

# THE SPEX SPEAKER

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## SOME APPLICATIONS OF SPECTROSCOPY IN THE PETROLEUM INDUSTRY

Tying up at the edge of a sprawling American petroleum refinery, a huge supertanker has made another 6000-mile round trip. Crews attach hose lines, and its cargo—a foul-smelling, gooeey, black liquid—starts to flow. Soon it will be distilled, cracked, reacted, filtered, blended, crystallized, washed. So it is converted into a host of fuels, lubricants, waxes, greases, illuminating gases, carbon-black, asphalt, and thousands of petrochemicals. It's the job of the analyst to monitor intermediates during processing, to maintain and improve quality and uniformity of the end products. To a large degree, the analyst's tools are spectrographs—optical and x-ray emission, absorption utilizing many regions of the electromagnetic spectrum, mass and nuclear magnetic resonance. But, let's begin by getting an idea of the production process.

### Production of Gasoline

Preliminary crude oil is first subjected to distillation. Here, several fractions or "cuts" are taken. In one boiling range, straight-run gasolines are formed. In others, the products are petroleum ether, kerosene, fuel oils, naphthas, lube oils and residua. Unlike the proverbial pig's squeal, nothing is wasted. The residual fraction is either reworked to obtain asphalt and coke or used directly as a bunker fuel to power ships and electrical generating plants.

From the standpoint of volume, gasoline is of most interest to the producer. Straight-run gasolines are no longer adequate. Gasoline for a modern automobile engine must:

- 1) boil and flash within close limits to insure complete volatilization in the carburetor and burning in the cylinders. This also improves rate of acceleration and decreases crankcase oil dilution.
- 2) contain certain portions vaporizing within even smaller limits to impart good starting and good mileage qualities.
- 3) have a vapor pressure low enough to prevent vapor lock which disrupts gasoline flow to the carburetor.
- 4) have an octane rating high enough to prevent knocking or pre-ignition. Modern engines use high compression in order to improve efficiency and reduce engine weight. In turn, the high compression requires a high octane rating.
- 5) be stable in storage and not deposit gums.

- 6) be non-corrosive. Sulphur is an offender and must be kept very low, particularly in cold weather when it reacts with condensed water vapor to form corrosive acids.
- 7) burn completely without causing spark-plug or engine deposits.
- 8) have a non-objectionable odor.
- 9) be modified for the season of the year in order to maintain good operational qualities regardless of the weather.
- 10) contain additives to resist self-oxidation, corrosion and counteract carburetor icing under certain weather conditions.

By and large, modern refinery operations center about the problem of meeting all of these specifications for quality gasolines. Cracking breaks down higher-boiling compounds into lighter ones of superior octane rating. In the presence of catalysts, reactions are favored which yield desirable compounds and reduce the yield of unstable, unsaturated hydrocarbons. The actual yield of gasoline from crude is doubled through cracking operations. Modern refineries have facilities for polymerization to produce and recover olefinic waste gases; for solvent extraction to rid lube oils and diesel fuels of unsuitable hydrocarbons and for catalytic reforming to produce superior gasolines.

Another step in gasoline production is the removal of corrosive sulphur compounds and skunk-like mercaptans. And finally, materials such as tetraethyl lead, TCP and organic boron compounds are added to reduce deposits and surface ignition and to boost octane rating even more. Like the physician who prescribes penicillin as a cure, and, at the same time, a medicine to curb its side effects, gasoline formulators add chlorine and bromine compounds to the TEL. They improve the volatility of the lead and reduce the formation of lead engine deposits as the gasoline burns.

### Other Products

Fortunately, the desirable properties of diesel fuels dovetail with those of gasoline. That is, the boiling range is relatively high, where for gasoline it is relatively low. Further, the method of ignition dictates a low octane value rather than high. These specifications permit the profitable use of fractions unsuitable for gasoline.

Fuel oils and kerosenes are usually straight-through distillates. Our home fuel oil supplier states that an anti-corrosion

agent is added. Otherwise, water settles out in the storage tank and, as some of us have unhappily experienced, it eventually rusts through the tank. Bunker No. 6, a residual fuel, is sold as a waste product. It contains traces of many metals which do not distill off in processing. Vanadium is the most troublesome one. Under certain conditions, it causes corrosion and pitting of boiler tubes. In fact, at least one experimental tanker with a steam engine designed to use bunker fuels is equipped with a simple spectrographic laboratory to check fuel oils before delivery.

