VIII.—*On some Derivatives of Phenylacetic Acid.*

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FOR our knowledge of the derivatives of phenylacetic acid we are
chiefly indebted to Radziszewski (*Ber.*, **2**, 207), who first investigated
the conditions under which substitution may take place either in the
aromatic nucleus or in the side-chain.   My investigation of some of
the derivatives of this acid has led to results differing somewhat from
those of the above-mentioned chemist.   I here propose to give an
account of these results, together with some further particulars rela-
tive to the bromonitrophenylacetic acids, &c., the study of which I
undertook, whilst in Bonn, at the proposal of Professor Kekulé, and

burg's prism, but Ladenburg throws doubts on Graebe's proof of the symmetri-
cal character of naphthalene.

short notices of which have already appeared in another Journal (*Ber*, **10**, 530 and 1657).

Nitro-derivatives.—Phenylacetic acid when nitrated yields two isomeric nitro-derivatives, viz., the para- and the ortho-, the former constituting the chief portion of the yield. The method of separating these two acids by the different solubilities of their barium salts, does not give satisfactory results. If, however, the acid prepared from the less soluble salt be crystallised repeatedly from a hot mixture of alcohol and water, an acid is obtained, crystallising in long needles, and melting at 150—151°, which proves to be paranitrophenylacetic acid.

(1.) *Paranitrophenylacetic acid* is soluble in hot water, from which it crystallises in long, yellowish-white, brittle needles; it is sparingly soluble in cold water, and easily in alcohol and ether. Its analyses gave the following results :—

I. 0·3066 gram yielded 0·5978 gram CO_2, and 0·1109 gram H_2O.

II. The "NO_2" was determined by Limpricht's method (*Ber.*, **11**, 35), 10 c.c. of stannous chloride solution diluted to 250 c.c. gave a solution, 10 c.c. of which required 10·83 c.c. of iodine solution. 1 c.c. of iodine solution contained 0·01108 gram $I = 0·000667$ gram NO_2. 0·2132 gram of acid heated with 10 c.c. $SnCl_2$ solution, diluted to 250 c.c., gave a solution 10 c.c. of which required 7·64 c.c. of iodine.

$$25 \times (10·83 - 7·63) \times 0·000667 = 0·005336 \text{ gram } NO_2.$$

				Found.	
				I.	II.
C_8	=	96	53·03	53·16	—
H_7	=	7	3·86	4·01	—
NO_2	=	46	25·41	—	25·02
O_2	=	32	17·67	—	—
		181	100·00		

This acid, when oxidised by a mixture of potassium bichromate and sulphuric acid, yielded paranitrobenzoic acid, m. p. 230°; the combustion of which gave the following numbers :—

0·3064 gram yielded 0·5682 gram CO_2 and 0·088 gram H_2O.

	Calculated for $C_6H_4NO_2CO_2H$.	Found.
C.	50·29	50·52
H	2·99	3·16

Methyl paranitrophenylacetate, $C_6H_4.NO_2.CH_2CO_2CH_3$, crystallises from alcohol in yellowish-white plates, by slow evaporation of the alcoholic solution it is obtained in thick striated plates; it melts at 54—55°.

Ethyl paranitrophenylacetate crystallises from dilute alcohol in thin, shining leaflets, melting at 62—64°.

The *barium salt* is obtained in beautiful light yellow transparent crystals by allowing the aqueous solution to evaporate slowly. These crystals appear to be six-sided plates; as on exposure to air they effloresce and become opaque, their exact crystalline form could not be determined. The crystals quickly dried between filter-paper, gave results (I) corresponding to the formula $Ba(C_8H_6NO_4)_2 + 7H_2O$; whilst the air-dried opaque crystals gave results (II) for the formula $Ba(C_8H_6NO_4)_2 + 2H_2O$.

I. 0·5358 gram heated at 110—115° lost 0·113 gram, and gave 0·1959 gram $BaSO_4$.

	Calculated for $Ba(C_8H_6NO_4)_2 + 7H_2O$.	Found.
Ba	21·99	21·48
H_2O	20·22	21·08

II. 0·2912 gram heated at 100—110° lost 0·0214 gram, and gave 0·1285 gram $BaSO_4$.

	Calculated for $Ba(C_8H_6NO_4)_2 + 2H_2O$.	Found.
Ba	25·70	25 92
H_2O	6·75	7·34

The aqueous solution of the barium salt gives with lead acetate solution a white precipitate, which partially dissolves when boiled and recrystallises on cooling; with silver nitrate solution a white curdy precipitate soluble in ammonia and nitric acid, and finally with ferric chloride a brownish precipitate.

