`_pd_meas_rocking_angle` is used to record the angular range through which the sample is rocked, where 360 indicates that one complete revolution occurs during each counting period. Numbers greater than 360 are possible.

3.3.8.9. Use of an incident-intensity monitor

For radiation sources for which the intensity may vary, such as synchrotron-radiation sources, the intensity of the incident radiation is measured using an incident-intensity monitor. This value may be specified for every data point using `_pd_meas_counts_monitor` (or `_pd_meas_intensity_monitor`) by including this data item in the loop with the diffraction intensities. For some instruments, counting times are set so that the same number of monitor counts are measured for each data point. If this is the case, `_pd_meas_counts_monitor` will be the same for every data point and need not be included in the loop.

3.3.8.10. Recording detector livetime

The detector deadtime is often more a function of the counting electronics than of the intrinsic properties of the detector. In these circumstances, the counting circuit may provide a gating signal that indicates when the electronics are processing an event *versus* when the circuit is idle and waiting for an event to process. From this gating signal, a detector livetime signal can be generated. Livetime is a better way to correct intensities than applying a deadtime correction, because if appreciable numbers of events are processed but are not counted (for example, counts due to fluorescence), the actual deadtime can be quite high, even though the recorded number of counts can be quite low. To use the livetime signal, the count time can be multiplied by the livetime or the livetime can be treated as a monitor (see Section 3.3.8.9). If an incident-intensity monitor and a livetime are both available, the `_pd_meas_intensity_monitor` value can contain the incident intensity times the livetime.

3.3.9. Use of pdCIF for Rietveld refinement results

One of the major aims of the development of the pdCIF definitions was to be able to communicate the results of Rietveld refinements, and this is expected to be the most common use for pdCIF. To aid the development of software that prepares pdCIF output from Rietveld refinements, this section describes the blocks and loops to be found in a pdCIF, noting variations due to the type of Rietveld refinement. Programmers may also wish to look at the *GSAS2CIF* program, which creates CIFs for a wide range of types of diffraction data, and for multiple data sets and phases (Toby *et al.*, 2003).

It is valuable for the CIF to contain the structural model(s), the observed powder-diffraction intensities and the calculated powder-diffraction intensities so that the fit of the model to the observed diffraction pattern can be viewed graphically. It is the present author's firm belief that it is impossible to judge the quality of a Rietveld refinement by *R* factors or any other numerical metric, since these values describe not just how well the structural model fits the measurements, but also how well the background and peak shape are fitted as well. Very poor models can have good *R* factors and χ^2 values if there is a significant amount of non-Bragg scattering that has been well fitted. On the other hand, with high-resolution observations measured to excellent precision, even trivial imperfections in the peak shapes can result in poor agreement factors. There is no substitute for the visual examination of a plot of the observed and calculated patterns, optimally at more than one magnification level. The program *pdCIFplot* (Toby, 2003) plots the observed and calculated powder-diffraction intensities in a pdCIF and allows the fit to be examined in more detail than can be provided by a figure showing the whole profile at once.

3.3.9.1. A single phase

When a single set of diffraction measurements is used to model a single phase, a pdCIF will usually contain only one block. There will be several important loops present.

One loop will contain atomic parameters, such as coordinates. The unit cell must also be specified.

A second loop will contain the reflection table.

A third loop will contain the observed (or processed) diffraction measurements and the simulated pattern. Other items that should be included in this loop are the least-squares weights (usually σ^{-2}, where σ is the standard uncertainty) so that it is possible to determine the quality of the fit in individual regions. Weight values of zero can also be used to indicate that data points have been excluded from the refinement. Since background fitting is quite important in Rietveld analysis, it is also valuable to include the background values. Thus, this loop should specify:

(i) the ordinate of the Rietveld plot, using one or more of: `_pd_meas_2theta_scan`, `_pd_meas_time_of_flight`, `_pd_proc_2theta_corrected`, `_pd_proc_d_spacing` or `_pd_proc_recip_len_Q`; alternatively the ordinate can be specified using either `_pd_meas_2theta_range_*` or `_pd_proc_2theta_range_*`, where `_*` is `_min`, `_max` and `_inc` outside the loop.

It is recommended that all CIFs describing the results of a Rietveld refinement include either `_pd_proc_d_spacing` or `_pd_proc_recip_len_Q`.

(ii) The observed (or processed) intensity values, using the items `_pd_meas_counts_total`, `_pd_meas_intensity_total`, `_pd_proc_intensity_total` or `_pd_proc_intensity_net`.

(iii) The background, using the item `_pd_proc_intensity_bkg_calc`.

(iv) The least-squares weights, using the item `_pd_proc_ls_weight`. If these weights are not specified, then it must be presumed that all points have been used in the refinement and that the weights are the reciprocal of the intensity values (if `_pd_meas_counts_total` was used) or the reciprocal of the intensity standard uncertainties, if specified.

(v) The calculated pattern should appear using either `_pd_calc_intensity_net` or `_pd_calc_intensity_total`.

It is good practice always to include at least one data item from each entry in the list above.

Apart from the information contained in these loops, information from almost all sections of the pdCIF dictionary can be valuable. Such items include data items that define how the diffraction measurements were made, how the sample was prepared and characterized, how the refinement was performed, and least-squares parameters and *R* factors. A template and an example pdCIF showing the combined use of pdCIF and core data items form part of the *Acta Crystallographica* instructions for authors at http://journals.iucr.org/services/cif/powder.html.

