

LABORATORY EQUIPMENT

FOR THE OIL AND FAT INDUSTRY

Authorities in this category of the industry contribute their views on the selection of equipment, new needs, and solution of problems for the benefit of Journal readers, who may wish to evaluate their own laboratories in the light of these thoughtful comments.

Helping the Customer Select Equipment

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40 KEEP ABREAST of modern and ever changing analytical procedures required today in industrial laboratory testing, control, and research the progressive laboratory supply house, too, must maintain an efficient research and development department with adequate laboratory facilities. There are two important functions for such a facility: the development of new, more precise apparatus to shorten the work of the analyst and the ability to take care of customer requirements, no matter what type of research, control, or testing is involved. Necessary is a well-equipped reference library with the latest technical publications, including the standard methods and analytical procedures published by recognized technical organizations such as A.O.C.S., A.S.T.M., A.O.A.C., A.C.S. and others with a continuous review of current literature to find changes in methods or apparatus specified. The library serves also as a reference for correspondents to provide customers with helpful information concerning specific uses of apparatus.

Space does not permit opportunity to review in detail the variety and types of laboratory equipment now available to industry. Emphasized and recommended whenever possible today are automatic instruments for analysis. Typical of these are the automatic titrator for measuring olefins and mercaptans quickly; the *beta* ray H/C meter which utilizes radioactive strontium 90 to measure in minutes the hydrogen-to-carbon ratios in liquid hydrocarbons; the direct-reading, infrared moisture balance for fast and accurate moisture measurements of a great variety of products; and electronic switches for automatic control of constant-temperature baths, ovens, and similar devices.

The supply house with a competent staff will recommend apparatus and instruments that will save the most for the laboratory worker in time and expense. It will, by following standard methods, supply a list of apparatus to perform the required tests most suitably and efficiently. Recommendation of equipment depends primarily upon the work the chemist has planned for his company and what he is trying to accomplish. The fully equipped laboratory with proper instrumentation will be determined by the chemist's own selection of methods he plans to follow. But the apparatus source can, from technical experience and previous installations, make helpful recommendation of items and even of methods.

SUGGESTED lists of apparatus used for laboratory operations are frequently prepared in advance to give immediate technical service when needed, and others are prepared on request. Typical is a list of laboratory apparatus for a soil testing laboratory based on simplified but accurate methods of analysis following the Association of Official Agricultural Chemists, American Potash Institute, and other similar published techniques. The grower who wishes to know the nutritional deficiencies of the soil will find a list such as this a valuable guide in the selection of laboratory equipment needed for determining fertility requirements of his soil. In the "Official and Tentative Methods" of the American Oil Chemists' Society such determinations as acid values, ash, chlorides, color, fatty acids, glycerol, iodine value, moisture, nitrogen, oil content, refractive index, specific gravity, and titer test are typical of what may be needed in another laboratory. Each test requires the use of specific apparatus.

Other similar but varied lists include apparatus and chemicals for the analysis of grain and stock feeds for moisture, crude fat, crude fiber, protein, and ash; for the qualitative and quantitative analysis of jams and jellies; for artificial breeding of cattle; for grading nonfat, drymilk solids; quality control of aviation gasoline, jet fuel, Diesel fuel, etc.; for laboratory sampling of coal and coke; for chemical analysis of steel, cast iron, open hearth steel, etc.; chemical analysis of Portland cement; and many more similar techniques.

An example of list making, copies¹ of which are available for the asking, is a 16-page list which summarizes all the commonly used methods for determining moisture with suggested equipment. The determination of moisture is common to a great many laboratory operations and is an extremely important test for the complete analyses of hundreds of raw materials, materials used in a manufacturing process, and materials needed for the finished product. In concise form this list describes oven drying methods including air, infrared, and vacuum ovens; the Karl Fischer method; the centrifuge method; the solvent distillation method and several others being currently used.

Helping the customer select equipment involves close contact with current analytical methods, it requires knowhow in preparation of suggested lists based on those methods, and it means the development of new and improved scientific devices to help shorten laboratory work and give more efficient plant control as progress is made to even greater automation in the laboratory of the future.

New Needs in Laboratory Equipment

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H^{ROM DISCUSSIONS with members of our laboratory staff the author has developed a list of potentially useful, but as yet unavailable, laboratory equipment. Many of the items mentioned here may have been fabricated by isolated individuals for their own private use, but to our knowledge these are not available as stock items from any laboratory-equipment supply company.}

One item of interest to those working with oilseeds would be a laboratory-scale steam cooker. Also for this same group is needed an instrument to measure rapidly and accurately the moisture content of oil seeds, extracted oil, and residual meal. Frequently it is necessary to know the official cup loss on an extracted oil sample. Usually the required 1–1.5 liters of oil is unavailable; consequently equipment for measuring loss on small samples of crude oil would be very helpful.

¹Pamphlet No. 2021, "A Summary of Methods and Suggested List of Equipment for the Determination of Moisture," Central Scientific Company, 1700 Irving Park road west, Chicago 13, Ill.

In the process of solvent extraction a modified Soxhlet flask is needed, so designed as to permit continuous introduction of fresh solvent and segregation of the extract. This relatively minor change would help avoid the heat damage often resulting from prolonged reflux of miscella. Frequently the process of continuous liquid-liquid extraction is called for. When the sample size is larger than that which can be handled by the Craig apparatus, as is usually the case, the laboratory worker must build his own extractor.

