

## THE SULFUR-IODINE AND OTHERS THERMOCHEMICAL PROCESSES STUDIES AT CEA

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### *Abstract*

The thermochemical Sulphur-Iodine process is studied by CEA with the objective of massive Hydrogen production using the heat at high temperature coming from a very high temperature reactor. A two folds programme was set up in 2000. The main part is devoted to the study of the basic process, including optimisation of a detailed flow-sheet using existing knowledge from literature, thermodynamics measurements of each section of the process and studies for a hydrogen production plant. Several devices are currently under test phase to get precise measurements of the Bunsen section and the HI distillation liquid vapour equilibrium. Measurements techniques have been prepared to obtain data under pressure and temperature values outside the existing domain. In parallel, an operating flow-sheet was studied that gives the best efficiency one could obtain today when building a loop using either a reactive or a direct distillation, i.e. 35%. From the analysis of those flow-sheets, an innovative programme was derived that could lead to raising the efficiency up to 50%. This program includes membranes studies, process operating point optimisation (by reducing water and iodine quantity, and by optimising temperature and pressure in each device of the flow-sheet).

In parallel, international collaborations are used to develop laboratory loops to get experience from materials and components, and to verify the existence of limited side reactions and of limited recirculating products. A Bunsen section is under construction in CEA/Marcoule to be coupled later on to two other sections, sulphuric decomposer and HI distillation, built in the US by SNL and GA respectively, thanks to an International NERI contract.

On the other hand, several cycles are presented with a first set of analysis: the hybrid Sulphur cycle, the UT\_3 cycle and Cerium-Chloride cycle.

## Introduction

The thermochemical Sulphur-Iodine process is being studied by the CEA, the French Atomic Energy Authority, with the objective of massive hydrogen production using high-temperature heat from a very high temperature reactor. A two-fold programme was set up in 2000. The main part is devoted to the study of the basic process, including optimisation of a detailed flow-sheet using existing knowledge from literature, thermodynamics measurements of each section of the process and studies for a hydrogen production plant. In parallel, operating flow-sheets was studied that gives the best efficiency one could obtain today when building a loop. From the analysis of those flow-sheets, an innovative programme was derived that could lead to raising the future efficiency. This programme includes membranes studies and process optimisation.

Besides the two main streams of the CEA programme, S/I cycle and HTE studies and development, a limited programme considers alternative cycle that could backup these two if optimization fails. In a first part, a methodology is being studied, that can be applied to each process and could lead to a more standardise comparison of advantages and disadvantages. Another point concerns economic evaluation of the cycles that is not reported here. Finally, several cycles are presented with a first set of analysis.

## Technical issues

### *The Bunsen section (section I)*

The Bunsen reaction involves an excess of both water, to make the reaction spontaneous, and iodine, to induce the phase separation which is a key point of the process. However, such excesses are quite unfavourable for the following HIX section (as will be described below). Hence, research and development efforts are devoted to find new operation points for the Bunsen reaction with lower amounts of  $I_2$  and  $H_2O$ , in order to find the best compromise between completion of reaction, phase separation, limitation of side reactions and energy loss in this low temperature exothermic step [1].

The experimental study of the Bunsen reaction which is currently underway at the CEA has required the development of analytical methods to allow a quantitative determination of the total amount of  $H^+$  ions by potentiometry, Sulphur by ICP-AES, iodine by UV-visible spectro-photometry after iodine reduction, and total water by density, in each phase. Specific devices have also been designed (Figure 1), ranging from simple glass devices to develop the analytical methods to a tantalum pressure vessel allowing studying the Bunsen reaction in actual process conditions, including the possibility of varying the  $SO_2$  pressure.