Paramidophenylacetic acid, $C_6H_4 \begin{cases} NH_2 \\ CH_2CO_2H \end{cases}$, is prepared by the reduction of the paranitro acid by tin and hydrochloric acid; it crystallises from hot aqueous solutions in white lustrous leaflets, which become brown on exposure to air, and melt at 199—200° with decomposition. The analysis of this acid gave the following numbers:—

0·2357 gram gave 0·5452 gram CO_2 and 0·129 gram water.

			Found.
C_8.	96	63·57	63·08
H_9	9	5·96	6·06
N	14	9·27	—
O_2.	32	21·20	—
	151	100·00	

(2.) *Orthonitrophenylacetic acid.* — The alcoholic mother-liquors from the crystallisation of the paranitro-derivative, when concentrated

yield a body crystallising in white opaque tufts of needles, melting at $112-117°$. A similar product is obtained by decomposing the more soluble barium salt with hydrochloric acid. By allowing the solution of this body in methyl or ethyl alcohol to evaporate slowly, large well-formed crystals separate out, which were collected and recrystallised from alcohol. By this means a few grams of these crystals were obtained, melting at $137-138°$. When crystallised from hot water, in which they are sparingly soluble, white shining needles separate out at first, and on standing minute crystals appear, having a form similar to the larger ones. Both the needles and the minute crystals melt at $137-138°$.

The combustion of the acid crystallised from water gave the following results :—

0·302 gram yielded 0·585 gram CO_2 and 0·1133 gram H_2O.

	Calculated for $C_6H_4{}^{NO_2}_{CH_2}CO_2H.$	Found.
C.........	53·03	52·64
H	3·86	4·13

This acid when oxidised by boiling with a solution of potassium permanganate, yields orthonitrobenzoic acid subliming in white needles, and melting at $141-143°$. Moreover, upon reduction it is converted into oxindol, which was recognised by its melting point, $120°$, and its reaction with nitrous acid. From these facts I conclude that the second nitro- acid, m. p. $137-138°$, is *orthonitrophenylacetic acid*.

For the following description and measurements of the crystals of orthonitrophenylacetic acid, I am indebted to Mr. Baker.
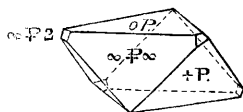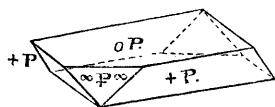
Orthonitrophenylacetic Acid.

Monoclinic. Fig. 1. $0P + P \infty P\infty$; tabular through $0P$.
    ,, 2. $0P + P \infty P\infty$ in equilibrium, together with $\infty P2$.
    $\breve{a} : \bar{b} : c = 1·0 : 0·594; \ \mathsf{L} = 97° 17'$.

|	Found.		Calculated.	
$0P : +P$.....................	=	$51° 45'$		$51° 45'$
$\infty P\infty : +P$.....................	=	108 9		108 15
$0P : \infty P\infty$	=	97 17		97 17
$+P : +P$ (in clino-diagonal)	=	94 28		94 28
$\infty P2 : \infty P2$ (in ortho-diagonal)....	=	81 16		81 0

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Barium orthonitrophenylacetate crystallises from its aqueous solutions in white lustrous scales, which are more easily soluble in water than the crystals of the corresponding salt of the para-acid. The analysis of this salt shows its formula to be $Ba(C_8H_6NO_4)_2 + 2H_2O$; the water cannot be directly determined as the salt is decomposed at $100—110°$.

0·495 gram gave 0·2121 gram $BaSO_4$, *i.e.*, 25·17 per cent. Ba, whilst $Ba(C_8H_6NO_4)_2 + 2H_2O$ requires 25·70 per cent. Ba.

Aqueous solutions of this barium salt give no immediate precipitate with solution of lead acetate; on standing, however, the lead salt separates out in tufts of fine needles. Silver nitrate and ferric chloride give reactions similar to those with the solutions of the salt of the para-acid.

