

Pitfalls of data mining: triclinic polymorph of 2,2-aziridinedicarboxamide revisited

**Marcin Podsiadło, Kamil
Dziubek and Andrzej Katrusiak***

Faculty of Chemistry, Adam Mickiewicz
University, Grunwaldzka 6, 60-780 Poznań,
Poland

Correspondence e-mail: katran@amu.edu.pl

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Several procedures have been employed for validating structural models refined on poor quality single-crystal diffraction data. Analysis of intra- and intermolecular distances in the structures of 2,2-aziridinedicarboxamide polymorphs proved to be a robust means, and a means independent of the chosen unit cell and symmetry, of detecting several incorrect atom-type assignments in the reported structure of the triclinic polymorph of 2,2-aziridinedicarboxamide [Brückner (1982), *Acta Cryst.* B38, 2405–2408]. The corrected model, refined in the space group $P\bar{1}$, rules out the existence of any conformational polymorphism in this compound. Small differences in the powder-diffraction patterns calculated for the original and corrected structures of the triclinic polymorph illustrate the sensitivity of the above method for polymorph validation.

1. Introduction

Efficient tests for validating and comparing crystal structures are increasingly important and needed. The crystallographic databases contain a huge number of structural data: the latest Cambridge Structural Database (CSD; Allen, 2002) alone consists of nearly 400 000 entries of organic and metal-organic compounds (<http://www.ccdc.cam.ac.uk>), most of which include the atomic positions. Thus, literature studies may require screening of a considerable amount of data which should be efficiently analysed. A possibility exists that despite rigorous validation procedures applied by the specialized crystallographic journals prior to publication of crystal structures, and by the crystallographic databases, some of the structures may be published with some kind of error (Marsh, 1999; Herbstein *et al.*, 2002; Marsh *et al.*, 2002; Clemente & Marzotto, 2004; Flack *et al.*, 2006). Therefore, new methods of visualizing (Katrusiak, 2001; Bruno *et al.*, 2002), validating (Ferguson, 2002; Spek, 2003), comparing (Boldyreva *et al.*, 1997; Dziubek & Katrusiak, 2004a) and classifying (Belsky *et al.*, 1995; Zorky, 1996) crystal structures are sought. Recently, *distance–distance plots* were successfully applied for resolving the question of polymorphism of 3 α ,4 α -dihydro-4 β ,10-dimethyl-2-phenyl-1*H*,3*H*,5*H*-pyrrolo[3,4-*b*]carbazol-1,3-dione (Dziubek & Katrusiak, 2004b). There are also crystallographic studies which are experimentally challenging, such as real-time imaging of crystal structures in microseconds, determinations of crystal structures at extreme conditions of high temperature and pressure, structural determinations of organic compounds and zeolites from powder data or investigations of very complex (proteins) or highly imperfect structures (*e.g.* undergoing phase transitions). Such experiments often provide insufficient data for unequivocal struc-

tural determinations and can be aided by efficient visualization and validation procedures. Also the information about crystal structures of chemical compounds becomes increasingly important because of patent litigations, particularly in the pharmaceutical industry. Efficient means of distinguishing similar phases and identifying possible errors are necessary.

In this study we have tested validation procedures based on intramolecular and intermolecular contacts in polymorphs α and β of 2,2-aziridinedicarboxamide (Brückner, 1982), denoted (I). These polymorphs were originally denoted with letters *A* and *B*, respectively. Compound (I) is regarded as a unique organic compound exhibiting polytypism (Fichtner & Grell, 1984; Bernstein, 2002; Fábíán *et al.*, 2004), of conformational polymorphs (Bernstein & Hagler, 1978), and as one of the structures with the largest Z' in the CSD. A search of the CSD, Version 5.27 updated in May 2006 (Allen, 2002), for the structures with $Z' \geq 10$ revealed 31 entries. However, some of these structures are disordered and refined to a high R factor, some were shown to be refined in a symmetry which is too low and Z' was overestimated (Nekola *et al.*, 2002; Hao, Chen *et al.*, 2005; Hao, Parkin & Brock, 2005), and for some of these structures atomic coordinates have not been archived at all. Only nine structures with $Z' \geq 10$ were considered to be reliable by Hao, Parkin & Brock (2005), who rejected erroneous and suspicious cases; two additional structures [refcode LANBOS, $Z' = 16$ (Banerjee *et al.*, 2005) and LANXOO02, $Z' = 12$ (Dobrzycki *et al.*, 2005)] were announced more recently. Thus, the convincing evidence of $Z' \geq 10$ appears to exist for approximately ten structures, a very small fraction of the structures collected in the CSD. One of them is the β polymorph of 2,2-aziridinedicarboxamide.

