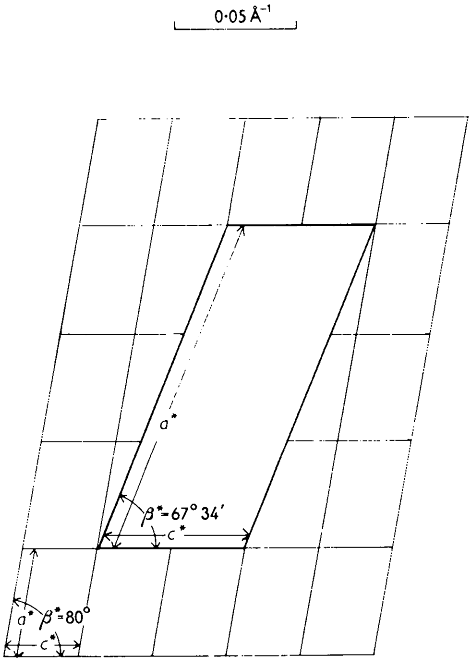

Fig. 1. The a^*c^* face of the reciprocal cell of the new form of lauric acid superimposed on the a^*c^* plane of the reciprocal lattice of the γ-form.

the unit-cell dimensions of the latter form from powder photographs. For lauric acid the dimensions were

$$a = 22{\cdot}1,\ b = 5{\cdot}24,\ c = 32{\cdot}0\ \text{Å};$$
$$\beta = 99°\ 58',\ \alpha = \gamma = 90°.$$

It was recently decided to try to check these figures by single-crystal methods.

A small quantity of γ-lauric acid was dissolved in ether and the solution allowed to evaporate slowly to dryness. The resulting material consisted of a tangle of fibres together with a few small plates. Three of these plates were selected for single-crystal examination: the remaining material was ground and a powder photograph of it taken. This photograph was identical with that used in the determination of the cell dimensions of the γ-form. Weissenberg and oscillation photographs were taken of each of the three single crystals and the unit cell was found to be triclinic with

$$a = 7{\cdot}45,\ b = 5{\cdot}40,\ c = 17{\cdot}5\ \text{Å};$$
$$\alpha = 96°\ 53',\ \beta = 113°\ 8',\ \gamma = 81°\ 7'.$$

This cell is thus different from that of the powdered fibres and does not account for all the lines on the powder photograph. There is, however, a simple relationship between the two cells which is illustrated in Fig. 1, in which the a^*c^* face of the single crystal reciprocal cell is shown superimposed on the a^*c^* plane of the reciprocal lattice of the powdered fibres. The perpendicular separations of the a^*c^* planes in the two reciprocal lattices do not differ by more than three percent.

The value of the long spacing ($1/c^*$) in the single crystal is 16·0 Å which is just half of that of the γ-form. It is of course possible that the spacing is really twice as long as reported, but no trace of weak spots to support this supposition could be found. The cell dimensions of the β form of stearic acid have been reported by von Sydow (1955) as

$$a = 5{\cdot}591,\ b = 7{\cdot}404,\ c = 49{\cdot}38\ \text{Å};\ \beta = 117°\ 22',$$

and the space group as $P2_1/a$. The short dimensions of this cell are not unlike those given above for the lauric acid plates but the space group is different and even if the long spacing of the lauric acid plates were doubled it would not agree with the value to be expected for the β-form of lauric acid.

Thus, although the unit-cell dimensions now reported bear some resemblance to those of both the β and γ-forms, they do not agree closely with either: they are quite different from those of the α-form and so it appears that a fourth crystalline form of lauric acid must be added to those previously known.

References

LOMER, T. R. (1955). *Nature, Lond.* **176**, 653.

PIPER, S. H., MALKIN, T. & AUSTIN, H. E. (1926). *J. Chem. Soc.* p. 2310.

SYDOW, E. VON (1955). *Acta Cryst.* **8**, 557.

THIBAND, J. & DUPRÉ LA TOUR, F. (1930). *C. R. Acad. Sci., Paris*, **191**, 200.

Acta Cryst. (1961). **14**, 313

The structure of crystalline uranium.

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It was proposed by Cash (1935) that the use of powder data in structure determinations of substances available only in powder form could be developed into a more powerful technique if photographs were made with as many different consecutive $K\alpha$ wavelengths as possible, so that as much of the diffraction pattern as possible could be observed in the high-resolution back-reflection region of the camera. The present work was undertaken in 1936 by one of us (A. H. C.) to test this notion. As uranium was then available only as a powder, it seemed a suitable and interesting test substance. But before these

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* Deceased, 19 August 1936. Mr Cash performed all of the experimental work reported here and had measured all the films before his untimely death.

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results were published, the work of Jacob & Warren (1937) appeared, and the project was abandoned. Since, however, there is current interest in the uranium parameter and our value differs somewhat from that of Jacob & Warren, our results are summarized briefly below.

