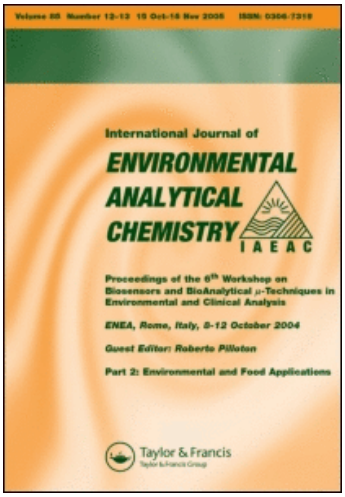


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# A Method of Analysis for Paraffinic Waxes on Ducks

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# A Method of Analysis for Paraffinic Waxes on Ducks

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A method has been developed for the analysis of individual alkanes in ducks and typical results are reported. Recoveries averaged 87% (+12% SD) with a detection limit of 0.05 mg/kg. Alkanes were observed on the duck skin at widely varying levels (e.g. 0.16 to 7.0 mg/kg skin for hexacosane) but were not observed in the duck flesh. These levels would therefore correspond to a total hydrocarbon concentration of 1 to 32 mg/kg duck which are small compared to other food products. Evidence is presented that the major source of these alkanes is from wax used in the duck defeathering process.

KEY WORDS: Analysis, duck, wax, hydrocarbons.

## INTRODUCTION

A number of methods of analysis of paraffin waxes in various foods have been reported. Lawrence and Iyengar[1] have described a packed column gas chromatographic (GC) method for fresh fruits and vegetables. Little sample preparation is required for these materials; the foods are simply dipped in chloroform and the solvent concentrated and applied directly to the GC. Oils and fats require rather more clean-up and Franzke and Kroll[2] have reviewed a number of methods for analysing mineral oils in these matrices. They have successfully used both thin layer chromatography (TLC) with phos-

phomolybdic acid detection and column chromatography of the saponified sample with gravimetric determination.

Although the presence of hydrocarbons on fruit and vegetables and in oils and fats has been established little work seems to have been done on meat. Recent changes in slaughterhouse procedures has lead to an increased likelihood of some poultry coming into contact with paraffin waxes. The application of mechanical plucking operations to waterfowl has proved difficult due to incomplete removal of the feathers. To overcome this problem it is now usual, after mechanical defeathering, for ducks to pass through a bath of molten wax. On removing the duck from the waxbath a coat of solid wax forms over the bird. When this is removed any remaining feathers are taken with it. Gross contamination of the bird with pieces of wax is most unusual but it is possible that trace residues of the wax remain.

In this paper we describe a suitable sample clean-up and capillary GC analysis of paraffin waxes on duck skins.



## METHODS

### Sample preparation

Duck skin (about 100 g) including subcutaneous adipose tissue was removed from a number of places on the duck and homogenised. Flesh samples were then removed taking care to avoid any cross contamination from the duck skin. Samples were stored frozen (−18°C) until ready for analysis. All glassware was cleaned in chromic acid and thoroughly rinsed before use. Millipore purified water and glass redistilled solvents were used throughout.

### Extraction procedure

Duck skin (2.0 g), potassium hydroxide (3 g), water (2 ml), ethanol (30 ml), internal standard ($C_{20}H_{42}$ 6 μg/ml, $C_{34}H_{70}$ 30 μg/ml in toluene) (200 μl) and three antibumping granules were refluxed together for 30 minutes. The digest was quantitatively transferred to a 250 ml separating funnel with water (50 ml) and hexane (75 ml). After extraction into hexane the aqueous phase was discarded and the hexane was further washed with water (50 ml) and alternately

0.5 N potassium hydroxide (20 ml) three times and water (20 ml) twice before a final wash with water (50 ml). The hexane was dried over Aristar grade anhydrous sodium sulphate which was then washed with more hexane (25 ml). The combined hexane solutions were reduced to about 1 ml by rotary evaporation.

