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Engelhard Corporation

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Platinum Group Metal Technology Development

DE-FC36-03GO13104

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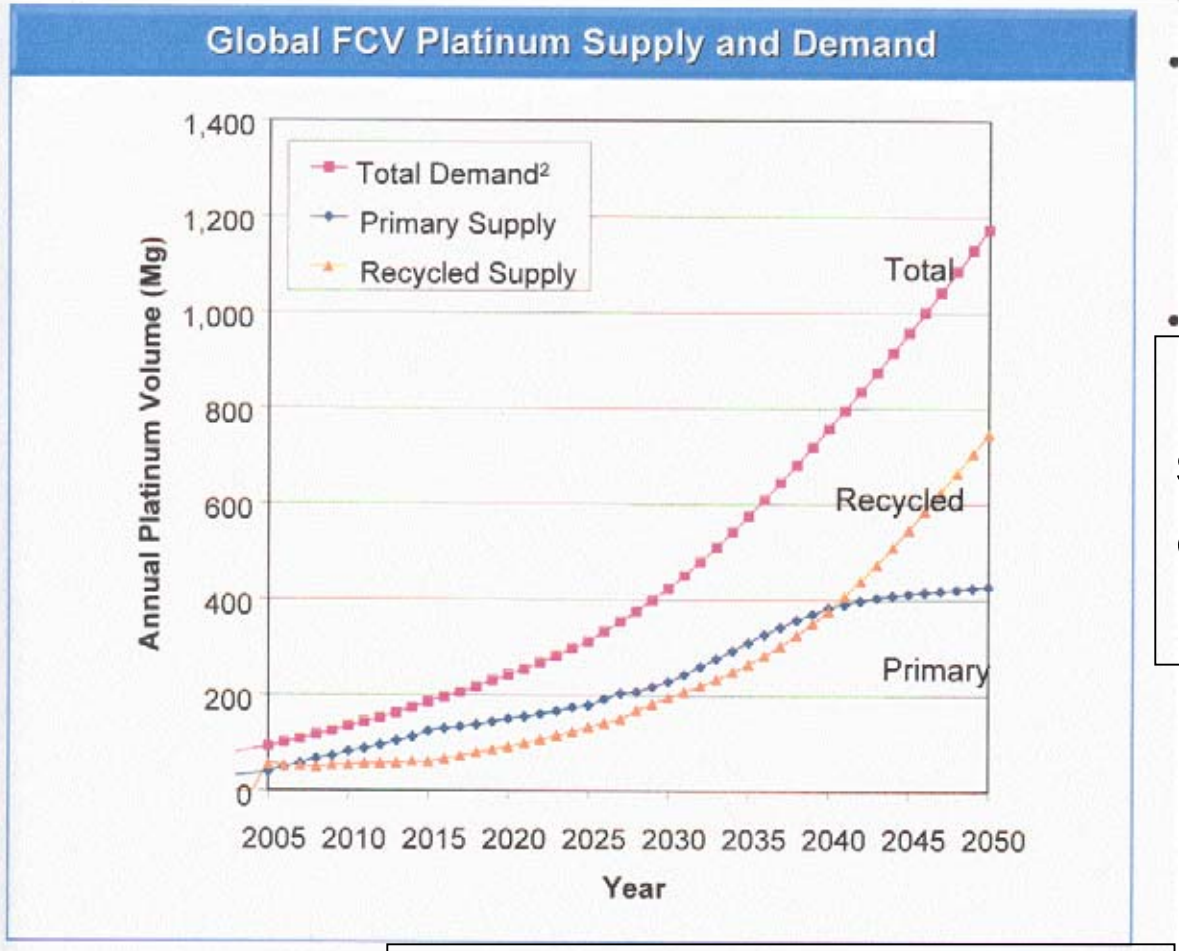
Pt Recycling is Crucial to Successful Implementation of Fuel Cells



Recycling will supply the bulk of Pt required in 2050

Refiners, like Engelhard, become 'miners'