Contrasted with cheap fuel and bunker oils, great stress is placed on the quality of lubricating oils which protect expensive machinery. These must:

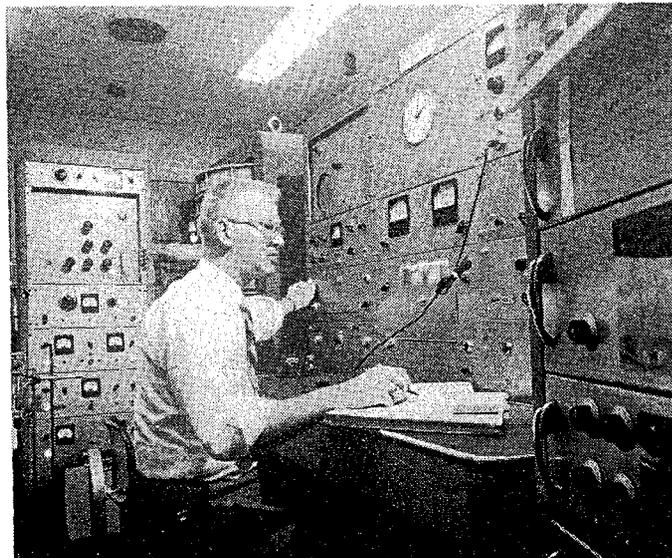
- 1) have good lubricating qualities for the purposes intended.
- 2) be stable for long periods under operating conditions.
- 3) be non-corrosive.
- 4) have low carbon residue on evaporation.
- 5) have proper flash and fire points, color, etc.
- 6) have small viscosity changes with temperature changes especially to aid in low temperature starting.
- 7) have high detergency.

High detergency (H.D. may also mean heavy duty) is a recent development in oils and is obtained largely through the use of metallo-organic additives. These hold all foreign particles in suspension and so help reduce wear on the moving parts. Diesel oils must have high detergency because foreign particles, even atmospheric dust, can clog the injection pump if they get into the firing chamber. Modern oils are age-stabilized, also fortified against the formation of varnishes and gummy deposits on the pistons and cylinder walls.

In addition to fuels and lubricants, other cuts in the distillation process yield substances ranging from highly volatile petroleum ether through the waxes to the semi-solid asphalts. Today, refineries also produce illuminating gas as a by-product which often is blended with natural gas before it is piped to the consumer. Catalytic towers are capable of rearranging molecules to produce many chemicals used as solvents, insecticides, medicines, intermediates and starting compounds.

### Mass Spectroscopy

The type of spectroscopy which is probably of greatest importance to the operations of a refinery is mass spectroscopy.



**MASS SPECTROMETER**—This massive installation cuts to minutes analytical work which ordinarily would take days. It analyzes gaseous and liquid petroleum products to determine the components and percentages of each in a given sample. Information obtained is computed on IBM machines.

Below, is a reproduction of part of a chart turned out by an IBM 402 Accounting Machine about 30 minutes after a sample was taken from a cracking tower to P. L. Work's lab at the Tidewater Oil Company's Delaware Refinery. The first column lists 17 components on which the production engineers keep tabs. The third column gives the percentage of these components (a decimal point is omitted before the last digit). In the fourth column, the air is subtracted and the percentage figures recalculated; and in the fifth column, the hydrogen sulfide and carbon dioxide values are subtracted to give figures based on the total hydrocarbon content. Other figures on the complete chart give the date, certain check figures and preliminary data needed to make the calculations.

While the computers are whirring away, the mixture is, of course, still reacting. The figures lose some of their accuracy in the thirty minutes of analysis time. Older analytical tech-

3089 CHECK PRESSURE	20848						11513
3089							11513
3089							11513
3089		1102					11513
3089 AIR	32	13	14	14	285		11513
3089 NONCONDENSIBLES	28				038 CR		11513
3089 H <sub>2</sub> S	34						11513
3089 CARBON DIOXIDE	44				099 CR		11513
3089 HYDROGEN	2	1	1	1	013	351	11513
3089 METHANE	16				074 CR	664	2 11513
3089 ETHENE	26				116 CR	988	3 11513
3089 ETHANE	30				003 CR	988	4 11513
3089 PROPENE	42				042 CR	1 54	5 11513
3089 PROPANE	29	7	7	7	148	1 54	6 11513
3089 BUTADIENE	54	6	6	6	124	1 72	7 11513
3089 BUTENES	56	479	485	485	10169	1158	8 11513
3089 ISOBUTANE	43	202	205	205	4295	1283	9 11513
3089 N BUTANE	58	190	193	193	4039	1242	10 11513
3089 PENTENES	70	29	29	29	609	1328	11 11513
3089 ISOPENTANE	57	70	71	71	1495	1434	12 11513
3089 N PENTANE	72	3	3	3	066	1417	13 11513
		1000	1014	1014	21243*	20871	

Portion of chart taken directly from IBM 402 Accounting Machine showing concentration figures for 17 components.

niques, however, requiring days would force a shut-down of the tower if the production department were to make use of the data. Even the mass spectrometer, before the days of the electronic computer, had too long a delay time for efficient use of the data. Analyzing a 17-component system involves the solution of an equal number of simultaneous equations—a time-consuming job if handled by man-power alone.

