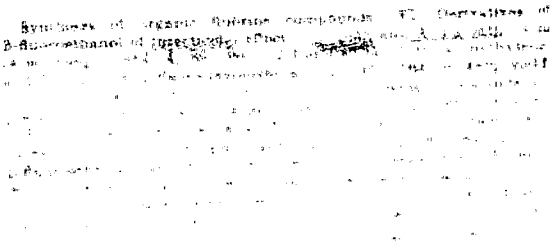
CLAH, G.



E-2

OLAH, GY.

HUNGARY/Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref Zhur-Khimiya, No 6, 1957, 19282.

Author : Olah Gy., Pavlath A., Kuhn I., Herr F.
Inst :
Title : Synthesis of Organic Fluorine Compounds. XVI. Preparation of Fluorine Derivatives of Pyribenzamine.

Orig Pub: Magyar Tud. Acad. Kem. Tud. Oszt. Kozl., 1955, 6, No 3-4, 327-330.

Abstract: Three methods of synthesis of fluorine derivatives of pyribenzamine (N,N-dimetnyl-N'-benzyl-N'-(α -pyridyl)-ethylenediamine) are developed. o-and m-fluoropyribenzamines (I and II) were obtained and their antihystamine activity was compared. In a guinea-pig, 1 γ of hystamine neutralizes the action of 0.05-0.1 γ m-fluoropyribenzamine (III), 1 γ II and 10 γ I.I, II, III are less toxic, than other haloid substituted pyribenzamines. 0.1 mole N,N-dime-

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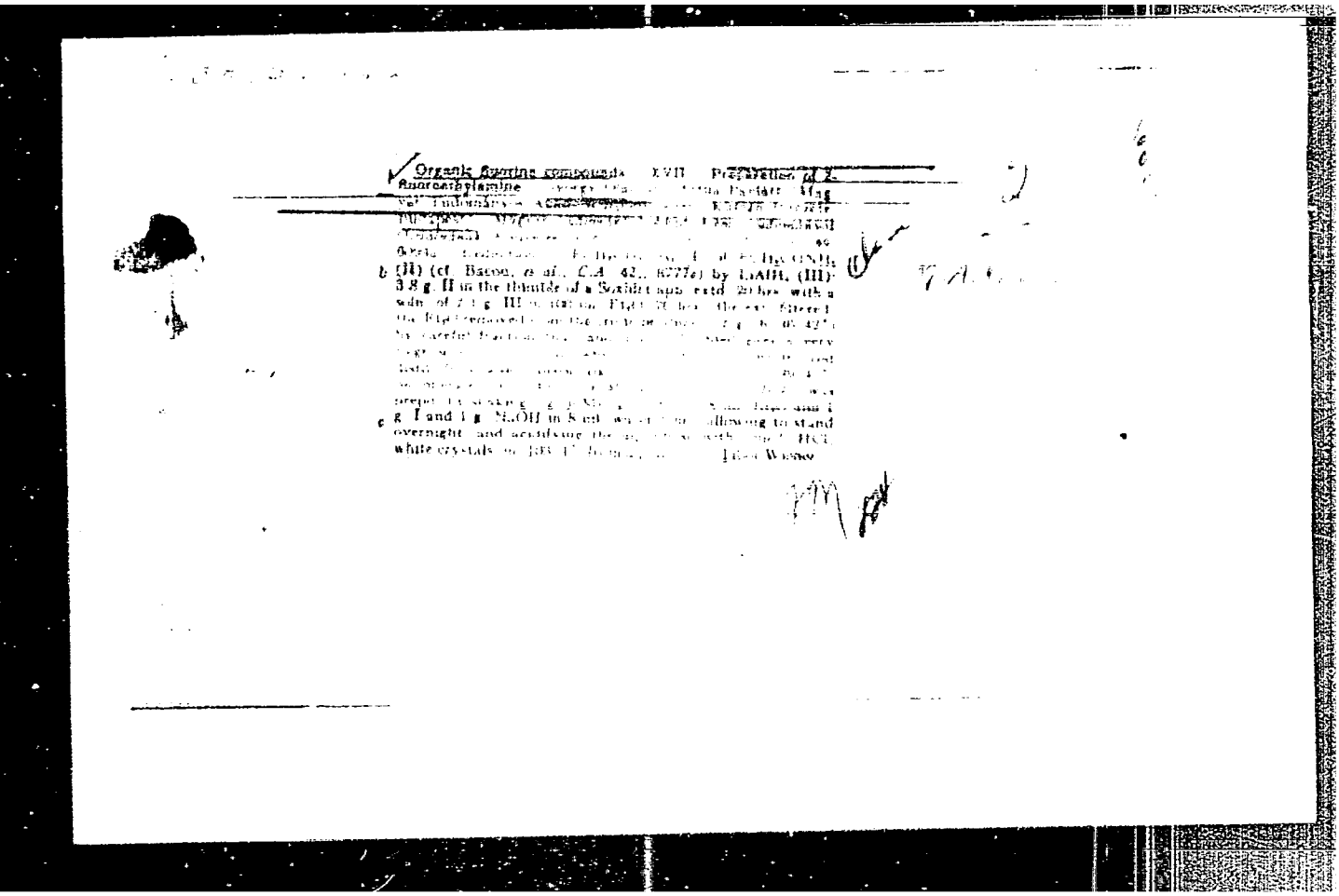
Abs Jour: Ref Zhur-Khimiya, No 6, 1957, 19282.

thylethylenediamine is boiled 12 hrs with 0.05 mole 4 - fluoropyridine (IV), from the filtrate is isolated N, N-dimethyl-N'-(4 -pyridyl)-ethylenediamine (V), yield 42.6%, b.p. 150-152°/25 mm. To a suspension of 0.1 mole NaOH in 30 cc toluene is added in drops (stirring, 105°) 0.1 mole V, boiled 2 hours, then at 50° is added in drops 0.09 mole o-$FC_6H_4CH_2Br$, on cooling 10 cc water is added and from the toluene layer and is isolated 4 g. V and I, yield 34.5%, b.p. 165-175°/3 mm; monohydrochloride, m.p. 198-200°. Analogically are obtained: from m-$FC_6H_4CH_2Br$ --II, yield 40%, b.p. 148-155°/1 mm; monohydrochloride, m.p. 145-147°; from n-$FC_6H_4CH_2Br$ --III, yield 56.2%. 0.1 mole n-$FC_6H_4CH_2NH_2$ (VI) is boiled 8 hours with 0.05 mole IV, cooled the solid mass is extracted with 200 cc n-hexane, the hot solution is filtered, and after cooling from the filtrate crystalize n-fluorobenzyl- -aminopyridine (VII); with the mother

Abs Jour: Ref Zhur-Khimiya, No 6, 1957, 19282

liquor the reaction mass is extracted two times more, yielding VII 83%, m.p. 95°. To a suspension 0.05 mole NaOH in 40 cc toluene is added in drops 0.05 mole VII, boiled with stirring for 3 hours, then at 50° is added a solution 5.4 g. $(CH_3)_2NCH_2CH_2Cl$ (VIII) in 10 cc toluene, boiled 6 hours, from the cooled off filtrate is obtained III, yield 58.8%. 0.1 mole hydrochloride VIII is boiled 2 hours with 0.2 mole VI in 200 cc toluene, shaken with 60 cc 6N NaOH, is obtained N,N-dimethyl-N'-(n-fluorobenzyl) -ethylenediamine (IX), b.p. 132-134°/12 mm, yield 32%. 0.1 mole IX boiled with 0.05 mole IVin 25 cc toluene 5 hours is shaken with 30 cc 6N NaOH is obtained III, yield 61.5%. Part XV see RZhKhim, 1956, 39629.



Organic fluorine compounds. XVII. Preparation of 2-
Anaroaniplamine

b (II) (cf. Bacon, et al., C.A. 42, 8277e) by LiAlH4 (III).
3.8 g. II in the thimble of a Soxhlet app. extd. 20 hrs. with a
soln. of 2.1 g. III in

c g. I and 1 g. NaOH in 5 ml. [illegible] allowing to stand
overnight and acidifying the [illegible] with HCl
white crystals of [illegible]

Chem. Ca

20 Synthesis of organic fluorine compounds. IX. Monomolecular reduction of nitro fluorobenzenes. Bimolecular reduction of nitro fluorobenzenes. XI. The preparation of several aromatic fluorine derivatives. [illegible]
1955 No. 1-2 pp. 65-92 3 tables

The corresponding fluoroquinoxydihydroxydiphenyls were obtained by reducing the ortho, meta or para nitro fluorobenzenes with zinc and ammonium chloride. By ferric chloride oxidation the fluorophenyl hydroxylamines were transformed into the thio-corresponding fluoro nitrosobenzene isomers. Chloronitrobenzenes were prepared by Schiemann's method; reduction by means of stannous chloride) and the fluorophenylhydrazines were obtained from the corresponding diazotized fluoroanilines by way of sulphur dioxide reduction. Investigating the bimolecular reduction of nitro fluorobenzenes to alkaline media it was found that only the meta-derivative [illegible] the fluoroazoxybenzenes or fluoro azobenzenes were obtained in extremely good yields by means of sodium arsenite. Reduction by means of zinc dust and ammonium chloride or by means of sodium amalgam in the presence of ethyl alcohol. Attempts to prepare the fluoro hydrazobenzenes by this method were unsuccessful. The investigations on aromatic fluorine derivatives included the chloromethylation of fluorobenzene. The elaboration of a simple method for the preparation of p-fluorobenzoic acid from p-chloronitrobenzene and the preparation of p-fluorobenzaldehyde and p-fluoroacetophenone by the [illegible]

OLAH, G.

Distr: 4E2c(j)

Synthesis of organic fluorine compounds. XII. Nitration of fluorobenzene. György Olah, Attila Pavlath, I. Kuhn, and Gy. Varsányi (Central Research Inst. Chem., Hungarian Acad. Sci., Budapest). *Acta Chim. Acad. Sci. Hung.* 7, 451-42(1955)(in English); cf. *C.A.* 50, 11251a; 52, 6288i.—PhF was nitrated by the following methods and the proportion of $FC_6H_4NO_2$ isomers formed detd. from the ultraviolet absorption spectra (reagents, % yield, temp. of reaction, and % *p*-, *o*-, and *m*-isomers given): 35 g. HNO_3 (sp. gr. 1.41) and 128 g. concd. H_2SO_4, 84.5, −10°, 90, 8, —; 40 g. $H_2SO_4.H_2O$, 25 g. 80% H_2SO_4, and 30 g. $NaNO_3$, 65, 60–70°, 90, 8, —; $AcONO_2$, 71.2, 0°, 90, 4, —; $BzONO_2$, 60.2, cooled, 100, —, —; $CCl_4 + N_2O_4$, 93.8, cooled, 72, 28, —; liquid N_2O_4, — (5.5 g. from 25 g. PhF), 130°, —, 78, 16, 6; N_2O_4 vapor, — (4 g. from 90 g. PhF), room temp., 90, 10. Ionic reactions give largely *p*-isomers, mixed type give 72–8% *p*- and 16–28% *o*-isomers, and the presumably radical type mechanism 90% *o*- and 10% *m*-isomers. 1,2,4-$FC_6H_3(NO_2)_2$ (I), b$_5$ 153–6°, was prepd. in 49% yield from 57 g. PhF with 70 g. $H_2SO_4.H_2O$ and 75 g. fuming HNO_3 at −5 to 0° in 24.8% yield from 101 g. 2,4-$ClC_6H_3(NO_2)_2$ in 101 g. $PhNO_2$ heated 2 hrs. at 105–10° in ultraviolet light with 20 g. dry KF, and the process repeated 3 times, and in 79.8% yield, m. 24°, from 4 g. PhF cooled and stirred with 9.5 g. N_2O_5 added in small portions. I (1 g.) added to 4 g. 60% oleum at 0° with 3 g. $H_2SO_4.H_2O$ and 3 g. fuming HNO_3, the mixt. heated to 90° 2 hrs. and to 130° 10 hrs., cooled, and poured onto ice yielded 30% *sym*-$FC_6H_2(NO_2)_3$ (II), m. 35°. Further nitration of 5 g. I with 5 g. N_2O_5 added with cooling, the mixt. warmed slowly, heated 1 hr. on a boiling H_2O bath, cooled, poured [illegible] dried in ether yielded a mixt. contg. 50% II. II [illegible] sive. Curves of the absorption spectra of [illegible] $FC_6H_4NO_2$ between 270 and 300 A. are given and [illegible] of quant. analysis by absorption spectra [illegible] 50, 70–80) completed for mixts. of [illegible] wt. XIII. Derivatives of 2-fluoro-[illegible] Olah, Attila Pavlath, and I. [illegible] cf. *C.A.* 49, 6540i, 52, 3604f.—[illegible] were [illegible] by [illegible] *urethans*, $RNHCO_2CH_2CH_2F$, were [illegible] mole $ClCO_2CH_2CH_2F$ (I) to 0.2 mole RNH_2 cooled in 50 ml. Et_2O, allowing the mixt. to stand overnight, filtering if necessary, drying the filtrate, and evapg. the Et_2O (R. cryst'g. solvent, m. p., and [illegible] (cm.): *Ph*, —, 51.5°, 58.7; *o*-MeC_6H_4, —, 74.5°, [illegible], —; *m*-MeC_6H_4, —, —, —, 171–4°, 68.6; *p*-MeC_6H_4, —, —, —, —; *p*-FC_6H_4, [illegible] hexane, 75°, 80.6; *p*-$FC_6H_4CH_2$, hexane, 56°, 94.0; $(3C_6H_4$, hexane, 64°, 90.8; *p*-BrC_6H_4, hexane, 93°, —; *p*-ICC_6H_4, hexane, 111–12° (yellow), —, —; *o*-NC_6H_4, 30% EtOH, 62° (yellow), 30% EtOH, *m*-NC_6H_4, 30% EtOH, 51–2°, 81.1; *p*-NC_6H_4, 30% EtOH, 124–5° (yellow), 88.7; *N*-phenyl-*N*-methyl, —, — (b$_{12}$ 119–20°), 7°), 92.4; *N*-phenyl-*N*-ethyl, —, — (b$_{12}$ 121–2°), 93.2; *α*-pyridyl, EtOH, 123.5°, 81.4; *β*-pyridyl, EtOH, 139°, 85.2; *γ*-pyridyl, EtOH, 75.9; *γ*-pyridyl, EtOH, 139°, 87.8; *N,N*-dimethyl, —, — (b$_{12}$ 135–40°), —, —; *N,N*-diethyl, —, — (b$_{12}$ 138–40°), —, —; *N,N*-dibenzyl, —, — (b$_{12}$ 138–40°), —, —; *N,N*-cyclohexyl (II), Et_2CO, 92°, 83.2; *N,N*-cyclopentamethylene, —, —, 121°, 85.1. Similarly prepd. were: *N,N*-diethyl, —, —, 115°, 94.2; *N,N*-bis(2-fluoroethyl), —, —, —, —; *fluoroethyldiurethan*, H_2O, —, 115°, 94.2; *N,N*-bis(chloroethyldiene)-2-fluoroethylamine, $MeCO_2$, 179°, 85.1;

Synthesis of organic ...

2-fluoroethylurethan N-(2-fluoroethyl)carboxylate, Me_2CO, 68-9°, 56.4. With esterase blocking agents (e.g. diisopropyl fluorophosphate), toxic doses of these compds. administered to animals produced no toxic symptoms. The compds. are being tested as growth inhibitors for exptl. cancerous tumors. XV. Decomposition reactions of derivatives of fluoroacetic acid. György Oláh, Attila Pavláth, and Gyula B. Major. *Ibid.* 451–3.—Fluoroacetates (I) with NH₄OH give the fluoroacetamide whose 15% H_2O soln. is stable. This soln. treated with chloride of lime (Hofmann reaction) decomp. completely. 2-Fluoroethanol (II) is also completely decompd. on alk. oxidation with chloride of lime. Biol. effects of I and II are similar. XVII. Preparation of 2-fluoroethylamine. György Oláh and Attila Pavláth. *Ibid.* 461-3.—See *C.A.* 50, 10642a.

