ruthenium solution, all divided by the gram atoms of ruthenium per liter in the eluted solution.

The charge per species is found from the concentration dependence of the exchange of ruthenium with a known ion—in our case hydrogen ion

$$\mathrm{Ru}_{b/a}\mathrm{X}^{+b} + b\mathrm{H}^{+}{}_{\mathrm{R}} = \mathrm{Ru}_{b/a}\,\mathrm{X}^{+b}{}_{\mathrm{R}} + b\mathrm{H}^{+}$$

$$Q = \frac{[\mathrm{RU}_{b/a}\mathrm{X}^{+b}]_{\mathrm{R}}\,[\mathrm{H}^{+}]^{b}}{[\mathrm{Ru}_{b/a}\mathrm{X}^{+b}]\,[\mathrm{H}^{+}]^{b}{}_{\mathrm{R}}}$$

where X denotes anions in the ruthenium species, R designates the resin phase and concentrations are indicated by brackets. The ratio of ruthenium concentrations is proportional to the ratio of hydrogen ion concentrations to the $-b$ power. From two equilibrations at different hydrogen ion concentrations b may be calculated. Conditions were chosen such that the great majority of the resin was in the hydrogen ion form and most of the ruthenium was present in the resin phase. There is an unpredictable change in activity coefficients in the aqueous phase but the method is believed to be sufficiently accurate in most cases because b must be an integer as well as a multiple of a.

It can be shown that any changes in hydrolysis on exchange do not affect the results.

This method has been used to establish the formulas of the species Ru^{+3} and RuCl^{+2}.

DEPARTMENT OF CHEMISTRY AND
RADIATION LABORATORY OF THE
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIF.
HOWARD H. CADY
ROBERT E. CONNICK

RECEIVED JULY 10, 1957

SEMI-QUANTITATIVE GAS CHROMATOGRAPHY

Sir:

We have found relationship (1) reasonably accurate in relating the fraction by weight of a component in a mixture to the corresponding area in a liquid–vapor chromatogram obtained using helium as the carrier gas and a tungsten wire thermal conductivity gage as detector

$$\left(\frac{m_i}{W}\right) = \frac{A_i\sqrt{M_i}}{\sum\limits_i A_i\sqrt{M_i}} \qquad (1)$$

where (m_i/W) is the fraction by weight of the ith component, A_i the planimeter-determined area under the peak on the chromatogram corresponding to the ith component and M_i the molecular weight of the ith component.

Clearly the equation may be applied only to mixtures which are completely resolved and such that all components appear in the chromatogram.

Component	Known mole %	Known wt. %	Found area %	Calcd. wt. %
Ethanol	43.4	45.4	47.3	44.6
Water	46.6	19.1	31.1	18.3
1,8-Cineole	10.0	35.5	21.5	37.1

The equation has been applied with comparable results to chromatograms of several mixtures: α and β-pinene plus ethanol; α and β-pinene plus benzene; methyl ethyl ketone, n-propyl alcohol and p-xylene.

Although the expression was arrived at empirically, the appearance of the square root function is not unreasonable on the basis of kinetic theory. Further, it is apparent that the expression may be utilized to determine the molecular weight of a component from the chromatogram of a mixture of known weight composition of the material of unknown molecular weight with a known substance. Although the square root function through its leverage places a premium on accurate determination of the areas on the chromatogram, we have had some success in this utilization of the relationship.

DEPARTMENT OF CHEMISTRY
AND CHEMICAL ENGINEERING
STANFORD UNIVERSITY
STANFORD, CALIFORNIA
RICHARD H. EASTMAN

RECEIVED JUNE 7, 1957

TETRAACETYL DIBORATE AND SO-CALLED "BORON ACETATE"

Sir:

There has been considerable confusion in the literature over the constitution of the product obtained by the reaction between orthoboric acid and acetic anhydride. Two types of compounds have been reported at various times. One, said to melt around 121°, has been called boron triacetate, $(\mathrm{CH_3CO_2})_3\mathrm{B}$,[1,2,3] and the other, m.p. 150–152°, pyroboron acetate, $(\mathrm{CH_3CO_2})_2\mathrm{BOB}(\mathrm{O_2CCH_3})$.[4,5,6] Both have been supported by analytical data but, in several cases, the authors used identical procedures only to obtain different products.

We have repeated some of the previous preparations, our results agreeing with those of Gerrard and Wheelans[6]; no evidence was obtained for boron triacetate, although the methods described for its preparation[1,2] were carefully followed. The analytical data for $(\mathrm{CH_3CO_2})_2\mathrm{BOB}(\mathrm{O_2CCH_3})_2$ were improved and extended and the name *tetraacetyl diborate* is proposed.

The reaction between orthoboric acid and acetic anhydride was found to occur according to the equation

$$2\mathrm{H_3BO_3} + 5(\mathrm{CH_3CO_2})_2\mathrm{O} \longrightarrow (\mathrm{CH_3CO_2})_2\mathrm{BOB}(\mathrm{O_2CCH_3})_2 + 6\mathrm{CH_3CO_2H}$$

Orthoboric acid (0.534 mole) and acetic anhydride (1.96 mole) were heated together at 50–100° with vigorous stirring in a nitrogen atmosphere until all the solid had dissolved. On cooling, the product crystallized as colorless needles. One sample was purified by recrystallization from a benzene–chloroform mixture, another by precipitation from solution in dry acetic acid by the addition of ether. Both melted at 147–148°.

All filtrations were carried out using a pressure of dry nitrogen and the products were dried in vacuum at room temperature before being transferred to a dry box for subsequent handling.

The acetate content was determined by titration with standard base, the end-point (around pH 6.7)

---

(1) A. Pictet and A. Geleznoff, *Ber.*, **36**, 2219 (1903).

(2) H. Cook, L. Ilett, B. Saunders and G. Stacey, *J. Chem. Soc.*, 3125 (1950).

(3) T. Ahmad and M. Khundkar, *Chem. and Ind.*, 248 (1954).

(4) O. Dimroth, *Ann.*, **446**, 97 (1925).

(5) H. Meerwein and H. Maier-Hüser, *J. prakt. Chem.*, **134**, 51 (1932).

(6) W. Gerrard and M. Wheelans, *Chem. and Ind.*, 758 (1954).