323. *The Synthesis of* N-*Alkyl*-2-*oxo*cyclo*pentanecarboxyamides.*

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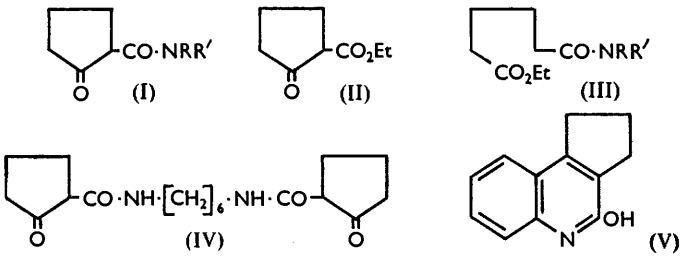
N-Alkyl-2-oxo*cyclo*pentanecarboxyamides result from the action of metallic sodium on *N*-alkyladipamic esters in boiling benzene. Yields are low for the lower homologues of the series but rise to 60—70% for amides derived from amines having a chain-length of 4 carbon atoms or more. The method is not applicable to the synthesis of *NN*-dialkyl-2-oxo*cyclo*pentanecarboxyamides.

N-Alkyl-2-oxo*cyclo*pentanecarboxyamides (I; R = H, R′ = Alkyl), which were required for studies of the thermal decomposition of Nylon 66 polymer during 1952—1956,[1] appear to have attracted no previous attention. Although several *N*-aryl analogues (I; R = H, R′ = Aryl), prepared by reaction of the ester (II) with amines, have been reported,[2] attempts by Mr. O. B. Edgar of these laboratories to extend these procedures to the aliphatic series were relatively unsuccessful. We have now found, however, that ethyl *N*-alkyladipamates (III; R = H, R′ = Alk) cyclise to the requisite β-keto-amides (I) under the base-catalysed conditions normally employed for the classical Dieckmann condensation,[3] *e.g.*, with sodium in boiling benzene, the experimental procedure being based



upon the optimum conditions for conversion of diethyl adipate into ethyl 2-oxo*cyclo*pentanecarboxylate (II). These conditions include the addition of a small amount of ethanol to eliminate or reduce any induction period,[4] and use of 1·4—1·5 atomic proportions of sodium.[5] The yields of amides (I) were lowest from amines of short chain-length and progressively increased to 60—70% for amines of chain-length equal to or greater than C_4 (see Table 2, p. 1626). The structure of a cyclisation product was demonstrated conclusively only for the butylamide, the structures of the homologues being inferred from their similar infrared absorption spectra and chemical properties.

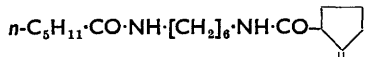
The butylamide afforded a crystalline 2 : 4-dinitrophenylhydrazone and semicarbazone and with ethanolic ferric chloride gave an intense royal blue colour similar to that given by the ester (II). Its infrared absorption was characterised by principal bands at 1742 cm.$^{-1}$ (*cyclo*pentanone ring), 3356, 3135, 1645, and 1507 cm.$^{-1}$ (CO·NH), and 2959, 2915, 1475, 1464, and 1385 cm.$^{-1}$ (CH_2 and Me). In hydrolytic behaviour the amide paralleled the ester: thus " ketonic " fission ensued in boiling 5N-hydrochloric acid, and " acidic " fission in boiling 20% aqueous sodium hydroxide.

The cyclisation procedure has been extended to the preparation, in 81% yield, of the

[1] Goodman, *J. Polymer Sci.*, 1954, **13**, 175; 1955, **17**, 587.
[2] (*a*) Blount, Perkin, and Plant, *J.*, 1929, 1975; (*b*) Linstead and Bao-Lang Wang, *J.*, 1937, 707; Ahmad and Desai, *Proc. Indian Acad. Sci.*, 1937, **5**, *A*, 543; Barany and Pianka, *J.*, 1947, 1420; Clemo and Mishra, *J.*, 1953, 192.
[3] Dieckmann, *Ber.*, 1894, **27**, 102, 965.
[4] Linstead and Meade, *J.*, 1934, 940.
[5] Pinkney, *Org. Synth.*, Coll. Vol. II, p. 116; Cornubert and Borrel, *Bull. Soc. chim. France*, 1930, **47**, 301.

