

# Przykłady zastosowania techniki FT-IR w badaniu materiałów asfaltowych

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14 listopad 2013



# Zakres prezentacji

**FT-IR** (**Fourier Transform Infrared spectroscopy**)

**Teoria**

**Przykłady**

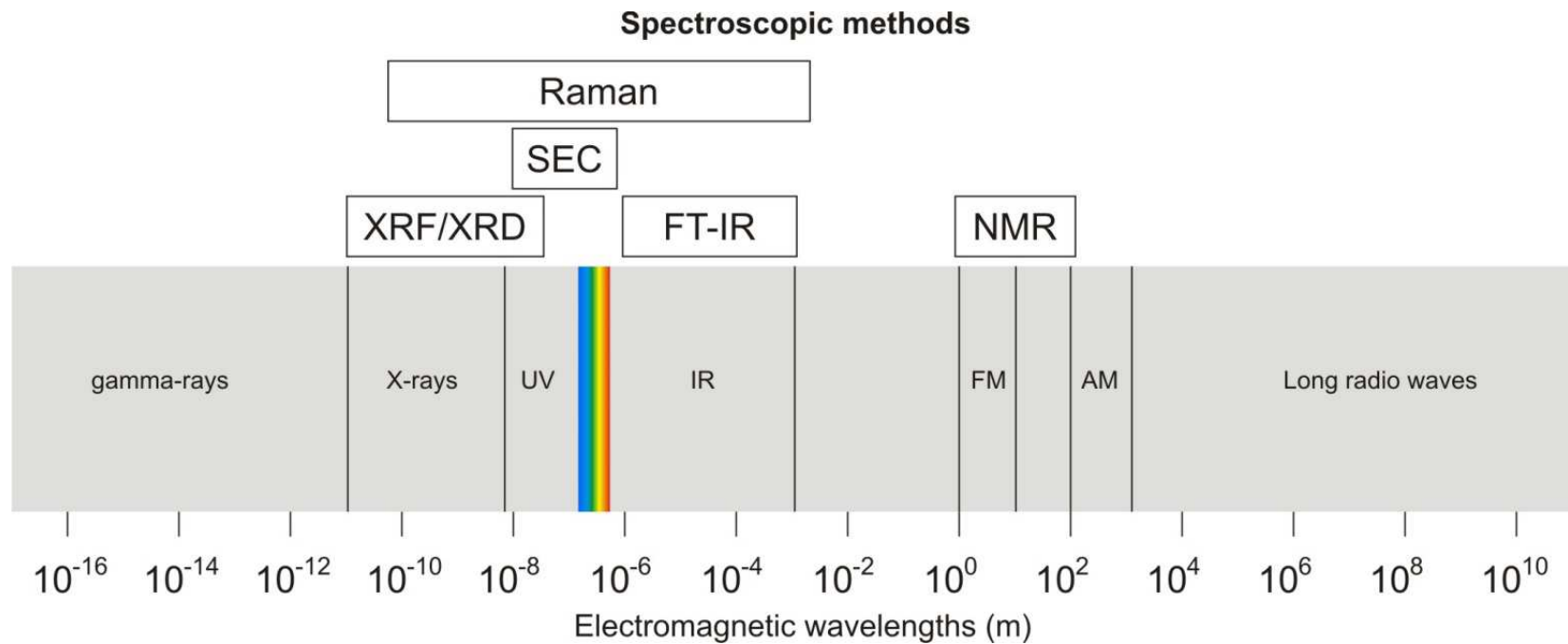
**Literatura**

**Fourierowska spektroskopia  
w podczerwieni**



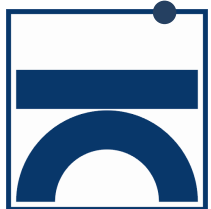
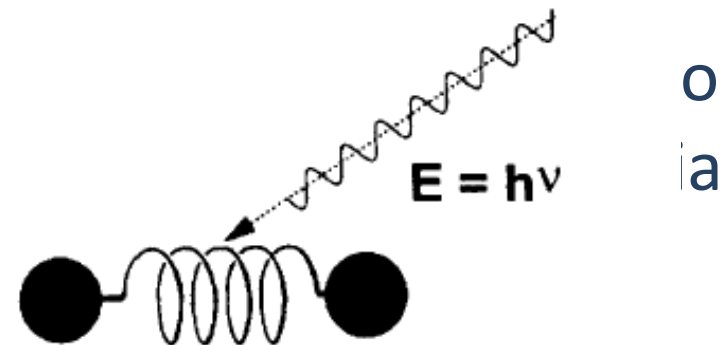
# Teoria – widmo światła

- Widmo elektromagnetyczne



# Teoria - interakcja

- IR opiera się na interakcji pomiędzy promieniowaniem podczerwonym a cząsteczkami (*= grupa dwóch lub więcej atomów utrzymywanych razem wiązaniem chemicznym*)
- Promieniowanie podczerwone jest wchłaniane przez materiał i ilość wchłoniętego promieniowania jest mierzona jako funkcja energii
- Promieniowanie jest wchłaniane, gdy jego częstotliwość pokrywa się z częstotliwością drgań chemicznych cząsteczki

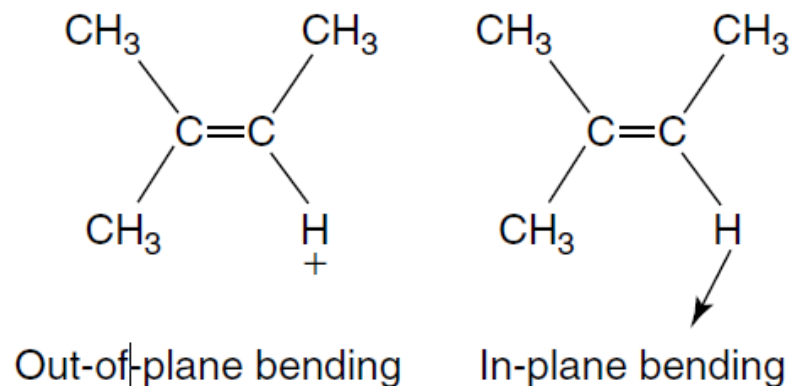
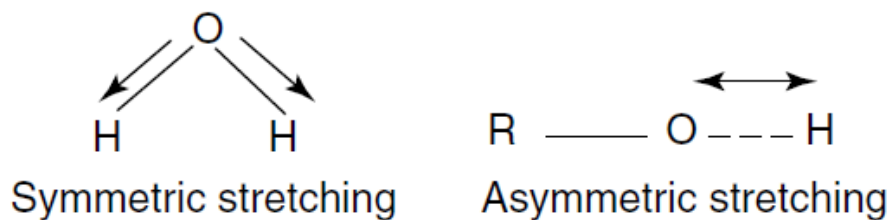
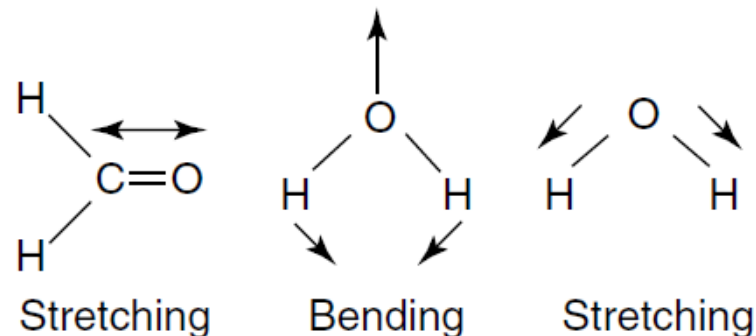


Wchłonięte promieniowanie jest odczytywane przez „skok” na wykresie (=widmo IR)

# Teoria - wiązania

- Drgania wiązań mogą być:

- Rozciągające
- Zginające
- W płaszczyźnie
- Poza płaszczyzną
- Symetryczne
- Asymetryczne
- Słabe/średnie/mocne

