

Characterization of Artisanal Cheeses by GC and GC/MS Analysis of Their Medium Volatility (SDE) Fraction

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Volatile compounds from six artisanal cheese varieties have been isolated by a micro-SDE (simultaneous distillation-extraction) procedure. The composition of the fractions has been analyzed by GC and GC/MS. About 70 components have been identified, and 46 have been quantified in the 17 samples studied. The composition depends mainly on cheese type and ripening stage; methyl ketones and free fatty acids are the most abundant components in blue varieties. Composition data obtained by this method seem to be useful for the characterization of cheeses.

INTRODUCTION

The study of the volatile components of cheeses is an interesting field since cheese aroma depends on their concentration. Several reviews summarize the present knowledge of volatile formation (Adda, 1984), profiling in different cheese types (Badings, 1984) and analytical techniques for their study (Reineccius and Anandaraman, 1984). The volatile chromatographic pattern has been used in the classification of some cheese varieties (Aishima and Nakai, 1987).

Artisanal cheeses from the Asturias region (a mountainous area in northern Spain) are highly prized products with intense flavors. Although two varieties, Cabrales (Alonso et al., 1987) and Gamonedo (Gonzalez de Llano et al., 1987), have been investigated, no data have been published on the composition of their volatile fractions.

The micro-SDE apparatus, proposed by Godefroot et al. (1981), is a simple and reliable method that affords quantitative results for a wide range of volatile compounds of cheese, with satisfactory reproducibility (Frutos et al., 1988).

As part of a project on the study of Spanish cheeses, the volatile fraction of different artisanal cheeses has been obtained by the SDE technique and their qualitative and quantitative composition has been determined by GC and GC/MS. Our main objective was the characterization of the artisanal cheeses studied from the composition of their volatile fraction: these data will be used in further studies on sensorial quality and ripening processes.

MATERIALS AND METHODS

Seventeen samples from six different cheese varieties were purchased at the local markets of Asturias. For each variety, the samples collected can be considered typical specimens; samples at different ripening stages were chosen when possible. Their characteristic features are summarized in Table I.

A micro simultaneous distillation-extraction (SDE) apparatus (Chrompack) similar to that described by Godefroot et al. (1981) was used with pentane as solvent. *n*-Nonadecane was chosen as internal standard, as previously described (Frutos et al., 1988). All reagents were of analytical grade.

Separation was carried out by capillary GC, using two columns of different polarity, which are described in Table II, in a Hewlett-Packard HP-5890 gas chromatograph with a Spectra-Physics 4270 integrator.

Quantitative data were obtained with the SE-30 column (number 1 in Table II).

Identification of components was achieved by comparing GC retention times (in one or both columns) with those of standard compounds and by GC/MS. Mass spectra (EI mode, 70 eV)

were recorded with a Konik 2000 gas chromatograph coupled to a VG 12-250 mass spectrometer, using the two mentioned columns.

Calculations were carried out in a Cyber 855 using the ARTHUR program package (Duewer).

RESULTS AND DISCUSSION

Characteristic chromatograms of every cheese type are shown in Figures 1-6. Figures 1-4 correspond to those obtained with the SE-30 column for A-D varieties. The chromatograms obtained with the SP-1000 column for E and F varieties are in Figures 5 and 6. The components identified by GC and GC/MS are listed in Tables III and IV. The concentration range for the six varieties studied, calculated from the chromatograms obtained with the SE-30 column, is shown in Table III.

The fractions corresponding to mold-ripened varieties (A-C in Table I) were richer in components than those corresponding to the other three types; the main peaks were assigned to methyl ketones and free fatty acids (FFA).

Total FFA content was higher in samples of the blue varieties. Octanoic and decanoic acids were, in general, the more abundant FFA of the SDE extracts in the samples examined. Other FFA of shorter (butanoic and hexanoic) and longer (hexadecanoic and octadecanoic) chain length were detected but not quantified. Relative proportions of FFA were different from those found by other methods; short-chain acids (up to pentanoic) could remain preferentially dissolved in the aqueous phase, while the low volatility of high-chain acids (tetradecanoic and longer) would discriminate against their presence in the volatile fraction. Discrimination against several components caused by their volatility or chemical characteristics is a problem common to all fractionation methods (Belitz and Grosch, 1986).

Odd carbon number methyl ketones from 7 to 17 carbon atoms were present in all samples. These compounds are considered an important part of the aroma of blue cheeses (Dartey and Kinsella, 1971). The highest concentration corresponded to 2-heptanone and 2-nonanone (the last one reaches 80.5 $\mu\text{g/g}$ in sample 1 of the Gamonedo variety); these are also the most abundant in other blue cheese types (Adda, 1984). 2-Pentanone was detected in some samples. We have not found previous references to 2-heptadecanone in cheeses. Since methyl ketones are formed by β -oxidation and decarboxylation of FFA, the origin of 2-heptadecanone must be octadecanoic acid, which was found in free form in our samples and also in many other types of cheese (Woo and Lindsay, 1983; Alonso et al.,

