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Preparation of Some Derivatives of *p*-Hydroxybenzenesulphonamides. I.

Some *N*-Alkyl Derivatives of *p*-(Carboxymethoxy)benzenesulphonamides

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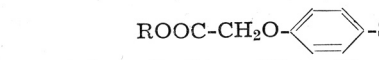
A series of products with the general formula



(where R, R′ or R″ = -H, $-CH_3$ or $-C_2H_5$),

formed by condensation of corresponding sulphonamides with derivatives of halogenacetic acid, or by condensation of corresponding sulphochlorides with some amines is described. These compounds, which are closely related to phenoxyacetic acid and to sulphonamides, are expected to have some phytohormonal properties.

Much progress has been made in the past twenty years in the field of synthetic plant hormones. Different authors have prepared a number of synthetic compounds with phytohormonal activity. Other investigators have studied the connection between the chemical constitution of organic compounds and their phytohormonal activity.

The first attempts with synthetic compounds as phytohormones were made with derivatives of indol-(3)-carbonic acid series[1]. Afterwards experiments were made with alkyl- and halogen- derivatives of benzene, naphtalene and other aromatic compounds[2,3,4,5]. These hormone-like substances induced very variable responses[6,7].

The importance of substituted phenoxy and benzoic compounds in the field of plant hormone research and some of the possible practical applications of these substances were pointed out by Zimmerman and Hitchcock in 1942[8]. They established the significant property of the wellknown weed killer 2,4-D (2,4-dichlorophenoxyacetic acid).

Recently attempts were made to use a great number of very different substances as plant hormones[9,10,11,12]. Ribiero[13] established that sulphanilamides posses some phytohormonal activity on the germination of rice. Other authors found that sulphonamides inhibit root-growth in the higher green plants[14,15,16].

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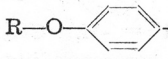
Bhardwaj and Rao[17] investigated the relative toxicity of some sulpha-drugs to wheat plants.

It would be interesting to investigate the phytohormonic activity of the derivatives of phenoxyacetic acid having in their *para* position a sulphanilamido group or *N*-substituted sulphonamide group. Compounds having in the same molecule two biologically active groups: ROOC-CH_2O-C_6H_4- with phytohormonic and -$C_6H_4SO_2NR'R''$ with bacteriostatic activity were until now unknown. For this reason we describe in the present paper the preparation of some derivatives of the above mentioned series.

The compounds were prepared by condensation of the corresponding *p*-sulphonamidophenoxides with esters of halogencarboxylic acids, or by condensation of corresponding phenolates with monochloroacetic acid. To this end we prepared as intermediate products *N,N*-dimethyl-*p*-hydroxybenzenesulphonamide (I)[18] and *N,N*-diethyl-*p*-hydroxybenzenesulphonamide (II) from the corresponding amines by diazotization and decomposition of diazonium salts by the usual methods[19,20,21]. Compound II was not yet been described.

TABLE I

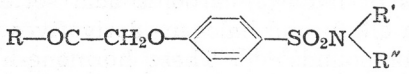
List of prepared intermediates



Compound	R	R'
I	H—	—$N(CH_3)_2$
II	H—	—$N(C_2H_5)_2$
III	$CH_3OOC—CH_2$—	—Cl
IV	$C_2H_5OOC—CH_2$—	—Cl

TABLE II

List of prepared substituted derivatives of p-carboxymethoxybenzenesulphonamide



Compound	R	R'	R''
V	CH_3O—	—H	—H
VI	HO—	—H	—H
VII	C_2H_5O—	—CH_3	—H
VIII	HO—	—CH_3	—H
IX	CH_3O—	—CH_3	—CH_3
X	C_2H_5O—	—CH_3	—CH_3
XI	HO—	—CH_3	—CH_3
XII	CH_3O—	—C_2H_5	—C_2H_5
XIII	HO—	—C_2H_5	—C_2H_5
XIV	NH_2—	—H	—H

Some esters (V, VII, IX, X, XII) were also prepared by condensation of the corresponding sulphochlorides with the appropriate amines. For this purpose two new sulphochlorides: p-(carbmethoxymethoxy)benzenesulphonyl chloride (III) and p-(carbethoxymethoxy)benzenesulphonyl chloride (IV) were prepared. They were obtained by chlorosulphonation of the methyl-phenoxyacetate[22] and ethyl-phenoxyacetate[23] using the method described by Smiles and Stewart[24,25].

