**Registry No.** MIU, 2440-60-0; MIU sulfate, 294227-58-5; L-lysine, 56-87-1; L-homoarginine, 156-86-5; barium hydroxide, 12009-08-4.

LITERATURE CITED

Chervenka, C. H.; Wilcox, P. E. Chemical derivatives of chymotrypsinogen. II Reaction with O methylisourea. *J. Biol. Chem.* **1956**, *222*, 635–647.

Greenstein, J. P. Studies of multivalent amino acids and peptides. II The synthesis of certain derivatives of lysylglutamic acid. *J. Biol. Chem.* **1935**, *109*, 541–544.

Hagemeister, H.; Erbersdobler, H. Chemical labelling of dietary protein by transformation of lysine to homoarginine: a new technique to follow intestinal digestion and absorption. *Proc. Nutr. Soc.* **1985**, *44*, 133A.

Hettinger, T. P.; Harbury, H. A. Guanidated cytochrome c. II. *Biochemistry* **1965**, *4*, 2585–2589.

Hughes, W. L.; Saroff, H. A., Jr.; Carney, A. L. Preparation and properties of serum and plasma proteins. XXII. A crystallizable guanidated derivative of human serum albumin. *J. Am. Chem. Soc.* **1949**, *71*, 2476–2480.

Hurrell, R. F.; Carpenter, K. J. Mechanisms of heat damage in proteins. 4. The reactive lysine content of heat-damaged material as measured in different ways. *Br. J. Nutr.* **1974**, *32*, 589–604.

Kapfhammer, J.; Muller, H. Guanido acids and guanido peptides. *Z. Physiol. Chem.* **1934**, *225*, 1.

Kassell, B.; Chow, R. B. Modification of the Basic Trypsin Inhibitor of Bovine Pancreas. The ϵ-amino groups of lysine and the amino-terminal sequence. *Biochemistry* **1966**, *5*, 3449–3453.

Kenchington, A. W. Chemical Modification of the side chains of gelatin. *Biochem. J.* **1958**, *68*, 458–467.

Klee, W. A.; Richards, F. M. The Reaction of O-methylisourea with bovine pancreatic ribonuclease. *J. Biol. Chem.* **1957**, *229*, 489–504.

Maga, J. A. Measurement of available lysine using the guanidation reaction. *J. Food Sci.* **1981**, *46*, 132–134.

Mauron, J.; Bujard, E. Guanidation, an alternative approach to the determination of available lysine in foods. *Proc. 6th Int. Nutr. Congr.* **1964**, 489–490.

Means, G. E.; Feeney, R. E. Acylating and similar reagents. *Chemical Modifications of Proteins*; Holden-Day: San Francisco, 1971; Chapter 5.

Shields, G. S.; Hill, R. L.; Smith, E. L. Preparation and properties of guanidated Mercuripapain. *J. Biol. Chem.* **1959**, *234*, 1747–1753.

Siriwan, P.; Bryden, W. L. Determination of endogenous amino acids using homoarginine. *Proceedings of the Poultry Husbandry Research Foundation*, Symposium, University of Sydney, Australia, 1987.

Received for review November 29, 1988. Revised manuscript received April 25, 1989. Accepted June 30, 1989.

# Retinol, Total Carotenoids, β-Carotene, and Tocopherols in Total Diets of Male Adolescents in The Netherlands

Wim van Dokkum,* Rudi H. de Vos, and Jaap Schrijver

Division of Nutrition and Food Research TNO, CIVO Institutes, Zeist, The Netherlands

Over a period of 2.5 years, every 3 months 221 different food items forming a "market basket" were purchased, prepared, and divided into 23 food commodity groups. The market basket was based on a study of the dietary intake of 18-year-old males. In the (homogenized) food groups vitamin A, β-carotene, and total carotenoids as well as α-, β-, γ- and δ-tocopherol were determined by chemical analysis. The analyzed total daily amount of vitamin A (1481 retinol equivalents or 4937 IU) can be considered as more than sufficient when compared to Dutch recommendations for male adolescents. Also the vitamin E intake (22.4 IU) meets the requirements. The group meat and meat products shows the highest contribution to the daily amount of retinol (37%); for the carotenoids, the groups leafy vegetables and root vegetables are the most important sources. The group butter, margarine, oils is the most important source for the average daily supply of all analyzed tocopherols (32–49% contribution). Regarding seasonality, highest daily amounts of most vitamins were observed in the months November/December, while lowest daily intakes seem applicable in May.

During the period 1976–1978 the first total diet study in The Netherlands with the market basket approach was carried out (van Dokkum et al., 1982; de Vos et al., 1984).

The aim of total diet studies is to monitor the exposure to additives and contaminants through habitual diets and to estimate the health risk for the consumer, by comparing the actual, analyzed contents with the acceptable daily intake (ADI) as established by FAO/WHO.

Total diet studies as defined and recommended by the FAO/WHO (WHO, 1976, 1985) are also well suited for evaluating the nutritional quality of well-defined diets (EuroNut, 1988).

In the 1976–1978 study, of all vitamins only α-tocopherol was determined; the present study was designed to include other fat-soluble vitamins as well.

