Antioxidant Constituents in Sage (*Salvia officinalis*)†

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The antioxidant compounds of oleoresin of sage (*Salvia officinalis*) were separated by column chromatography and high-performance liquid chromatography. Six major compounds were purified and identified by IR, MS, and ^{1}H NMR spectrometry as carnosol, carnosic acid, rosmadial, rosmanol, epirosmanol, and methyl carnosate. Their antioxidative activity was measured with an accelerated test, and their content was quantified in sage and in four commercial rosemary extracts.

INTRODUCTION

In food processing, lipidic oxidation not only causes a loss in nutritional and gustative quality of foods but also generates oxidized products such as free radicals which lead to various undesirable chemical reactions. To avoid or delay this autoxidation process, antioxidants have been used for over 50 years.

The recent consumer interest in "natural" products also requires natural antioxidative substances to replace conventional antioxidants such as BHT and BHA.

The spices, notably the Labiatae family, are well-known for their antioxidative properties, and two plants, especially, rosemary and sage, have been reported to have strong characteristics (Chipault et al., 1956; Cort, 1974; Watanabe and Ayano, 1974; Saito et al., 1976; Bishov et al., 1977; Gerhardt and Schröter, 1983; Houlihan and Ho, 1985). Rosemary has been extensively studied for its antioxidative principles as well as for its industrial and commercial exploitation (Brieskorn and Dömling, 1969; Chang et al., 1977, 1987; Wu et al., 1982; Löliger, 1989; Schuler, 1990; Chen et al., 1992). The current hypothesis is that sage contains the same antioxidants as rosemary but this fact has been proven for only three compounds: carnosol, rosmanol, and rosmadial (Nakatani, 1989). On the other hand, 9-ethylrosmanol ether was identified only in sage (Djarmati et al., 1991).

Sage was shown to possess a strong antioxidative efficiency comparable to rosemary when tested against methyl linoleate oxidation in an apolar medium (Cuvelier et al., 1991). We report here the content in major antioxidants of sage oleoresin.

MATERIALS AND METHODS

Materials. The oleoresin of *Salvia officinalis* was obtained from René Laurent Co. (Grasse, France).

Isolation of the Antioxidants from Sage. A primary fractionation of the sage oleoresin was carried out by column chromatography, and then individual compounds were isolated and collected by high-performance liquid chromatography.

(*a*) *Column Chromatography.* Seventy-five grams of sage oleoresin diluted in hexane (Prolabo R.P. Normapur) was loaded on a 1-m-long column (5-cm diameter) containing 0.5 kg of silica gel, Kieselgel 60 [Merck (0.063–0.2 mm)]. The elution was carried out by 10 L of each solvent in the following sequence: hexane–diethyl ether 95:5 (F1); hexane–diethyl ether 85:15 (F2); hexane–diethyl ether 50:50 (F3). Just before use, the commercial diethyl ether (Prolabo R.P. Normapur) was distilled to eliminate traces of BHT. The collected fractions were concentrated under reduced pressure, at 40 °C, and then stored at 4 °C.

(*b*) *Semipreparative High-Performance Liquid Chromatography (HPLC).* The sage fractions were dissolved in methanol (Carlo Erba RS, CLHP grade), stirred for 3 min in an ultrasonic bath, and then filtered (Nalgene 190-2020 filters). The filtrate was immediatly injected into HPLC. The separation was performed on a Waters 600E apparatus equipped with a gradient system and a radial compression column (Waters RCM, 25 × 100 mm), filled with C_{18} Prep Nova-Pak (6 mm). At each time, 200 μL of a 10% methanolic extract was injected. The elution conditions were the following: methanol–1% acetic acid mixture (75:25) during 20 min, linear gradient for 25 min to 85:15, the last proportion being maintained for 15 min. The compounds collected after complete evaporation of the solvent in a rotary evaporator, under reduced pressure and at 40 °C, were stored under nitrogen at −20 °C.

Structural Identification of the Sage Antioxidants. The identification of the sage compounds collected from semipreparative HPLC was made by IR, mass spectrometry, and nuclear magnetic resonance (NMR).

