Fusion of Three Sensory Modalities for the Multimodal Characterization of Red Wines

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***Abstract*—This work represents the first attempt to develop a sensory system, specifically designed for the characterization of wines, which combines three sensory modalities: an array of gas sensors, an array of electrochemical liquid sensors, and an optical system to measure color by means of CIElab coordinates. This new analytical tool, that has been called "electronic panel," includes not only sensors, but also hardware (injection system and electronics) and the software necessary for fusing information from the three modules. Each of the three sensory modalities (volatiles, liquids, and color) has been designed, tested, and optimized separately. The discrimination capabilities of the system have been evaluated on a database consisting of six red Spanish wines prepared using the same variety of grape (tempranillo) but differing in their geographic origins and aging stages. Sensor signals from each module have been combined and analyzed using pattern recognition techniques. The results of this work show that the discrimination capabilities of the system are significantly improved when signals from each module are combined to form a multimodal feature vector.**

***Index Terms*—Electronic nose, electronic panel, electronic tongue.**

I. INTRODUCTION

THE LAST few years have witnessed a great deal of research in the area of artificial olfactory systems [1]–[3], with a number of efforts focusing on the discrimination of wines [4], [5]. Discrimination of alcoholic beverages has been shown to be a challenging problem due to the notorious cross selectivity of odor sensors to water and ethanol, requiring the use of preconcentration techniques to reduce these effects [6], [7]. The initial success of "electronic noses" motivated the idea of constructing an "electronic tongue" using an array of non-selective electrochemical sensors coupled with chemometric methods for the recognition and discrimination of complex liquids [8]–[10]. Early efforts in this field involved signal generation from potentiometric sensors [11]. Voltammetric sensors have also been used to classify liquid samples and beverages [10], [12]–[15]. These sensors are advantageous due to their high versatility. They can be used with a range of voltages, wave forms, and electroactive materials to obtain sensing units with different selectivity.

It has been demonstrated that the simultaneous utilization of electronic noses and electronic tongues can increase the amount of information extracted from the samples [4], [16]. However, the complete characterization of beverages requires taking into account their visual aspects.

The objective of this work is to develop an innovative system that integrates a sensory system for the analysis of volatiles (commonly referred to as an "electronic nose"), a sensory system for the analysis of liquids (an "electronic tongue"), and a device able to measure the color of the samples (an "electronic eye"). The system has been specifically designed for the characterization and discrimination of wines.

To reduce the abovementioned cross selectivity of gas sensors to water and ethanol, the proposed system combines an array of metal-oxide sensors with solid phase micro extraction (SPME).

One of the novelties of the work lies in the development of a multi-channel taste sensor based on new voltammetric electrodes of rare-earth bisphthalocyanines and conducting polymers [13], [14], which produce a variety of electrochemical responses when exposed to wines.

In addition, their electrochemical properties can be modulated by introducing chemical modifications in the sensitive materials [17], [18]. Finally, information about the color properties of the wine is captured in our system by measuring the transmittance at wavelengths representative of the psycho-physics attributes of color [19].

Finally, a common software that allows to collect the outputs of each sensor modality and to carry out the data treatment in a combined manner to form a multimodal feature vector was developed.

Using this method, signals from each sensory modality were combined, and the discrimination capabilities of the integrated system were evaluated using pattern recognition software.

II. EXPERIMENTAL

The gas sensor array was constructed using fourteen inorganic sensors from Figaro and FIS. (Table I). The sensors were mounted in a stainless steel chamber with a volume of 150 ml. The analyzes were conducted with a custom-made odor-delivery system combining the gas sensor array with a preconcentration system based on solid-phase microextraction (SPME). Three milliliters of the wines were placed in 10 ml vials, and

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TABLE I
ARRAY OF FOURTEEN MOS GAS SENSORS SELECTED FOR THE STUDY

ID	Sensors	R /Ω	$(\%\ \Delta r/r_o)_{1day}$
S1	SB-AQ1A	9.12E+03	2
S2	SB-AQ4	4.37E+03	4
S3	SB-95	5.70E+03	2
S4	SB-11A	4.41E+03	3
S5	SP-12A	3.71E+04	3
S6	SB-50	8.22E+02	1
S7	SPMW1	4.81E+04	0.5
S8	TGS-2620	6.39E+04	0.5
S9	SP-19	1.20E+06	1
S10	TGS-880	1.78E+05	4
S11	TGS-2611	5.02E+04	4
S12	TGS-822	8.36E+04	8
S13	TGS-2610	4.91E+04	5
S14	TGS-825	1.05E+04	8

