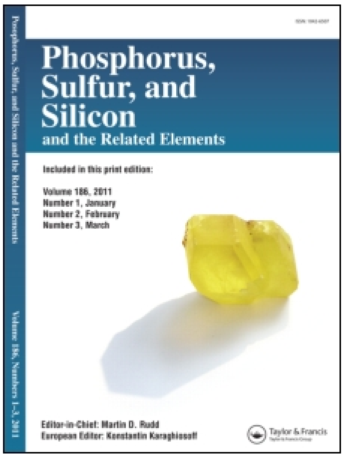

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ON THE SEARCH FOR THE REGIOSELECTIVE PHOSPHORYLATION OF 1,2,4-TRIAZINES BY CYCLIC, ACYCLIC PHOSPHITES AND TRIPHENYLPHOSPHINE

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ON THE SEARCH FOR THE REGIOSELECTIVE PHOSPHORYLATION OF 1,2,4-TRIAZINES BY CYCLIC, ACYCLIC PHOSPHITES AND TRIPHENYLPHOSPHINE

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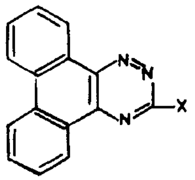
3-Triazine derivatives **1a** and **1b** react with phosphorus (III) reagents, to give regioselectively phosphorylated products at C-3 or N-2 depending on the nature of the substituent at C-3 and the phosphorus reagent used. 3-chlorotriazine **1a** reacts with cyclic phosphites **2a**, **b**, and acyclic phosphites **3a–c** to give the phosphonate products **6** and **10**, respectively. On the other hand, reaction of **1a** with **2c** leads to the formation of its 3-dimethylamino-derivative **8**. Reaction of **1a** with triphenylphosphine gave the phosphonium salt **11**. Solvolysis of **11** with aqueous methanol gave 3-hydroxytriazine **1b** and triphenylphosphine. Treatment of 3-hydroxy-triazine **1b** with 2-chloro-1,3,2-dioxaphospholane **2d** results in formation of phosphoramidite **12** at the N-2.

Key words: 1,2,4-Triazines, cyclic phosphites, trialkyl phosphites, triphenylphosphine.

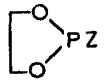
INTRODUCTION

Recently, a series of highly biologically active compounds derived from phosphorylated condensed heterocyclics containing nitrogen were described.[1–5] The arrangement of the nitrogen atoms, specifically, in the 1,4 position in the heterocyclic nucleus, results in excellent insecticides whose acaricidal activity is maintained against highly resistant strains.[5] Related to these groups are 1,2,4-triazine derivatives which provided well known pesticides.[4–6]

Several reports have appeared dealing with the study of reactions and synthesis of 3,5-disubstituted 1,2,4-triazines.[7–10] Thus, the preparation and study of new phosphorus derivatives from 3-chloro-, and 3-hydroxyphenanthro[9,10-e]1,2,4-triazines **1a** and **1b**, respectively, seemed a worthwhile endeavour.



1a, X=Cl
1b, X=OH



2a, Z = OCH_3
2b, Z = OC_2H_5
2c, Z = $N(CH_3)_2$
2d, Z = Cl

$P(OR)_3$

3a, R = CH_3
3b, R = C_2H_5
3c, R = C_3H_7-i

TPP

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RESULTS AND DISCUSSION

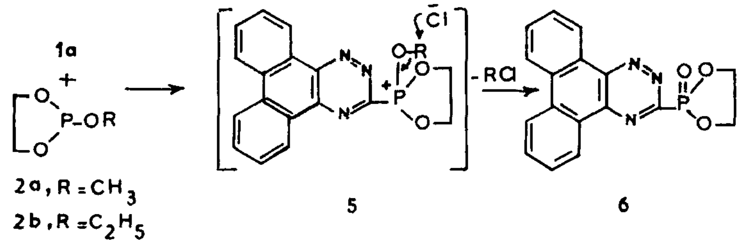
The reaction of tertiary phosphites with 3-chlorotriazine **1a** would be expected to parallel the reactions of phosphorus (III) esters with alkyl halides. In this case, according to Michaelis-Arbuzov mechanism, the first formed quaternary intermediate is a quasiphosphonium salt **4** which is either stable[11] or decomposes to other products (Equation 1).[11,12]

$$(RO)_3P + R'\text{-}X \longrightarrow X^- \underset{\mathbf{4}}{R\text{-}O\text{-}P^+(OR)_2R'} \longrightarrow \text{products} \quad (1)$$

Indeed, adducts from **1a** and the cyclic phosphites **2a**, **2b**, and **2c** or acyclic phosphites **3a–c** fall within this latter group and their mode of decomposition depends upon the type of phosphite ester used.

