PPXRD Round Robin



International Centre for Diffraction Data

Fangling Needham, Cyrus Crowder, Timothy Fawcett

This document was presented at PPXRD - Pharmaceutical Powder X-ray Diffraction Symposium

Sponsored by The International Centre for Diffraction Data

This presentation is provided by the International Centre for Diffraction Data in cooperation with the authors and presenters of the PPXRD symposia for the express purpose of educating the scientific community.

All copyrights for the presentation are retained by the original authors.

The ICDD has received permission from the authors to post this material on our website and make the material available for viewing. Usage is restricted for the purposes of education and scientific research.



PPXRD-8 Round Robin - History



Who

Interested in pharmaceutical materials

What

API + Excipient + XRPD Standard

Where

Powder Diffraction Laboratories (Synchrotron)

When

Deadline – before PPXRD-8

Why

Sample/instruments/analysis variables

Who



- PPXRD7: take home specimen attendees
- Post PPXRD7: mailed specimen attendees of past PPXRD organizing committee member synchrotron facility ICDD

What





PPXRD-8 Round Robin (MSDS)



Silicon STABILITY

stable at normal temperatures and pressures CONDITIONS TO AVOID

generating dust, heat,

sparks, open flames

MATERIALS TO AVOID

halogens, metal carbide, metals, oxidizing materials, metal salts, acids

HAZARDOUS

DECOMPOSITION

PRODUCTS

toxic oxides of silicon

HAZARDOUS

POLYMERIZATION

will not occur

Acetaminophen

STABILITY

stable

MATERIALS TO AVOID

oxidizing agents.

HAZARDOUS DECOMPOSITION PRODUCTS

carbon monoxide, carbon dioxide,

nitrogen oxides.

HAZARDOUS POLYMERIZATION

will not occur

Mannitol

STABILITY

stable when keep container tightly closed

in a dry and well-ventilated place

MATERIALS TO AVOID

strong oxidizing agents

HAZARDOUS DECOMPOSITION

PRODUCTS

formed under fire conditions

carbon oxides

PPXRD-8 Round Robin



Quantitative ResultsSpecimen Preparattion

```
Result
Acetaminophen/Mannitol/Silicon
Ratio = / /
Specimen Support
Specimen type and size of support
(Cavity, Zero Background...) =
```

PPXRD-8 Round Robin Instrument



Diffractometer system

Goniometer, Spinner, Radiation type wavelength

Incident beam

Radius (mm) = Voltage (kV) = Current (mA) =

Soller slit size =

Divergence slit

Variable (Y/N) =

or Fixed (size) =

Diffracted beam

Radius (mm) =

Soller slit size =

Anti-scattering slit size =

Filter material thickness =

Detector name/type =

PPXRD-8 Round Robin



Data Collection Analysis

```
Data Collection Software =
Data Analysis Software =
Scan
Scan
Scan range (° ) =
Step size (° ) =
Count Time =
```

Additional Information

Where



Laboratory Diffractometer, Synchrotron

Columbia

Mexico

France

Germany

Czech republic

Israel

Canada

USA

When



(A Few Surprises)

BEGINNIG

Why



