Mediated Electrochemical Oxidation (MEO) Based Technology

LLNL-97003436

Final Report

CRADA No. TC-0349-92

Date: 7-18-96

TACT:

Revision: 1

A. Parties

The project is a relationship between the Lawrence Livermore National Laboratory (LLNL) and EOSystems, Inc.

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Lawrence Livermore National Laboratory
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B. Project Scope

The goal of this CRADA was the continued research and development by LLNL, and the commercialization by EOSystems, Inc., of the waste treatment technology known as Mediated Electrochemical Oxidation. MEO is a non-thermal electrochemical technology developed in part at LLNL for the destruction of organic waste streams; this technology has wide applications in the government, manufacturing, biomedical and industrial sectors. The system uses an electrochemical cell to generate highly oxidizing 'mediators' in an acidic aqueous solution, which subsequently react with organic waste and convert it to carbon dioxide and water.

The broad research responsibilities of LLNL in this CRADA were the investigation of numerous cell electrode materials and materials of construction, the evaluation of the process chemistry, and the testing of a flow visualization cell and a functional prototype. Major deliverables included: a determination of suitable electrode materials, an investigation of the destruction efficiency for numerous organic substrates, the construction and testing of a flow visualization cell, and the testing of a functional prototype commercial cell. The responsibilities of EOSystems included the definition of the market and potential customers, the design and engineering of the flow visualization and prototype cells, and the commercialization of the MEO units. Deliverables included the selection of the process and ancillary systems, the design of a flow visualization cell, and the design and construction of a prototype cell. In general, most of the deliverables were met by both partners, although unexpected technical difficulties delayed some of the delivery dates and forced the adoption of a modified statement of work. However, the primary, original project goals were completed on-time and on-budget.

C. Technical

The technical aspects of this project can be broken down into three categories. The first area of research was an investigation of various materials for use as electrodes and in the construction of the cell and associated systems. The second area was the investigation of the process chemistry, and the determination of destruction efficiencies for various organic substrates. The final area was the design, construction, and testing of several prototype systems. In all of

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these areas, design and engineering aspects were shared by both LLNL and EOS, while all experimental work was done at LLNL. A chart is included below which lists these categories and their completion dates. Along with the technical issues, several issues relating to the commercialization of the technology were addressed by EOSystems. These issues are discussed in the Partner Contribution section of this report.

Part I:

	<u>Completed Tasks</u>	<u>Completion date</u>
I-1	Electrode development	
I-1-1	Investigation of Pt-Ir sputtered coatings on Ti	December 1994
I-1-2	Testing of commercially available electrode materials (e.g., anodes: Pt electroplated on Ti, DSA type electrodes; cathodes: Cu, Ni, Ni alloys)	March 1995
I-1-3	Testing of refractory materials for anodes and cathodes (e.g., Nb, Ta, Zr – electroplated with Pt for anodes)	March 1996
I-1-4	Development of inexpensive bipolar electrode materials (e.g., Cu or Ni alloys coated with refractory materials)	July 1995
I-2	Materials of construction	
I-2-1	Corrosion testing of moldable plastics	June 1995
I-2-2	Stress testing of selected plastics	September 1995

Part II:

	<u>Task</u>	<u>Completion date</u>
II-1	MEO Process Development for Organics Destruction	
II-1-1	Selection of process chemistry	August 1995
II-1-2	Development of process for non-chlorinated miscible organics	September 1995
II-1-3	Development of process for non-chlorinated immiscible organics	November 1995

Part III:

III-1	Prototype Development	
III-1-1	Flow visualization unit fabrication and testing	March 1995
III-1-2	Prototype design and fabrication	March 1996
III-1-3	Prototype testing	April 1996
III-2	Commercialization of MEO Process for Organics Destruction	
III-2-1	Further testing of prototype system	in progress
III-2-2	Conduct detailed market surveys and identify potential customers	December 1994
III-2-3	Begin production of MEO systems for sale	December 1996

It was anticipated early in the CRADA that Pt/Ir coating sputtered on titanium substrates would provide a suitable electrode material for use in the cobalt sulfate/sulfuric acid solution used in the MEO process. Corrosion of the electrodes was perhaps the single most difficult technical hurdle of this project, as the combination of acidic media, highly oxidizing species, chlorine species, free radicals, and organic fragments drastically shorten the useable lifetime of many common (and inexpensive) electrodes materials. However, after extensive investigation of these and other electrode materials, it was determined that none could be found which were economically feasible for use in the EOSystems commercial cell. Thus, after two years of pursuing the cobalt sulfate/sulfuric acid based MEO system, the decision was made to switch to a cobalt nitrate/nitric acid based system. Platinum coatings on titanium substrates had been effectively used in such nitric acid based systems in the past, with drastically reduced corrosion. A detailed list of the electrode and materials of construction data for both sulfuric and nitric acid based systems is included in the following tables. To summarize, the most suitable construction material is Kynar (PVDF); the most suitable anode material is platinum on titanium, and the most suitable cathode material is titanium.