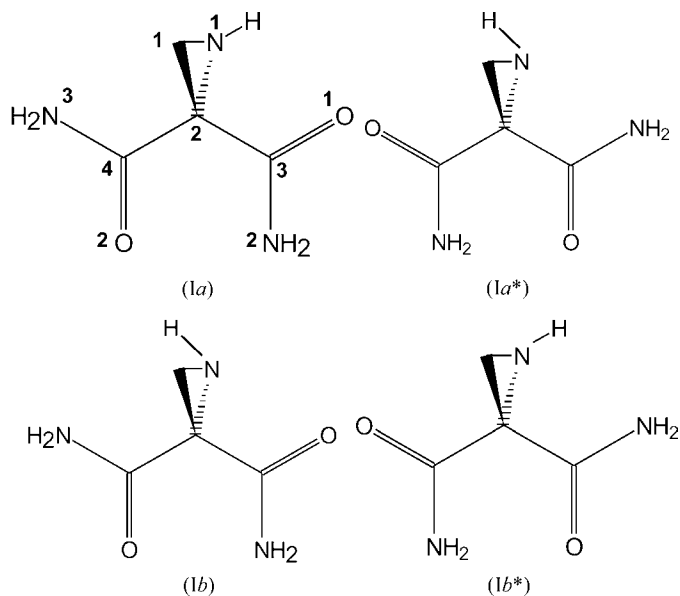
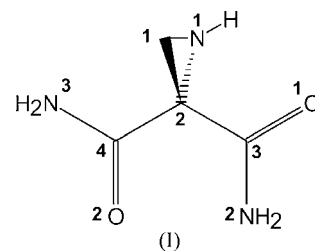


Figure 1

Conformations *anti-syn* (*Ia*), *syn-anti* (*Ib*), and their enantiomers [(*Ia**) and (*Ib**)] – the conformation descriptors refer to the positions of the amide groups with respect to the aziridine ring, the first one for that on the side of the H atom at the nitrogen of the aziridine ring.



Polymorph α is tetragonal, in the space group $P4_21_2$ and $Z = 32$ ($Z' = 4$); polymorph β is triclinic, in the space group $P1$ and $Z = 16$. The experimental data for the β sample, because of the low quality of the crystals, was insufficient for precisely refining the structure – the final R factor reported by Brückner (1982) for 2534 independent F values with $F \geq 2.5\sigma_F$ (of 5683 measured reflections) was $R_1 = 0.07$. Owing to the low quality of the data (and the possibility of twinning as mentioned by the author), the positions of the O and N atoms in the amide groups were designated only tentatively (Brückner, 1982). Four molecular conformations, (*Ia*), (*Ib*), (*Ic*) and (*Id*) (see Figs. 1 and 2), were reported among the symmetry-independent molecules in polymorph β . Only one conformation (*Ia*) was observed in polymorph α , as is consistent with the NMR results, suggesting the formation of an intramolecular N–H...O bond and a considerable barrier of 75.8 (13) kJ mol⁻¹ for the inversion of the aziridine nitrogen (Trapentier *et al.*, 1983). A search of the geminal dicarboxamides [the molecules containing the moiety >C(CONH₂)₂] in the CSD (Version 5.27 as above) resulted in 38 hits, 31 of which contained atomic positions. In 21 of them the N–C=O planes of amide groups were substantially twisted (the interplanar angle above the threshold of 45°, arbitrarily chosen by us); hence these structures could not be compared with that of 2,2-aziridinedicarboxamide. The remaining ten structures with both amide groups roughly planar are:

- six structures where the molecules adopt the conformation (*Ia*)/(*Ib*) (*anti-syn/syn-anti*);
- two structures with the (*Ic*) form of molecules, which however act as the bidentate ligands coordinating metals through the O atoms;
- one structure exhibiting disorder, for which molecular forms (*Ia*)/(*Ib*) were postulated (Usha & Venkatesan, 1979);

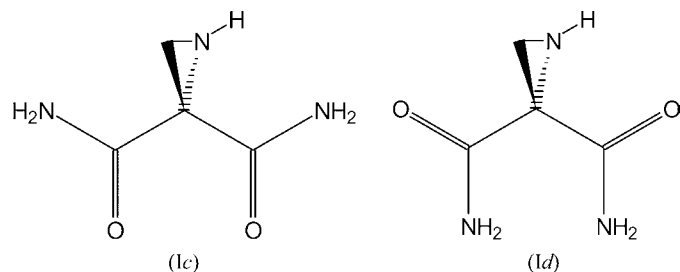


Figure 2

Two unlikely conformations reported in the β structure (Brückner, 1982): *syn-syn* (*Ic*) and *anti-anti* (*Id*).

Table 1

Crystal data and experimental details of the corrected structure of the triclinic polymorph of 2,2'-aziridinedicarboxamide (polymorph β').

The information about the X-ray experiment are reported after Brückner (1982)

Crystal data	
Chemical formula	C ₄ H ₇ N ₃ O ₂
<i>M_r</i>	129.13
Cell setting, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	293 (2)
<i>a</i> , <i>b</i> , <i>c</i> (Å)	15.829 (2), 12.381 (2), 12.391 (2)
α , β , γ (°)	90.00 (10), 74.30 (10), 83.10 (10)
<i>V</i> (Å ³)	2319.5 (6)
<i>Z</i>	16
<i>D_x</i> (Mg m ⁻³)	1.479
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.12
Data collection	
Diffractometer	Philips PW1100
Data collection method	$\theta/2\theta$ step scan
Absorption correction	None
No. of measured, independent and observed reflections	5683, 5683, 2534
Criterion for observed reflections	<i>I</i> > 2.5 σ (<i>I</i>)
<i>R</i> _{int}	0.000
θ_{\max} (°)	22.2
Refinement	
Refinement on	<i>F</i> ²
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.095, 0.218, 1.21
No. of reflections	2534
No. of parameters	585
H-atom treatment	Mixture of independent and constrained refinement
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 35.3943P]$, where $P = (F_o^2 + 2F_c^2)/3$
(Δ/σ) _{max}	< 0.0001
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.49, -0.38

Computer programs used: *SHELXL97* (Sheldrick, 1997), *XP* (Siemens, 1990).

(iv) the structure of the triclinic 2,2'-aziridinedicarboxamide. Thus, while the (*Id*) form can be ruled out because of steric hindrances, form (*Ic*) exists only in the molecules that form coordinate bonds. Otherwise, the (*Ic*) form of 2,2'-aziridinedicarboxamide is energetically unfavoured compared with (*Ia*)/(*Ib*) because of the stabilizing effect of the intramolecular N(aziridine)—H...O hydrogen bond. It was our intention to compare the intermolecular interactions of the molecules in their different crystal environments. It has been found that several very short and chemically unreasonable intramolecular contacts can be eliminated and significant differences in molecular interactions can be considerably reduced by altering the assignments of atomic types and locations of H atoms in the original model of polymorph β . The alternative model of crystal structure β , built only of molecules (*I*) in conformation (*Ia*) (the same as in polymorph α), has been refined on the original deposited structure factors and compared with the original model. The original and new structures of polymorph β have been subjected to a series of traditional and new tests aimed at detecting possible errors in these determinations, and the general usability of these tests has been discussed.

2. Experimental

The Supplementary Publication No. SUP 36838 containing the structure factors of polymorph β (*F_o* and *F_c* tables) was bought from the British Library Lending Division. We have typed 2534 *F_o* magnitudes into a computer file, as the quality of the copies was insufficient for any optical character-recognition programs. In this way we succeeded in recovering all the reflection data measured by Brückner (1982), however, the e.s.d.s of the reflections were not deposited and could not be obtained from the author either. The PDF file scanned from microfilm with *F_o* and *F_c* values obtained from the IUCr Electronic Archive was incomplete (2 pages missing) and its low quality did not allow to read about 10% of the data. Therefore, this source of the structure factors could not be used.

