to room temperature and placed in the refrigerator overnight. The precipitate is filtered off, and the filtrate treated with twice its volume of water and placed in the refrigerator for several hours.

The precipitate of 4,4′-diacetylaminodiphenyl sulfone weighs about 30 g. The melting point should be between 275–278° (uncor.), and the material in the melting point tube should not become very dark on melting. In case the material is not very pure, it is dissolved in boiling acetic acid, and 10 cc. of superoxol added. It is then placed in the refrigerator and cooled overnight.

The residue obtained by precipitating the filtrate with water is filtered off and saved until a sufficiently large number of residues is obtained. These are combined and dissolved in boiling glacial acetic acid, and 5 cc. of superoxol for each 10 g. of residue added. The solution is cooled overnight in the refrigerator and the precipitate filtered off. The residue from each run amounts to about 10–20% of the weight of thioaniline used.

4,4′-Diaminodiphenyl Sulfone.—To 10 g. of 4,4′-diacetylaminodiphenyl sulfone is added a solution of 25 cc. of concentrated hydrochloric acid (sp. gr. 1.2) in 75 cc. of water. The mixture is refluxed for one and one-half to two hours when all of the solid is dissolved. Decolorizing carbon is then added and refluxing continued for one hour. It is filtered while hot, the filtrate cooled, ammonium hydroxide added until the solution is almost neutral and then cooled again. When the solution is cold, an excess of ammonia is added. The precipitate is filtered off, dissolved in water with just enough hydrochloric acid added to dissolve the amine, and decolorizing carbon added. It is allowed to stand for fifteen minutes or more and then filtered. The filtrate is made alkaline with ammonia, cooled, and filtered. The product weighs about 7 g., and melts at 172–174° (uncor.). It may be recrystallized from methyl alcohol if a purer product is desired, the pure 4,4′-diaminodiphenyl sulfone melting at 175–176° (uncor.).

THE LILLY RESEARCH LABORATORIES
INDIANAPOLIS, INDIANA
RECEIVED SEPTEMBER 9, 1940

NEW COMPOUNDS

ADDITION COMPOUNDS OF ZINC, CADMIUM, COBALT AND NICKEL HALIDES WITH 1,4-DIOXANE

1,4-Dioxane was dehydrated by refluxing with sodium metal for two hours, allowing to remain in contact with anhydrous magnesium sulfate for twenty-four hours, and distilling. Dioxane thus purified gave no color reaction with anhydrous copper sulfate. The anhydrous halides were commercial preparations.

Two procedures for the preparation of the compounds were employed. If the halide is appreciably soluble in dioxane at room temperature, a 25-ml. portion of the solvent was saturated by adding an excess of finely powdered halide. After standing for several days the solution was decanted, and then evaporated in a desiccator over concentrated sulfuric acid until crystals separated out. The compounds were separated from the mother liquor by decantation and pressing between layers of filter paper. This is designated in the Table as procedure I.

When the halide is slightly soluble, it was allowed to remain in contact with the dioxane for several days before removing and separating from the solvent as described above, procedure II.

Formulas were determined by analysis for halide content by Mohr's method. The experimental percentages given are the mean of two or more determinations.

TABLE I

Formula	Halide content, %		Color	Procedure
	Calcd.	Found		
$ZnCl_2\cdot(C_4H_8O_2)$	31.6	31.4	White	I
$ZnCl_2\cdot 2(C_4H_8O_2)$	22.7	21.9	White	*a*
$ZnBr_2\cdot 2(C_4H_8O_2)$	39.8	40.0	White	I
$ZnI_2\cdot 2(C_4H_8O_2)$	51.2	51.1	White	I
$CdCl_2\cdot(C_4H_8O_2)$	26.1	25.7	White	II
$CdCl_2\cdot {}^1/_2(C_4H_8O_2)$	31.2	31.6	White	*b*
$CdBr_2\cdot(C_4H_8O_2)$	44.3	44.3	White	II
$CdI_2\cdot(C_4H_8O_2)$	55.8	55.9	White	II
$CoCl_2\cdot(C_4H_8O_2)$	32.6	32.7	Pale lavender	II
$CoBr_2\cdot 2(C_4H_8O_2)$	40.5	40.3	Dark blue	II
$CoI_2\cdot 3(C_4H_8O_2)$[e]	44.0	44.2	Greenish-blue	I
$CoI_2\cdot 2(C_4H_8O_2)$	51.9	51.9	Greenish-blue	II
$CoI_2\cdot(C_4H_8O_2)\cdot 2H_2O$	58.1	57.7	Bright green	*c*
$CoI_2\cdot(C_4H_8O_2)\cdot 4H_2O$	53.6	53.8	Pink	*d*
$NiCl_2\cdot(C_4H_8O_2)$	32.6	32.5	Pale yellow	II
$NiBr_2\cdot(C_4H_8O_2)$	52.1	52.0	Bright orange	II
$NiI_2\cdot 2(C_4H_8O_2)$	51.9	51.8	Yellow	I

[a] Separated on cooling solution to 0°C. [b] Formed when the mono-1,4-dioxane compound stands over sulfuric acid. [c] Formed by exposing the anhydrous to atmospheric moisture for 2 hours. [d] Formed by exposing the anhydrous compound to atmospheric moisture for 2–3 days. [e] Very hygroscopic and unstable.

ST. LOUIS UNIVERSITY SR. RODERICK JUHASZ, S.C.N.
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2,4-DIHYDROXYBENZAL 2,4-DINITROPHENYLHYDRAZONE

Immediately upon the addition of 1.4 g. of 2,4-dihydroxybenzaldehyde, dissolved in 10 cc. of 95% alcohol to 2 g. of 2,4-dinitrophenylhydrazone, dissolved in the least possible amount of dilute sulfuric acid, a bright red crystalline precipitate began to form. It was recrystallized from hot amyl alcohol: m. p. 286° decompn., yield 87%; insoluble in water, methanol, ligroin, benzene, chloroform, ether, and acetone; slightly soluble in hot higher alcohols; soluble in ethylene glycol, hot nitrobenzene, and dilute alkalies. It decomposed upon continued heating in most solvents. *Anal.* Calcd. for $C_{13}H_{10}O_6N_4$: N, 17.61. Found: N, 17.90.

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RECEIVED OCTOBER 3, 1940

SUBSTITUTED AMIDES

In the course of a comprehensive study of the insecticidal value of substituted amides, it was necessary to prepare cer-