```
################################################################################
### CIF submission form for results of Rietveld refinements (IUCr journals)###
###                              Version 10 February 2005  ###
################################################################################
```

This is an electronic "form" for submitting the results of a Rietveld
refinement of a model against powder diffraction data to an
IUCr journal as a Crystallographic Information File (CIF). Full details
of the CIF format are given in the paper "The Crystallographic Information
File (CIF): a New Standard Archive File for Crystallography" by S. R. Hall,
F. H. Allen and I. D. Brown [Acta Cryst. (1991), A47, 655-685].
#
The current version of the powder CIF dictionary, which contains definitions
of the terms staring _pd_, may be obtained from
http://www.iucr.org/iucr-top/cif/pd/index.html. Other terms are defined in
the core CIF dictionary at http://www.iucr.org/iucr-top/cif/core/index.html.
#
Note that all fields should be numeric or character type EXCEPT those which
are flagged as 'text' - free-form text of any length may be included in
these latter fields provided the text block begins and ends with a semicolon
as the first character of a new line. Note also that the query marks
'?' are significant as placeholders, and should not be deleted where a data
item is not given, UNLESS the accompanying data name is also deleted.
Lines should not exceed 80 characters in length. The comments following
a hash symbol '#' may be deleted if wished.


```
#==============================================================================
data_global

_audit_update_record
;
2010-06-25 # Formatted by publCIF
;
#==============================================================================

loop_
  _audit_conform.dict_name
  _audit_conform.dict_version
  _audit_conform.dict_location
    cif_core.dic .  ftp://ftp.iucr.org/pub/cif_core.dic
    cif_pd.dic .  ftp://ftp.iucr.org/pub/cif_pd.dic
```

1. SUBMISSION DETAILS

```
_publ_contact_author_name           'Hiroshi Kojitani'
_publ_contact_author_address              # Address of author for correspondence
;
Department of Chemistry
Gakushuin University
1-5-1 Mejiro, Toshima-ku
Tokyo 171-8588
Japan
```

```
;
_publ_contact_author_email       hiroshi.kojitani@gakushuin.ac.jp
_publ_contact_author_fax         +81-3-5992-1029
_publ_contact_author_phone       +81-3-3986-0221

_publ_contact_letter
;?
;

_publ_requested_journal          'American Mineralogist'
_publ_requested_coeditor_name        ?
_publ_requested_category             ?   # Acta Cryst. C: one of FI/FM/FO/AD
                            # Acta Cryst. E: one of EI/EM/EO/AD
```

#===

2. PROCESSING SUMMARY (IUCr Office Use Only)

```
_journal_data_validation_number     ?

_journal_date_recd_electronic       ?
_journal_date_to_coeditor           ?
_journal_date_from_coeditor         ?
_journal_date_accepted              ?
_journal_date_printers_first        ?
_journal_date_printers_final        ?
_journal_date_proofs_out            ?
_journal_date_proofs_in             ?
_journal_coeditor_name              ?
_journal_coeditor_code              ?
_journal_coeditor_notes
; ?
;
_journal_techeditor_code            ?
_journal_techeditor_notes
; ?
;
_journal_coden_ASTM                 ?
_journal_name_full                  ?
_journal_year                       ?
_journal_volume                     ?
_journal_issue                      ?
_journal_page_first                 ?
_journal_page_last                  ?
_journal_paper_category             ?
_journal_suppl_publ_number          ?
_journal_suppl_publ_pages           ?
```

#===

3. TITLE AND AUTHOR LIST

```
_publ_section_title
;
Structure refinement of high-pressure hexagonal aluminous phases
```

K~1.00~Mg~2.00~Al~4.80~Si~1.15~O~12~ and
Na~1.04~Mg~1.88~Al~4.64~Si~1.32~O~12~
;
_publ_section_title_footnote
.

