Data preservation in pressure measurement

Kamil Filip Dziubek
LENS European Laboratory for Nonlinear Spectroscopy, 50019 Sesto Fiorentino, Italy
dziubek@lens.unifi.it

Abstract. Suggestions concerning practical pressure scales are largely focused on the selection of primary calibrants, criteria for candidate reference points and choice of the equations of state. Meanwhile, preserving and archiving data related to pressure measurements need also considerable attention. These data, as well as metadata items, corresponding to the pressure determination method are often missing in publications, making it difficult to assess the soundness of the applied approach and validate the reliability of the results. Even if the relevant information is reported, it can be difficult to track down if buried in the article text or supplementary material. Therefore, only using a consistent standardized format one can conform to the requirements of modern science research data being FAIR, i.e. Findable, Accessible, Interoperable and Reusable. Existing data structures can serve as a good starting point, if properly adapted to host specific information related to the pressure measurement. In particular, essential high-pressure data and metadata can be encompassed within Crystallographic Information Framework (CIF), a widely accepted and robust standard file structure for the archiving and distribution of crystallographic information. This review is thus intended to indicate recommendations for data items to be preserved along with the associated measured pressure values.

1. Introduction
Precise and accurate determination of two basic thermodynamic parameters, temperature and pressure, is crucial for reliable interpretation of experimental data. Despite analogies, however, temperature and pressure metrology are diverse in terms of applied methods and underlying physical basis.

In situ temperature measurement in static spectroscopy and diffraction experiments at non-ambient temperature is usually considered trivial and comes down to the readout of a thermo-sensor located possibly close to the investigated sample. This is significantly different in high-pressure high-temperature studies with laser heating, where the radiation pyrometry is a conventional method of temperature determination and the measurement accuracy depends on a number of factors including the unknown wavelength dependence of sample emissivity, temperature distribution within a sample, and aberrations of optics applied in the experimental setup [1, 2].

On the contrary, the most accurate absolute pressure measurements employing primary pressure standards, based on the direct force/area determination, are usable only in a very limited pressure range, e.g. mercury manometers (up to few hundred bars) or force-balanced piston gauges (up to 2-3 GPa) [3, 4]. Such secondary standards as Bourdon tubes, pressure transducers and manganine wire gauges are employed in hydraulic systems and can also work just in a moderate pressure regime. From a practical
point of view, however, two main types of standards are useful in a broad pressure range in static high-pressure experiments, and as such will be addressed here in detail:

(i) equation-of-state calibrants, which enable to calculate the pressure via the equation of state (EoS) relating pressure \( P \) and specific volume \( V \) (molar volume or a unit-cell volume of a defined structure) at the given temperature \( T \) according to the formula \( p_r = p(V, T) \), or in a more general case the thermal equation of state \( p = p(V, T) \). This volume is usually determined in a diffraction experiment using X-ray or neutron, where the pressure marker is placed next to the sample. Noteworthy, most EoS calibrants are primary standards, exploiting \( p-V \) relationships based on the reduced dynamic compression data, ultrasonic measurements, combination of diffraction with Brillouin scattering or computed EoS. This type of gauges can be used in diamond anvil cells (DACs), but also in such pressure devices as Paris-Edinburgh or large-volume presses, where optical access to the sample is not possible;

(ii) optical sensors, which work on the basis of changes in optical properties in the function of pressure. They include luminescence sensors (e.g. R1 ruby fluorescence line [5]), Raman sensors (e.g. high-frequency edge of the diamond anvil Raman band [6]) or infrared absorption sensors (e.g. asymmetric stretching vibrations of nitrite or nitrate ions [7]). Optical sensor pressure scales are usually calibrated against the EoS standards. The application of such gauges is limited to pressure devices providing optical access to the sample, such as DACs. Its undeniable advantage is no need for access to X-ray diffraction instrumentation in order to measure the pressure. Moreover, ruby fluorescence systems are versatile and easy to set up on existing equipment like e.g. infrared and Raman spectrometers, facilitating online pressure measurement without moving the sample.

Pressure determination in dynamic compression is an important but separate topic and one outside the scope of this paper. Therefore, data preservation policies regarding pressure measurement in dynamic experiments would deserve further work.

2. Data management and data flow
Defining and developing the strategy of data management in pressure measurement requires a wider perspective. The general agreement is that nowadays the data should meet standards for being FAIR – findable, accessible, interoperable, and reusable [8]. Hence, the strong motivation to tackle the issue of management of primary data in scientific research has sprung up in many fields, including structural biology and chemistry. In 2011 the International Union of Crystallography (IUCr) established the Diffraction Data Deposition Working Group to ‘address the growing calls within the crystallographic community for the deposition of diffraction data images’. Indeed, numerous case studies show that preserving and sharing raw diffraction data in various areas of crystallographic research improve the quality of structural analysis and validation process [9]. Recently, major steps were accomplished toward the implementation of FAIR data policy in macromolecular crystallography publications [10].

