

Figure 14. Infrared Spectra of Plant Stream Olefin Concentrates from Silica Gel Adsorption

Table V. Comparison of Olefin Types in Refinery Products^a

Plant Stream	Alpha	Internal Double Bond
Catalytically cracked gasoline	9	14
Thermally reformed gasoline	13	13
Thermally cracked gasoline	43	32
Thermally cracked gasoline after hydrotreatment	10	50

^a To enable comparison of relative concentration in stream, ratios are corrected to total olefin basis of stream.

mally cracked gasoline blend, and (4) a hydrotreated product of (3). The spectrum of the olefin concentrate of the hydrotreated product was shown in Figure 13 at a liquid hourly space velocity of 10, and the spectra of the olefin concentrates of the other three gasolines were determined for the region 9.5 to 12.5 microns, and are shown in Figure 14.

Using representative olefin type calibration data as shown in Figure 10, the olefin distributions listed in Table V were obtained. In these analyses the olefin type concentrations were corrected to the total olefin concentration of the original sample as determined by bromine number. The sums of the uncorrected concentrations of the olefin types were from 3 to 12% below the value found by bromine number.

The infrared portion of the presented application required less than 0.5 ml. of sample and approximately 30 minutes of analytical time.

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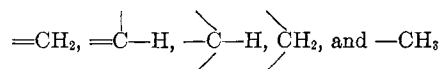
Infrared Analysis of Organic Mixtures

Using C—H Band Structure Resolved by a Lithium Fluoride Prism

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THE present-day availability of lithium fluoride prisms for the various commercially available infrared spectrometers extends the usefulness of infrared spectroscopy very greatly. It is well known (1, 9) that lithium fluoride provides a large increase in dispersion over sodium chloride in the 2 to 5.5 μ (5000 to 1820 cm^{-1}) region. This brings out structure characteristics, particularly in the spectra due to the stretching vibrations of the C—H valence bonds. Fox and Martin (6) and Rose (8) have shown that different types of substitutions in hydrocarbons

produce different, recognizable C—H absorption bands. Fox and Martin considered the following groups:



The bands due to these groups all lie near 3.4 μ and are so closely spaced that they are not satisfactorily resolved in the ordinary instrument that employs a sodium chloride prism.

With a prism of sodium chloride the usual infrared spectrometer does not resolve the structure of the absorption bands due to C—H groups. A lithium fluoride prism which possesses high dispersion in this spectral region resolves these bands very satisfactorily and makes them available for analytical determinations. Such a use of a lithium fluoride prism has been explored and a number of specific analyses have been developed. This method of analysis allows the examination of compounds and mixtures in dilute solutions of carbon tetrachloride as well as the analysis of compounds subject to complex formation such as hydrogen bonding and the analysis of mixtures possessing large differences of intensity of absorption in the sodium chloride region. It makes possible a new approach to systems of very similar isomers; and it extends the usefulness of instruments equipped with such a prism for molecular structure determination work.

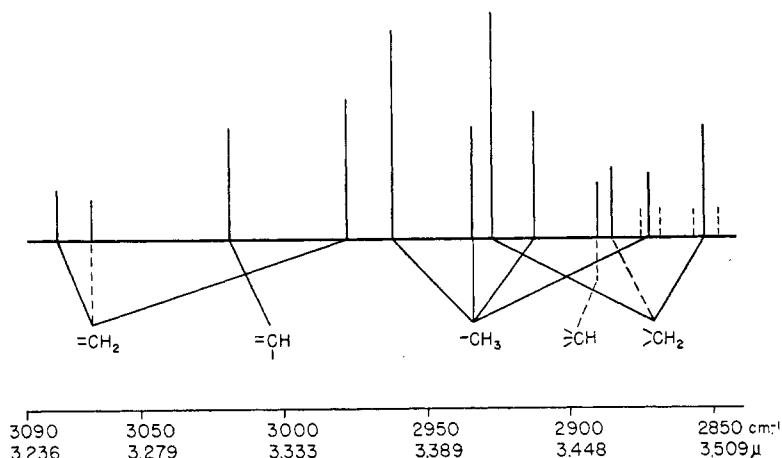


Figure 1. Absorption Frequencies Observable for Different Classes of C—H Groups in Hydrocarbons When Sufficient Resolution Is Achieved with Lithium Fluoride Prism

Data from Fox and Martin (6)

The use of a lithium fluoride prism, however, makes possible the utilization of the C—H structural bands for analytical applications. The present paper reports some of the results obtained in these applications.

