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ON THE DETERMINATION OF THE LATTICE TYPE AND UNIT CELL PARAMETERS OF A CRYSTAL USING ELECTRON DIFFRACTION

K.S. Milliken

Metal Physics Section

MINERALS RESEARCH PROGRAM

November 1975

Physical Metallurgy Research Laboratories
CANMET Report 76-6

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Catalogue No. M38 - 13/76-6

Nº de catalogue M38-13/76-6

Price subject to change without notice

Prix sujet à changement sans avis préalable

Information Canada
Ottawa, 1975

Information Canada
Ottawa, 1975

PHYSICAL METALLURGY RESEARCH LABORATORIES
CANMET REPORT 76-6

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by
K.S. Milliken*

ABSTRACT

The goniometer stage of a Philips EM300 transmission electron microscope has been used as a measuring device in lattice determination. Hewlett-Packard 9810A programs have been written to control the lattice calculations. The use of the programs is described and two examples are given.

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LABORATOIRES DE RECHERCHE EN MÉTALLURGIE PHYSIQUE
RAPPORT DE CANMET 76-6

À PROPOS DE LA DÉTERMINATION DU TYPE DE RÉSEAU ET DES PARAMÈTRES DE MAILLE ÉLEMENTAIRE D'UN CRISTAL À L'AIDE DE LA DIFFRACTION ÉLECTRONIQUE

par
K. S. Milliken*

RÉSUMÉ

Le porte-objet du goniomètre d'un microscope électronique de transmission Philips EM300 a été utilisé pour déterminer le réseau. Des programmes Hewlett-Packard 9810A on été écrits afin de contrôler les calculs de réseau. L'utilisation des programmes est décrite et deux exemples sont donnés.

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INTRODUCTION

Programs have been written to facilitate determination of the lattice constants and orientation of a crystal, using data from three mutually primitive diffraction spots observed in a Philips EM 300 transmission electron microscope, at crystal orientations measured with a double-tilt holder in a goniometer stage. The equipment required to run these programs is a Hewlett-Packard 9810A programmable desk calculator equipped with printer, 111 registers, 1012 program steps, Mathematics ROM, and Printer Alpha ROM.

The present report is intended as a guide for scientists who want to obtain rapid results by the procedure to be described. It does not explain the details of the calculations which are automated by the programs.

This procedure has been applied to the determination of the lattice constants of precipitates and matrices and their relative orientations, and also to the indexing of X-ray powder patterns by single crystal electron diffraction from individual powder particles.

To measure the positions of the diffraction spots and the orientations of the images of the tilt axes on the photographic plate, the following coordinate system has been chosen. Let the shadow of the beam stop define the direction of the Y-axis and take the origin at the transmitted beam spot (which is photographed by a short second exposure of the same plate). See Figure 1, which shows a row of electron diffraction spots as seen by an observer seated at the EM 300 console. The vectors T1 and T2 are drawn in the directions of the images of the tilt axes of the Philips goniometer. T1 refers to the axis whose direction is fixed horizontally in the microscope column. T2 refers to the short axis mounted in the double-tilt specimen holder and it is only horizontal, as shown in Figure 1, when the reading on the T1 degree circle is zero.

The directions of the images of the goniometer axes depend on microscope conditions. They are shown in Figure 1 for the conditions we normally use. We have found that, in diffraction patterns obtained with the function switch set at D + I, the intermediate lens set at 2 (fine control at minimum) and the high voltage at 100 kV, the image of the T1 axis is located 77.1 degrees clockwise from the X-axis. The magnitude of this angle, 77.1, is stored in program steps 359 to 362 of side 1 of both programs T1, T2 COMPLETE and T1, T2 VISIBLE. The user must determine this angle for the particular settings used on his EM 300 and then change the above program steps accordingly. We have a FORTRAN program which optimizes this parameter, so after much experience we know that 77.1 is correct for us within a few tenths of a degree. The user should probably try a few values when running data for a known cubic crystal and then choose the value which gives the most symmetry to the output of the COMPLETE program. See also the footnote to page 4.