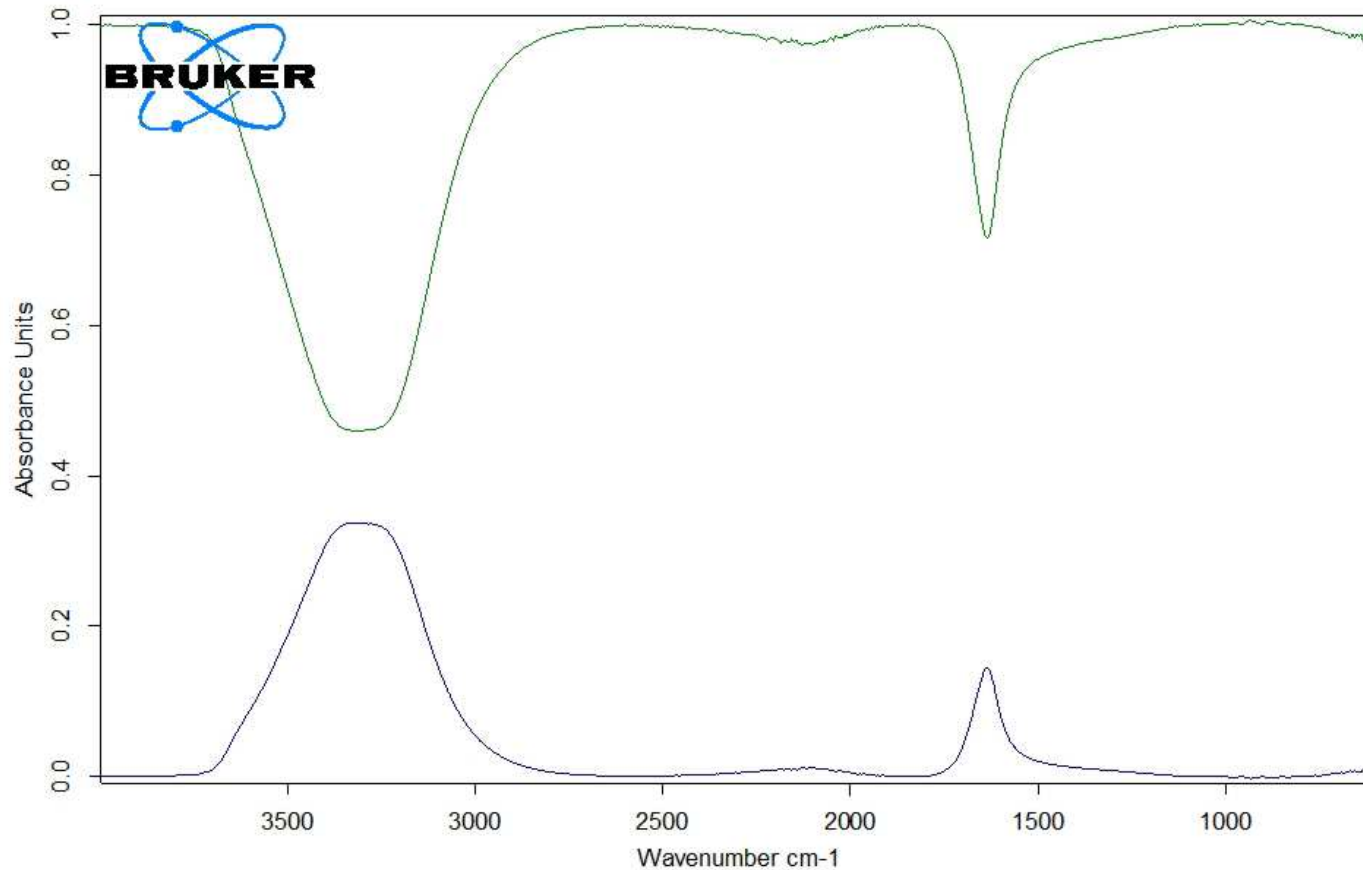


# Teoria – widmo IR

- Analiza widma (=wykresu) polega na odpowiednim przypisywaniu „skoków” do drgań cząsteczki
- Może być wiele rodzajów drgań nawet dla prostych cząsteczek
- Skoki na widmie mogą się pokrywać co utrudnia analizę



# Teoria – przykład widma



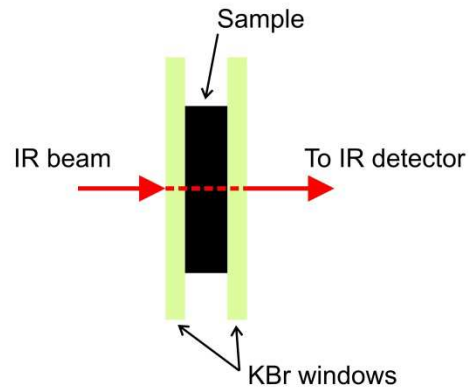
C:\OPUS_7.2.139.1294\Data\MIR Basic Demodata\Quant_LambertBeer\Example 1\Sugar 0% _ Water 100%.0	Sugar 0% / Water 100%	2010-09-17
C:\OPUS_7.2.139.1294\Data\MIR Basic Demodata\Quant_LambertBeer\Example 1\Sugar 0% _ Water 100%.0	Sugar 0% / Water 100%	



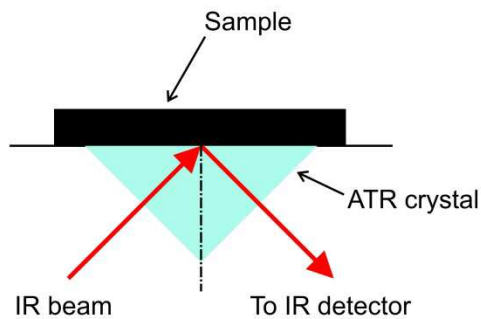
# Teoria – sposoby pomiaru

- Przenikanie (Tr)
- Osłabione całkowite odbicie (ATR)
- Rozproszone odbicie (DRIFT)

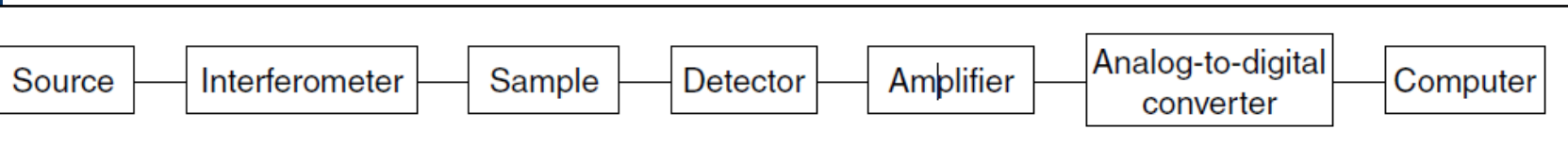
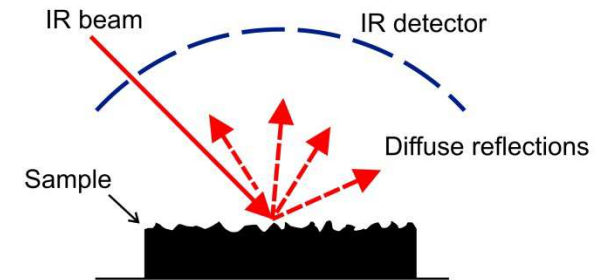
Transmission



