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**A VERSATILE BURNER FOR
FLAME SPECTROMETRY**

by

G. ROSSI, P. BENETTI

(Euratom)

and

N. OMENETTO

(University of Pavia)

1971



Joint Nuclear Research Centre
Ispra Establishment - Italy
Chemistry Department
Analytical and Inorganic Chemistry

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Printed by L. Vanmelle, Ghent
Luxembourg, March 1971

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Luxembourg, March 1971 — 20 Pages — 7 Figures — B.Fr. 40,—

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Results are then presented to illustrate the burner performances in atomic absorption with multipass optical system, in flame emission and in atomic fluorescence. The comparison of the absorption and emission data with those given by other more conventional burners shows that comparable and better results are achieved with the described burner.

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* Work in part taken from the Master thesis of P. Benetti prepared during his undergraduate fellowship at the J.R.C. Ispra

ABSTRACT

A versatile short path water cooled laminar flow burner for flame spectrometry is described. The burner is mounted on a pre-mixing nebulizing chamber and can be safely operated with most of the analytically useful flames such as Air-diffusion/or N₂-diffusion/H₂, Air-H₂, N₂O-H₂, Air-C₂H₂ and N₂O-C₂H₂. A flame separation attachment is described by which a stream of an inert gas "lifts off" the secondary reaction zone from the interconal zone in which most of the measurements are made. The effect of the separation in terms of background and associated noise is discussed.

Results are then presented to illustrate the burner performances in atomic absorption with multipass optical system, in flame emission and in atomic fluorescence. The comparison of the absorption and emission data with those given by other more conventional burners shows that comparable and better results are achieved with the described burner.

KEYWORDS

FLAMES
SPECTROMETRY
FLUORESCENCE
ATOMIZATION
MEASUREMENTS
QUALITATIVE ANALYSIS

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INTRODUCTION *)

It is well known that the problem of atomization is the fundamental one in the techniques of atomic absorption, atomic emission and atomic fluorescence spectrometry. Doubtless a flame burning at the atmospheric pressure is not the ideal means of atomizing and several other techniques (1-7) have been tried in attempts to obtain atomic vapours from a large number of elements. Nevertheless, the flame remains the most used one essentially because of its simplicity and of the wide range of temperatures and gas environments which can be presently obtained.

The widespread use of new flame mixtures and consequently of new terminology could perhaps make some confusion in assessing the essential characteristics of the flame used. Therefore, a brief recall of the fundamental terms involved in the flame itself and in the equipment used to feed the flame with a sample solution will be given here.

When only stationary flames are considered, a general subdivision is made between diffusion and laminar flames. It is a common practice to consider diffusion flames as turbulent flames supported by a "direct injection or total consumption" burner and laminar flames as pre-mixed long path flames supported by an "indirect nebulizer" burner. As a matter of fact, however, it can be shown that also diffusion flames can be considered as laminar flames supported on pre-mixing chamber as well as pre-mixed flames can be supported on total consumption burners although they have experimentally shown a partial turbulent character. Therefore, it is no more allowed to connect diffusion flames with turbulent burners and laminar flames with pre-mix burners. This becomes evident when the wide variety of flames presently used and collected in Table I is considered.

All flames presented in this table have been evaluated in atomic absorption (AA), atomic emission (FE) and atomic fluorescence spectrometry (AF) (8-17). Long path laminar flames with pre-mix

*) Manuscript received on November 18, 1970

burners are almost exclusively used in atomic absorption while turbulent diffusion flames and laminar-turbulent diffusion flames are claimed to be superior for emission and fluorescence measurements.

The relative merits of laminar and turbulent flames as far as it concerns sensitivity, background, versatility, chemical interferences, stability, efficiency in use of sample etc. have been widely discussed. Surely one of the most striking advantages of the laminar flames consists in the fact that well defined reaction zones can be observed. In turn this allows one to study the complicated chemistry involved in radicals formation and sample atomization.