(*a*) *IR Spectrometry (Perkin-Elmer 297 Apparatus).* The crystallized compounds were analyzed in KBr pellet and the liquid ones on a thin film between two NaCl crystals.

(*b*) *Mass Spectrometry (MS).* The mass spectra of the compounds were performed on a Hewlett-Packard 5995 apparatus (70 eV, one spectrum/s) by direct introduction of a tiny amount of product (about 10^{-8} g) laid in a capillary. In the ionization chamber, the temperature was raised by steps of 10 °C/min from 100 to 300 °C.

(*c*) *NMR Spectrometry.* ^{1}H NMR (250 and 400 MHz) spectra were run on an AC 250 and an AM 400 Brüker using trimethylsilane (TMS) as an internal standard. The compounds were diluted in methanol or acetone.

Analytical HPLC and UV Spectrophotometry. Sage and rosemary extracts were analyzed by HPLC using a HP 1040A photodiode array detector as the individual compounds eluted from a C_{18} Hypersil column, with the same gradient mixture described for the semipreparative process. Each identified compound was quantified from the peak area obtained at 284 nm using standard curves established previously (Cuvelier, 1992). The UV spectra of the individual compounds were obtained.

Test Method of Antioxidative Power (AOP). The antioxidative activity was measured according to the method described by Cuvelier et al. (1990) based on the disappearance of methyl linoleate in a lipophilic solvent, under strong oxidizing conditions, i.e., 110 °C and intensive oxygenation by bubbling pure oxygen with a flow rate of 7 mL/min. The half-life of the methyl linoleate, taken as reference value, is calculated from the kinetic curve of its disappearance. Points were measured every half hour, and the complete disappearance took less than 8 h when an initial concentration of 4% in dodecane was used, without antioxidant. Antioxidative efficiency was assessed by the relative increase of the half-reaction time. The efficiency varied according

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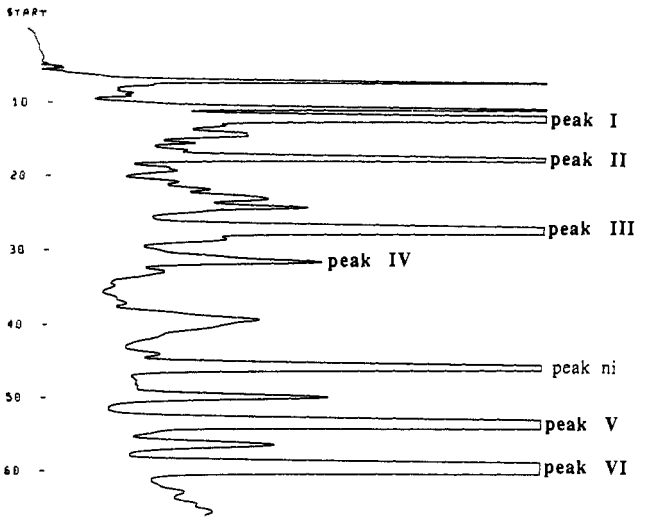


Figure 1. HPLC chromatogram from fraction F_3 of sage: column, C_{18} Hypersil; eluent, methanol–1% acetic acid (gradient from 75:25 to 85:15)

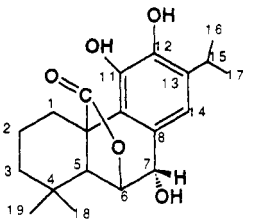
to the nature of the antioxidant and its concentration in the medium. To compare the efficiency of plant extracts, we determined the quantity of each (EQ, efficiency quantity) required to double the half-reaction time of the control and related it to the quantity of BHT providing the same effect. $EQ_{antioxidant}/EQ_{BHT}$ was called Eq_{BHT} (equivalent quantity). The lesser was Eq_{BHT}, the stronger was the antioxidant. To make this means easier, $1/Eq_{BHT}$, proportional to the activity, was used.

RESULTS

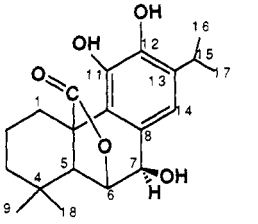
The antioxidative efficiency of the crude sage oleoresin was 3.5 times less than BHT. A primary fractionation of the oleoresin on column chromatography gave a first inactive fraction and then two fractions (F2 and F3) which showed a strong antioxidative activity: half of the activity of BHT for fraction F2 and a little less for fraction F3. These two fractions reacted strongly with Folin–Ciocalteu reagent, showing a high phenolic content. The residual extract eluted with methanol did not show any noticeable antioxidative activity.

