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Burkhard Ziemer* and Annegrit Rabis

Institut für Chemie, Humboldt-Universität zu Berlin, Hessische Strasse 1-2, 10115 Berlin, Germany
Correspondence e-mail: burkhard=ziemer@chemie.hu-berlin.de

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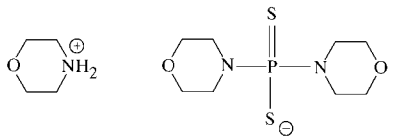
It was planned to sythesize morpholinedithiomonometaphosphoryl morpholinide by reaction of pyridinedithiomonometaphosphoryl chloride with 4-(trimethylsilyl)morpholine. But due to traces of water the title compound was formed as by-product, (I).



(I)

Experimental

A solution of 4-(trimethylsilyl)morpholine in benzene was slowly dropped to a suspension of pyridinedithiomonometaphosphoryl chloride in benzene. After one hour stirring at 333 K, the solid product was filtrated and washed with benzene. For the studied crystal of a ΔF^2 based absorption correction was carried out. The calculation was performed with *ABSCOR* (Stoe & Cie, 1997), a modification of *DIFABS* (Walker & Stuart, 1983). In contrary of *DIFABS*, *ABSCOR* loads F^2 values instead of F values.

Crystal data

$C_4H_{10}NO^+\cdot C_8H_{16}N_2O_2PS_2^-$	$Z = 2$
$M_r = 355.45$	$D_x = 1.389$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.355$ (2) Å	Cell parameters from 4803 reflections
$b = 8.291$ (3) Å	$\theta = 2.6$–$25.0°$
$c = 14.374$ (5) Å	$\mu = 0.420$ mm^{-1}
$\alpha = 95.78$ (4)°	$T = 180$ (2) K
$\beta = 98.68$ (4)°	Prism, colorless
$\gamma = 98.51$ (4)°	0.41 × 0.20 × 0.19 mm
$V = 850.1$ (5) Å^3	

Data collection

Stoe IPDS diffractometer	2881 independent reflections
φ-oscill., φ-incr. = 1.5°; 147 exposure scans	1833 reflections with >2sigma(I)
Absorption correction: refdelf see *Experimental*	$R_{int} = 0.1013$
$T_{min} = 0.847$, $T_{max} = 0.925$	$\theta_{max} = 25.24°$
6844 measured reflections	$h = -8 \rightarrow 8$
	$k = -10 \rightarrow 10$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$(\Delta/\sigma)_{max} = 0.012$
$wR(F^2) = 0.137$	$\Delta\rho_{max} = 0.536$ e Å^{-3}
$S = 1.040$	$\Delta\rho_{min} = -0.462$ e Å^{-3}
2876 reflections	Extinction correction: *SHELXL*93
197 parameters	Extinction coefficient: 0.0033 (29)
H atoms treated by a mixture of independent and constrained refinement	

Data collection: *IPDS*-2.75 (Stoe & Cie, 1997); cell refinement: *IPDS*-2.75 (Stoe & Cie, 1997); data reduction: *IPDS*-2.75 (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *XSTEP* (Stoe & Cie, 1997); software used to prepare material for publication: *SHELXL*93 (Sheldrick, 1993).

References

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