Moreover, one can take advantage of the possibility of separating with simple devices the secondary reaction zone from the interconal zone in which most of the spectroscopic observations are made. This effect combines a considerable reduction of background with an increase in the atom formation efficiency (18-23). On the other hand, it is also clear that there is no one flame and one burner design which can be considered of universal use in the three techniques. By taking into account the different requirements of the AA, FE and AF methods, one can derive the suitability of the essential features of the flame as shown in Table II.

From this Table it becomes apparent that the greatest flexibility is given by a short path laminar flame provided with flame separation possibility.

These considerations led us to realize a versatile burner with the following characteristics

- 1) easy in igniting various gas mixtures
- 2) laminarity in a wide range of gas flow
- 3) possibility of using laminar diffusion flames and pre-mixed laminar flames diluted with an inert gas
- 4) flame separation attachment
- 5) water cooling
- 6) safe in operation and noiseless
- 7) short path together with maximum flame density

EXPERIMENTAL

Burner design and characteristics

In designing a burner upon which a flame has to be stabilized, a detailed knowledge of the principles of fluid flow as well as of the reactive nature of the combustible mixture is required. It is not necessary to go here into the details of the fundamental parameters involved in this field because they were amply and clearly described in the literature (10). By taking into account the burning velocity, the stream velocity and the quenching effect of the walls in the case of the most critical gas mixture to be used ($N_2O-C_2H_2$), the calculations made led us to the burner design sketched in Fig. 1. Obviously, the same burner geometry will satisfactorily support most of the other analytical flames. The burner is made of a water cooled stainless steel tube having the following dimensions: ext. diam. 28 mm; int. diam. 14 mm; length 80 mm. The external diameter is reduced to 24 mm for a length of 22 mm at the lower extremity of the tube. A stainless steel cap which forms the burner head and contains five parallel slots 0.5 mm wide and 15 mm deep, is screwed on the upper extremity of the tube. The five slots were obtained by mating six machined and surface-ground stainless steel plates. Extreme care was taken to obtain surfaces as smooth as possible in order to avoid any turbulence in the slots and to assure the gas tight between the plates. More conventional burner heads (Meker or Lundegardh type) can be easily interchanged with the described head. Flame separation is achieved with a stream of argon or nitrogen flowing through a series of concentric circular slots (0.5 mm wide, 20 mm deep) surrounding the burner head. These slots were conveniently made with a series of brass rings held in position with a series of spacers (four for each brass ring) placed at 90° each other. The rings are soldered to a brass chamber in which the gas is introduced tangentially via two opposite copper tubes. The water cooling has been found effective in minimizing carbon deposits when using hydrocarbon flames. Moreover, as it is well known (10) a slightly more flexibility in

the size of the burner port and in the stream flow for a safe operation is achieved.

The burner can be easily fitted on any commercial premixing nebulizing chamber like Perkin Elmer or Techtron, although a home made premix expansion chamber provided with auxiliary supply of oxidizing gas has been used (Fig.2). Fig. 3 shows details of the burner head and separator attachment. Adjustments have been provided in order to move the burner both vertically and laterally for optical alignment.

Several flames have been tested with the described burner. Among these the laminar A/or N_2/H_2 diffusion flames, the Air- H_2 , N_2O-H_2 , Air- C_2H_2 and $N_2O-C_2H_2$. In the last two flames the secondary reaction zone is easily "lifted off" from the interconal zone with the separator device. All analytically useful flames are thus included. As far as the gases flow rates are concerned, the following pressures were maintained at the tank gauges: Air 45 psi, H_2 30 psi, N_2O 22 psi, C_2H_2 12 psi. The flow was then adjusted with a Jarrell-Ash gas control unit. Flash back were never experienced when the $N_2O-C_2H_2$ was lit via the Air- C_2H_2 flame.