Photographs were prepared in a vacuum precision camera similar to that of Bradley & Jay (1932) using the $K\alpha$ wavelengths of all elements from zinc to chromium, inclusive, except nickel. The uranium was somewhat fine-grained and did not produce very sharp lines, but the wealth of data from the back-reflection region and the many different estimates of the larger spacings produced measurements good to about 0·005 Å or better. These were easily indexed by elementary methods; the precision and resolution of the spacing measurements are in general more important in the indexing of powder data than is the method used. Lattice constants were derived by a least-squares fitting of seven fully resolved measurements of superior precision. Consideration of resolved lines showed the extinctions: hkl when $h+k$ is odd and $h0l$ when l is odd, indicating space groups *Cmcm* or *Cmc*. The former was assumed and produced a satisfactory structure. The resulting cell (Cu $K\alpha = 1\cdot5418$ Å) data are:

$$a = 2\cdot848 \pm 0\cdot004,\quad b = 5\cdot861 \pm 0\cdot007,\quad c = 4\cdot957 \pm 0\cdot005\ \text{Å}\ ,\quad Z = 4;$$

$$\varrho(\text{calc.}) = 19\cdot1\ \text{g.cm.}^{-3},\quad \varrho(\text{obs.}) = 18\cdot9\ \text{g.cm.}^{-3}$$

(Driggs & Lilliendahl, 1930).

Simple considerations of observed intensities showed that the four uraniums were in position *c* (*Internationale Tabellen*, 1935) with y approximately 0·1; and from the *observed* intensities, which were visual estimates, the permissible range of y was considerable. However, by considering reflections permitted by the space group but never observed on any film and others which are very weak, observed with some wavelengths and not with others, it was possible to put rather close limits on y. The final estimate was $y = 0\cdot102 \pm 0\cdot002$. Jacob & Warren gave $0\cdot105 \pm 0\cdot005$, which is consistent with our value, but less precise.

In calculating intensities, scattering factors for U were taken from the *Internationale Tabellen* (1935) and were not corrected for anomalous dispersion since for a centrosymmetric crystal with only one position occupied, this effect can alter intensities only in a way qualitatively similar to the influence of the thermal motions of the atoms, and the dispersion effect can be partially absorbed into the temperature and scale factors. The calculated intensities were corrected for multiplicity, absorption (cylindrical samples), and Lorentz-polarization factor; a temperature factor was multiplied into the uranium scattering factors. Calculated and observed spacings and intensities are compared in Table 1 for those reflections that are completely resolved.

The structure, except for small differences in bond lengths, is the same as that described by Jacob & Warren and discussed by Pauling (1940). For one atom the interatomic distances to neighbors are: 2 of 2·751 Å, 2 of 2·848 Å, 4 of 3·258 Å, and 4 of 3·343 Å. The two shortest distances (2·751 A) make with each other an angle of 128° 30′. It agrees within its own experimental error with the new and much more precise determination of Sturcken & Post (1960). We are indebted to them for prepublication information of their results.

Table 1. *Calculated and observed values of spacings and intensities**

d(obs.)	d(calc.)	hkl	I(obs.)	I(calc.)
—	2·925	020	< 15	9
2·556	2·557	110	115	117
2·510	2·517	021	130	167
2·466	2·473	002	80	90
2·267	2·271	111	115	116
—	1·888	022	< 15	10
1·773	1·778	112	130	146
—	1·608	130	< 15	12
1·526	1·529	131	115	157
1·460	1·462	040	30	29
1·432	1·436	023	60	73
1·422	1·421	200	40	40
—	1·402	041	< 20	23
1·384	1·386	113	40	53
1·345	1·348	132	17	16
—	1·278	220	< 15	5
1·255	1·258	042	40	42
1·151	1·151	133	100	88
1·1132	1·1133	114	100	58
1·093	1·094	043	15	13
1·0814	1·0818	150	60	45
—	1·057	151	< 15	0
1·0191	1·0190	240	30	29
1·0102	1·0102	223	100	76
0·9987	0·9981	241	25	24
0·9917	0·9911	152	80	82
—	0·980	134	< 15	10
—	0·975	060	< 15	11
0·954	0·956	061	15	17
—	0·888	224	< 15	7
0·876	0·875	312	80	62
0·866	0·867	243	40	30
—	0·852	330	< 15	6
0·825	0·824	006	30	31
—	0·819	045	< 15	10
—	0·802	170	< 15	3
—	0·763	172	< 15	4
0·758	0·757	333	120	150
0·747	0·746	314	120	140
0·738	0·736	350	100	130

* Resolved reflections only.

References

Bradley, A. J. & Jay, A. H. (1932). *Proc. Phys. Soc.* **44**, 563.

Cash, A. H. (1935). Master of Science Thesis, Cornell University, June 1935.

Driggs, F. H. & Lilliendahl, W. C. (1930). *Ind. Eng. Chem.* **19**, 516.

Internationale Tabellen (1935). Berlin: Borntraeger.

Jacob, C. W. & Warren, B. E. (1937). *J. Amer. Chem. Soc.* **59**, 2588.

Pauling, L. C. (1940). *The Nature of the Chemical Bond*, 2nd ed., p. 143. Ithaca, New York: Cornell University Press.

Sturcken, E. F. & Post, B. (1960). *Acta Cryst.* **13**, 853.