di-β-ketoamide (IV) from the ester (III; R = H, R′ = $[CH_2]_6$·NH·CO·$[CH_2]_4$·CO_2Et) with 2·9 atomic proportions of sodium, and to the formation, in 67% yield from ethyl adipanilate (III; R = H, R′ = Ph), of 2-oxo-*N*-phenyl*cyclo*pentanecarboxyamide (I; R = H, R′ = Ph),[2] which, with concentrated sulphuric acid at 100°, gave 2-hydroxy-3 : 4-*cyclo*pentenoquinoline [2a, 6] (V). However, by the Dieckmann procedure ethyl *NN*-dialkyladipamates, such as (III; R = R′ = Pr^n or Bu^n), gave resins having none of the expected properties, whilst ethyl adipamate (III; R = R′ = H) under these conditions furnished only 9% of the ester (II) and no amide. Hence the reaction appears to be restricted to esters derived from primary amines. That cyclisation proceeds at all, however, is somewhat surprising in view of the greater electron-attracting propensities of ester groupings than of amides. This feature would be expected to lead to activation of the methylene group adjacent to the ethoxycarbonyl system rather than to that of the CH_2 in the α-position to the CO·NHR.[7] Activation of the appropriate methylene site may conceivably arise by a charge redistribution within an ionised transition state or by interchange of ester and amide groups within such a state.

The foregoing cyclisation has been used in a synthesis of an amide carrying an extended



(VI)

(VII)

(VIII)

N-substituent. Thus, the diamide (VI) has been obtained in 65% yield from the ester (VII; R = EtO_2C·$[CH_2]_4$·CO), which was prepared from 6-aminohexanonitrile (VIII; R = H) by way of (VIII; R = *n*-C_5H_{11}·CO) and (VII; R = H).

Experimental

Ethyl Adipamate (III; R = R′ = H).—Prepared by ammonolysis [8] of the acid chloride [9] (b. p. 114°/11 mm.) of ethyl hydrogen adipate [10] (b. p. 106—108°/0·1 mm., m. p. 28—29°), this ester formed colourless needles, m. p. 77°, from ethyl acetate–light petroleum (b. p. 80—100°) (Found: C, 55·3; H, 8·7; N, 8·0. Calc. for $C_8H_{15}O_3N$: C, 55·5; H, 8·7; N, 8·1%). Rauscher and Tucker [8] give m. p. 77—78°.

Ethyl N-*Alkyladipamates* (III; R = H, R′ = Alk).—The acid chloride (204 g., 1·06 moles) of ethyl hydrogen adipate in benzene (200 ml.) was added during 90 min. to a stirred mixture of the amine (1·07 moles), pyridine (93·5 g., 1·18 moles), and benzene (200 ml.) at 0—5°. Then the mixture was kept at 100° for 2 hr., benzene and excess of pyridine were removed in steam, and the product was extracted with chloroform (2 × 250 ml.; 3 × 75 ml.). After washings with 2N-hydrochloric acid (3 × 100 ml.), water (2 × 100 ml.), aqueous sodium hydrogen carbonate (2 × 100 ml.), and water (2 × 100 ml.), the combined chloroform solutions were dried ($MgSO_4$) and evaporated. Distillation of the residue afforded a small forerun and then the pure *ethyl* N-*alkyladipamate* (see Table 1).

N-*Alkyl*-2-*oxo*cyclo*pentanecarboxyamides* (I; R = H, R′ = Alkyl).—The ethyl *N*-alkyladipamate (0·874 mole) and ethanol (2 ml.) were added in one portion to an agitated suspension of powdered sodium (30 g., 1·305 g.-atom) in benzene (750 ml.), and the mixture gradually heated to boiling under reflux. Reaction quickly set in with, in the early stages, much frothing; the sodio-derivative began to separate after 2 hr. The mixture was boiled under reflux with stirring for 20 hr., then cooled and poured into 2N-hydrochloric acid (750 ml.), and the benzene layer separated. The aqueous residue was extracted further with benzene (5 × 75 ml.), and the combined benzene solutions were washed with aqueous sodium hydrogen carbonate

[6] (*a*) Beer, McGrath, and Robertson, *J.*, 1950, 3283; (*b*) Witkop, Patrick, and Rosenblum, *J. Amer. Chem. Soc.*, 1951, **73**, 2641.
[7] Cf. Hauser and Renfrow, *ibid.*, 1937, **59**, 1823; 1938, **60**, 463.
[8] Rauscher and Tucker, *J. Amer. Chem. Soc.*, 1954, **76**, 3599.
[9] Blaise and Koehler, *Bull. Soc. chim. France*, 1910, **7**, 219.
[10] Brown, Armstrong, Moyer, Anslow, Baker, Querry, Bernstein, and Safir, *J. Org. Chem.*, 1947, **12**, 163.

