Hill, R. J. & Howard, C. J. (1986). Report AAEC/M112. Australian Atomic Energy Commission, Lucas Heights Research Laboratories, Australia.

Howard, C. J. (1982). *J. Appl. Cryst.* **15**, 615–620.

Howard, C. J., Ball, C. J., Davis, R. L. & Elcombe, M. M. (1983). *Aust. J. Phys.* **336**, 507–518.

Jones, H. (1960). *The Theory of Brillouin Zones and Electronic States in Crystals*, 1st ed. Amsterdam: North-Holland.

Kisi, E. H. (1988). Thesis, Univ. of Newcastle, NSW, Australia.

Lindahl, T. & Westman, S. (1969). *Acta Chem. Scand.* **23**, 1181–1190.

Ljung, H. & Westman, S. (1970). *Acta Chem. Scand.* **24**, 611–617.

Morton, A. J. (1974). *Phys. Status Solidi A*, **23**, 275–289.

Morton, A. J. (1975). *Phys. Status Solidi A*, **31**, 661–674.

Morton, A. J. (1976). *Phys. Status Solidi A*, **33**, 395–403.

Morton, A. J. (1977). *Phys. Status Solidi A*, **44**, 205–214.

Morton, A. J. (1979). *Acta Metall.* **27**, 863–867.

Morton, A. J. (1985). Inst. Phys. Conf. Ser. No. 78, ch. 9, p. 343. Bristol: Institute of Physics.

Morton, A. J. (1987). Private communication.

Rietveld, H. M. (1969). *J. Appl. Cryst.* **2**, 65–71.

Sande, M. van, van Landuyt, J. & Amelynckx, S. (1979). *Phys. Status Solidi A*, **55**, 41.

Sande, M. van, van Landuyt, J., Avalos-Borja, M., Torres-Villesenor, G. & Amelynckx, S. (1980). *Mater. Sci. Eng.* **46**, 167–173.

Sarah, N., Rajasekharan, T. & Schubert, K. (1981). *Z. Metall.* **72**(10), 732–735.

Stokhuyzen, R., Brandon, J. K., Chieh, C. & Pearson, W. B. (1974). *Acta Cryst.* **B30**, 2910–2911.

Westman, S. (1965*a*). *Acta Chem. Scand.* **19**, 1411–1419.

Westman, S. (1965*b*). *Acta Chem. Scand.* **19**, 2369–2372.

Westman, S. (1972). *Chem. Commun. Univ. Stockholm*, **4**, 1–37.

Wiles, D. B. & Young, R. A. (1981). *J. Appl. Cryst.* **14**, 149–151.

Willey, L. A. (1973). In *Metals Handbook* 8th ed., Vol. 8. Cleveland: American Society for Metals.

Acta Cryst. (1991). B**47**, 843–848

Accurate Structure Analysis with Synchrotron Radiation. An Application to Borazone, Cubic BN

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Abstract

An accurate room-temperature structure analysis has been carried out on borazone, cubic BN, using synchrotron radiation. X-ray diffraction data were collected on the five-circle diffractometer at HASYLAB during dedicated runs (3·7 GeV) of DORIS II at a wavelength of $\lambda = 0{\cdot}5000\,(1)$ Å up to $\sin\theta/\lambda = 1{\cdot}45$ Å^{-1}. Comparison with accurate tube data measured with Ag $K\alpha$ radiation [Will, Kirfel & Josten (1986). *J. Less. Common Met.* **117**, 61–71] resulted in a general agreement factor of 0·0135 (0·006 omitting the four lowest-order reflections). Refinements with multipole expansions up to the hexadecapole level yielded R values as low as 0·0046. The final R value of a joint data set, prepared by weighted averaging of tube and synchrotron data, was 0·0077. Ensuing deformation density maps agree well with expectation. The analysis and the refinements of the synchrotron radiation data, as well as the comparison with the X-ray tube data, support our previous findings on cuprite, Cu_2O, and corundum, α-Al_2O_3 [Kirfel & Eichhorn (1990). *Acta Cryst.* A**46**, 271–284], indicating that high-quality X-ray diffraction data can be obtained at a synchrotron radiation source within normal beam-time allocation.

