linear for dextran, glucose, and fructose and quadratic for sucrose.

CONCLUSION

Of the three types of resin tested, the application of Aminex A5 proved to be reliable for the separation of sucrose, glucose, and fructose since reproducible results could be obtained and good linearity response could be achieved as measured by peak heights. Aminex Q15S showed great potential as the method for analyzing juice samples free of dextran as one complete separation can be achieved in 5 min with overlap injections in this all-aqueous column system.

Best resolution between sucrose and reducing sugars was achieved with Aminex Q150S (K^+ form). Also Aminex Q150S is superior for the rapid fractionation of juice samples that have been subjected to a significant degree of microbial attack. We have found that a column packed with this resin can last as long as 4 months without the need of any regeneration.

Samples need only to be clarified by centrifugation and deionized before injection. Samples containing as much as 15% mud have been clarified and analyzed successfully by this procedure without any adverse effects on the column packing material.

The advantage of this system based on Aminex resins over adsorption chromatography is that since the products of deterioration elute first, it is possible to know exactly when the run is over regardless of whether these deterioration products are present or not in the sample. In contrast, adsorption chromatography does not permit this prediction since the deterioration products would be held indefinitely on the column system (Wong-Chong and Martin, 1978).

We have found this all-aqueous system to be well suited for the rapid and accurate analysis of juice samples in research work. The method shows great potential for routine laboratory analysis in the sugar cane industry.

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Determination of Chloramine-T in Foodstuffs

Antonius T. G. Steverink* and Henk Steunenberg

A gas chromatographic method for the quantitative determination of chloramine-T (C-T) in milk, ice cream, whole egg, mechanically deboned poultry meat, and croquettes has been developed. After hydrolysis of the C-T and precipitation and filtration of proteins and lipids, the hydrolysis products, *p*-toluenesulfonamide (*p*-TS), is extracted with ethyl acetate. Dried and concentrated extract is subjected to gas–liquid chromatography (column: 10% OV-7 on 100–120 mesh Gas-Chrom Q), using a flame ionization detector. This method is capable of detecting 1 ppm of C-T and the average recovery is 80%.

Chloramine-T (C-T) [(*N*-chloro-*p*-toluenesulfonamide) sodium] is a widely used disinfectant. For long it was generally believed to be nonpoisonous, although several cases of death and serious poisoning due to mistaking C-T tablets for headache tablets have been reported (Serin, 1949).

Provided C-T is used as a disinfectant for plant sterilization in food industry in the prescribed concentration (3 g/L), and the cleaned objects are well rinsed, less than 1 ppm of C-T will be found in foodstuffs (Van de Haar and Veenkamp, 1977). However, there are indications that C-T is added to various foodstuffs as a preservative in order to suppress bacterial growth (Brouwer, 1956; Van Gils, 1970). The potential toxic property of C-T led to a preferred level of 1 ppm of C-T in foodstuffs (Van Gils et al., 1975).

Although there are already a number of reports describing a variety of detection procedures for C-T in various food products, they are not specific enough (Van Gils, 1970; Van Gils and Hidskes, 1972), time consuming (Brouwer, 1956), or not sensitive enough (Rondags and Beljaars, 1978) or applicable only for one food product (Van Gils and Hidskes, 1972; Van Gils et al., 1975; Rondags and Beljaars, 1978).

Stavric et al. (1974) reported a GLC/FID method for the ortho isomer of toluenesulfonamide (detection limit 0.05 ppm). Using the same procedure these authors also obtained separation of *p*-TS. This paper describes a method for isolation and determination of C-T as *p*-TS in mechanically deboned poultry meat (MDPM), ice cream, milk, whole egg, and croquette.

Spelderholt Institute for Poultry Research, Ministry of Agriculture and Fisheries, 7361 DA Beekbergen, The Netherlands.

EXPERIMENTAL SECTION

Reagents. Carrez I reagent: 10.6 g of potassium ferrocyanide trihydrate (Baker analyzed reagent) in 100 mL of distilled water.

