

Structural Chemistry of Akdalaite, $\text{Al}_{10}\text{O}_{14}(\text{OH})_2$, the Isostructural Aluminum Analogue of Ferrihydrite

John B. Parise ^{1,2,3,*}, Bingying Xia ⁴, Jack W. Simonson ⁴, William R. Woerner ⁵, Anna M. Plonka ⁶, Brian L. Phillips ¹ and Lars Ehm ^{1,7}

¹ Department of Geosciences, Stony Brook University, Stony Brook, NY 11794, USA;

Brian.phillips@stonybrook.edu

² Department of Chemistry, Stony Brook University, Stony Brook, NY 11794, USA

³ Joint Photon Sciences Institute, Stony Brook University, Stony Brook, NY 11794, USA

⁴ Department of Physics, Farmingdale State College, Farmingdale, NY 11735, USA; xiab@farmingdale.edu (B.X.); simonsjw@farmingdale.edu (J.W.S.)

⁵ EAG Laboratories, 625-B, Alfred Nobel Dr, Hercules, CA 94547, USA; william.woerner@gmail.com

⁶ Department of Materials Science and Chemical Engineering, Stony Brook, NY 11794, USA;

Anna.Plonka@stonybrook.edu

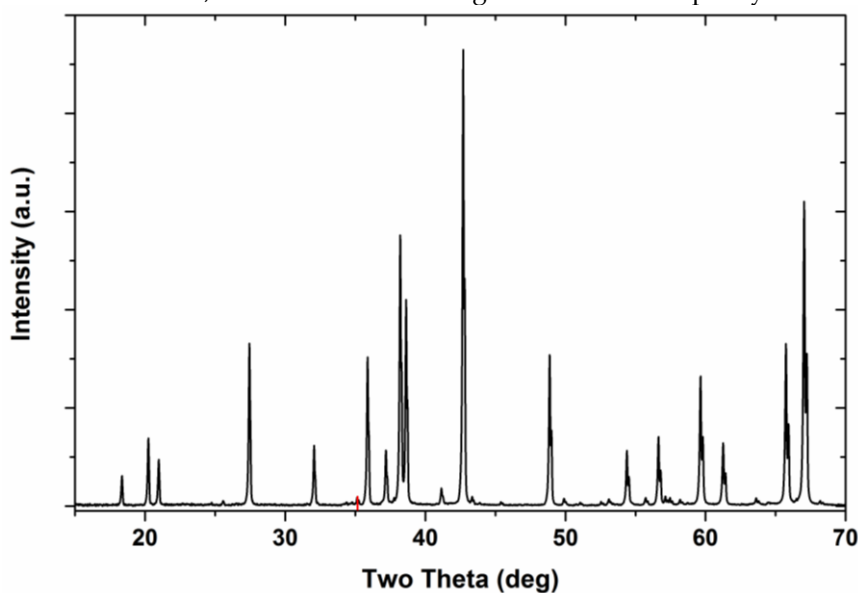
⁷ Mineral Physics Institute, Stony Brook University, Stony Brook, New York 11794, USA;

Lars.Ehm@stonybrook.edu

* Correspondence: john.parise@stonybrook.edu

1. Initial Characterization

The synthesized akdalaite samples were first inspected with laboratory PXRD and the crystal morphology was investigated with SEM. Akdalaite synthesized using the Al-floc starting material was nearly pure akdalaite with minor corundum impurity but had a needle morphology (Figure 4). The akdalaite synthesized from nanocrystalline boehmite also had a minor corundum impurity but had a hexagonal plate morphology suitable for SC-XRD (Figure 5). The 1-g sample synthesized from boehmite for NPD utilized a much longer capsule (5.5 cm) and was likely subject to a higher thermal gradient in the cold-seal vessel, which resulted in a higher amount of impurity corundum (Figure 6).



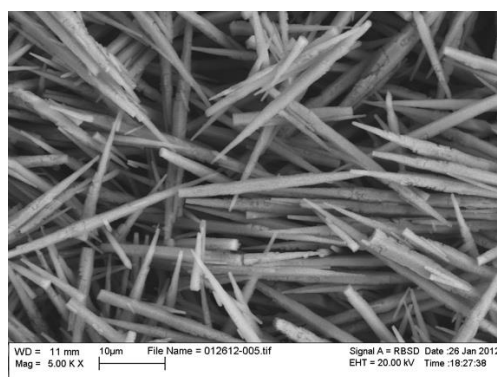


Figure S1. The PXRD pattern (top) and SEM image (bottom) of akdalaite synthesized from Al-floc showing only a minor impurity of corundum (red tick).

