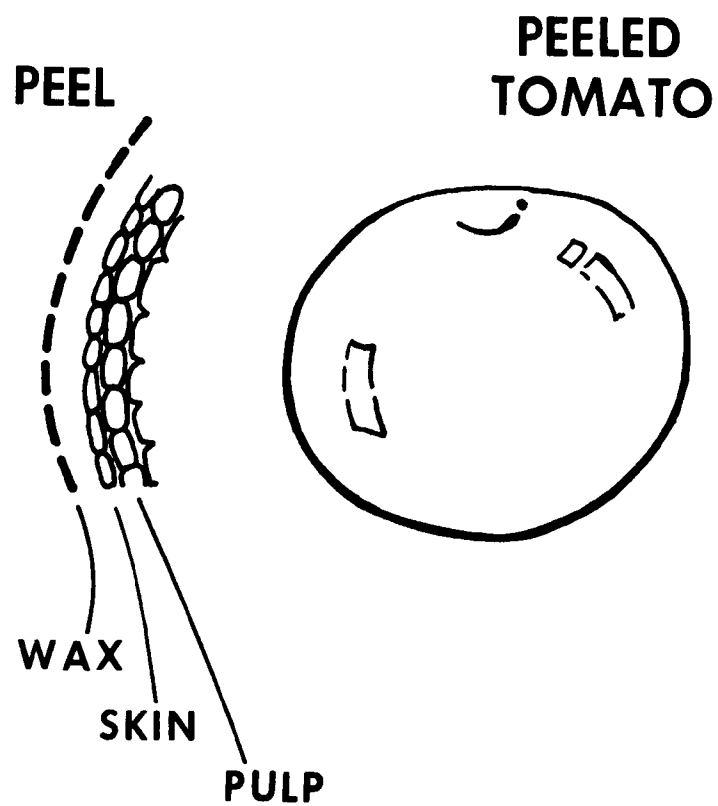


Figure 1. Tomato peel component diagram.

Consideration was also given to utilization of additional tomato solids derived from tomato pomace through an acid treatment similar to that demonstrated with macerate (4) (5).

The recovery is accomplished by acidifying the peel, then separating the peel into pulp (96%) and skin (4%) fractions. Skin is discarded. The recovered peel pulp contains salt and could be used in formulating tomato sauce or similar salted products. The experimentation, developments, and analyses spanned two processing seasons from June 1975 through December 1976. The 1975 experimentation centered on a continuous processing of peel from a regular cannery peeling operation, taking the peel as discharged from normal operations. The primary goal was to evaluate the commercial feasibility of recovering tomato peel pulp as food-grade material from tomato peelings and pomace. The material evaluated was 100% pulp (from peel) without combination with other tomato material as would be expected in normal cannery operation. This permitted the true identity and character of the recovered pulp to be evaluated in terms of the USDA Standards for Grade and FDA Standards of Identity.

During the first year of experimentation, the peelers were operated by the cannery personnel to conform to production needs, and experimentation involved only the pulp recovery which had to adapt to the cannery operation without any influence by the experimentors. Originally, 1976 was projected to emphasize process optimization with the caustic peel residual. However, as a result of the high peeling-aid residues found in 1975, work in 1976 was primarily developmental and emphasized the reduction of this peeling-aid residue in the recovered pulp.

The present FDA Standards of Identity for tomato pulp allow material from several sources. One source is the liquid obtained from peel residuals resulting from tomato peeling operations. The present (1976) Standards were written when steam and water peeling were prevalent. Those Standards are being revised, and it is expected that the revised Standards will be available in 1978.

## SECTION 2

### CONCLUSIONS

1. Tomato pulp can be recovered from caustic peel which amounts to 15% or more of the fresh tomato weight. Current practice is to discard the caustic peel. When acidified and screened, about 96% of the peel is recoverable pulp, and 4% is waste skin. Therefore, waste disposal volume is reduced by 96%.
2. Recovered pulp can meet USDA Grade A Standards for color, flavor, and absence of defects.
3. The recovered pulp contains salt, has an intense red color (maximum Grade A score), has a thick consistency that is due to insoluble materials, and is suitable for use in sauces, etc.
4. The preferred method of controlling the peeling aid level in the recovered pulp is an octanoic (caprylic) acid pretreatment of the tomatoes, instead of using peeling aid directly in the caustic applicator. Although steam-peelable tomatoes can be peeled by octanoic acid alone (without caustic), caustic is generally needed for current California varieties such as VF-145B-7879.
5. Pulp recovery is an economically feasible process in which the capital investment can be recovered in 1-year; thereafter, the net return is about \$200/operating hour for a 40 t/hr caustic peeling operation.



## SECTION 3

### RECOMMENDATIONS

1. Industry should seek regulatory clarification and acceptance of the process and products associated with the recovery of pulp from caustic peel, particularly on the: (a) pulp, (b) acidification, and (c) use of octanoic (caprylic) acid.
2. Industrial (long-term) demonstrations on a full-scale processing basis are needed to determine: (a) the pretreatment bath stability and replenishment frequency, (b) the extent of peeling-aid carryover to the caustic bath and into the product, and (c) the safety requirements such as the need for flow-diversion of off-control material.
3. Each cannery needs to determine the optimum process flow, equipment, and pulp use for its local situation.
4. A rapid, fatty-acid analysis should be developed to facilitate control of the octanoic acid (caprylic acid) peeling-aid concentration in the pretreatment bath to aid process control by cannery production personnel.

## SECTION 4

### MATERIALS AND CONTROL INSTRUMENTS

Tomatoes--In both years the tomatoes were field run as used by the canneries. There was no selection for experimental use; it was felt that any selecting would build in bias, and such selection would not be readily identifiable. In a peeling operation it is the extremes of tomato peelability that sets the conditions rather than the average tomato. Tomato peelability was judged using 10-lb samples taken from each run of 2,000 lbs. In 1975, the cannery processed the variety VF-145 almost solely. In 1976, the variety was usually the UC-134, with occasional varieties 198 and VF-145. Since a control run was made each day, the comparisons in peeling were based on peeling variables, eliminating the need to consider the tomato variety, harvest period, or growing location.

Sodium Hydroxide--The "caustic" (50% w/w sodium hydroxide aqueous solution; also called caustic soda or lye) used by canneries and in the pilot peeling studies was a standard food grade manufactured by the electrolytic diaphragm process. Currently, the chlor-alkali industry generally uses an asbestos diaphragm to produce the sodium hydroxide. A discussion on asbestos with respect to the chlor-alkali industry and its products was presented in 1975 (35).

Proposed rules have been published by the Food and Drug Administration regarding asbestos fibers in foods (6,7). However, as yet there have been neither conclusions about the medical significance of ingested asbestos fibers nor regulations promulgated on this subject.

Limited analyses were made in both years to determine the asbestos content in the recovered pulp, in the caustic bath, and on field-run tomatoes before and after washing. Although washing reduced the quantity of soil-borne asbestos on the whole tomatoes, asbestos fibers were found in the recovered pulp. The levels were about the same as have been reported as background in California drinking waters. Results from the limited number of analyses indicate that the major contributing source of asbestos is field soil, not the caustic bath.

The electrolytic mercury cell was formerly used by the chlor-alkali industry, but caustic soda from this process is no longer generally available. There is a newer caustic production process which utilizes an ion-exchange membrane, hence avoiding the use of either mercury or asbestos, but material so produced is also not generally available (8).

Hydrochloric Acid--In both years, the hydrochloric acid was a standard commercially available food grade 22° Baume (35.21% w/w HCl, 9.84 lb/gal).

In 1975 it was received from a bulk truck and stored in a 500 gal plastic tank provided with a calcium carbonate scrubber to control acid fumes.

Peeling Aids--These were commercially available sodium 2-ethylhexyl sulfate (40% aqueous solution), sodium mono- and di-methyl naphthalene sulfonates (40% aqueous solution), a proprietary mixture of C<sub>5</sub> to C<sub>9</sub> saturated fatty acids with an odd number of carbons (Faspeel, BASF Wyandotte Corp., Wyandotte, Michigan), hexanoic acid (caproic acid, 99%), and octanoic acid (caprylic acid, 92%). Faspeel is available only from the one source; all the others have multiple sources and are sold under a variety of trade names.

Peel pH Recording Controller--This Leeds and Northrup (North Wales, PA) pH equipment was a standard commercially available instrument, originally purchased in 1969, and used without modification other than to replace electrodes. It consisted of Leeds and Northrup Type No. 992-820-0925-6-008-485 having a Model R recorder, Series 80 electro-pneumatic controller with a pH 0 to 10 range and 3 to 15 psig output, and used an Electrode Assembly No. 7782 containing Measuring Electrode No. 117089, Reference Electrode No. 117106, and Temperature Compensator No. 352145. These units were suitable for tomato pulp at temperatures ranging from 80 to 212°F.

Final Pulp pH Recorder--Same type of unit as that used for Peel pH Recording Controller.

Incoming Peel pH Recorder--Prior to acidification and irrespective of the place of acidification, the incoming caustic peel was monitored by a Beckman (Fullerton, CA) Type No. 8710-2-04-4-1-0-0-1 Recorder with a pH 4 to 14 range and using a pH Electrode Station Model No. 300-0-1-03-1-2-1-0, containing a No. 636460 Remote Amplifier-Transmitter, No. 19033 Reference Electrode, No. 19505 Measuring Electrode, and a No. 19586 Temperature Compensator.

Laboratory pH Meter--Corning (Corning Scientific Instruments Co., Medford, MA) Model 475010 bench-type indicating meter with a pH 0 to 14 range and having a Beckman Combination Electrode No. 476051 and Corning Automatic Temperature Compensator No. 476097.

Acid Control Valve--Acid addition was modulated by a Research Control Valve (Research Control Co., Tulsa, OK) Type 75HB (Hastalloy B) with a 1/4-in. body, an orifice of C<sub>v</sub> = 0.30 equal percentage, and with a pneumatic actuator for 3 to 15 psig signal from the pH Recording Controller, air-to-open.

Hot-Break Temperature Controller--A standard thermocouple type instrument and pneumatic actuated steam modulating valve were used. The recording controller was a Honeywell (Philadelphia, PA) No. Y152P(13)-PH-96-K1-(13) with a 0 to 300°F range, 3 to 15 psig pneumatic output, and temperature sensing with a Honeywell No. 2T1M13G6-5 Type T copper constantan sheathed thermocouple. The controller was equipped with both proportional band and reset, but with the relatively large volume of liquid to be heated, the proportional band was quite adequate without reset.

Pretreatment Temperature Controller--Same as the Hot Break Temperature Controller.

Lye Applicator Temperature Controller--Same as the Hot Break Temperature Controller.

## SECTION 5

### EXPERIMENTAL PROCEDURES

#### 1975 EXPERIMENTATION--PEEL PROCESSING AND PULP CHARACTERIZATION

A flow diagram of the 1975 cannery peel-pulp recovery system is shown in Figure 2. Peel was experimentally processed on a daily basis during 1975 using peel as received from conventional caustic peeling at the Tillie Lewis Foods, Plant W, Antioch, California. This cannery processed VF-145 tomatoes through washing and sorting, then about 40 t/hr were diverted to their peeling operation. The diverted tomatoes were next immersed in a caustic bath; this was a typical industrial situation using 10-12% w/w sodium hydroxide with up to, but not exceeding, 0.2% w/w sodium 2-ethylhexyl sulfate at 200-210°F and with a nominal half-minute immersion. From this bath the tomatoes went into two types of mechanical peel removers, a flat-bed disc type followed by peel-tag removal rolls (FMC PR-20 Tomato Peel Remover, 1 machine; FMC Corporation, San Jose, California) or rotary-cylinder, rubber disc types (Magnuson Model C Peel Scrubbers, 4 machines; Magnuson Engineers, Inc., San Jose, California). All of these peel removers discharged the tomato peelings into a central screw conveyor which in turn emptied into a receiving tank. At this tank the peelings were picked up by a centrifugal pump and sent through a 3-in. stainless tube a distance of 200 ft to an outside yard where the experimental process equipment was located. At this yard the peelings passed into the experimental process system (Figure 3) or into the cannery waste-peel tank. In general, the experimental system utilized peel pumped directly from normal cannery peeling operations, and received this about 5.4 minutes after the clean tomatoes first entered the caustic applicator. This peel was "normal" material rather than closely controlled laboratory samples.

The primary experimental variables in the pulp recovery system were: (1) the extractor screen size and paddle clearance, (2) place of acidification, either before or after fractionation of pulp from the skin and miscellaneous seeds and fibers, (3) hot-break temperature, (4) lag time between the hot break and canning, (5) evaporator temperature and degree of pulp-solids concentration, and (6) the heat-processing time of cans. These variables were evaluated in terms of: (1) pulp yield, (2) product quality, and (3) tomato Standards of Identity.

Peel was received continuously at 10-30 gpm, and acidified with food-grade hydrochloric acid either immediately in the Peel Tank (48 gal pulp volume, Figure 4) or later in the Hot-Break Vessel (250 gal pulp volume, Figure 5); this acidification was continuously controlled by an automatic pH recording controller. Peel flowed continuously into each of these vessels and constant volumes were maintained by overflow weirs. A tramp-metal trap (Figure 6) was used to protect the pumps and other equipment.

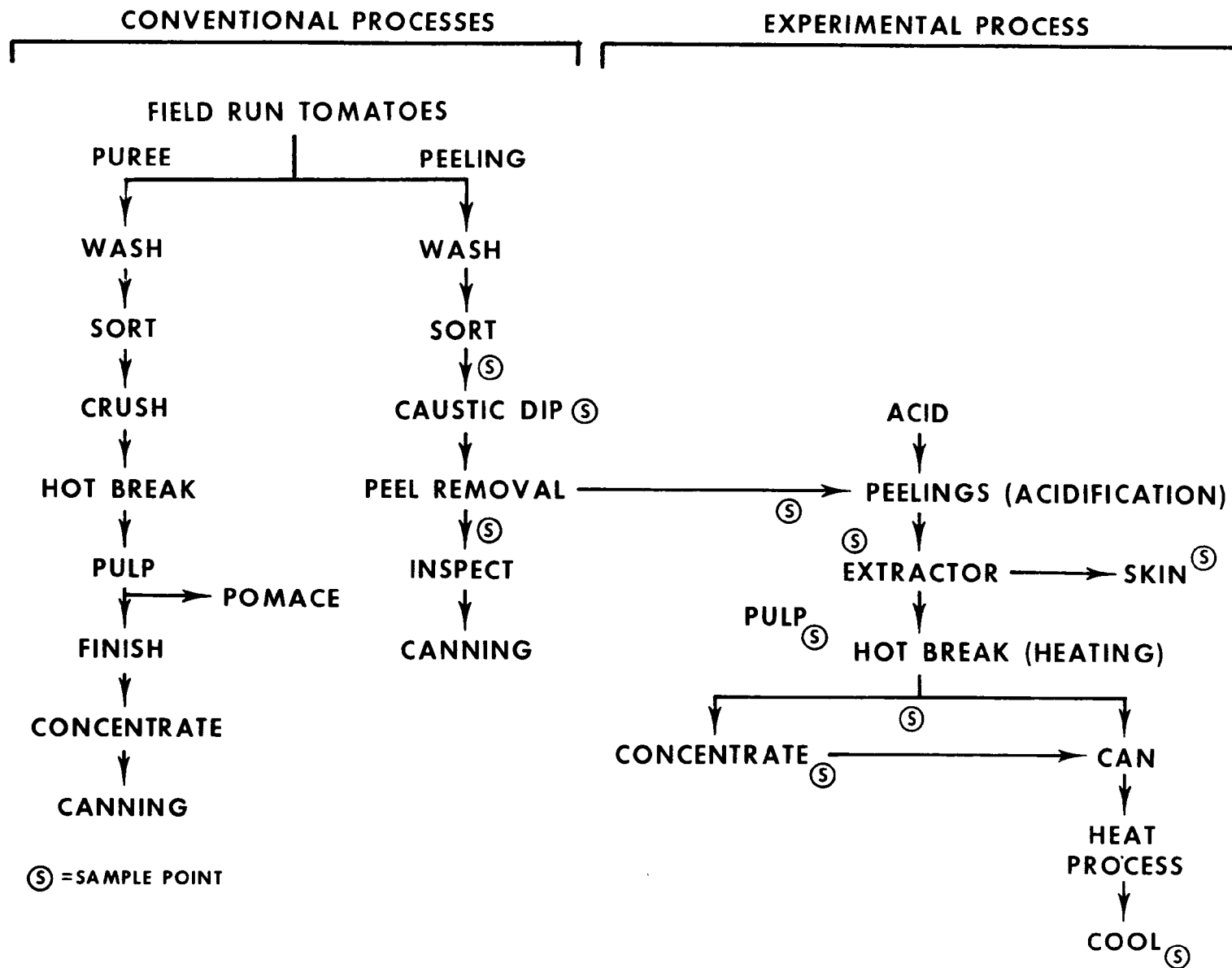


Figure 2. 1975 process flow diagram.

