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1. *The Crystal Structure of Potassium Sulphamate.*

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A complete determination of the crystal structure of potassium sulphamate, $NH_2 \cdot SO_3K$, has been made by means of three-dimensional Fourier syntheses. The three oxygens and the nitrogen atom in the $[NH_2 \cdot SO_3]'$ group are arranged in an almost regular tetrahedron around the sulphur atom, the bond lengths being S–O = 1·44 A and S–N = 1·57 A (both ± 0·03 A). The structure appears to be purely ionic and there is no evidence of hydrogen bridges between neighbouring anions. Although the position of the hydrogen atoms cannot be determined directly, there is very strong evidence that they are attached to nitrogen and not to oxygen atoms.

Although numerous crystallographic investigations of salts of sulphur acids have been carried out, the majority of investigations have assumed a tetrahedral distribution of S–O bonds, 1·50 A in length, and only in very few cases have direct and independent measurements of the lengths and angles of the bonds in the acid radical been obtained. The most accurate of these have been on sodium sulphate (Zachariasen and Ziegler, *Z. Krist.*, 1932, **81**, 92) and potassium pyrosulphite (Zachariasen, *Physical Rev.*, 1932, **40**, 923); in the former, the S–O distance was found to be 1·49 A, and in the latter the values were 1·44, 1·45 and 1·49 A, the valency angles in the pyrosulphite group ranging from 102° to 116½°. The accuracy of the fractional atomic co-ordinates in potassium pyrosulphite was estimated by Zachariasen to be 0·01, so that the above interatomic distances are probably accurate to about 0·07 A.

Our attention was first drawn to potassium sulphamate by the statement (Mellor, " Comprehensive Treatise on Inorganic and Theoretical Chemistry," Vol. 8, p. 638) that it is isomorphous with sulphamic acid; this proved not to be the case, but we decided to carry out a complete analysis of the potassium salt with the object of measuring the S–O and S–N bond lengths to a higher degree of accuracy than had hitherto been obtained in investigations of sulphur acid salts. An additional reason for studying this substance was the possibility that some form of hydrogen bridge might occur between nitrogen and oxygen atoms of adjacent radicals.

The analysis has been carried out chiefly by means of three-dimensional Fourier syntheses based upon quantitative measurements of the reflection intensities from 309 lattice planes, and the interatomic distances derived from it are probably accurate to 0·03 A. The results show that potassium sulphamate has an ionic structure of a new type and contains no hydrogen bridges. The nitrogen and oxygen atoms in the tetrahedral sulphamate ion are clearly differentiated by their distances of 1·57 and 1·44 A, respectively, from the sulphur atom, and the equality of the three S–O distances shows that the ion is definitely $[NH_2 \cdot SO_3]'$, and not $[NH \cdot SO_2 \cdot OH]'$.

Reasonably good crystals of potassium sulphamate were readily grown, and the unit cell and space-group were determined in the usual way. The dimensions of the unit cell, which contains four molecules of $NH_2 \cdot SO_3K$, are $a = 8{\cdot}32$, $b = 8{\cdot}28$, and $c = 5{\cdot}90$ A, and the systematic extinctions are $\{hk0\}$ absent for h odd and $\{0kl\}$ absent for k odd, indicating as the space-group either Pba (C_{2v}^5) or $Pbma$ (D_{2h}^{11}); the latter was adopted because the crystals showed no evidence of a polar character, and nothing contrary to this emerged from the subsequent analysis. The general position in the space-group *Pbma* is eight-fold and it is therefore reasonably certain that the four potassium, four nitrogen, and four sulphur atoms in the unit cell all lie on special positions. It is further permissible to assume that of the twelve oxygen atoms in the unit cell, four (O_I) lie on special positions, and eight (O_{II}) on general positions. The special positions are (cf. "International Tables for the Determination of Crystal Structures," Vol. 2, p. 134):

(a) (000); $(\tfrac{1}{2}\ 0\ 0)$; $(0\ \tfrac{1}{2}\ 0)$; $(\tfrac{1}{2}\ \tfrac{1}{2}\ 0)$
(b) $(0\ 0\ \tfrac{1}{2})$; $(\tfrac{1}{2}\ 0\ \tfrac{1}{2})$; $(0\ \tfrac{1}{2}\ \tfrac{1}{2})$; $(\tfrac{1}{2}\ \tfrac{1}{2}\ \tfrac{1}{2})$
(c) $(\tfrac{1}{4}\ 0\ z)$; $(\tfrac{3}{4}\ 0\ \bar{z})$; $(\tfrac{1}{4}\ \tfrac{1}{2}\ z)$; $(\tfrac{3}{4}\ \tfrac{1}{2}\ \bar{z})$
(d) $(x\ \tfrac{1}{4}\ z)$; $(\bar{x}\ \tfrac{3}{4}\ \bar{z})$; $(\tfrac{1}{2} + x\ \tfrac{1}{4}\ \bar{z})$; $(\tfrac{1}{2} - x\ \tfrac{3}{4}\ z)$

If the three oxygen atoms are attached to each sulphur with any non-planar distribution of bonds, the positions (*a*), (*b*), and (*c*) for the latter atoms are excluded since they require central or axial symmetry. The four sulphur atoms in the unit cell therefore most probably lie on the planes of symmetry with co-ordinates (*d*). The necessity for reasonable atomic distances within the acid radical then requires the four nitrogen and the four special oxygen atoms to lie on the planes of symmetry.