The acids (VI, VIII, XI, XIII) were prepared by refluxing the esters with dil. sodium hydroxide.

Tables I and II show the substances which were prepared. With some of these substances a preliminary research on their phytohormonic activity was carried out and the results will be published elsewhere[26].

EXPERIMENTAL

The melting points are uncorrected

N,N-*Diethyl*-p-*hydroxybenzenesulphonamide* (II)

Into a solution of 5 g. (0.0219 mole) of N^1,N^1-diethyl-p-aminobenzenesulphonamide[27] (yield: 91%; m. p. 103—104°) in a mixture of 5 ml. conc. sulphuric acid and 80 ml. water, cooled to —3 to —4°, a solution of 2 g. (0.0289 mole) of sodium nitrite in 12 ml. water was gradually added until a slight excess persisted. Soon after the diazotization was complete, a solution of diazonium salt was added dropwise to 100 ml. of water warmed to 80—85° under vigorous stirring. The diazonium salt suspension (or solution) was kept during this addition in an ice water bath. The mixture was held at 80—85° for 30 minutes, then charcoal was added and the mixture filtered. The filtrate was neutralised with a hot solution of barium hydroxide (about 100 g. into 600 ml. of water). After cooling, the barium sulphate was removed, and the filtrate evaporated to about 400 ml. On standing overnight 4.7 g. of a crystallized product were obtained which were purified by recrystallization from ethanol-water. 3.7 g. (74% yield) of colorless crystals were obtained. For analysis the substance was recrystallized from ethanol-water; m. p. 49—50°.

Anal. 18.771 mg. subst.: 1.019 ml. N_2 (20°, 745 mm)
$C_{10}H_{15}NO_5S$ (229.29) calc'd.: N 6.11%
found: N 6.20%

p-(*Carbmethoxymethoxy*)*benzenesulphonyl chloride* (III)

Into 125 ml. (220 g.; 1.888 mole) of freshly distilled chlorosulphonic acid 50 g. (0.301 mole) of methyl ester of phenoxyacetic acid[22] (yield: 82%; b. p. 133/12 mm) were added dropwise under constant and vigorous stirring. The temperature of the reaction mixture was between 3—5°. The speed with which the ester was dropwise added had to be regulated according to the disappearance of the bubbles of hydrogen chloride from the reaction mixture. When all the ester had been added the reaction mixture had still to be stirred for half an hour at a temperature of 5° and then poured on about 500 g. crushed ice. A reddish oil was obtained which solidified upon rubbing and mixing. The crystalline mass was sucked dry on a sintered glass funnel and washed with cold water. To obtain it pure the product was dissolved in cold acetone, the solution shaken with charcoal and filtered. This filtered solution was added dropwise into water under vigorous mixing. The product was recrystallized six times, m. p. 63—64°; colourless hexagonic prisms, yield: between 65—70%. The product is stable for several days only at a temperature below 4°. For further syntheses two or three recrystallization were enough.

p-(*Carbethoxymethoxy*)*benzenesulphonyl chloride* (IV)