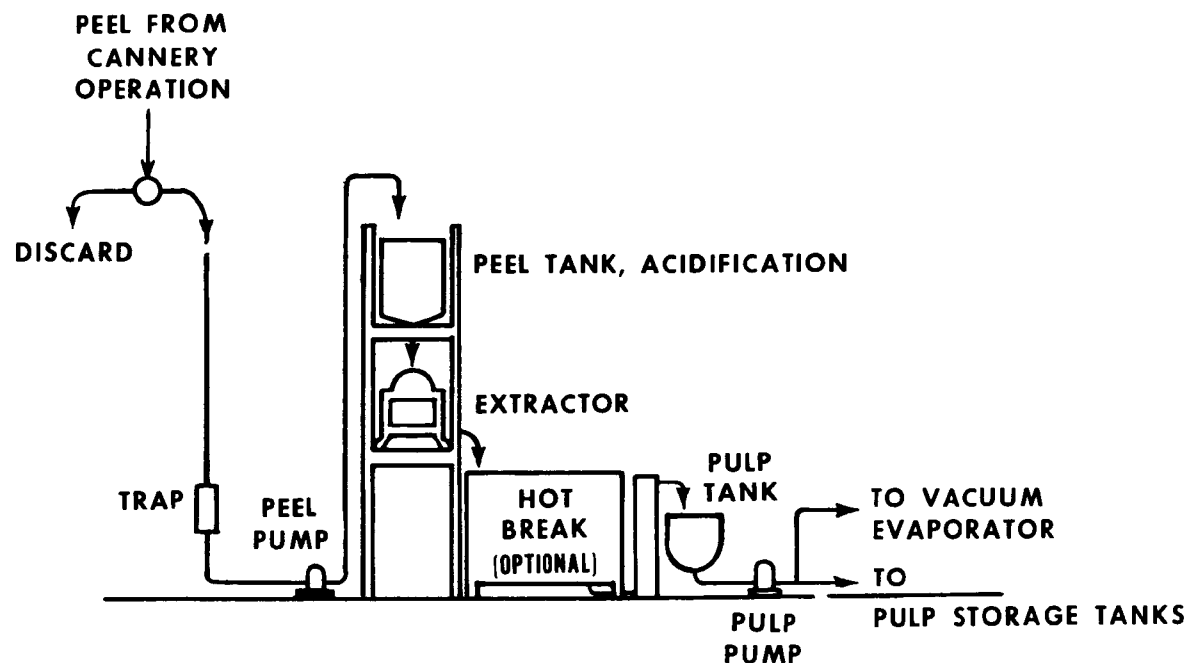


Figure 3. 1975 equipment profile.

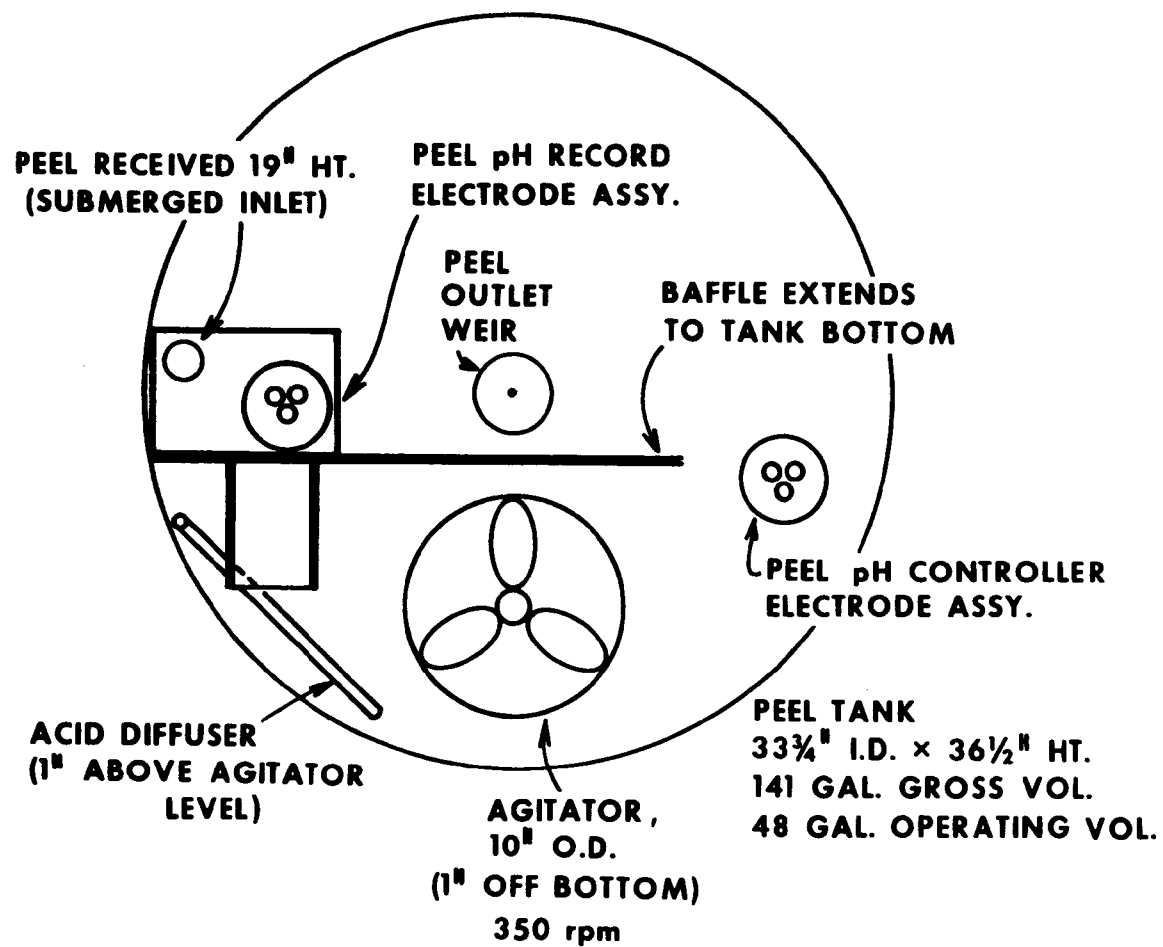


Figure 4. Peel tank, 1975.



STEAM COIL, 69 rpm  
 24" COIL OD, 1.5" OD TUBE,  
 23 TUBE TURNS, TWO 4" x  
 84" PADDLES ON COIL OD,  
 55 sq ft HEATING SURFACE  
 COIL DRIVE, 3 hp  
 STEAM FLOW CONTROLLED  
 AUTOMATICALLY BY  
 PULP TEMPERATURE

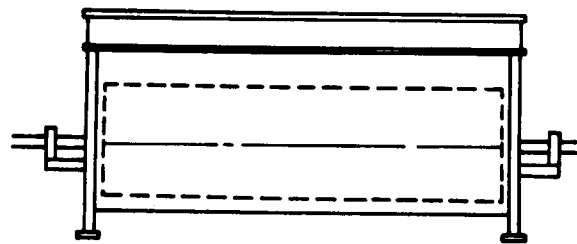
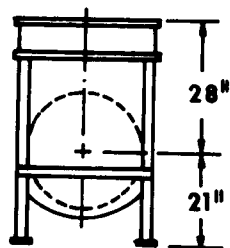
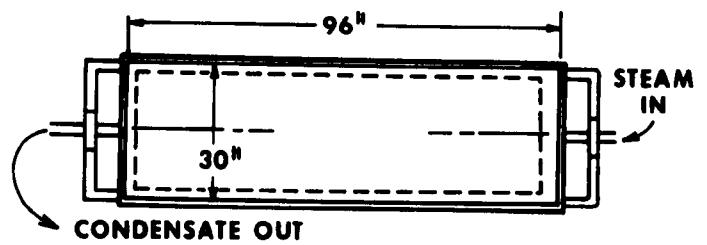


Figure 5. Hot-break vessel, 1975.

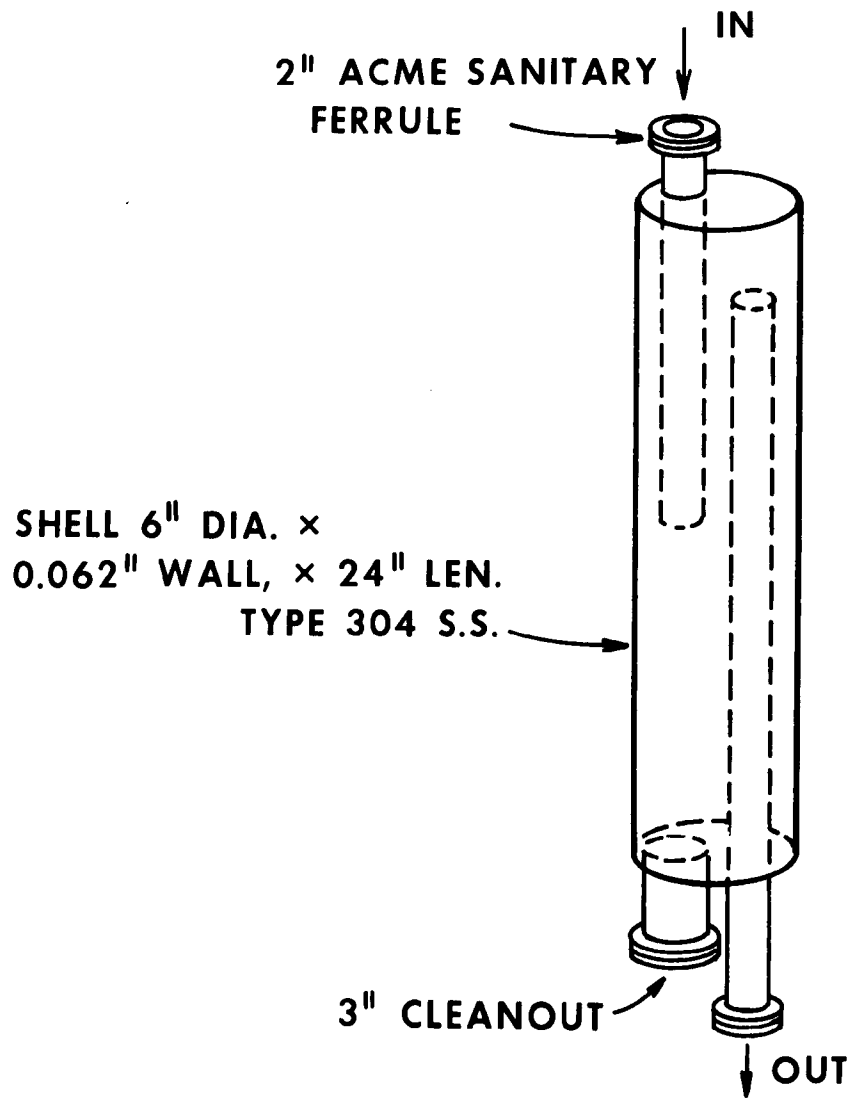


Figure 6. Tramp metal trap, 1975.

A Hot-Break Vessel (Figure 5) was provided to inactivate enzymes that might be present and to reduce subsequent microbiological growth by avoiding holding at the incoming 120°F pulp temperature for an extended period. This heating also provided thermal-exposure testing since caustic exposed tomato pulp is more susceptible to color and flavor changes than material which has only been processed at the normal tomato pH of 4.2-4.5.

Next the pulp flowed into the Pulp Tank for a final check and recording of pH. A material (mass) balance was made for each trial by weighing the Extractor waste (primarily skin and seeds) and measuring the volume of the recovered pulp. Recovery was determined with 400-1,000 gal. batches; the weight of the recovered pulp and Extractor waste was equal to that of the incoming peel. Recovered peel pulp was concentrated in 1,000 gal. batches at 160-200°F to concentrations of 10-20% TS (total solids) in the cannery single-stage vacuum evaporator.

The pH was measured and recorded at three positions in the process: (1) the incoming peel pH was continuously recorded, (2) the acidification was controlled and recorded at the peel tank or optionally at the hot break, and (3) the final pulp was continuously recorded. The acid diffuser in the peel tank was a 1/2-in. Schedule 80 plastic (Kynar) pipe drilled with 14 holes, 0.062-in. diameter on 1/2-in. centers. This acid diffuser was positioned as shown in Figure 4. Mixing was quite violent at the 350 rpm agitator speed, and the alkaline peel reached pH 4.2 within an estimated five seconds of entering the mixing portion of the tank. The diffuser for the Hot Break Tank had 16 holes, 0.062-in. diameter on 2.75-in. centers, and was positioned at the inlet end of the tank, parallel to the coil axis; the pH electrode assembly was on the outlet end.

Both fresh and canned samples were made up for subsequent analyses. All canned samples were hand filled into size 211 x 400 unenameled cans, sealed with a double seamer, heat processed in boiling water for 40 minutes, and cooled to 100°F in 75°F water. These canned samples were analyzed and judged on a 100% recovered-pulp basis without blending into other tomato materials.

#### 1976 LABORATORY STUDY ON PEELING AIDS

The 1976 laboratory experimentation was primarily directed towards solving the peeling-aid residue problem which was identified during the 1975 pulp-recovery experimentation. In the spring, laboratory peeling tests were conducted on tomatoes using about 70 compounds, including many surfactants (surface-active agents), to determine the chemical structure that best aided peeling. The wetting action (interfacial tension) of the potential peeling aids was checked, but surface-wetting action alone was found to provide little assistance in selecting peeling aids because enhancement of peeling seemed to be primarily due to chemical activity. Potential peeling aids were applied in two ways: (1) directly in the caustic bath in the traditional manner, and (2) as a pretreatment prior to immersing the tomatoes in the caustic bath. The purpose of the pretreatment was to apply only enough peeling aid to permeate the skin and assist the caustic to act more effectively in the caustic applicator. It was also assumed that the

optimum temperature and immersion time for applying the peeling aid might differ from those used for the caustic application.

#### 1976 PILOT SCALE MODIFIED CAUSTIC PEELING

Modified caustic peeling is the term used to indicate the application of the peeling aid as a pretreatment prior to immersion of the tomatoes into caustic (Figure 7). The best peeling conditions found in the laboratory tests were incorporated in a 1-t/hr pilot line at Hunt-Wesson Foods, Plant A, Hayward, California during the 1976 tomato processing season. This line operated on the regular cannery tomatoes, usually Variety UC-134. Final washing, sorting, and peeling were carried out solely on the pilot equipment. Peeling was performed continuously, typically in 45 minute runs, with samples taken during steady-state conditions.

This Hayward cannery received tomatoes usually in bulk 20-t loads (tractor with two trailers) as is typical for California canners. The tomatoes were removed from the trucks by the cannery personnel through a water wash-out and carried by a flume into a sump; from the sump, they were elevated out and spray washed, passed over a screen to remove gross trash and tomatoes less than 1.2-in. in diameter and then flumed. Prior to further cannery washing and sorting, part of the tomatoes were diverted from this flume to the pilot-peeling line.

In the experimental system these tomatoes were immersed in water, elevated out, and passed over a 1-ft x 10-ft rubber disc flat-bed scrubber having water sprays (10 gpm total); this was the final washing. The tomatoes were then passed over a sorting belt for hand sorting; the degree of hand sorting was varied to compare mold counts in the recovered pulp. The pretreatment immersion was in a 17-in. x 10-ft trough having a paddle type, positive displacement conveyor which controlled the immersion time. Immersion time could be varied from 15 seconds to three minutes. The heated solution was recirculated from entry to exit at about 20-gpm, and was controlled and varied from 75°F to 200°F, depending on the experiment. From this pretreatment, the tomatoes were removed on an open-mesh elevator for a variable draining time of 10-seconds to 2 minutes. After draining, the tomatoes dropped into the Caustic Applicator for a 10-sec to 2-min immersion time in 11% (w/w) sodium hydroxide at 210°F. A commercial applicator, such as the FMC Hi-Ton Tomato Peeler, has a drain period of about 50% of the immersion time, which not only removes excess caustic solution but provides a further period for the caustic to act on the tomato. The pilot caustic applicator did not have a similar drain period so this was simulated by a variable-speed, open-mesh belt normally held to 11 seconds residence time. Tomatoes then passed over rotating slitting blades and onto a 12-in. x 10-ft set of flat-bed rubber-disc peel removers which were operated without water sprays to provide undiluted peel. This unit was the same as that used for washing and had discs of 4.25-in. diameter, spaced 0.9-in. apart, on 3-in. centers, and turning at 425 RPM in the direction of the tomato travel; the bed was pitched upward 10-in./10-ft in the direction of the tomato travel which gave a nominal tomato residence time of 30 seconds. This dry removal of peel is increasingly being practiced commercially.

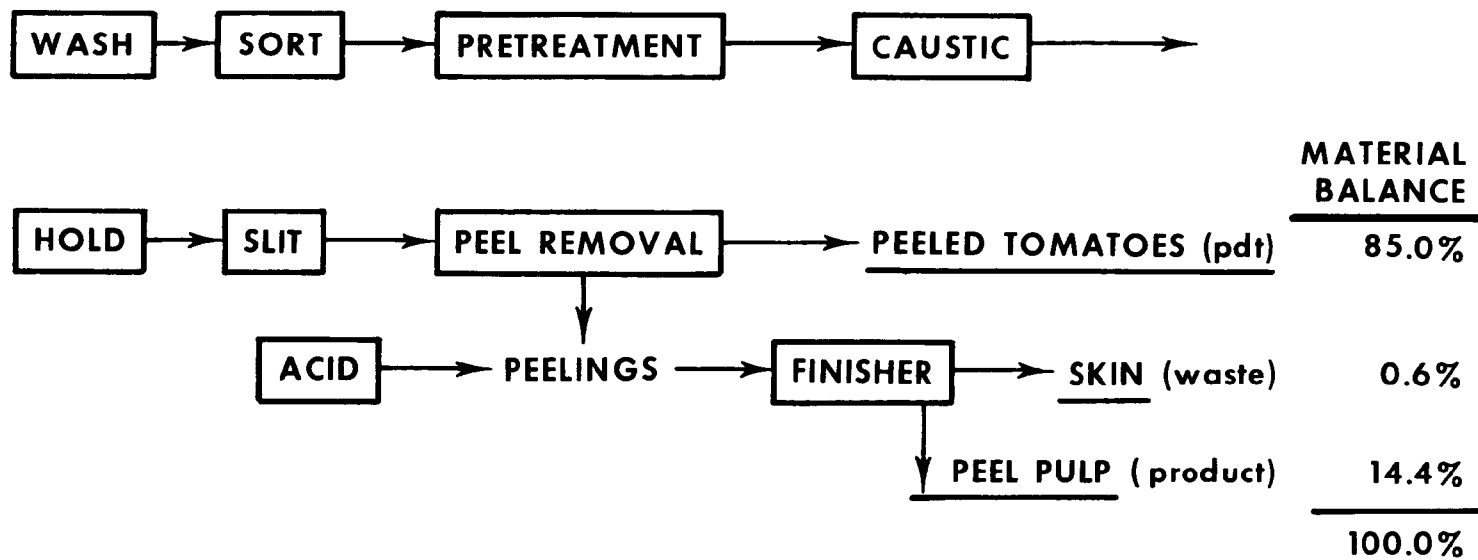


Figure 7. Modified-caustic peeling, 1976.