the SPME fiber coated with 100 μm of polydimethyl siloxane (PDMS, Supelco) was introduced in the gas-phase of the vial for 15 min at room temperature (25 °C). The fiber was then placed in a heated injection port and desorbed in two steps. The first desorption was carried out at 50 °C to eliminate traces of water and ethanol adsorbed by the fiber. This eluent was discarded. A second thermal desorption was carried out at 250 °C for 30 min. The eluted vapors were injected in the test chamber. Data collection was performed through a commercial data acquisition card (PC-LPM-16, National Instruments). The sensors were polarized using a constant voltage of 5 V provided by a programmable power supply (FAC-662B). The scan rate used to measure changes in the resistance of the sensors was 0.5 s.

Poly (3-methylthiophene) (P3MT), polyaniline (PANI) and polypyrrole (Ppy) films were grown electrochemically by chronoamperometry (CA) onto a platinum wire mounted in a plastic holder. Electrochemistry was carried out in a potentiostat/galvanostat (Model 263A, EG&G). The reference electrodes were $Ag/AgNO_3$ for the nonaqueous media and Ag/AgCl for aqueous systems. The counter electrode was a platinum wire. Table II summarizes the preparation conditions for the polymeric sensors.

Three rare-earth bisphthalocyanines: praseodymium $(PrPc_2)$, gadolinium $(GdPc_2)$, and lutetium $(LuPc_2)$ bisphthalocyanines, were synthesized following previously published procedures [20] and used to prepare carbon paste electrodes (CPE). The CPE were prepared by mixing the corresponding phthalocyanine with carbon powder (UltraCarbon, Ultra F purity) (15% w:w). Nujol was used as a conglomerant [14]. The array of voltammetric sensors formed by the phthalocyanine and the conducting polymer electrodes was immersed in the wines under study. The sensing units were used as working electrodes in cyclic voltammetry experiments between -1.0 and 1.5 V at a scan rate of 0.1 Vs^{-1}. The cyclic voltammograms were registered in a custom-made potentiostat/galvanostat that allows the response of each sensor to be registered in a sequential manner. Following the measurement of each wine, a cleaning step consisting with 0.1 mol.L^{-1} KCl was carried out.

TABLE II
PREPARATION CONDITIONS FOR THE CONDUCTING POLYMER-BASED SENSING UNITS

Compound	**Conditions**		
Monomer	*Solvent*	*Polimerization Conditions*	*Electrolyte*
3-MET	ACN	1.3 V, 60 s	$LiClO_4$, 0.1 M
Aniline	Water	0.9 V, 60 s	HCl, 2 M
Aniline	Water	0.9 V, 60 s	TFA, 2M
Pyrrole	Water	0.8 V, 60 s	$K_4Fe(CN)_6$, 0.1 M
Aniline/BSA	Water	0.8 V, 60 s	H_2SO_4, 2 M

TABLE III
LIST OF LEDS USED TO OBTAIN THE WINE SPECTRA

LED #	Wavelength (nm)	Supplyer
Led 1	475	AGILENT
Led 2	568	HP*
Led 3	503	AGILENT
Led 4	639	AGILENT
Led 5	644	HP*
Led 6	628	AGILENT
Led 7	594	AGILENT
Led 8	635	HP*
Led 9	525	AGILENT
Led 10	585	HP*
Led 11	492	HP*

Transmittance spectra were recorded using a series of eleven LEDS, selected to cover the range from 780 to 380 nm (Table III). The spectrum of each wine was reconstructed using these values, and the CIElab coordinates were calculated accordingly [21].

Once the three parts developed were tested and optimized separately, common parts were built up. They included an autosampler that distributes a certain volume of each sample to each one of the measuring systems, and a common software that allows to collect the outputs of each sensor modality and to carry out the data treatment in a combined manner to form a multimodal feature vector.

The samples under study were six Spanish red wines from two regions (Rioja and Ribera de Duero) prepared from the same variety of grape (Table IV). The wines were classified as Young (no aging), Crianza (total aging of 24 months) and Reserva (total aging of 36 months). The Oenological Center of Castilla y León characterized the wines chemically and by a human panel test following international regulations [19].

