Food Chemistry 36 (1990) 135–148

Headspace of Roasted Ground Coffee as an Indicator of Storage Time

Heikki Kallio,[a] Merja Leino,[a,b] Karla Koullias,[b]
Sinikka Kallio[b] & Jukka Kaitaranta[b]

[a] Department of Chemistry and Biochemistry, Laboratory of Food Chemistry,
University of Turku, SF-20500 Turku, Finland
[b] Oy Gustav Paulig Ab, POB 15, SF-00981 Helsinki, Finland

(Received 17 April 1989; revised version received and accepted 7 June 1989)

ABSTRACT

Volatiles emitted by roasted, ground coffee stored in laminate bags were analysed by a static headspace gas chromatographic–mass spectrometric (GC–MS) procedure. Ratios of several pairs of compounds in the headspace, such as thiophene/butanedione, 2-methylfuran/butanedione, acetone/butanedione, thiophene/propanal, 2-methylfuran/propanal and acetone/propanal increased with storage time. With CO_2 *or air in the bags, the model predicted storage time with linear correlation coefficients of 0·96 and 0·99, respectively. The shares of most of the 38 measured compounds in coffee stored at 37°C for 117 days were not dramatically influenced by the presence of oxygen. Carbon disulphide, dimethyl sulphide and dimethyl disulphide disappeared in bags containing air, probably by oxidation.*

INTRODUCTION

The hundreds of volatile aroma compounds which have sensory threshold values low enough to contribute to the aroma of roasted coffee are responsible for only a fraction of the total flavour. A delicious and balanced volatile aroma is an essential, but not an adequate, prerequisite of first class brewed coffee. The quality of roasted coffee beans relates to changes in the chemical composition of the headspace vapour. Easier than resolving the entire secret of the chemistry of fine coffee aroma is the use of a small number of volatiles as statistical indicators of quality.

Food Chemistry 0308-8146/90/$03·50

The variety and origin of coffee beans, their storage conditions as well as the roasting process, are controlled by the producers and manufacturers. Once delivered to thousands of stores and millions of consumers, a chance to bring about a quality verification no longer exists. Most of the chemical changes which take place after roasting have to be considered as unsatisfactory. The critical factors in preventing rancidity of coffee are the storage temperature, penetration of oxygen and loss of volatiles through diffusion.

Tests with different packing materials and modified atmospheres clearly show the importance of the hermetic sealing of the package (Vitzthum & Werkhoff, 1979; Tressl *et al.*, 1979; Radtke-Granzer & Piringer, 1981; Ito *et al.*, 1983; Liardon *et al.*, 1984). The quality changes due to the physical loss of the volatiles may even exceed those caused by oxidation (Ito *et al.*, 1983; Liardon *et al.*, 1984).

Coffee in a permeable package is especially susceptible to rapid changes (Vitzthum & Werkhoff, 1979; Radtke-Granzer & Piringer, 1981). Vitzthum and Werkhoff (1979) measured the ratios 2-methylfuran/2-butanone (M/B) and methanol/2-methylfuran (M/M) in roasted beans by using headspace analysis. The 'aroma index' M/B decreased in air-permeable packages linearly by about 0·1 unit a day from the initial value of 3·3 during the 10 days after roasting. The ratio M/B varied widely depending on the origin of the coffee beans, roasting level, package material, etc. These, among other things, showed the index M/B to decrease more slowly in beans than in ground coffee.

The decrease of M/B takes place both due to oxidation and loss of 2-methylfuran through diffusion. This was verified when roasted beans were stored in vacuum packages for different periods (Vitzthum & Werkhoff, 1979). The ratio M/B remained constant for at least 7 months with beans but, after grinding, the index M/B decreased rapidly. Evaporation at the surface caused a quicker drop of M/B than deeper layers in ground, unpacked coffee. On the other hand, the ratio M/M, increased in air-permeable bags during 12 weeks' storage.

