and stacking fault composition planes seems unlikely (Fleet, 1982*a*). No evidence for either coherent or incoherent twinning in magnetite has been observed in the recent studies by the author. Striated magnetites, such as 2741, have been subjected to particularly close scrutiny in this regard. The striations appear to be the traces of {111} growth planes.

One explanation consistent with the available data is that the defects were introduced during crystal growth. The present limited data do point to a correlation between point defect concentration and density of growth striations. Hence the interstitial cation defects may be concentrated on {111} growth planes. However, the density of such planes would have to be appreciably greater than the microscopically resolved striation density to account for the defect concentration observed in magnetite 2741.

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Structure of Dealuminated Linde Y-Zeolite; $Si_{139\cdot7}Al_{52\cdot3}O_{384}$ and $Si_{173\cdot1}Al_{18\cdot9}O_{384}$: Presence of Non-framework Al Species

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Abstract. Neutron powder diffraction techniques ($\lambda = 2\cdot3841$ Å) have been used to study samples of Linde Y-zeolite treated with NH_3/steam and gaseous $SiCl_4$. The respective compositions from chemical analysis are $Si_{139\cdot7}Al_{52\cdot3}O_{384}$ and $Si_{173\cdot1}Al_{18\cdot9}O_{384}$ ($Z = 1$), and Si/Al framework ratios from magic-angle spinning nuclear magnetic resonance are 10·5 and 10·8. The space group is $Fd3m$, origin at center ($\bar{3}m$). A combination of Rietveld refinement and difference Fourier techniques gives clear evidence for the presence of a four-coordinated Al species in the center of the sodalite unit in both materials, but no six-coordinated species nor the previously reported $Na^+AlCl_4^-$ species could be detected in the difference maps. The refinements give $a = 24\cdot358\,(1)$ Å, $V = 14451\,(1)$ Å^3, $R_{wp} = 10\cdot77$, $R_e = 6\cdot57$, $R_I = 6\cdot95$ for the NH_3/steamed sample at 293 K; $a = 24\cdot188\,(1)$ Å, $V = 14152\,(2)$ Å^3, $R_{wp} = 10\cdot61$, $R_e = 6\cdot02$, $R_I = 5\cdot61$, for the $SiCl_4$-treated sample at 293 K; and $a = 24\cdot171\,(1)$ Å, $V = 14122\,(2)$ Å^3, $R_{wp} = 10\cdot48$, $R_e = 5\cdot95$, $R_I = 5\cdot29$ for the $SiCl_4$-treated sample at 573 K.

Introduction. The removal of aluminum from the framework of Y-type zeolite by the action of steam (Scherzer, 1978, and references therein) or by treatment with gaseous $SiCl_4$ (Beyer & Belenykaja, 1980) has been discussed extensively. Such treatments produce so called 'ultrastable' zeolite Y, a form of the compound resistant to degradation and the mainstay of the petroleum cracking industry. Recent applications of magic-angle spinning nuclear magnetic resonance (MASNMR) have confirmed the removal of aluminum from the framework of the zeolite under the conditions of steaming (Maxwell, van Erp, Hays, Couperus, Huis & Clague, 1982) and also the entrainment of

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extra-framework Al species, resistant to removal from the zeolite, subsequent to dealumination (Klinowski, Thomas, Fyfe & Gobbi, 1982; Klinowski, Thomas, Fyfe, Gobbi & Hartman, 1983). The MASNMR evidence suggests this to be an octahedral Al species.

Pluth & Smith (1982) have reported the presence of a tetrahedrally coordinated extra-framework Al species in the sodalite units of type-A zeolite. Sodalite units also occur in Linde Y-zeolite, and this unit might be expected to contain Al species difficult to remove since access to it is restricted to six-ring and four-ring openings (Figs. 1 and 2). Unwashed samples of dealuminated zeolite Y are known to contain $AlCl_4^-$ species (Klinowski, Thomas, Fyfe, Gobbi & Hartman, 1983).

We report here a structural investigation of two Y-type zeolites using neutron powder diffraction techniques, one dealuminated with the use of NH_3/steam and one with the use of $SiCl_4$; the main objectives being to determine structural changes occurring during Al removal from the framework and to attempt to locate any entrained Al species. Prior to this work, refinements of zeolite Y using neutron powder diffraction data have been reported by Jirák, Vratislav, Zajácek & Bosácek (1977), Jirák, Vratislav & Bosácek (1980), and by Bosácek, Beran & Jirák (1981). These authors inferred the position of hydrogen atoms associated with the framework and refined their occupation factors but did not report the use of Fourier techniques in their studies.