2-Methoxy-1,3,2-dioxaphospholane **2a** and 2-ethoxy-1,3,2-dioxaphospholane **2b** reacted with 3-chlorotriazine **1a** in refluxing benzene to give in each instance a yellow substance in ~60% yield and were proved to have a ring-retained phosphonate structure **6**, the normal Michaelis-Arbusov product (Scheme I). No intermediate was isolated. Methyl chloride or ethyl chloride, evolved during the reaction and were detected by ^{1}H NMR. The infrared spectrum of **6** exhibited strong absorption at 1258 cm^{-1} typical for P=O. The oxaphospholane ring remained intact since the ^{1}H NMR spectrum of **6** showed two sets of a doubled triplet (J_{PH} = 11.5 Hz) at 3.8 and 4.2 ppm. The ^{13}C NMR spectrum of **6** exhibited a doublet at 158.3 ppm (J_{CP} = 243 Hz) is assigned to a carbon bearing no hydrogens and a large coupling constant suggests direct bonding to phosphorus (N=C—P). It showed also two doublets (J_{COP} = 6.7 Hz) at 62.2 and 62.6 ppm which are due to methylene carbons.[13] Moreover, the ^{31}P NMR spectrum in CDCl$_3$ gave only one detectable peak at δ = +11.4 ppm which is in agreement with the assigned phosphonate structure.[14] Its mass spectrum showed a molecular ion at m/z = 337, calcd. 337.29.

When **1a** was treated with 2-dimethylamino-1,3,2-dioxaphospholane **2c**, (which is more basic than the respective trialkyl analogues), the reaction took a different course and led to the production of 3-dimethylamino-1,2,4-triazine **8**. Pertinent NMR and analytical data are presented in the experimental section. Moreover, compound **8** was unequivocally obtained by treatment **1a** with dimethylamine. Examination of the reaction mixture prior to work up by ^{31}P NMR spectroscopy indicated the presence of 2-chloro-1,3,2-dioxaphopholane **2d** (δ = 167.6 ppm). A

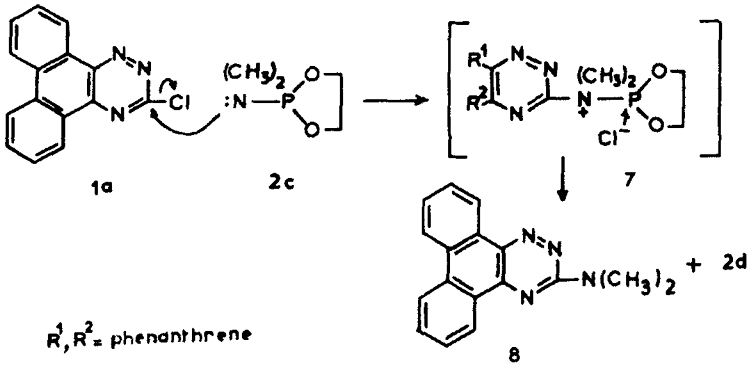


SCHEME I

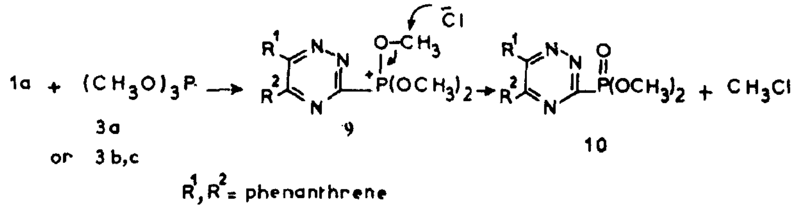
possible mechanism for the aforementioned C-amination process is depicted in Scheme II. This is based on direct attack by the nitrogen lone pair electrons of the anionic centre in **2c** with **1a** by an addition-elimination mechanism[15]; this would give an intermediate **7** which would suffer by attack of Cl^- at the P to produce **8** and **2d**.[16] This application of tertiary phosphorus reagent to induce C-amination, is analogous to their potentialities as alkylating agents for several functional groups.[17-20]