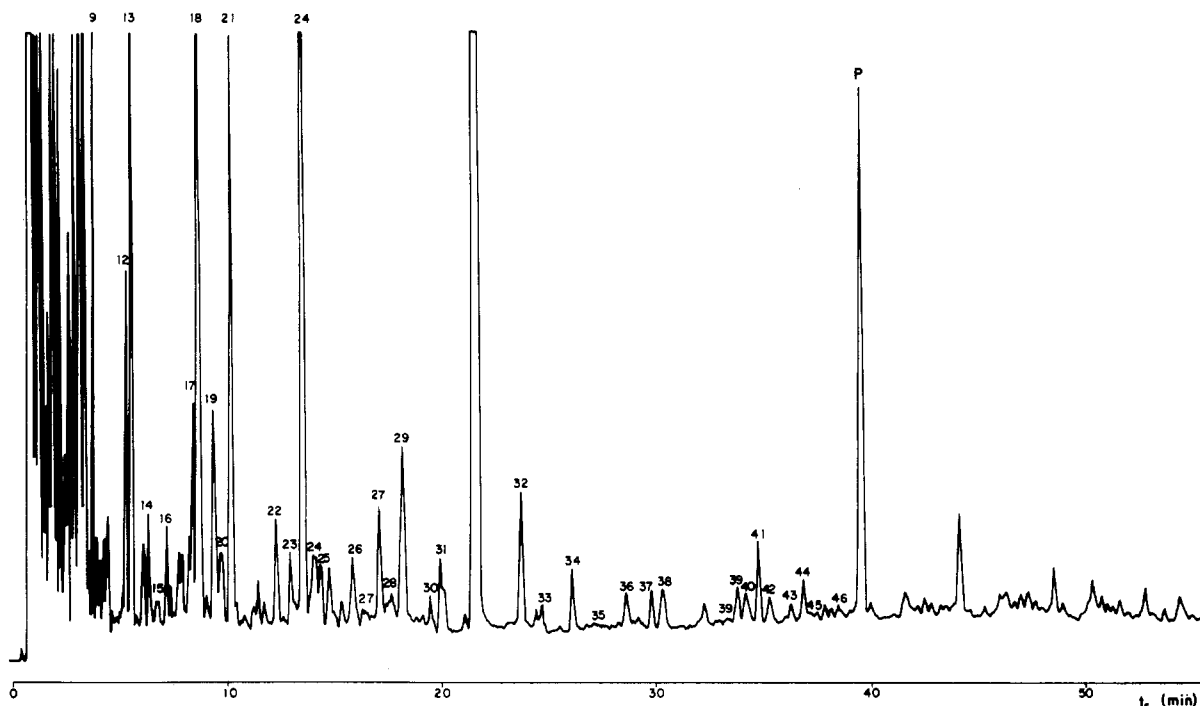


Figure 1. Capillary gas chromatogram of an SDE extract from a Gamonedo cheese. Temperature was programmed from 60 to 250 °C at 2 °C/min on a 22 m × 0.3 mm (i.d.) SE-30 column. The peak numbers correspond to the numbers of Table III; P corresponds to *n*-nonadecane (internal standard).

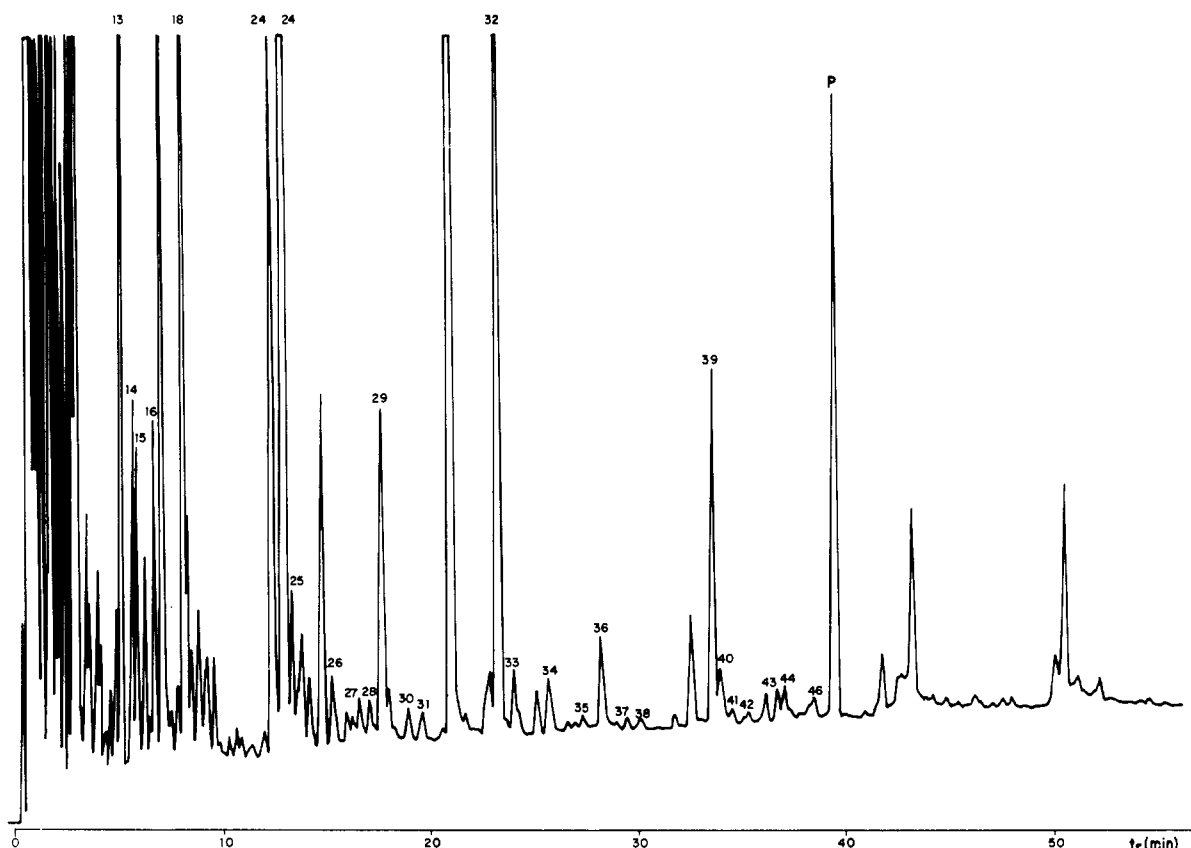


Figure 2. Capillary gas chromatogram of an SDE extract from a Cabrales cheese. Temperature was programmed from 60 to 250 °C at 2 °C/min on a 22 m × 0.3 mm (i.d.) SE-30 column. The peak numbers correspond to the numbers in Table III; P corresponds to *n*-nonadecane (internal standard).

