manufacturing may be dissociation of actomyosin to myosin A and actin by PP together with Mg^{+2}. On the other hand, TP becomes effective after its decomposition to PP by the TPase in meat. Actually, however, TP could be of use when a long curing period is used, for, unlike PP which becomes ineffective through its hydrolysis by PPase in meat, TP exhibits its influence for longer time period through its hydrolysis by the TPase in meat. The effect of polyphosphates such as HP may be confined to an enhancement of solubility and extractability of myosin B by increasing the ionic strength under the conditions of sausage manufacture, unless TP or PP are produced from HP spontaneous reversion or other type of decomposition. As pointed out by Hashimoto *et al.* (*10*), the important factor for the binding properties of sausage is the quality of the extracted proteins, and not the amount of protein extracted from meat.

These experimental results show that the effects of various inorganic polyphosphates on the physicochemical properties of myosin B will reflect the behavior of the muscle structural protein in meat in the presence of these phosphates.

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FOOD FLAVORS AND ODORS

Volatile Sulfur Compounds in Potatoes

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A LARGE VARIETY of simple sulfur compounds have been isolated from higher plants (*36*), and many such compounds have been found in vegetables (*1, 12, 14, 17, 24*), fruit juices (*22, 30*), meat extract (*4*), beer (*7–9*), wine (*38*), tea (*23*), and coffee (*21, 28*). The importance of sulfur-containing compounds to flavor lies in their extremely low odor thresholds, ranging in the order of 5 to 20 p.p.b. in water solution for compounds such as hydrogen sulfide, methyl mercaptan, and dimethyl mono- and disulfides (*7–9, 29, 34*).

Although gas chromatography has long since proved itself to be indispensable in flavor research, it has had limited application in the analysis of volatile sulfur compounds. The chromatographic behavior of a number of mercaptans and sulfides, including their response using a thermistor detector, has been studied by Baumann and Olund (*3*).

The first part of the work presented here is concerned with gas chromatography of a series of simple organic sulfur compounds, and preliminary studies of the response of the hydrogen flame ionization detector to these compounds. Reports on the behavior of the highly sensitive hydrogen flame ionization detector toward classes of compounds other than hydrocarbons are relatively few and have been reviewed by Ettre (*19*). In the second part, the volatile sulfur compounds from cooking potatoes were investigated by these gas chromatographic techniques. With the exception of hydrogen sulfide, which was identified chemically, identification of potato volatiles is based on two criteria: functional reaction—i.e., precipitation of mercaptans, sulfides, and disulfides with mercuric chloride—and retention time.

Part I. Gas Chromatography of Sulfur Compounds

Experimental Procedure. GAS CHROMATOGRAPHY APPARATUS. The dual-column gas chromatography apparatus, including the detectors, was constructed in the laboratory (*11*). Three different columns were used. Two were of 5-foot × 0.21-inch i.d. stainless steel—one packed with 80 to 100-mesh firebrick coated with 20% diethylene glycol succinate polyester (DEGS) and the other packed with 60 to 80-mesh firebrick coated with 30% Apiezon M. The third column was a 1000-foot × 0.034-inch i.d. nylon capillary coated with silicone SF 96(100). The DEGS column was operated at 38° C. and 28 ml. per minute nitrogen flow; the Apiezon column was operated at 115° C. and 30 ml. per minute nitrogen flow; and the capillary column was operated at 40° C. at 10 and 20 pounds per sq. inch nitrogen pressure. The nitrogen carrier gas was moistened by passing it through a glass-fiber wick which dipped into distilled water. Temperature fluctuations were minimized with "Thermistemp" temperature controllers utilizing thermistor probes. The columns were operated with hydrogen flame ionization

