1. **Project Name:** **Advanced Chlor-Alkali Technology**

2. **Lead Organization:** Los Alamos National Laboratory
   Los Alamos, NM 87545

3. **Principal Investigator:** Jerzy Chlistunoff
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4. **Project Partners:** Los Alamos National Laboratory (electrochemical reactor design and testing). Point of contact: Jerzy Chlistunoff – phone 505-667-7192, E-mail: jerzy@lanl.gov
   Negotiations with PPG Industries are in final stages.

5. **Date Project Initiated and FY of Effort:** 10/01/2001, 2nd fiscal year

6. **Expected Completion Date:** 09/30/2004

7. **Project Technical Milestones and Schedule:**

Description	Planned Completion	Actual Completion
Report effects of cathode hardware on caustic current efficiency (CCE)	12/31/02	12/31/02
Complete testing of cathode hardware effects on CCE	6/30/03	
Complete long-term testing of the hardware designed to reduce decline in CCE	9/30/04	
Introduce fully automated power backup	Date unknown	
Report effects of cathode structure and composition on the cell performance stability	3/31/04	
Extended testing of the cathode structure providing best performance stability or evaluation of alternative approaches	9/30/04	
Identify unsupported catalysts giving highest performance	11/30/03	
1 month life test of highest performing unsupported catalyst	2/15/04	
Complete evaluation of different catalysts and carbon-free structures	9/30/04	
Demonstrate stability of negligible peroxide levels with newly designed cathode structure during long term operation	3/31/02	3/31/02
Modify the new cathode structure to achieve better performance stability	9/30/02	9/30/02

Report effects of elimination of carbon from cathode structure	11/30/03	
Complete comparative evaluation of carbon free and carbon supported structures for both peroxide reduction and overall electrode performance	6/30/04	
Complete testing and evaluation of different electrocatalytic surfaces of the patterned flow field as peroxide decomposition catalysts	2/28/03	2/28/03
Identify most corrosion-resistant material and/or coating of the cathode hardware for further testing	3/31/03	3/31/03
Complete evaluation of the material/coating for the cathode hardware	9/30/04	
Report highest operational current density	8/31/04	
Modify the anode structure to achieve better cell performance	4/30/03	4/30/03
Demonstrate low-cost, non-machined anode flow field	8/31/03	
Complete comparative testing of cation exchange membranes	6/30/03	
Report lowest viable cell clamping pressure	6/30/04	
Evaluate operation of a taller cell at high throughput	9/30/04	

8. **Past Project Milestones and Accomplishments:**

Previous research indicated that our advanced chlor-alkali cells could be operated at much higher current densities than conventional membrane cells, without significant penalty in cell voltage. But, caustic current efficiency at high throughputs was noticeably lower. Other problems encountered included corrosion of the cathode hardware components, sometimes severe, and generation of unacceptably high levels of sodium peroxide by-product. The peroxide formation problem was virtually eliminated in 2001 by introducing a new cathode structure, which is now the subject of a patent application. Although long-term effectiveness of the structure in destroying the peroxide by-product was demonstrated, it was also found that the cathode was more susceptible to flooding by caustic solution, an undesirable phenomenon that led to an increase in cell voltage and a corresponding decrease in cathode lifetime. These shortcomings helped establish guidelines for our research efforts for this past year. The focus has been on increasing caustic current efficiency, reducing cell corrosion, improving flooding characteristics of the peroxide-destroying cathode structure and finding complementary methods for peroxide decomposition.

We introduced modifications to the cathode structure aimed at reducing its flooding susceptibility. These modifications resulted in better flooding resistance, but increased peroxide generation rates. While still quite low initially, the peroxide generation rates increased slowly over time. Analysis of the results led to defining further steps to be taken to achieve both excellent flooding characteristics and consistently low peroxide levels.

Catalysis of peroxide decomposition by the cathode hardware is considered a potentially effective complementary way of reducing peroxide generation.

In a search for corrosion resistant materials for the cathode hardware, it was found that gold plated nickel and stainless steel parts exhibited poor corrosion resistance in a concentrated caustic/oxygen environment. Silver plated nickel parts on the other hand exhibited excellent corrosion resistance, both under normal operating conditions and during extended power outages. The latter materials are also less expensive.

