

UYGULAMA NOTU

Gaz Kromatografi

G006

GC-HS-FID ile Kanda Alkol Analizi

HAZIRLAYANLAR

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KONU:

Headspace yöntemiyle gaz kromatografide “Kanda Alkol Analizi”.

ÇALIŞMANIN AMACI:

Özellikle adli vakalarda kişilerin kanında bulunan alkol ve yabancı madde miktarının promil cinsinden hesabı yapılmalıdır.

METOD ve MATERYALLER:

Bu çalışmamızda Shimadzu GC-2010 Plus Gaz Kromatografi sistemiyle kanda alkol tayini yapılmıştır. Uygulama Headspace gaz örnekleme ünitesi ve FID (Alev İyonlaştırma Dedektörü) kullanılarak gerçekleştirilmiştir.

Headspace sistemleri, GC ve GCMS sistemleri ile gerçekleştirilen VOC, bakiye solvent, aroma, koku, alkol gibi uçucu bileşik analizleri için ideal çözümlerdir.



NUMUNE HAZIRLAMA:

2 g örnek tartılır. 20 mL MeOH ilave edilir. 2000 rpm ‘de ve 20 C ‘de santrifüjlendikten sonra üst faz şırınga ucu filtreden geçirilerek cihaza enjeksiyon yapılır.

ANALİTİK KOŞULLAR:

GC için:

Instrument Parameter

Select Line Line1 Line2

SPL1 | Column | FID1 | General | Add. Flow

Temperature : 40.0 C

Injection Mode : Split

Sampling Time : 1.00 min

Carrier Gas : He

Flow Control Mode : Linear Velocity

Pressure : 80.1 kPa

Total Flow : 50.0 mL/min

Column Flow : 2.57 mL/min

Linear Velocity : 40.0 cm/sec

Purge Flow : 3.0 mL/min

Split Ratio : -1.0

INJ.Program Purge Flow Redraw

	Rate	Flow	Hold Time
0	-	3.0	0.00
1	0.00	0.0	0.00
>	0.00	0.0	0.00

Total Program Time : 0.00 min

Advanced...

Column Information (RTx-BAC Plus)

Length : 30.0 m Film Thickness : 1.80 um

Inner Diameter : 0.32 mm ID

Tamam iptal Uygula Yardım

Instrument Parameter

Select Line Line1 Line2

SPL1 | Column | FID1 | General | Add. Flow

Temperature : 40.0 C

Equilibration Time : 0.5 min

Column Information (RTx-BAC Plus)

Serial Number : 18004

Installation Date : 17/03/15

Column Max. Temp : 240 C

Length : 30.0 m

Inner Diameter : 0.32 mm ID

Film Thickness : 1.80 um

Description :

Column Oven Temperature Program Redraw

	Rate	Temperature	Hold Time
0	-	40.0	5.00
1	0.00	0.0	0.00
>	0.00	0.0	0.00

Total Program Time : 5.00 min

Tamam iptal Uygula Yardım

Instrument Parameter

Select Line Line1 Line2

SPL1 | Column | FID1 | General | Add. Flow

Temperature : 240.0 C

Signal Acquire

Sampling Rate : 40 msec

Stop Time : 5.00 min

Delay Time : 0.00 min

Subtract Detector : None

Flow Program Makeup Redraw

	Rate	Flow	Hold Time
0	-	30.0	0.00
1	0.00	0.0	0.00
2	0.00	0.0	0.00

Total Program Time : 0.00 min

Makeup Gas : He H2 Flow : 40.0 mL/min

Makeup Flow : 30.0 mL/min Air Flow : 400.0 mL/min

Tamam iptal Uygula Yardım

Instrument Parameter

Select Line Line1 Line2

SPL1 | Column | FID1 | General | Add. Flow

APC1 Pressure : 50.0 kPa

APC2 Pressure : 0.0 kPa

APC3 Pressure : 0.0 kPa

APC Program APC1 Redraw

	Rate	Pressure	Hold Time
0	-	50.0	0.00
1	0.00	0.0	0.00
2	0.00	0.0	0.00

Total Program Time : 0.00 min

Tamam iptal Uygula Yardım

Headspace İçin:

kanda alkol.hsm (Update) - HS-20 Method Editor

File Settings Help

Oven Temp.: 65.0 C Equilibrating Time: 15.00 min
Sample Line Temp.: 150.0 C Pressurizing Time: 0.50 min
Transfer Line Temp.: 150.0 C Pressure Equilib. Time: 0.10 min
Load Time: 0.50 min
Load Equilibrating Time: 0.10 min
Injection Time: 0.50 min
Needle Flush Time: 0.00 min
GC Cycle Time: 6.00 min