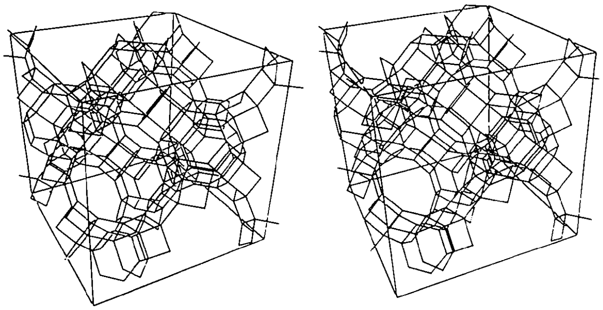

Fig. 1. Framework structure of zeolite Y. Nodes represent Si/Al sites. Oxygen atoms, omitted for clarity, lie at the approximate midpoint of the straight line segments (*ORTEP*; Johnson, 1965).

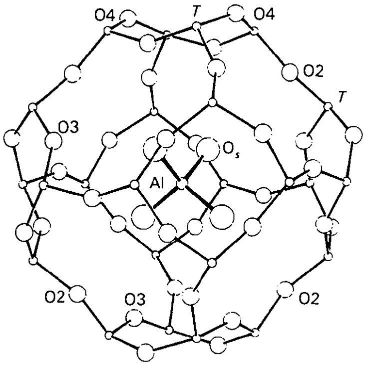

Fig. 2. Drawing (Johnson, 1965) of the sodalite unit in zeolite Y showing the disposition of the entrained tetrahedral aluminate species. Small circles are T(=Si/Al) atoms, larger circles are the oxygen atoms.

Table 1. *Experimental details for samples of dealuminated Linde Y-zeolite*

Sample treatment		
Sample designation	D_2O–Y	Si–Y
Treatment	NH_3/steam	$SiCl_4$/Ar
Temperature/time	823 (10)K/6 h	823 (10)K/6 h
Wash	No	H_2O
Si/Al framework ratio (^{29}Si MASNMR)	10·5	10·8
Al per unit cell (NMR)	16·7	16·3
Chemical analysis	$Si_{139\cdot7}Al_{52\cdot3}O_{384}$	$Si_{173\cdot1}Al_{18\cdot9}O_{384}$
Neutron diffraction		
Monochromator	Pyrolytic graphite (002)	
Analyzer	Pyrolytic graphite (004)	
Wavelength	2·3841 Å	
Collimation	20–40–40–20′ arc	
Monochromator mosaic spread	~25′ arc	
Sample container	Al can ~16 mm in diameter	
Angular ranges scanned	D_2O–Y	7·0–127·9°
	Si–Y	7·0–121·7°
	Si–Y–HT	7·0–128·3°
Angular step	0·1°	

R-factor definitions

$R_{wp} = 100[\sum w_i(y_o-y_c)^2/\sum w_i(y_o)^2]^{1/2}$

$R_e = 100[(N-P+C)/\sum w_i(y_o)^2]^{1/2}$

$R_I = 100[\sum|I_{obs}-I_{calc}|/\sum I_{obs}]$

w_i is the weight = $1/(y_{obs})$; y_{obs} is the observed intensity; y_o is the background-corrected intensity; y_c is the calculated intensity; N is the number of statistically independent observations; P is the number of refined parameters; and C the number of constraint functions. I_{obs} and I_{calc} are the observed and calculated integrated intensities determined according to the approximations of Rietveld (1969*a*, *b*).

Experimental. Two 15 g batches of stabilized zeolite Y (LZ-Y82) were dealuminated using NH_3/steam and gaseous $SiCl_4$ (Table 1). The NH_3/steamed sample (henceforth designated D_2O–Y) was not washed, while the $SiCl_4$-treated sample (designated Si–Y) was washed on filter paper with H_2O until no trace of chloride could be detected in the filtrate. It was hoped that in this fashion a comparison between material with a significant amount of some Al species trapped in its pores and one with essentially no extra-framework species could be made. Measurements using ^{29}Si MASNMR confirmed that both materials had similar Si/Al framework ratios (Table 1). The Si/Al chemical ratios obtained by chemical analysis (Table 1) and the cell dimensions (Breck & Flanigen, 1968) indicate that D_2O–Y contains significant extra-framework material.

Both samples were dehydrated and exchanged with D_2O on a vacuum rack. This served to decrease the background due to the incoherent scattering of neutrons by any residual hydrogen. Before being loaded into sample containers in a helium dry box, the zeolites were dehydrated at 573 K for 16 h in a vacuum better than 0·13 Pa.

