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# Highly stable novel inorganic hydrides from aqueous electrolysis and plasma electrolysis

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## Abstract

After  $10^4$  h of continuous aqueous electrolysis with  $K_2CO_3$  as the electrolyte, highly stable novel inorganic hydride compounds such as KH,  $KHCO_3$  and  $KH$  were isolated and identified by time of flight secondary ion mass spectroscopy (ToF-SIMS). The existence of novel hydride ions was determined using X-ray photoelectron spectroscopy (XPS) and solid state magic-angle spinning proton nuclear magnetic resonance spectroscopy ( $^1H$  MAS NMR). A novel hydride ion formed by plasma electrolysis of a  $K_2CO_3$ ,  $Rb_2CO_3$ , or  $Cs_2CO_3$  electrolyte was also observed by high resolution visible spectroscopy at 407.0 nm corresponding to its predicted binding energy of 3.05 eV. © 2002 Elsevier Science Ltd. All rights reserved.

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## 1. Introduction

Alkali and alkaline earth hydrides react violently with water to release hydrogen gas which subsequently ignites due to the exothermic reaction with water. Typically metal hydrides decompose upon heating at a temperature in the range of 250–1000 °C. These saline hydrides, so called because of their saltlike or ionic character, are the monohydrides of the alkali metals and the dihydrides of the alkaline-earth metals, with the exception of beryllium.  $BeH_2$  appears to be a hydride with bridge type bonding rather than an ionic hydride. Highly polymerized molecules held together by hydrogen-bridge bonding is exhibited by boron hydrides and aluminum hydride. The synthesis of hydrides such as Group I hydrides by direct reaction of hydrogen with the corresponding metals and their chemistry is well known [1–4]. But, the synthesis of hydrides by electrolysis and plasma electrolysis has been essentially unexplored. Since atomic hydrogen and alkali ions may be present in these systems, novel catalytic reactions may be possible.

It was previously reported that a new chemically generated plasma source has been developed that operates by incandescently heating a hydrogen dissociator and a catalyst to provide atomic hydrogen and gaseous catalyst, respectively, which react to produce an energetic plasma called a resonance transfer (rt)-plasma [5,6]. It was extraordinary, that intense vacuum ultraviolet (VUV) emission was observed at low temperatures (e.g.  $\approx 10^3$  K) and an extraordinary low field strength of about  $1-2$  V  $cm^{-1}$  from atomic hydrogen and certain atomized elements or certain gaseous ions which singly or multiply ionize at integer multiples of the potential energy of atomic hydrogen,  $E_h = 27.2$  eV where  $E_h$  is one hartree. The theory has been given previously [7,8].

The ionization of  $Rb^+$  to  $Rb^{2+}$ ,  $Cs$  to  $Cs^{2+}$ , and an electron transfer between two  $K^+$  ions each provide a reaction with a net enthalpy of about  $E_h$ . The presence of each of these gaseous reactants formed an rt-plasma with atomic hydrogen having strong VUV emission. Significant line broadening of 18, 12, and 12 eV was observed from an rt-plasma of hydrogen with  $KNO_3$ ,  $RbNO_3$ , and  $CsNO_3$ , respectively, compared to 3 eV from a hydrogen microwave plasma. Remarkably, a stationary inverted Lyman population was observed in the case of an rt-plasma formed with potassium and rubidium catalysts, and a collisional radiative model was used to determine that the observed overpopulation was

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