Chemical analysis shows that the wines under study have similar characteristics. In particular, the six wines have similar ethanol content ranging from 12.2 to 14.5%vol. This is due to the fact that they are prepared not only using the same variety of grape (tempranillo) but also with a similar vinification method and, in the case of Crianza and Reserva wines, a similar aging process in oak barrels.

TABLE IV
WINE SAMPLES UNDER STUDY

Sample	Geographic origin	Vintage	Classification
W1	Rioja	2001	Young
W2	Rioja	1999	Crianza
W3	Rioja	1998	Reserva
W4	Ribera Duero	2001	Young
W5	Ribera Duero	1999	Crianza
W6	Ribera Duero	1998	Reserva

Seven different samples were extracted from each wine, and measured with each of the three sensory systems. Each of the seven replicates was measured on a different day. Principal component analysis (PCA) of the resulting database was carried out using Matlab software V 5.3.

III. RESULTS

A. Headspace Analysis

In order to reduce the presence of water and ethanol contents in the test chamber, the analysis of volatiles present in the headspace of wines was carried out by using a SPME preconcentration technique. Suitable SPME parameters, including absorption time, desorption time and desorption temperature, were determined by GM chromatography so as to eliminate up to 95% of water and ethanol content with a minimum loss of volatiles. Given that SPME requires the use of inert carrier gas such as nitrogen, only those sensors that showed good baseline stability (drift <5%) under nitrogen were selected for the analysis (Table I). Exposure of the gas sensor array to the aromas of wines produced an expected decrease in sensor resistance. The percentual changes were high, ranging from 50–80%, and reversible. A polar plot of the average signals of the sensors for two wine samples, ethanol and water is shown in Fig. 1 in terms of relative resistance changes. The contour of these polar plots differs from one wine to another, illustrating the discrimination capabilities of the array. The standard deviation (n = 7 samples) for the 14 sensors ranged between 1% and 12%.

The maximum height of each sensor transient was used to form a 14-dimensional feature vector. To avoid a bias toward sensors with intrinsically larger responses, each feature was first preprocessed with a conventional auto-scaling transform [22]

$$x_d^{(n} = \frac{x_d^{(n} - \mu_d}{\sigma_d}$$

where μ_d and σ_d are the mean and standard deviation of feature d across the entire dataset, and $x_d{}^n$ is the value of feature d of example n in the database. This same procedure was also employed for electronic tongue and color measurements.

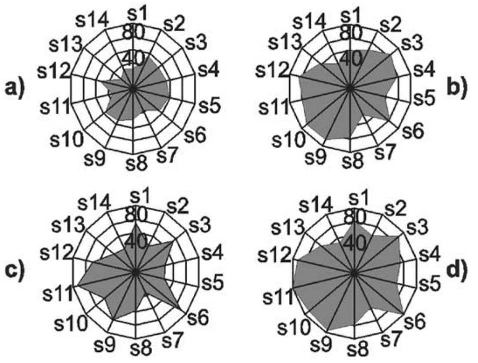

Fig. 1. Polar plots of the percentage variation in sensor resistance for (a) water, (b) ethano, (c) wine W4, and (d) wine W1.

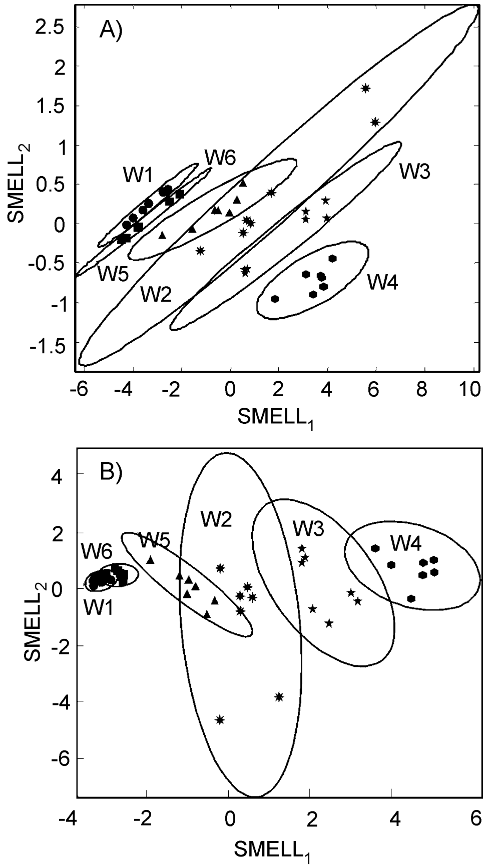

Fig. 2. Principal component decomposition of gas sensor-array data (a) before and (b) after multivariate drift reduction.