The neutron diffraction data were collected at 293 K at the high-flux beam reactor at the Brookhaven National Laboratory and also at 573 K for the sample Si–Y (designated Si–Y–HT). Even in 'dry' boxes, the loading of zeolite samples can introduce some moisture, and the refinement of data collected at 573 K provides an opportunity of distinguishing adsorbed H_2O from species less likely to desorb at these temperatures. A summary of experimental details is given in Table 1.

The data were analyzed using the Rietveld technique (Rietveld, 1969*a*, *b*; Hewat, 1973). Fourier, interatomic distance and interatomic angle calculations were performed using programs described by Finger & Prince (1975). Drawings of the structure were carried out using *ORTEP* (Johnson, 1965). All computations employed local VAX 11/780 computers.

The scattering from the T sites (T designating the averaged atom representing the scattering from the Si and Al atoms which are randomly distributed in the tetrahedral sites of the framework of zeolite Y) was fixed at a value consistent with the Si/Al ratio determined from ^{29}Si MASNMR (Klinowski, Thomas, Fyfe & Gobbi, 1982). The site was assumed to be fully occupied, this having been established in previous studies of dealuminated zeolite Y (see, for example, Gallezot, Beaumont & Barthomeuf, 1974). The scattering amplitudes used were $b(\mathrm{Si}) = 4{\cdot}149$, $b(\mathrm{Al}) = 3{\cdot}449$, and $b(\mathrm{O}) = 5{\cdot}805$ fm (Koester & Rauch, 1981).

Background was estimated at various points between the Bragg peaks and subtracted from the observed diffraction pattern prior to structural refinement. The peak shapes were presumed to be Gaussian except below 14° in 2θ where the correction introduced by Rietveld (1969*a*, *b*) for peak asymmetry was carried out. No allowance was made for adsorption, and peaks from the Al sample holder were excluded from the refinement. The quantity minimized was $\sum w[y_o-(1/c)y_c]^2$ (Table 1), where $w = 1/y_i(\mathrm{obs})$, $y_i(\mathrm{obs})$ being the observed intensity before background subtraction.

With the use of the atomic parameters of Baur (1964) for the framework atoms as starting values, refinement of the data for D_2O–Y led to the following discrepancy indices defined in Table 1: $R_{wp} = 11{\cdot}94$, $R_e = 6{\cdot}62$, $R_I = 9{\cdot}42$. A difference Fourier map at this stage contained two prominent peaks near the center of the sodalite unit (Fig. 2) at ($\frac{1}{8}, \frac{1}{8}, \frac{1}{8}$) and at (0·085, 0·085, 0·085). From the distance between the peaks (~1·7 Å) they were assigned to Al_s and O_s, respectively (s for sodalite). Since their occupation factors, when refined independently, were approximately in the ratio $f(\mathrm{Al}_s)/f(\mathrm{O}_s) = \frac{1}{4}$, the occupancy of the site Al_s was henceforth constrained to be $f(\mathrm{O}_s)/4$. Constraints were also placed on the thermal parameters for the framework oxygens, Al_s and O_s (Table 2), since allowing them to vary caused very little change in the discrepancy index R_{wp}. The results of a final refinement in which a scale factor, symmetry-independent positional parameters, isotropic thermal parameters, profile parameters, the cubic cell constant, and an asymmetry parameter (21 in all) were varied are given in Table 2. A diagram showing the final fit between observed and calculated diffraction patterns has been deposited.*

Table 2. *Refined atomic parameters and their e.s.d.'s at 293 K for Linde Y-zeolite dealuminated using* NH_3/*steam* (D_2O–Y) *and* $SiCl_4$ (Si–Y) *and at* 593 K *for* Si–Y (Si–Y–HT)

The data set Si–Y–HT is Si–Y collected at 593 K.