TIAX

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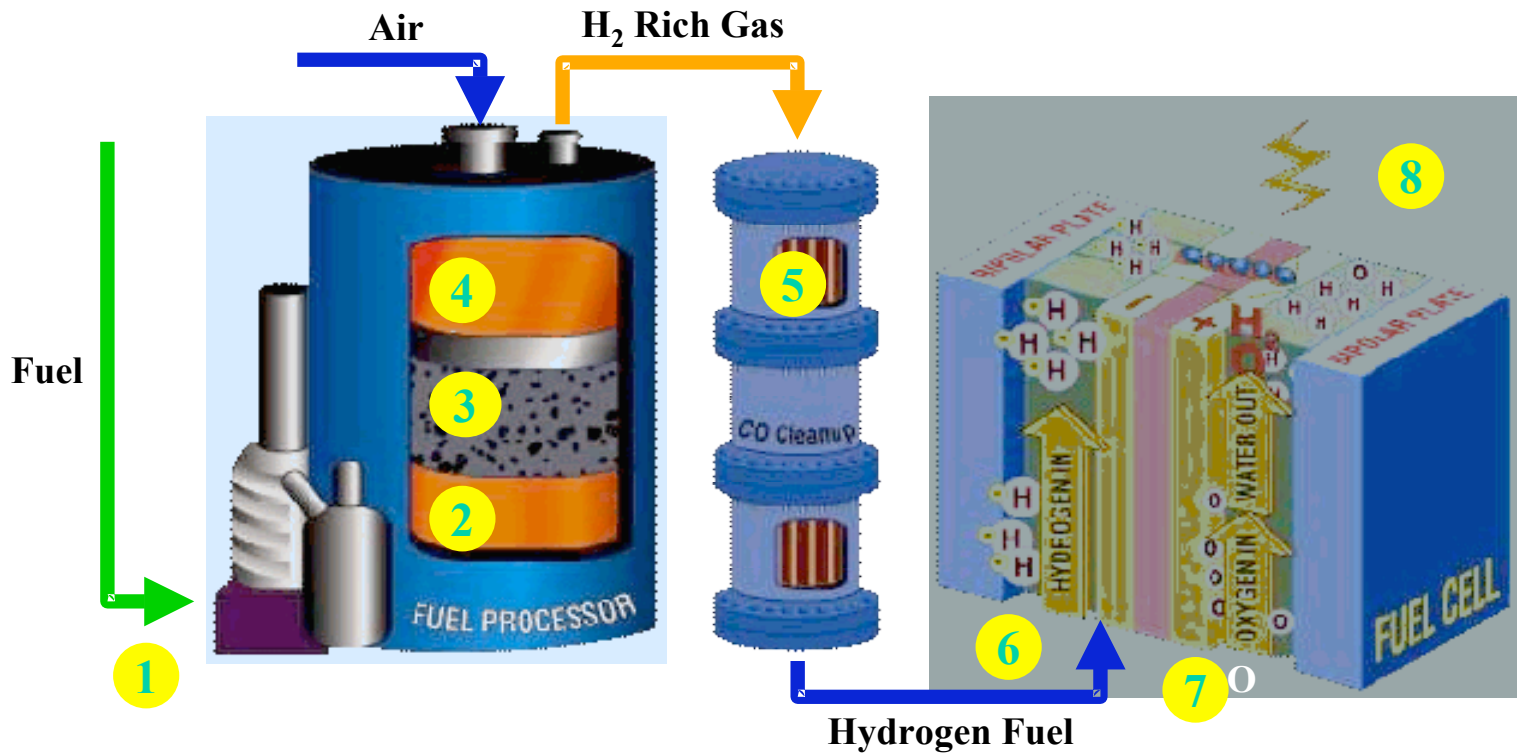
Schematic of a Fuel Cell System With Reformer



1. Sulfur Removal
2. Catalytic Partial Oxidatio
3. Steam Reforming
4. Water Gas Shift
5. PROX
6. Anode
7. Cathode
8. Emission Control

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Objectives of DOE Pt Recycling Project (Started 11/2003)

Recycle all PM-containing catalysts in a fuel cell 'system'

- **Develop a commercially-acceptable, environmentally-friendly process for recovering and recycling Pt** and Ru **from membrane electrode assemblies (MEAs)**
  - **Develop a process that does not emit pollutants, especially HF**
  - Evaluate Ru recovery from MEA's
- **Develop a process for PM recovery from metal monoliths**
- Maximize precious metal (PM) yield from ceramic catalysts

*Principal objectives in **bold**.*

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What Makes a Refining Process Commercially Acceptable?

