By employing this preliminary concentration 100 lb. of zinc oxide could be put through the distillation process in four or five runs using a 12-liter Pyrex flask, whereas before this method was used it required 25 to 30 runs; also, the quantity of material from that process which had to be worked over for gallium was correspondingly decreased.

CONTRIBUTION FROM THE
CHEMICAL LABORATORY OF THE
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RECEIVED JANUARY 9, 1929
PUBLISHED MAY 6, 1929

Determination of Ethylene by Absorption in a Solution of Silver Nitrate.—During the course of a study of the catalytic hydrogenation of acetylene[1] the author observed that a solution of silver nitrate is capable of absorbing ethylene. A preliminary test at that time tended to indicate that the reaction might be used for the quantitative determination of the latter gas.

A search of the literature revealed that reactions between ethylene and silver salts have been previously observed by Ross and Trumbull,[2] Gluud and Schneider[3] and Lommel and Engelhardt.[4] The feasibility of using a solution of a silver salt alone as an absorbing medium in the determination of ethylene has apparently never been investigated.

Reactions between ethylene and salts of a few other metals have also been observed. In this connection, attention may be directed to the suggestion[5] for the use of a solution of mercuric nitrate over mercury as a quantitative absorbing agent for this gas.

The apparatus used during a brief investigation of the usefulness of a solution of silver nitrate as an absorbent in the determination of ethylene has been described previously by the author.[6] In its use the absorbing solution is passed slowly over and around the gaseous sample and is removed continuously at the bottom. Such an apparatus is particularly advantageous in cases where, as appeared to be probable in this case, there is a tendency toward an equilibrium condition involving incomplete absorption.

The table below records the results of a comparison of the absorption of ethylene from a mixture consisting essentially of nitrogen and ethylene by solutions of silver nitrate of various concentrations. The volumes of

[1] Morris and Reyerson, *J. Phys. Chem.*, **31**, 1332 (1927).

[2] Ross and Trumbull, THIS JOURNAL, **41**, 1180 (1919).

[3] Gluud and Schneider, *Ber.*, **57B**, 254 (1924).

[4] Lommel and Engelhardt, *ibid.*, **57B**, 848 (1924).

[5] See Treadwell-Hall, "Quantitative Analysis," 7th ed., John Wiley and Sons, Inc., New York, p. 698.

[6] Morris, THIS JOURNAL, **49** 979 (1927).

solutions and the time requirements shown, while not necessarily representing the exact minima in every case, do give indication of what may be expected in a satisfactory operation carried out as rapidly as a careful observation of the progress of the absorption appears to justify.

TABLE I

THE ABSORPTION OF ETHYLENE BY SOLUTIONS OF SILVER NITRATE

Concn. of soln., %	40	20	10	5
Vol. of soln. used, cc.	12	28	40	80
Time for absorption, sec.	55	75	180	240
Red. in vol. of 50 cc. sample, cc.	34.8	34.9	34.9	34.7

The absorption by the more concentrated solutions was more rapid. The use of concentrated solutions is also recommended as a consequence of the fact that the completion of absorption can be more readily judged when using them rather than the slower absorbing dilute solutions.

In an additional experiment a comparison was made between a solution of silver nitrate and other reagents commonly used in the quantitative absorption of ethylene. The value for the ethylene content of a mixture of this gas with nitrogen as indicated by absorption with fuming sulfuric acid agreed closely with that obtained on the same mixture when a 20% solution of silver nitrate was used. The fact that absorption by bromine gave a slightly different value was attributed to a change in the composition of the gaseous mixture during the time interval between tests.

There are certain advantages in the use of silver nitrate for this purpose. It is interesting to note, for instance, that, in accordance with the observation of Lommel and Engelhardt,[4] the ethylene may subsequently be removed from the solution by evacuation. Recovery of the absorbing solution is thus possible. Evaporation can readily be applied to restore it to the concentration desired for subsequent determinations. Whether heat alone would free the solution from ethylene as well as concentrate it remains to be investigated. A solution of silver nitrate has, moreover, a decided advantage over the commonly used absorbents, bromine and strong sulfuric acid, in that the chemical and physical properties of these latter make them anything but pleasant reagents to handle.

There are several points in connection with the reaction between ethylene and silver nitrate which seem to be worthy of further study. Of primary interest is the question of the composition and properties of the reaction product. The possibility of increasing the effectiveness of absorption by adding acids such as sulfuric or phosphoric to the solution of silver nitrate also merits investigation.[4] As a final consideration attention may be directed to the fact that acetylene may be determined by titration of the nitric acid released when this gas reacts with a solution of silver nitrate. The possibility of determining both acetylene and ethylene in a mixture

of the two by the use of a single absorbing agent, a solution of silver nitrate, is at once apparent.

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RECEIVED JANUARY 12, 1929
PUBLISHED MAY 6, 1929

[CONTRIBUTION FROM THE RESEARCH LABORATORIES, CHEMICAL DEPARTMENT OF THE CITY OF CHICAGO MUNICIPAL TUBERCULOSIS SANITARIUM]

MENTHOL STUDIES. II. MENTHYL ESTERS OF THE 2-NITRO-4-CARBOXYPHENYLARSONIC AND PHENYLARSENIOUS ACIDS

BY K. LUCILLE McCLUSKEY

RECEIVED OCTOBER 3, 1928 PUBLISHED MAY 6, 1929

The first report[1] on the synthesis of aromatic terpene compounds to be used in chemotherapeutic studies in experimental tuberculosis included menthyl esters of the nitro- and aminocinnamic acids. Due to the great insolubility in water caused by the menthol, the menthyl esters of the aminocinnamic acids could not be made water-soluble through their hydrochlorides. Since a water-soluble product is desirable, the introduction of arsenic into the aromatic terpene group was thought to be desirable for two reasons; first, to increase the solubility in water and second, to confer probably greater therapeutic properties.

A few organic arsenicals have been used in experimental studies in tuberculosis therapy, and arsenic in the form of arsenic acid was tried early in the treatment of tuberculosis. Sodium cacodylate, atoxyl, arsacetin and neosalvarsan have no tuberculocidal properties, according to Arkin and Corper,[2] but earlier investigators (Buchner, Weismayr, Pokhorow)[3] comment favorably upon the worth of arsenic in the treatment of tuberculosis because of its favorable influence on metabolism, although they conclude that it is not a true chemotherapeutic agent with a specific action on human tubercle bacilli.

The new menthol arsenicals described here were made with the hope that they might possess some favorable pharmacological action.

Experimental

2-Nitro-4-methylphenylarsonic Acid.—This compound was first prepared by Jacobs, Heidelberger and Rolf[4] using a different technique in the arsonation process from that described here. 3-Nitro-4-toluidine (110 g.) is stirred by means of a mechanical stirrer in 20% hydrochloric acid (500 cc.) until the hydrochloride is formed, and then diazotized at 0° with sodium nitrite (55 g.) dissolved in water (250 cc.). The diazo solu-

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[1] McCluskey and Sher, THIS JOURNAL, **49**, 452 (1927).

[2] Arkin and Corper, *J. Infectious Diseases*, **18**, 333 (1916).

[3] Quoted by Weismayr in Ott's, "Chem. Path. der Tuberc.," **1903**, p. 480–481.

[4] Jacobs, Heidelberger and Rolf, THIS JOURNAL, **40**, 1585 (1918).