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IAFMM

# international association of fish meal manufacturers

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Our ref. SMB/RE/1990/12/
SCWGANAL+SCWGPROC

Your ref.

Date 22nd December 1989

## RESEARCH REPORT
## 1989-3

TO: ALL MEMBERS OF THE SCIENTIFIC WORKING GROUPS ON ANALYSIS AND PROCESSING

Dear Sirs

THE OXYGEN BOMB TEST

* I have pleasure in enclosing a copy of the Oxygen Bomb Test as submitted by Pesca Peru.

Yours faithfully
International Association of Fish Meal Manufacturers

S.M. Barlow
Director General

Encl.

# THE OXYGEN BOMB TEST

## A RELIABLE METHOD TO ASSESS OXIDATION STABILITY OF FISH MEAL
## A CONVENIENT PROCEDURE TO EVALUATE ANTIOXIDANT EFFECTIVENESS IN FISH MEAL

Submitted to the Scientific Committee Working Groups on Analysis and Processing of the International Association of Fish Meal Manufacturers IAFMM by PESCAPERU.

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Chemical Consultant
Representative of PESCAPERU

December 8, 1989

## INTRODUCTION

Oxidation stability of oils and fats is routinely evaluated by one of the following universally accepted methods:

a) SCHAAL TEST: Samples 50 grams each of the oil/fat are placed in several beakers in an oven at 60 C. Peroxide index of consecutive samples plotted against time will determine induction period. Duration of the test: several days.

b) SWIFT TEST: Air is bubbled through a sample of oil/fat at 100 C. Peroxide index of aliquotes plotted against time will determine induction period. Duration of the test: several hours.

c) THE RANCIMAT: Based on the Swift test, but instead of determining peroxide index, the head space gases (polar aldehydes and ketones from oxidizing fat) are bubbled in a water trap, where continuous conductometric evaluation versus time will readily indicate induction period. Duration of the test: several hours.

d) THE OXYGEN BOMB: A sample of the oil/fat is oxidized at 100 C with pure oxygen at high pressure. An automatic graph indicating pressure drop versus time is recorded from which the induction period can be easily determined. Duration of the test: several hours.

In the case of a fatty food, compound feed or fish meal, extracting the oil or fat in order to perform tests (a) or (b) would cause some peroxide decomposition. Moreover, separating the oil or fat from the other ingredients, like for example pro-oxidants (trace heavy metals, porphyrins, etc.), or antioxidant synergists (aminoacids, polar lecithins, etc.) would produce unrealistic stability results.
The RANCIMAT is non-applicable to fish meal since the high basic concentration of polar compounds coming from protein

degradation (volatile amines) would distort results.
Therefore, the only generally accepted method adequate to evaluate fish meal stability against oxidation and hence, the effectiveness of natural or added antioxidants, is the Oxygen Bomb test. (1) (2)
One of the advantages of using this method is that it evaluates the TOTAL effectiveness of antioxidants, whether naturally occurring or added. It also reflects antioxidant interaction with degradation products and synergism with other antioxidants. This is not achieved by just measuring antioxidant remnant concentration in fish meal. This measurement of antioxidant remnant concentration should not be used as a criterion for fish meal stability. (3) We think there are still a lot of unanswered questions in regard to the stability problem.
In our view, the only thing clear up to now is that we add antioxidants to stabilize fish meal to prevent quality detereoration, heating and/or spontaneous combustion.
One of the things that is not clear to us up to now is how we define what a stable fish meal is. We also seem not to have a consensus on a physical, chemical or the like method to prove or disprove if a fish meal is stable or not, not to mention if such method will enable us to make predictions about fish meal stability. All we know is, according to the IMO Code, that a fish meal is safe for maritime transportation when it has a remnant antioxidant concentration of at least 100 parts per million (ppm). Unfortunately, there seems to be no unanimous opinion yet as to what method should be used to measure the amount of remnant antioxidant in fish meal.
As far as we know, this 100 ppm figure has no technical or scientific justification. It was suggested to IMO by us back in 1973, 16 years ago, to move antioxidant treated fish meal from class 4 (spontaneously combustible) to class 9 which covers eventually dangerous materials. But time has shown, again and again, that this particular piece of regulation is unreliable and costly. It is unreliable, because, in some instances, fish meal has caught fire even when the Ethoxyquin remnant concentration was well above 150 ppm.
It is costly, because the fish meal industry in Chile, for example, has to add up to 1,200 ppm or more antioxidant to the fish meal in order to comply and be able to export. On the other hand, in Peru, since 1963, for 26 years to the present, based on the work of Flanzy et al (4) and especially that of the Eastman Chemical Company published as of 1954, we routinely perform Oxygen Bomb Tests with fish meal samples of export lots, and most especially with samples of either old lots, or lots in which eventual and sporadic heating has occured. The challenge of time has proved that we must be doing something right, and that the Oxygen Bomb Test is reliable to assess stability in fish meal, even in lots where the Ethoxyquin remnant concentration is well below 100 ppm.

## THE OXYGEN BOMB METHOD

The Oxygen Bomb Test, according to norm ASTM D-525 was adopted in 1964 to evaluate gasoline stability against accelerated oxidation by measuring induction period.
The induction period is the time elapsed between the moment in which the bomb is placed in boiling water and the time in the recording graph preceded by a drop

in pressure of 2 lb/in2 in 15 minutes, and followed by a drop in pressure of at least 2 lb/in2 in the next 15 minutes. This norm specifies using a 50 ml sample, 100 lbs/in2 initial oxygen pressure and 100 C temperature.