ATR

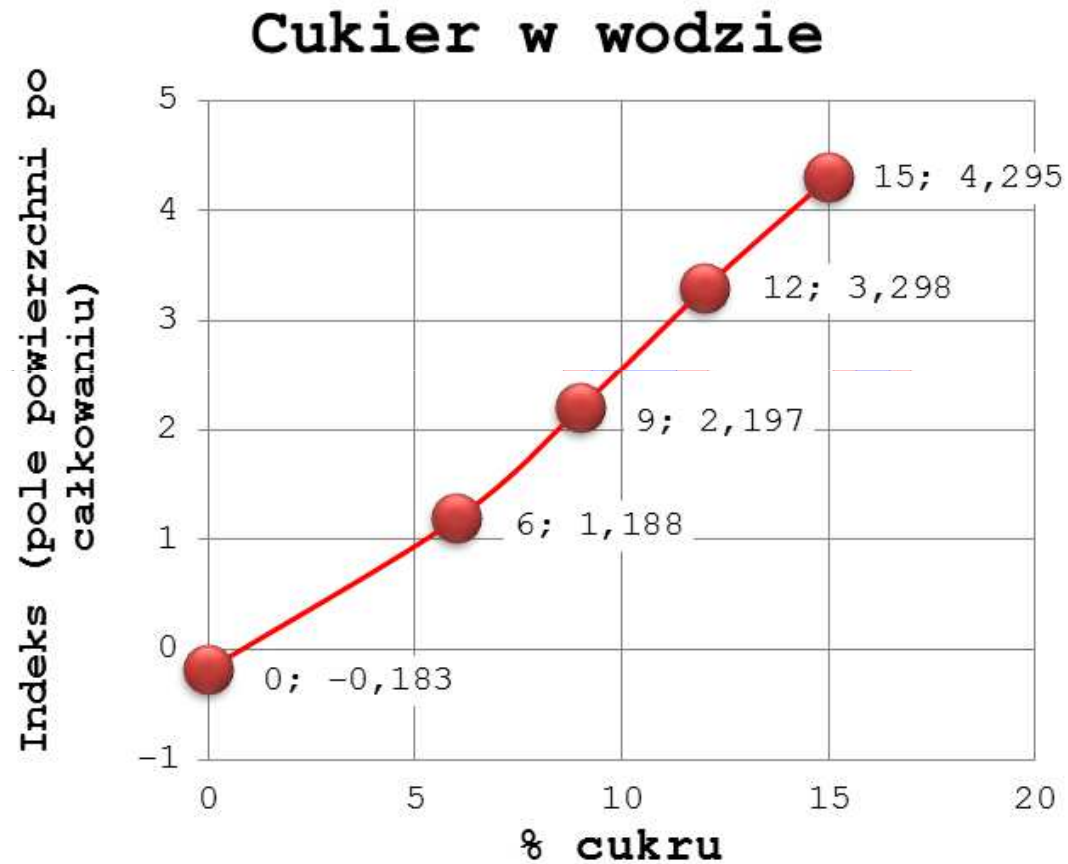


DR

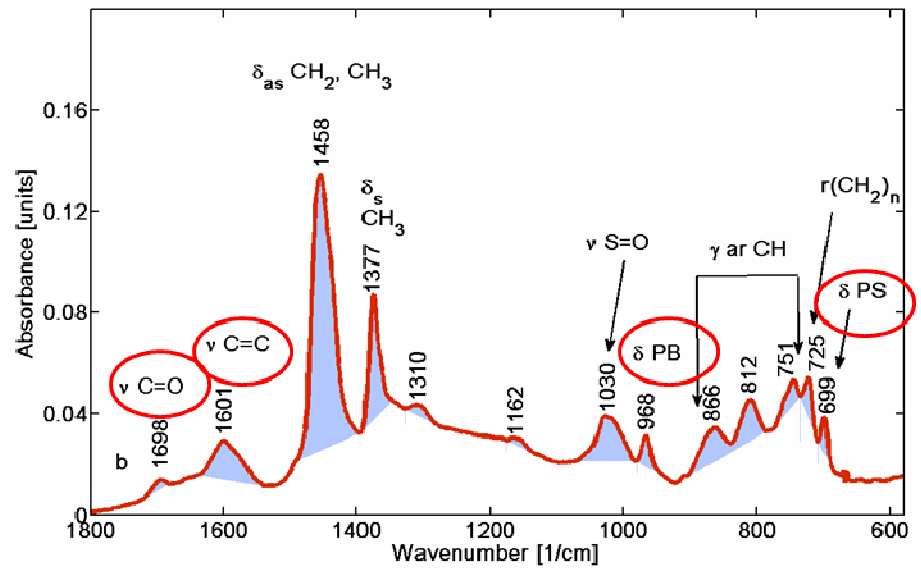
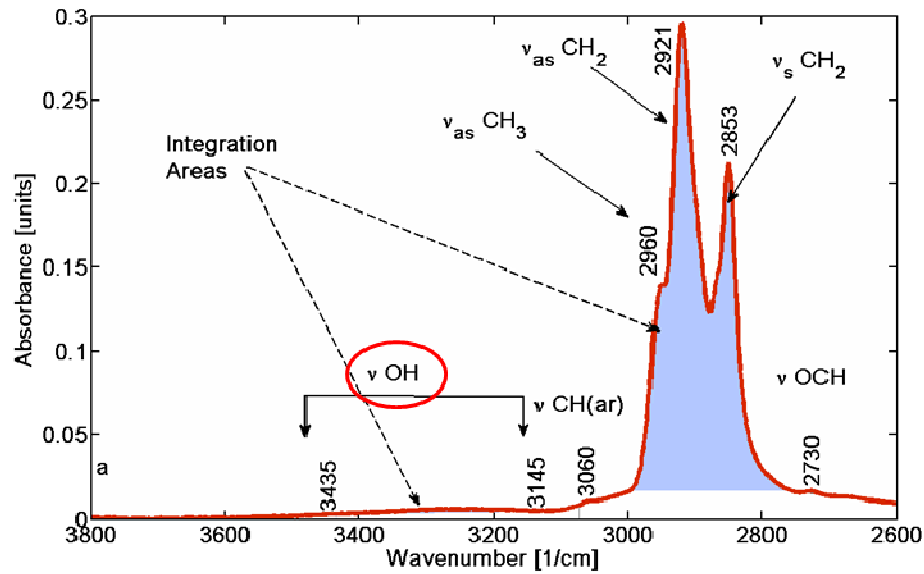




# Prosty przykład



# Przykładowe widmo asfaltu



# Grupy funkcyjne w asfalcie

**Table 4.3** Characteristic infrared bands of oxygen-containing compounds

Wavenumber (cm <sup>-1</sup> )	Assignment
3600	<i>Alcohol and phenols</i> Alcohol O–H stretching
3550–3500	Phenol O–H stretching
1300–1000	C–O stretching
1100	<i>Ethers</i> C–O–C stretching
2900–2700	<i>Aldehydes and ketones</i> Aldehyde C–H stretching
1740–1720	Aliphatic aldehyde C=O stretching
1730–1700	Aliphatic ketone C=O stretching
1720–1680	Aromatic aldehyde C=O stretching
1700–1680	Aromatic ketone C=O stretching
1750–1730	<i>Esters</i> Aliphatic C=O stretching
1730–1705	Aromatic C=O stretching
1310–1250	Aromatic C–O stretching
1300–1100	Aliphatic C–O stretching
3300–2500	<i>Carboxylic acids</i> O–H stretching
1700	C=O stretching
1430	C–O–H in-plane bending
1240	C–O stretching
930	C–O–H out-of-plane bending
1840–1800	<i>Anhydrides</i> C=O stretching
1780–1740	C=O stretching
1300–1100	C–O stretching

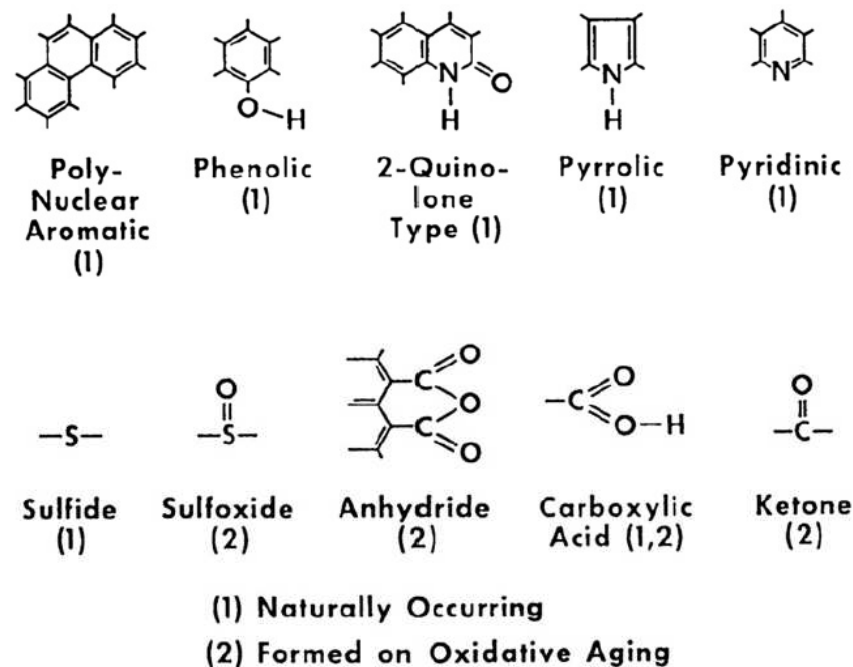
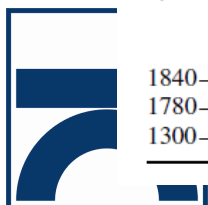
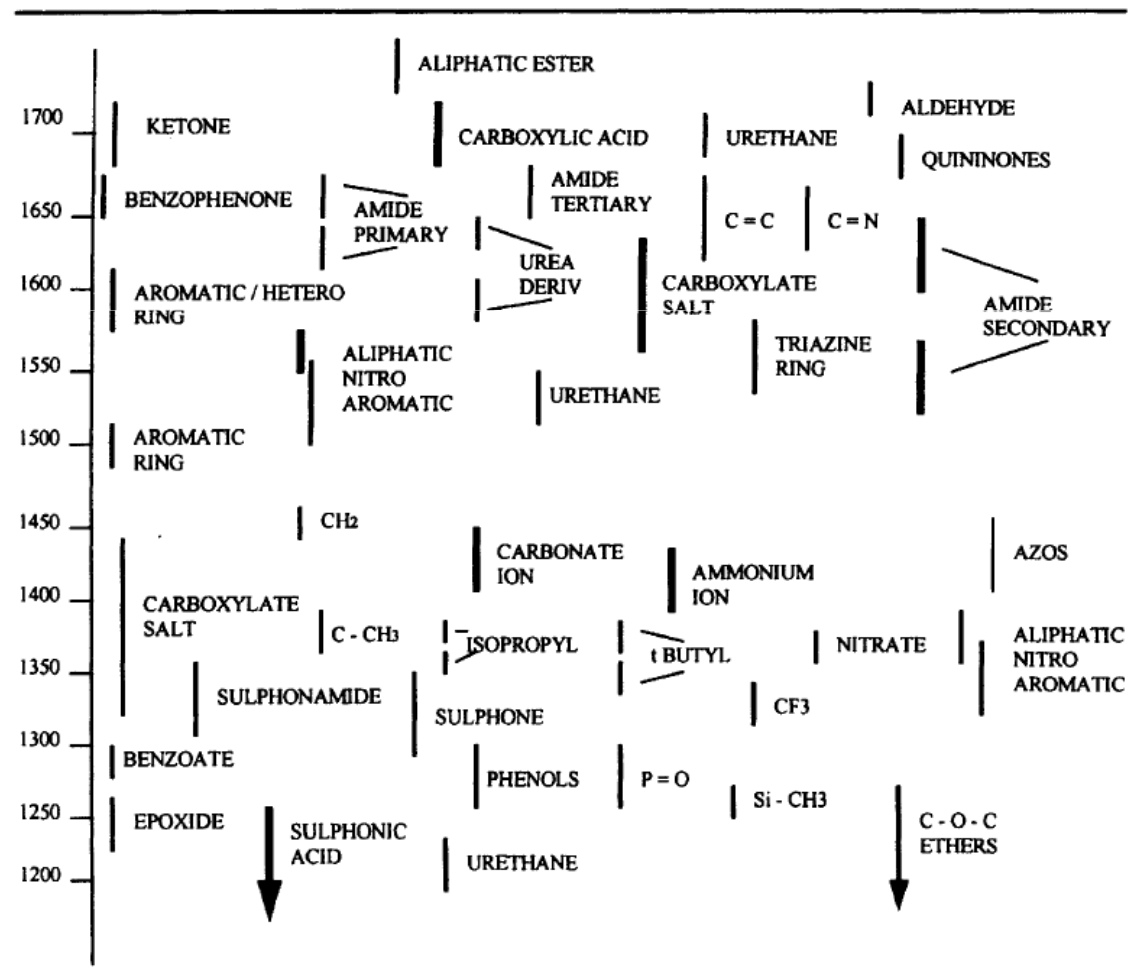


Fig. 2. Functional groups present in bitumen reprinted from [26] with permission by National Academy of Science.