(3.) *Bromo-derivatives.*—Bromophenylacetic acid was prepared by an application of Barth and Weselky's method for such cases, viz., by treating phenylacetic acid and mercuric oxide suspended in water, with the calculated quantity of bromine in small quantities, at the same time cooling by immersion in water. The reaction which takes place is expressed by the following equation:—

$$2C_7H_7CO_2H + HgO + 2Br_2 = 2C_7H_6BrCO_2H + HgBr_2 + 2H_2O.$$
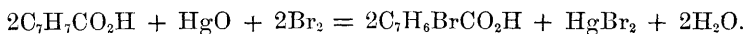
After the completion of the reaction an excess of caustic soda is added, and the filtrate from the mercuric oxide acidified with hydrochloric acid; the bromo-acid separates out as a white crystalline precipitate, and after washing with cold water is crystallised from hot dilute alcohol. The chief portion separates out from this solution as a slightly coloured oil, which on standing solidifies to a crystalline mass; some also crystallises out in tufts of white needles, both portions melting at $76°$. The alcoholic mother-liquors on concentration yield an acid having a somewhat higher melting point. This latter portion is converted into the barium salt either by boiling its aqueous solution with barium carbonate or with baryta-water, and in which case the excess of barium is removed by precipitation with carbonic acid. The concentrated aqueous solutions of this salt yield a white crystalline deposit formed of opaque nodular masses; these deposits were crystallised from hot water, and finally decomposed by hydrochloric acid.

Parabromophenylacetic Acid.—The acid thus obtained is dissolved in hot water, from which solution it separates on cooling in long, flat lustrous needles, resembling the crystals of benzoic acid, and melting at $114—115°$. Its analysis shows it to be a monobromophenylacetic acid, and its melting point is the same as the parabromophenylacetic acid prepared by Loring Jackson, and W. Lowery (*Ber.*, **10**, 1210) from parabrombenzyl bromide.

The analysis gave the following results :—

I. 0·256 gram gave 0·4188 gram CO_2 and 0·0786 gram H_2O.

II. 0·2396 gram gave 0·198 gram AgBr and 0·0065 gram Ag.

				Found.	
				I.	II.
C_8	=	96	44·65	44·60	—
H_7	=	7	3·25	3·39	—
Br	=	80	37·20	—	37·14
O_4	=	64	17·90	—	—
		247	100·00		

Orthobromophenylacetic Acid.—The mother-liquors, containing the more soluble barium salt, which on further evaporation yield no crystalline deposit, were precipitated with a solution of lead acetate, and the white precipitate so formed after washing with cold water was boiled with dilute sulphuric acid. The filtrate from the lead sulphate on cooling yielded crystals of an acid, which, after several crystallisations from hot water, is obtained in the form of long lustrous flat needles, melting at 103—104°, the analysis of which gave the following results :—

I. 0·2104 gram gave 0·3436 gram CO_2 and 0·0584 gram H_2O.

II. 0·257 gram gave 0·2148 gram AgBr and 0·0066 gram Ag.

|	Calculated.	Found.	
		I.	II.
C_8	44·65	44·53	—
H_7	3·25	3·04	—
Br	37·20	—	37·43
O_2	17·90	—	—
	100·00		

These results, together with the fact that when this acid is oxidised by boiling with a solution of potassium permanganate, orthobromobenzoic acid is formed, melting at 143—144°, prove this acid to be orthobromophenylacetic acid. The orthobromobenzoic acid gave the following results for the bromine determination; 0·1952 gram gave 0·1754 gram AgBr, and 0·0027 gram Ag, giving 39·24 per cent. Br, whereas the formula $C_6H_4<^{Br}_{CO_2H}$ requires 39·83 per cent. Br.

Orthobromphenylacetic acid may be obtained in well-formed crystals by the slow evaporation of its solution in glacial acetic acid, for the following description of which I am indebted to Mr. Baker :—

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Orthobromphenylacetic Acid.

System monoclinic $\breve{a} : \bar{b} : \bar{c} = 1\cdot000 : 0\cdot657 : 1\cdot767$; $\text{L} = 99^\circ\ 44$.
Forms observed, $\infty\text{P} \cdot 0\text{P} \cdot \infty\text{P}\infty \cdot \text{R}\infty \cdot -\frac{1}{2}\text{P}\infty \cdot \cdot -\text{P}$.