(6 × 75 ml.) and water (2 × 75 ml.); these washings which contained some unidentified acid (*N*-alkyladipamic acid?) were discarded. The dried benzene solution was evaporated, and distillation of the residue then afforded the N-*alkyl*-2-*oxo*cyclo*pentanecarboxyamides* (see Table 2).

TABLE 1. *Ethyl* N-*alkyladipamates* (III).

No.	R	R′	Yield (%)	B. p./mm.	n_D^{20}	Forerun Yield * (%)	Forerun B. p./mm.
1	H	Me	61·6	132—134°/0·1	1·4575	15·5	60—120°/0·1
2	H	Et	79·3	124—126/0·1	1·4560	8·3	62—117/0·1
3	H	Pr^n	72·0	138—140/0·15	1·4549	4·2	80—136/0·15
4	H	Bu^n	81·5	134/0·09	1·4540	4·7	80—130/0·09
5	H	*n*-C_6H_{13}	75·5	158—160/0·15	1·4568	8·0	70—143/0·15
6	H	Ph	79·2	194—196/0·45	1·5273	4·7	94—198/0·5
7	Pr^n	Pr^n	66·9	119—120/0·1	1·4549	13·7	86—130/0·1
8	Bu^n	Bu^n	71·0	136—138/0·15	1·4569	14·5	82—140/0·15
9	H	$[CH_2]_6$·NH·CO·$[CH_2]_4$·CO_2Et	87·0	M. p. 111—112° (from EtOAc)	—	—	—

No.	Found (%) C	H	N	Formula	Required (%) C	H	N
1	57·3	9·2	7·7	$C_9H_{17}O_3N$	57·7	9·15	7·5
2	59·9	9·5	6·4	$C_{10}H_{19}O_3N$	59·7	9·5	7·0
3	61·5	9·7	6·3	$C_{11}H_{21}O_3N$	61·4	9·8	6·5
4	63·0	10·0	5·6	$C_{12}H_{23}O_3N$	62·85	10·1	6·1
5	64·9	10·0	4·9	$C_{14}H_{27}O_3N$	65·3	10·6	5·4
6	66·9	7·3	5·3	$C_{14}H_{19}O_3N$	67·4	7·7	5·6
7	65·0	10·3	5·2	$C_{14}H_{27}O_3N$	65·3	10·6	5·4
8	67·3	11·0	5·0	$C_{16}H_{31}O_3N$	67·3	10·95	4·9
9	61·5	9·4	6·0	$C_{22}H_{40}O_6N_2$	61·65	9·4	6·5

* Calc. as ethyl *N*-alkyladipamate.

TABLE 2. N-*Substituted* 2-*oxo*cyclo*pentanecarboxyamides* (I).

R	R′	Yield (%)	B.p./mm.	M. p.	Found (%) C	H	N	Formula	Required (%) C	H	N
H	Me	9·5	80°/0·09	—	Unsatisfactory			$C_7H_{11}O_2N$	59·55	7·85	9·9
H	Et	38·0	—	84° [a]	62·0	8·1	9·1	$C_8H_{13}O_2N$	61·9	8·4	9·0
H	Pr^n	47·0	98/0·2	—	63·7	8·9	8·0	$C_9H_{15}O_2N$	63·9	8·9	8·3
H	Bu^n	65·0	103/0·05	—	65·5	9·5	7·7	$C_{10}H_{17}O_2N$	65·5	9·35	7·65
H	*n*-C_6H_{13}	62·0	130/0·1	—	68·4	9·8	6·4	$C_{12}H_{21}O_2N$	68·2	10·0	6·6
H	Ph	67·0	—	102—103 [b]	70·3	6·3	6·9	$C_{12}H_{13}O_2N$	70·9	6·5	6·9
H	*	81·0	—	98 [c]	63·9	8·6	7·7	$C_{18}H_{28}O_4N_2$	64·3	8·4	8·3

[a] From EtOAc. [b] From EtOAc–light petroleum (b. p. 80—100°) (lit.,[2,6] m. p. 104°). [c] From EtOAc–light petroleum (b. p. 60—80°).