In this paper we report results of the second total diet study, carried out in 1984–1986, regarding vitamins A and E. In this study the approach of a "market basket" survey was chosen, based on a dietary intake by a defined population group. The composition of the diet analyzed comprised the average total diet of 18-year-old males. This group probably has the highest food consumption as compared to other age categories and consequently is likely to have the highest intake of additives and con-

0021-8561/90/1438-0211$02.50/0 

**Table I. Market Basket of 18-Year-Old Males (g/Day)**

| | food group | main group | sub-groups |
|---|---|---|---|
| 1 | cereal products | 329 | |
| 1A | bread | | 249 |
| 1B | biscuits | | 48 |
| 1C | rice, macaroni, etc. | | 32 |
| 2 | potatoes and potato products | 260 | |
| 2A | potatoes | | 230 |
| 2B | potato products | | 30 |
| 3 | vegetables | 210 | |
| 3A | leafy vegetables | | 61 |
| 3B | other vegetables | | 33 |
| 3C | soups | | 116 |
| 4 | root vegetables | 16 | |
| 5 | legumes | 18 | |
| 6 | fruits | 256 | |
| 6A | fresh fruits | | 130 |
| 6B | canned fruits and fruit juices | | 126 |
| 7 | meat, poultry, eggs | 141 | |
| 7A | meat and meat products | | 108 |
| 7B | poultry and eggs | | 33 |
| 8 | fish | 9 | |
| 9 | milk and dairy products | 602 | |
| 9A | milk | | 316 |
| 9B | milk products and dairy products | | 286 |
| 10 | oils and fats | 75 | |
| 10A | butter, margarine, oils | | 64 |
| 10B | nuts | | 11 |
| 11 | sugar and sweets | 78 | |
| 12 | drinks and drinking-water | 1617 | |
| 12A | drinks | | 1257 |
| 12B | drinking-water | | 360 |
| | misc group (noodles, pizza, etc.) | 20 | |

taminants, the initial rationale to perform total diet studies.

## MATERIALS AND METHODS

In 1982 and 1983 the food consumption of 187 18-year-old males was surveyed (dietary history method with emphasis on the food consumption over the previous 14 days). The data obtained resulted in a total diet consisting of 426 food products. Only food items that contributed by more than 0.02% (by weight) to the total diet were incorporated in the final market basket. To this end, various products were aggregated into groups of similar food items on the basis of food type and nutrient content. Finally, 221 food products formed the market basket of the present study. These products contributed about 98% to the weight and energy content of the average total diet of male adolescents.

During a period of $2^1/_2$ years (1984–1986), every 3 months the complete set of (221) foods was purchased in Zeist, a town of 60 000 inhabitants, in the center of The Netherlands. The sites of purchase were varied, a wide variety of product brands was chosen, and seasonal variation in the consumption of fruits and vegetables was taken into account. In this way 10 samplings (market baskets) were collected in the period mentioned.

The various food items were prepared in the way they would normally be served and eaten. Cleaning and cooking were carried out according to standard procedures and supervised by a dietitian. After preparation, the foods were combined into 23 commodity classes, representing the basic 2-week diet of 18-year-old males. Foods in each of these 23 groups were homogenized and frozen at −20 °C.

Samples from each mixture were analyzed for the presence of retinol, total carotenoids, β-carotene, and four tocopherols (α, β, γ, δ). Table I shows the composition of the market basket divided into the 23 commodity groups.

## ANALYTICAL PROCEDURES

**Vitamin A** was determined in all groups in which food items of animal origin were incorporated. The vitamin A concentration (total *all-trans*-retinol = retinol + esters) was analyzed by

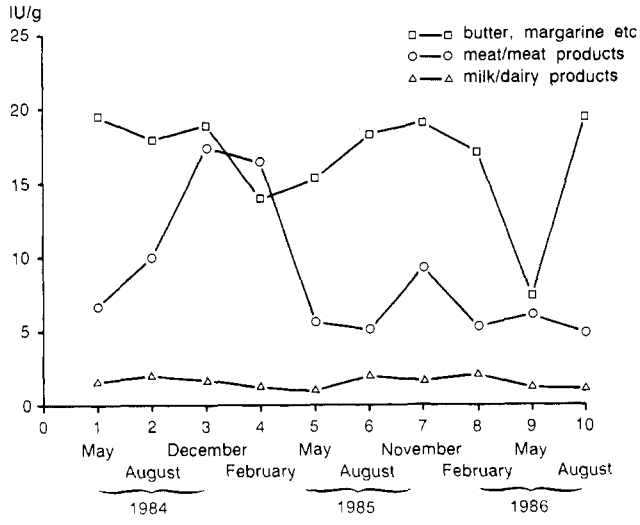


**Figure 1.** Seasonal differences of the retinol content in the three core food groups of the market basket of 18-year-old males.