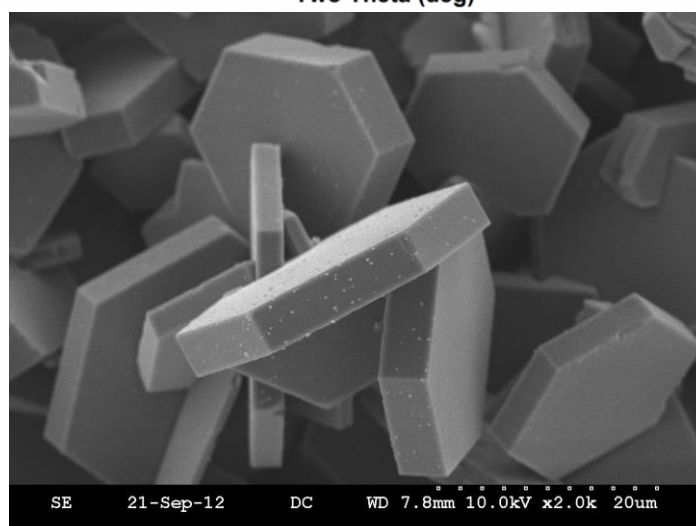
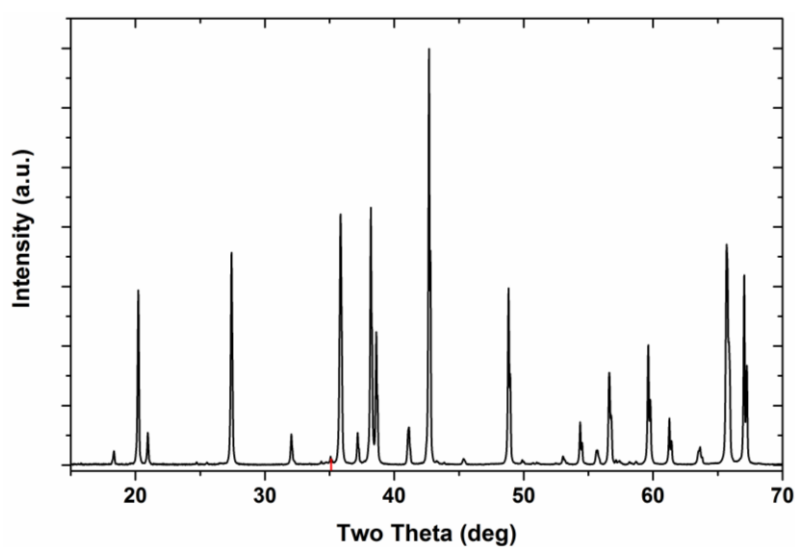


Figure S2. The PXRD pattern (top) and SEM image (bottom) of akdalaite synthesized from boehmite in a 2-cm-long capsule showing only a minor impurity of corundum (red tick).

2. Solid State NMR of Natural and Synthetic Akdalaite

For the synthetic sample, the tetrahedral Al gives a signal from +65 to +35 ppm, broadened by second-order quadrupolar effects (Figure S4).

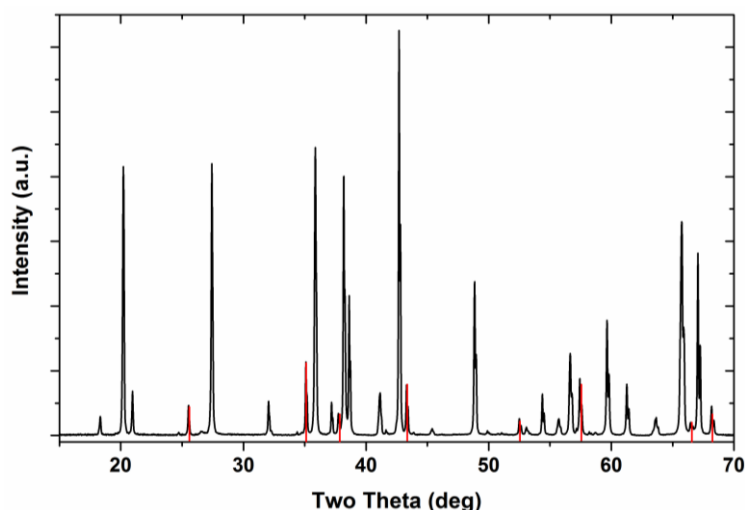


Figure S3. The XRD pattern of akdalaite synthesized from boehmite in a 5.5-cm-long capsule showing corundum (red tick) present as a minor phase.

The six-coordinate Al yields a complex feature from +20 to -10 ppm. The resonances in the synthetic spectrum display sharp features owing to the high crystallinity and purity of the sample, while the spectrum of the natural sample contains signal in the same shift regions but is less resolved due to contributions from paramagnetic impurity elements in akdalaite as well as the other accessory phases that comprise the sample (σ -Al₂O₃, fluorite, and mica). Additional minor, narrow peaks for four-coordinate Al occur in the spectrum of the synthetic sample at +72 and +67 ppm that likely arise from impurities but which cannot be assigned to any known phase based on literature data.

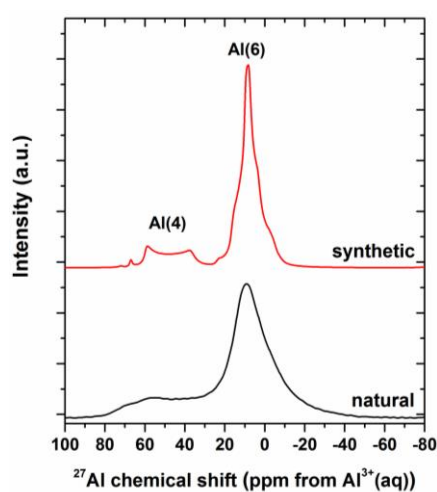


Figure S4. ²⁷Al MAS NMR spectra of synthetic crystalline akdalaite and natural akdalaite

3. High-Energy Powder X-ray Total Scattering

High-energy X-ray total scattering data were collected at the 11-ID-B beamline at the APS using ~20 mg of synthetic akdalaite in a Kapton capillary. The 2D diffraction data were collected at 58.26 keV ($\lambda = 0.2128 \text{ \AA}$) using a Perking-Elmer XRD 1621 detector. The geometry of the experimental setup was calibrated with a CeO₂ standard (NIST 674a) and the data were integrated into a 1D pattern using the program Fit2D.[1] The total scattering function ($S(Q)$) and pair distribution function (PDF, $G(r)$) were obtained using a Q_{max} of 25 \AA^{-1} with the program PDFgetX2 with standard and area detector geometry corrections [2,3].

