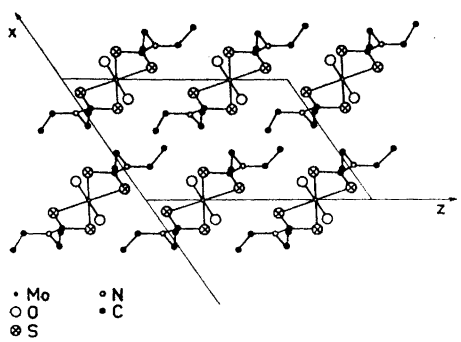

Fig. 1. The structure of $MoO_3((C_2H_5)_2NCS_2)_2$. Schematic drawing showing the *xz* projection.

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An Improved Preparation of Cellulose Layers for the Thin-layer Chromatography of Amino Acids

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Among the several thin-layer systems described for the resolution of amino acids, one of the most useful is that described by Jones and Heathcote,[1] in which they demonstrate the two-dimensional resolution of 23 naturally occurring amino acids on cellulose layers. Their procedure has the advantage that both developing solvents are easily prepared single-phase mixtures.

According to our results, using the system referred to above, the separation of leucine and isoleucine is disturbed or even prevented by uneven solvent flow in the second dimension. This is caused by an undulating, yellow-coloured band of impurities which forms along the solvent front in the first dimension. As the movement of leucine and isoleucine is almost as fast as the solvent front, it is difficult to isolate the disturbing band of impurities by breaking the layer between it and the compounds in question. This problem can be solved by making the cellulose layers according to the following procedure.

To a mixture of 50 ml water and 40 ml ethanol is added 15 g of cellulose powder (MN 300), and the slurry stirred vigorously for about 2 min with an electric stirrer designed to exclude air bubbles. The slurry is immediately spread on five 20×20 cm glass plates, previously cleaned with detergent solution, and well rinsed, to a layer of thickness 0.30 mm using Shandon equipment. After air drying overnight the plates were placed in a chromarack and washed by developing them in the first solvent-mixture until the solvent front almost reached the top of the plates. After air drying for about 24 h the plates were ready for use. Treatment of the plates with steam after washing, for faster removal of the washing solvent-mixture, did not improve the separation in question. This method of preparation gives a very pure and extremely strong layer of cellulose which in dry condition is only about 0.05 mm thick.

Sample spots were applied in amounts of 1—10 µl from 0.05 % solutions in isopropanol to which had been added enough 1 N hydrochloric acid to dissolve the compounds. After developing in the first dimension with isopropanol:formic acid:water (40:2:10), (about 4 h for a 12 cm length of run at room temperature), the plates were air dried overnight. The thin yellow line indicating the solvent front was isolated from the chromatogram by scoring the layer just behind it. Development in the second dimension was carried out for about 3.5 h using the system *tert.*-butanol:2-butanone:0.9 1ammonia: wa-

ter (50:30:14:6). In both cases the development was started immediately after the addition of solvent, and without tank saturation.

For the detection of the amino acids on the chromatogram a ninhydrin-collidine reagent was used. The reagent consisted of ninhydrin (0.15 g), acetic acid (10 ml) and 2,4,6-trimethyl-pyridine (1.25 ml), made up to a final volume of 50 ml with ethanol.[2] After spraying, the plates were placed on a hot plate (125°C) and the shape and colour of the spots marked with a pencil as they appeared. Not only was identification possible from the positions of the spots, but the process was also made easier by noting the speed of development, intensity and shade.

In addition to the 23 amino acids resolved by Jones and Heathcote, the system also provides a separation for norleucine, β-alanine, 4-aminobutyric acid, and 2-aminoglucose, some of which are present in complex humic hydrolysates. Norleucine (9.3, 5.9) is positioned close to, but definitely separated from, leucine (9.2, 5.4) and isoleucine (8.9, 4.8), where the parentheses give the R_F values for the first and second dimensions. β-Alanine (5.0, 1.1) and α-alanine (6.1, 1.3) separate well, but the latter may be partly overlapped by 4-aminobutyric acid (5.8, 1.2) which is positioned between them. Nevertheless all three can be recognized when present together in about equal amounts. 2-Aminoglucose (2.9, 3.3) is situated near serine (3.3, 1.8), but well separated from it.

According to the technique outlined above, we have analysed samples arising from different treatments of raw humus. The system works satisfactorily and also resolves several ninhydrin-positive spots which could not be assigned to any of the compounds mentioned above.

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Redetermined Crystal Structure of FeS_2 (Pyrite)

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The crystal structure of pyrite (FeS_2) was one of the very first to be determined by the method of X-ray diffraction. This early (1914) work by Bragg [1] and Ewald and Friedrich [2] depended on the use of single crystals, as are required by the Laue method. Suitable crystals were available in the form of the mineral pyrite. The only redetermination of this structure on the basis of an improved single crystal technique was reported in 1932 by Parker and Whitehouse,[3] whereas other investigators have employed various powder techniques on natural as well as synthetic samples.

Chemical intuition and elementary semi-theoretical considerations led Pauling and Huggins [4] (see also Pauling [5]) to suggest that the shortest (*i.e.* bonding) interatomic Fe–S and S–S distances in the pyrite and marcasite modifications of FeS_2, respectively, should be virtually equal. These expectations were found [4] to be confirmed with respect to the bonding Fe–S distances (which at that time were reported to be identical), whereas the short S–S pair distance appeared to differ considerably (2.09–2.14 Å *versus* 2.25 Å, respectively) between the two modifications. According to their empirical scheme of tetrahedral radii, Pauling and Huggins [4] furthermore deduced a predicted S–S distance of 2.08 Å, which together with the above evidence led them to propose that the parameter values for marcasite reported by Buerger [6] were probably inaccurate. Provoked by this criticism,[4,7] Buerger [8] repeated the determination of the marcasite structure and confirmed his original [6] parameter values with improved accuracy. However, the question concerning the distinction between the S–S pair distance in pyrite and marcasite remained unresolved, since the positional parameter of the pyrite structure might equally well be inaccurate. This view is in fact supported by the scattered values reported over the years for the S–S pair distance in pyrite (ranging from 2.09 to 2.17 Å, the latter value by Elliott [9] being the most