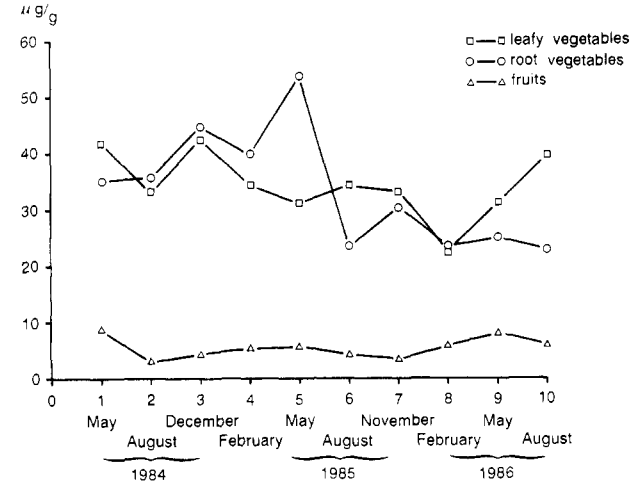


**Figure 2.** Seasonal differences of the total carotenoid content in the three core food groups of the market basket of 18-year-old males.

high-performance liquid chromatography (HPLC) (de Ruyter and de Leenheer, 1976) after alkaline saponification of the samples (Speek et al., 1985, 1986). Depending on the expected content, 1–10 g of a carefully homogenized food sample was mixed with 10 mL of 10% (w/v) sodium ascorbate solution and 5 mL of an aqueous solution containing 12% (w/v) sodium sulfide ($Na_2S$) and 70% glycerol. This mixture was saponified in a receiver on a boiling water bath with refluxing for 30 min after the addition of 50 mL of a solution of 2 M KOH in ethanol.

After the mixture was cooled to room temperature with use of running tap water, 100 mL of diisopropyl ether was added and the resultant mixture thoroughly mixed with the contents of the receiver. After separation of the layers, the top layer was transferred to a separation funnel containing 100 mL of 5% (w/v) KOH and the contents were thoroughly shaken for 30 s. After separation, the bottom layer was run off and discarded. The ether layer remaining in the funnel was washed three times with 100 mL of distilled water to remove KOH, followed by drying with pieces of filter paper. Absolute ethanol (5 mL) was used in case of emulsion formation. The concentration of vitamin A in the extract was brought to ca. 1 IU/mL by dilution or by concentration under vacuum rotation evaporation, followed by dissolution of the extract in diisopropyl ether. Recovery tests were carried out for added *all-trans*-retinyl acetate in amounts of the same order of magnitude as could be expected in the various food groups. The average recovery of the added *all-trans*-retinyl acetate was 96.0% (range 80–114%; $n$ = 228). The detection limit was about 0.05 IU of *all-trans*-retinol/g of food (depending on the extent the extract

**Table II. Mean Concentrations and Ranges of Compounds in 23 Food Groups Forming the Average Total Daily Diet of 18-Year-Old Males**

**A. Retinol, Total Carotenoids, and β-Carotene**

| food group[a] | retinol, IU/g | | total carotenoids, μg/g | | β-carotene, μg/g | |
|---|---|---|---|---|---|---|
| | mean | range | mean | range | mean | range |
| 1A | 0.03 | 0–0.2 | 0.60 | 0.30–1.0 | 0.01 | 0–0.06 |
| 1B | 2.29 | 1.1–3.5 | 1.81 | 1.1–2.7 | 0.36 | 0.1–0.6 |
| 1C | 0.08 | 0–0.7 | 0.66 | 0.4–1.1 | 0.02 | 0–0.1 |
| 2A | 0 | 0 | 0.75 | 0.2–1.0 | 0.01 | 0–0.1 |
| 2B | 0.01 | 0–0.1 | 5.23 | 1.5–11.0 | 0.22 | 0–0.8 |
| 3A | 0 | 0 | 34.40 | 22.6–42.6 | 5.70 | 3.8–8.2 |
| 3B | 0 | 0 | 9.73 | 1.7–18.8 | 2.00 | 1.3–2.9 |
| 3C | 0 | 0 | 2.89 | 0.6–5.1 | 0.77 | 0.1–1.6 |
| 4 | 0 | 0 | 33.50 | 22.9–54.0 | 20.30 | 9.6–32.2 |
| 5 | 0 | 0 | 11.60 | 9.5–14.1 | 2.03 | 1.4–2.7 |
| 6A | 0 | 0 | 5.46 | 3.0–8.7 | 0.28 | 0.2–0.4 |
| 6B | 0 | 0 | 2.40 | 1.6–3.2 | 0.19 | 0–0.3 |
| 7A | 8.68 | 4.90–17.4 | 1.11 | 0.5–1.9 | 0.35 | 0–0.7 |
| 7B | 2.46 | 1.60–3.3 | 5.36 | 4.2–6.9 | 0.10 | 0–0.2 |
| 8 | 5.79 | 1.50–16.0 | 1.02 | 0.4–2.4 | 0.19 | 0–0.3 |
| 9A | 0.85 | 0.60–1.1 | 0.19 | 0.1–0.4 | 0.12 | 0–0.3 |
| 9B | 1.56 | 1.00–2.1 | 0.44 | 0.2–0.6 | 0.32 | 0.2–0.5 |
| 10A | 16.70 | 7.40–19.5 | 5.95 | 3.6–7.0 | 3.37 | 0.6–5.1 |
| 10B | 0 | 0 | 0.99 | 0.2–2.2 | 0.07 | 0–0.2 |
| 11 | 0.20 | 0–0.8 | 0.25 | 0–0.5 | 0.04 | 0–0.1 |
| 12A | 0.02 | 0–0.2 | 0.07 | 0–0.3 | 0.01 | 0–0.1 |
| 12B | 0 | 0 | 0 | 0 | 0 | 0 |
| misc | 0.32 | 0–0.5 | 3.98 | 2.5–7.6 | 0.42 | 0.2–0.9 |