When stored 10 days in conventional packages allowing penetrating air, the total content of the volatile compounds decreased in the headspace of the roasted beans by a factor 0·5. The contents of butanedione, propanal, 2-ethylfuran, 2,5-dimethylfuran, thiophene, methyl formate and methyl acetate decreased by more than 80% (Vitzthum & Werkhoff, 1979). In 2 weeks at room temperature two-thirds of the sulphur-containing aroma compounds were lost while methylthioacetate, thiophene, thiophene-3-aldehyde and 3-methylthiophene decreased by over 80% (Vitzthum & Werkhoff, 1979). The loss of these compounds in frozen beans was only 1%.

Radtke-Granzer and Piringer (1981) observed the aroma changes of roasted coffee beans in conventional packages where oxygen exchange occurred. They analyzed the alteration of the contents of 2-methylpropanal, 3-methylbutanal, butanedione, 2-methylfuran and carbon disulphide at room temperature by headspace GC–MS analysis. The total content of the non-sulphur compounds mentioned above decreased by 50% during 74 days' storage and that of CS_2 by 75%. Increasing the storage temperature speeded up the loss of the compounds. Tressl *et al.* (1979) also showed a rapid decrease in 2-pentadecanone during 3 weeks' storage of beans.

Ground coffee stales five to ten times faster than beans (Vitzthum & Werkhoff, 1979). The amounts and ratios of volatile compounds of ground coffee change more rapidly than those of the beans even in hermetically sealed bags. However, the composition of the gas atmosphere (air, reduced oxygen content or nitrogen) seems to have a smaller effect than would be expected when escape of the volatile compounds is prevented (Ito *et al.*, 1983; Liardon *et al.*, 1984). Storage temperature is always of primary importance for the keeping quality.

Liardon *et al.* (1984) showed, by using multivariate analyses, gradual changes in volatiles of ground coffee stored in capsules containing air or reduced-oxygen atmosphere. The effect of varying oxygen content on the chemical changes was much smaller than that of the storage time (0 to 27 weeks at 20°C). Unfortunately, the structures of the coded aroma compounds were not listed in the paper.

In ground coffee stored in an evacuated package, the content of 2-methylfuran decreased very slowly, whereas that of butanedione dropped faster (Piringer, 1983). Their ratio was shown to be linear versus log time but, of course, dependent on the origin and the lot of the coffee. Radtke-Granzer and Piringer (1981) analysed the contents of 2-methylpropanal, 3-methylbutanal, butanedione and 2-methylfuran in ground coffee packed in reduced oxygen (partial pressure 0·5–3·0%) or nitrogen atmosphere at 23°C for 2 years. The total content of the four compounds decreased below 89 mg/kg, which was stated to be the lower limit for good quality coffee. The content of 2-methylfuran did not decrease at all when the content of oxygen was reduced.

As shown in the previous examples, the permeability of the packing material is of primary importance. This was also verified by analysing vacuum packed ground coffee, which kept the content of 4-vinylguajacol constant over a year, whereas in permeable material the content decreased, due to diffusion or oxidation, over 1 month from 24 to 11 mg/kg (Tressl *et al.*, 1979).

The amounts of several strongly flavoured, heterocyclic, pyrolysis-induced compounds such as 1-furfuryl-2-methylpyrrole, furfuryl mercaptan, 5-methylfurfuryl mercaptan as well as 4-vinylguaiacol were found by Tressl *et al.* (1979) to increase in stored roasted coffee beans. The rise of mercaptans had a lag-phase of 1 to 7 days. 5-Methylfurfuryl mercaptan, in particular, was reported to be responsible for the staling of coffee.

The aim of this study was to verify the changes of some volatile compounds in roasted ground coffee during storage, both under CO_2- and air-headspace in gas-impermeable packages. The relative contents of the compounds were measured with a static headspace GC–MS procedure and a model indicating that the storage time was based on the changes between the ratios of various compounds.

MATERIAL AND METHODS

Coffee samples

The coffee samples were of a filter ground, commercial brand 'Juhlamokka' processed by Oy Gustav Paulig Ab (Helsinki, Finland) from a blend of selected Arabica coffee qualities. Coffee, 125 g, was packed in 370 ml polyester–Al–polyethylene laminate bags filled with either air or flushed with CO_2 (headspace about 70 ml) and stored at 37°C for 0 to 4 months (accelerated staling process). In addition, reference samples of the coffee bags were kept at −18°C for synchronous analyses.