- **Process must permit quantification of the precious metal content of lot (weigh, sample, analyze)**
- **Precious Metal Recovery Meets or Exceeds Industry Standard**
- **Processing Cost is comparable to reference method**
- **Safety Concerns are Minimal**

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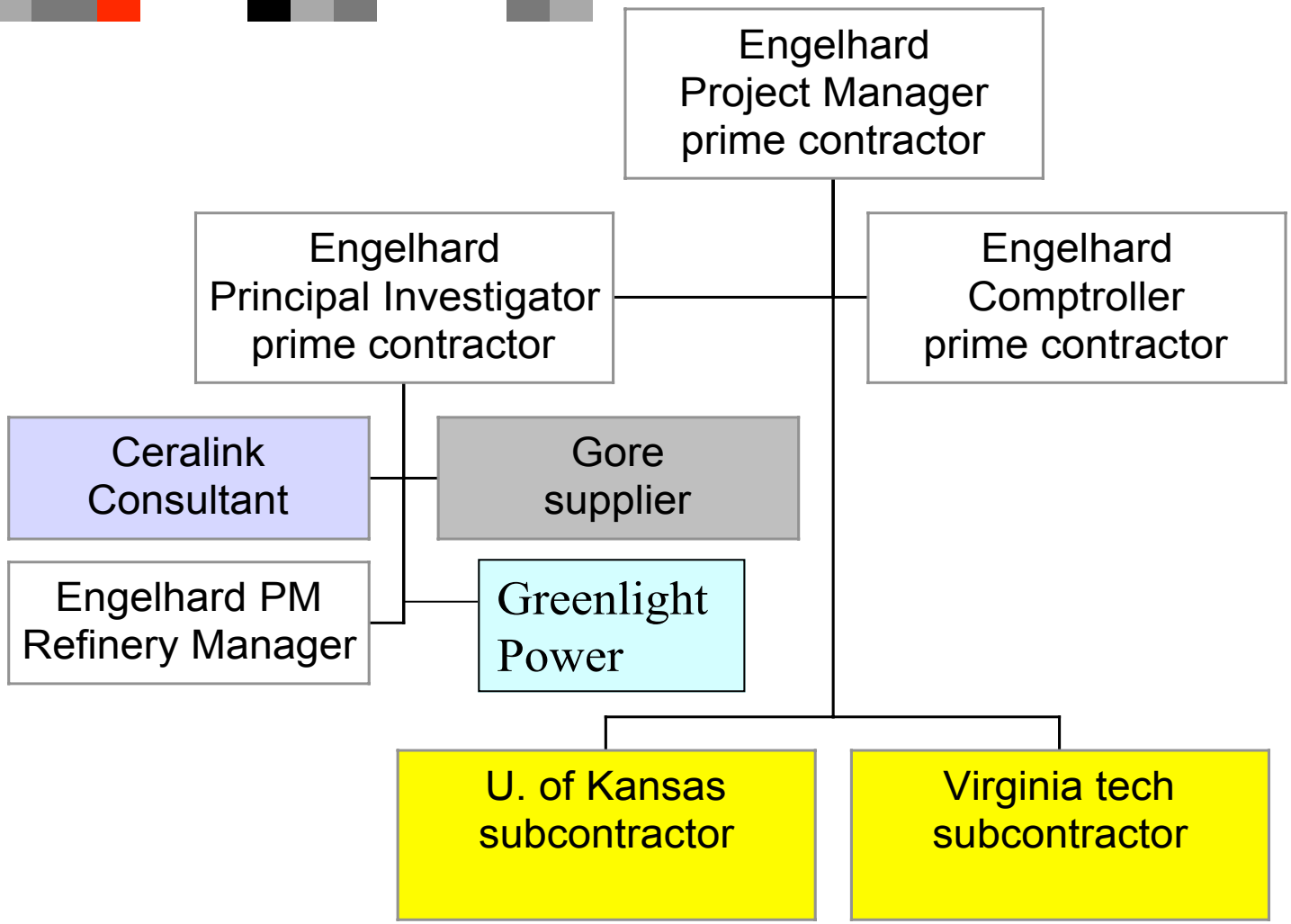
Organization Chart for Precious Metal Recovery/Recycling Project