**B. Tocopherols**

| food group[a] | α-tocopherol, μg/g | | β-tocopherol, μg/g | | γ-tocopherol, μg/g | | δ-tocopherol, μg/g | |
|---|---|---|---|---|---|---|---|---|
| | mean | range | mean | range | mean | range | mean | range |
| 1A | 2.59 | 1.5–5.4 | 1.18 | 0–3.1 | 8.44 | 5.6–12.6 | 0.15 | 0–0.5 |
| 1B | 17.00 | 14.0–19.8 | 1.45 | 0.8–2.2 | 19.30 | 3.1–34.2 | 5.36 | 2.2–10.5 |
| 1C | 1.13 | 0–2.3 | 0.31 | 0–0.5 | 1.69 | 0.5–3.4 | 0.56 | 0–3.0 |
| 2A | 0.65 | 0–1.2 | 0 | 0 | 0.02 | 0–0.2 | 0 | 0 |
| 2B | 12.40 | 5.8–24.0 | 1.76 | 0.6–5.7 | 34.70 | 11.1–62.0 | 20.80 | 8.6–34.3 |
| 3A | 8.88 | 5.5–11.6 | 0.04 | 0–0.3 | 4.63 | 2.0–8.2 | 0.38 | 0–1.4 |
| 3B | 4.30 | 3.3–6.1 | 0.03 | 0–0.2 | 2.99 | 0–3.9 | 0.22 | 0–0.6 |
| 3C | 1.92 | 1.3–3.2 | 0.05 | 0–0.4 | 1.06 | 0.7–1.4 | 0.07 | 0–0.5 |
| 4 | 6.58 | 3.2–12.5 | 0.04 | 0–0.4 | 2.22 | 0–4.8 | 0.52 | 0–1.9 |
| 5 | 1.24 | 1.0–1.4 | 0 | 0 | 19.20 | 15.1–25.0 | 0.69 | 0.4–1.5 |
| 6A | 3.37 | 2.1–5.5 | 0.10 | 0–0.4 | 0.15 | 0–0.5 | 0 | 0 |
| 6B | 2.83 | 2.4–3.2 | 0.02 | 0–0.2 | 0.10 | 0–0.3 | 0.06 | 0–0.3 |
| 7A | 11.30 | 2.7–20.3 | 0.94 | 0–2.0 | 15.50 | 0.7–30.6 | 6.87 | 1.1–15.4 |
| 7B | 17.70 | 11.3–22.6 | 0.42 | 0–0.9 | 12.70 | 6.0–19.4 | 2.88 | 0–6.2 |
| 8 | 32.70 | 18.9–65.3 | 0.52 | 0–1.2 | 21.10 | 7.1–33.0 | 6.96 | 1.1–17.4 |
| 9A | 1.39 | 0.3–6.5 | 0.03 | 0–0.2 | 0.85 | 0–8.0 | 0.31 | 0–1.9 |
| 9B | 2.36 | 1.6–4.0 | 0.01 | 0–0.1 | 0.23 | 0–1.0 | 0.25 | 0–1.9 |
| 10A | 108.00 | 84.5–136.7 | 7.03 | 0–14.5 | 187.0 | 99.2–313 | 52.90 | 21.7–148.0 |
| 10B | 78.80 | 12.0–120.0 | 3.79 | 0–6.7 | 135.0 | 74.8–177 | 23.30 | 8.5–34.6 |
| 11 | 3.03 | 1.9–3.8 | 0.12 | 0–0.5 | 9.16 | 7.0–11.8 | 1.48 | 0.9–2.9 |
| 12A | 0.21 | 0–1.0 | 0.05 | 0–0.5 | 0 | 0 | 0 | 0 |
| 12B | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| misc | 5.16 | 1.9–9.6 | 0.33 | 0.2–0.5 | 6.28 | 1.9–12.4 | 1.82 | 0.8–4.0 |

[a] The code numbers refer to the food groups as mentioned in Table I.

was concentrated for analysis); the limit of quantitation was about 0.30 IU/g of food.

The amounts of **total carotenoids** and of **β-carotene** were determined according to Speek et al. (1986) by colorimetry and HPLC, respectively. Saponification and extraction of samples were carried out as described above for vitamin A. The diisopropyl ether extract was evaporated by vacuum rotation. The residue was dissolved in the HPLC eluents. Recovery tests were carried out for added β-carotene. The average recovery of added β-carotene in HPLC analysis was 96.9% (range 68–126%; $n$ = 210).

In the colorimetric analysis of total carotenoids, the recovery of the same added β-carotene was 98.6% (range 81–126%; $n$ = 204).

The detection limit was about 0.05 μg of β-carotene/g of food; the limit of reliable quantitation was 0.20 μg of β-carotene/g of food for the HPLC determination of β-carotene and about 0.10 μg of β-carotene/g of food for the colorimetric analysis of total carotenoids.

