

UYGULAMA NOTU

İyon Kromatografi

I001

Dual IC ile Patlayıcılarda İyon Tayini

HAZIRLAYAN

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Ant Teknik Cihazlar Ltd. Şti.

KONU:

Patlayıcı Maddelerde Anyon ve Katyon Eşzamanlı Tayini

ÇALIŞMANIN AMACI:

Adli olaylarda meydana gelen patlamalarının sebebinin doğal ya da illegal yollardan oluşumunu takip edebilecek ve buna sebebiyet verecek kimyasal bileşimin kantitatif tayinini yapabilmektir.

METOD ve MATERYALLER:

Çalışma Shimadzu marka Dual Ion Chromatography cihazıyla yapılmıştır.

Kullanılan Standart Türleri:

Merck firmasının ürettiği kimyasal iyon standartları kullanılmıştır.

Ekstraksiyon Yöntemi:

Örnekler yağlı ise ilk olarak Aseton ekstrakte edilip, su ile hacme tamamlanmıştır. Suda çözünen bileşikler hacme direkt bu çözgen ile tamamlanıp; 0,45um naylon filtreden süzülerek cihaza enjeksiyon yapılır.

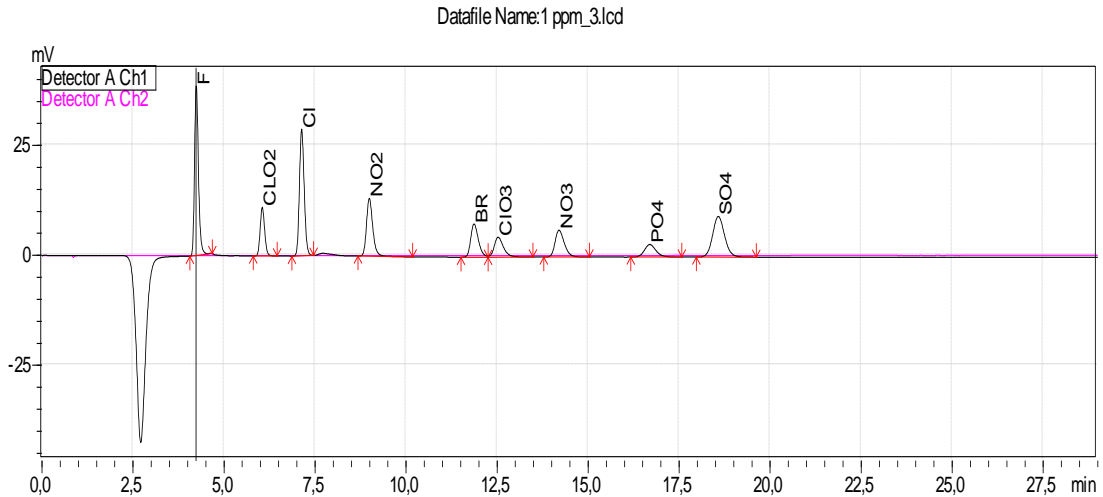
Hesaplama: Numune değerlendirmesi ilk olarak kalitatif olarak ve ardından dış standart - kalibrasyon eğrisi metoduyla kantitatif olarak tayini gerçekleştirilmiştir.

ANALİTİK KOŞULLAR:

| | | | |
|-----------------------|--|-----------|-------------|
| Cihaz | : HIC-20A Dual IC | Akış Hızı | : 1 mL/dk |
| Kolonlar | : IC-SA3 ve IC-C4 (250 mm x 4.6 mm x 0.25 um) | Dedektör | : CDD-10Asp |
| Kolon Fırın Sıcaklığı | : 40 °C | Süre | : 40 dk |
| Enjeksiyon Hacmi | : 40 uL | | |

