Tabelle 1. *Gitterkonstanten und Achsenverhältnisse*

Formel	a_0	c_0	c/a
$CsFe(SeO_4)_2$	$5{\cdot}04_3$ Å	$9{\cdot}02_8$ Å	$1{\cdot}79_0$
$RbFe(SeO_4)_2$	$4{\cdot}99_7$	$8{\cdot}53_1$	$1{\cdot}70_7$
$TlFe(SeO_4)_2$	$5{\cdot}01_2$	$8{\cdot}48_6$	$1{\cdot}69_3$
$NH_4Fe(SeO_4)_2$	$5{\cdot}00_4$	$8{\cdot}46_5$	$1{\cdot}69_1$
$RbGa(SeO_4)_2$	$4{\cdot}90_7$	$8{\cdot}59_2$	$1{\cdot}75_0$
$TlGa(SeO_4)_2$	$4{\cdot}93_1$	$8{\cdot}55_2$	$1{\cdot}73_4$
$NH_4Ga(SeO_4)_2$	$4{\cdot}91_9$	$8{\cdot}51_8$	$1{\cdot}73_1$
$RbAl(SeO_4)_2$	$4{\cdot}89_7$	$8{\cdot}54_9$	$1{\cdot}74_6$
$TlAl(SeO_4)_2$	$4{\cdot}88_4$	$8{\cdot}49_5$	$1{\cdot}73_9$
$NH_4Al(SeO_4)_2$	$4{\cdot}89_2$	$8{\cdot}47_0$	$1{\cdot}73_1$

Die *d*-Werte werden im *X-Ray Powder Data File* veröffentlicht.

Die Volumeninkremente sind von gleicher Grössenordnung wie bei den Doppelsulfaten und lassen sich analog erklären. Die Inkremente für den Übergang SeO_4–SO_4 schwanken geringfügig um den Werte 15,4 . 10^{-24} cm^3 pro Formeleinheit; den gleichen Wert fand auch Haussühl (1961) für die Alaune.

Der Deutschen Forschungsgemeinschaft danke ich für ihre finanzielle Unterstützung.

Literatur

Franke, W. & Henning, G. (1965). *Acta Cryst.* **19**, 870.
Haussühl, (1961). *Z. Kristallogr.* **116**, 371.
Vegard, L. & Maurstad, A. (1928). *Z. Kristallogr.* **69**, 519.

Acta Cryst. (1966). **20**, 921

Crystal data for 2-amino-6-hydroxypyridine.

By Brahama D. Sharma, *Department of Chemistry, Oregon State University, Corvallis, Oregon* 97331, *U.S.A.*

(*Received* 29 *November* 1965)

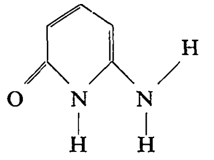
As a part of our interest in bases closely related to those found in nucleic acids we have examined crystals of 2-amino-6-hydroxypyridine, $C_5H_6N_2O$, by the methods of X-ray diffraction. Acicular crystals, elongated along **a**, were obtained by recrystallization from ethanol. Crystal data, as determined from the analysis of rotation and Weissenberg (zero and upper level) photographs about *a* and *b* axes, are:

Triclinic

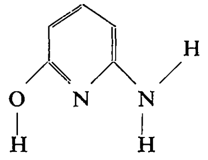
$a = 4{\cdot}85 \pm 2$ Å $\quad \alpha = 98°$
$b = 9{\cdot}30 \pm 3 \quad \beta = 91$
$c = 13{\cdot}57 \pm 5 \quad \gamma = 92$

The density measured in methylchloroform and benzene mixture by the flotation method is 1·298 g.cm^{-3}. Calculated density for four ($C_5H_6N_2O$. $\frac{1}{2}H_2O$) formula units per unit cell is 1·306 g.cm^{-3}. Presence of water of crystallization was also confirmed by elementary analyses (Found: C, 51·23; H, 6·22; N, 22·71 %. Required: C, 50·42; H, 5·88; N, 23·53 %), and drying of the recrystallized material.

Heavily exposed (72 hours, unfiltered radiation) Weissenberg photographs show a marked fall-off of intensity beyond a *d* value of 1 Å. No further crystallographic work on this compound, which is likely to have either of the tautomeric structures (I) and (II), is contemplated.



1,6-Dihydro-2-amino-6-oxo-pyridine
(I)



2-Amino-6-hydroxy-pyridine
(II)

The author gratefully acknowledges the support, in part, by the donors of the Petroleum Research Fund, administered by the American Chemical Society and in part by the Research Council of the Graduate School, Oregon State University.

Acta Cryst. (1966). **20**, 921

Note on reliability indices*.

By George M. Brown, *Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.*

(*Received* 19 *November* 1965)

Continued use by crystallographers of the discrepancy indices (or residuals) R_k given by the equation

$$R_k \equiv \frac{\Sigma \big|\, |F|_o^k - |F|_c^k \big|}{\Sigma |F|_o^k} \qquad (k = 1 \text{ or } 2) \qquad (1)$$

justifies some remarks on R_k values from experience in this laboratory in neutron-diffraction analysis. We have found that for a set of $|F|_o^2$ and $|F|_c^2$ values at convergence in least-squares refinement the index R_1 may be as large as R_2, or even somewhat larger (Table 1). This finding has caused reactions among various crystallographic colleagues varying from mild surprise to incredulity, apparently because the usual result in X-ray analyses based on data recorded photographically is that R_1 is about one-half of R_2.