Peel dropped onto a full-length pan and flowed down to a 10-gal. pot where the peel was acidified, then separated into skin and pulp fractions with a Langsenkamp Indiana Laboratory Pulper (Langsenkamp Inc., Indianapolis, Indiana) equipped with a 0.033-in. screen. This recovered pulp was hot filled at 115-190°F into 211 x 400 enameled cans, processed for 45 minutes in boiling water, and cooled to about 100°F in 75°F water. For a material balance on each trial, both the recovered pulp and peeled tomatoes were weighed, typically 1,000 to 2,000 lb/trial.

A typical set of experiments would involve altering one variable, such as pretreatment temperature, residence time, peeling-aid concentration, or the type of peeling aid. The first run would be with plain caustic preceded by a pretreatment, for example, at 150°F, 0.5 minute immersion in the pretreatment bath, without peeling aid. The weight percent of peeled tomatoes was calculated from the tomatoes in a 10-lb sample which had at least 99% of the peel removed. The pan under the rubber discs would catch the peel which would be swept into a pot and acidified every 4 to 8 minutes with hydrochloric acid. This acidification time corresponded to the delay between peel removal and acidification experienced in 1975. When peeling at 2,000 lb/hr, there was insufficient peel to screen continuously; therefore, the acidified peel was accumulated until the end of a run, then weighed and put batchwise through the finisher to separate recovered peel pulp from the skin and miscellaneous seeds and fiber. The 0.5 inch clearance used in 1975 when processing continuously was inadequate with the small batches. Therefore, a 3/32-in. clearance was used; even so, the 1976 pomace was wetter than in 1975.

## SAMPLE LOCATIONS

### 1975 Stations

Samples of tomato materials were collected at several locations (Figure 2) in both the commercial and experimental processes during each of the test runs. These locations were similar for both 1975 and 1976.

Station 1--Peel residuals were sampled at the peel tank preceding the extractor. Aliquots of approximately 300 ml were collected at the beginning, during the middle, and toward the end of each test run. A composite sample was made of these aliquots in 1-quart plastic containers. In all cases, samples collected at this station were obtained prior to acidification.

Station 2--Recovered pulp samples were collected at the discharge from the extractor in the same manner described above. When acidification of the pulp was conducted after extraction, samples collected at this point were alkaline; when acidification was conducted before extraction, the samples were acidic.

Station 3--Acidified and heated pulp was sampled from the pulp tank following the hot break. Aliquots of 400 to 500 ml were collected about 1/4 hour after steady-state conditions were achieved and toward the end of each run. These aliquots were composited and cooled in 1-quart plastic

containers. These samples were identified as "Sample 3." When additional test conditions were imposed (such as concentrating, blending, or holding), additional single samples of approximately one liter were obtained and identified by an alphabetical suffix (e.g., 3A, 3B, etc.).

Station 4--Recovered pulp was canned at the end of each test run. The hot pulp was obtained from the pulp tank following the hot break, placed into 211 x 400 unlined cans, sealed and heat processed by immersion in boiling water (100°C) for 40 minutes. The cans were cooled by immersion in cold (23°C) water. When additional test conditions were imposed, as described above, additional cans of material were preserved in a like manner. Each set of cans was assigned an appropriate code. However, for analytical purposes, the additional samples were assigned alphabetical suffixes (4A, 4B, etc.).

Station 5--Samples of tomato skin and seeds ejected by the extractor were collected in plastic bags. Approximately 100 to 200 g of material were collected at the beginning, during the middle, and toward the end of each run. These portions were composited for laboratory analysis.

Station 6--Whole tomatoes entering the conventional caustic applicators (baths) were sampled. About 8 to 12 tomatoes were randomly picked at the beginning and at the end of each test run. The two portions were composited in plastic bags for laboratory analysis.

Station 7--Peeled tomatoes exiting the disc peelers were sampled. About 8 to 12 tomatoes were randomly picked from the various units at the beginning and end of each run; the two portions were composited in plastic bags.

Station 8--Juice discharged from the conventional finishers was collected during the middle and at the end of each run. The 450 to 500 ml aliquots were composited and cooled in 1-quart plastic containers.

Station 9--Conventionally prepared tomato paste was sampled about 1/2 hour after each test run. About 300 g of material were collected in plastic bags. When other conventionally prepared tomato products, such as juice or sauce, was used to blend with the recovered pulp, additional samples were obtained. The latter materials were identified by an alphabetical suffix. Appropriate codes were also assigned to cans of these materials.

Station 10--Pomace from the conventional pulpers was collected in plastic bags. Each sample consisted of 100 to 200-g portions which were collected during the middle and toward the end of each test run.

All uncanned samples were held on ice during the compositing period, as well as during transport from the test site to the laboratory. Upon delivery to the laboratory, the samples were immediately placed under refrigeration (4°C). Analyses were performed as quickly thereafter as practicable.

Samples which were collected for ascorbic acid (Vitamin C) determinations (dichlorophenolindophenol method) were immediately weighed (20 g) and mixed with a solution of metaphosphoric - glacial acetic acid. The acidified samples were held on ice until delivery to the laboratory. These samples

were then frozen until analysis could be performed. Samples collected for  $\beta$ -carotene (Vitamin A) determinations and protein analysis were also iced until delivery and frozen in the laboratory prior to analysis.

The canned samples were cooled at the test site, transported to the laboratory, and held under refrigeration (4°C). This precautionary measure was taken to preclude spoilage, a concern due to the uncertainty of adequate processing at 100°C.

### 1976 Stations

The 1976 sample positions corresponded to those in 1975. Recovered peel pulp was canned as a single strength, acidified pulp, then heat processed after sealing. All samples were taken from the pilot line, none from the cannery. For instance, the fresh, washed tomatoes came from the pilot sorting belt which corresponded to the 1975 Station 6, and so forth. Some samples, such as the caustic bath, were from a similar station as in 1975, but of a different character. For instance, the caustic in 1976 had to be changed between each trial to eliminate prior materials affecting subsequent tests; therefore, the 1976 caustic was "fresh" and the 1975 caustic was "mellowed", a point which would not show up based on caustic content. A "water dip" was used to cushion the fall of the tomatoes as they came off the disc peeler, and served to rinse the tomatoes before they were elevated into barrels for weighing. Since this was not a typical cannery bath or flume, samples would not have been representative of a commercial situation.

### ANALYTICAL METHODS

In both years the collected samples were analyzed for parameters considered most significant for the respective sampling location. Emphasis was placed on obtaining data relative to the quality of the recovered pulp. The significance and major parameters evaluated for each sampling location are identified below. Most of these analyses were conducted on frozen and canned samples at the NFPA, Berkeley and the WRRRC, Albany laboratories. Fresh material was analyzed in the laboratory trailer at the experimental site. Field analyses during 1975 were primarily Agtron (M500-A) and Munsell colors, salt, pH, and alkalinity, carried out on the incoming peel and acidified pulp; in 1976, these analyses were NTSS, pH, alkalinity (for acid requirement), peeling efficiency, lye concentration, and serum viscosity. The purpose was to provide guidance for the experimental processing because the Berkeley and Albany laboratory data would not be available until after the experimental period. Where there was duplication of data from the field and research labs, the correlation was good. Additional details pertaining to the analytical procedures may be found in the references cited.

pH--The pH of all samples was measured to determine the relative acidity or basicity of each. Since the pH of solutions greatly influences the resistance of microorganisms to heat, as well as their ability to grow, the Federal Food and Drug Administration has arbitrarily established pH 4.6 as the value differentiating low-acid products from high-acid commodities.



Thermal processing regulations have been promulgated for low-acid foods (9). The natural pH of raw tomatoes processed in California normally fall pH 4.2 and 4.4.

Measurements were made electrometrically by direct insertion of a combination electrode into fluid sample. The pH of semi-dry pomace/skin samples was obtained by adding 2 to 3 parts (by weight) of water to 1 part of sample, stirring, and measuring after equilibrium had apparently been attained (about 30 minutes). Whole tomatoes were homogenized prior to pH measurement.

Total Solids (TS)--Since most commercial tomato products are concentrated to varying degrees, the amount of moisture present in tomato pulp is of interest. Additionally, the amount of moisture present in tomato pomace may serve as an index of the operating efficiency of pulpers and finishers which are used to extract pulp (juice) from crushed tomatoes. Moisture was determined by drying an appropriate weight of sample, based on an approximation of the solids percentage in the sample, in a vacuum oven (29 inches) for 4 hours at 70°C. Total solids were then calculated and expressed as "percent dry weight" (10). As tabulated, the total solids data include salt.

Natural Tomato Soluble Solids (NTSS)--Estimation of the total solids and specific gravity of tomato products may be obtained by measuring the refractive index (Brix) of the tomato serum (11). Natural tomato soluble solids are also the basis for establishing United States Standards for Grades of tomato products. Therefore, refractive index is commonly used by the industry in quality control. Direct readings at 25°C were taken for most of the samples collected during the study. Serum from samples containing higher solids percentages was obtained by centrifugation. All acidified samples were corrected for salt; results are expressed as "percent sucrose."

Salt (Sodium Chloride)--Salt is a commonly used seasoning for many tomato products. Since sodium chloride is formed by neutralization of sodium hydroxide with hydrochloric acid, the quantity of salt in the recovered tomato pulp was of interest. Total chlorides were measured by potentiometric titration with silver nitrate and the use of a silver electrode (12). Laboratory results are expressed as "percent salt (sodium chloride.)".

Alkalinity, Total--Total alkalinity measurements were conducted to determine the relative quantity of residual caustic (sodium hydroxide) contained in the collected samples. A 5.0 g sample was titrated with 0.1N H<sub>2</sub>SO<sub>4</sub> to an arbitrarily selected end-point of pH 4.2.

Consistency and Viscosity--The consistency of tomato products is affected by the amount of and extent of degradation of pectin, as well as the size, shape and quantity of thixotropic and insoluble materials contained therein. The United States Standards for Grades of tomato products specify limits for this parameter. Therefore, consistency measurements were made with a Bostwick Consistometer; results are expressed in centimeters in 30 seconds (14). Viscosity measurements, performed on free-flow samples, were

made by timing a specified volume through a capillary tube (15). In general usage, "consistency" is the term for the fluid-flow resistance of viscous foods, such as tomato pulp, with a relatively great amount of insoluble solids; "viscosity" is used for those fluids without appreciable insoluble solids, such as tomato serum.

Samples (chilled, then frozen) were checked for possible pectin enzyme activity before the hot break. Samples of alkaline peel pulp were acidified to approximately pH 4.2 either: (a) manually in the field laboratory or (b) automatically in the process (before the hot-break). Then they were cooled below 70°F within 5 minutes of acidification, packed in ice for about 3 hours, and stored at 0°F until evaluated for pectin enzyme activity. In one case the process acidified sample was held, without hot-break, for 90 minutes at 100 to 120°F before chilling and then stored cold in the manner described above; this hold was to check short term color and microbiological stability.

The activity of the pectic enzymes in the acidified samples was determined in 1975 by measuring changes in consistency after mixing the test samples with a high consistency tomato juice, which served as a pectin substrate. The test sample was added to the substrate at a ratio of 1:10, and the consistency was measured at 25°C over a period of 1 hour or more by the efflux-pipet method.

Further serum viscosity tests were made in 1976 by neutralizing the caustic with acid before peel removal so as to isolate viscosity changes occurring in the caustic bath from those taking place afterward.

Vitamins--Tomatoes and tomato products are considered to be significant sources of Vitamin A ( $\beta$ -carotene) and Vitamin C (ascorbic acid) in a normal adult daily diet. Both of these nutrients can be degraded through chemical treatment, heat, and/or oxidation. Since the recovered pulp was subjected to all three of these conditions, the vitamin content of the final recovered material was of concern. Vitamin A was determined spectrophotometrically; results are expressed as "International units per 100 g" (16). (Appendix A).

The initial analysis of Vitamin C (ascorbic acid) was made by the 2,6-dichlorophenolindophenol visual titration method, which is unsuitable if there are other reducing agents present or if some of the Vitamin C activity is due to dehydroascorbic acid (25). Inorganic reducing agents are usually minimal in tomato products but organic reducing substances, collectively termed reductones, are often formed in products which have undergone extensive heat treatment. Such reductones are formed by the action of alkali on sugar. An alternative method that avoids interference by inorganic reducing agents and assays total Vitamin C by including dehydroascorbic acid is the method of Roe and co-workers (17). This method involves the oxidation of ascorbic acid to dehydroascorbic acid, subsequent transformation of dehydroascorbic acid to diketogulonic acid, and coupling of this product with 2,4-dinitrophenylhydrazine under carefully controlled conditions to give red-colored osazones. A comparison of the color produced in samples and

ascorbic acid standards is used as a means of determining ascorbic acid content. The absorption of light by the pigment formed is maximum at a range of 510 to 520 m $\mu$ . Since any reductones present form osazones that absorb in the region of 450 to 490 m $\mu$  and these absorb more intensely than the red osazone of ascorbic acid (i.e., diketogulonic acid), a more accurate assay of ascorbic acid in the presence of reductones is obtained by reading absorption at 540 m $\mu$  rather than at 510 to 520 m $\mu$ . However, if reductones are present in large amounts, high estimates of ascorbic acid will nevertheless be obtained due to the interfering absorption of the reductones; therefore, a chromatographic separation is required to separate the osazones of ascorbic acid from those of the reductones before making absorption readings at 520 m $\mu$ .

The usual chromatographic separation of osazones by both column and thin-layer chromatography was modified to a separation by column chromatography alone, using a development solvent of acetic acid, ethyl acetate, and methylene chloride (3:20:97). In all cases, a sample with a known level of ascorbic acid added was also assayed by chromatographic isolation and colorimetric determination of the osazone at 520 m $\mu$ .

Peeling Aids--Peeling aid additives are carried out of the caustic baths by the tomatoes. Residues are removed from the peeled product during the peel removal and washing/rinsing operations. These residues appear in the peel residual and in the effluent wash waters whenever the latter operation is performed. Samples collected from the test system were analyzed for peeling aids; selected samples from conventional operations served as controls. Concentrations of sodium 2-ethylhexylsulfate and sodium mono- and di-methyl naphthalene sulfonates in appropriate samples were determined by a methylene-blue dye transfer method (Appendix B). Fatty-acid peeling aids were determined by esterification of the fatty acid and quantification by gas-liquid chromatography (GLC) (Appendix C).

Pesticides--The samples were extracted by the method in the Food and Drug Administration's Pesticide Analytical Manual (19). The extracts were cleaned through activated florisil. Toxaphene and thiodan were detected in their respective florisil eluates by GLC using the electron-capture detector. Parathion was detected by GLC using the flame photometric phosphorus detector. All pesticide analyses were performed by Stoner Laboratories, Santa Clara, California.

Toxaphene is a complex mixture of compounds manufactured by the chlorination of camphene and related compounds. Treatment with base caustic in the laboratory causes dehydrochlorination which appears during GLC as a shift to earlier eluting compounds (using methyl or phenyl silicone columns). A similar shift to earlier eluting compounds can be seen in old toxaphene residues in environmental soils, and similar shifts were visible in some samples.

By varying the strength of base used during hydrolysis, standards were synthesized with varying degrees of decomposition (as evidenced by their shift to shorter retention times). The toxaphene in some of the samples was like the (Stoner) mildly decomposed standard, and some were like the (Stoner) strongly decomposed standard.

Mold and Insect Fragments--The Federal Food, Drug, and Cosmetic Act in Section 402(a)(4) precludes the sale of products which "may have become contaminated with filth", whether or not such contaminants may pose hazards to health. "Defect action levels" have been established by the FDA for natural or unavoidable defects in foods for human use where no health hazards exist. Mold and insect fragments, resulting from field, transportation or storage conditions, fall into this category. Since these exist largely on the surface of the raw product, there was interest whether the pulp recovered from peel residuals might contain excessively high counts of these contaminants. Randomly selected canned samples were microscopically examined for mold and insect fragments (20). Mold is reported as percent of positive microscope fields; insect fragments are reported as total counts of fragments per 200 g. (Note: defect action levels are established for *Drosophila* fly eggs and/or larvae per 100 g).

Bacterial Counts--Tomato pulp samples were taken before and after the hot break, and checked for mesophilic (30°C incubation), thermophilic (50°C incubation), and total plate counts (30°C incubation). Plating was on glucose-tryptone agar. Vegetative cells (mesophilic) are more heat labile than the spores (thermophilic) so comparing the counts can indicate survival and reinoculation, equipment cleanliness and heat processing adequacy.

Quality Factors--The United States Standards for Grades of Canned Tomato Puree (Tomato Pulp) establishes the criteria classifying the quality of canned product (21). Factors for which scores are assigned are color and defects (dark specks, seeds, tomato peel, and other extraneous materials); non-scored factors are subjective evaluation of flavor and odor. The texture of graded samples are also routinely noted.

The above quality factors were evaluated by inspectors of the Fruit and Vegetable Quality Division, Food Safety and Quality Service, U.S. Department of Agriculture, Stockton, California. Color scores were determined by the specified Munsell color disc comparison procedure or by the use of a Hunter colorimeter. Color and defect scores are based on a maximum of 50 points for each factor. Canned recovered pulp samples were submitted for quality evaluation. These results were of primary concern since they would largely determine the commercial acceptability and utility of the recovered materials. In the lab trailer, color was measured with the Agron and Munsell instruments during 1975 but not during 1976.

Nitrogen and Amino Acids--Tomato pulp samples were analyzed for total nitrogen content and basic amino acids before and/or after centrifugation into soluble and insoluble fractions. The two fractions were obtained by centrifuging samples at 35000 G for 10 minutes. The insoluble solids were washed twice by re-suspending in distilled water to about 1.5 times the original volume and centrifuging. The washes were combined with the soluble fraction, and the solubles were concentrated by vacuum distillation at 45-50°C to near their original volume. In all cases the assay results for a fraction (soluble or insoluble) were expressed on the basis of the whole sample weight equivalent. The total nitrogen content of each sample was determined by Kjeldahl procedure (22). On the basis of the Kjeldahl results,

samples of 0.8 to 3.2 mg N were hydrolyzed in 6-N HCl (33). After the HCl was thoroughly removed by vacuum distillation at 45 - 50°C, the sample was concentrated to dryness with a rotary evaporator and suspended in a measured volume of pH 2.2 buffer. Then the sample was filtered and the protein hydrolysate was analyzed for amino acids using an automatic amino acid analyzer (Beckman Model 120, Phoenix Model K8000 B, or Durrum D-500) (23).