The sulfur-containing volatiles from cooking potatoes have been analyzed by gas chromatographic and chemical techniques. The gas chromatographic techniques are discussed, preliminary studies of the response of columns with hydrogen flame ionization detectors to organic sulfur compounds are described, and the retention times of a series of reference compounds are given. The analysis of potato volatiles for sulfur-containing compounds is presented. Such compounds, produced by cooking potatoes, were concentrated in a sulfur train as mercuric chloride complexes, and, upon regeneration, were analyzed by gas chromatography. Tentative identification was based on relative retention times on one to three columns. Methyl mercaptan and dimethyl disulfide were the major constituents found in the regenerated vapor. Present in smaller amounts were: ethyl mercaptan, dimethyl sulfide, methyl ethyl disulfide, and methyl isopropyl disulfide. Several other compounds were detected in only trace amounts. The mercaptans found were presumed to be formed, at least in part, from disulfides upon regeneration. Hydrogen sulfide, determined by its reaction to form methylene blue, was produced in relatively large amounts (200 to 500 p.p.b. per hour) and over extended periods from cooking either fresh or dehydrated potatoes. Mechanisms whereby simple sulfur compounds may be generated in cooking food materials are briefly discussed.

detectors and a 1-mv. Varian recorder, Model G-14.

Determination of Response of Flame Ionization Detectors to Sulfur Compounds. The response to various sulfur compounds was obtained directly by injecting 1 to 20 μl. of dilute aqueous solutions (10 p.p.m., w./v.). An alternate method, limited to compounds with low boiling points which would evaporate rapidly and completely, consisted of measuring 1 μl. of a single compound with a 1-μl. Hamilton precision syringe into a calibrated, 1-liter Erlenmeyer flask covered with aluminum foil. The air inside the flask was then agitated with a magnetic bar stirrer for 30 seconds to ensure complete evaporation and mixing of the added compound, after which 1 ml. of vapor was removed for injection into the gas chromatographic column.

Source of Reference Sulfur Compounds. Most of the sulfur compounds were obtained from either Eastman Kodak or Aldrich Chemical Companies. *n*-Propyl isopropyl sulfide and di-*n*-propyl sulfide were prepared from *n*-propyl bromide and the sodium salts of isopropyl and *n*-propyl mercaptans in quantities sufficient for gas chromatographic analysis (*25*). In addition, small amounts of methyl ethyl disulfide and methyl isopropyl disulfide were prepared by oxidation of the appropriate mercaptan mixtures with hydrogen peroxide (*25*). *n*-Butyl mercaptan was generated by reduction of di-*n*-butyl disulfide with zinc and HCl (*31*).

Results and Discussion. Response of Hydrogen Flame Ionization Detector to Sulfur Compounds. The response of the hydrogen flame ionization detector is generally considered to be roughly proportional to carbon content, particularly with hydrocarbons (*16*). However, sulfur compounds have been reported to give a response as low as 1/100 of that produced by carbonyls and esters (*26*).

Initial experiments did show that the response to sulfur compounds was somewhat low and even erratic, but with repeated use of the columns, the response improved. This suggested that irreversible adsorption had occurred in the chromatographic train.

Plots of molar response against number of carbon atoms per molecule for available data have shown that members of various classes of organic compounds follow nearly straight-line relationships with slopes that do not vary greatly from a theoretical slope based on the response of *n*-heptane (*19*). A similar plot of organic sulfur compounds, using two sampling techniques (Figure 1) shows, that responses of these compounds are not more than a few-fold less than theoretical responses based on benzene. Aqueous samples of mercaptans produced the lowest response of all compounds tested, however, in a manner strictly proportional to carbon content. The other compounds showed no such proportionality, although on one column, diethyl sulfide and dimethyl disulfide approached a response linear with that of benzene.

Unexpectedly, the injection of vapor samples resulted in considerably greater response and better reproducibility for methyl and ethyl mercaptan and even benzene. The response to the mercaptans increased sufficiently to form, now, a linear relationship among the three compounds. The reason for the low response associated with aqueous samples is not clear, unless the effect is related to the volatilization of these compounds when in solution. Apparently, however, the hydrogen flame ionization detector is capable of responding to sulfur compounds in a manner which does not differ greatly from other groups of compounds, and under certain conditions, near theoretical response may be expected.