**Figure 1. Glass device for the study of the Bunsen reaction**

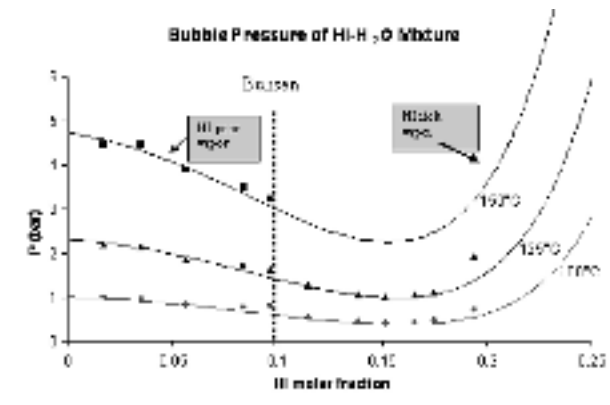


### The HIx section (section III)

In the S-I cycle, HI decomposition must be achieved from the HIx mixture produced during the Bunsen reaction described above. Four main difficulties have to be overcome:

- the extraction of HI from the HIx mixture is difficult because of the presence of an azeotrope (Figure 2) in the mixture, which prevents simple distillation;
- the extraction of HI from the HIx mixture requires very large heat exchanges, due to the large heat capacity induced by the high water content of the mixture;
- the decomposition reaction is slow and incomplete.

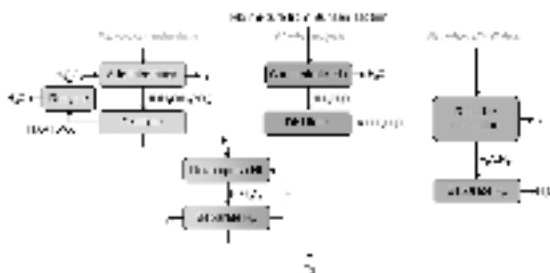
**Figure 2. Azeotropic lines of HIx mixture**



Three main options are currently being considered for the HI section (Figure 3):

- Extractive distillation was proposed by General Atomics [2]: the introduction of phosphoric acid induces first the separation of iodine, and then allows simple distillation of HI. HI is then decomposed in gaseous (or possibly liquid) phase around 450°C to yield H<sub>2</sub>, which has to be separated from the gaseous mixture using membranes.
- The present Japanese scheme favours electrodialysis [3], which removes some water of the HIx mixture to concentrate it beyond the azeotropic limit. Excess HI is then removed by simple distillation. The final decomposition and extraction steps are the same as in the extractive distillation process.
- Reactive distillation was proposed in the 1980s by RWTH Aachen [4]. HIx distillation and HI decomposition are performed in the same reactor at 350°C. A liquid-gas equilibrium is obtained in the middle of the column, I<sub>2</sub> is solubilised in the lower liquid phase and a mixture of gaseous H<sub>2</sub> and water is recovered at the top of the column.

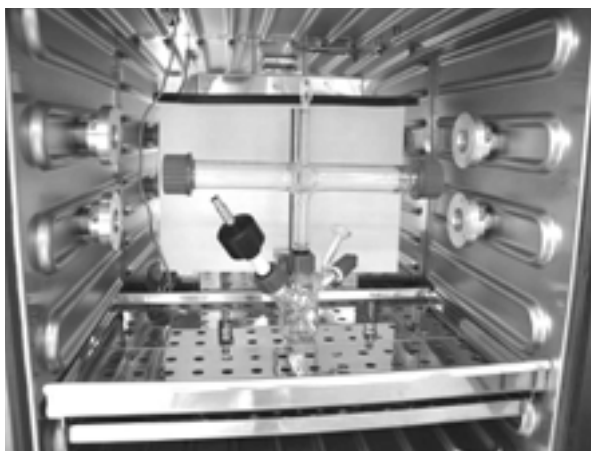
**Figure 3. Schematic representation of the main options for the HI section**



Apart from the membranes required for H<sub>2</sub> separation in the extractive distillation and electrolysis schemes, membranes could be used at other places in the cycle. In particular, pervaporation membranes [5] could allow removing water from the HI<sub>x</sub> mixture, thus providing a low energetic cost alternative to bypass the azeotrope.

Reactive distillation is the reference scheme chosen by the CEA [6]. However, the amount of hydrogen produced during the process depends closely on the I<sub>2</sub> and HI concentrations in the vapour, which means that the correct evaluation of the actual efficiency of reactive distillation requires the knowledge of liquid-vapour equilibrium data for the HI<sub>x</sub> mixture. The model currently used to represent this equilibrium is based on total pressure measurements performed at RWTH Aachen, and CEA has launched a program to measure the relevant partial pressures under process conditions (up to 300°C and 5 MPa). Like for the Bunsen section, this programme involves specifically designed experimental devices (Figure 4) as well as the development of suitable analytical methods. In particular, we have developed optical diagnostics (FTIR spectrometry, UV-visible spectrophotometry, Spontaneous Raman Scattering) to measure the composition of the vapour phase in the very concentrated conditions encountered in the process.