# Grupy funkcyjne na widmie

**Table 2(c)** Double bond and fingerprint region ( $1700\text{--}1300\text{ cm}^{-1}$ ) functional group correlation chart

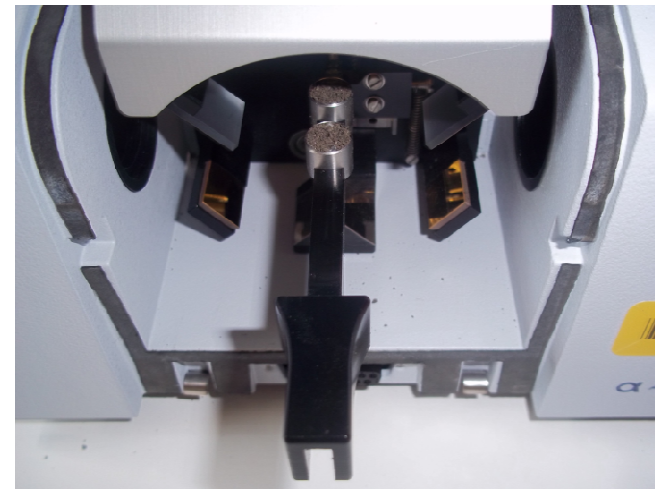
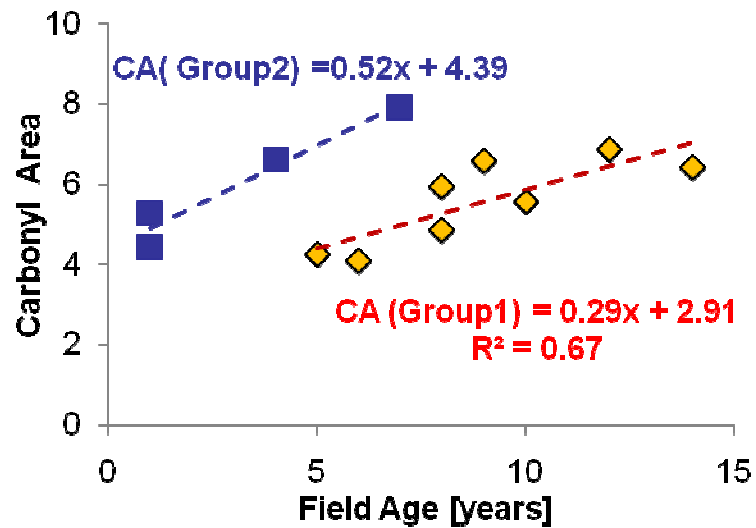
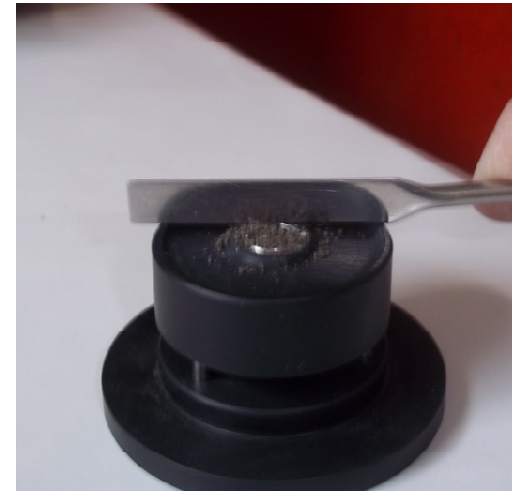


# Przykłady

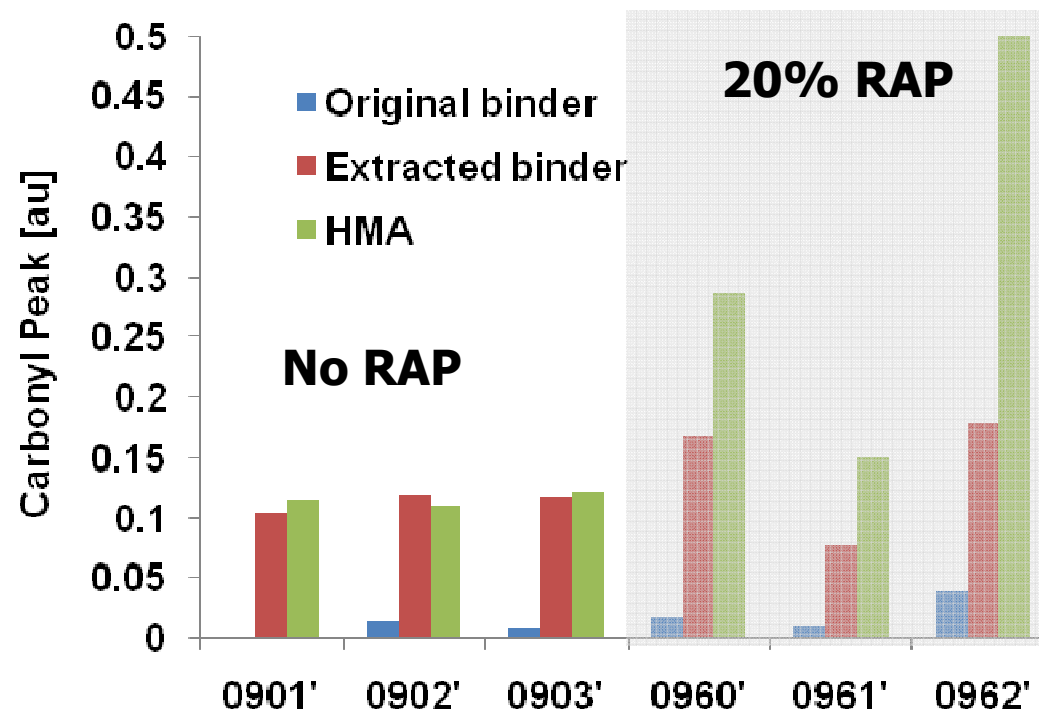
- Przykłady
  - Lepiszcza (daktyloskopia, wykrywanie, ilościowanie)
  - Mieszanki (wykrywanie, ilościowanie)
  - Destrukt asfaltowy
  - Zaawansowana analiza (2D CS, PCA)
  - Różne materiały budowlane
  - W laboratorium i w terenie