C. Technical (cont.)**Materials testing-COBALT SULPHATE/SULFURIC ACID BASED MEO UNITS****Construction Materials**

- Survivors:**

<u>Materials</u>	<u>Hours</u>	<u>Comments</u>
<u>Plastics:</u>		
PVDF#1(Kynar), HMW PE	5100	
CPVC	1740	
HD & UHMW PE	3300	-2%wt
UHXL PE	2680	+1%wt
ECTFE	1870	
ETFE	1870	
PCTFE	1870	
PEEK	1870	-6% wt
PFA	1870	
PVDF#2	1870	
PP	1440	<ul style="list-style-type: none"> •-3%wt •Battery-grade copolymer resin from Exxon.
<u>Gasket Materials:</u>		
Viton gasket	4090	+17% wt.
Viton O-ring	4090	+16% wt.
Kalrez	4090	+9% wt.
Chemraz	4090	
<u>Metals:</u>		
Tantalum	5720	
Niobium	3380	

• **Failures:**

<u>Materials</u>	<u>Hours</u>	<u>Comments</u>
<u>Plastics</u>		•Arbitrary failure criterion for wt. change: 20% in 4000 hr (about 1/2 year).
PP	1440	•Battery-grade copolymer resin, Rexene. •Only -1%wt but brittle fracture during handling.
PPS (30% glass, Ryton)	1520	-8%wt
PVC (2: white, gray)	5120	+30% wt.
CPVC (2)	4090, 3200	+38%, +50% wt.
Valox (black, GE)	1870	-10%wt, much material loss around hole.
<u>Clear plastics:</u>		
PC	300	Opaque after 300 h but still intact after 1200 hr
PMMA	2700	+50% wt., semi-transparent
PP	500	Crumbly. Type of resin unknown.
PVC	350	Brown, opaque
<u>Metals:</u>		
Titanium	2900	-65 % wt at 60±5°C but very little wt loss during 950 hr at 35±5°C
Incoloy 825 (Ni alloy)	1010	-6% wt. at 190h, gone at 1010h.

Electrode Materials

<u>Materials</u>	<u>Hours</u>	<u>Comments</u>
<u>Clad: Nb/Cu/Nb/Pt (1)</u>	300	Slight cupping.
<u>Nb clad with Pt (6)</u> <i>Anode Survivors (3/3)*</i> <i>Cathode Survivors (3/3)*</i>	 230-740 (450 avg) 300-900 (600 avg)	 •No clear failures; Small loss of Pt at edge of one is not unexpected because of shearing.
<u>Nb electroplated with Pt (15)</u> <i>Anode Survivors (310)</i> <i>Anode Failures (710)</i> <i>Cathode Survivors (5/5)</i>	 200-1000 (650 avg) 20, 40 660-1650 690 300-1150 (700 avg)	 EPI and Intrepid HEEC (discontinue testing because 2 out of 3 failed prematurely) EPI (4). •EPI and HEEC
<u>Sputtered Ta/I-825 Ni/Ta/Pt (2)</u>	4 90	Pt coating cracked open. All layers gone except Ta cathode.
<u>Ta electroplated with Pt (2)</u> <i>Anode tests invalid</i>	 260, 540	 Both anode failures are an artifact of embedded alumina polishing particles.
<u>Ti electroplated with Pt (18)</u> <i>Anode Survivors (7/13)</i> <i>Anode Failures (6/13)</i> <i>Cathode Failures: (5/5)</i>	 600-750 80-1300 (400 avg) 40-400 (100 avg)	 Variable anode lifetimes but all cathodes failed by hydriding. Bare Ti likewise hydrides.
<u>Ti sputtered with Pt/Ir (5) or Ir (1)</u>	200 avg	All coatings disappeared, at least in part.