Cathode hardware modifications aimed at improving caustic current efficiency did not lead to the desired effect. However the modifications unexpectedly resulted in a quite beneficial reduction of the cell voltage, with a corresponding lowering of energy consumption. The savings amount to approximately 8% at the high throughput technology target of 10 kA/m^2.

A substantial increase in caustic current efficiency at high throughputs was achieved through modifications of the anode structure. A patent disclosure relevant to this development is in preparation.

9. **Planned Future Milestones:**

An updated project schedule is presented in the table above. Our plans for the next year address major barriers towards scaling up and commercializing the technology, including studies on the remaining causes of performance losses and some cost related issues.

Oxygen diffusion cathodes with carbon-supported Pt catalyst, currently used in the project, exhibit low corrosion resistance during power outages. Corrosion of carbon support during down time leads to the catalyst loss and significant decline of the electrode performance. We plan to explore possibility of introducing electrodes with unsupported catalysts that will exhibit significant corrosion resistance during down time. Depending on the outcome of the study, a decision will be made as to the final selection of the cathode composition and structure.

Caustic current efficiency, cell voltage and caustic purity critically depend on performance of the ion-exchange membrane separating the cathode and anode compartments. Different membranes will be tested to select the one that offers the best overall performance.

Anode flow-field is responsible for transport of brine and chlorine in the anode compartment and thus for the overall anode performance. Machined serpentine channel, DSA coated titanium flow-field, currently used in our cells, guarantees transport efficiency, but its cost is relatively high. We plan to introduce easily scalable, non-machined flow-field that will lower the cell manufacturing cost without compromising the anode performance.

10. **Issues/Barriers:**

LANL requirements forced a shutdown of experimental operations pending electrical safety upgrades to the chlor-alkali test apparatus. This process took longer than expected and led to delays in programmatic work and consequent schedule changes. The modified schedule in the table above includes all the tasks called out in the initial proposal, but there is now little time for additional experiments that may arise due to unexpected experimental results.

A critical component to developing this technology requires the manufacture of electrodes with unsupported catalysts. Some difficulties may arise since standard methods used for preparation of membrane electrode assemblies (MEAs) for fuel cells, from which this technology is derived, may not be applicable in the chlor-alkali cells due to difficulty in obtaining proper ion-exchange polymeric binder and unsuitable thermal and swelling properties of specialized chlor-alkali membranes.

11. **Intended Market and Commercialization Plans/Progress:**

The new technology under development in this project offers significant energy savings (~30%) when compared to state-of-the-art membrane technology. It also offers higher throughputs than conventional technologies, which reduces initial capital investment costs. Projected savings do not take into account the energy value of the hydrogen by-product generated in conventional cells. This material could potentially be captured and used but is currently typically vented to the atmosphere. The new oxygen-consuming membrane cells are also more compact than the mercury and diaphragm cells currently in use, requiring less space as the older cells are replaced with the new technology cells.

The ultimate goal of this research is to develop the technology to the point where it will become sufficiently attractive to scale-up and implement. The most natural way to achieve this goal is through a close collaboration with a representative of the chlor-alkali industry. Negotiations with a potential strategic partner, PPG Industries, are in the final stages.

12. **Patents, publications, presentations:**

1-patent application in process: Method for suppression of peroxide generation in chlor-alkali cells with oxygen-consuming cathodes.
1-invention disclosure in submittal process: Method for improving caustic current efficiency in a chlor-alkali membrane cell.
2-presentations:
L.Lipp, S.Gottesfeld, J.Chlistunoff, *Effects of Operating Conditions on Selected Performance Characteristics of Oxygen-Depolarized Chlor-Alkali Cell*. Centennial Meeting of the Electrochemical Society, Philadelphia, May 2002
L.Lipp, S.Gottesfeld, J.Chlistunoff, *Zero-Gap Chlor-Alkali Cell With Oxygen-Consuming Cathode: Hardware Effects on the Cell Operation*. 203rd Meeting of the Electrochemical Society, Paris, France, April-May 2003