	Calculated.	Found.
$\infty\text{P} : \infty\text{P}$ (in ortho-diagonal)	$112^\circ\ 37'$	$112^\circ\ 37'$
$0\text{P} : \infty\text{P}\infty$ (L)	99 44	99 44
$0\text{P} : = \frac{1}{2}\text{P}\ \infty$	142 51	142 48
$\infty\text{P} : -\text{P}$	163 6	163 15
$0\text{P} : \text{R}\infty$	110 40	110 54
$0\text{P} : \infty\text{P}$	95 23	95 24
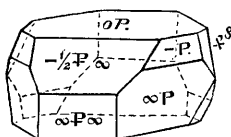
From the above experiments bromine in the cold acts on phenyl-acetic acid in a similar manner to nitric acid, producing para- and ortho-derivatives, in each case the melting point of the "para" is about 12° higher than that of the "ortho." Further, these results show that the parabromphenylacetic acid described by Radziszewski (*loc. cit.*), melting at 76°, is a mixture of the two isomerides. A similar remark may be made with regard to the para- and ortho-nitro-phenylacetic acids described by the same chemist, the former melting at 114° and the latter at 98°.

In one of the recent numbers of the *Berliner Berichte* (**12**, 1764), Maxwell publishes a description of paranitrophenylacetic acid, to which he attributes the melting point 151·5—152°, which agrees tolerably well with my determination, as does the description of the methyl- and ethyl-ethers of this acid. The barium salt is described as anhydrous and crystallising in needles, a difference probably due to the method of preparation.

(4.) *Bibromo-derivatives.*—To prepare a bibromo-acid crude bromo-phenylacetic acid (m. p. 76°) was sealed up in a strong glass vessel with the calculated quantity of bromine, and exposed for several months to the action of sunlight. Thus a brownish liquid was pro-duced, and on opening the vessel quantities of hydrobromic acid were given off. The product after washing several times with water, was converted into the methyl-ether, as all attempts to obtain a crys-talline product by crystallisation from alcohol had proved futile, yielding a yellowish viscous mass only. The methyl-ether when dis-tilled under reduced pressure chiefly came over at 220—230°; this

portion was saponified by boiling with caustic potash. The potassium salt decomposed with hydrochloric acid, and an acid obtained which crystallises from water in white shining needles, melting at 114—115°. But a small quantity of this acid was obtained; the bromine determination shows it to be a bibromophenylacetic acid. 0·276 gram gave 0·3521 gram AgBr and 0·0022 gram Ag, which corresponds to 54·78 per cent. Br, whilst $C_6H_4Br_2CH_2CO_2H$ requires 54·42 per cent. Br. Together with this acid other acids are formed, but in quantities nsufficient for analysis.

(5.) *Bromonitro-derivatives.*—Short notices of the preparation and separation of two bromonitrophenylacetic acids and the corresponding bromamido-derivatives have already appeared (*Ber.*, **10**, 530 and 1637). The following is a more detailed account of these acids, and also of a third isomeride. As in the preparation of these acids I used a bromophenylacetic-acid, melting at 76°, the formation of three bromonitro-acids is not astonishing, as the bromo-acid contained both the para- and ortho-derivatives; it renders doubtful, however, the constitution attributed to the bromonitrophenylacetic acid melting at 167—169°.

Parabromometanitrophenylacetic acid, $C_6H_3\Big\langle\begin{array}{l}NO_2\\Br\\CH_2CO_2H\end{array}$, crystallises

from its hot aqueous solutions in small flat greenish-yellow needles, melting at 113—114°. It dissolves in hot water, but is almost insoluble in cold water, and is easily soluble in alcohol and ether.

The analysis of this acid gave the following results:—

I. 0·3005 gram gave 0·4076 gram CO_2 and 0·0735 gram H_2O.

II. 0·252 gram gave 0·1736 gram AgBr and 0·0055 gram Ag.

III. The "NO_2" determination was made by Limpricht's method, 0·2123 gram gave 0·036768 gram NO_2.

			Found.		
			I.	II.	III.
C_8	96	36·92	36·97	—	—
H_6	6	2·30	2·66	—	—
NO_2......	46	17·68	—	—	17·31
Br	80	30·76	—	30·90	—
O_2	32	13·34	—	—	—
	260	100·00			

This acid when oxidised by potassium bichromate and sulphuric acid yields parabromometanitrobenzoic acid, melting at 197—199°, described by Hübner, Ohly, and Philipp (*Liebig's Annalen*, **143**, 248). Its analysis gave the following results:—

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I. 0.2675 gram gave 0.3391 gram CO_2 and 0.05485 gram H_2O.
II. 0.289 gram gave 0.3635 gram CO_2 and 0.0577 gram H_2O.
III. 0.3 gram gave 0.221 gram AgBr and 0.0057 gram Ag.

