BATCH DISTILLATION

1. BATCH DISTILLATION WITH FRACTIONAL COLUMN

1.1 INTRODUCTION

Batch distillation is commonly used in pharmaceutical industry, where mass flows are small and there is no need for continuous distillation. Batch distillation is used for purifying solvents, when the bottom product is e.g. formed salt or heavy organic component. The solvent is purified to distillate. A batch distiller can be also used in multicomponent distillation. Purity of the fractions depends on number ideal stages, reflux ratio and thermodynamic nature of the mixture.

A schema of a batch distiller with fractional column is shown in Figure 1.

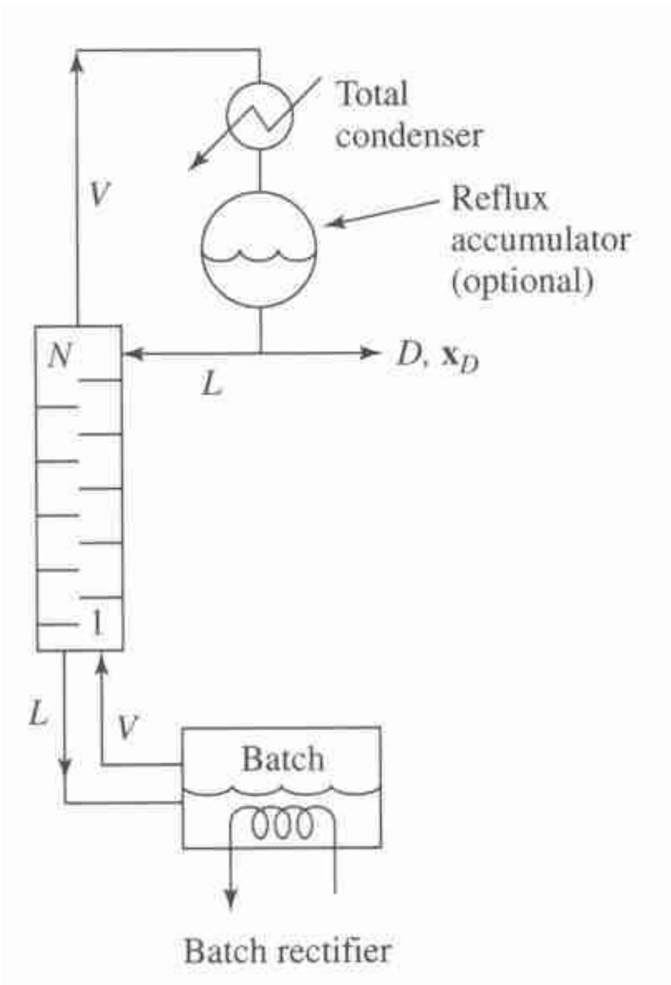


Figure 1. A batch distiller with fractional column.

The batch distiller can be operated with two different strategies: constant reflux ratio or constant concentration of distillate. The operation of batch distillation can be analyzed using McCabe-Thiele diagram for binary mixture as shown in Fig 2.

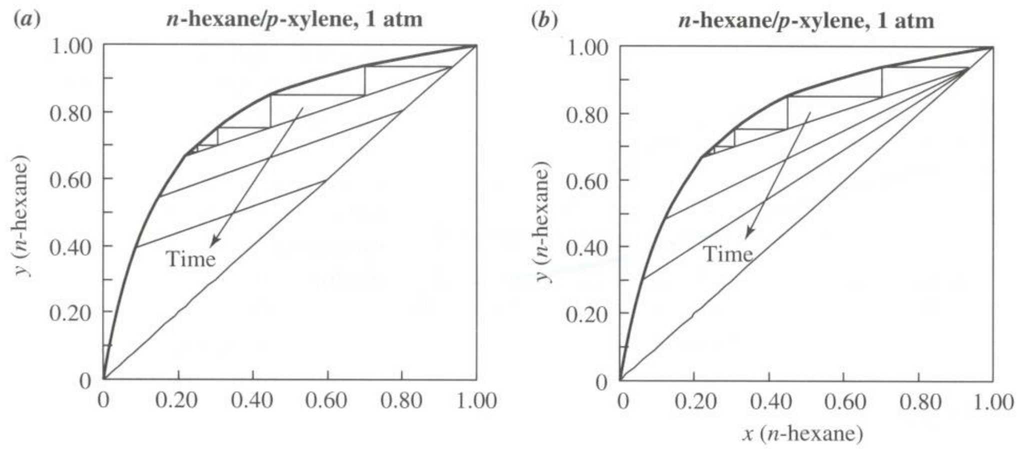


Fig 2. McCabe-Thiele diagram for a batch distillation, (a) constant reflux ratio, (b) constant concentration of distillate.