Gas Chromatography of Reference Sulfur Compounds. Table I presents the retention times relative to dimethyl disulfide for most of the common mercaptans, sulfides, and disulfides in the boiling range indicated. The values are averages of three to five determinations. The order of elution was strictly according to boiling point for the Apiezon column. A similar relationship for a group of some of these compounds has been reported for the more polar column, didecyl phthalate (*3*). With the silicone-coated capillary column, some exceptions to this order are evident. Resolution of mixtures containing compounds with nearly the same boiling points was far superior on the capillary column. For example, mixtures of ethyl mercaptan with dimethyl sulfide, *n*-propyl mercaptan with methyl ethyl sulfide, and isobutyl mercaptan with diethyl sulfide, which appeared as single peaks on the Apiezon column, were clearly separated on the capillary column.

The DEGS column was of value in the detection of mono- and disulfides only, since mercaptans could not be eluted unless overloading amounts were applied. With the elimination of such a large group of compounds, resolution of mono-

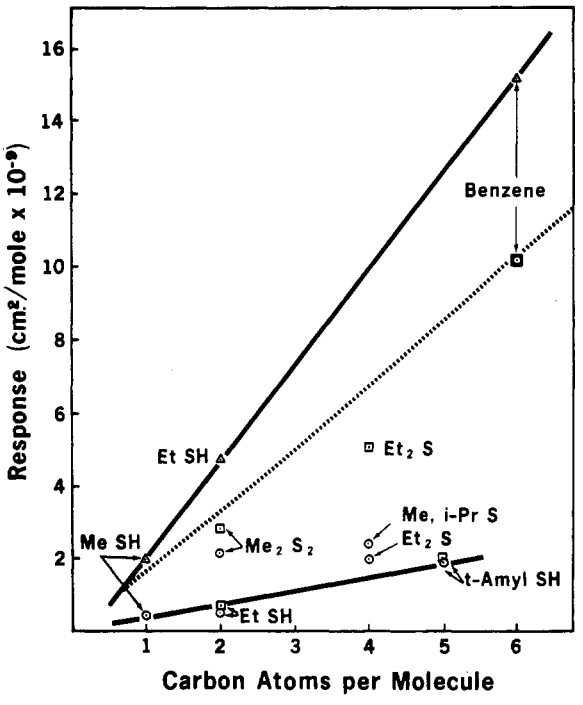

Figure 1. Response of columns with hydrogen flame ionization detectors to sulfur compounds

Aqueous samples — Apiezon column ⊡; nylon capillary column ⊙
Vapor samples — nylon capillary column △

The response of the detector used with the Apiezon column was normalized to match that of the detector used with the nylon capillary column by the factor 1/2.5

Table I. Retention Times, Relative to Dimethyl Disulfide, of Mercaptans, Sulfides, and Disulfides on Various Columns

Compound	Boiling Point, ° C.	Relative Retention Time on: Apiezon	Silicone (capillary)	DEGS
Methyl mercaptan	6	0.185	0.476	
Ethyl mercaptan	36	0.263	0.508	
Dimethyl sulfide	38	0.276	0.519	0.103
Isopropyl mercaptan	56	0.327	0.548	
t-Butyl mercaptan	64[a]	0.385	0.595	
Allyl mercaptan	63–66[b]	0.405	0.589	
Methyl ethyl sulfide	65–67	0.430	0.624	0.170
n-Propyl mercaptan	67	0.448	0.615	
Methyl isopropyl sulfide	85	0.589	0.755	0.209
Isobutyl mercaptan	88	0.657	0.763	
Diethyl sulfide	91	0.680	0.837	0.267
Methyl *n*-propyl sulfide	96	0.739	0.889	0.311
t-Amyl mercaptan	97	0.807	0.898	
n-Butyl mercaptan	97	0.812	0.882	
Ethyl isopropyl sulfide	107	0.917	1.08	0.332
Dimethyl disulfide	110[c]	1.00	1.00	1.00
Diisopropyl sulfide	119	1.17	1.40	0.351
Ethyl *n*-propyl sulfide	119	1.22	1.41	0.490
Isoamyl mercaptan	119–121	1.25	1.26	
n-Amyl mercaptan	126	1.55	1.57	
n-Propyl isopropyl sulfide	132	1.62	1.92	
Methyl ethyl disulfide	135[d]	1.72	1.70	1.57
Diallyl sulfide	138–140	1.74	2.06	
Di-*n*-propyl sulfide	143[c]	2.22	2.67	
Methyl isopropyl disulfide	...	2.44	2.54	
Diethyl disulfide	154[c]	2.92	3.18	1.72
Diisopropyl disulfide	174[c]	3.18	3.36	