Figure 1 presents an absorption band diagram synthesized from the empirical data of Fox and Martin (6) for hydrocarbons. This shows the positions and multiplicity of the absorption bands due to the various types of carbon-hydrogen groupings. The relative heights of the lines are some indication of the intensities of the observed absorption bands. However, neither these nor the wave-length positions may be regarded as exact, as variations occur in going from compound to compound. The dashed lines below the base line represent absorption bands observed only in some of the cases studied. The dashed lines above the base line are for the cases where a single band splits into a doublet. Despite these limitations it is evident from the figure that with the satisfactory resolution of these bands, which is possible using a lithium fluoride prism, the scope of applications of infrared spectroscopy may be greatly enlarged. As more data become available in the future it will be possible to determine the specific limitations and generalities of such data as those presented in Figure 1.

It is to be observed in Figure 1 that the absorption bands for the saturated carbon-hydrogen groupings occur at longer wave lengths than do those for the unsaturated groupings. This immediately suggests the use of the C—H bands for the quantitative analysis of mixtures of olefins and paraffins. These systems may be handled, as well as ones containing aromatics and oxygenated compounds. Because of the relatively narrow spectral

region and the limited number of bands available, the number of components per sample cannot be as large as can be handled when a sodium chloride prism is used in the longer wave-length region. However, the advantages of the present method lie in the handling of special types of mixtures which often have only a small number of components.

One advantage in the use of the C—H absorption band structure lies in the availability of an excellent solvent for this region. Carbon tetrachloride is very transparent throughout this region and many compounds and systems of compounds are soluble in it. It is so transparent that cells up to several centimeters in length can be used. This makes it possible to examine compounds and mixtures in dilute solutions. Under these conditions the inherent difficulties due to molecular association such as hydrogen bonding may be avoided. Also it makes possible the simultaneous analysis in the same mixture of compounds that may have large differences of intensity of absorption in the longer wave-length

regions such as paraffins and some of the polar or unsaturated materials. In some cases, mixtures of homologous compounds which do not have appreciably different spectra in the long wave-length region can be satisfactorily analyzed by this method. A further advantage of the method is a spectrometric one. Because the spectral region used is near the maximum for the radiation curve, the scattered light is very weak. In fact, it is so weak that the authors have detected none at all. This eliminates a correction procedure that must be applied at the longer wave lengths for best results. A further advantage lies in the extension of usage to analytical problems of an instrument equipped with a lithium fluoride prism and otherwise primarily used for molecular structure determination work. The use of the samples in solution also gives greater accuracy, in that it allows a true correction for the absorption, reflection, and scattering of the radiation beam by the cell itself. This is done by comparing the transmittance of the sample in solution with the transmittance of the solvent alone.

The accuracy attainable for a specific mixture depends upon the differences in intensity of absorption that can be utilized. In some cases where unique absorption bands can be found, the average errors are of the order of 0.1 to 0.2% of total sample. Where the wave length and intensity discrimination are not very good, the average errors may be of the order of 1.0% of total sample. High accuracy was not an all-important aim of the present work. Rather, a compromise in reasonable accuracy, rapidity of analyses, and ease of analyses by nontechnically trained personnel was desired. All calibration data and synthetic samples were processed under routine conditions.