The constant reflux ratio is technically easier to carry out and to operate than operating with constant concentration. Control equipment is needed to stabilize concentration of the distillate. Batch distillation is a dynamic operation, because concentrations in boiler, inside column, in reflux vessel and in distillate receiver are changing all the time. Therefore, distillation time is most essential variable in batch distillation.

2. OPERATING THE LABORATORY BATCH COLUMN

The batch distiller, located in the student's lab, has a reflux controller with a time switch. The controller gives 24 V DC to a coil which rises a ground glass joint with magnet. The ground glass joint when rised releases the flow to a distillate receiver or when closed there is total reflux. Temperatures are measured with Pt100 sensors, which send signal to the data logger and PC stores the temperatures into a log file.

In this experiment a binary or ternary mixture is to be distillate. The concentrations, depends on the mixture, are determined by refractive index or by refractive index and density. There are two measuring equipment for refractive index: manual and semi-automatic. Density is measured with mass-volume method or with a densimeter.

Binary mixture, methanol-isopropanol, is measured only with a manual reflective index meter. Methanol-water and isopropanol-water binary mixtures are measured with a manual or a semiautomatic reflective index meter and also with mass-volume method.

Supplies for the batch distiller:

- batch distillation equipment
- stop watch
- erlenmeyers
- thermometer, Nokeval + PC-logger
- safety mask

- isopropanol + water, minimum boiling point azeotrope
- methanol + water, no azeotropes
- methanol + isopropanol, no azeotropes

Methanol is poisonous. Avoid eye or skin contacts. Inhalation of vapour is also harmful.

2.1 MEASUREMENTS

1) Check condition of rubber tubes.
2) Turn on cooling water circulation.
3) Check that the bottom flask and the distillation receiver are clean.
4) Check that the valves of the distillation receiver are moving smoothly.
5) Check that sensors of thermometer are in the condenser and in the boiler.
6) Make the mixture for batch distillation. Use components and concentrations given by the assistant. Pure the mixture to the bottom flask.
7) Turn on the Nokeval data logger.
8) Turn on magnetic mixer and heating path.

9) Follow temperatures in the data logger.
10) Use total reflux i.e. reflux controller is turned off.
11) Let temperatures normalize.
12) Check the minimum reflux ratio with XY-diagram.

13) When temperatures are normalized, turn on the reflux controller with reflux ratio greater than minimum reflux ratio. Take immediately sample of the distillate.

Repeat phases 14)-16) five times, when 10 %, 20 %, 30 % and 50 % of volume of the batch has been distilled. Write down the clock time of data logger computer every time the sample is taken.

14) Determine mass flow of distillate with scales and stop watch (5 min). Take a sample of distillate and determine refractive index.

15) Define the reflux ratio with timing. Measure two minutes time how long has the reflux controller been in ON-position.

16) Observe flooding of the column. If the column is flooding, decrease heating power.

17) Do not distillate too long. Leave the mixture in the bottom flask at least ¼ of initial volume.

18) Stop distillation by turning off heating path. Leave the mixer on. Let the cooling water circulation be on atleast 30 minutes,

19) Turn off data logging and cooling water circulation.

20) Pure the distillate and the bottom product to the storing vessel.