3.1 X-ray Total Scattering and Pair Distribution Function Analysis

Crystalline akdalaite was studied with X-ray total scattering so as to compare the real space PDF to that of 6-line ferrihydrite (6-nm diameter particles) and evaluate the structural relationship between the two compounds. The PDF of two isostructural compounds cannot be compared directly, as the difference in the metal–oxygen (M–O) distances will propagate through the PDF, increasing the off-set of related pairs with distance. The differences in M–O distance can be accounted for in an empirical manner by comparing the PDFs of known crystalline structural analogues. Corundum (Al_2O_3) and hematite (Fe_2O_3) were chosen to determine the shift in pair correlations between aluminum and ferric iron, as the structure has a mix of edge, corner, and face sharing octahedra. There is no ideal Al and Fe analogue that has both octahedral and tetrahedral coordination with only +3 metal oxidation states. The PDFs for corundum and hematite were calculated from structure files using the program PDFgui with a Q_{max} of 25 \AA^{-1} [4–6]. The overlaid PDFs of corundum and hematite are shown in Figure 15. While being structurally analogous, the intensity of the related peaks in the PDFs are very different, as the intensity is still a function of the scattering power of the atoms. From the PDFs, the r-shift (Fe–Al Δr) for identical peaks is plotted as a function of average distance of those peaks in r (Figure 16). There is a strong linear correlation between the amount of r-shift between equivalent peaks and distance, so the equation of the trend line in Figure 16 becomes the r-shift equation (Equation S1) to apply to Al compound PDFs to equate to Fe compounds. The new distance (r_n) is determined after applying the trendline equation as a function of original distance (r_o):

$$r_n = r_o + [(0.0561 * r_o) + 0.0157]. \quad (\text{S1})$$

The PDF of corundum was shifted using Equation S1 and is shown with the PDF of hematite in Figure 17. It now becomes clear that corundum and hematite are isostructural, as the peaks in the PDF now overlay each other. The peak position alignment is not perfect after applying the r-shift to corundum, as it is an empirical correction but nonetheless a powerful approach for comparing structurally analogous compounds.

At a first glance, the PDF patterns of crystalline akdalaite and 6-line (6 nm) ferrihydrite (data from [7]) appear to have nothing in common (Figure 18, top). However, after the akdalaite data are shifted using the empirical Al–Fe r-shift equation (Equation S1) derived from the corundum–hematite data, the peaks in both datasets align (Figure 18, middle). The patterns are easiest to compare when they represent the same size regime as well. The akdalaite data was additionally enveloped to mimic the data of spherical nanoparticles with a diameter (d) of 30 \AA using the envelope function (f_e) of Equation S2, which is multiplied to the akdalaite $G(r)$ [8]. The $\theta(d - r)$ term is a step function that eliminates distances outside the diameter of the particle, as it is 1 when $r < d$, and 0 otherwise:

$$f_e(r, d) = \left[1 - \frac{3}{2} \frac{r}{d} + \frac{1}{2} \left(\frac{r}{d} \right)^3 \right] \theta(d - r). \quad (\text{S2})$$

In comparing the akdalaite data that has been linearly shifted and enveloped to a comparable particle size of ferrihydrite (Figure 18), it becomes obvious that the PDF patterns are extremely similar and share a common structure. These similarities between the adjusted akdalaite and 6-line ferrihydrite PDFs provide new evidence that ferrihydrite and akdalaite are indeed isostructural.

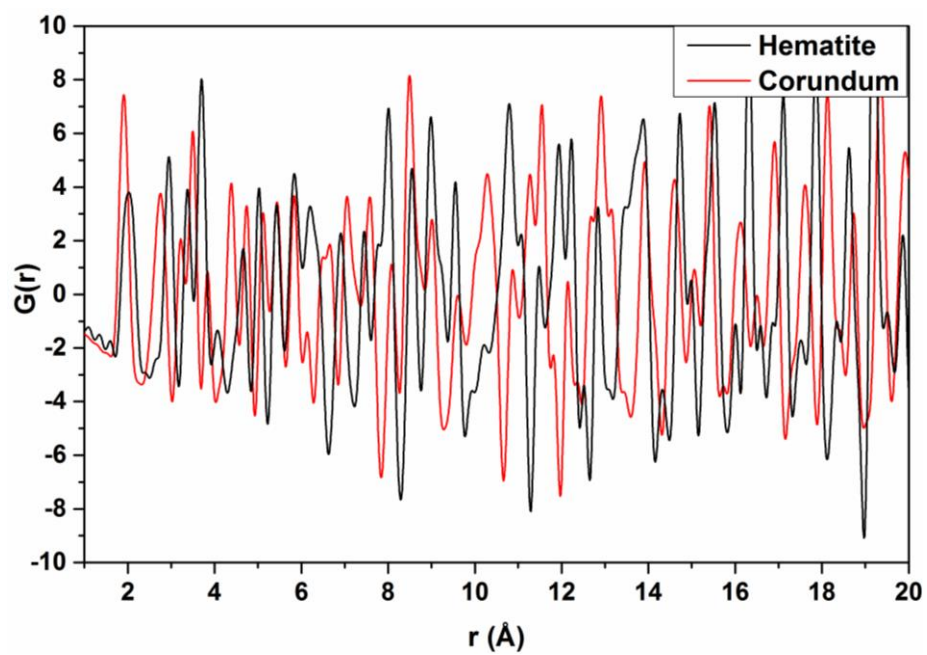


Figure S5. Calculated pair distribution functions (PDFs) of hematite and corundum.

