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APPENDICES

APPENDIX A

FACTOR ANALYSIS PROGRAM

```
%This program is used for  Quantitative Analysis of Mixed Xylene
%by applying Factor Analysis with FT- IR  Spectra
%Modified by Pornphen Neamjoy
```

1. Input_Data.mat (Data Loading)

```
%%%%%%%%%%%%  FILE IN DATA %%%%%%%%%%%%
%%%% Load experimental data in ASCII formatted file%%%%%
file_in=char('Cal.01','Cal.02', 'Cal.03','Cal.04' ..... 'Mix.01');
```

2. Mixture_Matrix.mat (Manage the experimental data into matrix form)

```
%%%%%%%Construct Calibration Set %%%%%%%
Input_Data;
d=[ ] ;
for i=1:size(file_in,1);
  temp=load(file_in(i,:));
  d=[S temp(:,2)] ;
end
%%%%%%%Plot All Mixture Spectra %%%%%%%%%%%
X = temp(:,1);
Y = S (: ,:) ;
%%%%% Check number of Mixture in Mixture Matrix %%%%%%%%%%
size (d)
%%%%% Number of column is the nimber of mixtures %%%%%%%%%
```

3. TestPCA.mat (Principle component analysis process)

```
% PCA Process to find the number of comonent
% and reproduce spectra.
clear;
Mixture_Matrix;
[m,n] = size (D);
Z = d * d';
[L,lamda] = eig (Z);
%1. Determination of Activecomponent in the spectra set
% number of nonzero element of N equal to
% the number of component.
N = diag(lamda);
```

4. TestEFA.mat (Evolving factor analysis process)

```
function [e,efl,ebl]=efa(d,ns)
% ns is the number of rows of this matrix
% e  is the abstract concentration profiles found by EFA
clear;
Mixture_Matrix;
ns=size(d,2); % number of factor ( column ) in matrix d
x=d(:,1:ns);
[nw,ns]=size(x);
minn=min(nw,ns);
ef=ones(ns-1,ns-1).*1e-33;
eb=ef;
% forward analysis
disp('FORWARD ANALYSIS')
```

```
n=2;
while n<=ns
l=svd(x(:,1:n));
nl=size(l);
ef(1:nl,n-1)=l(1:nl,1);
efl(1:nl,n-1)=2*log10(ef(1:nl,n-1));
eflmin=min(min(efl));
efl(efl==0)=ones(size(find(efl==0)))*eflmin;
n=n+1;
end
ef=ef';
efl=efl';
% backward analysis
disp('BACKWARD ANALYSIS')
x=x(:,ns:-1:1);
n=2;
while n<=ns
l=svd(x(:,1:n));
nl=size(l);
eb(1:nl,n-1)=l(1:nl,1);
ebl(1:nl,n-1)=2*log10(eb(1:nl,n-1));
eblmin=round(min(min(ebl)));
ebl(ebl==0)=ones(size(find(ebl==0)))*eblmin;
n=n+1;
end
eb=eb';
ebl=ebl';
% evolving factor analysis plots
```

```
% 1) plot the singular values (ef,eb)
xforward=[2:ns];
xbackward=[ns :-1 :2];
hold off;
subplot (211) , plot ( xforward,ef,xbackward,eb)
plot(xforward,ef,xbackward,eb)
title('evolving factor analysis')
% 2) plot the log of eigenvalues (ef ef^2, eb eb^2)
maxvalue=round(max(max(ebl)))+1;
minvalue=round(min(min(ebl)))-1;
[maxvalue,minvalue];
minvalue= 0 ;% input(' min. value of log efa plots ? ');
efl(efl<minvalue)=ones(size(find(efl<minvalue)))*minvalue;
ebl(ebl<minvalue)=ones(size(find(ebl<minvalue)))*minvalue;
subplot (212) ,plot (xforward,efl,xbackward,ebl)
plot(xforward,efl,xbackward,ebl)
title('evolving (log eig) factor analysis')
% 3) the arranged conc. profiles for the num. of factors
%% Num. of factors to be considered ? result from PCA %%%%
nf=3;
axis;
while nf~=0
   else,
   e(i,j)=eb(ii,jj);
   end
   if e(i,j)==0.0, e(i,j)=1.0e-30; end
end
end
```

Insert the number of component in the mixture

```
plot(xforward,e)
title('arranged efa (svd) profiles')
nf=0; %('other num. of components to be considered:? ');
end
   e(i,j)=ef(i,j);
e(2:ns,:)=e(1:ns-1,:)
```

5. SLR.mat (Simple linear regresstion process)

```
% Iteration process to find  the true result
% from the rough concentration (e) from TesEFA .mat
clear ;
Mixture_Matrix;
Test_EFA;
Ci = ans';
%Simple Linear Regrssion
nonnegativity = 1;
normalize     = 1;
k=0;
Residual = 1;
%% Factor to be consideration before run program%%
itmax=50; % number of iteration process
n=14;  % number of mixture
while norm( Residual )> 1.0e-8 & k< itmax;
  k = k+1;
  if k == 1
    Sr = (d* Ci')*(inv(Ci*Ci'));
  else
    Sr = (d* Cr')*(inv(Cr*Cr'));
```