## SECTION 6

### RESULTS AND DISCUSSION

#### General

The results from 1975-76 showed that tomato pulp recovered from caustic peelings has food potential. Product grades varied according to the processing method, and these results showed the necessity of processing the tomato pulp with care so as to achieve its full potential. Experimentation spanned two years. In 1975, tomato peel was taken directly from a commercial caustic peeling operation; this recovered pulp was characterized as to its color, grade, and conformity to the Standards of Identity. In 1976 process changes were made, then tried on a pilot peeling line to minimize the peeling-aid residuals found in the 1975 recovered pulps.

While the 1975 results (Table 1) showed there is food potential in recovered pulp, there was one major detraction--the peeling aid residue at 150-450 ppm. Of the currently used commercial peeling aids (sodium 2-ethylhexyl sulfate, sodium mono- and di-methyl naphthalene sulfonates, or fatty-acid mixtures containing predominately odd-numbered carbons), none seem suitable for clearance as food additives. Even if an additive-grade peeling aid were currently available, the 150-450 ppm residue is large enough to raise questions about declaring it on the product label as an additive.

While it is possible to peel tomatoes without a peeling aid, it is generally acknowledged that higher caustic concentration or increased temperature is required; these lead to higher peel losses, and as a result, the peeled tomato quality suffers since the vascular veins become more pronounced. The peeling aid residue level possibly could be reduced to less than that found in 1975, but it was considered improbable to reduce the residue to near zero once a peeling aid was introduced into the caustic peeling system. Therefore, a modified caustic peeling was developed in 1976 by using food grade chemicals as peeling aids and applying them as a precaustic treatment instead of adding directly to the caustic bath.

In normal cannery operations, the juice and pulp supplies are interconnected so that these materials can be shunted between the different sources and utilization points to satisfy changing production requirements. Therefore, in actual cannery practice recovered peel pulp could be combined with juice and macerate from other sources before processing into standard products such as tomato sauce, catsup, fill juice for whole-canned tomatoes, or other salted products. During acidification and possibly at the rubber-disc peel removal point, a small amount of water might be incorporated into

TABLE 1. SUMMARY OF TYPICAL 1975-1976 RECOVERED PEEL PULPS

Item	Typical Single-Strength Tomato Pulps		
	1975	1976	Conventional Processing (a)
Recovery of peel %w/w	96.7%	95%	n/a
NTSS, (natural tomato soluble solids), %w/w	5.3	5.2	5.4
Salt, (sodium chloride), g/100 gm	3.-	1.1	0.08
Total solids, (salt free), %w/w	5.6	5.9	5.71
Vitamin A, (beta-carotene), I.U./100 gm	665	---(b)	516
Vitamin C, (ascorbic acid), mg/100 gm	nil	nil	10.7
Color grade, (per puree std)	A-C	A	A
Flavor grade, (per puree std)	C	A-C	A
Peeling-aid residue, pulp ppm (c)	150 - 450	0 - 30	0 (d)
Insecticide residue, pulp product, toxaphene ppm (f)	0.4	trace	trace
Insecticide residue, skin waste, toxaphene ppm	5-60	34	7 (e)

trace positive amount less than 0.08 ppm.

n/a not applicable (no current commercial recovery).

(a) industry 4-yr averages, except salt which is 2-yr average.

(b) not analyzed.

(c) sodium 2-ethylhexyl sulfate in 1975, octanoic acid in 1976.

(d) no peeling aid used in conventional juicing/pulping.

(e) pomace from juicing, seeds with proportionally less skin than in peel-pulp recovery.

(f) tolerance is 7.0 ppm in canned and fresh tomatoes

the peel. This water must be removed, either through subsequent concentration such as for tomato sauce, or during the evaporation that occurs from holding tanks. This water could be removed either before or after combining with other pulps. There seems little processing justification for keeping recovered pulp isolated for use in a special product since there is an insufficient quantity of recoverable pulp to maintain a separate processing line.

The various process and product aspects are discussed below, including the unit operations, recovered pulp characteristics, and economic and regulatory considerations.

Washing and Sorting--Peel pulp recovered during the 1975 season from the typical cannery peeling operation (Tillie Lewis Foods, Plant W) had low mold, insect fragment, and bacterial counts that were well within regulatory tolerances. This showed that the cannery had an excellent washing and sorting system consisting of counter-current water flow and fine sprays, all with a relatively low total water usage of 750 gal/ton of tomatoes for the entire plant. Tomato washing consisted of the primary dump tanks, a secondary flume, a third flume, and a final fresh-water rinse as the tomatoes passed over roller conveyors. Water was chlorinated to 5 ppm. In 1976 tomatoes were received before commercial processing and washed in the pilot systems; the final rinse over rubber-discs corresponded to the previous year's use of rollers. Sorting in 1976 was varied from zero to 20% of the peeled tomato weight; the higher sorting was necessary when the California State Grade Certificate showed 3% mold. While the Grade Certificate is an indication of mold on in-coming raw product, the tomatoes were graded up to 24 hours prior, so the actual mold count could have been higher at the time of experimental processing. An alternative to hand sorting is the use of high-pressure water sprays (70-120 psig) as practiced for a number of years by most canners to remove broken and moldy tomatoes.

Washing and sorting methods to remove contaminants from raw commodities vary between canneries. A guide to the tolerances for the so called "natural or unavoidable defects" in tomato products, such as mold, rot, insects, etc., is published periodically by FDA (30).

Peeling Aids--A peeling aid (also called surfactant, surface-active agent, wetting agent) is used by all commercial canneries in the caustic applicator (bath). The purpose of the peeling aid is to promote uniform peel removal. Without it, yellow shouldered areas on the tomato tend not to be peeled. While a caustic peeling solution alone may peel satisfactorily on occasion, there are times when changing the temperature, immersion time, or caustic strength is not sufficient to effect quality peeling. Particularly at such times, the addition of a peeling aid can improve peeling in a rather empirical manner. If the tomato is given a prolonged submergence in the caustic or if the caustic concentration is increased, the peeling losses rise and the vascular (white) veins of the tomato become more prominent. The result is a poorer quality product and greater loss. These comments pertain more to the relatively "tough skinned" VF-145 processing tomato variety which accounts for perhaps 60% of the processing tomatoes grown in the U.S.A. There are various materials



permitted as washing or peeling aids (26), but none of these are permitted as additives in tomato products. Their use is permitted with the provision that after leaving the caustic applicator, the tomatoes will be washed with water, and there will be negligible peeling aid remaining on the whole tomato. The permitted aids are water soluble; hence, water is the conventional means of removal.

Sodium 2-ethylhexyl sulfate (Emersal 6465; Emery Industries Co., Cincinnati, Ohio) was used at the cannery during 1975. Subsequent experimentation showed that sodium mono- and di-methyl naphthalene sulfonates and fatty acids responded similarly with respect to residues in the recovered pulp. The sensitivity of the analytical method for sodium 2-ethylhexyl sulfate is about 25-50 ppm because natural tomato constituents interfere slightly with the analysis. The ratio of peeling aid to sodium in the caustic applicator was about the same as that found in the recovered peel pulp. The levels of peeling aid in the peel pulp were usually in the range of 150-450 ppm; such levels are not acceptable for tomato products. No peeling aid residue was found on peeled whole tomatoes after peel removal. The detection level was about 25 ppm for sodium 2-ethylhexyl sulfate and 1 ppm for fatty acids.

During the spring 1976 laboratory peeling study, the C<sub>6</sub> and C<sub>8</sub> saturated, monocarboxylic fatty acids were the most effective peeling-aids. Octanoic (caprylic) acid was the principal one used because it was as effective and less volatile than hexanoic acid. It is commercially available in a food grade (27) at a price competitive with peeling aids currently used in commercial tomato peeling. The 150°F pretreatment was as effective, or more so, as using the peeling aid directly in the caustic applicator. For easy-peeling tomatoes, such as those suitable for steam peeling, it was possible to peel without caustic by using a 1 to 2 min. immersion in a 150°F aqueous bath containing 0.2% w/w octanoic acid. While it may be convenient to apply the peeling aid with caustic, there is no inherent reason why it requires the same application temperature, pH, and immersion time as the caustic.

Peeling aids are commonly referred to as "wetting agents", but the most effective ones may do more than reduce the interfacial tension between the tomato surface and the caustic. Some wetting agents, such as sodium oleate or sodium lauryl sulfate, will show high-wetting improvement, but they will have little effect on peeling whether applied as a pretreatment or directly in the caustic bath. Others, such as sodium 2-ethylhexyl sulfate and sodium mono- and di-methyl naphthalene sulfonates, perform better when applied directly in the caustic bath than when used as a pretreatment. The most effective peeling aids appear to react chemically and (or) to disrupt the cell structure and allow enzyme action. This is illustrated by peeling tomatoes with only an acidic aqueous solution of octanoic acid at 150°F; this is discussed further under "Carboxylic Acid Peeling".

Octanoic acid does have a characteristic aroma (rancid, cheesy), and is used at up to about 400 ppm in synthetic blue cheese. In single strength tomato juice, less than 10 ppm is indistinguishable from plain tomato juice. The flavor threshold is about 20 ppm, and 50 ppm has a flavor identifiable as a fatty acid. This threshold would be higher in spiced sauces. This 20 ppm threshold is significantly higher than the 10 ppm proposed level for

general use in the GRAS affirmation under good manufacturing practice as a direct food ingredient as published in the June 1977 Federal Register (28). People differ in their threshold sensitivity to caprylic acid, but in another study, 19 ppm was found to be the odor threshold for a normal person to caprylic acid in buffered water at pH 4.8 (32).

Carboxylic Acid Peeling--The carboxylic acid peeling was accomplished with a 150°F aqueous solution containing 0.2% octanoic (caprylic) acid at about pH 3.6 and with a one to three minute immersion. This completely peeled the tomato varieties Tropic, Walter, Roma-VF, and VF-145-21-4 (contains a uniform-ripening gene). For the VF-145B-7879, UC-134, 198, and 13L, which are typical California processing tomatoes, the skin was loosened and peeling aided, but a subsequent caustic application was generally needed. The peeling loss averaged about 5% with octanoic acid as compared to 12% for caustic using commercial peeling aids. The difference was visually dramatic because caustic peel was red due to the adhering pulp, whereas the octanoic acid peel was a translucent, pale yellow because no pulp adhered. Other secondary treatments than caustic were atmospheric steam (30 sec.) and 900°F superheated steam (7 sec.). Both steam treatments provide additional peel loosening capability, particularly the 900°F steam which splits the skin and makes peel removal easier.

While octanoic acid performed best among the candidates as a peeling agent, all the C<sub>4</sub> to C<sub>8</sub> saturated monocarboxylic acids showed the most promise. Time was limited and an extensive pursuit of the ideal peeling agent or aid was not feasible. Octanoic acid occurs naturally in coconut oil and blue cheese, is readily available commercially in a food grade, and is priced similar to the currently used peeling aids. A food-grade peeling aid should be biologically metabolized in predictable fashion by both humans and animals or microorganisms associated with man, and octanoic acid fits this requirement. This is currently being considered in a proposed affirmation of the GRAS status of caprylic acid (28). With the wide-spread use of steam peeling in Europe, the carboxylic-acid peeling seems to be potentially competitive or an aid to steam peeling because of peeling varieties such as the San Marzano and Roma (31). The use of octanoic acid not only could improve peel removal, but it could reduce the duration of steam exposure and heat which softens the tomatoes unduly and increases peel loss.

Peeling Aid Pretreatment--During the 1976 tests pretreatment temperatures were varied from 75°F to 210°F. Initially, 150°F was chosen so as to be below enzyme-inactivation temperature. Experimentation showed that below about 140°F the peeling aid pretreatment was less effective or required long immersion, such as up to 10 minutes. Above about 170°F, even with a short dip, the tomatoes became increasingly soft and the peel loss increased. Overall, 150°F was best, with the 140°F to 160°F range being practical. Even a 150°F water (100%) pretreatment usually showed some peeling improvement, but use of a peeling aid was definitely better.

When a pretreatment is used, fatty acids offer a potential advantage over sodium 2-ethylhexyl sulfate (SEHS) and sodium mono- and di-methyl naphthlene sulfonates (SNS) because they tend to loosen the tomato skin while in an aqueous solution as discussed under "Carboxylic Acid Peeling".

The limited data and visual observation indicate better peeling when hexanoic (caproic) and octanoic (caprylic) acids were used as a pretreatment than when used directly in the caustic applicator. When the peeling aid is used directly in the caustic applicator, then all four of these peeling aids were about equally effective on an active ingredients basis. As commercially sold, the fatty acid peeling aids contain 100% active ingredients, and the SEHS and SNS contain 50% as these latter ones are distributed as water solutions. Although all peeling aids were used singularly in this experimentation, mixtures of fatty acids and other chemicals may offer additional peeling benefits, and further study would be beneficial. One example is that octanoic acid has a limited solubility of 0.12% in water at 150°F, and this level can be increased by the addition of acetic acid which will act as a co-solvent. The object of this work was to find a food-grade peeling aid and application method to obtain the full benefits of a peeling aid and to minimize residuals in the recovered pulp. Hence, the data were not necessarily of a nature to differentiate relative effectiveness of currently used peeling aids.

Modified-Caustic Peeling--The term "Modified Caustic Peeling" is used to indicate the tomato pretreatment with a peeling aid prior to the caustic applicator instead of the standard commercial practice of putting peeling aid in the caustic bath.

The 1976 cannery pilot peeling was mostly with the tomato variety UC-134 because it was usually the only tomato available at the cannery. This variety is also considered at least as difficult to peel as the more prevalent VF-145-7879. When the VF-145, 198, and 13L were peeled, they responded similarly to that of the UC-134 with respect to peeling aid pretreatment efficiency and residue in the recovered pulps.

When comparing peeling methods with a pilot line, it is possible to simulate commercial conditions but nearly impossible to simulate all aspects of the actual scale. Tomatoes change with each truck load and even within the load. Duplicating caustic solution is more than similar temperature and caustic concentrations because the usual cannery solution takes about one week to reach equilibrium between caustic and disintegrated tomato solids. Either the industry collectively, or each processor individually, will need to consider long term process factors such as: (a) pretreatment bath stability and replenishment frequency, (b) peeling aid carryover to the caustic bath and the peeling aid (octanoic acid) level in the recovered pulp, and (c) safety features such as the need of flow diversion for off-control material.

Peel Removal--Any peel removal system can be used to supply a pulp recovery system if the recovered pulp is not diluted with water. Water sprays may exist on peel removal systems either to provide lubrication, rinse peel from the equipment, or to remove peel from the tomato. If the water added to the peel is incidental, such as 1% of the peel weight, then this amount probably would not be significant if it is removed during further processing or by normal evaporation from holding tanks. Both the FMC PR-20 Tomato Peel Remover and the Magnuson Model C Peel Scrubbers supplied peel for the 1975 recovery. In the FMC remover, the peel is removed in two stages; the first stage is a horizontal bed of rubber discs, and the second is a flat bed of "pinch" rollers. In this case, peel was taken only

from the rubber disc section because the roll section had water sprays. In the Magnuson, the peel comes off in a one-stage rotary cage, disc unit. Both machines can provide recoverable peel in a production situation, and the choice between these or other machines would seem to be a matter of individual preference.

A flat-bed disc unit was used in the 1976 pilot peeling tests. The peeler was quite sensitive to the degree of loading because peel removal depends not only on the discs contacting the tomato but also on the inter-tomato contact and rubbing. With a 1-ft wide pilot peeler loaded at 1-t/hr of tomatoes, there was not sufficient inter-tomato contact for optimum peel removal. An 8-in. width would have been more appropriate, but the width would have introduced another variable and was therefore kept at a constant 12-in. for all trials. When the flat-bed type is used commercially, it is often used in combination with "pinch" rolls as on the FMC unit; the pilot system would have benefited from such "pinch" rolls.

Peel Flow--In 1975 the equipment was initially operated at 20-30 gpm, which was the full flow coming from the cannery. Although the full flow could be handled by the experimental equipment units, surges in the flow taxed the heating capacity of the hot break to the limit of the available steam pressure (45 psig). Also, overflows from the caustic applicators were discharged into the peel troughs, creating an acid demand that exceeded the acid flow capacity; the caustic overflow was a local condition which could not be modified at the time. Subsequently, peel flow into the peel tank was controlled to about 15 gpm by installing a 1-inch angle valve at the peel tank. This valve controlled the peel flow reasonably well, reduced the effect of the caustic overflow, and provided more stable heating and pH control. However, tomato skin occasionally clogged the valve port. These flow stoppages were corrected by installing a positive-displacement pump (Waukesha 55 D0, 130-520 rpm variable-speed drive, Waukesha Foundry Co., Waukesha, Wisconsin) to meter in the peel material. Later, the caustic carryover into the peel rose to the equivalent of 3% salt; to avoid excessive acid consumption, the peel flow was reduced to about 10 gpm.

The 1976 system peel dropped from the rubber discs onto an underlaying pan. This peel was then manually scraped into the acidification container about every 4 to 8 minutes. Peel flow was typically 0.4 gpm. The times from tomato immersion in the caustic applicator to peel removal, and to acidification were similar to 1975. The 1976 operation simulated a normal peeling operation and did not have to contend with the unreasonable caustic variations experienced in 1975. The contrast between these two years served to emphasize the production control required.

