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| Chem 216 |
| Analysis of the Components in an Over-the-Counter Analgesic Drug Using Ultraviolet Spectroscopy and HPLC |
| Lab Report 4 |
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| **4/19/2010** |

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# Purpose:

The purpose of this experiment is to quantitatively determine the composition of caffeine, acetaminophen, and salicylic acid in a type of Solpadine and aspirin tablets mixed together, using two methods; HPLC and UV spectroscopy.

# Procedure:

Refer to the Lab manual

# Data:

## UV:

### Stock solution preparation:

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Stock name | Mass added (mg) | Vdissolved  (ml) | aliqoute volume (ml) | Vdilution (ml) |
| Caffeine | 14.6 | 100 | 2 | 100 |
| Acetaminophen | 13.9 | 100 | 2 | 100 |
| Salicylic acid | 15.6 | 100 | 2 | 100 |
| Phenacetine | 34.6 | 100 | - | - |
| Aspirin tablet | 649.7 | 500 | - | - |
| Solpadein tablet | 136.0 |

Table the masses of standards and unknowns added to the solutions and the dilutions that occurred.

### UV spectra:

Figure Salycilic acid standard absorbance spectrum

Figure 2 Acetaminophen standard Absorbance spectrum

Figure Caffeine standard Absorbance Spectrum

Figure the absorbance spectrum of solution containing solpadein and aspirin Trial 1

Figure the absorbance spectrum of the solpadein aspirin solution trial 2

## HPLC:

### Unknown solution:

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|   | Area Run 1 (254nm) | Area Run 2 (254nm) | average | Area (225nm) |
| Acetaminophene | 2672162 | 919516 | 1795839 | 7440739 |
| Caffeine | 878774 | 84049 | 481411.5 | 222546 |
| Aspirin | 118708 | 11237 | 64972.5 | 981674 |
| Phenacetin | 6195014 | 582047 | 3388530.5 | 2828388 |

Table HPLC data of unknown

### Standard solution:

|  |  |  |  |
| --- | --- | --- | --- |
|   | Area Run 1 | Area Run 2 | average |
| Acetaminophene | 14481978 | 3272372 | 8877175 |
| Caffeine | 181278 | 38665 | 109971.5 |
| Aspirin | 1468 | 21733 | 11600.5 |
| Phenacetin | 6096440 | 1400607 | 3748524 |

Table HPLC data of standards

# Calculation:

## Molarity of each stock solution:

### Sample calculation:

[Caffeine] = $\frac{m (mg)}{V\left(total\right)(l)}×\frac{V alliquot(ml)}{V\left(dilution\right) ml}=\frac{14.6mg}{0.100l} ×\frac{2mL}{100mL}= 2.92ppm$

Similarly,

|  |  |
| --- | --- |
|  | Concentration(ppm) |
| Caffeine | 2.92 |
| Salicylic acid | 3.12 |
| Acetaminophen | 2.78 |

## Absorptivity calculation:

Modified Beer-Lambert Law: **A**λ = **a**λ(1/ppm.cm) **b**(cm) **c’**(ppm)

At 201nm for Salycilic acid, a = $\frac{A(201nm)}{B\left(cm\right).c^{'}(ppm)}= \frac{0.61487}{1 ×3.12}= 0.19707$

|  |  |  |  |
| --- | --- | --- | --- |
| Standard name | wavelength (nm) | Absorbance | a (absorptivity) |
| Salicylic acid (3.12ppm) | 206 | 0.52452 | 0.16812 |
| 205 | 0.57633 | 0.18472 |
| 204 | 0.61289 | 0.19644 |
| 203 | 0.63078 | 0.20217 |
| 202 | 0.62996 | 0.20191 |
| 201 | 0.61487 | 0.19707 |
| Acetaminophene (2.78 ppm) | 206 | 0.19298 | 0.06942 |
| 205 | 0.22586 | 0.08124 |
| 204 | 0.26157 | 0.09409 |
| 203 | 0.29501 | 0.10612 |
| 202 | 0.32545 | 0.11707 |
| 201 | 0.35241 | 0.12677 |
| Caffeine (2.92 ppm) | 206 | 0.18605 | 0.06372 |
| 205 | 0.1779 | 0.06092 |
| 204 | 0.166 | 0.05685 |
| 203 | 0.14961 | 0.05124 |
| 202 | 0.12876 | 0.04410 |
| 201 | 0.10652 | 0.03648 |