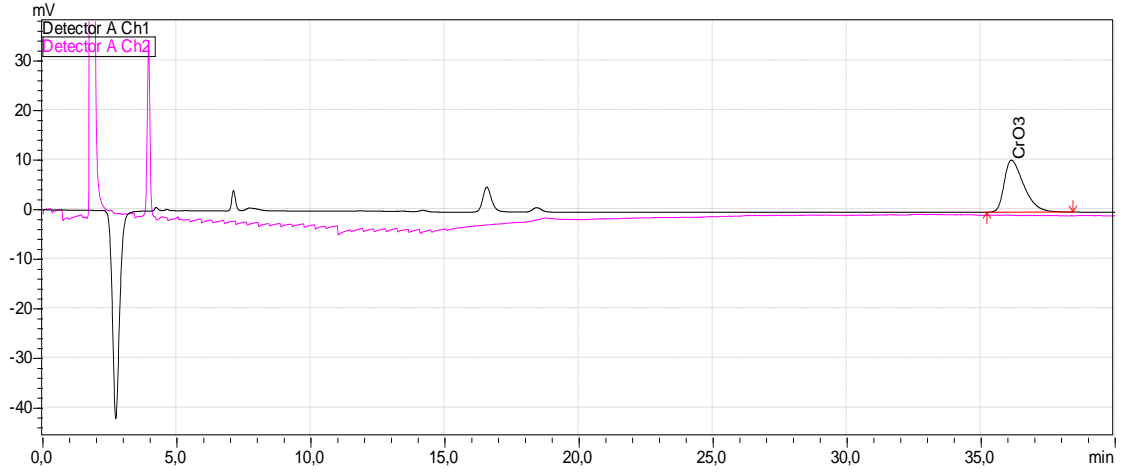
SONUÇLAR ve GÖRÜŞLER

1. Anyon Standart Karışımı



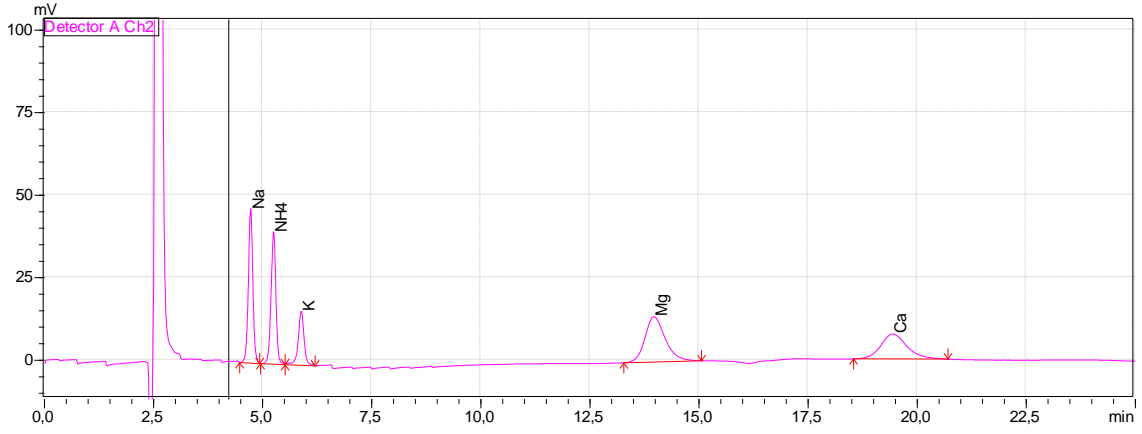
2. Kromat Standart Karışımı

Datafile Name:10 ppm kr_3.lcd
Sample Name:10 ppm kr_1.lcd



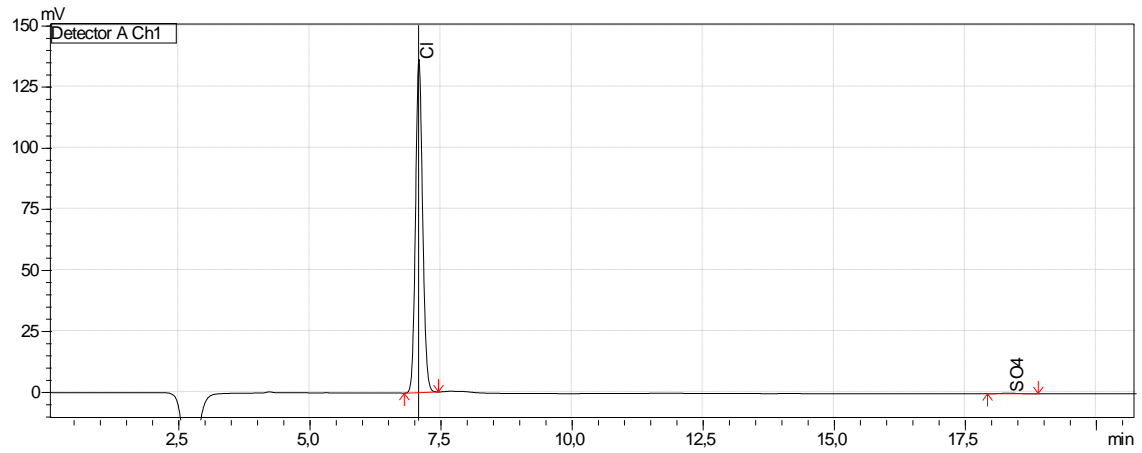
3. Katyon Standart Karışımı

Datafile Name:20 ppm katyon_2.lcd
Sample Name:20 ppm katyon_2.lcd



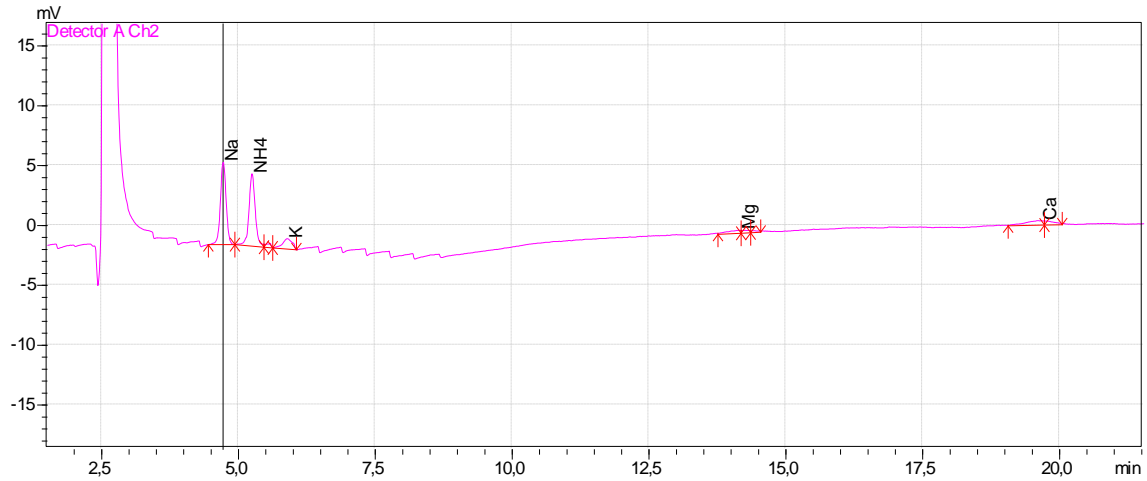
4. Numune (Anyon Sonucu)

Datafile Name:NH4Cl.lcd



