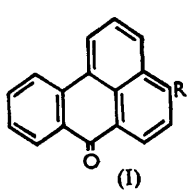
NOTES.

379. *Catalytic Action of Quinones in the Oxidation of* 4-*Methyl*-meso*benzanthrone to* meso*Benzanthrone*-4-*carboxylic Acid.*

By WILLIAM BRADLEY and K. H. SHAH.

THE catalytic effect of quinones on oxidation is well known [1] and the present is a further example in which the oxidant is nitrobenzene and barium hydroxide. In attempts to prepare *meso*benzanthrone-4-carboxylic acid (I; R = CO_2H) from 4-methyl*meso*benzanthrone (I; R = Me) by using an alkali and nitrobenzene with a trace of a copper salt the reaction was capricious and the yields were low. The addition of various quinones improved both the yield and the smoothness, 1 : 2-benzanthraquinone being the most effective of the quinones tried The Table shows the yields of *meso*benzanthrone-4-carboxylic acid prepared under the following conditions.[2] 4-Methyl*meso*benzanthrone (10 g.) and the selected quinone (0·5 g.) were stirred in nitrobenzene (100 ml.) at 160—180° for 2 hr. whilst barium hydroxide octahydrate (25 g.) and a trace of copper sulphate were added portionwise.



(I)

[1] Perkin and Spencer, *J.*, 1922, 479.
[2] B.P. 277,670; G.P. 576,176, 479,917; Copp and Simonsen, *J.*, 1942, 209.

Heating was continued for 1 hr. longer and then the alkali-soluble part of the product was separated.

Quinone	Yield of *meso*benzanthrone-4-carboxylic acid (g.)
None	1·0 or less
Anthraquinone	4·0
1 : 4 : 5 : 8-Tetrachloroanthraquinone	4·2
1 : 2-Benzanthraquinone	7·0
Anthanthrone	4·5
1′ : 2′-6′ : 7′-Dibenzopyrene-7 : 14-quinone	4·5
Flavanthrone	0·25

The relative amounts of 4-methyl*meso*benzanthrone, the quinones and nitrobenzene employed, and the yields of *meso*benzanthrone-4-carboxylic acid obtained indicate that nitrobenzene is the ultimate oxidant and that the quinones act as catalysts, presumably by functioning as hydrogen-acceptors. However, a high oxidation–reduction potential is not sufficient in itself. Flavanthrone which is very easily reduced is ineffective, probably on account of its very low solubility. No oxidation was observed when nitrobenzene was replaced by anisole.

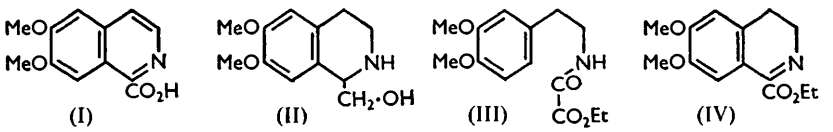
Department of Colour Chemistry and Dyeing,
The University, Leeds, 2.
[Present address (K. H. S.): The University, Bombay.] [*Received, December 19th*, 1958.]

---

380. *The Synthesis of Calycotomine.*

By A. R. Battersby and T. P. Edwards.

The alkaloid calycotomine, $C_{12}H_{17}O_3N$, has been isolated [1] in the optically active and the (±)-form from *Calycotome spinosa* and *Cytisus proliferus*. Its properties and the oxidation of the alkaloid to 6 : 7-dimethoxy*iso*quinoline-1-carboxylic acid (I) leave little doubt [2] that it has the structure (II).



A considerable quantity of calycotomine was required in connection with other work, and we describe here the synthesis of the alkaloid. This has not previously been accomplished though earlier attempts have been made.[2]

Ethoxalyl chloride with 3 : 4-dimethoxyphenethylamine gave the amide (III) together with the bisamide of oxalic acid. When the former was cyclised over phosphoric oxide in boiling toluene, it yielded, rather slowly, the 3 : 4-dihydro*iso*quinoline (IV). Both reducible groups in this base were attacked by lithium aluminium hydride in boiling tetrahydrofuran, and the product (II) was identical with natural (±)-calycotomine. The structure of the alkaloid is thus confirmed.