The flame was imaged on the slit of a Jarrell-Ash 0.5 m Ebert monochromator equipped with variable slits and RCA 1P28 or 1P21 photomultipliers. Signals were amplified with a Keithley μ m ammeter and displayed on a 0-10 mv Bristol strip chart recorder.

Results and discussion

The usefulness of the separator device as far as it concerns the flame emissivity has been first checked with hydrocarbon flames. Only the principal and strongest molecular bands have been considered. The results are shown in Table III. Obviously the greatest advantage is achieved when the nitrous oxide acetylene flame is used. As already described in the literature the pale blue outer mantle is considerably "lifted off" and the red interconal zone is increased in length. This results in an enormous reduction of the OH emission, in a slight increase in the CN emission (when A

is used as separating gas) and in a lower background noise. As expected the lowering of background was not so significant when the Air-C₂H₂ flame was considered.

The different appearances of the separated flames as compared with the normal ones clearly results from the Figures 4-7.

Atomic absorption, emission and fluorescence measurements were performed for a few elements in order to have a rather complete assessment of the versatility of the described burner. Comparison has been made with conventional laminar burners (10 cm and 5 cm slot) for AA and FE. No comparison was possible for AF.

Atomic absorption measurements.

The flame obtained by the circular burner is tall and narrow and therefore it cannot provide ^{the} same analytical sensitivity as that supported by long path burners. Nevertheless, simple optical arrangements can be used in order to increase the effective absorption path length. Thus, a comparison has been made between a standard 10 cm slot Air-C₂H₂ burner and the circular burner equipped with five passes system. Fuel and oxidizing flows were varied as well as flame region was chosen until the maximum absorption signal was observed for both burners. The other parameters like monochromator slit, lamp current etc. were held constant. No scale expansion has been used throughout all measurements. The results obtained are presented in Table IV.

Some observation can be drawn from this table :

- 1) there is practically no difference between the absorption sensitivity given by the conventional burner and the circular burner with multipass system.
- 2) Flame separation does not result in a significant improvement in the absorption sensitivity. However it must be borne in mind that the greatest utility of the separated flame in AA is to be expected in the ultraviolet region where the absorption of the flame itself plays a much more important role.

Unfortunately no measurements could be performed in this region because of the lack of the primary sources.

Atomic emission measurements.

Recent work (11) has demonstrated that the use of a conventional 5 cm slot burner operated with Air-C₂H₂ and N₂O-C₂H₂ flames provides near optimum sensitivity and versatility in most flame emission work when compared to cylindrical shape burners. Therefore selected elements were nebulized in a conventional 5 cm slot burner and in the circular burner. All measurements were performed with the N₂O-C₂H₂ flame. The best region of observation experimentally found was imaged on the monochromator slit. Slit dimensions were retained unchanged for both burners while gas flows were adjusted for maximum response. The results are collected in Table V. No effort was made for giving detection limits for all elements investigated but care was taken in order that a direct comparison between the two burners could be evident together with an evaluation of the separator usefulness.

The conclusion which can be drawn from the Table is that both burners certainly provide comparable performances. However, for a comprehensive understanding of the comparison data, the influence of various factors on the individual results must be taken into account. Among the others the flame temperature (depending on flame composition) the background emission, the inherent noise and the atomization capability are playing a predominant role, although it is cumbersome to establish the resulting effect since, in some instances, they could mutually offset. For example, the temperature of the N₂-separated flame has been reported (22) to be some 300°C lower than the corresponding temperature of the normal flame, but the increase in the height of the reducing CN interconal zone results in an improved atomization efficiency.

Therefore in order to have a critical evaluation of the different signals obtained, the behaviour of each element in connection with the above-cited parameters should be investigated. However this study was considered out of the scope of the present work. It must be also noted that the same slit set-up was used only to establish a comparison of the relative emission intensities. As a matter of fact, when flame separation is used the lower background

and the associated lower noise would permit to use wider slits and higher gain settings. As an example, in the case of Molybdenum (3193 \AA) even if the magnitude of the signals obtained is the same, a 10-fold reduction of the background and associated noise has been observed with the separated flame. The obvious gain in terms of detection limit does not need to be stressed.