1987). A series of methyl ketones with an even number of carbons from 8 to 16 (mainly 2-decanone) were detected in the three types of blue cheeses. Nonen-2-one was found in these types of cheeses. It has also been reported in some French cheeses (Dumont and Adda, 1978; Gallois and Langlois, 1990). 2-Alkanols (heptanol and nonanol),

2-methylpentanol, and 2-nonenol were also present; they have been described as related with methyl ketones formed by *Penicillium* (King and Clegg, 1979).

δ -Lactones have been found in most samples. They were difficult to measure in variety A, since many interfering components were present. Mass spectrometry has shown

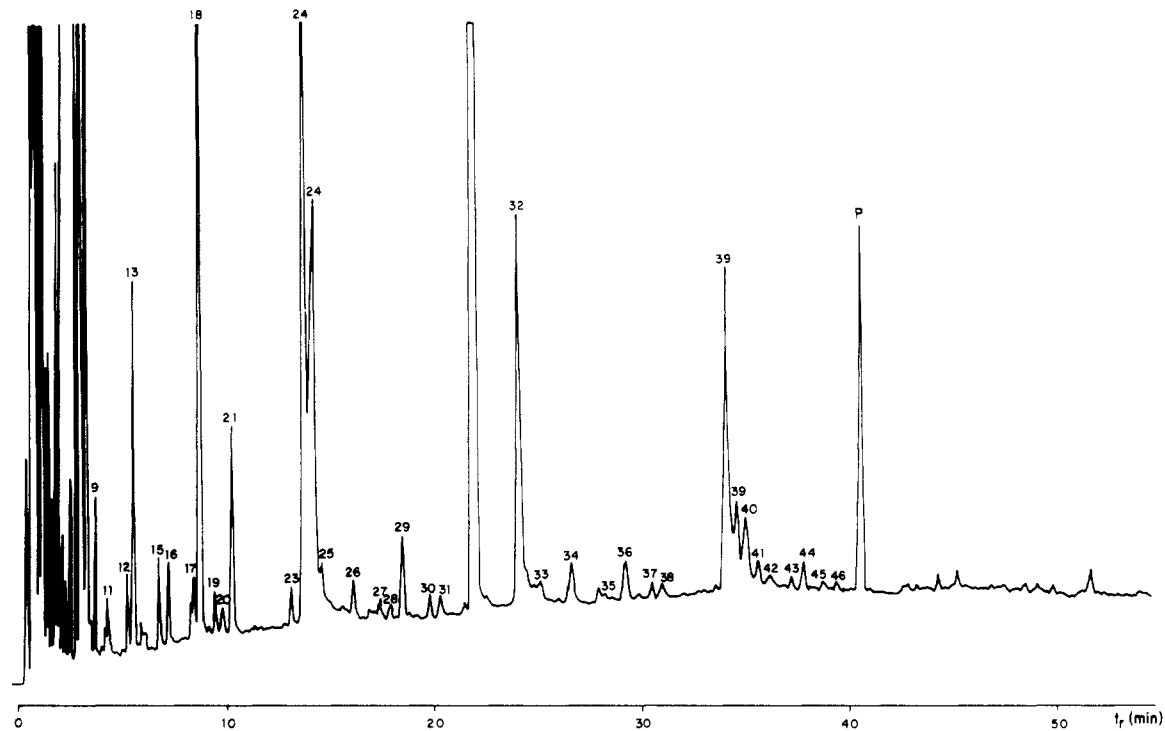


Figure 3. Capillary gas chromatogram of an SDE extract from a La Peral cheese. Temperature was programmed from 60 to 250 °C at 2 °C/min on a 22 m × 0.3 mm (i.d.) SE-30 column. The peak numbers correspond to the numbers of Table III; P corresponds to *n*-nonadecane (internal standard).