2.2 SPECIFICATION

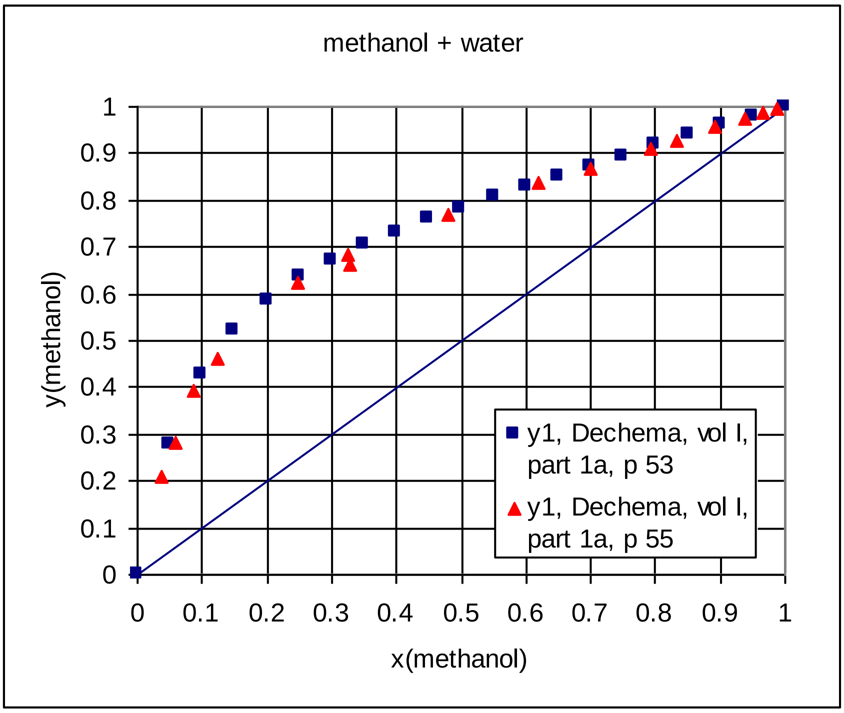
Determine at sample taking time the concentrations of distillate and batch using temperature data of the logger.

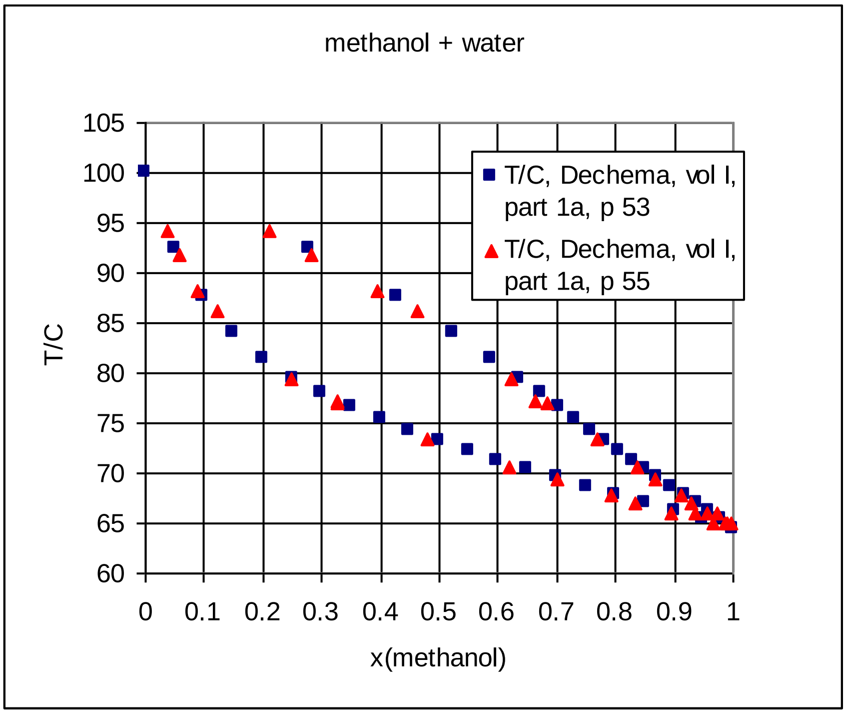
Calculate mole flows of vapour and liquid in top of the column.

Define the number of ideal stages.