Rough comparison of the intensities of the orders of {001}, {010}, and {100} is sufficient to show that the potassium ions cannot lie on positions (*a*), (*b*), or (*d*), and we accordingly assign them positions on two-fold rotation axes with co-ordinates (*c*). The atomic arrangement in potassium sulphamate is thus

4 K in $(\tfrac{1}{4}\ 0\ z_1)$; $(\tfrac{3}{4}\ 0\ \bar{z}_1)$; $(\tfrac{1}{4}\ \tfrac{1}{2}\ z_1)$; $(\tfrac{3}{4}\ \tfrac{1}{2}\ \bar{z}_1)$
4 S in $(x_2\ \tfrac{1}{4}\ z_2)$; $(\bar{x}_2\ \tfrac{3}{4}\ \bar{z}_2)$; $(\tfrac{1}{2} + x_2\ \tfrac{1}{4}\ \bar{z}_2)$; $(\tfrac{1}{2} - x_2\ \tfrac{3}{4}\ z_2)$
4 N in $(x_3\ \tfrac{1}{4}\ z_3)$; $(\bar{x}_3\ \tfrac{3}{4}\ \bar{z}_3)$; $(\tfrac{1}{2} + x_3\ \tfrac{1}{4}\ \bar{z}_3)$; $(\tfrac{1}{2} - x_3\ \tfrac{3}{4}\ z_3)$
4 O_I in $(x_4\ \tfrac{1}{4}\ z_4)$; $(\bar{x}_4\ \tfrac{3}{4}\ \bar{z}_4)$; $(\tfrac{1}{2} + x_4\ \tfrac{1}{4}\ \bar{z}_4)$; $(\tfrac{1}{2} - x_4\ \tfrac{3}{4}\ z_4)$
8 O_{II} in $(x_5\ y_5\ z_5)$; $(x_5\ \tfrac{1}{2} - y_5\ z_5)$; $(\bar{x}_5\ \tfrac{1}{2} + y_5\ \bar{z}_5)$;
$(\bar{x}_5\ \bar{y}_5\ \bar{z}_5)$; $(\tfrac{1}{2} + x_5\ y_5\ \bar{z}_5)$; $(\tfrac{1}{2} + x_5\ \tfrac{1}{2} - y_5\ \bar{z}_5)$;
$(\tfrac{1}{2} - x_5\ \tfrac{1}{2} + y_5\ z_5)$; $(\tfrac{1}{2} + x_5\ \bar{y}_5\ z_5)$

and ten parameters remain to be determined from intensity measurements.

From several series of oscillation photographs, together with direct comparisons of selected reflection intensities with the (400) rocksalt reflection, it was possible to obtain the absolute structure amplitudes for 309 planes; these were used to carry out appropriate syntheses as indicated below. The procedure adopted was to find the approximate co-ordinates of as many atoms as possible by means of a three-dimensional Patterson (F^2) synthesis (for which a knowledge of the phases of the structure amplitudes is not necessary), and from the atomic arrangement thus indicated to calculate the phase angles required for evaluating sections of the three-dimensional Bragg (F) synthesis; from this by successive refinement accurate values of all the parameters were eventually obtained.

Experience has shown that in the analysis of a structure of many parameters, two-dimensional Patterson syntheses are of little value except in those cases where a small proportion of the atoms in the unit cell are of markedly higher atomic number than the remainder (*e.g.*, organo-metallic compounds), and such syntheses were therefore not used in the present instance. In a structure such as that under consideration a three-dimensional Patterson synthesis can be expected to show definite peaks due to vectors between atoms of moderate atomic number (*i.e.*, K–S) and probably also those due to vectors between moderately heavy atoms and light atoms (*i.e.*, K–O, K–N, S–O and S–N). It is not likely that O–O and N–O vectors will be distinguishable except where, by chance, two or three such vectors coincide.

Since the y-co-ordinates of the potassium ions are 0 and $\tfrac{1}{2}$, while those of S, N, and O_I are $\tfrac{1}{4}$ and $\tfrac{3}{4}$, a section at $y = \tfrac{1}{4}$ of the three-dimensional Patterson synthesis

$$P_{(xyz)} = \sum_{-H}^{H} \sum_{-K}^{K} \sum_{-L}^{L} F^2_{hkl} \cos 2\pi(hx + ky + lz) \quad . \quad . \quad . \quad . \quad . \quad (1)$$