In 1963, CERPER, the Peruvian Government Quality Control Agency for Fisheries Products, adopted a similar procedure for testing the stability of fish meal, using following experimental conditions for the Oxygen Bomb Test:

Equipment: Oxygen Bomb plus automatic recorder, available from:

TESTING MACHINES INC.
400 Bayview Avenue
Amityville, New York 11701
Phone: (516) 842-5400 Fax: (516) 842-5220
Telex 96-1302
TMI number 16-00-00, Koehler p/n K10500 Oxidation Stability of Gasoline Bomb (double unit), composition gaskets, flexible seamless helical bronze tubing and two pen recorder, cost aprox.US$ 3,700.00

Fish meal sample: 60 grams
Initial Oxygen Pressure: 60 lbs/in2. When the Oxygen Bomb is placed thereafter in the boiling water bath, initial pressure rises to around 70 lbs/in2 in about half hour.
Duration of test: 3.5 hours
Stability criterion for fish meal: If oxygen pressure drops 10 lbs/in2 or more in any of the three hours following initial 0.5 hour, the fish meal is very reactive and needs to be reprocesed with additional antioxidant. If pressure drops less than 10 lbs/in2 per hour, the fish meal is stabilized, even if the antioxidant remnant concentration is less than 100 ppm.

Graphs Ia and Ib depict results of the Oxygen Bomb Test on untreated fish meal versus fish meal treated with 500 and 1000 ppm synergistic antioxidant blends.

The data to establish figures for the stability criterion for fish meal using the Oxygen Bomb Test was obtained in the early 1960's from several fish meal samples of lots that actually caught fire during maritime transportation. Since then, to our knowledge, none of the lots that passed this stability criterion, has ever caught fire during maritime transportation.

From the theoretical point of view, (5), one might be able to calculate the rate of acceleration of the fish meal oxidation in the Oxygen Bomb as follows:

a) The reaction among unsaturated compounds in fish meal and oxygen can be considered as second order, where:

$A + B = \text{Products}$, and

$$\text{rate} = -d[A]/dt = k.[A][B] \text{ moles/lit x sec}$$

$$\text{rate} = -d[O2]/dt = k.[O2][\text{fat}] \text{ moles/lit x sec}$$

Therefore, the rate of reaction is directly proportional to the oxygen concentration or pressure. Since the initial reaction is carried out at around 70 lbs/in2 equivalent to 5 times atmospheric pressure (14.5 lbs/in2), and since pure oxygen is used in the test, that is, at 5 times greater concentration than in air, then, the rate of reaction will be increased by a factor of 5 x 5 = 25.

b) The variation of the reaction rate with temperature is given by the Arrhenius equation:

$$k = A\, e^{-E/RT}$$

This equation is valid for all heat promoted reactions, whether homogeneous or heterogeneous, catalyzed or not, in solution or in the gas phase. From this equation, we can calculate that for every 10 C temperature rise, the rate constant k is doubled. Therefore, k, from a reaction at room temperature (20 C) will increase its value at 100 C like two to the eight, that is:

$$2^8 = 256$$

Consequently, the total increase in rate for the reaction of oxygen with fish meal during the Oxygen Bomb Test is:

$$25 \times 256 = 6{,}400$$

## ADVANTAGES OF THE OXYGEN BOMB METHOD OVER THE MEASUREMENT OF ANTIOXIDANT REMNANT CONCENTRATION TO EVALUATE POSSIBLE DANGER OF FISH MEAL HEATING OR AUTOCOMBUSTION.

a) The analytical determination of the antioxidant remnant concentration in fish meal implies, at best, a STATIC concept in the determination of its stability, because it only provides you with a figure on the amount of antioxidant in ppm's in a given moment, with no further information as to the fish meal's resistance or readiness to react with oxygen. Moreover, the semi-sinusoidal variation of the remnant antioxidant concentration with time is well documented (6) so that the result of one such determination cannot be properly located in said curve (ascending or descending ?) without performing a series of analysis during a time span for the same sample.
In contrast, the Oxygen Bomb Test offers a DYNAMIC follow up of the fish meal oxidation process under accelerated conditions, allowing, not only to determine in a reliable way its stability at a given moment, but also enabling us to make certain predictions of its oxidation behaviour in the future. In fact, (1 b) the peroxidation rate of fish meal fat inside the Oxygen Bomb is three to four times greater than the one observed for its fat during the Swift test.

Laboratory work done at CERPER confirms that there is a straight line relationship between oxygen absorption by fish meal inside the Oxygen Bomb and Ethoxyquin loss in a given time. Four samples fish meal in duplicate were submitted to the Oxygen Bomb Test. Besides measuring oxygen pressure drop versus time, the Ethoxyquin remnant concentration was measured at the beginning of the test, after one hour and after three hours in the duplicate sample. Following results were obtained:

| SAMPLE | INITIAL PPM ETQ | PPM ETQ AFTER 1 HR O/B | LB/IN2 PRESS.DROP 0.5-1.5 HR | PPM ETQ AFTER 3 HRS O/B | LB/IN2 PRESS.DROP 0.5-2.5 HR |
|---|---|---|---|---|---|
| A | 169 | 103 | 6 | 66 | 12 |
| B | 135 | 53 | 5 | 21 | 10 |
| C | 93 | 40 | 8 | 14 | 17 |
| D | 42 | 22 | 9 | 10 | 22 |

In Graph II a) we can see the relationship between pressure drop in lbs/in2 between 0.5 and 2.5 hours Oxygen Bomb Test.