<u>Ti4O7 electroplated with Pt</u>		No real failures but not sufficiently tested.
<i>Anode Survivors (1/1):</i>	225	Conduction fine but Pt beginning to flake off.
<i>Cathode Survivors (1/1):</i>	225	
<u>Ti thermally coated with IrO2; DSA (12)</u>		•Failures probably from Cl poisoning.
<i>Anode Failures at 70°C (12/12):</i>	20-600 (200 avg)	Returned 10 to producers, EC and EPI, for failure analysis. EPI suspected metal/oxide interface oxidation at temp > 60°C but results below refute this.
<i>Anode Failures at 55°C (2/2):</i>	120, 135	
<u>Cu or Ni electroplated with Pt (10)</u>		
<i>Anode Failures (5/5):</i>	20-105	All failed by heavy cavitation with a shell of Pt remaining.
<i>Cathode Failures: (5/5)</i>	10-105	Pt peeling.
<u>Bare metal anodes (2)</u>		
Pt	400	Corrosion rate=7µm/1000h but some may be artifact from surface strain.
<u>Bare metal cathodes: (35)</u>		Only Nb and Ta have survived.
• Survivors:		
Nb (7/7)	200-1650 (500 avg)	•55°C
	1650	•70°C
		•No hydrides
Ta (3/3)	480-1700 (900 avg)	•55°C
		•1 mil electrodeposit at 620h.
• Failures:		
Cu	215	•55°C
		•2mil loss = 120 MPY (mils/yr)
	75	•55°C
(7)	40-600	•70°C
		•Large corrosion losses
Ni	620	•55°C
		•50mil loss = 700 MPY
(5)	30-490	•70°C
		•Large corrosion losses
Ni alloy, I-825	515	•55°C
		•2mil loss = 35 MPY
Ti (2)	100, 650	•70C
		•Heavily hydrided
316SS	30	•70C
		•Heavily corroded
Ni	110	•Estimated 1mil loss = 80 MPY, but requires metallography later.
Pb (6)	40-710	Not suitable: nil corrosion at 70°C but overpotential too high (1.2V versus 0.1V Pt, 0.4V Nb).

C. Technical (cont.)**Materials testing-COBALT NITRATE/NITRIC ACID BASED MEO UNITS****Construction Materials**

No new materials of construction were tested in the nitric acid based system, as previous research by LLNL demonstrated that PVDF (Kynar) and Viton are suitable for these applications.

Electrode Materials**I. Anodes**

Solid Pt: Duration of testing was 1957 hours, and the projected rate of Pt dissolution (measured by weight loss) is approximately 1.2 mm (50 μ inches) per 1000 hours.

Electroplated Pt on Ti: Two electrodes tested; still effective after 1610 and 1835 hours.

Platinum clad on Nb: Survived 1800 hours with no appreciable corrosion.

Electroplated Pt on Nb (HEEC): Two electrodes tested; coating gone after 16 and 20 hours.

Ni aluminide on Ni: Electrode dissolved after 5 hours.

II. Cathodes

Titanium: Approximate average 7 % weight loss per 1000 hours, otherwise appeared fine.
Total of 2000 hours.

Stainless Steel (316L): Lost significant fraction of weight after 16 hours.

Niobium: Still working after 50 hours; testing stopped because of high cost of Nb.

C. Technical (cont.)

SUMMARY OF ORGANICS DESTRUCTION DATA

Organic	0.5 M CoSO ₄ in 4 M H ₂ SO ₄		0.5 M Co(NO ₃) ₂ in 4 M HNO ₃	
	Run #, Conditions	D.E. *	Run #, Conditions	D.E. *
Toluene	20A,10amps,35C,surf,28hrs	54%	42A,10amps,35C,surf,6hrs	100%
2,4-D	40B,7.5amps,55C,6hrs	33%	64A,7.5amps,55C,6hrs	93%
			43A,10amps,35C,7hrs	46%
Methyl methacrylate	28B,10amps,35C,7hrs	48%	44A,10amps,35C,7hrs	80%
Acetone	20B,10amps,35C,5hrs	62	45A,10amps,35C,7hrs	65%
Acetone	21B,10amps,55C,15hrs	41%	46A,10amps, 55C,6hrs	87%
Methy isobutyl ketone	19B,10amps,35C,15hrs	62%	47A,10amps,35C,6hrs	70%
Methyl ethyl ketone	24B,10amps,35C,14hrs	38%	48A,10amps,35C,7hrs	69%
Propylene Glycol	23B,10amps,35C,8hrs	64%	49A,10amps,35C,6hrs	85%
AP tar	22A,10amps,35C,5hrs	76%	50A,10amps,35C,5hrs	100%
Acetic Acid	47B,10amps,35C,17hrs	73%	51A,10amps,35C,8hrs	82%
Nitromethane	16A,10amps,35C,2.5hrs	39% ¹	52A,10amps,35C,5hrs	0% ¹
Tributyl phosphate	31B,10amps,35C,7hrs	56%	53A,10amps,35C,6hrs	50% ²
Epichlorohydrin	26B,10amps,35C,2.5hrs	87%	54A,10amps,35C,6hrs	100%
Trichloro ethylene	48B,10amps,35C,4hrs	33%	55A,10amps,35C,6.5hrs	15% ³
Dichlorobenzene	27B,10amps,35C,7hrs	41%	56A,10amps,35C,5hrs	4%
Vinyl acetate	30B,10amps,35C,3hrs	48%	57A,10amps,35C,6.5hrs	55%
Styrene	32B,10amps,35C,4hrs	37%	58A,10amps,35C,5hrs	38%
Butyl acrylate	38B,7.5amps,55C,6hrs	26%	61A,7.5amps,55C,5hrs	61%
			59A,10amps,35C,6hrs	64%
Barlox surf.	21A,10amps,35C,5hrs	87%	60A,10amps,35C,5hrs	91%
Diamino toluene	43B,10amps,35C,6hrs	100%	62A,10amps,35C,6hrs	95%
ethylene glycol	45B,10amps,35C,3 hrs	100%	97A,10amps,35C,6hrs	100%
Dodecane	26A,10amps,35C,45hrs, emulsion	0%	78A,10amps,55C,30hrs, 2%emuls.	66% ⁴
Dinitrotoluene	not done (insoluble)		63A,10amps,35C,6hrs	100%
Dichloro propanol	23A,10amps,35C,119hrs	99%	66A,10amps,35C,98hrs	72% ⁵
Isopropanol	18A,10amps,35C,20hrs	54%	65A,10amps,35C,53hrs	61%

