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SODIUM SAMPLING DEVICE ON A CIRCUIT FOR IMPURITY ANALYSES

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Summary

The equipment described was developed in consideration of the following two essential requirements:

Absence of any contamination during the sampling and transport operations leading to the analytical equipment.

Simultaneous recovery of several samples, each remaining representative of the sodium in the circuit.

For this purpose, sampling was carried out entirely under vacuum in a Pyrex glass equipment, which limits the temperature of the sodium to 240°C. The samples are obtained in the form of sealed ampules under vacuum and each contains 2-3 g of sodium.

The analysis for oxygen by the amalgamation technique, conducted with samples obtained by this method, reveals a very satisfactory reproducibility in the range of concentrations below 20 ppm.

Introduction

The problem of sampling assumes primary importance when it is not possible to analyze the impurities present in the sodium on the circuit itself and by a continuous process. In the case of oxygen particularly, it is useless to seek analytical methods permitting the determination of concentrations of less than 10 ppm if a sampling method that excludes all risks of detectable contamination is not available.

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The importance of the problem has not escaped numerous other authors, who have proposed a great variety of solutions [1-6].

Under these conditions, we concentrated on the development of a sampling device which would satisfy the following two essential requirements:

The most rigorous elimination possible of any source of contamination during the operations of sampling and transport to the analytical equipment.

Simultaneous recovery of several samples, each remaining representative of the sodium in the circuit.

Choice of the Method

With consideration of the above two requirements, we studied the various solutions which are possible for the problems posed by the sampling atmosphere, the nature of the receiver and the physical state of the sodium during sampling.

1. Sampling atmosphere:--In order to prevent any contamination, sampling must be made either in an inert atmosphere (anhydrous and deoxidized argon or helium) or under vacuum. The latter solution was adopted for the following reasons:

The continuous control of the vacuum is simpler and more reliable than the determination of the purity of a gas; and

The absence of purification systems eliminates the need for controls and frequent regeneration;

The method is then homogeneous with respect to our analytical method by amalgamation which is carried out in vacuum.

In this connection, the sampling tests in an inert atmosphere carried out by us revealed much greater scattering of the analytical results than for those samples obtained under vacuum.

2. Nature of the container:--The choice of a vacuum lead us to the use of Pyrex glass which permits sealing and opening of the ampules under vacuum. Furthermore, this glass has the advantage compared to metals that it can be easily degassed and that it does not have a superficial oxide skin which can constitute a source of contamination.

On the other hand, its use limits the sampling temperature to about 240°C . In fact, we were able to demonstrate that an ampule of sodium containing 10-12 ppm oxygen retained this same content after heating to 235°C for 1 hr. and 20 min., although the glass then began to assume a very slight coloration. At higher temperatures, in contrast, the attack of the glass becomes rapidly noticeable: after a period of 3 min. at 320°C , the oxygen content of a sodium of the same initial purity increases to 30 ppm.

3. Physical state of the sodium during sampling:-- Analytical uncertainties make it necessary to obtain several samples from the same sampling operation. In the case of the analysis for impurities in a liquid metal, we do not believe that sampling in the solid state can remain representative of the sodium in the circuit. In fact, a phenomenon of impurity segregation frequently occurs during solidification, which results in a heterogeneous distribution in the metal. Under these conditions, the analytical results

obtained from samples removed in the solid state become questionable, unless the entire metal removed is analyzed and an average is taken.

In our case, sampling is carried out in the liquid state at about 200°C, which in the case of oxygen guarantees homogeneity for concentrations up to 20 ppm.

Equipment and Procedure

The sampling equipment (Fig. 1) consists of a distributor (A) of stainless steel and a receiver (B) of Pyrex glass.

The distributor A essentially includes the following:

The sodium feed tube (1) welded at the top to the shut-off valve and equipped with an outer jacket containing a shielded resistance for preheating and a thermocouple for measurement of the sodium temperature;

A clamp (2), welded on the tube jacket (1) and containing the delivery line from the pumping aggregate (3);

A flexible membrane (4), each end of which is welded to a clamp. The upper clamp (5) is attached on piece (2), tightness being assured by a torus joint; the lower clamp (6) holds the glass equipment (B) and forms one unit with a mechanical system permitting horizontal and vertical movement.

The receiver (B) includes:

A flanged collar (7) for the torus joint;

A pot (8) reserved for draining sodium;

A bulb (9) intended for the collection of the sodium to be analyzed and provided with 8 ampules of thin glass on its large periphery.

Sampling takes place in the following manner:

The equipment is previously placed under a high vacuum, about $5 \cdot 10^{-5}$ mm Hg, and degassing of the glass equipment is accelerated by heating with a blow-pipe;

After heating tube (1) to about 150°C , the tubing of the drain pot (8) is connected opposite the end of the flow tube by means of the adjusting mechanism of the membrane. The shut-off valve is then opened and purging is effected.

After closing of the valve, another adjustment of the membrane permits the tubing of bulb (9) to be brought opposite the end of tube (1) and sampling takes place by again maneuvering the valve;

The bulb (9) is sealed with a blow-pipe and disconnected from the equipment.

During all of these operations, the vacuum is controlled by a gauge.

After heating to 200°C with a drier, the sodium of the bulb is transferred into eight ampules which are sealed under vacuum and sent to the analysis;

With the sampling completed, the membrane (4) is removed, permitting a complete cleaning of the equipment.

Discussion

It should be noted first of all that risks of leakages are limited to a maximum degree in this equipment; the number of joints has been reduced to two and their conditions of use practically exclude any possibility of leakage. In particular, the adjustment of the glass equipment takes place on a horizontal plane by translation rather than rotation around the membrane, in order to prevent any stress on the torus joint.

Furthermore, the equipment can be completely cleaned after each operation, thus preventing any risk of later contamination.

On the other hand, while the shape of the glass equipment might appear to be complex, its design makes any false maneuver practically impossible and finally simplifies the sampling operation.

In practice, the analysis for oxygen by the amalgamation technique, carried out with samples obtained by this method, is found to have a very satisfactory reproducibility in the concentration range below 20 ppm. For purposes of illustration, we present the results obtained by two different laboratories using five ampules obtained from the same sampling:

Laboratory A:	12, 15 ppm
Laboratory B:	11, 14, 15 ppm.

Conclusion

In its present version, this equipment is the result of numerous successive adaptations suggested by experience. In consideration of the delicate nature of the sampling operation, it seems that this technique is sufficiently simple and reliable to be currently utilized in liquid sodium installations in order to analyze for oxygen and probably for other elements.

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Fig.1. Schéma du dispositif de prélèvement.
.. A: distributeur - B: récepteur ..

Fig. 1.--Diagram of the sampling equipment.
(A) Distributor. (B) Receiver.