obtained 5 g. (85%) of pure product, pale yellow crystals from ethanol, m. p. 154–155°.

Anal. Calcd. for $C_{19}H_{17}NO_6$: C, 64.2; H, 4.8. Found: C, 64.5; H, 4.7.

Ethyl 2-Hydroxy-6-methoxy[1]benzopyrano[4,2-de]-quinoline-1-carboxylate, XV.—From 4 g. of XIV, with 450 ml. of 1% alcoholic sodium ethoxide, there was obtained 3.4 g. (89%) of product, yellow needles from alcohol, m. p. 272–274°.

Anal. Calcd. for $C_{19}H_{15}NO_5$: C, 67.6; H, 4.5. Found: C, 67.5; H, 4.4.

Ethyl 2-Chloro-6-methoxy[1]benzopyrano[4,2-de]-quinoline-1-carboxylate, XVI.—From 2 g. of XIV, treated with 10 ml. of phosphorus oxychloride, there was obtained 1.7 g. of product, yellow crystals from benzene–ligroin, m. p. 161–162°.

Anal. Calcd. for $C_{19}H_{14}ClNO_4$: C, 64.1; H, 4.0. Found: C, 63.8; H, 4.0.

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Chemical Degradation of Isotopic Succinic Acid[1]

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In connection with biochemical studies it was necessary to determine the distribution of isotopic carbon in samples of succinic acid isolated from biological sources. It has been found that pyrolysis of the barium salt in a high vacuum at 500° results in a satisfactory conversion of the carboxyl carbon of succinic acid to barium carbonate. The course of this reaction is uncertain. In addition to barium carbonate and some carbon, products such as carbon dioxide, carbon monoxide, methane, ethane, ethylene and hydrogen were identified by mass-spectrometric analysis.

The accompanying table, giving the results of the pyrolysis of carboxyl- and methylene-labeled succinates, shows that the barium carbonate satisfactorily represents the carboxyl carbon. Evidently there is some contamination of the carboxyl carbon by methylene carbon, but this is so small as to introduce only a negligible error. This slight enrichment of C^{13} in the non-labeled carboxyl position was not an artifact, since non-isotopic barium succinate invariably yielded barium carbonate with the normal C^{13} abundance. This enrichment may be due to oxidation of the methylene carbon by traces of oxygen; or possibly, to transfer of oxygen between barium carbonate and the accompanying residual carbon.

TABLE I

C^{13} DISTRIBUTION IN SYNTHETIC LABELED SUCCINIC ACIDS

Atom % C^{13} excess

	Over-all	Carboxyl carbon Calcd.	Carboxyl carbon Found
Carboxyl-labeled	2.41	4.82	4.80
Methylene-labeled	3.10	0.00	0.04 ± 0.02
Unlabeled	0.00	.00	.00 ± .01

Experimental

Preparation of Isotopic Succinic Acids.—Carboxyl-labeled succinic acid was prepared by refluxing ethylene dibromide with isotopic potassium cyanide, according to the procedure of Vanino.[2] The dinitrile was saponified with alkali without isolation, and after removal of neutral substances by extraction with ether, the succinic acid was isolated by acidification and continuous ether extraction. Yields ranged between 85 and 95%.

The methylene-labeled acid was prepared by a 4-step process giving an over-all yield of about 40%. Barium carbonate was reduced to the carbide according to the procedure of Cramer and Kistiakowsky[3] and the acetylene obtained therefrom reduced to ethylene by a modification of the method of Patterson and du Vigneaud.[4] This was converted to ethylene dibromide by addition of bromine and the former converted to succinic acid by the same procedure used for the carboxyl-labeled acid.

Preparation and Pyrolysis of Barium Salts.—About 20 mg. of the acid is dissolved in 1 ml. of water, 1 ml. of 20% barium chloride is added, and the solution brought to neutrality with dilute ammonia. Two volumes of 95% ethanol are added and the precipitated barium salt centrifuged, washed successively with alcohol and ether, and dried thoroughly in a vacuum.

The barium salt is transferred to a small glass tube, which is then sealed to the vacuum line or attached by means of a standard taper joint. After evacuation to a low pressure the salt is heated to 500° in an electric furnace. After about an hour the tube is cooled, the dark-colored residue is treated with dilute sulfuric acid and the evolved carbon dioxide collected for mass-spectrographic analysis.[5] It is important to avoid even traces of oxygen in this degradation since in its presence some of the methylene carbon will be oxidized to carbon dioxide and contaminate the carboxyl carbon.

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(3) Cramer and Kistiakowsky, *J. Biol. Chem.*, **137**, 549 (1941).

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Methoxyacetone

BY RAYMOND P. MARIELLA AND JOHN L. LEECH

In continuing our investigations of unsymmetrical ketones, it became necessary to prepare a large quantity of methoxyacetone.

It was found that the wet oxidation of the inexpensive and easily-available 1-methoxy-2-propanol (Dowanol 33B) with chromic acid at room temperature goes conveniently in one step to give methoxyacetone. The present method is adapted from that of Petrov[1] and gives methoxyacetone in much shorter time than other published methods[2–5] although in a somewhat lower yield.

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