Today, the mass spectrometer lab consists mainly of computing equipment as shown in the photograph. Raw intensity figures from the Consolidated Electrodynamics Corp. mass spectrometer are fed to a Spectro Sadic which is basically an analog to digital converter. From here, the data go to an IBM 514 card puncher and next to a 602A. This computer solves the simultaneous equations through an inverted matrix type of calculation and punches a set of answer cards. The IBM 402 produces the chart analysis record from the answer cards.

At the Standard Oil Company (Indiana) Whiting Refinery, F. W. Porsche reports that the mass spectrometer's major contribution is in pilot plant design. Here it is used to study yields of various components under different operating conditions. Such studies enable the engineers to work out conditions yielding optimum blends. Another extensive application of the mass spectrometer at Whiting is for the determination of such gases as butane in streams. Economically, butane is more valuable in gasolines than in fuel oils. Accordingly, constant checks are made to make sure that butane is not diverted into fuel-oil streams.



**MACHINE MAGIC**—IBM machines at Tidewater Oil Company's Delaware Refinery calculate and print out information from the Mass Spectrometer in the refinery's Quality Control Laboratories. Sorter (left) collates the punch cards. Calculating punch (center rear) determines the results. Machine at right prints out final results in visible form.

To summarize the great contribution of the mass spectrometer, it enables production to obtain, with sufficient speed, the desired chemical blend. Years ago, refineries had essentially a single source of crude oil. With it, production could use the same basic processing techniques and expect to produce a reasonably uniform end product. Today, crude oil is obtained from all parts of the world. Compositions vary so enormously that production procedures must be shifted continually to maintain required properties of the end products.

## Emission Spectroscopy

One of the most important non-routine applications of the spectrograph is in connection with corrosion problems in the refinery. So bothersome is corrosion that full-time engineers constantly make the rounds to hunt down potential trouble spots. It has been estimated that 2¢ on the cost of every gallon of gasoline is the price levied by corrosion from the time it is taken from the ground to when it is delivered to the motorist. Sulphur is the most offensive element, combining, as it does, to form sulphuric acid, hydrogen sulfide and mercaptans. In tanks, pumps, pipe-lines and elsewhere, chromium and other resistant steels are used to offset corrosion despite their relatively high cost.

Faced with this problem, the emission spectrographer is often called upon to examine deposits in order to aid the engineers in finding the corroding metal piece. By determining the approximate percentage of the elements in a deposit, for instance, the spectrographer can frequently pin-point the exact alloy. From specification data, the engineers can then spot the failing part. In this fashion, Paul W. Ziegler, of Sun Oil's Marcus Hook Refinery Laboratory, was recently able to guess at the type of chrome steel responsible for a corrosion deposit. The engineers were then able to locate the bad part in a cracking tower. Mr. Ziegler also told of a novel example of the use of his spectrograph in connection with a new organic corrosion inhibitor. At the refinery, this inhibitor was found to be quite successful in preventing corrosion in a water tank. Normally, a colorimetric method would be used to check the water periodically for iron. Here, however, the organic inhibitor interferes with the colorimetric test and so a spectrographic check is used at regular intervals on the water.

The emission spectrograph is routinely used to check on metallic impurities in all stages of production. Crude oil, charge stock and residual fuels are analyzed for vanadium and nickel, especially because of the damage these elements can do. Vanadium is oxidized by heat either in a cracking tower or in a boiler to yield low melting vanadates or vanadium pentoxide. Despite the fact that the vanadium in the oil may be present in trace quantities, these products deposit rapidly. The deposits are extremely corrosive to metals and poisonous to catalysts. Another bad actor is arsenic which, as low as few ppb, will poison platinum catalysts. Catalysts, themselves, are checked for evidence of build-up of poisons. Consisting of platinum mixed in aluminum oxide, a single charge of a reforming catalyst may cost upwards of \$1 million. Accordingly, every precaution is taken to increase periods between regeneration or replacement.

The procedure for analyzing catalysts, used by H. E. Knauer and E. Wohlfarth, of Socony Mobil Research Laboratory in Paulsboro, N. J., makes use of powders. Catalysts are first ground and then mixed with a buffer in a fixed ratio for qualitative or semi-quantitative analysis. It is not always necessary to determine the exact percentages of tramp elements. Production engineers are more interested in making sure that they are kept below certain values.