The analysis of fractions F2 and F3 by HPLC revealed seven major peaks (Figure 1) which were collected individually by preparative HPLC. They were analyzed by IR, ^{1}H NMR, and mass spectrometry. Their identification was deduced from structural data compared to those published in the literature for the antioxidative diterpenes found in rosemary (Wenkert et al., 1965; Nakatani and Inatani, 1981, 1983, 1984; Inatani et al., 1982; Wu et al., 1982). The unidentified major peak mentioned in Figure 1 is probably not pure because it was impossible to interpret precisely the NMR spectra. Further investigations of MS and NMR spectra are being done to verify a tentative identification of isorosmanol which has been described by Nakatani and Inatani (1984).

Compounds **I** and **II** appeared to be isomers and were identified respectively as **rosmanol** and **epirosmanol**. Rosmanol: MS, *m/z* (%): 346 (M^+, 85), 300 (59), 287 (76), 273 (52), 269 (26), 232 (35), 231 (100), 218 (34), 215 (68); UV λ_{max} 214, 288 nm; IR ν_{max} 3500, 3300, 2950, 1745, 1460, 1295, 1280, 1220, 1205, 1170, 1120, 1080, 1060, 1005 cm^{-1}; ^{1}H NMR (acetone) δ 0.89 (3H-19, s), 1.02 (3H-18, s), 1.16 (6H-16,17, t), 1.3–1.9 (5H-1ax,2,3, m), 2.28 (1H-5, s), 3.28 (2H-15, 1eq, m), 4.50 (1H-6, d), 4.62 (1H-7, d), 6.86 (1H-14, s). Epirosmanol: UV λ_{max} 196, 230, 288 nm: ^{1}H NMR (acetone) δ 0.91 (3H-19, s), 1.02 (3H-18, s), 1.18 (6H-16,17, dd), 1.3–1.9 (5H-1ax,2,3, m), 1.96 (1H-5, s), 3.28 (2H-15, 1eq, m), 4.69 (2H-6,7, br s), 7.00 (1H-14, s). The structural



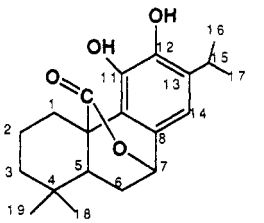
compound I: rosmanol



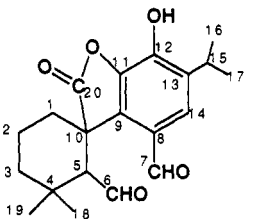
compound II: epirosmanol

difference of these two compounds, located at the configuration of C-7, was verified by NOE measurement of NMR spectra. In compound **I**, no NOE was observed between H-5 and H-6 or H-7. On the other hand, in compound **II**, a sharp NOE occurred between H-5 and H-7. These observations assigned an axial configuration of H-7 in compound **I**, rosmanol, and an equatorial one in compound **II**, epirosmanol. This determination confirmed the proposal of Nakatani and Inatani to resolve the discrepancy between former experiments (Nakatani and Inatani, 1981, 1984; Inatani et al., 1982).

Compounds **III** and **IV** were identified respectively as **carnosol** and **rosmadial**. Carnosol: MS, *m/z* (%), 330