Samples (air and CO_2 at +37°C and −18°C) were prepared and analysed eleven times over the 4-month test period. Thirty-five grams of ground coffee mixed in a breaker was weighed into a 500 ml Erlenmeyer bottle and closed with a headspace valve (J & W Scientific, Folsom, CA) as stated earlier (Kallio *et al.*, 1987). All the samples were prepared in duplicates. Equilibration of the headspace took place at 37°C for 19 h. The temperature of the reaction chamber was thus the same as that of the storage of the coffee bags.

GC and GC–MS analysis

The headspace volatiles were analysed on a Varian Aerograph 3700 gas chromatograph equipped with an FI-detector (Walnut Creek, CA) and a Hewlett–Packard 3388A integrator (Palo Alto, CA). The fused silica open tubular column (DB-1701, 30 m × 0·254 mm i.d., d_f 0·25 μm, J & W Scientific) was connected to the on-column injector which was kept at room temperature. The detector temperature was maintained at 250°C and the inlet pressure of the He-carrier at 90 kPa, which gave the carrier gas velocity 27 cm/s at 20°C.

Before the injection procedure the He-pressure was lowered to 60 kPa, the oven chilled with solid CO_2 to +10°C and a loop of the front end of the column placed in liquid N_2 (Kallio *et al.*, 1987). The coffee headspace sample (2·0 ml) was injected at the speed 0·5 ml/min onto a capillary column from a 10 ml gas tight Hamilton syringe (Hamilton, Reno, NV). The pressure was adjusted to 90 kPa and, after 3 minutes' stabilization, the cold trap was removed and the oven temperature programming, 10–100°C at 2°C/min, was started. Two identical bags of coffee were analysed each time in parallel.

The Kovats' indices (I_K) of the volatiles were determined with co-injection of a vapour mixture of *n*-alkanes (C_5–C_{12}). Comparison of the authentic compounds to the volatiles identified by EI–MS was made using the same co-injection manner.

The electron impact mass spectral (EI–MS) analyses (ionization energy 70 eV) were carried out on a VG 7070E spectrometer employed with the VG-11-250 data system and a Dani 3800 HR 2 ch gas chromatograph (VG, Wythenshawe, Manchester, UK). In addition to the DB-1701 column and program used in the GC analysis, the MS analyses were also performed with a DB-Wax column (30 m × 0·253 mm i.d., d_f 0·24 μm, J & W Scientific). The temperature programming in the latter case was carried through from 30 to 200°C at a rate of 2°C/min after 10 min at 30°C.

Quantitative measurements

The total content of the volatiles in each coffee sample was defined by integrating the arbitrary peak areas from t_0 to the compound number 38 (5-methyl-2-furaldehyde) of a chromatogram. No internal standards were used. The relative percentages of individual compounds were calculated from the total contents of volatiles on the chromatograms.

RESULTS AND DISCUSSION

The qualitative analyses with two liquid phases having different polarities and selectivities were necessary to verify the structures of some compound pairs having relative retentions (alpha-values) close to one. The primary stationary phase used in this work was DB-1701 and the DB-Wax phase was used for verification.

A coffee headspace chromatogram analysed with the DB-1701 column is presented in Fig. 1. The compounds numbered from 1 to 38 were identified according to their mass spectra and Kovats' indices (I_K). The GC profiles in Fig. 1 differs clearly from several other coffee headspace chromatograms published earlier (Arackal & Lehman, 1979; Vitzthum & Werkhoff, 1979; Ott & Liardon, 1981). This is partly due to the coffee and partly due to the