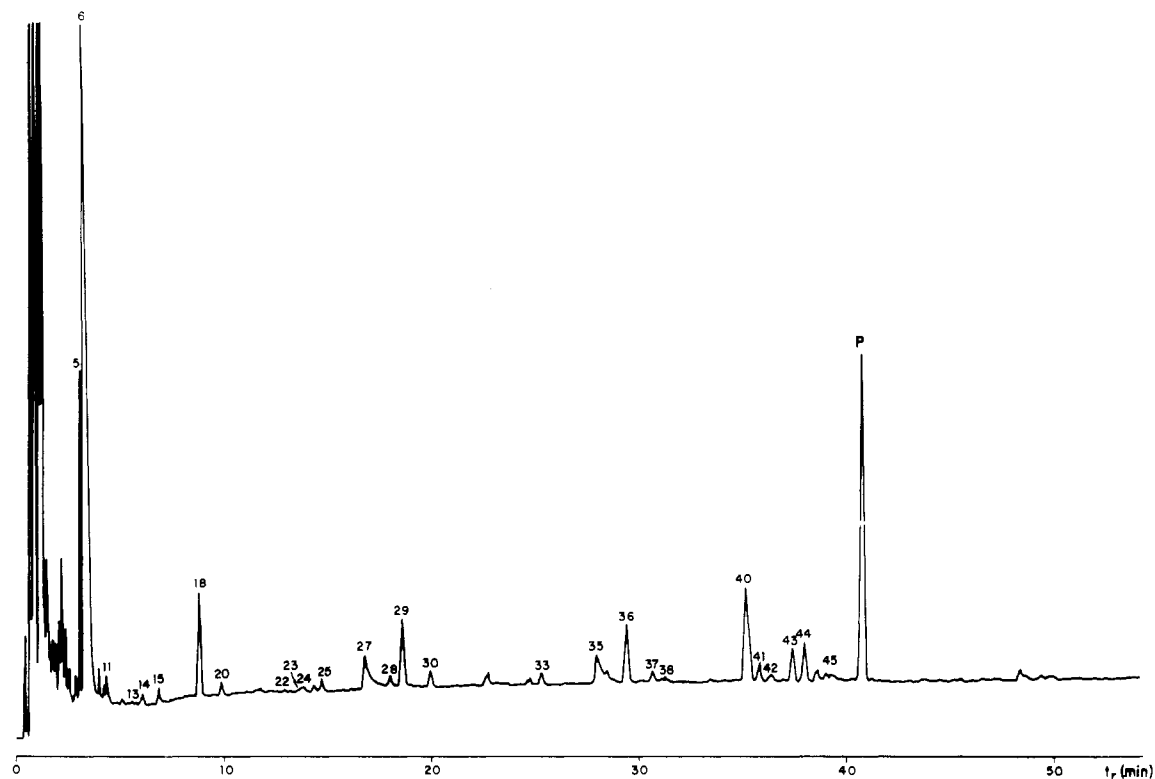


Figure 4. Capillary gas chromatogram of an SDE extract from a Peñamellera cheese. Temperature was programmed from 60 to 250 °C at 2 °C/min on a 22 m × 0.3 mm (i.d.) SE-30 column. The peak numbers correspond to the numbers in Table III; P corresponds to *n*-nonadecane (internal standard).

the presence of γ -decalactone in some samples. The presence of lactones has been correlated with the age and aroma of Cheddar (Wong et al., 1973).

Even carbon number ethyl esters from butanoate to hexadecanoate were present in most of the varieties studied. The higher levels were found in the blue cheeses; their quantitation was difficult since several acids and

ethyl esters coeluted in the SE-30 column. Other ethyl esters, unsaturated with 14, 16, and 18 carbons and branched with 17 and 18 carbons, have also been found, mainly in variety B. Methyl esters from hexanoate to tetradecanoate were found. Their concentration was maximum in blue cheeses and minimum in Peñamellera cheese (variety D). 2-Phenylethyl esters (acetate, pro-

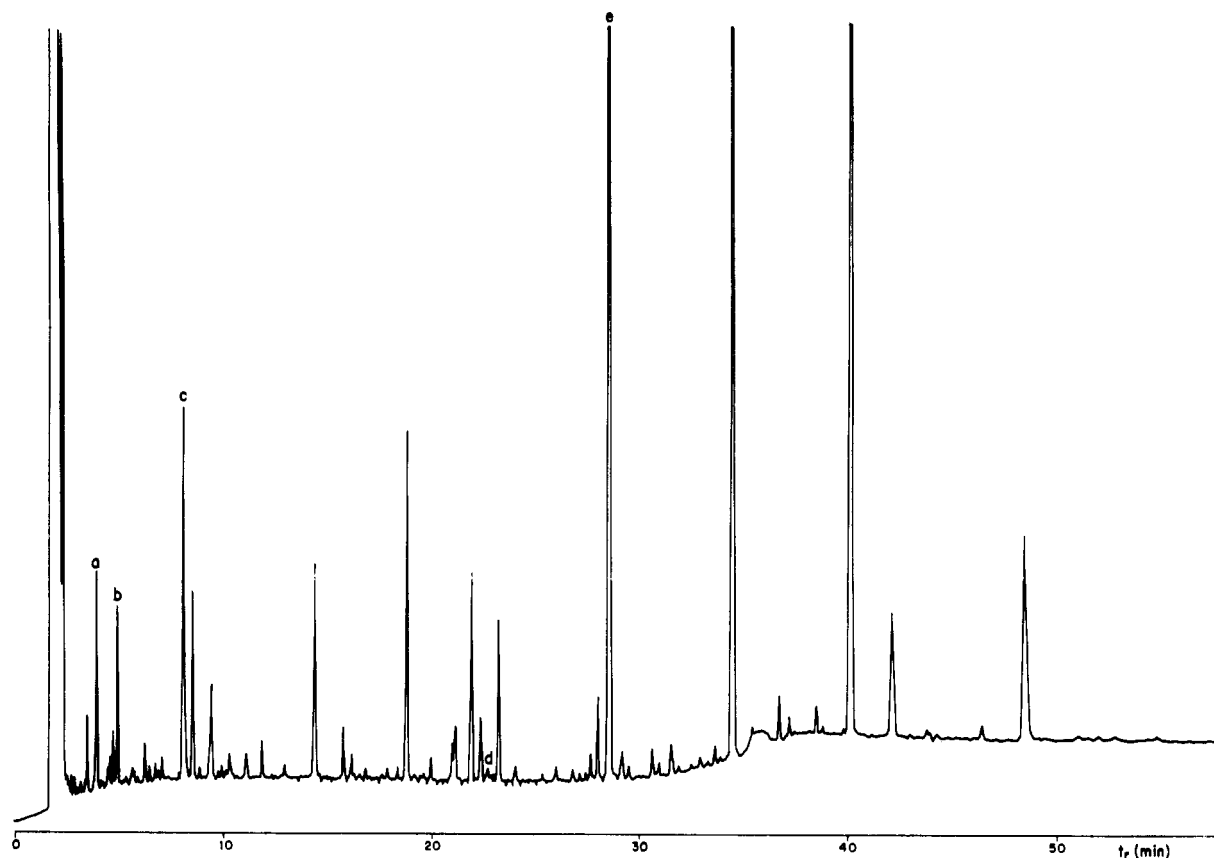


Figure 5. Capillary gas chromatogram of an SDE extract from a Los Beyos cheese. Temperature was programmed from 60 to 210 °C at 4 °C/min on a 25 m × 0.2 mm (i.d.) SP-1000 column. The peak numbers correspond to (a) 2-pentanone, (b) ethyl butanoate, (c) methyl hexanoate, (d) butanoic acid, and (e) hexanoic acid.