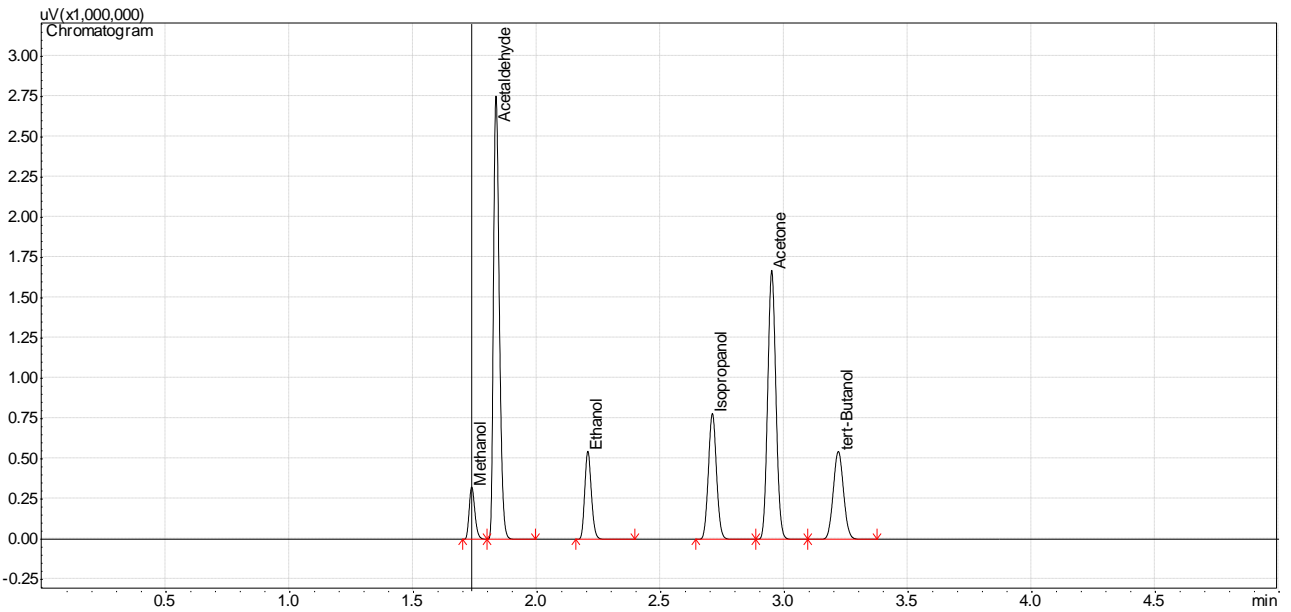
Shaking Level: 2
Multi Injection Count: 1