In Graph II b) we can see the relationship between total Ethoxyquin concentration in ppm and time elapsed in the Oxygen Bomb Test.

In Graph II c) we can see the relationship between TOTAL Ethoxyquin LOSS in ppm's in 3 hours and pressure drop in lbs/in2 between 0.5 and 2.5 hours Oxygen Bomb Test for the four samples.

b) If even a fraction of a fish meal lot has been inadequately treated with antioxidant during production, for example, due to clogging of the spray nozzle, to lack of antioxidant in the dispensing tank, to maladjustment of the dosifying screw, to high viscosity of the antioxidant at low ambient temperatures, to crystallization of antioxidant in the tank or connecting pipes, etc, etc, that particular lot (and in a ship's hull, the entire cargo) is in imminent danger of catching fire. Before shipment, a representative sample of this lot will contain fish meal particles with and without antioxidant. The remnant antioxidant concentration determination will give you an AVERAGE figure without any indication of the inherent danger. In contrast, performing the Oxygen Bomb Test will immediately warn you, through the excessive oxygen pressure drop, that the fish meal is potentially dangerous during storage and/or maritime transportation.

c) The Oxygen Bomb Test enables us to measure the effectiveness of added antioxidants to fish meal, whether only one is added or whether a synergistic blend of antioxidants is used. The Oxygen Bomb Test quantifies the TOTAL effect of said stabilizers in fish meal, their synergism, antagonism, interaction with their degradation products and with other naturally occuring compounds. This cannot be said from the determination of antioxidant remnant concentrations because, even though we could find out said concentrations, neither synergism nor antagonism would be accounted for in such data.

PRELIMINARY CHILEAN RESULTS USING THE OXYGEN BOMB TEST VERSUS ANTIOXIDANT REMNANT CONCENTRATION TO ASSESS STABILITY OF FISH MEAL TREATED WITH PURE ETHOXYQUIN AND WITH SYNERGISTIC BLENDS OF ETHOXYQUIN AND BHT

During the 29th Annual Conference of the IAFMM, the Chilean delegation informed the Working Committe on Processing on some short and long term results using the oxygen bomb test versus antioxidant remnant concentration to assess stability of fish meal treated with pure Ethoxyquin and with synergistic blends of Ethoxyquin and BHT.

If, in said Chilean short term experiments, you carefully note the oxygen absorption rate of both treated fish meals, you will find out that the meal treated with the synergistic blend of Ethoxyquin and BHT absorbed 43% LESS oxygen in the Oxygen Bomb Test than the fish meal treated with pure Ethoxyquin. Thus, following experimental results performed on September 25, 89 were reported to CORPESCA by the Catholic University in Valpariso, Chile:

| | TIME | PSI | PRESSURE DIFF. EACH FULL HOUR |
|---|---|---|---|
| ETQ TREATED | 10.00 | 60 | |
| FISH MEAL | 10.30 | 70.8 | |
| | 11.00 | 69.6 | |
| | 11.30 | 66.4 | 4.4 |
| | 12.00 | 63.2 | 6.4 |
| | 12.30 | 59.4 | 7.0 |
| | 13.00 | 55.5 | 7.7 |
| | 13.30 | 51.1 | 8.3 |

TOTAL PRESSURE DROP: 33.8 PSI

| | | | |
|---|---|---|---|
| SYNERGISTIC | 3.30 | 60 | |
| ANTIOXIDANT | 4.00 | 71.2 | |
| BLEND TREATED | 4.30 | 70.6 | |
| FISH MEAL | 5.00 | 68.9 | 2.3 |
| | 5.30 | 65.6 | 5.0 |
| | 6.00 | 63.9 | 5.0 |
| | 6.30 | 61.9 | 3.7 |
| | 7.00 | 60.6 | 3.3 |

TOTAL PRESSURE DROP: 19.3 PSI

This, of course, indicates to us that the fish meal treated with the synergistic blend is more stable than the one treated with pure Ethoxyquin. Thus, the correct conclusion would be that in short term, experiments indicate that the synergistic blend is performing better as an effective antioxidant than pure Ethoxyquin.

For the abovementioned Chilean short and long term experiments, you cannot possibly use the concept of remnant antioxidant concentration, in this case Ethoxyquin, to compare stability, because in the synergistic blend series, you are starting

with ONE THIRD the amount Ethoxyquin than the standard fish meal used for comparison. It is only logical to expect that the fish meal with ONE THIRD the amount of Ethoxyquin will run out of it sooner. The remarkable thing is that the fish meal treated with the synergistic antioxidant blend of Ethoxyquin and BHT showed still good stability during the first hour on the Oxygen Bomb Test after nine months production.
Notwithstanding, the Chilean long term experiments conclusively show, as you can see from Graph III, that by using synergistic blends of antioxidants, savings in Ethoxyquin in the order of 700 grams per metric ton of fish meal can be achieved:

| | DAYS | | | | |
|---|---|---|---|---|---|
| PPM ETHOXYQUIN CONSUMPTION | 0-60 | 60-120 | 120-180 | TOTAL | SAVINGS |
| PURE ETQ/sacked fish meal | 748 | 142 | 226 | 1,116 | |
| PURE ETQ/pelletized fmeal | 1,003 | 90 | -13 | 1,080 | |
| SYNERG.BLEND sacked fmeal | 310 | 69 | 42 | 421 | 695 |
| SYNERG.BLEND pelletizd fm | 267 | 17 | 24 | 308 | 772 |