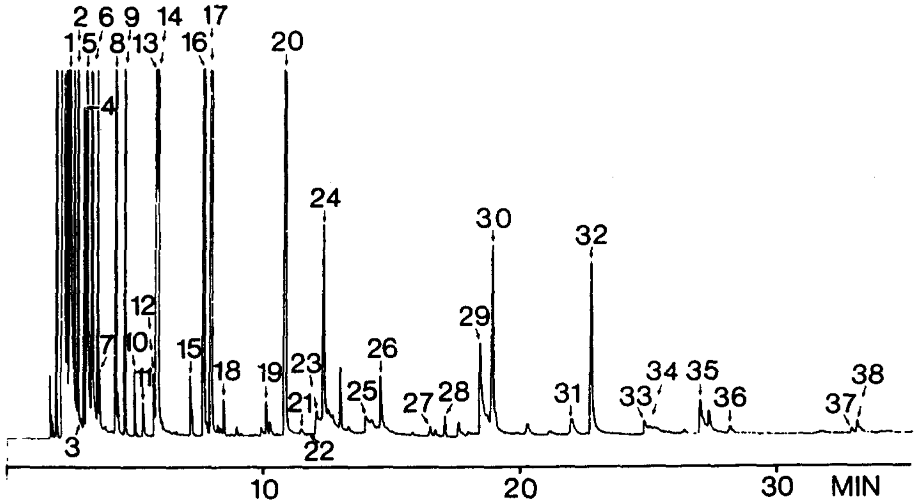

Fig. 1. A headspace chromatogram of roasted ground coffee packed in a gas tight bag containing air atmosphere, analysed one day after packing using a DB-1701 capillary column.

sampling technique. We did not use any Tenax or other concentration devices prior to the on-column cold trapping. This verified the transfer of the volatiles from the syringe into the column as near 100% as possible. If the responses of any of the minor compounds at the upper part of the chromatogram have to be intensified for quantitative purposes, the volume injected can be increased.

For the sampling process, a rather long incubation period in special reaction chambers was used instead of using the storage bags. The aim of this procedure was to verify identical conditions for each sample and to use the ground, aged coffee *per se* as the material to be studied. Comparison of the coffee stored in CO_2- and air bags showed that the composition of the samples in the hermetically sealed reaction chambers did not change too much during the 19 h equilibration period.

The trends of the changes of the volatile compounds analysed by the DB-1701 phase are presented in Table 1. The I_K-values of the compounds the range of the percentile shares of the volatiles (coffee stored at +37°C for 117 days in air or CO_2), the correlation coefficients between the measurements and the linear regression lines (r), and the changes of the relative contents are listed. The percentage shares and the absolute contents, i.e. partial pressures of the individual compounds in the headspace bottles, varied over a wide range depending on the age of the coffee. The digits were not corrected according to the different FI-detection responses of the compounds. The standard deviation of the GC analysis of the minor peaks is higher than that of the main compounds. A headspace analysis is always a compromise between overloading the column with the major compounds and the low quantitative accuracy of the minor compounds with low partial vapour pressures above the sample, as is the case in any chromatographic analysis.

A typical analogy of the trends in both packing lines can be seen in Table 1. When the share of a compound was rising in CO_2-packed coffee, it also rose in air-containing bags. The same could also be recognized among the diminishing volatiles. Thus, most of the relative changes seen during the testing period did not primarily take place due to oxidative reactions. The clearest exceptions to this rule were the behaviour of dimethyl sulphide, carbon disulphide and dimethyl disulphide. Carbon disulphide gives an extremely low response in FID which resulted in a very small chromatographic peak and low correlation coefficients shown in Table 1. In the CO_2-packed coffee, dimethyl sulphide increased 17% during the 117 days with the linear correlation coefficient (r) 0·74, and in the air bags, vice versa, declined 119% ($r = -0{\cdot}80$). Dimethyl disulphide almost doubled its share in the CO_2 samples ($r = 0{\cdot}64$) but remained constant in air packages. The results concerning the surprising similarity between the air- and CO_2-bags are limited, however, to only the compounds listed in Table 1 and are not general in the aroma of roasted coffee.

