Table II. Characteristic Infrared Absorption Bands of 1,3-Bis(o-hydroxybenzyl)-2-substituted Imidazolidines

Compound No.	Aromatic CH Stretch, Cm.$^{-1}$	Aliphatic C—H Stretch, Cm.$^{-1}$	—OH Stretch, Cm.$^{-1}$	OH Deformations[a] and C—O Stretch, Cm.$^{-1}$	C=C Stretch, Cm.$^{-1}$	C—H Out of Plane Deformations, Cm.$^{-1}$	Specific Absorptions, Cm.$^{-1}$
1	[b]	[b]	[b]	1230	1590	695	None
				1335	1470	725	
					1400[c]	740	
2	3030	2860	3367	1250	1610	825	C—Cl
				1335	1590	755	702
					1485		
					1410[c]		
3	3020	2773	3367	1240	1610	759	C—Cl
				1325	1590	821	652
					1480	870	
					1460		
					1390[c]		
4	3040	2793	3333	1230	1613	691	None
				1345	1585	754	
					1560	781	
					1450	860	
					1410[c]		
5	3040	2809	3333	1235	1600	767	C≡N
				1360	1470	823	2221
					1410[c]		
6	3030	2810	3330	1245	1615	757	C—CS—
				1325	1590		720
					1490		
					1400[c]		
7	3030	2790	3340	1230	1590	715	None
				1325	1470	750	
					1400[c]	815	

[a] Each compound showed multiple absorptions (7 to 10 bands) in 1180- to 1400-cm.$^{-1}$ region; bands assigned to OH deformations and C—O stretch were those having strongest absorption. [b] Specific absorptions could not be assigned because of broad band in 3500- to 2500-cm.$^{-1}$ region. [c] This absorption is also in region for —OH deformation or —C—O stretch.

(*1*). The cooled reaction mixture was filtered and the collected solid purified as indicated in Table I.

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N,N'-Disubstituted *P*-Phenylphosphonothioic Diamides

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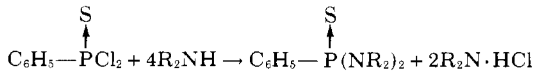
Several *N,N'*-disubstituted *P*-phenylphosphonothioic diamides were synthesized and characterized. Yields, melting points, infrared data, and elemental analyses are given for the reported compounds.

SEVERAL *N,N'*-disubstituted *P*-phenylphosphonothioic diamides were synthesized by the reaction of phenylphosphonothioic dichloride with primary and secondary amines using standard methods (*1, 2*). The compounds are listed in Table I.



$$C_6H_5\text{—}\overset{S\atop\uparrow}{P}Cl_2 + 4R_2NH \rightarrow C_6H_5\text{—}\overset{S\atop\uparrow}{P}(NR_2)_2 + 2R_2N\cdot HCl$$

With the exception of the *N,N'*-bis(ethoxyethyl) derivative, which was a straw-colored oil, the compounds were colorless, crystalline solids. The infrared spectra were consistent in each instance with the desired structure. Characteristic absorptions are shown in Table II. Expected absorptions due to specific groups for each compound were also noted.

EXPERIMENTAL

Phenylphosphonothioic dichloride was purchased from the Aldrich Chemical Co. Other reactants were also obtained commercially (reagent grade) and used without further purification, except 2-aminothiazole (technical grade) which was recrystallized from toluene. Reaction solvents were dried over sodium. Melting points were taken on a Thomas-Hoover melting point apparatus and are uncorrected.

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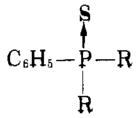
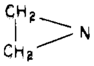
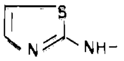
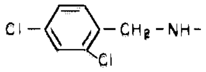
Table I. Characterization of Some *N,N'*-Disubstituted *P*-Phenylphosphonothioic Diamides