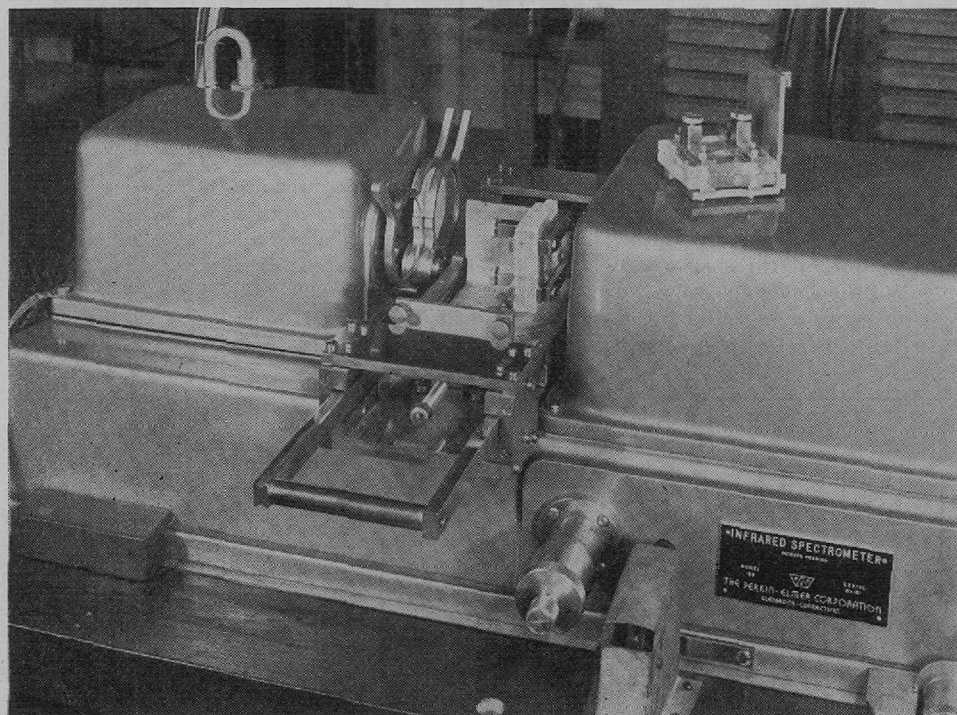


Figure 2. Liquid Absorption Cells and Mounting Arrangements Used in Analytical Work

An assay of the accuracy of the method under routine conditions may be made from the results reported for the synthetic samples below.

EXPERIMENTAL METHODS AND TECHNIQUES

The instrument on which the present work was done was a Perkin-Elmer Model 12B infrared spectrometer. Sodium chloride and lithium fluoride prisms are used in it interchangeably. A cell-in-cell-out arrangement is used wherein the absorption cells are securely clamped to a movable carriage. In Figure 2 may be seen one of the cells clamped in place and another one resting on top of the instrument. The type of cell used has been described (3). The carriage arrangement is the same as used in routine gas analysis (4). The liquid cells are equipped with a mounting frame which allows them to be held in the movable carriage by means of thumbscrews. The cell employs two needle valves, one fabricated to act as a filling piston when loading the cell. These cells have interchangeable parts and are easily fabricated and repaired. The thickness used most commonly in the present work was 0.006 inch.

For quantitatively measuring the radiation intensities at the various wave lengths, the same system of null measurements (4) was used. A high sensitivity galvanometer is used merely as an indicator and the thermocouple signals are balanced out by means of a low voltage obtained from a resistor and potentiometer network. Difficulties due to nonlinearity or change of sensitivity of the high sensitivity galvanometer are thereby eliminated. The instrument is equipped with an Amphenol connector for the thermocouple outlet. With this arrangement either the galvanometer and null system equipment or the automatic recorder may be connected at will. It has been found very convenient to use the automatic recorder to obtain a spectrum of the sample throughout the wave-length region used. This may then be examined to determine what compounds are present or, if the sample is a calibration standard, to determine the most desirable wave lengths to use in the analytical procedure. However, for quantitative measurements of transmittance at specific wave lengths consistently more accurate data are obtained manually, using the null method. The same equipment and general methods have been in use here for several

years for the quantitative analysis of multicomponent mixtures of hydrocarbons, using a sodium chloride prism and the longer wave-length region.

