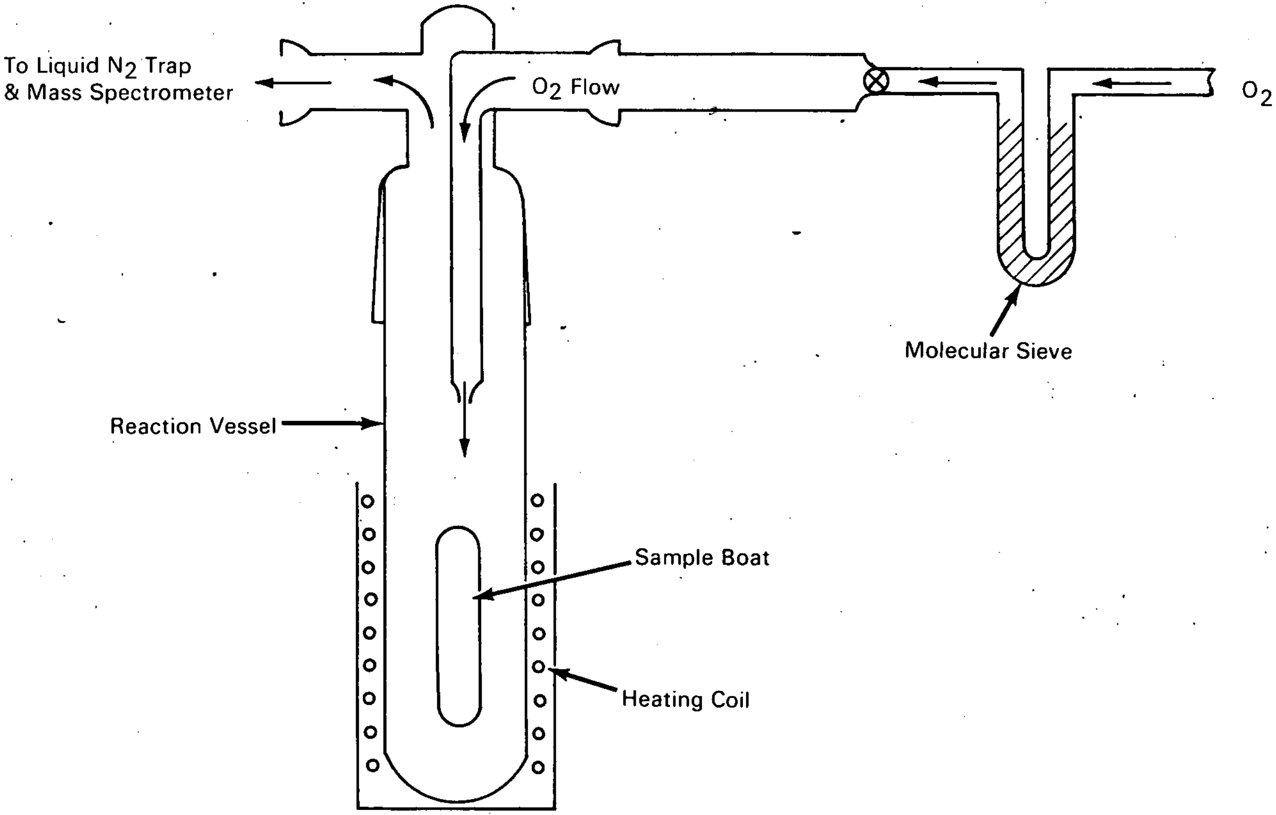
April 1970
Brief 70-10017

NASA TECH BRIEF

Determination of Hydroxyl Content in Impure Magnesium Oxide



Schematic of Laboratory Apparatus for OH Determination

The problem:

To devise an analytical method for the quantitative determination of the hydroxyl (OH) content in samples of magnesium oxide (MgO). Most of the impurities present in MgO can readily be determined by standard chemical and instrumental methods. However, there has been no reliable method for determining the amount of OH present in samples of MgO containing this impurity. Attempts to liberate OH (in the form of H_2O) from samples of MgO by raising the temperature to as high as 3300°C failed to give reproducible results. Reduction with carbon at elevated

(continued overleaf)

temperatures was also unsuccessful. Mass spectrographic methods are subject to inaccuracies, especially at high OH content. Infrared methods are feasible only for transparent forms of MgO, and these methods do not enable the analyst to differentiate between the OH bound in the superficial H_2O associated with the MgO and the OH chemically bonded to the magnesium (as in $Mg(OH)_2$, for example).

The solution:

A three-step thermal process based on hypothesis and experimental work indicating that the OH may be associated with the MgO in three different ways: (1) in superficial H_2O adsorbed on the MgO particles, (2) chemically bound OH in the form of $Mg(OH)_2$, and (3) chemically bound OH that substitutes for an O atom in the lattice and is present as Mg•OH. The OH in the latter chemical species is by far more tightly bound than the OH in $Mg(OH)_2$ and cannot be liberated (in the form of H_2O) in a reproducible manner by heating alone as in the case of $Mg(OH)_2$. The key (third) step in the process is the conversion of Mg•OH to $Mg(OH)_2$ by heating the sample in an oxygen atmosphere.

How it's done:

The weighed sample to be analyzed is first heated at 100°C in a vacuum (maintained in a degassed reaction vessel) for at least 30 minutes. During this step, surface water is liberated from the sample and the resultant effluent (water vapor plus other gases) is condensed in a liquid nitrogen trap. The trap is then isolated and warmed to expand the gases into a known volume at a measured pressure, and the amount of water in the gas is determined by mass spectrometry. In the second step, the sample is heated in the evacuated reaction vessel to 500°C, and the evolved water is analyzed as in the first step. The reaction in this step is represented by $Mg(OH)_2 \rightarrow MgO + H_2O$. In the final step, which is the new feature of the method, the sample is heated at 500°C in an oxygen atmosphere to convert the tightly bond Mg•OH to water in accordance with the following equations:

$$2Mg{\bullet}OH + 1/2\,O_2 \rightarrow Mg(OH)_2 + MgO$$

$$Mg(OH)_2 \rightarrow MgO + H_2O$$

The O_2 pressure in this step can range from 1 to approximately 130 torr, and the water evolved is analyzed as in the preceding steps. The OH corresponding to the water released from the sample in each of the three steps is calculated from the stoichiometric relation, OH/H_2O. The total OH, or the portion corresponding to the water released in each step, can then be expressed in weight percent of the sample analyzed.

Notes:

1. With suitable modification, this analytical method can be adapted to large-scale production of OH-free MgO.
2. The following documentation may be obtained from:

   Clearinghouse for Federal Scientific
   and Technical Information
   Springfield, Virginia 22151
   Single document price $3.00
   (or microfiche $0.65)

   Reference: NASA-CR-96385 (N68-33378), Development of Chemical Analysis Techniques for Advanced Materials

Patent status:

This invention is owned by NASA, and a patent application has been filed. Royalty-free, nonexclusive licenses for its commercial use will be granted by NASA. Inquiries concerning license rights should be made to NASA, Code GP, Washington, D.C. 20546.

Source: R. B. Iden, E. R. Blosser, and E. H. Hall of
Caltech/JPL
under contract to
NASA Pasadena Office
(NPO-10774)