3.3.9.2. Multiple phases

When more than one phase is present, multiple CIF blocks are needed. The resulting CIF will contain much the same information as would be found in a single-phase pdCIF, as described in Section 3.3.9.1. However, there will be a separate block for each phase containing information specific to that phase, such as the unit cell and the loop containing the atomic parameters.

The CIF will usually (see Section 3.3.7) contain one additional block with the observed and calculated pattern and a reflection table, as well as the other data items that define how the

diffraction measurements were made, how the refinement was performed *etc.* While reflection tables for each phase can be placed in each phase block, it is better to include a single reflection table in the block that contains the diffraction data. This block will also contain a phase table that uses the block pointer `_pd_block_diffractogram_id` to link to the phase blocks. The phase blocks can also be linked to the data block using the block pointer `_pd_phase_block_id`. For most Rietveld refinements, each phase is allowed to have different profile parameters, so `_pd_proc_ls_profile_function` should also be included in the phase-table loop.

3.3.9.3. One phase, multiple sets of measurements

It is fairly common to use more than one diffraction data set to determine a model for a single phase. Some examples include: combined refinement using both neutron and X-ray powder diffraction; use of multiple X-ray wavelengths to make use of anomalous dispersion; and the use of single-crystal X-ray and powder neutron diffraction data in a single refinement. For these cases, there will be a CIF block for each data set. Each of these blocks will contain a reflection table and a loop with the observed and calculated diffraction intensities, as described in Section 3.3.9.1.

As explained in Section 3.3.7, the resulting structural parameters could be placed in a block with one of the sets of diffraction data. However, it is better practice to create one additional block for these parameters, as it then becomes clear that the result is from a combined refinement. This is indicated by linking the phase block and the data-set blocks using a loop of `_pd_block_diffractogram_id` values in the phase block. The data-set blocks can also have a link to the phase information using the block pointer `_pd_phase_block_id`.

3.3.9.4. Multiple sets of measurements and phases

Multiple data sets may be used for mixtures as well as single phases. This is becoming increasingly common as more complex materials are studied using powder diffraction. The treatment of this case follows logically from that of Sections 3.3.9.2 and 3.3.9.3. If there are M diffraction data sets and P phases, there will be P blocks containing the crystallographic parameters for each phase. There will be M blocks with the observed and calculated diffraction intensities, as well as reflection tables. Depending on the Rietveld software, there may be $M \times P$ sets of some parameters, for example phase fractions and profile descriptions. These parameters may be placed in the phase table loop within the data-set block(s).

Ideally, the same specimen, or at least the same sample, will be used for all measurements. Sometimes, however, different samples are used for combined refinements to extend the number of observations, despite the possibility that the samples might have slightly different structures or compositions. If there are S samples, there will be an additional S blocks that record the sample and specimen preparation and characterization information. Thus, in this case there will be a total of $M + P + S$ blocks.

As before, the phase blocks will use the block pointers `_pd_block_diffractogram_id` to link to the data-set blocks. Likewise, the data-set blocks will have phase tables with `_pd_phase_block_id` values that link to the phase blocks. The sample blocks can use both `_pd_block_diffractogram_id` values and `_pd_phase_block_id` values to link to the the diffraction data and the analysis results. This is shown in the CIF in Example 3.3.7.1. The program *GSAS2CIF* (Toby *et al.*, 2003) can create CIFs for multiple sets of measurements and phases.

Example 3.3.10.1. *Phase identification using a reflection table and a phase table.*

```
loop_
  _pd_peak_2theta_centroid
  _pd_peak_id
     3 A1
     4 B1
     6 A2
     8 B2
     9 A3
    12 A4
    15 A5
    16 B3

loop_
  _refln_index_h
  _refln_index_k
  _refln_index_l
  _pd_refln_peak_id
  _pd_refln_phase_id
  ? ? ? A1 Phase1
  ? ? ? A2 Phase1
  ? ? ? A3 Phase1
  ? ? ? A4 Phase1
  ? ? ? A5 Phase1

loop_
  _pd_phase_id
  _pd_phase_name
  Phase1  'component 1'
```

3.3.10. Other pdCIF applications

As mentioned above, there are other applications for pdCIF than the storage of unprocessed measurements and the reporting of the results of a Rietveld refinement. This section describes the use of data items in other common pdCIF applications.

3.3.10.1. Simulated intensities

It is common to simulate a diffraction pattern from a known or hypothetical structural model. The structural model is recorded in CIF using core data items, such as `_atom_site_label`, `_atom_site_fract_x`, `_atom_site_fract_y`, `_atom_site_fract_z`, `_atom_site_U_iso_or_equiv` and `_atom_site_occupancy`, as well as the unit cell in `_cell_length_*` and `_cell_angle_*`. Calculated reflection intensities can be recorded using `_refln_index_*` and `_refln_F_squared_calc`, as described in Section 3.3.5.4. The simulated pattern can be recorded using `_pd_calc_*` data items, as described in Section 3.3.5.2.

The simulated diffraction pattern will be determined not only by the structural parameters, but also by the type of experiment that is being simulated. For example, it is good practice to define data items to specify the type of radiation in `_diffrn_radiation_probe`, the wavelength in `_diffrn_radiation_wavelength` and the profile in `_pd_proc_ls_profile_function`.

3.3.10.2. Phase identification and indexing

For phase identification, a CIF will include unprocessed measurements, as described in Section 3.3.8. Sample characterization information, for example chemical analysis information, can often aid phase determination. Characterization information is described in Section 3.3.4.1. Similarly, sample preparation information can also be quite valuable (see Section 3.3.4.1). Since preferred orientation or other artifacts of the measurement can make phase