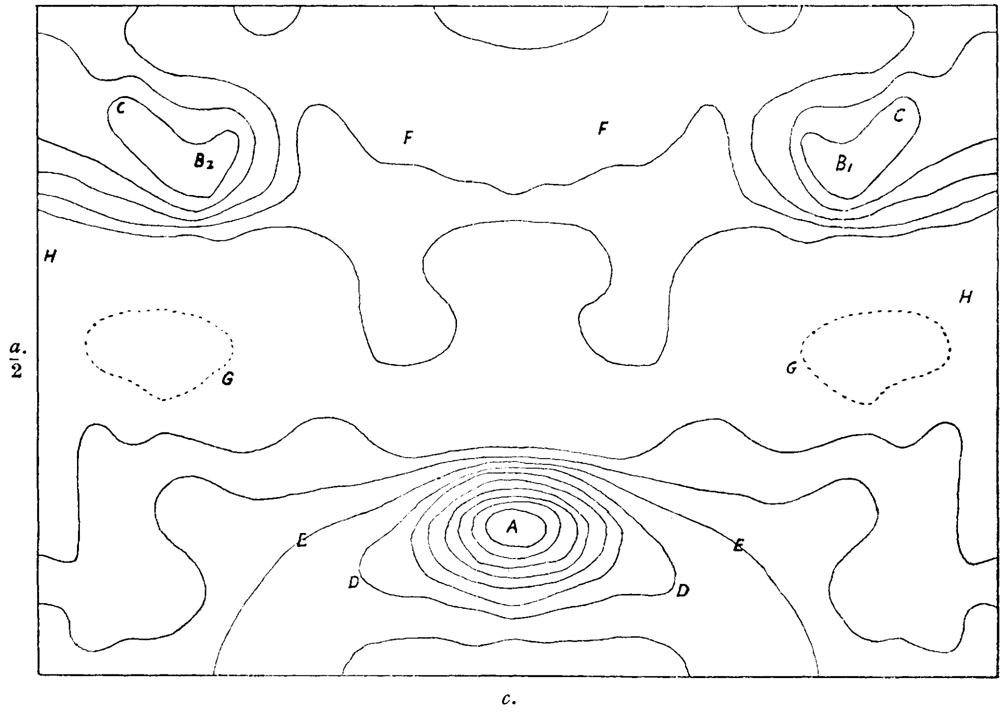
should exhibit maxima corresponding to the interatomic vectors K–S, and probably also K–N and K–O_I. For the space-group *Pbma*, the required section of (1) is

$$P_{(x\frac{1}{4}z)} \equiv 4\sum_{0}^{H}\sum_{0}^{L}\left\{\sum_{0}^{K}[(-1)^{k/2}F^2_{hkl}]\right\}\cos 2\pi hx \cos 2\pi lz \quad . \quad . \quad . \quad . \quad (2)$$

(summed for terms with k even only), and this was evaluated with the result shown in Fig. 1, plotted over half the cell with an interval of 5000 between contours. There are three very prominent maxima in the diagram, two, B_1 (0·36, 0·86) and B_2 (0·36, 0·14), being related by a plane of symmetry, while A (0·14, 0·50) is approximately twice the height of B.

Examination of the list of co-ordinates given above shows that there should be four K–S peaks in the diagram, with co-ordinates $(\frac{1}{4}+x_2,\ z_1+z_2)$; $(\frac{1}{4}+x_2,\ -z_1-z_2)$; $(\frac{1}{4}-x_2,\ z_1-z_2)$; $(\frac{1}{4}-x_2,\ z_2-z_1)$. It is evident that B_1 and B_2 are two of these and that A consists of the other two superimposed. From the co-ordinates of A and B given above we thus deduce $x_2 = 0{\cdot}11$, $z_2 = 0{\cdot}18$ and $z_1 = 0{\cdot}68$. Owing to the presence of the plane of symmetry it is difficult to determine the exact z-co-ordinates of the two peaks making up the apparent single peak A, and the above values of z_1 and z_2 are therefore only approximate. The elongation of the peaks A and B suggests that there are probably K–O or K–N peaks at about C (0·42, 0·08) and D (0·08, 0·33). The distinction between oxygen and nitrogen is of no importance until the final stages of the analysis are reached, and, assigning arbitrarily the co-ordinates x_3, z_3 to the atom concerned with the potassium in these maxima, we find $x_3 = 0{\cdot}33$ and $z_3 = 0{\cdot}29$. On account of the indefinite nature of the peaks these figures also are very approximate. So far we have the following co-ordinates :

FIG. 1.



K	$\frac{1}{4}$	0	0·68, etc.,
S	0·11	$\frac{1}{4}$	0·18, etc.,
N	0·33	$\frac{1}{4}$	0·29, etc.

Assuming that the sulphamate radical has a roughly tetrahedral form, with bond lengths of about 1½ A, we can readily calculate that the approximate co-ordinates of O_I are either (0·03, ¼, 0·36) or (0·13, ¼, 0·90). In the former case the K–O_I peaks would occur at *H* and *G* (Fig. 1), whereas if the latter figures are correct the corresponding peaks are at *E* and *F*. As the values of $P_{(x, \frac{1}{4}, z)}$ in both the positions *H* and *G* are nearly zero, the co-ordinates of O_I may be taken to be approximately

O_I 0·13 ¼ 0·90, etc.

Finally, by again using the assumption of a tetrahedral sulphamate group, we can deduce that for the oxygens in general positions the approximate co-ordinates are probably

O_{II} 0·03 0·09 0·25, etc.

Sufficient information was thus obtained to enable Bragg syntheses to be carried out. In order to get the greatest possible resolution of every atom, three-dimensional syntheses were used throughout. As it is exceedingly laborious and also unnecessary to effect the summation over the whole unit cell, only syntheses along certain lines and over certain planes were carried out; these were

(*a*) along the line (¼ 0 z) to obtain z_1;
(*b*) over the plane (x ¼ z) to determine x_2, z_2, x_3, z_3, x_4, and z_4;
(*c*) along the line ($x_5\,y\,z_5$), approximate values of x_5 and z_5 deduced previously being used, to obtain the parameter y_5;
(*d*) over the plane ($x\,y_5\,z$), the value of y_5 from (*c*) being used to determine x_5 and z_5.