Table I. Characteristics of the Studied Cheeses

variety		samples	milk	type of cheese	mass	rind	typical ripening period, days
code	name						
A	Gamonedo	1, old 2, old	mixture ^a cow	semihard	some greenish-blue veins	salted, smoked, and wrapped in fern leaves	60-180 in caves
B	Cabrales	3, fresh 4, ripe 5, old 6, ripe	cow cow mixture ^a cow	semihard	blue veins	salted and wrapped in chestnut tree leaves	60-180 in caves
C	La Peral	7, ripe 8, old	cow cow	semihard	blue veins of <i>P. Roqueforti</i> (spores are added to milk)		60-180
D	Peñamellera	9, ripe 10, ripe 11, fresh	cow mixture ^a mixture ^a	semihard	white	salt added	7-15
E	Los Beyos	12, ripe 13, ripe	cow cow	semihard	white	salted and smoked	15-60
F	Afuega'l pitu	14, white, fresh 15, white, ripe 16, red, fresh 17, red, ripe	cow cow cow cow	fresh	white or red, depending on if curd is mixed with salt and/or red pimento	without rind	15-60

^a These cheeses were made from a mixture of cow, goat, and sheep's milk; this is common practice in Asturias region.

panoate, isobutanoate, and butanoate) were found in most samples, their level being higher in blue varieties and very low also in the D variety. 2-Phenylethanol was also found in all the varieties. These types of esters have been found in many types of cheeses (Dumont et al., 1974; Guichard et al., 1987; Gallois and Langlois, 1990).

Several terpenes (α -pinene, γ -terpinene, and 1,8-cineole) were detected in some samples. Their presence in cheese is probably not related to the ripening processes but to the diets of cows grazing in high mountain pastures

(Dumont and Adda, 1978). They have been found in other varieties (Meinhart and Schreier, 1986; Guichard et al., 1987).

Substances such as indole, acetophenone, and 2-phenylethanol are considered degradation products of amino acids (Adda, 1984; Badings, 1984). Chlorinated hydrocarbons have been also found in other cheeses (Liebich et al., 1970; Dumont et al., 1974), but no explanation was given. Isoamyl esters (butanoate, isobutanoate, and pentanoate) were also found. Some S-containing compounds,

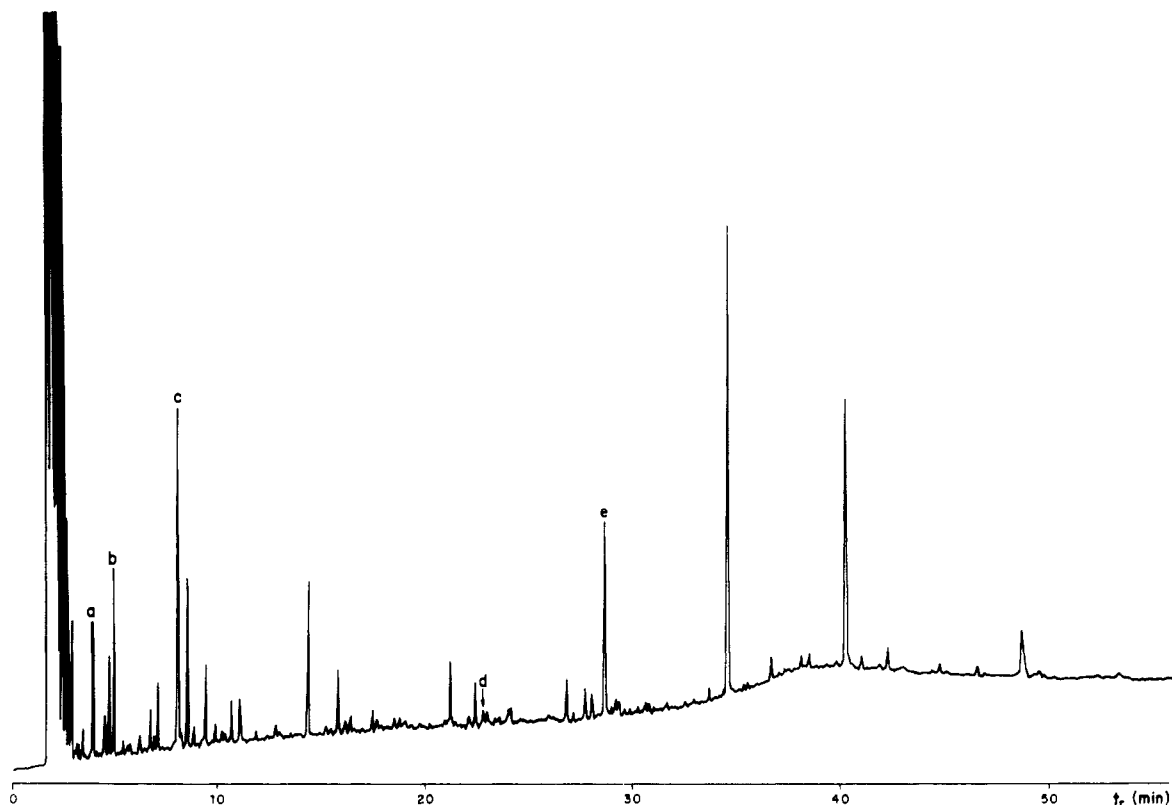


Figure 6. Capillary gas chromatogram of an SDE extract from an Afuega'l pitu cheese. Temperature was programmed from 60 to 210 °C at 4 °C/min on a 25 × 0.2 mm (i.d.) SP-1000 column. The peak numbers correspond to (a) 2-pentanone, (b) ethyl butanoate, (c) methyl hexanoate, (d) butanoic acid, and (e) hexanoic acid.