Into 23 ml. (40.5 g.; 0.348 mole) of freshly distilled chlorosulphonic acid 10 g. (0.055 mole) of ethyl ester of phenoxyacetic acid[23] (yield: 30—35%; b. p. 122/11 mm.) were added dropwise as was described in III. The reaction mixture was poured on about 150 g. of crushed ice. A light reddish oil was obtained which was kept for

several days in a cool place. Under repeated rubbing with a glass rod it solidified into a grey mass. This pastry-like mass was sucked dry on a sintered glass funnel and washed with cold water. The product was purified as previously described. Yield between 54—74%. For further syntheses the product was recrystallized three times. The product is stable for several days only at a temperature below 4°C.

p-*(Carbmethoxymethoxy)benzenesulphonamide* (V)

A suspension of 9 g. (0.034 mole) of *p*-(carbmethoxymethoxy)benzenesulphonyl chloride (III) was mixed with about 10 ml. of water and 10 ml. of 25% ammonium hydroxide. The reaction mixture was periodically well stirred, and left overnight. The crystalline product was filtered, washed with water and recrystallized from hot 60% ethanol. For analysis the substance was recrystallized from 60% ethanol; long, thin colourless needles, m. p. 210° (yield: 80—90%).

Anal. 1.83 mg. subst.: 0.098 ml. N_2 (32° 754 mm)
$C_9H_{11}NO_5S$ (245.25) calc d.: N 5.71%
found: N 5.94%

p-*(Carboxymethoxy)benzenesulphonamide* (VI)

A suspension of 1.3 g. (0.0053 mole) of V was refluxed for 20 minutes on a warm water bath with 20 ml. of 10% sodium hydroxide. To the reaction mixture charcoal was added, warmed to boiling and filtered. The filtrate was acidified with hydrochloric acid (1:1) to *p*H 1. After cooling, 1 g. of crude product (yield: 81%) was recrystallized from hot water. For analysis the substance was recrystallized from water; colourless needles, m. p. 194—195°.

Anal. 12.52 mg. subst.: 18.95 mg. CO_2, 4.25 mg. H_2O
$C_8H_9NO_5S$ (231.23) calc'd.: C 41.55; H 3.92%
found: C 41.31; H 3.79%

The same product was obtained by analogous hydrolysis of *p*-(carbethoxymethoxy)benzenesulphonamide, which we have described previously[28]. The microscopic forms of these crystals were the same, and the m. p. of their eutectic mixture was unchanged.

N-*Methyl*-p-*(carbethoxymethoxy)benzenesulphonamide* (VII)

Into a solution of 5 g. (0.018 mole) of IV in 40 ml. of acetone a cold solution of 1 g. (0.032 mole) of methylamine in 20 ml. of ether and a solution of 0.5 g. of sodium carbonate in 15 ml. of water were added simultaneously and dropwise under stirring. During this operation, the reaction mixture was cooled in ice water. After that, the flask with the reaction mixture was well closed and left at room temperature for 24 hours. The water layer was removed and the layer of acetone-ether solution was dried over sodium sulphate. The solvent was removed under reduced pressure on a warm water bath. The yellow-brown syrupy residue crystallized on standing and gave 2.4 g. of substance. The crude product was recrystallized from 60% ethanol. For analysis the substance was recrystallized from 60% ethanol; colourless flat prisms, melting at 82.5—83.0° (yield: 49%).

Anal. 15.76 mg. subst: 27.89 mg. CO_2, 7.64 mg. H_2O
1.94 mg. subst.: 0.882 ml. N_2 (25°, 759 mm)
$C_{11}H_{15}NO_5S$ (273.30) calc'd.: C 48.34; H 5 53; N 5.13%
found: C 48.29; H 5.42; N 5.20%

N-*Methyl*-p-*(carboxymethoxy)benzenesulphonamide* (VIII)

300 mg. (0.0011 mole) of VII was hydrolysed in 5 ml. of a 10% solution of sodium hydroxide, as was described in VI.

The product was recrystallized for analysis from 96% ethanol. Colourless hexagonal prisms melting at 184—185°. Yield: about 90%.