[a] (*27*). [b] (*35*), 67–8° C. also reported (*6*). [c] (*25*). [d] (*10*). All other boiling points (*15*).

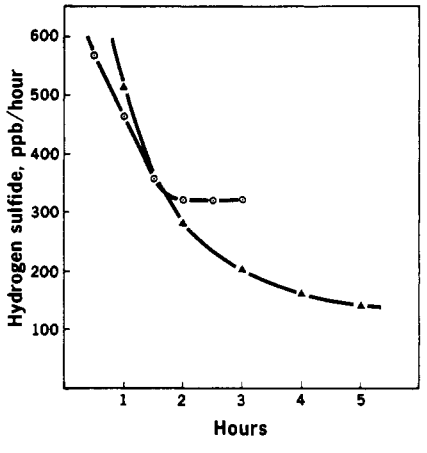

Figure 2. Rate of hydrogen sulfide production from cooking potatoes

▶ Fresh potatoes
⊙ Potato granules, on reconstituted basis

and disulfides was no problem. Examination of the data in Table I for the DEGS column shows that the inclusion of a second sulfur atom in the molecule to form a disulfide greatly increased its retention time and completely subordinated the relationship to boiling point.

Part II. Sulfur-Containing Volatiles from Cooked Potatoes

Experimental Procedure. Collection of Volatile Sulfur Compounds from Cooking Potatoes. Peeled Russet Burbank potatoes ($2^1/_2$ kg.) were blended with water in a Waring Blendor and placed with a total of 4 liters of water in a 12-liter flask. The flask was fitted for reflux, and steam from a steam generator was passed into the potato mixture. Nitrogen, H.P., was added to the steam line at 100 ml. per minute. The effluent nitrogen was led through a volatile sulfur train consisting of an anhydrous calcium chloride drying tube, a solid lead acetate tube, two traps each with 30 ml. of 4% mercuric cyanide, and four traps each with 30 ml. of 3% mercuric chloride (*17*). Upon refluxing, the lead acetate quickly darkened, due to the presence of considerable quantities of hydrogen sulfide. No precipitate formed in the mercuric cyanide traps, suggesting a lack of mercaptans in the volatiles. However, in a subsidiary experiment, a minimum of 7.5 mg. of methyl mercaptan or 20 mg. of ethyl mercaptan was required before any precipitate would form in a mercuric cyanide trap under these conditions. After initial runs, the mercuric cyanide traps were eliminated and the number of mercuric chloride traps increased to seven. The precipitate, which formed slowly in the mercuric chloride traps, was present even in the seventh of the series, and thus, the collection of volatile sulfur compounds was not considered to be quantitative.

To avoid collecting volatile degradation products which might be produced after prolonged steaming, the potato mixture was discarded and replaced with a fresh mixture after 2 hours of refluxing. This process was repeated several times until sufficient precipitate of sulfur volatiles had accumulated in the traps. Under these conditions, the precipitate of mercuric chloride complexes formed at a rate of 3 to 4 p.p.m. in 2 hours. The combined precipitate was dried and stored in a desiccator.

Gas Chromatography of Volatile Sulfur Compounds from Cooking Potatoes. For gas chromatography of the sulfur-containing volatiles, 2 to 3 mg. of the dried precipitate of mercuric chloride complexes were placed in a 25-ml. Erlenmeyer flask and covered with a boiled rubber serum cap. One-half milliliter of 6*N* HCl was injected through the cap, and 2 to 10 ml. of the regenerated volatiles were removed for injection into the chromatography column. It was necessary to use HCl which had been

filtered through activated charcoal to remove interfering impurities. The gas chromatographic apparatus was the same as that described in Part I.

