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COMMISSION OF THE EUROPEAN COMMUNITIES

**TRACE LEVEL DETERMINATION OF
OXYGEN IN ORGANIC COOLANTS AND
ULTRA-MICRO DETERMINATION OF
OXYGEN IN C-H-O ORGANIC COMPOUNDS**

by

A. COLOMBO

1972



**Joint Nuclear Research Centre
Ispra Establishment-Italy
Chemistry Division**

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The method is based on a catalytic pyrolysis and reduction in hydrogen stream and on the final measurement of the formed water by means of a commercial Keidel electrolytic hygrometer.

Although it has not been applied to compounds containing atoms other than carbon, hydrogen and oxygen it seems at last theoretically that small and simple variations of the apparatus or of the analytical procedure could render the method able to analyse substances containing other atoms.

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ABSTRACT

A method of high sensitivity which requires the use of small samples has been developed for the determination of the oxygen contained at trace level in the organic coolants used in nuclear reactors and for the ultra-microanalysis of oxygen-rich organic C-H-O compounds.

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KEYWORDS

OXYGEN
IMPURITIES
ORGANIC COOLANT
MICROANALYSIS
ORGANIC OXYGEN COMPOUNDS
CATALYSTS
EFFICIENCY
ELECTROLYTIC CELLS
QUANTITATIVE ANALYSIS

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1. INTRODUCTION

The use of organic coolants in nuclear reactors requires the determination of their total oxygen content. The Schütze method (1), which involves pyrolysis and reaction of the sample with heated carbon to produce carbon monoxide which may be determined in various ways, has given rise to difficulties in its application by modification of a commercial apparatus (2). Therefore a reinvestigation has been made of the Ter Meulen (3) method, which briefly consists of vaporizing the sample in a stream of hydrogen which flows over a cracking catalyst followed by a hydrogenation catalyst, thus converting the oxygen of the sample to water which may be measured.

As we possess a commercial Keidel electrolytic hygrometer (4) for the continuous determination of traces of water vapour in gases, it seemed logical to use such an instrument for the final measurement, as it would permit the achievement of two aims:

- The construction of a simple apparatus, which from previous experiences seemed to be the premise for getting results.
- The reasonable certainty of avoiding trouble during the vaporization of the samples (3), by limiting the weight of the substance, in consideration of the inherent ability of the hygrometer to measure very small amounts of water.

Instruments of this type have been already used in organic elemental microanalysis: Nakamura (5) reports on their use for the determination of carbon and hydrogen; the same author (6) employs a home-made instrument for determining oxygen on oxygen-rich samples weighing 3-4 mg according to the Schütze approach and conversion of the carbon monoxide to water through oxidation to carbon dioxide followed by a passage through a LiOH converter.

During the development of the present work, we perceived that the final apparatus was able to perform not only the analysis of oxygen in traces on samples of low weight (less than 30 mg), but also the ultra-micro analysis on oxygen-rich compounds (sample weight less than 0,5 mg) without any modification.

2. APPARATUS

The figures 1 and 2 give a diagram of the apparatus.

Before reaching the refrigerant and the metal line which go to the electrolytic cells of the hygrometer, the hydrogen passes through a Deoxo* catalyst which diminishes its oxygen content to less than 1 ppm volume to volume (v/v); a bed of 4 Å molecular sieve pellets for drying; a plastic tube and finally a quartz tube (inner diameter 8 mm, thickness 2 mm) the different parts of which serve for loading, vaporizing, cracking and reducing the samples.

Apart from the plastic tube, all rubber or plastic connections are avoided as they act as uncontrollable sources of moisture inside the apparatus; in particular, after the drying bed, brazing of metal parts is also avoided and all connections are made with Swagelok compression (for quartz to metal or metal to metal) or vacuum wax sealed cone and socket joints (for quartz to glass or glass to glass).

The argon serves only for flushing the hydrogen when it is necessary for one reason or another.

Details of the various parts are given below.