A silica Sep-pak (Waters Assoc.) was prewashed with 40 ml hexane. The concentrated extract was applied to it and eluted with a further 3 ml of hexane used to rinse the flask. The entire eluate was collected, taken to dryness and redissolved in toluene (200 $\mu$l).

### Gas chromatographic (GC) analysis

A BP1 (equivalent to SE30) silica capillary column, 25 m, 0.32 mm ID, phase thickness 1.0 $\mu$m, was used for analysis of the alkanes using helium carrier gas with a temperature programme of 110°C (for 1 min) to 170°C (for 1 min) at 25°/min then to 280°C (for 30 min) at 5°/min. Splitless injections (2 $\mu$l) were performed with an injector temperature of 250°C and FID detector at 300°C.

Quantitation was achieved by comparison of peak heights with those of appropriate standards of known concentration. Pure standards of heptacosane ($C_{27}H_{56}$), nonacosane ($C_{29}H_{60}$) and hentriacontane ($C_{31}H_{64}$) were not available so that no attempt has been made to quantitate these alkanes. These compounds have however been tentatively identified in duck skins by comparison of retention times with the appropriate peak of the wax chromatogram. Two internal standards, eicosane and tetratriacontane, were used to ensure that recovery was constant over the entire volatility range. Since tetratriacontane was less susceptible to interference this was used for recovery calculations.

## RESULTS AND DISCUSSION

The results of twelve analyses of duck skin for paraffinic hydrocarbons are shown in Table I. Recoveries of both internal standards were good and averaged 87% for tetratriacontane with a standard deviation of 12%. The detection limit varied between 0.03 mg/kg and 0.05 mg/kg depending on the response factor for a particular alkane. The chromatogram of a duck skin extract (Figure 1) shows the efficiency of the clean-up with the *n*-alkanes being the only signifi-

**Table I** Paraffinic hydrocarbons on duck skin (mg/kg)

| Hydrocarbon | Duck Sample | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | A | B | C | D | | E | F | G | H | I |
| | | | | Skin | Flesh | | | | | |
| Heneicosane $C_{21}H_{44}$ | 0.14 | 0.05 | 0.08 | 0.08 | <0.03 | 0.08 | 0.30 | 0.14 | 0.06 | 0.17 |
| Docosane $C_{22}H_{46}$ | 0.11 | 0.08 | 0.14 | 0.10 | <0.03 | 0.42 | 1.18 | 0.58 | 0.19 | 0.45 |
| Tricosane $C_{23}H_{48}$ | 0.24 | 0.14 | 0.31 | 0.23 | <0.03 | 1.37 | 3.20 | 1.40 | 0.49 | 0.13 |
| Tetracosane $C_{24}H_{50}$ | 0.37 | 0.26 | 0.63 | 0.33 | <0.03 | 2.39 | 4.72 | 2.19 | 0.59 | 0.11 |
| Pentacosane $C_{25}H_{52}$ | 0.61 | 0.33 | 0.87 | 0.38 | <0.03 | 3.72 | 5.29 | 2.38 | 0.74 | 0.22 |
| Hexacosane $C_{26}H_{54}$ | 0.80 | 0.56 | 1.42 | 0.44 | <0.05 | 5.64 | 7.00 | 2.86 | 1.01 | 0.16 |
| Octacosane $C_{28}H_{58}$ | 0.49 | 0.32 | 0.89 | 0.20 | <0.05 | 3.64 | 3.12 | 1.23 | 0.39 | 0.11 |
| Triacontane $C_{30}H_{62}$ | 0.22 | 0.28 | 0.77 | 0.08 | <0.05 | 1.85 | 1.17 | 0.44 | 0.16 | 0.05 |
| Dotriacontane $C_{32}H_{66}$ | Not analysed | 0.17 | 0.28 | 0.11 | <0.05 | 0.47 | 0.50 | 0.13 | 0.05 | Not analysed |
| Eicosane $C_{20}H_{42}$ (Std) | 87% | 74% | 118% | 134% | 84% | 87% | 97% | 92% | 76% | 119% |
| Tetratriacontane $C_{34}H_{70}$ (Std) | 90% | 66% | 94% | 106% | 74% | 89% | 93% | 78% | 91% | 98% |