The loop structure below should contain the names and addresses of all
authors, in the required order of publication. Repeat as necessary.

loop_
     _publ_author_name
     _publ_author_footnote
     _publ_author_address
 'Kojitani, Hiroshi '   #<--'Last name, firstname'
; ?
;
;
Department of Chemistry
Gakushuin University
1-5-1 Mejiro, Toshima-ku
Tokyo 171-8588
Japan
;
 'Iwabuchi, Takemi '   #<--'Last name, firstname'
; ?
;
;
Department of Chemistry
Gakushuin University
1-5-1 Mejiro, Toshima-ku
Tokyo 171-8588
Japan
;
 'Kobayashi, Makoto '   #<--'Last name, firstname'
; ?
;
;
Department of Chemistry
Gakushuin University
1-5-1 Mejiro, Toshima-ku
Tokyo 171-8588
Japan
;
 'Miura, Hiroyuki '   #<--'Last name, firstname'
; ?
;
;
Division of Earth and Planetary Sciences
Graduate School of Science
Hokkaido University
N10-W8, Kita-ku
Sapporo 060-0810
Japan
;

'Akaogi, Masaki '   #<--'Last name, firstname'
; ?
;
;
Department of Chemistry
Gakushuin University
1-5-1 Mejiro, Toshima-ku
Tokyo 171-8588
Japan
;

#===

4. TEXT (For Acta Cryst. C and E, include the text of your paper in the CIF)

_publ_section_synopsis
.
_publ_section_abstract
;

As possible Na- and K-host minerals in the lower mantle, hexagonal aluminous
phases with $K_{1.00}Mg_{2.00}Al_{4.80}Si_{1.15}O_{12}$ and
$Na_{1.04}Mg_{1.88}Al_{4.64}Si_{1.32}O_{12}$ compositions were synthesized at 22--25
GPa and 1500\%C. The K-rich hexagonal aluminous phase was synthesized for the
first time. Crystal structures of both hexagonal aluminous phases were refined
using the Rietveld method. Obtained interatomic distances and bond angles were
compared to published data on $CaMg_2Al_6O_{12}$ hexagonal aluminous phase. The
general chemical formula of the hexagonal aluminous phase is represented as
[M3][M2]$_2$[M1]$_6$O$_{12}$ where the small-, middle-, and large-sized cations
occupy the M1, M2, and M3 sites, respectively. Changes of size and shape of
$M1O_6$ octahedra by the substitution of Si^{4+} for Al^{3+} in the M1 site make
it possible to adjust the size of the M2 and the M3 sites to accommodate Na^+
and Mg^{2+} in the M2 sites and Na^+ and K^+ in the M3 sites. Along the
$NaAlSiO_4$-$MgAl_2O_4$ join, stability of hexagonal aluminous phase in a
relatively wide compositional range of 30--50 mol% in $NaAlSiO_4$ component can
be explained by possible replacement of Mg^{2+} by Na^+ in the M2 site due to
closer ionic radius of Na^+ to Mg^{2+} than K^+ and by shrinkage and
deformation of $M1O_6$ octahedra with the coupled substitution: $^{M2}Mg^{2+}$ +
$^{M1}Al^{3+} \rightarrow ^{M2}Na^+ + ^{M1}Si^{4+}$.
;
_publ_section_comment
;
(type here to add)
;
_publ_section_exptl_prep   # Details of the preparation of the sample(s)
                  # should be given here.
;
High-pressure high-temperature syntheses of the K- and the Na-hexagonal
aluminous phases were made using a Kawai-type multi-anvil high-pressure
apparatus at Gakushuin University. In the synthesis of the K-hexagonal
aluminous phase, the gel with $KMg_2Al_5SiO_{12}$ composition was held for 1
hour at 1500\%C and at 20 GPa. After quenching to room temperature at the
pressure, the sample was decompressed to ambient pressure. Similarly, the
Na-hexagonal aluminous phase was synthesized by keeping the starting mixture

of MgAl~2~O~4~ spinel and NaAlSiO~4~ high carnegieite in the mole ratio of 3:2
at 22 GPa and 1500\%C for 2 hours
;
_publ_section_exptl_refinement
;
The crystal structure of CaMg~2~Al~6~O~12~ hexagonal aluminous phase (Miura
<i>et al.</i> 2000) was adopted as the model structure.
;
_publ_section_references
;
Kojitani, H., Hisatomi, R. & Akaogi, M. (2007). Am. Mineral., 92, 1112-1118.