Comment:
Kanda Alkol Metodu

Ready NUM

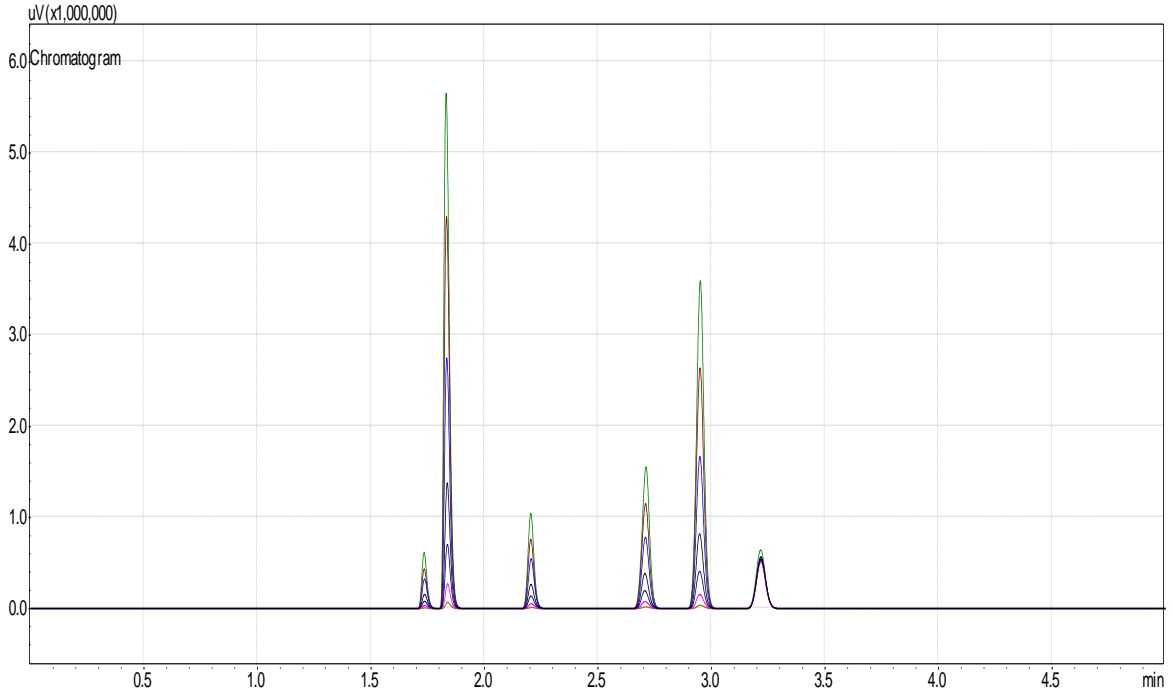
Kromatogramlar:

Standart analizi örnek kromatogramı



Standart analizi örnek kromatogramı - karşılaştırmalı

Not: 1 promil = 1000 ppm



Data1:1000 ppm std mix_1.gcd (CH1)
Data5:4000 ppm std mix_1.gcd (CH1)

Data2:200 ppm std mix_1.gcd (CH1)
Data6:500 ppm std mix_1.gcd (CH1)

Data3:2000 ppm std mix_2.gcd (CH1)
Data7:50 ppm std mix_1.gcd (CH1)

Data4:3000 ppm std mix_1.gcd (CH1)

Kalibrasyon Eğrileri:

Not: İç Standart (ter-Butanol) Değerlendirme Metodu Kullanılmıştır

ID#:1 Name:Methanol

$f(x)=0,126512279526*x+0,0$

$R=0,999452661629$ $R^2=0,998905622837$

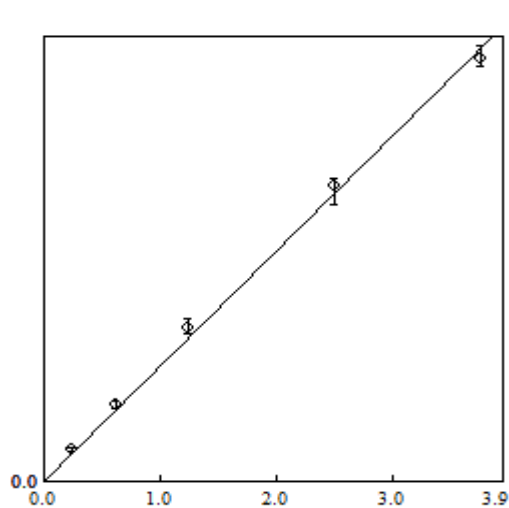
MeanRF:0,133367888245 RFS:8,96572780502e-003 RFRSD:6,7225536244

CurveType:Linear

ZeroThrough:Weight on Origin

WeightedRegression:None

Internal Standard



No.	Conc.	Area
1	0,250	0
2	0,625	0
3	1,250	0
4	2,500	0
5	3,750	0

ID#:1 Name:Methanol

$$f(x)=0,126512279526*x+0,0$$

R=0,999452661629 R²=0,998905622837

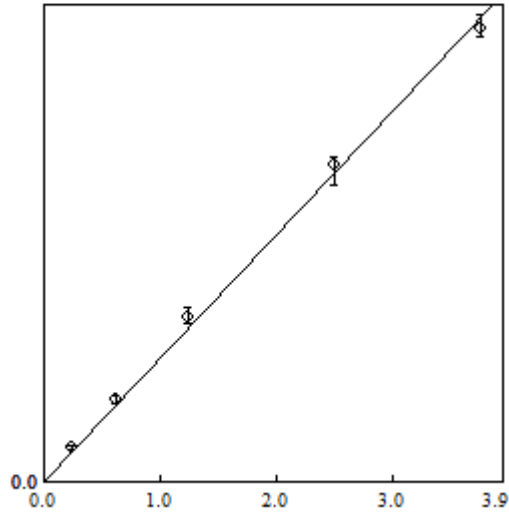
MeanRF:0,133367888245 RFSD:8,96572780502e-003 RFRSD:6,7225536244