Peel Alkalinity--The daily (1975) measured acid use is shown in Table 2, and laboratory pH adjusted alkaline (caustic) pulps are shown in Figure 8. This provides guidance for estimating acid requirements which are expressed as pounds of acid used per pound of tomato solids (w/w). If the caustic applicators had not been piped to overflow into the peel, the salt in the recovered acidified peel pulp would have been about 1.1% as in 1976. The present trend among processors is to recycle any overflow as makeup to the

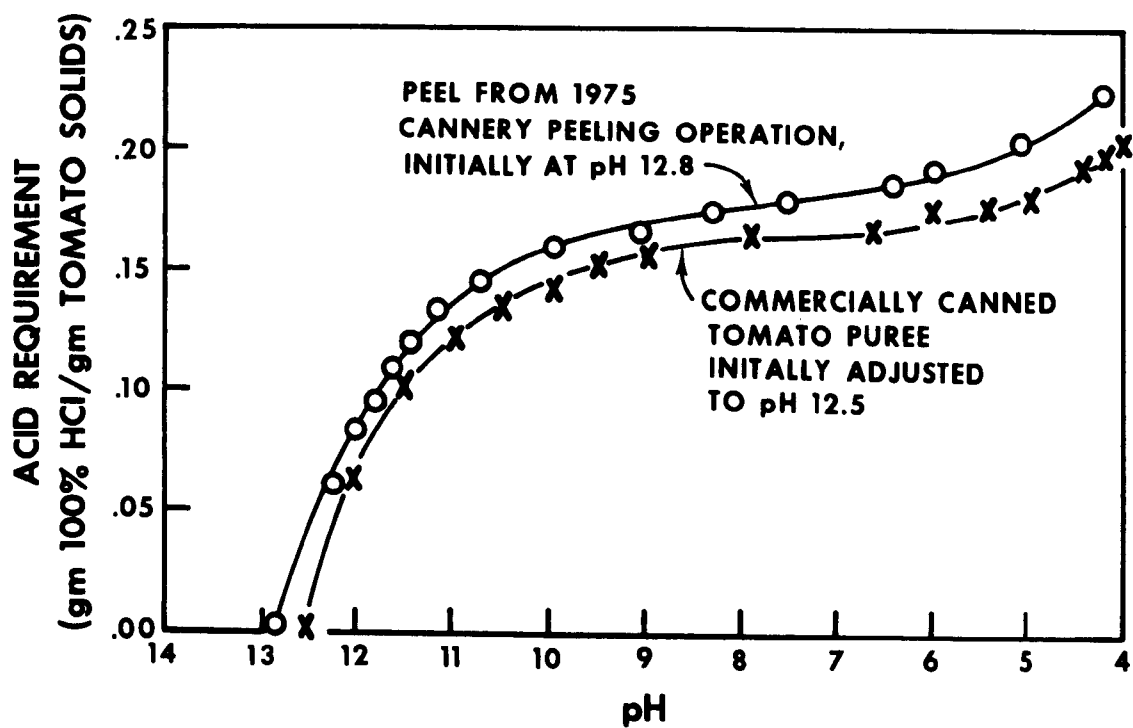


Figure 8. Acid required to reduce pH of caustic peel and pulp.

applicators after separating suspended solids, and to empty the caustic applicators less frequently than once per week.

Acidification--Early and rapid acidification is necessary because the tomato character is more stable at the natural pH 4.2 than at pH 11-12. At the high pH both color and flavor deteriorate with time and temperature, though microbiological growth is inhibited. Once acidification commences, a rapid decrease, in less than 10 seconds, from pH 11 to 4.2 is necessary because an off odor was sometimes detected when the acidification paused at pH 6-10 for several minutes.

Acidification time for the incoming peel was estimated to be 5-10 seconds to reach pH 4.2 in the peel tank, and 30 seconds when acidification was in the hot break vessel. If the peel was allowed to stand between approximately pH 6 to 10 for about 30 minutes, particularly above 100°F, off odor (sulfury) was pronounced; the odor seemed most objectionable at about pH 9. Color increasingly darkened from pH 10 down to about pH 7, then improved below pH 5. Below pH 5, the color was good down to pH 2. Peel received and held at pH 12 generally had a better color than if reduced and held at pH 7-10.

Acidification can be performed either before or after the skin is separated from the pulp. Since pulp recovery was 96% or better, there was little reason to be concerned with acid economy by acidifying after the skin removal, although both were tried. This leaves the processor the freedom of ultimately separating pulp and seeds either in an individual machine or combining this separation with other pulping operations in the cannery. For general pH control, ease of operation, and product quality, acidifying in the peel tank was best. The tank configuration shown in the diagram (Figure 4) was suitable for pH control at flows up to 20 gpm.

There is a definite preference for acidifying the pulp before heating since pulp held in the alkaline state is more susceptible to thermal discoloration (browning). There was no significant recovery difference in product or extractor operation whether the peel was extracted in an alkaline or acid condition.

Salt (Sodium Chloride)--Acidification of the 1976 caustic peel resulted in about 1.1% salt in the recovered pulp (Table 6). This salt content is similar to that found in tomato sauces. Raw whole (unsalted) tomatoes contain 0.08% w/w salt; canned peeled whole tomatoes, 0.4%; canned tomato juice, up to 0.8%; catsup, 2.7%; and special sauces, such as chili, cocktail, and pizza, up to 3% salt.

The amount of salt is a reflection of the caustic in the peel. As previously explained in this report, the (high) 3% salt in 1975 (Table 2) was a unique, atypical condition due to the caustic bath overflowing into the peel. Therefore, it is the nominal 1.1% salt which needs to be considered when utilizing recovered pulp both as a source of pulp and salt. To control the salt content in the final product, the salt from acidification can be managed in a number of ways, two of which are: (1) the salt content can be continuously monitored (analyzed) through either automatic or manual titration

for chloride or by selective ion electrode for sodium, and when blended with conventional unsalted pulp material, the salt normally added to products can be adjusted accordingly, or (2) the recovered pulp can be blended with conventional unsalted pulp so that the salt contribution will be insignificant, and the regular salting procedure can be used.

Pulp Extraction--The basic pulp extraction information was obtained in 1975 on a continuous flow basis at rates which would be similar to a

TABLE 2. ACIDIFICATION DATA (1975)

Date (1975)	Acidifying period (minutes)	Peel pulp flow (gpm)	Caustic Application (% w/w NaOH) (a)	Acid Used g 100% HCl g tomato solids (b)	Salt in Peel pulp (% NaCl)
Sep 03	180	--	20.	0.19	--
05	90	--	30.	0.15	--
08	180	--	6.9	0.22	1.8
09	120	--	16.7	0.17	1.9
11	237	--	15.5	0.30	5.4
16	105	10.0	16.7	0.--	--
17	--	--	17.5	--	2.6
am 19	135	12.8	9.6	0.34	3.3
pm 19	100	--	10.	0.21	2.6
22	90	10.6	8.	0.42	3.3
am 23	114	10.6	8.	0.34	3.4
pm 23	90	--	7.9	0.32	3.0
24	120	10.0	7.7	0.39	3.5
25	222	11.4	7.9	0.41	3.7
26	120	11.2	7.7	0.34	3.0
29	150	10.6	10.1	0.25	3.0
30	225	11.3	8.7	0.27	3.4
Oct 01	126	10.7	8.6	0.30	2.6
02	190	--	12.	0.23	2.9
03	228	11.3	7.9	0.29	5.1
07	153	--	6.	0.41	4.4
am 08	114	10.5	8.7	0.33	3.3
pm 08	72	--	9.1	0.28	2.7
14	205	10.4	14.5	0.15	1.7
15	115	10.5	6.9	0.46	3.9
16	51	10.8	11.	0.51	--
Avg			11.3	0.30	3.2

(a) Average of all caustic applicators in operation (normally 3).

(b) To bring pulp to pH 4.2.

commercial operation (Table 3). In 1976 the quantities were too small so extraction was on a batch basis and therefore the data were inappropriate for evaluation of yields. In 1975 with the Extractor (FMC Model 50; FMC Corporation, San Jose, California) positioned as shown in Figures 2 and 3, the best pulp recovery and skin extraction was obtained with a 0.033-in. screen and 0.5-in. paddle-screen clearance. Larger screen sizes (0.060 and 0.125-in.) and small clearance (0.187-in.) allowed too many skin particles and seed fragments from broken tomatoes to pass through the screen and remain with the pulp. Within the 1975 conditions, recoveries greater than about 98% had visually obvious skin and seeds going through the screen instead of being separated from the pulp. This 0.033-inch screen is typical of many cannery operations. Normal recovery with these settings averaged 96.8% on a wet basis (82.7% dry-solids basis).

The 0.5-in. clearance was necessary because closer clearances, such as the common 0.05-in. cannery practice, ground the skin and incorporated it into the pulp. Caustic action had already loosened the tomato cells so a wider clearance was necessary. Skin and seed fragments are undesirable because they are graded as physical defects in products; also, the waxy skin layer carries the insecticide since the insecticides are fat soluble. Thus, rejecting the skin is important both as a grade physical defect and as an insecticide residue control for products. The 1975 regulatory tolerance for toxaphene was 7.0 ppm. Typically, the recovered pulp contained 0.4 ppm toxaphene and the skin waste had 5-60 ppm.

To elaborate on the paddle-screen clearances, 0.5-in. and 0.19-in. were used. Pulp recovery was about 96% on a wet basis with the 0.5-in. and 99% with the 0.19-in. The 99% recovery occurred because the skin was comminuted and passed through the screen with the pulp. At no time during the trials was there any tendency for the screen to become clogged at either clearance. The extractor waste-outlet gate was open at all times to minimize the back pressure on the waste and allow it to pass through without restriction. The 0.5-in. clearance has the added advantage of greater machine life due to much less machine vibration and less pressure on the screen.

A 750 rpm extractor speed was used throughout 1975. This is a standard speed for the machine, normally giving good equipment life, and with a 96.8% average pulp recovery, there was no need to experiment with various speeds which would have detracted from more important experimentation.

Vacuum Evaporation--In 1975 four 1,000 gal batches of pulp were concentrated; three were recovered peel pulp and the fourth was conventional cannery juice. The first trial on recovered pulp used a 200°F heating temperature in the evaporator coils and a 15-in Hg vacuum; this was too high a temperature and darkened the pulp excessively. The other three runs used an 180°F temperature and 20-inch Hg vacuum and gave improved color. Even lower temperatures should be considered because the acidified, recovered peel is more apt to darken on exposure to heat than is the conventional juice. During 1976 recovered pulp was not vacuum concentrated because it was felt that there was adequate information from the prior year.



TABLE 3. SUMMARY OF 1975 PROCESSING CONDITIONS AND SOLIDS RECOVERY

Date (1975)	Peeler		Peel Flow Rate (gpm)	Acid. Point (b)	Pulp Extractor		Temperature, (F)		Solids Recovery	
	NaOH (% w/w)	PA (ppm) (a)			Screen Size (inch)	Clear (inch) (c)	Inc. Peel (d)	Hot Break	Wet Basis (%)	Dry Basis (%)
9/3am	--	--	20.0	HB(e)	0.125	0.5	--	--	96.7	85.9
9/5am	--	--	30.0	HB	0.125	0.5	--	--	--	--
9/8am	--	--	5.45	HB	0.125	0.5	--	--	98.7	91.6
pm	--	--	8.33	HB	0.125	0.5	--	--	98.1	90.4
9/9am	--	--	16.67	HB	0.125	0.5	--	--	97.3	88.6
9/11am	--	1780	16.7	HB	0.060	0.5	--	200	97.4	85.2
am	--	1780	--	HB	0.060	0.5	--	198	97.1	83.9
pm	--	1780	14.3	HB	0.060	0.5	123	200	97.5	87.3
9/16pm	9.98	1780	16.7	HB	0.060	0.187	122	198	98.9	94.6
9/17	--	--	17.5	HB	0.060	0.187	120	195	95.7	82.2
9/19am	12.85	993	9.6	PT(f)	0.060	0.187	120	200	99.4	93.9
pm	12.85	--	10.0	PT	0.060	0.187	129	200	99.1	91.7
9/22pm	10.6	--	8.0	PT	0.060	0.5	127	201	97.4	87.0
9/23am	10.6	--	8.0	PT	0.033	0.5	127	200	97.1	81.8
pm	10.6	1137	7.9	HB	0.033	0.5	131	199	96.5	69.1
9/24am	9.97	977	7.7	HB	0.033	0.5	136	201	97.1	85.0
9/25am	11.40	1535	7.9	PT	0.033	0.5	121	199	98.7	91.9
9/26pm	11.17	1280	7.7	PT	0.033	0.187	120	200	98.9	88.3
9/29am	10.63	1206	10.1	HB	0.033	0.187	117	195	98.8	90.1
9/30am	11.27	1510	8.7	PT	0.033	0.5	130	200	--	--

(continued)

TABLE 3. (continued)

Date (1975)	Peeler		Peel Flow Rate (gpm)	Acid. Point (b)	Pulp Extractor		Temperature, (F)		Solids Recovery	
	NaOH (% w/w)	PA (ppm) (a)			Screen Size (inch)	Clear (inch) (c)	Inc. Peel (d)	Hot Break	Wet Basis (%)	Dry Basis (%)
10/1am	10.67	1133	8.6	PT(f)	0.033	0.5	132	200	96.4	82.5
10/2am	--	1492	11.3	PT	0.033	0.5	--	194	96.4	82.3
10/3am	11.33	1640	7.9	PT	0.033	0.5	140	201	97.1	84.6
10/7am	--	960	6.0	PT	0.033	0.5	141	127	96.7	82.4
10/8am	10.48	1262	8.7	PT	0.033	0.5	130	200	96.9	85.0
pm	10.48	1283	9.1	HB(e)	0.033	0.5	126	198	96.8	--
10/14am	10.44	1465	12.3	PT	0.033	0.5	112	193	95.7	82.1
am	10.44	1465	16.6	PT	0.033	0.5	113	199	96.0	83.3
10/15am	10.51	1058	6.9	PT	0.033	0.5	138	131	96.6	82.6
10/16am	10.80	1275	11.0	PT	0.033	0.5	137	137	--	--
Average, of all trials									97.4	85.9
Average, for trials with 0.033-inch screen and 0.5-inch clearance									96.8	82.7

(a) PA = Peeling aid, sodium 2-ethylhexyl sulfate

(b) Acidification point

(c) Paddle-screen clearance

(d) Incoming

(e) Hot break

(f) PT = Peel tank

Prior to the 1975 cannery experiments, laboratory experimentation was undertaken to determine if the color darkening, due to high alkaline conditions or heat, occurred in the tomato pigment, the non-serum solids, or in the serum. In both cases, darkening was in the serum. Separations were made of these three fractions by centrifuging and solvent extractions, and the fractions were cycled from pH 4.2 to 11, down to pH 2, and returned to pH 4.2. They were observed visually for significant changes. The pigment fractions (lycopene and carotenoids) and solids fractions had only slight color changes. The serum fractions had substantial darkening at pH levels above 8. This darkening was semi-reversible (perhaps 95%), but with each alkaline cycle (more severe and not commercially typical) the serum fractions became progressively darker. Results were similar whether whole pulp or fractions were cycled.

#### Unit Operation Times

An early choice was faced on experimental equipment location. Either it could be placed inside the cannery adjacent to the peel removal or outside. Each had an advantage. If inside, the time between caustic application and acidification could be reduced perhaps by three minutes, but there was not sufficient space available to locate all the equipment together, and the experimental flexibility would have been drastically reduced and control impaired. With the equipment outside, full flexibility in utilizing the experimental equipment was retained. Subsequent experimentation in 1975-76 indicated that even acidifying the peel directly out of the caustic applicator did not appreciably change the product over that obtained from the 1976 modified caustic peeling line.

Time increments for each unit operation are shown in Table 4 for the peel recovery performed in 1975 at the cannery. A similar time sequence was followed in 1976. Ideally, the time exposure to the caustic should be zero to minimize potential flavor and color changes. On a practical basis, immersion in the caustic is necessary for peeling, and it is in this immersion that the principal changes and conditioning occur.

When the peel was received at 110°F, the color was virtually the same as at the time of peel removal; peel received at 140°F was appreciably darker, such as when the caustic applicator solution overflowed into the peel. The 30°F temperature increase, rather than the higher caustic content, was probably the predominant cause of the darkening. As discussed under vacuum evaporation, acidified recovered peel pulp is more sensitive to thermal darkening than conventional juice. This displays the importance of controlling the process throughout, not just taking any peel and "throwing" in acid.

The time between the peel removal and acidification (about 4.4 minutes) could be shortened, but there was not sufficient indication that a shorter time would improve quality appreciably. In 1976 tomatoes were taken directly from the caustic applicator and quickly plunged in a mild hydrochloric acid solution to "immediately" acidify the tomato peel; then the peel was removed by hand and the pulp judged for pectin content, consistency, enzyme activity, and color. There was no major difference between the pulp from this "instantaneous" acidification and pulp that was held at pH 11 for 5-10 minutes at

100-110°F. If there is a choice, the quicker acidification is preferred as it reduces potential adverse changes in color and flavor.

Product Color, Flavor and Vitamins--Product color and flavor differed between 1975 and 1976, illustrating how the peeling operation and recovery techniques affect the recovered pulp. As stated before, when the caustic applicator overflowed, the recovered pulp darkened, had poor flavor, and 2 to 3 times the normal salt content. But even under this very adverse, atypical condition, the color and flavor could be a food grade, albeit at times a standard Grade C rather than Grade A.

TABLE 4. UNIT OPERATIONS TIMES - 1975

Operation	Equipment (a)	Time (minutes)		Nominal Temperature (°F)
		Increment	Total (b)	
Lye application	C	0.5	0.5	210
Peel Removal	C	0.5	1.0	200
Peel Conveyor	C	0.6	1.6	180
Peel Pump and Pipe	C	3.8	5.4	180-110 (c)
Peel Tank (d)	E	5.0	10.4	120
Extractor	E	0.5	10.9	110
Hot Break	E	25.0	35.9	200
Pulp Tank	E	0.8	36.7	200
Canning	E	10.0	46.7	185
Heat Processing	E	35.0	81.7	212
Cooling	E	30.0	111.7	90

(a) C = cannery system, E = Experimental system

(b) After entering caustic applicator

(c) Peel cooled as pumped through 200 ft pipe.

(d) Acidification to pH 4.2 about 0.1 minute after entry into peel tank, or about 5.5 minutes after tomato entered caustic applicator.