The reaction of 3-chlorophenanthro[9,10-e]1,2,4-triazine (**1a**) with acyclic phosphites was also studied. **1a** was found to react in the absence of solvent with excess trimethyl-, triethyl, and triisopropyl phosphites **3a–c** to give the phosphonates **10** in ~70% yield. The assignments of structure **10** for the products were based on the following data. Correct elemental analyses and molecular weight determinations by (MS) were obtained on all three products. The infrared spectra of **10** showed the presence of absorption bands around 1670, 1255, 1080, and 875 cm^{-1}. Those at 1670, 1255, and 1080 cm^{-1} were tentatively assigned to C=N, P=O, and P—O—C stretching vibrations, respectively. The peak at 875 cm^{-1} was probably due to the C—P frequency.[21] The ^1H NMR spectrum of **10b** showed signals at δ 1.52 (t, 6H, —C—CH_3, J_{HP} = 11.5 Hz); 4.15 (q, 4H, —CH_2, J_{HP} = 11.5 Hz). Moreover, compounds **10** gave ^{31}P NMR signals at δ = ~8.5 ppm *vs.* 85% H_3PO_4. Even though these values are at much lower magnetic fields than those frequently recorded for phosphonates, similar observations were previously reported on different occasions.[14,22]

Scheme III shows a mechanism for the reaction of **1a** with trialkyl phosphites **3a–c**, which is analogous to the corresponding reaction of cyclic phosphites **2a, b**. The first formed quaternary intermediate is a quasiphosphonium salt **9**. According



SCHEME II



SCHEME III

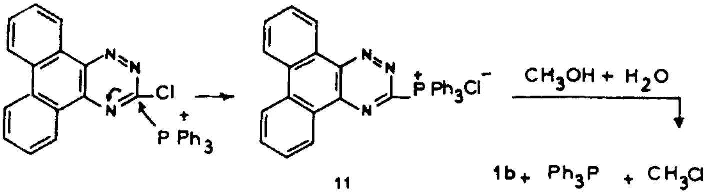
to Michaelis-Arbusove reaction, the high nucleophilic activity of the counter anion, such as chloride, causes rapid dealkylation, yielding the final products **10** and alkyl chloride. The later process is helped by the formation of the strong P=O bond.

The reaction of 3-chloro-1,2,4-triazine **1a** with triphenylphosphine (TPP) was sluggish in ether or toluene. However, the reaction was found to be completed after refluxing the reactants in xylene for 10 h to give rise to the usual quaternary phosphonium salt **11** by SN^2 attack. Treatment of the hygroscopic salt **11** with aqueous methanol yielded 3-hydroxy-1,2,4-triazine **1b**, triphenylphosphine, and methyl chloride (Scheme IV). Solvolysis products were isolated and identified (cf. Experimental). This result analogous to the common Michael nucleophilic displacement by phosphines at alkyl halides.[12]

