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Crystal data for $Zn(NO_3)_2 . 4H_2O$ and $Zn(NO_3)_2 . 2H_2O$.

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Unit-cell dimensions and space groups for $Zn(NO_3)_2.2H_2O$ and $Zn(NO_3)_2.4H_2O$ have been determined on the basis of rotation and Weissenberg photographs. Crystals of $Zn(NO_3)_2.4H_2O$ are isostructural with $Ni(NO_3)_2.4H_2O$.

As a part of our interest in nitrate hydrates of bivalent metals, we have examined crystals of zinc nitrate tetrahydrate and zinc nitrate dihydrate. Both compounds were prepared by methods already described (Mellor, 1952). The crystallization was performed in a dessicator over calcium-chloride or phosphorus pentoxide. The crystals obtained were colorless and quite hygroscopic monoclinic prisms. For further examination the crystals were sealed in capillary tubes. Crystal data, as determined from the analysis of rotation and Weissenberg photographs, are listed in Table 1 along with the data for $Ni(NO_3)_2 . 4H_2O$ (Gallezot, Weigel & Prettre, 1967) for comparison. The densities were determined by flotation in bromoform–ethyl iodide.

Similar values of lattice constants and the same space-group extinctions for zinc nitrate tetrahydrate and nickel nitrate tetrahydrate, whose crystal structure was solved recently (Gallezot *et al.*, 1967) suggest either isomorphism or close relationship of their structures. The determination of the crystal structure of $Zn(NO_3)_2 . 2H_2O$ is in progress.

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Table 1. *Crystal data*

	a (Å)	*b* (Å)	*c* (Å)	*β*	*V* (Å³)	*Z*	Space group	D_m (g.cm⁻³)	D_x (g.cm⁻³)	Reference
$Zn(NO_3)_2.2H_2O$	5·754 ±0·006	5·978 ±0·005	8·557 ±0·005	91°2′ ±20′	294·34	2	$P2_1/c$	2·50	2·54	This work
$Zn(NO_3)_2.4H_2O$	5·321 ±0·003	27·36 ±0·02	5·730 ±0·003	113°40′ ±20′	764·03	4	$P2_1/n$	2·22	2·27	This work
$Ni(NO_3)_2.4H_2O$	5·305 ±0·01	27·24 ±0·03	5·705 ±0·01	114° ±20′	753	4	$P2_1/n$	2·21	2·24	Gallezot *et al.* (1967)

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An I.C.T. Fortran program for least-squares refinement of crystal-structure cell dimensions.

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A computer program is described for the accurate calculation of unit-cell dimensions when the 'd_{hkl}' values (or simply line positions) and *hkl* indices are known. A weighted least-squares extrapolation method is used with provision for an eccentricity correction. The cubic, tetragonal, hexagonal, rhombohedral and orthorhombic crystal systems are included, and both single-crystal and powder data are acceptable. Various forms of data input and output are available.

The program was designed to be as general as possible whilst remaining simple to use. The crystal systems included are cubic, tetragonal, hexagonal, rhombohedral and orthorhombic. The rhombohedral indices must be referred to hexagonal axes. The input data required are the *hkl* indices and either 'd_{hkl}' values or 'line positions'. This latter can be in the form of 2θ values, or distances measured on the film from some arbitrary origin. Although the program was designed primarily for measurements made on films mounted in the Straumanis setting, it may be used without change with other film arrangements.

The calculation is made with each reflexion weighted according to the scheme suggested by Hess (1951)

$$w = 1/[\sin^2(\pi - 2\theta)] .$$

Using the Nelson & Riley (1945) function to allow for the effect of absorption and beam divergence on the measurements, it can be shown that

$$\partial(\sin^2\theta) = -\mu/2\,.\,\sin^2 2\theta\,.\,(1/\sin\theta + 1/\theta)$$

where μ is the systematic error constant for a particular film.

The program uses the relation

$$\sin^2\theta \simeq \alpha A + \beta B + \gamma C + \partial(\sin^2\theta);$$

hence the difference between the observed and theoretical values of $\sin^2\theta$ is

$$\Delta = \sin^2\theta - \alpha A - \beta B - \gamma C - \delta D\,.$$

By the method of least-squares the best values of A, B, C, D are those for which $\Sigma_i(w\,.\,\Delta_i^2)$ is a minimum. The standard deviations of these quantities are also calculated.

For accurate work provision is made for inclusion of an eccentricity correction, which can be varied independently from the data.

Data derived from Debye–Scherrer photographs and single-crystal rotation photographs are equally acceptable.

The print-out includes θ, observed d_{hkl}, calculated d_{hkl}, the weight, the value of the Nelson–Riley function, and the differences between observed and calculated $\sin^2\theta$. Some variations are possible.

The program is written in I.C.T. 1900 Fortran (Fortran IV) for use on the 1905 computer. The listing together with full instructions for use are available from the authors on request. A similar, separate program has been written for the monoclinic system, for which a listing is available.

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Crystal data on some oxoiodates.

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Crystal data and crystallographic descriptions are given of $Sr(IO_3)_2.H_2O$, $Ca(IO_3)_2.6H_2O$, $Sr(IO_3)_2.6H_2O$, $CdH_3IO_6.3H_2O$, $CaH_3IO_6.3H_2O$, $Sr(IO_4)_2.7H_2O$.