When either program is executed, the user must put in data specifying the location of the observed diffraction spots for three reciprocal-lattice vectors. The program treats this input triplet of vectors as the defining basis $\underline{A}^*$, $\underline{B}^*$, $\underline{C}^*$ of a triclinic reciprocal lattice $\underline{R}^*=H\underline{A}^* +K\underline{B}^*+L\underline{C}^*$.

INPUT DATA

(after each entry, press CONTINUE)

(1) L*LAMBDA. This is the number of millimeters on the photographic plate which are equivalent to one reciprocal Ångström in the reciprocal lattice. If the operator keeps the objective lens strength constant and maintains focus in bright field during tilting by using the height adjustment device on the goniometer, then $L\lambda$ will remain essentially constant. For our conditions $L\lambda \simeq 35$ millimeter Ångströms.

(2) θ. This is the angular polar coordinate of the diffraction spot or row of spots, measured in degrees counterclockwise from the X-axis.

(3,4) R2 and R1. These are the coordinates of two diffraction spots measured on a millimeter coordinate line along the above row.

(5) N. This integer is the order of the spacing between the readings R2 and R1. The program will calculate the primitive spacing, (R2-R1)/N, between spots on the row being measured. (R2-R1)/N is the radial polar coordinate, r, shown in Figure 1. One may discover as the analysis proceeds that the first order was absent, and rerun the data with N doubled. Because there is a non-linear radial distortion in the plates, it is wise to confine measurements to the central region.

(6) T1. This is the reading on the degree circle of the first goniometer axis, which is always horizontal. $-45° < T1 < +45°$. Positive rotations are defined as follows. If a right-hand screw were rotated so as to advance in the direction of the arrow, on the drawing of the image of the T1 axis, then the value of T1 would increase.

(7) T2. This is (in degrees) the angle of rotation about the second goniometer axis, which is horizontal only when T1 = 0. Readings from the Philips scale of 0 to 200 must be converted to degrees before entry into the HP9810A. Our calibration agrees with that of Pumphrey and Bowkett, (Rev. Sci. Instr. vol. 42, 216, 1971). We disagree with the Philips calibration by about 5° at the extremities.

Load data for the other two vectors by repeating, entries (2) through (7) twice. These three vectors should be chosen to be mutually primitive, i.e., the parallelepiped formed from these three vectors should contain no reciprocal lattice points within it or on its surface or edges except at the eight corners. The following experimental procedure usually gives three mutually primitive vectors. After observing the two shortest non-collinear vectors of a reasonably dense net, the sample should be tilted in such a manner* that the most densely occupied row of spots through the origin is held in view until another reasonably dense net is observed. The shortest vector of the new net, not on the held row, completes the required triplet. Unless a space group extinction occurs, which is not compensated by double diffraction, these three vectors will be mutually primitive. Reiteration of this procedure, to obtain neighbouring nets of higher density, will lead to more accurate results. On the other hand, the tilt coordinates of the densest nets will be listed in the output, so an exhaustive search on the microscope is not warranted.

*In general, the row can only be held by rotating the crystal alternately about both axes. If a row happens to have such a direction that it can be held while using only the T1 axis, then the direction of the row coincides with the direction of the image of the T1 axis, and program steps 359 to 362 can be loaded accordingly. With a Philips rotation holder, one can put a row parallel to the image of the T1 axis, if a single crystal is used, such as silicon, sliced at right angles to any direct lattice vector.

OUTPUT

After each of the three vectors is entered there are three lines of output. These contain the rectangular components of the vector, in reciprocal Ångströms, with respect to the rectangular axes T1, T2, and the optic axis T1 x T2, when the crystal is in the orientation T1 = 0, T2 = 0. This output is normally not used. However, there may occasionally be difficulty in choosing, from more than three observed vectors, a set of three which are mutually primitive. Then, analysis of these rectangular components, for all observed vectors, can reveal a mutually primitive triplet.

The next output is the volume of a parallelepiped of the direct lattice, in cubic Ångströms. If the input triplet happens to be second order, this volume will be half the volume of a primitive cell. If the input triplet happens to consist of coplanar vectors, this volume will be very large.

Then, after loading the balance of the program, there is a delay of less than three minutes for calculations. This is followed by printing of the list, which is a series of records each six words long. The words are:

(1) R**2. This is the square of the length of a direct-lattice vector, in square Ångströms.