HYDROGEN SULFIDE DETERMINATION IN POTATO VOLATILES. Peeled fresh potatoes, blended in water, were refluxed with steam in an apparatus similar to that described above, except that a smaller flask (3-liter) was used. Appropriate sample sizes were 10 to 50 grams mixed with 200 ml. of distilled water. A stream of nitrogen swept the hydrogen sulfide into a trap containing an absorbing solution of 2% zinc acetate. The hydrogen sulfide in the absorbing solution was determined by a colorimetric procedure based on the reaction forming methylene blue from hydrogen sulfide, *p*-aminodimethyl aniline, and ferric chloride (*7*, *8*). For comparison, dehydrated potato granules, prepared at the authors' laboratory, were substituted for fresh potatoes.

Results and Discussion. VOLATILES FROM COOKING POTATOES. Potato volatiles, regenerated from the mercuric chloride complex, were gas chromatographed on each of the three columns. A prominent peak, consistently found with all three columns, possessed the same retention time as that of dimethyl disulfide, and was chosen as an index for calculating relative retention times.

The results of chromatography of potato volatiles are given in Table II. Ten peaks were resolved on the Apiezon column, and of these six could be compared to reference compounds. Two unidentified peaks were very close to the position of methyl mercaptan and diethyl sulfide and, hence, obscured their detection. As mentioned above, some mercaptan-sulfide mixtures were not resolvable; thus, small amounts of ethyl mercaptan or methyl ethyl sulfide would have been lost in the peak for methyl sulfide and *n*-propyl mercaptan, respectively. The peaks coinciding with *t*-butyl mercaptan, *n*-propyl mercaptan, and methyl *n*-propyl sulfide were very small, and the first was found only on this column. The small amount plus lack of confirmation on the capillary column makes identification of *t*-butyl mercaptan questionable.

The DEGS column, from which mercaptans could not be eluted, resolved six peaks, only three of which were related to reference compounds.

From a total of 14 peaks resolved using the nylon capillary column, 11 could be associated with reference compounds. Since mixtures could be resolved with this column that could not be resolved on the Apiezon column, peaks corresponding to methyl and ethyl mercaptans were found. The peaks corresponding to diethyl sulfide and methyl *n*-propyl sulfide were small and not always observed, even though each was detected on another column.

Table II. Potato Volatiles and Associated Reference Compounds

Reference compound	Relative Retention Times, Apiezon	Silicone (capillary)	DEGS	% by Weight
Methyl mercaptan		0.472		45
Ethyl mercaptan		0.506		5
Dimethyl sulfide	0.272	0.515	0.105	2
Isopropyl mercaptan	0.329	0.543		0.5
t-Butyl mercaptan	0.379			...
n-Propyl mercaptan	0.459	0.613		0.2
Methyl ethyl sulfide		0.625		0.2
Diethyl sulfide		0.839	0.271	<0.05
Methyl *n*-propyl sulfide	0.723	0.885		...
Dimethyl disulfide	1.00	1.00	1.00	45
Methyl ethyl disulfide		1.70		0.7
Methyl isopropyl disulfide		2.55		1

An estimate of the relative amount, by weight, of each compound was made from the total peak area obtained from the regenerated potato volatiles chromatographed on the nylon capillary column and corrected for response based on proportionality to carbon content (*16*).

As shown in Table II, 90% of the mixture consisted of methyl mercaptan and dimethyl disulfide, with ethyl mercaptan and methyl sulfide making up most of the remainder. By comparison, the other compounds are present in quite minor amounts, especially diethyl sulfide and methyl *n*-propyl sulfide, whose relative amounts were not estimated.

