Appendix A. TOPAS input file showing the CaF_2 structural model and the nontronite supercell model used for supercell mode calibration. The Rietveld quantification formula is reversed and used to refine the interlayer cation occupancy of the nontronite supercell model. This is used to determine the Bulong nontronite unit cell mass and volume with known weight fractions of standard mixtures. The bold text highlights the novel calibration coding developed for this paper.

```
'--------------------------------------------------------------------------------
'CaF2 generated from best CaF2 synchrotron pattern fitting.
str
        prm cmCaF2 = Get(cell_mass);
        prm cvCaF2 = Get(cell_volume);
        LVol_FWHM_CS_G_L( 1, 0, 0.89, 0,@, 1000 min =500; max =10000;,@, 1000 min =500; max =10000;)
        e0_from_Strain( 0,@, 0.05 min =0; max =0.05;,@, 0.05 min =0; max =0.05;)
        r_bragg 1.39482605
        phase_name "Calcium Fluorite"
        MVW(312.300, 163.213765`_0.000194236569, 100.000`_0.000)
        scale !scCaF2 0.00554598749
        space_group Fm-3m
        Phase_LAC_1_on_cm( 110.76187`_0.00013)
        Phase_Density_g_on_cm3( 3.17734`_0.00000)
        Cubic(!afluorite  5.46494`_0.00000 min =5.45; max =5.49;)
        site Ca1 num_posns  4 x =0; : 0.00000 y =0; : 0.00000 z =0; : 0.00000 occ Ca+2 1 beq 0.5888 min 0.1 max 1
        site F1 num_posns  8 x =1/4; : 0.25000 y =1/4; : 0.25000 z =1/4; : 0.25000 occ F-1 1 beq 0.7937 min 0.1 max 1
'--------------------------------------------------------------------------------
'turbostratically disordered smectite d(001) approx. 15 A
'structure of TOT layer (cis-vacant) by: Tsipurski et al., Clay Minerals 19(1984), 177-193 (modified)
'model for turbostratic disorder by: Ufer et al., Z. Kristallogr. 219(2004), 519-527
str
        prm interlayer 0.39816`_0.00631
        'local scnon 0.00041`_0.00000 min 0.0000001 max 1 'scale factor
        prm mshkl 0.01999`_0.03602_LIMIT_MIN_5e-005 min 0.00005 max 0.02 'strain of non-basal reflection
        prm ms00l 6.73911`_0.32386 min 0.001 max 10 'strain of basal reflection
        prm cshkl 15.63019`_0.40663 min 10 max 1000 'size of non-basal reflections
        prm cs00l 9.20520`_0.17236 min 1 max 20 'size of basal reflections

        'occupancies of cations
        prm !pMg 0.065 min 0 max 1 prm !pNi 0.065 min 0 max 1 prm !pFe 0.6 min 0 max 1 prm !pAl 0.2375 min 0 max
1 'octahedral position, Mg + Fe + Al +Ni < 1.
        prm !ptrans 1 min 0 max 1 'mixing parameter for cis- and trans-vacancy; 0 => trans-vacant.
        prm !altet 0.08375 min 0 max 1 prm sitet =1-altet; 0.91625 'tetrahedral site: Al + Si = 1.
        prm pNa = interlayer 0.717;:0.31488`_0.00098 min 0 prm pMgI = interlayer 0.287;:0.12604`_0.00039 min 0
prm pH2O = pNa+pMgI;: 0.44092`_0.00105 min 0 'interlayer H2O
```

```
        'atom absolute position in c direction to avoid a stretching/shortening of the TOT layer by varying c0
        prm !zT 2.7135
        prm !zO11 1.0955
        prm !zO12 1.0553
        prm !zO2 3.3668

        prm !layer 9 'layer: supercell factor for elongation in c direction
        prm cnon 15.61679`_0.00516 min 14 max 15.8 'subcell c

        lor_fwhm = If(And(H==0,K==0), 0.1 Rad Lam/(Cos(Th) cs00l), 0.1 Rad Lam/(Cos(Th) cshkl)); 'thinner in 00l direction
        lor_fwhm = If(And(H==0,K==0), ms00l Tan(Th), Sqrt((mshkl Tan(Th))^2 + L^2/(Get(c)^2 L^2 + Get(c)^4 ((H/Get(a))^2+(K/Get(b))^2))) ); 'aniso strain and additional l-dependent broadening to avoid "ripples"
        scale_pks = If(And(H==0,K==0),If(Mod(L,layer),0,layer),1); 'scaling of classes (00l and hkl) and removal of redundant 00l reflections
        'scale =scnon;
        scale = cmCaF2 cvCaF2 scCaF2/Get(cell_mass)/Get(cell_volume)/0.100948577134299*0.899051422865701; :8.1020183e-006_4.48e-007

        r_bragg  2.35613318
        phase_name "Bulong supercell"
        MVW( 999.686`_0.654, 6620.83728`_2.49863214, 98.741`_0.070)
        'corrected_weight_percent  42.9984672_0.601779145
        space_group 5
