

of their sensitivity for measuring low impurity concentrations, such high sensitivity or Micro TCDs tend to over-range when exposed to the high concentrations of ethylene oxide in liquid ethylene oxide refined product streams. In addition, the peaks for acetaldehyde and ethylene oxide are close. Therefore, the ethylene oxide thermal conductivity signal may distort the acetaldehyde signal, resulting in inaccurate acetaldehyde concentration determinations. One known solution for this type of problem is to backflush the gas chromatograph or to use a splitter. However, because of the small amounts of impurities being measured and the sensitivity of micro TCDs, the backflush technique would produce a significant baseline upset in the carrier gas signal, yielding inaccurate concentration determinations. The splitter technique is generally not consistent. At impurity levels typically found in ethylene oxide purification processes, the splitter technique could introduce an error on the order of 25%.

[0012] Also, using thermal conductivity detectors forecloses the use of temperature programmable ovens to speed up the composition analysis because changes in temperature will affect the thermal conductivity of the gas. In certain cases, adjusting the oven temperature causes the compounds in the sample being tested to separate more distinctly from one another (better resolution), yielding clearer and more distinct compound peaks from the gas chromatograph effluent detector. However, because adjustments to the oven temperature will affect the thermal conductivity of the sample, the resulting thermal conductivity signals will not be indicative of the concentrations of the compounds comprising the sample.

SUMMARY

[0013] In accordance with the present disclosure, an automated method of detecting impurities in a liquid ethylene oxide stream having at least one impurity is provided. The method detects impurities in the liquid ethylene oxide stream as the liquid ethylene oxide stream is produced by a processing unit. The method comprises injecting a sample volume from a liquid ethylene oxide stream into a gas chromatograph train having a gas chromatograph effluent stream, and generating at least one signal indicative of the corresponding concentration of the at least one impurity.

[0013a] In accordance with a first aspect of the present disclosure, an automated method for determining the concentration of impurities in a liquid ethylene oxide stream having at least one impurity as the liquid ethylene oxide stream is produced by a processing unit is disclosed, comprising: injecting a sample volume from the liquid ethylene oxide

stream into a gas chromatograph train with one or more columns and having a gas chromatograph effluent stream; and generating at least one signal indicative of the corresponding concentration of the at least one impurity.

[0013b] In accordance with a second aspect of the present disclosure, method of operating an ethylene oxide purification column having a liquid ethylene oxide product stream is provided, the method comprising: performing the method of claim 1 to determine the concentration of at least one impurity in the liquid ethylene oxide product stream, wherein the processing unit comprises the ethylene oxide purification column, and the liquid ethylene oxide stream comprises the liquid ethylene oxide product stream; updating a mathematical model relating a predicted concentration of the at least one impurity in the liquid ethylene oxide product stream to selected ethylene oxide purification column process variables based on the at least one signal indicative of the corresponding concentration of the at least one impurity; calculating a predicted concentration of the at least one impurity in the liquid ethylene oxide product stream based on values of the selected process variables and the updated mathematical model; and adjusting at least one ethylene oxide purification column process variable based on the predicted concentration of the at least one impurity.

[0014] In accordance with the present disclosure, a system for determining the concentration of at least one impurity in a liquid ethylene oxide product stream is provided. The system comprises a gas chromatograph train having a gas

chromatograph effluent stream, wherein the gas chromatograph train is in selective fluid communication with a liquid ethylene oxide sample chamber that is in selective fluid communication with the liquid ethylene oxide stream and a carrier gas stream. The system also includes a gas chromatograph effluent detector, wherein the gas chromatograph effluent stream is in fluid communication with the GC effluent detector and a vent stream, and when the gas chromatograph effluent stream is fluidly coupled to the gas chromatograph effluent detector, the gas chromatograph effluent detector generates a signal indicative of a concentration of at least one impurity in the gas chromatograph effluent stream.

[0014a] In accordance with a third aspect of the present disclosure, a system for automatically determining the concentration of at least one impurity in a liquid ethylene oxide stream produced by a processing unit is provided, a gas chromatograph train having a gas chromatograph effluent stream, wherein the gas chromatograph train is in selective fluid communication with a liquid ethylene oxide sample chamber that is in selective fluid communication with the liquid ethylene oxide stream and with a carrier gas stream; a gas chromatograph effluent detector, wherein the gas chromatograph effluent stream is in selective fluid communication with the gas chromatograph effluent detector and a vent stream, and when the gas chromatograph effluent stream is fluidly coupled to the gas chromatograph effluent detector, the gas chromatograph effluent detector generates a signal indicative of a concentration of at least one impurity in the gas chromatograph effluent stream.

[0015] In accordance with a fourth aspect of the present disclosure, a system for controlling the concentration of at least one impurity in a liquid ethylene oxide stream from an ethylene oxide purification column is provided. The system comprises a computer readable medium having stored thereon a set of computer executable instructions comprising a mathematical model relating a predicted value of the concentration of the at least one impurity to values of a plurality of ethylene oxide process variables such that when executed by a computer processor, the computer executable instructions (i) calculate a predicted value of the concentration of the at least one impurity from the mathematical model and the values of the plurality of ethylene oxide purification column process variables; and (ii) update the mathematical model based on the predicted value of the concentration of the at least one impurity and at least one value of the concentration of the at least one impurity determined by the system for determining the concentration of at least one impurity; and a controller that

automatically adjusts the value of one or more of the plurality of ethylene oxide process variables based on the predicted value of the concentration of the at least one impurity.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] FIG. 1 is a schematic view of an ethylene oxide reaction system and a downstream refining unit;

[0017] FIG. 2 is a schematic view of an ethylene oxide refining unit;

[0018] FIG. 3 is a schematic view of an ethylene oxide impurity detection system comprising a sample conditioning system and a GC analyzer system; and

CLAIMS

WHAT IS CLAIMED IS:

1. An automated method for determining the concentration of impurities in a liquid ethylene oxide stream having at least one impurity as the liquid ethylene oxide stream is produced by a processing unit, comprising:

injecting a sample volume from the liquid ethylene oxide stream into a gas chromatograph train with one or more columns and having a gas chromatograph effluent stream; and

generating at least one signal indicative of the corresponding concentration of the at least one impurity.

