

LABNOTE

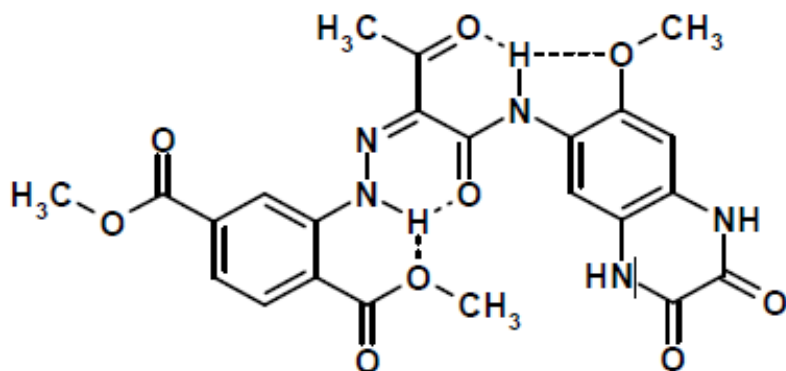
QUALITATIVE PDF ANALYSIS OF PIGMENT YELLOW 213¹⁾, C₂₃H₂₁O₉N₅

QUALITATIVE PDF ANALYSIS OF PIGMENT YELLOW 213¹⁾, C₂₃H₂₁O₉N₅ USING A STOE STADI P LABORATORY DIFFRACTOMETER WITH CU-, MO- OR AG-K_{α1} RADIATION FROM SEALED TUBES AND A DECTRIS MYTHEN 1K DETECTOR

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SETUP & RESULTS

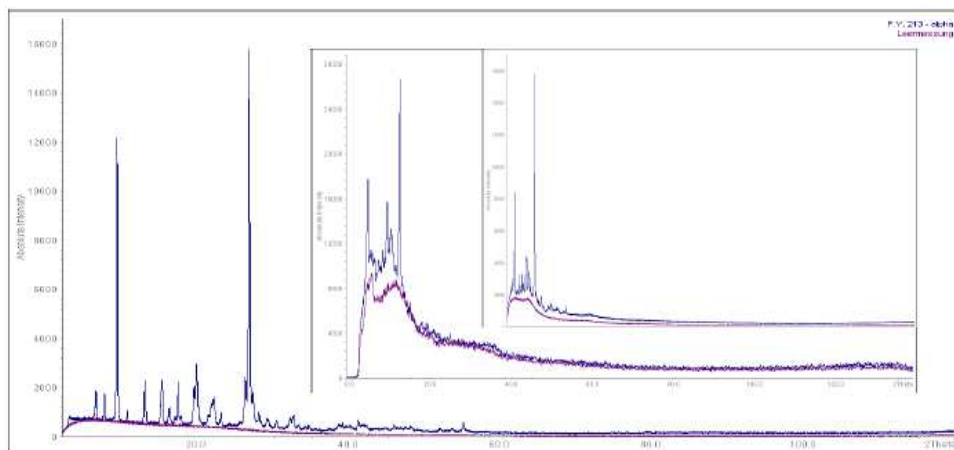
To observe the influence of the wavelength and the detector noise using a laboratory powder diffractometer for the PDF determination and the maximum evaluable Q-value a 1mm glass capillary has been filled with Pigment Yellow 213¹⁾ (PY213) and adjusted on a goniometer head of a Stoe Stadi P powder diffractometer in Debye-Scherrer geometry at the Stoe & Cie application laboratory.



Data has been taken from set-ups with Cu-, Mo- and Ag-anode sealed tubes and the respective Ge(111)-monochromator for pure K_{α1}-radiation as well as a Dectris MYTHEN 1K detector with a wavelength optimised chip (320, 450 or 1000 μm thickness), a linear and an IP-PSD.