Izumi, F. & Ikeda, T. (2000). Mater. Sci. Forum, 321/324, 198-204.

Miura, H., Hamada, Y., Suzuki, T., Akaogi, M., MIyajima, N. & Fujino, K.
(2000). Am. Mineral., 85, 1799-1803.
;
_publ_section_figure_captions
;

;
_publ_section_acknowledgements
;
This study was partially supported by Grants-in-Aid (No. 18540478 to H. K. and
No. 19340166 to M. A.) from Japan Society for the Promotion of Science.
;

#==

If more than one structure is reported, sections 5-10 should be completed
per structure. For each data set, replace the '?' in the data_? line below
by a unique identifier.

#==

data_K-hex

loop_
  _audit_conform.dict_name
  _audit_conform.dict_version
  _audit_conform.dict_location
    cif_core.dic . ftp://ftp.iucr.org/pub/cif_core.dic
    cif_pd.dic . ftp://ftp.iucr.org/pub/cif_pd.dic

5. CHEMICAL DATA

_chemical_name_systematic
; ?
;
_chemical_name_common   ?
_chemical_formula_moiety   'K1.00 Mg2.00 Al4.80 Si1.15 O12'
_chemical_formula_structural   'K1.00 Mg2.00 Al4.80 Si1.15 O12'
_chemical_formula_analytical   'K1.00 Mg2.00 Al4.80 Si1.15 O12'
_chemical_formula_sum   'K1.00 Mg2.00 Al4.80 Si1.15 O12'

```
_chemical_formula_weight        441.51
_chemical_melting_point         ?
_chemical_compound_source       ?     # for minerals and
                              # natural products


loop_
    _atom_type_symbol
    _atom_type_description
    _atom_type_scat_dispersion_real
    _atom_type_scat_dispersion_imag
    _atom_type_scat_source
    _atom_type_scat_length_neutron     # include if applicable
  ?   ?   ?   ?   ?    ?


#==========================================================================

# 6. POWDER SPECIMEN AND CRYSTAL DATA

_space_group_crystal_system    hexagonal
_space_group_name_H-M_alt    P63/m
_space_group_name_Hall       -P6c

loop_
    _symmetry_equiv_pos_site_id
    _symmetry_equiv_pos_as_xyz       #<--must include 'x,y,z'
    1   x, y, z
    2   -y, x-y, z
    3   -x;y, -x, z
    4   -x, -y, z+1/2
    5   y, -x+y, z+1/2
    6   x-y, x, z+1/2


_cell_length_a             8.8168(2)
_cell_length_b             8.8168(2)
_cell_length_c             2.7684(1)
_cell_angle_alpha          90.0
_cell_angle_beta           90.0
_cell_angle_gamma          120.0
_cell_volume               186.37(1)
_cell_formula_units_Z          1
_cell_measurement_temperature    293
_cell_special_details
; ?
;

# The next three fields give the specimen dimensions in mm.  The equatorial
# plane contains the incident and diffracted beam.

_pd_spec_size_axial            ?     # perpendicular to
                              # equatorial plane

_pd_spec_size_equat            ?     # parallel to
                              # scattering vector
                              # in transmission
```

```
_pd_spec_size_thick              ?        # parallel to
                                 # scattering vector
                                 # in reflection


# The next five fields are character fields that describe the specimen.

_pd_spec_mounting                        # This field should be
                                 # used to give details of the
                                 # container.
; powder were fixed using aceton on a quartz or a Si plate holder.
;
_pd_spec_mount_mode              'reflection'     # options are 'reflection'
                                 # or 'transmission'

_pd_spec_shape                   'flat_sheet'     # options are 'cylinder'
                                 # 'flat_sheet' or 'irregular'


_pd_char_particle_morphology     ?
_pd_char_colour                  white      # use ICDD colour descriptions


# The following three fields describe the preparation of the specimen.
# The cooling rate is in K/min.  The pressure at which the sample was
# prepared is in kPa.  The temperature of preparation is in K.


_pd_prep_cool_rate               ?
_pd_prep_pressure                20000000
_pd_prep_temperature             1773


# The next four fields are normally only needed for transmission experiments.
_exptl_absorpt_coefficient_mu        ?
_exptl_absorpt_correction_type       ?      # include if applicable
_exptl_absorpt_process_details       ?      # include if applicable
_exptl_absorpt_correction_T_min      ?      # include if applicable
_exptl_absorpt_correction_T_max      ?      # include if applicable


#=============================================================================

# 7. EXPERIMENTAL DATA

_exptl_special_details
; ?
;


# The following item is used to identify the equipment used to record
# the powder pattern when the diffractogram was measured at a laboratory
# other than the authors' home institution, e.g. when neutron or synchrotron
# radiation is used.


_pd_instr_location
; ?
;
_pd_calibration_special_details          # description of the method used
                                 # to calibrate the instrument
```

