Example 3.5: Product and Process Development in Gas-Solids Fluidized Bed Linear Low Density Polyethylene Manufacturing Process

Start Date:	April 2, 2005
End Date:	October 11, 2005
FYE:	December 31, 2005

Issues addressed Related to SR&ED:

1. Claiming Plant trials with continuous large-scale multi-stage commercial production process
2. EP and CP/ED Continuous Plant Trials
3. Number of plant trials and length of plant trials in support of SR&ED project
4. Excess materials consumption used in Plant Trials (gas flaring, additives consumption)
5. Proxy vs. Traditional Methods

Preamble:

This example has been developed for illustration of the concepts that distinguish experimental production (EP) from commercial production with experimental development (CP/ED) in chemical sector shop floor SR&ED projects. The example is intended to provide guidance on how to apply the principles contained in Chemicals Guidance Document 3 Part 1 [1] and SR&ED AP 2002 02R2 [2] for the review of chemical sector claims.

Background:

A generic commercial-scale fluidized bed gas- phase polyethylene reactor is comprised of a reaction vessel, a bed of polymer, which is in various stages of polymerization, a gas distribution plate, inlet and outlet piping, a compressor, a cycle gas cooler, a product discharge system, and a gas-flaring device (see Figure 1). A description of this type of process is given in References [3] and [4]. Additionally the polymerization system includes resin storage bins, and an extruder to mix additives into and pelletize the resin. The company was attempting to develop new polymer products and the capability to produce these products in a gas phase reactor using a new metallocene catalyst. The pilot-scale system in which the testing was done does not accurately simulate the commercial gas phase reactor because there are fundamental design differences between the pilot-scale reactor and the full-scale gas fluidized bed.

For the purpose of this example the system shown in Figure 1 is operated at pressures from 1.5 MPa – 2.5 MPa, at temperatures ranging from 70 to 95°C and at an average throughput rate of 20,000 kg per hour. The reactor is filled with a bed of about 45,000 – 50,000 kg of dry polymer particles that is vigorously agitated by a high velocity gas stream. The gas stream is comprised of a mixture of ethylene, optionally a comonomer, nitrogen, and hydrogen and enters the reactor from the bottom, and passes through a perforated distribution plate. Rapid circulation of the gas stream serves two purposes: fluidization of the particle bed and removal of the heat of polymerization. The unreacted gas stream enters an expanded disengagement zone at the top of the reactor, is separated from the entrained polymer particles, and is then compressed, cooled, and recycled back into the reactor. Product properties are controlled by adjusting reaction conditions (temperature, pressure, flow rates, etc.). Computer models are used to determine the required reaction conditions based on catalyst type and the specific product being produced.

1A. Scientific or Technological Objectives

The technological objective was to develop a new polymer product using a newly formulated metallocene catalyst that has recently had approval for a world patent, and to better understand the range of process parameters needed to operate the gas phase fluidized bed reactor process. The project involves experimental development in the fields of chemical reaction engineering and polyethylene manufacturing technology.

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1B. Technology or Knowledge Base or Level

Prior company experience was with another family of catalyst, which exhibited significantly different characteristics for the polyethylene grades that could be manufactured. In this project, Company XYZ will endeavour to produce polyethylene film grade products in a gas phase fluidized bed reactor with a newly formulated metallocene catalyst. Bench and pilot-scale development of the new catalyst was claimed as part of the company's SR&ED submission in the prior years. Results from the bench and pilot testing showed that there was a problem with entrained catalyst from the reactor, as well as, another separate problem associated with neutralizing excess catalyst activity in the extrusion area with excess additives. It was not known if these problems would also be apparent in the full-scale system since the pilot reactor was of a different design type.

Prior to the start of this project, the company did not have any relevant plant experience for polyethylene production using the newly developed metallocene catalyst in the commercial-scale fluidized bed. This resulted in a significant technological uncertainty associated with the reactor itself since the pilot reactor was of a fundamentally different design. Some of the technological issues that needed to be resolved with the entire multi-stage process shown in Fig. 1 included:

1) Hydrodynamics, heat and mass transfer characteristics of the gas phase reactor would be different than that known for the pilot scale reactor;
2) Highly active metallocene catalyst remains after the product is deactivated, and is present in significant quantities, even after the product leaves the product discharge system.
3) Excess purging of recycle gases (flaring) increases the cost of the product and impacts on pollution to the environment.
4) Excess active catalyst could cause reactions in the holding bins, which would be a safety hazard (fire) since the bins are not purged.
5) Temperature runaway due to the exothermic nature of the reaction could result in a serious risk of meltdown inside the reactor, which would result in an unplanned reactor shutdown, and an expensive maintenance and upgrade cost.
6) The gas phase reaction in the fluidized bed reactor was very sensitive to minor variations in catalyst loading and impurities in the feed stream.

