Column chromatography lab report pdf template word free

GC is continuing to be used in a number of fields as an analytical tool due to certain advantages like: Shorter run times Greater sample throughput Cheaper columns Higher signal to noise ratio Lower bleed (thinner films) High resolution power compared to others. So we cannot identify or quantitate them. Sensitivity in detection is very high with thermal conductivity detectors. To do this equal volume of hexane and ethanol in a small vial were combined and injected. Stan + Mouthwash Int. Nevertheless, a peak for xylene was seen (determined by comparing the Rt with the Rt of toluene from Experiment 3). Discussion and Conclusion In this lab, using the technique of Gas Chromatography, four different experiments were performed. The other peak (although small) which is seen occurs at Rt = 1.600 is close to Rt for xylene (Rt for 10 % xylene is 1.600). For 2 %= 1 ml each of toluene or para-xylene (separate), for 4 % = 2ml For 6 % = 3 ml, for 8 % = 4 ml, and for 10 % = 5 ml to 50 ml with hexane. The soil sample was deliberately contaminated with toluene and xylene and placed in a vial for 10 minutes. Share this: Facebook Twitter Reddit LinkedIn WhatsApp GC-1 (Gas Chromatography) Experiment 1, 2, 3 & 4 Experiment 1:- Determination of ethanol content of a mouthwash using an internal standard Experiment 2:- Determination of oxygenates in gasoline Experiment 3:- Qualitative and Quantitative analysis of BTEX (Benzene, Toluene, Ethylbenzene and Xylene) Experiment 4:– Determination of volatile compounds by headspace analysis Introduction & Theory The experiments performed in this lab were based on Gas chromatography – specifically gas-liquid chromatography. Observations, Calculations and Results Instrument: GC SST Instrument settings: Injector temperature: 200 degree C Detector temperature: 250 degree C Oven temperature: 80 degree C Attenuation: between 4 and 16 Carrier gas pressure: 8-10 psi FID range: 1 Sample Retention time Compound present Soil Sample 1 1.033 1.533 Toluene P-Xylene Soil Sample 2 1.050 1.483 Toluene P-Xylene From Observation Table # 7, last experiment, we know retention times of both toluene and p-xylene. Then, solutions of internal standard with unknown solution and ethanol were made and injected into the GC. So it is concluded that the sample had no toluene in it. In Experiment 2: ethanol content in gasoline mixture was calculated using a standard curve of ethanol made with pure ethanol. Inject pure hexane to establish its identity. The amount of ethanol that was found to be present in this sample was 0.00505%. The sample is transported through the column by the flow of inert, gaseous mobile phase. Now, three injections (0.3 uL each) were made of each of these solutions into the GC. (Some industrial applications of GC include: Identification and quantification of ubiquitous pollutants in the environment: analysis of various classes of persistent organic contaminants in air, water, soils, sediments and biota GC Analysis of Antioxidants Determination of ethanol in gasoline Analysis and quality assessment of alcoholic beverages – Quantitative and qualitative assessment of Alcohols in blood Aromatics (benzene, toluene, ethylbenzene, xylene) Flavors and Fragrances Permanent gases (H_2, N_2, O_2, Ar, CO_2, CO, CH_4) Hydrocarbons Pesticides, Herbicides, PCBs, and Dioxins Solvents (20of%20gas%20chromatography.pdf) As we can see, the varied applications of GC in industry and its advantages over other methods, performing of these GC experiments is industrially justified. The peaks for both toluene and xylene could be detected by GC. 10 ml of each was diluted to 100 ml in volumetric flask. GC: Schematic diagram ( ) In Experiment 1: the ethanol content in a mouthwash was determined. The column itself contains a liquid stationary phase which is adsorbed onto the surface of an inert solid. It was allowed to stand for 5 minutes. The