CONCLUDING REMARKS:

Based on our own laboratory and field results, PESCAPERU has stabilized over 100,000 metric tons fish meal, or roughly one third of our yearly production, with synergistic antioxidant blends of Ethoxyquin and BHT, and used the Oxygen Bomb Test to assess its stability. It seems to us, obviously, that fish meal can be stabilized by antioxidant synergistic blends, that the consumption of Ethoxyquin is much lower, and that there are significant safety and economical advantages for the fish meal industry to be using them.
Regardless of the outcome of additional Chilean experiments, no matter how many times we repeat laboratory or field experiences and come to right or wrong conclusions, the fact is that since 1963 Peru has successfully used the Oxygen Bomb Test to assess oxidation stability of fish meal, and since 1986 Peru has used, and still uses, synergistic antioxidant blends in Peruvian fish meal, and has had no known problems neither during maritime transportation nor in its use by customers at final destinations all over the world.

SUMMARY OF SUBMISSION:

For safety and economical reasons, PESCAPERU submits to the IAFMM Scientific Committee Working Groups on Analysis and Processing a proposed ADDITION (this is NOT a modification) to the IMO Code to regulate the maritime transport of fish meal treated with synergistic blends of permitted antioxidants as follows:

- on page 9020-2 of the IMDG Code (Class 9), (see attached), in Observations (a), between, "Stabilization of fish meal should be achieved to prevent spontaneous combustion:
  by effective application of between 400 and 1000 mg/kg (ppm) Ethoxyquin, or between 1000 and 4000 mg/kg (ppm) BHT (butylated hydroxytoluene)",

and,

"at the moment of production.",

insert:

"or any proven effective synergistic blend thereof"

also, after, "Antioxidant remnant concentration should be no less than 100 mg/Kg (ppm) at the time of shipment."

add:

"When synergistic antioxidant blends are used instead, in lieu of the 100 ppm remnant concentration, a representative sample of the fish meal lot should pass the Oxygen Bomb Test."

- on same page, in Observations (b), between, "anti-oxidant concentration at the time of shipment should be stated and must exceed 100 mg/Kg (ppm),"

and,

"packing, number of bags and total weight of consignement,"

insert:

"or, if the fish meal was treated with a synergistic antioxidant blend instead, in lieu of antioxidant concentration, the certificate should state that the fish meal was stabilized with an adequate amount of a proven effective synergistic blend of Ethoxyquin and BHT, and is not prone to spontaneous combustion as assessed by the stability test with the Oxygen Bomb,"

PESCAPERU seeks approval of said proposed addition to the IMO Code by the Scientific Committee of the IAFMM and requests support from IAFMM Headquarters, Potters Bar, in obtaining further approval by IMO in London.

ATTACHED GRAPHS:

Ia and Ib: Oxygen Bomb Test results on untreated fish meal versus treated with 500 and 1,000 ppm antioxidant synergistic blends.

IIa,b + c: Relationships between antioxidant (Ethoxyquin) remnant concentration, Ethoxyquin loss, time and oxygen absorption in fish meal during the Oxygen Bomb Test.

III : Ethoxyquin consumption in fish meal in Chilean long term experiments.

IV : Page 9020-2 of the IMDG Code (Class 9).

REFERENCES

(1) a) E.Bennett et al, "Studies of the Oxygen Bomb Method for determining shortening stabilities", JAOCS Vol.41, 505-507 (1964)

b) Cooper et al, "An improved apparatus for Oxygen Bomb Testing" JAOCS Vol 56, 1-5 (1979) and references therein.

(2) H.Pardun, "Methoden zur Bestimmung der Haltbarkeit von Fetten", Suesswarentechnik 3, 25-30 (1980).

(3) G.Vargas, "OXIQUIN SUPER, Nuevo antioxidante sinergista para harina de pescado", PESCA, Lima, pgs.14-17, Jul-Aug 1988.

(4) a) J.Flanzy, G.Rocquelin, A.Pihet, "Mesure de l'Absorption d'Oxygene par les Farines de Poisson. Application a leur Stabilisation par les Antioxygenes", Ann.Zootech., 1962, 11 (4), 263-272.

b) Eastman Chemical Products, Inc., Kingsport, Tenn.:

- "TENOX BHT IN FISH MEALS", Eastman Customer Service Laboratory, Brochure published ca.1954

- Publication No.ZG-195, page 7, June 1973.

(5) B.Stevens, "Chemical Kinetics", Chapman-Hall, London 1970.

(6) A.A.Spark, "Ethoxyquin in Fish Meal", 23 rd FIRI Annual Report (South Africa), page 59 (1969).