**Figure 4. Device for the study of partial pressures around 120°C**



***The Sulfuric acid section (section II)***

This section appears to be the best known one of the cycle, because of the experience gained in the sulphuric acid industry. In the proposed flow-sheet [6], sulphuric acid is concentrated through a series of flashes starting from low pressure. It is then dehydrated, before SO<sub>3</sub> is decomposed into SO<sub>2</sub>. This decomposition being only partial, non decomposed SO<sub>3</sub> is recombined with water, which allows to recovering its heat content.

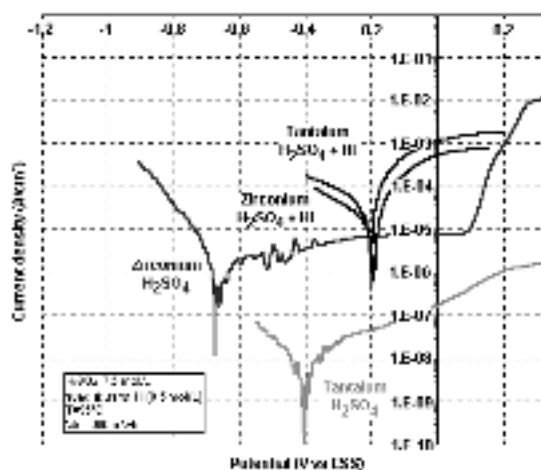
The main remaining questions concern the high temperature step of the process, namely  $\text{SO}_3$  into  $\text{SO}_2$  decomposition. The reaction, which requires a temperature in the  $870^\circ\text{C}$  range, will take place in a reactor and use the heat from a VHTR. This reactor will therefore also be a very high temperature heat exchanger, which will raise technological issues. Furthermore, a low pressure will be sought on the reaction side, whereas the heat transfer fluid will more likely be at high pressure. Finally, the temperature provided by a VHTR is not high enough to avoid the use of a catalyst for the reaction, and the long term resistance of this catalyst under the severe conditions that prevail will have to be ensured.

### Corrosion of materials

The Sulphur-Iodine cycle is very demanding on materials, which are exposed to very corrosive species at elevated temperatures and pressures.

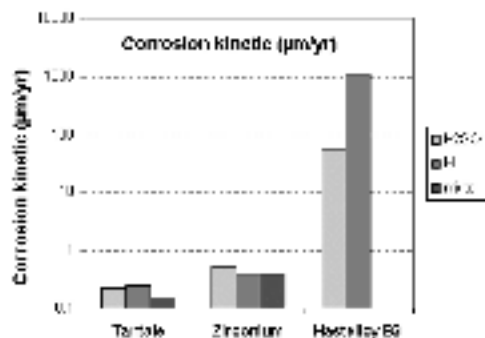
A literature review was conducted, which provided some materials as candidates for the section I (Bunsen) of the process: ceramics ( $\text{SiC}$ ,  $\text{Si}_3\text{N}_4$ ,  $\text{Al}_2\text{O}_3$ ), glass, fluorocarbons, Tantalum and Zirconium or Ni alloys. However, corrosion tests are necessary to assess the maximum temperature and acidity acceptable conditions, the long term behaviour and the corrosion mechanisms.

**Figure 5. Electrochemical tests on metallic materials**



As a first step, electrochemical and dipping tests have been conducted in each acid produced by the Bunsen-reaction (aqueous sulphuric acid and a mixture of hydriodic acid, iodine and water named  $\text{HIx}$ ), up to  $95^\circ\text{C}$ , so that first operating ranges are given for the candidate materials. Immersion tests performed in separate acids up to  $140^\circ\text{C}$  showed that Tantalum and Zirconium seem to be the most metallic relevant materials; nevertheless localized corrosion has been observed on Zirconium in liquid Bunsen condition ( $\text{H}_2\text{SO}_4\text{-HI-I}_2$  10wt%-10wt%-70wt%), (Figures 5 and 6).