# Przykłady – DRIFT (odcinki teren.)

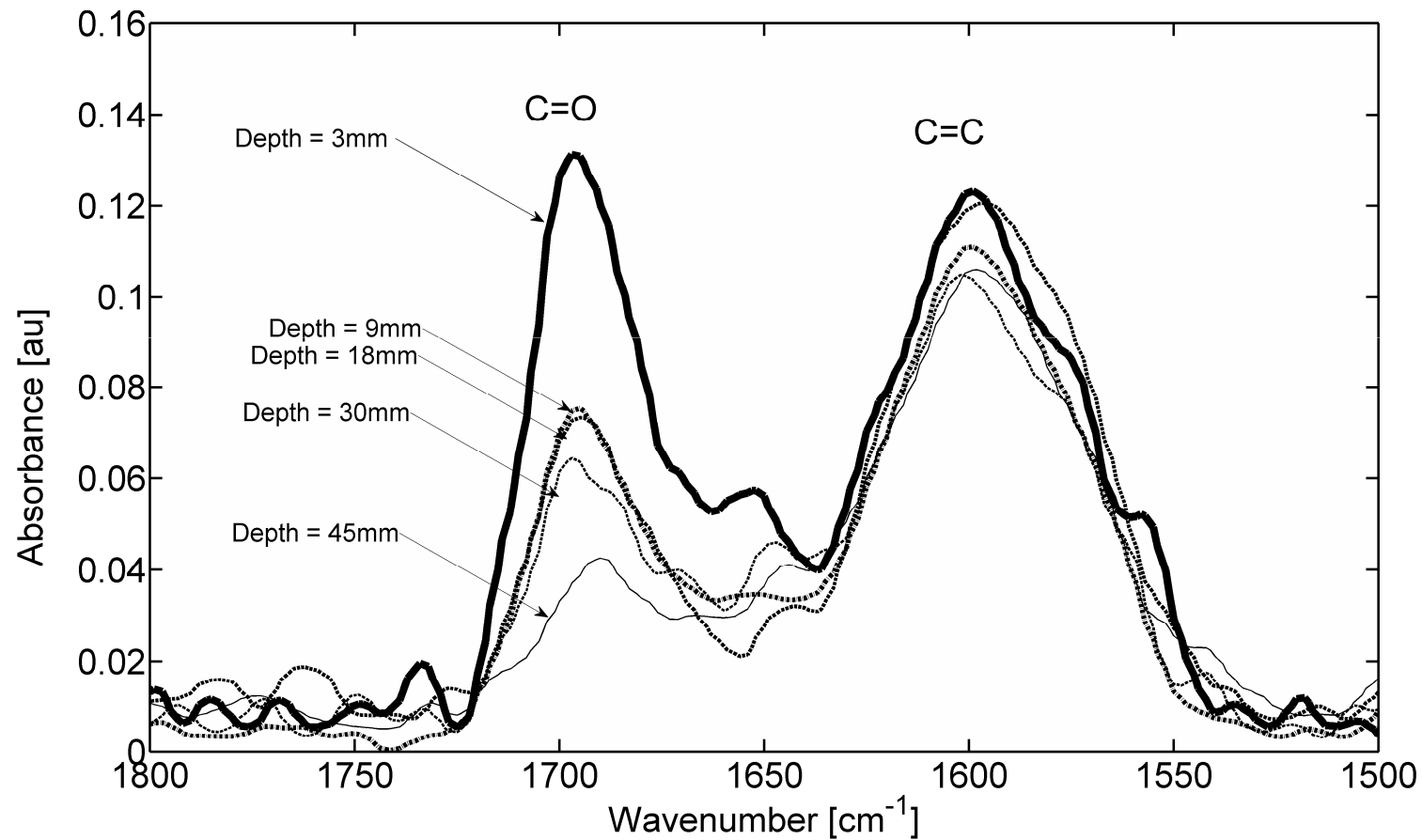


# Przykłady – LTPP (RAP)



Odcinki LTPP, 12 lat

# Przykłady – LTPP (RAP)

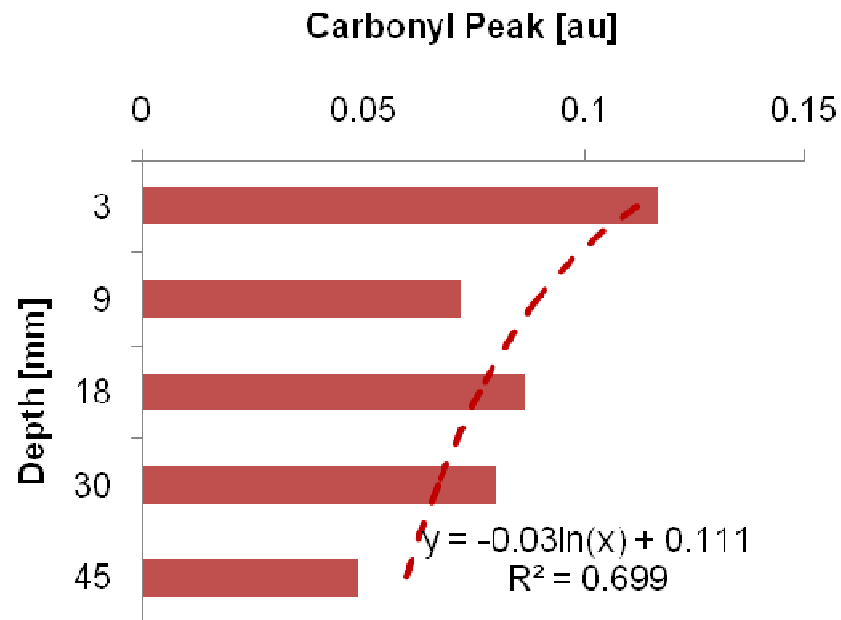


Odcinki LTPP, 12 lat

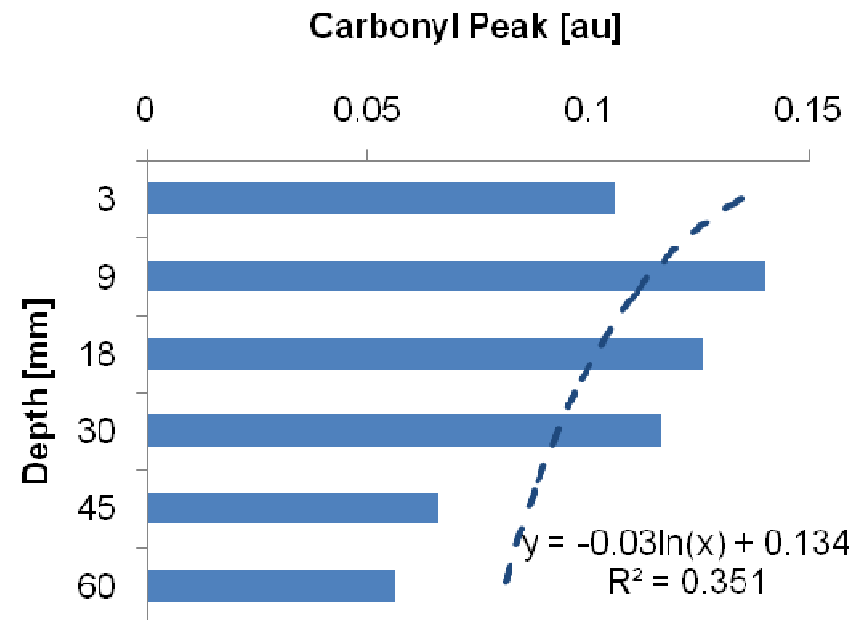


# Przykłady – LTPP (RAP)

## Section 0901 (no RAP)

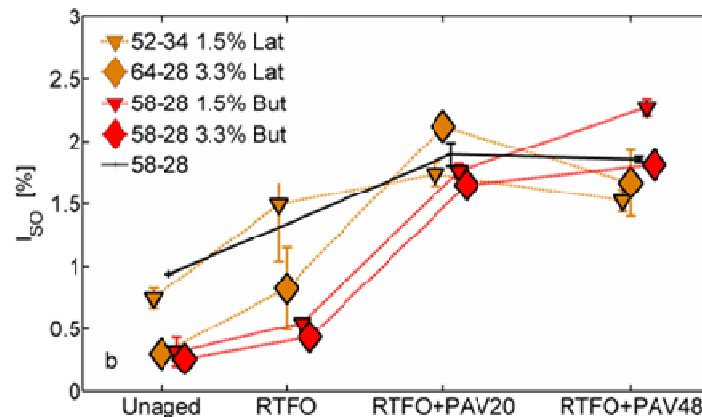
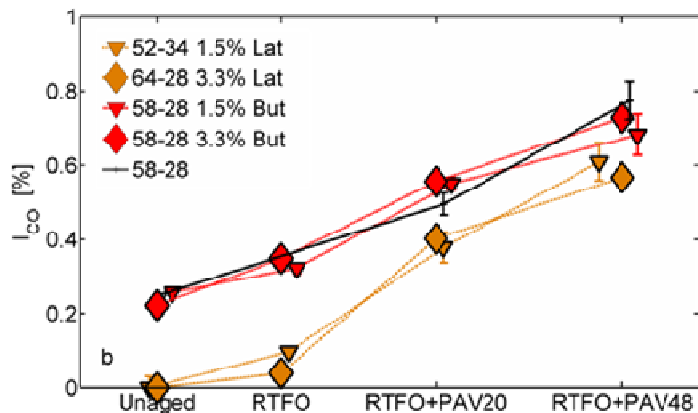
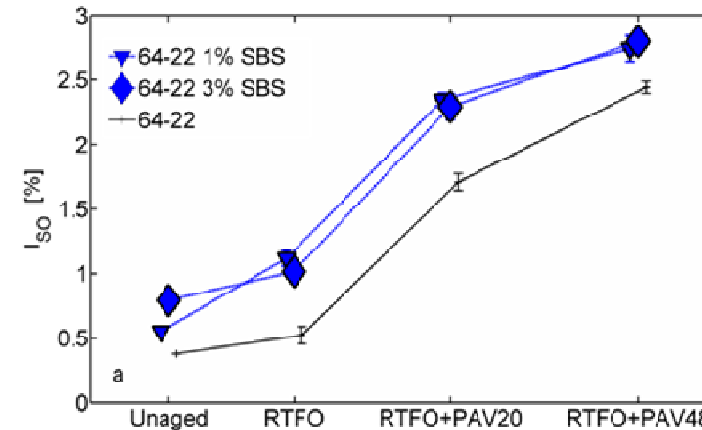
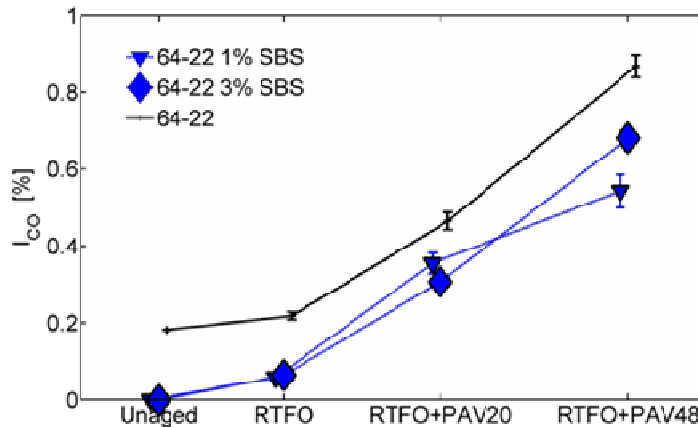


## Section 0962 (20% RAP)



Odcinki LTPP, 12 lat

# Przykłady – starzenie w lab.



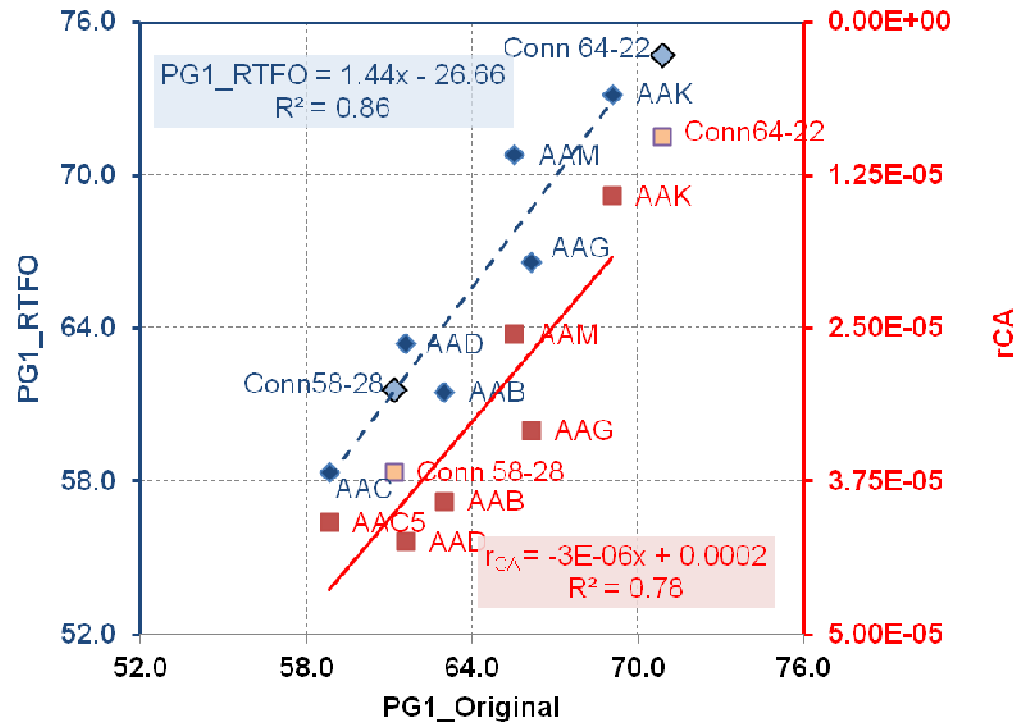
**C=O**

**S=O**

Grupa hydroksylowa (OH), ketonowa (C=O) i sulfotlenkowa (S=O)