A practical test for the determination of the age of coffee has to be reliable and as simple as possible. From the analytical point of view this means that we chose, for the test, only a few compounds which were easy to separate in a short time and had a sufficiently regular change. To fulfil these requirements in our statistical model we applied linear regression and used the linear correlation coefficient (r) as a criterion for the selection of the compounds. In accordance with the above, we chose three compounds having the highest positive correlation coefficients (Table 1) in both atmospheres (thiophene, 2-methylfuran and acetone) and two compounds with the highest negative r-values butanedione and propanal). The abbreviations of the percentile shares of the five compounds are: thiophene = T, acetone = A, 2-methylfuran = M, butanedione = B and propanal = P. In addition to the high linear correlations, the percentage increase or decrease of each was close to the other in both CO_2 and air bags. The compounds and the regression model were not designed to predict the sensory quality of the coffee headspace. They were chosen only to explore factors indicating the storage time of the coffee.

The five compounds chosen were not the only ones of potential importance, but the model was made simple to use. Some additional alternatives could have been, e.g., 2-methylpropanal, 2-methylbutanal, 2-butanone, 3-hydroxy-2-butanone, acetic acid, furan, 3-methylfuran, 2,5-

TABLE 1

Trends of the Relative Contents of the Volatiles in the Headspace of Hermetically Sealed Ground Coffee followed for 117 Days at 37°C

Compound			*CO_2 atmosphere at 37°C*			*Air atmosphere at 37°C*		
DB-1701 phase		*IUPAC-name*	*Range, % of the total area*	*r^b*	*Change, % of the initial value*	*Range, % of the total area*	*r^b*	*Change % of the initial value*
No.	*I_K*							
1	516	methanol	9·4–14·9	0·63	+27	9·1–13·5	0·83	+33
2	536	furan	1·5–2·6	0·78	+41	1·8–2·6	0·81	+29
3	575	carbon disulphide	0^a–0·4	0·13	+9	0^a–0·1	−0·19	−21
4	580	dimethyl sulphide	0·7–0·9	0·74	+17	0·0–0·7	−0·80	−119
5	585	propanal	1·6–3·0	−0·78	−38	1·8–3·3	−0·91	−38
6	592	acetone	9·8–13·3	0·89	+32	9·9–12·7	0·96	+27
7	602	methyl acetate	0·2–0·5	0·87	+103	0·2–0·3	0·51	+33
8	638	2-methylpropanal	9·2–11·9	0·81	+21	9·3–12·0	0·78	+19
9	646	2-methylfuran	3·3–5·0	0·95	+42	3·3–4·9	0·91	+37
10	666	3-methylfuran	0·2–0·3	0·96	+57	0·2–0·3	0·82	+35
11	672	*n*-butanal	0·1–0·2	0·23	+15	0·1–0·2	−0·34	−20
12	679	ethyl acetate	0·3–0·6	0·56	+44	0·2–0·3	0·70	+36
13	691	2-butanone	1·3–2·0	0·84	+35	1·3–2·0	0·78	+39
14	693	butanedione	3·7–5·3	−0·92	−31	3·5–5·3	−0·89	−33
15	722	thiophene	0·2–0·4	0·91	+81	0·2–0·4	0·98	+84
16	729	3-methylbutanal	3·2–5·5	0·50	+22	4·8–5·9	0·63	+13