**Figure 6. Corrosion rates of metallic materials**



The next step will be the study the long term behaviour of selected materials in separate and both acids, under the standard temperature and pressure conditions given by the flow-sheet of the process, in a pressurised reactor.

### Flow-sheet evaluation

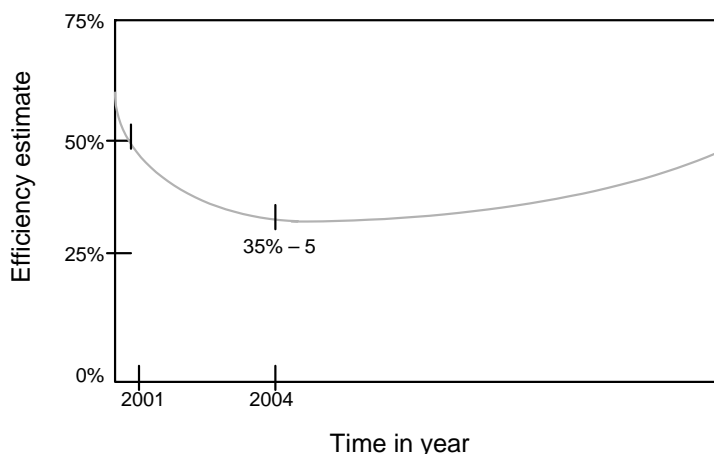
Another important fact is to describe the flow-sheet of the process to derive with sufficient accuracy the efficiency that remains the main criteria for the process evaluation. Obviously, we need to describe in detail each part of the process, including the various types of losses that can reduce the efficiency.

Several sub-sections are needed by the decomposition of a global reaction into several steps. For instance the sulphuric acid decomposition needs at least two or three steps, the first one to eliminate most of the water, the last one to crack the  $\text{SO}_3$ . So you have to look precisely at the way you want to operate, and for instance to introduce a re-circulation circuit and separate chemical reactors. Technology means are essential as they represent an actual part of the losses. Sometimes a component should not be feasible, for instance a compressor at high temperature or a heat exchanger with no temperature pinch. The way by which heat transfers and conveyance can be organized is dependent of the technology and also of the spatial organisation of the process.

One can illustrate the scientific approach generally used in process evaluation by drawing a curve giving the estimated efficiency versus time spent for research. In a first step, expert and manager judgments are used to enhance the ability of the process on a rough thermodynamic basis. The estimated efficiency is high. As a second step, a provisional flow-sheet is drawn and first thermodynamic calculations give a more realistic view based on existing or extrapolated data from literature. Some difficulties are arising, but there is no way to quantify them. Efficiency is decreasing. The third step needs more effort. It includes a first set of research. Detailed operational flow-sheets are presented based on the one hand on known technology and on the other hand on detailed calculation including some kinetics features. New experimental data become available. Process losses are accounted for. Laboratory scale experiments for the global process are scheduled and designed. Efficiency is low but realistic. Next step is carrying out a R&D programme in order to increase the efficiency and overcoming the difficulties encountered before. This is the longer step.

The example of the Sulphur-Iodine process studies is illustrated on Figure 7 based mainly on the work done at CEA and together with SNL and GA in the US, in the framework of an International NERI contract. For the last step, not yet achieved, several tracks are yet identified as described above in this paper.

**Figure 7. Estimated efficiency of a Hydrogen production process as a function of time spent in research. The case of the S/I process**



This curve shows that efficiency evaluation involves several steps so that to compare various processes among them, one needs to know the step he reached, that is depending on the level of knowledge he has got on the process. For that purpose, we try to identify clearly the various steps and to fix some data that could be used in each of them. For instance step 1 is based only on thermodynamics evaluation. Step 2 deals with reversible heat and work requirement calculations of the reactions, giving an upper bound of the efficiency. Step 3 needs detailed flow-sheet calculations including components efficiencies such as pumps, compressors and heat exchangers pinch. In this later step, we propose to set up a list of standard parameters to be used in the calculation. For instance the pinch for a gas-gas heat exchanger could be set to 50°C as a reference; the heat coming from the reactor is at 950°C, and so on . This work is underway in collaboration with ANL in the US.