While similar strategies in powder diffraction are still in the stage of development and implementation, a concise and thoughtful review by Aranda highlights the main challenges in this field and presents a step-by-step algorithm for data processing and analysis [11]. Following this idea, one can define primary raw data as the signal acquired by the X-ray detectors – either point detectors (such as scintillation or proportional counters), line detectors (like position sensitive or real-time multiple strip detectors) or area detectors (e.g. image plates, multi-wire proportional counters, Charge Coupled Device cameras, Complementary Metal-Oxide-Semiconductor detectors). In considering such ‘raw’ data, however, it should be born in mind that they are often already processed to some extent by in-built detector firmware that applies automatically distortion or flood-field corrections. The raw data are subsequently processed, in most cases involving human participation in the decision making. In case of area detectors, this operation includes detector calibration, applying masks to exclude unwanted regions (e.g. beam stop shadows, overexposed areas, defective pixels), imposing instrument geometry, correcting for the polarization of the X-rays, and finally radially integrating to obtain one-dimensional powder diffraction patterns, usually showing intensity in the function of scattering angle \( 2\theta \). At this stage, such one-dimensional diffractograms (processed raw data in Aranda’s terminology) can be further analyzed (using also some additional data such as wavelength, initial information about the
crystal phases, symmetry and chemical composition) to yield \textit{derived data} (depending upon the purpose of the study: lattice parameters, atomic positions, phase contents and total amorphous content, domain size, microstrain).

This approach can be straightforwardly applied to pressure determination using the EoS calibrants. The unit-cell volume calculated from lattice parameters or even the individual diffraction lines of the standard (\textit{i.e.} derived data), can serve to determine the pressure using a known EoS (in case of non-ambient temperature studies, the temperature should be also provided as an additional input parameter). In this respect, the pressure values are the \textit{final derived data}. It is worth noting that data flow in pressure determination using optical sensors can be described per analogy to the powder diffraction data processing pipeline outlined above. In this regard, the collected luminescence, Raman or infrared spectra are \textit{primary raw data}, while numerical parameters of the specific spectral features (frequency or wavelength corresponding to the peak maximum or inflection point), calculated after background subtraction and curve fitting with appropriate line shape functions, are \textit{derived data}. Substituting these values to the relevant pressure scale formula yields \textit{final derived data}: the calculated pressure. An overview of the whole procedure is shown in Figure 1.

\textbf{Figure 1.} Schematic data flowchart of the pressure determination procedure showing a pathway from primary raw data to final derived data, \textit{i.e.} the calculated pressure. Meshing gears symbolize schematically data processing.

Original raw diffraction data are mostly stored or planned to be stored in large-scale facilities. It is expected that soon the authors of scientific publications will be required to provide a permanent link to the raw data and associated processed data sets pertaining to their articles. This part of the pressure determination procedure with EoS calibrants should be therefore appropriately covered by general recommendations concerning data sharing in crystallography. As regards the optical sensors, repositories and databases focused on preserving raw spectroscopy data are not numerous, although constant progress has been made recently also in this field [12]. However, these resources still do not offer archiving processed data at different levels, including such details as the data processing method (background treatment and the choice of line shape function). Since all the aforementioned issues are more general concerns in the area of crystallographic and spectroscopic research, it has been decided to narrow the scope of this paper to the last step of data processing illustrated in Figure 1, namely the pressure calculations with the diffraction or spectroscopy derived data as an input.

\section{Requirements of data format}
Existing data structures can be conveniently extended to host data and metadata related to pressure measurement. One of the ongoing activities of the IUCr Commission on High Pressure (CHP) is defining essential data descriptors for high pressure research [13]. This can be effectively achieved in practice utilizing the syntax of CIF (Crystallographic Information File) [14], in which each specific piece of information (entry), called a \textit{data item}, is defined by a \textit{data name} (identifier) and an associated \textit{data
value, which can be numerical or textual. The numeric and character data values are distinguished in the base CIF specification, which recognizes four main data types, two of which are relevant to the high pressure research: (i) numb – a decimal base number supplied as an integer, a floating-point number or in scientific notation and (ii) char – a character or text data value (if it contains white spaces, it must be enclosed within single quote characters). Such a structured layout based on the ASCII character tag-value pairs makes CIF files not only human-readable, but also machine-parsable. An important advantage of the CIF concept is also its flexibility and extensibility. Data items are formalized in CIF dictionaries: the central coreCIF and also more specific dictionaries, developed for particular subdomains like macromolecular crystallography, powder diffraction, magnetic structures, electron density studies, etc. New specialized CIF dictionaries are planned to cover other branches of crystallography, including high pressure research. The high-pressure CIF dictionary should contain, among other essential descriptors of experimental details, the specifics of pressure measurement [15]. Currently, only few CIF data names relevant to high pressure studies exist in official dictionaries; they have been listed in Table 1.