Experimental.—Ethyl N-3 : 4-*dimethoxyphenethyloxamate* (III). A solution of 3 : 4-dimethoxyphenethylamine (100 g.) in dry ether (500 ml.) was added slowly to a stirred solution of ethoxalyl chloride [3] (38 g.) in dry ether (750 ml.), and stirring was continued for 1 hr. after the addition was complete. Water (500 ml.) was then added and the solution was filtered to

[1] White, *New Zealand J. Sci. Tech.*, 1944, **25**, *B*, 152.
[2] *Idem, ibid.*, 1951, **33**, *B*, 38.
[3] Adickes, Brunnert, and Lucker, *J. prakt. Chem.*, 1931, **130**, 168.

remove the crude di-(3 : 4-dimethoxyphenethylamide) of oxalic acid, m. p. 165—171° (lit.,[4] m. p. 173—174°). The ethereal layer from the filtrate was washed with an excess of dilute hydrochloric acid, the washings were combined with the main aqueous solution, and this solution was then extracted thrice with ethyl acetate. Evaporation of the combined solutions in ether and ethyl acetate left a gum (74 g.) which crystallised from ether to give *ethyl* N-3 : 4-*dimethoxyphenethyloxamate* (54 g.), m. p. 71—72°. A portion was distilled at 150° (bath)/0·1 mm. for analysis (Found: C, 59·7; H, 4·9; N, 6·8. $C_{14}H_{19}O_5N$ requires C, 59·8; H, 4·9; N, 6·7%).

Ethyl 3 : 4-*dihydro*-6 : 7-*dimethoxy*isoquinoline-1-*carboxylate* (IV). " Celite " (50 g.), previously dried at 100°, was added to a solution of the foregoing amide (24 g.) in anhydrous toluene (300 ml.), and the mixture was heated under reflux. Phosphoric oxide (50 g.) was then added, followed by a further portion (25 g.) after 10 min. The mixture was then heated for 35 min., being stirred several times. After being cooled, the excess of phosphoric oxide was decomposed by water, and the " Celite " was filtered off and washed with dilute hydrochloric acid and hot benzene. The organic layer of the filtrate was shaken with dilute hydrochloric acid, and these washings were combined with the main aqueous layer. After the aqueous solution had been extracted with ether, it was adjusted to pH 9 with potassium carbonate and extracted thrice again with ether. Evaporation of the latter dried ethereal solution left the *dihydroisoquinoline* (IV) as a gum (6 g.) which crystallised from ether as needles, m. p. 79—80°.

Unchanged starting material (12 g.) was recovered by evaporation of the combined solution in toluene, benzene, and ether above and was cyclised as before, to give again a basic and a neutral fraction. The process was repeated on the latter fraction and the combined yield of dihydro*iso*quinoline (IV) was 10·8 g. (48%) (Found: C, 64·1; H, 6·7; N, 4·8. $C_{14}H_{17}O_4N$ requires C, 63·9; H, 6·5; N, 5·3%).

(±)-1 : 2 : 3 : 4-*Tetrahydro*-1-*hydroxymethyl*-6 : 7-*dimethoxy*isoquinoline (II). A solution of the above dihydro*iso*quinoline (1·04 g.) in anhydrous tetrahydrofuran (50 ml.) was added dropwise to a stirred, boiling solution of lithium aluminium hydride (0·45 g., 3 mol.) in tetrahydrofuran (50 ml.). The mixture was heated under reflux for 1 hr. After the addition of the minimum amount of water to decompose the excess of hydride, the solution was filtered, the collected solid was washed thoroughly with boiling chloroform, and the washings were added to the main solution. This was evaporated under reduced pressure to leave a solid (0·9 g.) which was dried over phosphoric oxide *in vacuo*, then boiled thrice with benzene (total 100 ml.). The extracts were concentrated to 25 ml., the tetrahydro*iso*quinoline (II) crystallising (0·46 g., 53%); it had m. p. 132—134° raised to 133·5—134·5° by further recrystallisation (Found: C, 64·3; H, 7·7; N, 6·2. Calc. for $C_{12}H_{17}O_3N$: C, 64·6; H, 7·6; N, 6·3%). The m. p. was unchanged on admixture with natural (±)-calycotomine, m. p. 133—134°, in the same bath. The infrared spectra of the synthetic and the natural base (in Nujol) were identical.