*Destruction Efficiency defined as ratio of volume of CO₂ produced to that theoretically predicted from the amount of organic added to cell.

¹ Destruction efficiency increased upon addition of ethylene glycol: 85% for H₂SO₄ and 100% for HNO₃.

² Run still producing significant CO₂ at end.

³ Ethylene glycol added midway through run.

⁴ D.E. increased to 100% under very slow feed rates and with 8% HNO₃.

⁵ D.E. increased to near 100% under slower feed rates

C. Technical (cont.)**PROTOTYPE CELL TESTING**

A bench-scale prototype cell was designed and engineered by EOSystems and Fluoroware, Inc., and tested at LLNL. The cell is constructed of Kynar, and employs titanium cathodes and platinum on titanium anodes. The prototype cell is of a bipolar configuration, and contains two channels. Tests were conducted at approximately 50 °C in a cobalt nitrate/nitric acid media using a power supply of 400 amps.

TEST A (Ethylene Glycol)

Feed rate of ethylene glycol was 167 ml EG/hr (average over entire run); this feed rate was assuming a coulombic efficiency of virtually 100%. Based on this feed rate, the overall destruction efficiency was about 89%.

Run Time (hours)	Temp. (°C)	Voltage (volts)	EG Feed	Total EG added (ml)	TOC Anolyte (mg C/liter)	TOC Catholyte (mg C/liter)
0	22	8.45	off	0	<0.1	-
0.25	32	-	turned on	0	-	-
1.25	55	7.6	on	180	-	-
2.00	65	7.5	on	290	1059	9
4.0	65	-	turned off	626	806	344
>4.0	to 83	-	off	626	-	-

Representing everything in terms of carbon,

Carbon added: 268 gms C in 60 liters (626 ml EG in 60 liters)

Carbon remaining: 29.9 gms C in 60 liters ((0.806*20)+(0.344*40))

Destruction Efficiency: $(268-29.9)/268 = 89\%$

TEST B (Acetone)

Problems were encountered with the pumping of acetone into the cell; Tygon and Viton tubing were unsuitable for this purpose, while Phar-med was acceptable. The feed rate, 82 ml/hr, was 50% of that coulombically possible (theoretical maximum of 161ml in 1.17 hrs).

Run Time (hours)	Temp. (°C)	Voltage (volts)	Acetone Feed	Total Acetone added (ml)	TOC Anolyte (mg C/liter)	TOC Catholyte (mg C/liter)
<0	-	-	off	0	feed problems	feed problem
0	45	-	turned on	0	230	77
1	55	7.6	on	82	-	-
1.17	60	7.5	turned off	82	1116	195

Representing everything in terms of carbon,

Carbon added: 40.1 gms C in 60 liters (82 ml acetone in 60 liters)

Carbon remaining: 22.4 gms C in 60 liters (((1.116-0.230)*20)+((0.195-0.077)*40))

Destruction Efficiency: $(40.1-22.4)/40.1 = 44\%$

Note: This test was insufficient in length to accurately measure destruction efficiencies.

TEST C (Toluene Emulsion)

The toluene emulsion consisted of a 1:1 mixture of toluene and water, by volume, with about 1% of Barlox 12 surfactant added. The overall feed rate *normalized to pure toluene*, 31.8 ml/hr, was 36% of that coulombically possible (theoretical maximum of 243 ml in 2.75 hrs).