| Dictionary | Data name | Definition/data typea |
|------------|-----------|-----------------------|
| coreCIF    | _diffrn_measurement_specimen_support | The physical device used to support the crystal during data collectionb [char]. |
| coreCIF    | _diffrn_ambient_environment | The gas or liquid surrounding the sample, if not airc [char]. |
| coreCIF    | _diffrn_ambient_pressure | The mean hydrostatic pressure in kilopascals at which the intensities were measured [numb]. |
| coreCIF    | _diffrn_ambient_pressure_gt | The mean hydrostatic pressure in kilopascals above which (*_gt) or below which (*_lt) the intensities were measured. These items allow for a pressure range to be given [numb]. |
| coreCIF    | _cell_measurement_pressure | The pressure in kilopascals at which the unit-cell parameters were measured (not the pressure at which the sample was synthesized) [numb]. |
| coreCIF    | _exptl_crystal_pressure_history | Relevant details concerning the pressure history of the sample [char]. |
| coreCIF    | _exptl_crystal_recristallization_method | Describes the method used to recrystallize the sample. Sufficient details should be given for the procedure to be repeated. The temperature or temperatures should be given as well as details of the solvent, flux or carrier gas with concentrations or pressures and ambient atmospheree [char]. |
| mmCIF      | _exptl_crystal_grow.pressure | The ambient pressure in kilopascals at which the crystal was grown [numb]. |
The standard uncertainty (estimated standard deviation) of _exptl_crystalGrow.pressure [numb].

Preparation pressure of the sample in kilopascals. This is particularly important for materials which are metastable at the measurement pressure, _diffrn_ambient_pressure [numb].

The maximum and minimum values of the pressure in kilopascals defining the interval within which the modulation wave vector(s) were measured [numb].

---

### 4. Proposed data descriptors for pressure determination

Here the main goal is defining the minimal set of essential data and metadata guaranteeing reproducibility of the results. For the sake of clarity and coherence, this task was divided into four parts: data for pressure measurements with EoS calibrants, data for pressure measurements with optical sensors, metadata and error analysis.

#### 4.1 Data for pressure measurements with EoS calibrants

The first data item that needs to be addressed is the nature of the gauge material. Since the EoS calibrants are usually either chemical elements or very simple inorganic compounds, the textual data values can be simply defined using their chemical formula, or a name of a specific crystal polymorph to avoid ambiguities, e.g. 'Au', 'Pt', 'Mo', 'cBN' (cubic boron nitride), 'diamond' or 'quartz'. It would be convenient to prepare an extendable list of all the standards to enable syntactic recognition via parsing.

Form, accuracy and the range of applicability of a thermodynamic EoS are central for correct pressure determination. As Anderson and Mamnone rightly pointed out, “an equation of state which is used as a primary pressure standard has to be, like Caesar’s wife, above suspicion” [16]. There are only several forms of EoS conventionally used in extreme pressure regime, like e.g. third-order Birch-Murnaghan [17], Vinet [18] or Holzapfel AP2 (adapted polynomial of second order) [19]. They can be therefore straightforwardly assigned to corresponding data names such as 'BM3', 'Vinet' and 'AP2'. Providing the EoS is given in function of the other two state variables, which is further reduced to $p_T = p_T(V)$ for the isothermal case, its general expression should be included in a definition of the data descriptors. The equilibrium zero-pressure volume at a given temperature $V_0T$, isothermal zero-pressure bulk modulus $K_0T$ and its pressure derivatives, also evaluated at zero pressure ($K_0’T$, $K_0''T$), can be conveniently included as separate data items. Other material-dependent parameters, such as mean electronic density $Z/V_0$ (where $Z$ is the number of electrons per atom and $V_0$ is the molar volume at $p = 0$) used in the Holzapfel AP2 EoS, can be defined analogously.

Accurate determination of pressure at non-ambient temperature is a more challenging task. As a first approximation, the effects of pressure and temperature can be accounted for separately, using a known isothermal EoS after considering the volume thermal expansion defined by $a(T) = V^{-1}(\partial V/\partial T)_p$. This $V$-
$T$ relationship can be regarded as the isobaric EoS. In particular, Fei [20] formulated a polynomial expression for fitting experimental data over a specific temperature range $a(T) = a_0 + a_1 T + a_2 T^2$. An alternative approach is to derive $p$-$V$-$T$ EoS within the frame of the Mie-Grüneisen model. In this way the Debye temperature $\theta_D$, Grüneisen parameter $\gamma$ and its volume dependence coefficient $q$ are optimized by fitting to the compression data at different temperature [21]. In any case, a set of parameters specific for a given pressure gauge ($a_0$, $a_1$, $a_2$ or $\theta_D$, $\gamma Q$) can be defined as separate data items together with the associated $V$-$T$ or $p$-$V$-$T$ relations used to calculate the pressure. The measurement temperature is another thermodynamic variable that needs to be included in the data set. In the coreCIF dictionary its value is coupled to the _diffrn_ambient_temperature data name.

