thetic hydroxyaspartic acid. On the basis both of chromatographic behaviour and reduction products it can be regarded as proved that the amino acid studied is hydroxyaspartic acid.

The spot which travelled slower in butanol-acetic acid (R_F ~0.04) gave on reduction with HI and phosphorus glutamic acid and in addition glycine, serine, and alanine. It may be dihydroxyglutamic acid, but this cannot be regarded as proved.

Because there was no proof of the homogeneity of the isolated bound hydroxylamine fraction, it was subjected to paper electrophoresis (pyridine-acetic acid buffer, 1 000 V, 10 mA, pH 6.5, 4 h). Upon this bound hydroxylamine divided into two fractions, one of which behaved neutral and the other slightly basic (Fig. 2). On hydrolysis the neutral fraction gave hydroxyaspartic acid, glutamic acid, and alanine (in addition traces of valine and leucine), the basic fraction again the other unknown amino acids and besides the same amino acids as the neutral one. From the results it can be concluded that there are two hydroxylamine compounds, one giving hydroxyaspartic acid on hydrolysis, the other a still slower in butanol-acetic acid moving acidic amino acid (*cf.* above). If the components separated by electrophoresis are homogeneous they may be polypeptides, in which hydroxylamine is perhaps bound to one amino acid. The small amount of hydroxylamine formed on hydrolysis of these compounds — only about 1.5 % of the total N — arouses, however, the suspicion that the electrophoretically separated fractions may contain peptides as foreign substances. If this is the case nothing can be said about the chemical nature of the hydroxylamine compounds. Attention should, however, be called to the fact that hydroxyaspartic acid and another not fully characterized acid amino acid apparently belong to bound hydroxylamine in some way, since in *Azotobacter* cultures in which bound hydroxylamine was not formed, these amino acids were not found either.

A further elucidation of the problem demands more material and, accordingly, cultivation of *Azotobacter* on a larger scale. In any case it was proved that structurally complicated hydroxylamine compounds are accumulated in the culture solution of *Azotobacter;* that these compounds can be separated from amino acids by chromatography, and from each other by electrophoresis; that hydroxyaspartic acid is formed from one of these compounds on total hydrolysis and another not fully characterized amino acid from the other. In addition to these glutamic acid and alanine were formed from both.

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X-Ray Crystallographic Data on Miscellaneous Sulphur Compounds

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The unit cells and space groups of the following derivatives of divalent sulphur, selenium and tellurium were determined some years ago in the course of crystal structure work on analogous compounds, and have not been published before. The data were derived from oscillation and Weissenberg photographs on single-crystal specimens, using iron radiation, $\lambda(\mathrm{Fe}K\alpha) = 1.934$ Å, and in one case (tellurium dibenzenethiosulphonate) copper radiation, $\lambda(\mathrm{Cu}K\alpha) = 1.542$ Å.

Tellurium dimethylxanthate [1], $\mathrm{Te(S_2C\text{-}OCH_3)_2}$. Monoclinic prismatic, $a = 4.24$ Å, $b = 14.18$ Å, $c = 17.28$ Å, $\beta = 93°$. There are four molecules per unit cell; density, calc. 2.19, found 2.18 g/cm³. The space group, from systematic absences, is $C_{2h}^5 - P2_1/c$.

The crystals occur as brownish red prisms, extended along [100] and with {001} dominant. The repeat distance along the a axis, 4.24 Å, is shorter than twice the value, 2.2 Å, listed by Pauling [2] for the van der Waals radius of tellurium, but is not in discord with Briegleb's value [3], 1.9 Å, or with the non-bonded Te-Te approaches of 4.18 Å and 4.28 Å, respectively, found in *p,p'*-dichlorodiphenyl ditelluride [4] and monoclinic barium telluropentathionate dihydrate [5].

The crystals liberate tellurium more rapidly than do those of the ethyl compound described below, perhaps because of the close Te-Te contact.