The various tocopherols were analyzed according to Speek et al. (1985). The concentration of vitamin E in the extract was brought to ca. 10 μg/mL by dilution in $n$-hexane or by vacuum rotation evaporation followed by dissolution of the extract in $n$-hexane.

Recovery tests were performed by addition of α-tocopheryl acetate. The average recovery of added acetate was 98.9% (range 84–113%; $n$ = 218). No recovery tests were carried out for the other tocopherols. The detection limit was about 0.10 μg of tocopherol/g of food; the limit of reliable quantitation was about 1.0 μg of tocopherol/g of food. The total vitamin A activity of a food sample was calculated from retinol, total carotenoids, and β-carotene contents as indicated in the legend to Table III. Conversion factors for the various tocopherols are indicated as well.

## RESULTS AND DISCUSSION

Table II gives the mean concentration and the range of retinol, total carotenoids, β-carotene, and tocopherols

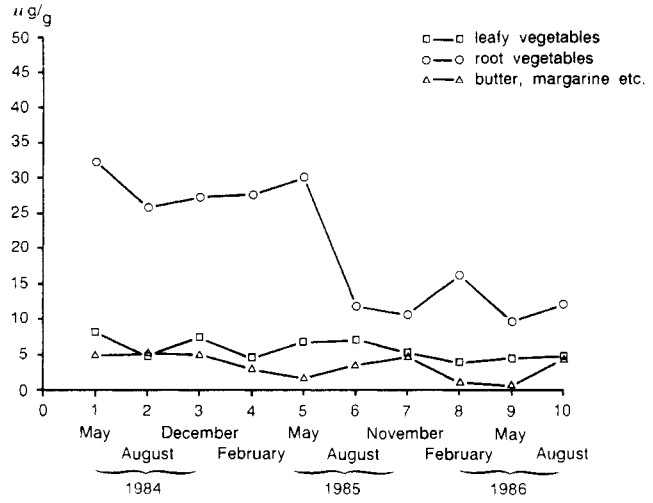


**Figure 3.** Seasonal differences of the β-carotene content in the three core food groups of the market basket of 18-year-old males.

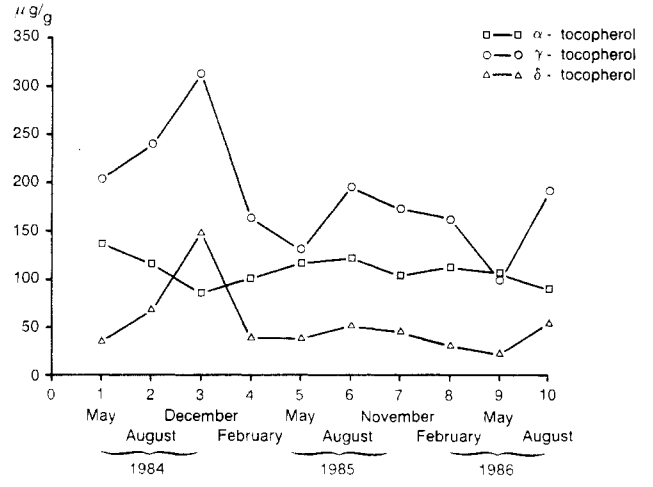


**Figure 4.** Seasonal differences of the α-, γ-, and δ-tocopherol contents in the group butter, margarine, oils of the market basket of 18-year-old males.

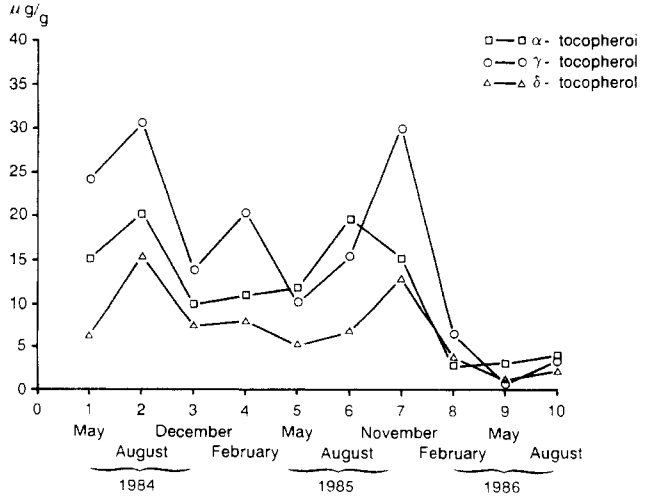


**Figure 5.** Seasonal differences of the α-, γ-, and δ-tocopherol contents in the group meat and meat products of the market basket of 18-year-old males.

in the 23 food groups. The range reflects the seasonal differences in vitamin content of the various food groups; seasonality of the concentration of the vitamins in the "core food groups" (those groups contributing most to the daily "intake" of the vitamins) is reflected in Figures 1–5.