1C. Scientific or Technological Advancement

The technological advancement being sought is the development of a stable gas-phase fluidized bed reactor process for the manufacture of linear low-density polyethylene (LLDPE) using a newly developed metallocene catalyst. The technological advancement being sought is in the field of chemical reaction engineering and polymer manufacturing technology.

No gas phase process employs a similar family of metallocene catalyst to efficiently produce linear low-density polymer products of acceptable grade. We do not know if our existing catalyst, which has only been demonstrated for a pilot-scale operation (with completely different design), would be suitable for a large-scale polyethylene processing application due to the exothermic profiles in the fluidized bed reactor, leading to a strong potential for a temperature runaway. Reactions in the gas phase system using the new catalyst are known to result in significantly higher temperature profiles based upon pilot scale results to date. The resulting reactor instability from temperature runaway could lead to an unstable process operation and large amounts of off-grade products.

Finally, we do not know if a method can be found to quench the residual metallocene catalyst activity, which is entrained with the product as it leaves the fluidized bed reactor discharge area.

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This is needed to reduce or preferably eliminate the excess additive consumption in the extrusion area.

1D. Description of Work in the Tax Year

Trial 1: Product Development with New Catalyst in Large-Scale Gas Phase Process.

Date: May 20-24, 2005 Trial Time: 3.5 days

The company investigated the use of a new metallocene catalyst in the gas phase bed. The product was not previously produced at this site. While they were running the commercial product trial the plant operations staff experienced problems controlling the process due to frequent temperature excursions in the reactor. By carrying out excess flaring they discovered that they could attain adequate temperature control. In Trial 1 plant personnel determined how much flaring was required to control temperature. The initial experimental grade product exhibited residual catalyst activity during the pelletization stage. This was identified through high additive consumption.

The company claimed the 3.5-day trial as EP. This included the time to transition from the prior production runs, as well as, targeting and achieving steady state processing conditions. Finally, there was the time needed to actually run the experimental period of the trial. It was necessary to run the reactor long enough to collect one blend bin of polymer (80,000 kg of polymer). This is required to have a homogenous product in the blend bin to alleviate fines and streamers that would result from blending smaller quantities.

Some of the product met the experimental specifications. The remainder was sold as off-grade product and the cost of materials were just recovered. Senior management was aware of the impacts on process stability, but fully endorsed the design and implementation of this plant trial.

The company claimed Trial 1 as EP.

Upon analyzing the results from Trial 1 the catalyst development team determined that the likely cause of the temperature excursions was catalyst carryover into the recycle loop, and proposed methanol addition into the reactor as a possible solution for residual catalyst activity.

Some of the product from Trial 1 was given to a customer ABC at no cost for applications testing. The product met the required specifications for a general-purpose product. However, the customer indicated that they would like to have a further improvement in film gel specification for another family of products. This work would be done in Trial 3.

Trial 2: Evaluation of methanol as an in-reactor catalyst deactivator.

Date: May 26-27, 2005 Trial Time: 2 days

In this trial the company investigated a catalyst deactivator in the reactor. In previous pilot plant studies, methanol was identified as an effective catalyst deactivator. However there was a substantial risk that the methanol could have adverse effects on the product properties and steady-state operations of the reactor.

In order to complete the experiment, a temporary methanol injection facility was installed. This was intended only for experimental use during Trial 2, and was claimed as dedicated SR&ED equipment in the submission.

During the experiment, methanol was injected into the reactor at rates varying from 0.5-5 kg/hr, and the impact on process stability and product characteristics was evaluated every 2 hours. It was determined that the MeOH effectively deactivated the catalyst at an addition rate of 3 kg/hour and it appeared to enhance process stability. However, product properties were negatively impacted.

The company claimed the 2-day trial as EP.

None of the product met all of the required specifications, and it was sold as off-grade product, and the cost of materials were just recovered. Senior management was aware of the impacts on process stability, but fully endorsed the design and implementation of this plant trial.