As shown in Table 1, the Vitamin A was about the same in the canned peel pulp as in the processed conventional pulp. Beta-carotene is quite stable, and since the outer surface (peel) of the tomato is richer than the whole tomato in this provitamin, the recovered pulp had a higher carotene value than conventional tomato pulp.

Ascorbic acid is so easily decreased in the presence of hot caustic that it was not present in pulps from either year (Table 1). Samples of recovered peel pulp were originally analyzed for ascorbic acid by visual titration, but, in view of the fairly high apparent levels of ascorbic found, and the possible presence of reductones, it was desirable to study representative canned samples to determine what portion of the apparent ascorbic acid titer was due to interfering substances. A direct photometric reading (at 540 m $\mu$ ) of the ascorbic acid osazone in the presence of the osazones of interfering compounds gave a value of 7 mg of ascorbic acid per 100 g of product instead of the 11 mg found by the visual titration of the reduced ascorbic acid. Since it became apparent that there was a large quantity of reductones present in the sample, it was necessary to chromatographically separate the ascorbic acid osazone from other interfering osazones in order to properly measure the total ascorbic acid content. The colorimetric determination of the isolated ascorbic osazone at 520 m $\mu$  showed that the samples actually contained less than 0.6 mg ascorbic acid per 100 g. It was concluded that for 1975 and 1976 processing, all significant ascorbic acid activity in the peel pulp was lost by exposure to high pH, high temperatures, and air (oxygen) contacts during the peeling process and subsequent processing.

Canned samples of 1975 and 1976 materials were stored to check the color stability. The 1975 samples were still a bright red after six months at 100°F in tinned lined cans (standard can for tomato products). As a further check and to eliminate the possible "bleaching" or inhibiting action of the tin, the 1976 samples were stored in double enameled cans. After nine months at 100°F, these samples were still "bright"; there was a "slight" darkening of the serum as happens to tomato products on storage, but this was less than that of a Grade A commercial puree (21). The color and flavor scores for the 1976 products are shown in Table 5. Color scores are based on the Hunter Color-Difference Meter and flavors were determined organoleptically; both were evaluated by the USDA, Food Safety and Quality Service, Fruit and Vegetable Quality Division, Processed Products Branch, Stockton, CA.

Consistency, Viscosity, and Pectin--In the recovered peel pulp there was a desirable, above average number of tomato whole cells which gave the pulp a heavier, thicker appearance compared to conventional tomato pulps. In general though, when the consistency or viscosity was measured, this pulp showed the consistency and viscosity of a good cold-break processed pulp (Table 6). Serum viscosity approached that of water which indicates degradation of the pectin as would be expected from the caustic exposure. Insoluble solids are generally the primary contributors to tomato-product consistency, but pectin is important because it serves to hold the insoluble solids in suspension and reduce the tendency for serum separation. The consistency of this recovered peel-pulp would be appropriate for pizza sauce or soups which normally may use a cold-break pulp because it is desirable to thicken with starch. For other sauces or pulps where a hot-break

material is normally used, combining recovered pulp with conventional pulp in a ratio of 1:3 results in a material with the consistency and character of a hot-break pulp.

Samples of peel pulps were evaluated for pectic enzyme activity in both 1975 and 1976, both in the laboratory and from the experimental equipment. It was concluded that the 1975 recovered peel pulp had little or no pectic enzyme activity as received when sampled before the heating; therefore, a hot break is considered unnecessary because there are no enzymes to inactivate. If, however, the process is modified so that there is substantially less exposure to caustic, then the hot break could be necessary. Adequate substrate was available in the test since the addition of 0.03% commercial enzyme (Rohm and Haas No. 59-L, Rohm and Haas Inc., Philadelphia, Penna.) to the test mixture resulted in a rapid loss of consistency. Without substrate addition, there was no change in pulp consistency over a period of 1 to 3

TABLE 5. STORAGE STABILITY OF RECOVERED PULP - 1976

Sample (a)	Color Score (b)				Flavor (b)				Total Solids salt free (%)
	Storage Temp. & Time				Storage Temp. & Time				
	34°F		100°F		34°F		100°F		
	9 mos.	1 mo.	3 mos.	9 mos.	9 mos.	1 mo.	3 mos.	9 mos.	
76007	51.0	51.0	50.9	50.4	FG	FG	FG	FG	6.6
76009	51.5	51.3	50.9	50.6	FG	G	FG	FG	6.8
76011	51.9	51.1	50.8	50.8	G	G	FG	FG	6.2
76013	51.6	51.6	51.2	51.2	G	G	FG	FG	7.1
76021	50.9	50.8	50.5	50.1	FG	FG	FG	FG	7.2
76023	50.8	50.9	50.7	49.5	G	FG	G	FG	6.1
76025	50.2	50.1	49.9	49.3	G	G	FG	G	5.6
76027	49.7	49.3	48.9	48.2	G	G	G	G	6.1
76031	49.3	48.5	48.0	47.0	G	G	G	G	6.4
Comm'l(c)	46.9	46.7	46.0	43.1	FG	FG	FG	FG	14.4

(a) Sample number as embossed on lids of canned samples; 76 = year an OXX is the sample and trial number.

(b) Color Grade A, is 45-50, Grade C is 40-44 for canned tomato pulp. Color determined using the Hunter Lab Color-Difference Meter D25D-2 with type A optical head and formula:  $\text{Score} = -122 + 4.139L - 0.081883L^2 + 130.572(a/\sqrt{a^2 + b^2})$ . For flavor, FG = fairly good. G = good.

(c) Pulp samples judged at total solids shown, except commercial sample was diluted to 8.5% NTSS.

TABLE 6. CANNED SAMPLES OF RECOVERED PULPS, 1976

Sample (1976) (a)	pH	NaCl (% w/w) (b)	NTSS (% w/w) (c)	Total Solids (% w/w) (d)	Flowtube Viscosity (sec) (e)	Quality Grade (f)
76003	3.7	1.07	3.8	5.4	50	A
76004	4.2	1.24	4.7	6.7	146	A
76005	4.0	.97	4.6	6.2	158	A
76006	4.1	2.22	4.5	7.3	206	A
76007	4.15	1.72	5.6	9.1	286	C
76009	4.25	.93	5.6	7.7	252	
76011	4.3	.94	5.0	6.8	213	
76013	4.45	.92	5.8	7.6	232	
76015	4.15	.90	5.2	7.2	242	A
76017	4.05	1.31	5.0	7.5	406	A
76019	4.1	1.38	6.3	8.6	534	C
76021	4.1	1.13	5.7	7.8	280	
76023	4.05	1.42	4.5	6.6	218	A
76025	4.1	.94	4.5	6.5	140	A
76026	4.3	1.04	4.2	5.8	173	C
76026 OM	4.2	1.12	4.0	5.9	261	
76027	4.05	1.04	5.9	7.1	181	A
76029	4.1	1.46	5.7	7.7	122	A
76031	4.25	1.16	6.0	7.7	180	A
76033	4.0	0.98	4.6	6.3	178	C
76035	4.15	0.99	4.8	6.4	229	
75037	4.1	1.0	4.3	6.0	196	C
76039	4.1	0.92	4.6	6.2	217	C
76041	4.1	1.08	4.6	6.6	304	A
76043	4.15	0.84	4.6	6.4	302	
76045	4.1	0.92	4.9	6.6	312	A
Avg.	4.13	1.14	5.0	6.9	231	62%A
CCTJ (g)	4.2	0.50	6.4	6.7	70 (i)	A to C
CCTP (h)	4.3	0.17	12.2	12.75	-- (j)	A to C

(continued)

TABLE 6. (continued)

- (a) Sample number as embossed on lids; missing even-numbered cans contained peeled, whole tomatoes.
- (b) NaCl, sodium chloride (salt).
- (c) NTSS, natural tomato soluble solids; does not include NaCl from acidification.
- (d) Total Solids, includes NaCl from acidification.
- (e) Flowtube Viscosity, seconds per 5 ml sample.
- (f) Quality Grade (except CCTJ), samples graded as tomato puree for color, defects, and flavor using puree standards (Reference 21), without solids content qualification, by USDA Fruit and Vegetable Quality Division, Food Safety and Quality Service, Stockton, California. Ten samples graded C because of speck defects; samples had good flavor except nine which were fairly good, and one which had a tin flavor. Trial conditions varied as process variables were being investigated, primarily as related to pretreatment and peeling aids.
- (g) CCTJ, commercial canned tomato juice; typical averages, salted product, single strength.
- (h) CCTP, commercial canned tomato puree; typical averages, unsalted product, concentrated.
- (i) Juice range was 28-148 seconds.
- (j) Puree is much more viscous than juice, measured by a Bostwick consistometer, and Bostwick readings are not convertible to Flowtube Viscosity.

hours. The literature (29) indicates the sensitivity of pectin to caustic conditions; for example, a solution at room temperature and containing 0.03 g of pectin per 100 ml was completely hydrolyzed in 30 minutes with 0.4% sodium hydroxide, or in about 10 minutes with 0.08% sodium hydroxide. By contrast, the tomato surface is exposed in cannery operations to 10 to 18% caustic at 200 to 210°F for 30 seconds, then further exposed to about 1.5% caustic for 5 minutes at temperatures declining to 110°F. Caustic peeling depends not only on applying the caustic to the outer surface of the tomato but also on the caustic permeating the skin; therefore, if pectin is to be retained in the recovered pulp, consideration must be given to what occurs in the caustic bath.

Therefore, trials were made on acidifying the tomato surface before peel removal in lieu of acidifying the peel after removal. Tomatoes were dipped and cooled in dilute hydrochloric acid immediately after removal from the caustic bath. The peel was removed manually, the pulp taken from the skin, and serum viscosity and peroxidase activity were checked. Also, raw tomato pulp was raised to pH 11, heated, re-acidified, and checked. Results were similar in both cases though some "virgin" pulp was inevitably included when hand peeling and the pectin and peroxidase therefore appeared (incorrectly) to have been only partially affected by the caustic. In general, the serum viscosity approached 1 cp and the peroxidase was inactivated when the tomato or pulp was exposed to pH 11.4 and 180°F for 40 sec; at 80°F and 40 sec exposure serum viscosity was still nil and there was no peroxidase activity.



From these 1975 and 1976 results there is no apparent need to further consider enzyme inactivation, whether pectinase or peroxidase. However, since the acidified pulp has a temperature of 100 to 130°F, consideration should be given in a commercial installation to heating acidified peel with a hot macerate going to a pulper-finisher; this avoids putting in a heating operation solely for recovering peel pulp. Such a decision will be affected by both production and regulatory considerations.

Amino Acids--Since there is only about 1% protein in tomatoes, this would not normally be considered a significant dietary source of protein. This is reflected in the general use of tomatoes in sauces and catsup, the main exception being tomato juice which is considered a source of ascorbic acid (Vitamin C). However, amino acids are worthy of analysis because they can indicate the amount of exposure to alkali and heat. Such a chemical index of processing effects could be used for future process modifications to obtain optimum product quality. Certain amino acids are affected more than others by high pH and heat, and, therefore, their relative changes can be used as indices of process induced changes.

Amino acid levels in the acid hydrolysates of 1975 and 1976 samples of recovered peel pulps were determined and compared with those of fresh and conventionally processed tomato pulp. Exposure of tomato pulp to caustic during the peeling process resulted in the degradation of certain amino acids, the degree of which depended on the exposure (time, pH, and temperature). Arginine, which is known to be unstable to caustic, was significantly lower in the recovered pulp samples than in either the fresh or the conventionally processed samples. The ratio of arginine to a more stable amino acid, such as isoleucine or histidine, was chosen as an index of the product condition. For example, the mean arginine to histidine (A/H) ratio calculated for conventionally processed tomato (A/H = 1.30) was closer to that of raw tomato (A/H = 1.30) than was that of recovered pulp (A/H = 0.35 in 1975 and 1.06 in 1976). Thus, the data indicate that the unstable amino acids in the recovered pulp samples were altered to a greater degree than those in conventionally processed tomatoes. In conventionally caustic peeled tomatoes, such changes occur at the tomato surface, which is rinsed, and do not alter the general amino acid content. Nevertheless, the 1976 recovered peel pulps were altered much less than in 1975. Therefore, the A/H ratio was found to be a sensitive and objective indicator of pulp processing history that was also in general agreement with color and flavor grades.

On the basis of the Kjeldahl assays, it appears that about 24 to 27% of the total nitrogen (protein) content of the tomato samples exists in the insoluble fraction of both the conventional and the experimental products. This similarity suggests that there was little or no change in protein solubility resulting from exposure to heat and high pH.

Product Mold, Insect Fragments and Bacterial Counts--In 1975 the peel taken from the commercial peeling operation was almost devoid of mesophilic and thermophilic bacteria, and the levels were insignificant compared to those in normal cannery operations at a comparable point in processing. Bacterial counts were made to check the cleanliness of the equipment, to see if there was a significant difference before and after the heating

(hot break), and to judge the microbiological stability of the peel coming from the commercial peeling. As it turned out, the caustic peel is inherently microbiologically stable and normal cleanup and sanitation procedures would be adequate. Therefore, it can be assumed that the caustic-peeling operation at high pH and 200-210°F does have a strong bactericidal effect and that the caustic remaining on the peel has a bacteristatic action.

Also in 1975, the mold and insect-fragment counts on the canned, recovered pulp were uniformly low and reflect the thorough washing and sorting system at the cannery which was described earlier (see Washing and Sorting). These microscopic counts were well under the maximum mold count tolerance for tomato juice (20% of the Howard slide fields) and therefore similar to that normally encountered in commercial products. In so far as this cannery was typical of the industry, the existing raw product cleaning procedures would seem quite adequate if peel pulp is recovered. In 1976 there was experimental freedom to vary sorting. Truck loads with a California State Grading Certificate showing 1% or less mold required minimal or no sorting to stay within mold tolerance; if graded as 3% mold, up to 20% of the tomato weight might need to be removed so as to stay within tolerance. This 20% included both tomatoes showing mold and broken tomatoes which would subsequently disintegrate in the caustic bath or not yield properly peeled whole tomatoes. In general, when the California State load grade certificate shows 3% or more, particular attention must be given to sorting. The elapsed time between picking, State grading, and processing, as well as the seasonal period, will modify the amount of sorting needed.

Bacterial plate counts were made on samples from three consecutive days in 1975 (October 14, 15, 16) with varying types of "cleanup". Up to October 14, the equipment was flushed with water at the end of the operating day and the tomato pulp residues in open vessels were removed by hand scrubbing. For October 15, the equipment was washed as usual, but a 10 ppm chlorine solution was put into the equipment overnight; the upper surface of horizontal pipes was assumed to be free of tomato material. Preparatory to October 16, the piping and equipment was disassembled, hand scrubbed, and rinsed with 100 ppm chlorine solution, drained, and rinsed with potable water. Pulp samples were taken before and after the hot break, at the pulp tank, and from the 200-gal holding tanks used for the material balance. On October 14, the total plate count varied from 6,000 to 17,000 per ml of tomato peel pulp depending on sample location; this is a much lower count than might be expected in conventional cannery operations after the hot break stage, and is attributed to the caustic peeling operation. In the October 14 samples, vegetative cells were found but no spores. The implication is that the hot caustic peeling reduced the count, but that there was some re-inoculation, possibly from unclean experimental equipment or from the atmosphere since the vessels were open. On October 15 and 16, the counts were zero for both vegetative cells and spores. Since this equipment was not operated on a 24-hour basis, this information should not be extrapolated to a round-the-clock operation.

Pesticides--Various pesticides are applied in the tomato field to protect crops from the time of planting through harvesting. Although the use and application of all pesticides are closely regulated by the

Environmental Protection Agency, pesticide residues are unavoidably on the surface of delivered produce. To assure that the levels of pesticides in the final products are well below toxic concentrations, maximum permissible levels have been established by the FDA. Previous studies have shown that pesticide residues on raw tomatoes are contained almost exclusively in the waxy layer overlaying the skin. Since the peel residual, from which pulp was being recovered during this study, contained an extremely high percentage of skin, samples collected from the test system were analyzed for pesticide residues (Table 7). Crop histories of the tomatoes delivered to the plant indicated that toxaphene (a chlorinated camphene containing 67-69% chlorine; also called camphechlor) was used most widely. Thiodan (a chlorinated hydrocarbon) and parathion (an organophosphate) were used to a lesser extent. All of these are detectable by gas chromatography. Appropriate samples were analyzed for each of the three pesticides. The pesticide counts in 1976 were similar to 1975, but the 1976 skin waste and recovered pulps were obtained from a short time (two minutes) batch separation of pulp. Therefore, the 1975 results are considered to be more indicative of a commercial operation.

The legal tolerance (maximum) for toxaphene residue on foods was 7.0 ppm in 1975 and 1976. Table 7 shows both the average and range of toxaphene in the recovered pulp for 1975 with an average of 0.4 ppm in the recovered (100%) pulp, i.e. about 6% of tolerance, and none of the samples were above tolerance. The 35 ppm average in the skin fraction illustrates the need to separate the skin from the pulp. The effects of the finisher clearance and skin size on separation of the skin from the pulp was discussed under the heading "Pulp Extraction". A close clearance between the finisher paddles and screen will cause the skin to pass through the screen with the pulp, and this high skin content can result in a high pesticide content in the pulp. Regulations prohibit bringing over-tolerance materials into compliance by dilution. Therefore, prudent manufacturing practice dictates the need for an adequate fractionation of skin from the pulp.

U.S. Standards of Identity--Since the proposed process modifications and materials may or may not be totally covered by the existing U.S. Food and Drug Regulations, the FDA Bureau of Foods was consulted on the following concerns: (1) utilization of pulp derived from caustic peelings, (2) acidification of the caustic peel with food-grade hydrochloric acid, (3) use of food-grade caprylic (octanoic) acid as a peeling aid with residue present in the recovered pulp, and (4) the labeling requirements when recovered pulp is utilized. Whether a commercial installation and product would meet the existing, and currently being revised, regulations, or whether further technical and regulatory considerations are needed, will depend on FDA judgment. Pertinent regulations in 1975-76 were the former 21 CFR 53.10-0.40, tomato products; 21 CFR 121.1070, food-grade fatty acids; and 121.1091, chemicals used in lye peeling. A reorganization and republication of regulations on food for human consumption appeared on March 15, 1977 in 42 FR 14302-14306; this cross references the old and new section numbers. The United States Standards for Grades of Canned Tomato Puree and Canned Tomatoes are set forth in 29 FR 9838 (1964) and amended in 35 FR 3651 (1970) and 29 FR 7909 (1964).

