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Mechanism of sulfur transfer from 1,2,4dithiazolidine-3,5-diones to triphenylphosphines

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The mechanism of sulfurization of substituted triphenylphosphines with 4-(3- and 4-substituted)-1,2,4dithiazolidine-3,5-diones in acetonitrile, dichloromethane, tetrahydrofuran and toluene at 25 °C was studied. The reaction pathway involves rate-limiting initial nucleophilic attack of the phosphorus at sulfur followed by fast decomposition of the phosphonium intermediate to the corresponding phosphine sulfide, phenylisocyanate and carbonylsulfide. From the Hammett correlations and from the solvent dependency, it was concluded that the transition-state structure is very polar and resembles the zwitter-ionic intermediate. The extent of P–S bond formation and S–S bond cleavage is very similar in the solvents series, but the latter gradually decreases with the decreasing polarity of the solvent. Copyright © 2013 John Wiley & Sons, Ltd.

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INTRODUCTION

4-Substituted 1,2,4-dithiazolidine-3,5-diones are known from 1966 when Zumach and his co-workers prepared^[1,2] a modest series of compounds containing this skeleton. Ten years later, Barany and Merrifield found the 1,2,4-dithiazolidine-3,5-dione ring to be a very efficient orthogonal amino protecting group^[3] and since that time it has been extensively used in the synthesis of peptides,^[3–5] aminosugars in glycopeptides^[6–10] and peptide nucleic acids.^[11] More recently 1,2,4-dithiazolidine-3,5-diones were recognized as a nucleophilic isocyanate building block^[12–14] and used in the Mitsunobu reaction^[15,16] or in the synthesis of linear polyurethanes^[17] and sulfonyl ureas.^[18] The formation of an isocyanate from 1,2,4-dithiazolidine-3,5-dione ring most frequently involves sulfur transfer from the disulfide bridge to triphenylphosphine^[12,13,15–18] with simultaneous elimination of carbonylsulfide. However, not only triphenylphosphine has to be used for this purpose. Other P(III) compounds can also trigger the reaction. For example, the sulfurization of phosphites with the parent 1,2,4-dithiazolidine-3,5-dione was used for the efficient synthesis of phosphorothioate-containing oligodeoxyribonucleotides.^[19] The 1,2,4-dithiazolidine-3,5-diones can therefore be considered as a suitable sulfurization agent of various P(III) compounds. The sulfurization efficiency of 4-phenyl-1,2,4-dithiazolidine-3,5-dione towards various P(III) compounds was recently^[20] evaluated and compared with other known sulfurization agents, but the detailed mechanistic study of this reaction has not been published until now. Therefore, we decided to explore the kinetics and detailed mechanism for the reaction of 4-(3- and 4-subst. phenyl)-1,2,4-dithiazolidine-3,5-diones (1a-g) with triphenylphosphines (Scheme 1).

RESULTS AND DISCUSSION

Sulfurization reactions of triphenylphosphines with 4-(3- and 4-subst. phenyl)-1,2,4-dithiazolidine-3,5-diones (**1a-g**) were

studied in acetonitrile (ACN), dichloromethane (DCM), tetrahydrofuran (THF) and toluene (TOL) at 25 °C. Using ³¹P NMR, we have found that the reaction of all 1,2,4-dithiazolidines 1a-g with triphenylphosphines in CDCl3 and CD3CN proceeds smoothly and the only products are the corresponding triphenylphosphinsulphides together with phenylisocyanates and carbonylsulfide which is consistent with previous^[12,13,15-18] results. Similar experiment with the least reactive triphenyl phosphite used in our previous study^[20] surprisingly showed that the parallel oxidation of trivalent phosphorus occurs and the product therefore contains a mixture of the desired triphenyl phosphorothioate $(\delta(CDCl_3) = 53.7 \text{ ppm})^{[21]}$ and triphenyl phosphate $(\delta(CDCl_3) = -17.3 \text{ ppm})^{[22]}$ in the ratios 1:3 - 1:5 depending on the water content (based on integration of peaks at 53.7 ppm and -17.3 ppm in ³¹P NMR spectrum). This result could not be caused only by dissolved oxygen in deuterated solvent or by the presence of oxygen in the air above the sample in the NMR tube during measurement because the triphenyl phosphite itself does not undergo oxidation under these conditions. In order to avoid complications with undesirable oxidation, we performed kinetic study with triphenylphosphines only. The position of the transition state