# Przykłady – asfalty MRL

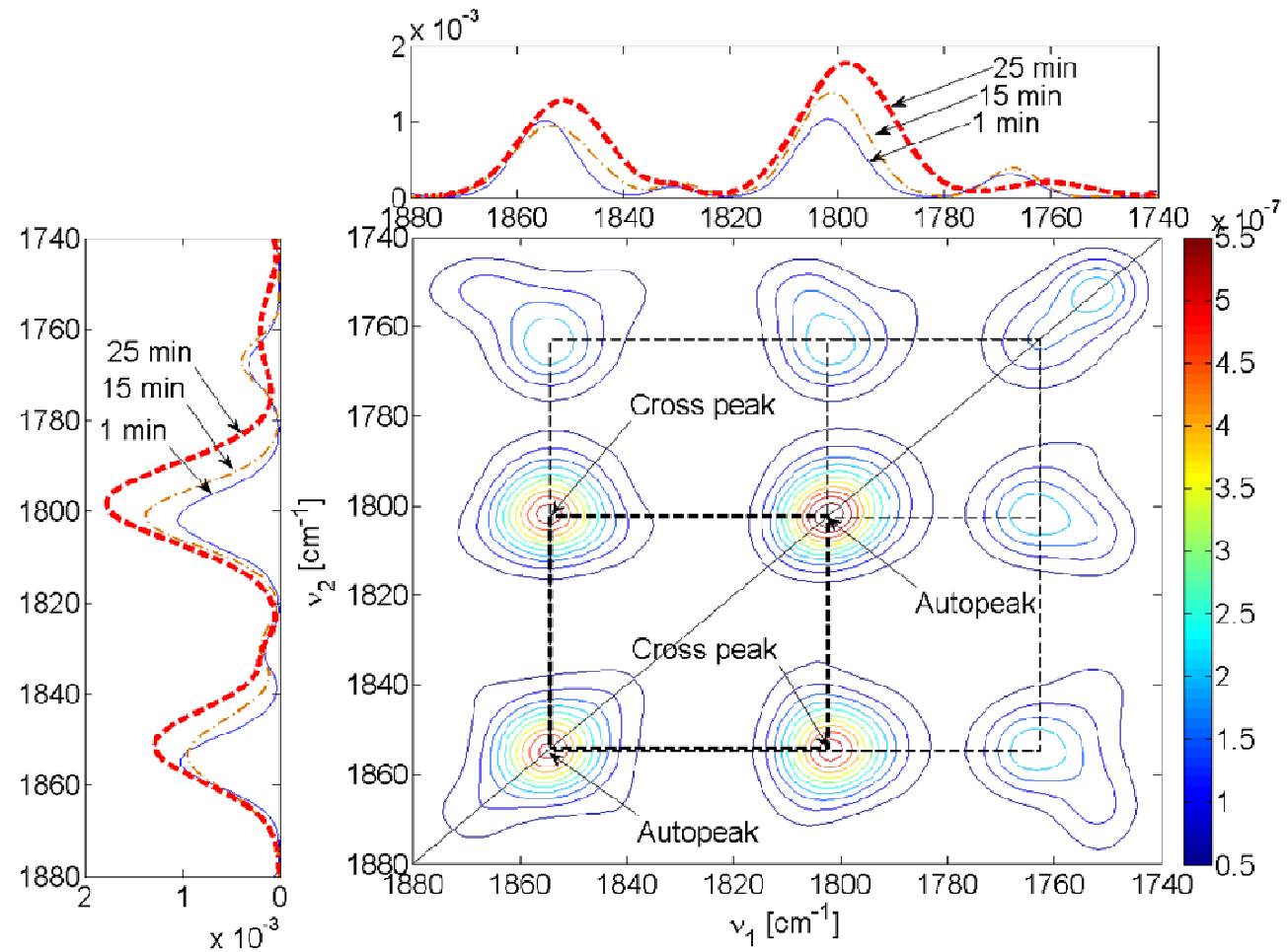


Binder ID	Source	True PG RTFO	r <sub>CA</sub> ATRAC
Conn 64-22	Venezuela	74.7	0.9
AAK	Venezuela	73.2	1.4
AAM	W Texas	70.8	2.6
AAG	Ca. Valley	66.6	3.3
Conn 58-28	Venezuela	61.6	3.7
AAB	Wyoming	61.5	3.9
AAC	Redwater	58.4	4.1
AAD	Ca. Valley	63.4	4.2

**Asfalty o większym średniej masie cząsteczkowej (Mw) starzej się wolniej**



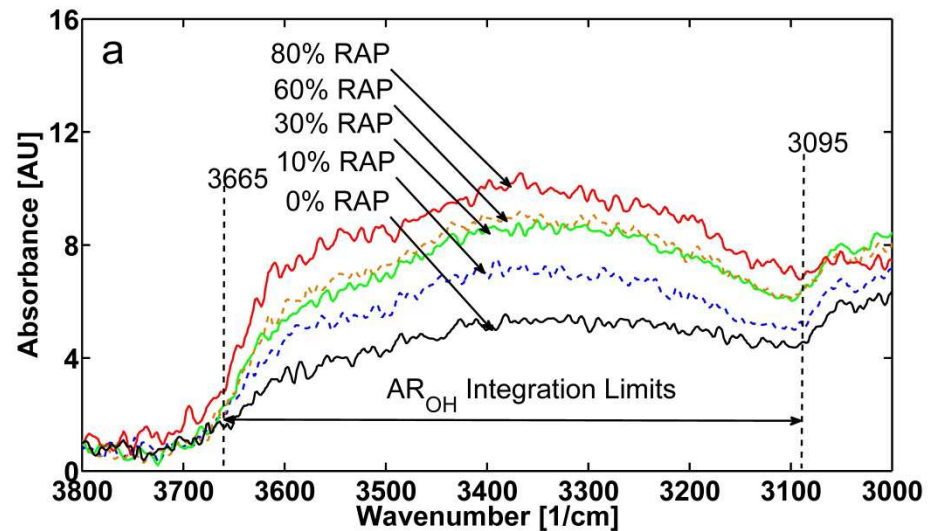
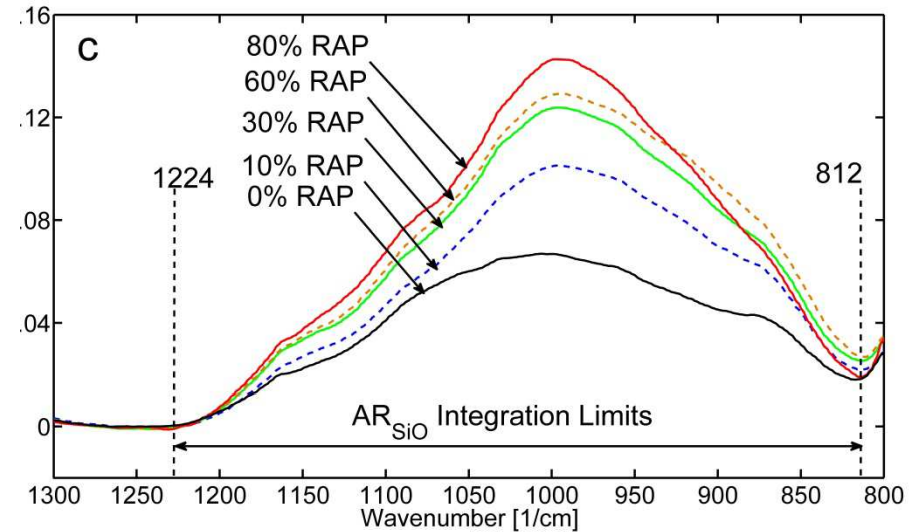
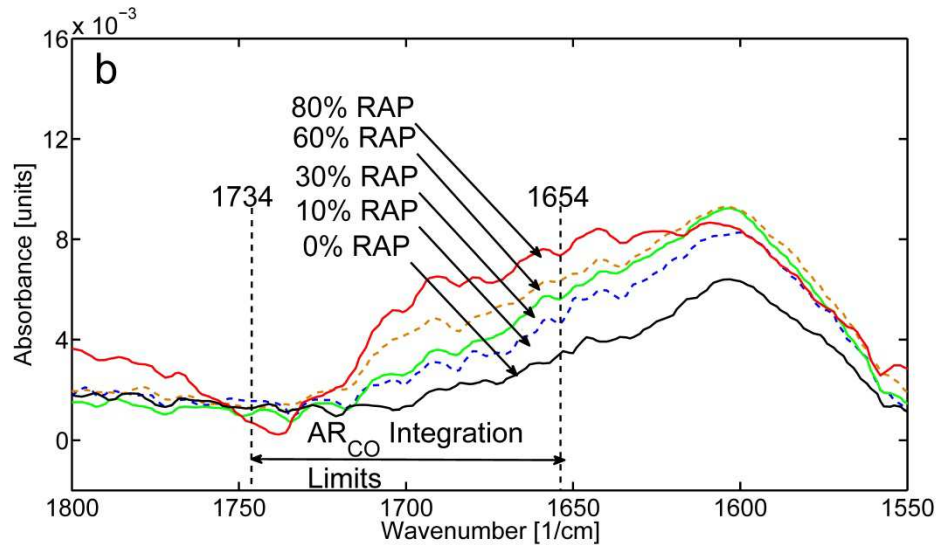
# Przykłady – 2D CS