A portion of the synthetic base was converted into the hydrochloride which crystallised from ethanol and had m. p. 193—195° alone or in admixture with the hydrochloride of natural (±)-calycotomine of m. p. 194—196° in the same bath. The two samples of hydrochloride had identical infrared spectra (in Nujol).

Grateful acknowledgment is made to Dr. E. P. White, Hamilton, New Zealand, for generous gifts of calycotomine derivatives and to the University of Bristol and the Ministry of Education for grants to T. P. E.

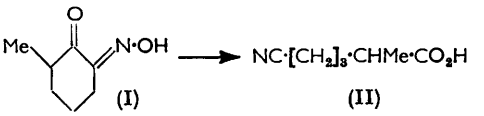
THE UNIVERSITY, BRISTOL. [*Received, January 19th,* 1959.]

[4] Child and Pyman, *J.*, 1929, 2010.

381. *The Second-order Beckmann Rearrangement of 2-Hydroxyimino-6-methylcyclohexanone.*

By E. B. McCall and B. B. Millward.

Wieland *et al.*[1] made use of the second-order Beckmann rearrangement [2] of an α-diketone monoxime to open a six-membered ring in a steroid nucleus. We now record the similar cleavage of 2-hydroxyimino-6-methyl*cyclo*hexanone (I) to 5-cyano-2-methylpentanoic acid (II).



(I) (II)

The oxime (I) was prepared by the method of Jaeger and van Dijk [3] but whereas they isolated two isomers, m. p. 167° (decomp.) and 65° respectively, we obtained one oxime, m. p. 80—82°. A satisfactory analysis for this unstable material could not be obtained but direct conversion into the cyano-acid (II) was effected in good yield by toluene-*p*-sulphonyl chloride in aqueous sodium hydroxide.

Experimental.—Ethyl 3-methyl-2-oxocyclohexanecarboxylate. This ester, b. p. 139—144°/32 mm., n_D^{20} 1·471, was prepared [4] in 44% yield from 2-methyl*cyclo*hexanone, n_D^{20} 1·449. The 2 : 4-*dinitrophenylhydrazone* crystallised from ethanol in orange leaflets, m. p. 87—87·5° (Found: C, 53·3; H, 5·5; N, 15·2. $C_{16}H_{20}O_6N_4$ requires C, 52·7; H, 5·5; N, 15·4%). The indazolone was formed as described for a thia-derivative of indazolone by Bennett and Scorah.[5] Recrystallisation from 95% methanol gave pale yellow prisms of 3 : 4 : 5 : 6 : 7 : 9-*hexahydro*-7-*methyl*-2-*phenylindazol*-3-*one*, m. p. 135—136° (Found: C, 73·6; H, 7·3; N, 12·1. $C_{14}H_{16}ON_2$ requires C, 73·6; H, 7·1; N, 12·3%).

Hydrolysis of the ester with 2N-sodium hydroxide solution followed by acidification regenerated 2-methyl*cyclo*hexanone, b. p. 70°/29 mm., n_D^{20} 1·449. The 2 : 4-dinitrophenylhydrazone crystallised from methanol in yellow leaflets, m. p. 139° (lit.,[6] m. p. 143°).

