matic); 6.31(m, 3 H, 3 CH, pyrrole ring); 5.86(bs, 1 H, NH, D_2O exchangeable); 5.73(s, 1 H, CH, β-lactam ring).

1-(4-Methylphenyl)-3,3-diphenyl-4-(2-pyrryl)-2-azetidinone (3b). UV(EtOH, nm): 244(ϵ 2.3 × 10^4), 295(ϵ 0.3 × 10^4); IR(Nujol, cm^{-1}): 1740(C=O, β-lactam) and 3410(N–H, pyrrole); NMR($CDCl_3$, δ ppm): 7.65(m, 2 H, aromatic); 7.10(m, 12 H, aromatic); 6.25(m, 3 H, three CH, pyrrole ring); 5.78(bs, 1 H, NH, D_2O exchangeable); 5.60(s, 1 H, CH, β-lactam ring); 2.25(s, 3 H, CH_3).

1-(4-Chlorophenyl)-3,3-diphenyl-4-(2-pyrryl)-2-azetidinone (3c). UV(EtOH, nm): 255(ϵ 2.4 × 10^4), 295(ϵ 0.4 × 10^4); IR(Nujol, cm^{-1}): 1745(C=O, β-lactam) and 3410(N–H, pyrrole); NMR($CDCl_3$, δ ppm): 7.55(m, 2 H, aromatic); 7.02(m, 12 H, aromatic); 6.22(m, 3 H, 3 CH, pyrrole ring); 5.72(bs, 1 H, NH, D_2O exchangeable); 5.63(s, 1 H, CH, β-lactam ring).

1-Diphenylmethyl-3,3-diphenyl-4-(2-thienyl)-2-azetidinone (3d). UV(EtOH, nm): 254(ϵ 0.4 × 10^4); IR(Nujol, cm^{-1}): 1740(C=O, β-lactam); NMR($CDCl_3$, δ ppm): 7.00(m, 23 H, aromatic); 5.58(s, 1 H, CH, benzhydryl); 5.46(s, 1 H, CH, β-lactam ring); Mass: m/z 471(M^+, 17), 304(16), 277(24), 276(43), 262(100), 261(31), 209(14), 194(98), 167(99).

1-(4-Methylphenyl)-3,3-diphenyl-4(2-thienyl)-2-azetidinone (3e). UV(EtOH, nm): 250(ϵ 2.6 × 10^4); IR(Nujol, cm^{-1}): 1750(C=O, β-lactam); NMR($CDCl_3$, δ ppm): 7.41(m, 17 H, aromatic); 5.91(s, 1 H, CH, β-lactam ring); 2.25(s, 3 H, CH_3).

1-(4-Chlorophenyl)-3,3-diphenyl-4-(2-thienyl)-2-azetidinone (3f). UV(EtOH, nm): 254(ϵ 2.8 × 10^4); IR(Nujol, cm^{-1}): 1740(C=O, β-lactam); NMR($CDCl_3$, δ ppm): 7.16(m, 17 H, aromatic); 5.93(s, 1 H, CH, β-lactam ring).

1-Diphenylmethyl-3,3-diphenyl-4-(2-furyl)-2-azetidinone (3g). UV(EtOH, nm): 258(ϵ 0.1 × 10^4); IR(Nujol, cm^{-1}): 1745(C=O, β-lactam); NMR($CDCl_3$, δ ppm): 7.16(m, 21 H, phenyl and furan CH_α,); 5.98(m, 1 H, furan CH_β); 5.80(bs, 2 H, furan CH_β, and benzhydryl); 5.23(s, 1 H, CH, β-lactam ring).

1,3,3-Triphenyl-4-(1-naphthyl)-2-azetidinone (3h). UV(EtOH, nm): 254(ϵ 2.8 × 10^4), 288(ϵ 1.4 × 10^4), 300(ϵ 0.9 × 10^4); IR(Nujol, cm^{-1}): 1740(C=O, β-lactam); NMR($CDCl_3$, δ ppm): 7.41(m, 22 H, aromatic); 6.50(s, 1 H, CH, β-lactam ring).

1,3,3-Triphenyl-4-(2-naphthyl)-2-acetidinone (3i). UV(EtOH, nm): 252(ϵ 1.8 × 10^4), 290(ϵ 0.2 × 10^4), IR(Nujol, cm^{-1}): 1740(C=O, β-lactam); NMR($CDCl_3$, δ ppm): 7.25(m, 22 H, aromatic); 5.86(s, 1 H, CH, β-lactam ring).