Photocopies of these reference articles are available upon request. Please contact:

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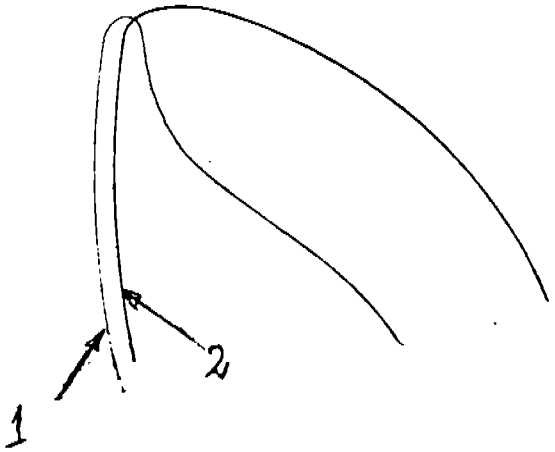
GRAPH I (a)

OXYGEN BOMB TEST

Sample 1 : Fish meal with no antioxidant

Sample 2 : Fish meal treated with 500 ppm synergistic antioxidant blend.




Empresa Pública de
CERTIFICACIONES PESQUERAS DEL PERU

Prueba : ABSORCION DE OXIGENO (BOMBA DE OXIGENO)

Muestra: Nº 1 Harina sin Antioxidante
Nº 2 Harina con Antioxidante (500 ppm.)
Antioxidante : "OXYKING"

16.01.86

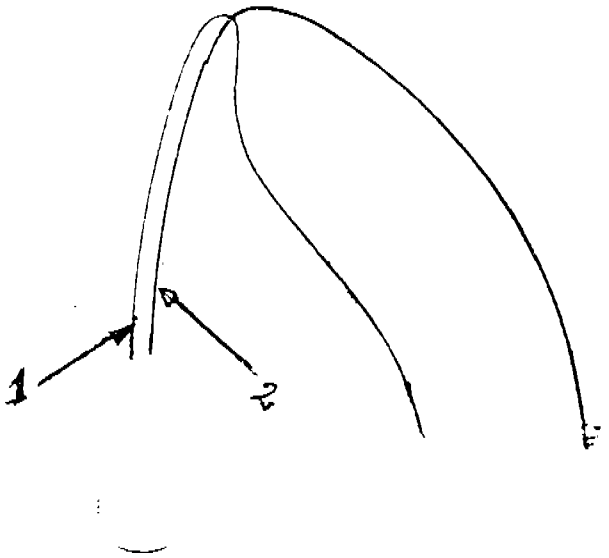
Dpto. ANALISIS Harina Pescado
16 ENE 1986
ENTREGADO

GRAPH I (b)

OXYGEN BOMB TEST

Sample 1 : Fish meal with no antioxidant

Sample 2 : Fish meal treated with 1,000 pp
synergistic antioxidant blend.