As a result of drift, the gas sensor responses were observed to be less repeatable than those from the other two modalities. For this reason, we employed a deflation procedure developed by Artursson *et al.* [23], which identifies the multivariate directions of drift as the first principal components from a calibration gas. Given a (row) feature vector x representing the response of the sensor array to a wine sample, the drift-compensation procedure is

$$x_{\text{drift-compensated}} = x - (x \cdot v_{\text{cal}})v_{\text{cal}}^T$$

where v_{cal} is the first eigenvector or loading (a column vector) of the covariance matrix of the calibration samples. To improve this drift-compensation procedure, two separate calibrants were used: distilled water and a 12%v/v dilution of ethanol.

PCA was carried out using feature vectors corresponding to the seven replicates in the database. Fig. 2 presents the PCA scatter before and after drift-compensation, where the

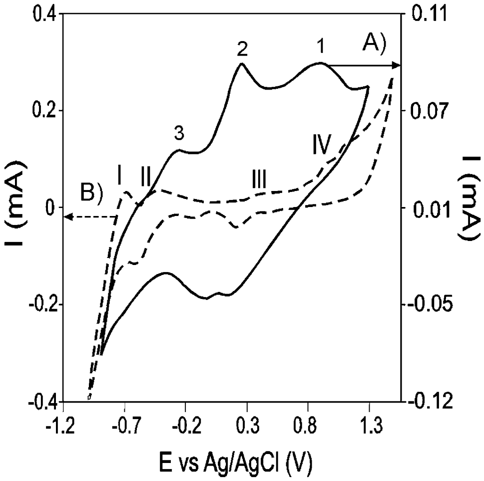

Fig. 3. CV of (a) $LuPc_2$ CPE and (b) P-3-MT immersed in W1.

ellipsoidal contours represent three standard deviations (Mahalanobis distance) from the mean. After drift-compensation, the first two principal components capture 92% of the variance (PC1:79%; PC2:13%). A certain degree of overlap between clusters is observed due to the similar characteristics of the wines under study.

B. Liquid Phase Analysis

The preparation method and the electrochemical characteristics of the liquid sensors have been previously published [13], [14]. It has been demonstrated that these electrodes show a rich electrochemical response toward model solutions of basic tastes. The response of the polymeric electrodes toward wines is illustrated in Fig. 3 for a P-3MT electrode immersed in wine sample W1. The curve is rather complex and shows a series of well defined peaks that can be associated to the oxidation and reduction of the polymer [18] (e.g., peak IV) or to redox processes related to the wine under study (e.g., peak II).

Sensors prepared from different polymers show similar behavior, but the curves differ in the position and the intensity of the peaks. This is illustrated in Table V, where the values of potential for peak I and for different sensing polymers exposed to wine W3 listed. Similarly, each sensor produced distinguishable electrochemical responses, in terms of shifts in the peak positions and changes in intensity, when exposed to different wines. Studies on the reproducibility of the sensors were also carried out. The voltammetric curves were highly reproducible, and only small shifts in the position of the peaks were observed (after ten consecutive scans CV 1–5%). Nevertheless, a gradual decrease in the intensity of the peaks could be noticed after consecutive scans.

The response of bisphthalocyanine-based CPE electrodes to the wines is also illustrated in Fig. 3. The graph represents the response of a carbon paste electrode of $LuPc_2$ when exposed to wine sample W1. In agreement with previously reported results [14], [17], the cyclic voltammogram presented the expected peaks corresponding to the one electron ring oxidation $Ln(III)Pc_2/Ln(III)Pc^+$ (peak 1) and one electron ring reduction $Ln(III)Pc_2/Ln(III)Pc^-$ (peak 3) of the phthalocyanine molecule. The presence of a peak at an intermediate voltage (peak 2) $E_{1/2} = 0.45$ V with definite anodic and cathodic waves can be attributed to electrochemical processes related to the wine. Sensing units with different selectivity toward liquid samples were prepared using different phthalocyanine molecules. The responses of $GdPc_2$ and $PrPc_2$ were similar, but the $E_{1/2}$ shifted to lower values when advancing in the lanthanide series. Table VI shows the values of peak 1 of the electrodes based on bisphthalocyanines when exposed to the six wines under study.