2-*Hydroxyimino*-6-*methyl*cyclo*hexanone* (I). The foregoing ester (36 g., 0·2 mole) was added to a stirred solution of potassium hydroxide (15 g., 0·27 mole) and sodium nitrite (13·8 g., 0·2 mole) in water (250 c.c.) under nitrogen. After 46 hr. the solution was cooled to 5° and treated dropwise with sulphuric acid (12·7 c.c. of 98% acid in 100 c.c. of water): carbon dioxide was liberated. The precipitated oxime (I) was collected, washed, and dried *in vacuo* to white prisms, m. p. 78—80° (18·2 g., 65%). Extraction of the filtrate with ether gave a crude oxime which crystallised from aqueous methanol in prisms, m. p. 80—82° (1·1 g.). The 2 : 4-*dinitrophenylhydrazone* crystallised from ethanol in orange prisms, m. p. 188—189° (Found: C, 48·8; H, 4·8; N, 21·6. $C_{13}H_{15}O_5N_5$ requires C, 48·6; H, 4·7; N, 21·8%).

5-*Cyano*-2-*methylpentanoic acid* (II). The oxime (9·5 g., 0·067 mole) was stirred in aqueous potassium hydroxide (12·8 g. in 44 c.c. of water) under nitrogen, and benzenesulphonyl chloride (9·8 c.c., 0·068 mole) was added dropwise (20 min.) at 20°. After a further 25 min. the solution was washed with chloroform, acidified, and extracted with chloroform, to yield 5-*cyano*-2-*methylpentanoic acid* (II), b. p. 142—143°/1·5 mm., n_D^{20} 1·451 (7·3 g., 77%) (Found: C, 59·9; H, 7·9; N, 9·8. $C_7H_{11}O_2N$ requires C, 59·6; H, 7·9; N, 9·9%). The p-*toluidide* crystallised from benzene in prisms, m. p. 86° (Found: C, 73·2; H, 7·5; N, 11·9. $C_{14}H_{18}ON_2$ requires C, 73·0; H, 7·9; N, 12·2%).

Hydrolysis of the cyano-acid with hot concentrated hydrochloric acid gave ammonium chloride and 2-methyladipic acid, which was crystallised with difficulty from benzene–light

[1] Wieland, Anner, and Miescher, *Helv. Chim. Acta*, 1953, **36**, 1803.
[2] Grob and Baumann, *ibid.*, 1955, **38**, 594.
[3] Jaeger and van Dijk, *Proc. Acad. Sci. Amsterdam*, 1936, **39**, 384.
[4] *Org. Synth.*, Coll. Vol. II, 1943, p. 531.
[5] Bennett and Scorah, *J.*, 1927, 196.
[6] Lecomte and Gault, *Compt. rend.*, 1954, **238**, 2538.

petroleum (b. p. 60—80°) in prisms, m. p. 55—60° (Found: C, 52·6; H, 7·9. Calc. for $C_7H_{12}O_4$: C, 52·5; H, 7·6%). Best and Thorpe [7] found m. p. 64°.

We thank Monsanto Chemicals Limited for permission to publish this work.

NICKELL LABORATORIES, MONSANTO CHEMICALS LIMITED,
RUABON, NORTH WALES. [*Received, January 29th,* 1959.]

[7] Best and Thorpe, *J.*, 1909, **95**, 712.

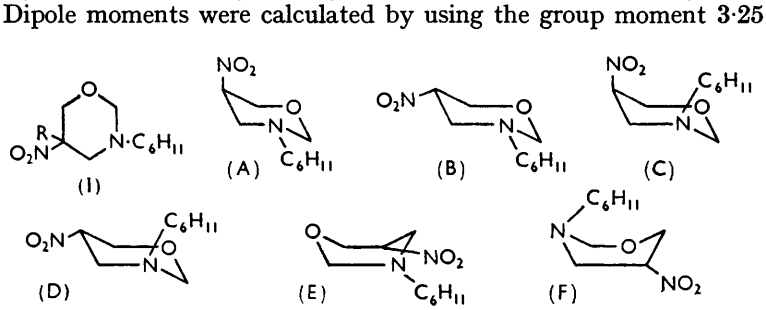
---

382. *The Stereochemistry of Some Tetrahydro-1 : 3-oxazine Derivatives.*

By (MRS.) D. GÜRNE and T. URBANSKI.