In the corrected model of polymorph β (denoted β'), all the molecules have been transformed into the (*Ia*) [or (*Ia**)] conformation. All the H atoms have been placed in calculated positions based on molecular geometry. Two models of the triclinic polymorph, the original β and the new β' model, have been refined with *SHELXL97* (Sheldrick, 1997). We attempted to refine Brückner's original β model (96 non-H atoms anisotropic and 48 isotropic, 77 H atoms included and 35 missing), however, in this refined model 66 of the displacement ellipsoids were non-positive definite. The number of measured reflections (2534), is small for the number of parameters (1377) in this model, *i.e.* less than 2 reflections/parameter. For these reasons the original β model could not be accepted and it was decided to repeat the refinement of the structure with all non-H atoms isotropic. The refinement of the isotropic β model gave *R*₁ = 0.11, which is considerably larger than that reported by Brückner of 0.07 for the partly anisotropic model with non-positive displacement ellipsoids. When all non-H atoms were assigned anisotropic temperature factors, most were refined to non-positive definite values. After correcting the atomic types it became apparent that polymorph β is centrosymmetric and the unit-cell origin has been appropriately shifted to a centre of inversion. Of all 72 non-H atoms in eight symmetry-independent molecules of model β' in the space group *P* $\bar{1}$, 54 could be refined with positive-definite anisotropic parameters. When the atomic types were reassigned to conform to the (*Ia*) and (*Ia**) conformations for all 16 molecules in the structure in space group *P*1, the *PLATON* ADDSYM-CHECK search (Spek, 2003) immediately indicated the *P* $\bar{1}$ space-group symmetry of the crystal. The crystal and refinement data of the centrosymmetric β' structure are summarized in Table 1.¹ The β' model of the triclinic polymorph has been deposited as a new entry in the Cambridge Crystallographic Database Centre.

¹ Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV5079). Services for accessing these data are described at the back of the journal.

3. Discussion

3.1. Comparison of the refined models

The verification of the hypothesis that the triclinic polymorph is centrosymmetric (model β') has been hampered by

