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PREPARATION, PROPERTIES AND STRUCTURE OF DI-(ACETAMIDINIUM)-DIFLUOROPENTATHIODIPHOSPHATE

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Plate-shaped, colorless crystals were grown from a solution, originating with the reaction of P_4S_{10} and NH_4F in CH_3CN. By a molar ratio of $4NH_4F/P_4S_{10}$, third of P_4S_{10} reacted to Di-(acetamidinium)-difluoropentathiodiphosphate, $[CH_3C(NH_2)_2]_2P_2S_5F_2$. Crystals are melting in the range of 134 - 142 °C under their decomposition. The crystal structure has been determined from three-dimensional MoK_α data, collected on a CAD-4 diffractometer. It crystallizes in the space group C2/c with four formula units in a cell of dimensions a = 10,600(5) b = 10,069(5) c = 15,129(8) Å β = 92,35(5)° with V = 1613,4 Å^3, D_c = 1,557 g.cm^{-3}, D_o = 1,49 g.cm^{-3}. The structure was solved by the direct methods program system MULTAN 82 and anisotropically refined by least-squares techniques to a final R = 0,037 based on 966 independent intensities. The hydrogen atoms were located from a difference map. The difluoropentathiodiphosphate-anion consists of two PS_3F-tetrahedra which are connected together by a common sulfur atom. This bridge-atom occupies a special equivalent position on a twofold rotation axis, therefore the anion shows the point symmetry 2. Bond distances and angles within the tetrahedra have the usual values (P-S_{bridge} = 2,075(1) Å, P-$S_{terminal}$ = 1,951(1) and 1,944(1) Å, P-F = 1,577(2) Å, ∢ P-S-P = 107,8(1)°. The nitrogen- carbon-atoms of the acetamidinium-cation are lying in plane. Bonds lengths are: C-C = 1,485(5) Å, C-N = 1,301(4) and 1,280(5) Å. There are hydrogen bonds in the structure.