CHAPTER 2

EXPERIMENTAL

2.1 Apparatus and chemicals

2.1.1 Apparatus

1. DC power supply Model 6612C 0-20V/0-2A, Agilent Technologies.
2. DC power supply Model 6613C 0-50V/0-1A, Agilent Technologies.
3. Multimeter Model UT60D (RS-232 serial port) by Uni-Trend International Limited, Hong Kong.
4. Multimeter Model UT30C by Uni-Trend International Limited, Hong Kong.
5. Dimmer 1200 watts.
6. Sensor head TGS 822 by Figaro Company Limited, U.S.A.
7. Syringe 1-10 μL by Supelco Company Limited.
8. Air pump, model 2.5 HP.
9. Micro-pipette; 2-10μL, 10-100μL and 100-1000μL by Biopette Company Limited.
10. Analytical Column 0.5 m. in length which is packed with 10% DEGS on chromosorb WHP, 80-100 mesh, made by course 205781.
11. Resister, 68 kΩ.
12. Chamber; home made.
13. Injector port; home made.
14. Thermocuple by Uni-Trend International Limited, Hong Kong.
15. Volumetric flask; 5mL, 100mL and 1000mL.

2.1.2 Chemicals

1. Ethanol, AR grade 99.7% (CH_3CH_2OH), Carlo Erba, France.
2. Acetone, AR grade 99.8% (CH_3COCH_3), Carlo Erba, France.

2.2 Preparation of the stock standard solution

2.2.1 Ethanol stock solution (1000 ppm)

Pipette 1.277 mL of AR grade ethanol (99.7%) into a 1- litre volumetric flask and make up to volume with reverse osmosis (RO) water. Mix well.

2.2.2 Stock standard solution of acetone (1000 ppm)

Pipette 1.267 mL of AR grade acetone (99.8%) into a 1- litre volumetric flask and make up to volume with RO water. Mix well.

2.2.3 Preparation of the standard ethanol and acetone concentration of 10.00, 20.00, 40.00, 80.00 and 160.00 ppm.

The standard ethanol and acetone solution with concentration of 10.00, 20.00, 40.00, 80.00 and 160.00 ppm are prepared by pipette acetone and ethanol 50, 100, 200, 400 and 800 μL of 1000 ppm acetone and ethanol stock solution of equal volume into each 5 mL volumetric flask, then adjust volume to 5 mL with RO water.

2.3 Basic measuring circuit

The basic circuit diagram is shown in Figure 2.1 Pins 2 and 5 are connected to the heater of the sensor unit, and the resistance between Pins 1 and 6 is designated as R_S. In pure air R_S is high. In the presence of any detectable gases, R_S changes with the variation of gas concentration.[44] Since V_c is a fixed voltage, the voltage on the resistor R_L is measured by a multimeter, If the load resistor (V_{RL}) increases while the R_S decreases, the change on R_S depends on the gas concentration.[45]

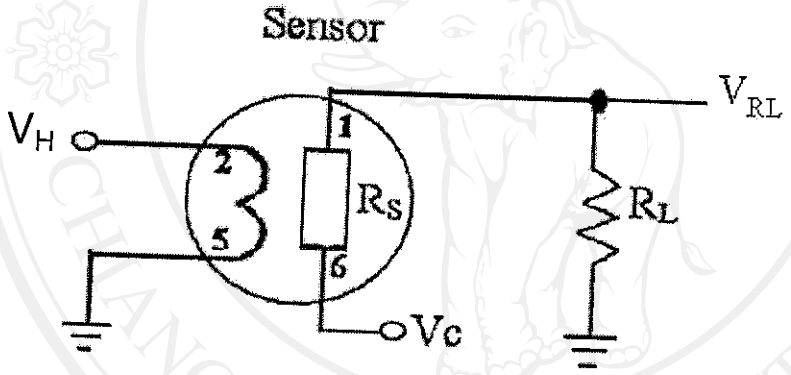

Figure 2.1 Basic circuit of a gas sensor.[44]

Where R_S is the resistance of the sensor, V_C is the circuit voltage, V_H is the voltage of heating coil, R_L is the load resistance, and V_{RL} is the output voltage in the resistance.

2.3.1 Effect of the flow rate

The flow rate of eluent is optimized by mixing ethanol and acetone standard solution of equal volume (1000 ppm). When 1 μL of these solutions is injected into the sensor system, the flow rate of carrier gas (air) is adjusted as 15, 26, 32, 42, 54, 80 and
112 mL/ min.

2.3.2 Optimization V_H of the heating coil

The operating temperature of the TGS 822 sensor is achieved by applying a voltage from dc source to the heating coil with varying voltage at 3.5, 4, 4.5, 5, 5.5, 6, 6.5 and 7 V respectively.

2.3.3 Optimization V_C of the circuit sensor

V_C is the circuit voltage, which is applied across R_S and R_L by a dc power supplier, from standard condition. The maximum V_C is 24 V. In this work V_C are varied as 1, 5, 8, 10, 13, 16, 18, 20 and 24 V .

2.3.4 Optimization R_L of the circuit sensor

R_L is the load resistance of circuit and R_S of TGS 822 sensor is calculated when the vapor of ethanol or acetone pass through the sensor head. The change of R_S depends on gas concentration. In this work the resistance of R_L are varied as 6, 10, 24, 38, 50, 68, 100, 120, 150, 180, 200 and 240 kΩ.

2.4 Experimental procedure

In this experiment, the schematic diagram for detection of ethanol and acetone sensitivity measurement is shown in Figure 2.2. The system for the detection of acetone and ethanol is constructed, which consists of a carrier gas, an injector port, an analytical column, a chamber with a sensor head, 2 dc power suppliers, a circuit sensor, a multimeter and a computer. The optimum condition of the system for the detection of acetone and ethanol is achieved by varying the flow rate of the carrier gas, V_H, V_C, R_L and the injector port temperature.