By using the above approximate co-ordinates obtained directly or by inference from the Patterson synthesis, the structure amplitudes for all planes with spacings down to 1·5 A were calculated. There was quite good general agreement between the values of the experimental and calculated structure amplitudes, so the signs obtained by calculation were allotted to the experimental values for the purpose of the syntheses (*a*)—(*d*). Graphs and contour maps of the results were plotted in the usual way, and a new set of parameters deduced. From these the structure amplitudes were calculated anew for all planes with spacings down to 1·2 A, and the syntheses were repeated with the new terms and such changes of sign as were necessary in the old ones. This process of successive refinement was continued, all available terms being used after the second set of syntheses, until the co-ordinates derived from a set of syntheses gave structure amplitudes having in every case the same sign as those from the previous set. This occurred at the fifth set of syntheses, and as the successive results appeared to converge in an alternating series, the means of the co-ordinates from the fourth and the fifth syntheses were adopted as final. These co-ordinates are given in Table I; the maximum probable error in them is almost certainly less than 0·004, since in no case do they differ from the results of either the fourth or the fifth synthesis by more than this amount.

TABLE I.

	x.	*y.*	*z.*		*x.*	*y.*	*z.*
K	¼	0	0·663	O_I	0·157	¼	0·937
S..................	0·137	¼	0·180	O_{II}	0·058	0·108	0·263
N	0·312	¼	0·285				

Fig. 2 is a composite representation of the results of the fifth synthesis, showing the section at $y = \frac{1}{4}$ with (inset) the section at $y = 0{\cdot}108$ through the oxygen atom in the general position, and with the potassium atoms indicated by circles. The contours are drawn at intervals of one electron per A^3, the zero and the first contour being omitted. Figs. 3 and 4 show the structure projected on (010) and (001) respectively, and a general indication of the agreement between observed and calculated structure amplitudes is given in Fig. 5.

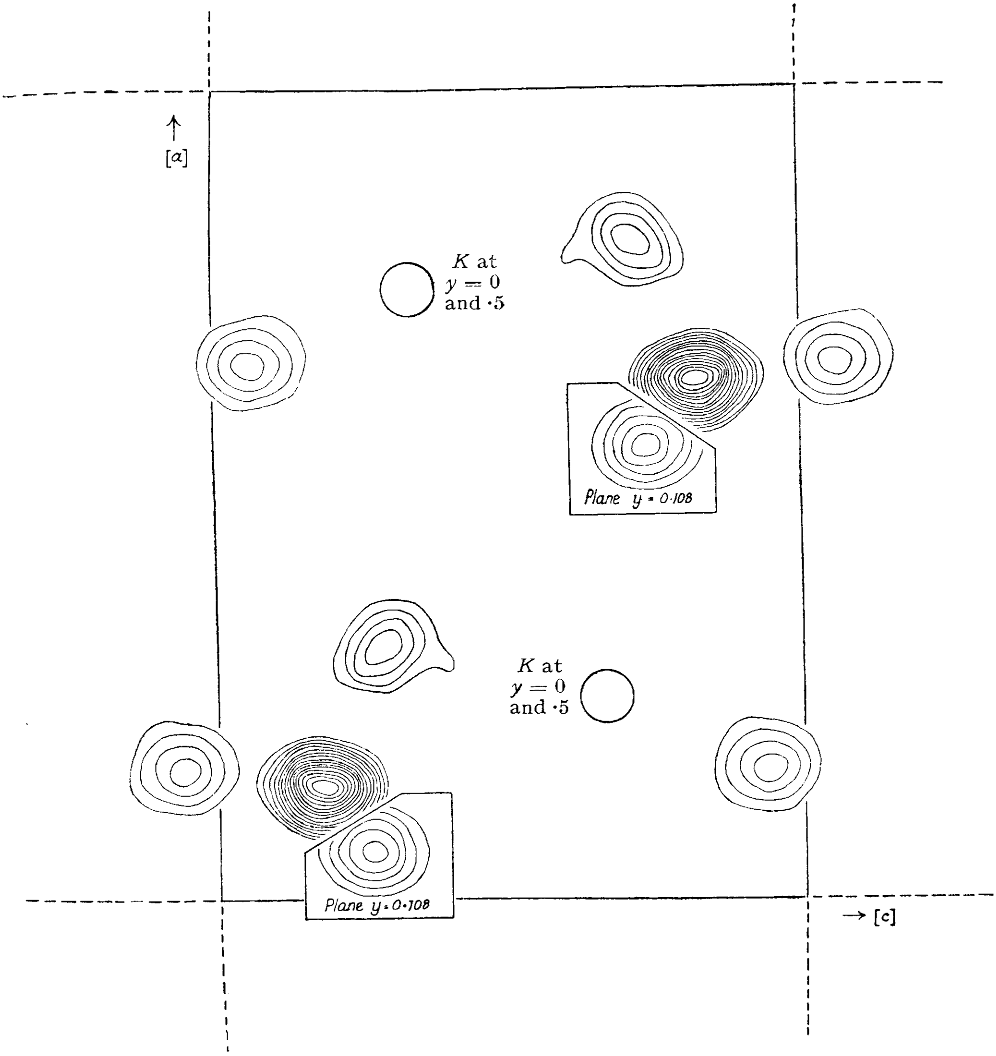
The preceding analysis yields the interatomic distances in potassium sulphamate with an accuracy of about 0·03 A. The dimensions of the sulphamate ion, which diverges very little from the exact tetrahedral form, are given in Table II. It will be observed that, although the nitrogen atom could not be distinguished from the oxygens by its electron

TABLE II.

