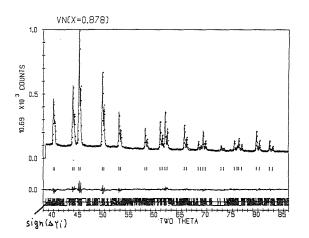12.4-1   RIETVELD REFINEMENT OF TWO SUB -
STOICHIOMETRIC VANADIUM NITRIDES. By L.Smrčok,
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Physical properties of VN_x depend on the V/N
ratio. The study of two such compounds (x=.89
and .73 determined by chemical analysis) was
carried out with X-ray diffraction data col -
lected using β-filtered MoKα radiation on a
Philips powder diffractometer at 10s/step,
$\Delta2\theta$=.05°. Calculations were performed with a
modified version of DBW2.9 program(Wiles&Young,
1981) employing the ML profile function. Re-
fined parameters included scale factor, iso -
tropic harmonic temperature factors B[$Å^2$],
population parameter of the non-metal site (n)
three FWHM parameters, zero-point, specimen
displacement and the lattice parameter. V and
N atoms were placed in the 4a and 4b Wyckoff
positions of the space group Fm3m, respective-
ly. Background was interpolated between 12
mean intensities, each calculated from the
three neighbouring observations, peak range :
+-12FWHM. The results for x=.89 are : a=
4.13470(2)Å, B(V)=.541(7), B(N)=.208(39), n=
.878(16), Rwp=.097, Rb=.014, S=1.23, d(Durbin-
Watson)=1.57, B's and n refined simultaneously
in the final cycles. For x=.73 : a=4.06951(6)
Å, B(V)=.345(9), B(N)=476(30), n=760(9), Rwp=
.136, Rb=.016, S=1.55, d=.73, but due to large
correlations and poorer fit the values of B's
and n were not obtained from the same cycle.
The TDS correction was not included in either
case for its maximum contribution is < 1.5%.
It should be emphasized that only the total B
parameters were determined in this work with-
out any attempt to separate them into a static
and a dynamic part. The results obtained can
be explained by assuming a systematic static
displacement of V-atoms adjacent to a N-va -
cancy along [100] away from the vacancy. These
static displacements lead to a shortening of
the V-N bonds in $VN_{.89}$ and subsequent reduc -
tion of amplitudes of the termal vibrations
of N-atoms in this compound. On the other hand
the high concentration of vacancies in $VN_{.73}$
leads to the compensation of static displa -
cements of V-atoms and to relaxation of N-
orbit resulting in increasing of the B(N)
value.

12.5-1   IS IT POSSIBLE TO DETERMINE A ZEOLITE
STRUCTURE AB INITIO FROM SYNCHROTRON-QUALITY POWDER
DIFFRACTION DATA? By Lynne B. McCusker, Clarendon
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The solving of new zeolite framework structures without
the benefit of single crystal diffraction data is a
problem that has plagued zeolite crystallographers for
years. A number of ingenious approaches have been
developed, but ultimately all rely upon the experience
and intuitive reasoning of the crystallographer.

Various characterization techniques can give clues about
features of the structure. For example, sorption data
can provide information about pore and cavity size,
electron diffraction unit cell and symmetry information,
29-Si MASNMR the number of chemically distinct Si sites
and their relative occupancies, and x-ray powder
diffraction the unit cell dimensions and impurity or
amorphous phase information. The usual approach is to
1) construct trial models which take into account all
the available data, 2) optimize the framework geometry
and assess its feasibility, 3) generate theoretical
powder diffraction patterns for reasonable models, and
4) compare these patterns with the observed one. It is
a long and tedious process with no guarantee of success.
Is there an alternative?

The advent of synchrotron radiation as a viable source
of high intensity x-rays has made very high resolution
x-ray powder diffraction possible. The resolution is
such that ab initio structure determination from powder
data has become a real possibility even for relatively
complex materials such as zeolites. This may be an
alternative to, or provide an additional piece of
information for, the present trial and error procedure.

Data from both the Daresbury and the Brookhaven
synchrotron facilities have been collected to examine
and evaluate this possibility. The data are now being
processed through a series of computer programs. The
critical steps are the extraction of reflection
intensities from the diffraction pattern (a modified
version of Pawley's ALLHKL program is being used), and
the selection of input parameters for the structure
determination package (G. Sheldrick's SHELXS-86 and
M. Woolfson's MULTAN-84).

Results on the test example Silicalite I, which has a
complex (but known) framework structure, and on a true
unknown will be presented.

VN(X=0.878)