Table Absorbance at six specific wavelengths

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | wavelength (nm) | Absorbance (trial 1) | Absorbance (trial 2) | Average |
| Dilute solpadeine and aspirin tablets | 206 | 0.18334 | 0.18657 | 0.184955 |
| 205 | 0.23428 | 0.23109 | 0.232685 |
| 204 | 0.28599 | 0.27855 | 0.28227 |
| 203 | 0.3391 | 0.32596 | 0.33253 |
| 202 | 0.39076 | 0.37578 | 0.38327 |
| 201 | 0.43771 | 0.41709 | 0.4274 |

A(α) = a(Ac, α)bCAc + a(As, α)bCAs+ a(Cf, α)bCCf

A(β) = a(Ac, β)bCAc + a(As, β)bCAs+ a(Cf, β)bCCf

A(γ) = a(Ac, γ)bCAc + a(As, γ)bCAs+ a(Cf, γ)bCCf

Wrong: in yellow

Solving this system of three equations and three unknowns such that α, β, and γ correspond to different wavelengths, we get:

CAc =2.25 ppm, CCf=2.21 ppm, CAs=1.00ppm

* Ccf in Solp-Asp=[(Ccf used in UVx0.1)/0.01]=22.1ppm and m Cf = 22.1\*0.5 =11.05mg

%Cfin the tablet mix= (11.05/785.7)\*100=1.41%

* CAc in Solp-Asp=[(CAc used in UVx0.1)/0.01]=22.5ppm and m Ac = 22.5 \*0.5 =11.25mg

%Acin the tablet mix= (11.25/785.7)\*100=1.43%

* CAs in Solp-Asp=[(CAs used in UVx0.1)/0.01]=10.0ppm and m As = 10.0\*0.5 =5.00 mg

%Asin the tablet mix= (5.00/785.7)\*100=0.64%

## HPLC:

* Cphenacetine ­ standard=34.6/0.1=346ppm

Cphenacetine in unknown=346ppm\*20/100=69.2ppm

* Detector responsesalicylic acid =Asalicylic acid/Aphenacitine =Csalicylic acidn/Cphenacitine = 0.019

Csalicylic acid=detector response\*Cphenacetine=0. 019\*69.2=1.32ppm

m salicylic acid = 1.32\*0.1 = 0.132mg

 %salicylic acidin the tablet mix= (0.132/785.7)\*100=0.017%

* Detector responsecaffeine =ACaffiene/Aphenacitine =CCaffiene/Cphenacitine= 0.142

 Ccaffeine = detector response\* Cphenacetine=0.142\*69.2 = 9.83ppm

m Caff = 9.83\*0.1 = 0.983mg

%Caffin the tablet mix= (0.983/785.7)\*100=0.125%

* Detector responseacetaminophen =Aacetaminophen/Aphenacitine =Cacetaminophen/Cphenacitine= 0.53

 Cacetaminophen = detector response\* Cphenacetine=0.53\*69.2 = 36.7ppm

m Ac = 10.3\*0.1 = 3.67mg

 %Acin the tablet mix= (3.67/785.7)\*100=0.47%

# Results:

## By UV:

Salycilic acid: 5.00 mg, 0.64%

Acetaminophen: 16.85 mg, 1.43%

Caffeine: 11.058mg, 1.41%

## By HPLC:

Salicylic acid: 0.132mg, 0.017%

Acetaminophen: 3.67mg, 0.47%

Caffeine: 0.983mg, 0.125%

# Conclusion:

There are major discrepancies between the two methods for the unknowns present in solution. It must be noted that we found it quite odd that a compound could give a negative absorbance, which is one of the reasons why we considered the HPLC more a more reliable instrument for quantization of the unknowns in the sample.