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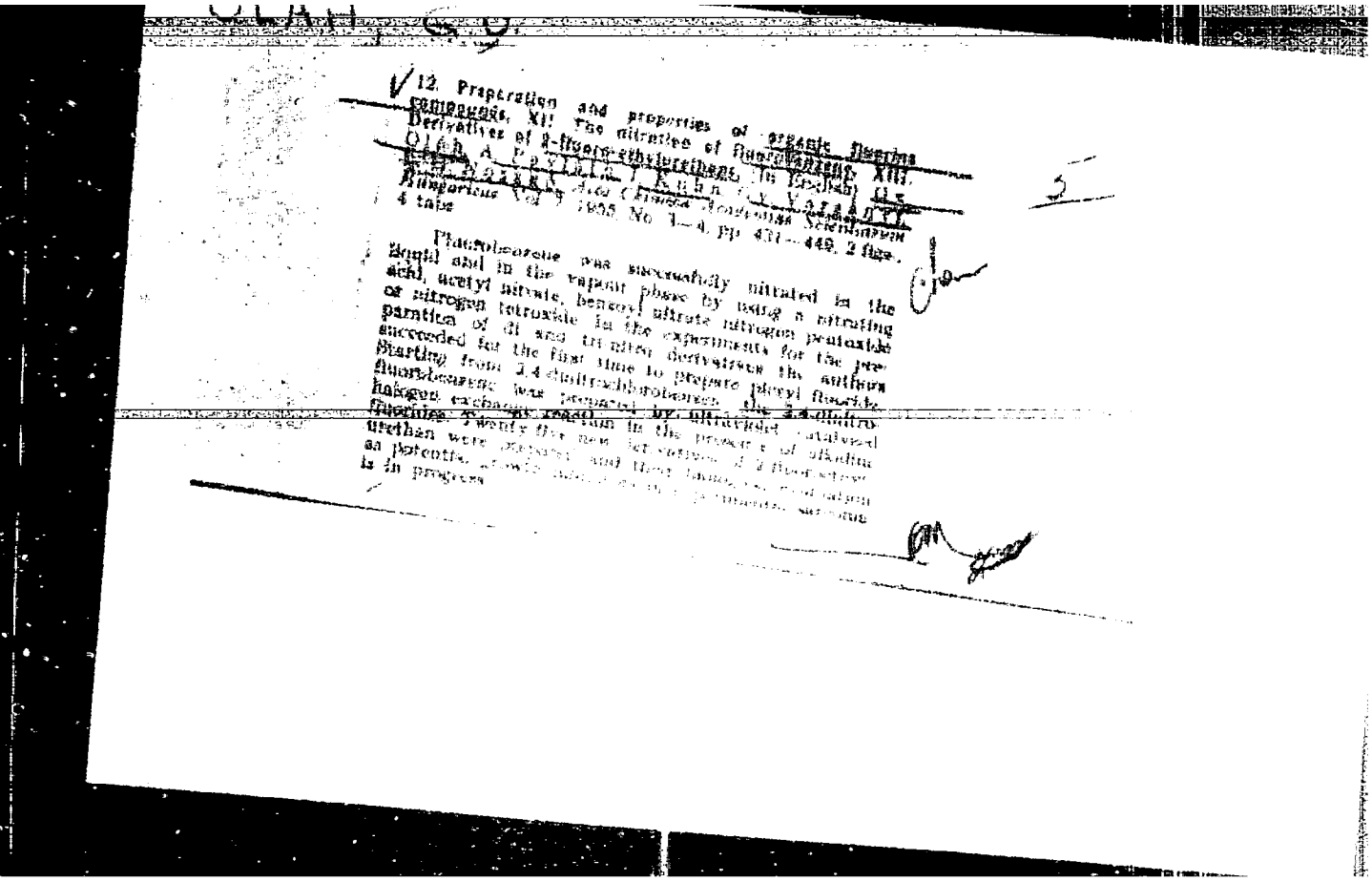

12. Preparation and properties of organic fluorine compounds. XII. The nitration of fluorobenzene. XIII. Derivatives of 2-fluoro-ethylurethane. In (with) [illegible]

[illegible] Acta Chimica Academiae Scientiarum Hungaricae (Budapest) 1955 No 1-4, pp 431-449, 2 figs., 4 tabs

Fluorobenzene was successfully nitrated in the liquid and in the vapour phase by using a nitrating acid, acetyl nitrate, benzoyl nitrate or nitrogen tetroxide in the experiments for the preparation of di and trinitro derivatives the authors succeeded for the first time to prepare pheryl fluoride. Starting from 2,4-dinitrochlorobenzene, the 2,4-dinitrofluorobenzene was prepared by nitrated dinitro-halogen exchange was prepared by nitrated 2,4-dinitro-fluorine. Twelve [illegible] in the presence of palladium urethan were prepared and their [illegible] as potential [illegible] is in progress

OLAH, GY.

E-2

HUNGARY/Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref Zhur-Khimiya, No 6, 1957, 19281.

Author : Olah Gy., Pavlath A., Major Gy B.
Inst :
Title : Synthesis and Investigations of Fluororganic Compounds. XV. Decomposition of Fluoroacetic Acid Derivatives.

Orig Pub: Acta chim. acad. sci. Hung., 1955, No 3-4, 451-453. See RZhKHim. 1956, 39629

Abstract: No abstract.

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Synthesis of organic fluorine compounds. IX. Bimolecular reduction of nitrofluorobenzenes. Gyula Oláh, Attila Pavláth, and István Kuhn (Hung. Acad. [illegible]). Acta Chim. Acad. Sci. Hung. 7, [illegible] (1955) (in English). cf. C.A. 49, [illegible]. —(Unless otherwise stated, the isomers in the following series of compds. are given in the order o-, m-, and p-.) A vigorously stirred suspension of [illegible] g. $FC_6H_4NO_2$ (I) in 250 ml. H_2O treated with [illegible] g. NH_4Cl, [illegible] g. pulverized Zn added over a 15-min. interval, [illegible] flask at [illegible]°, filtered [illegible] FC_6H_4NHOH [illegible] from gasoline, [illegible]° from C_6H_6, 80° (from C_6H_6 [illegible] g. and [illegible] g. [illegible] in [illegible] ml. H_2O treated with [illegible] g. pulverized Zn as above, the filtrate stirred into a soln. of [illegible] g. $FeCl_3$ in [illegible] g. ice, allowed to stand, and the yellow [illegible] removed from [illegible] and steam distd. In a Pregl-Faraday [illegible] gave white cryst. FC_6H_4NO, m. [illegible]°, 31°, [illegible]°. The $FC_6H_4NH_2$ (II), b. [illegible]°, [illegible] 187°, [illegible] [illegible]°, [illegible]°, [illegible] were prepd. by the Schiemann-Pil[illegible] method [illegible] C.A. 34, 1827[illegible]. II HCl [illegible] g. in [illegible] ml. [illegible] H_2SO_4 and 15 ml. H_2O diazotized with [illegible] g. $NaNO_2$ in [illegible] ml. H_2O, dild. with 1000 ml. [illegible] satd. with SO_2, [illegible] allowed to stand 1 hr., evapd. on a [illegible] bath, the residue [illegible] refluxed with 150 ml. EtOH, filtered [illegible] and allowed to stand [illegible] yielded $FC_6H_4NHNH_2.HCl$. The free bases were liberated from an aq. soln. of the HCl salt, the soln. treated with Et_2O, dried with K_2CO_3 and distd. in vacuo to give the base. $FC_6H_4NHNH_2.HCl$, m. 198 [illegible]°, 214 [illegible]°, [illegible]°; $FC_6H_4NHNH_2$, b[illegible] 80-81°, m. [illegible]° [illegible] needles, b[illegible] [illegible]°, m. 25.6°, light-sensitive crystals, b[illegible] [illegible]°, m. 48-[illegible]°. X. Bimolecular reduction of nitrofluorobenzenes. Ibid. [illegible] NaOH (0.35 mole) in [illegible]

Olah, Gyorgy

ml. H_2O added to 0.1 mole $PC_6H_4NO_2$ (I) in 15 ml. EtOH, the cooled mixt. stirred, 20 g. pulverized Zn added in small portions through a closed feed device, then 170 ml. EtOH, the mixt. kept on a hot H_2O bath 5 min., filtered, and the filtrate allowed to stand deposited (*p*-$EtOC_6H_4N{:}N$)$_2$, yellow lamellar crystals, m. 102°; (*m*-$PC_6H_4N{:}N$)$_2$ (II), orange lamellar crystals, m. 65.5°, and (*o*-$PC_6H_4N{:}N$)$_2$, m. 140–50°. To 0.1 mole treated with 0.033 mole As_2O_3 in a soln. of 0.2 mole NaOH in 10 ml. H_2O with vigorous stirring, the brown mixt. refluxed 5 hrs., cooled, filtd. with H_2O, filtered, and the product recrystd. from EtOH gave (*p*-$FC_6H_4N{:}NO$ (III), m. 58°, (*m*-$FC_6H_4N{:}NO$ (IV), yellow, m. 51°, acicular leaflets of the filtrate from o-I gave (*o*-$FOC_6H_4N{:}NO$ (V), m. 153°. NaOH (15 g.) in 60 ml. H_2O added to 0.082 mole I, the mixt. stirred 30 min. at 45–50°, treated with 0.06 mole glucose in 30 min., heated 30 min. on a H_2O bath, steam distd., then refrigerated, and the ppt. filtered and recrystd. from EtOH gave III, IV, and V, resp. *p*-$FC_6H_4NH_2$·HCl diazotized, 9 g. AcONa and 3 g. aniline added, and the mixt. allowed to stand overnight gave yellow *p*-$FC_6H_4N{:}NPh$ (VI), m. 69°. *p*-$FC_6H_4NH_2$·HCl (2.3 g.) added to 4.25 g. VI in 15 g. aniline, the soln. stirred 1 hr. on the H_2O bath at 52°, cooled, treated with 30 ml. 50% AcOH, and allowed to stand 15 min. gave red crystals of *p*-fluoro-*p*'-aminoazobenzene (VII), m. 220°. VII treated by the Sandmeyer method gave orange (*p*-$FC_6H_4N{:}N$)$_2$C$_6$H$_4$ (VIII), m. 110° (from EtOH). Pulverized Mg (3 g.) added to 5 g. I in 250 ml. abs. MeOH, the soln. refluxed 3 hrs. after the initial reaction subsided, boiled 1 hr. after the Mg had completely dissolved, most of the MeOH removed

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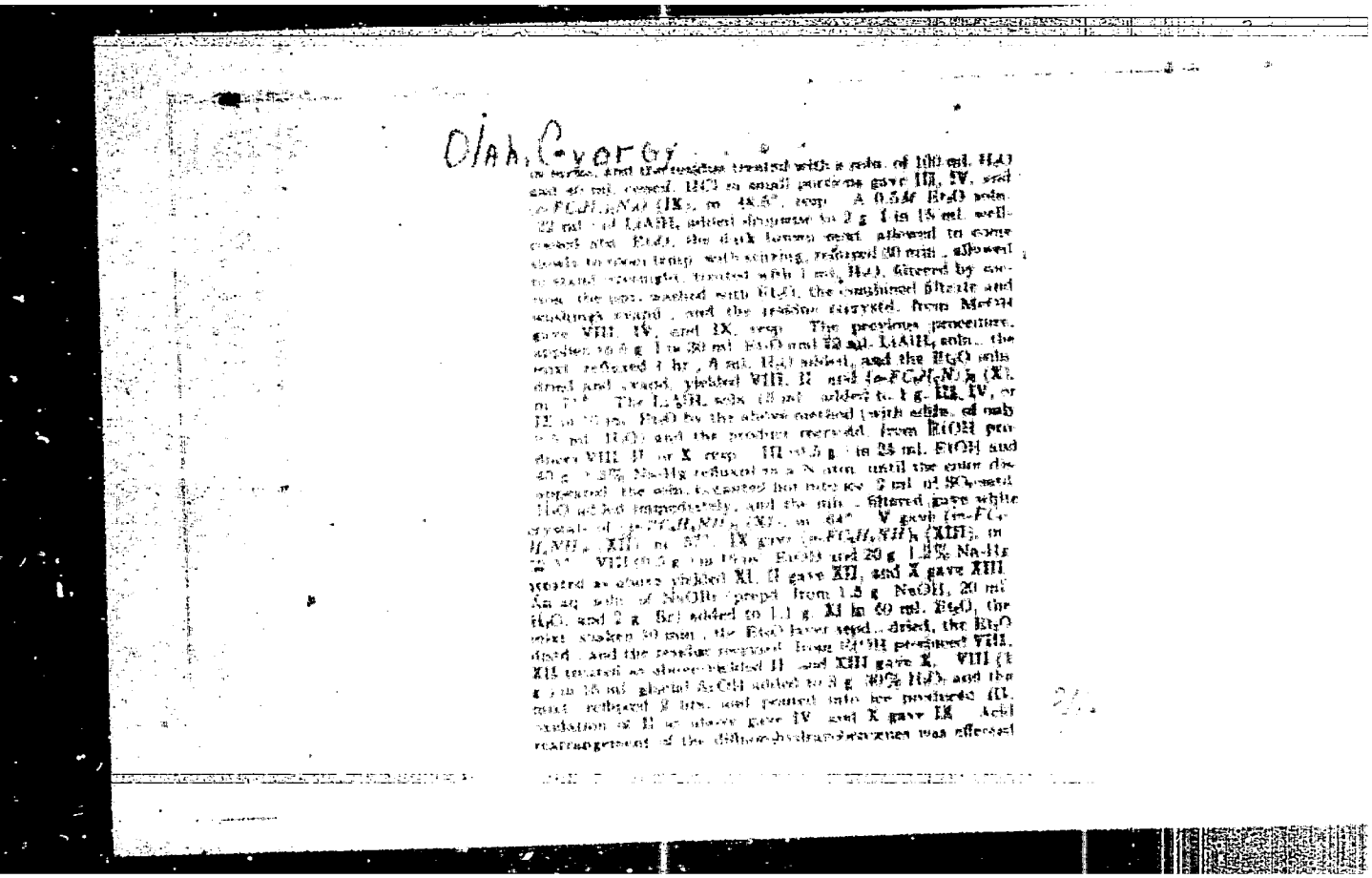

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Olah, Gyorgy

as follows: 1 g. XII, 5 ml. H_2O, and 8 ml. concd. HCl was heated 30 min. on a H_2O bath, then to boiling, cooled, the ppt. HCl salt filtered by suction, and the 2,4-$F(H_2N)C_6H_3$, m. 114–15°, liberated by adding 20 ml. 20% NaOH and filtering. Treatment of XIII as above produced 2,4-$F(H_2N)C_6H_3$, m. 105°, from XI no benzidine was obtained but repeated recrystn. gave some VIII, and steam distn. gave p-$FC_6H_4NH_2$. All m.ps. are uncor. On the basis of exptl. results a hypothetical mechanism of reaction for the reduction of I with $LiAlH_4$ is developed as a simple S_N2 type. XI. Preparation of several aromatic fluorine derivs. [illegible] Dry HCl fed 2 hrs. into a stirred mixt. of 100 g. PhF (I), 15 g. paraformaldehyde, and 15 g. anhyd. $ZnCl_2$, and the org. phase sepd., washed with H_2O, a 5% soln. of $NaHCO_3$, and again H_2O, dried with $CaCl_2$, and fractionated in vacuo gave p-$FC_6H_4CH_2Cl$ (II), b_{20} 89–90°, also obtained from 197 g. I, 50 g. paraformaldehyde, and 50 g. $ZnCl_2$ refluxed at 60°, dry HCl introduced for 8 hrs., and the org. layer sepd. after 1 hr. and treated as above. $(p$-$FC_6H_4)_2CH_2$, b. 133–4°, was also obtained. Boiling p-FC_6H_4Me (11 g.) irradiated with ultraviolet light in the presence of 0.2 g. PCl_5 and fractionated as above gave p-$FC_6H_4CCl_3$ (III), b_{20} 95–7°. II chlorinated in the presence of PCl_5 as described above gave III, b_{20} 98–101°. III (8 g.) refluxed 3 hr. with 8 g. KOH in 20 ml. H_2O, dild. with 30 ml. H_2O, acidified hot by a few drops of concd. HCl in the presence of Congo red, filtered hot, and allowed to cool gave white needles of p-$FC_6H_4CO_2H$ (IV), m. 182–3° (from hot H_2O). NaCN (40 g.) in 50 ml. H_2O and 65 ml. BuOH treated with 68 g. II dropwise with stirring in 30 min., the mixt. refluxed and stirred 3 hrs., cooled, filtered, the filtrate