oven temperature was decreased from 80 degree C to 70 and then to 60 degrees C to separate the two peaks. This conclusion was based on the fact that the peaks had comparable retention times as toluene and xylene. Observations, Calculations and Results Instrument: GC SST Instrument settings: Injector temperature: 200 degree C Detector temperature: 250 degree C Oven temperature: 60 degree C (Earlier 80 degree C and 70 degree C) Attenuation: between 4 and 16 Carrier gas pressure: 8-10 psi FID range: 1 Valve: Split 1 on Solution Oven temperature (in degree C) Retention time Pure Hexane 60 0.500 Hexane + Ethanol 80 Hexane: 0.483 Ethanol: 0.683 (Less resolved peaks) Hexane + Ethanol 70 Hexane: 0.483 Ethanol: 0.750 (Better resolved peaks) Hexane + Ethanol 60 Hexane: 0.500 Ethanol: 0.916 (well resolved peaks) Solution Peak Area Standard 0.008 %, 2186.8 Standard 0.02 % 3509.9 Standard 0.04 % 5296.3 Standard 0.08 % 8746.5 Sample (1st Injection) Sample (2nd Injection) 1982.2 2138.1 Calculating % of ethanol in sample Equation of line from standard curve : y = 89994x + 1605.1 where, y = peak area, x = concentration of ethanol in % From Observation table # 3 we have, Area of sample = 1982.2 and 2138.1 Putting these values in equation in place of y we get, 1982.1 = 89994x + 1605.1 & 2138.1 = 89994x + 1605.1 On solving for x we get, x = 0.00418 % and x = 0.00592 % Averaging the two values, we get x = 0.00505 % So, the % of ethanol in the given Unknown Ethanol in Hexane Sample D is 0.00505 %. Calculating amount of xylene in sample. The separation, determination and identification of many compounds withnegligibledifferences in boilingpoints is possible by this technique. References (20of%20gas%20chromatography.pdf) Share this: Facebook Twitter Reddit LinkedIn WhatsApp In Experiment 4: the technique of headspace analysis was used to determine the volatile compounds in a contaminated soil sample. Equal volumes of ethanol and internal standard were mixed. Next, ethanol standard solution and butanol (internal standard) (each2 ml to 100 ml water) were made. After doing this, the experiment was performed. And equal volumes of sample solution and internal solution were made (each 5 ml). Let the sample rest in the vial for about 10 minutes so that the volatile components gather in the headspace of the vial. So this peak would be for xylene. Complex mixture can be resolved into its components by this GC method. The amount of ethanol present in the unknown solution of ethanol was found to be 1.00 % and the ethanol content in mouthwash was found to be 0.98%. Get Help With Your Essay If you need assistance with writing your essay, our professional essay writing service is here to help! Essay Writing Service In Experiment 3: qualitative analysis of BTEX (Benzene, Toluene, Ethylbenzene and Xylene) was performed by injecting pure solutions of these compounds in GC. And qualitative analysis like determination of ethanol content in gasoline, mouthwash, etc. Firstly, we injected pure hexane and ethanol to find out their retention times and then an equal mixture was injected to see if the two peaks can be resolved or not. Inject this to the GC. We can find its concentration by making a standard curve for xylene from the data in Observation table # 7. Make 2,4,6,8 and 10 % solutions of each of toluene and para-xylene in 50 ml volumetric flasks. The value of peak area obtained in the chromatogram for the unknown sample was substituted in the equation from standard curve to find the ethanol concentration. This was done by using an internal standard of butanol. So, using headspace analysis, Toluene and p-xylene could be identified in the soil sample. This GC method is even used industrially to find ethanol content in gasolines, plus oxygenates like butyl ether that can contaminate drinking water. Experiment 4:– Determination of volatile compounds by headspace analysis Procedure Soil was taken in a sealed vial and 1-2 drops each of toluene and p-xylene were added to it. After 10 minutes, using a syringe, suck out 0.3uL of the headspace keeping in mind that the syringe doesn't touch the soil sample itself and headspace sample is taken from just the midway of the vial. In Experiment 2: ethanol content in gasoline mixture was calculated using a standard curve of ethanol made with pure ethanol (An unknown ethanol in hexane sample was used). Stan- 1.383 Mouthwash – 0.683 (Ethanol) 3373.4 2079.5 Using the formula, Rspl RI.S. = Cspl Rstd Cstd RI.S. where, R=response (peak area); spl=sample, I.S.