4.2 Data for pressure measurements with optical sensors
Data items associated with pressure determination using optical sensors can be defined analogously to these concerning pressure measurements with EoS calibrants. One of the most significant differences is that the EoS calibrants are always either pure chemical elements or well-defined stoichiometric compounds, while optical gauges are often doped or composite materials, like luminescence sensors ruby (Cr$^{3+}$: Al$_2$O$_3$) [5], Sm$^{3+}$:SrBaO$_7$ [22], Sm$^{3+}$:YAG [23], Sm$^{3+}$:MFCl (M=Sr,Ba) [24], commercial fluorescent polystyrene FluoSpheres® [25] or infrared sensors, e.g. dilute solid solutions of NaNO$_2$ or NaNO$_3$ in NaBr [7]. Hence, since such factors as a concentration of a dopant as well as residual strain generated during preparation of a sensor can affect its optical properties, it is critical to measure the reference value (e.g. wavelength of the R1 ruby fluorescence line at atmospheric pressure, $\lambda_0$) for the same sensor specimen before applying pressure (in an empty DAC, before loading) or at least for another piece of the gauge material coming from the same batch. Relying on default reference values can result in systematic errors, particularly considering that the defaults often differ between software pressure calculators, either these incorporated in laboratory ruby fluorescence systems or available on the web.

Optical gauge pressure scale formulas can be addressed similarly as the EoS data items, with their expressions included in the definitions. A recent proposal worked out by the AIRAPT Task Group on the International Practical Pressure Scale, provides a comprehensive list of published ruby scales [26]. As stated previously, these scales were derived by fitting experimental data of a ruby fluorescence sensor measured along with the EoS standards. The differences are mainly in the selection of EoS standards, criteria for reference points, choice of the EoS, the applied weighting scheme and the form of the fitting equation. The fitting procedure yields two or three fitting parameters (named usually A, B and C), which are constant characteristics of the scale. Therefore, the resulting equations give the pressure as a function of these parameters and two variables: the reference zero-pressure ruby R1 line wavelength $\lambda_0$, and the R1 line wavelength at the measured pressure $\lambda$. Pressure scales for Raman and infrared optical sensors are constructed likewise.

Pressure measurement with optical sensors at non-ambient temperature is usually performed using the room-temperature pressure scales, and additionally the linear or polynomial expressions relating a change in optical properties with temperature difference, e.g. in case of the ruby gauge $\Delta \lambda$ to $\Delta T$ [27]. These expressions, along with the associated coefficients, should be therefore defined as additional data items. It needs to be stressed that these temperature-dependence laws can be valid only over a limited temperature range, with a change of formula and coefficients while moving to a different temperature regime. A similar approach can be applied also for other luminescence and Raman sensors [27]. Alternatively, pressure scales can be determined with various parameters for several isotherms, as in an example of the NaNO$_2$ in NaBr infrared pressure gauge [28].

4.3 Metadata
Metadata, often referred to as ‘data about data’ or ‘information about information’ are data that describe and give information about other data, putting them in a context. One of the specific examples of metadata related to the pressure determination procedure is the analytical technique used to acquire primary data (X-ray or neutron diffraction, luminescence spectroscopy, infrared absorption spectroscopy, Raman scattering). Here belong also experimental details pertinent to the pressure...
determination procedure, like the line shape functions applied in curve fitting, type of the laboratory instrument, X-ray or neutron wavelength, luminescence or Raman excitation line wavelength. Other metadata include the items concerning data processing aimed at reducing raw data or processed raw data to derived data (i.e. the lattice parameters, the unit-cell volume, or in the case of optical sensors the wavelength or the frequency attributed to a characteristic spectral feature). Such information can be found in literature, in the article text, e.g. “the (200) reflection of the B1 phase of NaCl was used for pressure determination” [29] or “the unit-cell parameter of gold was determined by least-squares refinement of five diffraction lines [(111), (200), (220), (311), and (222))]” [30]. These descriptions are indeed textual metadata that could be included in a data set, providing that a relevant data item is defined.

A seemingly trivial, nevertheless significant metadata contextualizing the entire pressure determination procedure are literature references to published EoS and pressure scales used to calculate pressure. This information is usually included in research articles to give credit to the authors of a calibration but representing it as a data item which underpins the journal publication would comply with the FAIR principles.

Another important but often neglected information associated with pressure measurement is a $p$-$T$ working range of EoS and pressure scales. In principle, the calibrant material must be within the elastic behavior regime, but domains of applicability of EoS based on a specific model can be even narrower. This situation is especially exacerbated at high temperature, due to large uncertainties in calculating the thermal pressure and limitations of the working models [31]. Also, optical sensors have a constrained $p$-$T$ window, within which the measurement is reliable. Ruby becomes structurally metastable above 80-100 GPa and its fluorescence signal weakens with pressure [32]. The decrease in the intensity of luminescence and broadening of the spectral bands with temperature is another well-known limiting factor.