Carrez II reagent: 23.8 g of zinc acetate dihydrate (Baker analyzed reagent) and 3 g of anhydrous acetic acid (Baker analyzed reagent) in 100 mL of distilled water.

Carrez II solution: a 2.5% (v/v) dilution of Carrez II reagent with distilled water. 35% (w/v) basic lead acetate (Baker analyzed reagent) in distilled water; 2% (w/v) sodium sulfite heptahydrate (p.a., Merck) in distilled

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water; 5% (v/v) glacial acetic acid (Baker analyzed reagent) in distilled water.

Spiking solution: 10.0 mg of chloramine-T trihydrate (p.a., Merck) in 100 mL of distilled water.

(External) standard solution: 6.1 mg of *p*-toluenesulfonamide (pure, Koch-Light Laboratories Ltd., Colnbrook Bucks, England) in 100 mL of ethyl acetate (EtAc) (p.a., Merck). Anhydrous sodium sulfate (p.a., Merck).

Gas Chromatography. Analyses were performed on a Perkin-Elmer 3920b gas chromatograph, equipped with a flame ionization detector and a Philips PM 8222 recorder (Philips Nederland B.V., Eindhoven, The Netherlands). A 2 m × 3.175 mm ($^1/_8$ in.) stainless steel column was packed with 10% OV-7 (Applied Science Laboratories Inc., State College, PA) on 100–120 mesh Gas-Chrom Q (Applied Science Laboratories Inc.). Carrier gas (nitrogen) flow was 30 mL/min and the flame gases were hydrogen (140 kPa) and air (300 kPa). Column temperature started at 75 °C for 8 min and then was programmed at 32 °C/min to 245 °C and was kept at 245 °C for 16 min. Injector and detector/interface temperature were 250 and 300 °C, respectively. Under these conditions the retention time for p-TS was 15.33 min.

Procedure. Twenty-five grams of each product (milk, ice cream, whole egg, MDPM, and croquette) was mixed with 100 mL of distilled water in a beaker.

A. Milk–Ice Cream–Blank (100 mL of Distilled Water). Two and one-half milliliters of Carrez I reagent, 2.5 mL of Carrez II reagent, 2.5 mL of the sodium sulfite solution, and 2.5 mL of the basic lead acetate solution were added successively, with stirring, to remove proteins, lipids, stabilizers and emulsifiers, while C-T was hydrolyzed simultaneously to *p*-TS. The mixture was boiled with stirring (Heidolph magnetic stirrer/heating unit type MR 2-E-U, Heidolph-Elektro K.G., D-842 Kelheim, West Germany) 30 min, filtered on a 7-cm i.d. Büchner funnel with S & S 589[3] filter paper (Carl Schleicher & Schüll, 3354 Dassel, West Germany) and the filtrate was collected in a 250-mL beaker placed in a Witt filtration apparatus. Beaker and filter were washed with 20 mL of the Carrez II solution and 20 mL of distilled water. Filtrate and washings were transferred quantitatively to a 250-mL separatory funnel via a moistened cotton plug (to remove fatty matter), extracted with 2 × 50 mL of EtAc and the extracts were collected and dried with anhydrous sodium sulfate. The EtAc was evaporated in a Rotavapor (type RE, Büchi Laboratorium-Technik AG, CH-9230 Flawil, Switzerland) at 30 °C and under diminished pressure (water pump), the residue was transferred to a Reacti-vial (Pierce Eurochemie B.V., Rotterdam, The Netherlands) with 3 × 1 mL of EtAc and the solvent was removed in a stream of nitrogen. Finally the residue was redissolved in 250 μL of EtAc and 3 μL was injected in a GC column.