compound III: carnosol



compound IV: rosmadial

(M^+, 17), 287 (21), 286 (100), 271 (17), 215 (50), 204 (22), 202 (20); UV λ_{max} 210, 284 nm; IR ν_{max} 3500, 3300, 1715, 1590, 1455, 1350, 1320, 1305, 1200, 1130, 1030, 990, 920 cm^{-1}; ^{1}H NMR (methanol) δ 0.87 (6H-19,18, s), 1.18 (3H-16, d), 1.20 (3H-17, d), 1.30 (1H-3eq, m), 1.52 (1H-3ax, m), 1.59 (1H-2eq, m), 1.69 (1H-5, dd), 1.83 (1H-6ax, m), 1.91 (1H-2ax, m), 2.19 (1H-6eq, m), 2.56 (1H-1eq, ddd), 2.80 (1H-1ax, m), 3.25 (1H-15, m), 5.41 (1H-7, dd), 6.69 (1H-14, s). Rosmadial: MS, *m/z* (%), 344 (M^+, 20), 288 (22), 287 (100), 273 (19), 231 (16); UV λ_{max} 208, 290 nm; IR ν_{max} 3150, 2900, 1800, 1715, 1655, 1605, 1580, 1240, 1170, 1125, 1010 cm^{-1}; ^{1}H NMR (methanol) δ 1.24 (9H-16,17,19, m), 1.47 (3H-18, s), 1.5–2.4 (6H-1,2,3, m), 3.32 (1H-15, m), 4.14 (1H-5, s), 7.48 (1H-14, s), 9.62 (1H-6, d), 9.64 (1H-7, s). Our data matched well with the published data (Inatani et al., 1982; Wu et al., 1982; Nakatani and Inatani, 1983).

Compound **V** was identified as **carnosic acid** by means of MS and NMR data: MS, *m/z* (%), 332 (M^+, 8), 287 (22), 286 (100), 271 (19), 243 (24), 230 (48), 217 (16), 215 (18), 204 (20); ^{1}H NMR (methanol) δ 0.92 (3H-19, s), 0.99

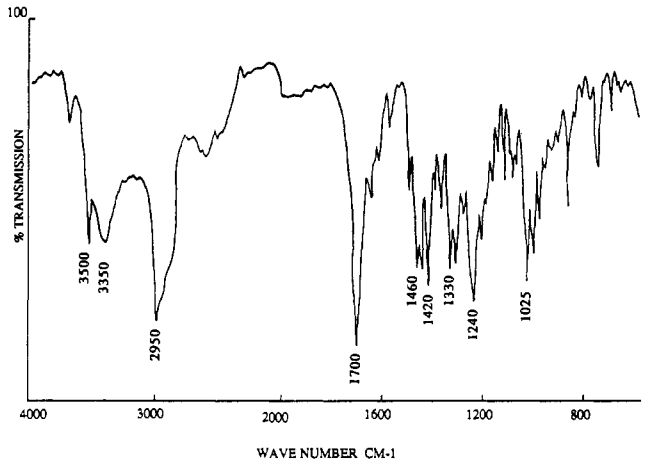
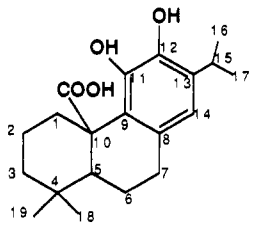
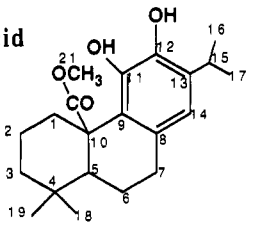


Figure 2. IR spectrum of methyl carnosate (compound **VI**).

(3H-18, s), 1.05 (1H-1ax, dd), 1.17 (6H-16,17, dd), 1.30 (1H-3eq, m), 1.4–1.6 (3H-2eq,3ax,7ax, m), 1.80 (1H-5, d), 2.04 (1H-2ax, m), 2.40 (1H-6ax, m), 2.77 (2H-6eq,7eq, dd), 3.18 (1H-15, sept), 3.50 (1H-1eq, d), 6.44 (1H-14, s); UV λ_{max} 202, 230, 284 nm; IR ν_{max} 1680, 1420, 1370, 1320, 1290 cm^{-1}. The published data (Wenkert et al., 1965) gave only a part of the NMR spectrum, which agreed with ours.



compound V: carnosic acid



compound VI: methyl carnosate

Compound **VI** was identified as **methyl carnosate**: MS, *m/z* (%), 346 (M^+, 8), 301 (23), 300 (100), 285 (24), 257 (16), 245 (10), 244 (44), 232 (15), 231 (15), 229 (16); UV λ_{max} 208, 228, 282 nm. Its IR and NMR spectra are reported in Figures 2 and 3, respectively. The molecular structure of methyl carnosate was inferred from the NMR spectrum, which was similar to that of carnosic acid. The major difference was the appearence of an additional CH_3 at δ 1.10 (3H-21, s).