The type of the pressure transmitting medium (PTM) is a crucial metadata item. Each PTM has its characteristic hydrostatic limit, beyond which the non-hydrostatic stress is generated [33-34]. Even for helium, considered the best PTM for its wide hydrostaticity range, above 100 GPa the uniaxial pressure components can reach ~1 GPa [35]. Besides, in multiphase solid mixtures one can observe the so-called Lamé effect resulting in erroneous pressure measurements [36]. It depends on the shear modulus of the PTM (matrix) and the difference between the bulk moduli of the pressure marker (inclusion) and the PTM. Temperature annealing of the compressed sample can be effectively used to mitigate this undesirable phenomenon. It is worth mentioning here that the existing coreCIF data items _diffrn_ambient_environment and _exptl_crystal_pressure_history can be used for storing information about the PTM and history of the sample, respectively.

4.4 Error analysis
In the high-pressure research there is a tendency to report statistical uncertainties alone within a single experiment and to neglect the systematic uncertainties associated with the pressure standard. The reasons for this situation are at least twofold: no guidelines and practical recommendations on error analysis, and lack of uncertainty assessment reported along with the EoS or secondary pressure scales. Therefore, careful analysis of the systematic uncertainties associated with the determination of pressure should be conducted in future studies.

The recent proposal for a new practical pressure scale includes the identification and quantitative estimation of principal sources of errors in the $p$-$V$ relation of diamond [26]: the choice of EoS form ($\pm 0.7 \%$), the scatter of $K_{0T}$ ($\pm 0.3 \%$) and the uncertainty of $K_{0T}^{\gamma}$ ($\pm 1 \%$). Convolution of these three terms yields the combined uncertainty in pressure for diamond as the primary EoS calibrant of ($\pm 1.3 \%$). Similar analyses would be desirable for all available pressure markers. Another interesting review on the accuracy of the determined EoS has been provided by Liu and Bi [37].

The uncertainty in pressure determination can be also derived from the propagation of uncertainty law, providing that all the individual uncertainties of EoS parameters ($V_0$, $K_{0T}$, $K_{0T}^{\gamma}$, $K_{0T}^{\gamma}$) are available together with the uncertainty in volume measurement. The effectiveness of this approach rests on the presumption that all the constituent uncertainties are independent and not correlated. In the case of EoS
calibrants the uncertainty in volume measurement is usually assessed from the diffraction experiment. However, in practice no detailed diffraction data analysis, like a Le Bail or Pawley fitting, is undertaken. If the pressure is determined based on one reflection of the standard only, the estimation of an error is virtually impossible. If a higher number of reflections is available, more complete reasoning can be performed [30].

In pressure measurements with optical sensors the errors in the \( p-V \) relationship should be convoluted altogether with that of \( V-\Delta \lambda \) dependence, to obtain the total uncertainty. Such an uncertainty associated with a newly proposed Ruby2020 gauge is estimated to be \( \pm2.5\% \) for the pressure range up to 150 GPa [26]. It is appropriate here to warn against a possible misunderstanding. In the already classical book of Eremets [38] the accuracy of pressure determination using the ruby fluorescence method is estimated as \( \pm0.03 \) GPa, which can be even improved to \( \pm0.01 \) GPa by fitting of the top of the \( R1 \) line [39]. This number is, however, the accuracy of the spectral \( R1 \) line measurement (\( \Delta \lambda \) of 0.01 nm, which corresponds approximately to \( \Delta p \) of 0.03 GPa), not the total uncertainty of the pressure determination, which should include also uncertainty of the ruby scale.

Apart from the accuracy of the diffraction or spectral measurement and the uncertainty of the EoS or pressure scale, other sources of error that may also contribute to the overall cumulative uncertainty should also be considered. As already mentioned, the non-hydrostatic stress [33-34] and other instrumental effects (like the Lamé effect) [36] are among the key factors to be addressed. Another source of systematic uncertainty may be related to a strain generated in a pressure gauge in non-hydrostatic conditions, which exhibits hysteresis and does not return to its initial value on pressure release, causing discrepancies between the pressure values measured in compression and decompression runs. It has been also noted that the ruby \( R1 \) fluorescence line shifts considerably with temperature. In the room temperature range, the temperature difference of 5 K results in the same \( \Delta \lambda \) shift as the pressure difference of \( \pm0.1 \) GPa. Therefore, any variation of temperature during the experiment and any shift from the measurement temperature of the reference \( p=0 \) wavelength, should be taken into account. In high- or low- temperature measurements exploiting thermal EoS or substantial temperature corrections, an accuracy of temperature determination should be appropriately included in error analysis. Furthermore, for experiments with membrane DACs, a pressure drift usually occurs during the data acquisition. Even after some stabilization time, the drift may remain at the level of \( \pm0.5 \) GPa, comparable to the uncertainty of the pressure measurement [40]. Hence, it is recommended to record the initial \( (p_i) \) and final \( (p_f) \) pressure value, at the beginning and just after the sample data collection and report the pressure as the arithmetic mean of \( p_i \) and \( p_f \), properly addressing the propagation of uncertainty.