2-D synchronous

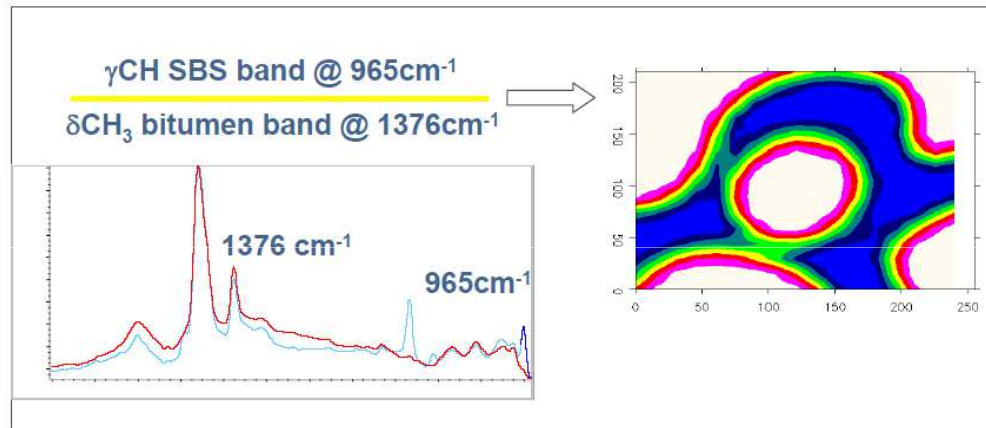
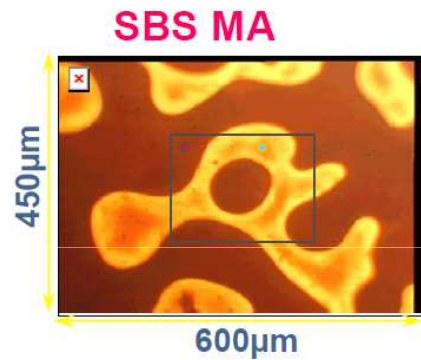


# Przykłady – RAP w lab.

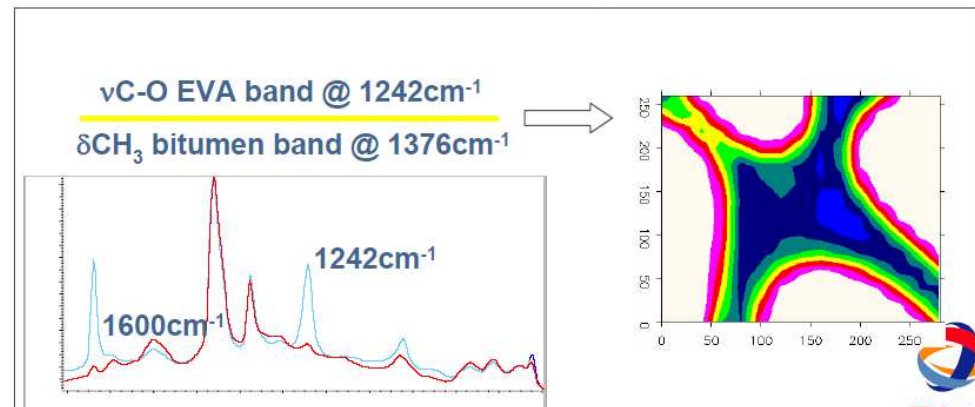
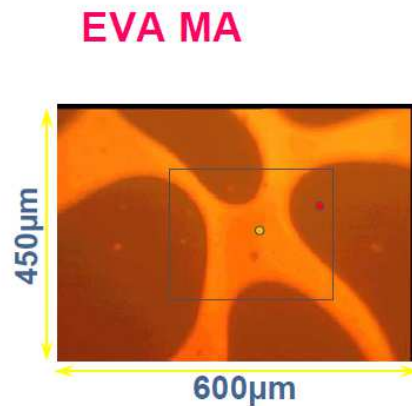


# Možna i tak

## FTIR MICROSCOPY of PmB's



▪Colors = various levels of polymer concentrations



# Abstrakcja?

*DETERMINING POLYMER ADDITIVE PERCENTAGES IN POLYMER MODIFIED ASPHALT CEMENTS*

*TXDOT DESIGNATION: TEX-533-C*

Test Procedure for

## **DETERMINING POLYMER ADDITIVE PERCENTAGES IN POLYMER MODIFIED ASPHALT CEMENTS**



**TxDOT Designation: Tex-533-C**

*Effective Date: August 199*

ASTM D5477 - 02

ASTM D5576 - 00(200

ASTM D5594 - 98(200

ASTM D6645 - 01

ASTM E1421 - 99(200

ASTM E1944 - 98(2007

FHWA

FHWA



**Standard Method of Test for**

## **Polymer Content of Polymer-Modified Emulsified Asphalt Residue and Asphalt Binders**

**AASHTO Designation: T 302-05**

**1. SCOPE**

Solvent-Reducible Paints  
Ash By X-Ray Fluorescence  
tenders in Paint by X-Ray Diffraction

n  
ctrometers  
e) Compounds Using an Infrared

Infrared Spectrophotometry  
iolet, Visible, and Near-Infrared

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# FT-IR publikacje/prezentacje

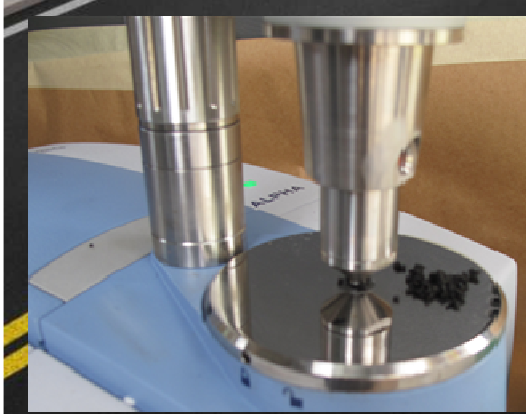
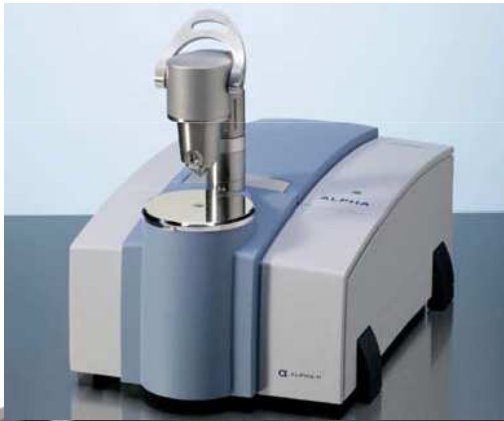
1. Yut I. and Zofka A., Fingerprinting of Chemical Admixtures in Fresh Portland Cement Concrete by Portable Infrared Spectrometer, TRR 2012
2. Yut I., Bernier A., and Zofka A., Development of a Compact Laboratory Aging Procedure for Asphalt Binders, Journal of AAPT, Vol. 81, 2012
3. Bernier A., Zofka A., Yut I., Laboratory Evaluation of Rutting Susceptibility of Polymer-Modified Asphalt Mixtures Containing Recycled Pavements, Construction and Building Materials, Vol. 31, 2012
4. Yut I. and Zofka A., Attenuated Total Reflection (ATR) Fourier Transform Infrared (FT-IR) Spectroscopy of Oxidized Polymer-Modified Bitumens, Applied Spectroscopy, Vol. 65 (7), 2011
5. Zofka A., Yut I., Shaw M., Chrysochoou M. et al., Evaluating Applications of Field Spectroscopy Devices to Fingerprint Commonly Used Construction Materials: Final SHRP2 R06 (B) Report, Transportation Research Board of National Academies, In Press, 2012
6. Yut I., Bernier A., Zofka A., Development of a Compact Laboratory Aging Procedure for Asphalt Binders, AAPT, April 4, 2012, Austin, Texas
7. Zofka A., Chrysochoou M., Yut I., Spectroscopic Evaluation of Recycled Asphalt Pavement Materials, Geo-Congress 2012, Oakland CA, March 27, 2012.
8. Yut I. and Zofka A., Spectroscopic Evaluation of Recycled Asphalt Pavement Materials, Paper 12-1259, TRB 91st Annual Meeting, Washington, DC, January 25, 2012
9. Yut I. and Zofka A., Fingerprinting of Chemical Admixtures in Fresh Portland Cement Concrete by Portable Infrared Spectrometer, Paper 12-1257, TRB 91st Annual Meeting, Washington, D.C., January 23, 2012
10. Zofka A., Yut I., Investigation of Rheology and Aging Properties of Asphalt Binder Modified with Coffee Berries, presented at AFK20 & AFK30 Joint Workshop on Alternative Binders for Sustainable Asphalt Pavements, TRB 91st Annual Meeting, Washington, D.C., January 22, 2012
11. Yut I., A. Bernier, and Zofka A., Spectroscopic Evaluation of Recycled Pavement Materials, 48th Peterson Asphalt Research Conference, Laramie, Wyoming, July 11-13, 2011
12. Yut I., Zofka A., Spectroscopic Evaluation of Oxidative Aging of Polymer-Modified Asphalt Binders: Laboratory Phase, Poster Presentation, New England Asphalt User/Producer Group Annual Meeting, October 6-7, 2010, Saratoga Springs, NY
13. Zofka A., Chrysochoou M., Shaw M., Mahoney J., Farquharson S., Yut I., Sun S., Zhang X., SHRP 2 R06(B): Evaluating Applications of Field Spectroscopy Devices to Fingerprint Commonly Used Construction Materials, NDE/NDT for Highways and Bridges: Structural Materials Technology (SMT) 2010, August 16-20, 2010, New York, New York.
14. Yut I., Zofka A., and Sun S.-P., Investigation of Oxidative Aging of Polymer Modified Binders by Spectroscopy Methods, Presented on 47th Peterson Asphalt Research Conference, Laramie, Wyoming, 12-14 July, 2010
15. Yut I., Zofka A., and Sun S.-P., Spectroscopic Evaluation of Polymer-Modified Hot-Mix Asphalt Mixtures, Presented on 47th Peterson Asphalt Research Conference, Laramie, Wyoming, 12-14 July, 2010
16. Yut, I., Investigation of Aging In Polymer Modified Binders by Using Spectroscopy Methods, Presented on Institute of Transportation Engineers' 6th Annual Student Symposium, April 7, 2010