        Phase_LAC_1_on_cm( 6.48560`_0.00594)
        Phase_Density_g_on_cm3( 0.25073`_0.00019)
        a anon  5.25575`_0.00063_LIMIT_MIN_5.25 min 5.25 max 5.35
        b !bnon  9.09592434 min 9.1 max 9.2
        c =layer*cnon; 'supercell c
        be benon  99.81479`_0.04551

        'Na+0.716Mg2+0.287[Si4+7.33Al3+0.67][Al3+0.95Fe3+2.40Ni2+0.26Co2+0.01Mg2+0.26Cr3+0.09]O20(OH)4 'Bulong Nontronite
'octahedral trans
        site Al1   num_posns 2  x =0;:0.00000  y =0;:0.00000  z =0;:0.00000 occ Al+3 =ptrans*pAl;         beq 0.5
        site Mg1  num_posns 2  x =0;:0.00000  y =0;:0.00000  z =0;:0.00000 occ Mg+2 =ptrans*pMg;       beq 0.5
        site Fe1   num_posns 2  x =0;:0.00000  y =0;:0.00000  z =0;:0.00000 occ Fe+3 =ptrans*pFe;         beq 0.5
        site Ni1   num_posns 2  x =0;:0.00000  y =0;:0.00000  z =0;:0.00000 occ Ni+2 =ptrans*pNi;         beq 0.5
'octahedral cis
        site Al2   num_posns 2  x =0;:0.00000  y 0.6540  z =0;:0.00000       occ Al+3 =(1-ptrans)*pAl;    beq 0.5
        site Mg2  num_posns 2  x =0;:0.00000  y 0.6540  z =0;:0.00000       occ Mg+2 =(1-ptrans)*pMg; beq 0.5
        site Fe2   num_posns 2  x =0;:0.00000  y 0.6540  z =0;:0.00000       occ Fe+3 =(1-ptrans)*pFe;    beq 0.5
        site Ni2   num_posns 2  x =0;:0.00000  y 0.6540  z =0;:0.00000       occ Ni+2 =(1-ptrans)*pNi;    beq 0.5
        site Al3   num_posns 2  x =0;:0.00000  y 0.3210  z =0;:0.00000       occ Al+3 =pAl;      beq 0.5
```

```
        site Mg3  num_posns 2  x =0;:0.00000  y 0.3210  z =0;:0.00000        occ Mg+2 =pMg;   beq 0.5
        site Fe3  num_posns 2  x =0;:0.00000  y 0.3210  z =0;:0.00000        occ Fe+3 =pFe;   beq 0.5
        site Ni3  num_posns 2  x =0;:0.00000  y 0.3210  z =0;:0.00000        occ Ni+2 =pNi;   beq 0.5
'tetrahedral
        site Si1  num_posns 4  x 0.4320  y =1/3;:0.33333  z =zT/(layer*cnon);        occ Si+4 =sitet;  beq 0.4
        site Al4  num_posns 4  x 0.4320  y =1/3;:0.33333  z =zT/(layer*cnon);        occ Al+3 =altet;  beq 0.4
        site Si2  num_posns 4  x 0.4320  y =2/3;:0.66667  z =zT/(layer*cnon);        occ Si+4 =sitet;  beq 0.4
        site Al5  num_posns 4  x 0.4320  y =2/3;:0.66667  z =zT/(layer*cnon);        occ Al+3 =altet;  beq 0.4

        site O7   num_posns 4  x 0.1730  y 0.7250  z =zO2/(layer*cnon);     occ O-2 1beq 0.8
        site O8   num_posns 4  x 0.1700  y 0.2680  z =zO2/(layer*cnon);     occ O-2 1beq 0.8
        site O9   num_posns 4  x 0.4170  y 0.6560  z =zO11/(layer*cnon);    occ O-2 1beq 0.8
        site O10  num_posns 4  x 0.3430  y 0.3470  z =zO11/(layer*cnon);    occ O-2 1beq 0.8
        site O11  num_posns 4  x 0.3340  y -0.0240  z =zO12/(layer*cnon);   occ O-2 1beq 0.8
        site O12  num_posns 4  x 0.4890  y 0.4960  z =zO2/(layer*cnon);     occ O-2 1beq 0.8
'interlayer
        site Mg   num_posns  4  x 0.50000  y 0.25000  z 0.05556   occ Mg+2 =pMgI;  beq 0.8
        site Na   num_posns  4  x 0.50000  y 0.25000  z 0.05556   occ Na+1 =pNa;   beq 0.8
        site O1   num_posns  4  x 0.35860  y 0.43735  z 0.06560   occ O-2 =pH2O;   beq 2.5
        site O2   num_posns  4  x 0.64140  y 0.06265  z 0.04551   occ O-2 =pH2O;   beq 2.5
        site O3   num_posns  4  x 0.07980  y 0.25000  z 0.04551   occ O-2 =pH2O;   beq 2.5
        site O4   num_posns  4  x 0.92020  y 0.25000  z 0.06560   occ O-2 =pH2O;   beq 2.5
        site O5   num_posns  4  x 0.35860  y 0.06265  z 0.06560   occ O-2 =pH2O;   beq 2.5
        site O6   num_posns  4  x 0.64140  y 0.43735  z 0.04551   occ O-2 =pH2O;   beq 2.5
'rigid body of the interlayer complex
prm !dMgO 2.41
rigid
point_for_site Mg
point_for_site Na
point_for_site O1 uz = dMgO;
point_for_site O2 uz =-dMgO;
point_for_site O3 ux = dMgO;
point_for_site O4 ux =-dMgO;
point_for_site O5 uy = dMgO;
point_for_site O6 uy =-dMgO;

rotate 45 qx 1 operate_on_points "O*"
rotate =180/Pi*ArcSin(1/Sqrt(3)); qy 1 operate_on_points "O*"
rotate 180 qz 1 operate_on_points "O*"
translate tx =0.5*anon+0.5*cnon*Cos(benon Deg); ty =0.5*bnon; tz =0.5*cnon*Sin(benon Deg); operate_on_points "O*
Mg Na"
```