1. Introduction

In a recent feasibility study, accurate room-temperature structure analyses have been carried out on cuprite, Cu_2O, and corundum, α-Al_2O_3 (Kirfel & Eichhorn, 1990; hereafter referred to as KE), using synchrotron radiation. This work served to assess the accuracy of single-crystal X-ray diffraction data that can be obtained with such a source. Experimental and data-processing problems were discussed in

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Table 1. *Crystallographic data of borazone, cubic* BN

a_0* (Å)	3·6159 (1)
V_c (Å^3)	47·277 (1)
Space group	$F\bar{4}3m$ (No. 216)
	$Z = 4$
Atoms	B 4(a) (0, 0, 0); $\bar{4}3m$
	N 4(c) ($\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{4}$); $\bar{4}3m$
Formula weight	$M_r = 24{\cdot}817$
Density (Mg m^{-3})	$D_x = 3{\cdot}48$
μ(Mo $K\alpha$) (mm^{-1})	0·240
μ(Ag $K\alpha$) (mm^{-1})	0·149

* Refined from 25 reflections ($21{\cdot}7 \leq 2\theta \leq 72{\cdot}3°$, $\lambda = 1{\cdot}54056$ Å).

detail. The choice of the compounds was determined by their structural simplicity, a center of symmetry, and, in particular, by the availability of careful X-ray tube measurements which could be employed for rigorous cross-checks.

The results of KE indicated that in spite of the relatively fast data collections complete sets of structure amplitudes can be derived with a quality which compares well with the reference data, provided proper attention is given to the performance of the experiments and to the data reduction.

The following contribution is intended to add further experimental evidence to this finding, this time from a single-crystal diffractometry study of cubic BN which was carried out in the course of an investigation of anomalous-dispersion effects of nitrogen (Grochowski, Serda, Kupcik & Eichhorn, 1989, 1990). As for cuprite and corundum, reflection data of BN can be compared with careful tube measurements (Josten, 1986), employed in an electron density distribution study by Will, Kirfel & Josten (1986; referred to as WKJ hereafter). Essentially, these tube data are Ag $K\alpha$ data, supplemented by data from additional measurements on the same crystal and on a second sample, using Mo $K\alpha$ radiation.

Because of the similarity of the experimental and computational procedures of the present work with that of KE, there is no need to report all steps in detail. Thus, emphasis is put on the assessment of data quality and on the data analysis. The latter includes least-squares refinements on the multipole expansion level, without, however, intending a thorough discussion of the electron density distribution in BN.

2. Structure and sample

The crystallographic data of cubic BN are summarized in Table 1. The structure of the zinc-blende polymorph has similarities with the isoelectronic diamond stucture (space group $Fd\bar{3}m$). It is, however, non-centrosymmetric ($F\bar{4}3m$). The close relationship between cubic BN and diamond, and the similarity of their physical properties has prompted great interest in the chemical bonding. Although the bonding is usually considered as essentially covalent, WKJ determined a charge transfer of 0·46 electrons from B towards N in keeping with the respective electronegativities. This experimental finding is in good agreement with theoretical calculations: 0·38–0·50 electrons (Coulson, Redel & Stocker, 1962), 0·46 electrons (Levin, Syrkin & Dyatkina, 1966), and 0·47 electrons (Spackman, 1984). Thus, taking diamond and LiF as representative of pure covalent and pure ionic bonding, respectively, bonding in BN can be assigned an ionic contribution of about 20%.