TABLE 7. PESTICIDE RESIDUES IN RECOVERED PEEL PULP

Sample, 1975	Toxaphene			Thiodan			Parathion		
	No. of Samples	Average (ppm)	Range (ppm) (a)	No. of Samples	Average (ppm)	Range (ppm) (a)	No. of Samples	Average (ppm)	Range (ppm) (a)
Whole tomatoes (fresh)	3	0.2	0.2 - 0.3	3	0.01	nd-0.02	3	nd	nd-nd
Peeled tomatoes (before canning)	3	nd	nd	3	nd	nd-nd	3	nd	nd-nd
Caustic peelings before pulp extraction	5	0.2	nd - 0.4	5	0.00(2)	nd-0.01	5	0.01	nd-0.06
Peel skin (after pulp extraction)(b)	20	35.	3. - 150.	7	0.04	nd-0.15	7	0.03	nd-0.08
Peel pulp (after pulp extraction)(b)	27	0.4	nd - 4.8	10	0.00(1)	nd-0.01	10	nd	nd-nd

nd = none detected

(a) Detection limit = 0.1 ppm for Toxaphene; 0.01 ppm for Thiodan and Parathion.

(b) Pulp Extractor normally set with 0.5-inch paddle-screen clearance; screen hole size varied from 0.033 to 0.125-inch. Range also includes other screens and clearances.

Currently, the tomato Standards of Identity are being revised and re-written by FDA with consideration given to the present CODEX regulations and present day needs. To the readers of these or past regulations, they should be aware of the fact that it is not only the wording of regulations that are of consequence, but the intent (unwritten) as well.

A proposed affirmation of GRAS status for caprylic acid as an indirect and direct human food ingredient was published on June 14, 1977 (28). This proposal is subject to public comment, revision, and approval. The significance of this GRAS status would place caprylic acid in the status of an ingredient rather than that of an additive, though possibly just as a flavor. Use levels are proposed in the new paragraph. The new paragraph 184.1025d (28) proposes that the ingredient (caprylic acid) be used in foods at levels not to exceed good manufacturing practices (GMP) and that for food categories not specifically mentioned, GMP results in maximum levels, as served, of 0.001% (10 ppm) or less. The expected use of recovered peel pulp in tomato products, as consumed, would be below this 0.001% level. The current regulations (Standards of Identity) do not address utilization of recovered tomato peel pulp directly, although the liquid from peels and cores may be used as a packing media for canned tomatoes and for manufacturing canned puree and catsup (155.190-.194). The Standards of Identity are now being revised and will influence the future utility of recovered peel pulp.

Effluents and Wastes--One of the prime considerations when initiating the project was not only to reduce liquid and solid effluents in terms of caustic, BOD, and COD, but to avoid creating new ones; this was successfully managed. There was no continuous liquid discharge except from washing the tomatoes, and this is present commercial practice. In the 1976 Modified Caustic Peeling system, there was a carryover of peeling aid from the Pretreatment to the Caustic Applicator, and from the Applicator to the disc peel remover. These carryovers were food-grade materials, not inedible peeling aids nor effluents. Since 96% of the peel is recovered as pulp, the normal peel effluent was drastically reduced. It was reduced by fractionating the 96% edible material from the 4% non-edible skin and seeds (from tomatoes disintegrated during processing). The skin, seeds, and fibers separated by the Extractor are normal processing wastes. Since these skins and seeds have been re-acidified, they are more acceptable and in smaller quantity than the current caustic peel for disposal on agricultural land or into a public sanitary landfill site. The pretreatment bath liquid was not operated for extended periods, and though BOD and COD measurements were made, these likely do not represent what might be experienced under commercial conditions.

Some canners currently operate their caustic applicators the full 3-mo. processing season without changing solutions; others change the caustic once a week. The pretreatment solution of octanoic (caprylic) acid and water is biodegradable, whereas some of the current peeling aids are not. After peel removal, all canners rinse or flume the peeled tomatoes and this practice would be unaffected by modified caustic peeling. Therefore, this modified caustic peeling and peel recovery, while not affecting liquid effluents, would decrease current peel solids discharge by 96%, and the discharge would have an improved pH character.

Economics--The economics for a cannery-peeling operation of 40 t/hr are summarized in Table 8. This assumed installation has the general features of a projected average situation containing the components of the Modified Caustic Peeling System. An actual installation, in a specific cannery, will vary according to available space, existing equipment, and production requirements for tomato products. Such a peeling operation might use two caustic applicators, such as the FMC Hi-Ton, and either two FMC PR-20 Peel Removers, four Magnuson Model C Peel Scrubbers, or equivalent dry mechanical peel removers that do not add water to the peel. The costs and values shown are presented as an example and should be adjusted with local data. These costs are based on 1976 information, a 12% recoverable peel, 60 days of operation per year, 16 hours of peeling each day, and a \$50/t pulp value. While the trend of costs is upward and is expected to continue up, the relative spread between costs and value is expected to remain about the same for the coming few years.

Capital and operating expenses include only those directly associated with pretreatment and pulp recovery, not the balance of the peeling process that is presumed to already exist. The capital cost could easily be smaller or greater, depending on whether existing equipment and utilities are readily adaptable or entirely new equipment would be needed. One example is that the pretreatment could be applied with an existing flume that is converted to heated, recirculated liquid, or it might require a special tank with controlled immersion time for the tomatoes, such as that used in the pilot installation during 1976.

There is a significant cost reduction in peel waste disposal because 96% of the peel can be recovered as pulp and only 4% remains as true waste for disposal. A cost of \$2.50/t was used for the current disposal of caustic peel, and this is a minimum as the range is \$2.50 to \$5.00/t. For the installed Capital Equipment, a significant potential difference exists between this estimate and an actual installation. The amount of electrical and piping supply installation is subject to existing facilities; for instance, if a flume is used for the pretreatment with peeling aid, a 50 hp electric motor might be required for a recirculation pump, and such a load could require adding major electrical power capacity in that area. The balance of the fixed and variable costs are fairly predictable, but for each year after 1976, about 10% per year should be added as an inflation factor.

The capital costs associated with peel pulp recovery can potentially be recovered within one year as indicated in Table 8. Therefore, the recovery of peel pulp appears to be self supporting, hence economically viable and attractive. Since other plant operations are not affected by pulp recovery, the economic risk is considered to be limited to the recovery process.

TABLE 8. PROJECTED ECONOMICS OF A COMMERCIAL INSTALLATION

Based on: 40 t/hr tomatoes x 12% peel = 4.8 t/hr peel 96% recovery = 4.608 t/hr pulp recovered. 60 dy/yr x 16 hr/dy = 960 hr/yr		1st Year Operation (\$/hr)	2nd Year and Thereafter (\$/hr)
<u>Recovered Peel-Pulp Value</u>			
Based on \$50/t gross value of pulp.		+ 230.40	+ 230.40
<u>Fixed Cost, installed Capital Equipment</u>			
Depreciated in 1 yr, 960 hr.			
Pretreatment (circulation, temp. control)	- 15,000		
Chemical Supplies (tanks, pumps, piping)	- 16,000		
Extraction (pulper, piping)	- 14,000		
Acidification (pH control, agitator)	- 15,000		
General (utilities, piping, etc.)	- 35,000		
Contingency	- 20,000		
(installed total)	-115,000	- 119.79	0.00
<u>Variable Costs</u>			
Direct Labor (operator, cleanup, QC, supv.)	- 12.48		
Startup labor, 1st year	- 24.96		
Indirect labor (mechanic, clerk)	- 3.13		
		- 40.57	- 15.61
Superintendence	- 0.94		
		- 0.94	- 0.94
Utilities, steam, 7,230 lb/hr	- 10.84		
water, 10 gpm	- 0.07		
electricity, 78 kw	- 1.57		
		- 12.48	- 12.48
Chemicals, fatty acid, 1.33 lb/hr	- 1.33		
hydrochloric acid, 150 lb/hr	- 6.00		
		- 7.33	- 7.33
Maintenance Supplies, 5%/yr of capital	- 5.99		
		- 5.99	- 5.99
Miscellaneous (operating & cleanup supplies)	- 1.00		
		- 1.00	- 1.00
<u>Value</u>		+ \$230.40	+ \$230.40
<u>Costs</u>		- 188.10	- 43.35
NET RETURN ON PEEL PROCESSING		+ \$ 42.30	+ \$187.05
Saving on Caustic Peel Disposal (\$2.50/t)		+ 12.00	+ 12.00
		+ \$ 54.30	+ \$199.05
OVERALL RETURN ON PEEL PROCESSING		or \$52,128.00 1st year	or \$191,088.20 thereafter

## REFERENCES

1. Schultz, W. G., R. P. Graham, and M. R. Hart. Pulp recovery from tomato peel residue. Proceedings of 6th National Symposium on Food Processing Wastes, Madison, Wisconsin, April 5-11, 1975.
2. Hart, M. R., R. P. Graham, G. W. Williams, and P. F. Hanni. Lye peeling of tomatoes using rotating rubber discs. Food Technology 28:38, 1974.
3. Ostertag, R., and K. Robe. Waterless peel removal. Food Processing 36:60, Jan. 1975.
4. Schultz, W. G., R. P. Graham, W. C. Rockwell, J. L. Bomben, J. C. Miers, and J. R. Wagner. Field processing of tomatoes, Part 1, process and design. J. Food Sci. 36:397, 1971.
5. Miers, J. C., J. R. Wagner, Nutting, M-D., W. G. Schultz, R. Becker, H. J. Neumann, W. C. Dietrich, and D. W. Sanshuck. Field processing of tomatoes, Part 2, quality and composition. J. Food Sci. 36:400, 1971.
6. 38 FR 27076. Asbestos Particles in Food and Drugs, Sept. 28, 1973.
7. 40 FR 26683-4. Electrolytic Diaphragm Process for Salt, 1975.
8. Dahl, S. A. Chlor-alkali cell features new ion-exchange membrane. Chem. Engr. 82:17, 1975.
9. 21 CFR 113. Thermally Processed Low-acid Foods Packaged in Hermetically sealed containers, 1977.
10. Horowitz, William (ed.). Official Methods of Analysis of the Association of Official Analytical Chemists. AOAC, Washington, D.C., 11th ed., 1970. p 560.
11. ibid. p 560.
12. National Canners Assn. Laboratory Manual for Food Canners and Processors, Vol. 2. The AVI Publishing Co., Inc., Westport, Conn., 1968. pp 291 - 292.
13. ibid. p 341.
14. ibid. pp 294 - 296.



15. Horowitz, W. loc. cit. p 369.
16. ibid. pp 769 - 771.
17. Assoc. Vitamin Chem. "Methods of Vitamin Assay", 3rd Ed., 1966.  
pp. 320-327,
18. ibid. pp 332 - 338.
19. Food and Drug Administration. Pesticide Analytical Manual, Vol. 1.  
U.S. Dept. of HEW, Washington, D.C., 1971 §212.13a(2).
20. National Canners Assn. loc. cit. pp 300 - 316, 324 - 325.
21. 35 FR 3651. Canned Tomato Puree (Tomato Pulp), 1970.
22. Assoc. Offic. Agr. Chem. "Official Methods of Analysis, 448, 1960.
23. Spackman, D. H., W. H. Stein, and S. Moore. Automatic recording  
apparatus for use in the chromatography of amino acids. Anal. Chem.  
30:1190, 1958.
24. Wagner, J. R., and J. C. Miers. Consistency of tomato products. Food  
Technology 21:920, 1967.
25. Horowitz, W. loc. cit. pp 777-778.
26. 21 CFR 173.315. Chemicals Used in Washing or to Assist in the Lye  
Peeling of Fruits and Vegetables, 1977.
27. 21 CFR 172.860. Fatty Acids, 1977.
28. 42 FR 30390-1. Caprylic Acid, 1977.
29. Joslyn, M. A. "Methods of Food Analysis", Academic Press, 1950.  
pp 455-56.
30. USDHEW, Public Health Service, Food and Drug Adm., HFF-342, Washington,  
D.C. 20204. "The Food Defect Action Levels", 11-12, August 1977.
31. Method of Peeling Fruits and Vegetables with Carboxylic Acids, SN 714,  
229. National Technical Information Service, U.S. Department of  
Commerce, 425 Thirteenth St., NW, Washington, D. C. 20204.
32. Amoores, J. E., D. Venstrom, and A. R. Davis. Measurement of Specific  
Anosmia. Perceptual and Motor Skills. 26: 143-164, 1968.
33. Kohler, G. O., and Palter R. Studies on methods for amino acid analysis  
of wheat products. Cereal Chem. 44:512, 1967.
34. Martin, J. T., and B. E. Juniper. "The Cuticles of Plants". Arnold  
Publ., 1970.

35. Beaman, D. R., F. P. Boer, C. T. Lichy, R. J. Moolenaar, F. W. Spillers, O. C. Taylor, and D. M. Young. Abestos in Water in the Chlor-Alkali Industry. The Chlorine Institute, 17th Chlorine Plant Managers' Seminar, New Orleans, Louisiana, February 5, 1975.

#### BIBLIOGRAPHY

- 29 FR 7909 Grades of Canned Tomatoes, July 24, 1969.
- 21 CFR 155.190(a) -.190(b), -.190(c) -.191, -.192, and -.194. Standards of Identity - Canned Tomatoes, Tomato Paste, Tomato Puree, Catsup, 1977.
- U.S.D.A. "Composition of Foods", Agriculture Handbook No. 8. U.S. Government Printing Office, Washington, D. C., 1963.
- Wilson, L., and Sterling, C. Studies on the Cuticle of Tomato Fruit. Zeitschrift fur Pflanzenphysiologie, Vol. 77, pp. 359-371, 1976.
- 42 FR 10004-10009. Canned Tomatoes, proposed United States Standards for Grades, 1977.
- 42 FR 1000-100013. Canned Stewed Tomatoes, proposed United States Standards for Grades, 1977.

## APPENDIX A

### Vitamin A Activity Method\*

#### Reagents

Acetone: Dry, alcohol-free reagent grade.

Petroleum Ether: redistill in all-glass apparatus collect 60-70° fraction

Adsorbent: mix for 1-2 hours 1+1 activated magnesia (MgO from Merck Co., Rahway, New Jersey) and Hyflo Super-Cel (diatomaceous earth from Fischer Scientific Company, Fair Lawn, New Jersey).

#### Extraction

Work in subdued light.

Weigh 10-50 grams sample into blender cup. Sample size depends on amount of International Units per 100 grams sample: less than 100 to 200 I.U./100 g. needs 50 grams sample, 200 to 1000 I.U./100 g. needs 25 grams sample, and more than 1000 ILU./100 g. needs 5-10 grams sample.

Add 200 ml 50:50 petroleum ether:ethanol. Add approximately 2 grams filter aid. Blend at high speed for 5 minutes. Filter through fast flow Whatman filter paper on Buchner funnel into 500 ml Erlenmeyer flask connected to vacuum. Rinse blender cup with water and then with petroleum ether into funnel.

Transfer filtrate to 500 ml separatory funnel rinsing first with petroleum ether then with water.

Scrape residue from filter paper back into blender cup. Add 100 ml 50:50 petroleum ether:ethanol. Blend at medium speed for three minutes.

Filter contents from second blending through same filter paper into same 500 ml Erlenmeyer flask. If filtrate is colored re-extract in the same manner until clear, each time transferring filtrate quantitatively to separatory funnel.

Drain water layer in separatory funnel to an Erlenmeyer flask. Swirl vigorously with 50 ml petroleum ether. Add back to separatory funnel. Drain and discard water layer. Wash filtrate with 100 ml portions of water until water layer is clear, about five times. Swirl with a rotary motion. Drain off as much water as possible. Filter through 10 grams anhydrous sodium sulfate into appropriate size beaker. Rinse separatory funnel and

---

\*For regular tomato materials @pH 4.0 to 4.5.

sodium sulfate with 50 ml petroleum ether into beaker. Evaporate extract to 50-25 ml under warm air in hot water bath.

### Column

Chromatographic tube should be 175 mm long with an outer diameter of 22 mm and a constriction at the bottom of 10 mm. A reservoir for holding eluant (solvent) at the top is convenient.

Plug base of tube with glass wool. Add loose adsorbent to 15 cm depth. Apply vacuum and tamp with tight fitting rod to 7 cm depth making sure surface is flat. Add 0.5 cc anhydrous sodium sulfate.

### Elution

Pre-wet column with petroleum ether. Adjust flow rate to 2-3 drops per second by air pressure from above. Do not let column go dry. Transfer entire extract to column. Load by rinsing beaker and reservoir with small portions of petroleum ether just as extract has reached about 1 cm above column.

Elute first carotene band with 5% acetone in petroleum ether. Collect in graduated cylinder. Elute second xanthophyll band with 10% acetone in petroleum ether and collect in graduated cylinder.

### Calibration of Spectrophotometer

Standard Stock Solution: 1.0 mM  
dry crystalline 1-(Phenylazo)-  
2-naphthol (C.I. Solvent Yellow;  
Sudan I) to constant weight in 70°F  
vacuum oven. Dissolve 0.1241 g in  
500 ml acetone-isopropanol (1 + 1).  
Store in dark.

Working Solution: 0.04 mM  
Dilute 20 ml stock solution to 500 ml with acetone-  
isopropanol (1 + 1). Store in dark.

Working solution should read 0.460 at 436 mμ and 0.561 at 474 mμ with slit width of 0.03. The instrument deviation factor, f, is the ratio of the theoretical absorbance to the actual. Assume that the working solution of dye has the same A as 2.35 mg carotenes per liter at 436 mμ and 2.38 mg xanthophylls per liter at 474 mμ.

Read first band at 436 mμ and second band at 474 mμ.