Trial 3: Investigation to Improve Film Gel Properties

Date: June 1, 2005 Trial Time: 1 day

The customer requested 200 tonnes of resin for their application, since the product met their specifications for their general-purpose film. However, ABC also had a high-end film that required a lower gel specification. While they were producing the 200 tonnes of general-purpose film resin, Company XYZ carried out additional experimentation in Trial 3 to determine the potential for reducing the film gel specification. XYZ knew that a variation in solids residence time could impact on the potential for film gels. Work from the pilot plant study provided the commercial plant with the range of operating conditions that ensured that the product would be on-specification. Hence there was no expected impact on the product quality or on the process.

The third trial was then done to investigate if changes to solids residence time could eliminate the gel problem. All other parameters were retained at the conditions used in Trial 1. It was determined that residence time did not reduce the film gel problem.

The entire product met all of the required specifications, and it was sold as prime A-grade product. Materials were not claimed.

The company claimed Trial 3 as CP/ED.

Trial 4: Evaluation of Another Deactivator X for Gas Phase Process

Date: Sept- October, 2005

The company's management decided to investigate another type of proprietary Deactivator X in the reactor that might have minimal impacts on the product properties.

During start-up for Trial 4, a failure in the gas feeds purification resulted in the reactor flooding with impurities. The operations team undertook an investigation of the purification trains to identify the source of the impurities. The problem associated with the trains was remedied over a 2-day time period, and these trains were then placed back on-line with the reactor.

At this point the operations team resumed the investigation of Deactivator X for the gas phase process. Processing conditions were maintained identical to those noted for Trial 2, and a total of two days was subsequently needed to complete the work pertaining to Deactivator X.

The company claimed the 2 days for Deactivator X part of the trial as EP, and also claimed claimed the two days needed to investigate the purification trains.

None of the product met all of the required specifications, and it was sold as off-grade product, and the cost of materials were just recovered. Trial 4 was terminated and the company will now focus on developing new products starting initially at the pilot plant, with subsequent scale-up to the commercial plant. No further plant trials in support of this project were claimed beyond Trial 4 for the current tax year claim.

List of Contractors

CONTRACTOR NAME	ROLE & RESPONSIBILITY
Contractor X	Installation of methanol injector

1E. Supporting Information

1. Description of the work of Contractor for Installation of Methanol Injector
2. Experimental operating instructions and Test methods
3. Detailed logs of start-up operations
4. Experimental design logic and methodology
5. Experimental operating instructions and corresponding closing reports
6. Process engineering team meeting minutes
7. Senior management sign-off for experimental trials
8. Pilot-scale Reactor Experiments, Trial Results and corresponding closing reports
9. Capital project documentation for Methanol Injector
10. Team meeting minutes