Atomic fluorescence measurements.

As mentioned before, it is not possible to draw a comparison between the performances of the described burner with those given by long path laminar burners.

On the other hand, it was felt unreasonable and hazardous to compare literature data since the role played in AF by the excitation source and optics is of a paramount significance.

The most attractive feature of the burner in respect of this technique lies in its versatility in supporting various flames, in the reduced scattering problems due to its sample feeding system and in the possibility of lessening the inconveniences due to the flame emission owing to the separator device. This will result in a wide flexibility in choosing the best flame environment for the various elements. For instance, the strongest Ga, In and Tl fluorescence has been observed in the Air-H₂ flame. On the other hand, the N₂ separated Air-C₂H₂ flame allowed the highest sensitivity to be reached for Cr and Fe. All these results were already reported in a previous paper (24) and will not be discussed here. At present, no data can be given on the described burner operated with the N₂O-C₂H₂ flame.

CONCLUSIONS

The preliminary results obtained in AA, FE and AF make the circular burner described of great versatility and usefulness in the flame methods.

The advantage of using the three techniques on the same burner

with sensitivities comparable to those obtained with widely used conventional systems in AA and FE is significant. All flame mixtures of analytical utility can be safely operated on the burner thus increasing its flexibility and allowing the analyst to choose the best flame for each particular problem.

Finally, the flame separation provided with it results in a combination of a high temperature, low background, reducing medium which favours the applicability of the flame methods to the refractory elements.

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TABLE I

STATIONARY FLAMES	DIFFUSION FLAMES	LAMINAR	$\left\{ \begin{array}{l} \text{A-H}_2 \\ \text{N}_2\text{-H}_2 \end{array} \right.$	$\left\{ \text{Not easily separated} \right.$
		TURBULENT	$\left\{ \begin{array}{l} \text{Air-H}_2 \\ \text{O}_2\text{-H}_2 \\ \text{O}_2\text{-C}_2\text{H}_2 \\ \text{N}_2\text{O-C}_2\text{H}_2 \\ \text{A/H}_2 \text{ entrained air} \end{array} \right.$	
	PRE-MIXED FLAMES	LAMINAR	$\left\{ \begin{array}{l} \text{A/H}_2 \\ \text{Air-C}_2\text{H}_2 \\ \text{N}_2\text{O-C}_2\text{H}_2 \\ \text{N}_2\text{O-H}_2 \end{array} \right.$	$\left\{ \text{Easily separated} \right.$
		LAMINAR-TURBULENT DIFFUSION	$\left\{ \begin{array}{l} \text{N}_2\text{O-C}_2\text{H}_2 \\ \text{N}_2\text{O-H}_2 \\ \text{Air-H}_2 \\ \text{O}_2\text{-H}_2 \end{array} \right.$	

TABLE II

Suitability of the essential characteristics for flame spectral methods

	Long path	Short path	Laminarity	Turbulency	Flame separation
AA	Excellent	poor *	very good	good	reasonable
FE	Good	very good	very good	reasonable	very good
AF	very poor	excellent	very good	good	very good

* except when special technics are used

TABLE III

Effects of separation on flame background

λ (A°)	Emitting species	Unseparated/separated intensity ratio		
		N ₂ -separated Air-C ₂ H ₂	N ₂ -separated N ₂ O-C ₂ H ₂	A-separated N ₂ O-C ₂ H ₂
2829	OH	2,17	29	20,5
2893	OH+NO	-	24,4	14,3
3064	OH	3	85	83
3089	OH	2,7	91	97
3360	NH	-	15	7,5
3435	CO (continuum)	2,6	-	-
3590	CN	-	1,2	0,42
3883	CN	-	1,3	0,42
4314	CH	2,3	-	-
4365	C ₂	1,5	-	-