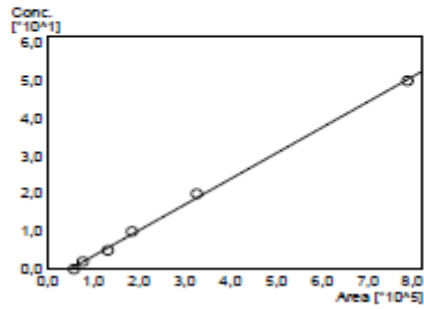
Numune (Kasyon Sonucu)

Datafile Name:NH4Cl.lcd



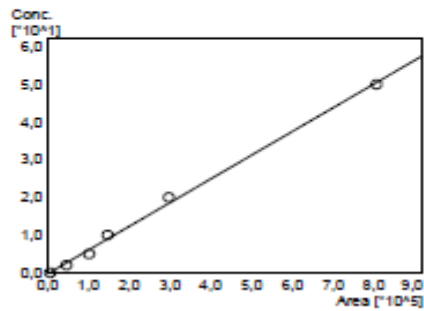
5. Kasyon için Kalibrasyon Eğrileri

ID# : 1
 Name : Na
 Quantitative Method : External Standard
 Function : $f(x)=6,83721e-005*x+3,33654$
 $Rr1=0,9992723$ $Rr2=0,9985451$ $RSB=2,571478e+000$
 MeanRF: 4,567229e-005 RFSO: 1,943621e-005 RFRSD: 42,555796
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