From a data viewpoint, standard uncertainties of pressure and all other numerical parameters (including \( V_0, K_{0T}, K'_{0T}, K''_{0T} \), the temperature, parameters of the optical pressure scales) can be simply associated with a parent data value. Any numerical CIF data value can contain an appended standard uncertainty number enclosed within parentheses.

5 Summary and perspectives

In the preceding section, the bare minimum of data and metadata has been identified as fundamental for full interpretation of pressure measurement in static experiments. For the sake of clarity, this set is summarized in Table 2.

### Table 2. Proposed data items relevant to pressure measurement.

| Data item(s)                  | Description                                                                                                                                   | Data type^a |
|-------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------|-------------|
| Pressure gauge material       | Data value can be straightforwardly defined using a chemical [char] formula, a name of a crystal polymorph, a common name, or a combination of a dopant and a host material name e.g. 'Au', 'Pt', 'Mo', 'cBN' (cubic boron nitride), 'diamond', 'quartz', 'ruby', 'Sm2+:SrB4O7', 'Sm3+:YAG', 'FluoSphere', 'NaNO2:NaBr'. |             |
Pressure measurement technique
Laboratory method used for pressure measurement: 'XRD' (X-ray diffraction), 'neutron' (neutron diffraction), 'luminescence' (luminescence spectroscopy), 'Raman' (Raman scattering), 'IR' (infrared spectroscopy).

Pressure measurement experimental details
Description of experimental details relevant to the laboratory technique (e.g. X-ray or neutron wavelength, luminescence or Raman excitation line wavelength, type of analytical instrument).

Pressure measurement data processing details
Description of data processing (software used in data processing, line shape functions applied in curve fitting, the number and Bragg indices of diffraction lines used for the determination of lattice parameters).

Lattice parameters and unit cell volume
Lattice parameters and/or unit cell volume as determined by X-ray or neutron diffraction.

Spectral characteristics
Characteristic values used for pressure determination (R1 ruby fluorescence line, high-frequency edge of the diamond anvil Raman band, characteristic infrared absorption frequencies).

Reference spectral characteristics
Characteristic values used for pressure determination at atmospheric pressure ($p = 0$).

EoS or pressure scale form
For example, 'BM3' (third-order Birch-Murnaghan) or 'Vinet'. The analytical form of the EoS or the pressure scale used for pressure determination should be provided in the definition.

EoS or pressure scale parameters
Numerical parameters of the EoS ($V_0, K_{0T}, K_{0T}', K_{0T}''$), thermal EoS ($\theta_0, \gamma_0 q$) or the pressure scale (A, B, C).

Temperature correction form
The temperature correction form should be provided in the definition (the set of parameters may be included).

Literature references
References to published EoS and pressure scales.

PTM
PTM used in the experiment.

EoS or pressure scale working range
$p$-$T$ window within which the measurement is reliable.

Measurement $T$
Temperature of the pressure sensor.

Reference measurement $T$
Temperature of the pressure sensor at $p = 0$ (associated with the reference spectral characteristics).

Pressure
Pressure determined using the applied EoS or pressure scale.

\(^a\) Reported in square parentheses.

Two examples of pressure measurement with an EoS calibrant and an optical sensor are presented in Table 3 to illustrate how the proposal outlined above would work in practice.

**Table 3. An example of data items relevant to pressure measurement using an EoS calibrant\(^a\) and an optical sensor.**

| Data item                        | Data value |
|----------------------------------|------------|
| Pressure gauge material          | Au         |
| $P$ measurement technique        | XRD        |
Pressure measurement experimental details: Synchrotron X-ray source at the 16-ID-B beamline (HPCAT) of the Advanced Photon Source (APS), beam size of \( \approx 4 \times 5 \mu \text{m} \), \( \lambda = 0.30622 \, \text{Å} \), MarCCD detector.

Pressure measurement data processing details: The collected images were integrated using FIT2D program \([41]\); the lattice parameter was determined by least-squares refinement of five diffraction lines [(111), (200), (220), (311), and (222)].

- Lattice parameter \( a = 3.9797(8) \, \text{Å} \)
- Unit-cell volume \( V = 63.03(4) \, \text{Å}^3 \)

EoS form: BM3 (third-order Birch-Murnaghan)

- EoS parameter \( V_0 = 67.850(4) \, \text{Å}^3 \)
- EoS parameter \( K_{0T} = 167 \, \text{GPa} \)
- EoS parameter \( K_{0T}' = 5.77(2) \)

EoS literature reference: Fei et al. PNAS 2007, 104, 9182–9186 \([42]\).