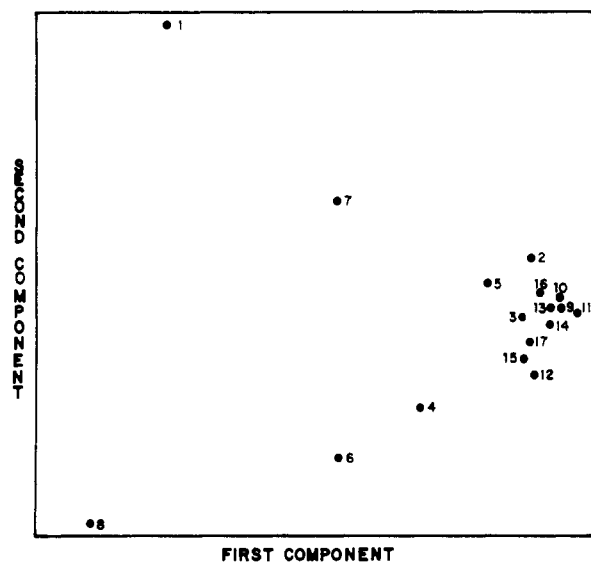


Figure 7. KARLOV (Harper, 1981) plot using the two first principal components for the 17 samples studied.

Table II. Chromatographic Conditions Used for the Analysis of SDE Fractions

stationary phase	tube	temp program
0.1 μm SE-30	0.3 mm × 22 m	60–250 °C, 2 °C/min
0.4 μm SP-1000	0.2 mm × 25 m	60–210 °C, 4 °C/min
apparatus	HP-5890 gas chromatograph, with FID	
carrier gas	nitrogen	
injection mode	split, ratio 1:30	
temp	300 °C	

such as thioesters and trithiapentane, were present in some of the samples studied, but they could be not quantified.

Several phthalates (dimethyl, diethyl, and dioctyl) were

found in different samples. These substances are well-known plasticizers which are frequently found in samples of different origins (Crompton, 1979); as some of them appear in blank assays, they have been supposed to be contaminants. Aliphatic hydrocarbons from 10 to 32 carbon atoms were also detected, but some of them seem to come from the pentane used in the SDE fractionation.

FFA are important components in cheese flavor, and their role is different according to the cheese variety: they give the main note to the flavor of some Italian cheeses (Woo and Lindsay, 1984) but only the background in Cheddar (Patton, 1962). Methyl ketones and 2-alkanols are typical components of the flavor of blue cheeses. Most of the esters found (ethyl, 2-phenylethyl, and isoamyl esters) have a sweet odor (fruity or floral) and may contribute to cheese aroma by smoothing the notes from acids and ketones. The role of lactones is difficult to estimate, because they have chiral structures; the two enantiomers (which cannot be resolved in achiral columns) usually have different organoleptic properties.

Most quantified components listed in Table II show a wide concentration range, both among and within varieties. The main reason for the variability among cheeses of the same variety is the different ripening stage of the selected samples, which seems to be an important source of variation (Guichard et al., 1987). On the contrary, the effect of milk species has been found to have less significance in other cheese types (Martinez-Castro et al., unpublished results).

The difference in composition between blue and white varieties is easily appreciated. The three blue varieties are richer in FFA, methyl ketones, and 2-alkanols than the other cheeses. Varieties B and C were also richer in most components than variety A, which showed few veins of *Penicillium*. White varieties were distinguished mostly by δ-lactones (very scarce in variety E), esters (present in

Table III. Concentration Range (Micrograms per Gram) for the Six Cheese Varieties Studied