Whether free mercaptans, as such, were present in the original vapor from cooking potatoes could not be ascertained under the conditions of the experiment, since the mercaptans which were detected can be accounted for, in whole or in part, by the decomposition known to occur upon formation and subsequent regeneration of mercuric chloride complexes of disulfides (*13*). This phenomenon was confirmed in the laboratory by detecting considerable amounts of methyl mercaptan formed by the treatment of dimethyl disulfide–mercuric chloride complex with acid. Thus, methyl ethyl and methyl isopropyl disulfides, which were present only in small amounts and not always found, and dimethyl disulfide undoubtedly contributed to the corresponding mercaptans that were observed. Accordingly, one might expect other higher boiling disulfides, representing other possible combinations of the mercaptans identified, to be originally present in vapor from cooking potatoes.

The lack of precipitate in the mercuric cyanide traps used in the initial runs while collecting potato volatiles does not mean that mercaptans were completely absent in the original vapor, however, since significant amounts of mercaptans were necessary to produce a visible precipitate. Using cold trap techniques, Self *et al.* reported the presence of both methyl and ethyl mercaptans, including hydrogen sulfide and dimethyl sulfide, in volatiles from cooked potatoes (*37*).

HYDROGEN SULFIDE PRODUCTION FROM COOKING POTATOES. As shown by the darkening of the trap containing solid lead acetate as the potatoes began refluxing, hydrogen sulfide was evolved in relatively large amounts. The rates of hydrogen sulfide production from cooking fresh potatoes and, for comparison, dehydrated potato granules are shown in Figure 2. The curves are representative in that the rate of hydrogen sulfide production decreased initially and then became nearly constant for periods up to several hours with little indication of dropping off. Variation among samples was large, with the average of several runs being 400 p.p.b. produced in the first hour. For dehydrated potato granules, sufficient precursors remain after processing to produce hydrogen sulfide at a rate comparable to that for fresh potatoes.

GENERAL MECHANISMS OF ORIGIN OF VOLATILE SULFUR COMPOUNDS IN FOODS. It may, on first thought, seem anomalous that such an array of highly odoriferous compounds occur in a food as bland as potatoes. However, it is probable that sulfur compounds are important in providing secondary flavoring characteristics for many cooked foods. A phenomenon consistently reported to occur with these compounds at extremely low concentrations is the loss of their characteristic odor. Common subjective descriptions are: "papery" or "yeasty" when they are present in beer, and "cowy" or "malty" when they are present in milk (*7*, *9*, *33*, *34*). Although the pathways and intermediates of sulfur metabolism in plants and animals represent an area largely unexplored, there is sufficient information available

to account for the appearance of these compounds in food products. Both primary and secondary mechanisms may be regarded as responsible for the production of volatile sulfur compounds during cooking. For example, the breakdown of the sulfur amino acids is thought to be the primary source for simple organic sulfur compounds (*36*, *39*). However, volatile sulfur compounds have also been shown to be produced directly from the protein in milk upon heating (*18*). To a lesser extent, the breakdown of other compounds common to all natural food materials, such as thiamine, biotin, coenzyme A, and glutathione, undoubtedly contribute to the volatile sulfur components. Sulfonium compounds, such as *S*-methyl methionine (*36*), decompose to yield dimethyl sulfide, whereas degradation of methionine, itself, yields chiefly methyl mercaptan (*2*). In view of the reactivity of sulfur compounds, many secondary reactions can occur to further increase the variety of volatile sulfur compounds produced during cooking. For example, the strongly nucleophilic S^{-2} can attack a group, such as methoxy, to yield the even more nucleophilic methyl-S^{-1}. Another such attack by this latter species produces dimethyl sulfide (*20*). Sulfide may also attack alcohols to give rise to mercaptans (*38*). Disproportionation among disulfides readily occurs due to their ability to form the free radical RS· (*5*). This radical may also be formed from mercaptans and polysulfides (*5*), and its addition to dienes to form branched sulfides has been observed (*32*). Polysulfides are quite unstable and readily break down to disulfides when distilled (*36*). By these and other such mechanisms, the mixture of sulfur compounds contributing to the volatiles of cooking food can be expected to be large and complex, indeed.

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