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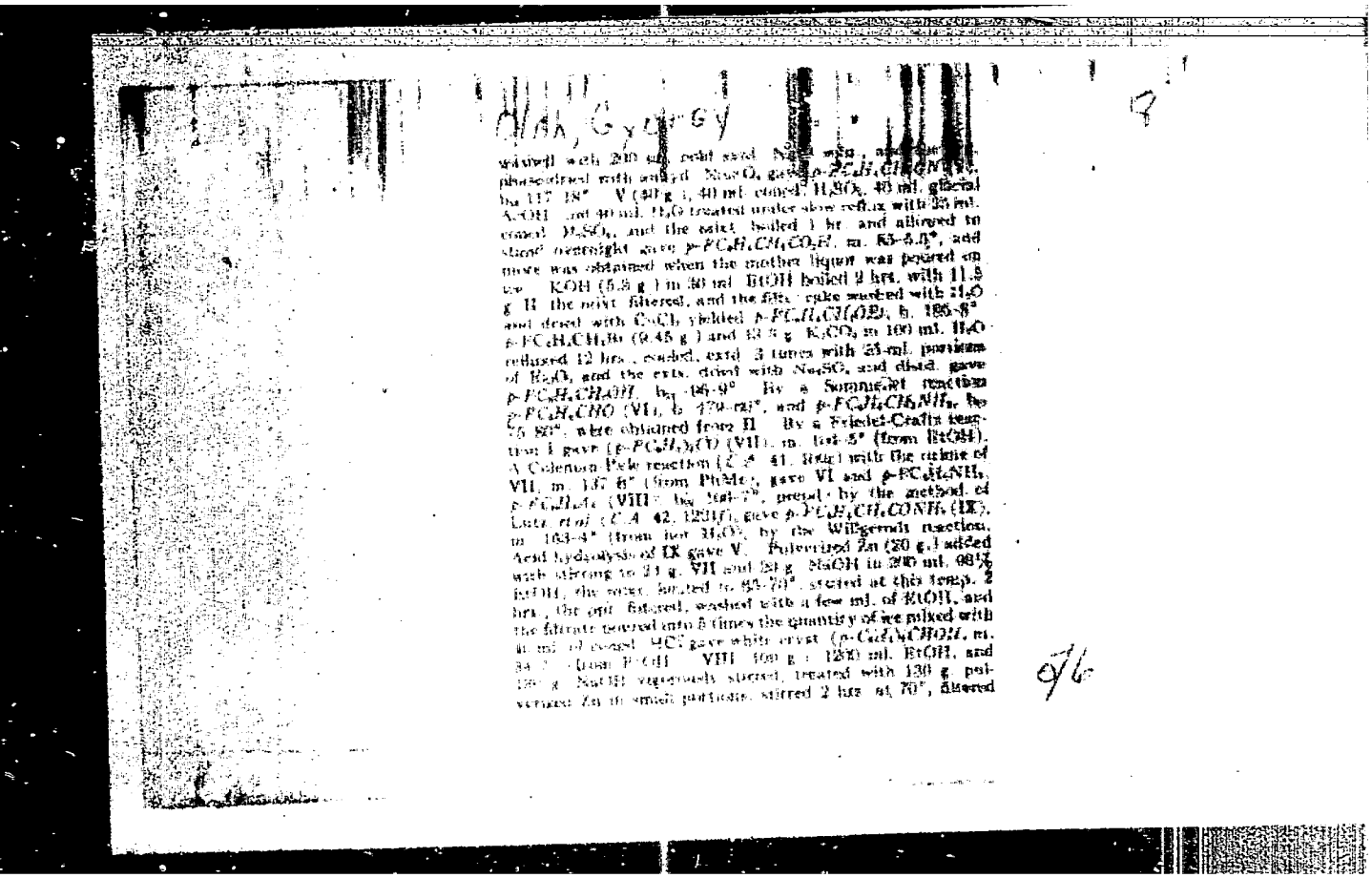
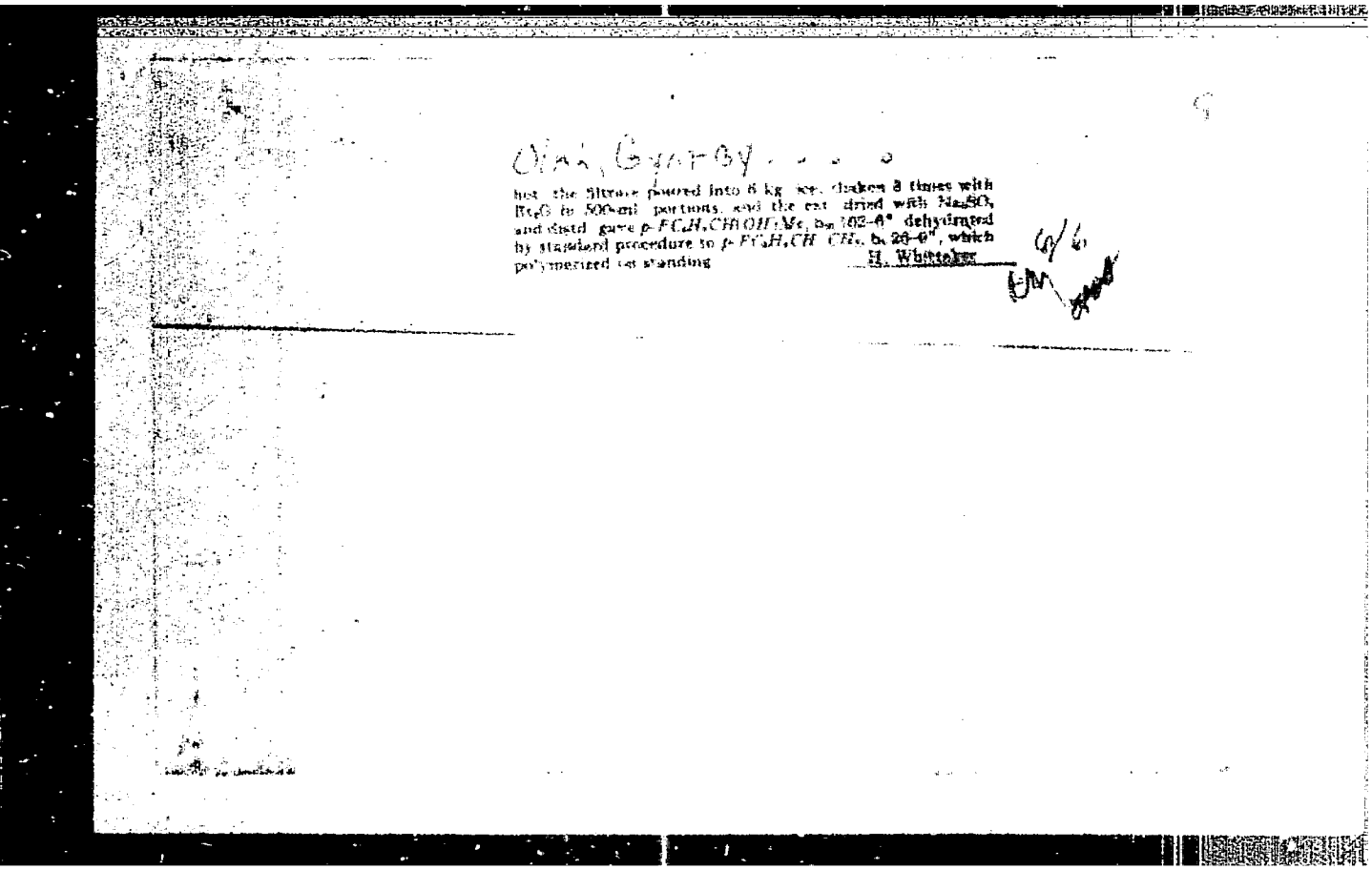
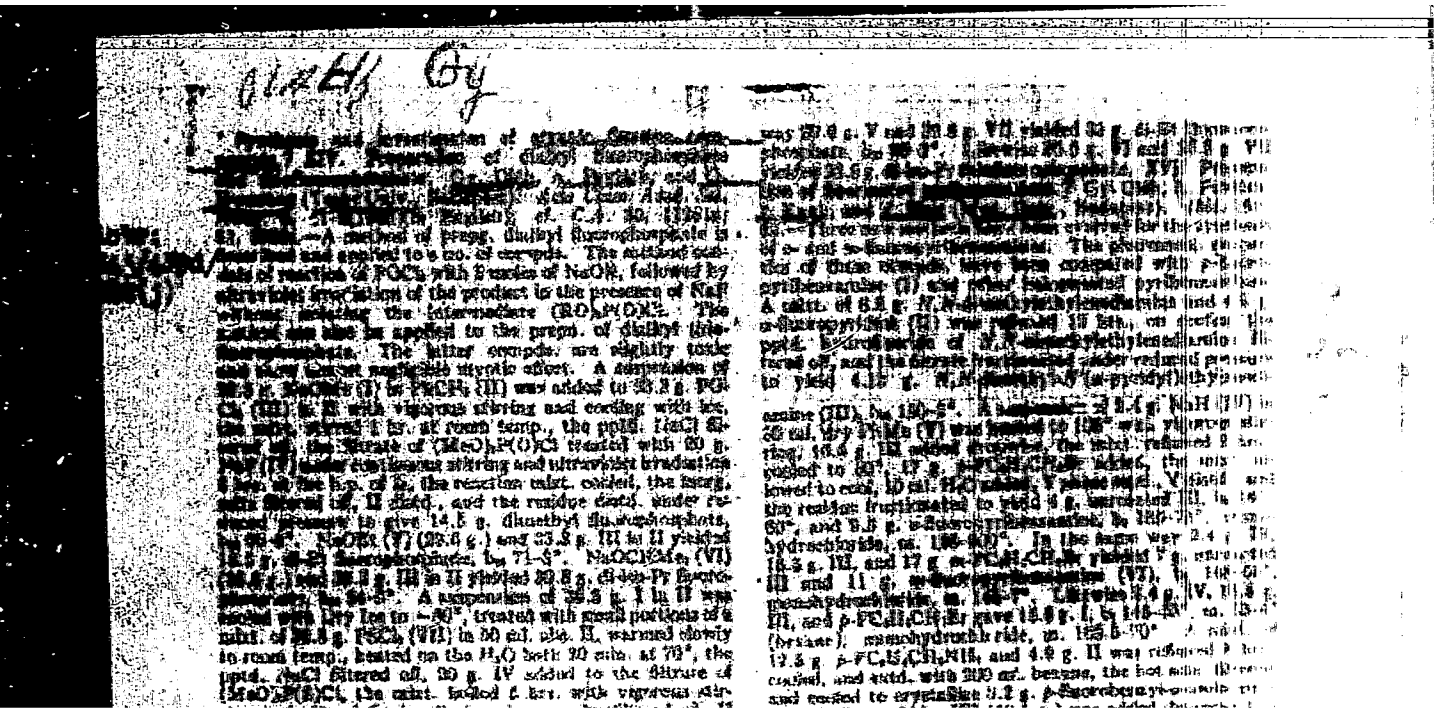


washed with 200 ml. cold satd. Na_2CO_3 soln., and the [illegible] phosphorized with satd. Na_2O_2 gave *p*-$FC_6H_4CH_2CN$ (V), bp. 117–18° V (40 g.), 40 ml. concd. H_2SO_4, 40 ml. glacial AcOH, and 40 ml. H_2O treated under slow reflux with 20 ml. concd. H_2SO_4, and the mixt. boiled 1 hr. and allowed to stand overnight gave *p*-$FC_6H_4CH_2CO_2H$, m. 85–6.5°, and more was obtained when the mother liquor was poured on ice. KOH (5.5 g.) in 30 ml. EtOH boiled 2 hrs. with 11.5 g. II the mixt. filtered, and the filter cake washed with H_2O and dried with $CaCl_2$ yielded *p*-$FC_6H_4CH(OEt)_2$, b. 105–8° *p*-$FC_6H_4CH_2Br$ (9.45 g.) and 12.5 g. K_2CO_3 in 100 ml. H_2O refluxed 12 hrs., cooled, extd. 3 times with 25-ml. portions of Et_2O, and the extn. dried with Na_2SO_4 and distd. gave *p*-$FC_6H_4CH_2OH$, bp. 96–9° By a Sommelet reaction *p*-FC_6H_4CHO (VI), b. 179–81°, and *p*-$FC_6H_4CH_2NH_2$, bp. 85°, were obtained from II By a Friedel-Crafts reaction I gave (*p*-$FC_6H_4)_2CO$ (VII), m. 101–5° (from EtOH). A Colemun-Pelc reaction (*C. A.* 41, [illegible]) with the oxime of VII, m. 137–8° (from PhMe), gave VI and *p*-$FC_6H_4NH_2$. *p*-FC_6H_4Ac (VIII), bp. 98–9°, prepd. by the method of Lutz *et al.* (*C. A.* 42, 1201f), gave *p*-$FC_6H_4CH_2CONH_2$ (IX), m. 163–4° (from hot H_2O), by the Willgerodt reaction. Acid hydrolysis of IX gave V. Pulverized Zn (20 g.) added with stirring to 21 g. VII and 24 g. NaOH in 200 ml. 95% EtOH, the mixt. heated to 65–70°, stirred at this temp. 2 hrs., the ppt. filtered, washed with a few ml. of EtOH, and the filtrate poured into 5 times the quantity of ice mixed with 40 ml. of concd. HCl gave white crystals (*p*-$ClC_6H_4)_2CHOH$, m. 84.5–5° (from PhH) VIII (60 g.) 120 ml. EtOH, and 120 g. NaOH vigorously stirred, treated with 130 g. pulverized Zn in small portions, stirred 2 hrs. at 70°, filtered



but the filtrate poured into 6 kg. ice, shaken 3 times with Et_2O in 500-ml. portions, and the ext. dried with Na_2SO_4 and distd. gave *p*-$FC_6H_4CH(OH)Me$, b$_{20}$ 102-6°, dehydrated by standard procedure to *p*-$FC_6H_4CH{:}CH_2$, b$_{20}$ 26-9°, which polymerized on standing.

H. Whitaker



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By: Dilthey, W. Hurdum

with stirring, cooled to 30°, [illegible] the mixture refluxed 6 hrs., cooled, the ppt. filtered off, V dried to give 8.1 g. I, b. 143-53°. A mixt. of 14.4 g. N,N-dimethylaminomethyl chloride hydrochloride and 25 g. VI in 200 ml. V was refluxed 2 hrs., cooled, shaken with 60 ml. 6N NaOH, V phase sepd., dried with Na_2SO_4, V distd., and the portion fractionated to give 9.1 g. N,N-dimethyl-N'-(o-chlorobenzyl)ethylenediamine (VIII), bp 122-4°. VIII (10.6 g.) and 4.9 g. II in 25 ml. V was refluxed 5 hrs., cooled, shaken with 30 ml. 6N NaOH, V phase sepd., dried over Na_2SO_4, and fractionated to yield 8.2 g. I, bp 135-5°.

A. R. Rice

2 May 3

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Ohab, G.

Nitryl perfluoride as a nitrating agent. G. Olah and S. Kuhn (Hung. Acad. Sci., Budapest, [illegible]). *J. Org. Chem.* 26, 237-8 (1961). [illegible] were nitrated with nitryl fluoride (O_2NBF_4) to the corresponding (I) according to the reaction [illegible] + O_2NBF_4 → [illegible] + HF + BF_3. I, which has a [illegible] carbon was prepd. from C_6H_6 and HF, or from [illegible] and BF_3, and was stable at room temp. for a long time. M. K. Reger

OLAH, G. (Dr.)

Hungary

Neure Entwicklung auf dem Gebiet der Theorie und Praxis der Hoch[illegible]

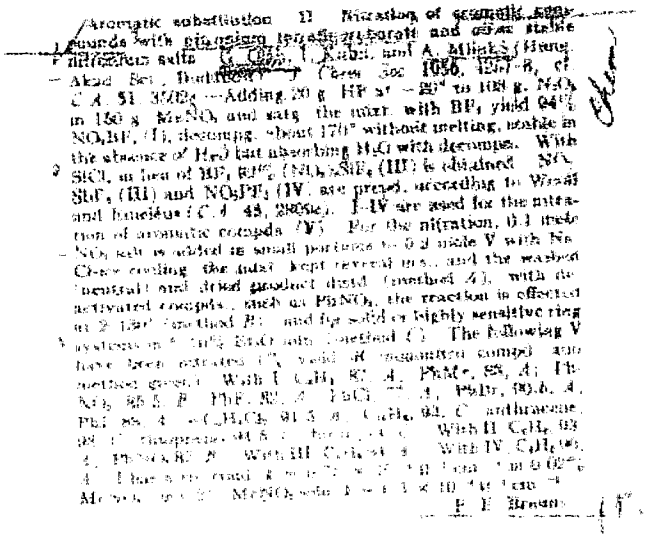
(Hauptjahrestag[illegible] Republik).

Aus Dem Tagungsprogramm - Nachmittags : Gruppe C:

Dr. G. OLAH, Budapest,"Über den Mechanismus der mit BF-BF_3 [illegible] Polymerization der Olefine. Beitrage zur Theorie der [illegible] Athylens."

SOURCE: Plaste und Kautschuk, October 1956, Unclassified.

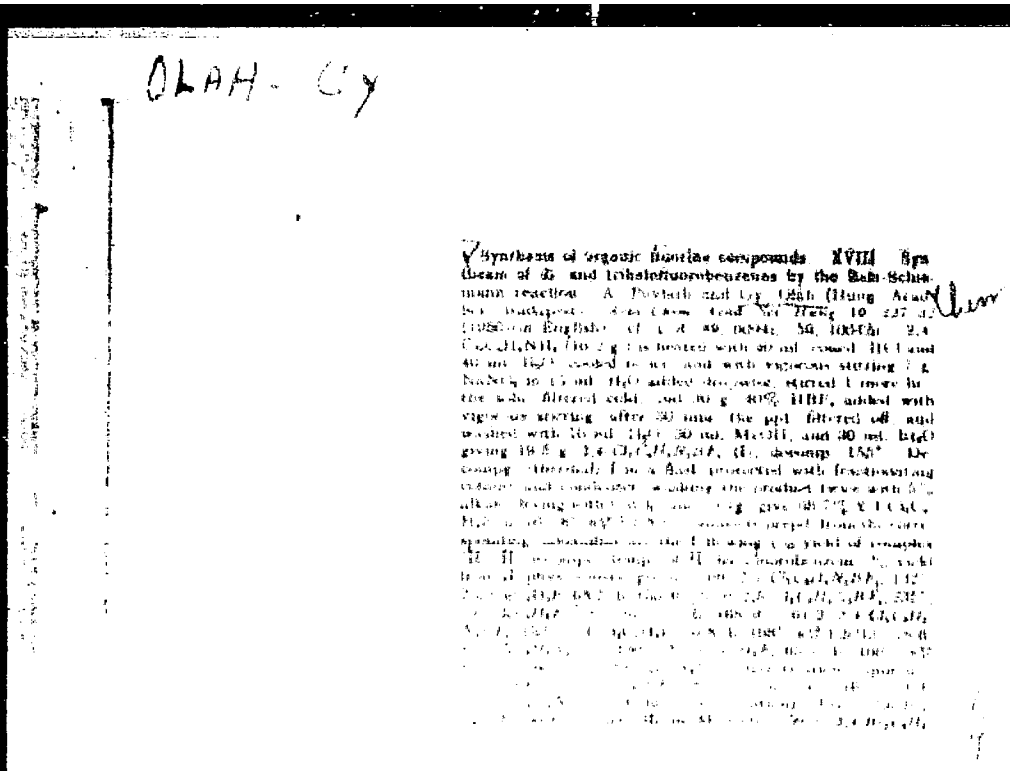
Olah, G.