; ?
;

```
_diffrn_ambient_temperature     293
_diffrn_source                  'rotating target'
_diffrn_source_target           Cr
_diffrn_source_type             ?
_diffrn_radiation_type          ?
_diffrn_measurement_device_type 'Rigaku RINT2500V'
_diffrn_detector                'NaI scintillation counter'
_diffrn_detector_type           ?       # make or model of detector

_pd_meas_scan_method            'cont'      # options are 'step', 'cont',
                                # 'tof', 'fixed' or
                                # 'disp' (= dispersive)
_pd_meas_special_details
; ?
;
```

The following six items are used for angular dispersive measurements only.

```
_diffrn_radiation_wavelength    2.2897
_diffrn_radiation_monochromator  'pyrolytic graphite monochromator'
```

The following four items give details of the measured (not processed)
powder pattern. Angles are in degrees.

```
_pd_meas_number_of_points       6500
_pd_meas_2theta_range_min       15
_pd_meas_2theta_range_max       145
_pd_meas_2theta_range_inc       0.02
```

The following three items are used for time-of-flight measurements only.

```
_pd_instr_dist_src/spec         ?
_pd_instr_dist_spec/detc        ?
_pd_meas_2theta_fixed           ?
```

#==

8. REFINEMENT DATA

Use the next field to give any special details about the fitting of the
powder pattern.

```
_pd_proc_ls_special_details
;
The atomic displacement parameters of two oxygen sites were fixed at 1.0 which
 was an average for oxides. The site occupancy fractions were based on the
results of the chemical analysis.
;
```

The next three items are given as text.

```
_pd_proc_ls_profile_function          'pseudo-Voigt'
_pd_proc_ls_background_function   'Legendre's polynomial'
_pd_proc_ls_pref_orient_corr
;
March-Dollase function
;

_pd_proc_ls_prof_R_factor          0.0754
_pd_proc_ls_prof_wR_factor          0.1011
_pd_proc_ls_prof_wR_expected    0.0757
_refine_ls_R_I_factor                   0.0470
_refine_ls_R_Fsqd_factor               ?
_refine_ls_R_factor_all                 0.0340

_refine_special_details
; ?
;

_refine_ls_matrix_type             ?
_refine_ls_weighting_scheme   ?  # options are 'sigma' (based on measured su's)
                      # or 'calc' (calculated weights)
_refine_ls_weighting_details      ?
_refine_ls_hydrogen_treatment    ?
_refine_ls_extinction_method      ?
_refine_ls_extinction_coef        ?
_refine_ls_number_parameters      ?
_refine_ls_number_restraints      ?
_refine_ls_number_constraints     ?

# The following item is the same as CHI, the square root of 'CHI squared'
_refine_ls_goodness_of_fit_all    ?

_refine_ls_restrained_S_all       1.3343
_refine_ls_shift/su_max           ?
_refine_ls_shift/su_mean          ?

# The following four items apply to angular dispersive measurements.
# 2theta minimum, maximum and increment (in degrees) are for the
# intensities used in the refinement.

_pd_proc_2theta_range_min         15
_pd_proc_2theta_range_max         145
_pd_proc_2theta_range_inc         0.02
_pd_proc_wavelength               2.2897

# Each refinement must be accompanied by a listing of the powder data
# in CIF format. Each listing should be sent as a separate file consisting
# of one data block containing a single powder profile. The value of
# _pd_block_diffractogram_id is used to associate each refinement with
# its corresponding powder profile, since it must match the value
# of _pd_block_id in the file containing the powder data. A template
# for supplying powder data in CIF format is available by ftp at
# ftp://ftp.iucr.org/pub/rietdataform.cif and an example is given
# at ftp://ftp.iucr.org/pub/rietdataxmpl.cif.
```