(2,3,4) X,Y,Z. These three integers are the indices of this direct-lattice vector $\underline{R} = X\underline{A} + Y\underline{B} + Z\underline{C}$, $\underline{A}$, $\underline{B}$, and $\underline{C}$ being the vector triplet which is reciprocal to the input vector triplet.

(5,6) T1, T2. These are the goniometer settings at which this direct-lattice vector will be parallel to the optic axis and at which a net should be observable.

There are two HP9810A versions of the program: T1, T2 COMPLETE and T1, T2 VISIBLE. They accept the same input data, and produce the same general form of output. Direct-lattice vectors which are known to be non-primitive are rejected from the output of both. Direct-lattice vectors which are equal and opposite are not both included in the output. Both versions list the nets in order of increasing R**2. This results in the most dense nets being at the top of the list, since the number of reciprocal-lattice points per unit area is inversely proportional to R.

T1, T2 COMPLETE calculates all non-primitive direct-lattice vectors within a hemisphere. Memory space is dimensioned for up to seventeen vectors, although occasionally the space is not completely filled because of an underestimate of the radius of the hemisphere. The output of COMPLETE is used for the selection of conventional unit cells, and the determination of their lattice parameters. However, it should be noted in the first place that if two lists of R**2 are identical, the corresponding two crystals are identical except for orientation. Lattice symmetry elements, conventional cells and indices, and lattice parameters can be selected without difficulty using the list, if the following pointers are kept in mind.

(1) Whenever two equal values of R^2 occur on the list, say X_1, Y_1, Z_1, R_1^2 and X_2, Y_2, Z_2, R_2^2 with $R_1^2 = R_2^2$, it follows that $(\underline{R}_1 + \underline{R}_2)\cdot(\underline{R}_1 - \underline{R}_2) = 0$ and hence that $X_1 + X_2$, $Y_1 + Y_2$, $Z_1 + Z_2$ is orthogonal to $X_1 - X_2$, $Y_1 - Y_2$, $Z_1 - Z_2$. When using uncorrected data, discrepancies of 3% between "equal" values of R^2 are common in this laboratory.

(2) Whenever a value of R^2 is clearly not paired with an equal, $\underline{R}$ must lie parallel to a symmetry plane or axis (except in the triclinic case).

3) In a hemisphere, the multiplicity of equal values of R^2, for a most general lattice vector $\underline{R}$, varies with the lattice type as follows:

Type	Multiplicity
Cubic	24
Hexagonal	12
Tetragonal	8
Trigonal	6
Orthorhombic	4
Monoclinic	2
Triclinic	1

T1, T2 VISIBLE is a version of T1, T2 COMPLETE which gains the space in memory to extend the list to higher values of R**2 by not remembering direct-lattice vectors for nets with T1, T2 coordinates which exceed the 45,30 degree physical limitations of the goniometer. It produces tilt coordinates for the seventeen most densely occupied nets in the visible range, in order of decreasing density of reciprocal-lattice points. Note that if the input triplet is not mutually primitive the nets will be disordered in the list, with respect to their true density, but nets should be found at the predicted tilt coordinates. The output of VISIBLE is used mainly to facilitate locating the most dense nets to photograph for complete analysis.

Two examples will illustrate the application of these programs to the prediction of tilt coordinates, the selection of conventional unit cells, and the determination of lattice parameters.

Examine the output of T1, T2 VISIBLE in example E1. The list does not mention the net observed at T1 = -15.5 and T2 = 35.3 because of the extreme value of T2. The tilt coordinates of the first predicted net at -5.7, -8.7 agree with those at which the first input vector was observed -6.0, -8.7.

Other nets were observed at

T1	24.0	21.5	-10.7	-33.8	7.2
T2	20.0	-10.8	11.2	18.7	26.5

These tilt coordinates are in agreement with the predictions on the list.