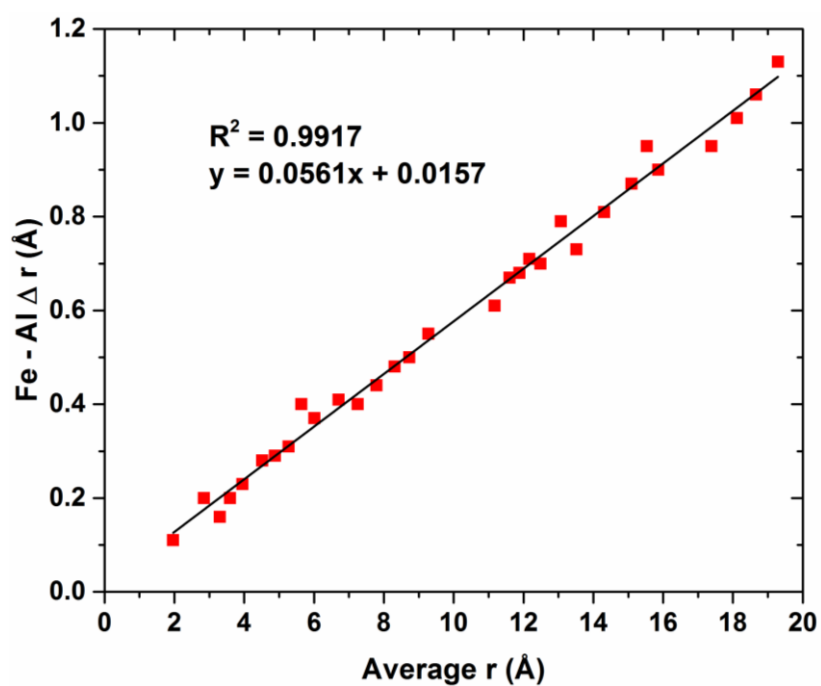


Figure S6. Plot of the r -shift of identical peaks in the calculated PDFs of hematite and corundum.

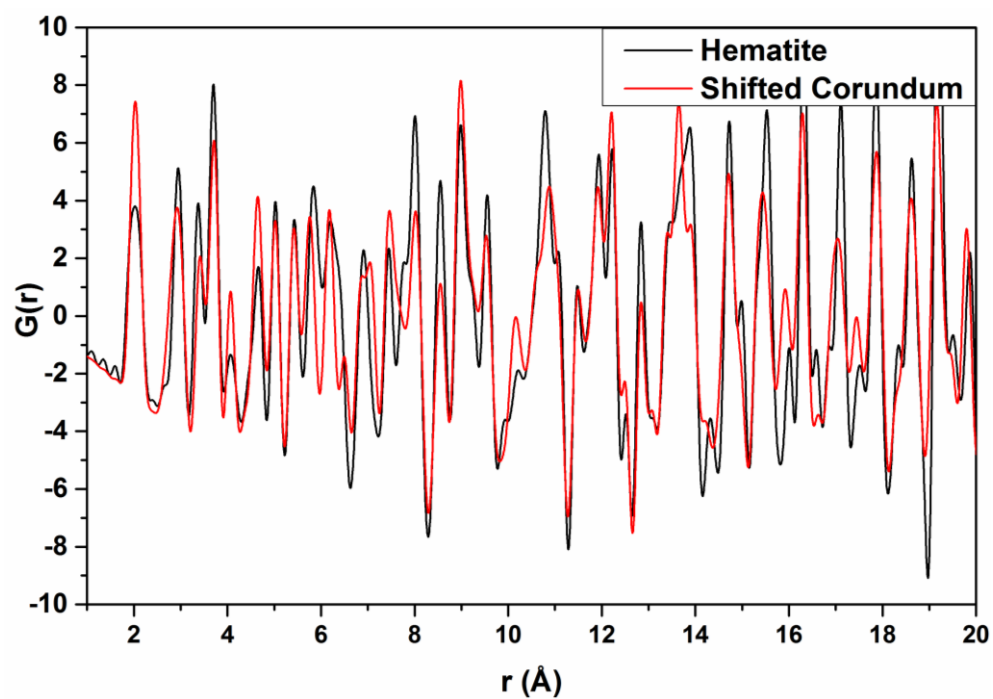
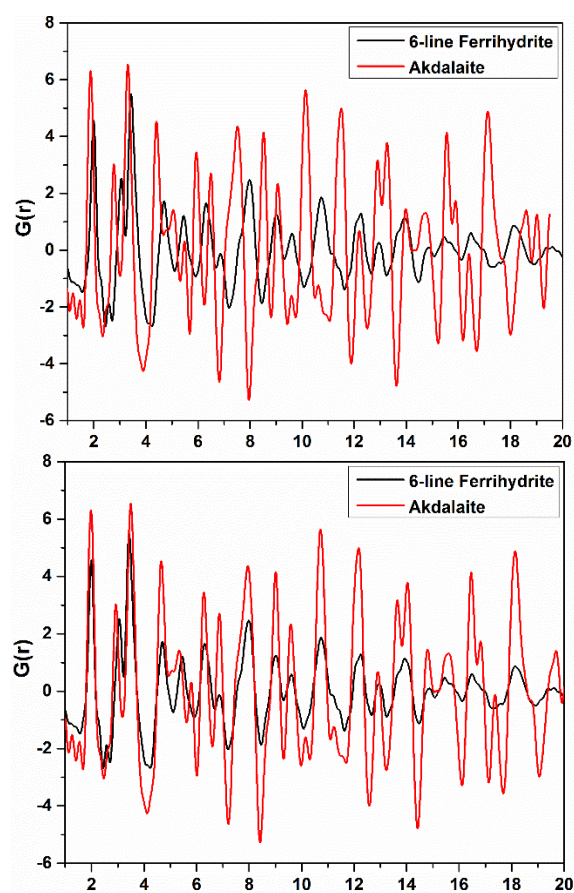


Figure S7. Calculated PDFs of hematite and corundum after the corundum data has been shifted to account for the different M–O differences with the fitted linear r -shift equation (Equation S1).



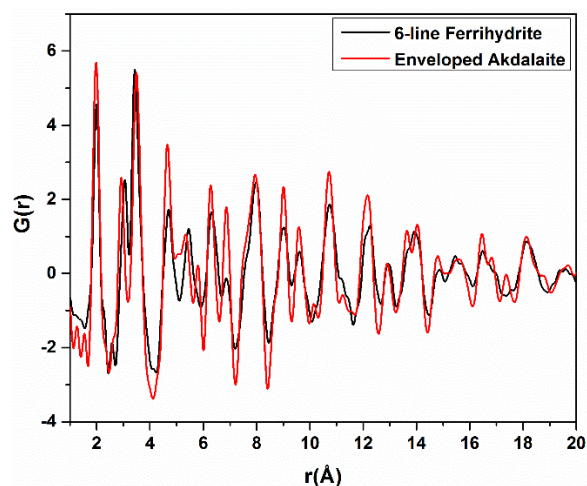


Figure S8. (top) Comparison of X-ray PDF data from crystalline akdalaite and 6-line ferrihydrite. (middle) X-ray PDF data from 6-line ferrihydrite and r-shifted akdalaite which has been adjusted to have approximately equivalent M–O distances as Fe–O. (bottom) Same shifted data as middle but the akdalaite data has had a spherical envelope function applied so intensities are representative of a 3-nm spherical particle.