At Tidewater, P. L. Work, like many other spectrographers in the petroleum industry, analyzes crude oils by first ashing the material and adding lithium carbonate as a matrix. Routine determinations of nickel, vanadium, iron, chromium and copper are made on all incoming crude oil. J. A. Kanehann and E. B. Childs of Socony Mobil Technical Service Laboratory in Brooklyn, N. Y. described a similar method for determining vanadium using silica and graphite as matrix buffers.

As part of their lubrication service program, some of the petroleum laboratories help the smaller railroads with their preventive maintenance programs (see SPEX SPEAKER, Vol. III, No. 2). Mr. Robert Hoffman, of Socony Mobil in Brook-

lyn, uses a powder method based on a matrix of lithium carbonate containing 1% indium oxide as an internal standard for such analyses.

Considering this large-scale use of the spectrograph by the railroads for checking wear, one natural question is whether the petroleum labs check the wear characteristics of various oils in automotive engines. Do different oils change wear rates on bearings and piston rings? The answer to the question is "yes" although specific data appear to be trade secrets. One laboratory reported that wear rates are studied by first diluting used oils 1:9 with mineral oil and then sparking with a rotating electrode technique. Another spectrographer stated that one of the essential tests on all experimental oils is a wear-rate study in which the spectrograph is used to obtain a good portion of the data. To some extent, however, this has been supplanted by tracer methods in which engine parts are made radioactive beforehand.

### X-Ray Spectroscopy

Many of the problems handled by emission may be solved equally well by x-ray spectroscopy. Although the former has a head start of several years, x-rays are being put to many routine uses. In one refinery, all determinations of TEL in gasoline will soon be switched to x-ray spectroscopy. Several refineries are controlling the content of additives to their oils through the determination of barium, calcium and zinc. The determination of chlorine and bromine in TEL; calcium and strontium in oil-bearing rocks; barium, titanium, zinc and other heavy metals in brines and other oil-field materials are promising applications of x-ray spectroscopy. It is also used to determine poisoning elements in catalysts as well as platinum, a catalytic element itself. In exploration studies, geologists are constantly searching for correlations between mineral formations and oil deposits. Both x-ray spectroscopy and diffraction are used here to determine crystal structure as well as the mineral elements present.

Earlier it was mentioned that corrosive sulphur is the most troublesome element in a refinery. Hundreds and even thousands of sulphur determinations per month are run. Faster,

less expensive analytical methods are constantly sought and here x-ray fluorescence seems quite promising.

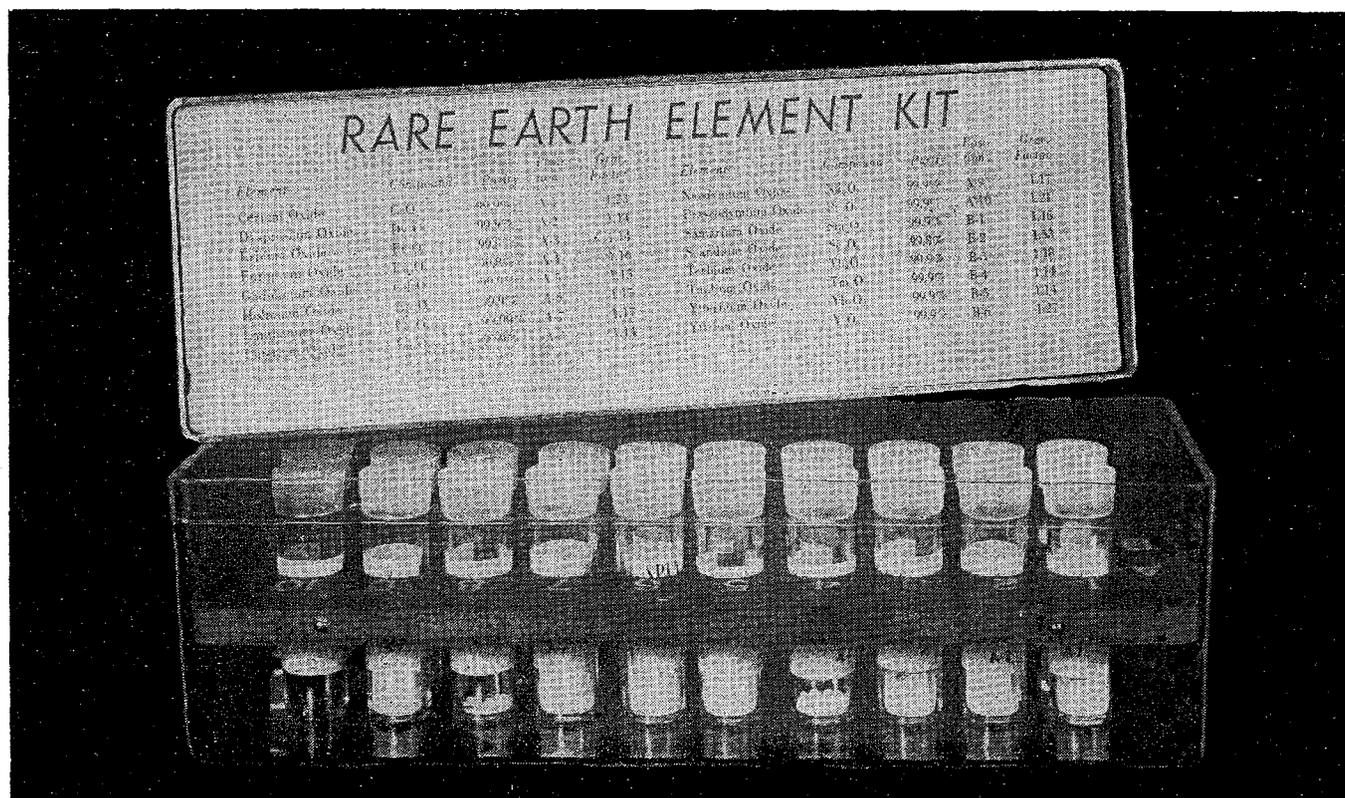
The x-ray problems involving sulphur are however, formidable. A light element, sulphur requires the use of a helium path for its x-ray detection. In addition, its compounds are broken down to elemental sulphur by an x-ray beam and thus precipitate out of a liquid sample. The effect is a constantly increasing intensity of sulphur radiation if an upside-down cell is used where the beam impinges on the bottom of a liquid sample through a Mylar window.