2.1. Hygrometer

An electrolytic hygrometer absorbs on phosphor pentoxide and in the mean time electrolyses continuously the water vapour present in a gas stream, giving rise to a current which is proportional to the mass of water flowing through the cell in the unit of time according to the Faraday's law. For greater details reference is made to a published work (7), which reports also about the experiences of different authors on hygrometers and on the handling of water in trace amounts. The instrument used in this case is the "Hygromite"**, which makes use of rhodium electrodes, on which the so called "recombination effect" viz. the phenomenon of partial reversion to water of the products of the electrolysis catalysed by the electrode materials in hydrogen streams, resulting in an electrolysis current higher than the true, is very small and independent of the concentration of water

*Engelhard Ind. Inc., Newark, N.J., USA

**Beckman Instruments Inc., Fullerton, Calif., USA

in the stream (7).

Its microamperometer is graduated in parts per million from zero to ten ppm (v/v) and gives directly the concentration of water in a gas stream when the gas flow is 100 cc STP*/min; it follows that 10^{-4} cc STP/min of water vapour through the cell at whatever flow of the gas corresponds to a reading of 1 ppm (v/v), which in turn corresponds to a current of 13,33 microampere.

The signal can be attenuated in five steps: X1, X3, X10, X30, X100 and this gives the possibility to measure a maximum of 10^{-1} cc STP/min of water vapour.

For this work the "Hygromite" has been dismantled and for improved efficiency two cells have been connected in series with respect to the hydrogen stream; the cells, filled with aqueous phosphoric acid solution (10% volume) according to the instructions manual, are energized in parallel by the electrical supply of the instrument: so the microamperometer indicates a current sum of the water which is passing in both the cells; through the facility incorporated in the "Hygromite", the ammeter is coupled to a potentiometric recorder**, having two chart speeds: 6" and 24"/hour which are useful respectively for samples giving low and high quantities of water (slower and faster peaks of water on the recorder).

2.2. Refrigerant and metal line

These connect the quartz tube containing the catalysts with the electrolytic cells, and are made of standard stainless steel tubes respectively of $\frac{1}{4}$ " and $\frac{1}{8}$ " ext. diameter; while the length of the line is kept as short as possible, the length of the refrigerant is 8 cm. Before installing, they are degreased with solvents, washed first with dilute hydrochloric acid (1/1), and then with a mixture water-nitric acid-hydrofluoric acid (85/10/5), followed by distilled water and ethanol. After this, the dried metal line is flamed with a Bunsen burner to a red heat whilst hydrogen is flushed through it.

*Standard Temperature and Pressure: 21°C; 760 mm Hg

**Beckman cat. no. 93502-Beckman Instr. Inc., Fullerton, Calif, USA

Such a chemical cleaning permits during the analyses a faster equilibration rate between the water carried by the hydrogen and the water absorbed on the metallic walls, accelerating its transfer process to the cells, which in turn means narrower and higher water peaks on the recorder.

The refrigerant, whose operational temperature is about 10°C, is installed with the aim of stopping the non gaseous products coming from the quartz tube after the pyrolysis and the reduction of the samples, which if condensed in the cells, would cover the phosphor pentoxide with an inert material which would greatly reduce its water absorption properties.

2.3. Cracking and hydrogenation catalysts

As the total oxygen of the samples is measured by integration of the current due to the water passing in the electrolytic cells, viz. by integration of the peaks which appear on the recorder, the problem was to find catalysts which, through the already mentioned adsorption-desorption phenomena which slow down the process of elution of the formed water, did not give rise to wide, flat and therefore not easily measurable peaks. Because of the small amounts of water handled by the apparatus (amounts of 50-55 μg of oxygen contained in the samples, give water peaks at full scale on the microamperometer set at X100 attenuation and the analyses are performed usually at X30 or X100 attenuation) the problem was not of scarce importance and not readily solved. Among the various materials tested, the best results have been obtained by the use of quartz wool at 950-960°C as cracking catalyst and of nickel promoted by thoria and supported on quartz wool at 370-380°C as hydrogenation catalyst.