TABLE IV

Comparison of atomic absorption sensitivity given by a standard 10-cm laminar flow burner and the circular burner with optical multipass system

Element	λ (A°)	Sensitivity *		
		Air - C ₂ H ₂		
		Laminar flow burner	Circular burner normal flame	circular burner N ₂ separated flame
Co	2407	0,18	0,26	0,18
Cr	3579	0,23	0,2	**
Cu	3247	0,08	0,08	0,08
Fe	2483	0,2	0,24	0,2
Mn	2795	0,08	0,1	0,08
Ni	2320	0,24	0,24	0,2

* The sensitivity is defined as the concentration (ppm) of the element giving 1% absorption (0,0044 absorbance)

** The fuel rich flame necessary could not be separated

TABLE V

Comparison of the emission values given by a standard 5-cm N₂O-C₂H₂ burner and the circular burner

Element	λ (A°)	Concentration $\mu\text{g/ml}$	Relative intensities (*)		
			5 cm slot	circular burner normal	circular burner A-separated
Al	3961	10	10,1	8	9,1
		50	50,5	38,6	45,5
		100	100	76	77,6
Cr (**)	4254	1	10	3,84	3,75
		5	51,5	19,9	18,9
		10	100	39,5	37,8
Mo	3193	10	12,7	11,4	10,6
		50	52	54,5	49
		100	100	108	103
Mo	3903	10	12,3	12,6	14,2
		50	52	58	67,5
		100	100	117	135
Ti	3998	10	11,6	12,7	16,6
		50	50	54,5	64,7
		100	100	119	140
V	4379	10	18	19,1	25,8
		50	57,7	76,5	97
		100	100	165	211

(*) instrumental sensitivity has been set in order to have a deflection of 100 divisions when aspirating the highest concentration with the conventional slot burner