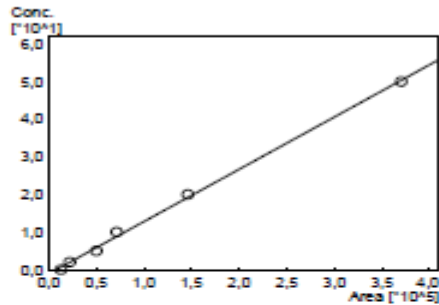
| # | Conc. (Ratio) | MeanArea | Area |
|---|---------------|----------|--------|
| 1 | 0 | 57263 | 57263 |
| 2 | 2 | 77526 | 75475 |
| | | | 78554 |
| | | | 78883 |
| 3 | 5 | 131583 | 127735 |
| | | | 134105 |
| | | | 134105 |
| 4 | 10 | 184951 | 181280 |
| | | | 184735 |
| | | | 184735 |
| 5 | 20 | 326354 | 338757 |
| | | | 338757 |
| | | | 301559 |
| 6 | 50 | 786354 | 669129 |
| | | | 632304 |
| | | | 657629 |

ID# : 2
 Name : NH4
 Quantitative Method : External Standard
 Function : $f(x)=6,30624e-005*x+0,299031$
 $Rr1=0,9984605$ $Rr2=0,9969236$ $RSB=5,437521e+000$
 MeanRF: 5,441027e-005 RFSO: 1,838499e-005 RFRSD: 33,789549
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



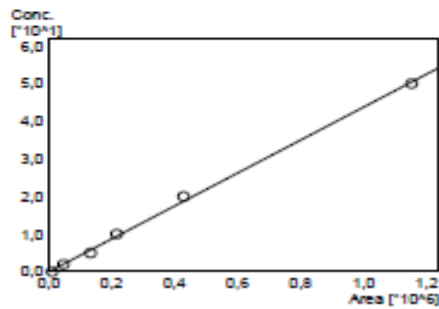
| # | Conc. (Ratio) | MeanArea | Area |
|---|---------------|----------|--------|
| 1 | 0 | 7212 | 7212 |
| 2 | 2 | 46341 | 50035 |
| | | | 42927 |
| | | | 47851 |
| 3 | 5 | 102297 | 96380 |
| | | | 102455 |
| | | | 105255 |
| 4 | 10 | 147384 | 157280 |
| | | | 147435 |
| | | | 147435 |
| 5 | 20 | 297707 | 312185 |
| | | | 312185 |
| | | | 268751 |
| 6 | 50 | 806495 | 674005 |
| | | | 863118 |
| | | | 894457 |

ID# : 3
 Name : K
 Quantitative Method : External Standard
 Function : $f(x)=0,000139097^*x-1,12248$
 $Rr1=0,9990632$ $Rr2=0,9981272$ $RSS=3,310141e+000$
 MeanRF: 1,132049e-004 RFSD: 3,827078e-005 RFRSD: 33,806644
 FITType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



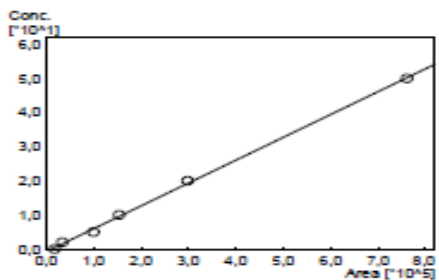
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|--------|
| 1 | 0 | 13030 | 13030 |
| 2 | 2 | 22387 | 19830 |
| | | | 23151 |
| 3 | 5 | 50209 | 48070 |
| | | | 51178 |
| | | | 51578 |
| 4 | 10 | 71655 | 78150 |
| | | | 68423 |
| | | | 68423 |
| 5 | 20 | 146251 | 153931 |
| | | | 153931 |
| | | | 150881 |
| 6 | 50 | 370340 | 318332 |
| | | | 384418 |
| | | | 388172 |

