3.2.2.1. Crystal cell parameters and measurement conditions

The categories describing the crystal unit cell and its determination are as follows:

CELL group
- CELL
- CELL_MEASUREMENT_REFLN

The data items in these categories are as follows:

(*a*) CELL

```
_cell_angle_alpha
_cell_angle_beta
_cell_angle_gamma
_cell_formula_units_Z
_cell_length_a
_cell_length_b
_cell_length_c
_cell_measurement_pressure
_cell_measurement_radiation
_cell_measurement_reflns_used
_cell_measurement_temperature
_cell_measurement_theta_max
_cell_measurement_theta_min
_cell_measurement_wavelength
_cell_reciprocal_angle_alpha
_cell_reciprocal_angle_beta
_cell_reciprocal_angle_gamma
_cell_reciprocal_length_a
_cell_reciprocal_length_b
_cell_reciprocal_length_c
_cell_special_details
_cell_volume
```

(*b*) CELL_MEASUREMENT_REFLN

- `_cell_measurement_refln_index_h`
- `_cell_measurement_refln_index_k`
- `_cell_measurement_refln_index_l`

```
_cell_measurement_refln_theta
```

The bullet (•) *indicates a category key.*

The CELL category includes two groups of data names: those characterizing a crystal unit cell, and those describing the experimental conditions relating to the unit-cell determination. It is a feature of the formal definition of the *category* classification unit in CIF dictionaries that these may be classed within the same category, whereas the Miller indices of the reflections used in the measurement of the unit cell belong to a different category. An argument could be made for dividing the CELL category into two categories to reflect the division drawn above between the cell parameters and their determination. However, the CIF dictionaries have been designed to have as few separate categories as possible, subject to the constraint that data items that are looped together in the same list must belong to the same category.

The individual dictionary definitions of the data items in this category are unambiguous, with the possible exception of `_cell_formula_units_Z`, which records the number of complete *chemical* formula units present in the unit cell, and *not* the number of repetitions of the asymmetric unit. In some instances the value of Z could be less than the number of repetitions of the asymmetric unit, such as when an internally symmetric molecular unit is positioned on a symmetry element and spans multiple asymmetric units. Of course, Z can be greater than the number of repetitions of the asymmetric unit (*i.e.* $Z' > 1$).

Note that the value associated with the data item `_cell_volume` is not independent, but can be derived from the other cell parameters. Within the core dictionary there are many cases of derivable items, both because they have traditionally been reported separately and because the presence of redundant information allows cross checking of the internal consistency of the data set.

Data items in the CELL_MEASUREMENT_REFLN category record details about the reflections used to determine the crystallographic cell parameters. The key items in this list are marked with bullets; all three of the h, k, l values are needed to identify a reflection.

3.2.2.2. Data collection

The categories describing data collection are as follows:

DIFFRN group

General description (§3.2.2.2.1)
- DIFFRN

Apparatus and instrumentation before the crystal (§3.2.2.2.2)
- DIFFRN_ATTENUATOR
- DIFFRN_RADIATION
- DIFFRN_RADIATION_WAVELENGTH
- DIFFRN_SOURCE

Apparatus and instrumentation at the crystal (§3.2.2.2.3)
- DIFFRN_MEASUREMENT
- DIFFRN_ORIENT_MATRIX
- DIFFRN_ORIENT_REFLN

Apparatus and instrumentation after the crystal (§3.2.2.2.4)
- DIFFRN_DETECTOR

Intensity measurements (§3.2.2.2.5)
- DIFFRN_REFLN
- DIFFRN_REFLNS
- DIFFRN_REFLNS_CLASS
- DIFFRN_SCALE_GROUP
- DIFFRN_STANDARD_REFLN
- DIFFRN_STANDARDS

The category group related to the diffraction experiment is broad and includes details of the apparatus as well as the measurements. The individual categories are grouped together according to location within the experimental setup (see Fig. 3.2.2.1) or the measurement of intensities.

3.2.2.2.1. *General description*

The data items in this category are as follows:

DIFFRN

```
_diffrn_ambient_environment
_diffrn_ambient_pressure
_diffrn_ambient_pressure_gt
_diffrn_ambient_pressure_lt
_diffrn_ambient_temperature
_diffrn_ambient_temperature_gt
_diffrn_ambient_temperature_lt
_diffrn_crystal_treatment
_diffrn_measured_fraction_theta_full
_diffrn_measured_fraction_theta_max
_diffrn_special_details
_diffrn_symmetry_description
```

These data items give an overview of the diffraction experiment. They are intended to be independent of the instrument, techniques or methodology of the experiment.

The items describing the ambient environmental conditions are reasonably self-explanatory. They are often absent from a CIF, because an author has not thought it necessary to provide

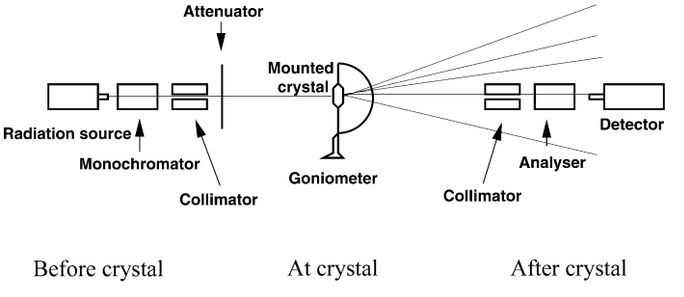

Fig. 3.2.2.1. Scheme of a diffraction experiment.