Insert number of mixture (Calibration +Test)

```
  end
  if nonnegativity ==1;
    Sr(Sr < 0) = ones(size(find (Sr< 0))).*1e-33;
for j=1:nf
  jj=nf+1-j;
for i=1:ns-1,
  ii=ns-i;
  if ef(i,j)<=eb(ii,jj),
end
  Cr =(inv(Sr'*Sr))* Sr' *d ;
  if nonnegativity ==1;
Cr(Cr < 0) = ones(size(find (Cr< 0))).*1e-33;
  end
  if normalize ==1;
    Crsum = sum(Cr);
    for i=1:n
      Cr (:,i) = Cr ( :,i)/ Crsum(i)
    end
  end
 end
%%%% Recheck reproduce spectra.
X = temp(:,1);
Y1= Sr (:,1 );
Y2= Sr ( : ,2 );
Y3= Sr ( : ,3 );
subplot (311) , plot ( X,Y1)
subplot (312) , plot ( X,Y2)
subplot (313) , plot ( X,Y3)
```

Result (Volume Fraction)

Result (Original Spectra)

APPENDIX B

SPECIFICATION OF THREE XYLENE ISOMER

1. ***o-xylene*** **(Carlo erba reagent)**

$C_6H_4(CH_3)_2$	Cas. No. 96-47-6
-FW	106.17
-Minimum assay	99 % (GLC)
-Freezing point	-25 °C ± 1.5 °C
-Boiling point	144.0 °C (b.r. ± 1.0 °C)
-Density @ 20 °C/ 4 °C	0.880 ± 0.005
-Refractive index at 20 °C/D	1.5058 ± 0.0030

2. ***p*-xylene (Carlo erba reagent)**

$C_6H_4(CH_3)_2$	Cas. No. 106-42-3
-FW	106.17
-Minimum assay	99 % (GLC)
-Melting point	13.5 °C ± 1.0 °C
-Boiling point	138.0 °C (b.r. ± 1.5 °C)
-Density@20 °C/ 4 °C	0.861 ± 0.005
-Refractive index at 20 °C/D	1.4958 ± 0.0030

3. ***m*-xylene** (Carlo erba reagent)

$C_6H_4(CH_3)_2$	Cas. No. 108-38-3
-FW	106.17
-Minimum assay	99 % (GLC)
-Melting point	-47.4 °C ± 1.5 °C
-Boiling point	139.0 °C (b.r. ± 1.0 °C)
-Density@20 °C/ 4 °C	0.864 ± 0.003
-Refractive index at 20 °C/D	1.4973 ± 0.0030

APPENDIX C
PREDICTED ERROR

1. Predicted residual error of sum of square (PRESS)

$$PRESS = \sum_{i=1}^{n} (y^* - y)^2$$

where: y^* = The actual value

y = The predicted value

n = number of testing sample

2. Variance of prediction (s^2)

$$s^2 = \sum_{i=1}^{n} \frac{(y^* - y - bias)^2}{n-1}$$

where: y^* = The actual value

y = The predicted value

n = number of testing sample

bias = mean of error. This can be written as $\overline{y^* - y}$

APPENDIX D
GAS CHROMATOGRAPHY TEST

Gas Chromatography Operating Conditions:

Standard References:

p-xylene	Merck 808691	98%
m-xylene	Merck 808688	99%
o-xylene	Merck 808697	99%

Experimental Setup:

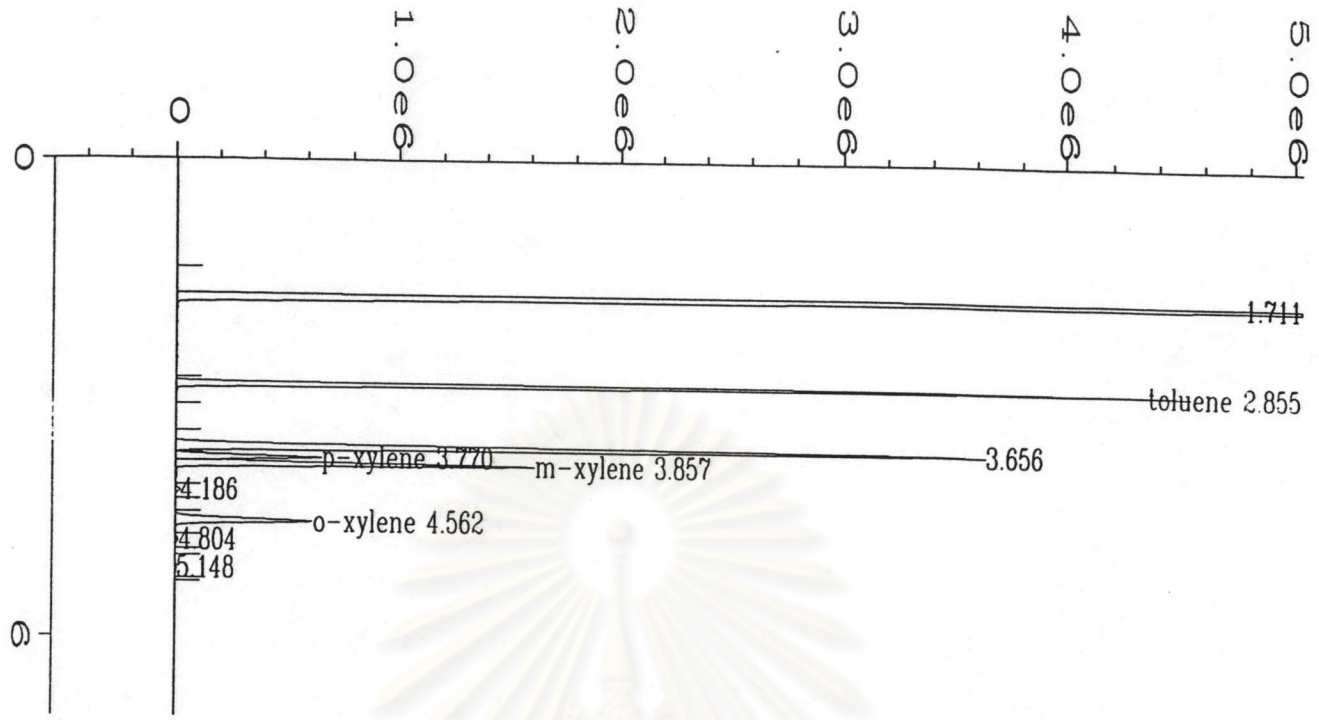
Column	Stabiwax 30 m length/ 0.53 mm ID./ 1.0 μm df
Flow rate	3 ml/min
Injector	150 °C 1.0 μl
Detector	FID 230 c

Gas Chromatography Test Result:

Mixed Xylene:	o-xylene	% weight	10.9
	m-xylene	% weight	23.2
	p-xylene	% weight	9.9

Remarks:

The chromatogram and detail were illustrated in page 62-63.



Internal Standard Report

Data File Name : C:\HPCHEM\1\DATA\KHANIT\VH759019.D
Operator : Khanit
Instrument : ANALYZER1
Sample Name : Mixed xylene
Run Time Bar Code:
Acquired on : 11 Jul 01 11:42 AM
Report Created on: 11 Jul 01 02:47 PM
Last Recalib on : 11 Jul 01 02:39 PM
Multiplier : 0.025

Page Number : 1
Vial Number : 2
Injection Number :
Sequence Line :
Instrument Method: XYLE_STA.
Analysis Method : XYLE_STA.
Sample Amount : 0.8695
ISTD Amount : 17.71

Sig. 2 in C:\HPCHEM\1\DATA\KHANIT\VH759019.D

Ret Time	Area	Type	Width	Ref#	Amount %	Name
2.855	9821491	PV T	0.034	1-I	50.914	toluene
3.770	1870500	VV T	0.044	1	9.901	p-xylene
3.857	4658004	VV T	0.045	1	23.329	m-xylene
4.562	2209841	BV T	0.056	1	10.924	o-xylene

แบบ วศ.

ที่ วว 0504/ 8660

ถึง สาขาวิชา ปิโตรเคมี และวิทยาศาสตร์พอลิเมอร์ จุฬาลงกรณ์มหาวิทยาลัย

กรมวิทยาศาสตร์บริการขอส่งรายงานการตรวจ วิเคราะห์ ทดสอบ วัตถุตัวอย่าง ตาม คำร้อง

ลงวันที่ 5 มิถุนายน 2544 เลขรับ 3080 วันที่ 5 มิถุนายน 2544

ซึ่งกรมวิทยาศาสตร์ ฯ ได้รับเมื่อวันที่ 5 มิถุนายน 2544

กรมวิทยาศาสตร์บริการ DEPARTMENT OF SCIENCE SERVICE 19 กรกฎาคม 2544

กอง เคมี
โทร. 2017227-8

รายงานการตรวจ วิเคราะห์ ทดสอบ

ชื่อวัตถุตัวอย่าง ตามที่ผู้ส่งเรียก	เครื่องหมาย ตรา ฯลฯ ที่ระบุตัวอย่าง	หมายเลข ปฏิบัติการ			
ไซลีนผสม (Mixed xylene)	-	VH.759	O-xylene	ร้อยละ	10.9
			m-xylene	ร้อยละ	23.2
			p-xylene	ร้อยละ	9.9

(นางสาวขนิษฐ พานชูวงศ์)
นักวิทยาศาสตร์ 4

หมายเหตุ ค่าธรรมเนียมการวิเคราะห์ 2,000.00 บาท (สองพันบาทถ้วน)

รายงานนี้ : - รับรองเฉพาะวัตถุตัวอย่างที่ได้ตรวจ วิเคราะห์ ทดสอบ เท่านั้น
- ไม่รับรองวัตถุหรือสินค้าที่ใช้รายงานนี้ในการโฆษณาหรืออ้างถึง

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