ID# : 4
 Name : Mg
 Quantitative Method : External Standard
 Function : $f(x)=4,38266e-005^*x+0,0501234$
 $Rr1=0,9991571$ $Rr2=0,9983149$ $RSS=2,978452e+000$
 MeanRF: 4,085586e-005 RFSD: 1,237932e-005 RFRSD: 30,299982
 FITType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 1 | 0 | 11874 | 11874 |
| 2 | 2 | 47859 | 36148 |
| | | | 46786 |
| 3 | 5 | 132859 | 60675 |
| | | | 146999 |
| | | | 131449 |
| 4 | 10 | 215757 | 137420 |
| | | | 215777 |
| | | | 215348 |
| | | | 215358 |
| 5 | 20 | 428926 | 444726 |
| | | | 444726 |
| | | | 397371 |
| 6 | 50 | 1152673 | 895801 |
| | | | 1234508 |
| | | | 1243711 |

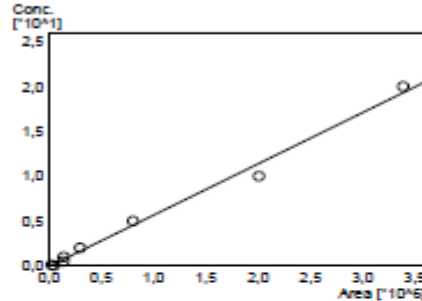
ID# : 5
 Name : Ca
 Quantitative Method : External Standard
 Function : $f(x)=6,70703e-005^*x+0,775485$
 $Rr1=0,9993898$ $Rr2=0,9987800$ $RSS=2,156279e+000$
 MeanRF: 5,924306e-005 RFSD: 2,144433e-005 RFRSD: 36,197152
 FITType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|--------|
| 1 | 0 | 16425 | 16425 |
| 2 | 2 | 32079 | 35721 |
| | | | 19125 |
| | | | 44425 |
| 3 | 5 | 100137 | 47825 |
| | | | 101483 |
| | | | 101483 |
| 4 | 10 | 154030 | 152670 |
| | | | 154710 |
| | | | 154710 |
| 5 | 20 | 298601 | 315505 |
| | | | 315505 |
| | | | 315505 |
| | | | 464588 |
| 6 | 50 | 751075 | 622225 |
| | | | 814862 |
| | | | 815862 |

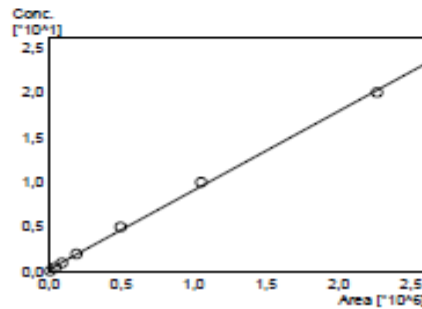
6. Anyon için Kalibrasyon Eğrileri

ID# : 1
 Name : F
 Quantitative Method : External Standard
 Function : $f(x) = 5,72676e-006 \cdot x + 0,0904740$
 $Rr1 = 0,9955931$ $Rr2 = 0,9912056$ $RSS = 3,025400e+000$
 MeanRF: 4,724531e-004 RfSD: 1,069400e-003 RFRSD: 226,350428
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



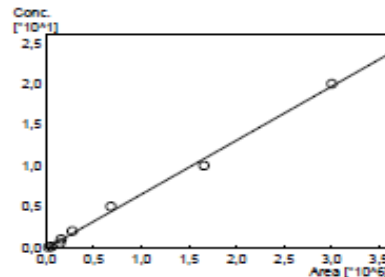
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 1 | 0 | 51351 | 51351 |
| 2 | 0.1 | 30253 | 49992 |
| | | | 30384 |
| | | | 30384 |
| 3 | 0.5 | 140536 | 140308 |
| | | | 140550 |
| | | | 140550 |
| 4 | 1 | 143879 | 140550 |
| | | | 255130 |
| | | | 250152 |
| | | | 5076 |
| | | | 5524 |
| | | | 5524 |
| 5 | 2 | 299048 | 293863 |
| | | | 293863 |
| | | | 588111 |
| | | | 588 |
| | | | 5355 |
| | | | 5524 |
| 6 | 5 | 805085 | 1628100 |
| | | | 159969 |

ID# : 2
 Name : CLO2
 Quantitative Method : External Standard
 Function : $f(x) = 8,82026e-006 \cdot x + 0,279680$
 $Rr1 = 0,9992276$ $Rr2 = 0,9984558$ $RSS = 4,901296e-001$
 MeanRF: 1,023605e-005 RfSD: 8,187482e-007 RFRSD: 7,996671
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