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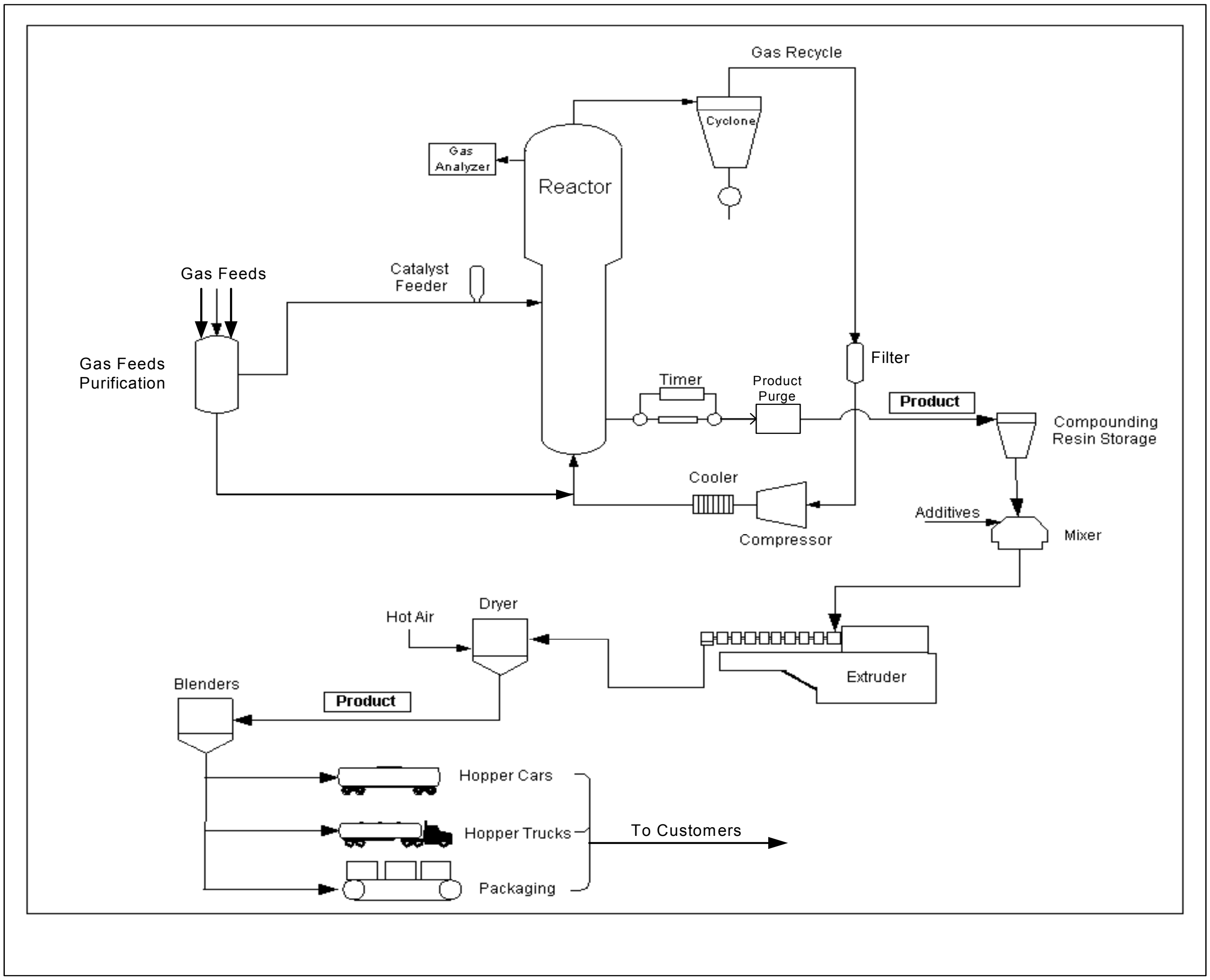

Figure 1: Continuous Multi-stage Gas-solids Fluidization Process for Large-Scale Polyethylene Production

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Table 1a: Expenditures Claimed using the Proxy Method

Total Directly Engaged Labor Cost Lab, Pilot Work and Plant Trials	$150,000
Material consumed/transformed • Product given to customers for Applications Testing (Trial 1) • Excess Additive Cost (Trial 1) • comonomer costs due to extra flaring (Trial 1)	$24,000 $20,000 $20,000 Total = $64,000
Capital Expenditures • Temporary Methanol Injection System (ASA for Trial 2)	$20,000
Trials 1-4 Total Claimed, excluding prescribed proxy amount (PPA)	$234,000
PPA = labour*.65	$150,000*0.65 = $97,500
Trials 1-4 Total Claimed, including PPA	$234,000+97,500= $331,500

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Table 1b: Expenditures Claimed using the Traditional Method

Total Directly Engaged Labour Cost Lab, Pilot Work and Plant Trials	$150,000
Material consumed • Product given to customers for Applications Testing (Trial 1) • Excess Additive Cost (Trial 1) • comonomer costs due to extra flaring (Trial 1)	$24,000 $20,000 $20,000 Total = $64,000
Capital Expenditures • Temporary Methanol Injection System (ASA for Trial 2)	$20,000
Trials 1-4 Total Claimed, excluding traditional overheads	$234,000
Listing of Traditional Overheads Incurred (all costs are either ASA attributable or directly related and incremental to SR&ED for 3 EP and 1 CP/ED Plant trials and Company Lab Facilities): 1. Utilities (steam, natural gas, electricity, water, nitrogen, air) – Total costs of $500,000 2. Benefits & Incentives – Total cost $80,000, less Managers and Admin. $5,000 = $75,000. 3. Contract services (Pipefitters, millwrights) - Total costs of $100,000. 4. Supplies - Total costs of $25,000. 5. Maintenance - Total costs of $150,000. 6. IT Costs - Total costs of $10,000. 7. Plant operating costs - Total costs of $100,000, $20,000 ineligible. 8. Technical Services - Total costs of $40,000. Total Overheads - Traditional Method	 $500,000 $75,000 $50,000 $25,000 $150,000 $10,000 $80,000 $40,000 $930,000
Trials 1-4 Total Claimed, including traditional overheads	$234,000 + $930,000 = $1,164,000