### Calculation

#### Carotenes

$$\text{I.U./100 g} = \frac{A_{436} \times f \times \text{final volume} \times 1667 \times 100}{196 \times \text{g on column} \times b}$$

where f = instrument deviation factor, 1667 = international units vitamin A activity per gram, and 196 = the extinction coefficient (A = .460/2.35 mg carotenes per liter), and b = cell length in cm.

#### Xanthophylls

$$\text{I.U./100 g} = \frac{A_{474} \times f \times \text{final volume} \times 1667 \times 100}{236 \times \text{g on column} \times b \times 2}$$

The activity is divided by two since this group of carotenoids have about 50% biological activity.

Reference: AOAC 39,014 - 39,023, Eleventh Edition, 1970.

## APPENDIX B

### Photometric Determination of Anionic Peeling Aids\*

[Sodium 2-ethylhexyl sulfate and sodium mono-  
and di-methyl naphthalene sulfonate]

#### Solutions and Chemicals

1. Chloroform (Spectrophotometric and Technical grade).
2. Methylene Blue Indicator.  
0.03 grams Methylene Blue (N. F. Powder).  
12.00 grams Concentrated Sulfuric Acid (ACS).  
20.00 grams Sodium Sulfate (anhydrous) ACS.  
Dissolve the above ingredients to 1 liter with deionized water (detergent free). Extract in a 1 liter separatory funnel by shaking with Technical grade Chloroform until the Chloroform layer does not show any variation when compared to an aliquot of Technical grade at 600 wavelength.
3. Deionized water (detergent free).
4. Sulfuric Acid 5N.  
Dilute 140 ml of Conc. Sulfuric Acid (ACS) to a liter with deionized water (detergent free).

#### Apparatus

1. Volumetric flasks-100 ml.
2. Volumetric pipettes - 1, 2, 4, 5, 10, 20 ml. sizes.
3. Separatory funnel, 250 ml & 1 liter.
4. 30 ml screw cap test tubes.
5. Photometer capable of measuring at 600 m $\mu$ .

#### Procedure

Pipette 5 ml. of caustic peel sample into a 100 ml volumetric flask and fill with deionized water (detergent free) to the mark, shake and pipette 5 ml of this diluted solution into a 250 ml separatory funnel. Add 5 ml of 5 N sulfuric Acid and 20 ml of Technical grade chloroform, stopper funnel, and shake for one-half minute. Permit time for the chloroform layer to separate and discard. Add another 20 ml of chloroform, stopper funnel, and shake for one-half minute. Permit time for the chloroform layer to

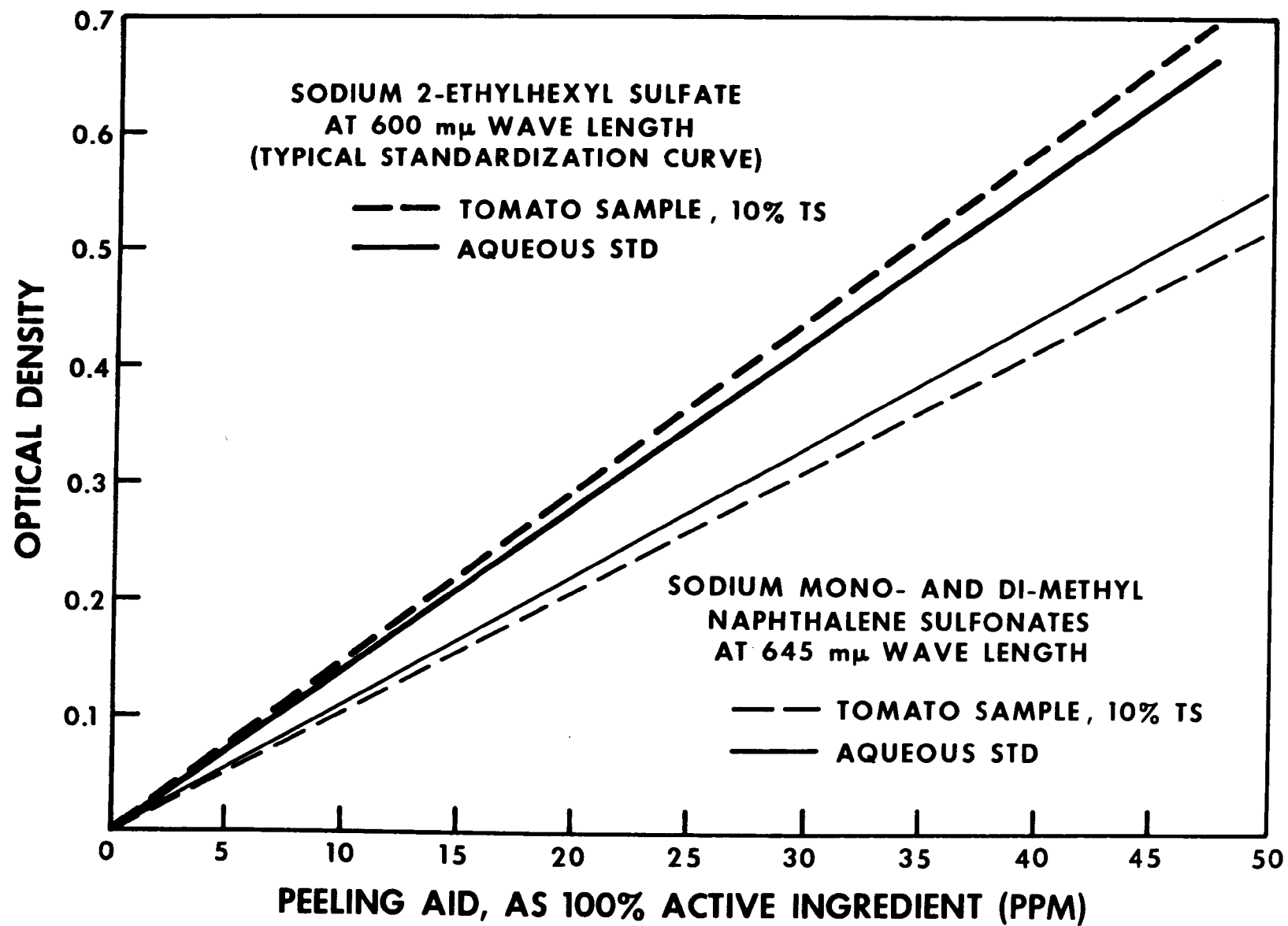
---

\*Modification of a methods published by Intercontinental Chemical Co.,  
P.O. Box 15318, Sacramento, CA 95813 and Union Carbide Inc., 270 Park Ave.,  
New York, NY 10017.

separate and discard. Add 20 ml of Methylene Blue solution and 20 ml of Spectrophotometric grade chloroform to a separatory funnel, shake for one full minute and allow layers to separate as before. Very carefully draw off the chloroform layer into a 30 ml screw cap test tube. Transfer chloroform to Photometric Cuvette and measure the optical density at 600 mμ.

To establish a standardization curve, make solutions containing exactly 0, 5, 10, 20, 30, 40, 50 ppm of peeling aid and analyze according to the outlined procedure, omitting the Technical grade chloroform steps. In a graph, plot ppm of concentration of peeling aid against the optical density as the examples in Figure B-1.





## APPENDIX C

### Determination of Fatty Acids in Processed Tomato Products.

#### Analytical Procedure

##### Principle

This method involves the extraction of fatty acids (and their salts) from processed tomato products and the formation of their propyl esters for their quantitative determination by gas liquid chromatography. This method is applicable to the analysis of  $C_6$  to  $C_{10}$  fatty acids in tomatoes that have been treated with such materials during their processing (peeling).

This procedure defines the chromatographic retention time and response of the propyl esters of hexanoic (caproic) acid, heptanoic (enanthic) acid, octanoic (caprylic) acid, nonanoic (pelargonic) acid, and decanoic (capric) acid. The residue of any one or more of these fatty acids is calculated on the basis of the area of the peak(s) in the sample extract representing the fatty acid in question. This procedure requires prior knowledge of the fatty acid(s) present in the sample, since the area of the propyl ester(s) of the residue is compared to the area of the propyl ester of a similar fatty acid that is added to the sample as an internal standard.

##### Apparatus

1. Extraction equipment:
  - a. 60 ml and 250 ml separatory funnels.
  - b. 25 and 50 ml graduates.
  - c. 50 ml boiling flask, 24/40 taper joint.
  - d. 300 mm West condenser with 24/40 taper joints.
  - e. steam bath.
  - f. 250 ml centrifuge bottles.
  - g. centrifuge.
  - h. 10 ml volumetric flasks or other vessels with glass stoppers.
  - i. 10 ml beakers.
  - j. Pasteur transfer pipets and bulb.
  - k. Pipets: 1 ml, 2 ml, 5 ml.
2. Gas chromatographic instrument with the following minimal characteristics:
  - a. Column oven operated with temperature programmed between 80°C and 170°C, at a rate of 2° to 5° rise per minute.
  - b. Sample injection port with heater characteristic necessary for operating about 85°C higher than the maximum necessary column oven temperature (255°C).

- c. Detector of the flame ionization type. If separately thermostated, it should be maintained at 255-265°C.
  - d. Column, 8-10ft long, 1/8 inch outside diameter, made of stainless steel or glass, packed with 10% by weight FFAP liquid phase on 60-80 mesh acid washed DMCS Chromosorb W.
  - e. Recorder, 1 to 100 mv range, 1-sec. full scale deflection with a chart speed of 1 mm per minute and an attenuator switch to change the recorder range required. The recorder should be equipped with an integrator if possible.
  - f. Helium, nitrogen, or argon; minimum purity 99.95 mol %.
  - g. Hydrogen, minimum purity 99.95 mol %.
  - h. Air, dry, dew point -75°F max.
3. Syringe (1 microliter) for sample injection.

### Reagents

- 1. Reagent Purity. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 2. Chloroform.
- 3. Boron Trifluoride - propanol (14% w/v). Store in refrigerator. (Applied Science Labs, Inc., \*State College, PA 16801).
- 4. 6N - Hydrochloric Acid.
- 5. Saturated solution of ammonium sulfate.
- 6. Anhydrous sodium sulfate.
- 7. Internal Standard. Use heptanoic acid when analyzing for hexanoic or octanoic acid. Use decanoic acid when analyzing for nonanoic acid or a mixture of C<sub>6</sub> to C<sub>9</sub> fatty acids that include heptanoic acid. Standard solution should be prepared in chloroform.
- 8. Standard Mixture of Fatty Acids. Quantitatively prepare a mixture of the fatty acids to be analyzed in approximately equal amounts, including the fatty acid chosen as the internal standard, e.g., about 2 mg of each fatty acid in a total of 2 ml chloroform.

### Procedure

#### 1. Extraction

- a. For samples of tomato pulp with expected fatty acid residuals

of up to about 150 ppm, weight accurately about 40 grams of pulp into a 250 ml separatory funnel. For larger residuals use a proportionately smaller sample size.

- b. Acidify with 1 ml 6N HCl.
- c. Add about 130 ml chloroform and an accurately measured amount of internal standard (ca 3 mg, preferably as an aliquot from a standard solution prepared with chloroform).
- d. Mix thoroughly for about 2 minutes. If necessary, transfer to a 25 ml centrifuge bottle and centrifuge sufficiently to break any emulsion that forms; then transfer to the original separatory funnel.
- e. Remove the chloroform extract (lower phase) to another 250 ml separatory funnel.
- f. Add 20 ml 0.5N  $\text{NaHCO}_3$  and mix thoroughly to extract fatty acids from chloroform.
- g. Allow phases to separate and discard chloroform (lower phase); if necessary, centrifuge.
- h. Acidify with 2 ml 6N HCl. Mix thoroughly until effervescence stops.
- i. If necessary to improve purity of the extract, repeat steps (c) to (h), using 5 ml in chloroform in step (c) and 5 ml 0.5N  $\text{NaHCO}_3$  in step (f) and 0.5 ml 6N HCl in step (h) in a small separatory funnel. Do not add more standard when repeating extraction.
- j. Transfer acidified aqueous phase to 60 ml separatory funnel and extract fatty acids with 2 ml chloroform.
- k. Transfer chloroform extract (lower phase) to a 10 ml beaker containing 0.4 g anhydrous  $\text{Na}_2\text{SO}_4$ .

## 2. Ester formation

- a. Decant the clear chloroform extract into a 50 ml boiling flask.
- b. Add 2 ml of boron trifluoride (14% w/v) propanol reagent.
- c. Reflux on steam bath for 15 minutes.
- d. Cool to room temperature; add 5 ml saturated ammonium sulfate solution and mix throughly. Transfer to 60 ml separatory funnel and allow phases to separate.
- e. Discard lower (aqueous) phase, and with a Pasteur pipet transfer organic phase to a 10 ml glass-stoppered flask containing about 0.4 g anhydrous  $\text{Na}_2\text{SO}_4$ .
- f. Refrigerate until chromatographic analysis.

## 3. Calibration

- a. Prepare the propyl esters of the standard mixture of the fatty acids by using 2 ml of a chloroform solution of the fatty acids (ca. 2 mg each) in step (a) of ester formation. Complete steps b-f of ester formation as for sample extracts.
- b. Chromatograph the ester solution and adjust the chromatographic conditions such that there are suitable retention times and good resolution of the fatty-acid propyl esters.

- c. Techniques pertaining to column conditioning, selection of carrier gas flow rates, peak resolution, sensitivity settings, sample injection, and attenuation control, etc., should be patterned after ASTM Method D 1983-69, AOCS Method Ce 1-62 or any other method suitable for the determination of fatty acid esters, including those suggested by the instrument manufacturer.
- d. Determine the detector response for each fatty-acid propyl ester relative to that of the chosen internal standard. The detector response for esters of fatty acids of similar molecular weight should be very similar. For all practical purposes, the relative detector response for adjacent peaks (e.g., C<sub>7</sub> and C<sub>8</sub> fatty acid propyl esters) may be assumed to be identical. Therefore, it is preferable to use heptanoic acid as an internal standard for assaying octanoic acid.

#### 4. Chromatographic analysis

- a. Once the optimum chromatographic conditions have been established (see calibration), chromatograph the propyl esters of the sample extract, which includes an internal standard of fatty acid (e.g., heptanoic acid).
- b. Identify the fatty acid propyl ester(s) peak(s) and the internal standard peak in the chromatograph of the sample extract.
- c. Integrate only these peaks, correcting for the detector response characteristics of each propyl ester.
- d. If no integrator is used, measure the height and width at half height of each peak in order to estimate its area.
- e. Calculate the residue of each fatty acid as follows:

$$\text{fatty acid residue, ppm} = R_a \frac{S}{W} \times 10^6,$$

where  $R_a$  = ratio of the subject peak area to the internal standard peak area

S = weight of internal standard in grams

W = weight of tomato sample in grams

#### 5. Interference

Analysis of tomatoes not treated with fatty acids may show small amounts of various naturally occurring compounds with retention times similar to those of the C<sub>6</sub> to C<sub>10</sub> fatty acid propyl esters. Individually, these usually amount to less than 1 ppm (fatty-acid equivalent), and very often they can be differentiated from the peaks of the esters being analyzed. If the fatty acid used in processing the tomatoes has an impurity similar to the fatty acid used as the internal standard, the results (compared against the standard) will be slightly low.

<b>TECHNICAL REPORT DATA</b> <i>(Please read Instructions on the reverse before completing)</i>		
1. REPORT NO. EPA-600/2-78-202	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE COMMERCIAL FEASIBILITY OF RECOVERING TOMATO PROCESSING RESIDUALS FOR FOOD USE	5. REPORT DATE September 1978 issuing date	6. PERFORMING ORGANIZATION CODE
	8. PERFORMING ORGANIZATION REPORT NO.	
7. AUTHOR(S) W. G. Schultz, H. J. Neumann, J. E. Schade, J. P. Morgan, P. F. Hanni, A. M. Katsuyama & H. Maagdenberg	10. PROGRAM ELEMENT NO. 1BB610	
9. PERFORMING ORGANIZATION NAME AND ADDRESS USDA, Western Regional Research Center Albany, CA, and National Food Processors Association, Western Research Laboratory, Berkeley, CA	11. CONTRACT/GRANT NO. EPA-IAG-D5-0795	
	13. TYPE OF REPORT AND PERIOD COVERED FINAL REPORT 5/75-8/77	
12. SPONSORING AGENCY NAME AND ADDRESS Industrial Environmental Research Lab. - Cinn, OH Office of Research and Development U.S. Environmental Protection Agency Cincinnati, Ohio 45268	14. SPONSORING AGENCY CODE EPA/600/12	
	15. SUPPLEMENTARY NOTES	
16. ABSTRACT A 2-year project was undertaken to determine the commercial feasibility of recovering pulp from the peelings of caustic peeled tomatoes. In 1975, peel from regular cannery operations was processed through a 20-gpm (5 t/hr) continuous-flow line. This processing consisted of acidifying the peel to pH 4.2 with food-grade hydrochloric acid, then separating the pulp from the skin with a paddle finisher (screen). Recovered peel pulp was found to be of food quality but contained high peeling-aid residues (150-450 ppm). Peeling aids in current use are approved for peeling but not as additives to the final product. In 1976, a 1-t/hr pilot peeling line was set up at a cannery to study modifications in the peeling process for the purpose of reducing peeling-aid residue in the recovered pulp. The principal modification was to pretreat the tomatoes by immersion in a 150°F aqueous bath (approximately pH 3.6) containing about 0.15% food-grade octanoic (caprylic) acid; subsequently, the tomatoes were immersed in caustic. The peel was removed with rubber-disc peelers. Recovered pulp met USDA Quality Grade A, and the octanoic acid levels were low (0 to 30 ppm). The proposed use of this recovered peel pulp is in combination with tomato pulp from regular sources for canned products such as tomato sauce, catsup, paste, and fill juice for peeled tomatoes. Economic analysis indicates an attractive ROI.		
17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Food Processing Canning Byproducts Economic Analysis	Tomato Peel Pulp Recovery	68D
18. DISTRIBUTION STATEMENT  RELEASE TO PUBLIC	19. SECURITY CLASS (This Report) UNCLASSIFIED	21. NO. OF PAGES 78
	20. SECURITY CLASS (This page) UNCLASSIFIED	22. PRICE