The lack of an inversion center in cubic BN causes problems with respect to a reliable electron density mapping, because the structure-factor phases are model-dependent. Apart from this intrinsic problem, cubic BN is well suited as a standard material for the purpose of this study. The high symmetry permits the collection of sufficient numbers of equivalent reflections for the determination of internal consistency as well as for the detection of multiple scattering. Furthermore, the substance is chemically stable, suffers no radiation damage, and the extreme hardness ($H = 10$) and the high Debye temperature of 1900 K (at 0 K) imply little thermal diffuse scattering (TDS). In fact, calculations based on the mean sound velocity of diamond ($v = 1{\cdot}75 \times 10^4$ m s^{-1}) indicate TDS correction factors $(1 + \alpha) \leq 1{\cdot}01$ up to $\sin\theta/\lambda = 1{\cdot}7$ Å^{-1} (Josten, 1986). Finally, the light elements preclude problems by absorption of radiation for $\lambda \simeq \lambda$(Ag $K\alpha$), and anomalous-dispersion effects are negligible within the present context.

Isometric dark-brown single crystals (General Electric, 100–125 mesh grade) with natural faces and dimensions of 0·08–0·25 mm were carefully examined by standard tube experiments (Cu $K\alpha$, Mo $K\alpha$). Particular attention was paid to the mosaic structure. The sample finally selected for the synchrotron radiation experiment had a maximum diameter of 0·24 mm and a mosaic spread of 0·28° (half-width of an ω-scan of $\bar{1}11$, using 0·5 Å synchrotron radiation). The choice of a smaller sample was precluded by twinning of smaller crystals (Kupcik, Grochowski & Serda, 1990).

3. Experimental

Data collection was carried out on the five-circle diffractometer at HASYLAB beamline D3 (Kupcik, Wendschuh-Josties, Wolf & Wulf, 1986; Wendschuh-Josties & Wulf, 1989) during dedicated shifts of the DORIS II storage ring. Average fills lasted up to a maximum of 3 h, with ring currents between 100 and 40 mA. Beam steering was with respect to beam port

Table 2. *Data collection and data processing*

Instrument	Five-circle diffractometer, HASYLAB
Monochromator	Flat double-crystal, Ge(111)
Collimator	1 mm diameter
Detector aperture (mm)	3 × 3
Detector–sample distance (mm)	248
Sample size (mm)	0·21 × 0·22 × 0·24
Temperature	Ambient (295 K)
Wavelength (Å)	$\lambda = 0{\cdot}5000\ (1)$
Harmonic suppression	$I_0(\lambda/3)/I_0(\lambda) < 10^{-3}$
Counter deadtime (μs)	3·8
Scan mode	ω-step scans
No. of steps	61
Step size (°)	0·04
Scan range (° in ω)	2·40
Scan speed (° s^{-1})	1·2–3·6 (0·5–1·5 s $step^{-1}$)
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	1·45
Test reflection	222
Interval	15 reflections
No. of reflections	420 (including standards)
Unique ($\bar{4}3m$)	52 reflections
Unobserved ($\lvert F_o\rvert < \sigma$)	None
$R_{int}(I)$	0·0075
$wR_{int}(I)$	0·0181

D where the diffractometer is located. The wavelength was set to 0·5000 (1) Å, and the beam was collimated to 1 mm diameter. For strong reflections ($I > 20\,000$ Hz) the intensity was reduced by inserting appropriate filter combinations. Initially, careful checks were made on both diffractometer and sample alignment. After optical centering and refinement of the sample's orientation matrix additional checks were made for crystal stability and beam homogeneity, beam contamination by higher harmonics, and optimum scan range and detector aperture. In addition, counter dead times and filter factors were determined, following the procedure of Chipman (1969). Details of data collection and data reduction are summarized in Table 2.

The complete data set (420 reflections, including the standards, one quarter of reciprocal space) was collected within 12 h beam time (comprising five fills). During the measurements the degree of linear horizontal polarization remained almost constant. It changed from 93·5 to 91·5% during a fill, thus posing no problems with the polarization correction.