It is helpful in considering the relation between R_1 and R_2 to define the fractional discrepancy f_k of an individual observation by the equation

---

* Research sponsored by the U.S. Atomic Energy Commission under contract with the Union Carbide Corporation.

Table 1. *Comparative values of residuals R_1 and R_2 in least-squares refinements of structures from neutron-diffraction data*

	R_1	R_2	σ	Remarks
α-D-Glucose (Brown & Levy, 1965)	0·060	0·059	1·097	37 of 1656 data omitted
Potassium hydrogen chloromaleate (Ellison & Levy, 1965)	0·123	0·092	1·015	4 of 1959 data omitted
K_2NbF_7 (Brown & Walker, 1966)	0·090	0·081	1·20	All 1355 data, no extinction corrections
	0·109	0·103	1·13	112 data omitted because of extinction
	0·087	0·070	1·11	All 1355 data, corrected for extinction

$$f_k \equiv \left|\,|F|_o^k - |F|_c^k\,\right| / |F|_o^k \tag{2}$$

and to rewrite R_k in the form

$$R_k = \frac{\Sigma f_k |F|_o^k}{\Sigma\, |F|_o^k}\,. \tag{3}$$

This expression emphasizes that R_k is the weighted average value of the absolute fractional discrepancy, each individual value being weighted by the corresponding $|F|_o^k$. Making use of the approximation $f_1 \simeq f_2/2$, valid for f_1 and f_2 values in the range of interest, one can write the specific forms for R_1 and R_2 as follows:

$$R_1 = \tfrac{1}{2}\, \Sigma f_2 |F|_o / \Sigma\, |F|_o \tag{4}$$

$$R_2 = \Sigma f_2 |F|_o^2 / \Sigma\, |F|_o^2\,. \tag{5}$$

It is well known that for X-ray film data the fractional observational errors are usually approximately constant over a wide range of $|F|_o$ values above a minimum value. From (4) and (5) it follows that the relation $R_1 \simeq R_2/2$ should be expected to hold, in agreement with the usual finding.

In the typical set of neutron-diffraction data, obtained by counter techniques, f_2 is by no means constant. Rather, it varies over a very wide range, usually from about 0·03 for the strongest reflections to the order of unity for the weakest observable reflections. In comparison with (4), the expression (5) weights the smaller fractional discrepancies associated with the larger $|F|_o$ values much more heavily than the larger discrepancies associated with the smaller $|F|_o$ values. Given the usual distribution of $|F|_o$ values in a set of data, it is understandable, therefore, that R_1 may equal or exceed R_2.

For X-ray data recorded by counter techniques higher precision can easily be achieved in measuring the weaker reflections, because of the better resolution against background allowed by the higher intensity of radiation in X-ray beams. Therefore the range of values of f_2 is usually not so large as it is for neutron data. It follows that one should expect for such data values of R_1/R_2 intermediate between those characteristic of X-ray film data and neutron data. The precise value in a given case will depend on the distribution of the fractional errors in the data, which will be determined by the details of the recording scheme and by the effects of any systematic errors that may be present.

By systematic analysis of the discrepancies, one may detect in the last stages of a structure refinement with neutron data the presence of extinction errors which are small in magnitude but which may affect fifty or a hundred data of the strongest reflections. The R_k values computed when these data are included in the refinements are always lower than those computed when the data are omitted, though the reverse is true for the corresponding values of the more significant quantity σ, the standard deviation of an observation of unit weight.* At first sight this finding may seem anomalous, but it is readily understood by reference to the expression (3) for R_x. When the data in error are included, their fractional discrepancies are reduced by a compensating adjustment of the scale factor on the observations, so that the discrepancies become small relative to those of many of the weak reflections. Then the large weights given these data by equation (3) ensure a value of R_k that is misleadingly low. In fact, the resulting R_k value may even be quite close to the value calculated after refinement on data corrected for extinction.

References

Brown, G. M. & Levy, H. A. (1965). *Science*, **147**, 1038.

Brown, G. M. & Walker, L. A. (1966). *Acta Cryst.* **20**, 220.

Ellison, R. D. & Levy, H. A. (1965). *Acta Cryst.* **19**, 260.

Hamilton, W. C. (1964). *Statistics in Physical Science*, p. 127 *et seq.* New York: Ronald Press.

---

* This quantity is defined by the equation

$$\sigma = \left[\frac{\Sigma\, w(|F|_o{}^2 - |F|_c{}^2)^2}{n-p}\right]^{\frac{1}{2}}$$

where the weight w is the reciprocal of the variance of an observation $|F|_o{}^2$, n is the number of observations, and p is the number of parameters fitted to the data set. See, *e.g.* Hamilton (1964).

Acta Cryst. (1966). **20**, 922

Crystallite size and lattice distortion parameters from X-ray line broadening.

By D. R. Buchanan, R. L. McCullough and R. L. Miller, *Chemstrand Research Center, Inc., Durham, North Carolina, U.S.A.*

(*Received* 14 *October* 1965 *and in revised form* 8 *December* 1965)

In a study of line broadening from gray irons and steels, Hauk & Hummel (1956) obtained vastly differing results for integral breadth analyses and the Fourier transform analysis of Warren & Averbach (1950). These discrepancies led others (Willets, 1965) to suggest alternate methods of line profile analysis in order to separate the effects of small