17	742	2-methylbutanal	8·0–11·7	0·93	+36	7·8–11·2	0·86	+32
18	748	2,5-dimethylfuran	0·1–0·2	0·93	+48	0·1–0·2	0·84	+43
19	780	phenol	0·1–0·2	0·80	+40	0·1–0·2	0·78	+34
20	793	2,3-pentanedione	4·7–6·0	0·17	+4	4·4–5·0	0·00	0
21	798	dimethyl disulphide	0·0–0·1	0·64	+82	0·0–0·1	0·03	−4
22	814	toluene	0·1–0·6	−0·39	−88	0·0–0·3	−0·04	−10
23	826	acetic acid	0^a–1·5	−0·62	−98	0^a–1·2	−0·62	−122
24	830	1-methylpyrrole	0·3–0·6	0·19	+15	0·3–0·4	0·32	+12
25	844	pyridine	0·0–0·2	−0·48	−55	0·0–0·3	−0·20	−36
26	855	3-hydroxy-2-butanone	0·1–0·4	−0·75	−64	0·2–0·4	−0·75	−47
27	872	2,4-dimethyl-3-pentanone	0·0–0·1	0·26	+20	0·1–0·1	0·82	+48
28	881	2-hydroxyphenol	0·1–0·2	0·59	+34	0·1–0·2	0·63	+24
29	895	2-methylpyrazine	0·4–1·5	−0·57	−46	0·4–1·1	−0·56	−39
30	904	dihydro-2-methyl-3(2)-furanone	1·0–2·0	0·09	+5	1·2–1·6	0·23	+7
31	949	2,5-furandione	0^a–0·2	−0·70	−119	0^a–0·2	−0·69	−150
32	960	2-furaldehyde	0·7–1·4	−0·64	−32	0·9–1·3	−0·81	−25
33	986	2,5-dimethylpyrazine	0·0–0·2	−0·14	−29	0·1–0·1	0·18	+9
34	987	2,6-dimethylpyrazine	0·0–0·2	−0·16	−27	0·0–0·1	−0·06	−8
35	1 014	2-furanmethanol	0^a–0·5	−0·21	−49	0·0–0·4	−0·63	−84
36	1 029	2-acetylfuran	0·0–0·1	0·18	+19	0·0–0·1	0·32	+18
37	1 090	furfuryl acetate	0·0–0·1	0·30	+29	0^a–0·1	0·19	+11
38	1 099	5-methyl-2-furaldehyde	0·0–0·2	0·31	+49	0^a–0·2	−0·38	−18

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[a] Below the detection threshold limit.

[b] Correlation coefficient for $y = a + bx$, where y is the percentile share of a compound and x the actual age.