No significant scatter was observed for the (222) standard reflection intensities. In particular, no influence of the ring current could be detected. Actually, the (222) is a weak reflection not normally chosen as a standard. Since, however, this measurement served to establish a reference data set for the determination of small anomalous-dispersion effects of nitrogen at larger wavelengths (> 1·5 Å), it was interesting to know how well the intensity of a weaker reflection – which is quite 'observable' with synchrotron radiation – could be reproduced throughout a full data collection. The internal agreement of the standard, $R(I) = 0{\cdot}012$, was even better than expected from the mean relative e.s.d. $\sigma(I)/I = 0{\cdot}020$. These findings indicated stability of the experimental conditions as well as correct beam monitoring.

4. Data analysis

4.1. *Data reduction*

The data-reduction procedure was essentially the same as described by KE. The data were neither corrected for absorption nor for TDS since both effects were considered to be negligible. This applies also to the contribution of anomalous dispersion (f', $f'' \leq 0{\cdot}002$ at $\lambda = 0{\cdot}5$ Å), so that the averaging of symmetry-equivalent and multiple reflections also included Friedel pairs. From 391 reflections two were rejected during averaging, their discrepancies probably being caused by multiple scattering. The final synchrotron data set (SYN) comprised 52 unique reflections (none unobserved) with an internal agreement of $R(I) = 0{\cdot}0075$, $wR(I) = 0{\cdot}018$ ($w = 1/\sigma^2$), implying good data quality.

4.2. *Comparison with tube data*

In order to estimate the agreement between the SYN data [52 reflections, $(\sin\theta/\lambda)_{max} = 1{\cdot}45$ Å^{-1}] and the WKJ tube data [76 reflections, $(\sin\theta/\lambda)_{max} = 1{\cdot}71$ Å^{-1}] a procedure similar to that described by KE was used. The SYN data were adjusted to the corresponding subset of WKJ by minimizing

$$[K|F_o(\mathrm{SYN})|\exp(-\Delta Bs^2) - |F_o(WKJ)|]^2$$

where K is a scale factor, $s = \sin\theta/\lambda$ and ΔB accounts for a difference in the mean isotropic temperature factor.

Case SYNA. Using all SYN data yielded an overall agreement between data SYN and WKJ of $R(F) = 0{\cdot}0135$, and $\Delta B = 0{\cdot}051\ (9)$ Å^2. The largest differences occured for the four lowest-order reflections (111, 200, 220, 311); in each case $|F(\mathrm{SYN})| < |F(\mathrm{WKJ})|$. The reason for this systematic discrepancy is not readily evident; a possible explanation will be discussed below.

Case SYNB. Repeating the same procedure, omitting the four lowest-order SYN reflections for the determination of K and ΔB, yielded $R(F) = 0{\cdot}006$ and $\Delta B = 0{\cdot}033\ (3)$ Å^2.

These results can be seen in Fig. 1, which shows for the two cases, SYNA and SYNB, the distributions of the internal R values as a function of $|F|$.

Recalling that different samples, different instruments, and different radiation sources are involved in this comparison, case SYNA can be considered as satisfactory, and case SYNB as excellent for the 48 reflections with $h^2 + k^2 + l^2 > 11$. Thus, as already found by KE, synchrotron data can compete very well with tube data.

A joint data set, BNJOI, was prepared from the weighted average of synchrotron and tube data, SYNA and WKJ. The e.s.d.'s, $\sigma(F)$, of these joint data were either derived from the scatter or assigned a minimum value of $0{\cdot}01|F|$. The largest relative difference between synchrotron and tube data, $\Delta|F| = -0{\cdot}376$, was found for reflection (200), which was subsequently assigned $\sigma(F) = \Delta|F|$. Finally, set SYNB was modified by substituting the four lowest-order reflections by the tube measurements, yielding set SYNB4.

