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SUMMARY REPORT - APRIL 1964
CONTRACT NO. AT(11-1)-865, PROJECT 11

MASTER

APDA SODIUM TECHNOLOGY LOOP

The current study program, initiated in March of 1964, is an extension of the investigations described in References 1 and 2 for the evaluation of in-line meters designed to detect impurities in operating sodium loops. Experiments are scheduled to determine the thermal stability of sodium contaminants, and evaluate new processes designed for sodium loop purification. A "back-to-back" installation of two Rhometers is being incorporated in the extended loop facility to provide continuous differential measurement of the contaminant separations effected by the various new purification units.

To improve the performance of the original design of the two Rhometers, an auxiliary control package has been designed. A series of bench tests are programmed prior to the installation of the modified Rhometers in the extended loop facility. Circuit modifications to the meters will follow the changes described in Reference 2. The modifications will extend the range of temperature compensation and correct the ambient temperature drift inherent in the original meters. Graduated dials have been specified to effect better adjustment of Rs and Ry to achieve bias stability. A low impedance potentiometer (Vernistat) will be installed to reduce the error which is introduced into the signal summation circuit by small inherent errors in the commercial type LS-33 transformer units employed to extend the temperature compensator range. These improvements are expected to increase the sensitivity of the meter, and to reduce the decade adjustments presently required to maintain signal stability as sodium temperature varies.

Construction drawings of the extended loop layout and supporting steel framework are 75% complete, and procurement of major equipment is 50% complete.

Supporting laboratory effort has been initiated by the Chemistry Section to develop analytical techniques for confirmation of indicated impurity levels. A summary of the present capabilities of the Section with respect to the analysis of total carbon, "carbonate" carbon, "oxygen" and hydrogen in sodium is reported along with the progress on the development of new methods or the improvement of existing methods.

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1. Total Carbon by High Temperature Combustion

The method for determining total carbon in sodium metal is based on the conversion of all carbon compounds in the sodium sample to sodium carbonate followed by the thermal decomposition of the carbonate to carbon dioxide at 1100 C. The conversion and decomposition process takes place over a period of two hours in an atmosphere of 10% oxygen in helium. The carbon dioxide evolved is swept into a liquid nitrogen trap and subsequently determined by gas chromatography. All components of the combustion system which are in contact with the hot zone of the combustion furnace are fabricated from quartz.

The accuracy and precision of this method will be determined as one of the first steps in the program. Some standardization work has been undertaken during this report period. The accuracy of the method has been demonstrated on Leco carbon structures and the recoveries range from 96- to 104-% with 30 μgm samples. Present indications are that the precision is better than ± 10 ppm on replicate samples containing less than 100 ppm total carbon. Significant progress was made in lowering the blank for the procedure to a consistent level of 2- to 5- μgm .

Improved handling techniques have diminished the possibility of sample contamination. This was accomplished by modifying the combustion tube to allow complete sample isolation during transfer from the glove box to the combustion furnace.

2. "Carbonate" Carbon by Alcohol Dissolution and Acid Decomposition

APDA has developed a method for the determination of the carbon dioxide evolved when sodium is dissolved in alcohol and subsequently treated with acid. The method is rapid and easily performed. It is based on reacting the sodium sample with absolute ethyl alcohol in an inert gas atmosphere at 0 C. The resulting solution is decomposed with acid to yield carbon dioxide which is trapped in liquid nitrogen and determined by gas chromatography.

The accuracy of the method ranges from 95- to 105-% as demonstrated by recoveries on standard sodium carbonate samples. The precision appears to be ± 3 ppm on replicate samples with "carbonate" carbon contents under 100 ppm. The blank for the system is consistently under 5 μgm of carbon.

It has not yet been determined that the method is specific for "carbonate" carbon. Preliminary work on the procedure has confirmed the suspicion that non-carbonate carbon in sodium can be converted to carbonate by reacting the sodium with water. However, it has been shown that this reaction does not occur in the present alcohol dissolution procedure.

3. "Oxygen" by Mercury Amalgamation

A variation of the original Pepkowitz procedure for separation of sodium oxide by mercury amalgamation is in use at APDA. the present procedure is carried out in a glove box under a purified argon atmosphere to protect the sodium sample from oxygen, carbon dioxide, and water during amalgamation and extraction. Specially designed separatory funnels are employed for isolating the oxides from the sodium. The oxides are subsequently measured by titrating the oxide solution with standard sulfuric acid. The titration is made using a recording pH meter and automatic titrimeter. This procedure is not specific for sodium oxide and other unamalgamated sodium compounds interfere with the results. Since no standard runs have been made using this procedure, the accuracy has not been determined.

4. Hydrogen in Sodium

The published methods for the determination of hydrogen in sodium do not appear to be reliable. Consequently, the analytical effort is directed toward developing an accurate method for hydrogen in sodium at a minimum level of 0.05 ppm. The program presently under investigation involves this goal, and is also directed toward the rapid development of a semiquantitative method that can be used on an interim basis while development work is in progress on the more accurate method.

The interim method proposed is based on the following sequential steps; the thermal decomposition of hydrogen containing compounds, the oxidation of the evolved hydrogen with copper oxide, and the absorption of the water formed on magnesium perchlorate. A mercury diffusion pump in the system will recirculate the gas to insure complete combustion of the hydrogen. After the combustion, evolution and absorption of hydrogen are complete, the increase in weight of the magnesium perchlorate will be determined and the amount of hydrogen calculated.

The more precise method involves the thermal decomposition of hydrogen compounds using an induction heater and absorption of the evolved hydrogen on an active metal such as cerium or lanthanum. The absorption rate will be observed by measuring the weight change of the metallic absorber with a Cahn Electrobalance which will be sealed in the gas collection system. Alternatively, the evolved hydrogen will be oxidized by passing through a hot palladium diaphragm into an oxygen containing atmosphere. The diaphragm will act as a catalyst to promote the oxidation of hydrogen to water. The water will then be weighed on the electrobalance.

Based on past experience, the major problem which occurs in the thermal decomposition of hydrogen compounds in sodium is the condensation of sodium vapor on the cooler parts of the system and the subsequent gettering of the evolved gases by the deposited sodium film. It is believed that the use of induction heating will minimize this problem; therefore, a 2-1/2 kw induction heater has been rented from Lepel High Frequency Laboratories. The unit has been set up and is in operating condition. Three empirical sodium melt tests have been run which showed that the unit has sufficient power. In one test, when approximately 10 grams of sodium were heated from room temperature to 500 F in three seconds, considerable deposition of a sodium mirror occurred. However, during the latest test performed, when the sodium was heated to 500 F slowly at low power, the extent of the sodium mirror deposited was considerably smaller.

Future tests will utilize quartz, Pyrex, nickel, and zirconium crucibles to determine which container produces the least sodium volatilization.

REFERENCES

1. APDA-149 "Evaluation of the Blake Sodium Resistivity Meter," October 1962, AEC Contract AT(11-1)-865, Project 3
2. APDA-163 "Operation of the Sodium Technology Loop for Contamination Meter Evaluations," March 1964, AEC Contract AT(11-1)-865, Project 3