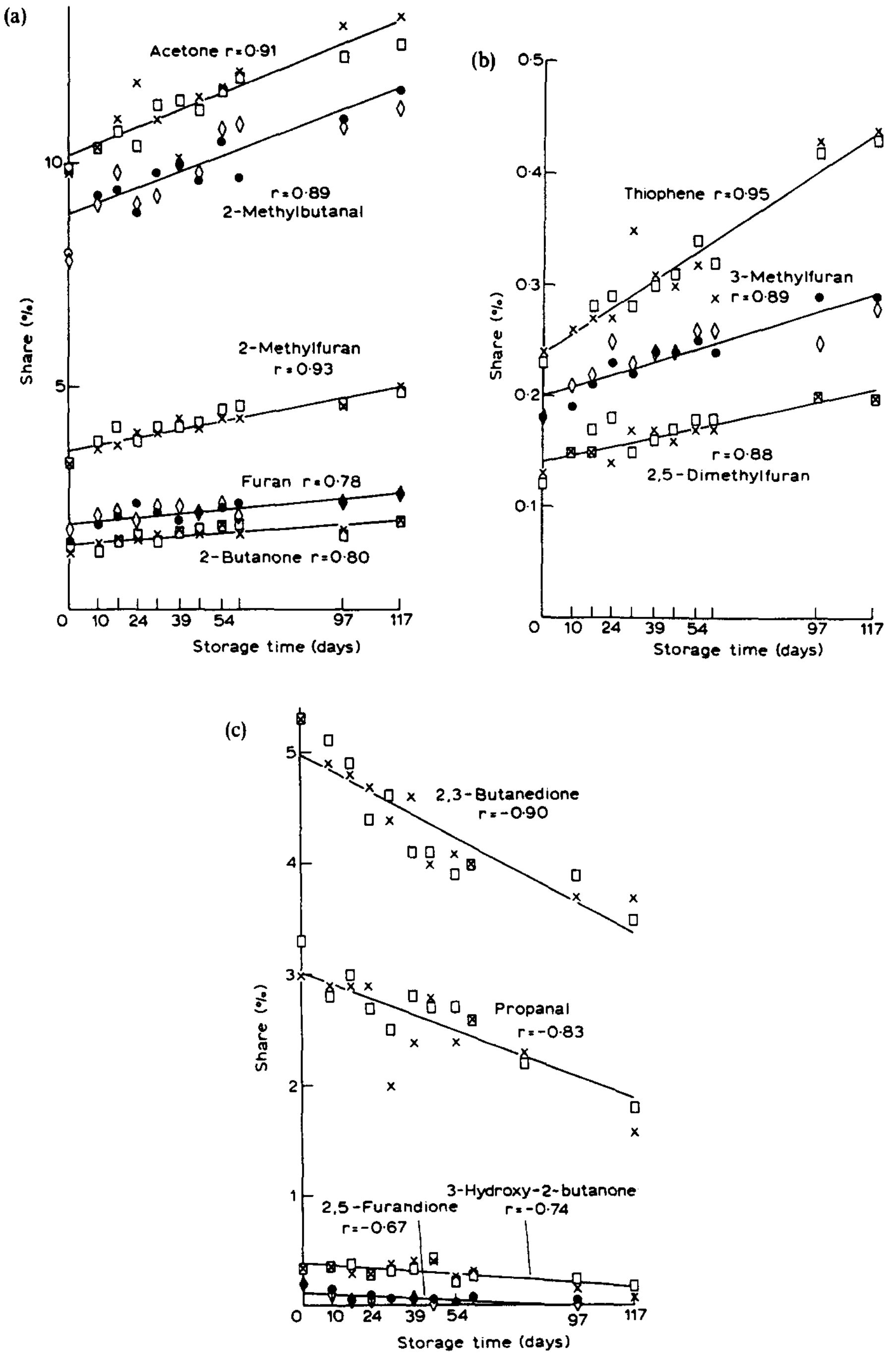

Fig. 2. The changes of percentile shares of some of the coffee headspace volatiles stored and analysed at 37°C. (□) and (◇) air atmosphere, (●) and (×) CO_2 atmosphere. *r*-Values are the linear correlation coefficients of the percentile shares versus actual age.

TABLE 2
Definitions of the Age Determination of Packed Coffee

*Storage time indicator** y	$y = a + bx$†	
	a	b
T/B	0·048	0·000 69
M/B	0·724	0·006 51
A/B	2·047	0·015 90
T/P	0·079	0·001 31
M/P	1·191	0·012 55
A/P	3·370	0·031 01

* Ratios of percentile shares of the compounds in the headspace of ground coffee; T = thiophene, M = 2-methylfuran, A = acetone, B = 2,3-butanedione, P = propanal.
† Actual storage time in days.

dimethylfuran, 2,5-furandione, 2-furancarboxaldehyde. The change of the relative contents of some of the indicator compounds in the coffee samples and the linear regressions are shown in Figs 2(a)–(c). Each dot is the mean of two identical coffee bags. There does not seem to be any apparent reason to use other than linear correlations in the simplified correlation calculations.

To make the test as independent of the amount of coffee and the test conditions as possible, we used in our model the ratios of the shares of the compounds (T/B, A/B...) instead of the individual %-values (T, B...). The definitions of the age of the coffee were the linear correlations T/B, A/B... versus actual age, i.e. the six regression lines $y = a + bx$ where y is the ratio and x the age. The parameters a and b calculated from our GC measurements are shown in Table 2.

To eliminate the effect of the possible occasional variation in one of the indicator compounds, the age of the coffee in a bag was specified as the mean of the six age determinations. The reliability of this method (repeatability of the analytical procedure) was tested with the total analysis material of the samples stored at 37°C and is shown in Table 3 and Fig. 3. The correlation coefficient of calculated age versus actual age was, in the case of CO_2 atmosphere, 0·96 and air atmosphere, 0·99.