B. Whole Egg. The egg/water mixture was heated with stirring still coagulation started and next proceeded as in A.

C. Croquette. The product was mixed with the distilled water for 30 min in a centrifuge tube, centrifuged 15 min at 3000 rpm (International PR-6, International Equipment Co., Needham Heights, MA), the upper liquid phase was decanted, and 25 mL of distilled water was added to the residue and after mixing and centrifuging the collected liquid phases from the centrifuge steps were treated as in A. Note: mixing and centrifuging must be performed at room temperature to prevent gel formation.

D. MDPM. After adjustment of the meat/water mixture to a pH of ca. 4.9 with a 5% solution of acetic acid, the mixture was boiled with stirring for 30 min and filtered on a previously heated and wetted 7 cm i.d. Büchner funnel with S & S 589[3] filter paper. The filtrate was treated as in A.

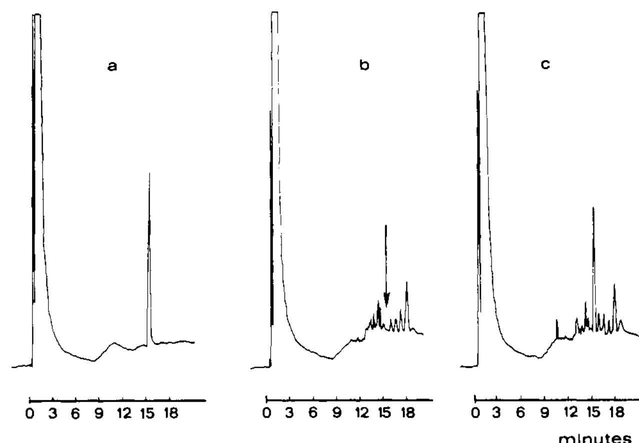

Figure 1. Chromatograms of (a) standard solution of *p*-TS, (b) whole egg blank, and (c) a whole egg sample with a 1 ppm dope of C-T under identical conditions. For GC conditions, see text.

Blanks. Distinction has to be made between the distilled water blank and the product blanks. I. A distilled water blank was carried out to check for interfering peaks from the chemicals used. Further, the procedure was tested by analyzing 25 g of distilled water doped with 1 ppm of C-T. II. Product blanks: each product was tested for interfering peaks. None of the analyzed products gave interfering peaks with the GC conditions of this paper. After these product blanks, each product was spiked with the spiking solution (25 μg of C-T in 250 μL of distilled water was added to 25 g of the product). Recoveries were determined by the ratio

$$\frac{\text{peak ht of } p\text{-TS in sample}}{\text{peak ht of standard sol. of } p\text{-TS}} \times 100\%$$

Figure 1 shows the chromatograms of (a) the standard solution of *p*-TS, (b) a whole egg blank, and (c) a whole egg sample spiked with 1 ppm of C-T.

Note: It is necessary to "saturate the GC system" by alternately injecting samples and standard solution of *p*-TS until a constant response has been obtained. If the system is used continuously, e.g., if the GC is equipped with an automatic injection system, only before the first analysis this saturation has to be performed.

RESULTS AND DISCUSSION

The Dutch "Keuringsdienst van Waren" (Food Inspection Service) consider 1 ppm of C-T in foodstuffs as an acceptable upper limit (Van Gils et al., 1975; Van de Haar and Veenkamp, 1977) although a suitable analytical method for this detectability limit was not available. Therefore, we initiated work in developing an appropriate analytical procedure. Our preliminary investigations with the *p*-TS standards gave excellent results on GLC using either flame ionization or nitrogen phosphorus detectors (Steverink, 1976). Initial analyses of MDPM, although well reproducible, gave recoveries of only 40–45% (Steverink, 1977). Longer boiling time as well as lower pH lead to higher recoveries as presented in Table I. An increase in boiling time to 40 min yielded no appreciable improvement and pH values of 4.5 and lower produced a gel-like mass unsuitable for further analyses. Therefore, a boiling time of 30 min and a pH of 4.9 were found to be the optimal conditions for isolation of C-T from MDPM. Under these conditions ten samples of MDPM were spiked with 1 ppm with the spiking solution and recoveries and standard