Tests for the scattered radiation intensity were made by the total absorber method (2). For some of these an absorption cell of 0.036-inch thickness was used. Almost any material containing C—H groups is suitable as solute for such tests in the 3.4 μ region, provided a strong enough concentration is used.

In the present work all dilutions were made volumetrically in graduated pipets and burets. In certain cases care must be taken to follow a definite procedure in making the calibration blends, diluting the samples, and obtaining the data, in order to avoid trouble caused by differences in evaporation rates. It is well known (7) that the high boiling points of the simple alcohols, in comparison with other compounds of similar molecular weights,

are due to the hydrogen bonding forces between the molecules. These hydrogen bonding forces, due to the hydroxyl groups, are responsible for molecular association between the molecules and the attendant reduction in vapor pressure. However, in dilute solutions in a solvent such as carbon tetrachloride the relative degree of hydrogen bonding is reduced because of the greater average distance between hydroxylated molecules. Consequently, the alcohol molecules in such a solution will escape much more rapidly, on a relative basis, than from a concentrated solution. This effect is so strong that the same procedure as regards time intervals must be used on the samples to be analyzed and on the calibration blends. Otherwise errors will arise due to loss of some of the material by evaporation. In Figure 3 may be seen the optical density plotted as a function of the number of times analyzed for a 2.5% by volume solution of ethyl alcohol in carbon tetrachloride. This sample was kept in a glass-stoppered bottle and analyzed on successive days. During these tests the concentration of ethyl alcohol changed radically. The

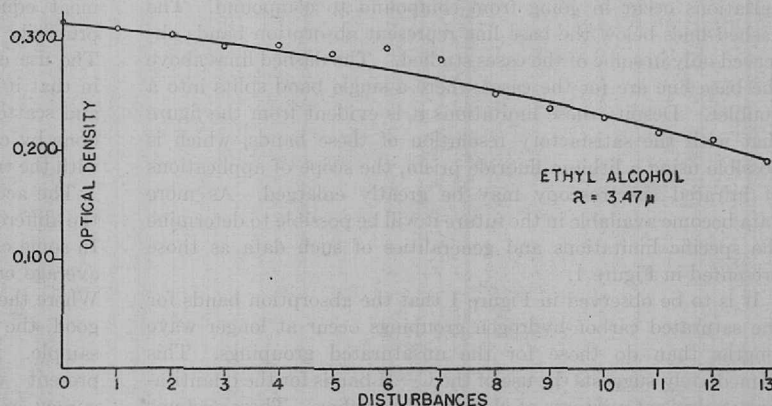


Figure 3. Optical Density of Dilute Solution of Ethyl Alcohol in Carbon Tetrachloride Solution as a Function of Number of Times Analyzed (Disturbances)

authors have found it most advisable to obtain the data for such a solution on the same day as it was prepared.

As Beer's law of absorption is obeyed by the compounds under investigation the calculations and handling of data are straightforward. The absorption, reflection, and scattering effects due to the cell may be eliminated by comparing the transmittance of the sample in solution with the transmittance of the solution alone. Actually this is accomplished by a suitable subtraction of optical densities.