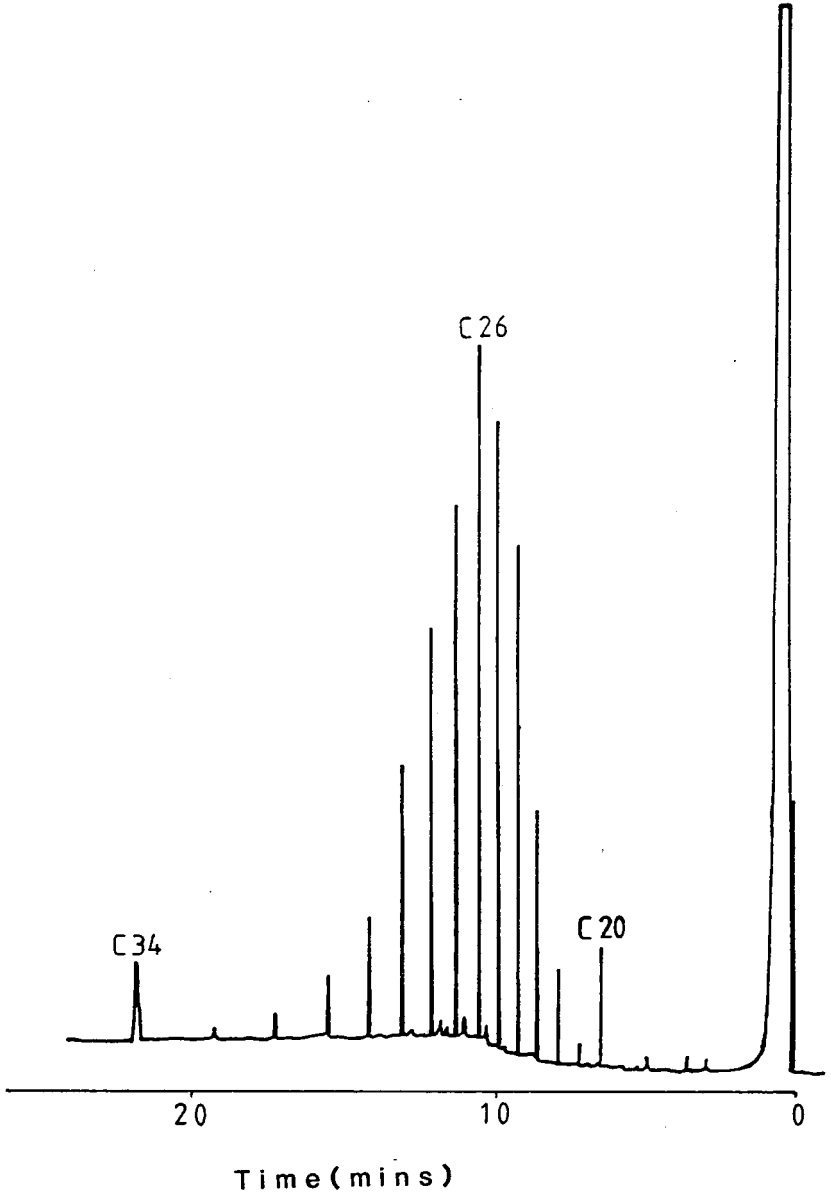


**Figure 1** GC/FID Chromatogram of sample E duck skin extract ( × 32).

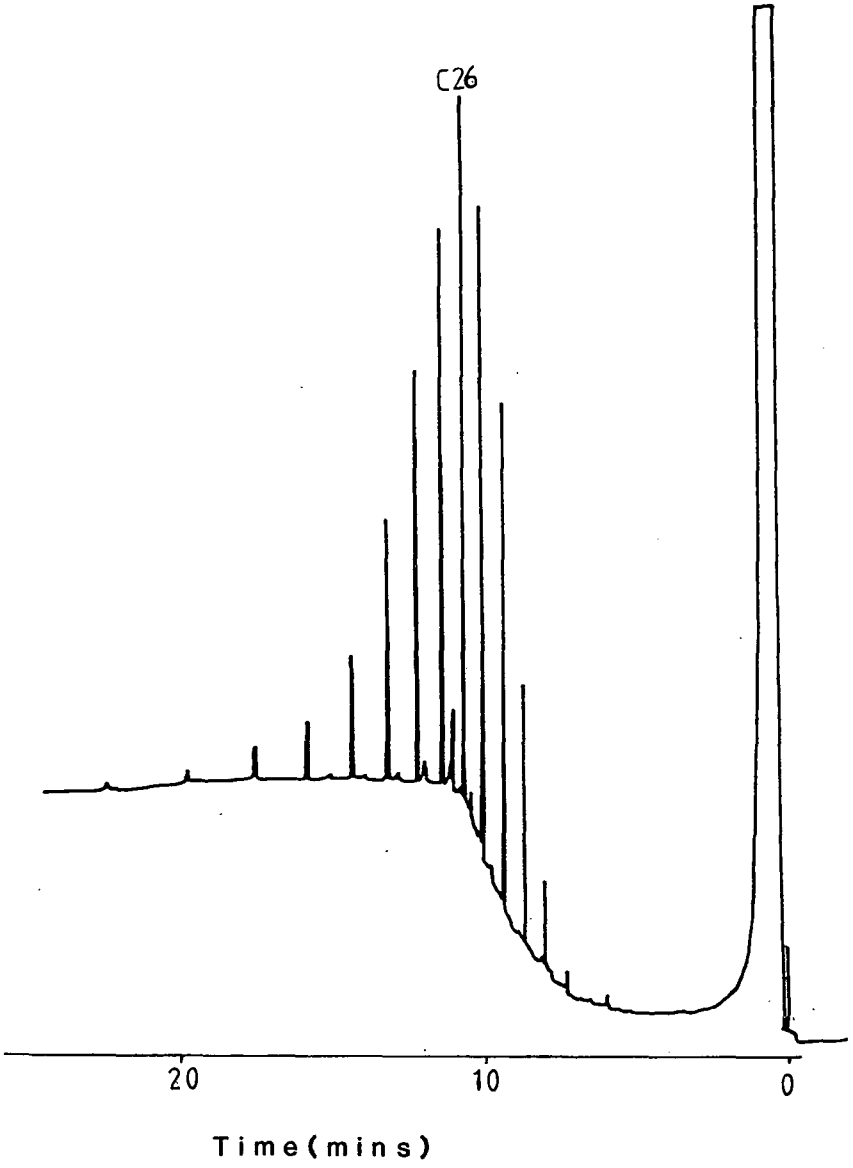


**Figure 2** GC/FID Chromatogram of wax sample (×4).

cant peaks in the chromatogram. The chromatogram of a sample of duck defeathering wax (Figure 2) taken from the slaughterhouse shows a remarkably similar profile to that of the duck skin extract and this "fingerprint" is strongly indicative that the alkanes on the duck skin came from this wax.

Analysis of the duck defeathering wax showed that the alkanes observable by GC only comprised 28% (w/w) of the wax. TLC showed only one spot at the solvent front when run with hexane as eluant, indicating the paraffinic nature of the wax. These results suggest that the remainder of the wax was composed of higher molecular weight alkanes which were not sufficiently volatile to permit analysis by GC.

The levels of hydrocarbons on the duck skin vary quite widely with hexacosane ($C_{26}H_{54}$) the most abundant alkane, ranging from 0.16–7.0 mg/kg. If the whole of the wax is deposited on the duck skin in proportion to the level of hexacosane (5.8%) then the amount of wax on the duck skin will range from 8–120 mg/kg skin. Since the duck flesh contains no detectable level of alkanes and the skin accounts for 26% of the bird weight, this is equivalent to 1–32 mg/kg on a whole duck basis. These values are considerably less than those reported for edible oils/fats[2] of 0.02–0.12% (200–1200 mg/kg) and should not be of concern in a food safety context.



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