Ensuing structure refinements will be referred to these data sets: SYN, SYNA, SYNB SYNB4 and BNJOI, respectively.*

4.3. *Spherical-atom refinements*

The positional and thermal parameters reported by WKF were taken as starting values for both a conventional spherical-atom refinement (SYN) and a high-order refinement (HO-SYN), the latter using a cutoff $\sin\theta/\lambda = 0{\cdot}8$ Å^{-1}. Refinements were based on $|F|$ with weights $w = [\sigma^2(F) + (0{\cdot}01F)^2]^{-1}$. Scattering factors for the neutral atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). An isotropic extinction correction (Becker & Coppens, 1974*a*,*b*) using a type 1 Lorentzian distribution, turned out to be insignificant, in agreement with the relatively large mosaic spread, and was therefore omitted from further calculations. Anomalous-dispersion corrections for B and N – which are very small for $\lambda = 0{\cdot}5$ Å – were taken from Cromer & Liberman (1970).

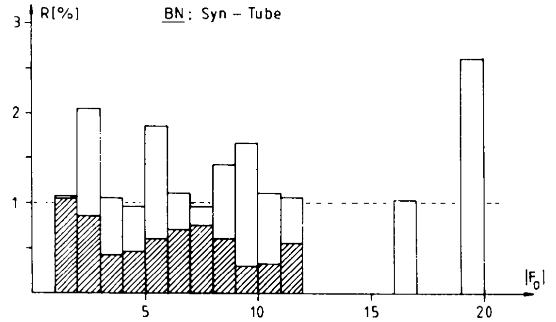

Fig. 1. Plot of the internal R value $R(F)$ between the synchrotron data SYN and the tube data WKJ as a function of $|F|$, in steps of $\Delta F = 1$. (*a*) SYNA data, all reflections, white histogram. (*b*) SYNB data, omitting 111, 200, 220, 311, shaded histogram.

Table 3. *Refinement results* (*see text for details*)

CT is the formal charge transfer in electrons from B to N. HO-SYN $0{\cdot}80 \le \sin\theta/\lambda \le 1{\cdot}45$ Å^{-1}, BNJOI $0{\cdot}24 \le \sin\theta/\lambda \le 1{\cdot}71$ Å^{-1}, other refinements $0{\cdot}24 \le \sin\theta/\lambda \le 1{\cdot}45$ Å^{-1}. Function minimized was $Q = \sum_h w_h[|F_o(h)| - k|F_c(h)|]^2$. Weighting scheme $w_h = 1/\max[\sigma^2(F), (0{\cdot}01F)^2]$ for MBNJOI and $w_h = 1/[\sigma^2(F) + (0{\cdot}01F)^2]$ otherwise. Dispersion correction $f'(\mathrm{B}) = 0$, $f''(\mathrm{B}) = 0$, $f'(\mathrm{N}) = 0{\cdot}001$, $f''(\mathrm{N}) = 0{\cdot}002$.

	IAM model		Multipole model				
	SYN	HO-SYN	MSYN	MSYNA	MSYNB	MSYNB4	MBNJOI
B(B)	0·204 (3)	0·207 (3)	0·192 (1)	0·243 (1)	0·230 (1)	0·235 (1)	0·223 (1)
B(N)	0·166 (3)	0·170 (3)	0·163 (1)	0·207 (1)	0·194 (1)	0·203 (1)	0·194 (1)
CT	–	-	0·14 (2)	0·14 (3)	0·13 (3)	0·36 (5)	0·34 (5)
R	0·0112	0·0052	0·0046	0·0051	0·0053	0·0049	0·0077
wR	0·0124	0·0078	0·0060	0·0080	0·0075	0·0060	0·0081
GOF	1·07	0·83	0·58	0·77	0·72	0·58	0·50
N_o	52	40	52	52	52	52	76
N_v	3	3	14	14	14	14	14