4. CIFs

4.1 Akdalaite Single Crystal X-ray Diffraction

```

=====
data_global

_audit_update_record
;
2019-04-14 # Formatted by publCIF

;
_publCIF_datablock.id '{4462011d-d757-489e-b5e4-26a3042b1191}'
_audit_creation_method      'Jana2006 Version : 09/06/2017'

```

1. PROCESSING SUMMARY (Crystals Office Use Only)

```

_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_paper_category             ?
_journal_techeditor_notes           ?
?

```

_journal_codem_ASTM	?
_journal_name_full	'Crystals'
_journal_year	?
_journal_volume	?
_journal_issue	?
_journal_page_first	?
_journal_page_last	?
_journal_suppl_publ_number	?
_journal_suppl_publ_pages	?

#=====

2. SUBMISSION DETAILS

_publ_requested_journal	Crystals
_publ_contact_author_name	'Dr. John B. Parise'
_publ_contact_author_email	john.parise@stonybrook.edu
_publ_contact_author_phone	'(631) 632-8196'
_publ_contact_author_address	'Department of Geosciences, Stony Brook University, NY-11790, USA'

#=====

3. TITLE AND AUTHOR LIST

loop_
 _publ_author_name
 _publ_author_address
 'Parise, John B.' "
 'Xia, Bingying' "
 'Simonson, Jack W.' "
 'Woerner, William R.' "
 'Plonka, Anna M.' "
 'Phillips, Brian L.' "
 'Ehm, Lars' "

#=====

4. TEXT

_publ_section_references
 ;
 Petricek, V., Dusek, M. & Palatinus, L. (2006). Jana2006. Structure Determination Software Programs. Institute of Physics, Praha, Czech Republic.
 Palatinus, L. & Chapuis, G. (2007). <i>J. Appl. Cryst.</i> 40, 786--790.
 Brown, I. D. (1996). <i>J. Appl. Cryst.</i> 29, 479--480.
 ;

#=====

data_I

_publcif_datablock.id '{5cf2115b-00ac-4fa8-a71a-f44b05ffcab7}'

#=====

5. CHEMICAL DATA

_chemical_formula_moiety	'Al5 H1 O8'
_chemical_formula_sum	'Al5 H1 O8'
_chemical_formula_weight	263.9

#=====

6. CRYSTAL DATA

_symmetry_cell_setting	hexagonal
_symmetry_space_group_name_H-M	'P 63 m c'
_symmetry_space_group_name_Hall	'P 6c -2c'
_symmetry_Int_Tables_number	186

loop_

_space_group_symop_id	
_space_group_symop_operation_xyz	
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
7	-y,-x,z
8	-x+y,y,z
9	x,x-y,z
10	y,x,z+1/2
11	x-y,-y,z+1/2
12	-x,-x+y,z+1/2

_cell_length_a	5.6244(3)
_cell_length_b	5.6244(3)
_cell_length_c	8.8417(3)
_cell_angle_alpha	90.000(4)
_cell_angle_beta	90.000(4)
_cell_angle_gamma	120.000(6)
_cell_volume	242.22(2)

_cell_formula_units_Z	2
-----------------------	---

_cell_measurement_reflns_used	4264
_cell_measurement_theta_min	2.42
_cell_measurement_theta_max	23.83
_cell_measurement_temperature	100
_cell_special_details	
?	

```

_exptl_crystal_density_diffn      3.6184
_exptl_crystal_density_meas      ?
_exptl_crystal_density_method    ?
_exptl_crystal_F_000             260

_exptl_absorpt_coefficient_mu     0.265
_exptl_crystal_description        'plate'
_exptl_crystal_size_max           0.02
_exptl_crystal_size_mid          0.005
_exptl_crystal_size_min          0.005
_exptl_crystal_size_rad          ?
_exptl_crystal_colour             'colorless'
_exptl_absorpt_correction_type    analytical
_exptl_absorpt_process_details

```

```
;
```

CrysAlisPro 1.171.38.46 (Rigaku Oxford Diffraction, 2015)

Analytical numeric absorption correction using a multifaceted crystal
model based on expressions derived by R.C. Clark & J.S. Reid.

(Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897)

Empirical absorption correction using spherical harmonics,
implemented in SCALE3 ABSPACK scaling algorithm.

```
;
```

```

_exptl_absorpt_correction_T_min    0.995
_exptl_absorpt_correction_T_max    0.999

```

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

7. EXPERIMENTAL DATA

```

_diffn_ambient_temperature        100
_diffn_source                     'synchrotron'
_diffn_source_power               ?
_diffn_source_voltage              ?
_diffn_source_current              ?
_diffn_radiation_type              synchrotron
_diffn_radiation_source            ?
_diffn_radiation_wavelength        0.41328
_diffn_radiation_monochromator     'synchrotron'
_diffn_measurement_device          'four-circle diffractometer'
_diffn_measurement_device_type     'Saxi-CrysAlisPro-abstract goniometer imported
SAXI images'
_diffn_detector                    'CCD plate'
_diffn_detector_area_resol_mean    ?
_diffn_measurement_method          '\f scans'
_diffn_measurement_specimen_support ?

_diffn_reflns_number              5136
_diffn_reflns_theta_min            2.43
_diffn_reflns_theta_max            23.77
_diffn_reflns_theta_full           23.13
_diffn_measured_fraction_theta_max 0.96