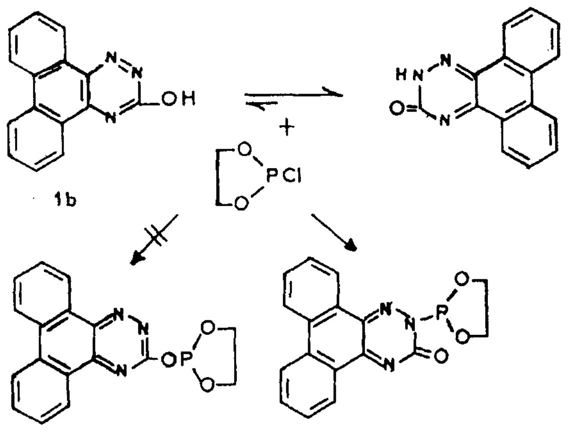
Extending our work on the potential phosphorylation of 1,2,4-triazines, the reaction of 3-hydroxyphenanthro[9,10-e]-1,2,4-triazine (**1b**) with 2-chloro-1,3,2-dioxaphospholane (**2d**) was investigated. When **1b** was allowed to react with **2d** in acetone solution and in the presence of anhydrous K_2CO_3, a yellow product **12** was obtained. Compound **12** was insoluble in 10% NaOH aq. and could be hydrolyzed to the parent hydroxytriazine **1b** upon heating with alcoholic KOH solution. Correct analytical values, molecular weight (MS) together with compatible IR, ^1H, ^{13}C, and ^{31}P NMR data were obtained for the product. The ^{31}P NMR shift recorded for **12** was δ 148.3 ppm, a value that lies in the range of ring-retained phosphoramidite shifts.[14] The IR spectrum revealed the presence of strong absorption band at 1675 cm^{-1} characteristic of a ring C=O group.[7] The presence of a C=O group in **12** also was confirmed by a signal at 196.6 pm in its ^{13}C NMR spectrum. Moreover, in the ^1H NMR spectrum of **12**, the ethylene protons of the dioxolane ring gave an AAMMX spin system. Thus, the protons of the two methylene groups, which are non-equivalent, appeared as two sets of a doubled triplet (J_{HP} = 10.5 Hz) at δ 3.75 and 4.32 ppm. Based upon these arguments, the alternative structure **13** can be excluded.

Formation of **12** instead of **13** can be explained in terms of the existence of **1b** in equilibrium with the tautomeric form **1c**. Thus it undergoes reactions characteristic of both forms.[7] Indeed, the behaviour of **1b** $\rightleftarrows$ **1c** toward halogenated phosphorus reagents is similar to its reaction with various alkylating agents which are known[7] to yield the corresponding N-alkyl derivatives. It is worthy of mention here that nucleophilic attack on nitrogen by chlorinated phosphorus reagents has been precedented in the literature.[18]

An inspection of these results clearly shows, firstly, that the substituents at the phosphorus (III) reagent may dramatically change the overall site of attack in the investigated reactions, and secondly, that both the cation and anion play an important role in this regard.



SCHEME IV



1b

13 **12**

SCHEME V

EXPERIMENTAL

All melting points are uncorercted. The IR spectra were run on a Perkin-Elmer Infracord Spectrometer Model 197 (Grating). The ^1H and ^{13}C NMR spectra were recorded with a Bruker Spectrometer Model WH-90 and 200 MHz and the chemical shifts were recorded in δ ppm relative to TMS. The ^{31}P NMR spectra were carried out on a Varian CFT 20 Spectrometer (*vs.* external 85% H_3PO_4). The mass spectra were run at (70 eV): MS-50 of Kratos (AEI) Spectrometer provided with data system. Elemental analyses were carried out at the microanalysis laboratory, National Research Centre, Cairo, Egypt. All experiments were carried out under a nitrogen atmosphere.

Reaction of 3-Chloro-1,2,4-triazine **1a** *with 2-Alkoxy 1,3,2-Dioxaphospholane* **2a**, **b**. A mixture of **1a**[23] (2.6 g, 0.01 mol) and the cyclic phosphite **2a** or **2b** ($\sim$3 ml) was refluxed in dry benzene solution (30 ml) until gas evolution stopped. The gas was collected by passing it through deuteriochloroform; ^1H NMR analysis showed it to be methyl—or ethyl chloride, respectively. After the reaction was completed (TLC, $\sim$3 h), the volatile materials were evaporated, *in vacuo*, and the residual substance was triturated thrice by cyclohexane and then crystallized from chloroform-pet.ether to give in both cases, the adduct **6** as yellow crystals (2 g, 62%), mp 213–15°C. Anal. Calcd. for $C_{17}H_{12}N_3O_3P$ (337.29): C, 60.54; H, 3.59; N, 14.46; P, 9.18. Found: C, 60.38; H, 3.52; N, 14.07; P, 9.13. IR(KBr) cm^{-1}: 1258 (P=O), 1085 (P—O—C), 785 (C—P). ^1H NMR ($CDCl_3$) δ: 3.8, 4.2 (2d of t, 4H, 2 OCH_2, J_{HP} = 11.5 Hz), 7.6–9.2 ppm (m, 8H, Ar—**H**). ^{13}C NMR ($CDCl_3$) δ: 62.2, 62.6 (2d OCH_2, J_{COP} = 6.7 Hz), 158.3 ppm (d, N=C—P, J_{CP} = 243 Hz). ^{31}P NMR($CDCl_3$) δ = 11.4 ppm. MS: m/z = 337 (18%).

