International Tables for Crystallography (2006). Vol. G, Section 3.3.4.3, pp. 119–120.

3.3.4.2. Description of the specimen used in the experiment

The data items in this category are as follows:

PD_SPEC
```
  _pd_spec_description
  _pd_spec_mount_mode
  _pd_spec_mounting
  _pd_spec_orientation
  _pd_spec_preparation
  _pd_spec_shape
  _pd_spec_size_axial
  _pd_spec_size_equat
  _pd_spec_size_thick
  _pd_spec_special_details
```

The PD_SPEC data items describe the specimen used to measure the diffraction data. The data item **_pd_spec_preparation** describes how the specimen that was used to measure the diffraction data was treated, not how the sample was prepared (PD_PREP) or characterized (PD_CHAR).

The PD_SPEC data items are also used to describe how the specimen was mounted for the diffraction experiment. For example, **_pd_spec_mount_mode** and **_pd_spec_orientation** describe the measurement geometry, while **_pd_spec_shape** and **_pd_spec_size_*** describe the specimen shape and size.

3.3.4.3. Instrument calibration and description

The data items in these categories are as follows:

(*a*) PD_CALIB
```
• _pd_calib_detector_id
  _pd_calib_2theta_offset
  _pd_calib_2theta_off_point
  _pd_calib_2theta_off_min
  _pd_calib_2theta_off_max
  _pd_calib_detector_response
  _pd_calib_std_external_block_id
  _pd_calib_std_external_name
  _pd_calib_std_internal_mass_%
  _pd_calib_std_internal_name
```

(*b*) PD_CALIBRATION
```
  _pd_calibration_conversion_eqn
  _pd_calibration_special_details
```

(*c*) PD_INSTR
```
  _pd_instr_2theta_monochr_pre
  _pd_instr_2theta_monochr_post
  _pd_instr_beam_size_ax
  _pd_instr_beam_size_eq
  _pd_instr_cons_illum_flag
  _pd_instr_cons_illum_len
  _pd_instr_dist_src/mono
  _pd_instr_dist_mono/spec
  _pd_instr_dist_src/spec
  _pd_instr_dist_spec/anal
  _pd_instr_dist_anal/detc
  _pd_instr_dist_spec/detc
  _pd_instr_divg_ax_src/mono
  _pd_instr_divg_ax_mono/spec
  _pd_instr_divg_ax_src/spec
  _pd_instr_divg_ax_spec/anal
  _pd_instr_divg_ax_anal/detc
  _pd_instr_divg_ax_spec/detc
  _pd_instr_divg_eq_src/mono
  _pd_instr_divg_eq_mono/spec
  _pd_instr_divg_eq_src/spec
  _pd_instr_divg_eq_spec/anal
  _pd_instr_divg_eq_anal/detc
  _pd_instr_divg_eq_spec/detc
  _pd_instr_geometry
  _pd_instr_location
  _pd_instr_monochr_pre_spec
  _pd_instr_monochr_post_spec
  _pd_instr_slit_ax_src/mono
  _pd_instr_slit_ax_mono/spec
  _pd_instr_slit_ax_src/spec
  _pd_instr_slit_ax_spec/anal
  _pd_instr_slit_ax_anal/detc
  _pd_instr_slit_ax_spec/detc
  _pd_instr_slit_eq_src/mono
  _pd_instr_slit_eq_mono/spec
  _pd_instr_slit_eq_src/spec
  _pd_instr_slit_eq_spec/anal
  _pd_instr_slit_eq_anal/detc
  _pd_instr_slit_eq_spec/detc
  _pd_instr_soller_ax_src/mono
  _pd_instr_soller_ax_mono/spec
  _pd_instr_soller_ax_src/spec
  _pd_instr_soller_ax_spec/anal
  _pd_instr_soller_ax_anal/detc
  _pd_instr_soller_ax_spec/detc
  _pd_instr_soller_eq_src/mono
  _pd_instr_soller_eq_mono/spec
  _pd_instr_soller_eq_src/spec
  _pd_instr_soller_eq_spec/anal
  _pd_instr_soller_eq_anal/detc
  _pd_instr_soller_eq_spec/detc
  _pd_instr_source_size_ax
  _pd_instr_source_size_eq
  _pd_instr_special_details
```

(*d*) Part of PD_DATA
```
  _pd_instr_var_illum_len
```

The bullet (•) indicates a category key.