After washing with aqua regia and distilled water, the dried cracking catalyst is put into the cracking zone of the quartz tube, where, once and for all, it is conditioned under working conditions by means of an organic coolant sample.

The hydrogenation catalyst is prepared by imbibing the quartz wool

(cleaned as mentioned previously) with a 20% aqueous solution of Ni $(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ containing thorium nitrate in a quantity sufficient to give 2% thoria on the nickel present; the wet mass is put in a muffle at 600°C to insure the destruction of the nitrates and finally charged in the apparatus where it is reduced for 24 hours under hydrogen flow at 400°C, venting during this time, the exit of the quartz tube to the atmosphere.

2.4. Vaporizer

It consists of a small electric furnace capable of temperatures up to 1050°C and therefore able to accommodate a wide variety of samples. For this work the temperature was always kept at 550°C without any inconveniences; the samples have been introduced by means of a boat technique: unless specified, quartz boats for the trace level, and small platinum boats hand made using platinum foil 0,03 mm thick for the ultra-micro work. The latter ones are compatible with the Cahn Gram Electrobalance*, which has been used for weighing samples less than 1 mg.

2.5. Plastic tube

Soft polyvinylchloride whose dimensions are 25 cm length, 8 mm ext. diameter, 1 mm wall thickness. Due to its permeability to atmospheric moisture, it gives inside the apparatus a steady and controlled quantity of water vapour of the order of $6 \cdot 10^{-3}$ cc STP/min (which corresponds under stationary conditions to a reading of ~ 60 ppm (v/v) on the micro-amperometer of the hygrometer and in turn to a base line of ~ 60 ppm (v/v) on the recorder), which accelerates very much the transfer to the cells of the water coming from the samples.

*Cahn Division/Ventron Instr. Corp., 7500 Jefferson St., Paramount Calif., USA

2.6. Hydrogen pressure and flow rate under working conditions

The hydrogen pressure is kept at 0,25 Atm g. by means of a two stage pressure regulator mounted on a gas cylinder: this avoids the entrance of air during the loading of the samples in the apparatus through the loading cone B14, which is usually closed by means of a greaseless glass stopper held in situ by springs.

The flow rate is relatively high and kept at 120-130 cc STP/min by means of the needle valve mounted immediately before the electrolytic cells: it represents a good compromise between the efficiency of the entire system and the necessity of a high rate for a fast elution of the water. The apparatus can be operated at a lower flow rate, but with some sacrifice in the detectability of water.

3. OPERATING PROCEDURE

With the temperature, pressure and flow rate at the operating values, the sample holder* is taken off from the vaporizer and put below the loading cone where is left to cool down for five minutes; at the end of this time a boat containing a sample is loaded in the sample holder and after a period of 2,5 minutes, which is necessary to return to the stationary condition, is pushed into the vaporizer where it is left for 10 minutes: the sample undergoes vaporization, cracking, reduction and its oxygen appears as a water peak above the steady base line of the recorder. The cycle can now be repeated on another sample and so on.

The area, which is taken from the start of a peak to the end of the 10 minute period in the vaporizer, is evaluated by the "cut and weigh" technique and, with allowance being made for the area given by the empty boats, (net area), gives the oxygen of the sample when multiplied by the ratio between the theoretical oxygen and the net area of a calibration substance (a pure oxygen-rich compound, e.i. benzoic acid), used in known amount.

*externally handled by means of a magnet

The calibration is made three times a working day, viz. at the beginning, at the middle and at the end of the analyses, using amounts of calibration compound such to give quantities of water in the range of those obtained from the samples, and what is finally used for the calculations is in fact the average value of the mentioned ratios.

Because an electrolytic hygrometer is in principle absolute, the calibration should not be necessary as the correspondency between the reading of 1 ppm (v/v) and the value of 10^{-4} cc STP/min of water passing through the cells provides a mean of calculating absolute results. Nevertheless the calibration is used mainly as the absolute efficiency of the apparatus is slightly less than 100%, probably due to incomplete reduction of the samples, or small irreversible water adsorptions on the various components of the system, or an incomplete 100% efficiency of the cells.