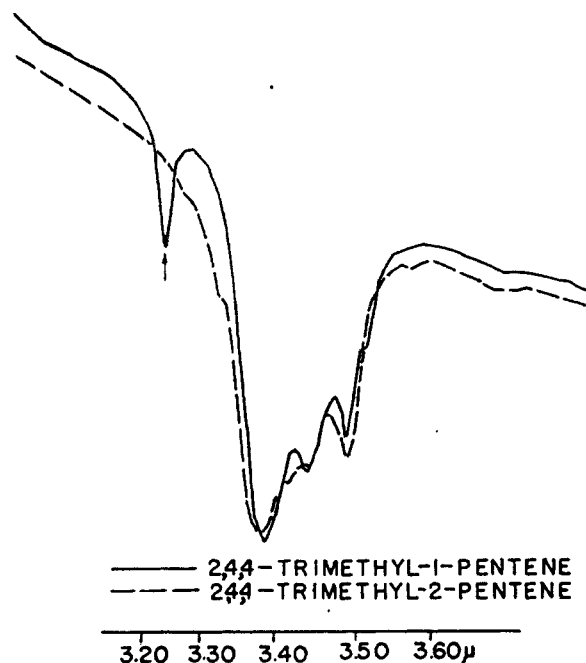


Figure 4. Infrared Absorption Spectra in C—H Region of Isomeric Trimethylpentenes

To see this let us consider the transmittance of a solution of pure compound at wave length λ_i . We have for the solution:

$$D_i = \log(I_0/I) = A_i C + K_i \quad (1)$$

where D_i is the optical density, I_0 is the incident radiation intensity, I is the transmitted radiation intensity, A_i is the calibration coefficient for the pure compound at wave length λ_i , C is the concentration of the solute, and K_i is an attenuation factor due to the absorption, reflection, and scattering by the cell and absorption by the solvent. Since the concentrations of solute used are small, between 1 and 6% for the present work, the K_i in Equation 1 may be evaluated by obtaining the optical density for the cell filled with solvent only. A subtraction of this value from the D_i of Equation 1 then gives the component of optical density due to the compound. With this method of obtaining the optical density, Beer's law was tested for a number of the compounds in the examples below and found to hold in each case.

With the above method of obtaining true optical densities and with the additivity of optical densities that occurs when Beer's law is obeyed, simple equations of the type below are obtained. Here a three-component system is assumed.

$$\begin{aligned} D_1 &= A_{11}C_1 + A_{12}C_2 + A_{13}C_3 \\ D_2 &= A_{21}C_1 + A_{22}C_2 + A_{23}C_3 \\ D_3 &= A_{31}C_1 + A_{32}C_2 + A_{33}C_3 \end{aligned} \quad (2)$$

Here D_1, D_2, D_3 refer to the true optical densities of the mixture at the three wave lengths chosen for operation; A_{11} is the calibration coefficient of the first compound at the first wave length, A_{23} is the calibration coefficient of the third compound at the second wave length, etc.; and $C_1, C_2,$ and C_3 are the concentrations of the three compounds. These equations may be readily solved for the concentrations by matrix methods (2) or by successive

approximations. In the method of successive approximations concentrations C_2 and C_3 are assumed to be zero in the first equation and it is solved directly for C_1 . This value is used in the second equation with the assumption that C_3 is zero and a value for C_2 is determined. Next the values of C_1 and C_2 obtained in the first two steps are used in the third equation and it is solved for C_3 . The cycle is repeated with the values of C_2 and C_3 from the latter two steps substituted in the first equation and a new value of C_1 is determined. The second equation is then solved for C_2 using the most recent values of C_1 and C_3 , etc. Several cycles can be quickly run with a semi-automatic or automatic calculating machine in a few minutes.

A wave-length calibration of the C—H region was prepared from the data of Fox and Martin (5, 6). No attempt was made to make a highly precise calibration. In practice the wave lengths for specific analyses are specified in terms of the instrument vernier readings. For that reason specific wave lengths are not given below. For some of the examples the wave-length values may be appraised from the figures. It is believed that these may be relied on to within about 0.005μ or better.

APPLICATION TO SPECIFIC ANALYSES

From the information given in Figure 1 a prediction may often be made as to whether or not a specific mixture can be analyzed. For example, a binary mixture containing two compounds with differences in types of unsaturated groups may generally be analyzed with ease.