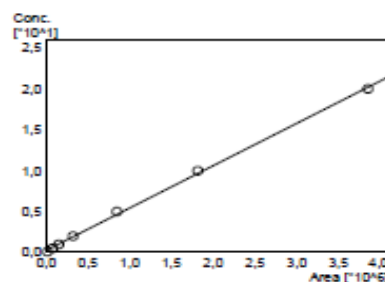
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 2 | 0.1 | 6978 | 8940 |
| | | | 8996 |
| | | | 8996 |
| 3 | 0.5 | 46930 | 46637 |
| | | | 46926 |
| | | | 46926 |
| 4 | 1 | 91061 | 92035 |
| | | | 92035 |
| | | | 85016 |
| 5 | 2 | 191600 | 191369 |
| | | | 192509 |
| | | | 190933 |
| 6 | 5 | 496662 | 509601 |
| | | | 487654 |
| | | | 392230 |
| 7 | 10 | 1052692 | 1034163 |
| | | | 1049154 |
| | | | 1074760 |
| 8 | 20 | 2066503 | 2051436 |
| | | | 2308721 |
| | | | 2299342 |

ID# : 3
 Name : Cl
 Quantitative Method : External Standard
 Function : $f(x) = 5,99917e-006 \cdot x + 0,127196$
 $Rr1 = 0,9979354$ $Rr2 = 0,9958751$ $RSS = 1,419025e+000$
 MeanRF: 7,560777e-005 RfSD: 1,816603e-004 RFRSD: 228,481203
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



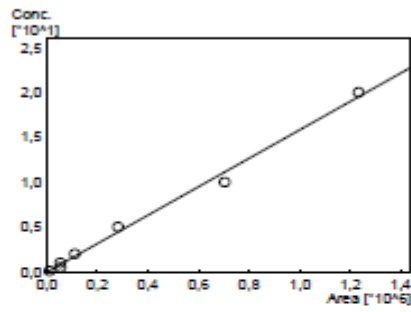
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 1 | 0 | 34449 | 34449 |
| 2 | 0.1 | 55877 | 54634 |
| | | | 55652 |
| | | | 55652 |
| 3 | 0.5 | 144179 | 143662 |
| | | | 144497 |
| | | | 144497 |
| 4 | 1 | 158963 | 157381 |
| | | | 160110 |
| | | | 36208 |
| | | | 74833 |
| | | | 56774 |
| | | | 56774 |
| 5 | 2 | 274608 | 489182 |
| | | | 489718 |
| | | | 463816 |
| | | | 54318 |
| | | | 54318 |
| | | | 46779 |
| 6 | 5 | 681040 | 1300671 |
| | | | 141348 |

ID# : 4
 Name : NO2
 Quantitative Method : External Standard
 Function : $f(x) = 5,17582e-006 \cdot x + 0,277345$
 $Rr1 = 0,9994087$ $Rr2 = 0,9988178$ $RSS = 3,752455e-001$
 MeanRF: 6,086588e-006 RfSD: 5,614069e-007 RFRSD: 9,223672
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



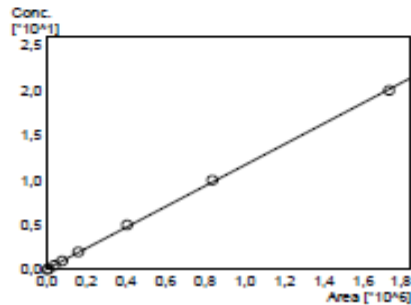
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 2 | 0.1 | 14902 | 15000 |
| | | | 14845 |
| | | | 14845 |
| 3 | 0.5 | 77219 | 77955 |
| | | | 78896 |
| | | | 78896 |
| 4 | 1 | 151385 | 151381 |
| | | | 144899 |
| | | | 159285 |
| 5 | 2 | 320824 | 320190 |
| | | | 322011 |
| | | | 322982 |
| 6 | 5 | 846555 | 874980 |
| | | | 825901 |
| | | | 82497 |
| 7 | 10 | 1815682 | 1782027 |
| | | | 1798992 |
| | | | 1866028 |
| 8 | 20 | 3850956 | 3804242 |
| | | | 385312 |
| | | | 3807049 |