compd	I_R^a	cheese variety					
		Gamonedo A	Cabrales B	La Peral C	Peñamellera D	Los Beyos E	Afuega'l pitu F
1 2-heptanone	866	6.3-14.3	0.6-11.4	17.0-31.0	0.1-0.8	0.2-1.8	nd ^b -1.4
2 ethyl hexanoate	980	0.3-2.9	0.6-2.8	0.6-2.2	0.1-0.2	0.8-1.4	0.2-2.5
3 2-nonenone	1051	2.8-3.8	0.2-2.6	3.0-3.8	nd-0.02	nd-0.06	nd
4 acetophenone	1058	nd	nd-1.2	nd	0.06-0.2	0.1-0.2	nd-0.2
5 2-nonanone	1067	1.5-80.5	2.8-14.3	36.0-38.0	0.6-1.3	0.3-0.7	0.07-1.4
6 2-phenylethanol	1078	0.1-0.5	nd-1.8	0.6-1.2	0.09-5.5	0.1-0.2	0.04-0.1
7 2-nonanol	1085	2.4-13.0	1.1-8.8	0.3-5.6	nd-tr ^c	nd-0.2	nd
8 <i>n</i> -undecane	1100	0.02-0.04	nd	nd-tr	nd-0.1	0.09-0.1	nd
9 methyl octanoate	1107	0.2-1.3	0.2-0.6	0.3-0.5	nd-0.02	0.02-0.06	nd-0.7
10 unknown	1118	0.1-0.2	nd-0.2	tr-0.09	nd-0.02	tr-0.03	tr
11 unknown	1130	0.1	tr-0.2	0.1-0.2	0.04-0.06	0.1	0.03-0.2
12 2-decanone	1168	0.2-0.6	nd-0.3	0.2	nd	nd	nd
13 ethyl octanoate	1179	0.08-2.5	0.09-13.9	0.9-1.6	tr	0.2-0.3	0.4-0.9
14 octanoic acid	1188	0.2	nd-3.3	nd-4.0	0.02-0.04	0.3-9.1	0.05-8.6
15 2-phenylethyl acetate	1215	0.08-5.7	nd-1.7	0.3-10.8	nd-0.04	0.6-0.8	0.5-2.5
16 isoamyl ester	1228	0.2-0.4	0.07-0.7	0.2-15.0	nd	0.02-0.06	tr-0.1
17 indole	1263	0.5-0.6	0.04-0.2	0.2	nd-tr	nd-tr	nd-0.05
18 2-undecanone	1270	4.6-9.3	0.7-3.3	3.2-3.5	0.4-0.5	0.06-0.4	0.06-0.8
19 unknown	1289	0.1-0.4	nd-0.5	0.2-0.4	nd	nd	nd
20 <i>n</i> -tridecane	1300	0.05-0.3	nd-0.2	0.1-0.2	0.03-0.06	0.03-0.1	0.02-0.09
21 methyl decanoate + 2-phenylethyl propanoate	1309	1.4-2.2	0.2-1.4	0.8-1.3	nd	nd-0.04	nd-0.2
22 unknown	1354	0.3-0.8	nd	nd	tr	nd	nd
23 unknown	1369	0.08-0.3	nd-0.3	0.1-0.3	tr	tr-0.02	0.02-0.1
24 ethyl decanoate + decanoic acid	1381, 1388	0.3-3.9	0.5-38.3	6.9-70.8	tr	0.1-7.6	0.5-6.1
25 2-phenylethyl isobutanoate + <i>n</i> -tetradecane	1400	0.2-0.6	0.4-1.0	nd-0.4	0.05-0.06	0.03-0.08	tr-0.4
26 unknown	1429	0.2-0.3	0.05-0.3	nd-0.2	nd	0.02-0.04	nd-0.02
27 δ -decalactone + 2-phenylethyl butanoate	1446	0.4-0.5	0.03-0.4	0.03-0.06	tr-0.4	tr-0.2	nd-0.1
28 unknown	1463	0.07-0.1	0.09-0.2	0.05-0.06	0.03-0.1	0.02-0.05	0.01-0.1
29 2-tridecanone	1474	0.5-0.7	0.5-1.2	0.4-0.7	0.2-0.4	tr-0.2	0.5-1.2
30 <i>n</i> -pentadecane	1500	0.08-0.1	0.03-0.1	0.1	0.07-0.08	0.1	0.05-0.1
31 methyl dodecanoate	1509	0.03-0.3	nd-0.5	0.09-0.1	nd	nd	nd-0.03
32 ethyl dodecanoate + docecanoic acid	1577	0.2-0.5	0.03-4.8	2.3-11.5	nd	0.03-0.2	0.1-0.4
33 <i>n</i> -hexadecane	1600	0.05-0.07	nd-0.08	nd-0.03	0.04-0.06	0.03-0.07	0.05-0.07
34 unknown	1626	0.2-0.3	0.07-1.3	0.1-0.3	nd	nd-tr	nd
35 δ -dodecalactone	1651	0.05-0.1	nd-0.08	0.1-0.2	0.1-0.3	0.08-0.1	nd-0.3
36 2-pentadecanone	1676	0.2-0.3	0.2-0.4	0.2	0.1-0.4	nd-0.1	0.02-0.3
37 <i>n</i> -heptadecane	1700	0.06-0.1	0.03-0.07	0.06-0.07	0.05-0.1	0.03-0.05	0.04-0.2
38 methyl tetradecanoate	1708	0.04-0.2	nd-0.3	0.1-0.2	0.02-0.09	nd-0.04	0.02-0.1
39 ethyl tetradecanoate + tetradecanoic acid	1779, 1769	0.3-1.5	0.2-1.8	2.6-5.3	nd	0.03-0.1	0.1-0.2
40 unknown	1788	0.2-0.6	nd-0.2	0.7-0.8	0.08-0.7	0.05-0.2	0.2-1.4
41 <i>n</i> -octadecane	1800	0.1-0.3	0.04-0.1	0.2-0.3	0.08-0.1	0.06-0.1	0.1-0.8
42 unknown	1812	0.1-0.2	0.03-0.06	0.2	nd-0.04	0.04-0.1	tr-0.3
43 unknown	1833	nd-0.2	nd-0.1	0.1-0.2	0.01-0.2	0.03-0.09	nd-0.2
44 unknown	1843	0.3	0.1-0.2	0.2	0.1-0.3	0.06-0.3	0.1-0.2
45 δ -tetradecalactone	1859	0.07-0.09	nd	0.08-0.1	0.05-0.1	tr-0.1	nd-0.04
46 2-heptadecanone	1878	0.09-0.1	0.02-0.09	0.1	nd-tr	tr-0.04	nd-0.03

^a I_R , experimentally determined retention linear indices on the SE-30 column. ^b nd, not detected. ^c tr, trace level, $\leq 0.01 \mu\text{g/g}$.