Aromatic substitution. II. Nitration of aromatic compounds with nitronium tetrafluoroborate and other stable nitration salts. G. Olah, T. Kuhn, and A. Mlinko, Hung. Acad. Sci., Budapest. Chem. Soc. 1956, 1254–8; cf. C.A. 51, 3028i.—Adding 20 g. HF at −20° to 100 g. NO_2F in 150 g. $MeNO_2$ and satg. the mixt. with BF_3 yield 94% NO_2BF_4 (I), decomps. about 170° without melting, stable in the absence of H_2O but absorbing H_2O with decomps. With SiO_2, in lieu of BF_3, 83% $(NO_2)_2SiF_6$ (II) is obtained. NO_2SbF_6 (III) and NO_2PF_6 (IV) are prepd. according to Wiesl and Jander (C.A. 45, 2805e). I–IV are used for the nitration of aromatic compds. (V). For the nitration, 0.1 mole NO_2 salt is added in small portions to 0.1 mole V with $MeNO_2$ or CH_2Cl_2 cooling the mixt. kept several hrs., and the washed (neutral) and dried product distd. (method A), with deactivated compds., such as $PhNO_2$, the reaction is effected at 2–150° (method B); and for solid or highly sensitive ring systems in CH_2Cl_2 soln. (method C). The following V have been nitrated (% yield of mononitro compd. and method given): With I C_6H_6, 87, A; $PhMe$, 88, A; $PhNO_2$, 85.5, B; PhF, 82, A; $PhCl$, —, A; $PhBr$, 90.6, A; PhI, 88, A; o-$C_6H_4Cl_2$, 91.5, A; C_6H_5I, 93, C; anthracene, 99, C; naphthalene, 91.5, C; benzil, —, C. With II C_6H_6, 93, A; $PhNO_2$, 82, B. With III C_6H_6, 94, A. With IV C_6H_6, 95, A; $PhNO_2$, 85, B. [illegible] $MeNO_2$ soln. [illegible]

E. E. Braun

OLAH - C

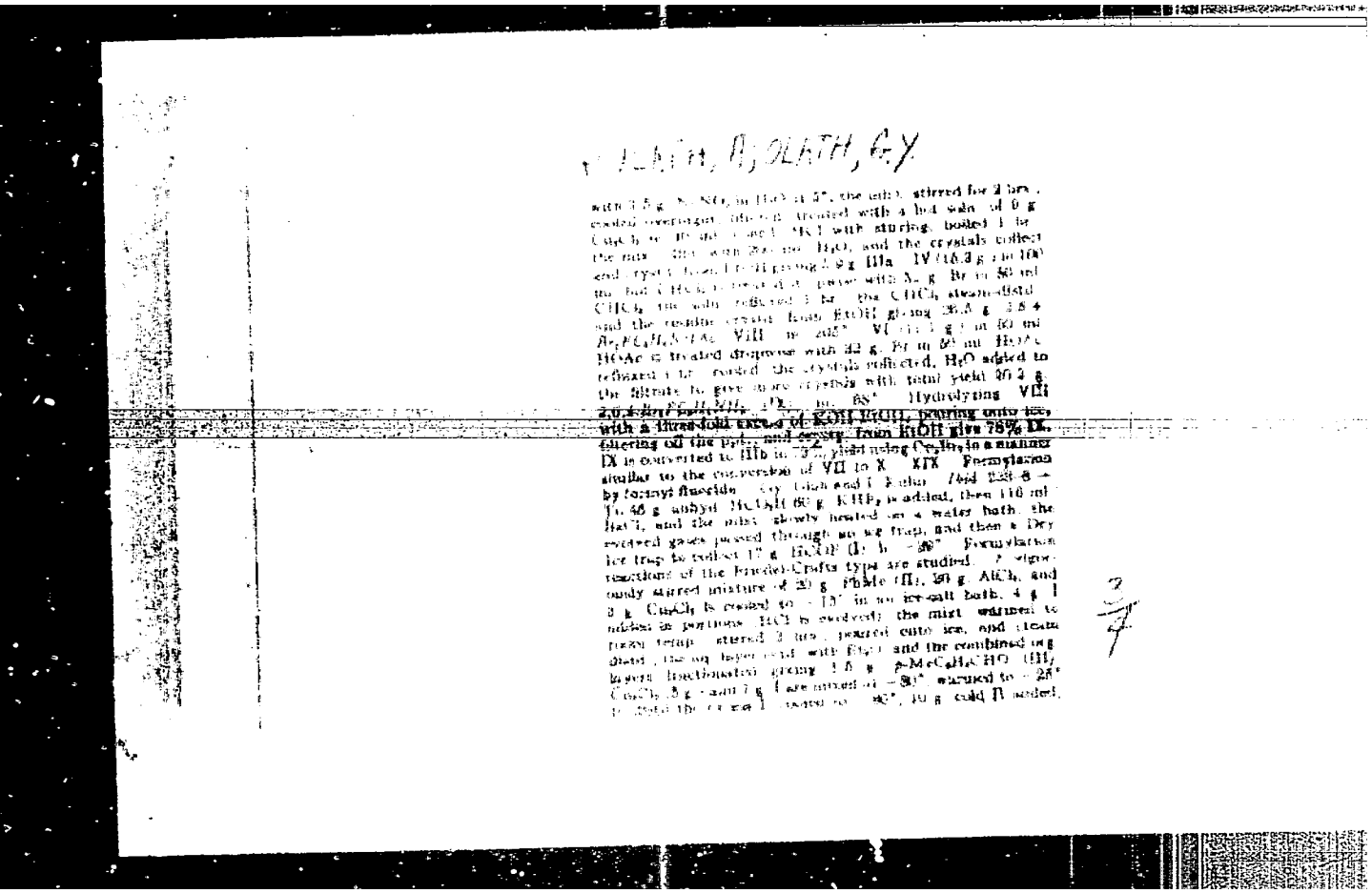


Synthesis of organic fluorine compounds XVIII. Synthesis of di- and trifluoromethylbenzenes by the Balz-Schiemann reaction. A. Furdik and Gy. Olah (Hung. Acad. Sci., Budapest). Acta Chim. Acad. Sci. Hung. 10, 237-42 (1956) (in English). Cf. C.A. 49, 10844i. 2,4-$C_6H_3(NH_2)_2$, 10.8 g., is heated with 40 ml. conc. HCl and 40 ml. H_2O, cooled to 0°, and with vigorous stirring 7 g. $NaNO_2$ in 15 ml. H_2O added dropwise, stirred 1 more hr. the solid filtered, and 30 g. 40% HBF4 added with vigorous stirring after 30 min. the ppt. filtered off, and washed with 30 ml. HCl, 30 ml. MeOH, and 30 ml. Et_2O, giving 16.5 g. 2,4-$(CF_3)_2C_6H_3N_2BF_4$, dec. decomp. 155°. Decomp. thermally in a flask provided with fractionating column and condenser, shaking the product twice with 5% alkali, drying with $CaCl_2$, and distg. gave 68.2% 2,4-$(CF_3)_2C_6H_3F$, n_D^{20} 1.4 [illegible] purified from the corresponding chlorides via the Beilstein test. 2,4-$(CF_3)_2C_6H_3F$, b. 112° [illegible]

PAVLATH A., LAH, G.Y.

2,4,5-(or 2,4,6-dimethoxy)-3(6)-, 2,4-Br$_2$C$_6$H$_2$F, 86.8, by
mp 1 mg + 5[illegible] 2,4,5-(C$_6$H$_5$)$_2$C$_6$H$_2$NH$_2$ (II) 15.7 g is heated to
boiling with 60 ml. conc. HCl and 40 ml. H$_2$O, cooled to
0°, and 7 g. NaNO$_2$ in 25 ml. H$_2$O added dropwise, the mixt.
stirred for 6 hrs. let stand overnight in the cold, filtered,
the filtrate treated with 80 g. 40% HBF$_4$, let stand 1 hr.
the ppt. filtered off, and washed with 10 ml. ice H$_2$O, 30 ml.
MeOH and 30 ml. Et$_2$O, giving 16.5 g. 2,4,5-(C$_6$H$_5$)$_2$C$_6$H$_2$N$_2$BF$_4$
(III), decomp. 170°. Thermal decompn. of III in a flask
protected by a condenser, rectn. of the product with Me$_2$CO,
clarification, evapn. of the Me$_2$CO, and cryst. from EtOH

gave [illegible] 2,4,5-(C$_6$H$_5$)$_3$C$_6$HF$_2$ (IIIa), 68.6, m. 82°. Similarly
prepd. from the corresponding tribromoanilines are (data re-
corded as above): 2,4,5-Br$_3$C$_6$H$_2$NH$_2$, 200°; 2,4,5-
Br$_3$C$_6$HF$_2$, 96.6, m. 28°, bp$_{0.1}$ 101°; 87.4, 2,4,6-(C$_6$H$_5$)$_3$C$_6$H$_2$N$_2$BF$_4$
(IIIb), 68.6, m. 82°; 52.1 2,4,5-
[illegible] 2,4,5-Br$_3$C$_6$H$_2$F, 90.6, m. 41°; 87.7,
[illegible] 2,4,5-Br$_3$C$_6$H$_2$F, 45.8, m. 30°;
[illegible] 2,4,6-Br$_3$C$_6$H$_2$F, 60.4, m.
[illegible] 2,4,6-Br$_3$C$_6$H$_2$N$_2$BF$_4$, 198°; 2,4,6-Br$_3$C$_6$H$_2$N (IIIb),
[illegible] m. 80°; p-(C$_6$H$_5$)$_2$NHAc (V), 15.5 g. in 100 ml. not
[illegible] treated with the Cl from 9 g. KClO$_3$, the CHCl$_3$
removed by distn., the cooled residue filtered off, and re-
crystd. from EtOH giving 17 g. 2,6,4-Cl$_2$FC$_6$H$_2$NHAc (VI),
m. 193°. Similar chlorination of p-FC$_6$H$_4$NH$_2$ (VI, gives
22.7% 2,6,4-Cl$_2$FC$_6$H$_2$NH$_2$ (VII), m. 43°. Treating VII with
a five-fold excess of Ac$_2$O and pouring the mixt. into H$_2$O
gave V. Hydrolyzing V with a three-fold excess of KOH-
EtOH, pouring the mixt. onto ice, filtering off, and cryst.
from EtOH gave 70% VII. VII (9 g.) is heated to boiling
with 20 ml. concd. HCl and 20 ml. H$_2$O, cooled, diazotized



PAVLATH, A.; OLATH, G.V.

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the reaction mixt. said with BF_3, warmed to room temp. in 6 hrs., steam distd., and the distillate treated as above giving 2 g. III. I (3 g.) is said with BF, at −80°, 5 g. acid II added, the mixt. warmed to room temp. overnight, poured onto ice, said with Et_2O, and the Et_2O soln. distd., giving 1.5 g. III. To 10 g. BF, cooled in liquid air, 7 g. I (previously cooled to −100°) is added, the mixt. warmed to −110° to remove the excess BF, and the solid melted, giving 17 g. I-BF, complex (IV), m. −112°. Melted IV has a high BF, tension, and decomp. above −80°. IV (1.75 g.) is decomped with excess solid, said soln. of K_2SO_4 giving HCO_2H, H_2SO_4, and KBF. II is added to 5 g. IV at −80°, let stand for 5 hrs., the soln. warmed to room temp. slowly, stored overnight, said with Et_2O after an ice hydrolysis, the Et_2O soln. dried and fractionated to give 2 g. III. Similarly IV is treated with MeOPh to give a small amt. of p-$MeOC_6H_4CHO$, PhOH, and polymers, with alc. MeOH giving 58% HCO_2Me, with PrOH giving 60% HCO_2Et, and with PrOH giving 47% HCO_2Pr. To an Et_2O soln. of 5 g. MeMgI cooled to −70° is added 5 g. I in the cold, warmed to 0° in stand 3 hrs., the mixt. cooled to −20° and decomped. with H_2O, and the org. layer sepd. and distd. [illegible] g. AcH. Similarly I reacts with PhMgBr giving [illegible]

H. S. Nutter

OLAH, G.

4

84. Synthesis and investigation of organic fluorine compounds XIX. Formylation by formyl fluoride. (In English) By: Olah, G. Kuhn, S. Acta Chimica Academiae Scientiarum Hungaricae Vol. 10, 1956, No. 1—3, pp 133—136

On investigating Friedel-Crafts type formylation reactions which can be carried out directly with formyl fluoride, it has been demonstrated that boron trifluoride is the most adequate catalyst. Reaction of boron trifluoride with formyl fluoride at liquid air temperature yielded a formyl-borofluoride complex. As shown by its reactivity this complex appeared to be an $-F\cdots BF_3$ coordination complex which has the advantage of easily splitting off formonium cations. The structure of the complex is represented by the formula $(CHO)-BF_4$. With the aid of this complex various aromatic compounds (such as toluene, anisole) could be directly formylated. From the reaction of the complex with alcohols the corresponding alkyl formates were obtained, whereas formyl fluoride treatment of alkyl or aryl magnesium halogenides resulted in the formation of the corresponding aldehydes

FM

OLAH, G.

HUNGARY/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref. Zhur.-Khimiya, No II, 1958, 36288.

Author : Olah G., Kuhn I.
Inst : Not given.
Title : Synthesis and Investigation of Fluorganic Substances. XXI. Synthesis of Acetaldehydefluoride and of Aliphatic Fluoromethylketones.

Orig Pub: Magyar tud. akad. Kem. oszt. lozl., 1956, 7, No 3-4, 481-483.

Abstract: No abstract. Refer to Ref. Zhur.-Khimiya, 1957, 63581.

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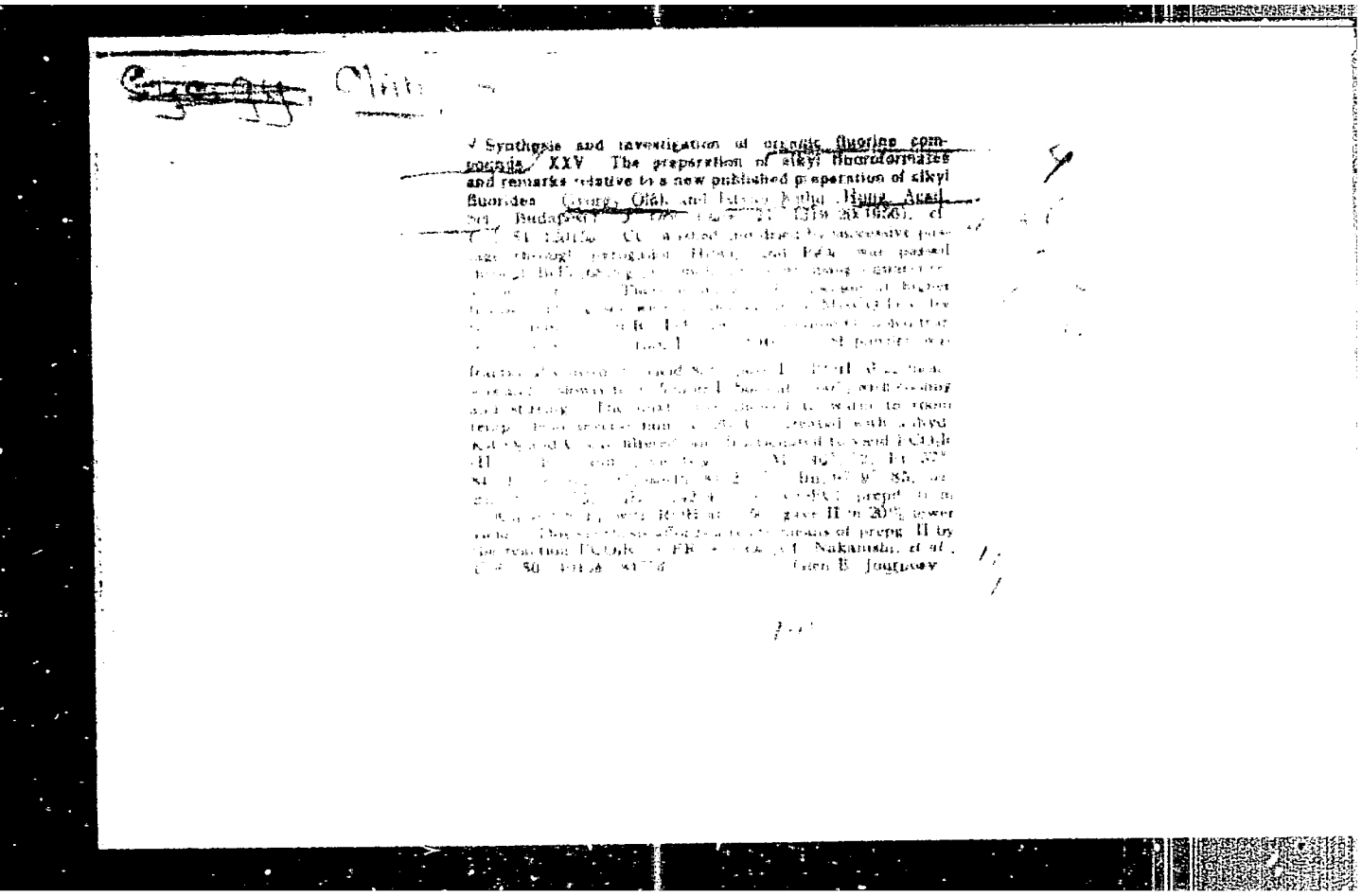
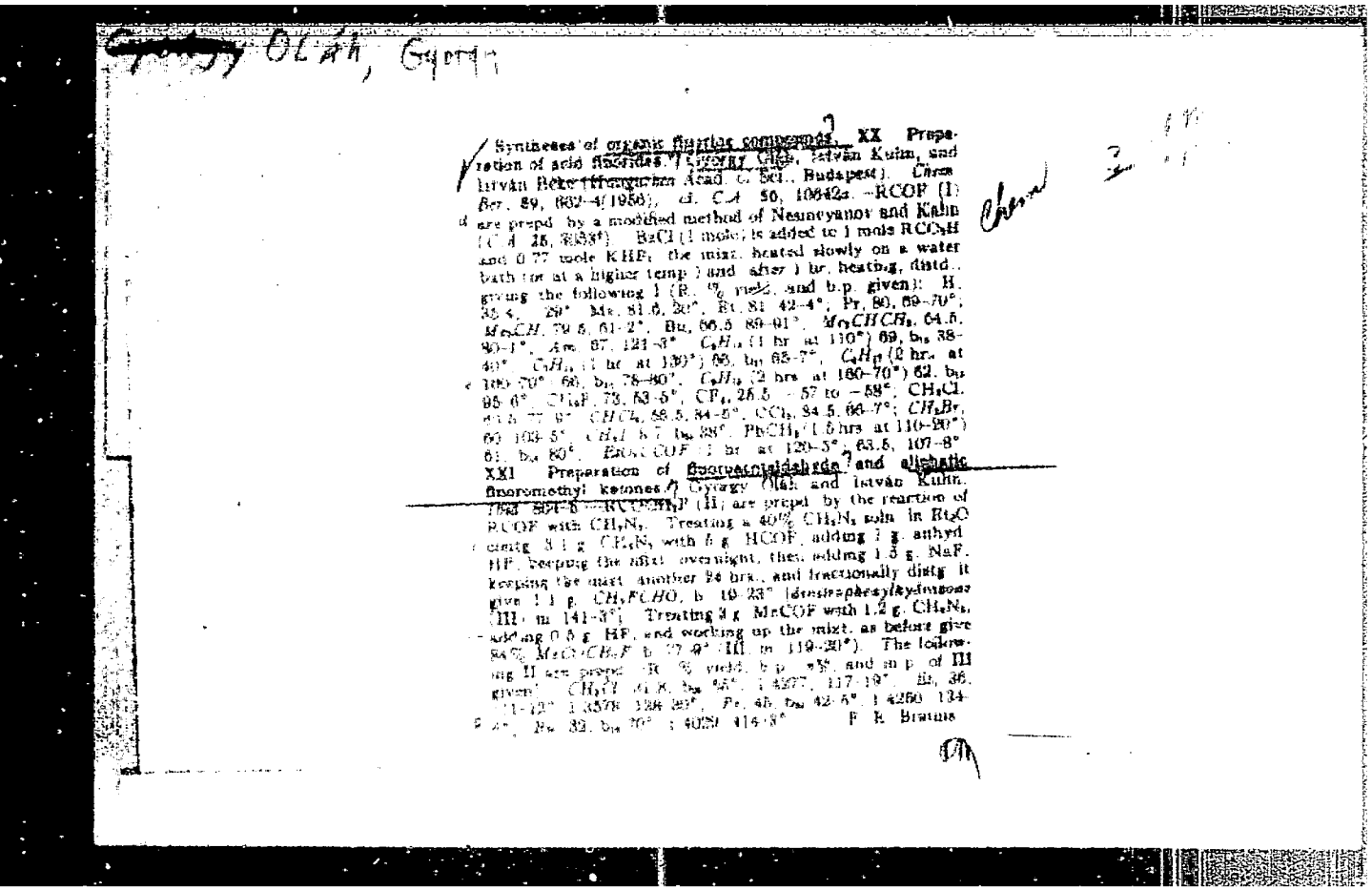


Synthesis and investigation of organic fluorine compounds. XXV. The preparation of alkyl fluoroformates and remarks relative to a new published preparation of alkyl fluorides

OLAH, G.

