

Here again the concentration law gives too small quantities. We are inclined to accept the often suggested explanation of hydrargyration of the dissolved sodium.

TABLE VII  
CONCENTRATION EFFECT AND TEMPERATURE COEFFICIENTS

$C_1$	$C_2$	$C_1/C_2$	$\frac{R \ln C_1/C_2}{F}$	$\frac{\Delta \pi}{\Delta T}$
0.416	0.1978	2.109	0.0642	0.0915
0.416	0.1102	3.780	0.1145	0.1550
0.1978	0.0396	4.990	0.1380	0.1625
0.0396	0.01972	2.010	0.0600	0.0610

Further theoretical considerations are postponed until the thermochemical data are ready for publication.

We are greatly indebted to the Carnegie Institution of Washington for much of our apparatus.

### Summary

1. Improvements have been effected in the electrolytic preparation and the subsequent manipulation of pure liquid sodium amalgam, as well as in the apparatus for the measurement of the electromotive force of sodium amalgam concentration cells with aqueous electrolyte.

2. The electromotive forces of many concentration cells of liquid sodium amalgam have been measured at 25° and a few cells also at 15° and 35°.

3. The deviations of these potentials from the simple concentration-law were found to be greater in sodium amalgam than with any other mercurial solution hitherto studied in detail.

4. The heats of transfer of sodium from one amalgam to another calculated by the Helmholtz equation, are found to be unusually large.

CAMBRIDGE 38, MASSACHUSETTS

### NOTES

**The Rapid Analysis of Potassium Perchlorate.**—In reviewing the various methods proposed for the analysis of potassium perchlorate, it is noted that the only principle that has been successfully applied is that of heating the perchlorate, either alone or mixed with some other substance. In the practical application of the principle the loss by volatilization of the resulting potassium chloride is the greatest source of error.

The method of heating the perchlorate with manganese dioxide has given such satisfactory results that it has been thought worth while to record the procedure.

A 0.5g. sample of potassium perchlorate is intimately mixed in an agate mortar with 1.0 g. of manganese dioxide. The charge is transferred to a porcelain crucible and heated for 15 minutes at a temperature of 600–

700°. After cooling, the mass is extracted with hot water, filtered, and the chlorine determined by either Mohr's or Volhard's method. The results obtained average about 0.2 to 0.3% low, indicating a small loss by volatilization, but are sufficiently accurate for most purposes. Should greater accuracy be desired, the method of Lamb and Marden<sup>1</sup> of heating the perchlorate alone in a glass test-tube and retaining the fumes evolved with a plug of asbestos wool, may be employed. When manganese dioxide is used, a blank is necessary inasmuch as commercial as well as native manganese dioxide always contains a small amount of chlorine.

DEPARTMENT OF CHEMISTRY  
UNIVERSITY OF WISCONSIN  
MADISON, WISCONSIN  
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VICTOR LENHER AND MARTIN TOSTERUD

### Electrolytic Reduction of Chromic Chloride to the Divalent Salt.<sup>1</sup>—

The problem of chromium reduction arose in connection with the work done at the Edgewood Laboratory on a method for producing ethylene from acetylene by the use of a solution of chromous chloride as a reducing agent. It was proposed to convert the chromic chloride produced by the acetylene treatment back to the divalent salt by means of electrolytic reduction. The investigations described in the present communication were undertaken to find conditions whereby it would be possible to obtain satisfactory yields of chromous chloride.

In the electrolytic reduction of chromic to chromous chloride the principal source of inefficiency is the production of hydrogen gas at the cathode. In some of our preliminary attempts at reduction an insoluble deposit formed on the cathode. The formation of such a coating of course represented inefficiency as far as the production of chromous chloride was concerned. However, the conditions under which the experiments reported herewith were carried out were such as to prevent the formation of any such deposit, and we may therefore regard the production of hydrogen gas as the only source of inefficiency in these particular experiments. Just what caused the deposit to appear in the preliminary work is unknown, but it may have been due to impurities in the chromic chloride solution, or to too high current density when the chromic concentration of the solution became small.

The following procedure was adopted as a means of determining the part of the current used for reduction purposes: the hydrogen evolved from the cathode in a given time and by a known amount of current was measured and reduced to standard conditions; the total hydrogen which would have been evolved by the current, had none of it been used for reduction pur-

<sup>1</sup> Lamb and Marden, *THIS JOURNAL*, **34**, 812 (1912).

<sup>1</sup> Published by permission of General Amos Fries, Chief of the Chemical Warfare Service.