THE preparation of derivatives of tetrahydro-5-nitro-1 : 3-oxazine derivatives from primary nitro-paraffins, formaldehyde, and ammonia or primary amines has been described.[1] Their stereochemistry has now been examined. The dipole moments indicate that the ring is in the chair form with nitro- and the *cyclo*hexyl group in the axial and the equatorial conformation respectively.

We prepared the products (I; R = Me, Et, Pr^n, Pr^i, and Bu^n) from nitroethane,[2] 1-nitropropane,[3] 1-nitrobutane,[3] 1-nitro*iso*butane, and 1-nitropentane respectively by condensation with formaldehyde and treatment of the resulting 1 : 3-diols with formaldehyde and *cyclo*hexylamine. *cyclo*Hexylamine was chosen as it yields crystalline products. Dipole moments were calculated by using the group moment 3·25 D for NO_2



and the bond moments C–O 0·86 (bond length 1·43 Å) and C–N 0·45 D (bond length 1·47 Å), and the CNC and COC angles 111° and 110° 42′ respectively. Experimental values found were 4·45, 4·42, 4·41, 4·56, 4·46 D for the compounds (I; R = Me, Et, Pr^n, Pr^i, Bu^n respectively); these were referred to the chair form (A) with an axial nitro-group, for which the calculated value is 4·37 D. For form (B) with an equatorial nitro-group the calculated value is distinctly lower (μ 2·73 D). For both these calculations the *cyclo*hexyl ring is assumed to be equatorial since the axial conformations (C and D) give lower values (3·74 and 2·56 D respectively).

Boat forms give calculated dipole moments ranging from 2·17 (lowest) (conformation E) to 3·74 D (highest) (conformation F).

EXPERIMENTAL

3-cyclo*Hexyltetrahydro-5-nitro*-5-iso*propyl*-1 : 3-*oxazine*.—2-Nitro-2-*iso*propylpropane-1 : 3-diol (prepared from 1-nitro*iso*butane and formaldehyde) (0·1 mol., 16·3 g.) was mixed with 30%

[1] Hirst, Jones, Minahan, Ochynski, Thomas, and Urbański, *J.*, 1947, 924; Urbański and Lipska, *Roczniki Chem.*, 1952, **26**, 182; Urbański, Kolesińska, and Piotrowska, *Bull. Acad. Polon. Sci., Cl. III*, 1955, **3**, 179; Gürne and Urbański, *ibid.*, p. 175; *Roczniki Chem.*, 1957, **31**, 855; Eckstein, Sobótka, and Urbanski, *ibid.*, 1956, **30**, 133.

[2] Senkus, *J. Amer. Chem. Soc.*, 1950, **72**, 2967.

[3] Gürne and Urbański, *Bull. Acad. Polon. Sci., Cl. III*, 1956, **11**, 221; *Roczniki Chem.*, 1957, **31**, 869.

aqueous formaldehyde (0·15 mol., 15 ml.). Sodium hydrogen carbonate (0·5 g.) and freshly distilled *cyclo*hexylamine (0·1 mol., 9·9 g.) were added dropwise. The temperature rose by 10—15° and was kept at 60—65° for 3 hr., with stirring. The colourless resinous *oxazine* was washed with water, dissolved in hot ethanol, and left to crystallise (yield, 8·5 g., 33%). On repeated crystallisation from ethanol it formed needles, m. p. 55—57° (Found: C, 60·8; H, 9·8; N, 11·4. $C_{13}H_{24}O_3N_2$ requires C, 61·0; H, 9·5; N, 10·9%).

