3. CIF DATA DEFINITION AND CLASSIFICATION

indicate the connection to the initial CIF, since the original block ID is retained.

A potential future use for block pointers may be to reference non-CIF data files that contain large two- and three-dimensional data structures. This is expected to become increasingly important as neutron and synchrotron instruments are constructed that cover increasing ranges of solid angle. As mentioned in Section 3.3.2, CIF is not well suited to these complex, large and possibly irregular measurement arrays. The NeXus format has been developed by a consortium of synchrotron and neutron laboratories to address these concerns and is currently being used for a variety of scattering applications (NeXus, 1999). The NeXus format is based on the platform-independent HDF binary standard (HDF, 1998). The use of block pointers to resolve references to non-CIF documents will require additional definitions.

3.3.8. pdCIF for storing unprocessed measurements

While many researchers prepare a CIF only when a project is complete, there are good reasons for preparing a pdCIF when the diffraction data are measured, as this is the best time to document how the measurement was performed. Much of the instrumental information will remain unchanged for all pdCIFs from a given diffraction instrument, so it is a good idea to prepare a file that describes each of the common settings for an instrument. This file will probably contain some of the following data items and their associated values:

(i) The `_pd_instr_*` items, such as the instrument type in `_pd_instr_geometry`, the size of the instrument and the collimation in `_pd_instr_dist_*` and `_pd_instr_divg_*`, and monochromatization in `_pd_instr_monochr_*` (see Section 3.3.4.3)

(ii) Depending on how the calibration is performed, it may be appropriate to include `_pd_calib_*` items.

(iii) Information about the radiation source should be specified using the `_diffrn_radiation_*` and `_diffrn_source_*` data items.

(iv) Detector information should be specified using `_diffrn_detector_*` items, for example, the detector type in `_diffrn_detector_type` and perhaps calibration values such as the deadtime (in `_diffrn_detector_dtime`).

A second section of the pdCIF will contain information specific to the experiment, such as the diffraction conditions (*i.e.* pressure and temperature) recorded using the `_diffrn_ambient_*` data items. Sample and specimen information will appear in the `_pd_prep_*`, `_pd_spec_*` and `_pd_char_*` data items.

A third section of the pdCIF contains the observations. The data items used to specify the unprocessed observations will vary with the type of instrument used, as described in Sections 3.3.8.1 to 3.3.8.10 below.

3.3.8.1. Single pulse-counting detectors

In the most common measurement method, where a single pulse-counting detector is scanned over a range of 2θ, the `_pd_meas_*` entries (see Section 3.3.4.4) will be of the form shown in Example 3.3.8.1. If the data were scanned using a variable step size, the observations might be given as shown in Example 3.3.8.2. Note that when `_pd_meas_counts_*` is used, the values given must be counts, so that the standard uncertainty will be the square root of the intensity values. This means that the intensity values must not be scaled, for example if the values were counts per second; otherwise the statistical uncertainty estimates will be incorrect.

Example 3.3.8.1. *Measurements from a single pulse-counting detector with constant-step scan.*

```
_pd_meas_2theta_range_min   5.0
_pd_meas_2theta_range_max  65.0
_pd_meas_2theta_range_inc   0.02
_pd_meas_number_of_points  3001
_pd_meas_scan_method        step
_pd_meas_step_count_time    10
loop_
   _pd_meas_counts_total
   10 16 23 18 30 45 58 123 80 67 32 21 12 ...
```

Example 3.3.8.2. *Measurements from a single pulse-counting detector with variable-step scan.*

```
_pd_meas_number_of_points  3001
_pd_meas_scan_method        step
_pd_meas_step_count_time    10
loop_
   _pd_meas_2theta_scan
   _pd_meas_counts_total
   5.00 10 5.02 16 5.04 23 5.06 18 5.07 30 5.08 45
   ... ...
```

3.3.8.2. Detectors that do not count pulses

When the method used to detect intensities does not count individual quanta as they hit the detector, for example, the digitization of intensities recorded on film or on an imaging plate, or even with data recorded using a detector having a built-in deadtime correction, the standard-uncertainty values are not the square root of the intensities. [Note that when the actual deadtime correction is known, it is best to incorporate this scaling into the monitor value (see `_pd_meas_counts_monitor` in Section 3.3.4.4) or else save the uncorrected measurements and create a second set of corrected intensity values as `_pd_proc_intensity_net` (see Section 3.3.5.1).] The `_pd_meas` entries for an experiment using non-pulse-counting detection will look like the examples given in Section 3.3.8.1, except that the data loop will be in the form

```
loop_
   _pd_meas_intensity_total
   10 16 23 18 30 45 58 123 80 67 32 21 12 ...
```

or

```
loop_
   _pd_meas_2theta_scan
   _pd_meas_intensity_total
   5.00 10 5.02 16 5.04 23 5.06 18 5.07 30 5.08 45
   ... ...
```

If standard uncertainties for the intensity values are known, they can be given using the conventional notation

```
loop_
   _pd_meas_2theta_scan
   _pd_meas_intensity_total
   5.00 10(10) 5.02 16(11) 5.04 23(13) 5.06 18(12)
   5.07 30(18) ...
```

Note that when `_pd_meas_intensity_*` is used, it is best to specify `_pd_meas_units_of_intensity` as well.

3.3.8.3. Multiple detectors

At present, CIF does not offer the ability to construct true multi-dimensional data structures. However, many instruments with multiple detectors produce reasonably tractable numbers of data points. For such instruments, it is possible to include an additional data item, `_pd_meas_detector_id`, in the loop with the data to indicate the detector that made the observation.