Run Time (hours)	Temp. (°C)	Voltage (volts)	Emulsion Feed	Total Toluene added (ml)	TOC Anolyte (mg C/liter)	TOC Catholyte (mg C/liter)
0	40	-	turned on	0	387	185
1.33	40	-	on	35	-	-
2.75	40	-	turned off	88	502	213

Representing everything in terms of carbon,

Carbon added: 69.5 gms C in 60 liters (88 ml toluene in 60 liters)

Carbon remaining: 3.4 gms C in 60 liters ($((0.502-0.387)*20)+((0.213-0.185)*40)$)

Destruction Efficiency: $(69.5-3.4)/69.5 = 95\%$

UNRESOLVED ISSUES

After the final testing of the prototype bipolar cell, several more issues arose which need to be addressed during the final design/commercialization of the MEO system. It was noted that leaks with the prototype were common, and the seals along the electrodes and the internal channels in the cell must be better designed. Issues such as regeneration of the electrolyte, distillations, recovery of cobalt salts, NOx emissions, and the treatment of halogenated organics must be addressed.

D. Partner Contribution

The contributions of EOSystems have been focused primarily in the areas of marketing and commercialization. EOSystems also provided design and engineering work for the flow visualization and prototype cells. Below is a summary of the accomplishments of the Company.

While the MEO process is founded upon the electrochemical generation of a high potential metal ion oxidant for subsequent destructive reaction with organic waste materials, the commercial application of the method requires the integration of many unit operations and design factors into a "process". These include many details such as, design for manufacturability, process control design and implementation, and regulatory compliance issues. There are other critical chemical process issues that must also be addressed such as water removal from a nitric acid catholyte and re-oxidation of the cathodically produced nitrous acid. EOSystems has been working on these and other critical tasks over the term of the CRADA.

The cell patent application came out of the design for manufacturability effort. This effort also included a partnership with Fluoroware, Inc., of Chaska MN, the country's leading producer of fluorocarbon plastics for the semiconductor industry. At the present time, Fluoroware has assembled a team of 12 people to bring the EOSystems' cell design into high volume manufacturing using their injection molding expertise.

EOSystems has also formed a strategic partnership with Controlled Power Company of Troy, MI for the design and manufacture of the dc power supplies required to operate the electrochemical cells. Controlled Power is designing a power supply specifically for the MEO application for optimum operation and minimum cost.

EOSystems is also working with Molycorp, a division of Unocal. Molycorp operates a lanthanide mine at Mountain Pass, CA which is the world's largest mining operation devoted solely to the winning of the rare earth elements. Molycorp will supply the cerium used in the CEO variant of the process. They have also advised the Company on methods for the recovery and recycle of this mediator and will also do reprocessing of spent cerium anolyte solutions at the Mountain Pass location.

In the process chemistry area, EOSystems has been working with Rosenblad Design Group on the design of equipment for the removal of process water from the catholyte. This partnership has designed 3 different sizes of evaporation equipment for the EOSystems' applications. This unit operation operates at reduced pressure and utilizes a flash evaporation followed by a fractional distillation to produce a very low acid product water stream. The Company is also working with R.C. Costello and Associates on the design of the process equipment needed for the removal of volatile chlorine species from the anolyte.

In the regulatory area, EOSystems is working with CAL EPA to obtain certification of the technology under the aegis of AB2060 legislation. The first step involved a three-way partnership with EOSystems, LLNL and the University of California at Davis (UCD) funded by CAL EPA for the laboratory evaluation of the MEO process technology. EOSystems has applied to CAL EPA for support for the next phase which is a third party evaluation of the commercial unit.

EOSystems has applied for a patent on its electrochemical cell, "Electrochemical Cell for Processing Organic Wastes," G. Anthony Steward. This application covers the design of the PVDF cell for operation of the process. EOSystems, from inception, has been determined to make the MEO process cost-competitive with existing technologies. In order that this goal could be realized, the Company had to build its commercial processing units around an inexpensive electrochemical cell. Since commercial units are individually machined they are too expensive to use for a waste application. To overcome this limitation, EOSystems designed its cell using thermoplastic materials, such as PVDF (poly(vinylidene fluoride)), so that high volume manufacturing techniques like injection molding could be used to reduce manufacturing costs.

Finally, EOSystems has moved from the conceptual stage into its own laboratory and early manufacturing facilities in Sunnyvale, CA and is in the process of constructing the first of three pilot plant scale units for testing. It is expected that the first unit will be operational before the end of August 1996.

E. Documents/Reference List

LLNL

Reports:

*"Annual Review Advance Summary," June 1994, Zoher Chiba.

*"Development of Mediated Electrochemical Oxidation (MEO) Based Technologies 1995-Annual CRADA Review," April 1995, Zoher Chiba, report to EOSystems.

*"Final Report-Prototype "T" cell tests," April 1996, Bryan Balazs, report to EOSystems.