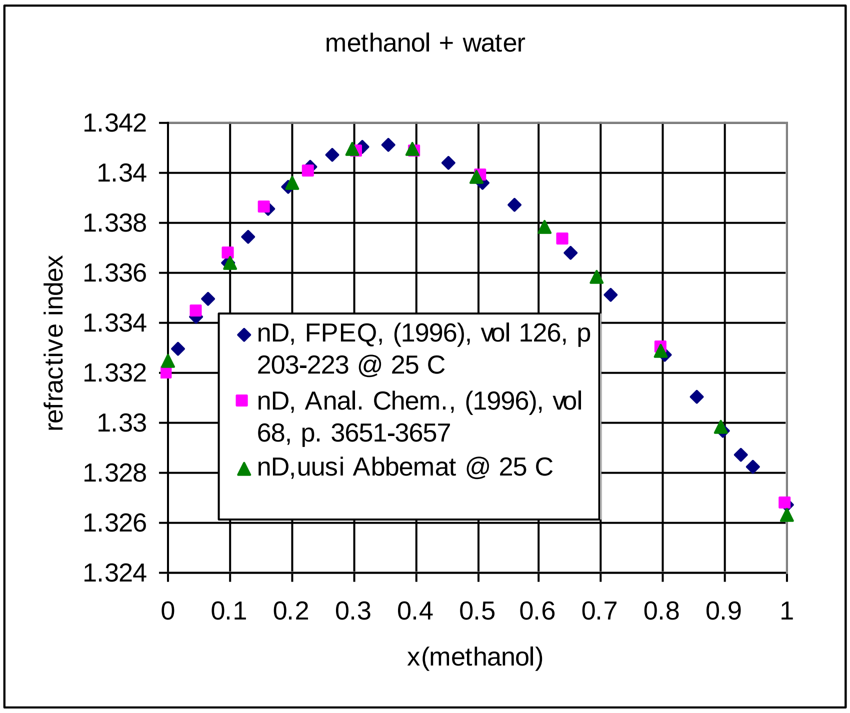
Measure height of the packings, and calculate HETP.

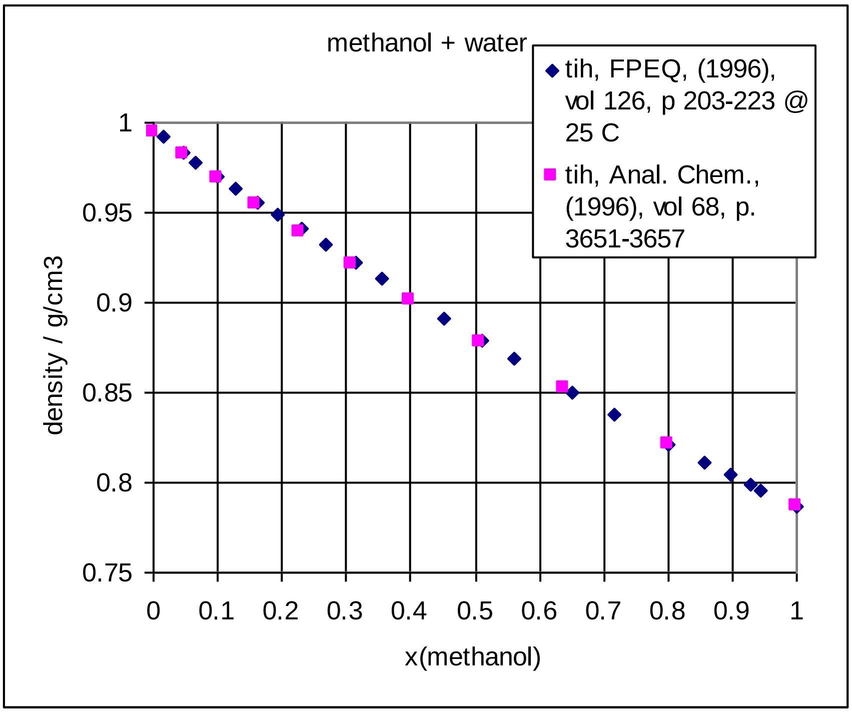
APPENDIX 1. EQUILIBRIUM DATA: METHANOL + WATER