Distances, A.				Angles.			
S–N	1·57	N–O_{II}	2·42	O_ISN	107°	O_{II}SN	106°
S–O_I	1·44	O_I–O_{II}	2·39	O_ISO_{II}	113½°	O_{II}SO_{II}	109½°
S–O_{II}	1·44	O_{II}–O_{II}	2·36				
N–O_I	2·38						

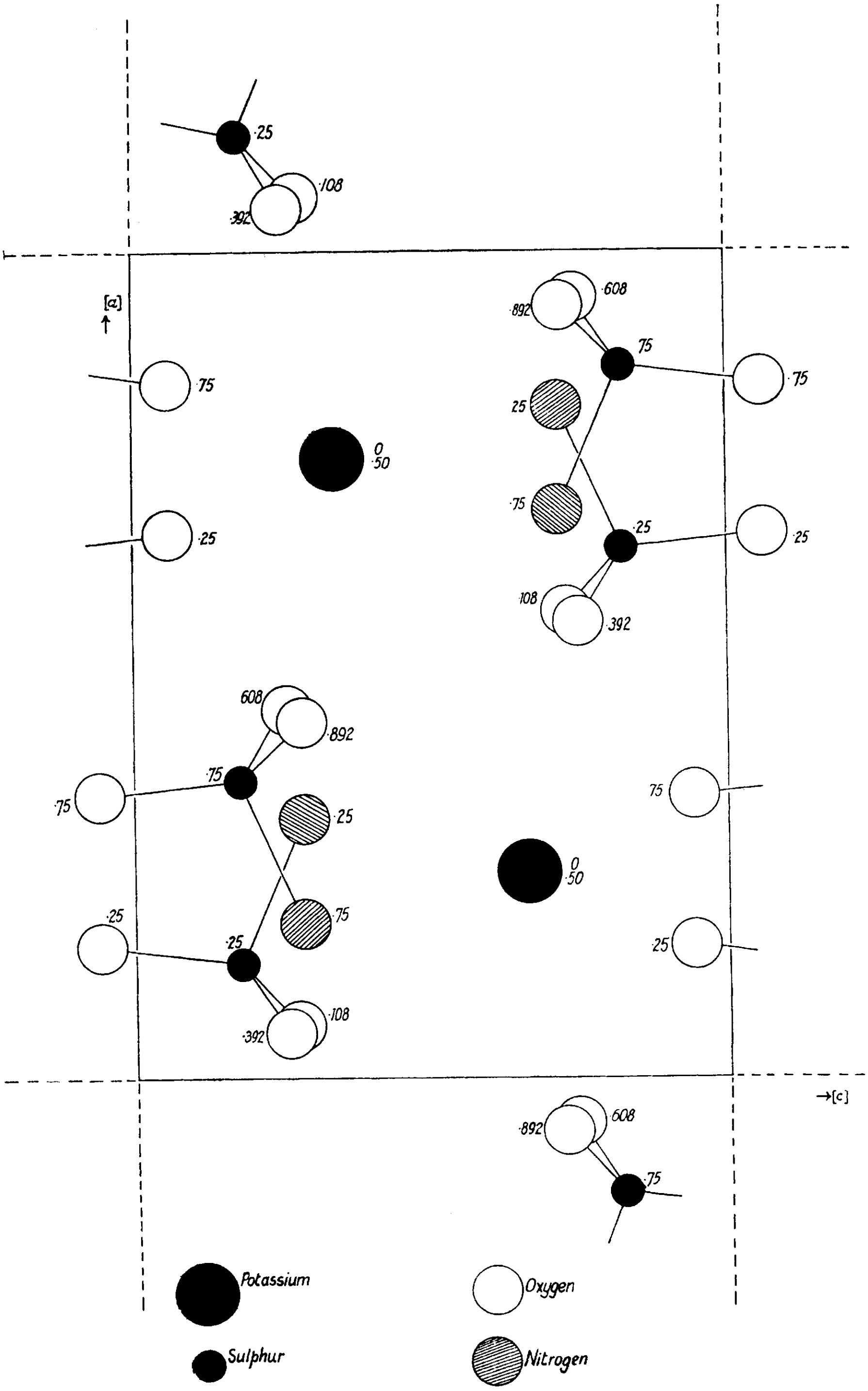
density (cf. Fig. 2), yet it is sharply differentiated by its greater distance from the sulphur atom; moreover, the equality of the S–O distances indicates that the two hydrogen atoms are attached to the nitrogen atom. The length of the S–O bond, 1·44 A, is slightly less than the mean, 1·48 A, of the distances found in potassium pyrosulphite (Zachariasen, *loc. cit.*) and sodium sulphate (Zachariasen and Ziegler, *loc. cit.*), and although this difference may be covered by the possible experimental errors, it seems probable that it is real, and due to the fact that the sulphamate ion carries only a single negative charge as compared with the double charge on the sulphate and pyrosulphite ions.

FIG. 2.