The COMPLETE list in example E1 shows that the first three vectors have about equal lengths (R^2 = 24.1, 24.5, 24.7) and the fourth vector has a unique (unpaired) length (R^2 = 27.3). The indices of the first three are not independent since $(01\bar{1})+(\bar{1}\bar{1}0)=(\bar{1}0\bar{1})$. Hence these three vectors will form an equilateral triangle, yielding a close-packed plane. Vectors 3 and 4 add to yield $(\bar{1}\bar{1}0)+(100)=(0\bar{1}0)$ which is vector 6. The sum of their squared lengths 24.7 and 27.3 is 52.0 which is about equal to 50.7, the squared length of vector 6. By Pythagorus' theorem, vector 4 is normal to vector 3 and similarly normal to the other vectors in the close-packed plane. Obviously vector 4 is the c axis of a hexagonal lattice with $c = \sqrt{27.3} = 5.2$ A° and $a = \sqrt{24.4} = 4.9$Å. Seven plates were measured and the FORTRAN program gave c = 5.43, a = 4.92. These values are in agreement with the translation lattice of quartz.

Examine the output of T1, T2 VISIBLE in example E2, and compare with the following tilt coordinates of the nets which were observed.

T1	34.5	8.0	7.0	-7.5	21.2	-20.5	-13.2
T2	25.3	35.3	-21.8	10.8	1.3	-16.0	23.2

The COMPLETE list in example E2 shows a lot of pairing of values of R^2 (equal within about 3%) so the crystal is probably monoclinic. The first vector (R^2 = 51.8) looks unique so it is either the monoclinic axis or in the monoclinic plane. The pairs give the following orthogonality relationships by sum and difference:

	at R^2 = 59		$(001)\cdot(203) = 0$,
	at R^2 = 68		$(0\bar{1}2)\cdot(0\bar{1}0) = 0$,
	at R^2 = 86		$(0\bar{2}1)\cdot(203) = 0$,
and	at R^2 = 137		$(\bar{2}0\bar{3})\cdot(021) = 0$.

The vector (203) is seen to be normal to a lattice plane which contains (001) and (021) and hence (010). So the monoclinic axis is (203). The shortest vectors in the monoclinic plane are (010) and possibly (001). A cell based on these last three vectors is second order since their determinant is 2. In fact, the face containing (203) and (001) is centered because (203)+(001) = 2(102). If the purpose is to use the Donnays' "Crystal Data" for crystal identification, one should label the two shortest vectors in the monoclinic plane as $\underline{c}$ and $\underline{a}$ with $c < a$, β non-acute, and type P, C, A or I. Hence base vectors for the conventional cell could be taken as

$\underline{a}$	=		$\underline{C}$
$\underline{b}$	=	$2\underline{A}$	$+3\underline{C}$
$\underline{c}$	=	$-\underline{B}$	

The c face of this cell is centered. $a = \sqrt{67.7} = 8.2$ Å. $b = \sqrt{170.8} = 13.1$ Å. $c = \sqrt{51.8} = 7.2$ Å. β can be obtained using the law of cosines:

$$(\underline{a}-\underline{c})\cdot(\underline{a}-\underline{c}) = a^2 + c^2 - 2\underline{a}\cdot\underline{c},$$

$$169.9 = 67.7+51.8-2\sqrt{67.7*51.8}\cos\beta,$$

$$\beta = 115°.$$

(One should be circumspect in the choice of a conventional cell for this particular example because there are two vectors with $R^2 = 68$. These data are not sufficiently accurate to determine which of the pair is the second shortest vector in the monoclinic plane). The above matrix, $001/203/0\bar{1}0$, also transforms the Miller indices of the input reciprocal-lattice vectors from (100), (010), (001) into (020), $(00\bar{1})$, (130) with respect to the chosen cell. The transpose of its inverse may be used to transform the indices X, Y, Z

of the output direct-lattice vectors into symmetric form.

$$x = 1.5X + Z$$
$$y = +0.5X$$
$$z = -Y$$

Analysis of all the data showed that this grain has the translation lattice of an orthoclase.

Listings of the programs T1, T2 VISIBLE and T1, T2 COMPLETE may be obtained by writing to the author. If recordings are desired, please enclose four of the Hewlett-Packard 6-in. magnetic cards.

ACKNOWLEDGEMENTS

The author is grateful to his colleagues J.T. Jubb, E.E. Laufer and W.N. Roberts for all the electron microscopy which forms the experimental basis of these programs.

