Example 3.6.5.3. *The crystal used in the determination of an HIV-1 protease structure (PDB 5HVP) described using data items in the* EXPTL *and* EXPTL_CRYSTAL *categories.*

```
_exptl.entry_id                    '5HVP'
_exptl.crystals_number             1
_exptl.method     'single-crystal x-ray diffraction'
_exptl.method_details
; graphite monochromatized Cu K(alpha) fixed tube
  and Siemens multiwire detector used
;
_exptl_crystal.id                  1
_exptl_crystal.colour              'colorless'
_exptl_crystal.density_percent_sol0.57
_exptl_crystal.description        'rectangular plate'
_exptl_crystal.size_max            0.30
_exptl_crystal.size_mid            0.20
_exptl_crystal.size_min            0.05
```

3.6.5.3.2. *Crystal growth*

The data items in these categories are as follows:

(*a*) EXPTL_CRYSTAL_GROW

```
• _exptl_crystal_grow.crystal_id
        → _exptl_crystal.id
  _exptl_crystal_grow.apparatus
  _exptl_crystal_grow.atmosphere
  _exptl_crystal_grow.details
  _exptl_crystal_grow.method
  _exptl_crystal_grow.method_ref
  _exptl_crystal_grow.pH
+ _exptl_crystal_grow.pressure
  _exptl_crystal_grow.seeding
  _exptl_crystal_grow.seeding_ref
+ _exptl_crystal_grow.temp
  _exptl_crystal_grow.temp_details
  _exptl_crystal_grow.time
```

(*b*) EXPTL_CRYSTAL_GROW_COMP

```
• _exptl_crystal_grow_comp.crystal_id
        → _exptl_crystal.id
• _exptl_crystal_grow_comp.id
  _exptl_crystal_grow_comp.conc
  _exptl_crystal_grow_comp.details
  _exptl_crystal_grow_comp.name
  _exptl_crystal_grow_comp.sol_id
  _exptl_crystal_grow_comp.volume
```

The bullet (•) *indicates a category key. Where multiple items within a category are marked with a bullet, they must be taken together to form a compound key. The arrow* (→) *is a reference to a parent data item. Data items marked with a plus* (+) *have companion data names for the standard uncertainty in the reported value, formed by appending the string* `_esd` *to the data name listed.*

Crystallization strategies and protocols are very varied and may not lend themselves to a formal tabulation. Common or well defined techniques may be indicated using the data item `_exptl_crystal_grow.method`, and a literature reference, where appropriate, may be given using `_exptl_crystal_grow.method_ref`. Frequently, however, a detailed description of methodology is required; this can be given in `_exptl_crystal_grow.details`. Example 3.6.5.4 shows how information about strategies that were attempted and proved unsuccessful can be recorded. In circumstances such as this, the data item `_exptl_crystal_grow.pH` would record the final pH.

Where the crystallization protocol is well defined, it is useful to list the individual components of the solution in the category EXPTL_CRYSTAL_GROW_COMP. Example 3.6.5.4 labels the solutions used as 1 and 2, in accordance with the convention that solution 1 contains the molecule to be crystallized and solution 2 (and if necessary additional solutions) contains the precipitant. However, it is permissible and may be preferable to use more explicit labels such as 'well solution' in the `_exptl_crystal_grow_comp.sol_id` field.

Example 3.6.5.4. *The growth of HIV-1 protease crystals (PDB 5HVP) described with data items in the* EXPTL_CRYST_GROW *and* EXPTL_CRYSTAL_GROW_COMP *categories.*

```
_exptl_crystal_grow.crystal_id     1
_exptl_crystal_grow.method         'hanging drop'
_exptl_crystal_grow.apparatus      'Linbro plates'
_exptl_crystal_grow.atmosphere     'room air'
_exptl_crystal_grow.pH             4.7
_exptl_crystal_grow.temp           18(3)
_exptl_crystal_grow.time    'approximately 2 days'
_exptl_crystal_grow.details
; The dependence on pH for successful crystal growth
  is very sharp. At pH 7.4 only showers of tiny
  crystals grew, at pH 7.5 well formed single
  crystals grew, at pH 7.6 no crystallization
  occurred at all.
;
loop_
  _exptl_crystal_grow_comp.crystal_id
  _exptl_crystal_grow_comp.id
  _exptl_crystal_grow_comp.sol_id
  _exptl_crystal_grow_comp.name
  _exptl_crystal_grow_comp.volume
  _exptl_crystal_grow_comp.conc
  _exptl_crystal_grow_comp.details
  1  1  1  'HIV-1 protease'  '0.002 ml'  '6 mg/ml'
; The protein solution was in a buffer containing
  25 mM NaCl, 100 mM NaMES/MES buffer, pH 7.5,
  3 mM NaAzide
;
  1  2  2  'NaCl'  '0.200 ml'  '4 M'
  'in 3 mM NaAzide'
  1  3  2  'Acetic Acid'  '0.047 ml'  '100 mM'
  'in 3 mM NaAzide'
  1  4  2  'Na Acetate'  '0.053 ml'  '100 mM'
; in 3 mM NaAzide. Buffer components were mixed
  to produce a pH of 4.7 according to a ratio
  calculated from the pKa. The actual pH of
  solution 2 was not measured.
;
  1  5  2  'water'  '0.700 ml'  'neat'
 'in 3 mM NaAzide'
```

3.6.6. Analysis

The mmCIF dictionary contributes several new categories and data items to the REFINE and REFLN category groups. These reflect common practices in macromolecular crystallography in refinement and in the handling of experimental observations.

A new category group, the PHASING group, has been introduced to provide a structured description of phasing strategies, as macromolecular crystallography differs strongly from small-molecule crystallography in how phases are determined. The data model for phasing in the current version of the mmCIF dictionary cannot describe all approaches to phasing yet. Additions and revisions to the data items in the PHASING group of categories are anticipated in future versions of the dictionary.

3.6.6.1. Phasing

The categories describing phasing are as follows:

PHASING group

Overall description of phasing (§3.6.6.1.1)

PHASING

Phasing via molecular averaging (§3.6.6.1.2)

PHASING_AVERAGING

Phasing via isomorphous replacement (§3.6.6.1.3)

PHASING_ISOMORPHOUS

Phasing via multiple-wavelength anomalous dispersion (§3.6.6.1.4)

PHASING_MAD

PHASING_MAD_CLUST