Table I. Influence of Boiling Time and pH in the Isolation Step on the Recovery of C-T from MDPM, Spiked with 1 ppm of C-T

boiling time, min	pH	% recovery
10	6.9	45
20	6.9	60
30	6.9	68
20	6.9	60
20	5.9	64
20	4.9	76
20	4.5	gel formation
30	4.9	84

Table II. Recoveries of C-T from Different Foodstuffs, Spiked with 1 ppm of C-T

product	no. of analysis	av % recov. ($P = 95\%$)	SD, %
dist. water	7	89.4 ± 3.7	4.0
milk	10	86.4 ± 6.3	8.8
ice cream	8	91.3 ± 7.5	9.0
MDPM	10	82.6 ± 4.2	5.9
whole egg	10	69.6 ± 2.4	3.4
croquette	9	77.6 ± 2.2	2.9

deviations were determined to check repeatability (reproducibility). Table II shows the results for MDPM and the other products for seven–ten samples of each product after spiking with 1 ppm of C-T.

The proposed method is suitable for the determination of C-T at a desired level in a wide range of products and can be performed with a readily controllable system (GC/FID), which is suitable for routine analysis. The recovery and reproducibility (Table II) of this method are in good agreement with the results of other authors (Van der Haar and Veenkamp, 1977; Rondags and Beljaars, 1978) but it is more sensitive and applicable to a variety of foods.

Finally two remarks: although system saturation can be performed easily, it takes at least 1 h. As an alternative, a glass column cannot be used because of the poor heat transfer that would make temperature programming ineffective. Possibly the new nickel tubing would help this problem.

In other laboratories another problem can be temperature programming with 32 °C/min. Most modern gas chromatographs can easily maintain this rapid rise. For older types which cannot it will be necessary to try a rate higher than 16 °C/min, for this rate does not yield a proper separation of *p*-TS from interfering peaks. In an early stage of our work a sample cleanup was tried (the EtAc extracts were washed with a 0.5 M solution of sodium hydroxide) but this led to lower recoveries (Steverink, 1977). Possibly another type of sample cleanup will do the job (e.g., column chromatography).

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High-Pressure Liquid Chromatographic Determination of Hydroxy-*s*-triazine Residues in Plant Material

K. A. Ramsteiner and W. D. Hörmann*

A specific method is described for quantitative determination of hydroxy-*s*-triazine residues in biological material. The hydroxy metabolites of atrazine, simazine, propazine, terbutylazine, and the corresponding methylthio and methoxy compounds are separated by high-pressure liquid chromatography on a silica gel column and detected at 240 nm with a UV spectrophotometer detector. The procedure involves the extraction of samples with methanol, cleanup with a strong cation-exchange resin, a polyacrylamide adsorption resin, and a styrenedivinylbenzene gel filtration column. The clean-up procedure described is not suitable for dealkylated hydroxy-*s*-triazine metabolites; however, those compounds are separated chromatographically also under the liquid chromatographic conditions given. Recoveries in the range of 70–113% indicated that this procedure is suitable to the residue analysis of hydroxy-*s*-triazines without derivatization of the metabolites with detection limits of 0.05 mg/kg.

An important pathway for the degradation of *s*-triazine herbicides in soil and other biotopes is their conversion to the corresponding hydroxy analogues (Jordan et al., 1970). These represent major alteration products after hydrolytic cleavage of the groups located in position 6. Hydroxy-*s*-triazines exist as tautomeric mixtures involving ring protonated structures (Chen, 1967; Jordan et al., 1970).

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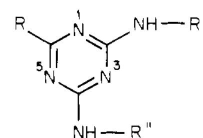

R = Cl, OCH_3, SCH_3

The chemistry of the hydroxy-*s*-triazines is not similar to that of alcohols, phenols, or amides, and due to their physicochemical properties (low volatility, melting points above 580 K, low solubility in organic solvents, chemical

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