Example 3.3.8.3. *Identifying intensities from multiple detectors.*

```
loop_
  _pd_meas_2theta_scan
  _pd_meas_intensity_total
  _pd_meas_detector_id
  5.0  10 A   25.0  16 B  45.0 23 C  65.0 18 D
  5.02 16 A   25.02 30 B  ...  ... ...
```

Example 3.3.8.4. *Measurements from an energy-dispersive X-ray diffraction experiment.*

```
_pd_meas_scan_method        disp
_pd_meas_2theta_fixed       6.5
_pd_calib_2theta_offset     0.1071
loop_
  _pd_meas_detector_id
  _pd_meas_counts_total
  _pd_proc_energy_detection
  _pd_proc_recip_len_Q
    0   180   6114.0 .714
    1   166   6141.2 .717
    2    11   6168.4 .721
    3    11   6195.5 .724
```

Example 3.3.8.5. *Measurements from a neutron time-of-flight diffraction experiment.*

```
_pd_meas_scan_method        tof
loop_
  _pd_meas_detector_id
  _pd_meas_time_of_flight
  _pd_proc_d_spacing
  _pd_meas_counts_total
  _pd_proc_intensity_incident
    88  1101.6  0.251658559  11843  4003
    88  1103.2  0.25202477   11934  4001
    88  1104.8  0.252391011  11906  3999
    88  1106.4  0.252757192  11773  3997
    88  1108.0  0.253123432  11707  3995
    ...
    150 1500.0  0.257258087   6559  3185
    ...

loop_
  _pd_calib_detector_id
  _pd_meas_2theta_fixed
    88   88.05
    150 148.29
```

2θ, `_pd_meas_angle_omega` and `_pd_meas_angle_chi` are defined where needed (Example 3.3.8.5).

TOF data are usually reduced to a small number of 'banks' consisting of intensity as a function of d space or Q, where multiple detectors are summed. Data in this form can be recorded using a loop containing `_pd_proc_d_spacing` and `_pd_proc_intensity_net`. A data block is needed for each bank.

3.3.8.6. Digitized film and image plates

To record intensities from digitized X-ray film or from image plates properly requires the storing of two-dimensional data structures, which in some cases can be accommodated through imgCIF (see Chapters 2.3 and 3.7). However, it is possible to record a one-dimensional scan using `_pd_meas_position` and `_pd_meas_intensity_total` (not `_pd_meas_counts_total`!). `_pd_proc_2theta_corrected` values can then be assigned using calibration information, and they can then be included in the same loop, as in Section 3.3.8.4.

3.3.8.7. Direct background measurements

For some diffraction experiments, particularly for the determination of radial distribution functions, measurements are made for background scattering from the diffraction instrument and from the sample container. When this is done, the values can be included in a single loop using `_pd_meas_counts_background`, `_pd_meas_counts_container` and `_pd_meas_counts_total`.

3.3.8.8. Noting sample orientation

For texture measurements, intensity measurements can be made as a function of different sample setting angles. These setting angles can be specified using `_pd_meas_angle_chi`, `_pd_meas_angle_omega` and `_pd_meas_angle_phi`. The change in these values may be specified by including these data items in the loop with the diffraction intensities. In some cases, it may be more convenient to separate measurements with different setting angles into different blocks. In this case, the values for the setting angle(s) that are invariant will be set outside of a loop.

It is common in powder diffraction to reduce preferred orientation and improve crystallite averaging by rocking or rotating the sample. This is indicated by specifying the axis used for rocking, usually φ for capillary specimens or χ for flat-plate specimens, as `_pd_meas_rocking_axis`. The data item

In Example 3.3.8.3, four detectors placed 20° apart are referenced with arbitrarily chosen labels A, B, C and D. Note that the detector characteristics will typically be specified in a separate calibration loop containing terms such as `_pd_calib_detector_id`, `_pd_calib_detector_response` and `_pd_calib_2theta_offset`. The labels given for `_pd_calib_detector_id` should match those in `_pd_meas_detector_id`.

3.3.8.4. Energy-dispersive X-ray detection

For energy-dispersive X-ray diffraction, an X-ray detector is placed at a fixed value of 2θ and a diffractogram is measured on a multichannel analyser. The channel number is then calibrated to yield photon energies. From the energy and 2θ angle, a d-spacing or Q value ($Q = 4\pi \sin\theta/\lambda$) is calculated for each diffraction point. Note that energy, d spacing or Q are not the experimental independent variable. Rather, they result from processing, since calibration information is required. The calibration equation should be described in `_pd_calibration_conversion_eqn`.

In Example 3.3.8.4, the nominal 2θ setting is 6.5°, but the actual position (determined by prior calibration) is 6.6071°, so the difference is indicated using a `_pd_calib_2theta_offset` value (see Section 3.3.4.3).

3.3.8.5. Neutron time-of-flight detection

Neutron time-of-flight (TOF) detection in theory should be no different from energy-dispersive X-ray detection, but TOF instruments record complex three-dimensional data structures, where diffraction intensities are recorded as a function of time for as many as several hundred detectors. For some instruments, both the position along the detector and the time of flight are recorded, so there may be effectively thousands of detectors. To add even further complexity, the data may be binned in different time steps for detectors at different 2θ values. CIF is likely to be cumbersome for the storage of unprocessed measurements from TOF instruments, owing to the one-dimensional nature of CIF, but it could be useful to translate files from one binary format to another using CIF as a common intermediate. To do this, a single loop is used for all data points, where each detector (or detector section, in the case of a position-sensitive detector) is assigned a detector ID. In a second loop, the detector ID values are defined. In addition to