CurveType:Linear

ZeroThrough:Weight on Origin

WeightedRegression:None

Internal Standard



No.	Conc.	Area
1	0,250	0
2	0,625	0
3	1,250	0
4	2,500	0
5	3,750	0

ID#:2 Name:Acetaldehyde

$$f(x)=1,19399208665*x+0,0$$

R=0,998893343872 R²=0,997787912431

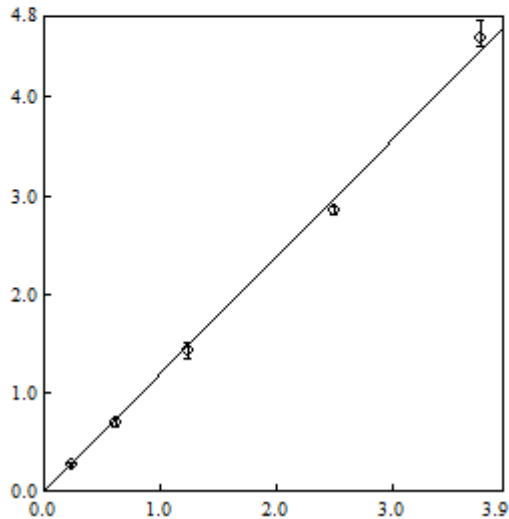
MeanRF:1,1400085099 RFSD:7,02277063259e-002 RFRSD:6,16027913091

CurveType:Linear

ZeroThrough:Weight on Origin

WeightedRegression:None

Internal Standard

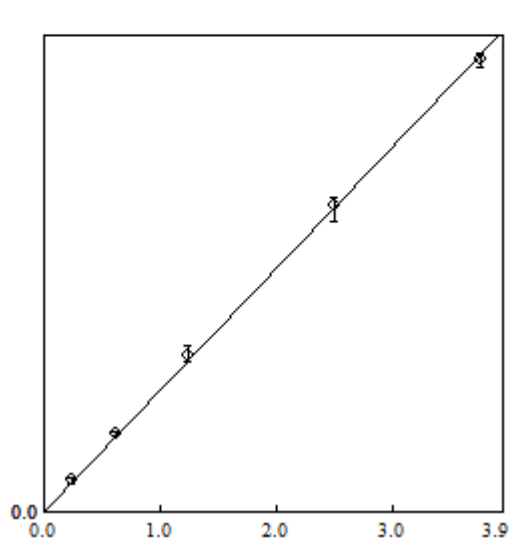


No.	Conc.	Area
1	0,250	0
2	0,625	1
3	1,250	1
4	2,500	3
5	3,750	5

ID#3 Name:Ethanol

$f(x)=0,250078893865*x+0,0$
 $R=0,999923504902$ $R^2=0,999847015656$
MeanRF:0,256550652744 RFSD:1,26021314357e-002 RFRSD:4,9121416379
CurveType:Linear
ZeroThrough:Weight on Origin
WeightedRegression:None

Internal Standard

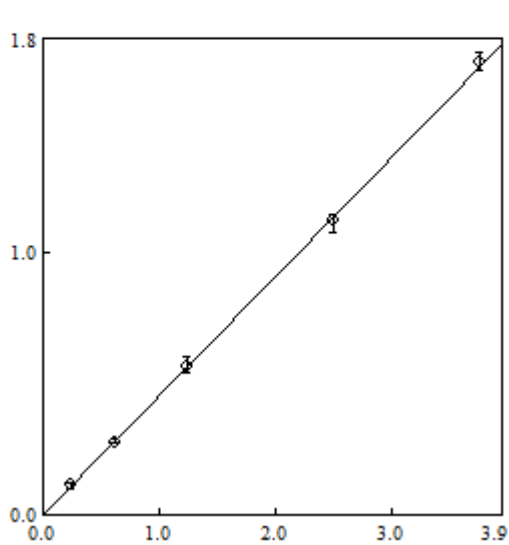


No.	Conc.	Area
1	0,250	0
2	0,625	0
3	1,250	0
4	2,500	1
5	3,750	1

ID#4 Name:Isopropanol

$f(x)=0,454047450188*x+0,0$
 $R=0,999831089441$ $R^2=0,999662207413$
MeanRF:0,451988531496 RFSD:1,88998239855e-002 RFRSD:4,18148308386
CurveType:Linear
ZeroThrough:Weight on Origin
WeightedRegression:None