The antioxidative activity of carnosic acid, methyl carnosate, rosmadial, carnosol, and rosmanol isolated from sage was measured by the AOP test (Cuvelier et al., 1990) and reported to that of BHT (Table 1).

The content in carnosol, rosmadial, carnosic acid, and methyl carnosate was quantified in four commercial rosemary extracts as well as in sage extracts by analytical HPLC. Rosmanol and epirosmanol were not measured because of their poor separation. Fortunately, they were only present in small amounts. In Table 2, the contents are expressed as grams of compound per gram of crude extracts. The measured antioxidative activity ($1/Eq_{BHT}$) of each crude extract was compared to the sum (S) of the activities brought by the four compounds calculated according to the equation mentioned in Table 2.

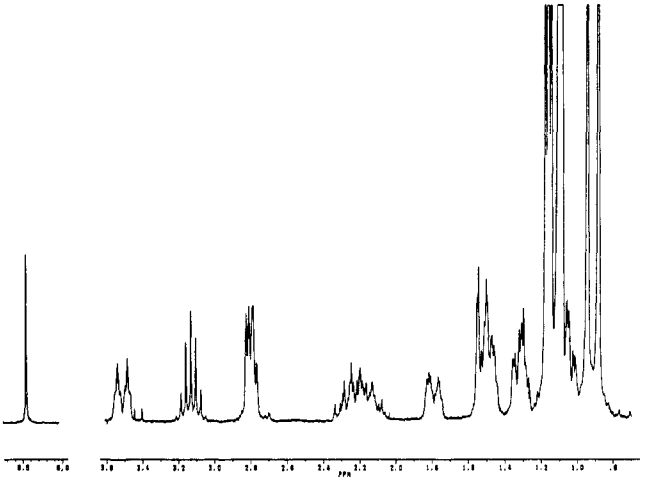


Figure 3. ^1H NMR spectrum of methyl carnosate (compound **VI**).

Table 1. Antioxidative Activity, Reported to That of BHT, of Major Compounds Isolated from Sage

	$1/Eq_{BHT}$ (wt equiv)	$1/Eq_{BHT}$ (molar equiv)
BHT	1	1
carnosic acid	0.24 ± 0.03	0.36 ● 0.05
methyl carnosate	0.14 ± 0.02	0.22 ± 0.03
rosmadial	0.13 ● 0.02	0.20 ± 0.04
carnosol	0.13 ± 0.02	0.19 ± 0.04
rosmanol	0.10 ± 0.02	0.16 ● 0.03

DISCUSSION

Carnosol, rosmadial, carnosic acid, rosmanol, and epirosmanol, which have already been found in rosemary, are also present in *S. officinalis*. In addition, the presence of isorosmanol is speculative. The resemblance of the compositions of these two Labiatae is now well established.

Methyl carnosate, revealed as a major compound, has not yet been reported elsewhere. It was present especially in fraction F3 of the sage and also in two commercial rosemary extracts (Table 2). It is possible that a part of this compound derived from carnosic acid, known for its instability (Wenkert et al., 1965). In fact, when a methanolic solution of carnosic acid was followed for several days, a marked decrease of carnosic acid was observed while carnosol, rosmadial, and the unidentified compound and later methyl carnosate increased (Figure 4). On the other hand, fresh methanolic preparations of fraction 2, particularly rich in carnosic acid, did not show any methyl carnosate, ruling out the possible degradation of carnosic acid during the HPLC procedure.

These facts raised the following question: is methyl carnosate a true antioxidant of rosemary varieties or an artifact of the preparation of oleoresin?

The antioxidative activity of all of these components is related to their phenolic structure: these compounds, except rosmadial, are diterpenes of the ferruginol type with two orthophenolic functions and one isopropyl group on the adjacent carbon. They are known as excellent antioxidants (Brieskorn and Dömling, 1969; Nakatani and Inatani, 1981, 1983). Comparison of the antioxidative efficacy of the major compounds isolated from sage, except for epirosmanol which was not measured, showed that carnosic acid had the greatest activity (Table 1). However, these compounds were 3–7 times less active than BHT, and therefore, their efficacy appeared to be lower than in published data. These differences may be explained by the instability of the purified compounds, which is increased by the heating at 110 °C of the oxidation test.