```

_diffn_measured_fraction_theta_full	0.98
_diffn_reflns_av_R_equivalents	0.0471
_diffn_reflns_av_unetI/netI	0.0063
_diffn_reflns_limit_h_min	-10
_diffn_reflns_limit_h_max	10
_diffn_reflns_limit_k_min	-10
_diffn_reflns_limit_k_max	6
_diffn_reflns_limit_l_min	-16
_diffn_reflns_limit_l_max	17
_diffn_radiation_probe	x-ray

#=====

8. REFINEMENT DATA

_reflns_number_total	712
_reflns_number_gt	669
_reflns_threshold_expression	'I>3 \s(I)'
_iucr_refine_instructions_details	

;

JANA .m50 file contents
Version Jana2006
title
cell 5.6244 5.6244 8.8417 90 90 120
esdcell 0.0003 0.0003 0.0003 0.004 0.004 0.006
spgroup P63mc 186 6
lattice P
symmetry x y z
symmetry -y x-y z
symmetry -x+y -x z
symmetry -x -y z+1/2
symmetry y -x+y z+1/2
symmetry x-y x z+1/2
symmetry -y -x z
symmetry -x+y y z
symmetry x x-y z
symmetry y x z+1/2
symmetry x-y -y z+1/2
symmetry -x -x+y z+1/2
unitsnumb 2
chemform Al5 O8 H
formtab -62
atom Al atradius 1.43 color 129178214
atom O atradius 0.74 color 254003000
atom H atradius 0.46 color 255204204
roundmethod 1
end

contour
end contour
dist

```

angles 1 lstype 1 dmax 3.4
bondval Al O 1.644 0.38
bondval H O 0.569 0.94
end dist
fourier
  addbord 0 maptype 6 intmethod 2 intrad 1.6
end fourier
refine
  fsquare 1
  distfix 0.9655 0.001 O1 H1;
  keep ADP riding O1 1.2 H1
end refine

```

JANA .m40 file contents

```

  8  0  0  1
11.21502 0.000000 0.000000 0.000000 0.000000 0.000000 100000
0.000000 0.000000 00
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000 000000
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000 000000
Al1  1  2  0.500000 0.834322 0.668644 0.977450
0.001949 0.001604 0.002127 0.000802 0.000150 0.000301 0101111010
Al2  1  2  0.166667 0.333333 0.666667 0.769781
0.001700 0.001700 0.001520 0.000850 0.000000 0.000000 0001101000
Al3  1  2  0.166667 0.333333 0.666667 0.160387
0.001535 0.001535 0.001810 0.000768 0.000000 0.000000 0001101000
O1  2  2  0.166667 0.000000 0.000000 0.097059
0.002204 0.002204 0.000928 0.001102 0.000000 0.000000 0001101000
O2  2  2  0.500000 0.971877 0.485938 0.112879
0.001681 0.001987 0.002184 0.000841-0.000354-0.000177 0101111010
O3  2  2  0.500000 0.165937 0.834063 0.873912
0.001375 0.001375 0.002381 0.000303 0.000503-0.000503 0101101110
O4  2  2  0.166667 0.666667 0.333333 0.866656
0.001903 0.001903 0.000749 0.000951 0.000000 0.000000 0001101000
H1  3  1  0.166667 0.000000 0.000000 0.206196
0.002135 0.000000 0.000000 0.000000 0.000000 0.000000 0001000000
----- s.u. block -----
0.030289 0.000000 0.000000 0.000000 0.000000 0.000000
0.000000 0.000000
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
Al1  0.000000 0.000029 0.000058 0.000406
0.000109 0.000129 0.000164 0.000064 0.000048 0.000095
Al2  0.000000 0.000000 0.000000 0.000409
0.000154 0.000154 0.000292 0.000077 0.000000 0.000000
Al3  0.000000 0.000000 0.000000 0.000409
0.000145 0.000145 0.000276 0.000072 0.000000 0.000000
O1  0.000000 0.000000 0.000000 0.000444
0.000281 0.000281 0.000468 0.000141 0.000000 0.000000
O2  0.000000 0.000143 0.000072 0.000414
0.000234 0.000193 0.000281 0.000117 0.000228 0.000114
O3  0.000000 0.000088 0.000088 0.000415

```

```

0.000250 0.000250 0.000349 0.000258 0.000089 0.000089
O4          0.000000 0.000000 0.000000 0.000434
0.000333 0.000333 0.000531 0.000166 0.000000 0.000000
H1          0.000000 0.000000 0.000000 0.000519
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
----- Saved points -----
0
----- Fourier maxima -----
  1  1  1
max1  1  1  1.000000 0.322195 0.160714 0.065852 000 0 0 0
    0.90 0.27
max2  1  1  1.000000 0.359965 0.178571 0.473271 000 0 0 0
    0.65 0.20
max3  1  1  1.000000 0.289340 0.142857 0.442000 000 0 0 0
    0.53 0.17
max4  1  1  1.000000 0.517857 0.040437 0.211264 000 0 0 0
    0.49 0.11
max5  1  1  1.000000 0.669643 0.330357 0.364701 000 0 0 0
    0.63 0.10
max6  1  1  1.000000 0.330357 0.669643 0.057162 000 0 0 0
    1.02 0.09
max7  1  1  1.000000 0.051945 0.026786 0.736595 000 0 0 0
    0.43 0.09
max8  1  1  1.000000 0.595630 0.068857 0.690769 000 0 0 0
    0.47 0.08
max9  1  1  1.000000 0.044643 0.088834 0.193203 000 0 0 0
    0.32 0.08
max10 1  1  1.000000 0.330357 0.669643 0.454532 000 0 0 0
    0.83 0.07
----- Fourier minima -----
  1  1  1
min1  1  1  1.000000 0.160714 0.321277 0.448063 000 0 0 0
    0.49 0.10
min2  1  1  1.000000 0.500000 0.003803 0.015336 000 0 0 0
    0.56 0.09
min3  1  1  1.000000 0.169643 0.337088 0.090665 000 0 0 0
    0.45 0.09
min4  1  1  1.000000 0.302633 0.151786 0.168497 000 0 0 0
    0.54 0.08
min5  1  1  1.000000 0.553571 0.106412 0.037598 000 0 0 0
    0.35 0.07
min6  1  1  1.000000 0.316398 0.160714 0.316943 000 0 0 0
    0.31 0.07
min7  1  1  1.000000 0.824503 0.166046 0.762748 000 0 0 0
    0.71 0.07
min8  1  1  1.000000 0.356934 0.178571 0.376536 000 0 0 0
    0.38 0.07
min9  1  1  1.000000 0.633929 0.265238 0.111670 000 0 0 0
    0.43 0.07
min10 1  1  1.000000 0.000000 0.000000 0.333704 000 0 0 0
    0.59 0.07
-----