KSM:gt

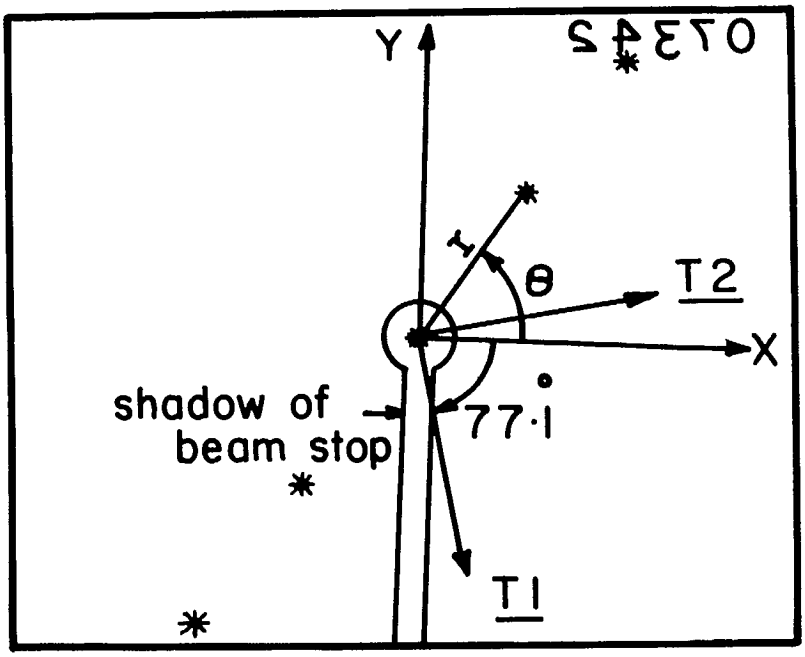

figure 1

Fig. 1. This diagram illustrates how the shadow of the beam stop is used in choosing the coordinate system for measuring the location of a diffraction spot on a photographic plate. The images of the goniometer tilt axes, T1 and T2, are shown for the microscope conditions specified in the text.