_pd_block_diffractogram_id   ?

Give appropriate details in the next two text fields.

_pd_proc_info_excluded_regions   ?
_pd_proc_info_data_reduction   ?

The following items are used to identify the programs used.

_computing_data_collection   'RIgaku application software'
_computing_cell_refinement   'RIETAN-2000 (Izumi and Ikeda, 2000)'
_computing_data_reduction   ?
_computing_structure_solution   ?
_computing_structure_refinement   'RIETAN-2000 (Izumi and Ikeda, 2000)'
_computing_molecular_graphics   ?
_computing_publication_material   ?

#==

9. ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

loop_
    _atom_site_label
    _atom_site_fract_x
    _atom_site_fract_y
    _atom_site_fract_z
    _atom_site_occupancy
    _atom_site_symmetry_multiplicity
    _atom_site_Wyckoff_symbol
    _atom_site_B_iso_or_equiv
    _atom_site_adp_type

  K   0   0   0.25   0.5   ?   2a 1.59(11) Biso
  Mg   0.66667 0.33333   0.25   1.0   ?   2d 0.47(8)   Biso
  Al   0.9915(2) 0.3472(2)   0.25   0.80 ?   6h 0.22(5)   Biso
  Si   0.9915(2) 0.3472(2)   0.25   0.19 ?   6h 0.22(5)   Biso
  O1   0.1318(3) 0.6021(3)   0.25   1.0   ?   6h 1.0   Biso
  O2   0.3206(4) 0.2074(3)   0.25   1.0   ?   6h 1.0   Biso

Note: if the displacement parameters were refined anisotropically
the U matrices should be given as for single-crystal studies.

loop_
    _atom_site_aniso_label
    _atom_site_aniso_U_11
    _atom_site_aniso_U_22
    _atom_site_aniso_U_33
    _atom_site_aniso_U_12
    _atom_site_aniso_U_13
    _atom_site_aniso_U_23
    _atom_site_aniso_type_symbol
     ? ? ? ? ? ? ? ?

```
#==============================================================================

# 10. MOLECULAR GEOMETRY

_geom_special_details              ?

loop_
    _geom_bond_atom_site_label_1
    _geom_bond_atom_site_label_2
    _geom_bond_distance
    _geom_bond_site_symmetry_1
    _geom_bond_site_symmetry_2
    _geom_bond_publ_flag
  K    O2   2.483(3)    ?   ?   y
  K    O2   2.843(3)    ?   ?   y
  Mg   O1   2.094(2)    ?   ?   y
  Al   O1   1.949(2)    ?   ?   y
  Al   O1   1.945(2)    ?   ?   y
  Al   O2   1.927(3)    ?   ?   y
  Al   O2   1.837(2)    ?   ?   y
  O1   O1   2.525(4)    ?   ?   y
  O2   O2   2.843(2)    ?   ?   y

    loop_
    _geom_angle_atom_site_label_1
    _geom_angle_atom_site_label_2
    _geom_angle_atom_site_label_3
    _geom_angle
    _geom_angle_site_symmetry_1
    _geom_angle_site_symmetry_2
    _geom_angle_site_symmetry_3
    _geom_angle_publ_flag
  O2  Al  O1  86.1(1)   ? ? ? y
  O1  Al  O1  90.7(1)   ? ? ? y
  O2  Al  O2  97.8(1)   ? ? ? y
  O1  Al  O1  80.8(1)   ? ? ? y
  O1  Al  O2  161.4(2)  ? ? ? y
  Al  O1  Al  99.2(1)   ? ? ? y
  Al  O2  Al  128.8(1)  ? ? ? y


data_Na-hex

loop_
  _audit_conform.dict_name
  _audit_conform.dict_version
  _audit_conform.dict_location
    cif_core.dic . ftp://ftp.iucr.org/pub/cif_core.dic
    cif_pd.dic . ftp://ftp.iucr.org/pub/cif_pd.dic

# 5. CHEMICAL DATA

_chemical_name_systematic
```