5-Butyl-3-cyclohexyltetrahydro-5-nitro-1 : 3-oxazine.—2-Butyl-2-nitropropane-1 : 3-diol (prepared from 1-nitropentane) (0·1 mol., 17·7 g.) with 30% aqueous formaldehyde (0·15 mol.) and *cyclo*hexylamine (0·1 mol.) in presence of hydrogen sodium carbonate (0·5 g.) gave, as above, the *oxazine* (14·5 g., 53%), needles, m. p. 57—59° (from alcohol) (Found: C, 62·2; H, 9·5; N, 10·8. $C_{14}H_{26}O_3N_2$ requires C, 62·3; H, 9·7; N, 10·4%).

Dipole Moments.—These were measured by the heterodyne-beat method, with benzene as a solvent, at 20°. The results were extrapolated by Le Fèvre and Vine's method. The experimental error was ±0·1 D.

The authors are much indebted to Professor W. Tomassi, Dr. H. Calus, and Miss H. Jankowska for measuring the dipole moments.

INSTITUTE OF ORGANIC SYNTHESIS, POLISH ACADEMY OF SCIENCES,
WARSAW 10, POLAND. [*Received, December 23rd*, 1958.]

---

383. 5- *and* 6-*Fluoro*-3-*indolylacetic Acid.*

By ERNST D. BERGMANN and ZVI PELCHOWICZ.

THE observations that 5-fluorotryptophan is an antagonist of tryptophan,[1] and 5-fluorotryptamine an antagonist of tryptamine,[2] led us to study the biological relation of 5- and 6-fluoro-3-indolylacetic acid to 3-indolyl-2-acetic acid (heteroauxin).

The two fluoro-compounds were synthesised by a conventional method:[3] the respective fluoroindoles were converted by formaldehyde and dimethylamine into 5- and 6-fluorogramine, and these compounds by potassium cyanide into the 5- and 6-fluoro-3-indolylacetonitrile, which were hydrolysed without isolation. The overall yields were 80%.

Preliminary experiments kindly carried out by Professor M. Evenari, Department of Botany, Hebrew University, have shown that the two compounds are germination-inhibitors.

Experimental.—5-Fluoroindole has been prepared by Allen, Brunton, and Suschitzky [4] from ethyl pyruvate *p*-fluorophenylhydrazone by Fischer's method, followed by hydrolysis and decarboxylation. We used Reissert's method.

5-Fluoro-2-nitrotoluene. 3-Methyl-4-nitroaniline, prepared [5] from *N*-acetyl-*m*-toluidine [m. p. 138° (from 50% ethanol)] (14 g.) was dissolved in hot concentrated hydrochloric acid (30 ml.), cooled in ice-salt, and treated with sodium nitrite (7·6 g.) in water (20 ml.). To the filtered solution, 50% fluoroboric acid (24 ml.) was added and the fluoroborate (11 g., 48%) filtered off, washed, and dried. It was mixed with half its quantity of talcum powder and decomposed in small batches at 100°. The product (30%), isolated by distillation with steam and extraction of the distillate with ether, boiled at 114°/30 mm. It has been obtained before by nitration of *m*-fluorotoluene.[6]

5-Fluoroindole-2-carboxylic acid. To a mixture of anhydrous ether (5 ml.) and anhydrous ethanol (10 ml.), potassium (3·2 g.) was added; when the reaction became slow, more ethanol (17 ml.) was added. After the metal had dissolved, the product was diluted with ether (125 ml.) and cooled, and successively ethyl oxalate (12·3 g.) and 5-fluoro-2-nitrotoluene (11 g.) were

[1] Bergmann, *Proc. k. ned. Akad. Wetenschap.*, 1954, *C*, **57**, 108; Sharon and Lipmann, *Arch. Biochem. Biophys.*, 1957, **69**, 219.
[2] Unpublished results from our laboratories.
[3] Snyder and Pilgrim, *J. Amer. Chem. Soc.*, 1948, **70**, 3770.
[4] Allen, Brunton, and Suschitzky, *J.*, 1955, 1283.
[5] Wibaut, *Rec. Trav. chim.*, 1913, **32**, 287.
[6] Schiemann, *Ber.*, 1929, **62**, 1794.

added. A red solid slowly separated and was filtered off after 24 hr. and dried. This potassium 5-fluoro-2-nitrophenylpyruvate (15·5 g., 75%) was heated at 100° (reflux condenser) with 35% ammonia solution (150 ml.) and water (175 ml.), with stirring, and a solution of ferrous sulphate heptahydrate (157·5 g.) in water (175 ml.) was added. After a further hour's heating, the solution was filtered and acidified. The solid powder so obtained was filtered, dried, and recrystallised from 50% alcohol. The product melted at 233° (decomp.); Allen *et al.*[4] reported 248—249° (decomp.).