*"Summary of Organics Destruction Tests for MEO Project September 1993 - June 1995," June 1995, Bryan Balazs, report to EOSystems.

Invention Disclosures:

4-96 CIL-9954, "Mediated Electrochemical Oxidation (MEO) of Organic Wastes Using a Co(III) Mediator in a Neutral pH Anolyte," G. Bryan Balazs, Patricia R. Lewis, filed by LLNL.

5-96 CIL-9989, "Mediated Electrochemical Oxidation (MEO) of Organic Wastes Using a Co(III) mediator in a Nitric Acid based System," G. Bryan Balazs, Zoher Chiba, Patricia R. Lewis, Norvell Nelson, G. Anthony Steward, filed by LLNL.

EOSystems

EOSystems delivered an invited presentation of the MEO technology to the US Army at their Alternative Technologies for Chemical Demilitarization Conference in Reston, VA, in September of 1995. As a result of this conference, MEO was selected as 1 of the 4 alternative technologies to be given an opportunity for further evaluation by the NRC (National Research Council). The AEA Technology-Subsea consortium was chosen to perform the MEO evaluation tests. EOSystems was deemed to be too early in its development to be able to undertake the testing within the Army's time frame.

The MEO CEO technology has received a lot of exposure in the trade press, most notably in a recent issue of Chemical Engineering (McGraw-Hill, May 1996) and on the cover of the May 1996 issue of R&D Magazine which showed a picture of the EOSystems' cell as installed at Pacific Northwest National Laboratory. In addition, the process design of EOSystems and PNNL was awarded a R&D-100 award for 1996 as 1 of the top 100 new technologies of the year.

Patents:

Patent application, "Electrochemical Cell for Processing Organic Wastes," G. Anthony Steward, filed by EOSystems.

F. Acknowledgment

Participant's signature of the final report indicates the following:

- 1) The Participant has reviewed the final report and concurs with the statements made therein.
- 2) The Participant agrees that any modifications or changes from the initial proposal were discussed and agreed to during the term of the project.
- 3) The Participant certifies that all reports either completed or in process are listed and all subject inventions and the associated intellectual property protection measures attributable to the project have been disclosed or are included on a list attached to this report.
- 4) The Participant certifies that if real property was exchanged during the agreement, all has either been returned to the initial custodian or transferred permanently.
- 5) The Participant certifies that proprietary information has been returned or destroyed by LLNL.

Technical report from EOSystems
is on file in the Industrial Partnerships
& Commercialization Office at LLNL.

[Industrial Participant]

Date

7-18-96

[LLNL Principal Investigator] Date

Attachment I -- Final Abstract
Attachment II -- Project Accomplishments Summary
Attachment III -- Final Quarterly Report

**Attachment I
Final Abstract
TC-349-92**

The project is a relationship between the Lawrence Livermore National Laboratory (LLNL) and EOSystems, Inc.

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EOSystems, Inc., located in Palo Alto, Calif., is a start-up company founded in 1992 to bring Mediated Electrochemical Oxidation (MEO) technology to market for the disposal of hazardous organic waste.

Title of Project	Development of Mediated Electrochemical Oxidation (MEO) Technology
Purpose	Destroy toxic and hazardous organic wastes. Treat mixed wastes.
Benefit to Industry	Research and develop a product with the help of LLNL resources.
Benefit to DOE/LLNL	Cheaper waste treatment at DOE and other government facilities.
Time Period	Three years, starting August 30, 1993
LLNL Contact	Bryan Balazs, (510) 423-5403

Overview

Lawrence Livermore National Laboratory and EOSystems, Inc., formed a partnership to commercialize a hazardous waste treatment technology initially investigated by LLNL as a means of treating radioactive wastes from weapons manufacturing plants. The process is called Mediated Electrochemical Oxidation, or MEO, and it employs chemical reactions, similar to those in a battery, to destroy organic wastes. The waste is introduced into a self-contained unit containing a solution that breaks down the waste when an electric current is applied. The units are inherently safe: they operate at ambient temperature and pressure in a closed-loop system that presents no risk of accidental release into the air. The planned units will be scaled in size to specific waste streams (the smallest unit will be roughly the size of a large desk). The units will efficiently destroy from 20 to 2,000 pounds of waste per day on site, eliminating the need for costly transportation to a disposal site. Hospitals, research organizations, and many industrial manufacturers generate this type of waste, which is now stored on site, then shipped to a waste treatment facility for incineration—a disposal method that is no longer acceptable.