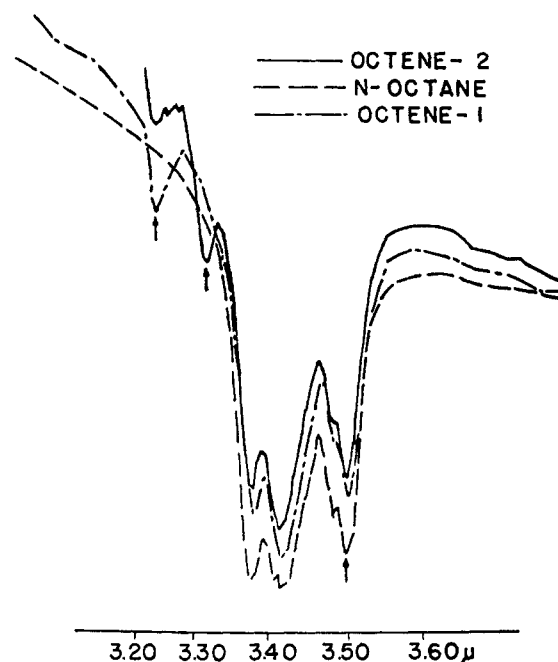


Figure 5. C—H Structure Absorption Bands for 2-Octene, *n*-Octane, and 1-Octene

A specific example of this is a mixture of similarly branched trimethylpentenes. In Figure 4 may be seen the spectra of 2,4,4-trimethyl-1-pentene and 2,4,4-trimethyl-2-pentene. In these curves, which are reproductions of the automatically recorded spectra, the transmitted energy is plotted as a function of wave length. Here, as indicated by the arrow, may be seen the absorption band due to the terminal $=CH_2$ group in one of the compounds. Actually, the difference in absorption is great enough here to permit taking data at this point only. Then instead of having two equations of the type of Equations 2 we have but one plus the equation:

$$C_1 + C_2 = C \quad (3)$$

where C is the concentration of sample in solution. The results of analyses of two synthetic samples may be seen in Table I.

Table I. Analyses of Synthetic Mixtures of Binary Mixture of Trimethylpentenes

Compound	Synthetic, %	Calculated, %	Difference, %
2,4,4-Trimethyl-1-pentene	50.0	50.4	0.4
2,4,4-Trimethyl-2-pentene	50.0	49.6	-0.4
2,4,4-Trimethyl-1-pentene	30.0	30.6	0.6
2,4,4-Trimethyl-2-pentene	70.0	69.4	-0.6

Table II. Analyses of Synthetic Mixture of Hydrocarbons Containing Paraffins and Unsaturation

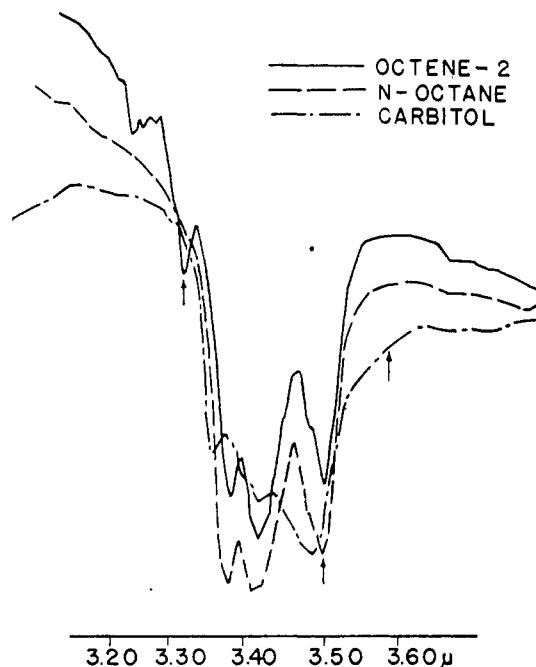
Compound	Synthetic, %	Calculated, %	Difference, %
<i>n</i> -Octane	20.0	18.9	-1.1
2-Octene	30.0	31.0	1.0
1-Octene	50.0	50.1	0.1
<i>n</i> -Heptane	50.0	49.9	-0.1
Benzene	50.0	50.1	0.1
<i>n</i> -Heptane	75.0	75.1	0.1
Benzene	25.0	24.9	-0.1

