droxide, ice being added to keep the temperature below 20° C. The resulting precipitate was dissolved in ether and the ethereal solution dried over anhydrous sodium sulfate. After removal of the ether, the residue, if solid, was crystallized from petroleum ether or purified by fractional distillation at reduced pressure (Table III), if liquid.

Preparation of 1,10-Phenanthrolines. To a well-stirred mixture of 0.1 mole of the appropriate 8-aminoquinoline, 18 ml. of 80% arsenic acid, and 100 ml. of 87% phosphoric acid heated to 100° C., 0.2 mole of the α,β-unsaturated ketone was slowly added. Stirring was continued for 3 hours while the temperature was slowly raised to 130° C. The mixture was cooled, made alkaline with concentrated potassium hydroxide and the resulting precipitate removed by filtration. The filtrate and residue were extracted with hot benzene. After removal of benzene, the product was purified by crystallization from a suitable solvent (Table IV).

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1,3-Bis(o-hydroxybenzyl)-2-Substituted Imidazolidines

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Several 1,3-bis(o-hydroxybenzyl)-2-substituted imidazolidines were synthesized and characterized. Yields, melting points, and elemental analyses are reported for the new compounds.

USING THE PROCEDURE of Billman and Dorman (*1*) several 1,3-bis(*o*-hydroxybenzyl)-2-substituted imidazolidines were synthesized and tested for herbicidal activity.

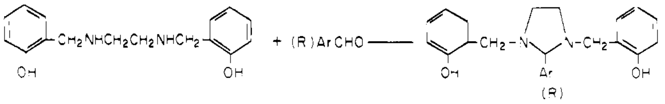


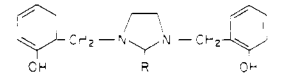
Table I summarizes yields, melting points, and elemental analyses of compounds prepared. A list of characteristic infrared absorption bands is shown in Table II. The infrared spectrum of each compound was consistent with the desired structure.

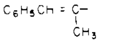
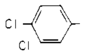
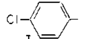
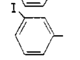
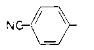
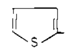
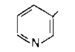
EXPERIMENTAL

N,N'-Bis(*o*-hydroxybenzyl)ethylenediamine was obtained from the Dow Chemical Co. Other reactants were also obtained comercially and used without further purification. Melting points were taken on a Hershberg melting point apparatus and are uncorrected. Infrared spectra were recorded on a Perkin-Elmer Model 21 spectrophotometer using potassium bromide as a matrix.

To a hot solution of *N,N'*-bis(*o*-hydroxybenzyl)ethylenediamine (0.1 mole) in 500 ml. of methanol was added 0.1 mole of the aldehyde dissolved in a minimum amount of methanol. The reaction mixture was refluxed for 1 hour

Table I. 1,3-Bis(o-hydroxybenzyl)-2-substituted Imidazolidines



Compound No.	Compound	R	% Yield	M.P., ° C.		Analyses % C	 % H	 % Cl	 % N
1	1,3-Bis(*o*-hydroxybenzyl)-2-(α-methy-10-styryl)imidazolidine	 $C_6H_5CH=\underset{CH_3}{\underset{\vert}{C}}-$	80	115–16[a]	Calcd. Found	78.0 78.0	7.1 6.9	. . .	7.0 7.1
2	1,3-Bis(*o*-hydroxybenzyl)-2-*p*-chlorophenylimidazolidine		85	155–56[b]	Calcd. Found	70.0 70.0	5.9 5.8	9.0 8.9	7.1 7.1
3	2-(3,4-Dichlorophenyl)-1,3-bis(*o*-hydroxybenzyl)imidazolidine		88	106–07[a]	Calcd. Found	64.3 64.5	5.2 5.3	16.5 16.5	6.5 6.8
4	1,3-Bis(*o*-hydroxybenzyl)-2-*m*-iodophenylimidazolidine		87	138–39[b]	Calcd. Found	56.8[c] 57.0	4.8 4.8	. . .	5.8 5.7
5	2-(*p*-Cyanophenyl)-1,3-bis(*o*-hydroxybenzyl)imidazolidine		65	142–43[a]	Calcd. Found	74.8 74.6	6.0 6.1	. . .	10.9 10.8
6	1,3-Bis(*o*-hydroxybenzyl)-2-(2-thienyl)-imidazolidine		90	178–79[d]	Calcd. Found	68.8[e] 68.9	6.1 6.1	. . .	7.6 7.7
7	1,3-Bis(*o*-hydroxybenzyl)-2-(3-pyridyl)-imidazolidine		82	182–83[f]	Calcd. Found	73.1 73.0	6.4 6.4	. . .	11.6 11.8

[a] Recrystallized from methanol. [b] Recrystallized from 2-propanol. [c] Analyzed for iodine: Calcd. 26.1; found 25.8. [d] Recrystallized from pyridine. [e] Analyzed for sulfur: Calcd. 8.8; found 8.7. [f] Recrystallized from ethanol.

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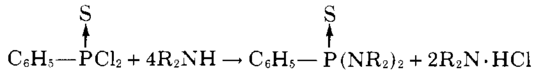
Several *N,N'*-disubstituted *P*-phenylphosphonothioic diamides were synthesized and characterized. Yields, melting points, infrared data, and elemental analyses are given for the reported compounds.

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.



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

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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