Isolation of a carbonaid intermediate complex of the electrophilic aromatic substitution reaction and its application as a substitution agent. G. Olah and I. Kuhn (Hung. Acad. Sci., Budapest). Naturwissenschaften 43, 60 (1956). The alkylfluoroniumate could be prepd. through the reaction of alkyl or alicyclic fluorides with BF_3. The isolated complex was used to alkylate C_6H_6, PhMe, etc. O_2NF and BF_3 gave nitronium borofluoride, a powerful nitrating reagent, which was used to nitrate C_6H_6, PhMe, PhF, PhCl, $PhNO_2$, 1,2,5-$F_3C_6H_3$, and 2,4-$(O_2N)_2C_6H_3$. Sulfonium borofluoride was used to sulfonate aromatic compds.

Felix Szmuszkovicz



Olah, Gyorgy

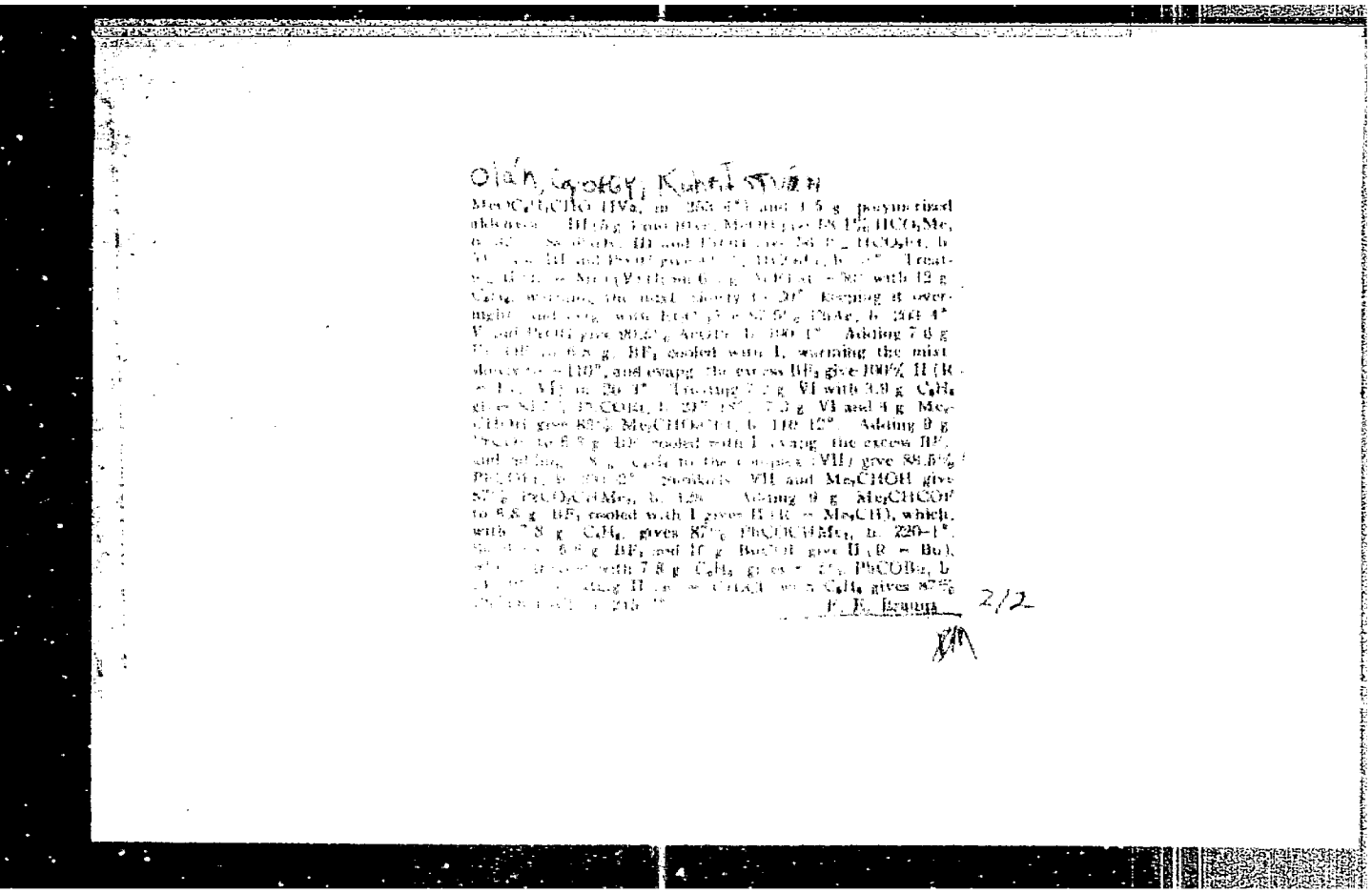
Syntheses of organic fluorine compounds. XX Preparation of acid fluorides. Gyorgy Olah, Istvan Kuhn, and Istvan Bekey (Hungarian Acad. of Sci., Budapest). *Chem. Ber.* 89, 862-4 (1956); cf. *C.A.* 50, 10042a. —RCOF (I) are prepd. by a modified method of Nesmeyanov and Kahn (*C.A.* 35, 8338⁸). $BzCl$ (1 mole) is added to 1 mole RCO_2H and 0.77 mole KHF_2, the mixt. heated slowly on a water bath (or at a higher temp.) and after 1 hr. heating, distd., giving the following I (R, % yield, and b.p. given): H, 35%, 29°; Me, 81.6, 20°; Et, 81, 42-4°; Pr, 80, 69-70°; Me_2CH, 79.5, 61-2°; Bu, 66.5, 89-91°; $MeCH_2CH_2$, 64.5, 90-1°; Am, 87, 121-3°; C_5H_{11} (1 hr. at 110°) 69, b_{15} 38-40°; C_6H_{13} (1 hr. at 130°) 66, b_{11} 65-7°; C_7H_{15} (2 hrs. at 160-70°) 60, b_{15} 76-80°; C_8H_{17} (2 hrs. at 160-70°) 62, b_{15} 95-6°; CH_2F, 73, 53-5°; CF_4, 25.5, −57 to −58°; CH_2Cl, 61.5, 77-8°; $CHCl_2$, 58.5, 84-5°; CCl_3, 84.5, 66-7°; CH_2Br, 60, 103-5°; CH_2I, 67, b_{15} 88°; $PhCH_2$ (1.5 hrs. at 110-30°) 61, b_{14} 80°. $BrCH_2COF$ (1 hr. at 120-5°), 63.5, 107-8°

XXI Preparation of fluoroacetaldehyde and aliphatic fluoromethyl ketones. Gyorgy Olah and Istvan Kuhn. *Ibid.* 869-73.—$RCOCH_2F$ (II) are prepd. by the reaction of RCOF with CH_2N_2. Treating a 40% CH_2N_2 soln. in Et_2O containg 3.1 g. CH_2N_2 with 5 g. HCOF, adding 1 g. anhyd. HF, keeping the mixt. overnight, then adding 1.5 g. NaF, keeping the mixt. another 24 hrs., and fractionally distg. it give 1.1 g. CH_2FCHO, b. 19-23° (*dinitrophenylhydrazone* III, m. 141-3°). Treating 3 g. MeCOF with 1.2 g. CH_2N_2, adding 0.5 g. HF, and working up the mixt. as before give 84% $MeCOCH_2F$, b. 77.4° (III, m. 119-20°). The following II are prepd. (R, % yield, b.p., $n^{25}D$, and m.p. of III given): CH_2Cl, 41.6, b_{744} 95°, 1.4277, 117-19°; Et, 36, 71-2°, 1.3978, 126-28°; Pr, 45, b_{744} 42-5°, 1.4260, 134-5°; Bu, 32, b_{744} 70°, 1.4050, 116-18°.

F. R. Bruns

OLAH, GYORGY

Acylation reactions with acid fluorides in the presence of boron trifluoride, including formylation with formyl fluoride. György Olah and István Kuhn (Hungarian Acad. of Sci., Budapest). *Chem. Ber.* 89, 866-70(1956). cf. preceding abstr. — RCOF (or Ac$_2$O) with BF$_3$ at the temp. of liquid air (I), stable complexes, *acylfluoroborates*, [RCO]$^+$BF$_4^-$ (II). The formyl complex [HCO]$^+$BF$_4^-$ (III) formylates C$_6$H$_6$, PhMe, and PhOMe directly. Adding 5 g. CuCl to 7 g. HCOF at −80°, evapg. the excess HCOF at −25°, adding 16 g. PhMe at −80°, satg. the mixt. with BF$_3$, keeping it 6 hrs., allowing the temp. to rise to 20°, and steam distg. the residue gave 2 g. *p*-MeC$_6$H$_4$CHO (IV), b$_{20}$ 204°. Satg. 3 g. HCOF at −80° with BF$_3$, adding 5 g. PhMe, warming the mixt. slowly to 20°, keeping it overnight, adding ice, and extg. with Et$_2$O gave 30% IV. Adding 7 g. HCOF cooled to −100° to 10 g. BF$_3$ cooled with I and warming the mixt. slowly give III m. −112°. Hydrolysis of 1.26 g. III with satd. K$_2$CO$_3$ soln. and titration of the soln. require 4.37 cc. 0.1 *N* NaOH (calcd. 4.42 cc.). Treating 6 g. III with 10 cc. PhMe at −80° and warming the mixt. after 6 hrs. give [illegible]% IV, 2,4-dinitrophenylhydrazone (IVa), m. 233-4°. Similarly 7 g. III and 9.5 g. PhOMe yield 0.5 g. *p*-



Olah, George; Kuhn, Stephen

$Me_3C_6H_2CH_2Cl$ (IVa, R = Me), m. 256–8° and 1.5 g. polymerized chloride were treated with 10.6 g. Me_3CHCO_2Me, b. 87–8°, and 15.5 g. BF_3 at 0–5° to give 85.1% Me_3CHCO_2Me, b. 87–8°. Similarly, IVa and Me_3CHOH gave 78.2% Me_3CHCO_2Me, b. 87–8°. Treatment of 15 g. Me_3CH with 6 g. $AlCl_3$ at −80° with 12 g. $MeCOCl$, warming the mixt. slowly to 20°, keeping it overnight, and treating with ice gave 57.5% $Me_2CHCOMe$, b. 104–4.5°. IVa and $PhCOCl$ gave 90.6% $ArCOPh$, b. 180–1°. Adding 7.6 g. $Me_2CHCOCl$ to 6.8 g. BF_3 cooled with L, warming the mixt. above −10°, and evapg. the excess BF_3 gave 100% II (R = Me). VI, m. 26–8°. Treating 7.2 g. VI with 9.9 g. C_6H_6 gave 85.7% $PhCOEt$, b. 207–8°. 7.5 g. VI and 4 g. Me_2CHOH gave 88% $Me_2CHCO_2CHMe_2$, b. 110–12°. Adding 9 g. $Me_2CHCOCl$ to 8.5 g. BF_3 cooled with L, evapg. the excess BF_3, and adding 8.5 g. C_6H_6 to the complex VII gave 88.6% $PhCOEt$, b. 208–9°. The products VII and Me_2CHOH gave 87% $Me_2CHCO_2CHMe_2$, b. 120°. Adding 9 g. $MeCHClCOCl$ to 8.5 g. BF_3 cooled with L gave II (R = $MeCHCl$), which, with 7.8 g. C_6H_6, gives 87% $PhCOCH(Cl)Me$, b. 220–1°. Similarly, 8.5 g. BF_3 and 10 g. $BuCOCl$ give II (R = Bu), which with 7.8 g. C_6H_6 gives 87% $PhCOBu$, b. [illegible] g. II (R = CH_2Cl) with C_6H_6 gives 87% [illegible] 215°.

P. E. Pruna 2/2

Olah Gyorgy

N-Formylation of amines with formyl fluoride. Gyorgy Olah and Istvan Kuhn (Hung. Acad. Sci., Budapest). Chem. Ber. 89, 2211-12 (1956).--Adding with stirring and NaCl-ice cooling 5.75 g. HCOF to 0.2 mole appropriate amine in 30 cc. abs. Et_2O and allowing the temp. to rise to 15° give the corresponding RR'NCHO, of which the following are prepd. (R, R', % yield, and b.p. or m.p. given): H, Et, 89, b. 196-8°; Et, Et, 84, b. 176-8°; H, Ph, 81, m. 46-7°; Et, Ph, 92, b. 120°; Ph, Ph, 94, m. 71-2°. N-formylpiperdine, 90, b. 220-2°.

F. E. Braun

OLAH, GYORGY

Preparation of nitrosamines, alkylnitrites, and alkyl [illegible] esters with nitrosyl and nitryl tetrafluoroborates. Olah, Ladislaus Noszkó, Istvan Kerekes, and Michael [illegible] (Polytech. Chem. Tech. Univ., Budapest) *Chem. Ber.* 99, 2974-7 (1966).—Adding 11.7 g. ONBF, with stirring and ice-cooling to 0.2 mole secondary amine, stirring the mixt. 10 min., fractionally distg. the filtered soln. yield the following RR'NNO (R, R', % yield, and b. p. given): Et, Et, 88, 178°; Et, Ph, 80, b_{20} 130°; Me, $PhCH_2$, 88, b_{10} 168°; RR' = $O(CH_2CH_2)_2$, 78, b_{20} 139-40°; RR' = $(CH_2)_5$, 88, 216°. Adding 23.5 g. ONBF, in small portions to MeOH or EtOH with stirring and ice-cooling gives 58% MeONO, b. −14 to −10° or 87.5% EtONO, b 16-20°, resp. Adding 0.3 mole ONBF, to 0.3 mole PrOH, BuOH, or iso-AmOH and 6 g. anhyd. Na_2CO_3 at −10° gives 36.4% PrONO, b 46-50°, 45% BuONO, b 78-8° or 48% iso-AmONO, b 87-105°, resp. Adding 13.8 g. ONBF, in small portions to 0.3 mole sugar acetate, air gives the following $RONO_2$ (R, % yield, and b. p. given): Me, 87, 84-5°; Et, 92.5, 87-8°; Pr, 87, 100-1°; Bu, 91, 135-6°; C_5H_{11}, 85.5, b_{10} 70-1°; $C_6H_{13}F$, 89, b 130-8° (n_D^{20} 1.394); $C_6H_{13}Cl$, 85, 149-50°; $C_6H_{13}Br$, 85.5, 166-7°; CF_3CH_2, 75, 2 (n$_D^{20}$ 1.358). E. E. R.

OLAH, G.

Isolation of the stable boron trifluoride–hydrogen fluoride complexes of the methylbenzenes; the onium (or σ complex) structure of the Friedel-Crafts complexes. G. Oláh, I. Kuhn, and A. Pavlath (Hung. Acad. Sci., Budapest). *Nature* 178, 693–4(1956); cf. McCaulay, *et al.*, *C.A.* 45, 933h.—Stable BF_3–HF complexes of some methylbenzenes were isolated at low temp. Melting points and specific cond. of the following complexes were detd.: toluene, −66°, 0.8×10^{-4} ohms^{-1} cm.$^{-1}$; *m*-xylene, −55°, 2.0×10^{-4} ohms^{-1}cm.$^{-1}$; mesitylene, −15°, 1.6×10^{-4} ohms^{-1}cm.$^{-1}$; hexamethylbenzene, −10°, 0.6×10^{-3} ohms^{-1} cm.$^{-1}$. P. M.

OLAH, G.

