[On line chemical composition
of analyzer development]
Status Report

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MASTER

Program/Project Identification No.: DE-FC07-88ID-12691	2. Program/Project Title: On-Line Chemical Composition Analyzer Development	3. Reporting Period: 2/1/92 to 4/30/92
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INTRODUCTION

A summary of the status of the Phase II project tasks relevant to this time period is as follows:

1. a) Steady-state analysis - 100% complete. A first approximation model is complete.
Modifications will be incorporated as necessary throughout the project.
- b) Dynamic analysis - 50% complete.
2. a) Test plan Development - 100% complete.
- b) Review of test plan 100% complete.
3. a) Site Preparation - 100% complete.
- b) Supervision of installation - 90% complete.
4. c) Control system decision - 20% complete.
8. a) Seminars - 50% complete.
- b) Papers - 50% complete.

A. Summary of Steady-State Analysis

The steady state work presented in the last quarterly report was found to be satisfactory and has been used for the dynamic and simulation work started this quarter. It is important to note that additional steady-state analysis will probably be required as the work progresses. The column selected for the field test is also being studied by Tennessee Eastman Company (TEC). The purpose of these studies is to improve the operation and control of the column. As modifications and improvements are implemented it may be necessary to rerun some of the steady state as well as the dynamic studies. The initial steady state analysis is complete at this time.

B. Summary of the Dynamic Analysis and Simulation

Dynamic studies of the distillation column are currently proceeding. A dynamic simulation of TEC's No. 32 Azeotropic Distillation Tower was developed in the following manner. To provide a starting case, the steady state model (stage to stage composition and temperature profiles) and operational data [including: (1) the flow rates, (2) stream compositions, (3) temperatures, and (4) pressures] were used to model the column. The

resulting computer simulations were then reviewed with TEC operations' personnel to get their comments and suggestions. Using the current dual ended temperature control scheme, the model was also tested to ensure that it reflects reality as well as possible. Based on the operating data, the temperature profile on the simulation closely matches the actual operation.

Based on a TEC concern that the vapor liquid equilibria model might not be adequate, a study of an alternative model has been completed. The old model used the Wilson equation to capture the nonidealities of the liquid and vapor phase association (Van't Hoff coefficients) to capture the nonidealities of the vapor. The new model uses vapor phase association and the NRTL equation and parameters to model the liquid nonidealities. Theoretically, the NRTL equation can model the extremely nonideal behavior such as phase splitting (i.e. two liquid phases) whereas the Wilson equation cannot. This new model for the ternary system results in a slightly different column temperature profile and composition profile. Dynamic perturbation studies with this model should provide further insights into the nature of azeotropic distillation.

The goal of the dynamic studies is to determine how the composition information can be best used to control the production. Perturbation studies using temperature controllers and composition controllers have been made. While not complete, these studies have yielded insights into the nature of azeotropic distillation. For instance, variation studies have shown the existence of multiple steady states (operating profiles) in the column. Current plans call for using ternary phase diagrams to confirm these results. These studies will aid in development of the strategy to use the composition analyzer to control the distillation column.

Another study that is underway is attempting to quantify the amount of variation entering the column. Six months of operational data was obtained from Tennessee Eastman Company. Analysis of the data has yielded an idea of the amount of variation in the flow rates of the feeds (one liquid and one vapor) as well as the amount of variation in the compositions of the reflux and feeds. Also, an attempt to capture the pressure and temperature variations of the feed and reflux is being made. It is important to determine these quantities in order to ensure that the control strategy will be able to handle the variation. Developing the strategy also requires some level of understanding of how the column responds to these variations when under current control (i.e. dual ended temperature control) and in the case of no control.

The following summarizes the status of the dynamic studies: (1) The dynamic model is complete. (2) A new set of vapor liquid equilibria equations/parameters are available for investigation of azeotropic distillation. (3) Perturbation studies and (4) analysis of the amount of variation entering the column are continuing. (5) Preliminary results indicate that multiple steady states do exist in the column.

C. Test Plan:

The Phase III test plan included in the first quarterly report has been approved by DOE. A copy of the letter indicating approval is appended to this report.

D. Field Installation of Sample Cell and Spectrometer

The field test of the Raman Analyzer on a production column at Tennessee Eastman Co. is still in the initial phase. At this date an enclosure has been designed and fabricated for the sample cell. The enclosure was designed to be intrinsically safe for a process area with explosive volatile gases. Special considerations were necessary since a relatively high power laser source is brought to the cell using fiber optics. The enclosure is a NEMA rated box with a number of additional safeguards and fail safe devices which will trip the power to the laser during any abnormal operation.