No previous data are available for the length of the S–N bond, but from the known

FIG. 3.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·108
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→[c]
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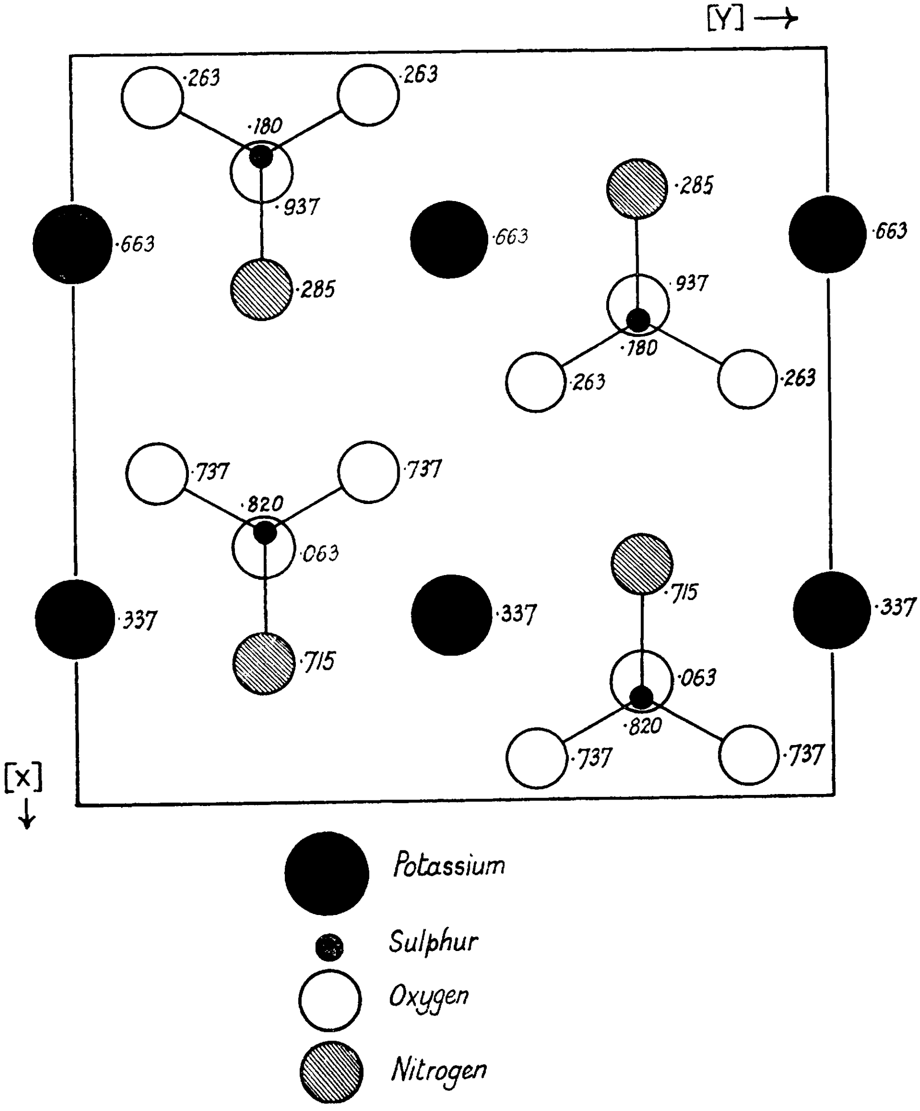
608
·75
Potassium
Oxygen
Sulphur
Nitrogen

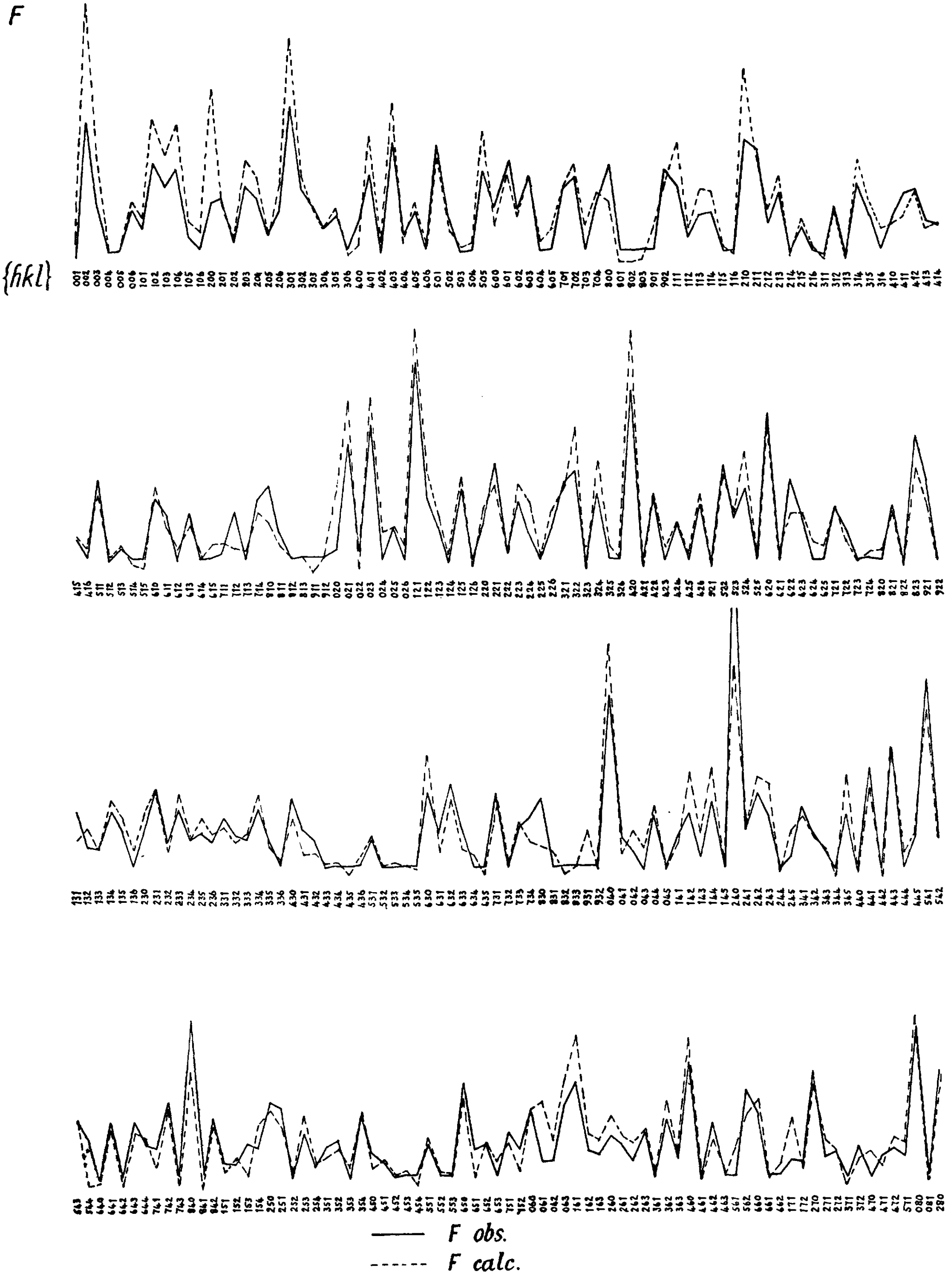
atomic radii it may be inferred that in an ion of this kind the bond from sulphur to a nitrogen atom (as distinct from an amino-group) would be about 1·54 A, and the fact that the value (1·57 A) now obtained experimentally is greater than this affords further evidence that the hydrogen atoms are attached to the nitrogen atom, since this would presumably increase the S–N distance slightly.