Tellurium diethylxanthate [1], $\mathrm{Te(S_2C\text{-}OC_2H_5)_2}$. Monoclinic prismatic, $a = 9.35$ Å, $b = 6.17$ Å, $c = 21.21$ Å, $\beta = 91°$. Four

molecules per unit cell; density, calc. 2.01, found 2.03 g/cm³. Space group, from systematic absences, $C_{2h}^5-P2_1/c$.

The crystals were obtained as long, red prisms, bounded by {001} and {100}, with the latter occasionally dominant.

Tellurium dibenzenethiosulphonate, $Te(S_2O_2C_6H_5)_2$, *monoclinic dimorph.* $a = 13.93$ Å, $b = 7.71$ Å, $c = 15.72$ Å, $\beta = 96°$. Density, calculated for four molecules per unit cell, 1.88 g/cm³, as compared with 1.86 g/cm³ for the orthorhombic dimorph [6]. The space group, from systematic absences, is $C_{2h}^5-P2_1/c$.

The orthorhombic dimorph (I) of this compound, the crystal structure of which was described recently [7], was obtained on crystallization from chloroform [6]. The monoclinic dimorph (II) usually occurred when benzene was used as a crystallization medium instead of chloroform, and also when the compound was crystallized from dilute chloroform solutions on addition of ether. Occasionally, mixtures were obtained, small amounts of I separating along with II from benzene, and II together with I from chloroform. Chemical analysis of the X-ray sample of II gave 26.6 % Te, calc. 26.9 %. The dimorphs appear to be about equally stable, no transition between them being observed at room temperature or on heating to about 150°C.

Tetragonal crystals, containing 75 mole % of tellurium dibenzenethiosulphonate and 25 mole % of sulphur dibenzenethiosulphonate, have been observed. The latter compound, and also the selenium analogue, crystallize in a tetragonal space group [6,8].

Disulphur dibenzenethiosulphonate [9], $S_2(S_2O_2C_6H_5)_2$. Orthorhombic bipyramidal, $a = 11.36$ Å, $b = 12.90$ Å, $c = 22.47$ Å. Density, calculated for eight molecules per unit cell, 1.66 g/cm³. This figure may be compared with the X-ray densities, 1.58, 1.57 and 1.62 g/cm³, respectively, of the analogues containing one, two and three sulphur atoms less [8,10]. The space group, from systematic absences, is $D_{2h}^{15}-Pbca$.

This hexathionic compound crystallized as plates {001} with plate edges parallel to the *ab* diagonals.

Strontium selenopentathionate dihydrate, $SrSe(S_2O_3)_2 \cdot 2H_2O$. Orthorhombic bipyramidal, $a = 4.94$ Å, $b = 10.19$ Å, $c = 21.83$ Å. Density, calculated for four formula units per unit cell, 2.58 g/cm³. Systematic absences, $0kl$ when $k + l$ is odd, $hk0$ when h is odd. These data indicate that the salt is isomorphous with orthorhombic barium pentathionate [11] and selenopentathionate [12] dihydrates, the space group of which is $D_{2h}^{16}-Pnma$. The crystals have the same morphology as the barium salts, and show the same perfect cleavage along the *c* plane.

The crystals were obtained by recrystallization from aqueous methanol of a crude sample prepared from the sodium salt [13] by metathesis with strontium perchlorate. No chemical analysis was made, but the X-ray data in connection with the known composition of the barium salts appear sufficient to establish the formula given above.

No further crystallographic work on the compounds is contemplated.

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The Nature of the Sulphur-Sulphur Bond in Thiosulphate and Thiosulphonate Ions

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Thiosulphate and thiosulphonate ions, $[S-SO_3]^{--}$ and $[S-SO_2R]^-$, are apparently (beside the polysulphide ions) the only relatively stable species with sulphur-sulphur bonds where one of the sulphur