Table IV. Other Compounds Identified in the Cheeses Studied

peak ^a	component	identification		component	identification	
		GC	GC/MS		GC	GC/MS
d	butanoic acid	+	+	2-heptanol	+	+
e	hexanoic acid	+	+	2-methylpentanol	-	+
	hexadecanoic acid	+	+	2-nonenol	-	+
	octadecanoic acid	+	+	naphthalene	-	+
b	ethyl butanoate	+	+	methylnaphthalene	-	+
	ethyl hexadecanoate	+	+	methylethylphenol	-	+
	ethyl tetradecanoate	-	+	anetol	-	+
	ethyl hexadecanoate	-	+	<i>p</i> -methylanisole	-	+
	ethyl octadecanoate	-	+	<i>p</i> -dimethoxybenzene	-	+
c	methyl hexanoate	+	+	dichlorobenzene	-	+
	δ -octalactone	-	+	trichlorobenzene	-	+
	δ -hexadecalactone	-	+	trithiapentane	-	+
	γ -decalactone	-	+	isoamyl butanoate	-	+
a	2-pentanone	+	+	isoamyl isobutanoate	-	+
	2-octanone	+	+	isoamyl pentanoate	-	+
	2-dodecanone	+	+	α -pinene	-	+
	2-tetradecanone	-	+	γ -terpinene	-	+
	2-hexadecanone	-	+	1,8-cineole	-	+

^a In Figures 5 and 6.

variety A in higher concentrations), and FFA (almost absent in variety D).

In spite of the different characteristics of the samples studied, some of their components seem to be related.

The subroutines KADISP and HIER in the ARTHUR package (Harper, 1981) permit the grouping of the components according to their correlation coefficients. Two main groups are formed; the first includes 2-heptanone, most of the fatty acids, and ethyl esters, while the second is formed by the ketones having from 9 to 15 carbon atoms. The metabolic pathways that produce the compounds in each group seem to be related.

The composition of the samples can be shown graphically with a minimum loss of information by using the principal components technique (Wolff and Parsons, 1983). The plot coordinates in Figure 7 were calculated from the original composition data obtained by GC with column 1 (Table II) using the KARLOV and KAPRIN routines in ARTHUR (Harper, 1981). The two first components explain 94.5% of total variance. The first component is related with the total volatile content; the second component has a negative weight for most ethyl esters and acids and a positive weight for ketones and methyl esters. Samples rich in the components of the first group are plotted at the bottom of Figure 7, while samples rich in ketones are plotted near the top.

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Registry No. 2-Heptanone, 110-43-0; ethyl hexanoate, 123-66-0; 2-nonenone, 27936-13-6; acetophenone, 98-86-2; 2-nonanone, 821-55-6; 2-phenylethanol, 60-12-8; 2-nonanol, 628-99-9; *n*-undecane, 1120-21-4; methyl octanoate, 111-11-5; 2-decanone, 693-54-9; ethyl octanoate, 106-32-1; octanoic acid, 124-07-2; 2-phenylethyl acetate, 103-45-7; isoamyl ester, 123-51-3; indole, 120-72-9; 2-undecanone, 112-12-9; *n*-tridecane, 629-50-5; methyl decanoate, 110-42-9; 2-phenylethyl propanoate, 122-70-3; ethyl decanoate, 110-38-3; decanoic acid, 334-48-5; 2-phenylethyl isobutanoate, 103-48-0; *n*-tetradecane, 629-59-4; δ -decalactone, 705-86-2; 2-phenylethyl butanoate, 103-52-6; 2-tridecanone, 593-08-8; *n*-pentadecane, 629-62-9; methyl dodecanoate, 111-82-0; ethyl dodecanoate, 106-33-2; dodecanoic acid, 143-07-7; *n*-hexadecane, 544-76-3; δ -dodecalactone, 713-95-1; 2-pentadecanone, 2345-28-0; *n*-heptadecane, 629-78-7; methyl tetradecanoate, 124-10-7; ethyl tetradecanoate, 124-06-1; tetradecanoic acid, 544-63-8; *n*-octadecane, 593-45-3; δ -tetradecalactone, 2721-22-4; 2-heptadecanone, 2922-51-2; butanoic acid, 107-92-6; hexanoic acid, 142-62-1; hexadecanoic acid, 57-10-3; octadecanoic acid, 57-11-4; ethyl butanoate, 105-54-4; ethyl hexadecanoate, 628-97-7; ethyl tetradecenoate, 71178-24-0; ethyl hexadecenoate, 27710-66-3; ethyl octadecenoate, 28555-06-8; methyl hexanoate, 106-70-7; δ -octalactone, 698-76-0; δ -hexadecalactone, 7370-44-7; γ -decalactone, 706-14-9; 2-pentanone, 107-87-9; 2-octanone, 111-13-7; 2-dodecanone, 6175-49-1; 2-tetradecanone, 2345-27-9; 2-hexadecanone, 18787-63-8; 2-heptanol, 543-49-7; 2-methylpentanol, 105-30-6; 2-nonanol, 22104-79-6; naphthalene, 91-20-3; methyl-naphthalene, 1321-94-4; methylethylphenol, 30230-52-5; anethole, 104-46-1; *p*-methylanisole, 104-93-8; *p*-dimethoxybenzene, 150-78-7; dichlorobenzene, 25321-22-6; trichlorobenzene, 12002-48-1; trithiapentane, 7529-06-8; isoamyl butanoate, 106-27-4; isoamyl isobutyrate, 2050-01-3; isoamyl pentanoate, 2050-09-1; α -pinene, 80-56-8; γ -terpinene, 99-85-4; 1,8-cineole, 470-82-6.