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Pyridinium dihydrogenmonothiophosphate

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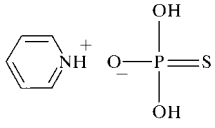
By reaction of pyridine dithiomonometaphosphoryl fluoride with ethyldiphenylphosphine in the presence of traces of water, we isolated crystals of the title compound, (I). In the structure of this salt, two kinds of hydrogen bonds were detected. They link N—H (part of the cation), as well as one O—H group (part of the anion), to the phosphoryl O atom of the anion. The donor–acceptor distances are 2.654 (1) and 2.568 (1) Å, respectively. The P—O and P—S bond lengths are 1.514 (2) and 1.967 (1) Å, respectively, which fall therefore in the range of a 1.5-fold bond.



(I)

Experimental

According to Fluck *et al.* (1973), a solution of ethyldiphenylphosphine in benzene was slowly dropped into a suspension of pyridine dithiomonometaphosphoryl fluoride in benzene with stirring. After 1 h, the solid product was filtered and recrystallized from a solution in benzene/acetonitrile.

Crystal data

$C_5H_6N^+\cdot H_2PSO_3^-$
M_r = 193.15
Monoclinic, $P2_1/n$
a = 6.7837 (13) Å
b = 9.0268 (11) Å
c = 13.237 (2) Å
β = 101.02 (2)°
V = 795.7 (2) Å^3
Z = 4
D_x = 1.612 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1775 reflections
θ = 2.5–25.0°
μ = 0.564 mm^{-1}
T = 180 (2) K
Plate, yellow
0.40 × 0.28 × 0.12 mm

Data collection

Stoe IPDS diffractometer
φ-oscill., φ-incr. = 1.5°, 133 exposure scans
Absorption correction: see below; T_{min} = 0.806, T_{max} = 0.935
4980 measured reflections
1540 independent reflections
1260 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 26.04°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 10$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.077
S = 1.041
1538 reflections
126 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

For the studied crystal, a ΔF^2-based absorption correction was carried out. The calculation made with *ABSCOR* (Stoe & Cie, 1997), a modification of *DIFABS* (Walker & Stuart, 1983). Unlike *DIFABS, ABSCOR* works with F^2 values instead of F. All H atoms were refined [C—H 0.89 (2)–0.91 (2) Å].

Data collection: *IPDS*-2.87 (Stoe & Cie, 1997); cell refinement: *IPDS*-2.87 (Stoe & Cie, 1997); data reduction: *IPDS*-2.87 (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XSTEP*-2.18 (Stoe & Cie, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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