(**) no valid explanation can be offered for the reduced sensitivity

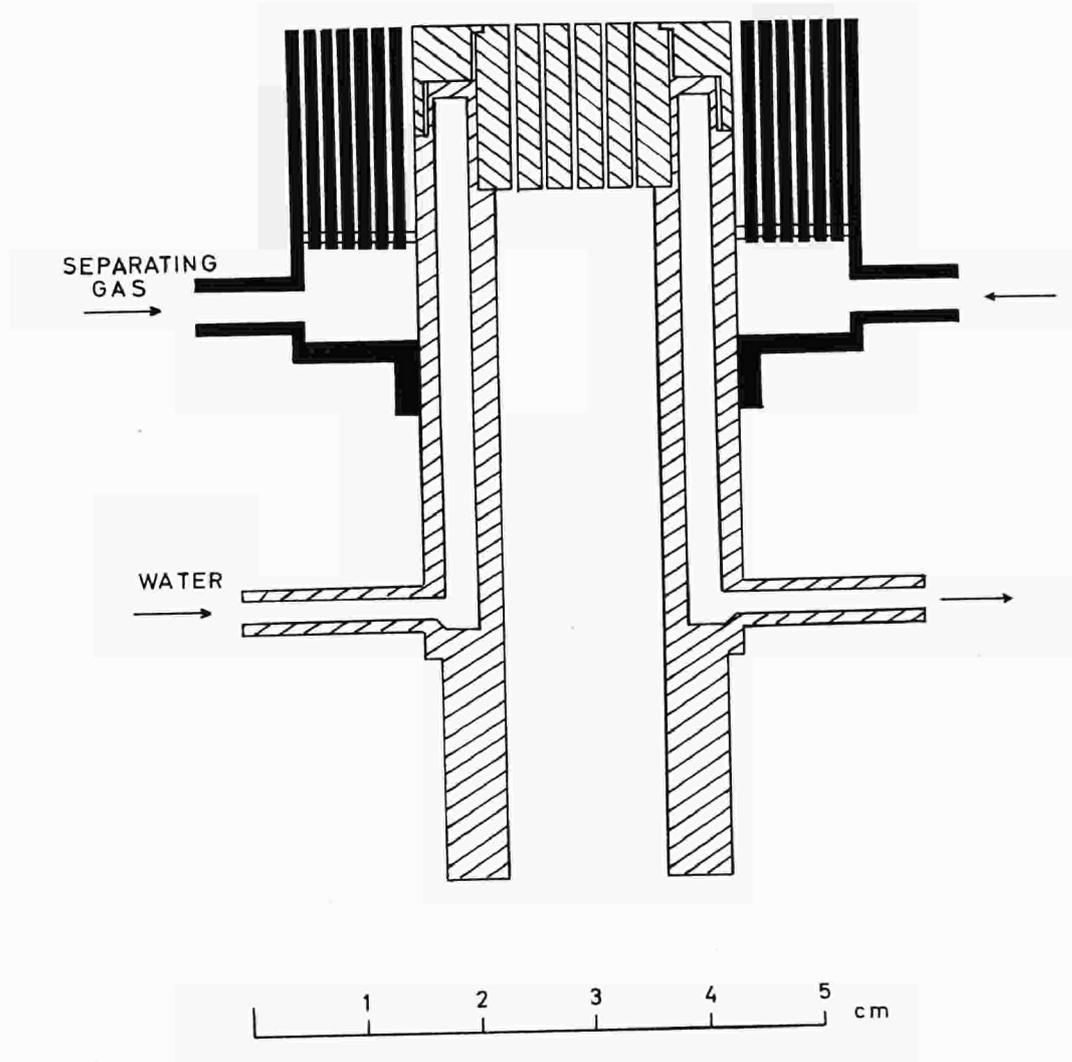


Fig. 1 : Cataway sketch of the burner

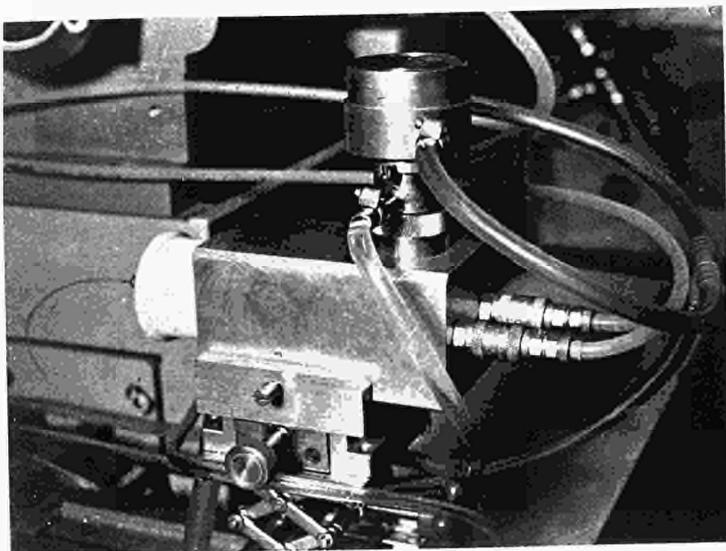


Fig. 2 : View of the burner assembly

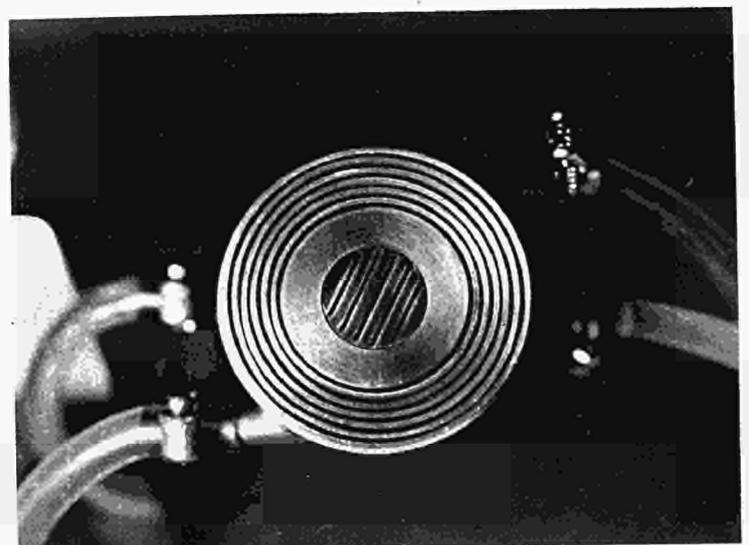


Fig. 3 : Front view of the burner head surrounded by the flame separator



Fig. 4 : Appearance of the normal Air/C₂H₂ flame
feeded with Lithium salts



Fig. 5 : Effect of N₂ separation on the flame of Fig. 4.
The lifting of the secondary reaction zone and the permanence of