G. R. Clark and R. E. Hunt of the International Nickel Research Laboratory in Bayonne, N. J., have tackled the problem of sulphur determination in fuel oil by a rather clever scheme. Exactly five drops of the fuel oil are placed on a 1-1/2" diameter by 3/16" thick Whatman ashless filter tablet. After a waiting time of three minutes, during which the oil diffuses through the tablet, the x-ray beam is switched on. Using a NaCl crystal, a helium tunnel and a flow proportional counter, sensitivity down to about 0.02% sulphur is attained. Accuracy is ample for routine checks on No. 4 fuel oil as it is delivered to a metallurgical furnace. High sulphur in fuel oil, in addition to being corrosive to furnace parts, has been found to have other detrimental effects.

### Acknowledgments

The preparation of this article was made possible by the following scientists who so generously permitted interviews: *John Hansen* and *Charles Hodgkins* of Esso Research and Development Corporation in Linden, N. J.; *R. Pomatti* of Texaco in Beacon, N. Y.; *Paul W. Ziegler* of the Sun Oil Co., Marcus Hook, Pa.; *Henry Knauer* and *Ernest Wohlfarth* of Socony Mobil Research and Development Laboratory, Paulsboro, N. J.; *V. J. Tkac* and *Paul L. Work* of Tidewater Control Laboratories in Delaware City, Del.; *E. B. Childs* and *Robert Hoffman* of Socony Mobil Technical Service Laboratory in Brooklyn, N. Y.; *F. W. Porsche* and *T. C. Yao* of Standard Oil Company (Indiana) in Whiting; *G. R. Clark* and *R. E. Hunt*, International Nickel Co., Bayonne, N. J.

—A. J. MITTELDORF.



# NEW RARE EARTH STANDARDS

Now available are standards designed to determine the rare earths as well as scandium and yttrium semi-quantitatively. Similar to our popular standards for the determination of 43 of the more common elements, the rare earth standards are used in the same manner. For the analysis of a substance containing rare earths, it is diluted with spectrographic grade lithium carbonate by factors of 10 and 100. The diluted materials are then matched against the accurately prepared standards. The highest standard contains 0.50% of the sixteen elements; the three others 0.050%, 0.0050% and 0.00050% of the same elements.

The analytical method suggested uses a dc arc of about 6 amperes with a complete burn taking about 2-1/2 minutes. Unweighed samples are packed into a 3/16" dia. shallow, necked electrode (Spex 4004); the upper electrode is a pointed 1/8" rod (Spex 4019). A stepped sector or filter is recommended in order to obtain optimum registry of each line. The three-lens method of illumination recommended is the optical technique of focusing the light from the arc on an aperture which masks out the incandescent electrodes. This improves the line to background ratio. The SA#2 emulsion is used because of its moderate, uniform contrast and because its sensitivity extends to the 4500A region (for europium). A 10-micron slit with a spectrograph having a dispersion of at least 5A/mm is recommended.