4.4. *Multipole model and deformation properties*

Multipole refinements (indicated by adding an 'M' to the above data set's name) were carried out using the program *VALRAY* (Stewart, 1976; Stewart & Spackman, 1981). The formalism was the same as described by KE, and need not be repeated here. Systematic studies of the influence of the model and weighting scheme, as well as of the intrinsic problem of structure-factor phasing due to the non-centrosymmetry of borazone, were not attempted, being beyond the goals of this work. Thus, the electron density results may be biased by the model, refinement strategy and weighting scheme. Since, however, the same model was used in all refinements, differing results may be safely attributed to the respective data (and to the sample) rather than to the model. With the atomic parameters from the multipole model, the static deformation density in the section containing the B—N zigzag bonds was calculated by direct-space integration according to Stewart (1976). Again, details have already been given by KE. Dynamic deformation density maps $\Delta\rho(F_o - F_c^{\mathrm{IAM}})$ were calculated, using the F_c^{mult} multipole model phases for F_o. Here, F_c^{IAM} is the structure factor for spherical neutral atoms calculated with the thermal displacement parameters taken from the multipole refinement.

5. Results and conclusions

Results of the various IAM and multipole model refinements are summarized in Table 3. The quality of the SYN data as well as the influence of the chemical bonding can be appreciated from the results of refinements SYN, HO-SYN and MSYN. Clearly, the 12 low-order reflections with $\sin\theta/\lambda \le 0{\cdot}8$ Å^{-1} carry the bulk information about the charge redistribution, which is successfully modelled in MSYN.

Discrepancies between corresponding parameters of these refinements, using the original SYN data,

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* Lists of structure factors for both synchrotron and tube data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54346 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

can be attributed to extremely high correlations between the scale factor and the temperature factors.

Compared with the values quoted in the beginning (0·38–0·50 electrons) the original synchrotron data SYN, and also both adjusted data sets, SYNA and SYNB, yield too small a charge transfer from B towards N. Since the SYNB4 data, as well as the joint data, BNJOI, produce a value in better agreement, it must be concluded that primarily the four lowest-order reflections are required for adjustment of the monopolar functions of the atoms. These reflections appear to be systematically too low in the synchrotron data SYN. In view of the excellent agreement of the remaining reflections with the tube data, errors due to questionable filter factors or problems in the data-reduction procedure seem very unlikely. Thus, we suspect that the investigated crystal was not completely stoichiometric. In fact, a lack of N or a small amount of N on the B site changes the relative distribution of the lowest-order calculated structure factors towards that of the observations. No significant results could, however, be obtained from refinements with variable atomic occupancy factors (*i.e.* variable core populations).

Figs. 2 and 3 give further illustrations of the main refinement results. The differently modelled charge transfers of MSYN and MBNJOI are clearly evident in the respective dynamic and static deformation density distributions. Differences of $\Delta\rho$ occur in the rear of the atoms, *i.e.* charge accumulations are observed at B (MSYN) in contrast to N (MBNJOI). On the other hand, the density accumulations on the B—N bond are very similar, and the polarization

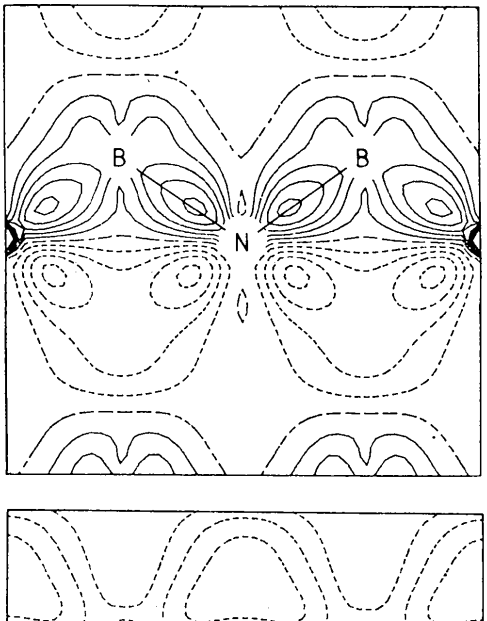

Fig. 2. Static model deformation density $\Delta\rho_{\rm stat}$ for BN from refinements MSYN (top) and MBNJOI (bottom), including the monopoles, in the plane (110). Horizontal axis [110], vertical axis [001]. B—N bonds are indicated. Contours at 0·05 e Å^{-3}, negative contours broken, positive full.

