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First Results on the Use of Luminometry in the Consideration of the Stability of Flavourings during Storage

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Introduction

Numerous papers have been published on organoleptic alterations of flavourings induced by inappropriate containers; also on methods used to assess specific properties of packaging materials and modifications (1–18) caused by inadequate packaging and/or storage conditions used for flavouring products.

Appropriate packaging cannot be specified, in the case of flavourings, merely in terms of such container materials as are assumed to be adequate for shipping the product from manufacturer to user. As a matter of fact, the shipping container then becomes the storage container, storage time and conditions being known only to the user. Criteria ought to be adopted for the packaging of flavourings that should be drastically different from those applicable to the packaging of foodstuffs in general, because the inherent «aggressiveness» resulting from high concentrations of certain compounds that are typical of commercial flavouring products must be considered.

One of our current research efforts consists of the evaluation of simple analytical methods that would enable us to assess the behaviour of packaged and stored flavouring products. The goal, in other words, is to determine the significance of analytical findings and to correlate such findings with certain organoleptic evaluations.

Luminometry is one of the analytical methods used: there is reason to believe that several correlations may exist between luminometric data and certain involution phenomena, and that, in a broader meaning, information may be obtained about the status of packaged flavouring products while being stored.

This contribution contains the results of experiments concerning the storage of orange oil and lemon oil and follows a paper given at «Euro Food Pack» in Vienna (19).

Experimental

Analytical principles and methods

Luminescence is the generation of light by a succession of events producing single photons of light. Thus the analytical principle of luminometry is the mea-

surement of single photons of light emitted by the decay of excited species which might be produced by chemical or biological reactions.

The amount of light has been found to be directly proportional to the concentration of the reactants over a wide range of concentrations. The light may be measured with a luminometer. The luminometer, in its simplest form, is an instrument containing a light detection device that responds proportionately to the light produced by chemical reactions in a chamber observed by a measuring device. The Packard Pico-Lite uses a side-window photomultiplier tube to measure the light generated by reactants mixed directly in front of the photocathode in a 6 x 50 mm glass cuvette. In our case the essential oils drawn out at fixed times from several containers used for packaging and storage are the subjects of our measures. The use of luminometry in this case is justified by the following idea: if during storage, because of the catalytic effect due to transfer products from containers or to the action of light or temperature or to spontaneous instability of one or more components, excited compounds in the mass of the examined flavour are developed, it must be possible to measure the photons of light emitted and to correlate such measurements with the behaviour of the compound in the storage conditions considered.

The results obtained show that this method may provide information useful for the identification of the less suitable materials for containers (or of the less suitable storage conditions).

Instrumental

The block diagram of the Pico-Lite luminometer system is shown in figure 1. The Pico-Lite can be divided into the two units that make up the system: the analyzer and the detector.

The detector contains the photomultiplier tube, which detects the luminescent reactions, and a manual sample changer for six samples. The top knob includes syringe guides/ports and a septum, which allows the sample to be injected in complete darkness.

A heating element is included in the detector, which allows samples to be tested at controlled temperatures from ambient to 44 °C. For subambient temperature tests, the detector must be connected to a cold water source (bath).

The analyzer contains the microprocessor, which controls the various functions of the instrument. Sensor keys are used to set up the instrument to perform the timing, counting, calculating, etc. LED displays show count delay time, count time, set and actual temperature, program number, background subtraction number, normalization constant, accumulated counts, and sample sequential number. The analyzer has the capability for the operator to select any one of seven present counting programs. The analyzer controls allow the delay and count times and temperature settings to be adjusted. Sample volumes may range from 10 μl to 0.3 ml.

