Short Communications

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Preliminary X-ray crystallographic data for methyl 3-*O*-(1-carboxyethyl) shikimate δ-lactone.

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Optically active methyl 3-*O*-(1-carboxyethyl) shikimate δ-lactone (I) was synthesized by Sprecher & Sprinson (1962) as part of their studies of aromatic amino acid biosynthesis. The exact configuration of the substituents on the carbon α to the carbonyl group in the lactone ring is unknown.

Compound (I) was crystallized by slow cooling from benzene. The procedure yields highly birefringent triclinic laths, elongated along **c**, and frequently twinned on the (100) face. Traces of α and β lie in the (100) plane.

The X-ray crystallographic data were obtained from oscillation and precession photographs (Cu $K\alpha$). The density was determined by the modified density gradient tube procedure (Low & Richards, 1952; Richards & Thompson, 1952).

$a = 11{\cdot}32 \pm 0{\cdot}03$ Å $\alpha = 101{\cdot}8 \pm 0{\cdot}6°$
$b = 8{\cdot}48 \pm 0{\cdot}03$ $\beta = 99{\cdot}3 \pm 0{\cdot}6$
$c = 6{\cdot}52 \pm 0{\cdot}03$ $\gamma = 102{\cdot}1 \pm 0{\cdot}6$

Space group $P1$

$D_m = 1{\cdot}413 \pm 0{\cdot}005$ g.cm^{-3}.

Molecular weight as determined, assuming two molecules per unit cell, 248 ± 6.

Formula weight for $C_{11}O_6H_{14}$, 242.

No further work on this structure is planned.

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References

Low, B. W. & Richards, F. M. (1952). *J. Amer. Chem. Soc.* **74**, 1660.

Richards, F. M. & Thompson, T. E. (1952). *Anal. Chem.* **24**, 1052.

Sprecher, M. & Sprinson, D. B. (1962). Private communication.

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A simple multi-film intensity technique for the precession camera.

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A recent short communication by Williams (1965) described a modified Zoltai screen (Zoltai, 1963) for the Buerger precession camera, which enables any screen setting to be achieved, hence allowing the photography of zero and upper levels. Among other things the screen eliminates the progressive doubling of spots on successive films of a multi-film pack (caused by the wrong crystal-to-film distance) by the non-recording of one reflexion of each pair. Thus intensity data can be recorded much more rapidly, especially for upper levels where the exposure times are longer than for the zero level.

A much simpler method to overcome the disadvantage of partial doubling of spots on a pack of films without any adaptation of the precession camera is to misset deliberately the cassette position so as to produce doubling of spots on all films of the pack. This method works for any level which can be photographed in the ordinary way. The convenience of the method is limited by the spacings of the spots recorded on the films; if the spacings are too small the photographs become difficult to interpret owing to partial overlap of spots with different indices. The method can thus not be used for crystals with large repeat distances. The doubling becomes zero at the limit of the recorded area of the reciprocal-lattice plane, since points there enter and leave the sphere of reflexion at the same time.

For zero-level photographs the film cassette must be displaced towards the crystal, whereas for upper levels the film cassette can be displaced away from the crystal (giving photographs similar to that figured by Buerger, 1964, p. 95). For zero-level photographs a displacement of the film cassette towards the crystal of 0·5–1 mm is usually adequate, if the crystal is not too large. For upper-level photographs the cassette must be sufficiently displaced away from the crystal so that the distance of the film nearest the crystal

is greater than the correct setting – a displacement of 2 mm is usually adequate. The doubling can be determined from curves given by Zoltai (1963). The method is most applicable to upper-level photography, because of the relatively longer exposure times needed. The exposure time for the pack of films must of course be doubled, as with the Zoltai screen, but a saving of time is achieved because up to six films can be exposed simultaneously.

The method has one further possible advantage – because the cassette is displaced away from the crystal for upper-level photography, the value of ζ which can be recorded, before mechanical fouling of the cassette and the camera occurs, is increased slightly and thus in certain cases one higher level than normal can be recorded.

References

BUERGER, M. J. (1964). *The Precession Method in X-ray Crystallography*, p. 95. New York: John Wiley.
WILLIAMS, P. P. (1965). *Acta Cryst.* **19**, 859.
ZOLTAI, T. (1963). *Amer. Min.* **48**, 759.

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The effect of absorption errors in a crystal structure refinement.