The area given by the empty boats is an average value obtained analysing each working day two boats, which have the same characteristics of those used for the samples.

The figure 3 shows a typical water peak, from a sample of HB40*, weighing 11,3 mg, and containing 2600 ppm weight (ppm w) oxygen.

4. RESULTS

All the results presented in this section have been obtained by the use of benzoic acid as a calibration compound, but, as the Table 3 shows, other substances can be employed.

4.1. Trace level analysis

Table 1 gives the typical results obtained on mixtures prepared from pure oxygen-rich compounds and OM2* and Table 2 a comparison between this and the activation analysis method on various organic coolant samples.

*OM2 composition= 1% ϕ_2 ; 14-16% o- ϕ_3 ; 79-81% m- ϕ_3 ; 3-5% p- ϕ_3 ; solid product
HB40 composition= 18% o- ϕ_3 ; 82% hydrogenated ϕ_3 ; liquid product
OMD composition= 24-26% ϕ_2 ; 54-58% o- ϕ_3 ; 18-20% m- ϕ_3 ; 1% p- ϕ_3 ; liquid product

The mixtures have been prepared in the platinum boats, weighing directly the compound in the boat by means of the Cahn Electrobalance, and adding then the OM2 on a normal balance.

Whilst the results of Table 1 are in close agreement with the theoretical value, Table 2 shows an evident discrepancy between the chemical and the activation analysis; this is, for the moment, unexplained as both methods measure the total oxygen (chemically bound in the organic molecule, chemically bound as trace water in the sample, and dissolved) and as the chemical analysis has been performed on the same samples previously subjected to activation analysis in vacuum tight plastic capsules, which have been opened to the atmosphere only just before submitting the samples to the chemical method. From both our and other experiments (8) and from the solubility and reactivity of the oxygen and of the moisture toward the organic coolants, it seems highly improbable that samples exposed to air for such a short time will change their oxygen content so drastically. For the standard deviations given, it appears that the reproducibility of the method is of the order of 5% relative or 40-70 ppmw, which is the greatest.

As the blank value of the empty quartz boats is of the order of 0,7-1,5 μg oxygen (whether the boats have been recently "conditioned" in the apparatus or not), the apparatus can easily detect 50 ppmw oxygen on samples weighing 30 mg. Higher weights have not been used, as they can perturb too much the hydrogen flow, which in turn perturbs the base line of the recorder.

The maximum amount of oxygen contained in the samples, should be less than 50-55 μg (see section 2.3.).

At the end of each working day, the apparatus is opened between the quartz tube and the refrigerant and, under argon flow, the end of the tube is cleaned with a flame from the small deposits whilst the refrigerant is washed with solvents. While these deposits do not show adsorption properties towards the water, they cause when in too large amounts, irregularities along the average value of the base line, thus impairing the evaluation of the area of the peaks. Such an operation, the dismantling and the reinstalling of the various parts included, takes no more than half an hour.

In the figure 4 a comparison is given between a typical base line and another which exhibits irregularities due to accumulated deposits.

4.2. Ultra-micro analysis

Table 3 and Table 4 show typical results obtained on oxygen-rich compounds.

The best results (Table 3) are obtained when the samples are used in relatively high weights, provided the requirement, already mentioned, that the samples must contain less than 50-55 μg of oxygen, is met. At a sacrifice of precision and accuracy, the apparatus is able to work on samples of lower weight (Table 4); the drop in precision and accuracy is to be charged mainly to the relatively higher importance of small fluctuations of the base line on the magnitude of small peak areas and in the difficulty of weighing with an accuracy better than 2-3 μg .

The blank value of the empty platinum boats is of the order of 0,35-0,70 μg oxygen.

As, due to the small amounts of substance used, no deposits arise, therefore it is unnecessary to clean the end of the quartz tube and the refrigerant at the end of the working day.

As measured for all the determinations of Tables 3 and 4 (and for the pertinent calibrations) the absolute efficiency of the apparatus was in average 93,5% with fluctuations from day to day in either directions.