; ?
;
_chemical_name_common   ?
_chemical_formula_moiety   'Na1.04 Mg1.88 Al4.64 Si1.32 O12'
_chemical_formula_structural   'Na1.04 Mg1.88 Al4.64 Si1.32 O12'
_chemical_formula_analytical   'Na1.04 Mg1.88 Al4.64 Si1.32 O12'
_chemical_formula_sum   'Na1.04 Mg1.88 Al4.64 Si1.32 O12'
_chemical_formula_weight   423.86
_chemical_melting_point   ?
_chemical_compound_source   ?   # for minerals and
                           # natural products

loop_
    _atom_type_symbol
    _atom_type_description
    _atom_type_scat_dispersion_real
    _atom_type_scat_dispersion_imag
    _atom_type_scat_source
    _atom_type_scat_length_neutron   # include if applicable
  ?   ?   ?   ?   ?   ?

#===

6. POWDER SPECIMEN AND CRYSTAL DATA

_space_group_crystal_system   hexagonal
_space_group_name_H-M_alt   P63/m
_space_group_name_Hall   -P6c

loop_
    _symmetry_equiv_pos_site_id
    _symmetry_equiv_pos_as_xyz   #<--must include 'x,y,z'
  1   x, y, z
  2   -y, x-y, z
  3   -x;y, -x, z
  4   -x, -y, z+1/2
  5   y, -x+y, z+1/2
  6   x-y, x, z+1/2

_cell_length_a   8.7274(2)
_cell_length_b   8.7274(2)
_cell_length_c   2.7663(1)
_cell_angle_alpha   90.0
_cell_angle_beta   90.0
_cell_angle_gamma   120.0
_cell_volume   182.47(1)
_cell_formula_units_Z   1
_cell_measurement_temperature   293
_cell_special_details
; ?
;

The next three fields give the specimen dimensions in mm. The equatorial
plane contains the incident and diffracted beam.

```
_pd_spec_size_axial              ?       # perpendicular to
                                 # equatorial plane

_pd_spec_size_equat              ?       # parallel to
                                 # scattering vector
                                 # in transmission

_pd_spec_size_thick              ?       # parallel to
                                 # scattering vector
                                 # in reflection

# The next five fields are character fields that describe the specimen.

_pd_spec_mounting                        # This field should be
                                 # used to give details of the
                                 # container.
; powder were fixed using aceton on a quartz or a Si plate holder.
;
_pd_spec_mount_mode              'reflection'    # options are 'reflection'
                                 # or 'transmission'

_pd_spec_shape                   'flat_sheet'    # options are 'cylinder'
                                 # 'flat_sheet' or 'irregular'

_pd_char_particle_morphology     ?
_pd_char_colour                  white    # use ICDD colour descriptions

# The following three fields describe the preparation of the specimen.
# The cooling rate is in K/min.  The pressure at which the sample was
# prepared is in kPa.  The temperature of preparation is in K.

_pd_prep_cool_rate               ?
_pd_prep_pressure                22000000
_pd_prep_temperature             1773

# The next four fields are normally only needed for transmission experiments.
_exptl_absorpt_coefficient_mu    ?
_exptl_absorpt_correction_type   ?       # include if applicable
_exptl_absorpt_process_details   ?       # include if applicable
_exptl_absorpt_correction_T_min  ?       # include if applicable
_exptl_absorpt_correction_T_max  ?       # include if applicable

#==============================================================================

# 7. EXPERIMENTAL DATA

_exptl_special_details
; ?
;

# The following item is used to identify the equipment used to record
# the powder pattern when the diffractogram was measured at a laboratory
# other than the authors' home institution, e.g. when neutron or synchrotron
```