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Registry No. 1, 3469-17-8; **2a**, 4089-09-2; **2b**, 14479-37-9; **2c**, 51305-60-3; **2d**, 105090-43-5; **2e**, 5918-69-4; **2f**, 13533-31-8; **2g**, 54220-18-7; **2h**, 890-50-6; **2i**, 18263-29-1; **3a**, 105090-44-6; **3b**, 105090-45-7; **3c**, 105090-46-8; **3d**, 105090-47-9; **3e**, 105090-48-0; **3f**, 105090-49-1; **3g**, 105090-50-4; **3h**, 105090-51-5; **3i**, 105090-52-6; **4**, 3893-33-2.

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Phase-Transfer-Catalyzed Preparation of Triaryl Phosphorothionates

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Triaryl phosphorothionates have been prepared in good yield from thiophosphoryl chloride and three equivalents of a phenol by a phase-transfer-catalyzed process.

Triaryl phosphates are commonly prepared by treatment of phosphorus oxychloride ($POCl_3$) with three equivalents of a phenol (*1*). A number of effective catalysts have been identified to promote this reaction, such as halide salts of calcium, magnesium, and aluminum (*2*). This contrasts the behavior of thiophosphoryl chloride ($PSCl_3$) which does not react with phenol even under quite forcing conditions. In the presence of aluminum chloride, phenol and thiophosphoryl chloride remained unchanged after several hours at reflux. Triaryl phosphorothionates have been prepared from $PSCl_3$ and three equivalents of a phenol in the presence of an acid acceptor such as triethylamine (*3*) or by the oxidation of a triaryl phosphite with sulfur (*4*). This appeared to constitute a problem for us, since we were in need of large amounts of a variety of pure triaryl phosphorothionates in connection with another study.

Triaryl phosphates and diaryl benzenephosphorothionates have very recently been prepared by the phase-transfer-catalyzed reaction of phenols with $POCl_3$ (*5*, *6*) and $C_6H_5PSCl_2$ (*7*), respectively. We now wish to report a general synthesis of triaryl phosphorothionates by phase-transfer catalysis, which is both simple and effective (eq 1).

$$\underset{\mathbf{1}}{3HOAr} + \underset{\mathbf{2}}{PSCl_3} \xrightarrow[\substack{CH_2Cl_2 \\ PTC}]{NaOH/H_2O} \underset{\mathbf{3}}{(ArO)_3P{=}S} + 3NaCl \qquad (1)$$

As can be seen from Table I, the isolated yields obtained for this process were generally in the 80 percentile range. The products were identified by their spectral data (see Table II), and melting points where known. For all new compounds satisfactory high-resolution mass spectral data as well as correct elemental analyses were obtained. The tris(p-chloro-

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Table I. Triaryl Phosphorothionates Prepared by Phase-Transfer Catalysis

no.	Ar	yield,[a] %	mp, °C	lit. mp °C	mass spectrum calcd	mass spectrum found
3a	C_6H_5	86	55	54[b]		
3b	$4H_3C-C_6H_4$	84	93–94	93–94[c]		
3c	$2,4(H_3C)_2C_6H_3$	76	63.0–64.5		426.1419	426.1428
3d	2naphthyl	83	94.5–96.0		492.0949	492.0957
3e	$3,5(H_3C)_2C_6H_3$	80	92–94		426.1419	426.1438
3f	$4Cl-C_6H_4$	88	84–86	108.5[d] 113[e] 85–86[f]	443.9310	443.9310
3g	$4O_2H-C_6H_4$	82[g]	177–179	174[h]	477.0032	477.0029
3h	$4NC-C_6H_4$	88	158–159		417.0336	417.0336
3i	$4Br-C_6H_4$	86	96.0–97.5	88–99[f]	575.7797	575.7799

[a] Yield of purified product after recrystallization from *n*-heptane; elemental analyses (C, H, N, P, S, Cl, Br) were submitted for review and agreed with the appropriate theoretical values. [b] Yamasaki, T. *Science Rep. Inst. Tohou Univ.* **1954**, *6*, 172; *Chem. Abstr.* **1955**, *49*, 6858i. [c] *Beilstein 6* (3) 1372. [d] Mel'nikov, N. N.; Shevetsova, S.; Kagan, M. Y.; *Zh. Obshch. Khim.* **1960**, *30*, 2931; *Chem. Abstr.* **1961**, *55*, 9321a. [e] Kamai, G.; Koshkina, E. S. *Tr. Kazan. Khim-Tekhnol. Inst.* **1955**, 11: *Chem. Abst.* **1956**, *50*, 6347a. [f] Mel'nikov, N. N.; Khokhlov, D. N. *Zh. Obsch. Khim.* **1953**, *23*, 1357; *Chem. Abstr.* **1954**, *48*, 9903e. [g] Recrystallized from acetone. [h] Ketelarr, J. A. A.; Gersmann, H. R. *J. Am. Chem. Soc.* **1950**, *72*, 5777.