$$C_6H_5-\overset{\overset{S}{\uparrow}}{\underset{R}{P}}-R$$

No.	Compound	R	Yield, %	M.P., ° C.	Empirical Formula		% C	% H	% P	% S
1	*N,N'*-Diallyl-*P*-phenyl phosphonothioic diamide	$CH_2=CH-CH_2-NH-$	55[a]	51–54	$C_{12}H_{17}N_2PS$	Calcd. Found	57.2 57.1	6.8 6.1	12.2 12.4	... ...
2	Bis(1-aziridinyl)phenyl phosphine sulfide		9[b]	105–07.5	$C_{10}H_{13}N_2PS$	Calcd. Found	53.5 53.5	5.8 5.8	13.8 14.2	14.3 14.5
3	*N,N'*-Bis(ethoxyethyl)-*P*-phenylphosphonothioic diamide	$C_2H_5O-CH_2CH_2-NH-$	69[c]	Oil	$C_{14}H_{25}N_2O_2PS$	Calcd. Found	53.2 52.6	8.0 8.1	9.7 9.7	10.1 9.8
4	*N,N'*-Bis(2-thiazolyl)-*P*-phenyl phosphonothioic diamide		60[d,e]	210–11 (dec.)	$C_{12}H_{11}N_4PS_3$	Calcd. Found	42.6 42.5	3.3 3.3	9.2 9.3	28.4 28.8
5	*N,N'*-Bis(2,4-dichlorobenzyl)-*P*-phenylphosphonothioic diamide		28[f,g]	101–02.5	$C_{20}H_{17}Cl_4N_2PS$[h]	Calcd. Found	49.0 48.9	3.5 3.3	6.3 6.3	6.5 6.5
6	Bis(2-phenylhydrazinyl) phenylphosphine sulfide	$C_6H_5NH-NH-$	53[i,j]	199.5–200.5 (dec.)	$C_{18}H_{19}N_4PS$	Calcd. Found	61.0 61.3	5.4 5.6	8.7 8.9	... ...

[a] Recrystallized from absolute ethanol to yield white crystalline solid. [b] Crude solid dissolved in benzene-pentane (1 to 1), filtered through anhydrous sodium carbonate and cooled to yield white crystalline solid. [c] Crude oily product heated to 132° C./0.40 mm. to remove unreacted dichloride and amine, n_D^{20} 1.5508. [d] Refluxed 1 hour. [e] Crude solid recrystallized from absolute ethanol to yield white powder. [f] Refluxed 3 hours. [g] Crude oil crystallized by trituration in absolute ethanol, then recrystallized from ethanol to yield white powder. [h] Cl, calcd., 28.9; found, 29.3. [i] Refluxed 5½ hours. [j] Crude solid recrystallized from benzene to yield white powder.

Table II. Characteristic Infrared Absorptions of *N,N'*-Disubstituted *P*-Phenylphosphonothioic Diamides

Compound No.	P→S (Stretching), Cm.$^{-1}$	P—N (Stretching), Cm.$^{-1}$	P—C_6H_5 (Stretching), Cm.$^{-1}$	CH Deformations, Cm.$^{-1}$
1	655	715	1439	750 690
2	670	710 730	1439	750 695
3	650	720	1445	750 695
4	650	718	1439	748 693
5	622	715 722	1435	800 748 688
6	650	743 752	1437	750 690

Infrared spectra of the solid compounds dispersed in KBr matrices were recorded by a Perkin-Elmer Model 21 spectrophotometer. The infrared spectrum of a thin film of *N,N'*-bis(ethoxyethyl)-*P*-phenylphosphonothioic diamide was also recorded.

Procedure for Compounds 1, 2, and 3 (*1, 2*). A solution of phenylphosphonothioic dichloride (0.75 mole) in anhydrous ether (200 ml.) was added dropwise with stirring to a solution of amine (2.92 moles) in anhydrous dried ether (500 to 1000 ml.) in a nitrogen atmosphere at room temperature. The reaction mixture was allowed to stand at room temperature overnight under a nitrogen blanket. The precipitated amine hydrochloride was removed by filtration and the filtrate was concentrated in vacuo to yield an off-white solid. The crude product was purified as described in the footnotes in Table I.

Procedure for Compounds 3, 4, and 5 (*1, 2*). A solution of phenylphosphonothioic dichloride (0.50 ml.) in anhydrous benzene (1000 ml.) was added dropwise with stirring to a solution of the amine (1.90 moles) in anhydrous benzene in an atmosphere of nitrogen at room temperature. In the preparation of *N,N'*-bis(2-thiazolyl)-*P*-phenylphosphonothioic diamide, it was necessary to dissolve 2-aminothiazole in dry ether (1 liter).

To prepare the *N,N'*-bis(2,4-dichlorobenzyl)-*P*-phenylphosphonothioic diamide derivative, phenylphosphonothioic dichloride (0.114 mole), amine (0.227 mole), and triethylamine (as hydrochloride acceptor) (0.227 mole) were used. The reaction mixture was refluxed (Table I), and the precipitated hydrochloride salt was removed by filtration. The filtrate was concentrated in vacuo to an oil or solid. Purification was effected as described in the footnotes in Table I.

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