**Figure 6. Infrared Absorption Structure in C-H Region for Benzyl Alcohol, *n*-Octanol, and Methyl Ethyl Ketone**

Continuing with hydrocarbon systems we see in Figure 5 that satisfactory wave-length discrimination is obtainable for the ternary system of *n*-octane, 2-octene, and 1-octene. The wave lengths used for analysis are indicated by the arrows. At about 3.26 μ may be seen the band characteristic of the terminal =CH₂ group of 1-octene, at about 3.32 μ the band characteristic of the =CH group of 2-octene, and at about 3.50 μ the band characteristic of the CH₂ groups, which is stronger for *n*-octane than for the others. The 2-octene shows some of the bands characteristic of the other compounds, because the sample used for recording was not pure.

Although definite differences in spectra are found, the differences in intensity of absorption are not great (Figure 5). Despite this, fair accuracy is obtainable, as may be seen in Table II.

Table II also gives the results for two synthetic samples of the binary system of *n*-heptane and benzene, for which considerably better accuracy is possible.

**Figure 7. C-H Structure of Absorption Bands for 2-Octene, *n*-Octane, and Carbitol**

In the preceding type of examples predictions could be made in advance concerning the success of the method. In other cases, such as the examples discussed below, an idea of the feasibility cannot be reliably formulated until the spectra of the pure compounds are obtained. A number of ternary mixtures of particular interest in the study of azeotropes may be easily analyzed. In general, these systems contain a paraffin, an aromatic or olefin, and an oxygenated compound. Other systems containing all oxygenated compounds may in some cases be analyzed with good accuracy.

In Figure 6 may be seen the spectra in the 3.4 μ region of benzyl alcohol, *n*-octanol, and methyl ethyl ketone. Here it is observed that three distinct and unique wave lengths, indicated by arrows, may be chosen for analysis. Table III gives the results on synthetic samples made up to test the accuracy on such a system.

Table III. Analyses of Synthetic Mixtures of the System: *n*-Octanol-Methyl Ethyl Ketone-Benzyl Alcohol

Compound	Synthetic, %	Calculated, %	Difference, %
<i>n</i> -Octanol	30.0	30.3	0.3
Methyl ethyl ketone	30.0	29.4	-0.6
Benzyl alcohol	40.0	40.3	0.3
<i>n</i> -Octanol	25.0	24.5	-0.5
Methyl ethyl ketone	50.0	50.5	0.5
Benzyl alcohol	25.0	25.0	0.0

In some cases the spectra of the compounds in the mixtures to be analyzed have such similar spectra that distinctive wave lengths may not be found, but sometimes it is possible to utilize the difference of intensity on the shoulders of the bands for analytical application.

An example of such a case is seen in Figure 7, which is for the system 2-octene-*n*-octane-Carbitol (diethylene glycol monoethyl ether). Here the arrows again illustrate the wave lengths used for analysis. For the Carbitol, utilization is made of its increased absorption in the long wave-length shoulder of its

Table IV. Analyses of Synthetic Samples of Ternaries of *n*-Octane, 2-Octene, Carbitol, and Ethylbenzene

Compound	Synthetic, %	Calculated, %	Difference, %
Carbitol	47.4	47.1	-0.3
<i>n</i> -Octane	18.0	17.6	-0.4
2-Octene	34.6	35.3	0.7
Carbitol	37.5	37.2	-0.3
<i>n</i> -Octane	25.0	25.3	0.3
Ethylbenzene	37.5	37.5	0.0

Table V. Analyses of Synthetic Samples of Ternaries Containing a Paraffin, an Unsaturate, and an Oxygenated Compound