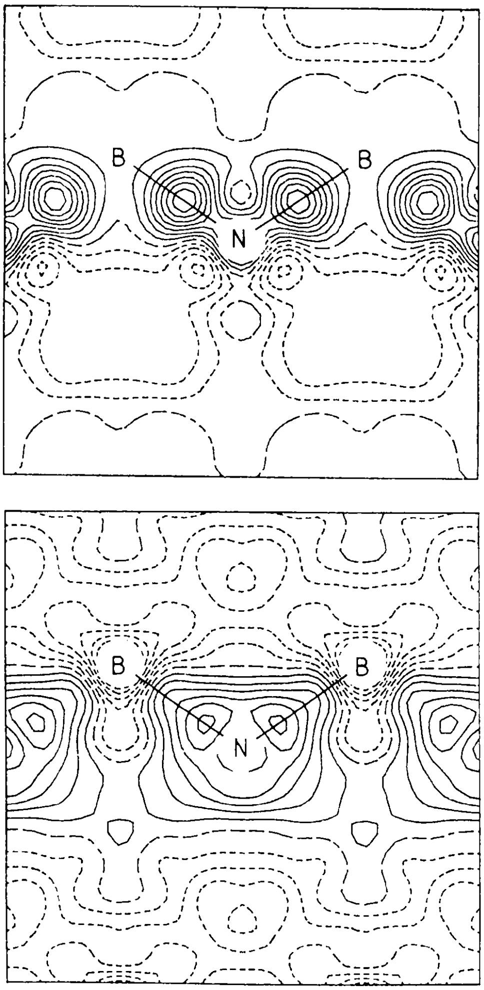

Fig. 3. Dynamic difference density $\Delta\rho(F_o - F_c^{\rm IAM})$ based on the parameters from refinements MSYN (top) and MBNJOI (bottom). $(\sin\theta/\lambda)_{\rm max} = 0·85$ Å^{-1}. Orientation and contours as in Fig. 2.

towards N is also in agreement with the electronegativities of the elements B and N. The mean error in the bond region of $\Delta\rho(F_o - F_c^{\mathrm{IAM}})$ (Fig. 3) is approximately $\sigma(\Delta\rho) \simeq (1/V)[\Sigma(\Delta F)^2]^{1/2} = 0{\cdot}003$ e Å^{-3} (Cruickshank, 1949). Considering, finally, the residual density distributions (Fig. 4), one finds for both cases almost featureless maps, particularly at the atom sites, though, with respect to the bond region, refinement MSYN is a little less satisfactory than MBNJOI.

These few comments may suffice to address the inherent difficulties which are encountered in the charge density study of a compound like borazone, even employing apparently high-quality X-ray diffraction data.