```

```

----- Atom objects -----
-----

;

_refine_ls_structure_factor_coef      Fsqd
_refine_ls_R_factor_gt               0.0255
_refine_ls_wR_factor_gt              0.0618
_refine_ls_R_factor_all              0.0275
_refine_ls_wR_factor_ref             0.0621
_refine_ls_goodness_of_fit_ref       2.33
_refine_ls_goodness_of_fit_gt        2.39
_refine_ls_number_reflns             712
_refine_ls_number_parameters         32
_refine_ls_number_restraints         1
_refine_ls_number_constraints        2
_refine_ls_weighting_scheme          sigma
_refine_ls_weighting_details         'w=1/(\s^2(I)+0.0004I^2^)'
_refine_ls_hydrogen_treatment        reffall
_refine_ls_shift/su_max              0.0146
_refine_ls_shift/su_mean             0.0060
_refine_diff_density_max              1.00
_refine_diff_density_min             -0.70
_refine_ls_extinction_method         'none'
_atom_sites_solution_primary         iterative
_atom_sites_solution_secondary       difmap
_refine_ls_abs_structure_details
; 331 of Friedel pairs used in the refinement
;

loop_
  _atom_type_symbol
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
Al  0.0201  0.0167
'International Tables Vol C tables 4.2.6.8 and 6.1.1.1'
H  0.0000  0.0000
'International Tables Vol C tables 4.2.6.8 and 6.1.1.1'
O  0.0018  0.0018
'International Tables Vol C tables 4.2.6.8 and 6.1.1.1'

_computing_data_collection
'CrysAlisPro 1.171.38.46 (Rigaku OD, 2015)'
_computing_cell_refinement
'CrysAlisPro 1.171.38.46 (Rigaku OD, 2015)'
_computing_data_reduction
'CrysAlisPro 1.171.38.46 (Rigaku OD, 2015)'

loop_
  _restr_distance_atom_site_label_1

```

```

_restr_distance_site_symmetry_1
_restr_distance_atom_site_label_2
_restr_distance_site_symmetry_2
_restr_distance_target
_restr_distance_target_weight_param
O1 . H1 . 0.9655 0.001

```

```

#=====

```

9. ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

```

loop_
_atom_site_label
_atom_site_type_symbol
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_adp_type
_atom_site_U_iso_or_equiv
_atom_site_site_symmetry_multiplicity
_atom_site_occupancy
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Al1 Al 0.83432(3) 0.66864(6) 0.9775(4) Uani 0.00193(10) 6 1 d . . .
Al2 Al 0.333333 0.666667 0.7698(4) Uani 0.00164(14) 2 1 d . . .
Al3 Al 0.333333 0.666667 0.1604(4) Uani 0.00163(13) 2 1 d . . .
O1 O 0 0 0.0971(4) Uani 0.0018(2) 2 1 d . . .
O2 O 0.97188(14) 0.48594(7) 0.1129(4) Uani 0.00198(17) 6 1 d . . .
O3 O 0.16594(9) 0.83406(9) 0.8739(4) Uani 0.0019(2) 6 1 d . . .
O4 O 0.666667 0.333333 0.8667(4) Uani 0.0015(3) 2 1 d . . .
H1 H 0 0 0.2062(5) Uiso 0.0021 2 1 d . . .

```

```

loop_
_atom_site_aniso_label
_atom_site_aniso_type_symbol
_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
Al1 Al 0.00195(11) 0.00160(13) 0.00213(16) 0.00080(6) 0.00015(5) 0.00030(10)
Al2 Al 0.00170(15) 0.00170(15) 0.0015(3) 0.00085(8) 0 0
Al3 Al 0.00154(15) 0.00154(15) 0.0018(3) 0.00077(7) 0 0
O1 O 0.0022(3) 0.0022(3) 0.0009(5) 0.00110(14) 0 0
O2 O 0.0017(2) 0.00199(19) 0.0022(3) 0.00084(12) -0.0004(2) -0.00018(11)
O3 O 0.0014(3) 0.0014(3) 0.0024(3) 0.0003(3) 0.00050(9) -0.00050(9)
O4 O 0.0019(3) 0.0019(3) 0.0007(5) 0.00095(17) 0 0