In connexion with an investigation of the structures of the oxoanions of iodine(V) and iodine(VII) (Ferrari, Braibanti & Tiripicchio, 1965), the crystal data of some oxoiodates of bivalent cations have been determined by X-ray Weissenberg methods (Cu $K\alpha$ radiation, $\lambda=1{\cdot}5418$ Å). The I^V compounds were prepared by concentrating solutions of the corresponding periodates in concentrated hydrochloric acid and the I^{VII} compounds by evaporating solutions obtained by dissolving the metal carbonates in aqueous periodic acid. All the compounds are colourless. Densities were measured by the pycnometer method.

(1) $Sr(IO_3)_2\,.\,H_2O$. Stable, shiny, monoclinic crystals. Optically biaxial. Observed forms: {001}, {100}, {110}, {$\bar{1}$01}, {$\bar{1}$11}, {$\bar{1}$12}. Rotation axis for the X-ray analysis: [010]. $a=13{\cdot}12\pm1$, $b=7{\cdot}74\pm1$, $c=8{\cdot}95\pm1$ Å, $\beta_x=133°6'\pm10'$, $\beta_{\rm gon}=132°35'\pm8'$, $V=668$ Å^3, $Z=4$, $D_m=4{\cdot}522$, $D_x=4{\cdot}520$ g.cm^{-3}, $(a:b:c)_x=1{\cdot}695:1:1{\cdot}156$, $(a:b:c)_{\rm gon}=1{\cdot}709:1:1{\cdot}160$. No piezoelectric effect was observed. Space group: $C2/c$ or Cc (from systematic absences). From the crystal data it appears to be a member of the isostructural series $M^{II}(XO_3)_2\,.\,H_2O$ (M^{II} = Ca, Sr, Ba, Pb and X = Cl, Br, I) (Groth, 1908).

(2) $Ca(IO_3)_2\,.\,6H_2O$. Shiny, short piezoelectric prisms. Orthorhombic. Rotation axis: [001]. $a=23{\cdot}12\pm1$, $b=15{\cdot}03\pm1$, $c=6{\cdot}43\pm1$ Å, $V=2236$ Å^3, $Z=8$, $D_m=2{\cdot}967$, $D_x=2{\cdot}957$ g.cm^{-3}. Space group: $Fdd2$ (from systematic absences). The X-ray data correspond to the goniometric measurements given by Marignac (1857), Eakle (1896) and later by Mélon (1935): $(a:b:c)_x=1{\cdot}538:1:0{\cdot}428$, $(a:b:c)_{\rm gon}=1{\cdot}547:1:0.428$ (from Mélon's data).

(3) $Sr(IO_3)_2\,.\,6H_2O$. Small, prismatic, orthorhombic crystals. Rotation axis: [001]. $a=23{\cdot}57\pm1$, $b=15{\cdot}28\pm1$, $c=6{\cdot}50\pm1$ Å, $V=2341$ Å^3, $Z=8$, $D_m=3{\cdot}205$, $D_x=3{\cdot}097$ g.cm^{-3}. Isostructural with (2).

(4) $CdH_3IO_6\,.\,3H_2O$. Prismatic, monoclinic crystals which lose $4H_2O$ at 120° and the last $\frac{1}{2}H_2O$ at 170°C. Observed forms: {010}, {011}, {021}, {$\bar{1}$02}, {$\bar{1}$21}. Rotation axis: [100]. $a=5{\cdot}96\pm1$, $b=14{\cdot}38\pm1$, $c=9{\cdot}78\pm1$ Å, $\beta_x=121°19'\pm15'$, $\beta_{\rm gon}=120°25'\pm11'$, $V=723$ Å^3, $Z=4$, $D_m=3{\cdot}605$, $D_x=3{\cdot}604$ g.cm^{-3}, $(a:b:c)_x=0{\cdot}414:1:0{\cdot}680$, $(a:b:c)_{\rm gon}=0{\cdot}409:1:0{\cdot}672$. No piezoelectric effect was observed in agreement with the space group ($P2_1/c$) deduced from the systematic absences. The crystals are probably the same as those studied by Rammelsberg (1869) and quoted in Groth (1908) as possibly orthorhombic.

(5) $CaH_3IO_6\,.\,3H_2O$. Monoclinic prisms, which alter in air. Observed forms: {010}, {100}, {011}, {$\bar{1}$02}, {$\bar{1}$22}. Rotation axis: [001]. $a=5{\cdot}85\pm1$, $b=14{\cdot}56\pm1$, $c=9{\cdot}98\pm1$ Å, $\beta_x=120°7'\pm6'$, $\beta_{\rm gon}=120°5'\pm12'$, $V=736$ Å^3, $Z=4$, $D_m=2{\cdot}920$, $D_x=2{\cdot}886$ g.cm^{-3}, $(a:b:c)_x=0{\cdot}402:1:0{\cdot}679$, $(a:b:c)_{\rm gon}=0{\cdot}401:1:0{\cdot}684$. Isostructural with (4).

(6) $Sr(IO_4)_2\,.\,7H_2O$ or $SrH_2I_2O_9\,.\,6H_2O$. Formula given on the basis of the chemical analysis. Thick, opaque, triclinic crystals. No piezoelectric effect was observed. Rotation axis: [001]. $a=6{\cdot}21\pm1$, $b=11{\cdot}15\pm1$, $c=11{\cdot}63\pm1$ Å, $\alpha=55°25'\pm8'$, $\beta=73°35'\pm1°23'$, $\gamma=64°56'\pm52'$, $V=597$ Å^3, $Z=2$, $D_m=3{\cdot}311$, $D_x=3{\cdot}309$ g.cm^{-3}. The compound seems to correspond to that described by Rammelsberg (1868) as a hexahydrate.

Further structural work is in progress at this Institute for (1), (2) and (4) only.

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