				Found.		
				I.	II.	III.
C_7	$=$	84	34.14	34.54	34.29	—
H_4	$=$	4	1.62	2.23	2.21	—
N	$=$	14	5.69	—	—	—
Br	$=$	80	32.52	—	—	32.73
O_4	$=$	64	26.03	—	—	—
		246	100.00			

Barium parabromometanitrophenylacetate, $Ba(C_8H_5BrNO_4)_2 + H_2O$, crystallises in yellowish plates or needles united in concentric groups, is soluble in water, but less soluble than the salt of the α-bromitro-acid.   The analysis of this salt gave the following results : 0.569 gram heated at 110—$120°$ lost 0.154 gram, and gave 0.1969 gram $BaSO_4$, corresponding to 2.70 per cent. H_2O and 20.33 per cent. Ba, whilst the formula requires 2.79 per cent. H_2O and 20.35 per cent. Ba.

A dilute solution of the barium salt gives with silver nitrate a white curdy precipitate soluble in nitric acid, with copper acetate a bright blue precipitate, and with lead acetate a white precipitate, which dissolves on boiling.

The methyl ether forms yellowish-white needles usually grouped together, melting at 40—$41°$.   Its ethyl ether has only been obtained as an oily liquid.

$Parabromometamidophenylacetic$ $acid$, $C_6H_3 {\Big<} {{NH_2} \atop {Br} \atop {CH_2CO_2H}}$, obtained by

the reduction of the nitro-acid with tin and hydrochloric acid, crystallises from water in white silky needles, which colour slowly on exposure to the air.   It is less soluble in water than the α-bromamido-acid, but is easily soluble in alcohol and chloroform, and sparingly in ether.   It melts at 133—$134°$.

Its analysis gave the following result :—0.2156 gram gave 0.3315 gram CO_2 and 0.0714 gram H_2O.

	Calculated for $C_6H_3NH_2.Br.CH_2CO_2H.$	Found.
C	41.73	41.92
H	3.47	3.66

Its hydrochlorate, $C_7H_7BrNCO_2H.HCl + H_2O$, crystallises in white

needles, united in concentric groups, becoming red on exposure to the air. It is soluble in cold water. The analysis of this salt gave the following results* :—

I. 0·2636 gram gave 0·1282 gram AgCl and 0·0041 gram Ag.

II. 0·2208 gram gave 0·275 gram CO_2 and 0·084 gram H_2O.

III. 0·333 gram gave 0·2168 gram of AgBr.

	Calculated.	Found.		
		I.	II.	III.
C...............	33·72	—	33·96	—
H	3·86	—	4·21	—
Br	28·12	—	—	27·68
Cl	12·47	12·51	—	—

α-*Bromonitrophenylacetic acid* crystallises from water in yellowish-white, branching needles, melting at 167—169°. It is easily soluble in alcohol and ether, is insoluble in cold water, but soluble in hot water, and less soluble in a mixture of alcohol and water than the isomeric acid already described. The following are the results of its analysis :—

I. 0·303 gram yielded 0·4106 gram CO_2 and 0·0776 gram H_2O.

II. 0·5884 gram yielded 0·7955 gram CO_2 and 0·1354 gram H_2O.

III. 0·5335 gram yielded 0·3861 gram AgBr and 0·004 gram Ag.

IV. 0·2172 gram by Limpricht's method gives 0·037852 gram " NO_2."

	Calculated.	Found.			
		I.	II.	III.	IV.
C	36·92	36·93	36·86	—	—
H.............	2·30	2·83	2·54	—	—
Br	30·76	—	—	31·32	—
NO_2..........	17·68	—	—	—	17·42
O.............	13·34	—	—	—	—
	100·00				

The barium salt, $Ba(C_8H_5BrNO_4)_2 + 4H_2O$, forms yellow transparent needles, united to form concentric groups, is soluble in hot water, less soluble in cold, and more soluble than the salt of the isomeride already described. The following results confirm the above formula : —0·4718 gram heated at 110—115° lost 0·046 gram, and gave

* In I, the chlorine was determined by adding silver nitrate to the aqueous solution of the hydrochlorate ; in III, the substance heated with lime, &c., gave a mixture of silver chloride and bromide, and the amount of the latter determined by subtracting the silver chloride given by the chlorine determination.