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Scheme 1. Reaction of 4-(3- and 4-subst. phenyl)-1,2,4-dithiazolidine-3,5-diones (1a-g) with P(III) compounds

on the reaction coordinate and the charge distribution in the transition state can be determined from studies of the effect of solvent polarity on reaction rates and from studies of the transmission of substituent effects. Therefore, the kinetics of the sulfurization reaction were determined by monitoring the decrease in concentration of **1a–e** spectrophotometrically at 336 nm in the above-mentioned solvents in the presence of excess triphenylphosphine. The absorbance decreased exponentially with time from which was obtained a pseudo-first order rate constant which varied linearly with the concentration of the substituted triphenylphosphine (as an example see Fig. 1 for ACN) to give the corresponding second-order rate constant (k in Table 1).

From the data in Table 1, it is obvious that the second-order rate constants show a significant dependence on solvent polarity which indicates development of substantial charges in the transition state. Such observations are completely different^[20] to what was observed in the case of structurally similar 1,2,4-dithiazole-5-ones and 5-thiones where the influence of solvent



Figure 1. Dependence of the observed rate constant (k_{obs}) on the triphenylphosphine concentration (c_{Ph3P}) for the reaction of **1a** (\bullet), **1b** (\blacksquare), **1c** (\blacktriangle), **1d** (\square , inset) and **1e** (\bigcirc , inset) in acetonitrile at 25 °C



Figure 2. Hammett plot for sulfurization of triphenylphosphine with **1a-e** in acetonitrile (•), dichloromethane (•), tetrahydrofuran (\odot) and toluene (\Box) at 25 °C

polarity on the rate of sulfurization was only negligible. From the data in Table 1, it is also possible to plot corresponding Hammett correlations (Fig. 2) in each solvent whose positive-slopes reflect the negative charge developing on the leaving sulfur in the transition state.

These Hammett ρ -values are only slightly dependent on a solvent polarity, but their absolute values ($\rho_{ACN} = 2.49 \pm 0.17$, $\rho_{DCM} = 2.33 \pm 0.11$, $\rho_{THF} = 2.16 \pm 0.11$, $\rho_{TOL} = 1.90 \pm 0.16$) are higher than those found for the reaction of related 3-(3- and 4-subst. phenyl)-1,2,4-dithiazole-5-ones and 5-thiones with triphenyl phosphite^[20] ($\rho = 1.1-1.6$) or triphenylphosphine ($\rho = 1.7$; for source data see Table 10 in Supporting Information). This confirms the proposal that the transition state structures existing on the reaction coordinate are more polarized. It appears that the polarization of S–S bond in the transition state is enhanced by the increasing polarity of the solvent as seen from an increase in ρ -values changing from the lowest value found in non-polar TOL to the highest value found in strongly polar ACN. In other words, the S–S bond cleavage in the transition TOL.

To get a better insight into the reaction mechanism, especially into the extent of P–S bond formation in the transition state, we also studied the transmission of polar substituent effects in reaction of 4-phenyl-1,2,4-dithiazolidine-3,5-dione (**1a**) with substituted triphenylphosphines. Again, it was possible to determine second-order rate constant (*k* in Table 2) from the slopes of linear dependences of k_{obs} vs. $c(4-X-Ph_3P)$ and plot these constants against the sum of Hammett σ_p -constants (Fig. 3).