Reaction of **1a** *with 2-Dimethylamino-1,3,2-dioxaphospholane* **2c**. A mixture of **1a** (2.6 g, 0.01 mol) and the cyclic phosphite **2c** ($\sim$3 ml) was refluxed in dry benzene solution (30 ml) for 2 h. Working up the mixture as above yielded 1.6 g of **8** as yellow crystals in 57.6% yield, mp 167°C (ethyl alcohol). Anal. Calcd. for $C_{17}H_{14}N_4$ (274.33): C, 74.43; H, 5.14; N, 20.42. Found: C, 74.27; H, 5.02; N, 20.39. IR (KBr) cm^{-1}: 1245 (C—NCH_3), 1555 (N=N). ^1H NMR ($CDCl_3$): δ 3.24, 3.26 [2s, 6H, $N(CH_3)_2$], 7.6–9.3 ppm (m, 8H, Ar—**H**). ^{13}C NMR ($CDCl_3$): δ 32.1, 32.6 [2s, $N(CH_3)_2$], 152.8 ppm (N=C—NCH_3). MS: m/z = 274 (22%).

^{31}P NMR ($CDCl_3$) of the reaction mixture prior to working up had three resonances at δ 150.4 (unreacted **2c**), 167.6 (**2d**); 13.3 ppm (ring-retained phosphate, from air oxidation of the phosphite.

Preparation of **8**. A mixture of **1a** (0.5 g) and dimethylamine ($\sim$3 ml) in tetrahydrofuran (30 ml) was refluxed for 6 h. Evaporating the volatile materials and crystallizing the residue from ethanol furnished compound **8** as yellow crystals (0.4 g, 72%), mp, mixed mps, and comparative IR spectra.

Reaction of **1a** *with Trialkyl Phosphites* **3a–c**. The products **10a–c** were obtained upon heating trimethyl-, triethyl- and triisopropyl phosphites **3a–c** (6 ml) with **1c** (2.6 g, 0.01 mol) at 100°C in the absence of solvent for $\sim$2 h (TLC) and using the previous workup.

Compound **10a**, yellow needles (2.3 g, 67.3%) mp 144°C (chloroform-pet. ether, 1:1). Anal. Calcd. for $C_{17}H_{14}N_3O_3P$ (339.30): C, 60.18; H, 4.16; N, 12.38; P, 9.13. Found: C, 60.05; H, 4.09; N, 12.17; P, 9.01. IR (KBr) cm^{-1}: 1665 (C═N), 1258 (P═O), 1100 (P—O—C), 875 (C—P). ^1H NMR ($CDCl_3$): δ 4.16 (d, 6H, OC**H**$_3$, J_{HP} = 13.5 Hz), 7.7–9.6 ppm (m, 8H, Ar—**H**). ^{13}C NMR ($CDCl_3$): δ 52.7 (d, OCH_3, $^3J_{CP}$ = 6.8 Hz), 148.5 ppm (d, N═C—P, J_{CP} = 247 Hz). ^{31}P NMR ($CDCl_3$) δ = 8.4 ppm. MS: m/z = 339 (18%).

Compound **10b**, yellow needles (2.7 g, 74.8%) mp 185°C (chloroform-pet. ether, 1:1). Anal. Calcd. for $C_{19}H_{18}N_3O_3P$ (367.36): C, 62.12; H, 4.94; N, 11.44; P, 8.43. Found: C, 62.00; H, 4.78; N, 11.21, P, 8.53. IR (KBr), cm^{-1}: 1670 (C═N), 1255 (P═O), 1080 (P—O—C), 890 (C—P). ^1H NMR ($CDCl_3$): δ 1.52 (d of t, 6H, C.C**H**$_3$, J_{HP} = 11.5 Hz), 4.15 (q, 4H, CH_2, J_{HP} = 11.5 Hz), 7.5–9.1 ppm (m, 8H, Ar—**H**). ^{31}P NMR ($CDCl_3$): δ = 8.8 ppm. MS: m/z = 367 (32%).