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Because of the widely divergent opinions held by crystallographers on the subject of the extent to which failure to correct for absorption effects causes errors in both position and thermal parameters, we have carried out a series of calculations designed to investigate the question empirically. We have selected as a test compound bisethylenediaminepalladium(II) chloride, whose structure has recently been determined in this laboratory (Wiesner & Lingafelter, 1966). It contains one molecule of $Pd(C_2N_2H_8)_2Cl_2$ in a triclinic cell ($P\bar{1}$) of dimensions $a_0=6{\cdot}878$, $b_0=8{\cdot}325$, $c_0=4{\cdot}980$ Å, $\alpha=95°44'$, $\beta=101°25'$, $\gamma=108°52'$ ($\mu=191$ cm^{-1} for Cu $K\alpha$ radiation). Using the final atomic positions and a set of reasonable isotropic thermal parameters, we have calculated a set of structure factors corresponding to 97% of the Cu $K\alpha$ sphere. For each member of a series of assumed crystal shapes we then calculated the absorption effect for Cu $K\alpha$ radiation for each reflection and applied this effect to the calculated structure factor, in this manner arriving at a set of hypothetical 'observed' structure factors uncorrected for absorption. The absorption effect has been calculated for diffractometer geometry assuming the crystal to be mounted about the c axis on an Eulerian cradle, and intensities to be collected with the plane of the χ axis normal to the plane of the ω and 2θ axes and bisecting the angle between the incident and diffracted beams. These hypothetical F_o's have then been used to carry out full-

Table 1. *Parameter changes with estimated standard deviations in parentheses*

Thermal parameter changes and e.s.d. $\times 10^2$
Positional parameter changes and e.s.d. $\times 10$

Atom		Iα, IVα	Iω	IIα	IIω	IIIα	IIIω	IVω
Pd	*B*	−29 (1)	−2 (3)	−92 (2)	−57 (4)	−47 (3)	−5 (9)	−215 (4)
Cl	*x*	−5 (23)	−8 (53)	−4 (43)	−12 (87)	−15 (59)	−36 (177)	3 (83)
	y	1 (19)	1 (43)	3 (35)	1 (71)	−2 (48)	2 (144)	11 (67)
	z	1 (30)	−3 (70)	7 (57)	−8 (114)	−7 (78)	30 (232)	10 (109)
	B	−30 (3)	−2 (6)	−93 (5)	−56 (9)	48 (6)	−5 (19)	−2·17 (9)
N(1)	*x*	−23 (73)	−87 (168)	115 (137)	−119 (277)	−96 (193)	369 (538)	−21 (268)
	y	−60 (60)	−81 (136)	−12 (112)	45 (225)	−346 (157)	−840 (438)	176 (218)
	z	21 (97)	99 (220)	−70 (180)	−18 (363)	253 (252)	300 (705)	−88 (351)
	B	−23 (8)	5 (18)	−88 (14)	−49 (28)	−29 (20)	−16 (56)	−214 (25)
N(2)	*x*	93 (72)	80 (161)	65 (134)	113 (262)	124 (183)	282 (535)	−59 (262)
	y	−14 (58)	2 (131)	−85 (109)	−48 (213)	53 (149)	115 (436)	−315 (212)
	z	95 (94)	223 (211)	91 (176)	172 (343)	−439 (239)	−1063 (702)	371 (341)
	B	−31 (7)	−8 (17)	−92 (13)	−70 (26)	−48 (19)	−10 (56)	−217 (24)
C(1)	*x*	89 (100)	−9 (224)	231 (186)	−275 (369)	−223 (260)	106 (851)	−364 (364)
	y	−16 (81)	82 (182)	−93 (151)	73 (300)	88 (212)	590 (692)	−89 (296)
	z	186 (130)	161 (293)	240 (244)	441 (483)	471 (341)	3165 (1115)	11 (476)
	B	−29 (10)	−6 (24)	−93 (19)	−63 (37)	−35 (27)	+78 (93)	−219 (34)
C(2)	*x*	−9 (99)	18 (225)	−10 (186)	36 (371)	−64 (255)	154 (764)	98 (365)
	y	21 (81)	54 (183)	−8 (152)	93 (302)	22 (208)	234 (623)	−72 (299)
	z	9 (131)	55 (295)	−48 (245)	45 (487)	101 (355)	753 (1004)	−140 (481)
	B	−29 (10)	−4 (24)	−92 (19)	−60 (38)	−46 (26)	6 (81)	−217 (34)
	R	0·036	0·081	0·066	0·135	0·087	0·290	0·134
	l_1(mm)	0·064	0·064	0·127	0·127	0·064	0·064	0·510
	l_2(mm)	0·078	0·078	0·157	0·157	0·157	1·411	0·470
	l_3(mm)	0·098	1·947	0·122	1·951	0·122	1·463	0·488