The simple model presented above was not adequate when trying to distinguish between the packages of two atmospheres. Furthermore, the trends of carbon disulphide, dimethyl sulphide and dimethyl disulphide did not seem to be consistent enough for age verification. The multivariate analysis of the complete data is in progress. Liardon *et al.* (1984)

TABLE 3

Reliability Test of the Storage Time Determinations. Linear Correlation Equations of the Calculated Storage Times (definitions in Table 2) versus Actual Storage Times. (The test was carried out with the same analysis material of ground coffee which was used for the age definitions)

Storage time indicator	$y^* = a + bx\dagger$		
	a	*b*	*r*
CO_2 bags			
T/B	−4·96	0·98	0·96
M/B	−4·98	0·92	0·98
A/B	−1·54	0·94	0·98
T/P	−3·89	1·04	0·85
M/P	−7·04	1·08	0·87
A/P	−6·04	1·11	0·87
mean of all above	−4·71	1·01	0·96
air bags			
T/B	−3·84	0·95	0·99
M/B	−0·54	0·91	0·95
A/B	−2·15	0·89	0·97
T/P	−9·44	0·99	0·97
M/P	−7·51	0·96	0·96
A/P	−9·33	0·95	0·95
mean of all above	−5·46	0·94	0·99

* Storage time in days, calculated according to the definitions in Table 2.

† Actual storage time in days.

unfortunately published the compounds in their multivariate analysis only as code numbers, which makes accurate comparisons with our data impossible.

Our results and method cannot be compared with those of Vitzthum and Werkhoff (1979) or Piringer (1983) as their packages were gas-permeable. The exchange of air and escape of the volatiles through the wall material were not prevented. For example, in our experiments the ratios 2-methylfuran/2-butanone and methanol/2-methylfuran could not be used as indicators at all (Table 1).

The test conditions in the work of Radtke-Granzer and Piringer (1981) resembled those used in this work but for some reason the trends of the compounds they used as age indicators (2-methylpropanal, 3-methylbutanal, butanedione and 2-methylfuran) behaved differently.

The higher-boiling, good staling indicators reported by Tressl *et al.* (1979), 4-vinylguajacol, 1-furfuryl-2-methylpyrrole, furfurylmercaptan and

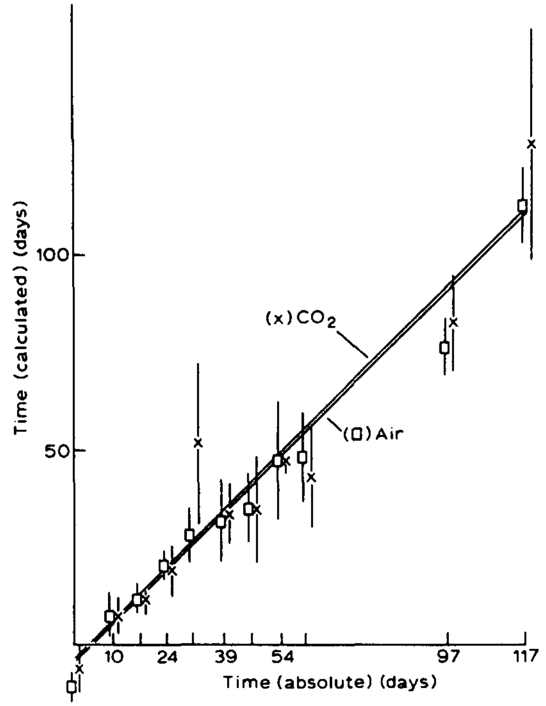

Fig. 3. Validity of the storage time determinations of ground coffee stored at 37°C. Standard deviations shown in bars.

5-methylfurfurylmercaptan, were not found at all in our work because of their low partial vapour pressures under the experimental conditions.

The determination of the age of coffee by means of the equations in Table 2 is valid only after calibration with the coffee and conditions in which it is studied. This concerns any method which is based on the contents and the changes of coffee volatiles. Without knowing the temperature history of the product an age determination does not make sense and is without any credibility. For comparison, the experiments were also carried out at −18°C, 50°C lower than the temperature of the accelerated ageing test. The cold storage did not give any basis for the temperature correction factors for the equations in Table 2. The changes at −18°C were so slow that the correlation coefficients of the equations percentile share versus time were very low. This also indicates that a coffee bag stored in a freezer can be used, if needed, as an unaltered reference for staling studies.

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