**Table III. Vitamins A and E in Total Diets of Male Adolescents**

| vitamin | total daily amt (range) | recommendations[a] (for male adolescents) |
|---|---|---|
| vitamin A | 2880 IU (2090–4120) | |
| carotenoids | 6.09 mg (5.27–7.00) | 3333 IU[c] |
| β-carotene | 1310 μg (700–1900) | |
| α-tocopherol[b] | 13.9 mg (10.4–17.1) | |
| β-tocopherol[b] | 1.05 mg (0.52–2.15) | 16 IU[d] |
| γ-tocopherol[b] | 19.1 mg (11.4–25.7) | (1.0 IU/g PUFA) |
| δ-tocopherol[b] | 5.10 mg (2.43–9.94) | |

[a] Provisional recommendations (Dutch Nutrition Council, to be published). [b] Calculated as acetate. [c] 1 RE (retinol equivalent) is defined as 1 μg of *all-trans*-retinol, which is equivalent to $^{10}/_{3}$ IU of vitamin A. The mean daily value for *all-trans*-retinol is 2880 IU of vitamin A (864 RE). The conversion factor is $^{1}/_{6}$ for β-carotene and $^{1}/_{12}$ for the other carotenoids analyzed, which means that 6 μg of β-carotene and 12 μg of the other carotenoids are equivalent to 1 RE. From this it can be calculated that 1310 μg of β-carotene and 4780 (6090 − 1310) μg of the other carotenoids together are equivalent to 617 RE or 2056 IU of vitamin A: [6090 − 1310]/12 + 1310/6 = 617 RE = 617/0.3 = 2056 IU of vitamin A. The total mean daily amount is thus 864 + 617 = 1481 RE (=4937 IU of vitamin A). [d] 1 IU of vitamin E is defined as 1 mg of (*dl*)-α-tocopheryl acetate = 0.74 mg of (*d*)-α-tocopheryl acetate = 1.47 mg of (*d*)-β-tocopheryl acetate = 7.14 mg of (*d*)-γ-tocopheryl acetate = 20 mg of (*d*)-δ-tocopheryl acetate. Assuming that all tocopherols present in the foods would have the *d* structure, it can be calculated that average vitamin E intake is 22.43 IU/day: 13.90/0.74 + 1.05/1.47 + 19.10/7.14 + 5.10/20.00 = 22.43 IU of vitamin E.

**Table IV. Three Food Groups with the Highest Percentage Contribution to the Total Amount of the Various Vitamins in the Average Diet of Male Adolescents**

vitamin A: meat and meat products, 37%; butter, margarine, oils, 28%; dairy products, 16%
carotenoids: leafy vegetables, 34%; fruits, 12%; root vegetables, 9%
β-carotene: leafy vegetables, 26%; root vegetables, 25%; butter, margarine, oils, 12%
α-tocopherol: butter, margarine, oils, 37%; meat and meat products, 10%; nuts, 6%
β-tocopherol: butter, margarine, oils, 32%; bread, 28%; meat and meat products, 11%
γ-tocopherol: butter, margarine, oils, 46%; bread, 11%; meat and meat products, 10%
δ-tocopherol: butter, margarine, oils, 49%; meat and meat products, 17%; potato products, 12%

The total daily amounts of the various vitamins in the average diet of male adolescents are presented in Table III. For each of the 10 total diet samplings, these values were calculated from the concentrations of the vitamin contents in the various food groups and the daily amounts of those groups in the total diet as mentioned in Table I. In Table III the mean value of the 10 samplings and the range are given, as well as the Dutch recommendations for daily dietary intake of the vitamins analyzed. For each vitamin the three food groups with the highest contribution to the total daily intake are presented in Table IV. For all analyzed vitamins, seasonality with respect to the total daily amounts is shown in Figures 6 and 7.

As could be expected, high concentrations of vitamin A were found in the food groups meat and meat products and butter, margarine, oils. The contribution of both food groups to the daily amount of vitamin A is high. Despite a low concentration of vitamin A in the group milk products and dairy products, the significance of this group for the daily supply of vitamin A is not to be

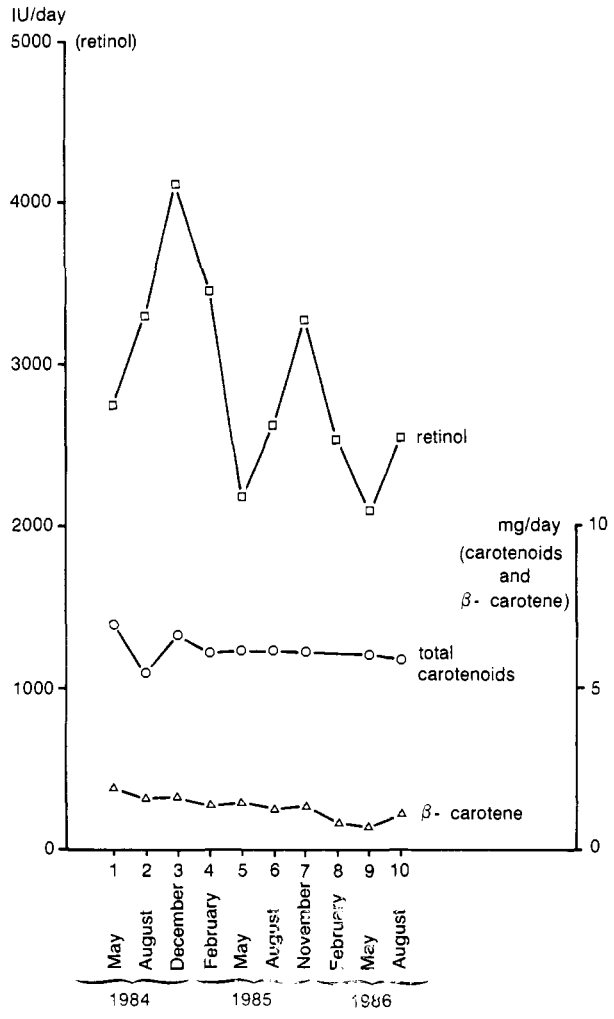


**Figure 6.** Daily amounts of retinol, total carotenoids, and β-carotene in the average diet of 18-year-old males over a period of 2.5 years.