# Dziękuję za uwagę

Ciekawe czy przyjedzie  
dziś inspektor z ftirem....

Eeeh... Wątpie...  
Może za 100 lat...



Narodowe Archiwum Cyfrowe, sygn. 1-G-7269-9

Budowa drogi Kraków-Mysłowice

Budowa drogi Kraków-Mysłowice. Robotnicy podczas pracy, w głębi widoczna maszyna do walcowania drogi.

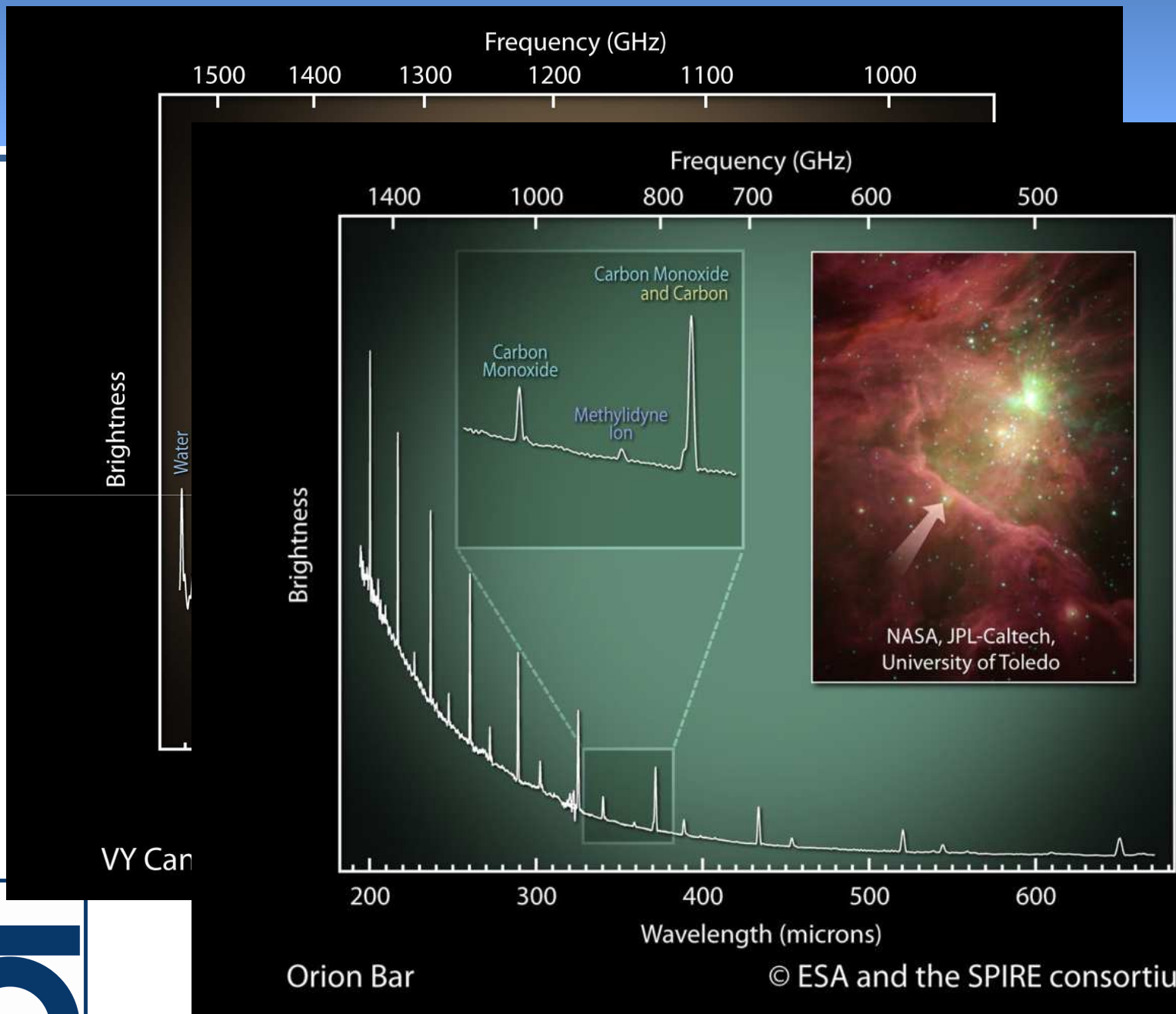
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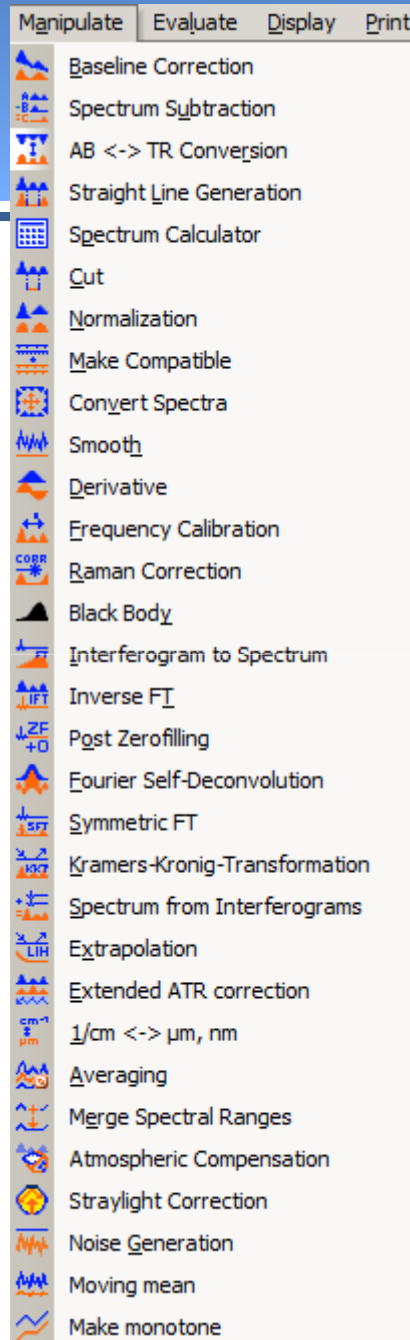
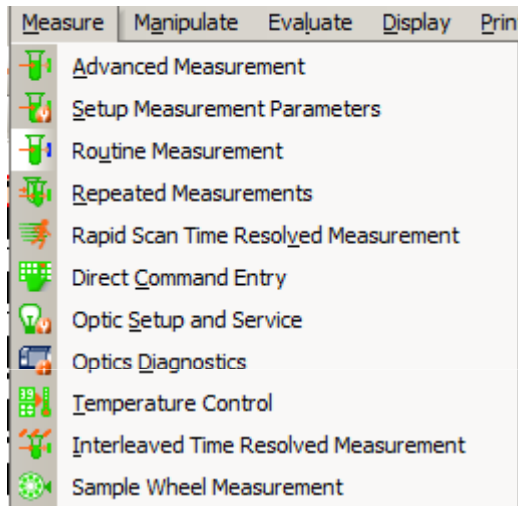
[azofka@ibdim.edu.pl](mailto:azofka@ibdim.edu.pl)

+slides

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meaningful specifications. The current specifications for petroleum asphalt are little more than a quality control exercise for the refiner's vacuum crude tower operations, hence there is little or no distinction among asphalts other than a set of viscosities at two elevated temperatures and a mix plant oxidation susceptibility. That is, all of many asphalts may be classified as, for example, AC-20, and all will be applied as if they are a single material. Yet, their performance characteristics in pavements often vary quite significantly. The results of using poorly classified asphalts have been rude and costly surprises in the form of various types of early failures with increasing frequency. It behooves the user, therefore, to determine what properties of asphalt binders are reflected in pavement performance and then select binders which have desirable performance properties. From a practical viewpoint, no doubt many petroleum residua will require modification to prepare asphalts that have desirable performance characteristics.