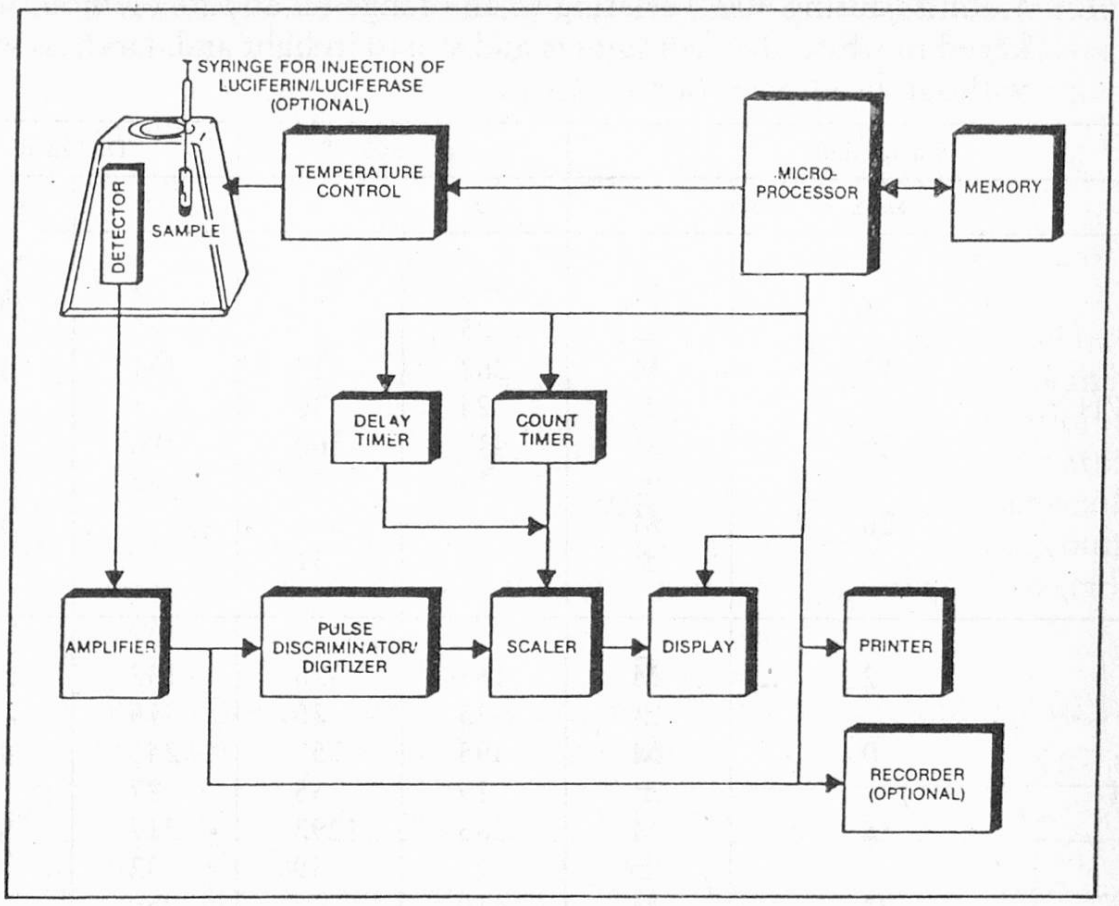

Fig. 1. Pico-Lite block diagram

Results and discussion

Some of the results that are reported here were obtained with a Pico-Lite 6100 luminometer (Packard Instruments C, Inc.), the purpose being merely to show the significance of the investigations and their potential value.

Counts are reported, in table 1, relating to an orange oil and to a lemon oil packaged in white glass containers and stored in light and darkness with headspace or without (air or CO_2).

The results may be interpreted if we compare separately the values for orange oil and lemon oil, respectively.

The data show in general the emission of a number of photons growing pari passu with storage time.

Growth was registered in all cases, although with wide variations with lower counts for all samples kept in the dark.

As CO_2 rather than air was contained in the headspace, counts were not reduced in samples exposed to light, whereas in samples kept in the dark, CO_2 kept counts to very low levels, comparable to those of products stored with no headspace.

Table 1. Counts (during 400") relating to an orange oil and to a lemon oil pakkaged in white glass containers and stored in light and darkness with or without headspace (air or Co_2).