Compound	Synthetic, %	Calculated, %	Difference, %
Methylcyclohexane	25.0	24.6	-0.4
Methyl ethyl ketone	35.0	34.7	-0.3
Toluene	40.0	40.7	0.7
Dichloroethyl ether	70.0	68.7	-1.3
<i>n</i> -Octane	10.0	10.9	0.9
Ethylbenzene	20.0	20.4	0.4
Cyclohexane	30.0	30.7	0.7
Ethyl alcohol	40.0	39.4	-0.6
Benzene	30.0	29.9	-0.1
Hexanol	40.0	40.3	0.3
<i>n</i> -Heptane	20.0	21.0	1.0
Heptenes (mixture)	40.0	38.7	-1.3

Table VI. Analyses of Synthetic Samples of Binary Mixtures of Methyl and Ethyl Alcohol

Compound	Synthetic, %	Calculated, %	Difference, %
Methyl alcohol	51.6	52.0	0.4
Ethyl alcohol	48.4	48.0	-0.4
Methyl alcohol	91.4	92.1	0.7
Ethyl alcohol	8.6	7.9	-0.7

C—H absorption region. Distinct bands near 3.40μ for *n*-octane are available but are not used, because the absorption there is too strong at the concentrations necessary to bring out adequate absorptions at the other wave lengths. The results for this system as well as those for a closely similar system in which the 2-octene is replaced by ethyl benzene may be seen in Table IV. In this latter system the difference of absorption intensity on the long wave-length shoulder was again utilized.

In the preceding discussion a few specific types of analyses found to be practical have been enumerated and the results given on some synthetic mixtures. The practicality of the method for such systems can best be determined after the absorption bands are automatically recorded. In Table V are given a few more results on other systems for which the spectra will not be shown. In Table VI are given the results of tests on synthetic samples of binary mixtures of methyl and ethyl alcohol.

The attainable accuracy depends upon the particular system being analyzed. The method has been found to give satisfactory results for almost all binary and ternary systems investigated. It is also suitable for the analysis of gas mixtures. It considerably augments the usefulness of the absorption spectroscopic techniques in use at this laboratory and is now used as a routine procedure. The time required per sample is approximately the same, except for the dilutions, as for analyses by infrared when a sodium chloride prism and the longer wave lengths are used.

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Magnetic Stabilizer for Direct Current Arcs in Spectroscopy

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IT IS generally recognized that although the direct current arc is a versatile source of excitation, it provides relatively poor reproducibility when used for quantitative spectrochemical analyses. Apparently this defect is due chiefly to wandering of the cathode "spot," which produces both wandering of the arc and fluctuations in amperage with attendant changes in the discharge temperature. Ways of improving reproducibility are available. Hasler and Harvey (1), Jeppesen, Eastmond, and Logan (2), and others have described electrodes specially shaped for the purpose. Jaycox and Ruehle (3) average the effects of wandering of the arc by rotating the lower electrode holding the sample. Mixing the powdered sample with a "spectroscopic buffer" may improve reproducibility. Recently Myers and Brunstetter (4) recommended a rotating magnetic field placed near the arc.

The present paper describes an instrument that eliminates almost completely the wandering of a central horizontal slice of the arc. Almost any part could be rendered free of fluctuations, but

in the present paper attention is directed only to that part from which the light is dispersed and photographed.

PRINCIPLE OF THE INSTRUMENT

Normally, a direct current arc is surrounded by a circular magnetic field, depicted by broken circles in Figure 1. By reason of a well-known principle, placing one pole of a horizontal bar magnet near the arc will cause the arc to move sideways in a direction at right angles to the magnet's polar axis. The magnetic field from a north pole moves the arc to the left of the magnet, and from a south pole moves it to the right, when the lower electrode is positive.

Figure 1 shows how the magnetic effect just described was applied to eliminate wandering of the central part of the arc. Figure 2 shows the magnetic stabilizer equipment in position, the two slits of the phototube housing opened wider than usual, in