	D_2O–Y	Si–Y	Si–Y–HT
T = Si/Al[192(i)]			
x	0·1254 (4)	0·1257 (4)	0·1260 (4)
y	0·9464 (4)	0·9460 (4)	0·9460 (4)
z	0·0359 (4)	0·0359 (4)	0·0362 (4)
B(Å^2)	2·3 (1)	3·1 (2)	3·1 (1)
O(1)[96(h)]			
$x = -y$	0·1057 (3)	0·1076 (2)	0·1076 (3)
z	0	0	0
B(Å^2)*	3·41 (7)	3·58 (7)	4·50 (7)
O(2)[96(g)]			
$x = y$	0·2535 (3)	0·2539 (3)	0·2536 (3)
z	0·1418 (4)	0·1406 (4)	0·1406 (4)
B(Å^2)*	3·41	3·58	4·50
O(3)[96(g)]			
$x = y$	0·1761 (3)	0·1747 (2)	0·1752 (3)
z	0·9656 (4)	0·9634 (4)	0·9633 (4)
B(Å^2)*	3·41	3·58	4·50
O(4)[96(g)]			
$x = y$	0·1774 (4)	0·1798 (3)	0·1799 (3)
z	0·3199 (5)	0·3217 (4)	0·3215 (4)
B(Å^2)	3·41	3·58	4·50
Al_s[8(a)]†			
$x = y = z$	$\frac{1}{8}$	$\frac{1}{8}$	$\frac{1}{8}$
Population	1·3	0·7	0·7
O_s[32(e)]†			
$x = y = z$	0·085 (2)	0·085‡	0·085‡
Population	5·2 (3)	2·8 (3)	2·8 (3)
a(Å)	24·358 (1)	24·188 (1)	24·171 (1)
R_{wp}	10·77	10·41	10·48
R_e	6·57	6·02	5·95
R_I	6·95	5·61	5·29

* Thermal parameters for framework oxygens constrained to be equal.

† Thermal parameters fixed at 3·0 Å^2, occupancy of Al_s constrained to be equal to $\frac{1}{4}$ that of O_s.

‡ Position fixed at $x = y = z = 0{\cdot}085$.

In order to test the effects of uncertainty in the fitting of the asymmetric peak corresponding to (111) on the occupancies of Al_s and O_s in the sodalite unit, this reflection was excluded from a trial refinement. The occupancies of the sites in question were unaffected,

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* Diagrams and numerical data corresponding to the observed and calculated diffraction patterns, final Fourier and difference Fourier maps, and the primary diffraction data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39454 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and all parameters were refined to within one e.s.d. of the values obtained when this peak was included.

A similar set of refinements was repeated for the sample treated with $SiCl_4$ with data taken at 293 K (Si–Y) and 593 K (Si–Y–HT). The results are given in Table 2,* and it can be seen that sites Al_s and O_s are also occupied in this material, although to a lesser degree. At this stage, an attempt was made to locate the D positions in the NH_3/steamed sample (D_2O–Y) from a difference map. The only noticeable feature was a small peak at (0, 0, 0·1) of comparable height to the residue about the framework oxygen and *T* sites. Refinement of its occupation factor (assuming it to be D^+) led to only a small decrease in R_{wp} (from 10·77 to 10·67 with $R_e = 6·57$). Its occupancy corresponds to 10·6 atoms of D per unit cell (compared to approximately 17 atoms of Al in the framework). The nearest D–framework-oxygen distances are D–O(2) (1·1 Å) and D–O(3) (2·1 Å) while D–O_s is 2·9 Å. Because of the uncertainty of the existence of scattering matter at this site, and its assignment, it has not been included in the tables. Refinement of the occupancy of this site does not yield sensible results in the case of the $SiCl_4$ treated and washed samples. No density could be found in any of the difference maps in the vicinity of the positions specified by Baur (1964) for H_2O or by Bosácek, Beran & Jirák (1981) for H^+.

Discussion. The three refinements presented here clearly indicate the presence of some tetrahedral aluminosilicate species in the center of the sodalite unit of zeolite Y. Unfortunately, the obvious questions concerning the nature of this species and its origin cannot be fully answered on the basis of the currently available structural analyses of this and A-type zeolites (Pluth & Smith, 1982, and references therein). Since it is present in both NH_3/steamed and $SiCl_4$-treated samples, it is unlikely to be the $Na^+AlCl_4^-$ species recently reported by Klinowski, Thomas, Fyfe, Gobbi & Hartman (1983) on the basis of ^{27}Al MASNMR measurement. The scattering from the site designated O_s is not in agreement with its being Cl rather than O. It may be entrained at the time of synthesis or may result from the dealumination process, and is resistant to removal by washing because of the slow desorption expected from a sodalite unit. Pluth & Smith (1982) have suggested the second explanation is the more plausible in the case of zeolite A.

In previous structure analyses of samples which have been ion-exchanged with single valence cations, no extra-framework species have been detected. On the other hand, divalent cation-exchanged samples do show such a species. Whether the species exists in both types of sample (but is forced to order by the presence of a divalent cation) or results from dealumination at room temperature by ion exchange is a question best addressed by the appropriate spectroscopic (especially MASNMR) techniques.