* 6-(2-Oxo*cyclo*pentanecarboxyamido)hexyl [cf. (IV)].

TABLE 3. 2 : 4-*Dinitrophenylhydrazones of amides from Table* 2.

R	R′	Solvent ‡	M. p.	Found (%) C	H	N	Formula	Required (%) C	H	N
H	Me	M	231—232° †	48·6	5·0	21·3	$C_{13}H_{15}O_5N_5$	48·6	4·7	21·8
H	Et	E	175—176	50·1	5·5	20·6	$C_{14}H_{17}O_5N_5$	50·1	5·1	20·9
H	Pr^n	E	177—178	51·2	5·3	20·3	$C_{15}H_{19}O_5N_5$	51·6	5·5	20·05
H	Bu^n	E	168	53·1	5·8	19·6	$C_{18}H_{21}O_5N_5$	52·9	5·8	19·3
H	*n*-C_6H_{13}	E	148	55·3	6·6	17·5	$C_{18}H_{25}O_5N_5$	55·2	6·4	17·9
H	Ph	E	190 [a]	56·7	4·6	18·4	$C_{18}H_{17}O_5N_5$	56·4	4·5	18·3
H	*	M	214 †	51·7	5·5	20·4	$C_{30}H_{36}O_{10}N_{20}$	51·7	5·2	20·1

* As footnote of Table 2. † With decomp. ‡ E = ethanol; M = 2-methoxyethanol.

[a] Beer *et al.*[6a] give m. p. 182°.

The 2 : 4-*dinitrophenylhydrazones* (see Table 3) were prepared in boiling ethanol containing hydrochloric acid, and the *semicarbazone* derivatives (see Table 4) by the sodium acetate method. Apart from 2-oxo-*N*-phenyl*cyclo*pentanecarboxyamide (I; R = H, R′ = Ph) which gave an intense green-blue colour, all the keto-amides had intense royal-blue ferric reactions in ethanol.

2-*Hydroxy*-3 : 4-cyclo*pentenoquinoline* (V).—A solution of *N*-phenyl-2-oxo*cyclo*pentanecarboxyamide (1 g.) in concentrated sulphuric acid (7·5 ml.) was kept at 100° for 15 min., then poured into water (400 ml.). Purification of the resulting precipitate, from aqueous acetic acid, gave 2-hydroxy-3 : 4-*cyclo*pentenoquinoline in pale yellow needles, m. p. 259° (Found: C, 77·9;

TABLE 4. *Semicarbazones of amides from Table 2.*

				Found (%)				Required (%)		
R	R′	Solvent *	M. p.	C	H	N	Formula	C	H	N
H	Me	M	219—220°	48·6	6·9	28·1	$C_8H_{14}O_2N_4$	48·5	7·1	28·3
H	Et	M	207—208	51·3	7·5	26·6	$C_9H_{16}O_2N_4$	50·9	7·6	26·4
H	Pr^n	M	210	53·2	8·5	—	$C_{10}H_{18}O_2N_4$	53·1	8·0	24·8
H	Bu^n	M	208 (decomp.)	55·0	8·3	23·1	$C_{11}H_{20}O_2N_4$	55·0	8·4	23·3
H	*n*-C_6H_{13}	M	209	58·2	8·9	20·7	$C_{13}H_{24}O_2N_4$	58·2	9·0	20·9
H	Ph	E	220—221 †	60·6	6·1	21·5	$C_{13}H_{16}O_2N_4$	60·0	6·2	21·5

* As in Table 3. † With decomp. (slow heating); 227° (decomp.) (rapid heating).

H, 6·1; N, 7·0. Calc. for $C_{12}H_{11}ON$: C, 77·8; H, 6·0; N, 7·6%), which gave a pale orange ferric reaction in alcohol and had an infrared absorption spectrum identical with that recorded by Witkop *et al.*[6] (lit., m. p. 256°,[2a, 6b] 262° [6a]).