FIG. 4.



In spite of the similarity of the ions concerned, and some similarity also of cell dimensions, the structure of potassium sulphamate as a whole bears no appreciable resemblance to that of potassium perchlorate. This difference, however, is not obviously due to any disturbing influence of the amino-groups, since the structure is a simple ionic one, in which the closest distance of approach between nitrogen and oxygen atoms of adjacent ions (3·15 A) is not small enough to suggest a hydrogen bridge of any appreciable strength, and indeed is slightly greater than the minimum O • • • O distance (3·08 A) found in sodium sulphate, although less than the O • • • O distances in potassium sulphamate itself. Some diminution

FIG. 5.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F obs.
F calc.

of the $NH_2\cdots O$ distance as compared with the $O\cdots O$ distances might in any case be expected on account of the more positive character of the amino-group.

The potassium ion is surrounded by six oxygen and two nitrogen atoms, and the greater distance of the latter again indicates that the hydrogen atoms are attached to the nitrogen. The principal inter-ionic distances (in A) are given below :

$K-O_I$	2·75	$K-O_{II}$	2·96	$N-O_{II}$	3·15	O_I-O_{II}	3·65
$K-O_{II}$	2·75	K–N	3·10	$O_{II}-O_{II}$	3·45	N–N	4·29

EXPERIMENTAL.

Potassium sulphamate was prepared by neutralising sulphamic acid with potassium hydroxide; large crystals were obtained by slow evaporation of the aqueous solution. The crystals are orthorhombic combinations of *c*{001}, *q*{021}, *r*{101}, and *o*{111} tabular on {001}. The pyramid faces are usually small; on one crystal *a*{100} was observed. The crystals described by Fock (Groth, "Chem. Kryst.," **2**, 719) were similar except that {111} was more prominent, and {101} was not observed.

Rotation and oscillation photographs about the principal axes were taken with Cu-*K*α and Mo-*K*α radiation. The observed density (by flotation) was 2·23 g./cm.³ (calc. for 4 molecules of $NH_2{\cdot}SO_3K$, 2·22). The axial ratios are $a:b:c = 0{\cdot}996:1:0{\cdot}711$ (cf. $0{\cdot}994:1:0{\cdot}710$; Fock, *loc. cit.*).

The reflection intensities of as many planes as possible (309) were measured on an arbitrary scale from oscillation photographs, and were afterwards put on the absolute scale by making direct comparisons of the intensities of 28 selected planes with the (400) rocksalt reflection.

TABLE III.