### Alternative cycles evaluation

Other cycles working at a temperature compatible with the use of a nuclear reactor, that is to say less than 1 000°C, are currently under evaluation at CEA.

The UT\_3 cycle from Tokyo University with Calcium Bromide has been studied including some experiments on basic reactions. It was shown that the transformation of  $\text{CaBr}_2$  in  $\text{CaO}$  was largely unfinished around 750°C with a bulk solidification. The transformation of  $\text{FeBr}_2$  in  $\text{Fe}_3\text{O}_4$  was not effective with preferably the formation of  $\text{Fe}_2\text{O}_3$  (in a range of 600 to 700°C) which modifies considerably the cycle. Also was noticed the azeotropic mixture  $\text{H}_2\text{O}$ - $\text{HBr}$  at 47%  $\text{HBr}$  and the eutectic  $\text{CaBr}$ - $\text{CaO}$  above 700°C. Finally, the MASCOT pilot experiment conducted in 1980 was considered too complex, far from an easy to ride process, especially due to the rapid ageing of the compounds and the formation of clusters. Knowing that Bromide is a toxic compound, this cycle has been given up.

The Sulphate cycle using a metal M (M = Fe, Ni, Co or Mn) has been evaluated on a theoretical basis. Tests are underway to verify the efficiency of the elementary reactions and the presence of side reactions. The process needs to transfer solids from one reaction to another, which is not easy generally speaking.

Concerning the Cerium Chloride cycle, a first flow-sheet has been issued and separate reactions were experimentally tested. Two of them indicated a correct behaviour with a good kinetics. Work is ongoing to achieve the experiment and to derive data for flow-sheet calculations.

The hybrid Sulphur cycle is the one that benefits from the most important program of evaluation, due to its synergy with the Sulphur-Iodine cycle on the sulphuric acid decomposition and the potential simplification that it introduces in the full process suppressing the Iodine utilisation. A first flow-sheet has been issued and calculations associated to this flow-sheet gives 35% efficiency at least but with some hypothesis on the electrolysis part. Work is underway to achieve a detailed flow-sheet at the same level of knowledge than the one used on the S/I process. Specific tests on the  $\text{SO}_2+\text{H}_2\text{O}$  electrolysis are scheduled to determine the operating voltage and to test various combinations of anodic, cathodic and electrolytic materials in order to enhance the efficiency.

## Conclusion

Thermochemical cycles, and in particular the Sulphur-Iodine cycle, have been the subject of renewed intense interest in the last years. The accurate evaluation of their industrial potential is difficult, as it involves many aspects, from scientific questions such as the knowledge of thermodynamic data to safety, acceptability and economic assessments.

In particular, as the Sulphur-Iodine cycle is investigated worldwide, the problems that have to be solved appear more and more clearly. However, the basic advantages of this cycle remain valid:

- it only involves the handling of fluids;
- it is purely thermochemical, which is associated with low electricity need and therefore high potential efficiency;
- its coupling to a VHTR seems promising.

The CEA has launched an integrated program to choose by 2008 the most promising way to produce hydrogen using the high temperature heat available from a VHTR. In order to develop its own expertise on thermochemical cycles assessment, the CEA has chosen to develop a scientific approach based on data acquisition (development of devoted devices and specific analytical methods) and modelling (physical models, flow-sheet analysis, systemic approach). This approach comprises:

- Development of a methodology for process comparison.
- Acquisition of basic thermodynamic data.
- Flow-sheet analysis and development.
- Pre-conceptual design of a hydrogen production plant coupled to a VHTR, including energy distribution and safety issues.
- Efficiency and cost analysis based on the previous items.

The CEA has chosen to concentrate on a limited number of processes, namely the Sulphur-Iodine cycle and high temperature electrolysis, with the hybrid Sulphur cycle as a back-up, cycles that have been selected as being the most promising. Experience gained on the evaluation of these options is built on to perform a more theoretical assessment of other potentially interesting cycles.