TABLE V
PEAK POTENTIALS (EXPRESSED IN VOLTS) OF PEAK I OBTAINED FOR POLYMERIC UNITS EXPOSED TO WINE W3

		P3MT	**PANI**	**PPY**	**PANI/ABS**
Peak I	Cathodic	-0.41	-0.78	-0.48	-0.68
	Anodic	-0.63	-0.89	-0.69	-0.77
	$E_{1/2}$	-0.52	-0.83	-0.58	-0.72

TABLE VI
POTENTIAL VALUES (IN VOLTS) OF PEAK 1 FOR $LnPc_2$ EXPOSED TO THE SIX WINE SAMPLES

Wine sample	$LuPc_2$	$GdPc_2$	$PrPc_2$
W1	0.921	0.967	0.972
W2	0.967	0.993	1.116
W3	0.985	0.984	1.086
W4	0.948	1.003	1.029
W5	0.915	0.941	1.006
W6	0.888	0.921	1.041

In addition, a certain sensor produces similar but distinguishable electrochemical responses when exposed to the different wines. The main differences rely in the intensity and the position of the peaks.

Subsequent scans were highly reproducible and the values of the potentials of peak 1 obtained from curves registered in consecutive scans, showed a coefficient of variation lower than 4%.

In order to explore the different sensitivities of the system, information from the phthalocyanine and conducting polymer sensors was combined into a feature vector, and processed with PCA (Fig. 4). In this case, the value of the potential of the anodic wave of peak 1 was used as the input feature for each sensor. As shown in the figure, the ellipses at three standard deviations are perfectly separated from each other with the exception of wines W5 and W1. The first two principal components capture 56% and 34% of the variance, respectively, indicating that the array of liquid sensors is less collinear that the array of gas sensors.

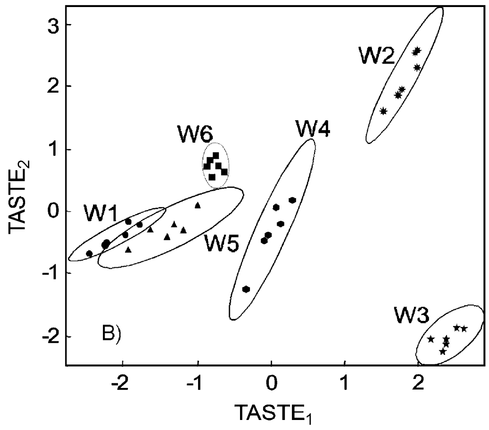

Fig. 4. Principal component decomposition of the liquid sensor-array data.

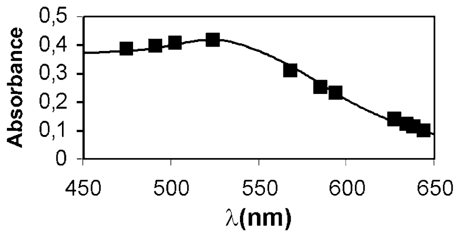

Fig. 5. Reconstructed spectrum for wine W5.

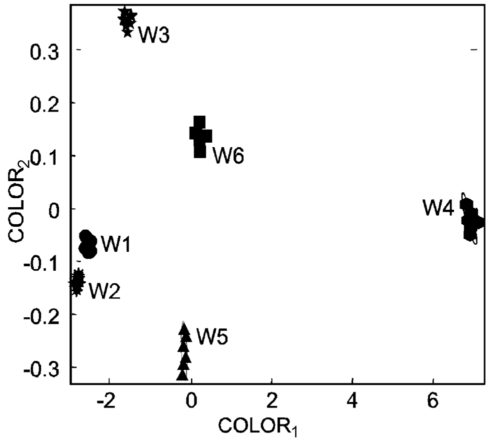

Fig. 6. Principal component decomposition of the LED-based color spectra.

C. Color Analysis

The LED system designed in our laboratory allows us to reconstruct the spectra of the wine samples under study. This spectral response is illustrated in Fig. 5 for a sample of wine W5. The 14 transmittance values of each wine sample were used to calculate the CIElab coordinates that were used as a feature vector for the multivariate analysis. The first and second principal components, shown in Fig. 6, capture 99.5% and 0.4% of the total variance, respectively. The PCA clusters are small , allowing for an easy discrimination of wines. In addition, the clusters corresponding to Ribera de Duero and Rioja wines appear in well-separated regions of the PCA scatterplot.