Table II. Spectral Data of Triaryl Phosphorothionates

no.	IR, cm^{-1}	^1H NMR, ppm
3a	1587, 1185, 1158, 939, 798, 751, 685	7.35 (brs. 5 H)
3b	1493, 1181, 940, 923, 821, 748	2.33 (s, 3 H), 7.17 (s, 4 H)
3c	2880, 1477, 1242, 1180, 1098, 940, 902, 808, 763, 687	2.22 (s, 3 H), 2.24 (s, 3 H), 6.90 (d, 1 H, $J = 7.5$ Hz), 6.96 (brs, 1 H), 7.20 (d, 1 H, $J = 7.5$ Hz)
3d	1242, 1210, 1157, 981, 968, 946, 938, 875, 870, 741	7.43 (m, 3 H), 7.74 (s, 4 H)
3e	1281, 1125, 1018, 951, 854, 677	2.30 (s, 6 H), 6.87 (s, 3 H)
3f	1471, 1182, 1157, 1081, 921, 824, 786, 770	7.31 (dd, 2 H, $J_{AB} = 8.3$, $J_{BP} = 1.5$ Hz), 7.35 (d, 2 H, $J_{AB} = 8.3$ Hz)
3g	1587, 1523, 1485, 1356, 1190, 1162, 930, 858, 800, 750	7.73 (dd, 2 H, $J_{AB} = 9.0$, $J_{BP} = 1.5$ Hz), 8.44 (d, 2 H, $J_{AB} = 9.0$ Hz)
3h	1193, 1161, 923, 834	7.38 (dd, 2 H, $J_{AB} = 9.0$, $J_{BP} = 2.0$ Hz), 7.78 (d, 2 H, $J_{AB} = 9.0$ Hz)
3i	1185, 1158, 925, 828, 798, 765, 670	7.13 (dd, 2 H, $J_{AB} = 9.0$, $J_{BP} = 1.5$ Hz), 7.54 (d, 2 H, $J_{AB} = 9.0$ Hz)

phenyl) phosphorothionate had been reported in the literature on three occasions; however, each reference contains a different melting point, so this material was also fully characterized.

Experimental Section

Melting points were determined on a Thomas Hoover apparatus and are uncorrected. Infrared spectra were determined with a Beckman Microlab MX-250 spectrophotometer as KBr disks; absorbance positions are reported in reciprocal centimeters (cm^{-1}). Proton magnetic resonance spectra were recorded on a Varian EM-390 spectrometer as solutions in chloroform-*d* unless otherwise stated. High-resolution mass spectra were recorded on a MAT instrument. Elemental analyses were determined by the General Electric Research and Development Center analytical services group.

***Preparation of Triphenyl Phosphorothionate*: *Typical Example*.** A 500-mL round-bottomed flask equipped with a reflux condenser, mechanical stirrer, and addition funnel was charged with phenol (56.4 g, 0.6 mol) and sodium hydroxide solution, (24.0 g, 1.2 mol in 150 mL of water). To this solution was added Aliquot 336 (2.25 g) and 150 mL of dichloromethane. The solution was stirred rapidly while thiophosphoryl chloride (33.9 g, 0.2 mol) was added dropwise from the addition funnel over a period of 0.25 h. The solution was stirred at room temperature for 2 h and then the contents of the flask poured into a separatory funnel. The phases were separated and the aqueous layer extracted with two 100-mL portions of dichloromethane. The combined organic extracts were washed with brine and then dried over anhydrous magnesium sulfate. The solution was filtered and concentrated and the oil taken up in 100 mL of hot *n*-heptane whereupon crystals of pure thiophosphate formed, 48.8 g, (86%); mp 55 °C.

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Heterocycles. 11. Synthesis of Substituted Benzo[*h*]quinazolines

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2-Arylidene-1-tetralones (I) were condensed with benzamidine or guanidine to give the corresponding substituted benzo[*h*]quinazolines II and III, respectively. The structures of all products were established by chemical and spectroscopic methods.

Aryl aldehydes were previously reacted with 1-tetralones (*1*) to yield 2-aryliden-1-tetralones (I). These were condensed with benzamidine to produce the corresponding 4-aryl-2-phenylbenzo[*h*]hexahydroquinazolines (IIa–i) (cf. Scheme I). The structures of the products are different from those previously mentioned (*2*) and were substantiated by spectral and chemical