5-*Fluoroindole.* The ammonium salt of the foregoing acid was heated at 250° for 30 min. and the product isolated by steam-distillation. It formed colourless plates, m. p. 46° (lit.,[4] 46°) (20%) (Found: C, 71·2; H, 4·3. Calc. for C_8H_6NF: C, 71·1; H, 4·4%).

6-*Fluoroindole.* The Reissert synthesis was employed (cf. Allen *et al.*[4]). 4-Fluoro-2-nitrotoluene [8] (b. p. 107—109°/24 mm., 56—57°/0·2 mm.) was obtained, as described above, from 4-methyl-3-nitroaniline. 4-Fluoro-2-nitrophenylpyruvic acid, recrystallised from aqueous alcohol, melted at 135°, 6-fluoroindole-2-carboxylic acid, recrystallised from xylene, at 246° (lit., 246°), and 6-fluoroindole at 72° (lit., 75°).

5-*Fluorogramine.* An ice-cold solution of 25% aqueous dimethylamine solution (1·8 ml.), glacial acetic acid (1 ml.) and 40% aqueous formaldehyde (0·75 ml.) was added to solid 5-fluoroindole (1·25 g.), which dissolved quickly. After 24 hr. at room temperature, an excess of 10% aqueous potassium hydroxide was added, and the precipitate filtered off and recrystallised from acetone (yield quantitative; m. p. 139—141°) (Found: C, 68·5; H, 6·7; N, 14·3. Calc. for $C_{11}H_{13}N_2F$: C, 68·7; H, 6·7; N, 14·6%). Quadbeck and Roehm [8] report m. p. 145—146°.

6-*Fluorogramine*, obtained analogously and in the same yield, and recrystallised from acetone, melted at 131—132·5° (Found: C, 68·4; H, 6·6; N, 14·5%).

5-*Fluoro*-3-*indolylacetic acid.* To a solution of 5-fluorogramine (22 g.) in ethanol (250 ml.), a solution of potassium cyanide (44 g.) in water (70 ml.) was added and the mixture refluxed for 90 hr., neutralised with hydrochloric acid, and evaporated to dryness. The residue was refluxed for 4 hr. with 2N-potassium hydroxide (150 ml.), and the solution was acidified with dilute hydrochloric acid and thoroughly extracted with ether. The ether residue (17·5 g., 80%) was purified by dissolution in sodium hydrogen carbonate (charcoal) and acidification and eventually by recrystallisation from isopropyl alcohol. This *acid* had m. p. 140—141° (Found: C, 62·1; H, 4·0; F, 10·3; N, 7·6. $C_{10}H_8O_2NF$ requires C, 62·2; H, 4·3; F, 10·0; N, 7·3%).

6-*Fluoro*-3-*indolylacetic acid*, prepared in the same manner and in the same yield, had m. p. 165—165·5° (from isopropyl alcohol) (Found: C, 62·3; H, 4·0; F, 10·4; N, 7·3%).

SCIENTIFIC DEPARTMENT, ISRAEL MINISTRY OF DEFENCE,
TEL-AVIV, ISRAEL.

[*Received, December 29th*, 1958.]

[7] Steck and Fletcher, *J. Amer. Chem. Soc.*, 1948, **70**, 439; Suschitzky, *J.*, 1953, **3326**.
[8] Quadbeck and Roehm, *Z. physiol. Chem.*, 1954, **297**, 229.