Analysis of Project:

The project involved the development of a gas phase process using a newly developed family of metallocene catalyst. The work was planned and carried out in a systematic fashion by qualified technical personnel. Plant trials (1-3) are commensurate with the needs of the SR&ED project. In plant trial 4 the work related to fixing the purification trains is not in support of the advancement sought in this project. Rather this work is related to maintenance of the operating facility and is not related to the development of the gas phase process. Only the work related to Deactivator X in Trial 4 is commensurate with the needs of the project. As such, the work for Trials 1-3, and the Deactivator X work in Trial 4 meets the definition of SR&ED in Subsection 248(1) of the Income Tax Act.

CRA Decision:

The Research and Technology Advisor (RTA) verified that there was a SR&ED project. During the technical review of the claim the RTA confirmed all of the following in support of Trials 1, 2, and 4 (Deactivator X work) being claimed as EP:

- The product from Trials 1, 2, and 4 was either given away, or it was sold as off-grade to second-tier customers, and the cost of materials was just recovered.
- There were significant process changes made during Trials 1, 2 and 4 including the testing of the new metallocene catalyst and alternative deactivators.
- Additional technical staff and R&D time (staff not normally allocated to plant trials work) was allocated to trials 1, 2, and 4 by senior plant management, in order to be able to better handle the increased volume of data associated with experimental test trials.
- Specific experimental operating instructions and other consistent records were prepared for Trials 1, 2 and 4 as part of the original project plan.
- Employees were involved in designing specific experiments, and monitoring and analyzing test data from all trials.

As a result of these findings by the Research and Technology Advisor (RTA), and the other technical considerations and factors as noted in the preceding project description, it was confirmed that Trials 1, 2 and 4 (Deactivator X work) were EP.

For Trials 1, 2, and 4 (Deactivator X work) work was claimed for all processing stages in the plant. Specifically, for a continuous process, a poor quality product from the Fluidized Bed Reactor would be expected to impact adversely on the quality of the product from each of the subsequent stages situated downstream of the reactor. Hence, all of this work (Trials 1, 2, and 4: Deactivator X part) was considered to be EP. The claimant chose not to include materials transformed during Trials 1, 2 and 4 since the product was sold and the recapture rules would have applied to the materials transformed.

Plant trial 3 was considered to be CP/ED. From this trial only the incremental labour costs were claimed as SR&ED expenditures. The materials and overheads associated with Trial 3 were not claimed. The labour cost for one operator (1 FTE x 24 hours) and one process engineer (for experimental planning, monitoring, and completing the analysis; 24 hours) was the only claim made for the Trial 3. The entire output product was sold as prime or “A-grade”.

During the review of the claim the Financial Reviewer (FR) confirmed that only incremental expenditures for the labour were claimed for Trial 3, and that the company had sufficient documentation to support their methodology.

As a result of these findings by the FR, and the other relevant technical considerations and factors as noted in the preceding project description, it was confirmed that the expenditure allocation for Trial 3 was appropriate for a CP/ED trial. It was also confirmed that the expenditures reported for Trials 1,2, and 4 (Deactivator X work) could be substantiated with the relevant documentation. The work related to the purification trains in Trial 4 was disallowed.

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References for Example 3.5

[1] Chemicals Guidance Document 3 Part 1 – Chemical Processes
http://www.cra-arc.gc.ca/taxcredit/sred/publications/chem3/chem3-README.html
http://www.cra-arc.gc.ca/taxcredit/sred/publications/chem3/chem3-LISEZ-MOI.html

[2] Application Policy SR&ED 2002-02R2: Experimental Production and Commercial production with experimental development work – Allowable SR&ED Expenditures.
http://www.cra-arc.gc.ca/taxcredit/sred/publications/ap2002-02r2-e.html
http://www.cra-arc.gc.ca/taxcredit/sred/publications/ap2002-02r2-f.html

[3] Brown, G.L., Warner, D.F., and Byon, J.H., “Exothermic Polymerization in a Vertical Fluid Bed”, United States Patent 4,255,542: “Exothermic Polymerization in a Vertical Fluid Bed” (March 10, 1981).

[4] McAuley, K.B. and MacGregor, J.F., “On-line Inference of Polymer Properties in an Industrial Polyethylene Reactor”, AIChE J. 37(6): 825-835 (June 1991).