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### Systematic error differences between two refined sets of position coordinates for $\text{Na}_3\text{PO}_3\text{CO}_2 \cdot 6\text{H}_2\text{O}$ .

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Half-normal probability plot analysis shows that systematic error is present in one or both sets of the refined position coordinates for  $\text{Na}_3\text{PO}_3\text{CO}_2 \cdot 6\text{H}_2\text{O}$  [Naqvi, Wheatley & Foresti-Serantoni, (1971). *J. Chem. Soc. (A)*, p. 2751] and that the least-squares derived standard deviations are too small by a factor of at least 1.3 and possibly by as much as 2.5.

Independent solution and refinement of a given crystal structure by two different laboratories is becoming increasingly frequent. Unless gross differences are found between the two sets of refined atomic position coordinates, detailed comparison is not often made. The purpose of this note is to point out the kind of information so readily available in a comparison by means of normal probability plot analysis. The example is taken from a recent study on trisodium phosphonoformate hexahydrate (Naqvi, Wheatley & Foresti-Serantoni, 1971). The first two authors used 2238 visually estimated structure factors (Set 1), the last

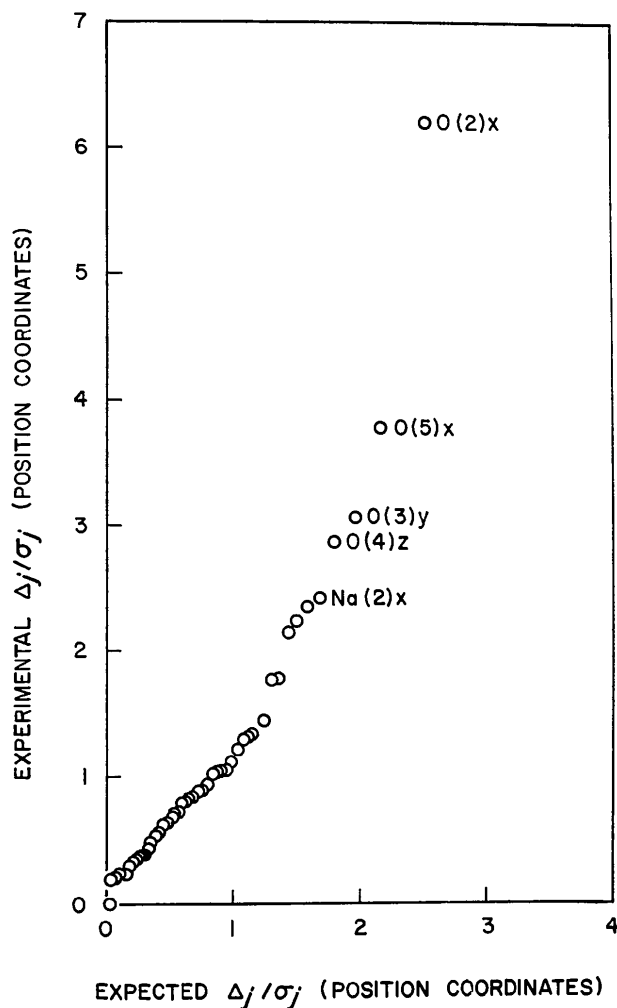


Fig. 1. Half-normal probability plot of  $\Delta_j/\sigma_j$  for the 48 position coordinates refined using Set 1 and Set 2 data.

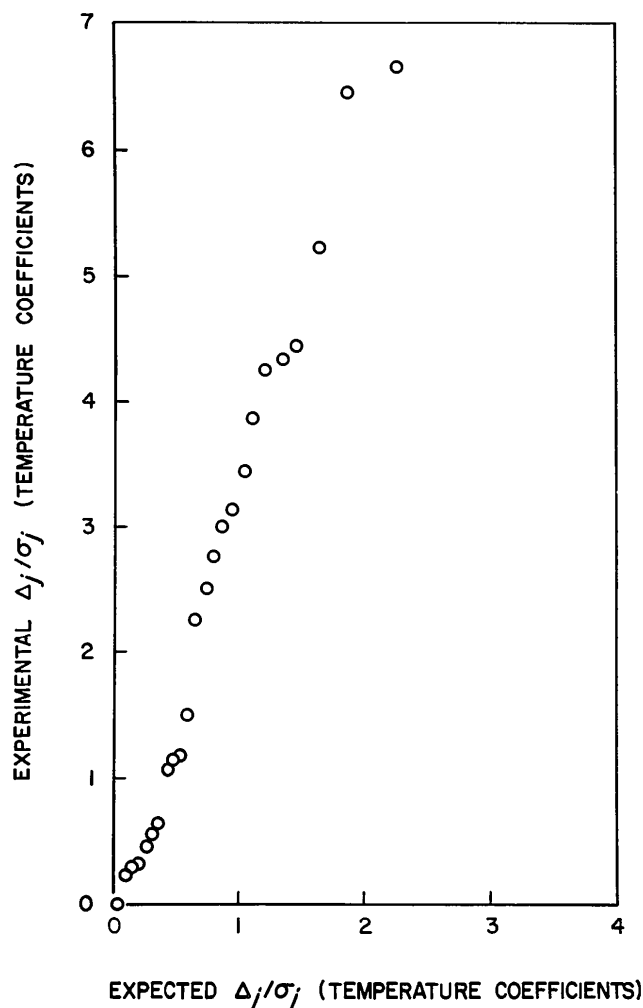


Fig. 2. Half-normal probability plot of  $\Delta_j/\sigma_j$  for the 24 anisotropic temperature coefficients obtained from Set 1 and Set 2 data, with estimated standard deviations as in text.

