Acta Cryst. (1973). B**29**, 143

The crystal structure of urea oxalic acid (2:1): a correction.

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(*Received* 9 *October* 1972; *accepted* 10 *October* 1972)

Incorrect cell constants have been given for urea oxalic acid (2:1) in the paper by Harkema, Bats, Weyenberg & Feil [*Acta Cryst.* (1972). B**28**, 1646–1648]. The correct values are: $a=5{\cdot}110$ (3), $b=12{\cdot}400$ (3), $c=7{\cdot}035$ (2) Å, $\beta=98{\cdot}13$ (7)°. Corrections to the original tables of bond distances and angles are given.

In the calculation on the crystal structure of urea oxalic acid (2:1) (Harkema, Bats, Weyenberg & Feil, 1972) a wrong set of cell constants was used. The correct values for the cell constants are: $a=5{\cdot}110$ (3), $b=12{\cdot}400$ (3), $c=7{\cdot}035$ (2), $\beta=98{\cdot}13$ (7)°. The error has no appreciable effect on atomic positional parameters. The reported U values can easily be corrected for the difference in cell constants. Changes in bond angles and distances for some bonds are significant; therefore, corrections to Tables 4 and 5 in our paper are given.

Table 4. *Bond distances* (Å)

C(2)–C(2′)	1·537 (5)	N(1)–H(4)	0·83 (2)
C(2)–O(2)	1·208 (3)	N(2)–H(2)	0·87 (4)
C(2)–O(3)	1·298 (5)	N(1)–H(5)	0·88 (3)
O(3)–H(1)	0·99 (4)	O(1)······O(3)	2·493 (5)
C(1)–N(1)	1·329 (6)	O(2)······N(2)	3·022 (5)
C(1)–N(2)	1·329 (4)	O(3″)····N(2)	3·127 (3)
C(1)–O(1)	1·261 (3)	O(2‴)···N(1)	2·911 (3)
N(2)–H(3)	0·85 (2)	O(1′)····N(1)	2·954 (4)

Table 5. *Bond angles* (°)

C(2′)–C(2)–O(2)	121·7 (2)
C(2′)–C(2)–O(3)	112·7 (3)
O(2)–C(2)–O(3)	125·7 (2)
C(2)–O(3)–H(1)	110 (3)
O(1)–C(1)–N(1)	120·0 (3)
O(1)–C(1)–N(2)	121·5 (2)
N(1)–C(1)–N(2)	118·5 (3)
C(1)–N(1)–H(5)	118 (3)
C(1)–N(2)–H(2)	119 (2)
C(1)–N(1)–H(4)	123 (3)
C(1)–N(2)–H(3)	120 (2)
H(4)–N(1)–H(5)	119 (3)
H(2)–N(2)–H(3)	119 (4)

It appears that the crystal structure of the title compound has been determined by another research group (Folting, Merritt, Schuch & Sturdivant, 1972). Comparison of results, which are now in excellent agreement, revealed the above mentioned error.

References

Harkema, S., Bats, J. W., Weyenberg, A. M. & Feil, D. (1972). *Acta Cryst.* (1972). B**28**, 1646–1648.

Folting, K., Merritt, L. L., Schuch, A. F. & Sturdivant, J. H. (1972). Private communication.

Acta Cryst. (1973). B**29**, 143

The formation of the plastic crystal phase in several pentaerythritol derivatives.*

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(*Received* 2 *August* 1972; *accepted* 14 *September* 1972)

Several substituted pentaerythritol compounds [$R-C(CH_2OH)_3$, where $R=COOH$, CH_3, NO_2 and NH_2] have been found to undergo phase transitions to plastic crystals. With the exception of $R=NH_2$ (which has only one detectable diffraction line in the plastic state), the crystals are *f.c.c.*, $a\simeq 8{\cdot}9$ Å.

Many compounds composed of tetrahedral, approximately spherical molecules undergo solid–solid phase transitions. Crystals of the highly disordered phase stable just below the melting point (phase I) are generally referred to as *plastic crystals* (Timmermans, 1961). Pentaerythritol, $C(CH_2OH)_4$, has been shown to undergo a transition at 452·7°K (Nitta & Watanabé, 1938) to just such a phase. As part of our attempts to define the sphericity of the molecular envelope that is required in order for plastic crystals to form (Rudman & Post, 1968; Rubin & Rudman, 1973), a series of substituted pentaerythritol compounds was selected for investigation.

The following compounds [$RC(CH_2OH)_3$] were studied: I tris(hydroxymethyl)acetic acid, $R=-COOH$; II 2-hydroxymethyl-2-methyl-1,3-propanediol, $R=-CH_3$; III 2-hydroxymethyl-2-nitro-1,3-propanediol, $R=-NO_2$: IV 2-amino-2-hydroxymethyl-1,3-propanediol, $R=-NH_2$. Samples purified by several recrystallizations from methanol and by vacuum sublimation gave identical results.

Each compound was studied using the following methods: (*a*) differential scanning calorimetry (DSC) from 120°K to above the melting point utilizing techniques described elsewhere (Silver & Rudman, 1970); (*b*) observation through a polarizing microscope over a temperature range from room temperature to above the melting point; and (*c*) X-ray diffraction flat-plate powder photographs (using nickel-filtered Cu K radiation) of the high-temperature phases. The high-temperature studies were carried out using a commercially available wound-filament quartz-tube gas heater* with compressed air.

The results of these studies (Table 1) show that as the samples are warmed, each of the compounds undergoes a solid–solid phase transition from an optically birefringent crystal to an isotropic crystal. Compounds II and IV have

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* The support of the National Science Foundation is gratefully acknowledged.

* Gas heater, Model SGH 114680, Sylvania Special Products Division, Exeter, New Hampshire 03833, USA.