the emitting atoms in the interconal zone is clearly shown

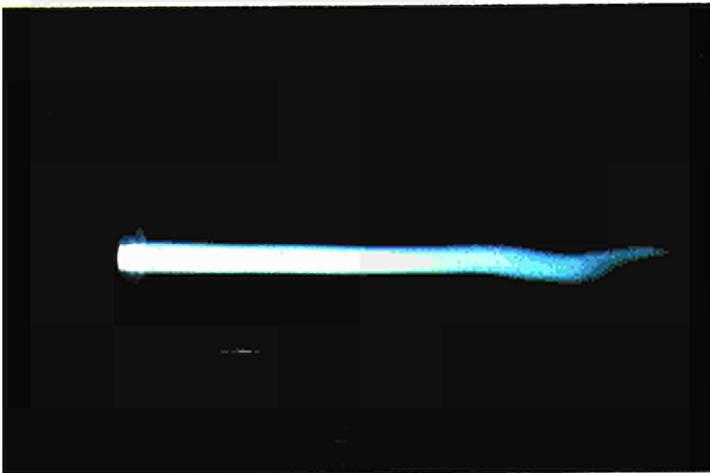


Fig. 6 : Appearance of the normal N₂O/C₂H₂ flame

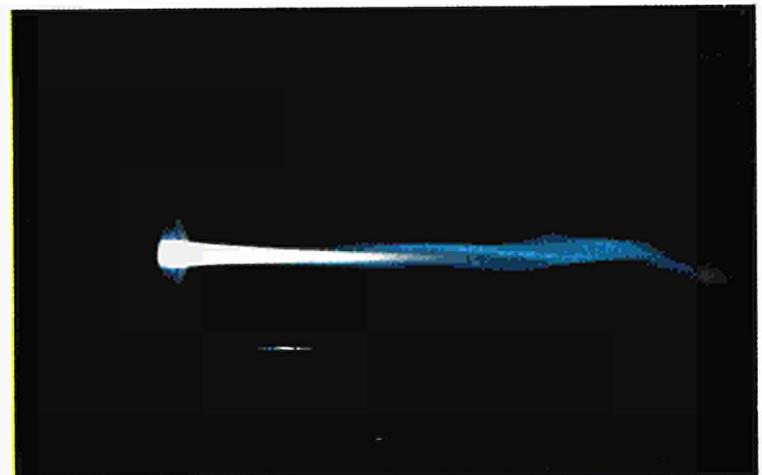


Fig. 7 : Effect of the N₂ separation on the flame of Fig. 6.
The pale blue outer mantle is lifted off while the interconal
reducing zone is markedly lengthened

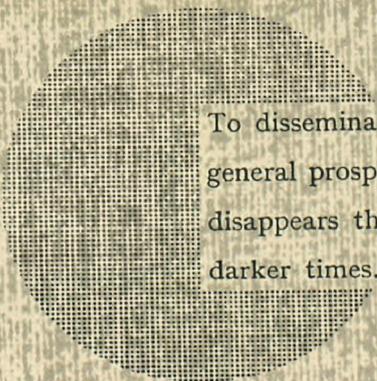
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Alfred Nobel

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