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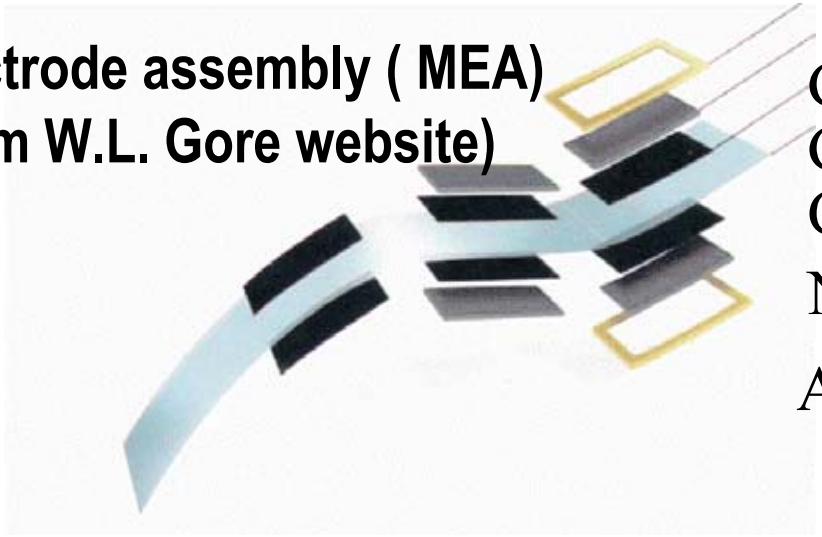
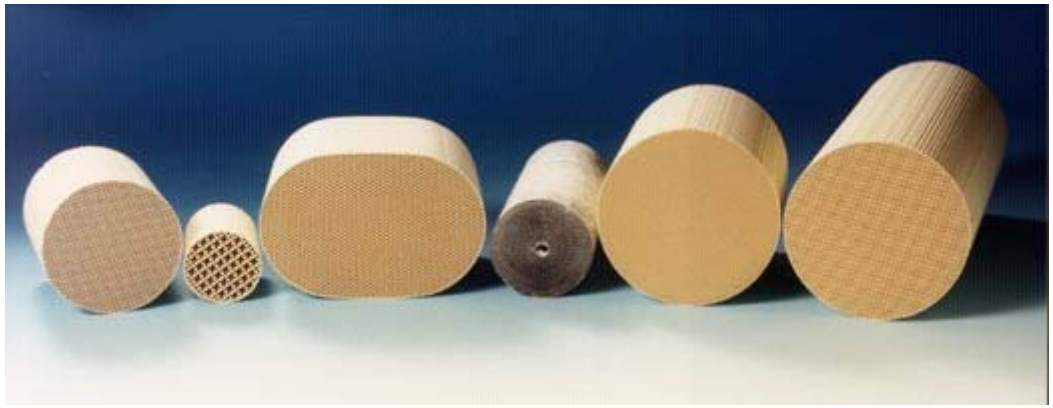
Examples of Fuel Cell Catalysts

Membrane electrode assembly (MEA)
(Adapted from W.L. Gore website)



Gasket
Gas diffusion layer
Cathode catalyst
Nafion membrane
Anode catalyst



Honeycomb monoliths (ceramic) - Used in a fuel cell reformer

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Budget for DOE Project No. DE-FC36-03GO13104

FY2003	$375,000	$300,000	$75,000.00
FY2004	$681,250	$545,000	$136,250
FY2005	$875,000	$700,000	$175,000
FY2006	$1,625,000	$1,300,000	$325,000
FY2007	$1,513,995	$1,211,700	$302,295
FY2008	$900,000	$720,000	$180,000

Engelhard has a 20% cost share obligation

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Technical Barriers and Targets

- Address barriers N (Cost) and O (Stack Material and Manufacturing Cost) of the HFCIT Program Multi-Year R&D Plan.

- Meet DOE technical targets for 2010 for fuel cell power systems
  - ➢ $45/kW cost target for Transportation
    - – Direct hydrogen
    - – Reformer-based operating on low-sulfur gasoline
  - ➢ $400 – 700/kw for Stationary
    - – PEM fuel cell system operating on natural gas or propane

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Overview of Technical Approach

- Develop environmentally-friendly processes for recovering and recycling the precious metals present in PEM fuel cell stacks and fuel reformers.
  - Investigate
    - leaching
    - industrial microwave methods
    - super critical carbon dioxide

  and select the preferred processes for recovering precious metals from the various types of catalysts present in PEM fuel cell systems
- **Build and operate pilot equipment to demonstrate process viability (Primary deliverable)**
- Estimate the economics of viable processes

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Safety Issues Related to the Pt Recycling Project

- Microwave Techniques
  - Microwave leakage
  - Electrical Shock for capacitor
  - HF formation
  - Overheating
- Supercritical Fluid Techniques
  - System under pressure
- Leach Techniques
  - Corrosive fumes

- Use a microwave leak detector and safety interlocks
- use proper insulation
- Properly vent and/or scrub exhaust fumes
- Monitor temperature
- Use Plexi-glass shields for protectiion
- Properly vent and/or scrub fumes