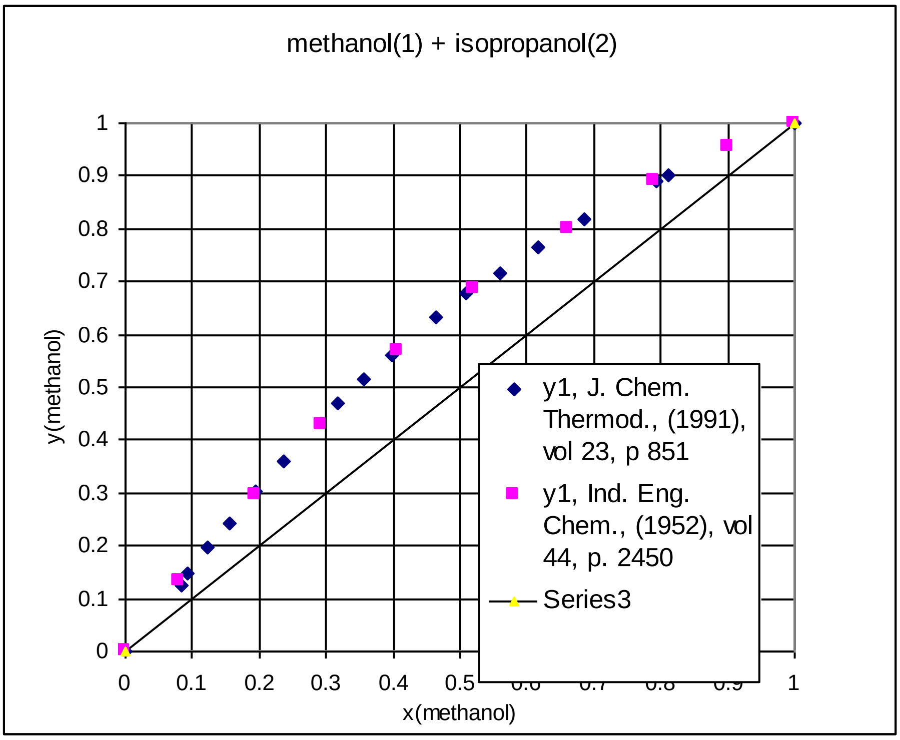


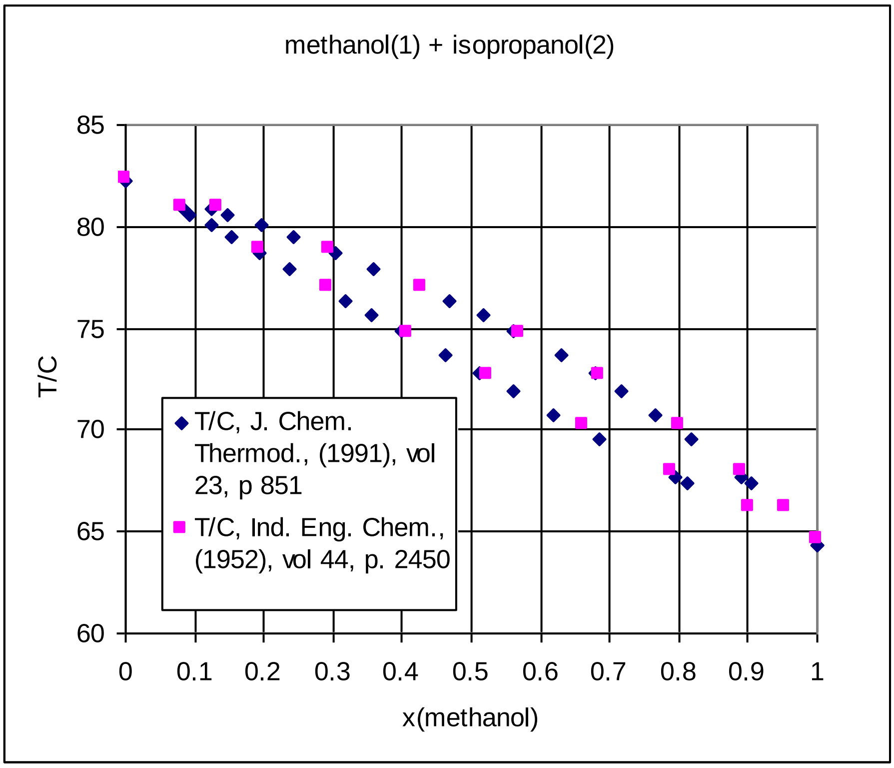
APPENDIX 1. EQUILIBRIUM DATA: METHANOL + WATER



