peaks due to the parent molecules were left, *i.e.*, until the CH_3^+ peak from CH_4 had been eliminated.

The results obtained using this method of analysis on a mixture of CH_4 and CD_4 are given in Table I. Table II shows a typical set of results obtained for the analysis of a mixture containing four of the methanes. The precision with which the values could be measured has been indicated.

TABLE I

ANALYSIS OF CH_4–CD_4 MIXTURE

Compound	Analysis at low electron voltage, %	Calculated composition, %
CH_4	66.8 ± 0.4	61.2
CHD_3	1.8 ± .2	1.9
CD_4	31.4 ± .5	36.9

TABLE II

ANALYSIS OF DEUTERATED METHANE MIXTURE

Compound	Analysis at low electron voltage, %	Calculated composition, %
CH_4	35.8 ± 0.6	33.3
CH_3D	33.9 ± .4	32.0
CHD_3	1.5 ± .2	1.6
CD_4	28.8 ± .4	33.0

It can be seen from the results given above that the method of Stevenson and Wagner gives rise to serious errors if used as a general method of analysis for mixtures of deuterated methanes. This is apparently due to the relatively large differences between the ionization potentials of the methanes, as determined by electron impact. For the worst case, that of mixtures containing both CH_4 and CD_4, the CD_4 estimation can be in error by about 15%. It appears likely that a mixture of any successive pair of methanes (CD_nH_{4-n}–$CD_{n+1}H_{3-n}$) can be analyzed by this method with about the same accuracy as a mixture of CH_4 and CH_3D. For methanes farther apart in the series the error increases rapidly.

Very little information is available concerning the effect of deuteration on the ionization potentials of higher hydrocarbons. However, it has been shown[2] that for both acetylene and ethylene the ionization potential of the undeuterated compound is the same as that of the completely deuterated compound within 0.02 e.v. Thus, it would seem that the method of Stevenson and Wagner could be used to analyze, with good accuracy, mixtures of deuterated higher hydrocarbons. The method would be restricted to those cases where the appearance potential of interfering fragments is a volt or two greater than that of the parent molecule.

NATIONAL RESEARCH LABORATORIES
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RECEIVED JULY 16, 1951

NEW COMPOUNDS

1,6-Dibenzyl-3,4-isopropylidene-D-mannitol

A sodium benzyloxide solution was prepared according to the directions of Tishler[1] from 5.5 g. of freshly cut sodium, rinsed with petroleum ether, and 110 ml. of dry (by distillation) benzyl alcohol. Three grams of 1,2;5,6-dianhydro-3,4-isopropylidene-D-mannitol[2] was added and dissolved, and the solution was heated with steam in a jacketed flask under reflux for 24 hours. The reaction mixture was then diluted with chloroform at 0° under vigorous stirring, the sodium benzyloxide was decomposed with cracked ice, and the organic layer which separated was dried over sodium sulfate. After removal of the chloroform at the water-pump, the benzyl alcohol solution was fractionated under high vacuum. The fraction collected at 0.02 mm. and a bath temperature of 160–190° was shown by acetone analysis to contain the desired product, and on redistillation at 0.02 mm. 1,6-dibenzyl-3,4-isopropylidene-D-mannitol came over at 195° (bath temperature). The product was a yellowish, viscous sirup having $n^{25}\text{D}$ 1.5395 and $[\alpha]^{23}\text{D}$ +10.8° (*c*, 5.2 in chloroform). An 0.25-mmole sample dissolved in glacial acetic acid absorbed 2.04 mmoles of hydrogen in 2.5 hr. at room temperature and atmospheric pressure on being shaken with pre-reduced Adams catalyst. No further consumption of hydrogen occurred during the following half-hour. Hydrogenolysis of the benzyl ether linkages and saturation of the aromatic rings requires 8 molar equivalents of hydrogen or 2.0 mmoles.

Anal.[3] Calcd. for $C_{23}H_{30}O_6$ (402.47): C, 68.63; H, 7.51; $(CH_3)_2CO$, 14.4. Found: C, 69.72; H, 7.40; $(CH_3)_2CO$, 14.0.

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(1) L. F. Fieser, "Experiments in Organic Chemistry," 2nd ed., D. C. Heath Co., Boston, Mass., 1941, p. 385.

(2) L. F. Wiggins, *J. Chem. Soc.*, 384 (1946). Our sample was prepared from the 1,6-ditosyl-2,5-diacetyl derivative.

(3) Carbon–hydrogen by the Micro-Tech Laboratories, Skokie, Ill. Acetone was determined by the method of Block and Bolling (R. J. Block and D. Bolling, "The Amino Acid Composition of Proteins and Foods," Thomas, Springfield, Ill., 1940, p. 223) after hydrolysis of the samples in the apparatus of Lester and Greenberg [*J. Biol. Chem.*, **154**, 177 (1944)].

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N-(2-Phenyl-2-ethoxyethyl)-2′-chloroethylamine Hydrochloride

A mixture of 6.0 g. (0.03 mole) of N-(2-phenyl-2-ethoxyethyl)-2-hydroxyethylamine[1] and 4.3 g. (0.036 mole) of purified thionyl chloride in 30 ml. of dry benzene was refluxed for nine hours. The solid, which precipitated during this period of reflux, was recrystallized from isopropyl alcohol, using charcoal to decolorize the solution. Four grams of product (51%), melting at 138–139° (cor.), was obtained.

Anal. Calcd. for $C_{12}H_{18}ClNO \cdot HCl$: N, 5.30. Found: N, 5.28.

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(1) I. A. Kaye and I. C. Kogon, THIS JOURNAL, **73**, 4893 (1951).

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Hexamethylenetetramine Salt of α-Bromobutyrolactone

To a solution of 140.1 g. (1.0 mole) of hexamethylenetetramine in 1750 ml. of hot 95% ethanol was added 165 g. (1.0 mole) of α-bromobutyrolactone.[1] After 22 hours at room temperature, the mixture was filtered and the precipitate washed with ethanol followed by ether. The white crystalline water-soluble product weighed 112.1 g. (37%) and melted with decomposition at 155.5–156° (cor.).

Anal. Calcd. for $C_{10}H_{17}BrN_4O_2 \cdot 2H_2O$: C, 35.21; H, 6.20. Found: C, 35.30; H, 6.20.

5-(β-Isothiuroniummethyl)-hydantoin Bromide.—The intermediate, 5-(β-bromoethyl)-hydantoin, was prepared by

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(1) J. E. Livak, E. C. Britton, J. C. Vander Weele and M. F. Murray, THIS JOURNAL, **67**, 2218 (1945). The yield of α-amino-γ-butyrolactone hydrobromide prepared by these investigators is incorrectly reported as 59.3%; it should be 50.8%. Following their directions the author obtained a 53.8% yield.