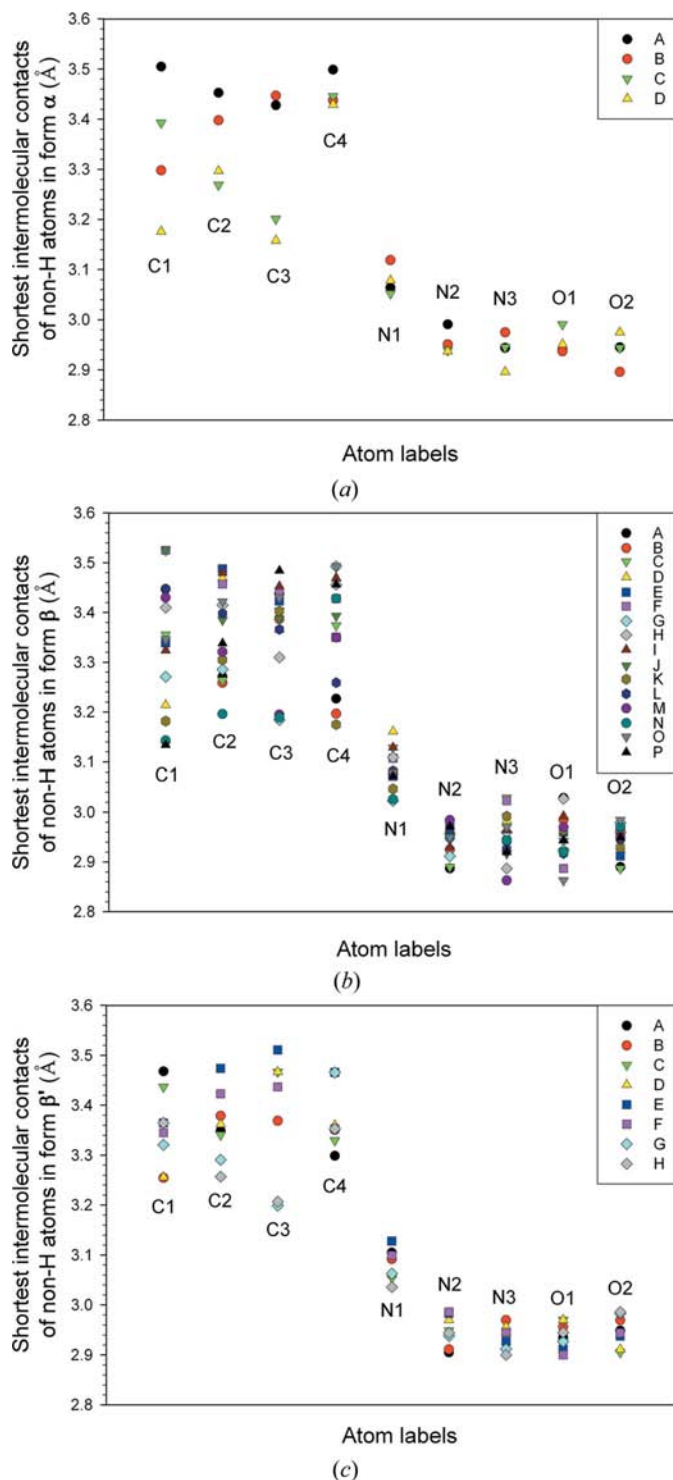


Figure 3 Scatterplots of the shortest intermolecular contacts in polymorphs (a) α , (b) β and (c) β' . The symmetry-independent molecules in polymorph α have been labelled with letters A–D, and in structure models β and β' with letters A–P and A–H, respectively.

some ambiguity concerning the original β non-centrosymmetric model:

(i) the β model was not complete, as 35 H atoms were missing;

(ii) 96 out of 144 non-H atoms in the β model were assigned anisotropic displacement parameters, while the refinement of this β model repeated by us resulted in almost all non-positive definite ellipsoids.

The corrected β' model could not be refined with all non-H atoms anisotropic, although the number of non-positive definite atoms was considerably smaller than in the β model (35 versus 77% of atoms, respectively). Moreover, the R factor calculated for the original β model was higher than that originally reported by Brückner (1982), which could be caused by excluding the weighting scheme based on the e.s.d.'s of the structure factors (now not available). Because of the poor quality and low completeness of the data, the models of structures β and β' have been refined with anisotropic

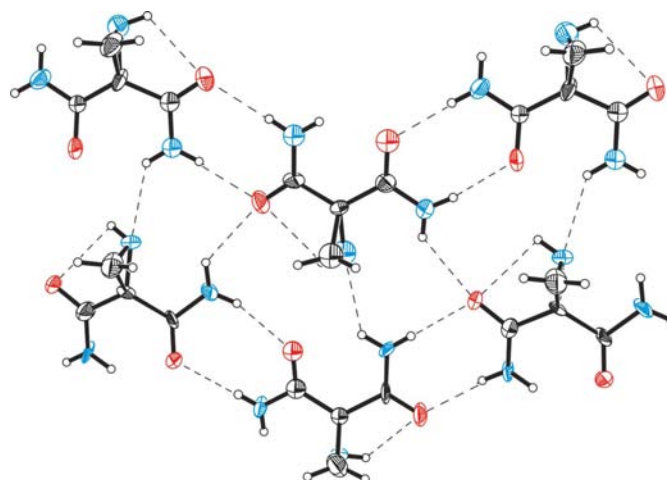


Figure 4

One layer of hydrogen-bonded molecules in polymorph β' viewed perpendicular to the crystallographic direction [100]. The displacement ellipsoids are plotted at the 50% probability level.

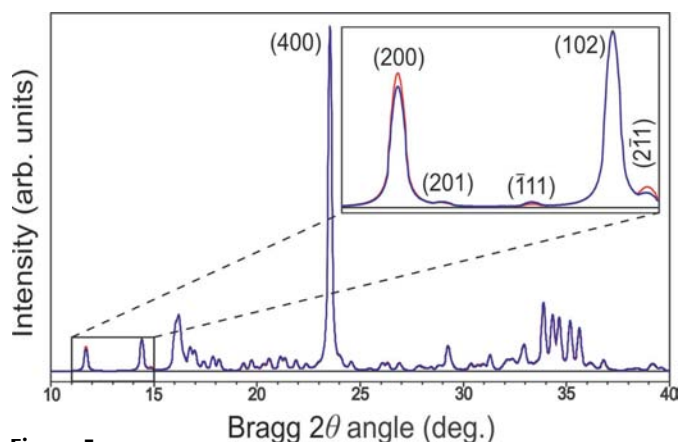


Figure 5

Powder-diffraction patterns calculated for models β (indicated in blue) and β' (red) for Cu $K\alpha$ radiation. The inset shows reflections (200) and (211) with the largest difference in intensity.