Compound **10c**, yellow needles (2.8 g, 72.6%) mp 157°C (chloroform-pentane, 1:1). Anal. Calcd. for $C_{21}H_{22}N_3O_3P$ (395.41): C, 63.79; H, 5.61; N, 10.63; P, 7.83. Found: C, 63.58; H, 5.47, N, 10.48; P, 7.85. IR (KBr)cm^{-1}: 1656 (N—C═N), 1275 (P═O), 1063 (P—O—C). ^1H NMR ($CDCl_3$): δ 1.52 (d, 12H, C.C**H**$_3$, J_{HP} = 13.5 Hz), 4.2 (sept., 2H, C**H**.C, J_{HP} = 13.5 Hz), 7.65–9.8 ppm (m, 8H, Ar—**H**). ^{31}P NMR ($CDCl_3$) δ: 8.6 pm. MS: m/z = 395 (25%).

Reaction of **1a** *with Triphenylphosphine TPP.* A solution of 2.6 g (0.01 mol) of chlorotriazine **1a** and 2.6 g (0.01 mol) of TPP in 50 ml of xylene was refluxed for 10 h. The mixture was filtered in a dry atmosphere and residual solvent was removed in a vacuum desiccator to give 3.8 g (78%) of **11**, mp > 280°C which is quite stable for running the spectroscopic analyses. Anal. Calcd. for $C_{33}H_{23}ClN_3P$ (528.05): C, 75.06; H, 4.39; Cl, 6.72; N, 7.96; P, 5.86. Found: C, 74.88; H, 4.26; Cl, 6.54; N, 7.73; P, 5.71. IR (KBr) cm^{-1}: 980 (P—Ph), 770 (N═C—P). ^1H NMR (DMSO): δ 7.3–9.8 ppm (m, Ar—**H**). ^{13}C NMR (DMSO): δ 162.4 ppm (d, N═C—P, J_{CP} = 147 Hz). ^{31}P NMR (DMSO): δ = 14.3 ppm.

Solvolysis of **11**. To a solution of 1 g of **11** in 30 ml of methanol was added 5 ml of water. The solution was boiled for 15 min., diluted with water, and extracted with chloroform. The chloroform solution was washed, dried, and evaporated. The residue was triturated by boiling benzene. Concentration of benzene precipitated triphenylphosphine which was isolated and identified. Crystallization of the residue from pyridine afforded the hydroxytriazine **1b** mp., mixed mps (284–86°C)[23] and comparative IR spectra.

Reaction of 3-Hydroxy-1,2,4-triazine **1b** *with 2-Chloro-1,3,2-Dioxaphospholane* **2d**. To a stirred solution of the hydroxy compound **1b** (2.5 g, 0.01 mol) in dry acetone (100 ml) was added 5 g of dry K_2CO_3 and stirring was continued at room temperature for 1 h. The chlorophospholane **2d** (1.6 ml, 0.013 mol) was added and the mixture was refluxed for 12 h. After removal of the inorganic material by filtration, the volatile materials were removed from the filtrate under reduced pressure. The residue was crystallized from cyclohexane to give 1.5 g of the phosphoramidite **12** (47.4%), mp 102°C. Anal. Calcd. for $C_{17}H_{12}N_3O_3P$ (337.29): C, 60.54; H, 3.59; N, 12.46; P, 9.18. Found: C, 60.44; H, 3.77; N. 12.28; P, 9.06. IR (KBr) cm^{-1}: 1675 (C═O), 1020 (P—O—C), 970 (P—N). ^1H NMR ($CDCl_3$): δ 3.75, 4.32 (2d of t, 4H, OC**H**$_2$, $^3J_{HP}$ = 10.5 Hz), 6.8–8.9 ppm (m, 8H, Ar—**H**). ^{13}C NMR ($CDCl_3$): δ 196.6 (C═O). ^{31}P NMR ($CDCl_3$): δ = 148.3 ppm. MS: m/z = 337.

Alkali-Hydrolysis of **12**. Compound **12** (0.8 g) was refluxed for 2 h with a solution of 10% NaOH aq. (10 ml) and ethanol (10 ml). After cooling the solution was acidified with 15% HCl. The ppt. so formed (0.55 g, 60%) was crystallized from pyridine and proved to be **1b** (mp, mixed mps and comparative IR spectra.[23]

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