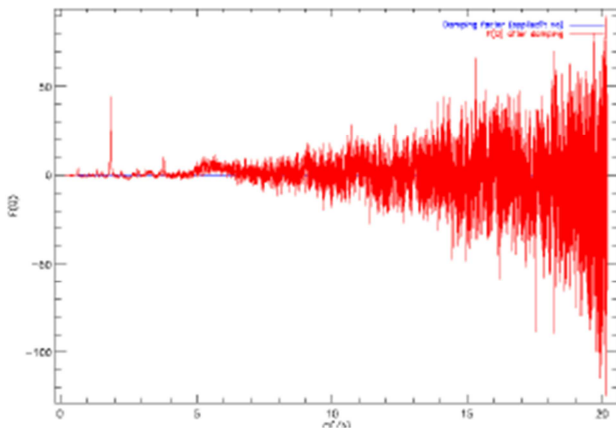
Data evaluation has been carried out by the Dept. of Inorg. Chemistry, Goethe University Frankfurt, using PDFgetX2²⁾.

Picture 1: Pigment Yellow 213, C₂₃H₂₁O₉N₅



Picture 2: Background and powder pattern of PY213 measured with Cu- and lin. PSD, Mo and IP PSD as well as Ag-radiation and MYTHEN 1K (large to small)

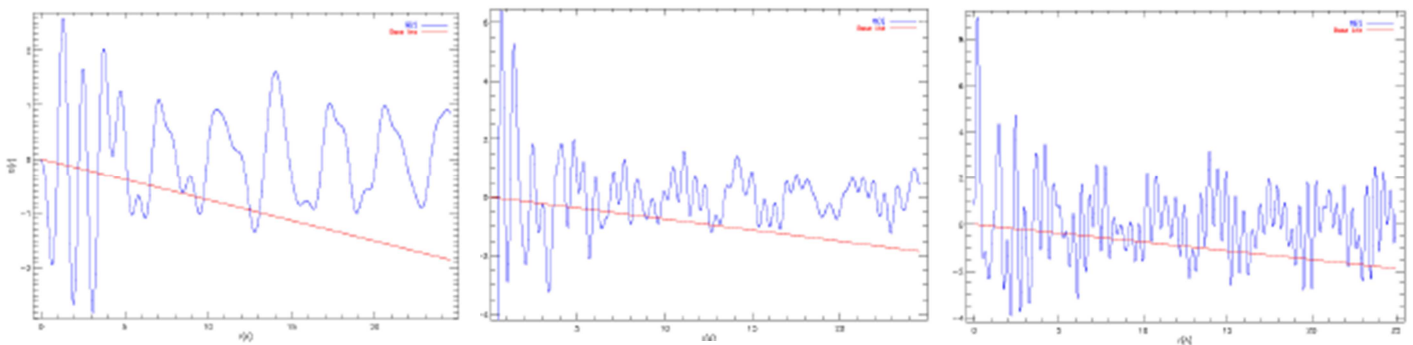
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Picture 3: F(Q) for PY213 with Ag-radiation

A Fourier transformation of the diffraction data $F(Q)$ with $Q=4\delta(\sin\theta/\lambda)$ yields the observed PDF $G(r)$, which shows the probability to find a pair of atoms in the distance r . The maximum theoretical Q value can be directly calculated from 2θ (max) of the goniometer.

For the Stadi P $Q(\text{theo max})$ could be 20 \AA^{-1} for Ag-radiation, but the observed $Q(\text{max})$ is much lower. $G(r)$ yields a $Q(\text{obs max})$ for Cu-radiation of appr. 7 \AA^{-1} , 10 \AA^{-1} for Mo- and 13 \AA^{-1} for Ag-radiation. $Q(\text{obs max})$ for ESRF Synchrotron data with $\lambda = 0.40 \text{ \AA}$ has been 14 \AA^{-1} ³⁾.



Picture 4: G(r) for PY213 measured with Cu-, Mo- and Ag-radiation (left to right)

As expected, the shortest wavelength yields the highest Q (obs max).

Because of the outstanding resolution and the low noise the Dectris MYTHEN 1K is the detector of choice and with the pure Ag $K_{\alpha 1}$ -radiation obtained by the Ge(111) monochromator the Stoe Stadi P powder diffractometer scores as well in XRD as in PDF calculation.



Picture 5: Stoe Stadi P with MYTHEN 1K

- 1) Schmidt, M.U. et al., Acta Cryst. (2009). B65, 189-199
- 2) Qiu, X., Thompson, J.W. & Billinge, S.J.L. J. Appl. Cryst. (2004). 37, 678.
- 3) Presentation of Martin U. Schmidt, EPDIC, Warszawa, Sept. 2008