PTM Ne

Measurement \( T \): 300 K

Pressure: 15.22(15) GPa

| Pressure gauge material | Ruby |
|-------------------------|------|
| \( P \) measurement technique | Luminescence |
| Pressure measurement experimental details | A custom-built fluorescence spectrometer with a 600 grooves/mm grating, and a 532 nm frequency-doubled Nd:YAG green laser, calibrated using the emission lines of a Ne lamp |
| Pressure measurement data processing details | The fluorescence spectrum was acquired using a custom software application and the \( R1 \) line was fitted with a Voigt profile |
| Spectral characteristics | \( R1 \) fluorescence line \( \lambda = 698.55(1) \, \text{nm} \) |
| Reference spectral characteristics | \( R1 \) fluorescence line at \( p = 0 \) \( \lambda_0 = 694.22(1) \, \text{nm} \) |
| Pressure scale form | \( p = A(\Delta \lambda / \lambda_0)[1+B(\Delta \lambda / \lambda_0)^2] \) |
| \( P \) scale parameter \( A \) | 1870 GPa |
| \( P \) scale parameter \( B \) | 5.63(3) |
| Temperature correction form | \( \Delta \lambda_{R1} = 0.000746(4) \, \Delta T - 3.01(25) \times 10^{-6} \, \Delta T^2 + 8.76(33) \times 10^{-9} \, \Delta T^3 \) |
| Working range: 296 K < \( T < 900 \, \text{K} \) |

Literature references:
- Pressure scale: Ruby2020 (Shen et al. 2020) \([26]\)
- Temperature correction: Datchi et al. HPR, 2007, 27, 447–463 \([27]\)

PTM \( \text{N}_2 \)

Measurement \( T \): 400(1) K

Reference measurement \( T \): 298(1) K

Pressure: 10.0(3) GPa

\(^a\) Adapted after \([30]\).
data and metadata are often stored as descriptive annotations in the article text or supplementary material. Such unstructured data resources are primarily human-readable only. Moreover, some laboratory logbooks records are neither published nor deposited in an archive and could be lost forever. To rescue information from looming digital darkness, using a standardized format enabling information handling via electronic data processing is mandatory. Only under such conditions, FAIR data principles can be turned into reality. Flexibility and extensibility of the CIF format (which has no formal distinction between data and metadata), makes it an effective data scheme for the preservation of essential aspects of pressure measurement. This is the first step toward the deposition of pressure-measurement related data in existing databases and the facilitation of efficient data mining.

Acknowledgments
The author gratefully acknowledges Dr. Guoyin Shen for valuable insights and fruitful discussions.

References
[1] Mezouar M, Giampaoli R, Garbarino G, Kantor I, Dewaele A, Week G, Boccato S, Svitlyk V, Rosa A D, Torchio R, Mathon O, Hignette O and Bauchau S 2017 *High Press. Res.* **37** 170–80
[2] Giampaoli R, Kantor I, Mezouar M, Boccato S, Rosa A D, Torchio R, Garbarino G, Mathon O and Pascarelli S 2018 *High Press. Res.* **38** 250–69
[3] Holzapfel W B 1997 Pressure determination in High-pressure Techniques in Chemistry and Physics: A practical approach ed W B Holzapfel and N S Isaacs (Oxford: Oxford University Press) chapter 2 pp 47–55
[4] Bini R and Schettino V 2014 *Materials Under Extreme Conditions: Molecular Crystals at High Pressure* (London: Imperial College Press) pp 128–40
[5] Forman R A, Piermarini G J, Barnett J D and Block S 1972 *Science* **176** 284–5
[6] Akahama Y and Kawamura H 2006 *J. Appl. Phys.* **100** 043516
[7] Klug D D and Whalley E 1983 *Rev. Sci. Instrum.* **54** 1205–8
[8] Wilkinson M D, Dumontier M, Aalbersberg I J, Appleton G, Axton G, Blomberg N, Boiten J W, da Silva Santos L B, Bourne P E, Bouwman J, Brookes A J, Clark T, Crosse M, Dillo I, Dumon O, Edmunds S, Evelo C T, Finkers R, Gonzalez-Beltran A, Gray A J G, Groth P, Goble C, Grethe J S, Heringa J, ‘t Hoorn P A C, Hooft R, Kuhn T, Kok R, Kok J, Lusher S J, Martone M E, Mons A, Packer A L, Persson B, Rocca-Serra P, Roos M, van Schaik R, Sansone S-A, Schultes E, Sengstag T, Slater T, Strawn G, Swertz M A, Thompson M, van der Lei J, van Mulligen E, Velterop J, Waagmeester A, Wittenberg P, Wolstencroft K, Zhao J and Mons B 2016 *Sci. Data* **3** 160018
[9] Helliwell J R, McMahon B, Guiss J M and Kroon-Batenburg L M J 2017 *JUCrJ* **4** 714–22
[10] Helliwell J R, Minor W, Weiss M S, Garman E F, Read R J, Newman J, van Raaij M J, Hajdu J and Baker E N 2019 *Acta Cryst.* **D** **75** 455–7
[11] Aranda M A G 2018 *J. Appl. Cryst.* **51** 1739–44
[12] El Mendili Y, Vaitkus A, Merkys A, Gražulis S, Chatteigner D, Mathevet F, Gascoin S, Petit S, Barateau J-F, Zaunett M, Secchi M, Mariott G, Kumar A, Cassetta M, Lutterotti L, Borovin E, Orberger B, Simon P, Hehlen B and Le Guen M 2019 *J. Appl. Cryst.* **52**, 618–25
[13] Dziubek K F, Wang L and Liu H 2019 *Materials Structure* **26** 91–3 http://www.xray.cz/ms/bul2019-2/chp.pdf
[14] Hall S R and McMahon B eds 1995 *International Tables for Crystallography Vol. G Definition and exchange of crystallographic data* (Springer: Dordrecht)
[15] Dziubek K F and Katrusiak A 2015 Metadata in high-pressure crystallography *Metadata for raw data from X-ray diffraction and other structural techniques A Satellite Workshop to the 29th European Crystallographic Meeting Programme and background materials* ed Helliwell J and McMahon B pp 13–14 https://www.iucr.org/__data/assets/pdf_file/0020/113924/programme2.pdf
[16] Anderson O L and Mammone J 1979 A Test of the NaCl Equation of State by the Behavior of the Shear Velocities at High Pressure High-Pressure Science and Technology ed Timmerhaus K D and Barber M S (Boston: Springer) pp 181–94