l *hk*.	0.	1.	2.	3.	4.	5.	6.	*l* *hk*.	0.	1.	2.	3.	4.	5.
00	+272	+ 3	−45	+32	+ 3	− 9	+31	63	− 31	−17	+34	+18	− 5	− 5
10		−13	−35	+27	+33	− 9	+ 5	73		+31	+ 5	−19	−22	
20	− 20	+21	+ 7	−27	+23	+10	−18	83	+ 29	+ 5	+ 5	− 5		
30		−56	+26	+24	−12	+16	− 5	93		+ 5	+ 5			
40	+ 8	−46	− 4	+37	−11	−18	+ 5	04	+ 66	+15	−10	+ 4	−23	+ 5
50		+42	−16	+ 4	− 5	−34		14		+16	−24	+ 8	+28	− 5
60	− 14	+31	+19	−30	+ 5	+ 5		24	−122	+18	+31	−23	+ 4	+ 8
70		−26	+30	+ 5	−21			34		−26	+17	−10	+ 4	+23
80	+ 23	5	5	− 5				44	− 4	−40	+ 4	+47	− 5	−16
90		+ 5	−33					54		+72	−15	−24	17	
11		−27	− 9	+17	+18	+ 4	− 5	64	+ 4	+24	+ 5	−24	+16	
21	− 44	−41	+14	+25	+ 4	−12	− 5	74		−15	+31	− 5		
31		− 3	+19	− 4	−28	−17	+ 5	84	+ 61	5	−25			
41	− 16	+24	+26	−15	−13	+11	+ 5	15		− 9	− 9	+16	+15	
51		+33	+ 4	− 8	− 5	+ 5		25	− 31	−29	+ 4	+19	+ 7	
61	+ 26	+20	− 4	−21	− 5	+ 5		35		+15	+17	− 4	−26	
71		−14	−21	+ 5	+25			45	+ 13	+ 9	+ 4	− 5	− 5	
81	− 30	−14	+ 5	+ 5				55		+16	+ 5	− 5		
91		+ 5	− 5					65	+ 38	+14	−16	− 5		
02	− 8	−45	− 7	+52	− 4	−15	+ 5	75		−20	−14			
12		+75	−26	−16	+ 4	−29	+ 5	06	− 29	−31	+10	+30		
22	− 17	+39	+ 7	−25	+14	+ 5	−16	16		+38	−14	−12		
32		−32	+36	− 4	−28	+ 5	+ 5	26	− 19	+16	+10	−20		
42	+ 65	+ 3	−26	+ 4	−17	+ 5	+24	36		− 4	+25	−11		
52		+ 4	−38	+20	+30	− 5		46	+ 45	+ 4	−23	+ 5		
62	− 57	+ 4	+33	−20	+ 5	+ 5		56		+ 5	−35			
72		−23	+15	− 5	− 5			66	− 26	+ 5	+ 5			
82	+ 5	−24	− 5	+49				17		+25	+ 9			
92		+33	− 5					27	+ 42	+11	−13			
13		−24	+12	+11	−24	−17	+ 5	37		+ 4	−15			
23	+ 17	+32	+13	−25	−14	+16	+13	47	+ 5	−11	−14			
33		−22	−16	+14	+25	+13	− 5	57		−16				
43	− 29	−17	+14	+ 4	− 5	− 5	+ 5	08	+ 58	+ 5				
53		−14	− 4	+ 4	+ 5	+ 5		28	− 42					

Relative Intensity Measurements.—A small crystal, as equally developed as possible, was used for a series of *b*-axis oscillation photographs, taken with Mo-*K*α radiation in order to minimise errors due to absorption. All {*h*0*l*} intensities were measured by means of a Cambridge microphotometer, and the {*hkl*} intensities were estimated by eye, the {*h*0*l*} intensities being used as standards. The results were corrected when necessary for oblique incidence of the *X*-rays on the film, and for the variation in angular velocity of the reflecting planes. A few planes were not measured on the molybdenum photographs and were obtained from those taken with Cu radiation.

Absolute Intensity Measurements.—A large crystal was used, and spectra obtained by reflection from the naturally occurring faces and from a surface ground parallel to (010). Each photograph had recorded on it, in addition to the sulphamate reflections, a (400) reflection from a ground cleavage slip of rocksalt, and direct photometric comparisons of the reflections were made. To reduce errors of photometry the conditions were arranged so that the photographic densities to be compared were as nearly as possible equal. The intensities measured were {200} (four orders), {001} (six orders), {020} (four orders), {101} (five orders), (401), (501), (601), (102), (103), (104), (403), (603), and (111). In some cases the reflecting planes were not parallel to the surface of the crystal, and the usual formula $Q = 2\mu\rho$, giving the integrated reflection per unit volume Q in terms of the linear absorption coefficient μ and the integrated reflection from an extended face ρ, had to be modified. The equation is readily shown to be

$$Q = \mu\rho\,[\operatorname{cosec}(\theta + \phi) + \operatorname{cosec}(\theta - \phi)]/\operatorname{cosec}(\theta + \phi)$$

where ϕ is the angle the reflecting plane makes with the surface of the crystal, and θ its glancing angle. The values of Q obtained from these absolute measurements were used to put all the intensities on the absolute scale. The structure amplitudes F were calculated by means of the usual formula for a mosaic crystal; they are listed in Table III with the signs used in the final synthesis.

Sulphamic Acid (with F. J. LLEWELLYN).—The crystals examined were similar in habit to those described by Fock (Groth, *op. cit.*, **1**, 129). The dimensions of the unit cell, which contains 8 molecules of SO_3,NH_3, are $a = 8{\cdot}06$, $b = 8{\cdot}05$, $c = 9{\cdot}22$ A, whence $a : b : c = 1{\cdot}001 : 1 : 1{\cdot}146$ (cf. 0·995 : 1 : 1·149, Fock, *loc. cit.*). Density observed by flotation, 2·12 g./cm.^{3} (Calc., 2·16). The missing reflections are $\{hk0\}$ for k odd, $\{h0l\}$ for h odd, and $\{0kl\}$ for l odd, so that the space-group is $Pcab$ (D_{2h}^{15}).

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