radiation is used.

```
_pd_instr_location
; ?
;
_pd_calibration_special_details        # description of the method used
                            # to calibrate the instrument
; ?
;

_diffrn_ambient_temperature     293
_diffrn_source                'rotating target'
_diffrn_source_target          Cr
_diffrn_source_type            ?
_diffrn_radiation_type         ?
_diffrn_measurement_device_type  'Rigaku RINT2500V'
_diffrn_detector              'NaI scintillation counter'
_diffrn_detector_type          ?      # make or model of detector

_pd_meas_scan_method           'step'      # options are 'step', 'cont',
                            # 'tof', 'fixed' or
                            # 'disp' (= dispersive)
_pd_meas_special_details
; ?
;
```

The following six items are used for angular dispersive measurements only.

```
_diffrn_radiation_wavelength     2.2897
_diffrn_radiation_monochromator   'pyrolytic graphite monochromator'
```

The following four items give details of the measured (not processed)
powder pattern. Angles are in degrees.

```
_pd_meas_number_of_points       6500
_pd_meas_2theta_range_min       10
_pd_meas_2theta_range_max       140
_pd_meas_2theta_range_inc       0.02
```

The following three items are used for time-of-flight measurements only.

```
_pd_instr_dist_src/spec          ?
_pd_instr_dist_spec/detc         ?
_pd_meas_2theta_fixed            ?
```

#===

8. REFINEMENT DATA

Use the next field to give any special details about the fitting of the
powder pattern.

```
_pd_proc_ls_special_details
;
```

The atomic displacement parameters for the two different oxygen sites were fixed
 at 1.0 for the Na-hexagonal aluminous phase. The atomic displacement parameter
 for the Na site was fixed at 2.0 which was reasonable and gave the lowest
 reliability factor. The site occupancy fractions were based on the results of
 the chemical analysis.
;

The next three items are given as text.

_pd_proc_ls_profile_function   'pseudo-Voigt'
_pd_proc_ls_background_function   'Legendre's polynomial'
_pd_proc_ls_pref_orient_corr
;
March-Dollase function
;

_pd_proc_ls_prof_R_factor   0.1027
_pd_proc_ls_prof_wR_factor   0.1366
_pd_proc_ls_prof_wR_expected   0.1014
_refine_ls_R_I_factor   0.0375
_refine_ls_R_Fsqd_factor   ?
_refine_ls_R_factor_all   0.0225

_refine_special_details
; ?
;

_refine_ls_matrix_type   ?
_refine_ls_weighting_scheme   ? # options are 'sigma' (based on measured su's)
                      # or 'calc' (calculated weights)
_refine_ls_weighting_details   ?
_refine_ls_hydrogen_treatment   ?
_refine_ls_extinction_method   ?
_refine_ls_extinction_coef   ?
_refine_ls_number_parameters   ?
_refine_ls_number_restraints   ?
_refine_ls_number_constraints   ?

The following item is the same as CHI, the square root of 'CHI squared'
_refine_ls_goodness_of_fit_all   ?

_refine_ls_restrained_S_all   1.35
_refine_ls_shift/su_max   ?
_refine_ls_shift/su_mean   ?

The following four items apply to angular dispersive measurements.
2theta minimum, maximum and increment (in degrees) are for the
intensities used in the refinement.

_pd_proc_2theta_range_min   10
_pd_proc_2theta_range_max   140
_pd_proc_2theta_range_inc   0.02
_pd_proc_wavelength   2.2897

Each refinement must be accompanied by a listing of the powder data
in CIF format. Each listing should be sent as a separate file consisting
of one data block containing a single powder profile. The value of
_pd_block_diffractogram_id is used to associate each refinement with
its corresponding powder profile, since it must match the value
of _pd_block_id in the file containing the powder data. A template
for supplying powder data in CIF format is available by ftp at
ftp://ftp.iucr.org/pub/rietdataform.cif and an example is given
at ftp://ftp.iucr.org/pub/rietdataxmpl.cif.