Using the conditions above, the following table was obtained. It shows the analysis of a sample of xenotime:

## Spectrochemical Semi-Quantitative Analysis of Xenotime Sample Using Spex Rare Earth L Standards

Element	Line A	Actual Analysis %	Spex Analysis %	Detection Limit %
Cerium	4186.6*	2.57	5.0	.001
Dysprosium	4046.0	4.36	4.0	.0005
Erbium	3372.8	3.42	4.0	.0002
Europium	4594.0	0.10	0.1	.0005
Gadolinium	3422.5	1.65	2.0	.001
Holmium	3891.0	1.0	2.0	.0005
Lanthanum	3949.1	1.8	1.0	.0005
Lutetium	2615.4	0.38	0.3	.0005
Neodymium	4303.6	1.69	0.5	.005
Praseodymium	4225.3	0.46	0.2	.005
Samarium	3634.3	0.70	0.3	.005
Scandium	4023.7	—	—	.0005
Terbium	3509.2	0.54	0.5	.005
Thullium	3462.2	0.43	0.5	.0005
Ytterbium	3289.4	1.96	3.0	.0005
Yttrium	3242.3	35.8	30.	.0005

\*This line is in a cyanogen band. It was verified with a high-dispersion Order Sorter on an Ebert spectrograph.

The method, while showing great promise is not the last word. Determinations of several rare earth elements are not as close to the "actual" analysis as those obtained for the more common elements. We do, nevertheless, feel that the standards and the method are a good start, especially in view of the present sparsity of materials of known composition, standards and methods of analysis.

Rare Earth Spex Mix contains 16 elements at 5.28% concentration. This may be used to check the purity of a rare earth compound. The unknown is diluted with Rare Earth Spex Mix so that additions of 0.1%, 0.01% (and lower if desired) are made. A comparison between the diluted and the original unknown quickly tells the spectrographer the level of the elements in the latter. Rare Earth Spex Mix may also be used for the preparation of semi-quantitative standards in a desirable matrix.

- 1030 **Rare Earth Element Kit**, for spectrochemical quantitative, semi-quantitative and qualitative analysis. Contains quantities ranging from 100 mg to 2 g of 16 high-purity compounds of the following elements: Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sm, Sc, Tb, Tm, Yb and Y.  
Per Kit ..... \$50.00
- 1031 **Rare Earth Spex Mix**, contains the same elements as #1030, each element at exactly 5.28% concentration.  
Per 2 grams ..... \$36.00
- 1032 **Rare Earth L Standards**, semi-quantitative standards for the determination of rare earth elements in unknown materials. One standard contains 0.50% of the 16 elements listed under #1030; the others 0.050%, 0.0050% and 0.00050%.  
Per set of 4 standards, 2 grams each ..... \$49.00
- 4004 **High-purity Graphite Preformed Electrodes**, 3/16" dia., necked crater 3/32" deep.  
Per 100 ..... \$18.00
- 4019 **High-Purity Graphite Preformed Electrodes**, 1/8" dia. pointed upper.  
Per 100 ..... \$18.00
- 1005 **Lithium Carbonate**, spectrographic grade.  
Per 10 grams ..... \$ 6.00

## MEETING NOTICES

Ninth Annual Fisk University Infrared Institute, August 25-29. Meetings will be held at the University in Nashville, Tennessee.

Fifth Ottawa symposium of the Canadian Association for Applied Spectroscopy, September 15-17, 1958. The Symposium will be held in the Lecture Hall, Victoria Museum, Metcalfe and MacLeod Streets, Ottawa, Ontario, Canada. All inquiries should be directed to W. J. Wright, Program Committee, c/o Noranda Copper and Brass Limited, Post Office Box 1238, Place D'Armes, Montreal, Quebec, Canada.

Thirteenth Annual Meeting of the Society for Applied Spectroscopy, November 6 and 7. Meetings will be held at the Hotel New Yorker. For information about the exhibition of equipment, contact Norman Gardner, International Testing Laboratories, 580 Market St., Newark, N. J. Dr. Mabel Wilson of Air Reduction Research Laboratories, Murray Hill, N. J., should be contacted for submission of papers and program information.

## DON LANDON JOINS US

We are pleased to announce the appointment of Donald O. Landon as Director of Research. Don comes to us from Sperry Gyroscope where he was in charge of emission spectroscopy in the Materials Laboratory. His prior employers include Consolidated Testing Laboratory and Armour Research Foundation. In both places he worked as an associate of Art Mitteldorf. Don's extensive experience in trace element techniques, in instrumentation and in the analysis of materials having complex spectra will be invaluable in our continual development of new products.