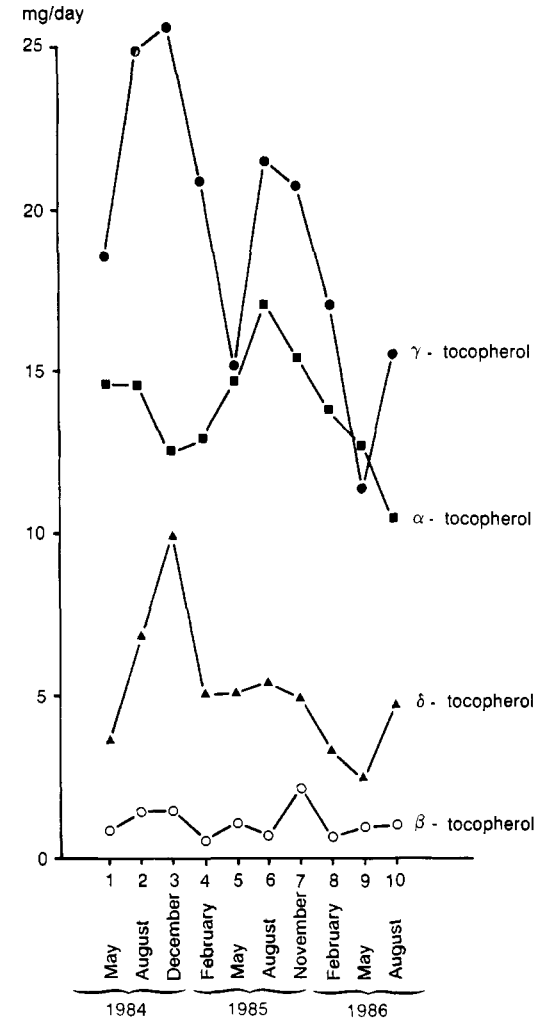


**Figure 7.** Daily amounts of tocopherols in the average diet of 18-year-old males over a period of 2.5 years.

neglected. The high content of total carotenoids in the group leafy vegetables results in a similarly high contribution to the mean daily amount of carotenoids. In spite of a high content of these provitamin A compounds, the group root vegetables does not contribute substantially to the daily amount of carotenoids.

The groups root vegetables, leafy vegetables, and butter, margarine, oils contribute together more than 60% to the total daily amount of β-carotene. The other foo groups are of minor importance for the daily β-carotene supply.

The group butter, margarine, oils is the most important one for the total daily intake of the four tocopherols analyzed (α, β, γ, δ). The contents of these vitamin E active compounds are highest in this food group.

The group meat and meat products contributes 10–17% to the daily amount of tocopherol. The high contribution of the group bread to the daily β-tocopherol supply is remarkable. As the consumption of nuts by the group of male adolescents is limited, the contribution of this group to the total daily amount of the various tocopherols is low in spite of the high contents found.

For most analyses the mean and median values are in good agreement. The range in the contents found is substantial for the group of tocopherols, particularly in the food groups potato products, biscuits, meat and meat products, fish, butter, margarine, oils, and nuts. This may be explained by the use of different types of oils and fats during (industrial) food manufacturing as well as by seasonal influences. However, from Figures 1–5 no clear seasonal influence on the contents of the vitamins in the core food groups could be observed. High concentrations of vitamin A in, e.g. meat/meat products, in the winter season 1984/1985 were not so obvious in 1985/1986. The high concentrations of γ- and δ-tocopherol in the group butter, margarine, oils in December 1984, were not observed in November 1985. A random variation of the vitamin concentrations in time seems therefore more applicable than a seasonal influence.

The Dutch recommended daily intake of vitamin A for male adolescents is 1000 retinol equivalents (RE): 1 RE = 1.00 μg of *all-trans*-retinol, which is 3.33 IU of vitamin A. On the basis of the conversion factors for total carotenoids and β-carotene (see Table III), it can be calculated that the average daily intake of vitamin A is 1481 RE (4937 IU). Approximately 60% (864 RE) originated from *all-trans*-retinol and about 40% from the provitamin A carotenoids. It can be concluded that the recommendation for vitamin A is almost met by the contribution of *all-trans*-retinol alone. The Dutch recommendation for vitamin E for male adults is 1.0 IU/g of polyunsaturated fatty acids (PUFA). Since the total amount of PUFA in the total diet of male adolescents is approximately 16 g/day (to be published), the recommended daily intake of vitamin E is 16 IU. Taking the various conversion factors for the tocopherols into account (see Table III), it can be calculated that the mean daily amount of vitamin E is approximately 22 IU, which means that the recommendation is met. More than 40% of this

daily amount of vitamin E originates from the group butter, margarine, oils, particularly from vegetable oils.