Anal. 14.11 mg. subst.: 22.90 mg. CO_2, 5 45 mg. H_2O
3.20 mg. subst.: 0.167 ml. N_2 (30°, 759 mm)
$C_9H_{11}NO_5S$ (245.25) calc'd.: C 44.07; H 4.52; N 5.71%
found: C 44.28; H 4.32; N 5 87%

N,N-*Dimethyl*-p-*(carbmethoxymethoxy) benzenesulphonamide* (IX)

Into a solution of 10 g. (0.0378 mole) of III in 40 ml. of cooled acetone 3.2 g. (0.0709 mole) of dimethylamine in 30 ml. methanol were added dropwise. The reaction mixture was stirred periodically, and placed in a refrigerator for two days. The crystalline product was filtered by suction. To the filtrate about 30 ml. of water were added and it was extracted with three 20 ml. portions of ether. The combined ether solutions were dried over sodium sulphate, the ether removed *in vacuo*, and the residue crystallized from 70% ethanol. Yield: 8.6 g. (89%) of crude product. An analytical sample was crystallized from 70% ethanol; colourless plates, m. p. 105—106°.

Anal. 13.19 mg. subst.: 23.24 mg. CO_2, 6.45 mg. H_2C
$C_{11}H_{15}NO_5S$ (273.31) calc'd.: C 48.34; H 5.53%
found: C 48.03; H 5.47%

N,N-*Dimethyl*-p-*(carbethoxymethoxy) benzenesulphonamide* (X)

A. By condensation of *N,N*-dimethyl-*p*-hydroxybenzenesulphonamide with ethyl ester of monobromoacetic acid

0.25 g. (0.0107 mole) of sodium were dissolved in 30 ml. of absolute ethanol and 2 g. (0.0098 mole) of *N,N*-dimethyl-*p*-hydroxybenzenesulphonamide (I)[18] (yield: 80%; m. p. 90—92°) were added after the sodium had disappeared. The reaction mixture was stirred and refluxed for two hours on a hot water bath. Ethanol was removed under reduced pressure and sodium phenoxide was evaporated to dryness *in vacuo* on an oil bath at 60°. To the finely pulverized residue 1.2 ml. (1.8 g.; 0.107 mole) of ethyl ester of monobromoacetic acid[29] were added. The reaction mass was well stirred and refluxed for five hours on an oil bath. In the beginning the temperature of the oil bath was 110°, but during the reaction the temperature was raised gradually to 130°. The reaction mixture became a syrupy substance. After cooling, the substance was extracted with three 50 ml. portions of warm absolute ethanol and filtered. The combined alcohol solution was passed through an Al_2O_3 column for purification. The solvent was evaporated to a small volume and after cooling (or sometimes after an addition of a few drops of water) crystallized yielding 1 g. of the product (36%). The product was recrystallized from 96% ethanol-water. For the analysis the substance was recrystallized from 96% ethanol; colourless needles with a m. p. 71—72°.

Anal. 16.600 mg. subst.: 0.688 ml. N_2 (24°, 745 mm)
$C_{12}H_{17}NO_5S$ (287.33) calc'd.: N 4.88%
found: N 4.67%

B. By condensation of IV with dimethylamine

A cooled solution of 5 g. (0.111 mole) of dimethylamine in 40 ml. of ether was added dropwise to a solution of 12.3 g. (0.044 mole) of IV in 20 ml. of acetone. There was a vigorous reaction. The reaction mixture was left standing overnight at room temperature. The solvent (acetone-ether) was removed under reduced pressure. The residue was a yellow-brown oil. After standing at 0°, the syrupy residue crystallized. A crude product was recrystallized from 96% ethanol-water. Colourless needles, m. p. 71—72°.

The microscopic forms of the crystals obtained by method *A* and method *B* were the same. The melting point of their eutectic mixture was unchanged: 71—72°.