Table 2

Intramolecular and intermolecular contacts (Å) involving the aziridine NH in the β' structure.

$D-H\cdots A$	$D\cdots A$ (Å)	$H\cdots A$ (Å)	$D-H\cdots A$ (°)
Intramolecular distances			
N1A–H1A \cdots O2A	2.68 (2)	2.33 (2)	103.1 (14)
N1B–H1B \cdots O2B	2.78 (2)	2.38 (2)	106.6 (15)
N1C–H1C \cdots O1C	2.80 (2)	2.37 (2)	109.5 (14)
N1D–H1D \cdots O1D	2.69 (2)	2.35 (2)	102.4 (14)
N1E–H1E \cdots O2E	2.77 (2)	2.47 (2)	100.5 (16)
N1F–H1F \cdots O2F	2.77 (2)	2.50 (2)	98.3 (15)
N1G–H1G \cdots O1G	2.78 (2)	2.39 (2)	106.7 (15)
N1H–H1H \cdots O1H	2.81 (2)	2.44 (2)	105.4 (16)
Intermolecular distances			
N1A–H1A \cdots O2D ⁱ	3.11 (2)	2.55 (2)	120.7 (17)
N1B–H1B \cdots O2C ⁱⁱ	3.24 (2)	2.85 (3)	107.4 (21)
N1C–H1C \cdots O1B ⁱⁱⁱ	3.12 (2)	2.64 (3)	114.4 (18)
N1D–H1D \cdots O1A ^{iv}	3.06(2)	2.72(3)	103.5 (20)
N1E–H1E \cdots O2H ^v	3.21 (2)	2.77 (3)	111.1 (22)
N1F–H1F \cdots O2G ^{vi}	3.21 (2)	2.69 (3)	117.5 (19)
N1G–H1G \cdots O1F ⁱⁱⁱ	3.16 (2)	2.79 (3)	106.0 (20)
N1H–H1H \cdots O1E ^{vii}	3.15 (2)	2.80 (3)	104.5 (22)

Symmetry codes: (i) $x, y+1, z-1$; (ii) $x, y-1, z$; (iii) x, y, z ; (iv) $x, y, z+1$; (v) $x, y-1, z+1$; (vi) $x, y, z-1$; (vii) $x, y+1, z$;

temperature factors retained only for the positive-definite atoms, while the non-positive atoms have been refined with isotropic factors. The non-centrosymmetric β model refined to $R_1 = 0.1056$ and the final parameters of the centrosymmetric β' model are listed in Table 1.

3.2. Intermolecular distances in structures α , β and β'

It is well known that close non-bonding distances in crystal structures are often characteristic of interacting groups. In specific cases, when assignments of atomic types based on the electron density in Fourier maps, molecular connectivity or dimensions are dubious for any reason, either during the initial stages of structure solving or due to poor quality and low resolution of the experimental data, the intermolecular contacts can be used as an additional criterion for distinguishing atoms. The specific well known features of the intermolecular contacts are the van der Waals radii of the atoms, and their possible involvement in hydrogen bonds or other types of weak interactions. The non-bonding contacts can also be employed for validating the crystal structures.

The inspection of intramolecular contacts in the structural model of polymorph β , containing improbable conformations (Ic) and (Id) (see Fig. 2), revealed abnormally short intramolecular N \cdots N and O \cdots O distances, and unreasonable geometry of the intermolecular hydrogen bonds forming chains arranged into layers. The closest intermolecular contacts in the structural model of polymorph α , and the two structural models β and β' are shown in Fig. 3.