Table 1. Second-order rate constants (*k*) for the reaction of 4-(3- and 4-subst. phenyl)-1,2,4-dithiazolidine-3,5-diones $(1a-e)^a$ with triphenylphosphine in ACN, DCM, THF and TOL at 25 °C

Compound	$k(ACN) (I \cdot mol^{-1} \cdot s^{-1})$	$k(\text{DCM}) (\mathbf{I} \cdot \text{mol}^{-1} \cdot \mathbf{s}^{-1})$	$k(\text{THF}) (I \cdot \text{mol}^{-1} \cdot \text{s}^{-1})$	$k(\text{TOL}) (I \cdot \text{mol}^{-1} \cdot \text{s}^{-1})$		
1a	33.9±0.6	$\textbf{20.2}\pm\textbf{0.1}$	3.62 ± 0.12	1.40 ± 0.02		
1b	11.0 ± 1.4	3.51 ± 0.06	$\textbf{0.828} \pm \textbf{0.018}$	0.31 ± 0.01		
1c	20.6 ± 0.2	6.30 ± 0.22	1.27 ± 0.03	0.44 ± 0.01		
1d	191.0 ± 2.8	48.9 ± 0.6	11.35 ± 0.14	3.16 ± 0.10		
1e	434.8 ± 33.9	120.0 ± 1.3	18.2 ± 0.2	4.72 ± 0.19		
^a Deartien with devivatives 1 r h was too fast even for storned flow reconvergences						

^aReaction with derivatives **1g,h** was too fast even for stopped-flow measurements.

phosphines in ACN, DCM, THF and TOL at 25 °C							
(4-X-Ph) ₃ P X	$k(ACN) (I \cdot mol^{-1} \cdot s^{-1})$	$k(\text{DCM}) (I \cdot \text{mol}^{-1} \cdot \text{s}^{-1})$	$k(\text{THF}) (\text{I} \cdot \text{mol}^{-1} \cdot \text{s}^{-1})$	$k(\text{TOL}) (I \cdot \text{mol}^{-1} \cdot \text{s}^{-1})$			
OCH3 CH3 H	$\begin{array}{c} 9488 \pm 389 \\ 1070 \pm 49 \\ 33.9 \pm 0.6 \end{array}$	$5601 \pm 207 \ 550 \pm 13 \ 20.2 \pm 0.1$	2367 ± 99 120 ± 2 3.62 ± 0.12	$699 \pm 18 \\ 46.7 \pm 1.6 \\ 1.40 \pm 0,02$			
Cl	1.20 ± 0.04	0.619 ± 0.025	$\textbf{0.338} \pm \textbf{0,008}$	0.0925 ± 0.0010			

Table 2. Second-order rate constants (k) for the reaction of 4-phenyl-1,2,4-dithiazolidine-3,5-dione (1a) with tris(4-subst.phenyl)





Figure 3. Hammett plot for sulfurization of 4-substituted triphenylphosphines with **1a** in ACN (\bullet), DCM (\bullet), THF (\bigcirc) and TOL (\Box) at 25 °C

From Fig. 3, it is clear that the slopes of Hammett correlations in individual solvents are virtually the same ($\rho_{ACN} = -2.66 \pm 0.17$, $\rho_{DCM} = -2.69 \pm 0.15$, $\rho_{THF} = -2.55 \pm 0.15$, $\rho_{TOL} = -2.63 \pm 0.27$) but their absolute values are again much higher than those obtained for reaction of related 3-(3- and 4-subst. phenyl)-1,2,4-dithiazole-5-ones and 5-thiones with triphenyl phosphites^[20] ($\rho = -1.07$ and -1.28) or triphenylphosphines ($\rho = -1.63$). Even smaller values were found for the reaction of 3-amino-1,2,4-dithiazole-5-thione and 3-dimethylamino-1,2,4-dithiazole-5-thione with triphenylphosphines ($\rho = -0.86$ and -0.92)²³. On the other hand, a high negative Hammett ρ -value^[24,25] was also found for the reaction of *m*- and *p*-substituted triphenylphosphines with elemental sulfur in benzene and TOL ($\rho = -2.5$) for which a highly polarized transition state was proposed.