5. CONCLUSIONS

In consideration of the small weights of sample needed, the proposed apparatus seems to work satisfactorily. The catalysts have never exhibited fatigue phenomena and the cells held for months before the need of regenerating them. This should be done when the absolute efficiency of the apparatus falls below 80-85%.

Despite that extensive tests have not been performed, it seems that the apparatus can be used without modification in the ultra-micro analysis of nitrocompounds, sulphur and halogen containing compounds (fluorine excluded); but while nitro and halogenated compounds do not seem to have deleterious effects on the cells or on the catalysts, no proof exists for the moment that the same applies to sulphur containing substances in consideration of the usually high poisoning power of sulphur on catalysts. A copper barrier at $\sim 400^{\circ}\text{C}$ placed between the cracking and the reduction catalyst could in this case solve the question of the sulphur interference.

Amino compounds seem to be the greatest problem, as they release probably, despite the unfavourable thermodynamic conditions, small amounts of ammonia which render in a short time the cells inefficient because of phosphate formation. The addition in the boats of some chlorine containing organic compound, could cancel this effect through ammonium chloride formation.

Metal-containing materials cannot be analysed by the usual Schütze method because part of the oxygen is retained by the metal; the present apparatus should offer definite advantages in the analysis of such materials as all oxygen compounds which are reduced by hydrogen directly in the boats or over the catalysts after decomposition should be analysable; in difficult cases the use of Indulin Base R.M., as proposed by Unterzaucher in 1952 and as confirmed by Gouverneur and Bruijn (8), should solve the problem.

The last two samples of table 4 are presented in order to show the ability of the apparatus to analyse materials other than simple C-H-O compounds; but further work is necessary in order to clarify completely what is exposed in this section.

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TABLE I
ANALYSIS OF MIXTURES

	OM2 mg	Compound mg	Ø ppm w theory	Ø ppm w found	Recovery %
Mix 1	a _{12,90}	Diphenylene oxide 0,4018	3465	3425	98,8
Mix 2	a _{9,10}	B-naphthol 0,1420	2305	2340	101,5
Mix 3	b _{12,95}	2-2'Dihydroxybiphenyl 0,1016	1970	1740	88,3
Mix 4	b _{9,90}	B-naphthol 0,1027	1770	1500	84,8
Mix 5	b _{12,50}	Benzophenone 0,1590	1730	1830	105,8
Mix 6	b _{8,40}	B-naphthol 0,0570	1380	1315	95,3
Mix 7	b _{11,80}	Diphenylene oxide 0,0352	920	1000	108,7

a = 610 ppm w O - b = 637 ppm w O
according to the described procedure after multiple determinations

Av. 97,6

TABLE 2
ANALYSIS OF ORGANIC COOLANTS

Sample	Range of weights mg	no. Det.	Ø ppmw	Std. Dev. b	Neutron activation analysis Ø ppmw
OM2 no. 1	15-30	4	415	41,5	205
OM2 no. 2	15-30	4	375	65,0	235
OMD	15-30	4	460	38,5	320
HB 40	5-10	4	2515	91,0	2905
Mix OMD/HB 40 ^a	10-20	4	1535	85,5	1325

a = 54,8 % w OMD; 45,2 % w HB 40

$$b = \sqrt{\frac{\sum (\bar{x} - x)^2}{n-1}}$$

TABLE 3

ANALYSIS OF OXYGEN-RICH COMPOUNDS - NORMAL WEIGHTS

Sample	Weight mg	O % w found	O % w Av. found	O % w theory	Error	Rel. Error %
Benzophenone	0,5220	8,57	8,72	8,78	-0,06	-0,68
	0,3619	8,52				
	0,3112	9,07				
Diphenylene oxide	0,3902	9,41	9,59	9,51	+0,08	+0,84
	0,3352	9,99				
	0,4150	9,36				
B-naphthol	0,3072	11,33	11,36	11,10	+0,26	+2,34
	0,2904	11,34				
	0,4268	11,40				
2-2' Dihydroxybiphenyl	0,2410	16,52	16,75	17,18	-0,43	-2,50
	0,2186	17,25				
	0,1942	16,48				
Citric acid	0,0652	59,70	58,87	58,29	+0,58	+1,00
	0,0542	59,90				
	0,0742	57,00				
						Av. + 0,20