Internal Standard



No.	Conc.	Area
1	0,250	0
2	0,625	0
3	1,250	1
4	2,500	1
5	3,750	2

ID#:5 Name:Acetone

$$f(x)=1,01157911992*x+0,0$$

R=0,999069398788 R^2=0,998139663594

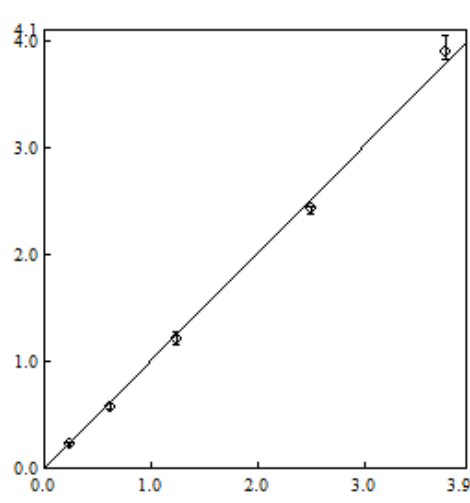
MeanRF:0,961822499943 RFSD:6,1524317825e-002 RFRSD:6,39663948688

CurveType:Linear

ZeroThrough:Weight on Origin

WeightedRegression:None

Internal Standard



No.	Conc.	Area
1	0,250	0
2	0,625	1
3	1,250	1
4	2,500	2
5	3,750	4

ID#:6 Name:tert-Butanol

$$f(x)=0,0*x+0,0$$

R=0,0 R^2=0,0

MeanRF:0,0 RFSD:-- RFRSD:--

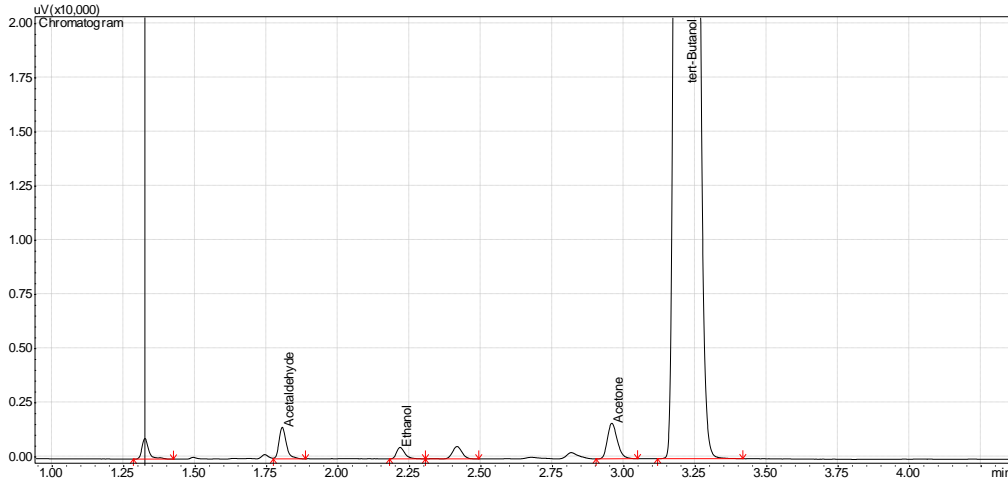
CurveType:Linear

ZeroThrough:Weight on Origin

WeightedRegression:None

Internal Standard

Numune Analizi Örnek Kromotogramı



Çalışmalarımız **DİCLE ÜNİ. MERKEZ ARAŞTIRMA LABORATUVARI**'nda gerçekleştirilmiştir.

Desteklerinden dolayı Sn. Mustafa Abdullah Yılmaz'a teşekkürlerimizi sunarız.

Bu Aplikasyon Notu uygulamanın gerçekleştirildiği tarihe ait bilgiler ışığında oluşturulmuştur.

Bu yayında yer alan bilgilerin referans gösterilerek başka bir yerde kullanılması Ant Teknik'in iznine tabidir.

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