Table 2. Content (in Grams per Gram of Extract) in Four Identified Compounds, Measured Antioxidative Activity ($1/Eq_{BHT}$), and Calculated Activity (S)[a] of Various Rosemary and Sage Extracts

extract	carnosol	rosmadial	carnosic acid	methyl carnosate	$1/Eq_{BHT}$ (wt equiv)	S (wt equiv)
rom 1, commercial rosemary extract (viscous)	0.027 ± 0.003	0.007 ± 0.002	0.26 ± 0.021	0.085 ± 0.006	1 ± 0.09	0.08
rom 2, commercial rosemary extract (powder)	0.057 ± 0.005	0.018 ± 0.002			0.7 ± 0.06	0.01
rom 3, commercial rosemary extract (powder)	0.15 ± 0.01	0.034 ± 0.001	0.012 ± 0.004		0.6 ± 0.06	0.02
sage F 2	0.052 ± 0.010	0.018 ± 0.002	0.19 ± 0.01		0.5 ± 0.07	0.05
sage F 3	0.075 ± 0.01	0.009 ± 0.004	0.029 ± 0.009	0.26 ± 0.02	0.4 ± 0.06	0.05
rom 4, commercial rosemary extract (viscous)	0.032 ± 0.008	0.012 ± 0.003	0.18 ± 0.02	0.13 ± 0.01	0.4 ± 0.07	0.06
sage oleoresin	0.036 ± 0.003	0.006 ± 0.001	0.057 ± 0.007	0.039 ± 0.005	0.3 ± 0.07	0.02

[a] $S = \sum(r_i \times 1/Eq_{BHT\,i})$, where r_i = weight ratio of the compound i in the extract and $1/Eq_{BHT\,i}$ = antioxidative activity of the compound i as given in Table 1.

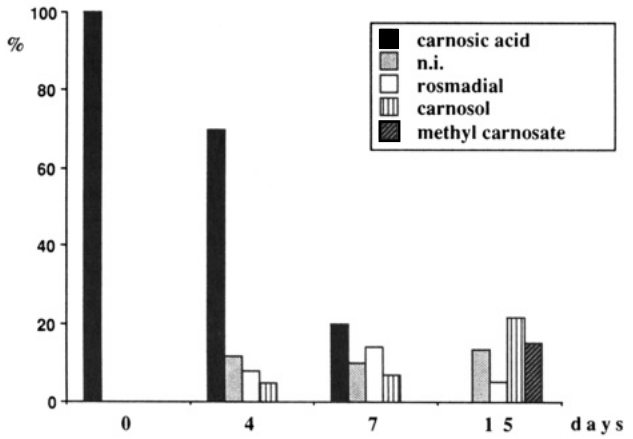


Figure 4. Evolution of a methanolic solution of carnosic acid at 0, 4, 7, and 15 days.

Moreover, the mixture of the five major compounds in a ratio identical to that found in crude oleoresin showed an activity much less than the crude oleoresin activity. It is obvious that the purification yield of these substances was not equal to 100%. It is also possible that the oleoresin contained some protective agents of the phenols as well as other antioxidants which were not taken into account. Some flavonoids, which may participate in the antioxidative activity of rosemary, have been isolated by Aeschbach et al. (1986). In sage, to our knowledge, such an element has not yet been shown, but today we cannot exclude such a hypothesis.

All of these considerations are enhanced by the data of Table 2, since the calculated activities (S) represent only 1.4–17% of the activities measured for the extracts ($1/Eq_{BHT}$).

Two extracts, rom 2 and 3, appeared to be particularly interesting: their commercial form was a powder that contained no methyl carnosate and almost no carnosic acid. Their high antioxidative activity, close to that of BHT, might be caused by a large peak of rosmarinic acid which was eluted with the solvent under the HPLC conditions used. On the other hand, rosmarinic acid seemed to be absent in the extracts rom 1 and rom 4 and in sage.

In conclusion, to establish a correlation between the antioxidative efficacy and the composition of all phenolic compounds, it is necessary to measure and take into account the efficacy of each.

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