author independently estimated microdensitometrically 1550 structure factors (Set 2) on a different crystal, but from the same source. The authors state that 'in general, the two sets of derived atomic coordinates agree well, though there are one or two exceptions'.

Half-normal probability plot analysis (Abrahams & Keve, 1971) requires the ordered, experimental values of  $\Delta_j/\sigma_j$  to be plotted against the expected values (Hamilton & Abrahams, 1972), where  $\Delta_j$  is the difference between the

corresponding  $j$ th coordinate obtained from Set 1 and Set 2, and  $\sigma_j$  is the pooled standard deviation of  $\Delta_j$ , i.e.  $\Delta_j = |p(1)_j - p(2)_j|$  and  $\sigma_j = \{\sigma^2 p(1)_j + \sigma^2 p(2)_j\}^{1/2}$ . Results derived from data containing a random normal distribution of error would give a linear plot of zero intercept and unit slope. Fig. 1 shows the half-normal probability plot based on the final atomic coordinates given for the 16 independent non-hydrogen atoms refined from Set 1 and Set 2 and the corresponding least-squares derived standard deviations.

Fig. 1 is not linear and demonstrates the presence of systematic error in one or both sets of atomic coordinates. The initial slope (all points with experimental  $\Delta_j/\sigma_j < 2.0$ ) is about 1.3: that for the final point, O(2) $x$ , is 2.5. In the absence of further experimental information to aid in partitioning the systematic error between Sets 1 and 2, it is assumed that the standard deviations in both sets are underestimated by a factor of at least 1.3 and possibly as much as 2.5.

Standard deviations were not given for the anisotropic

temperature coefficients for Set 2: these coefficients are listed for the four heaviest atoms, and Debye-Waller factors only for the remaining lighter atoms. Assuming all  $\sigma B_{ij}$  for Set 1 are equal at  $0.08 \text{ \AA}^2$  (as given by Naqvi *et al.*, except for some smaller value phosphorus  $\sigma B_{ij}$ ) and those for Set 2 equal at  $0.22 \text{ \AA}^2$ , Fig. 2 is obtained for the resulting 24 coefficients. The array in Fig. 2 is considerably more linear than in Fig. 1; the slope in Fig. 2 shows that these assumed standard deviations are underestimated by about a factor of 3.2.

#### References

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 NAQVI, R. R., WHEATLEY, P. J. & FORESTI-SERANTONI, E. (1971). *J. Chem. Soc. (A)*, p. 2751.

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**The crystal structure of toluene- $\alpha$ ,2-dicarboxylic acid,  $C_9H_8O_4$ : errata.** By M. P. GUPTA and M. SAHU, *Department of Physics, University of Ranchi, Ranchi-8, India*

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Corrected values of interatomic distances, bond angles and deviations from atomic planes are given for toluene- $\alpha$ ,2-dicarboxylic acid.

Our attention has been drawn by Professor Jerry Donohue (private communication) to some numerical errors in the geometrical factors reported in Tables 3, 4, 5 and 6 of our paper (Gupta & Sahu, 1971).

A recalculation of the above values has shown that the following changes should be noted (there are no errors in the atomic coordinates as published earlier).

Bond:	C(3)–C(4)	1.367 Å
H-bond:	O(1)–O(2)*	2.65
H-bond:	O(3)–bO(4)*	2.61

Bond angles:	
O(1)–C(1)–O(2)	120.0°
O(1)–C(1)–C(2)	112.1
O(2)–C(1)–C(2)	127.9
C(8)–C(9)–O(3)	113.8

O(3)–C(9)–O(4)	125.9
C(8)–C(9)–O(4)	120.3
C(7)–C(2)–C(3)	120.3
C(1)–C(2)–C(3)	123.1
C(1)–C(2)–C(7)	116.6

The deviations of atoms from the best plane through the central aromatic ring are given in parentheses below.

C(2)	(–0.007),	C(3)	(0.009),	C(4)	(0.008)
C(5)	(–0.028),	C(6)	(0.029),	C(7)	(–0.013)

Full details of the recalculated values are available from the authors.

#### Reference

- GUPTA, M. P. & SAHU, M. (1971). *Acta Cryst.* B **27**, 2469.