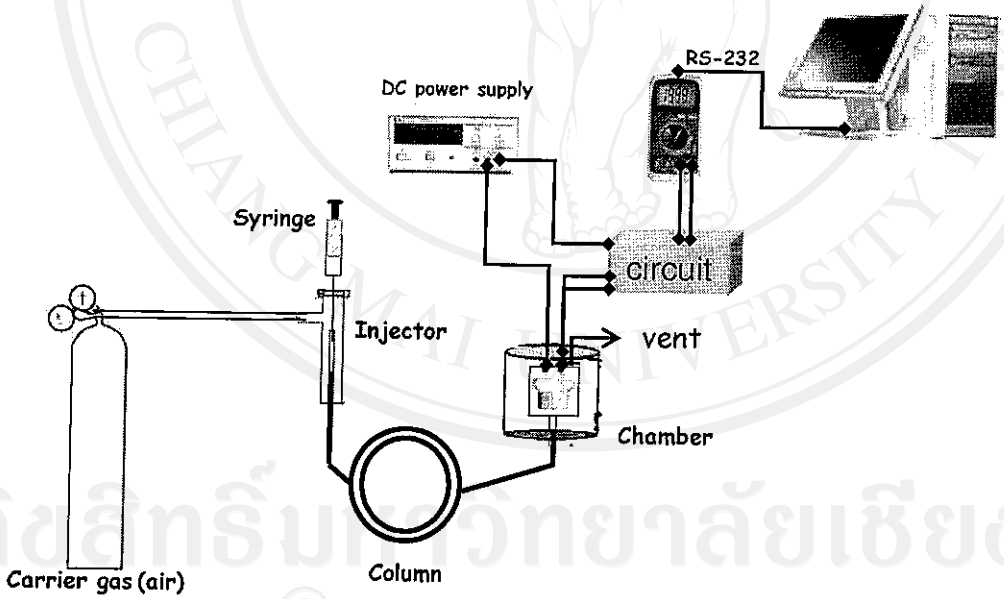

Figure 2.2 Schematic diagram for the detection of ethanol and acetone.

The process of analysis under optimum condition is shown in Figure 2.3. The optimum condition for the detection of acetone and ethanol is as follows :

(i) the flow rate of the carrier gas is 15 mL/ min.

(ii) circuit condition with V_H = 5.5 V, V_C = 20 V and R_L = 68 kΩ.

(iii) the temperature of the injection port is 150 °C.

A solution of ethanol and acetone with concentration of 10 mg/L, 20 mg/L, 40 mg/L, 80 mg/L and 160 mg/L are mixed together. 1 μL of the mixture is injected into the system and the response of the analytes are measured after the base line voltage (V_{air}) is stable. The chromatograms are shown on the monitor and the peak areas are recorded.

2.5 Characteristics of the procedure

2.5.1 Linearity range

Standard mixtures of ethanol and acetone with mixing proportion of equal volume are in the range from 10-160 ppm, when 1 μL of these solutions is injected into the sensor system under the optimum condition.

2.5.2 Limit of detection (LOD)

The standard solution of ethanol and acetone prepared in the range from 10-60 ppm and 5-40 ppm respectively. 1 μL of each concentration is injected into the sensor system under the optimum condition. In this work the TGS 822 response values in term of peak area are plotted against concentration of standard solution for

construction of the calibration graph. See Appendix for the calculation of the LOD of ethanol and acetone.

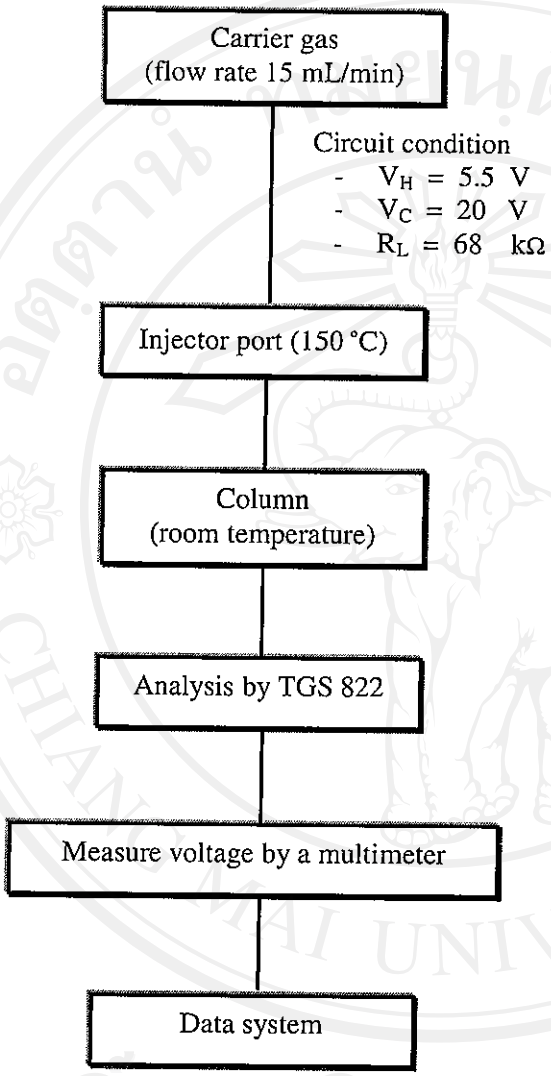

Figure 2.3 Process for the detection of ethanol and acetone under optimum condition.