2. The automated method of claim 1, wherein the step of injecting a sample volume from the liquid ethylene oxide stream into a gas chromatograph train having a gas chromatograph effluent stream comprises accumulating the sample volume of liquid ethylene oxide from the liquid ethylene oxide stream in a sample chamber that is in selective fluid communication with the liquid ethylene oxide stream and the gas chromatograph train, and fluidly coupling the sample chamber to the gas chromatograph train.

3. The automated method of claim 1, wherein the at least one impurity is selected from the group consisting of acetaldehyde, formaldehyde, carbon dioxide, and water.

4. The automated method of claim 1, wherein the step of injecting a sample volume of the liquid ethylene oxide stream into a gas chromatograph train comprises fluidly coupling a sample chamber containing the sample volume to a carrier gas stream such that the carrier gas introduces the sample stream into the gas chromatograph train.

5. The automated method of claim 4, wherein the step of fluidly coupling the sample chamber to the carrier gas stream comprises flash vaporizing the sample volume.

6. The automated method of claim 1, wherein the gas chromatograph train is contained in a temperature controlled housing operated at a first temperature, and the step of the injecting the sample volume into the gas chromatograph train comprises injecting

the sample volume into a carrier gas stream outside of the housing at a second temperature less than the first temperature.

7. The automated method of claim 1, wherein the step of generating the at least one signal indicative of the corresponding concentration of the at least one impurity comprises introducing the gas chromatograph effluent stream into a gas chromatograph effluent detector, and the automated method further comprises diverting a portion of the gas chromatograph effluent stream away from the gas chromatograph effluent detector, wherein the diverted portion of the gas chromatograph effluent corresponds to ethylene oxide.

8. The automated method of claim 7, wherein the at least one impurity comprises acetaldehyde, acetaldehyde has a retention time, ethylene oxide has a retention time, and the step of diverting a portion of the gas chromatograph effluent stream away from the gas chromatograph effluent detector comprises diverting the portion of the gas chromatograph effluent after the expiration of the acetaldehyde retention time and before the expiration of the ethylene oxide retention time.

9. A method of operating an ethylene oxide purification column having a liquid ethylene oxide product stream, the method comprising:

performing the method of claim 1 to determine the concentration of at least one impurity in the liquid ethylene oxide product stream, wherein the processing unit comprises the ethylene oxide purification column, and the liquid ethylene oxide stream comprises the liquid ethylene oxide product stream;

updating a mathematical model relating a predicted concentration of the at least one impurity in the liquid ethylene oxide product stream to selected ethylene oxide purification column process variables based on the at least one signal indicative of the corresponding concentration of the at least one impurity;

calculating a predicted concentration of the at least one impurity in the liquid ethylene oxide product stream based on values of the selected process variables and the updated mathematical model; and

adjusting at least one ethylene oxide purification column process variable based on the predicted concentration of the at least one impurity.

10. A system for automatically determining the concentration of at least one impurity in a liquid ethylene oxide stream produced by a processing unit;

a gas chromatograph train having a gas chromatograph effluent stream, wherein the gas chromatograph train is in selective fluid communication with a liquid ethylene oxide sample chamber that is in selective fluid communication with the liquid ethylene oxide stream and with a carrier gas stream;

a gas chromatograph effluent detector, wherein the gas chromatograph effluent stream is in selective fluid communication with the gas chromatograph effluent detector and a vent stream, and when the gas chromatograph effluent stream is fluidly coupled to the gas chromatograph effluent detector, the gas chromatograph effluent detector generates a signal indicative of a concentration of at least one impurity in the gas chromatograph effluent stream.

11. The system of claim 10, further comprising:

a gas chromatography analyzer system that comprises the gas chromatograph train and the gas chromatograph effluent detector, wherein an inlet stream to the gas chromatograph train is in selective fluid communication with the liquid ethylene oxide sample chamber, and when the gas chromatograph effluent stream is fluidly coupled to the vent stream, the gas chromatograph effluent stream bypasses the gas chromatograph effluent detector.

12. The system of claim 10, further comprising a multi-port separation valve connected to the gas chromatograph train.

13. The system of claim 12, further comprising a multi-port diversion valve connected to the multi-port separation valve and a multi-port diversion valve carrier gas inlet stream.

14. The system of claim 13, wherein the multi-port diversion valve is configured to selectively connect the gas chromatograph train to a gas chromatograph effluent detector and a vent stream.

15. A system for controlling the concentration of at least one impurity in a liquid ethylene oxide stream from an ethylene oxide purification column, comprising:

a computer readable medium having stored thereon a set of computer executable instructions comprising a mathematical model relating a predicted value of the concentration of the at least one impurity to values of a plurality of ethylene oxide process variables such that when executed by a computer processor, the computer executable instructions (i) calculate a predicted value of the concentration of the at least one impurity from the mathematical model and the values of the plurality of ethylene oxide purification column process variables; and (ii) update the mathematical model based on the predicted value of the concentration of the at least one impurity and at least one value of the concentration of the at least one impurity determined by the system of claim 10; and

a controller that automatically adjusts the value of one or more of the plurality of ethylene oxide process variables based on the predicted value of the concentration of the at least one impurity.