At the completion of the project, a prototype MEO unit was designed, built and tested. The results of the testing, done on the model organics ethylene glycol, acetone and toluene, indicate that the system is functioning as designed and meets performance expectations. The cell is designed for ease of manufacture, and is constructed of PVDF (Kynar) so that rapid techniques such as injection molding may be used in the construction. Several patents application have been filed on various aspects of this CRADA, and EOSystems was the recipient of an R&D100 award for the MEO process design. EOSystems intends to market this device later this calendar year.

Mediated Electrochemical Oxidation (MEO) Based Technology

Project Accomplishments Summary (Attachment II) CRADA No. TC-0349-92-1

Date: July 15, 1996

Revision: 1

A. Parties

The project is a relationship between the Lawrence Livermore National Laboratory (LLNL) and EOSystems, Inc.

University of California
Lawrence Livermore National Laboratory
7000 East Avenue, L-370
Livermore, CA 94550

EOSystems, Inc.
760 San Aleso Avenue
Sunnyvale, CA 94086

B. Background

The remediation of organic waste streams is an ongoing concern at many industrial, medical, and government facilities; treatment technologies currently employed include incineration, and physical separation methods such as chromatography or distillation. Mediated Electrochemical Oxidation (MEO) is an organic waste remediation technology which offers many advantages in terms of cost, safety, and applicability to a variety of waste streams, including those containing radionuclides. LLNL and EOSystems entered into this CRADA in order to optimize the MEO technology previously developed at LLNL, to the point of commercialization by EOSystems. EOSystems would gain invaluable research expertise and facilities, while LLNL/DOE would have access to a less costly method of hazardous waste treatment.

C. Description

The objectives of this CRADA were the continued research, and subsequent commercialization, of the MEO technology previously developed at LLNL. LLNL was responsible for the research and testing of the MEO systems, while EOSystems was responsible for marketing studies, securing investment capital, and design and development of a prototype commercial unit. Nearly all of the original goals of the CRADA were met, and a prototype of the system was designed, built and tested. At the end of the CRADA, EOSystems anticipated sales of the first commercial units within six months.

D. Expected Economic Impact

The remediation of hazardous waste streams is a crucial goal of many government, manufacturing, research, and medical facilities. The anticipated timescale of sale and implementation of the commercial units by EOSystems is at the end of calendar 1996. It is anticipated that the results of this CRADA will enable the cost-effective remediation of hazardous waste streams, avoiding the disadvantages of current treatment systems such as incineration. The near-ambient temperature operation of the MEO units offer substantial advantages in terms of safety, and this technology avoids the issue of formation/volatility of components such as dioxins. Because of this, positive public and political perception of this 'environmentally-friendly' technology is expected. As the demand for such environmental remediation projects is enormous,

it is anticipated that this technology development will lead to the creation of new jobs and companies.

E. Benefits to DOE

The remediation of hazardous waste streams is of both immediate and long-term concern to LLNL, and indeed, to the entire DOE complex. As mentioned, this technology is being transferred to the private and public sectors through this CRADA, further enhancing the public and political acceptance of the research.

F. Industry Area

Numerous industrial areas can benefit from this waste treatment technology; these include research, biomedical, manufacturing, waste treatment, petroleum/petrochemical, etc.

G. LLNL Point of Contact for Project Information

Bryan Balazs
Mail Code L-370
Lawrence Livermore National Laboratory
Livermore, CA 94550
(510) 423-5403 fax: (510) 423-0909 email: balazs1@llnl.gov

H. Company Size and Point(s) of Contact

EOSystems, Inc. is a start-up venture capital company with four employees. As of the end of the CRADA, there were no sales as the development was just completed. Contact persons at this company are as follows:

G. Anthony Steward (President)
Norvell Nelson (Vice President)

EOSystems, Inc.
760 San Aleso Avenue
Sunnyvale, CA 94086
Phone (408) 744-9180
Fax (408) 744-9184

I. Project Examples

LLNL is in possession of a bench-scale, operational prototype of the MEO system, containing all ancillary systems and hardware.

J. Release of Information

Bryan Balazs, Principal Investigator
Staff Chemist, Chemical Engineering Section, Chemistry and Materials Science Dept.
Phone: (510) 423-5403 Fax: (510) 423-0909 Email: bb@llnl.gov

RELEASE OF INFORMATION

I have reviewed the attached Project Accomplishment Summary prepared by Lawrence Livermore National Laboratory and agree that the information about our CRADA may be released for external distribution.

Unable to obtain Industrial Partner's signature.