Attempted Cyclisation of Ethyl Adipamate (III; R = R′ = H).—Ethyl adipamate (III; R = R′ = H) (7·0 g.) with sodium (1·35 g.) in benzene (100 ml.) gave ethyl 2-oxo*cyclo*pentanecarboxylate (0·58 g.), b. p. 116—118°/32 mm., n_D^{20} 1·4571 (blue ferric reaction in alcohol; infrared absorption) (Found: C, 60·9; H, 8·0. Calc. for $C_8H_{12}O_3$: C, 61·5; H, 7·75%). The 2 : 4-dinitrophenylhydrazone separated from ethanol in yellow needles, m. p. and mixed m. p. 128° (Found: C, 50·3; H, 4·9; N, 16·7. Calc. for $C_{14}H_{16}O_6N_4$: C, 50·0; H, 4·8; N, 16·7%).

Hydrolysis of N-*Butyl*-2-*oxo*cyclo*pentanonecarboxyamide*.—(a) *With* 5N-*hydrochloric acid*. The amide (5·7146 g., 1 mol.) was boiled under reflux for 3 hr. with 5N-hydrochloric acid (100 ml.) whilst nitrogen was led through the mixture; carbon dioxide (1·3662 g., 0·995 mol.) in the dried gases was absorbed on " Carbosorb " soda-lime asbestos. The aqueous residue was steam-distilled until a drop of the distillate no longer gave a precipitate with acidic 2 : 4-dinitrophenylhydrazine solution. The total steam-distillate was then diluted to 500 ml. and a 100 ml. portion was boiled under reflux for 1 hr. with 2 : 4-dinitrophenylhydrazine (1·35 g.) in concentrated sulphuric acid (35 ml.) and water (270 ml.). The cooled mixture was kept at 20° overnight and, next day, the yellow precipitate (1·4528 g., 0·88 mol.) was collected, washed, dried, and recrystallised from methanol to yield *cyclo*pentanone 2 : 4-dinitrophenylhydrazone, m. p. and mixed m. p. 144° (Found: C, 50·1; H, 4·4; N, 21·5. Calc. for $C_{11}H_{12}O_4N_4$: C, 50·0; H, 4·6; N, 21·2%).

The acidic residue from the steam-distillation was basified with 20% sodium hydroxide solution and boiled under reflux in a current of nitrogen. The gases were led through N-hydrochloric acid (50·00 ml.) and found by back-titration to contain *n*-butylamine (0·946 mol.), which was isolated as the 2 : 4-dinitrophenyl derivative, yellow needles (from methanol), m. p. and mixed m. p. 92°.

(b) *With* 20% *aqueous sodium hydroxide*. The amide (6·2767 g., 1 mol.) was boiled under reflux for 5 hr. with 20% aqueous sodium hydroxide (100 ml.) in a current of nitrogen. The gases were led into N-hydrochloric acid (50·00 ml.) and found by back-titration to contain *n*-butylamine (0·921 mol.), isolated as N-*n*-butyl-2 : 4-dinitroaniline, m. p. and mixed m. p. 92°.

No significant quantity of carbon dioxide (as measured by absorption on " Carbosorb " soda-lime asbestos) was liberated when the residual alkaline hydrolysate was acidified and boiled under reflux for 2 hr., but, from the cooled mixture, adipic acid (4·00 g., 0·799 mol.) was isolated by continuous ether-extraction and purified from water in needles, m. p. and mixed m. p. 151—152°.

N-(5-*Cyanopentyl*)*hexanamide* (VIII; R = *n*-C_5H_{11}·CO).—Hexanoyl chloride (37 g.) in benzene (35 ml.) was added dropwise to a stirred mixture of 6-aminohexanonitrile (30·8 g.), pyridine (21·7 g.), and benzene (120 ml.) at 0—5°. The mixture was stirred overnight at room temperature and next day was heated at 100° for 1 hr. Benzene and excess of pyridine were removed in steam, and the residue was extracted with chloroform (6 × 50 ml.). After successive washings with 2N-hydrochloric acid (6 × 75 ml.), aqueous sodium hydrogen carbonate

(6 × 50 ml.), and water (3 × 50 ml.), the extract was dried and the solvent evaporated. Distillation of the residue afforded N-(5-*cyanopentyl*)*hexanamide* (49 g.), b. p. 168°/0·1 mm. (Found: C, 69·2; H, 10·8; N, 13·1. $C_{12}H_{22}ON_2$ requires C, 68·5; H, 10·5; N, 13·3%).