## STILL MORE APPLICATIONS FOR THE MIXER/MILL AND WIG-L-BUG

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We've tried to keep our readers abreast of the unusual uses for our impact grinders, the Wig-L-Bug and the newer, larger capacity Mixer/Mill. Several novel applications and techniques have been reported recently. Ray Krammes, American Cyanamid in Bound Brook, N. J., reports that he uses the Wig-L-Bug to prepare water dispersions of dyes for electron microscopy. Small amounts of pigments are ground rapidly and uniformly using a stainless steel grinding vial.

We've already reported that some brittle metal sponges, such as platinum, are ground in plastic vials. Now Morris Slavin of Brookhaven National Laboratory and Arno Arrak of Belmont Smelting and Refining in Brooklyn have shown that high-purity bismuth may be similarly ground. This has permitted the incorporation of Spex Mix directly in bismuth pow-

## CORRECTION

In Vol. II, No. 2 of the SPEX SPEAKER, a calculation error has been pointed out in the article entitled "Semi-Quantitative Spectrochemical Analysis". On p. 2 column 2, l. 5 the sentence should read ". . . compounds divided into 43 . . ." Later, in par. 3, l. 17, the sentence should read, "It is made by mixing 0.100 weight units of Spex Mix with 1.24 weight units of graphite."

For those of you who may be concerned with your past analyses, please note that the actual instructions for using Spex Mix which appear on p. 3 under "Analysis of 'Pure' Materials" are given correctly. Errors were limited to the descriptive text.

der for subsequent analysis by a powder technique. Older techniques involved the extra step of dissolving the sample.

Wet grinding has proved very successful for reducing the particle size of many materials down to a few microns. Such powders are desirable for x-ray analysis, both diffraction and spectroscopic. Largely a function of the surface seen, x-ray intensity and reproducibility are increased as the particle size is reduced. Water is, of course, the best medium for preparing slurries. It has been used successfully on limonite and aluminum ores, open-hearth and blast furnace slags, silica and silica sand. Other liquids have been used where water is precluded. O. E. Brown, Manager of Marquette Cement's Research Laboratory in Chicago, suggested 1,1,1-trichloroethane for grinding cements which, of course, react with water.

William Kiley of Philips Electronics in Mt. Vernon, N. Y., had the problem of grinding samples of carnauba wax. Both hand and mechanical grinding proved futile: the chunks jumped out of the mortar. Using plastic jars and balls in the Mixer/Mill sufficient fines were obtained in 2 minutes for an x-ray diffraction sample.

## TYPICAL GRINDING RESULTS USING SPEX MIXER/MILL

MATERIAL	WEIGHT, g.	GRINDING METHOD	TIME, min.	RESULTS
Cement	20	Slurry with 1,1,1-trichloroethane; ceramic vial and 1 1/2" dia. ceramic ball	30	100% -300 mesh
Bauxite	3	Water slurry; tungsten carbide vial and one 1/2" ball	15	100% -300 mesh
Blast Furnace Slag	3	Water Slurry; tool steel grinding vial and three 1/2" dia. balls	20	100% -300 mesh
Open Hearth Slag	3	Water Slurry; tool steel grinding vial and three 1/2" dia. balls	20	100% -300 mesh
Sand	20	Water Slurry; high-alumina ceramic grinding vial and one 1/2" dia. ball	30	100% -300 mesh
Bismuth metal	5	Plastic jar with five 3/8" dia. plastic balls	20	100% -200 mesh
Limonite Ore	3	Water Slurry; tool steel grinding vial and three 1/2" dia. balls	30	100% -300 mesh
Zirconium carbide	15	Water Slurry; high alumina ceramic grinding vial and one 1/2" dia. ball	30	100% -300 mesh
Ferrous alloy (Hardness Rockwell C-30)	2	Tungsten carbide grinding vial with two 7/16" dia. balls	30	62% -300 mesh 83% -200 mesh
Tabular Alumina Balls	10	Tungsten Carbide grinding vial; three 7/16" dia. balls	30	100% -300 mesh
Carnauba Wax	5	Plastic jar; five 3/8" dia. balls	2	20% -300 mesh
Activated Carbon	10	Tool steel; two 1/2" dia. balls	10	90% -300 mesh
Copper shot	2	Tungsten carbide grinding vial	15	95% -300 mesh