TABLE 4

ANALYSIS OF OXYGEN-RICH COMPOUNDS - SMALL WEIGHTS

Sample	Range of weights mg	no. Det.	O % w found	Std. Dev.	O % w theory	Error	Rel. Error %
Benzophenone	0,06-0,15	4	9,36	0,45	8,78	+0,58	+6,60
Diphenyleneoxide	0,06-0,15	4	9,12	1,23	9,51	-0,39	-4,10
d-camphor ^a	0,10-0,25	2	9,02		10,51	-1,49	-14,17
β-naphthol	0,04-0,10	4	11,11	0,58	11,10	+0,01	+0,09
2-2' Dihydroxybiphenyl	0,04-0,12	4	15,83	0,39	17,18	-1,35	-7,85
Citric Acid	0,02-0,05	4	58,01	3,40	58,29	-0,28	-0,48
Copper(II) oxide	0,08-0,15	5	20,35	0,82	20,12	+0,23	+1,14
Silver nitrate	0,04-0,10	5	29,16	1,13	28,26	+0,90	+3,18
						Av. -0,20 ^b	

a = Results very low, because of sublimation before analyzing

b = d-camphor not included

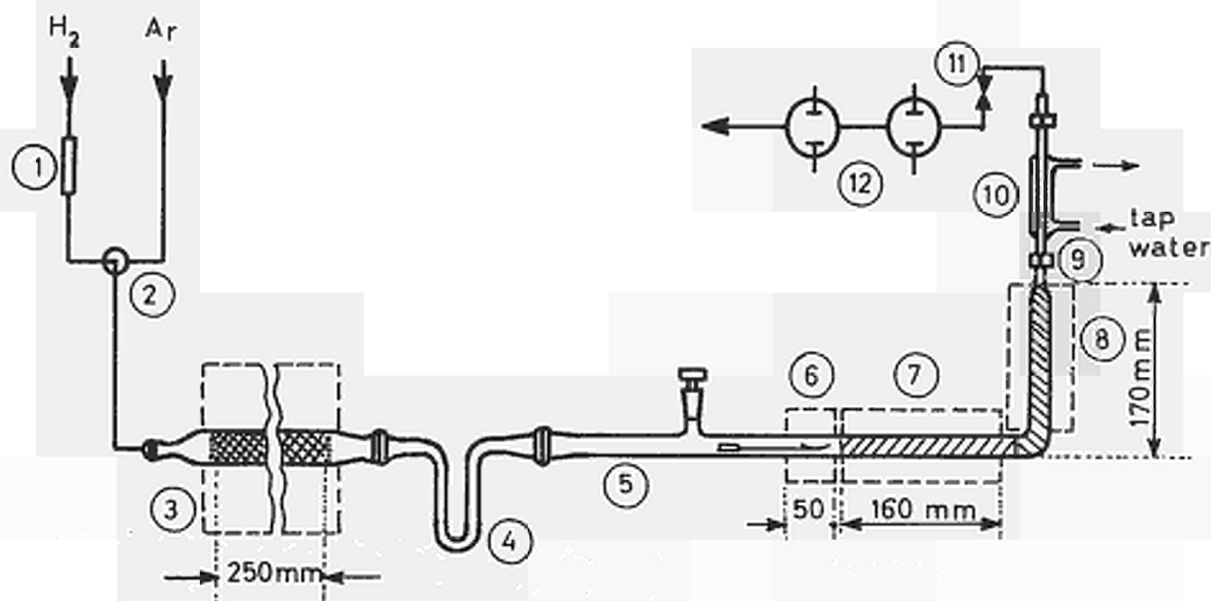


FIG.1

DIAGRAM OF THE APPARATUS

1=Deoxo catalyst 2=three ways flat glass stop cock 3=Molecular sieve pellets 4=Plastic tube 5=Quartz tube 6=Vaporizer 7=Cracking catalyst 8=Reduction catalyst 9=Swagelok joint (with teflon ferrules) 10=Refrigerant 11=Needle valve 12=Electrolytic cells