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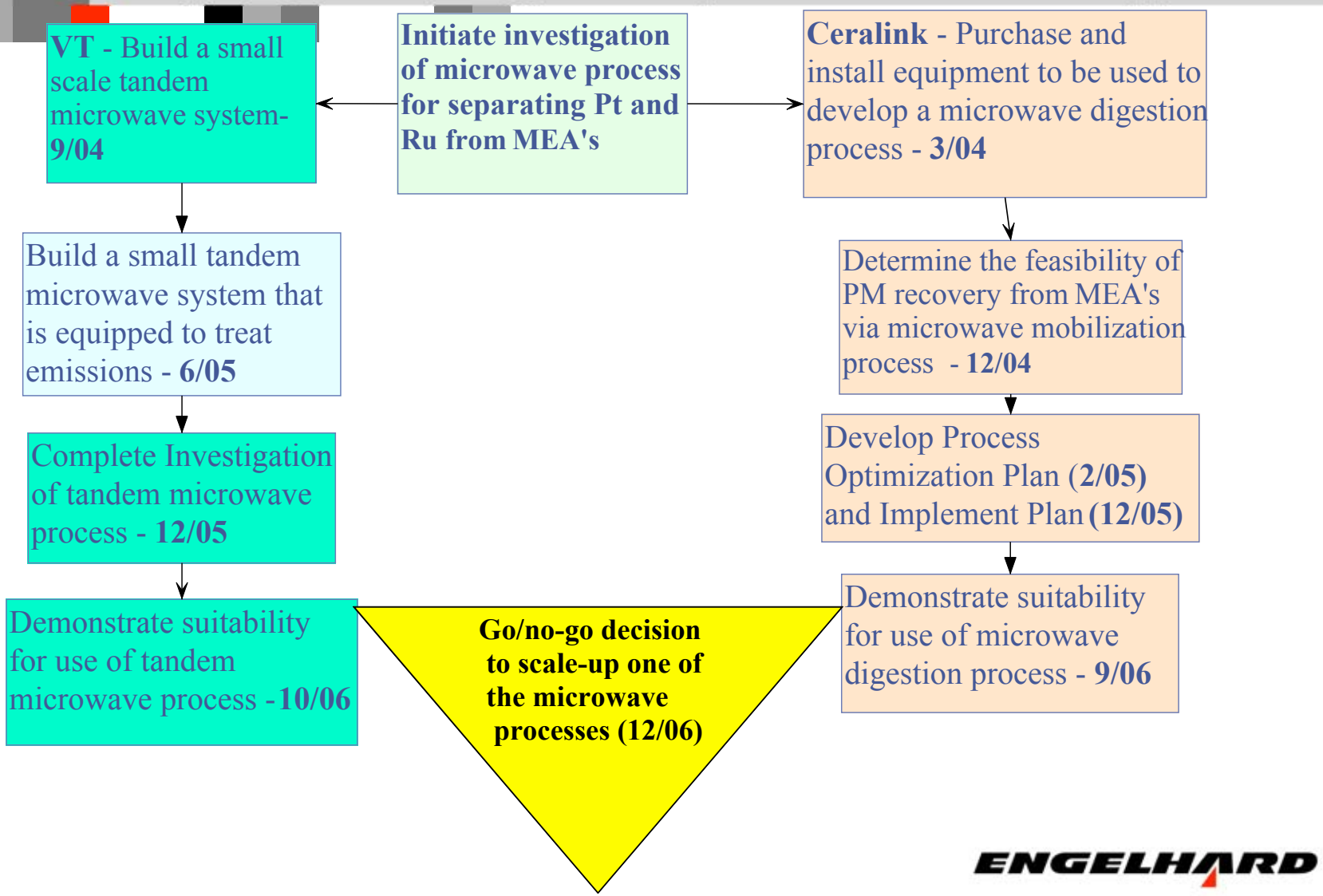
Milestone Schedule for Platinum Group Metal Recycling Technology Development – Microwave Approach