It becomes immediately apparent that the corresponding shortest contacts are more disperse in β than in β' . For example, the shortest contacts of C1 range between 3.134 and 3.525 Å (difference of 0.391 Å) in β versus 3.255–3.468 Å (difference of 0.213 Å) in β' ; of N1 between 3.023 and 3.161 Å (0.138 Å difference) versus 3.036 and 3.128 Å (difference of

0.092 Å); and for O1 between 2.863 and 3.029 Å (difference of 0.166 Å) versus 2.900 and 2.970 Å (difference of 0.070 Å), respectively. The dispersion of contacts in β' becomes very similar to that in α , which is reasonable because the molecules are likely to assume positions in similar environments. An analogous observation concerns longer van der Waals contacts – considerably narrower in β' compared with β . The observed widening of the ranges of contacts in β can be explained by the lower precision of atomic positions in this lower-symmetry model, resulting from the lower $N_{\text{data}}/N_{\text{parameters}}$ and the random errors of distances increased by *ca* 3 e.s.d.s. This spread of distances in β is additionally widened by systematic errors caused by the confused atomic types and H positions. Thus, the widening of the spread of distances in the β model can be considered as a measure of random and systematic errors. When considering the physical meaning of the observed distance distributions of models β and β' , the β' model is more likely since it has a more uniform distribution of intermolecular contacts. The similar distances reflect the absence of voids and strained contacts in the structure.

In the β' structure the aziridine nitrogen considerably contributes to the molecular conformation by hydrogen bonding the aziridine ring with the carboxamide oxygen (see Fig. 4). The dimensions of intramolecular aziridine NH \cdots O hydrogen bonds in β' are listed in Table 2, along with the next closest intermolecular contacts of the aziridine NH to the O atoms. The aziridine NH group is the H donor for the intramolecular hydrogen bond only.

3.3. Validation by powder diffraction pattern

Recently, a similarity criterion based on the comparison of powder diffraction patterns has been presented (van de Streek & Motherwell, 2005). In Fig. 5 the calculated X-ray powder diffraction patterns of the triclinic form for original and new models have been compared. Despite the changed space-group symmetry, exchanged N with O atoms, and misplaced H-atom positions, the X-ray powder diffraction patterns are very similar. The powder diffraction patterns differ only slightly in reflection intensities and therefore the powder diffraction method appears to be an efficient test for identifying the identical structures despite the possible atomic-type assignment errors (van de Streek, 2006). On the other hand, the validating procedures based on intra- and intermolecular distances can be very helpful in distinguishing different compounds and polymorphs with similar powder patterns. The powder method appears insensitive for distinguishing similar structural models. It should be noted that the (200) reflection, which according to the calculated powder patterns is sensitive to the structural differences between models β and β' (see the inset in Fig. 5), was absent from the list of measured reflection intensities.

4. Conclusions

The example of 2,2-aziridinedicarboxamide polymorphs illustrates the possible difficulties in the process of structural

data mining and data validation. Despite a very poor diffraction experiment, it has been shown without doubt that polymorph β is centrosymmetric. This conclusion has been based on:

- (i) conformational analysis and intramolecular distances;
- (ii) the spread of intermolecular distances;
- (iii) the comparison of the reliability factors of the least-squares refinements and the consistency of the refined models.

The structures determined from low-quality data require special attention. In such cases automated validating procedures can be considerably supported by chemical and supramolecular information. In polymorph β , both the molecular and supramolecular information indicate errors in the original model. Such errors misled all the validating procedures available to us and only after correcting the atomic types was the too low space-group symmetry immediately indicated by a *PLATON* ADDSYM check (Spek, 2003). Thus, structure refinements and chemical information can be considerably supported by the statistical analysis of non-bonding distances, which can be applied as an additional robust and powerful tool for validating structural models. Such a statistical analysis of non-bonding contacts can be efficiently applied both at the stage of solving a new structure and when mining huge structural databases. Naturally, one can argue that better data could definitely resolve the problem of crystal symmetry and atomic positions in polymorph β of 2,2-aziridinedicarboxamide, but in this particular case and many others (Brückner, 1982) collecting good quality data is a problem. Then one has to rely, tentatively at least, on computational methods of validating the results. Similarly, most of the researchers performing data mining are not usually prepared to verify all the dubious or very interesting structures (outliers) experimentally.

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