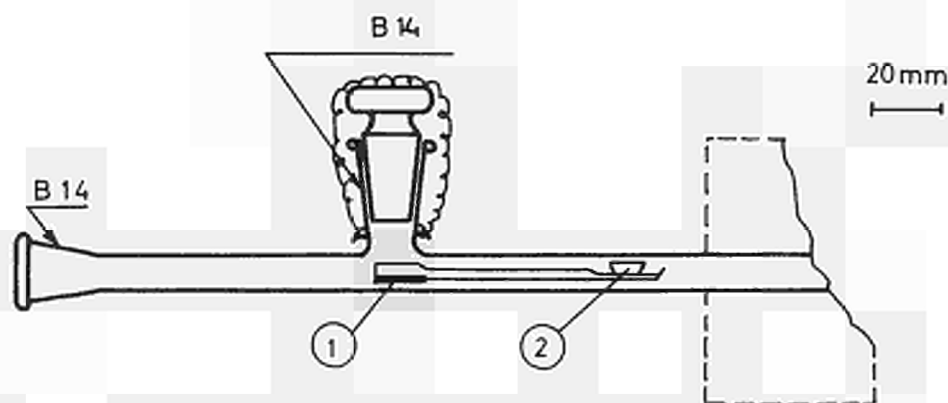


FIG.2

DETAILS OF THE LOADING ZONE

1=Iron welded to the terminal part of the quartz sample holder
2=Boat

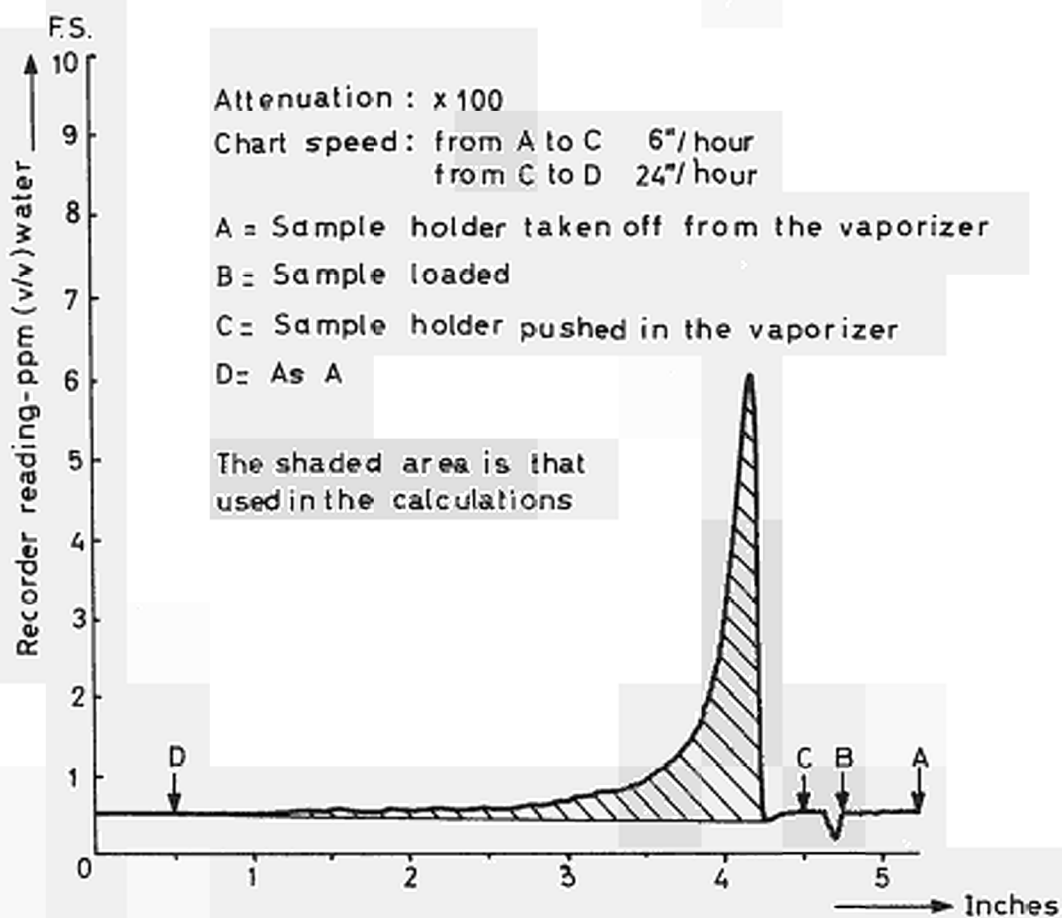


Fig. 3 TYPICAL WATER PEAK

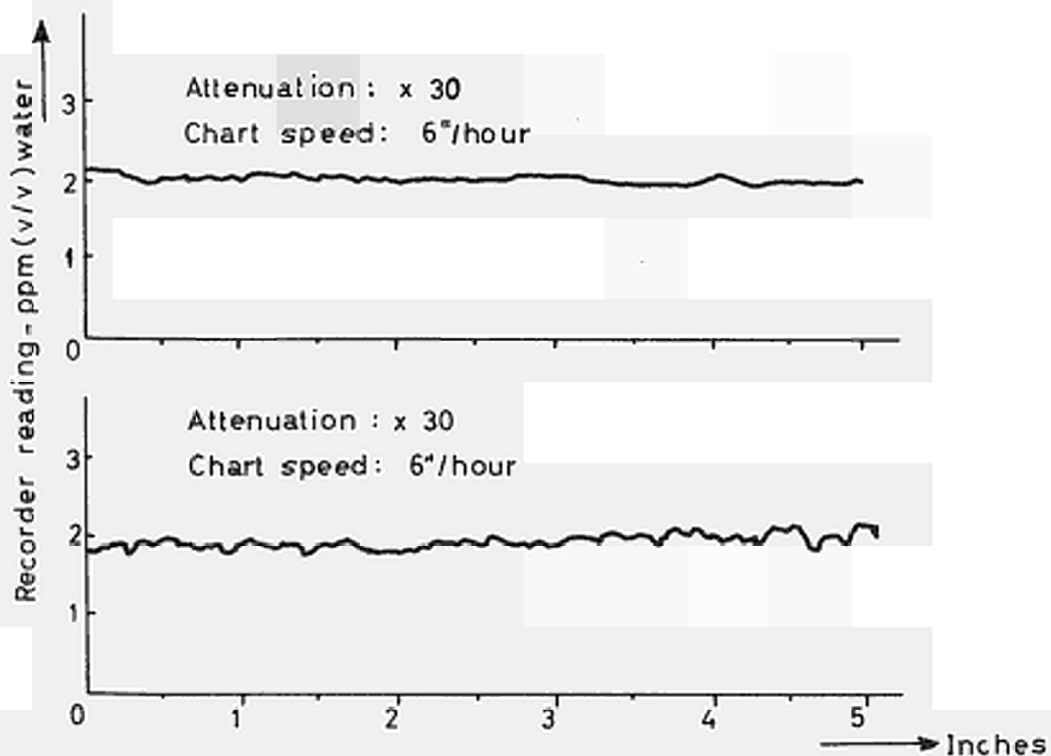
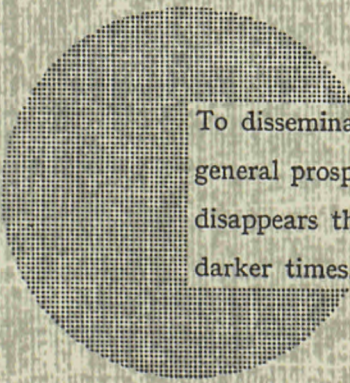


Fig. 4 TYPICAL (upper) AND IRREGULAR (lower) BASE LINE

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To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

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