[17] Birch F 1947 Phys. Rev. 71 809–24

[18] Vinet P, Smith J R, Ferrante J and Rose J H 1987 Phys. Rev. B 35 1945–53

[19] Holzapfel W B 1998 High Press. Res. 16 81–126

[20] Fei Y 1995 Thermal expansion Mineral Physics and Crystallography: A Handbook of Physical Constants ed Ahrens T J (Washington: American Geophysical Union) pp 29–44

[21] Fei Y, Li J, Hirose K, Van Orman J, Sanloup C, van Westrenen W, Komabayashi T and Funakoshi K 2004 Phys. Earth Planet. Inter. 143–144 516–26

[22] Lacam A and Chateau C 1989 J. Appl. Phys. 66 366–72

[23] Hess N J and Exarhos G J 1989 High Press. Res. 2 57–64

[24] Shen Y R, Gregorian T and Holzapfel W B 1991 High Press. Res. 7 73–75

[25] Picard A, Oger P M, Daniel I, Cardon H, Montagnac G and Chervin J-C 2006 J. Appl. Phys. 100 034915

[26] Shen G, Wang Y, Dewaele A, Syassen K, Wu C, Fratanduono D E, Eggert J, Klotz S, Dziubek K F, Loubeyre P, Fat'yanov O V, Asimov P D, Mashimo T, Wentzcovitch R M M 2020 High Press. Res. DOI:10.1080/08957959.2020.1791107 accepted

[27] Datchi F, Dewaele A, Loubeyre P, LeToullec R, Le Godec Y and Canny B 2007 High Press. Res. 27 447–63

[28] Ceppatelli M, Serdyukov A, Bini R and Jodl H J 2009 J. Phys. Chem. B 113 6652–60

[29] Lin C, Smith J S, Sinogeikin S V, Park C, Kono Y, Kenney-Benson C, Rod E and Shen G 2016 J. Appl. Phys. 119 045902

[30] Finkelstein G J, Dera P K and Duffy T S 2015 Am. Miner. 100 1821–33

[31] Angel R J, Miozzi F and Alvaro M 2019 Minerals 9, 562

[32] Liu Z, Cui Q and Zou G 1990 Phys. Lett. A 143, 79–82

[33] Angel R J, Bujak M, Zhao J, Gatta G D, and Jacobsen S D 2007 J. Appl. Cryst. 40 26–32

[34] Klotz S, Chervin J-C, Munsch P and Le Marchand G 2009 J. Phys. D: Appl. Phys. 42 075413

[35] Takemura K and Dewaele A 2008 Phys. Rev. B 78 104119

[36] Wang Y, Weidner D J and Meng Y 1998 Advances in Equation-of-State Measurements in SAM-85. Properties of Earth and Planetary Materials at High Pressure and Temperature: Geophysical Monograph 101 ed Manghnani M H and Yagi T (Washington: American Geophysical Union) pp 365–72

[37] Liu L and Bi Y 2016 Matter Radiat. Extremes 1 224–36

[38] Eremets M 1996 High Pressure Experimental Methods (New York: Oxford University Press) pp 176–7

[39] Stepanov A N 1987 Zh. Prikl. Spektr. 47, 251–4

[40] Dewaele A 2019 Minerals 9, 684

[41] Hammersley A P, Svensson S O, Hanfland M, Fitch A N and Hausermann D 1996 High Press. Res. 14, 235–48

[42] Fei Y, Ricolleau A, Frank M, Mibe K, Shen G and Prakapenka V 2007 Proc. Natl. Acad. Sci. 104, 9182–86