

## 3.3. CLASSIFICATION AND USE OF POWDER DIFFRACTION DATA

## 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  $2\theta$  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