The interpretation of the Hammett ρ -value in terms of absolute charge distribution in the transition state requires a reference reaction, ideally a corresponding ρ -value for an equilibrium reaction. Unfortunately, the only equilibrium studied for P(III) compounds concerns protonation of phosphines in nitromethane which gave^[26] Taft $\rho^* = -2.6$. The polarity of nitromethane in terms of relative permittivity ($\varepsilon_r = 38.97$) is virtually the same as for ACN ($\varepsilon_r = 37.50$) from which it can be concluded that the absolute charge on attacking phosphorus as well as on



Scheme 2. Structure of the zwitter-ionic intermediate T formed during sulfurization of triphenylphosphines with 4-(3- and 4-subst. phenyl)-1,2,4-dithiazolidine-3,5-diones (**1a–g**)

leaving sulfur is close to unit value (+1 and -1, respectively) and the transition structure closely resembles the structure of zwitter-ionic intermediate **T** in Scheme 2.

The great difference between the structures of these late transition states found here and the early transition states recently found^[20,23] for the related 1,2,4-dithiazole-5-one and 5-thione derivatives is probably caused by the poorer leaving ability of the sulfur. The slower S-S bond cleavage presumably occurs because there is some cross-conjugation^[27] between the leaving sulfur/nitrogen lone pairs and the carbonyl group (see Scheme 2). Such competition shifts the position of transition state on the reaction coordinate close to the highly polarized zwitter-ionic intermediate T together with substantial rate retardation (cf. value $k(\mathbf{1a}) = 33.9 \text{ I} \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$ from Tables 1 and 2 with value $k = 1.14 \cdot 10^3 \text{ I} \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$ found^[20] for the reaction of 3phenyl-1,2,4-dithiazole-3-thione with triphenylphosphine in ACN). Further evidence for efficient conjugation of the nitrogen lone pair with carbonyl group(s) comes from X-ray crystallographic analysis of compound 1f. The ORTEP diagram (Fig. 4) shows that the 1,2,4-dithiazolidine ring in 1f is essentially planar (torsion angles C1-S1-S2-C2 and S1-C1-N1-C2 are less than $0.54(7)^{\circ}$) and the plane of the benzene ring is strongly deviated from the plane of the parent heterocycle (dihedral angle is 63.4°. This observation supports the idea of efficient nitrogen lone pair delocalization with both carbonyl groups and no conjugation with benzene ring. Although a high degree of nitrogen lone pair conjugation is observed, the C-S and S-S bonds being attributed to be a single one similarly as in another four examples of 1,2,4-dithiazolidine-3,5-diones found in the literature.^[13,17,28,29] Typical values of all bond lengths were taken from Ref..^[30]



Figure 4. ORTEP view of compound **1f** (thermal ellipsoids at 40% probability level). Selected interatomic distances [Å]; angles and interplanar angle [°]: C1 S1 1.7715(15), S1 S2 2.0621(5), S2 C2 1.7608(13), C2 N1 1.382(2), N1 C1 1.3914(19), O1 C1 1.1994(19), O2 C2 1.206(2); C1 S1 S2 95.85(5), S1 S2 C2 95.76(6), S2 C2 N1 113.84(11), C2 N1 C1 121.50(12), N1 C1 S1 113.04(11) and interplanar angle of the phenyl ring versus the heterocyclic ring is 63.4

EXPERIMENTAL

Syntheses

All the 1,2,4-dithiazolidine-3,5-diones (**1a–g**) were synthesized and purified according to published general procedures^[31] in two steps from phenyl isothiocyanate (see Supporting Information).

Kinetic measurements

The kinetic measurements were carried out on a Diode Array Stopped-Flow SX.18 MV-R (Applied Photophysics) spectrophotometer or on a Hewlett Packard HP 8453 Diode Array spectrophotometer at 25 °C in 1 cm closable cells under pseudo-first order conditions (triphenylphosphines in large excess) in ACN, DCM, THF and TOL. The observed pseudo-first order rate constants k_{obs} were calculated from the measured time dependence of the absorbance at 336 nm with the help of an optimization program (See Tables 3-9 in Supporting Information). In all kinetic runs, the standard deviation in the fit was always less than 5% of the quoted value and was more usually between 0.2 and 0.4% of the quoted value. Solvents used for kinetic measurements were of HPLC quality and were dried and distilled under argon prior to use. All triphenylphosphines were from commercial sources and were used as received because the amount of corresponding oxidation product or another phosphoruscontaining compound was only negligible according to $^{\rm 31}{\rm P}$ NMR spectrum. Due to potential oxidation of triphenylphosphines, all the solutions were freshly prepared just before kinetic measurements.