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Preparation and investigation of deuterium compounds. I. A simple preparation of deuterium fluoride. G. Olah and J. Kuhn (Ungarischen Akad. Wiss., Budapest). Z. anorg. u. allgem. Chem. 287, 252-8 (1956). The reaction of heavy water, D_2O, with SOF_2, COF_2, and RCOF, where R is H, Me, Et, and Ph to yield DF was studied. The best results were obtained with BzF. BzF (168 g.) was cooled in Ag app., 5 g. of 99% D_2O was added, and the whole warmed slowly to 80-90° to distil off the DF, the distillate being condensed and stored at −15°. The DF was redistd. A 92% yield was obtained. John H. Wood

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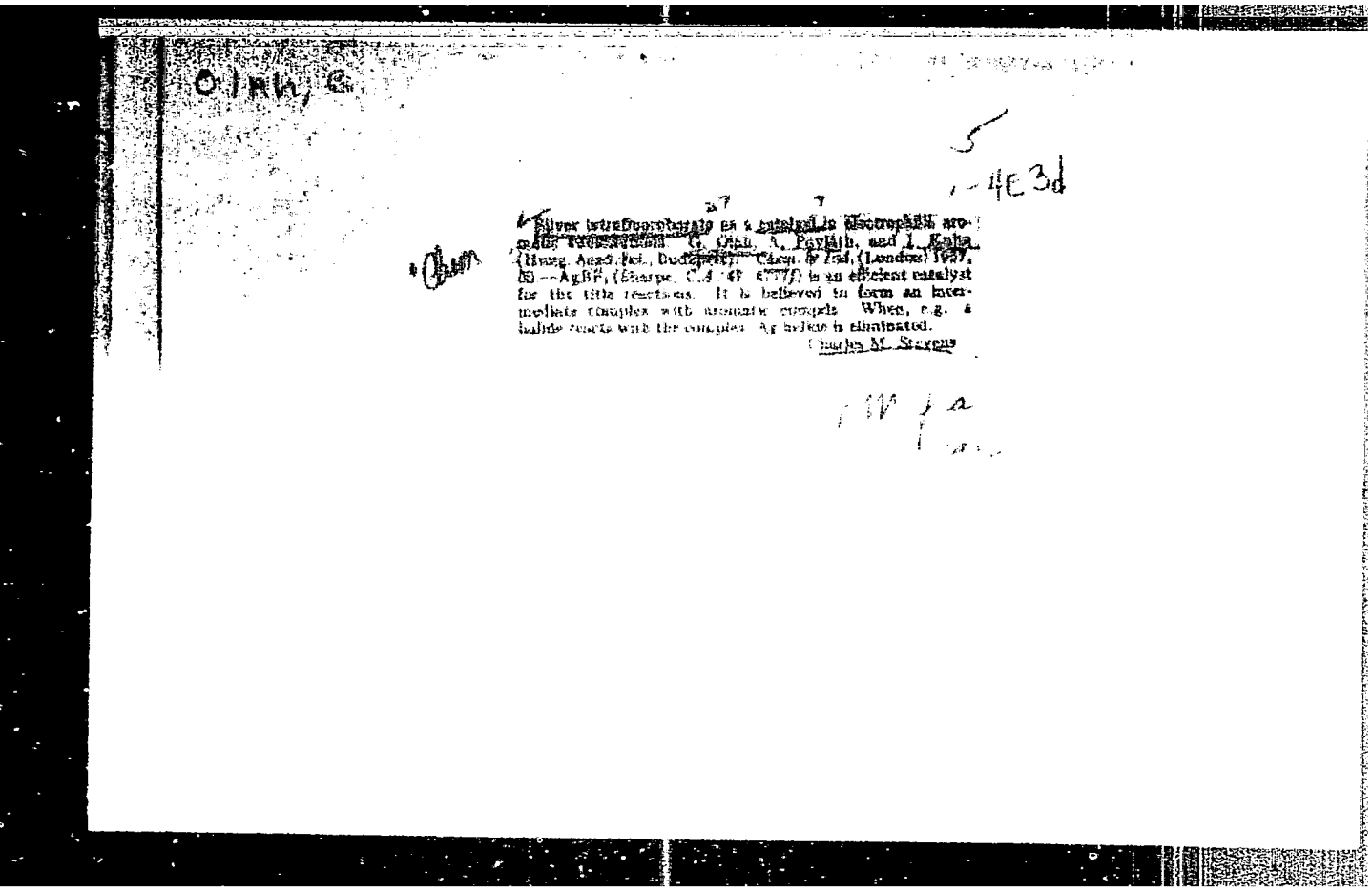
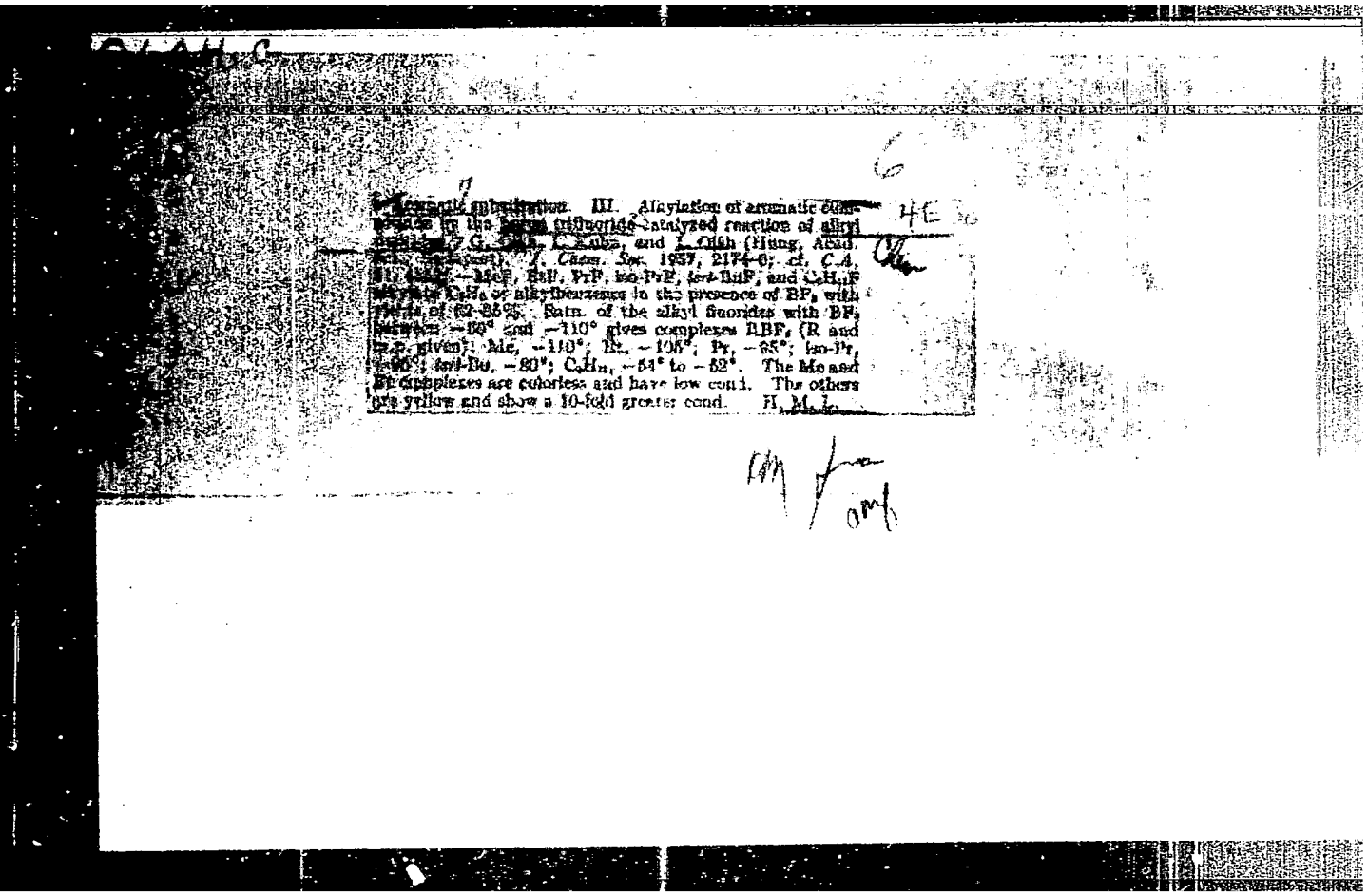
Chem

PM MIT



Silver tetrafluoroborate as a catalyst in electrophilic aromatic substitution. G. Olah, S. Kuhn, and J. Kuhn (Hung. Acad. Sci., Budapest). Chem. & Ind. (London) 1957, 530.—AgBF$_4$ (Sharpe, C.A. 49, 6777f) is an efficient catalyst for the title reactions. It is believed to form an intermediate complex with aromatic compds. When, e.g., a halide reacts with the complex, AgBF$_4$ is not eliminated.

Charles M. Stevens

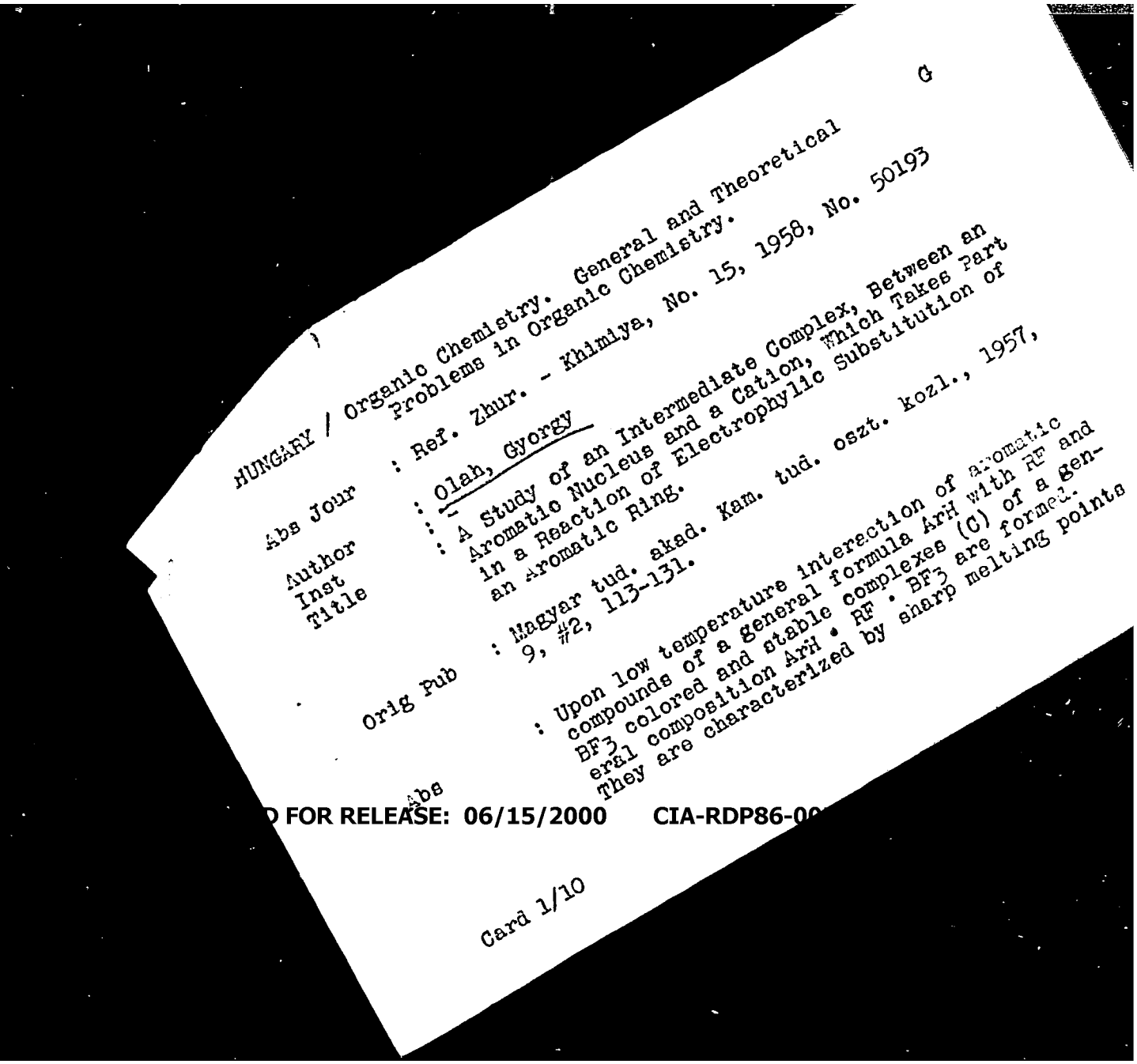


Aromatic substitution. III. Alkylation of aromatic compounds by the boron trifluoride-catalyzed reaction of alkyl fluorides. G. Baddeley, J. Kenner, and E. Ollis (Hung. Acad. Sci., Budapest). *J. Chem. Soc.* 1957, 2174–6; cf. *C.A.* 51, 4447f.—MeF, EtF, PrF, iso-PrF, tert-BuF, and $C_6H_{11}F$ alkylate C_6H_6 or alkylbenzenes in the presence of BF_3 with yields of 62–85%. Satn. of the alkyl fluorides with BF_3 between −50° and −110° gives complexes RBF_4 (R and m.p. given): Me, −110°; Et, −105°; Pr, −65°; iso-Pr, −90°; tert-Bu, −80°; C_6H_{11}, −51° to −52°. The Me and Et complexes are colorless and have low cond. The others are yellow and show a 10-fold greater cond. H. M. L.

Distr: 4E2c/4E3c/4E3d/4E2c(j)

41. A study of the electrophilic deuterization of toluene by deuterium fluoride and boron trifluoride. Gy. OLAH, A. Pavláth, I. Kuhn, Gy. Olah, I. Norko. *A Magyar Tudományos Akadémia Kémiai Tudományok Osztályának Közleményei.* Vol. 9, 1957, No. 1, pp. 39—42

In the course of the studies on electrophilic aromatic deuterization the reaction with deuterium fluoride was investigated in the presence of boron trifluoride. A simple, new laboratory procedure has been developed for the preparation of deuterium fluoride through the deuterolysis of organic acid fluorides such as benzoyl fluoride. Nuclear deuterization experiments have been made by substituting toluene. A study of the intermediary complexes of the electrophilic aromatic substitutions revealed that complexes of methylbenzenes with hydrogen fluoride and boron trifluoride consist in fact of protonated methylbenzenes of the onium ionic salt (or σ complex) type.



G

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry.

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

Author : Olah, Gyorgy

Inst :

Title : A Study of an Intermediate Complex, Between an Aromatic Nucleus and a Cation, Which Takes Part in a Reaction of Electrophylic Substitution of an Aromatic Ring.

Orig Pub : Magyar tud. akad. Kam. tud. oszt. kozl., 1957, 9, #2, 113-131.

Abs : Upon low temperature interaction of aromatic compounds of a general formula ArH with RF and BF3 colored and stable complexes (C) of a general composition ArH • RF • BF3 are formed. They are characterized by sharp melting points

Abs

Card 1/10

G

anic Chemistry. General and Theoretical
roblems in Organic Chemistry.

ef. Zhur. - Khimiya, No. 15, 1958, No. 50193

and a comparatively good electrical conductivity
(χ), and are σ complexes (σ-C) of structure
(I-XVIII). The latter substances when reacted
with one atom of C (sp3-hybrid) and with an elec-
tronic configuration of a pentadiene cation lead
to intermediate C, formed during the electro-
phylic substitution of an aromatic nucleus.
This is caused by the interaction of an aroma-
tic ring with the impinging cation. Structure
I is substantiated by its formation due to an
interaction of 3 (or 6?)-fluoro-1-methyl cy-
clohexadiene-1,4 (XVIII) with BF3. Upon a
careful thermal decomposition the complex is
obtained with good yields, corresponding to mono
R substitutes. The products (which consist in

Card 2/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

Author : Olah, Gyorgy
Inst : -
Title : A Study of an Intermediate Complex, Between an Aromatic Nucleus and a Cation, Which Takes Part in a Reaction of Electrophylic Substitution of an Aromatic Ring.

Orig Pub : Magyar tud. akad. Kam. tud. oszt. kozl., 1957, 9, #2, 113-131.

Abs : Upon low temperature interaction of aromatic compounds of a general formula ArH with RF and BF_3 colored and stable complexes (C) of a general composition ArH • RF • BF_3 are formed. They are characterized by sharp melting points

Card 1/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

and a comparatively good electrical conductivity (χ), and are σ complexes (σ-C) of structure (I-XVIII). The latter substances when reacted with one atom of C (sp^3-hybrid) and with an electronic configuration of a pentadiene cation lead to intermediate C, formed during the electrophylic substitution of an aromatic nucleus. This is caused by the interaction of an aromatic ring with the impinging cation. Structure I is substantiated by its formation due to an interaction of 3 (or 6?)-fluoro-1-methyl cyclohexadiene-1,4 (XVIII) with BF_3. Upon a careful thermal decomposition the complex is obtained with good yields, corresponding to mono R substitutes. The products (which consist in

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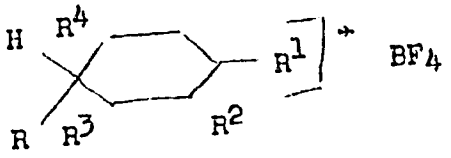
HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

the case when C is obtained from toluene, of a mixture of orto and para isomers) contain small quantities of di-R derivatives. A yield of a 50% corresponding to mono-deutero derivatives is obtained from V-VIII. The mere fact of isolation of the complexes and their properties indicate that intermediate C which determine the speed of reaction of electrophylic substitution in an aromatic nucleus are not π-complexes but σ-complexes of I-XVII type.



