SHORT PAPER

Dimethylnitrosamine in Frankfurters

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Summary—Samples of frankfurters from eight large producers were analysed for *N,N*-dimethylnitrosamine (DMNA), using gas chromatography, with an alkali-flame ionization detector for quantitative analysis and a mass spectrometer for confirmation. Only trace levels of a compound that was apparently DMNA were found in the products of five manufacturers. In one further sample a somewhat higher level of a compound presumed to be DMNA was found but again this could not be confirmed. Two samples of frankfurters, containing 84 and 11 μg DMNA/kg respectively, were found among 22 samples from one producer, and one sample out of 12 from another producer contained 48 μg DMNA/kg. These were confirmed. High sodium nitrite values found in the frankfurters from one manufacturer could not be correlated with nitrosamine formation.

Introduction

N-nitrosamines are formed by the reaction of nitrite with an appropriate amine. The curing of meat involves interaction of curing salts, particularly nitrite, with the meat components, and the possible presence of amines, or compounds that may be precursors of amines, in meat has resulted in the investigation of a number of classes of cured meat products for nitrosamines. Positive results have been reported in a few instances. Thus 40 μg diethylnitrosamine/kg was found in a Kasseler-type cured beef (Freimuth & Gläser, 1970) and low concentrations of *N*-nitrosamines (0·8–6 μg/kg) were reported in sausage, bacon and ham (Ender & Ceh, 1968). More recently, *N,N*-dimethylnitrosamine (DMNA) was found at levels of 10–80 μg/kg in five out of 59 samples of prepared meat products (Sen, 1972). Negative results, however, have been obtained in surveys of a larger number of products. Fifty-one samples of a variety of classes of meat products contained 5 μg DMNA/kg or less, and only one, a ham, contained 5 μg/kg confirmed by mass spectrometry (Fazio, White & Howard, 1971). Ten cooked hams, fresh or canned, were found to contain less than 10 μg DMNA/kg (Fiddler, Doerr, Ertel & Wasserman, 1971). At a somewhat lower level of sensitivity (25–60 μg/kg), nitrosamines could not be found in a number of samples of canned pork luncheon meat, Danish back bacon, English bacon, cured ham and fresh pork and beef (Telling, Bryce & Althorpe, 1971).

This paper describes the positive identification and quantitative determination of DMNA in commercially produced frankfurters.

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Experimental

Materials. One-pound packages of frankfurters were purchased from local retailers. The franks were cut in half lengthwise, and then each half was cut into thirds. Alternate pieces were taken for analysis from each half of the frankfurter and the remainder was frozen for storage. A total of 100 g of the product was ground and treated by a modification of the extraction procedure described by Howard, Fazio & Watts (1970). Briefly, the frankfurters were digested with methanolic KOH, an aliquot equivalent to 25 g meat was distilled, and the distillate was extracted with methylene chloride, washed with base and dried with anhydrous sodium sulphate and concentrated. The concentrate was put through an acid-treated Florisil* column, and the nitrosamines were then eluted with methylene chloride, concentrated and analysed by gas chromatography. The concentration of DMNA in every sample was corrected for the recovery of 20 μg DMNA/kg added to an aliquot of a control sample and simultaneously carried through the procedure.

Analytical procedures. Quantitative measurements for DMNA were carried out with a Varian Aerograph Model 1740-1 gas chromatograph using a 9 ft $\times$ $\frac{1}{8}$ in. outer diam. stainless steel column packed with 15% Carbowax 20M-TPA on 60–80 mesh gas chrom P. An alkali-flame ionization detector was used consisting of a KCl-coated coil prepared as described by Howard *et al.* (1970). Flow conditions were helium 58, hydrogen 45 and air 188 ml/min. The hydrogen and air flows were slightly adjusted as necessary to maintain maximum detector sensitivity. Injection port and detector temperatures were 190 and 250°C, respectively, and the column temperature was held isothermally at 115°C. The presence of DMNA was confirmed by the use of a gas chromatograph as described above interfaced with a DuPont Model 21–492 mass spectrometer. The temperature of the gas-chromatograph column, injection port and detector were 115, 200 and 230°C respectively. Helium carrier gas was used at a flow rate of 25 ml/min, and the hydrogen and air flows were 40 and 335 ml/min, respectively. The column effluent was split approximately 1:1, passing into the mass spectrometer via an inlet line heated at 200°C. Mass spectra were obtained at an ionizing voltage of 70 eV and an ion-source temperature of 200°C. Nitrite determinations were carried out by the procedure described by the Association of Official Analytical Chemists (1970).

Results and Discussion

Samples from one package of frankfurters from each of eight major meat-product processors were analysed for residual nitrite and DMNA concentration. The results are shown in Table 1. The first five products contained concentrations of apparent DMNA that were below the 10 μg/kg level established arbitrarily as the confirmable level of nitrosamine. Analysis of the frankfurters from company F showed 36 μg DMNA/kg in the sample. An effort to confirm the presence of DMNA was unsuccessful because of inadequate development of techniques at this stage of our studies. The sample of the product containing DMNA from company F was exhausted in the attempt to concentrate the nitrosamine. However, samples of the frankfurters from companies G and H contained 84 and 48 μg DMNA/kg, respectively, and these findings were confirmed by mass spectrometry.

A survey was instituted of frankfurters produced by companies G and H. Over a period of 2 months, a total of 22 samples of the product of company G and 12 of Company H were

*Reference to a brand or firm name does not constitute endorsement by the US Department of Agriculture over others of a similar nature not mentioned.

Table 1. *Dimethylnitrosamine and nitrite concentrations in frankfurters*

Company	No. of samples analysed	DMNA* (μg/kg)	Nitrite concentration (mg/kg)
A	1	3	6
B	1	Trace	20
C	1	8	7
D	1	9	7
E	1	2	10
F	1	36	4
G	1	84†	151
	1	11†	138
	20	0–6‡	6–230‡
H	1	48†	7
	11	0–6‡	5–76‡

*N,N-Dimethylnitrosamine, corrected for recovery of 20μg DMNA/kg added to an aliquot of the sample.
†Identity confirmed by mass spectrometry.
‡Range of values for the samples analysed.

purchased in various local food markets in a random manner to ensure that different lots of product were obtained. Samples of the frankfurter emulsion before stuffing and samples of product immediately after cooking and smoking were also obtained from the two plants involved. Of the 21 additional samples tested from company G, one contained 11 μg DMNA/kg, confirmed by mass spectrometry. None of the additional samples from company H contained confirmable quantities of DMNA.

Nitrite analyses carried out on the frankfurters showed that the residual nitrite concentrations of all products except those of company G were in the range of 4–76 μg/kg, the normal range found for this type of product. Most of the frankfurters of company G, however, contained residual nitrite in concentrations above 100 μg/kg; in fact, one sample had 230 μg/kg, which is greater than the legally permissible level (200 μg/kg). It is not possible, at this time, to relate higher concentrations of residual nitrite in a frankfurter with nitrosamine formation.

The formation of DMNA in the preparation of frankfurters appears to occur in a random manner, and the conditions leading to its formation are unknown at this time. As a result, detection of the nitrosamine and evaluation of its significance will be difficult unless large numbers of samples are analysed.

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