Calibration information can be placed in the PD_CALIB and PD_CALIBRATION categories. The **_pd_calibration_*** data items are descriptive and will not appear in a loop. The **_pd_calib_*** items may be looped to describe multiple detectors. Correction values for 2θ can be given using the **_pd_calib_2theta_offset** and **_pd_calib_2theta_off_*** data items. A calibration equation can be given using **_pd_calibration_conversion_eqn**. When multiple detectors are used, **_pd_calib_detector_response** is used to indicate the relative performance of each detector. The detector deadtime is specified using the core data item **_diffrn_detector_dtime** (which cannot be looped by detector).

If an internal standard is added to the sample for calibration, this information is specified using **_pd_calib_std_internal_name** to specify the material added and **_pd_calib_std_internal_mass_%** to specify the amount.

When a set of calibration intensities is measured using an external standard, it is possible to include the measurements and the derived results in a separate CIF block. A data block would then use **_pd_calib_std_external_block_id** to link to the block containing the calibration information. See Section 3.3.7 for a discussion of block pointers and block IDs. Note that the use of a unique name for the block ID allows the calibration information to be stored in a separate file, so that the calibration CIF need not be repeated in every CIF that references it.

The PD_INSTR section of the pdCIF dictionary contains terms that describe the instrument used. For example, the instrument or laboratory location is given using **_pd_instr_location**. The instrument type can be indicated using **_pd_instr_geometry**. The instrument geometry can be described in much greater detail using several data items. The geometry is described in terms of four regions of the experiment: radiation source to monochromator (**src/mono**); monochromator to specimen (**mono/spec**); specimen to analyser (**spec/anal**); and analyser to detector (**anal/detc**). If no monochromator is present, the first two regions are combined into radiation source to specimen (**src/spec**). If no analyser is present, the last two regions are combined into specimen to detector (**spec/detc**). Thus two, three or four sets of values describe the dimensions of the instrument and the collimation. For example, **_pd_instr_dist_src/mono** would be used to specify the distance between the radiation source and the monochromator. Alternatively, **_pd_instr_dist_src/spec** would be used to specify the distance between the radiation source and the specimen if no monochromator was present.

Two methods may be used to describe the slits limiting the divergence in the equatorial plane. The angular divergence allowed by the slits in degrees can be specified using the **_pd_instr_divg_eq_*** data items. Alternatively, the dimensions of the slits in the equatorial direction in millimetres may be specified using **_pd_instr_slit_eq_***. The dimensions of these slits in the axial direction, *i.e.* the direction perpendicular to the equatorial plane and containing the incident or diffracted beam as appropriate, are specified in millimetres using **_pd_instr_slit_ax_***. The axial slit lengths, along with the **_pd_instr_dist_*** distances, are useful for estimating the low-angle peak asymmetry (Finger *et al.*, 1994). Note that angular divergence in the axial plane is not a well defined concept for line-focus instruments, but can be specified, where appropriate, using **_pd_instr_divg_ax_***.

The axial and equatorial directions are shown schematically in Fig. 3.3.4.1. The equatorial plane contains the equatorial direction vectors, as well as the incident beam, the diffracted beam and the scattering vector. The axial plane is perpendicular to the equatorial plane and contains the sample centre, which is the point where the incident and diffracted beams meet. For area-detection instruments, the designations of axial and equatorial directions may be arbitrary.

Soller collimators are described using **_pd_instr_soller_eq_*** data items rather than **_pd_instr_ divg_eq_*** data items. It is common practice to specify the Soller collimation in arc-minutes (*e.g.* 30′). However, pdCIF defines these items to have units of degrees, so 30′ would be recorded in the CIF as 0.5. It is not usual to limit the axial divergence, except to reduce low-angle asymmetry, but if this is done, the **_pd_instr_soller_ax_*** data items can be used to define this.