ID# : 5
 Name : BR
 Quantitative Method : External Standard
 Function : $f(x) = 1,58558e-005 \cdot x + 0,0391014$
 $Rr1 = 0,9967708$ $Rr2 = 0,9935519$ $RSS = 2,046671e+000$
 MeanRF: 2,002330e-003 RFSO: 3,962443e-003 RFRSD: 197,891650
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



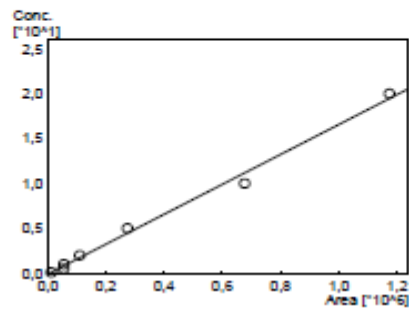
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|--------|
| 1 | 0.1 | 12126 | 11827 |
| 2 | | | 12276 |
| 3 | 0.5 | 55104 | 52925 |
| 4 | | | 52013 |
| 5 | 1 | 54313 | 52013 |
| | | | 108670 |
| | | | 107603 |
| | | | 111311 |
| | | | 1538 |
| | | | 1325 |
| | | | 1382 |
| 6 | 2 | 111443 | 222233 |
| | | | 221791 |
| | | | 220009 |
| | | | 1486 |
| | | | 1496 |
| | | | 1541 |
| 7 | 5 | 282085 | 578415 |
| | | | 561985 |
| | | | 567757 |

ID# : 6
 Name : ClO3
 Quantitative Method : External Standard
 Function : $f(x) = 1,15349e-005 \cdot x + 0,0956306$
 $Rr1 = 0,9998127$ $Rr2 = 0,999254$ $RSS = 1,288761e-001$
 MeanRF: 1,155685e-005 RFSO: 2,648530e-006 RFRSD: 22,917406
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



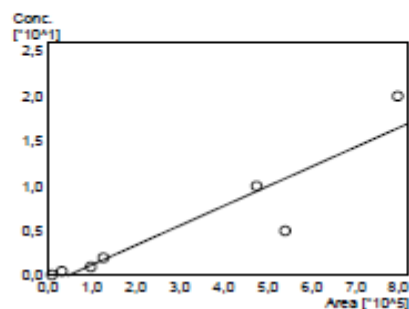
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 1 | 0 | 4281 | 4281 |
| 2 | 0.1 | 8982 | 8655 |
| | | | 9090 |
| 3 | 0.5 | 39882 | 4090 |
| | | | 39882 |
| | | | 39882 |
| 4 | 1 | 78419 | 80549 |
| | | | 74139 |
| | | | 80268 |
| 5 | 2 | 160198 | 160632 |
| | | | 160611 |
| | | | 159019 |
| 6 | 5 | 405533 | 416222 |
| | | | 39788 |
| | | | 403899 |
| 7 | 10 | 836965 | 832764 |
| | | | 833826 |
| | | | 833301 |
| 8 | 20 | 1729006 | 1715318 |
| | | | 1763637 |

ID# : 7
 Name : NO3
 Quantitative Method : External Standard
 Function : $f(x) = 1,66670e-005 \cdot x + 0,0980369$
 $Rr1 = 0,9966073$ $Rr2 = 0,9932262$ $RSS = 2,150062e+000$
 MeanRF: 7,555604e-004 RFSO: 1,665331e-003 RFRSD: 217,530960
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



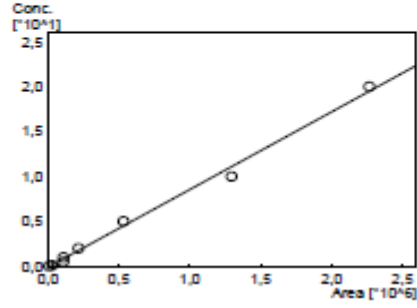
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|--------|
| 2 | 0.1 | 12563 | 12104 |
| | | | 12793 |
| | | | 12792 |
| 3 | 0.5 | 54661 | 54372 |
| | | | 54804 |
| | | | 54804 |
| 4 | 1 | 55542 | 106165 |
| | | | 98725 |
| | | | 108358 |
| | | | 8595 |
| | | | 8705 |
| | | | 8705 |
| 5 | 2 | 110073 | 214770 |
| | | | 215335 |
| | | | 217803 |
| | | | 6594 |
| | | | 6594 |
| | | | 6423 |
| 6 | 5 | 274148 | 556116 |
| | | | 533795 |
| | | | 538185 |