=internal standard C= Concentration For Unknown ethanol sample, we have, Rspl = 874.3, R I.S= 2878.8, Rstd = 1341.9, R I.S. = 2226.7 Cstd = 2 % (From observation table # 1 & 2) Putting these values in equation, 874.3 / 2878.8 / 1341.9 / 2226.7 X 2 = Cspl On solving we get, Cspl = 1.00 % For mouthwash, we have, Rspl = 2079.5 , R I.S= 3373.4, Rstd = 1341.9, R I.S. = 2226.7 Cstd = 2 % (From observation table # 1 & 2) Putting these values in equation, 2079.5 /3373.4 / 1341.9 / 2226.7 X 2 = Cspl On solving we get, Cspl = 0.98 % So, % alcohol in Mouthwash is = 0.98 % in Unknown ethanol sample = 1.00 % Experiment 2:- Determination of oxygenates in gasoline Procedure Develop a set of operating conditions that will satisfactorily separate ethanol from hexane. Stan +Unknown ethanol sample Int. The retention times obtained were recorded for each. Solution Peak Area Toluene 2 % 939.8 Toluene 4 % 1254.0 Toluene 6 % 1987.6 Toluene 8 % 2260.2 Toluene 10 % 3210.0 Para-xylene 2 % 455.8 Para-xylene 4 % 985.0 Para-xylene 6 % 1168.1 Para-xylene 8 % 1791.5 Para-xylene 10 % 2222.9 Sample Run 1 Sample Run 2 10500 (Rt = 0.733) 863 (Rt = 1.600) 2966 (Rt = 0.733) 181 (Rt = 1.600) From the sample analysis, we find that the two biggest peaks are seen at Rt = 0.733. From the chromatographs, the % of alcohol in sample were calculated. Run the individual standards and record the retention times. To conclude, we can say that we used GC for quantitative analysis like analysis of BTEX in chemicals, food, etc. The % ethanol was determined using calibration curve data and peak area data from step 5. This value neither corresponds to value of Rt for toluene (around 0.900) nor xylene (around 1.350). In the second part, standard solutions of toluene and xylene were made and calibration curves were made for each. Part B: Quantitative analysis Prepare a series of standard of toluene and para-xylene using hexane as a solvent. Next, the unknown sample of BTEX was injected to GC. They were 0.950 and 1.550. Experiment 1:- Determination of ethanol content of a mouthwash using an internal standard Procedure The instrument was set to the following parameters: Injector temperature: 200 degree C Detector temperature: 250 degree C Oven temperature: 80 degree C Attenuation: between 4 and 16 Carrier gas pressure: 8-10 psi FID range: 1 Valve: Split 1 on Dilute the unknown ethanol sample and mouthwash provided with water in a 1:10 ratio. Concentration, 0.2 ml = 0.2/25 = 0.008 %, 0.5 ml = 0.5/25 = 0.02 % 1.0 ml = 1.0/25 = 0.04 % 2.0 ml = 2.0/25 = 0.08 % These standards were injected into GC and a calibration curve was prepared using the peak area data obtained. Analyse an unknown sample of BTEX provided and find the % of toluene and para-xylene in it using the standard curve data. The gasoline mixture was extracted with water to extract the ethanol in it for further analysis by GC. The retention areas obtained were recorded and substituted in equations of standard curves to find the actual content of toluene and xylene in sample. The soil sample was also deliberately contaminated with toluene and xylene and placed in a vial for 10 minutes. This technique involves a sample being vapourised and injected onto the head of the chromatographic column. The water layer was taken using a Pasteur pipette and injected into GC. In Experiment 1: the ethanol content in a mouthwash