**Exercise #2: GC calculations and predictions**

 *Chem 6614 Chemical Instrumentation*

**TABLE OF RUN CONDITIONS:**

Tinitial = 50 oC, thold = 0

Temperature ramp = 20o C/min

Tfinal = 160 0C, thold= 1 minute

Tinlet = 300 oC

Column = HP-1 (`OV-1` non-polar siloxane phase 10 m, 350oC MAOT ) silica capillary (0.2 mm ID,0.33 µ)

Split ratio: 1:100

He column flow = 0.9 mL/min

FID detector: P(H2) = 40 psi, P(air) =75 psi

Sampling method: wet needle

Reference mixture 1: 33.3 % octane (125.6 oC) 33.3% heptane (98.5oC ) 33.3% hexane (68.7o C) v/v Reference mixture 2: 33.3% acetone (56 oC ) 33.3 % methyl ethyl ketone(79.6oC) cyclohexanone(155.6 oC)

**TABLE OF COLLECTED RUN DATA:**

**Chromatogram #R1: Known reference mixture data for alkanes**

Component tr,obs(min) Area (pA\*s)

Hexane 0.65 40.0

Heptane 0.90 65.1

Octane 1.10 98.2

**Chromatogram #R2: Known reference mixture data for ketones**

acetone 0.80 55.0

MEK 1.40 76.4

cyclohexanone 3.70 90.5

**Chromatogram A: unknown mixture of above components**

tr, obs(min) Area (pA\*s)

0.65 90.6

1.11 60.0

1.39 11.4

**2.1 Peak Assignment of Chromatogram A Unknown Mixture**

**Use Chromatographic data from R1 and R2 to assign the compounds appearing in Chromatogram A of the Unknown Mixture**

probable compound in A tr, obs(min) Area (pA\*s)

0.66 90.6

1.12 60.0

1.39 trace peak (no area computed by Instrument)

**2.2. Estimating Run behavior from Run conditions**

a) what is the total volume of the column ? \_\_\_\_ mL

b) how long will a single run minimally take from the time of injection to completion of the GC

 program cycle described in the run condition table above ?

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ min

c) given the flow rate for He, use your answer above to compute the time it takes for a `plate’ of He to traverse the column (= minimum retention time=dead or `void’ time…c.f. p. 766 of text)

 \_\_\_\_\_ = tmin = tdead volume (min)

d) Based on just temperature ramp, initial column temperature and the boiling points of the components, what do you estimate the theoretical retention times, tr theory should be for hexane, heptane and octane ?

Component tr obs(min) tr,theory(min)

Hexane 0.63

Heptane 0.92

Octane 1.15

Suggest a reason for why the theoretical retention times may not match observed.

**2.3. Estimating Unknown Mixture composition if only 1 column analysis required**

a) Assuming no changes in sensitivity or run character between unknown and known, compute the apparent sensitivity, Sk of the components in pA\*s/% using the known mixture data below

**Analysis of Reference Mixture R1 (hexane, octane) and R2 (MEK)**

Component Area (pA\*s) %V Sk = Area/%V

Hexane 40.0 33 40.0/33.0=1.21

Octane 98.2 33 U-Do-It

MEK 76.4 33

b) Given the Sk above, we can solve for the unknown %Vk unk (raw).For example, for hexane in unknown:

 Shexane = observed Area of hexane in unknown

 %Vhexane unk(raw)

 1.21 = 90.6

%V1 unk(raw)

%Vhexane unk (raw) = 90.6/1.21=74.97~75%

Finish the calculations for the other component with measurable Area (pA\*s) below

Component A=Area (pA\*s) Sk=A/33.3%Vk(raw)

Hexane 90.6 1.21 75

Octane 60.0 U-Do-It

MEK 76.4

c) Add up the %Vk(raw). They normally do not sum exactly to 100%. Rescale them to 100 as shown below: %Vk(final) = %Vk(raw) \* 100 and tabulate below.

 Σ%Vk(raw)

Component %Vk(raw) %Vk(rescaled)

Hexane 75

Octane

MEK

Σ%Vk(raw)

2.4.  **Estimating Unknown Mixture composition if 2 column analysis required**

**Chromatogram B: unknown mixture of above components**

Component tr, obs(min) Area (pA\*s) GC

hexane 0.65 50.0 non-polar instrument 1

octane 1.16 90.0 non-polar instrument 1

cyclohexanol 5.69 no area recorded polar instrument 2

 but signal observed

Here, you assume the hexanol is a minor component (e.g. probably less than 5% of the mixture) and compute the % hexane and octane as if they are the only major compounds present. You would report these and indicate that a trace (<5%) v% of MEK is present based on peak appearance, as is done below:

%Vhexane unk (raw) = 50.0/1.21=41.3

%Voctane unk (raw) = 90.0/1.80=50.0

Rescaled and reported as:

%Vhexane, rescaled ~ 100\*41.3/(41.3+50.0) ~45.2

%Voctane, rescaled ~ 100\*50.0/(41.3 +50.0) ~54.8

trace cyclohexanol (weak peak at 5.70 min)