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## TABLE OF CONTENTS

<b>FOREWORD</b> .....	3
<b>OPENING SESSION</b> .....	9
<i>Thierry Dujardin</i> Welcome address .....	11
<i>Osamu Oyamada</i> Opening Remark .....	13
<b>SESSION I</b>	
<b>The prospects for Hydrogen in Future Energy Structures and Nuclear Power’s Role</b> .....	15
<i>Chair: M.C. Petri</i>	
<i>M. Hori, M. Numata, T. Amaya, Y. Fujimura</i> Synergy of Fossil Fuels and Nuclear Energy for the Energy Future .....	17
<i>G. Rothwell, E. Bertel, K. Williams</i> Can Nuclear Power Complete in the Hydrogen Economy? .....	27
<i>S. Shiozawa, M. Ogawa, R. Hino</i> Future Plan on Environmentally Friendly Hydrogen Production by Nuclear Energy .....	43
<b>SESSION II</b>	
<b>The Status of Nuclear Hydrogen Research and Development Efforts around the Globe</b> .....	53
<i>Chair: M. Methnani, W.A. Summers</i>	
<i>M. Hori, S. Shiozawa</i> Research and Development For Nuclear Production of Hydrogen in Japan .....	55
<i>A.D. Henderson, A. Taylor</i> The U.S. Department of Energy Research and Development Programme on Hydrogen Production Using Nuclear Energy .....	73
<i>F. Le Naour</i> An Overview of the CEA Roadmap for Hydrogen Production.....	79
<i>Y. Sun, J. Xu, Z. Zhang</i> R&D Effort on Nuclear Hydrogen Production Technology in China .....	85

<i>A.I. Miller</i> An Update on Canadian Activities on Hydrogen .....	93
<i>Y-J. Shin, J-H. Kim, J. Chang, W-S. Park, J. Park</i> Nuclear Hydrogen Production Project in Korea.....	101
<i>K. Verfondern, W. von Lensa</i> Michelangelo Network Recommendations on Nuclear Hydrogen production .....	107
<b>SESSION III</b> <b>Integrated Nuclear Hydrogen Production Systems .....</b>	<b>119</b>
<i>Chairs: A. Miller, K. Verfondern</i>	
<i>X. Yan, K. Kunitomi, R. Hino and S. Shiozawa</i> GTHTTR300 Design Variants for Production of Electricity, Hydrogen or Both.....	121
<i>M. Richards, A. Shenoy, K. Schultz, L. Brown, E. Harvego, M. Mc Kellar, J.P. Coupey, S.M. Moshin Reza, F. Okamoto, N. Handa</i> H2-MHR Conceptual Designs Based on the SI Process and HTE .....	141
<i>P. Anzieu, P. Aujollet, D. Barbier, A. Bassi, F. Bertrand, A. Le Duigou, J. Leybros, G.Rodriguez</i> Coupling a Hydrogen Production Process to a Nuclear Reactor .....	155
<i>T. Iyoku, N. Sakaba, S. Nakagawa, Y. Tachibana, S. Kasahara, K. Kawasaki</i> HTTR Test Programme Towards Coupling with the IS Process.....	167
<i>H. Ohashi, Y. Inaba, T. Nishihara, T. Takeda, K. Hayashi Y. Inagaki</i> Current Status of Research and Development on System Integration Technology for Connection Between HTGR and Hydrogen Production System at JAEA .....	177
<b>SESSION IV</b> <b>Nuclear Hydrogen Technologies and Design Concepts .....</b>	<b>187</b>
<i>Chairs: K. Kunitomi, J.S. Herring, Y.S. Shin, T. Takeda</i>	
<i>K. Onuki, S. Kubo, A. Terada, N. Sakaba, R. Hino</i> Study on Thermochemical Iodine-Sulfur Process at JAEA .....	189
<i>S. Kubo, S. Shimizu, H. Nakajima, K. Onuki</i> Studies on Continuous and Closed Cycle Hydrogen Production by a Thermochemical Water-Splitting Iodine-Sulfur Process .....	197
<i>A. Terada, Y. Imai, H. Noguchi, H. Ota, A. Kanagawa, S. Ishikura, S. Kubo, J.Iwatsuki, K. Onuki, R. Hino</i> Experimental and Analytical Results on H <sub>2</sub> SO <sub>4</sub> and SO <sub>3</sub> Decomposers for IS Process Pilot Plant.....	205