```

#=====

4.1 Akdalaite Powder Neutron Diffraction

```

data_AKDALAITE-ISO-H-ANIS_publ
_audit_creation_method "from EXP file using GSAS2CIF"
_audit_creation_date      2019-04-14T07:36
_audit_author_name       Bill
_audit_update_record
; 2019-04-14T07:36 Initial CIF as created by GSAS2CIF
;
_publ_requested_journal   Crystals
_publ_contact_author_name 'Dr. John B. Parise'
_publ_contact_author_email john.parise@stonybrook.edu
_publ_contact_author_phone '(631) 632-8196'
loop_
_publ_author_name
_publ_author_address
'Parise, John B.' "
'Xia, Bingying' "
'Simonson, Jack W.' "
'Woerner, William R.' "
'Plonka, Anna M.' "
'Phillips, Brian L.' "
'Ehm, Lars' "

_cell_length_a      5.576099(17)
_cell_length_b      5.5761
_cell_length_c      8.77247(6)
_cell_angle_alpha   90.0
_cell_angle_beta    90.0
_cell_angle_gamma   120.0
_cell_volume        236.2180(20)
_symmetry_cell_setting hexagonal
_symmetry_space_group_name_H-M "P 63 m c"
_chemical_formula_sum 'Al5 H1 O8'
_chemical_formula_weight 263.9
_cell_formula_units_Z 2
loop_ _symmetry_equiv_pos_site_id _symmetry_equiv_pos_as_xyz
1 +x,+y,+z
2 x-y,+x,+z+1/2
3 -y,x-y,+z
4 -x,-y,+z+1/2
5 y-x,-x,+z
6 +y,y-x,+z+1/2
7 y-x,+y,+z
8 -x,y-x,+z+1/2
9 -y,-x,+z
10 x-y,-y,+z+1/2

```


11 +x,x-y,+z
 12 +y,+x,+z+1/2

ATOMIC COORDINATES AND DISPLACEMENT PARAMETERS

loop_

_atom_site_type_symbol
 _atom_site_label
 _atom_site_fract_x
 _atom_site_fract_y
 _atom_site_fract_z
 _atom_site_occupancy
 _atom_site_thermal_displace_type
 _atom_site_U_iso_or_equiv
 _atom_site_symmetry_multiplicity

Al

Al1	0.83409(19)	0.6683(4)	0.97855(33)	1.0	Uani	0.00505	6
Al							
Al2	0.33333	0.66667	0.7690(7)	1.0	Uani	0.00946	2
Al							
Al3	0.3333	0.6667	0.1609(4)	1.0	Uani	0.00595	2
O							
O1	0.0	0.0	0.0970(4)	1.0	Uani	0.00507	2
O							
O2	0.97280(18)	0.48642(9)	0.11433(27)	1.0	Uani	0.00588	6
O							
O3	0.16458(25)	0.83542(25)	0.87450(31)	1.0	Uani	0.00636	6
O							
O4	0.66667	0.33333	0.86817	1.0	Uani	0.00548	2
H							
H1	0.0423(12)	0.0212(6)	0.2134(7)	0.33333	Uiso	0.0152(12)	6

loop_ _atom_site_aniso_label

_atom_site_aniso_U_11
 _atom_site_aniso_U_12
 _atom_site_aniso_U_13
 _atom_site_aniso_U_22
 _atom_site_aniso_U_23
 _atom_site_aniso_U_33

Al1	0.00350(34)	0.00201(24)	0.00168(25)	0.0040(5)	0.0034(5)	0.0078(6)
Al2	0.0103(8)	0.0052(4)	0.0	0.0103(8)	0.0	0.0078(14)
Al3	0.0039(4)	0.00196(19)	0.0	0.0039(4)	0.0	0.0100(13)
O1	0.0047(4)	0.00236(20)	0.0	0.0047(4)	0.0	0.0057(8)
O2	0.00475(29)	0.00238(14)	-0.0014(4)	0.00505(25)	-0.00068(19)	0.0077(4)
O3	0.00586(34)	0.0037(5)	0.00210(16)	0.00586(34)	-0.00210(16)	0.0084(4)
O4	0.0060(5)	0.00298(27)	0.0	0.0060(5)	0.0	0.0045(10)

loop_ _atom_type_symbol

_atom_type_number_in_cell
 O 16.0
 Al 10.0

H 2.0

References

1. Hammersley, A.P.; Svensson, S.O.; Hanfland, M.; Fitch, A.N.; Hausermann, D. Two-dimensional detector software: From real detector to idealised image or two-theta scan. *High Pressure Res* **1996**, *14*, 235-248.
2. Qiu, X.; Thompson, J.W.; Billinge, S.J.L. PDFgetX2: a GUI-driven program to obtain the pair distribution function from X-ray powder diffraction data. *Journal of Applied Crystallography* **2004**, *37*, 678.
3. Chupas, P.J.; Qiu, X.Y.; Hanson, J.C.; Lee, P.L.; Grey, C.P.; Billinge, S.J.L. Rapid-acquisition pair distribution function (RA-PDF) analysis. *Journal of Applied Crystallography* **2003**, *36*, 1342-1347.
4. Farrow, C.L.; Juhas, P.; Liu, J.W.; Bryndin, D.; Bozin, E.S.; Bloch, J.; Proffen, T.; Billinge, S.J.L. PDFfit2 and PDFgui: computer programs for studying nanostructure in crystals. *J Phys-Condens Mat* **2007**, *19*.
5. Maslen, E.N.; Streltsov, V.A.; Streltsova, N.R.; Ishizawa, N.; Satow, Y. Synchrotron X-Ray Study of the Electron-Density in Alpha-Al₂O₃. *Acta Crystallogr B* **1993**, *49*, 973-980.
6. Maslen, E.N.; Streltsov, V.A.; Streltsova, N.R.; Ishizawa, N. Synchrotron X-Ray Study of the Electron-Density in Alpha-Fe₂O₃. *Acta Crystallogr B* **1994**, *50*, 435-441.
7. Michel, F.M.; Ehm, L.; Liu, G.; Han, W.Q.; Antao, S.M.; Chupas, P.J.; Lee, P.L.; Knorr, K.; Eulert, H.; Kim, J., et al. Similarities in 2- and 6-line ferrihydrite based on pair distribution function analysis of X-ray total scattering. *Chem Mater* **2007**, *19*, 1489-1496.
8. Neder, R.B.; Proffen, T. *Diffuse Scattering and Defect Structure Simulations: a cook book using the program DISCUS*; Oxford University Press: Oxford, 2008; Vol. 11, pp. 228.