# tricks of the trade

## SEIDEL EMULSION CALIBRATION KIT

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When plotted on ordinary log-log coordinate paper, photographic emulsion calibration curves are the typical S-shape figures known as H&D curves. For spectrographic computations, it is difficult to interpolate along the curved portions. The Seidel transformation, in which an arbitrary ordinate scale is substituted for the logarithmic scale, permits almost straight-line plots of calibration curves. A refinement of this technique was described in *Applied Spectroscopy*, Vol. 10, No. 1 (1956) by C. V. Wheeler, who showed that, when the preliminary curve is prepared on double Seidel coordinate paper, it, too, turns out to be almost a straight line. E. S. Hodge of Mellon Institute has gone another step forward by enlarging the graph paper to minimize measurement errors and by providing a scale which may be attached to the slider of a calculating board. With Hodge's technique, the Seidel calibration curve substitutes directly for ordinary log-log graph paper on the calculating board.

We are now supplying a kit containing 15 preliminary curve blanks in which the transmittances of two lines (or two steps) are both plotted on Seidel coordinates; 15 final calibration curve blanks in which the ordinate is given in the Seidel function and the abscissa in logs; Seidel coordinate strips for attaching to the slider of the calculating board; instructions for use.

1102 **Seidel Emulsion Calibration Kit**, as described above.  
Per Kit ..... \$8.00

## POLYETHYLENE GLOVES

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Impervious to practically all corrosive chemicals, lint-free and inexpensive enough to be considered expendable after a few wearings, polyethylene gloves will find a great many uses in your laboratory.

As pictured, the glove has separate compartments for the thumb and the forefinger. It may be used for either the left or right hand. A heat seal extends around the outside, making the glove watertight. Because it is so flexible, one size will accommodate any hand.

3906 **Polyethylene gloves**, as described above  
Per 12 pairs ..... \$2.50

## SPECTROGRAPHIC DETERMINATION OF URANIUM ISOTOPES

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A beautiful example of how far the emission spectrograph has come is some current work on uranium isotope determination. The resolution of several commercial spectrographs is so good that the closely-spaced isotope lines may be measured. In the past, even the parent lines of uranium could not be resolved on many instruments.

Neil Gordon reports that his laboratory regularly runs assays for uranium 235, 236 and 238 using an Ebert spectrograph. The eighth order of a 15,000 lpi grating is used on the reverse side of the grating. That is, the grating is rotated beyond the central image to the eighth order of the unblazed side. Here the intensity is greater than the normally used blazed side. For the analysis, a high-voltage ac arc serves as the source. The determination of uranium 234 is carried out by radio-chemistry techniques.

Further information may be obtained from Mr. Gordon at Westinghouse Atomic Power Department, P.O. Box 355, Pittsburgh 30, Pa. His laboratory analyzes uranium-bearing samples both for internal control and for outside customers.



BUREAU OF ANALYSED SAMPLES, LTD. ISSUES  
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So popular was the original set of low-alloy steel standards that incomplete sets as well as short ends were eagerly purchased when the full sets were depleted. They have now been re-issued in an improved form. Instead of 1/2" dia. rods, the new standards are in the form of blocks 1" x 1" x 1-1/2", equally suitable for x-ray or optical spectroscopy. To minimize matrix effects, some elements increase while others decrease in concentration so that the iron remainder in each standard is kept around 94%. The standards are especially valuable to the spectrographer who runs occasional analyses without prior knowledge of the alloy type. Steels in the AISI 1000, 2000, 3000, 4000, 6000 and 8000 series may be readily identified and analyzed quite closely. A composite table of mean values is printed inside the attractive box containing the standards.

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SPECTROGRAPHIC STANDARDS

S.S.No	Si %	Mn %	Ni %	Cr %	Mo %	V %	Cu %
1/1	0.43	1.54	1.24	0.53	1.50	0.64	0.53
2/1	0.27	0.34	2.25	0.42	1.06	0.23	0.20
3/1	0.65	0.84	1.01	0.99	0.65	0.50	0.38
4/1	0.21	0.26	3.28	0.28	0.23	0.15	0.34
5/1	0.15	0.15	4.99	0.20	0.31	0.22	0.25
6/1	0.23	1.00	0.21	2.35	0.53	0.18	0.17
7/1	0.10	0.55	0.27	2.95	0.17	0.12	0.13
8/1	0.95	0.40	0.55	1.30	0.79	0.35	0.15

Thus far the following elements have been standardized:

Chromium	0.2 - 3%	Nickel	0.2 - 5.0%
Copper	0.1 - 0.5%	Silicon	0.1 - 1%
Manganese	0.15 - 1.5%	Vanadium	0.1 - 0.6%
Molybdenum	0.2 - 1.5%		

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