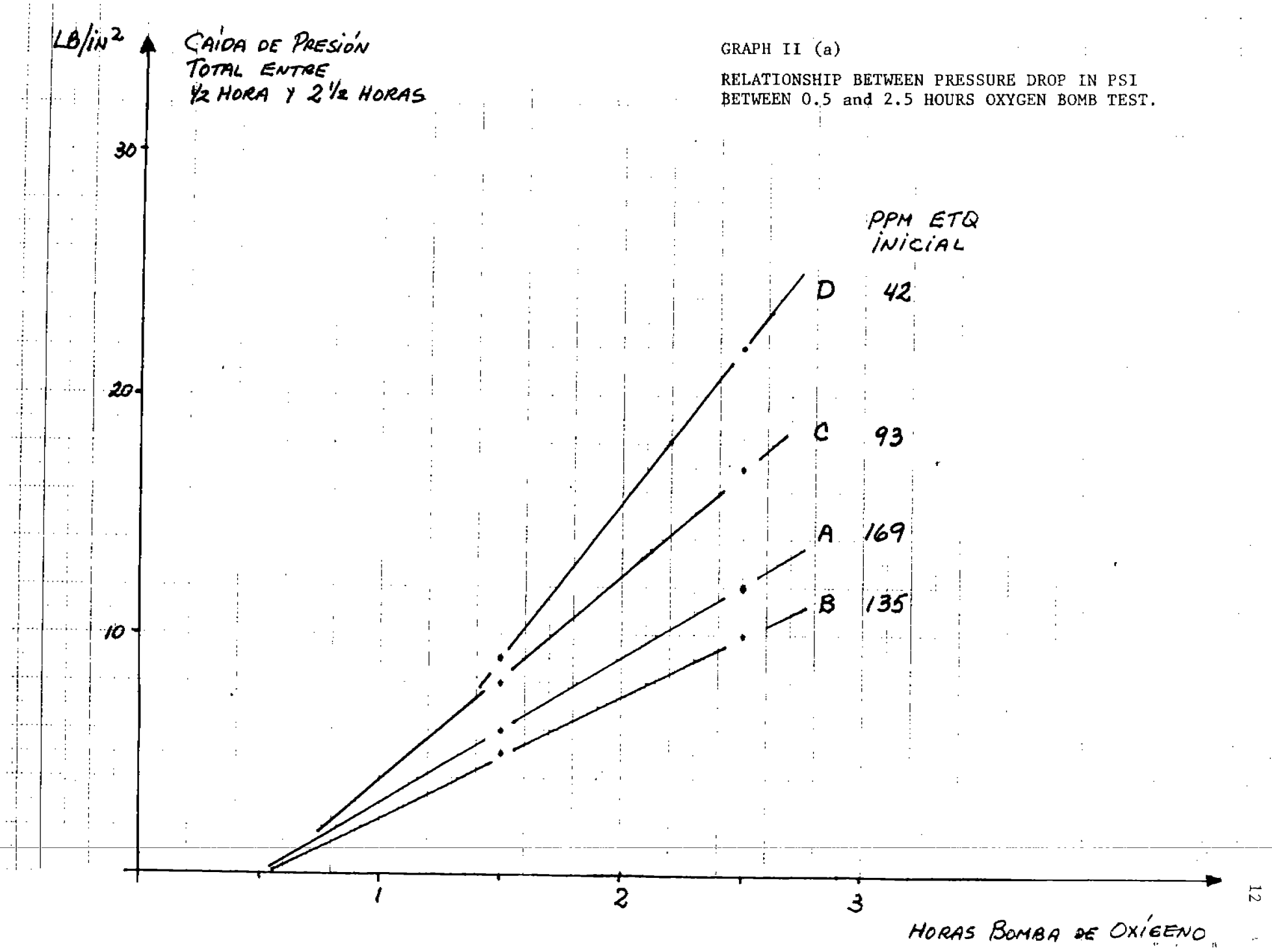



Empresa Pública de
CERTIFICACIONES PESQUERAS DEL PERU
Prueba : ABSORCION DE OXIGENO (BOMBA DE OXIGENO)
Muestra: Nº 1 Harina sin Antioxidante
Nº 2 Harina con Antioxidante (1,000 ppm.)
Antioxidante :"OXYKING"

15.01.86

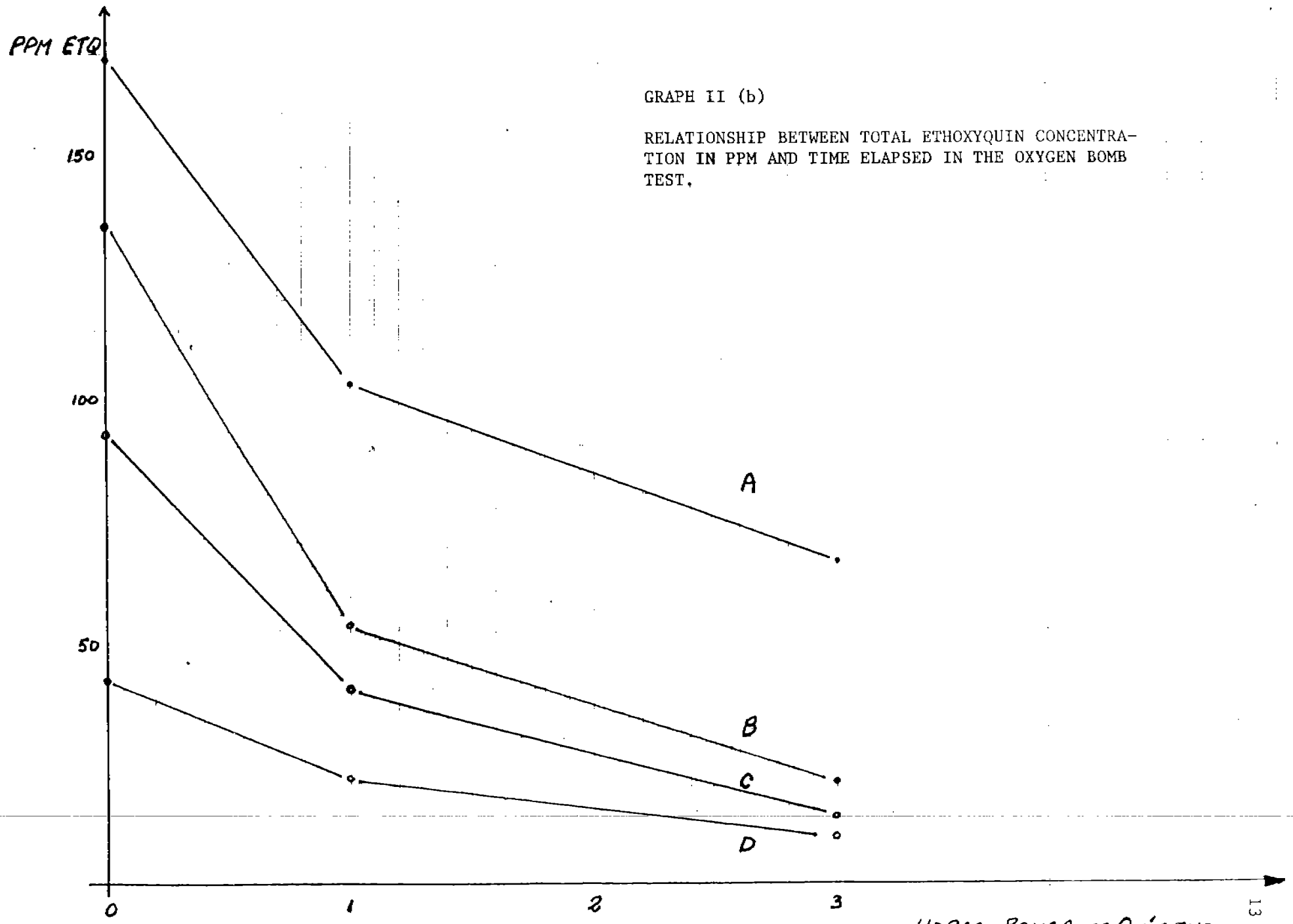
GRAPH II (a)

RELATIONSHIP BETWEEN PRESSURE DROP IN PSI BETWEEN 0.5 and 2.5 HOURS OXYGEN BOMB TEST.