Card 3/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

I. $R = R^2 = R^3 = R^4 = H$ and $R^1 = CH_3$
(or $R = R^1 = R^2 = R^4 = H$ and $R^3 = CH_3$)

II. $R = R^2 = R^4 = H$, $R^1 = R^3 = CH_3$

III. $R = R^2 = H$, $R^1 = R^3 = R^4 = CH_3$

IV. $R = H$, $R^1 = R^2 = R^3 = R^4 = CH_3$

V. $R = D$, $R^2 = R^3 = R^4 = H$ $R^1 = CH_3$
(or $R = D$, $R^2 = R^3 = R^4 = H$, $R^3 = CH_3$)

VI. $R = D$, $R^2 = R^4 = H$, $R^1 = R^3 = CH_3$

Card 4/10

VII. $R = D$ $R^2 = H$ $R^1 = R^3 = R^4 = CH_3$

VIII. $R = D$ $R^1 = R^2 = R^3 = R^4 = D$

IX. $R = C_2H_5$, $R^2 = R^3 = R^4 = H$ $R^1 = CH_3$

(or $R = C_2H_5$, $R^1 = R^2 = R^4 = H$, $R^3 = CH_3$)

X. $R = C_2H_5$, $R^2 = R^4 = H$, $R^1 = R^3 = CH_3$

XI. $R = C_2H_5$, $R^2 = H$, $R^1 = R^3 = R^4 = CH_3$

XII. $R = CHO$, $R^2 = R^3 = R^4 = H$, $R^1 = CH_3$

(or $R = CHO$, $R^1 = R^2 = R^4 = H$, $R^3 = CH_3$)

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

XIII. R = CHO, $R^2 = R^4 = H$, $R^1 = R^3 = CH_3$

XIV. R = CHO, $R^2 = H$, $R^1 = R^3 = R^4 = CH_3$

XV. R = CHO, $R^1 = R^2 = R^3 = R^4 = CH_3$

XVI. R = C_2H_5CO, $R^2 = R^3 = R^4 = H$, $R^1 = CH_3$

(or R = C_2H_5CO, $R^1 = R^2 = R^4 = H$, $R^3 = CH_3$)

XVII. R = NO_2, $R^1 = R^2 = R^4 = H$, $R^3 = CF_3$

Card 6/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

Below are listed in the order shown, melting points in °C and electroconductivity mhos (ohm^{-1} cm^{-1}) of the molten complexes, measured at the melting temperature -

I.	-65, 0.8	IX.	-80, 0.2	XIII.	-52, 0.5
II.	-55, 2.0	X.	-75, 0.1	XIV.	-16, 0.7
III.	-15, 1.6	XI.	-15, 0.2	XV.	-10, 0.7
IV.	-10, 0.6	XII.	-70, 0.3	XVI.	-2, 0.2
				XVII.	-50, -

Properties of deutero complexes V-VIII are practically the same as the corresponding properties of I-IV. A general method for preparation of complexes I-XVI consists of passing BF_3 through

Card 7/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur.- Khimiya, No. 15, 1958, No. 50193

a solution of RF in ArH while keeping the temperature between -25° and -80°. Upon the addition to $C_2H_5F \cdot BF_3$ of an equimolar amount of toluene at -80°, IX is obtained. By analogy XII is prepared by interaction of $[HCO]^+ BF_4^-$ (see Ref. Zhur. Khim. 1957, 77160) of 0.1 x 10^{-2} mhos-1) with toluene. By adding, (at -80°) 0.1 mole of NO_2F to 0.1 mole of $CF_3C_6H_5$, then by reacting the mixture with 0.2 grams of BF_3 at - 120°, and subsequently by mixing and warming to -100°, XVII is obtained. $[NO_2]^+ BF_4^-$ does not react with $CF_3C_6H_5$ even at + 100°. To 1 l of liquid NH_3 at -80° are added consecutively, 46 g Na, mixture of 92 g toluene with 64 g CH_3OH, 20 g CH_3OH, 18.5 g Na and 36 g CH_3OH.

Card 8/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry. G

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

This mixture is then stirred for 2 hours, and kept for about 12 hours. Subsequently NH_3 is driven off by vaporization and the mixture is decomposed by water. The yield of methyl cyclohexadiene (XIX) is 52.5%. Its b.p. = 114.5 -115°. Into a solution of 19.29 of XIX in 200 ml of CCl_4 are added 37.4 g of N-bromo succinimide (1 hour, mixing, 100 C). Stirring is continued for 4 hours, and the mixture is kept for about 12 hours at 0°. The yield of 3 (or 6?) -bromo-1-methyl cyclohexadiene-1,4 (XX) is 55.2%. Its $n^{20}D$ = 1.5678, and its decomposition temperature = 40°/0.01 mm. Into a solution of 17.3 g of XX in 20 ml of CH_3CN is added, upon

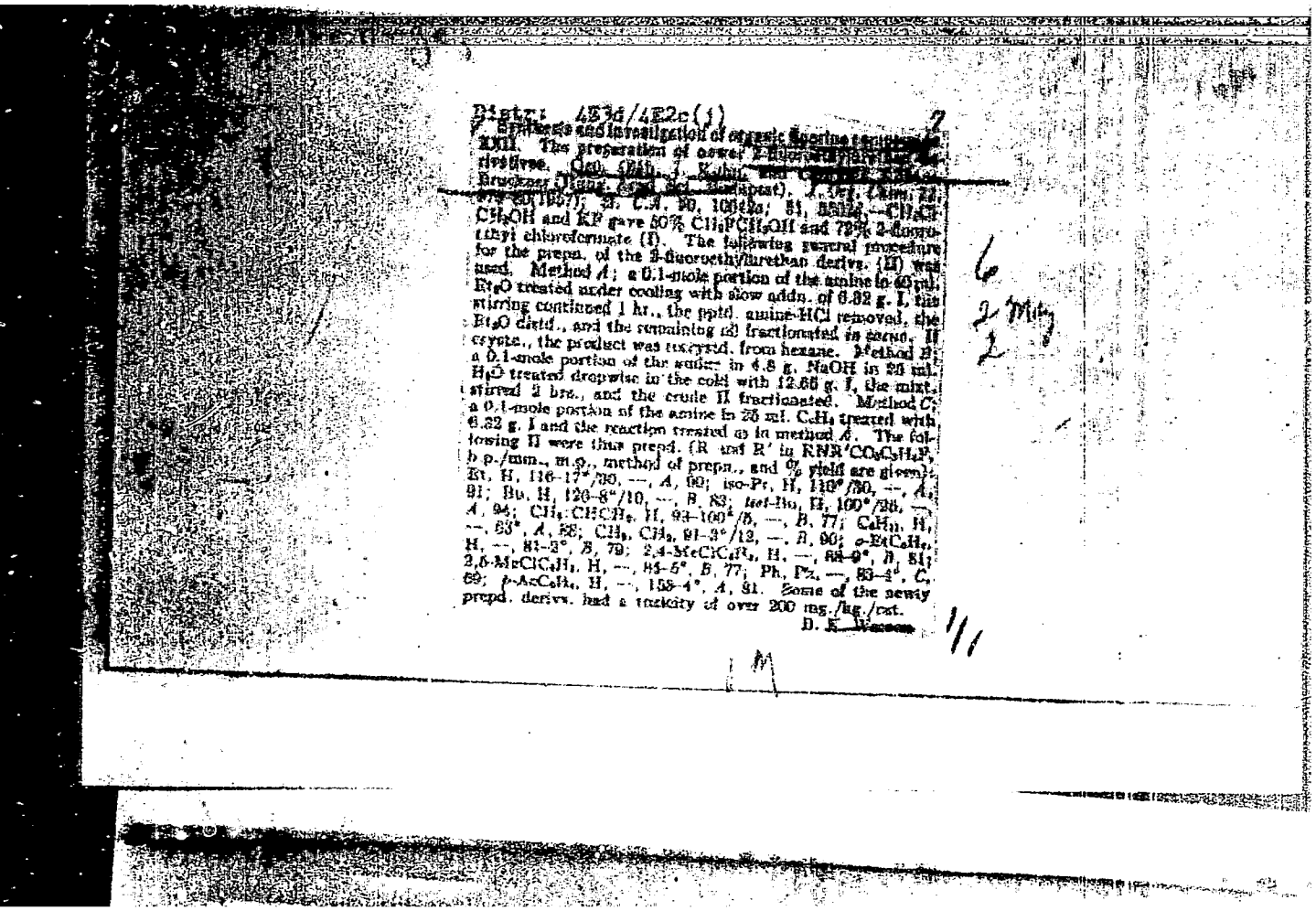
Card 9/10

HUNGARY / Organic Chemistry. General and Theoretical Problems in Organic Chemistry.

Abs Jour : Ref. Zhur. - Khimiya, No. 15, 1958, No. 50193

stirring, a solution of 14 g AgF in 50 ml CH_3CN. Stirring is continued for 6 hours, the solution is aged for 12 hours, then it is filtered, vaporized, washed with iced water and finally the remnants of the solvent are driven off (0.05 mm for 12 hrs.). The yield of XVIII is 46.6%. Its $n^{20}D$ = 1.4975. 5.5 g of XVIII is saturated with BF_3 at -50° - -65°. The measured magnitude of electroconductivity of the reacted mass (-65°) is 1 x 10^{-2} mhos. Upon thermal decomposition the yield of toluene is 24%.

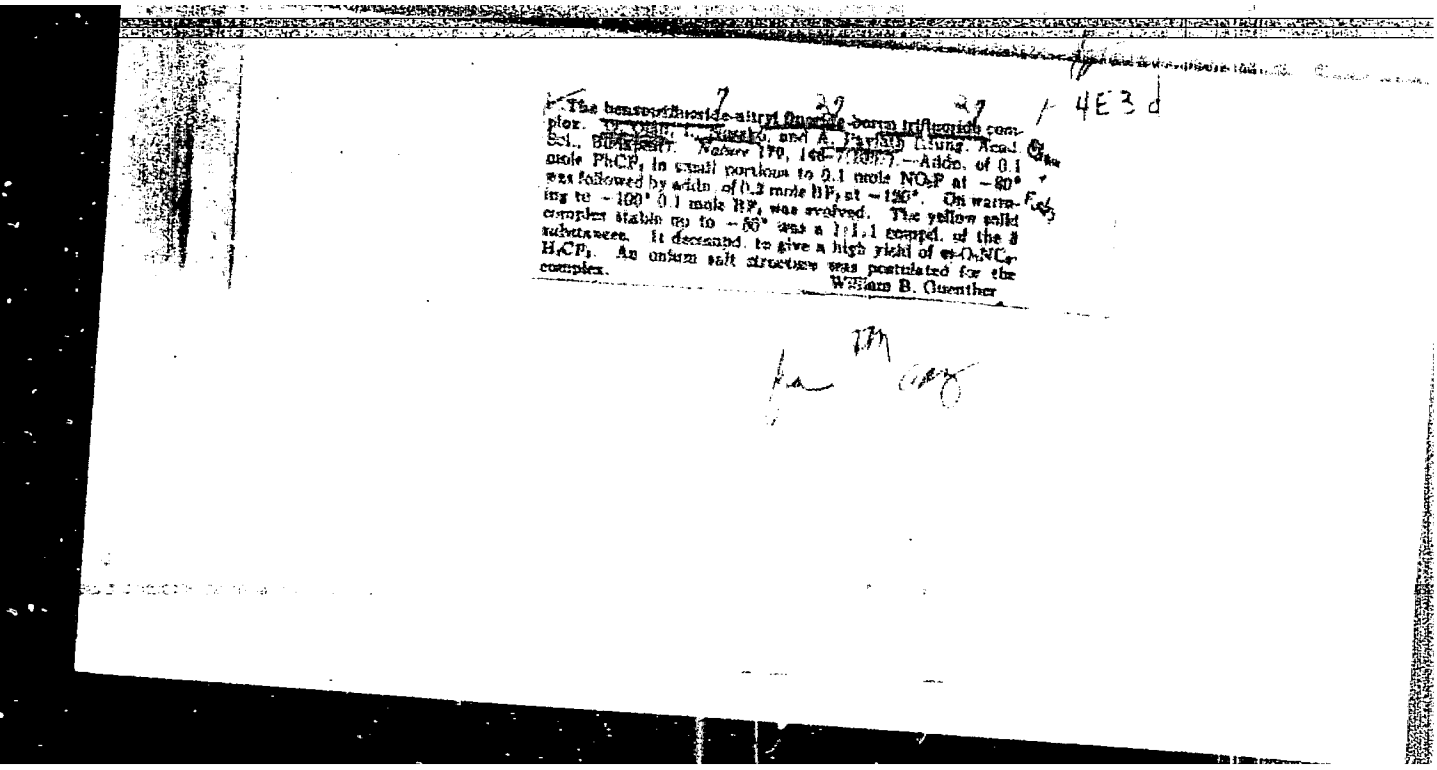
Card 10/10



Digest: 4536/4E2c(1)

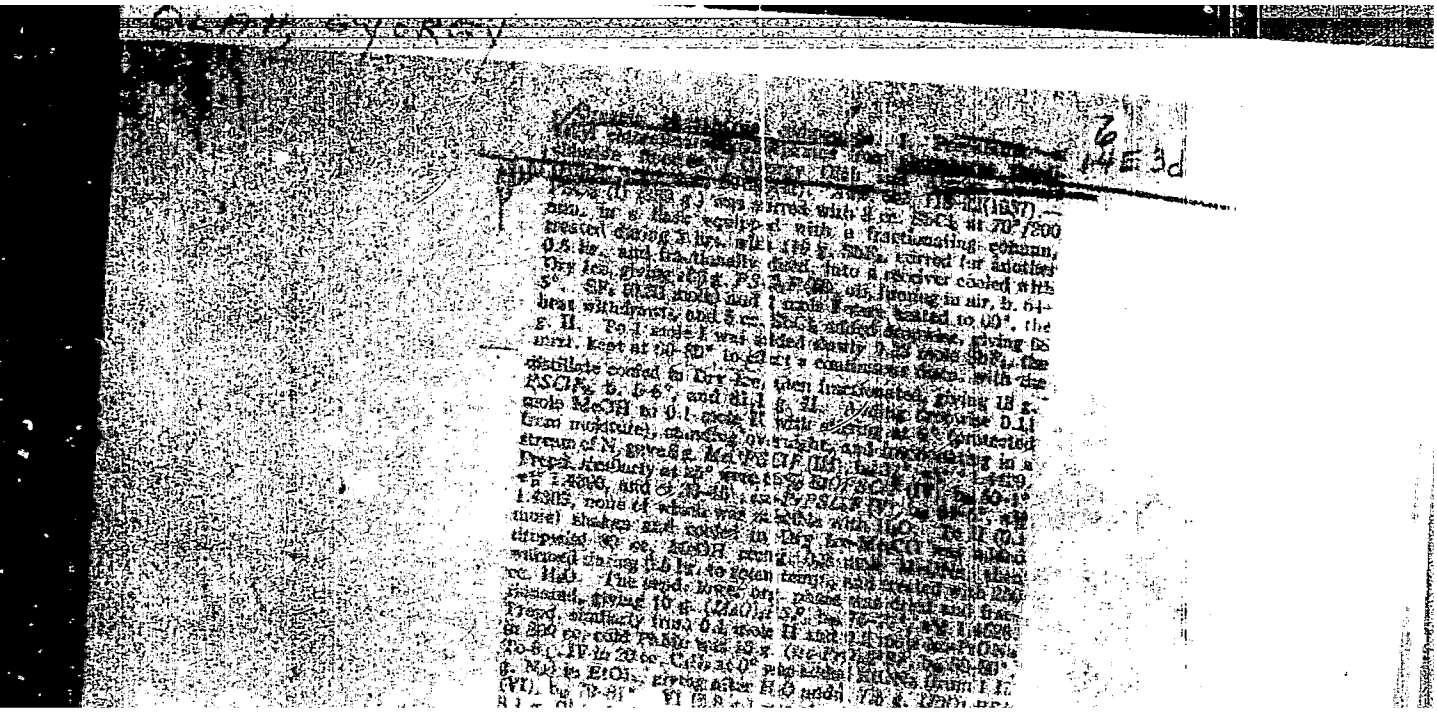
Synthesis and investigation of organic fluorine [illegible]
XXII. The preparation of some 2-fluoroethyl [illegible]
rivatives. Gen. Obšč. J. Kuhn, and [illegible]
Bruckner, Jiang, Gesch. der [illegible] Stuttgart), J. Org. Chem. U.S.S.R. 20, 1105(7); C. A. 50, 10042a; 51, 5512g.—ClCH$_2$CH$_2$OH and KF gave 50% ClCH$_2$CH$_2$OH and 72% 2-fluoroethyl chloroformate (I). The following general procedure for the prepn. of the 2-fluoroethylurethan derivs. (II) was used. *Method A*: a 0.1-mole portion of the amine in 40 ml. Et$_2$O treated under cooling with slow addn. of 6.32 g. I, the stirring continued 1 hr., the ppte. amine·HCl removed, the Et$_2$O distd., and the remaining oil fractionated in vacuo. If crysts., the product was recrystd. from hexane. *Method B*: a 0.1-mole portion of the amine in 4.8 g. NaOH in 25 ml. H$_2$O treated dropwise in the cold with 12.66 g. I, the mixt. stirred 2 hrs., and the crude II fractionated. *Method C*: a 0.1-mole portion of the amine in 25 ml. C$_6$H$_6$ treated with 6.32 g. I and the reaction treated as in method *A*. The following II were thus prepd. (R and R' in RNR'CO$_2$C$_2$H$_4$F, b. p./mm., m.p., method of prepn., and % yield are given): Et, H, 116–17°/30, —, *A*, 90; iso-Pr, H, 110°/30, —, *A*, 91; Bu, H, 120–8°/10, —, *B*, 83; *tert*-Bu, H, 100°/38, —, *A*, 94; CH$_2$:CHCH$_2$, H, 93–100°/5, —, *B*, 77; C$_6$H$_{11}$, H, —, 53°, *A*, 58; CH$_2$, CH$_2$, 91–3°/12, —, *B*, 90; *o*-EtC$_6$H$_4$, H, —, 81–3°, *B*, 79; 2,4-MeClC$_6$H$_3$, H, —, 88–9°, *B*, 81; 2,6-MeClC$_6$H$_3$, H, —, 85–5°, *B*, 77; Ph, Pz, —, 83–4°, *C*, 69; *p*-AcC$_6$H$_4$, H, —, 158–4°, *A*, 91. Some of the newly prepd. derivs. had a toxicity of over 200 mg./kg./rat.

D. K. Wessels



The benzenediazonium-aryl fluoride-boron trifluoride complex. [illegible] Kiss[illegible], and A. [illegible] Chim. Acad. Sci., Budapest. *Nature* 170, 166–7(1952).—Addn. of 0.1 mole $PhCF_3$ in small portions to 0.1 mole NO_2F at −80° was followed by addn. of 0.2 mole BF_3 at −120°. On warming to −100° 0.1 mole BF_3 was evolved. The yellow solid complex stable up to −50° was a 1:1:1 compd. of the 3 substances. It decomposed to give a high yield of *m*-$O_2NC_6H_4CF_3$. An onium salt structure was postulated for the complex.