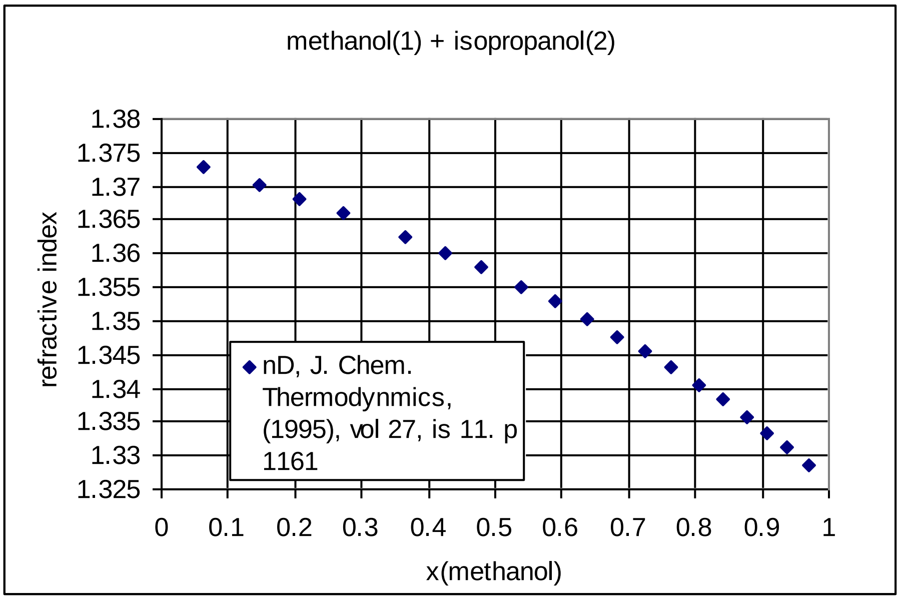


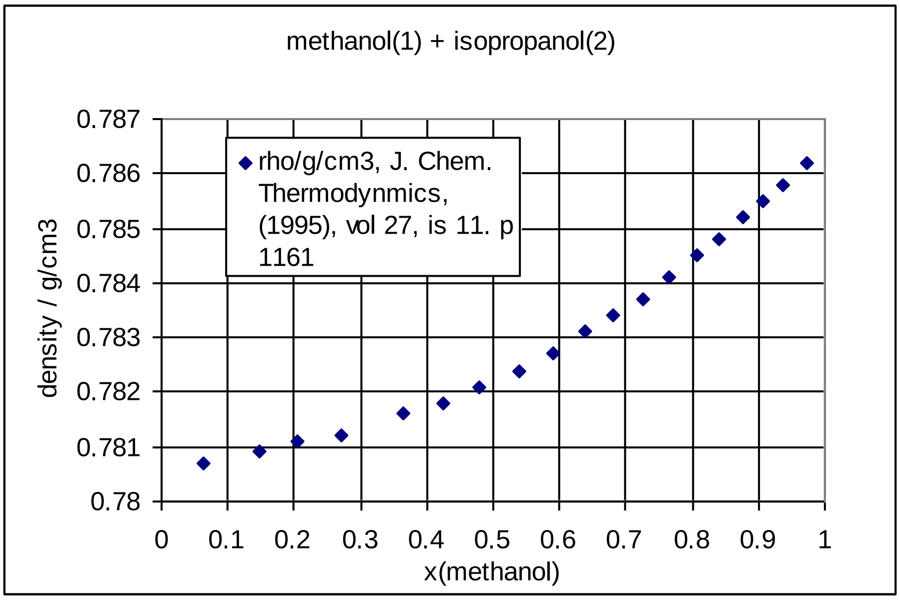
APPENDIX 2. EQUILIBRIUM DATA: METHANOL + ISOPROPANOL