0·1501 gram $BaSO_4$; or 9·74 per cent. H_2O, and 18·69 per cent. Ba, the formula requiring 9·99 per cent. H_2O and 18·84 per cent. Ba.

The aqueous solutions of the barium give with silver nitrate and lead acetate solution similar reactions to those of the isomeric acid ; copper acetate, however, gives a green precipitate.

The methyl-ether forms transparent, slightly yellow, flat, shining needles; it melts at 66—68°. The ethyl-ether forms yellowish needles.

α-Bromamidophenylacetic acid is obtained by reducing the corresponding α-nitro-acid with tin and hydrochloric acid ; it crystallises from water in white, brittle needles, which redden on exposure to the air, and melt at 167°, a decomposition taking place at the same time. It is soluble in alcohol and chloroform, but sparingly soluble in ether. Its analysis gave the following results :—

I. 0·2006 gram gave 0·3055 gram CO_2 and 0·0684 gram H_2O.

II. 0·2276 gram gave 0·1692 gram AgBr and 0·0099 gram Ag.

	Calculated.	I.	II.
C	41·73	41·52	—
H	3·47	3·78	—
Br	34·78	—	34·88

with "Found." spanning columns I. and II.

The hydrochlorate of α-bromamidophenylacetic acid,

$$C_8H_8BrNO_2HCl + H_2O,$$
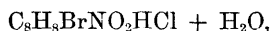
crystallises from water in long white needles, which become red on exposure to the air ; it is more soluble in water than the hydrochlorate of the isomeric acid already described. Its analysis gave the following results :—

I. 0·2404 gram gave 0·302 gram CO_2 and 0·0892 gram H_2O.

II. 0·220 gram gave 0·1065 gram AgCl and 0·0034 gram Ag.

III. 0·2029 gram gave 0·1319 gram AgBr.

IV. 0·9059 gram heated in a current of dry air at 90—100°, lost 0·0574 gram.

Calculated for $C_6H_3Br.NH_2CH_2CO_2H.HCl + H_2O.$	I.	II.	III.	IV.
C	33·74	34·23	—	—	—
H	3·86	4·11	—	—	—
Br	28·12	—	—	27·64	—
Cl	12·47	—	12·45	—	—
H_2O	6·32	—	—	—	6·33

with "Found." spanning columns I., II., III., IV.

The salts of this acid with the alkalis are easily soluble in water ;

their solutions give a dark green precipitate with copper acetate of the copper salt.

β-Bromonitrophenylacetic Acid.—Together with the above two bromo-nitrophenylacetic acids, a third is formed ; it was separated from the yellow crystalline mass obtained by extracting with chloroform the mother-liquors from the nitration of the bromophenylacetic acid. This extract was dissolved in glacial acetic acid, and the solution allowed to evaporate slowly, thus an acid was obtained crystallising in small transparent yellow prisms.   It melts at 162°, and when crystallised from hot water it forms long colourless needles, having the same melting point.   By reduction with tin and hydrochloric acid, it is converted into a bromamidophenylacetic acid, which crystallises from water in white leaflets, becoming brown on exposure to the air ; it melts at 186°.   Its hydrochlorate is less soluble in water than that of either of the above isomerides.   But a small quantity of these two acids has been obtained ; the analysis of the bromamido-acid gave the following results :—

I. 0·213 gram yielded 0·324 gram CO_2 and 0·072 gram H_2O.

II. 0·2385 gram gave 0·1821 gram AgBr and 0·0061 gram Ag.

	Calculated for $C_6H_3.Br.NH_2.CH_2CO_2H$	Found.	
		I.	II.
C	41·73	41·45	—
H	3·47	3·75	—
Br	34·78	—	34·29

From the parabromometamidophenylacetic acid, and the α-brom-amido-acid, I have attempted to obtain the amidophenylacetic acids by means of the action of sodium amalgam on the aqueous solutions of these acids ; the results of this action are, however, not very satisfactory.   Some further clue to the constitution of the above bromonitro-phenyl acetic acids I hope to obtain by the preparation of the nitro-paramidophenylacetic acids, and from which by means of the diazo reaction to prepare the corresponding bromonitro-derivatives.