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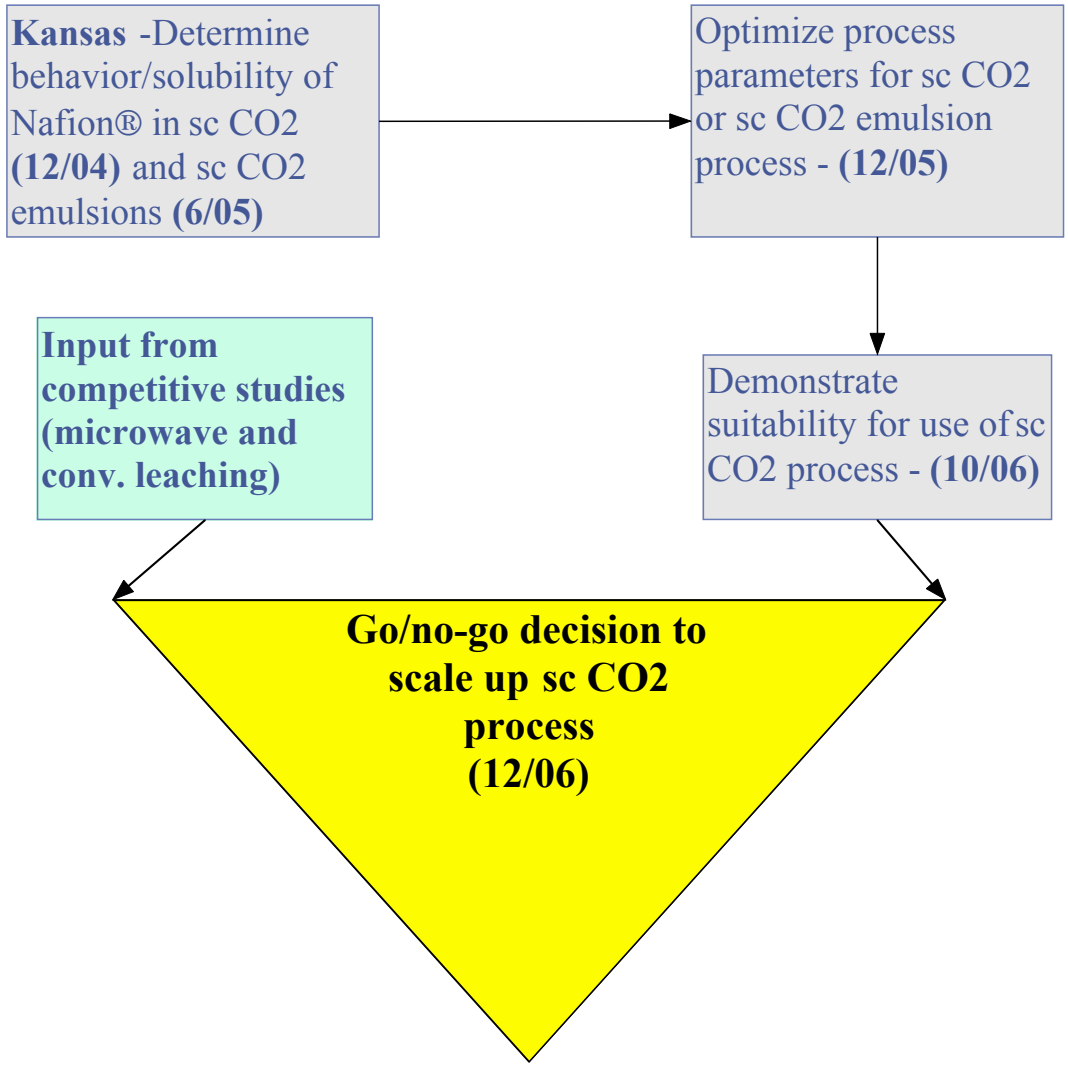
Milestone Schedule for Platinum Group Metal Recycling Technology Development- SC CO2 Approach