ID# : 8
 Name : PO4
 Quantitative Method : External Standard
 Function : $f(x) = 2,19150e-005 \cdot x + 0,993800$
 $Rr1 = 0,9203187$ $Rr2 = 0,8469865$ $RSS = 4,836778e+001$
 MeanRF: 3,374081e-005 RFSO: 5,627292e-005 RFRSD: 166,779957
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



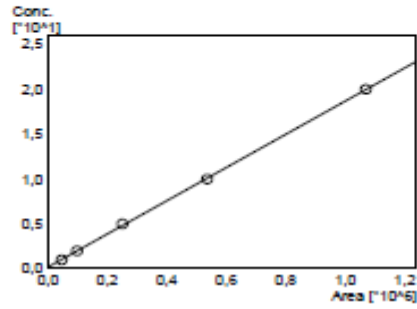
| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|--------|
| 2 | 0.1 | 9333 | 8870 |
| | | | 9564 |
| | | | 9564 |
| 3 | 0.5 | 32022 | 31868 |
| | | | 31709 |
| | | | 32099 |
| 4 | 1 | 58421 | 61506 |
| | | | 56826 |
| | | | 63641 |
| | | | 136281 |
| | | | 136282 |
| | | | 136282 |
| 5 | 2 | 126701 | 131878 |
| | | | 132281 |
| | | | 129041 |
| | | | 120976 |
| | | | 120976 |
| | | | 13095 |
| 6 | 5 | 338477 | 350951 |
| | | | 336378 |
| | | | 340101 |

ID# : 9
 Name : SO4
 Quantitative Method : External Standard
 Function : $f(x) = 8,65989e-005 \cdot x - 0,120803$
 Rr1=0,9972008 Rr2=0,9944095 RSS=1,923207e+000
 MeanRF: 2,215521e-004 RFSID: 5,210714e-004 RFRSD: 235,191307
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 1 | 0 | 6190 | 6190 |
| 2 | 0,1 | 31855 | 31158 |
| | | | 34494 |
| | | | 37204 |
| 3 | 0,5 | 109744 | 105211 |
| | | | 110011 |
| | | | 110011 |
| 4 | 1 | 110100 | 207581 |
| | | | 194555 |
| | | | 211900 |
| | | | 14555 |
| | | | 15928 |
| | | | 15928 |
| 5 | 2 | 216097 | 413738 |
| | | | 414106 |
| | | | 405193 |
| | | | 20525 |
| | | | 20525 |
| | | | 18500 |
| 6 | 5 | 532780 | 1065933 |
| | | | 1016214 |

ID# : 10
 Name : CrO3
 Quantitative Method : External Standard
 Function : $f(x) = 1,84893e-005 \cdot x + 0,197414$
 Rr1=0,9999108 Rr2=0,9999215 RSS=4,304394e-002
 MeanRF: 1,981796e-005 RFSID: 1,113919e-005 RFRSD: 5,620755
 FIType : Linear
 ZeroThrough : Not Through
 Weighted Regression : None
 Detector Name : Detector A



| # | Conc.(Ratio) | MeanArea | Area |
|---|--------------|----------|---------|
| 4 | 1 | 26587 | 47524 |
| | | | 46260 |
| | | | 46260 |
| 5 | 2 | 99053 | 67419 |
| | | | 67419 |
| | | | 69741 |
| 6 | 5 | 251016 | 250330 |
| | | | 251358 |
| | | | 251358 |
| 7 | 10 | 536269 | 534500 |
| | | | 537570 |
| | | | 533599 |
| 8 | 20 | 1069827 | 1047458 |
| | | | 1081826 |
| | | | 1080198 |

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.
 Aplikasyon Notu Ant Teknik tarafından önceden bildirilmeksizin değiştirilebilir.