N-(6-*Aminohexyl*)*hexanamide* (VII; R = H).—Hydrogen (15·4 l., 2·8 mol.) was absorbed when *N*-(5-cyanopentyl)hexanamide (49 g.) in dioxan (200 ml.) was hydrogenated at 100°/150 atm. over cobalt–kieselguhr (10 g.) in the presence of anhydrous ammonia (35 ml.). After catalyst had been removed, the solution was acidified with 2N-hydrochloric acid and evaporated to dryness. An aqueous solution (200 ml.) of the residue was washed with chloroform (3 × 75 ml.), then basified with 10% aqueous sodium hydroxide and extracted with chloroform (7 × 75 ml.). Distillation of the residue left on evaporation of the dried extract afforded *N*-(6-aminohexyl)hexanamide (43 g.), b. p. 148°/0·1 mm.; this compound rapidly carbonated and analysis was unsatisfactory.

Acylation with hexanoyl chloride and pyridine at 100° furnished 1 : 6-dihexanamidohexane, which crystallised from ethyl acetate in needles, m. p. and mixed m. p. 142—143° (Goodman,[11] 141—142°) (Found: C, 68·7; H, 11·6; N, 8·6. Calc. for $C_{18}H_{36}O_2N_2$: C, 69·2; H, 11·6; N, 9·0%).

Ethyl N-(6-*Hexanamidohexyl*)*adipamate* {VII; R = $EtO_2C{\cdot}[CH_2]_4{\cdot}CO$}.—Prepared by the dropwise addition of the acid chloride of ethyl hydrogen adipate (87 g.) in benzene (150 ml.) to an agitated ice-cold mixture of *N*-(6-aminohexyl)hexanamide (87 g.), pyridine (45 ml.), and benzene (250 ml.), this *diamide* (136 g.), isolated in the usual way with chloroform, was purified from ethanol and then ethyl acetate, and obtained as needles, m. p. 115—116° (Found: C, 64·4; H, 10·3; N, 7·8. $C_{20}H_{38}O_4N_4$ requires C, 64·8; H, 10·3; N, 7·6%).

N-(6-*Hexanamidohexyl*)-2-*oxo*cyclo*pentanecarboxyamide* (VI).—A stirred mixture of the preceding ester-amide (75 g.), absolute ethanol (1 ml.), benzene (400 ml.), and sodium (6·8 g.) was boiled under reflux for 16 hours. The *product*, isolated in the normal manner, crystallised from ethyl acetate in needles (42—47 g., 64—71·5%), m. p. 108—109°, which had an intense royal-blue ferric reaction in ethanol (Found: C, 66·3; H, 10·0; N, 9·1. $C_{18}H_{32}O_3N_2$ requires C, 66·6; H, 9·9; N, 8·6%). The infrared spectrum (Nujol mull) had principal absorption bands at 3378ms, 3115m, 2941—2890s, 1742ms, 1639s, 1541s, 1468s, 1385ms, 1261m, 1236m, 1217m, 1143m, 1064w, 980w, 952w, 838w, and 728m $cm.^{-1}$.

The amide (VI) formed a 2 : 4-*dinitrophenylhydrazone* as orange needles (from aqueous methanol), m. p. 170—171° (Found: C, 57·0; H, 7·3; N, 16·7. $C_{24}H_{36}O_6N_4$ requires C, 57·1; H, 7·2; N, 16·7%), and a *semicarbazone*, colourless needles (from aqueous methanol), m. p. 205—206° (Found: C, 60·0; H, 9·3; N, 18·8. $C_{19}H_{35}O_3N_5$ requires C, 59·8; H, 9·2; N, 18·4%).

Acid hydrolysis, essentially as above, gave carbon dioxide (0·89 mol.), volatile acid (0·934 equiv.), and *cyclo*pentanone 2 : 4-dinitrophenylhydrazone (1·06 mol.), m. p. and mixed m. p. 144°. A volatile acid was isolated and shown by paper chromatography to be hexanoic acid containing a trace of acetic acid. 1 : 6-Diaminohexane was also isolated and was shaken for 24 hr. with ethanol (30 ml.), anhydrous potassium carbonate (7·5 g.), and 1-fluoro-2 : 4-dinitrobenzene (2·3 g.); the mixture was then filtered, washed with water, and dried. The yellow solid (3·026 g., 1·09 mol.) separated from nitrobenzene in yellow needles, m. p. 205° alone or admixed with the authentic bis-2 : 4-dinitrophenyl derivative (lit.,[12] m. p. 205°).

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