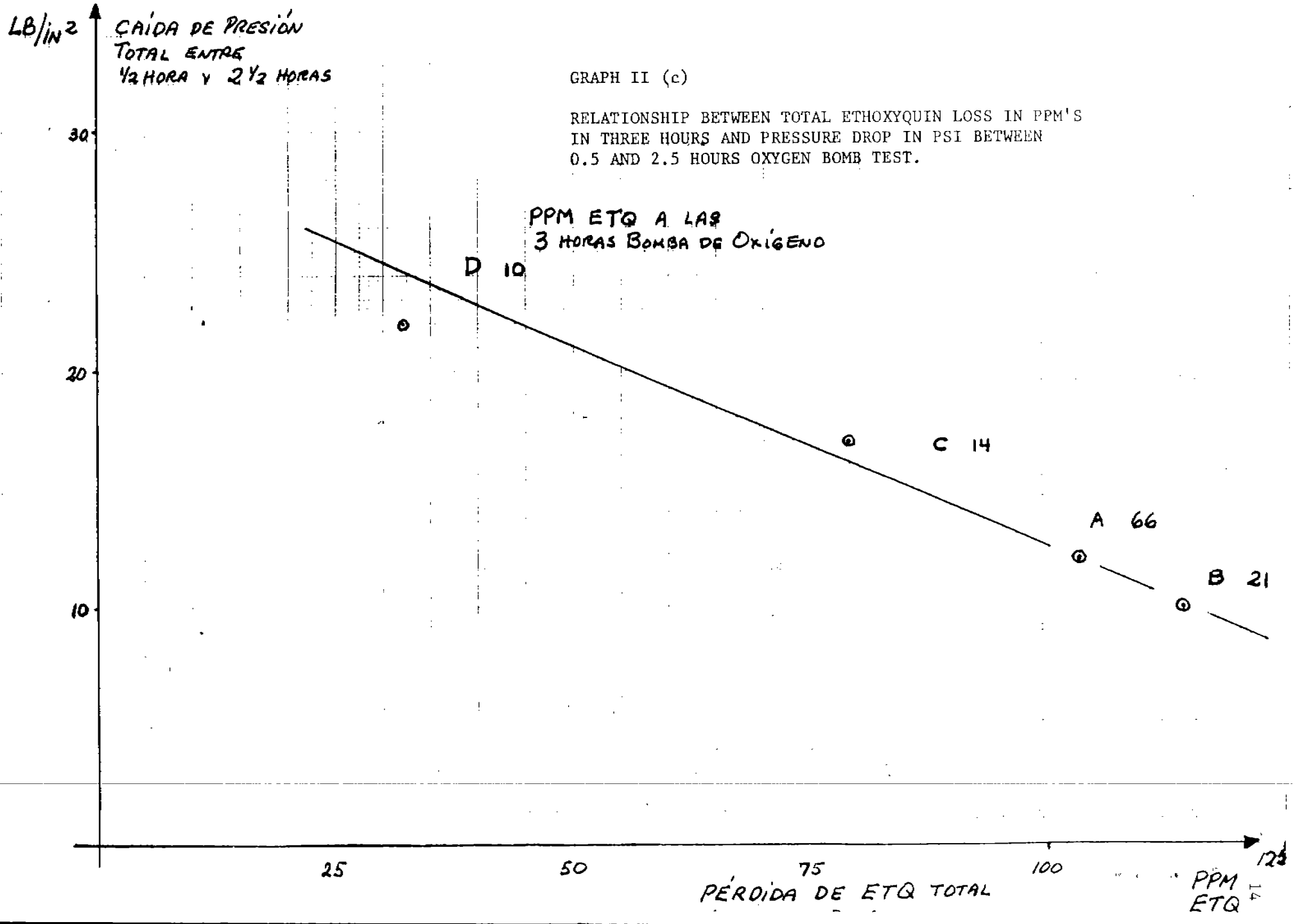
GRAPH II (b)

RELATIONSHIP BETWEEN TOTAL ETHOXYQUIN CONCENTRATION IN PPM AND TIME ELAPSED IN THE OXYGEN BOMB TEST.



GRAPH II (c)