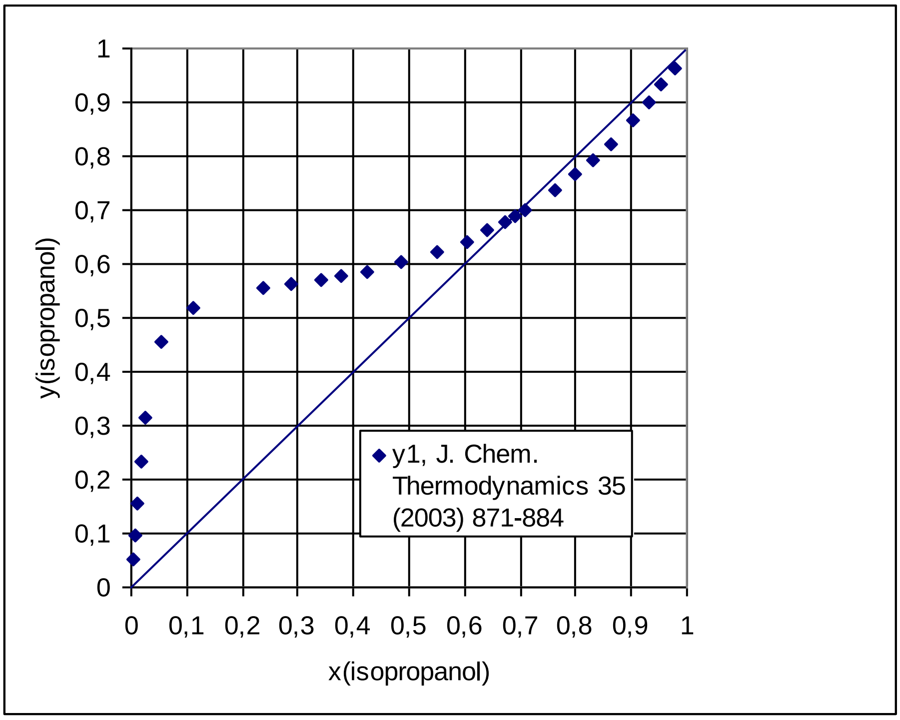


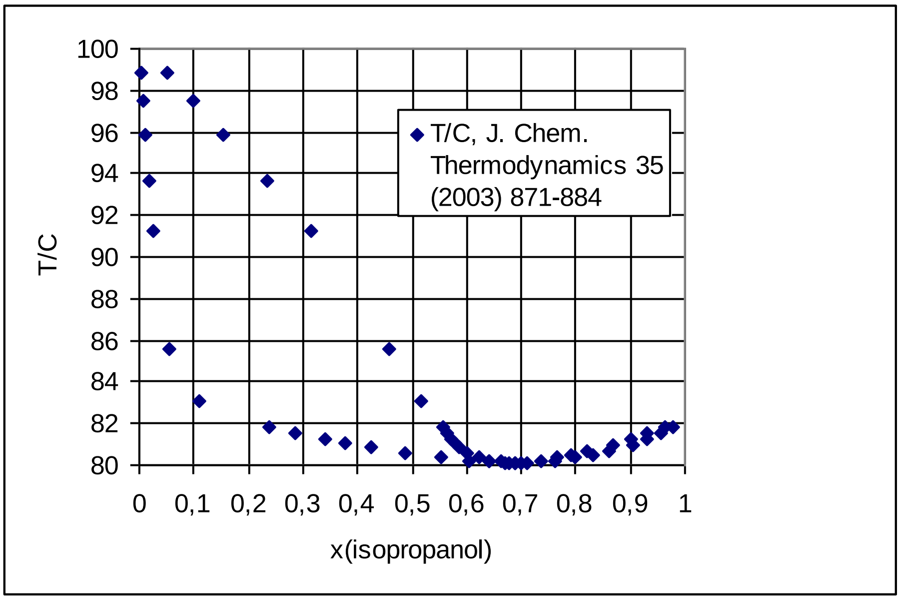
APPENDIX 2. EQUILIBRIUM DATA: METHANOL + ISOPROPANOL





APPENDIX 3. EQUILIBRIUM DATA: ISOPROPANOL +WATER





APPENDIX 3. EQUILIBRIUM DATA: ISOPROPANOL + WATER