_pd_block_diffractogram_id   ?

Give appropriate details in the next two text fields.

_pd_proc_info_excluded_regions   ?
_pd_proc_info_data_reduction   ?

The following items are used to identify the programs used.

_computing_data_collection   'RIgaku application software'
_computing_cell_refinement   'RIETAN-2000 (Izumi and Ikeda, 2000)'
_computing_data_reduction   ?
_computing_structure_solution   ?
_computing_structure_refinement   'RIETAN-2000 (Izumi and Ikeda, 2000)'
_computing_molecular_graphics   ?
_computing_publication_material   ?

#==

9. ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

loop_
    _atom_site_label
    _atom_site_fract_x
    _atom_site_fract_y
    _atom_site_fract_z
    _atom_site_occupancy
    _atom_site_symmetry_multiplicity
    _atom_site_Wyckoff_symbol
    _atom_site_B_iso_or_equiv
    _atom_site_adp_type

  Na   0   0   0.25 0.46 ? 2a 3.30(29) Biso
  Mg 0.66667 0.33333   0.25 0.94 ? 2d 1.66(10) Biso
  Na 0.66667 0.33333   0.25 0.06 ? 2d 1.66(10) Biso
  Al   0.9892(2) 0.3408(3) 0.25 0.77 ? 6h 0.32(7) Biso
  Si   0.9892(2) 0.3408(3) 0.25 0.22 ? 6h 0.32(7) Biso
  O1 0.1224(5) 0.5925(6) 0.25 1.0   ? 6h 1.0   Biso
  O2 0.3116(3) 0.2045(4) 0.25 1.0   ? 6h 1.0   Biso

Note: if the displacement parameters were refined anisotropically
the U matrices should be given as for single-crystal studies.

loop_

```
      _atom_site_aniso_label
      _atom_site_aniso_U_11
      _atom_site_aniso_U_22
      _atom_site_aniso_U_33
      _atom_site_aniso_U_12
      _atom_site_aniso_U_13
      _atom_site_aniso_U_23
      _atom_site_aniso_type_symbol
       ?  ?  ?  ?  ?  ?  ?  ?
```

#==

10. MOLECULAR GEOMETRY

_geom_special_details   ?

```
loop_
      _geom_bond_atom_site_label_1
      _geom_bond_atom_site_label_2
      _geom_bond_distance
      _geom_bond_site_symmetry_1
      _geom_bond_site_symmetry_2
      _geom_bond_publ_flag
  Na   O2  2.393(3)   ?   ?   y
  Na   O2  2.764(3)   ?   ?   y
  Mg   O1  2.128(3)   ?   ?   y
  Al   O1  1.903(5)   ?   ?   y
  Al   O1  1.940(3)   ?   ?   y
  Al   O2  1.890(4)   ?   ?   y
  Al   O2  1.817(3)   ?   ?   y
  O1   O1  2.373(6)   ?   ?   y
  O2   O2  2.764(3)   ?   ?   y

    loop_
     _geom_angle_atom_site_label_1
     _geom_angle_atom_site_label_2
     _geom_angle_atom_site_label_3
     _geom_angle
     _geom_angle_site_symmetry_1
     _geom_angle_site_symmetry_2
     _geom_angle_site_symmetry_3
     _geom_angle_publ_flag
  O2  Al  O1  90.7(2)   ? ? ? y
  O1  Al  O1  90.9(2)   ? ? ? y
  O2  Al  O2  99.2(2)   ? ? ? y
  O1  Al  O1  76.3(2)   ? ? ? y
  O1  Al  O2  161.2(2) ? ? ? y
  Al  O1  Al  103.7(2)  ? ? ? y
  Al  O2  Al  127.5(1) ? ? ? y
```

#==
Additional structures (sections 5-10 and associated data_? identifiers)
may be added at this point.
#==

The following lines are used to test the character set of files sent by
network email or other means. They are not part of the CIF data set.
abcdefghijklmnopqrstuvwxyzABCDEFGHIJKLMNOPQRSTUVWXYZ0123456789
!@#$%^&*()_+{}:"~<>?|\-=[];'`,./