Table 3. *Comparison of bond lengths* (Å) *and angles* (°) *for dealuminated Linde Y-zeolite*

E.s.d.'s are approximately ±0·01 Å for distances and ±1° for angles.*

	D_2O–Y	Si–Y	Si–Y–HT
T–O(1)	1·61	1·63	1·63
–O(2)	1·60	1·60	1·60
–O(3)	1·63	1·61	1·62
–O(4)	1·60	1·62	1·61
⟨*T*–O⟩	1·61	1·62	1·62
T–*T*(1)	3·03	3·03	3·03
–*T*(2)	3·06	3·04	3·04
–*T*(3)	3·08	3·09	3·10
–*T*(4)	3·08	3·10	3·10
⟨*T*–*T*⟩	3·06	3·07	3·07
O(1)–O(2)	2·64	2·65	2·66
–O(3)	2·62	2·64	2·63
–O(4)	2·63	2·61	2·61
O(2)–O(3)	2·62	2·63	2·62
–O(4)	2·61	2·62	2·63
O(3)–O(4)	2·67	2·69	2·70
⟨O–O⟩	2·63	2·64	2·64
4×(Al_s–O_s)	1·68	1·69†	1·69†
O(1)–*T*–O(2)	110	110	111
–O(3)	108	109	108
–O(4)	110	107	107
O(2)–*T*–O(3)	108	109	108
–O(4)	109	109	110
O(3)–*T*–O(4)	111	113	113
T–O(1)–*T*	140	136	136
–O(2)–*T*	148	151	151
–O(3)–*T*	143	147	147
–O(4)–*T*	145	140	140
4×(O_s–Al_s–O_s)	109	109·5†	109·5†

* The e.s.d. for Al_s and O_s is larger (±0·05 Å for distances) due to thermal disorder and lower occupancy.

† Constrained.

No diffraction evidence was found for an H or D species close to the entrained AlO_4 moiety, implying disorder of the O–H groups associated with it. No other types of aluminate species were detected, and it is therefore presumed that the octahedral species noted in ^{27}Al MASNMR studies of dealuminated Y (Klinowski, Thomas, Fyfe & Gobbi, 1982) are disordered over the channel system of zeolite Y.

Selected bond lengths and angles, shown in Table 3, are consistent with those expected for the framework zeolites with high Si/Al ratios.

The structural differences between D_2O–Y and Si–Y are manifested in the *T*–O–*T* angle. As expected for aluminosilicate frameworks, the TO_4 tetrahedra remain relatively rigid while they flex about the bridging oxygen to accommodate changes in cell dimension. The positional parameters for silicon cations of both samples are essentially invariant while the oxygen atoms show considerable shifts (Table 3).

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* See deposition footnote.

At present, the evidence for D^+ order is inconclusive; and a further neutron powder study combined with MASNMR of a carefully deammoniated sample of Linde Y-zeolite is planned in an attempt to resolve this point.

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Structure of Zr_2Be_{17}

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Abstract. $M_r = 335{\cdot}64$, hexagonal, $R\bar{3}m$, $a = 7{\cdot}538\ (1)$, $c = 11{\cdot}015\ (1)$ Å, $V = 541{\cdot}9\ (5)$ Å^3, $Z = 3$, $D_x = 3{\cdot}09$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 27{\cdot}1$ cm^{-1}, $F(000) = 444$, $T = 295$ K, $R = 0{\cdot}020$ for 951 unique reflections, e.s.d. for an observation of unit weight = 1·054. The compound is isostructural with the analogous niobium compound [Zalkin, Sands & Krikorian (1959). *Acta Cryst.* **12**, 713–715] with all the atoms of the structure located on special positions. The Zr atoms are positioned in a barrel-shaped cavity defined by 18 Be atoms with a 19th Be atom and another Zr atom capping both ends of the cavity.

Introduction. Beryllium forms intermetallic compounds with most of the transition metals in a rich variety of combining ratios. Although the stoichiometric atomic ratios of the binary compounds are relatively simple, their crystal structures generally are more complex as indicated by high numbers of formula units per unit cell. The correspondingly large unit cells lead to relatively dense sampling of reciprocal space in a diffraction experiment. The resulting dense set of structure factors is well suited to carry explicitly the subtle differences in valence electron density which distinguish different transition metals in the same structure type, or perhaps even the same transition metal in a variety of environments.

The current study is part of a systematic program of accurate structure determination of Be intermetallic compounds. This series of studies is designed to identify and evaluate compounds which could be used in detailed electron density analyses. In addition to the considerations already mentioned, other favorable properties of Be intermetallic compounds are their very small thermal parameters and low X-ray absorption coefficients when the Be:metal ratio is high. A tendency toward twinning is dealt with in these studies by careful photographic examination of each potential specimen crystal to ensure its suitability for data collection.

The title compound was originally studied as a powder by Zalkin, Bedford & Sands (1959). The present work is the single-crystal structure determination of Zr_2Be_{17}.

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