Although seasonality with respect to vitamin concentrations does not seem so obvious, Figures 6 and 7 show that, for most vitamins analyzed, highest daily amounts could be observed in November/December, while the daily intakes were lowest in May. But even when the lowest daily amounts of all vitamins are used to calculate total vitamin A or vitamin E intake, the recommendations for male adolescents are met.

In conclusion, the amounts of both vitamin A (or retinol equivalents) and vitamin E analyzed in total diets of Dutch male adolescents can be regarded as more than sufficient. For vitamin A, the group meat and meat products is the most important one for daily supply, for carotenoids the groups leafy vegetables and root vegetables are the most important sources, and the group butter, margarine, oils is the most important source for the daily vitamin E intake. A group of male adolescents formed the basis for our total diet study. For other age categories, consuming less food, or for groups or individuals with a more extreme dietary pattern, conclusions may diverge from ours. At present, a third total diet study is carried out, in which individual food products are analyzed rather than food groups. This approach will enable us to evaluate the quality of dietary food intake of any population group if their food consumption pattern (or market basket) is known.

**Registry No.** Vitamin A, 68-26-8; β-carotene, 7235-40-7; α-tocopherol, 59-02-9; β-tocopherol, 148-03-8; γ-tocopherol, 7616-22-0; δ-tocopherol, 119-13-1.

LITERATURE CITED

De Ruyter, M. G.; de Leenheer, A. P. Determination of serum retinol levels (vitamin A) by high-speed liquid chromatography. *Clin. Chem.* **1976**, *22*, 1593-1595.

De Vos, R. H.; van Dokkum, W.; Olthof, P. D. A.; Quirijns, J. K.; Muys, T.; van der Poll, J. M. Pesticides and other chemical residues in Dutch total diet samples (June 1976-July 1978). *Food Chem. Toxicol.* **1984**, *22*, 11-21.

EuroNut. Total diet studies in Europe. Report of an EC Workshop, Zeist, The Netherlands, 9-11 Nov 1987; van Dokkum, W., de Vos, R. H., Eds.; EuroNut Report 10; Agricultural University: Wageningen, The Netherlands, 1988.

Speek, A. J.; Schrijver, J.; Schreurs, W. H. P. Vitamin E composition of some seed oils as determined by high-performance liquid chromatography with fluorimetric detection. *J. Food Sci.* **1985**, *50*, 121-124.

Speek, A. J.; Temalilwa, C. R.; Schrijver, J. Determination of β-carotene content and vitamin A activity of vegetables by high-performance liquid chromatography and spectrophotometry. *Food Chem.* **1986**, *19*, 65-74.

Van Dokkum, W.; de Vos, R. H.; Cloughley, F. A.; Hulshof, K. F. A. M.; Dukel, F.; Wijsman, J. A. Food additives and food components in total diets in The Netherlands. *Br. J. Nutr.* **1982**, *48*, 223-231.

World Health Organization. Pesticide Residues in Food. Technical Report Series No. 592; Geneva: WHO, 1976.

World Health Organization. Guidelines for the Study of Dietary Intakes of Chemical Contaminants. WHO Offset Publication No. 87; Geneva: WHO, 1985.

Received for review February 6, 1989. Accepted July 13, 1989.

# Extraction and Analysis of Volatile Compounds in White Wines Using Amberlite XAD-2 Resin and Capillary Gas Chromatography

Charles G. Edwards*,† and Robert B. Beelman‡

Irrigated Agriculture Research and Extension Center, Washington State University, Prosser, Washington 99350, and Department of Food Science, 111 Borland Laboratory, The Pennsylvania State University, University Park, Pennsylvania 16802

A rapid and simplified technique for the analysis of major volatile compounds in wines has been developed by modification of a previously published procedure for beer analysis. Modifications include adjustment of the alcohol content of standards and samples to 10% (v/v) ethanol, use of larger sample size, use of two internal standards, and employment of a newly developed capillary column with a modified poly(ethylene glycol) stationary phase. Relative recoveries of higher alcohols, esters, and medium-chain fatty acids extracted from a white wine ranged from 90 to 114%. Precision, as measured by coefficients of variation, were less than 5% with the exceptions of isobutyl alcohol (22%) and decanoic acid (9%). Analysis of white wines fermented with and without insoluble grape solids and/or yeast ghosts revealed differences in the concentration of higher alcohols, esters, and medium-chain fatty acids in the bottled wines.

Various techniques for the extraction and analysis of volatile compounds in wines have included using extraction solvents such as carbon disulfide or Freon (Snyman, 1977; Nelson and Acree, 1978; Marais and Houtman, 1979) or have used other solvents or procedures (Cobb et al., 1978; Usseglio-Tomasset and Di Stefano, 1981; Simpson and Miller, 1984; Shinohara, 1985; Baumes et al., 1986). Although widely used for analysis of volatiles, these methods often require several hours for extraction and analysis of one wine sample, a major disadvantage to their use. Recently, a technique for analysis of volatile

† Washington State University.
‡ The Pennsylvania State University.

0021-8561/90/1438-0216$02.50/0 