NMR measurements

¹H, ¹³C and ³¹P NMR spectra were recorded on a Bruker Avance 3 400 MHz instrument. Chemical shifts (δ) are referenced to solvent residual peaks δ (DMSO-*d*₆) = 2.50 (¹H) and 39.6 ppm (¹³C), and δ (CDCl₃) = 7.27 (¹H) and 77.0 (¹³C).^[31] P NMR shifts are referenced to 85% phosphoric acid (external standard).

X-ray crystallography of compound 1f

The yellowish single crystals of **1f** suitable for X-ray determination were grown from hot diethyl ether and petroleum ether solution. The X-ray data for **1f** were obtained at 150 K using an Oxford Cryostream low-temperature device on a Nonius KappaCCD diffractometer with Mo K_x radiation ($\lambda = 0.71073$ Å), a graphite monochromator, and the Φ and χ scan modes. Data reductions were performed with the DENZO-SMN.^[32] The absorption was corrected by integration methods.^[33] Structures were solved by direct methods (Sir92)^[34] and refined by full matrix least-squares based on F^2 (SHELXL97).^[35] The hydrogen atoms were mostly localized on a difference Fourier map: however, to ensure uniformity of the treatment of the crystals, all hydrogen atoms were recalculated into idealized positions (riding model) and assigned temperature factors H_{iso}(H) = 1.2 U_{eq}(pivot atom) with C– H = 0.93 Å for the hydrogen atoms in the aromatic ring.

$$\begin{split} R_{\text{int}} &= \Sigma \mid F_o^2 - F_{o,\text{mean}}^2 \mid /\Sigma F_o^2, \\ \text{GOF} &= \left[\Sigma \Big(w(F_o^2 - F_c^2)^2 \Big) / \big(N_{\text{diffrs}} - N_{\text{params}} \big) \right]^{\$} \text{for all data}, \\ R(F) &= \Sigma \mid |F_o \mid - |F_c \mid | /\Sigma \mid F_o \mid \text{for observed data}, \\ wR(F^2) &= \left[\Sigma \Big(w(F_o^2 - F_c^2)^2 \Big) / \Big(\Sigma w(F_o^2)^2 \Big) \right]^{\$} \text{for all data}. \end{split}$$

Crystallographic data for structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 921142. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EY, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http:// www.ccdc.cam.ac.uk).

Compound 1f

 $\begin{array}{l} C_8H_4N_2O_4S_2, \ M=256.25, \ monoclinic, \ P \ 2_1, \ a=7.8220(3), \ b=7.8601(2), \\ c=7.9299(3) \ \text{\AA}, \ \beta=92.726(3) \ ^\circ, \ Z=2, \ V=486.99(3) \ \text{\AA}^3, \ D_c=1.748 \ g\cdot cm^{-3}, \\ \mu=0.545 \ mm^{-1}, \ T_{min}/T_{max}=0.887/0.932; -9\leq h\leq 10, \ -10\leq k\leq 8, \ -8\leq l\leq 10; \\ 3535 \ reflections \ measured \ (\Theta_{max}=27.5 \ ^\circ), \ 3515 \ independent \ (R_{int}=0.0242), \ 1875 \ with \ l>2\sigma(l), \ 145 \ parameters, \ S=1.090, \ R1(obs. data)=0.0194, \ wR2(all \ data)=0.0497; \ max., \ min. \ residual \ electron \ density=0.270, \ -0.191 \ e \ \text{\AA}^{-3}. \end{array}$

In the crystal packing of 1f (Fig. 4), no interesting interatomic interactions were observed except of non-classical H-bonding and short contacts between C-H \cdots O and N = O \cdots S-S atoms or moieties, respectively.

SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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