Storage time			Light exposed		Darkened	
	Days		O	L	O	L
WH	6	$\overline{M}$	238	272	85	165
		$\pm$	25	35	25	22
	13	$\overline{M}$	262	362	151	426
		$\pm$	24	30	31	27
	20	$\overline{M}$	437	560	286	444
		$\pm$	16	57	48	17
	26	$\overline{M}$		558		630
		$\pm$		31		48
H	2	$\overline{M}$	165	326	132	288
		$\pm$	23	25	18	17
	9	$\overline{M}$	395	758	241	413
		$\pm$	19	38	27	44
	16	$\overline{M}$	366	1292	217	540
		$\pm$	17	49	32	61
	22	$\overline{M}$	410		250	
		$\pm$	31		23	
C	6	$\overline{M}$	226	440	126	305
		$\pm$	40	36	14	40
	13	$\overline{M}$	365	968	177	457
		$\pm$	37	48	11	15
	20	$\overline{M}$	462	1284	247	456
		$\pm$	12	73	29	38

WH = without headspace H = headspace (air) C = headspace (CO_2)

Counts relating to essential lemon oil stored in light, with or without air or CO_2, show the greatest increase.

With reference to this it is shown from the organoleptic point of view that
- samples exposed to light with a headspace suffered marked organoleptic degradation;
- light was a decisive factor for the degradation of the organoleptic characteristics, but to a different extent for both oils;
- CO_2 in the presence of light doesn't permit to stop the degradation of organoleptic characteristics.

Obvious correlations emerge, therefore, between luminometric determination and degradation: very low counts were invariably associated with satisfactory storage conditions, as photon counts were indicative of a very low state of excitation of the product's constituents at the time of measurement.

However, the significance of counts has to be correctly explained. In fact, a very low state of excitation (very low counts) may indicate that either the changes being examined had not started yet, or that they have ceased at the time of measurement.

With regard to this, the results shown in table 2 should be considered: the counts are given here, which were obtained from the same lemon and orange oils stored in contact with three different metals, the surface-to-volume ration being the same (0.412 sq cm/ml) in all three instances. The highest count was attained, after just a few days of exposure, by the samples packaged in the presence of copper: after 10 and 15 days respectively for orange oil and lemon oil, the count was down to undetectable values and at the same time the organoleptic degradation was at its highest.

Table 2. Counts (during 400") obtained from an orange oil and from a lemon oil stored in contact with three different metals.

Storage time		AISI 316		Fe		Cu	
Days		O	L	O	L	O	L
3	$\overline{M}$	230	615	118	482	149	1138
	$\pm$	21	22	38	32	11	36
10	$\overline{M}$	333	528	129	475	NR	361
	$\pm$	18	20	22	27		15
15	$\overline{M}$	400	670	127	558	NR	NR
	$\pm$	19	15	35	34		

NR = not detectable

This phenomenon can easily be commented on if we consider that the values of the emission-speed of photons becomes negligible after the completion of the chemical transformation that causes the emission itself.

As a matter of fact, in the case considered the count for the lemon oil, for example, shows very high values already at a time corresponding to 3 days. This demonstrates a degradative transformation, which is already finished after a very short time. On the contrary, counts for lemon oil samples in contact with AISI 316 steel were still quite similar to the initial levels as the tests ended.

The experience obtained suggests that counts should only be considered after they have been statistically analysed.

Conclusion

Many of the experiments, which have been realized and some of which are reported here enable us to conclude that luminometry may be regarded as unusual technique, which may make itself very useful in the study of the behaviour of certain flavourings during storage under different conditions.

Although the technique doesn't permit the study of the fundamentals of the phenomenon of the degradation, it permits us to define the conditions under which modifications occur more or less rapidly, relating these to the organoleptic quality of the sample. Therefore this technique is considered useful in the gathering of data, which permits to define the optimal storage conditions concerning packaging material, temperatures and time.

Summary

Several correlations may exist between luminometric data and certain «involution» phenomena and information may be obtained on the status of packaged flavouring products during storage.

Some of the results that were obtained with a Pico-Lite 6100 luminometer are reported.

Zusammenfassung

Es könnten mehrere Beziehungen zwischen luminometrischen Angaben und einigen Involutionsphänomenen bestehen. Informationen über den Zustand von verpackten eingelagerten Aromen können verlangt werden.

Einige Ergebnisse, die mit dem Pico-Lite 6100 Luminometer erzielt wurden, werden mitgeteilt.

Résumé

Il est possible de démontrer de nombreuses corrélations entre les valeurs de luminométrie et les phénomènes de dégradation des arômes au cours du stockage. Des informations y relatives peuvent être demandées.

Les résultats d'expériences réalisés avec le Pico-Lite luminomètre 6100 sont présentés.

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