TYPEWRITTEN COPY OF PRINTED PAPER TAPE FOR EXAMPLE E1

T1,T2 VISIBLE
L * LAMBDA

34.700*
θ ,R2,R1,N 97.000*
93.750*
73.200*
2.000*

T1,T2 -6.000*
-8.700*
-0.291
0.030
0.048

θ,R2,R1,N 20.700*
88.850*
72.600*
2.000*

T1,T2 -15.500*
35.300*
-0.062
0.224
0.032

θ,R2,R1,N 141.400*
92.750*
76.450*
2.000*

T1,T2 -15.500*
35.300*
-0.127
-0.141
-0.138

VOLUME IN A**3
110.557

R**2,X,Y,Z,T1,T2
50.331
0.000
0.000
-1.000
-5.745
-8.726

51.148
1.000
1.000
-1.000
24.827
19.072

98.079
0.000
1.000
-2.000
21.874
-11.684

123.116
1.000
2.000
-2.000
41.849
9.265

130.785
1.000
0.000
-1.000
-10.618
9.422

131.450
1.000
-1.000
0.000
-34.581
16.180

132.882
2.000
1.000
-1.000
8.007
26.157

178.058
0.000
-1.000
-1.000
-25.860
-6.464

178.273
1.000
1.000
-2.000
9.887
4.579

193.980
0.000
2.000
-3.000
35.510
-13.524

T1,T2 COMPLETE
L * LAMBDA

34.700*
θ ,R2,R1,N 97.000*
93.750*
73.200*
2.000*

T1,T2 -6.000*
-8.700*
-0.291
0.030
0.048

θ ,R2,R1,N 20.700*
88.850*
72.600*
2.000*

T1,T2 -15.500*
35.300*
-0.062
0.224
0.032

θ ,R2,R1,N 141.400*
92.750*
76.450*
2.000*

T1,T2 -15.500*
35.300*
-0.127
-0.141
-0.138

VOLUME IN A**3
110.557

R**2,X,Y,Z,T1,T2
24.076
0.000
1.000
-1.000
63.729
-21.336

24.540
-1.000
0.000
-1.000
7.971
-54.261

24.685
-1.000
-1.000
0.000
-48.360
-75.212

27.331
1.000
0.000
0.000
-15.500
35.300

50.331
0.000
0.000
-1.000
-5.745
-8.726

50.736
0.000
-1.000
0.000
-45.842
-3.244

51.148
1.000
1.000
-1.000
24.827
19.072

51.668
1.000
-1.000
1.000
-53.754
60.565

53.296
2.000
1.000
0.000
18.495
61.825

53.412
2.000
0.000
1.000
-16.568
79.754

72.549
-1.000
1.000
-2.000
36.672
-44.311

72.982
1.000
2.000
-1.000
71.745
63.828

74.374
-2.000
-1.000
-1.000
-20.542
-62.666

98.079
0.000
1.000
-2.000
21.874
-11.684

98.884
-1.000
-1.000
-1.000
-26.411
-28.597

99.293
0.000
-2.000
1.000
-72.628
9.859

101.333
2.000
2.000
-1.000
41.848
45.113

TYPEWRITTEN COPY OF PRINTED PAPER TAPE FOR EXAMPLE E2

T1,T2 VISIBLE
L * LAMBDA

	35.040*	85.263
		-1.000
∅,R2,R1,N		-1.000
	-7.600*	-1.000
	16.090*	-43.622
	0.000*	-8.922
	3.000*	
T1,T2		168.508
	34.500*	-1.000
	25.300*	-1.000
	0.083	0.000
	0.118	-7.574
	-0.051	8.313
∅,R2,R1,N		
	81.900*	221.607
	10.760*	0.000
	0.000*	-1.000
	2.000*	2.000
		22.522
T1,T2	34.500*	-1.037
	25.300*	236.282
	-0.116	-2.000
	0.045	-1.000
	-0.089	-2.000
∅,R2,R1,N		-36.411
	-16.500*	18.718
	9.250*	248.185
	0.000*	-1.000
	1.000*	-2.000
T1,T2		0.000
	8.000*	-19.766
	35.300*	-16.388
	0.124	
	0.228	385.547
	0.049	-2.000
VOLUME IN A**3		-1.000
	350.039	-1.000
		-13.144
R**2,X,Y,Z,T1,T2		21.047
	67.667	387.087
	0.000	-1.000
	0.000	-1.000
	1.000	1.000
	34.500	8.618
	25.300	14.157
	69.048	395.580
	0.000	1.000
	-1.000	-1.000
	1.000	4.000
	7.209	41.255
	-22.435	-9.075

T1,T2 COMPLETE
L * LAMBDA

	35.040*	60.437	169.933
		1.000	0.000
∅,R2,R1,N		0.000	1.000
	-7.600*	2.000	1.000
	16.090*	72.471	39.408
	0.000*	-64.276	62.580
	3.000*		
		67.667	170.787
T1,T2		0.000	-2.000
	34.500*	0.000	0.000
	25.300*	1.000	-3.000
	0.083	34.500	-51.076
	0.118	25.300	60.072
	-0.051		
		69.048	173.707
∅,R2,R1,N		0.000	1.000
	81.900*	-1.000	-1.000
	10.760*	1.000	3.000
	0.000*	7.209	39.910
	2.000*	-22.435	-31.323
T1,T2			
	34.500*		
	25.300*	85.263	174.075
	-0.116	-1.000	0.000
	0.045	-1.000	-2.000
	-0.089	-1.000	1.000
		-43.622	-11.249
∅,R2,R1,N		-8.922	-45.247
	-16.500*		
	9.250*	87.168	192.477
	0.000*	1.000	-1.000
	1.000*	-1.000	0.000
		2.000	0.000
T1,T2			
	8.000*	23.995	7.894
	35.300*	-72.906	35.110
	0.124		
	0.228	135.963	197.418
	0.049	-1.000	1.000
		1.000	0.000
VOLUME IN A**3		-1.000	3.000
	350.039	4.242	59.228
		71.852	6.291
R**2,X,Y,Z,T1,T2			
	51.823	137.353	215.382
	0.000	-1.000	-1.000
	-1.000	-1.000	-2.000
	0.000	-2.000	-1.000
	-30.157	-70.239	-42.882
	-76.142	-83.122	-41.176
	58.790	168.508	
	-1.000	-1.000	
	0.000	-1.000	
	-1.000	0.000	
	-21.049	-7.574	
	44.399	8.313	

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