D. Sensor Fusion

It is interesting to note that, as illustrated in Figs. 2, 4, and 6, each instrument provides a complementary piece of information that aids in the discrimination of the six wines. For instance, wines that appear nearly overlapped in gas-sensor feature space can be easily discriminated with the liquid sensory system. These discrimination capabilities are further enhanced with the optical modality. It is also important to observe that the first principal component in the three instruments is clearly aligned with the direction of scatter between classes. This allows us to combine information from the three instruments into a three-dimensional vector consisting of each of these first principal components. This multi-modal representation is illustrated

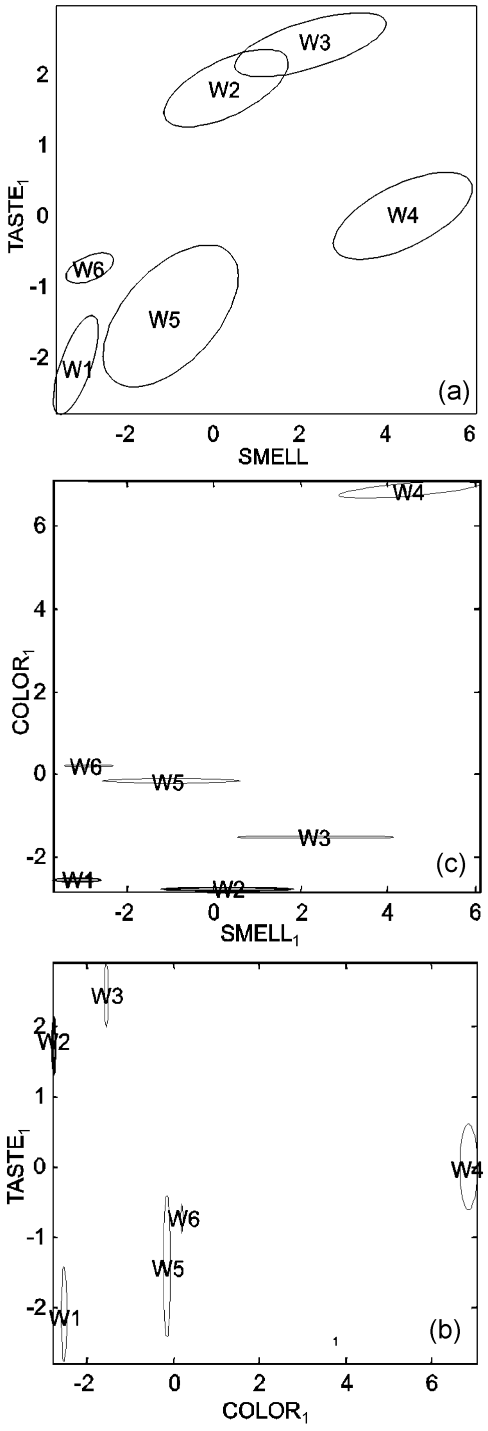

Fig. 7. Fusion of information from the three sensing modalities.

TABLE VII
CORRELATION COEFFICIENT BETWEEN THE DIFFERENT MODALITIES

	SMELL	TASTE	COLOR
SMELL	1.000	0.6106	0.6367
TASTE	0.6106	1.000	-0.1016
COLOR	0.6367	-0.1016	1.000

in Fig. 7 where samples have been replaced by an equiprobable contour at three standard deviations from the mean (Mahalanobis distance $d_{\mathrm{MAH}} = 3$).

Table VII presents the correlation coefficients between the three modalities, computed from the average response of each wine in Fig. 7. Color information is nearly uncorrelated with taste information. These results further support the notion that the three sensing modalities provide complementary discriminatory information.

IV. CONCLUSION

A system specifically designed for the characterization of wines has been designed. The proposed instrument categorizes wine samples using three types of sensory systems that evaluate the properties of the gas phase, the liquid phase, and the color of the wine samples separately.

In particular, an array of voltammetric sensors based on phthalocyanines and conducting polymers have been successfully used for the discrimination of wines. The differential cross selectivity displayed toward similar wines indicates that the array could be used for the discrimination of complex liquids. The discrimination capability of the system is significantly improved when the signals from each sensory subsystem are combined into a multimodal representation.

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