<i>M.A. Lewis, M.C. Petri, J.G. Masin</i> A Scoping Flowsheet Methodology for Evaluating Alternative Thermochemical Cycles .....	219
<i>S. Suppiah, J. Li, R. Sadhankar, K.J. Kutchcoskie, M. Lewis</i> Study of the Hybrid Cu-Cl Cycle for Nuclear Hydrogen Production.....	231
<i>M. Arif Khan, Y. Chen,</i> Preliminary Process Analysis and Simulation of the Copper-Chlorine Thermochemical Cycle for Hydrogen Generation .....	239
<i>W.A. Summers, J.L. Steimke</i> Development of the Hybrid Sulfur Thermochemical Cycle .....	249
<i>P. Anzieu, P. Carles, A. Le Duigou, X. Vitart, F. Lemort</i> The Sulfur-Iodine and Others Thermochemical Processes Studies at CEA .....	259
<i>K-K. Bae, K-S. Kang, S-D. Hong, C-S. Park, C-H. Kim, S-H. Lee, G-J. Hwang</i> A Study on Hydrogen Production by Thermochemical Water-splitting IS (Iodine-Sulfur) Process .....	269
<i>P. Zhang, B. Yu, L. Zhang, J. Chen, J. Xu</i> Present Research Status and Development Plan of Nuclear Hydrogen Production Programme in INET .....	277
<i>T. Nakagiri, T. Kase, S. Kato, K. Aoto</i> Development of the Thermochemical and Electrolytic Hybrid Hydrogen Production Process for Sodium Cooled FBR.....	287
<i>J.S. Herring, J.E. O'Brien, C.M. Stoots, G.L. Hawkes, P. Lessing, W. Windes, D. Wendt, M. Mc Kellar, M. Sohal, J.J. Hartvigsen</i> Progress in High-temperature Electrolysis for Hydrogen Production.....	297
<i>Y. Kato</i> Possibility of a Chemical Hydrogen Carrier System Based on Nuclear Power .....	309

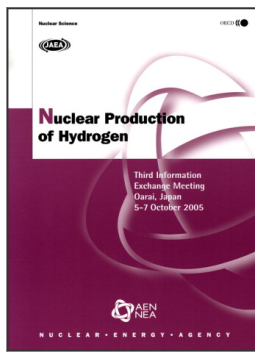
## **SESSION V**

### **Basic and Applied Science in Support of Nuclear Hydrogen Production .....**

*Chairs: Y. Kato, P. Anzieu, Y. Sun*

<i>C-H. Kim, B-K. Kim, K-S. Kang, C-S. Park, S-H. Lee, S-D. Hong, G-J. Hwang, K-K. Bae</i> A Study on the HI Concentration by Polymer Electrolyte Membrane Electrodialysis.....	321
<i>H-S. Choi, G-J. Hwang, C-S. Park, H-J. Kim, K-K. Bae</i> The Preparation Characteristics of Hydrogen Permselective Membrane for Higher Performance in IS Process of Nuclear Hydrogen Production .....	329
<i>H. Karasawa, A. Sasahira, K. Hoshino</i> Thermal Decomposition of SO <sub>3</sub> .....	337

<i>S. Fukada, S. Suemori, K. Onoda</i> Direct Energy Conversion by Proton-conducting Ceramic Fuel Cell Supplied with CH <sub>4</sub> and H <sub>2</sub> O at 600-800°C .....	345
<i>M. Ozawa, R. Fujita, T. Suzuki, Y. Fujii</i> Separation and Utilisation of Rare Metal Fission Products in Nuclear Fuel Cycle as for Hydrogen Production Catalysts?.....	355
<i>H. Kawamura, M. Mori, S-Z. Chu, M. Uotani</i> Electrical Conductive Perovskite Anodes in Sulfur-based Hybrid Cycle .....	365
<i>Y. Izumizaki, K-C. Park, Y. Tachibana, H. Tomiyasu, Y. Fujii</i> Generation of H <sub>2</sub> by Decomposition of Pulp in Supercritical Water with Ruthenium (IV) Oxide Catalyst.....	381
<b>SESSION SUMMARIES</b> .....	389
<b>RECOMMENDATIONS</b> .....	391
<i>Annex A: List of Participants</i> .....	393
<i>Annex B: Meeting Organisation</i> .....	411
<i>Annex C: Additional Presentations to the Second HTTR Workshop</i> .....	413



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