N,N-*Dimethyl*-p-*(carboxymethoxy) benzenesulphonamide* (XI)

A. By condensation of I with monochloroacetic acid

Into a suspension of 3 g. (0.0148 mole) of *N,N*-dimethyl-*p*-hydroxybenzenesulphonamide (I)[18] (yield: 80%; m. p. 90—92°) in 30 ml. of water a solution of 33% sodium hydroxide was added. A clear solution (about 2 ml.) of sodium phenoxide was obtained. To that solution a solution of 5.3 g. (0.0561 mole) of monochloroacetic acid in 15 ml. water was added dropwise, under stirring and at the same time so much of a 33% solution of sodium hydroxide to keep the *p*H permanently at 7—8. The reaction temperature was kept at 40°. After the addition was completed, the temperature was raised to the boiling point for a few minutes. After

cooling to 70—80°, the reaction mixture was neutralised with hydrochloric acid (1:2), charcoal added and the mixture filtered while still warm. The warm filtrate was acidified with hydrochloric acid (1:2) to Congo Red. The reaction mixture was allowed to stand in the refrigerator overnight and gave 2.1 g. (62%) of crystals. The product was recrystallized from water or ethanol. Na salt of XI melts at 322°. An analytical sample was crystallized from ethanol; colourless rhombic prisms melting at 175.5—176.0°.

Anal. 12.54 mg. subst.: 21.31 mg. CO_2, 5.60 mg. H_2O
3.36 mg. subst.: 0.176 ml. N_2 (29°, 757 mm)
$C_{10}H_{13}NO_5S$ (259.28) calc'd.: C 46.32; H 5.06; N 5.41%
found: C 46.37; H 4.99; N 5.58%

B. By hydrolysis of X or IX

1.2 g. (0.0042 mole) of X was hydrolysed in 17 ml. of a 7% solution of sodium hydroxide, similarly as it was described for VI. Colourless rhombic prisms, m. p. 175.5—176°.

By an analogous hydrolysis of IX in dilute sodium hydroxide, crystals were collected with m. p. 175.5—176°.

The microscopic forms of the crystals obtained by method *A* or method *B* were the same. The melting point of their eutectic mixture was unchanged: 175.5—176°.

N,N-*Diethyl*-p-*(carbmethoxymethoxy) benzenesulphonamide* (XII)

Into a cooled solution of 10 g. (0.0379 mole) of III in 30 ml. of acetone a solution of 10 g. (0.1369 mole) of diethylamine in 70 ml. of ether was added dropwise. After vigorous reaction, the mixture was left to stand overnight at room temperature. The solvent was removed and the oily residue obtained extracted twice with ether. The combined extracts were dried over sodium sulphate. The solvent was evaporated and the brown syrupy residue soon crystallized to give 10.4 g. (91%) of the crude product. The product was recrystallized for analysis from 60% ethanol; colourless prisms melting at 53.5—54.0°.

Anal. 18.39 mg. subst.: 34.81 mg. CO_2, 9.98 mg. H_2O
$C_{13}H_{19}NO_5S$ (301.35) calc'd.: C 51.83; H 6.35%
found: C 51.65; H 6.07%

N,N-*Diethyl*-p-*(carboxymethoxy) benzenesulphonamide* (XIII)

A. By condensation of *N,N*-diethyl-*p*-hydroxybenzenesulphonamide (II) with ethyl ester of monobromoacetic acid

0.23 g. (0.010 mole) of sodium were dissolved in 30 ml. of absolute ethanol. When the sodium had disappeared 2.3 g. (0.010 mole) of pulverized II were added. To the alcoholic mixture 1.2 ml. (1.8 g.; 0.0107 mole) of ethyl ester of monobromoacetic acid[29] were added and refluxed on a boiling water bath for two hours. After cooling, the reaction mixture was filtered, and the filtrate boiled with charcoal. The charcoal was removed by suction and the alcohol evaporated *in vacuo*. The residue was extracted several times with absolute ether and the combined solutions purified with charcoal and evaporated. The residue was a yellow oil. It was hydrolysed with 20 ml. of a 10% solution of sodium hydroxide, as described in VI. After being acidified and cooled, the crude crystals were recrystallized from 30% ethanol. For analysis the substance was recrystallized from 30% ethanol; colourless thin prisms, melting at 146—147°; yield: 39%.