Example 3.2.2.1. *Attenuation of reflection intensities indicated by reference to attenuator scaling factors.*

```
loop_
     _diffrn_attenuator_code
     _diffrn_attenuator_material
     _diffrn_attenuator_scale
          1   Zr   16.976

loop_
     _diffrn_refln_index_h
     _diffrn_refln_index_k
     _diffrn_refln_index_l
     _diffrn_refln_attenuator_code
     _diffrn_refln_angle_chi
     _diffrn_refln_scan_rate
     _diffrn_refln_counts_bg_1
     _diffrn_refln_counts_total
     _diffrn_refln_counts_bg_2
     _diffrn_refln_scan_width
     _diffrn_refln_elapsed_time

0  0 -16  .   0.0 4.12  28 127  36 1.516 19.43
3  4  -4  1   0.0 1.03  69 459  73 1.516 2082.58
```

information for experiments conducted under 'normal' conditions of room temperature and pressure, and in a standard atmosphere. However, 'normal room temperature' may span a range of many degrees Kelvin and might have a non-negligible effect upon cell dimension measurements, so the temperature should be given. As there is significant variability in the ambient temperature at which laboratory experiments may be carried out, it is not appropriate to assign a default value for **_diffrn_ambient_temperature**, since any numeric value chosen as a default could be misconstrued as an experimentally determined value. If the ambient temperature has not been measured, an author may supply a best estimate of the ambient temperature with a suitable standard uncertainty. Alternatively, known upper and lower limits for the temperature may be given using **_diffrn_ambient_temperature_lt** and ***_gt**. The same considerations hold true for ambient pressure.

The default for **_diffrn_ambient_environment** may be understood as 'air', although formally it is impossible in the dictionary to specify a default for a free-text field.

The **_diffrn_measured_fraction_theta_*** items are provided in this category as an indication of the completeness of a set of reflection measurements. They are not as general as the other items in this category, as they apply only to monochromatic X-ray diffraction experiments, and they do not reflect the way macromolecular crystallographers tend to analyse the completeness of a data set as a function of resolution. When used, they must be accompanied by the value of the monochromatic radiation wavelength **_diffrn_radiation_wavelength** and relate to the maximum θ angle for which the measured reflection count is considered as complete (**_diffrn_reflns_theta_full**).

The other textual data items are provided for comment on other aspects of the handling of the crystal prior to the intensity measurement (**_diffrn_crystal_treatment**), observations on the diffraction point symmetry, systematic absences and inferred space group or superspace group relationships (**_diffrn_symmetry_description**) and any other comment on the intensity measurement process as a whole that cannot be accommodated elsewhere (**_diffrn_special_details**).

3.2.2.2.2. *Apparatus and instrumentation before the crystal*

The data items in these categories are as follows:

(*a*) DIFFRN_ATTENUATOR
- **_diffrn_attenuator_code**

_diffrn_attenuator_material
_diffrn_attenuator_scale

(*b*) DIFFRN_RADIATION
_diffrn_radiation_collimation
_diffrn_radiation_filter_edge
_diffrn_radiation_inhomogeneity
_diffrn_radiation_monochromator
_diffrn_radiation_polarisn_norm
_diffrn_radiation_polarisn_ratio
_diffrn_radiation_probe
_diffrn_radiation_type
_diffrn_radiation_xray_symbol

(*c*) DIFFRN_RADIATION_WAVELENGTH
- **_diffrn_radiation_wavelength_id**

_diffrn_radiation_wavelength
_diffrn_radiation_wavelength_wt

(*d*) DIFFRN_SOURCE
† **_diffrn_radiation_source**
_diffrn_source
_diffrn_source_current
_diffrn_source_details
_diffrn_source_power
_diffrn_source_size
_diffrn_source_take-off_angle
_diffrn_source_target
_diffrn_source_type
_diffrn_source_voltage

The bullet (•) indicates a category key. The dagger (†) indicates a deprecated item, which should not be used in the creation of new CIFs.

Attenuator properties are described by data items in the DIFFRN_ATTENUATOR category. Where an attenuator is used to reduce the intensity of an X-ray beam, this category may be used to describe the attenuator and its scaling factor. Details of multiple attenuator settings or materials can be included and each is identified by a code. A matching code value (**_diffrn_refln_attenuator_code**) appears in the list of intensities against each reflection that must be scaled by the appropriate attenuation factor. In Example 3.2.2.1, the intensity of the second reflection has been reduced using a zirconium attenuator and must be multiplied by 16.976 to place it on the same scale as the first (and other unattenuated intensities).

The DIFFRN_RADIATION category describes the radiation used in the diffraction experiment and its experimental handling by collimation and monochromatization before it interacts with the sample. [Post-sample treatment of the radiation beam after diffraction (including passage through any analyser or collimator) is described by data items in the complementary DIFFRN_DETECTOR category.] Many of the data items in this category are descriptive. Additional information about the generation of the radiation is also found in the DIFFRN_SOURCE category.

The use of **_diffrn_radiation_probe** is strongly recommended as an unambiguous indicator of the probing radiation or particle type (its permitted values are `x-ray`, `neutron`, `electron` and `gamma`). The similar-sounding data name **_diffrn_radiation_type** allows for a more detailed description of the radiation type, such as `white-beam` or (using the CIF code for the Greek character α, \a) `'Cu K\a'` for copper $K\alpha$ radiation. In the case of monochromatic (or near-monochromatic) X-radiation, a better representation is given by the use of **_diffrn_radiation_xray_symbol**, which can have one of a limited number of values expressing the X-ray wavelength according to IUPAC conventions (*e.g.* K-L_3, corresponding to the older Siegbahn notation $K\alpha_1$). If this data item is used, the element used as the X-ray generator target must also be specified using the data item **_diffrn_source_target**. Software for reading CIFs should be aware of these two alternative representations.

If the radiation beam is monochromatic, the wavelength can be provided using **_diffrn_radiation_wavelength**.