William B. Guenther



OLAH, Gyorgyne

HUNGARY/Physical Chemistry. Radiochemistry. Isotopes. B-7

Abs Jour: Ref Zhur-Khim., No 13, 1958, 42474.

Author : Olah Gyorgy, Pavlath Attila, Kuhn Istvan, Olah Gyorgyne, Noszko Laszlo.

Inst :

Title : Study of Electrophilic Deutorization of Toluene with Deuterium Fluoride and Boron Trifluoride.

Orig Pub: Magyar tud. akad. Kem. tud. oszt. kozl., 1957, 9, No 1, 39-42.

Abstract: No abstract.

Card : 1/1

ABRAMOVICI, R.; VELICA, F.; OLAH, I.

Influence of the introduction of previously melted feldspar in the porcelain mass on its properties. Bul St si Tehn Tim 9 no.2:341-348 Jl-D '64.

OLAH, Imre,; SZABO, Gyorgy.

Methods to measure sensitivity of the cornea. Szemeszet 92 no.2: 93-96 June 55.

1. A Budapesti Orvostudomanyi Egyetem I.sz. Szemklinikajanak (igazgato: Radnot Magda egyetemi tanar, az orvostudomanyok doktora) koslemenye.

(CORNEA, physiol.
sensitivity, new measuring instrument (Hun))
(OPHTHALMOLOGY, appar. & instruments
instrument for measuring corneal sensitivity (Hun))

OLAH, Imre.

Principles and clinical application of electroretinography. Szemeszet 92 no.4:176-183 Dec 55.

1. A Budapesti Orvostudomanyi Egyetem I. sz. Szemklinikajanak (igazgato: Radnoit Magda egyetemi tanar, az orvostudomanyok doktora) kozlemenye.

(RETINA, physiol.
electroretinography, technic & clin. value, review (Hun))

OLAH, Imre

Changes in corneal sensitivity in trachoma. Szemeszet 93 no.2: 79-82 June 56.

1. A Budapesti Orvostudomanyi Egyetem I. sz. Szemklinikajanak (Igazgato: Radnot, Madga egyetemi tanar, az orvostudomanyok doktora) kozlemenye.

(TRACHOMA, physiol.
changes in corneal sensitivity (Hun))
(CORNEA, physiol.
sensitivity, changes in trachoma (Hun))

RADNOT, M.; OLAH, I.

The effect of light on the number of eosinophilic blood cells. Acta med. hung. 11 no.3:393-395 1958.

1. I. Augenklinik der Medizinischen Universitat, Budapest.
(EOSINOPHIL COUNT
eosinopenia induction by prolonged exposure of eyes to light in human volunteers (Ger))
(LIGHT, eff.
prolonged exposure of eyes to light inducing eosinopenia in human volunteers (Ger))

SOLTI, Ferenc; PETER, Agnes; OLAH, Imre; SIMONYI, Gusztav; ISKUM, Miklos; REV, Judit; HERMANN, Robert

Effect of sodium nitrate on the cerebral circulation, central retinal arterial pressure and cerebrospinal fluid pressure. Kiserletes orvostud. 13 no.3:305-310 Je '61.

1. Budapesti Orvostudomanyi Egyetem I. sz. Belklinikaja es Neurologiai klinikaja.

(NITRATES pharmacol) (BRAIN blood supply)
(RETINA blood supply) (CEREBROSPINAL FLUID pharmacol)

OLAH, Imre, dr.; FENYO, Egon, dr.

Correlation between headache and hypotension of the central artery of the retina. Orv. hetil. 102 no.27:1256-1258 2 Je '61.

1. Budapesti Orvostudomanyi Egyetem, Neurologiai Klinika.

(HEADACHE etiol) (RETINA blood supply)

SOLTI, F.; PETER, A.; OLAH, I.; ISKUN, M.; REV, J.; HERMANN, R.; REFI, Z.

Effect of nicotine on cerebral blood circulation and venous pressure. Kiserl. orvostud. 14 no.3:269-272 Je '62.

1. Budapesti Orvostudomanyi Egyetem I. sz. Belklinikaja es Idegklinikaja.

(BRAIN blood supply) (NICOTINE pharmacol)

OLAH, Imre, dr.; AMBORZY, Gyorgy, dr.

A new method for the diagnosis of thrombosis of the internal carotid artery. Ideggyogy. szemle 15 no.6:175-182 Je '62.

1. Budapesti Orvostudomanyi Egyetem Neurologiai Klinikajanak (Igazgato: Horanyi Bela dr. egyetemi tanar) kozlemenye.
(THROMBOSIS diag) (ANIOGRAPHY) (CAROTID ARTERIES dis)
(CEREBRAL EMBOLISM AND THROMBOSIS diag)

SOLTI, F.; PETER, A.; OLAH, I.; ISKUM, M.; REV, J.; HERMANN, R.; REFI, Z.

The acute effect of nicotine on cerebral blood flow and cerebral venous pressure. Cor vasa 5 no.3:197-202 '63.

1. First Medical Clinic and Neurological Clinic of the University Medical School, Budapest.
(CEREBROVASCULAR CIRCULATION) (RETINAL VESSELS)
(BLOOD PRESSURE) (BLOOD FLOW VELOCITY)
(NICOTINE)

OLAH, Imre; ARTAI, Janos; GALOS, Gizella

Changes in systemic and retinal hypertension in hypertensive patients influenced by hypotensive agents. Kiserl. orvostud. 16 no.4:439-443 Ag '64.

1. Budapesti Orvostudomanyi Egyetem Neurologiai Klinikaja es Robert Karoly krt.-i korhaz I Belgyogyaszati Osztalya.

HUNGARY

ROHLICH, Pal, TOROK, Laszlo, OLAH, Imre; Medical University of Budapest, Institute of Histology and Embryology (Budapesti Orvostudomanyi Egyetem, Szovet- es Fejlodestani Intezet).

"Ribosome Helices."

Budapest, A Magyar Tudomanyos Akademia Biologiai Tudomanyok Osztalyanak Kozlemenyei, Vol VIII, No 2, 1965, pages 179-188.

Abstract: Helical structure was observed earlier by the authors on the ergastoplasm of pigment cells in mollusk eyes. In the present, morphological study, pictures of the myoblast cells of 5 day-old chick embryos are presented in which the helical structures are clearly visible. Fixation was achieved with osmium and later with a combination of glutaric aldehyde and osmium. The dimensions of the helix are: width 450-600 Å, thickness of the line forming the helix 160 Å, distance between threads 250-500 Å. Helices with more than 10-20 threads were not found. If we assume that the helix is produced by polymerization of the ribosomes, about 150 of them may be present in the longest helix found. When the presence of helical ribosome structures is considered in different cell types, they are found mainly in differentiating cells where free ribosomes, not bound to membrane, are present. It seems that helical structures are needed not for the synthesis of proteins to be secreted but for the formation of the own, presumably filamentar, proteins of the cell. Detailed studies are needed to confirm this hypothesis. All 12 references are Western.

1/1

- 3 -

OLAH, Istvan

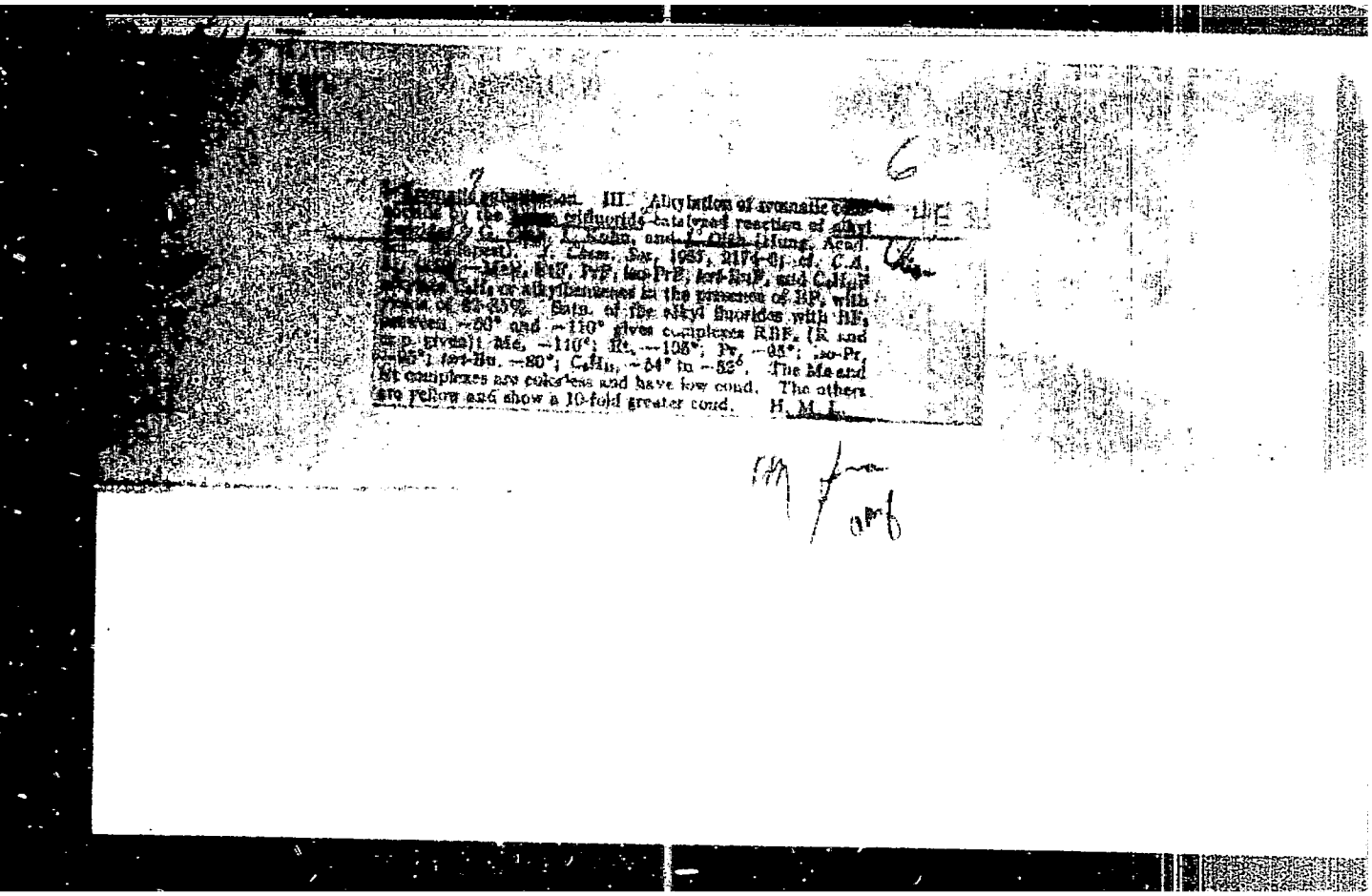
Operation of condenser loudspeakers. Radiotechnika 14 no.11:2 of cover N '64.

OLAH, Istvan

The AR 632 type "Panorama" radio set. Radiotechnika 14 no.7:268-270 Jl '64.

OLAH, Istvan

The AR 612 type "Pacsirta" radio receiver. Pt.2. Radiotechnika 14 no.8:299-302 Ag '64.



III. Alkylation of aromatic [redacted] fluorides—catalyzed reaction of alkyl [illegible] Acad. Sci., 1953, 2174-6; cf. C.A. [illegible] MeF, EtF, PrF, iso-PrF, tert-BuF, and $C_6H_{11}F$ [illegible] or alkylbenzenes in the presence of BF, with [illegible] of 85-95%. Rate of the alkyl fluorides with BF, between −50° and −110° gives complexes RBF. (R and [illegible] given): Me, −110°; Et, −108°; Pr, −95°; iso-Pr, −85°; tert-Bu, −80°; C_6H_{11}, −54° to −52°. The Me and Et complexes are colorless and have low cond. The others are yellow and show a 10-fold greater cond. H. M. L.

OLAH, J.

Mobile laboratory of soil mechanics. p. 243.

MELYEPITESTUDOMANYI SZEMLE. (Kozlekedes- es Kozlekedesepitestudomanyi Egyesulet) Budapest, Hungary, Vol. 9, no. 5, May 1959.

Monthly list of East European Accessions (EEAI), LC, Vol. 8, No. 8, August 1959.
Uncla.

HERMANN, Bela, dr.; OLAH, Jeno, dr.

Data on the etiopathogenesis of angina pectoris and myocardial infarct. Orv. hetil. 104 no.1:15-18 6 Ja '63.

1. Gyula Megyei Korhaz, I. Belosztaly.

(ANGINA PECTORIS) (MYOCARDIAL INFARCT) (STRESS)
(ERGOT ALKALOIDS, HYDROGENATED) (ACETYLCHOLINE)
(NEOSTIGMINE)

HERMANN, Bela, dr.; OLAH, Jeno, dr.; HELM, Vilmos, dr.

Relationship between angina pectoris and coronary atherosclerosis.
Orv. hetil. 105 no.32:1498-1500 9 Ag '64.

1. Gyulai Megyei Korhaz, 1. Belgyogyaszat es Korbonctani Osztaly.

BURKUS, Ferenc, okleveles gepeszmernok; RASZTOCZKY, Laszlo, okleveles mernok; OLAH, Jeno, okleveles mernok

Mechanization of road construction. Melyepitestud szemle 14 no.12:557-560 D '64.

1. Division Chief, Road and Railroad Planning Enterprise, Budapest (for Burkus). 2. Road and Railroad Planning Enterprise, Budapest (for Rasztoczky and Olah).

GYARMATI, Istvan, dr. (Budapest, XI., Budafoki ut 8); OLAH, Kalman (Budapest, XI., Budafoki ut 8)

Analysis of the rate entropy production by thermodynamic "oquations of motion." Acta chimica Hung 35 no.1:95-105 '63.

1. Department for Physical Chemistry, Technical University, Budapest.

OLAH, [redacted] Karoly] (Budapest XI, Sztoczek, u.2)

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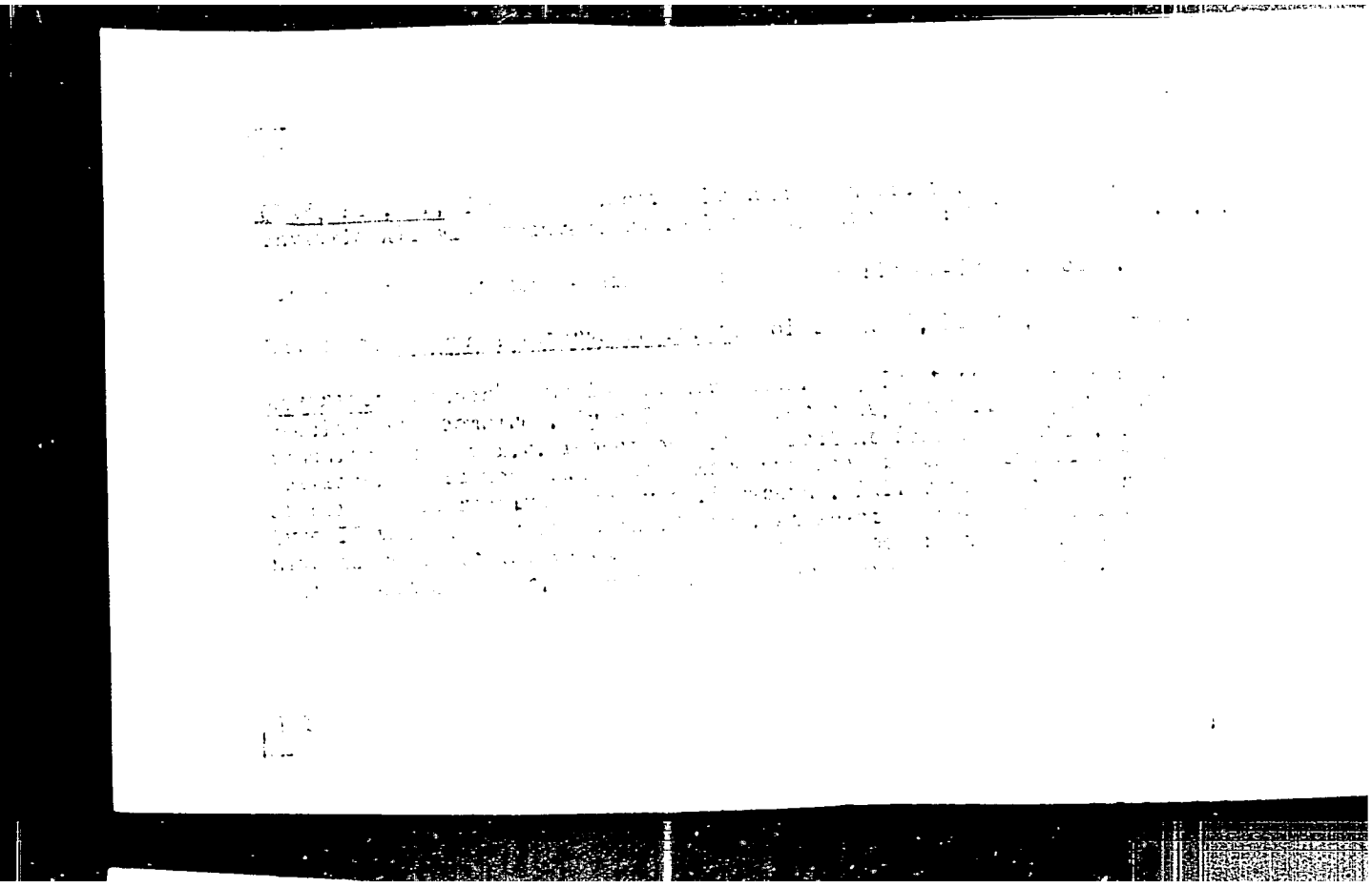

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Prague, Ceskoslovenska Fysiologie, Vol 15, No 2, Feb 66, p 75

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