Anal. 12.62 mg. subst.: 23.21 mg. CO_2, 6.59 mg. H_2O
2.70 mg. subst.: 1.176 ml. N_2 (25°, 759 mm)
$C_{12}H_{17}NO_5S$ (287.33) calc d.: C 50.16; H 5.96; N 4.88%
found: C 50.18; H 5.84; N 4.98%

B. By condensation of II with monochloroacetic acid

From 2.3 g. (0.010 mole) of II in 10 ml. water, 3.5 g. (0.0371 mole) of monochloroacetic acid in 10 ml. water and about 8 ml. of 33% solution of sodium hydro-

xide crystals of XIII similarly as was described previously in XI were obtained. Yield: 21%. Colourless thin prisms m. p. 146—147°.

C. By hydrolysis of XII

2.6 g. (0.0086 mole) of XII was hydrolysed with 30 ml. of 10% sodium hydroxide, as previously described in VI. Colourless prisms, m. p. 146—147°. Yield: 95%

The microscopic forms of the crystals obtained by methods *A*, *B* and *C* were the same. The melting point of their eutectic mixture was unchanged: 146—147°.

Amide of p-*(carboxymethoxy) benzenesulphonamide* (XIV)

A. To a solution of 1 g. (0.0045 mole) of V in 20 ml. of warm conc. ethanol 20 ml. of 25% ammonium hydroxide were added and refluxed on a boiling water bath for 25 hours. To the reaction mixture charcoal was added, and the mixture boiled for the next 15 minutes without a reflux condensor and then filtered. Soon a product crystallized, which was recrystallized from 60% ethanol. An analytical sample was recrystallized five times from 60% ethanol; colourless needles melting at 206.5—207.8°; yield: 53%.

Anal. 8.165 mg. subst.: 12.660 mg. CO_2, 3.250 mg. H_2O
$C_8H_{10}N_2O_4S$ (230.25) calc'd.: C 41.73; H 4.37%
found.: C 42.31; H 4.45%

B. The product XIV was obtained also by refluxing of *p*-(carbethoxymethoxy)-benzenesulphonamide[28] with 25% ammonium hydroxide.

Acknowledgement. We are indebted to Mrs. E. Jaeger-Stevčevski for the microanalyses. Some microanalyses (X and XIV) were made by Mrs. M. Rill and Mrs. M. Munk-Weinert.

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IZVOD

Priprava nekih derivata *p*-hidroksibenzensulfonamida. I. O nekim *N*-alkil derivatima *p*-(karboksimetoksi)benzensulfonamida

B. Stavrić i E. Cerkovnikov

Pripravljena je serija *N*-alkil derivata općenite formule ROOC—CH_2O—C_6H_4—$SO_2NR'R''$ (*p*-) (gdje su R, R' i R'' = vodik, metil ili etil). Opisana je sinteza devet spojeva iz toga reda (V — XIII) (vidi tabelu II), kao i amida *p*-(karboksimetoksi)-benzensulfonamida (XIV). Osim toga, opisana je još i priprava nekih međuprodukata i to dva sulfoklorida (III i IV), te fenola *N,N*-dietil-*p*-hidroksibenzensulfonamida (II) (vidi tabelu I).

Priređeni spojevi su po svojoj kemijskoj konstituciji u uskoj vezi s fenoksi-octenom kiselinom, te se od njih očekuje da posjeduju izvjesno fitohormonsko djelovanje. Rezultati fitohormonskih ispitivanja bit će objavljeni posebno.

ZAVOD ZA ORGANSKU KEMIJU
FARMACEUTSKI FAKULTET
ZAGREB

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