For constant-wavelength instruments, it is common to have a monochromator or filter either before the sample, or after the sample (an analyser), or sometimes both. This is described using **_pd_instr_monochr_pre_spec** and **_pd_instr_monochr_post_spec**. It is rare, but possible, to have both a filter and a monochromator in the same location. The BT-1 neutron powder diffractometer at NIST uses both a Cu(311) monochromator and a graphite filter to attenuate the $\lambda/2$ component. In this case, the two elements would be placed in a loop:

```
loop_  _pd_instr_monochr_pre_spec
  '5 seg. vert. focusing Cu(311) monochromator'
  '3 cm graphite filter'
```

Note that the monochromator and analyser takeoff angles are given using **_pd_instr_2theta_monochr_pre** and **_pd_instr_2theta_monochr_post**. It is useful to record these values for X-ray studies, as they are needed for proper polarization corrections.

In a conventional Bragg–Brentano diffractometer, the divergence slits limit the illumination area at the sample. However, since the φ axis (the sample θ axis) is usually set to bisect the 2θ angle of the detector, the actual length of the area of the sample that is illuminated changes with 2θ. One should choose divergence slits so that the beam does not illuminate areas outside the sample at the lowest diffraction angle used. An alternative method for data collection is to have a divergence slit that opens as 2θ increases, so that a constant area of the sample is illuminated. This is known as a θ-compensating slit. Using a θ-compensating slit provides a better signal-to-noise ratio at larger 2θ values, but means that the diffraction intensities have to be normalized to compensate for the change in illumination. It also introduces greater optical

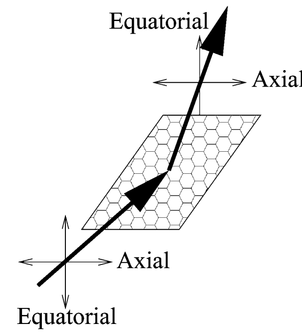

Fig. 3.3.4.1. The axial and equatorial directions in a powder-diffraction experiment.

aberrations with 2θ; a flat plate becomes an increasingly worse approximation to the curved sample geometry in the true Bragg–Brentano geometry.

The use of a variable divergence slit can be recorded in the form:

```
_pd_instr_cons_illum_flag      yes
_pd_instr_cons_illum_len       25.4
```

Note that if **_pd_instr_cons_illum_flag** is not specified, the value is assumed to be `no`, indicating that a fixed-width divergence slit has been used.

The beam size can be specified in two different ways: as the size at the source, using **_pd_instr_source_size_ax** and **_pd_instr_source_size_eq**, or as the size at the sample position, using **_pd_instr_beam_size_ax** and **_pd_instr_beam_size_eq**. Note that the size of the beam at the sample differs from the illumination length described above except when the sample is perpendicular to the beam. When a variable-divergence slit is in use, the beam size at the sample changes with 2θ, so if this size is known directly, the **_pd_instr_beam_size_eq** data item can be included in the loop containing the diffraction intensities. Similarly, in a constant-divergence instrument, where the illumination length changes with 2θ, the illumination length can be specified in the loop using **_pd_instr_var_illum_len**.

There are also several data items in the core CIF dictionary that should be present in the description of the instrument in a pdCIF. Use **_diffrn_radiation_probe** and **_diffrn_radiation_type** to specify the type of radiation used and **_diffrn_detector_type** to specify the detection type.

3.3.4.4. Observations and measurement conditions

The data items in these categories are as follows:

(*a*) Part of PD_DATA

```
  _pd_data_point_id
  _pd_meas_2theta_fixed
  _pd_meas_2theta_range_min
  _pd_meas_2theta_range_max
  _pd_meas_2theta_range_inc
  _pd_meas_2theta_scan
  _pd_meas_angle_2theta
  _pd_meas_angle_chi
  _pd_meas_angle_omega
  _pd_meas_angle_phi
  _pd_meas_counts_total
  _pd_meas_counts_background
  _pd_meas_counts_container
  _pd_meas_counts_monitor
  _pd_meas_datetime_initiated
  _pd_meas_detector_id
        → _pd_calib_detector_id
  _pd_meas_point_id
  _pd_meas_position
  _pd_meas_rocking_angle
  _pd_meas_step_count_time
  _pd_meas_time_of_flight
```


references