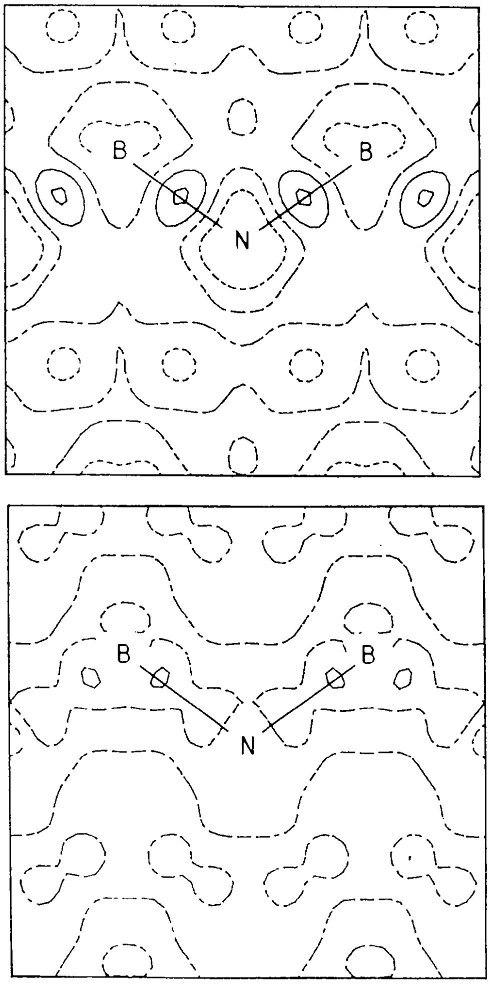

Fig. 4. Residual density for BN from refinements MSYN (top) and MBNJOI (bottom). $(\sin\theta/\lambda)_{\max} = 0{\cdot}85$ Å^{-1}. Orientation and contours as in Fig. 2.

The aim of this work was not primarily a reliable charge density study but mainly another investigation of the quality of X-ray diffraction data that can be collected at a synchrotron radiation source, using our 'standard' data collection and data reduction procedures developed in the course of our analyses of Cu_2O and Al_2O_3.

The results obtained on cubic borazone, BN, support the findings of our earlier feasibility study and indicate once more that high-quality X-ray diffraction data can be obtained at a synchrotron radiation source. Moreover, it should be recalled that the synchrotron data were collected within less than 12 h effective beam time, whereas measurement of the tube data (in which AK was involved) lasted over many weeks. This does not mean that data collections at a synchrotron radiation source are advocated in general, but it shows that complementary data can well be acquired within a rather limited beam-time allocation.

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References

BECKER, P. & COPPENS, P. (1974*a*). *Acta Cryst.* **A30**, 129–147.
BECKER, P. & COPPENS, P. (1974*b*). *Acta Cryst.* **A30**, 148–153.
CHIPMAN, D. R. (1969). *Acta Cryst.* **A25**, 209–214.
COULSON, C. A., REDEL, L. B. & STOCKER, D. (1962). *Proc. R. Soc. London Ser. A*, **270**, 357–372.
CROMER, D. T. & LIBERMAN, D. A. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CRUICKSHANK, D. W. J. (1949). *Acta Cryst.* **2**, 65–82.
GROCHOWSKI, J., SERDA, P., KUPCIK, V. & EICHHORN, K. (1989). *HASYLAB Jahresber.* pp. 381–382.
GROCHOWSKI, J., SERDA, P., KUPCIK, V. & EICHHORN, K. (1990). Proc. 32nd Pol. Crystallogr. Meet. Wroclaw, Poland.
JOSTEN, B. (1986). *Röntgenographische Untersuchung der Elektronendichteverteilung und der chemischen Bindung in kubischem Bornitrid*, BN. Dissertation, Univ. Bonn, Germany.
KIRFEL, A. & EICHHORN, K. (1990). *Acta Cryst.* **A46**, 271–284.
KUPCIK, V., GROCHOWSKI, J. & SERDA, P. (1990). *HASYLAB Jahresber.* pp. 295–296.
KUPCIK, V., WENDSCHUH-JOSTIES, M., WOLF, A. & WULF, R. (1986). *Nucl. Instrum. Methods*, **A246**, 624–626.
LEVIN, A. A., SYRKIN, Y. K. & DYATKINA, M. E. (1966). *J. Struct. Chem.* (*USSR*), **7**(4), 583–588 (in Russian).
SPACKMAN, M. A. (1984). Private communication.
STEWART, R. F. (1976). *Acta Cryst.* **A32**, 565–574.
STEWART, R. F. & SPACKMAN, M. A. (1981). *VALRAY Users Manual*. Preliminary Draft. Department of Chemistry, Carnegie-Mellon Univ., Pittsburgh, PA 15213, USA.
WENDSCHUH-JOSTIES, M. & WULF, R. (1989). *J. Appl. Cryst.* **22**, 382–383.
WILL, G., KIRFEL, A. & JOSTEN, B. (1986). *J. Less Common. Met.* **117**, 61–71.