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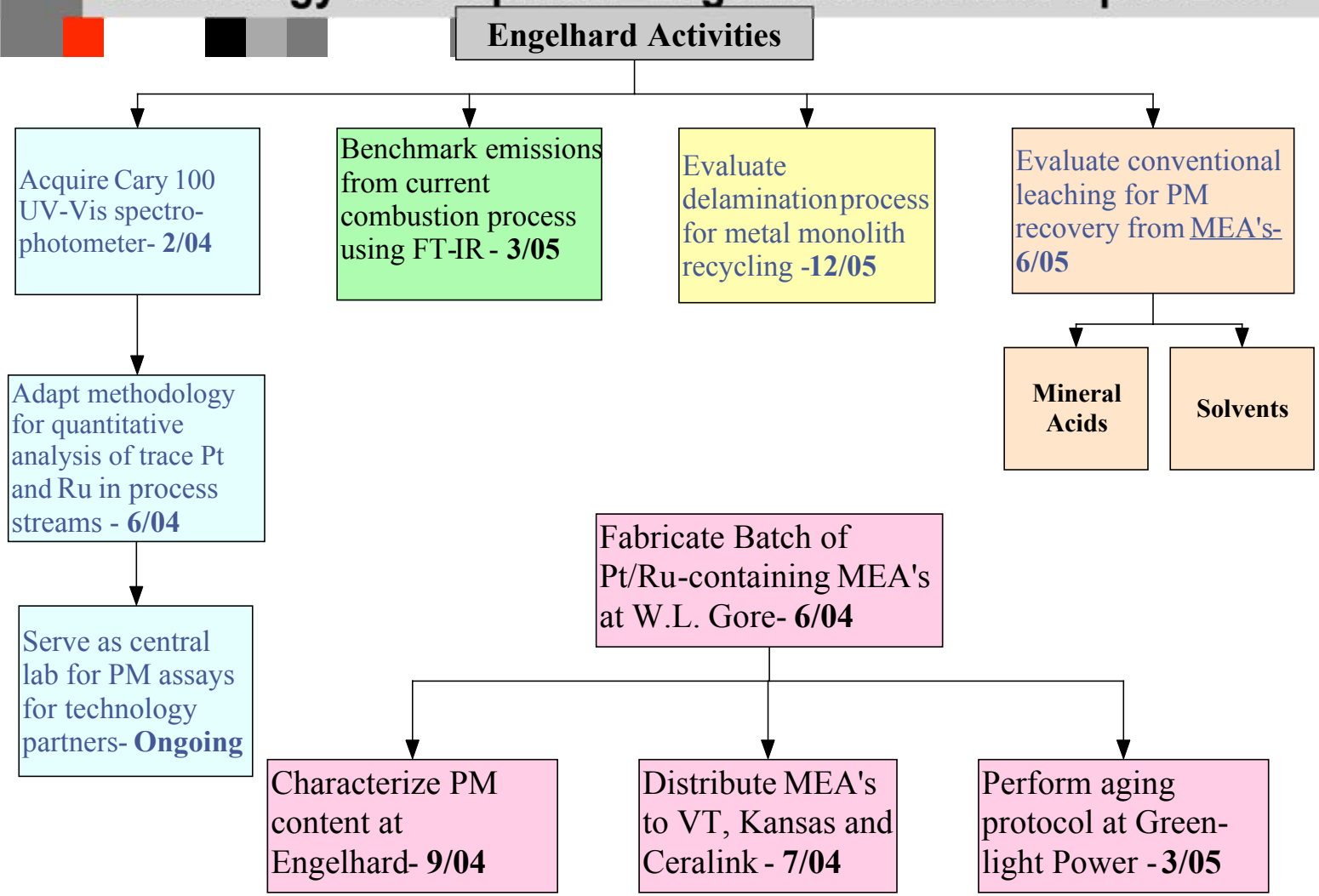
Milestone Schedule for Platinum Group Metal Recycling Technology Development- Engelhard Activities Up to 2006



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Milestone Schedule for Platinum Group Metal Recycling Technology Development- Engelhard Activities For 2006-2008

Task	Due Date
Place Order for large batch of MEA's	1/1/2006
Accept delivery of MEA's	6/1/2006
Estimate preliminary process cost	10/1/2006
Engineering plans for pilot plant due	12/15/2006
Final estimate of process cost	3/1/2007
Order Equipment for demonstration plant	4/1/2007
Build demonstration plant	12/1/2007
Demonstration plant operational	6/1/2008

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MEA Aging of Gore-fabricated MEA's

- Greenlight Power will assemble a stack and
  - Age with reformate using a stationary source cycle
  - Acquire electrochemical performance
  - Withdraw cells at 500, 1000 and 1500 hours for testing
  - Harvest the remainder at 2000 hours
- The suitability of proposed recovery methodology to aged MEA's will be determined
- Ruthenium migration across membrane will be evaluated through time lapse sampling of the stack

Major accomplishments In 1st year

- VT – Thermal Destruction of MEA with microwave energy
- Ceralink – Delamination of 3- and 5-layer MEA's
- University of Kansas – Construction of apparatus for phase studies
- Gore – Fabrication of MEA batch (end of 2nd quarter)
- Engelhard – Developing practical method for Pt and Ru determination with quantitative recovery