was determined. The areas obtained for each compound were then used to calculate the % alcohol in the sample. Standard solutions of toluene and xylene were made and calibration curves were made for each. Stan- 1.383 Unknown ethanol Sample- 0.683 (Ethanol) 2878.8 874.3 Int. From the standard curve (Graph # 2), we have the equation of line y = 217.04x + 22.45 where, y = peak area, x = concentration of ethanol in % From Observation table # 7 we have, Area of sample = 863 and 181 Putting these values in equation in place of y we get, 863 = 217.04x + 22.45 & 181 = 217.04x + 22.45 On solving for x we get, x = 3.87 % and x = 0.73 % Averaging the two values, we get x = 2.30 % Therefore, the sample (Unknown B) contains 2.30 % of xylene and no toluene. In Experiment 3: qualitative analysis of BTEX (Benzene, Toluene, Ethylbenzene and Xylene) was performed by injecting pure solutions of these compounds in GC. Observations, Calculations and Results Instrument: GC SST Instrument settings: Injector temperature: 200 degree C Detector temperature: 250 degree C Oven temperature: 80 degree C Attenuation: between 4 and 16 Carrier gas pressure: 8-10 psi FID range: 1 Valve: Split 1 on Solution Retention time Peak area Butanol 1.400 5452.57 Ethanol 0.700 3607.3 Butanol (I.S.) + Ethanol 1.400-Butanol (I.S) 0.700-Ethanol 2226.7 1341.9 Solution Retention time Peak area Int. Prepare ethanol standards: 0.2 ml, 0.5 ml, 1.0 ml and 2.0 ml in 25 ml DI water. Perform duplicate injections if both components can not be seen at one go. The GC method is used industrially to find ethanol content in gasolines, plus oxygenates like butyl ether that can contaminate drinking water.. Observations, Calculations and Results Instrument: GC SST Instrument settings: Injector temperature: 200 degree C Detector temperature: 250 degree C Oven temperature: 80 degree C Attenuation: between 4 and 16 Carrier gas pressure: 8-10 psi FID range: 1 Solution Retention time Chlorobenzene 3.866 Ethylbenzene 2.016 p-xylene 1.400 o-xylene 4.133 Toluene 1.500 From individual injections of hexane, toluene and xylene, it was seen that that the retention times for each of these were 0.500, 0.933 and 1.550 respectively. The unknown gasoline sample (Unknown Sample D: Ethanol in hexane) was taken and 5.0 of it was transferred to a vial. or BTEX in soil. No peaks were seen at this retention times even on duplicate runs of the sample. Now with the equation of calibration curve and the peak area of sample for xylene, the concentration of xylene present in the sample was calculated. The calculations for determining the % of ethanol were based on equation that was derived from the standard curve from ethanol standards. One can detect upto 100 ppm, while flame detectors, electron capture and phosphorus detectors can detect ppm, parts per billion or picograms respectively. Experiment 3:- Qualitative and Quantitative analysis of BTEX (Benzene, Toluene, Ethylbenzene and Xylene) Procedure Part A: Qualitative Analysis Take 1 ml each of Benzene, Toluene, Ethylbenzene and Xylene in separate vials. So on comparing these Rts with the Rts obtained in these chromatograms, we can identify the peaks as toluene or xylene as done in Observation Table # 8. A standard curve was made from the data obtained by running xylene standards. It was found to contain 2.30 % xylene. Then using a syringe, the headspace was sucked and injected to GC for performing a qualitative analysis (the retention times of BTEX from Experiment 3 were used). Prepare a calibration curve based on the peaks area data obtained. In the second part, quantitative analysis of toluene and p-xylene was done. Inject this mixture into GC and ensure two resolved peaks. 5.0 ml of water was added and mixed thoroughly in the vial. It was found that the unknown sample had not retention that matched with the retention times of toluene which was close to 0.9550.

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