The enclosure was designed by engineers and scientists at Tennessee Eastman, reviewed by engineers at the University of Tennessee, and constructed by DCS Inc. of Alcoa, Tennessee. The enclosure has been fabricated, inspected, and shipped to Kingsport. It is estimated that it will take two to three weeks for the installation of the box and the conduit. The conduit way is necessary to house and protect the fiber optic and electrical cables that connect the sample cell to the spectrometer and laser systems that will be located in the control room. The spectrometer will be delivered around the middle of June and the field test plan will start immediately after the installation is complete.

E. Control System Analysis and Design:

The dynamic control simulation will also be used in the analysis and design of present and proposed control strategies. During the past quarter a number of controllers were added to the simulation for such studies. These controllers presently include level controllers, temperature controllers, composition controllers, ratio

controllers and cascade controllers. The level controllers are used to control the bulk inventories in the column while the temperature and composition controllers maintain a temperature or composition setpoint for a particular stage in the column. The cascade controllers allow for setting the setpoint to another controller. For instance, the composition measurement from the Raman Analyzer can be compared to a composition setpoint. The resulting error can then be used to change the setpoint to the temperature controller. Although this describes a continuous process control strategy, a statistical process control strategy could also be used to determine when to make these setpoint changes. The ratio controller would allow one to use the ratio for two compositions or some combination of two or more process signals. Comparison of how well these strategies perform has been initiated and will proceed throughout the project.

F. Du Pont Technology Transfer Study:

A distillation column at an E.I. Du Pont de Nemours facility in Old Hickory Tennessee was also studied this quarter. The column under consideration at Du Pont is an acetic acid recovery unit similar to the field test column at Tennessee Eastman but with some major differences. The Eastman column is an azeotropic distillation of a feed consisting of acetic acid, water, and a solvent. The Du Pont column recovers only low purity acid from a feed which consists of water and many other organic components. The Du Pont column also has multiple feeds, each of which enters the column on a different tray. In terms of the operation and control of the column, it is also significant that each of the multiple feeds is a plant-wide recycle.

The Du Pont study involves an analysis of the column. It will focus primarily on the potential role of analyzers in the control of the column. It will consider the effectiveness of placing analyzers in the column (a la Tennessee Eastman) but will also consider analyzers placed in one or more of the various column feeds. At present steady state and dynamic simulations have been developed and verified for the Du Pont column. The control system studies are expected to start in early June and be completed by the end of the summer. It should be noted that a parallel and overlapping study is also being conducted of the plant-wide control problems of the multiple recycle systems. Initial studies indicate that many of the control problems that manifest at the distillation column could be reduced if not eliminated by better control over these recycle systems. (In control systems as in

medicine, and once of prevention is often worth a pound of cure). It is anticipated that this study will also show that analyzers are needed in parts of the plant other than the distillation column.

It is important to emphasize that the Du Pont study does not directly involve the Raman analyzer which is being tested at Tennessee Eastman. The purpose of the Du Pont study is to broaden our basic understanding of the role of analyzers in the operation and control of plants which use distillation columns. It is anticipated that Du Pont will implement those recommendations that can be addressed by present commercial analyzer technology. Other recommendations that require an on-line analyzer with higher performance than is presently available will simply have to wait until the initial Raman work is complete and has been commercialized in a package suitable for the rigors of the chemical industry.

G. Seminars:

The on-line composition project was reviewed at the April Measurement and Control Engineering Center Industrial Advisory Board meeting. A list of attendees for this review/seminar is appended.

H. Papers:

Presentations were made at two meetings this quarter and papers were prepared for inclusion in conference proceedings. SPIE, the society of Photo-optical Instrumentation Engineers, held a conference on Optically Based Methods for Process Analysis in Somerset, New Jersey, March 23-26, 1992. "On-line analysis of chemical composition using an FT-Raman spectrometer in the near-ir" was presented by Arlene Garrison at that meeting and a copy of the paper is appended to this report. Anatech '92, an international symposium on Analytical Techniques for Process Control, was held in Atlanta, GA, April 5-8, 1992. The paper from this presentation, "Distillation process control using FT-Raman spectroscopy" is appended and will be published in a special edition of the journal Process Control and Quality.

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