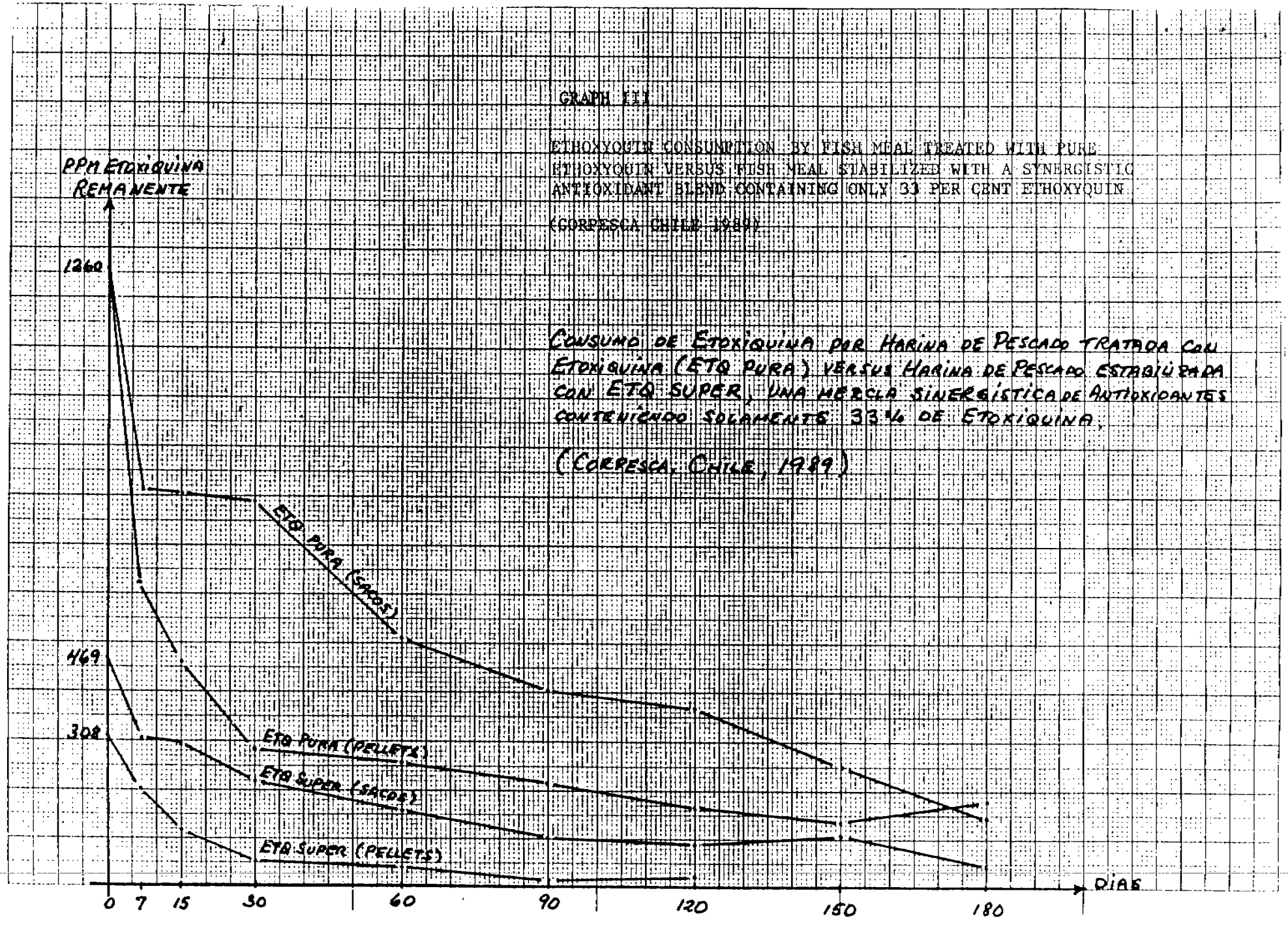
RELATIONSHIP BETWEEN TOTAL ETHOXYQUIN LOSS IN PPM'S IN THREE HOURS AND PRESSURE DROP IN PSI BETWEEN 0.5 AND 2.5 HOURS OXYGEN BOMB TEST.



GRAPH III

ETHOXYQUIN CONSUMPTION BY FISH MEAL TREATED WITH PURE ETHOXYQUIN VERSUS FISH MEAL STABILIZED WITH A SYNERGISTIC ANTIOXIDANT BLEND CONTAINING ONLY 33 PER CENT ETHOXYQUIN

(CORPESCA, CHILE, 1989)




CONSUMO DE ETOXIQUINA POR HARINA DE PESCADO TRATADA CON ETOXIQUINA (ETQ PURA) VERSUS HARINA DE PESCADO ESTABILIZADA CON ETQ SUPER, UNA MEZCLA SINERGÍSTICA DE ANTIOXIDANTES CONTENIENDO SOLAMENTE 33% DE ETOXIQUINA.

(CORPESCA, CHILE, 1989)

FISHMEAL,
FISHSCRAP,
(d) Anti-oxidant treated,
Moisture content: between 5% and 11%, by weight.
Fat content: not more than 18%, by weight

UN No. 2216

Formula

Properties

Brown to greenish-brown product obtained through heating and drying of fish.
Strong odour which may affect other cargo.
Liable to heat spontaneously unless of low fat content or effectively anti-oxidant treated.

**Observations**

(a) Stabilization of fishmeal should be achieved to prevent spontaneous combustion:
by effective application of between 400 and 1000 mg/kg (ppm) ethoxyquin, or between 1000 and 4000 mg/kg (ppm) BHT (butylated hydroxytoluene) at the moment of production.
The said application occurring no longer than twelve months prior to shipment.
Anti-oxidant remnant concentration should be not less than 100 mg/kg (ppm) at the time of shipment.

(b) Certificates from a recognized authority should state:
moisture content,
fat content,
details of anti-oxidant treatment for meals older than 6 months, anti-oxidant concentration at the time of shipment should be stated and must exceed 100 mg/kg (ppm),
packing, number of bags and total weight of the consignment,
temperature of fishmeal at the time of dispatch from the factory,
and the
date of production.

*Note:* No weathering/curing is required prior to loading

(c) The temperature of the cargo should not, at the time of loading exceed 35°C or 5°C above ambient temperature, whichever is higher.

(d) Temperature readings should be taken 3 times a day during the voyage and recorded.

(e) If the temperature of the cargo exceeds 55°C and continues to increase, ventilation to the hold should be restricted. If self-heating continues, then carbon dioxide or inert gas should be introduced.

 *(continued on next page)*