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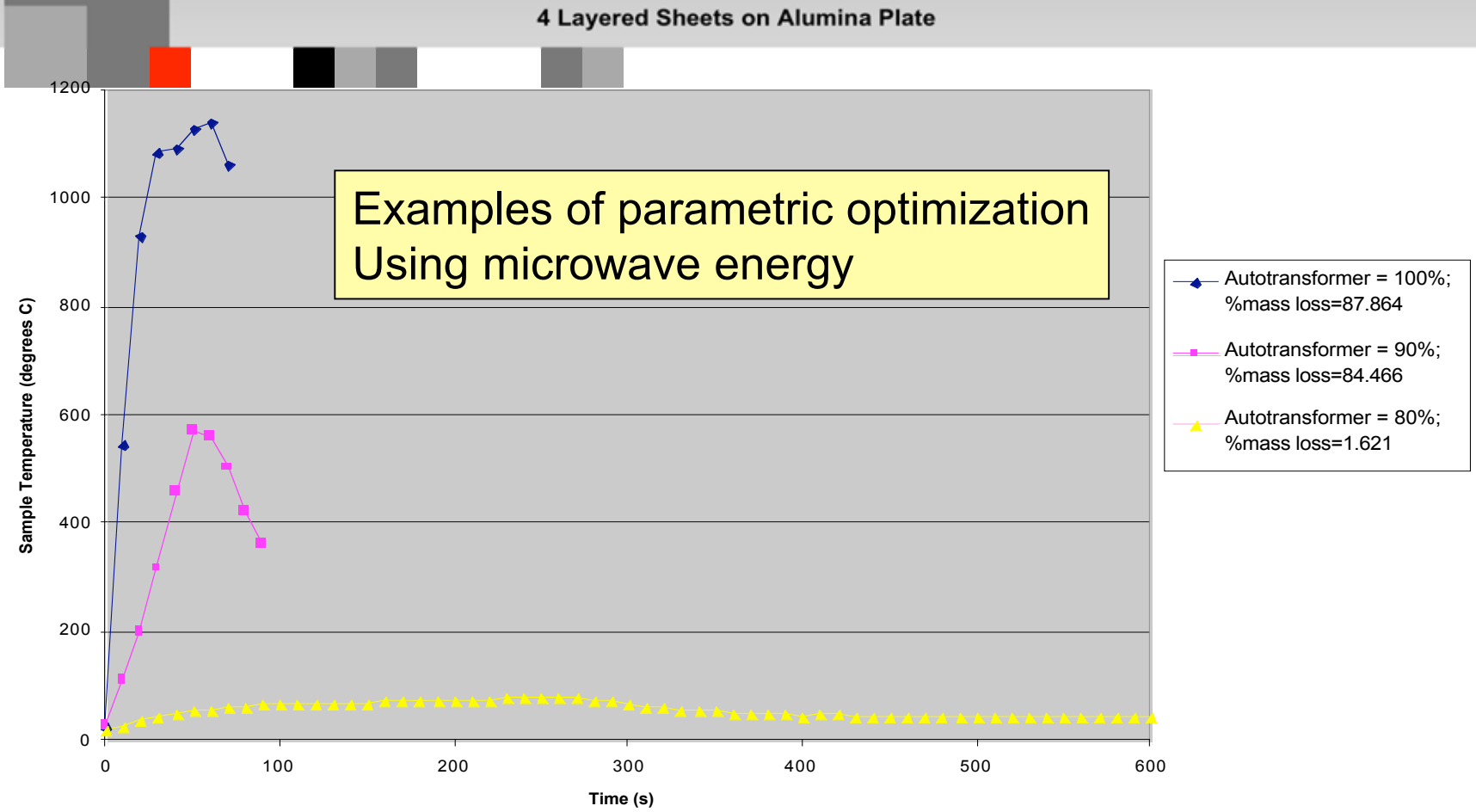

Figure 1. Results of microwave firing of three samples of 3-ply catalyst-coated Nafion stacked 4 sheets high on a microwave-transparent substrate.

Future Work (2004-2005)

- VT
  - Continue temperature versus time runs with MEA samples.
  - Transfer residues to Engelhard to assay Pt content and ash composition
  - Build a microwave unit with effluent treatment capability
- University of Kansas –
  - Continue phase analysis studies to determine favorable solvent system for Nafion.
- Ceralink
  - Evaluate acidic media and microwave conditions for optimizing precious metal recovery from MEA's

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Acknowledgement to Our Technology Partners

- Corporate - Project Administration and technical services
- Seneca - Refining expertise and facilities
- **Virginia Tech, Blacksburg, VA (Diane Folz, David Clark and Carlos Suchicital) Development of emission-free process involving microwave decomposition and sequestration of hazardous volatiles**
- **Ceralink, Alfred, NY (Holly Shulman) - Microwave assisted leaching of PM from Nafion**
- **University of Kansas, Lawrence KA (Bala Subramaniam) - Supercritical fluid treatment of MEA's to dissolve Nafion and permit separation of PM phase from Nafion**
- W.L. Gore, Elkton, MD - Supplier of MEA's
- Green Light Power, Vancouver, BC- Testing of stacks to generate aged MEA's

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