G. Anthony Steward
EOSystems, Inc.
President

Date

**Lawrence Livermore National Laboratory
Final Quarterly Report (Attachment III)**

Title: Development of Mediated Electrochemical...
Participant: EOSystems
DOE TTI No.: 93-LLNL-084-C1 & 93-LLNL-014-D1
CRADA No.: TC-0349-92
TACT: E&E
Account Numbers 4705-01 to 03
Date Accounts Closed: 6/14/96
Approved Funding Profile (\$K)

Reporting Period: 07/01/95 - 06/30/96
Date CRADA Executed: 8/30/93
DOE Approval Date: 8/27/93
Project Completion Date: 6/30/96
Scheduled Ending Date: 8/30/96
B & R Code (S): DP030101
35DP0301

	FY93	FY94	FY95	FY96	FYOUT	Total
LLNL Planned	507	413	700	380	0	2000
Participant In-Kind	400	700	900	0	0	2000
Participant Funds-In	0	0	0	0	0	0
WAS DP030101	507	413	700	248	0	1868
WAS 35DP0301	0	0	0	0	0	0
Total Costs	12	703	903	248	0	1866

DP030101	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	July	Aug	Sep	FYTD
FY93	0	0	0	0	0	0	0	0	0	0	0	12	12
FY94	24	20	16	31	54	88	70	59	87	71	66	119	703
FY95	171	102	71	77	85	88	75	93	49	28	33	31	903
FY96	34	34	35	23	25	68	37	-7	0	0	0	0	248
FYOUT	0	0	0	0	0	0	0	0	0	0	0	0	0

1866

35DP0301	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	July	Aug	Sep	FYTD
FY93	0	0	0	0	0	0	0	0	0	0	0	0	0
FY94	0	0	0	0	0	0	0	0	0	0	0	0	0
FY95	0	0	0	0	0	0	0	0	0	0	0	0	0
FY96	0	0	0	0	0	0	0	0	0	0	0	0	0
FYOUT	0	0	0	0	0	0	0	0	0	0	0	0	0

0

STAFF w/phone:

Lab PI: Bryan Balazs (510) 423-5403
Resource Manager: Ilene Detherow (510) 422-1853
DOE OAK: Jerry Scheinberg (510) 637-1653

Participant: Norvell Nelson (415) 494-6982
DOE HQ: M. Michaelis (202) 586-4105

TREAT AS BUSINESS SENSITIVE

Lawrence Livermore National Laboratory
Final Quarterly Report (Attachment III)

Reporting Period: 07/01/95 - 06/30/96
DOE TTI No.: 93-LLNL-084-C1 & 93-LLNL-014-D1
CRADA No.: TC-0349-92

Page 2

Milestones and Deliverables:

List the complete set of milestones for all phases of the CRADA. Continue on separate pages if necessary.
Report any changes from the original CRADA or previous quarterly report on the CRADA Change Form.

		Completion Date:	
		Scheduled	Actual
A1	Develop design crit & select prototype organic separator	6-95	*
A2	Validate design & bring prototype org sep into operation	9-95	
A3	Conduct operational tests	12-95	
A4	Couple organic separator and MEO operation	3-96	
A5	Eval factors for commercial application of separator	9-94	9-94
A6	Conduct market assess for sales of org sep capability	3-95	*
A7	Des scale-up parameters for org sep & build pilot plat unit	3-96	
A8	Productize unit & begin commercial sales	9-96	
B1	Extend corrosion resist coat process to full-size electrodes	9-95	
B2	Test & eval electrodes for perf in MEO proc for corrosion resist	12-94	6-95 (est.)
B3	Perf market survey & econ feas study for sales of electrodes	9-95	
B4	Determine manuf of new electrodes using LLNL procedures	3-96	
C1	Selection of process chemistry	9-94	9-94
C2	Development of test protocols for certification procedures	3-95	*
C3	Design & build equipment & conduct shakedown tests	3-95	*
C4	Demonstration of process compliance	6-95	*
C5	Certification review by EPA & CalEPA	3-96	
C6	Conduct detailed market survey of SQGs	3-95	*
C7	Productize unit for SQGs & begin commercial sales	3-96	

* The SOW and schedule are still being reworked with the partner.

Verification of participants' in-kind contribution was made in accordance with LLNL policy. Explain basis of verification:
See attached

Please initial: YES _____ NO _____

List any subject inventions by either party (include IL# for LLNL inventions), additional background intellectual property, patents applied for, software copyrights, publications, awards, licenses granted or reportable economic impacts
See attached list of publications.

Verification that all equipment and proprietary information has been returned to the initial owner or permanently transferred

Please initial: YES X NO _____

Accomplishments

Describe Technical/Non-Technical lessons learned and other observations.
Summarize causes/justification of deviations from original scope of work.

See Final Report

Reviewed by CRADA project Program Manager:

Date: _____

Reviewed by Karena McKinley, APL Partnering, IP&C:

Date: _____

Direct questions regarding this Report to IP&C Resource Manager, Susan Springer, at (510) 422-5507

TREAT AS BUSINESS SENSITIVE