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, % Calcd.	Halide content, % Found	Color	Procedure
$ZnCl_2 \cdot (C_4H_8O_2)$	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.
ST. LOUIS, MISSOURI L. F. YNTEMA
RECEIVED OCTOBER 30, 1940

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.

CHEMICAL LABORATORY ALFRED W. SCOTT
UNIVERSITY OF GEORGIA JOHN M. BURNS
ATHENS, GEORGIA
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-

Name	Formula	M. p., °C. (uncor.)	Nitrogen, % Calcd.	Nitrogen, % Found
N-(2,4-Xylyl)-propionamide	$C_2H_5CONHC_6H_3(CH_3)_2$	137–137.5	7.90	7.88
N-(2,5-Xylyl)-propionamide	$C_2H_5CONHC_6H_3(CH_3)_2$	138	7.90	7.95
N-(2,6-Xylyl)-propionamide	$C_2H_5CONHC_6H_3(CH_3)_2$	115.5–116.5	7.90	7.52
N-Xenyl propionamide	$C_2H_5CONHC_6H_4C_6H_5$	176 –177	6.22	6.36
N-Benzyllauramide	$C_{11}H_{23}CONHCH_2C_6H_5$	82 –82.5	4.84	4.87
m-Laurotoluide	$C_{11}H_{23}CONHC_6H_4CH_3$	54 – 56	4.84	4.76
N-Cyclohexylpalmitamide	$C_{15}H_{31}CONHC_6H_{11}$	94 – 95	4.15	4.24
N-Benzylpalmitamide	$C_{15}H_{31}CONHCH_2C_6H_5$	94.5– 95	4.05	3.94
o-Palmitotoluide	$C_{15}H_{31}CONHC_6H_4CH_3$	90 – 91	4.05	3.97
m-Palmitotoluide	$C_{15}H_{31}CONHC_6H_4CH_3$	74.5– 75.5	4.05	4.08
N-Cyclohexyl-2-furamide	$(C_4H_3O)CONHC_6H_{11}$	112 –112.5	7.25	7.45
N-Benzyl-2-furamide	$(C_4H_3O)CONHCH_2C_6H_5$	110.5–111	6.86	6.97
N-(2,4-Xylyl)-2-furamide	$(C_4H_3O)CONHC_6H_3(CH_3)_2$	104 –105	6.51	6.51
N-(2,5-Xylyl)-2-furamide	$(C_4H_3O)CONHC_6H_3(CH_3)_2$	89 – 90	6.51	6.33
N-(2,6-Xylyl)-2-furamide	$(C_4H_3O)CONHC_6H_3(CH_3)_2$	125 –126	6.51	6.43
N-1-Naphthyl-2-furamide	$(C_4H_3O)CONHC_{10}H_7$	155 –156	5.91	5.92
N-2-Naphthyl-2-furamide	$(C_4H_3O)CONHC_{10}H_7$	152 –153	5.91	5.87
N-2-Fluoryl-2-furamide	$(C_4H_3O)CONHC_{13}H_9$	201 –201.5	5.07	5.07
N-Xenyl-2-furamide	$(C_4H_3O)CONHC_6H_4C_6H_5$	171 –172	5.62	5.48

tain compounds that are not described in the literature. The entomological results will be published elsewhere.

The lauramides, palmitamides, and furamides were prepared from the respective acid chlorides and the appropriate amines Since these amides were insoluble in water, they were separated from the amine hydrochlorides by washing with water. The propionamides were obtained by refluxing the amine and propionic acid for several hours.

These compounds, with their melting points and nitrogen analyses, are listed in the table.

BUREAU OF ENTOMOLOGY AND PLANT QUARANTINE
U. S. DEPT. OF AGRICULTURE C. V. BOWEN
WASHINGTON, D. C. L. E. SMITH

RECEIVED OCTOBER 21, 1940

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p-CYCLOHEXYLBENZOPHENONE AND OXIME

Sixteen grams of phenylcyclohexane and 14 g. of benzoyl chloride were dissolved in 70 ml. of dry carbon disulfide and 12 g. of anhydrous aluminum chloride was added slowly over a fifteen minute period. A very vigorous reaction ensued with copious evolution of hydrogen chloride. After this the mixture was heated on a water-bath for one hour and then washed with water. It was now refluxed with excess 10% sodium hydroxide solution for one hour to hydrolyze any unreacted benzoyl chloride. The carbon disulfide solution was then washed and dried over anhydrous calcium chloride. The solvent was distilled off and the residual oil vacuum distilled. A colorless, refractive liquid was obtained boiling at 195–200° at 3 mm. pressure. This material on standing a few weeks turned to a crystalline white mass: melting point, 58–60°; yield, 10 g. (40%).

Anal. Calcd. for $C_{19}H_{20}O$: C, 86.36; H, 7.58. Found: C, 85.71; H, 7.85.

Determination of structure was accomplished after the method of Mayes and Turner[1] by oxidation with sodium dichromate in the presence of sulfuric acid. The acid obtained was isolated in practically quantitative yield: melting point, 193–195°. It is hence *p*-benzoylbenzoic acid and this establishes beyond a doubt that the ketone is *p*-cyclohexylbenzophenone.

Oxime.—The oxime was prepared by the method of Shriner and Fuson[2] with the exception that sodium acetate was substituted for sodium hydroxide. It was obtained as white needles which were recrystallized from dilute alcohol; melting point, 125–127°.

Anal. Calcd. for $C_{19}H_{21}ON$: N, 5.03. Found: N, 5.30.

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(1) Mayes and Turner, *J. Chem. Soc.*, 500 (1929).

(2) Shriner and Fuson, "Identification of Organic Compounds," John Wiley and Sons, New York, N. Y., 1935, p. 145.

DEPARTMENT OF CHEMISTRY RICHARD D. KLEENE
UNIVERSITY OF CHICAGO
CHICAGO, ILL.

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