In connection with refining a heavy duty, variable-speed motor is required for stirring. None of those available at present meets both of the above requirements. Although many types of agitators can be purchased, all of these familiar to the author tend to produce a vortex. Impellers of the type available for commercial units cannot be purchased as stock items for the laboratory.

Sensitive, direct-reading, analytical balances are now a reality. Needed are direct-reading, torsion balances accurate to perhaps 0.1 g. Likewise less expensive analytical balances reading directly to about 5 milligrams would find a market.

Especially useful for chromatographic studies would be a continuous, recording refractometer. Also an inexpensive recording instrument used to obtain a continuous record of temperature, pressure, etc., would certainly be welcome.

N OUR LABORATORY WE have developed equipment which functions very well for steam deodorization. The accompanying sketches (2, 3, and 4) show the design which we have used. This, or something similar, could well be a standard item.

Everyone who has participated in flavor panels recognizes the need for an instrument to measure accurately the development of flavor reversion or rancidity in fats.

Very helpful would be equipment to measure the viscosity of small, oil samples. Also an apparatus for the cryoscopic measure of molecular weight would find application.

A unit for chilling small samples of shortening (5-10 lb.) is needed. This should be designed to permit control of the volume of gas introduced.

In connection with process control work a portable fume hood would permit handling of hazardous solvents in the plant.

There is always a need for large, stainless-steel flasks (12 liters or larger) with accompanying steel condensers.

The above constitutes a partial list of new laboratory equipment which should be made available to our industry.



Courtesy, Central Scientific Company

Fig. 1. This infrared moisture-balance provides fast, accurate, and reproducible moisture determinations on a direct-reading scale.

Perhaps the reader can think of many more items which have been overlooked.

Equipment Problems and How They Are Solved

ELDEN D. HALLER, Arthur H. Thomas Company, Philadelphia, Pennsylvania

THE INTELLIGENT CHOICE of laboratory equipment is a constant challenge to the oil chemist. Proper selection can be vital to the speeding up and expansion of research and control operations.

The challenge to the oil chemist charged with improving



Fig. 2-4. Shown above are sketches of design used for steam deodorization.

FAT STABILITY APPARATUS

Mercury Regulator—Electronic Relay High Operating Temperatures—95° to 115°C Air pre-heating prevents sample cooling

Designed by E. H. Sargent & Co. for use in the determination of relative stability or keeping quality of lards, fats and oils, based on the formation of peroxides and aldehydes in the process of oxidative decomposition.

Adopted as standard, company-wide equipment by principal packing firms. This improved apparatus is now offered with a highly sensitive and extremely reliable temperature regulating system employing an adjustable mercury thermoregulator and the Sargent electronic relaying system.

The apparatus consists of a thermostatically controlled bath to maintain the samples at operating temperature, a pre-heating and distribution system to condition and regulate air passing through the sample, and twenty aeration tubes.



The mineral oil heating bath is contained in a sheet metal tank and is heated by three electrical immersion heaters supplying, respectively, auxiliary power for rapid attainment of operating temperature, constant power to supply in part that heat normally lost through conduction and radiation, and intermittent heat to an extent determined by the thermoregulator. Oil circulation to ensure uniformity of temperature is accomplished by a centrifugal immersion pump. Operating temperature may be adjusted over the range of 95° to 115° C with a regulation of $\pm 0.1°$ C.

The air distribution system consists of a glass manifold suspended from the cover and surrounded by the heating medium. Outlet tubulatures extend through the cover to each aeration position and are connected by segments of Neoprene rubber tubing through capillary orifices standardized at 2.33 milliliters of air per second. Inlet to the manifold is through a onefourth inch diameter glass tube of which a forty inch section is immersed in the heating bath and which terminates in a tee connection at the cover.

Aeration tubes are 25x200 mm, Pyrex brand test tubes equipped with rubber stoppers carrying inlet and outlet tubes oriented for convenience in connection to the manifold and in organoleptic testing.

Length, 42 inches; width, $7\frac{1}{2}$ inches; total height, $14\frac{1}{4}$ "; maximum power consumption, 1100 watts.

SARGENT

SCIENTIFIC LABORATORY INSTRUMENTS . APPARATUS . SUPPLIES . CHEMICALS

E. H. SARGENT & COMPANY, 4647 W. FOSTER AVE., CHICAGO 30, ILLINOIS MICHIGAN DIVISION, 8560 WEST CHICAGO AVENUE, DETROIT 4, MICHIGAN SOUTHWESTERN DIVISION, 5915 PEELER STREET, DALLAS 35, TEXAS SOUTHEASTERN DIVISION, 3125 SEVENTH AVE., N., BIRMINGHAM 4, ALA. the position of his organization in a complicated and highly competitive oil and fat industry demands the best use of technical training and the availability of modern laboratory tools. Present and future needs for equipment must be considered, and recommendations given to management regarding the exact instrumentation required properly to control quality and to pursue research problems.

Fortunately there is little need for guess-work on the part of the laboratory director. Literature surveys will acquaint him with typical applications for currently available instrumentation. Recent issues of his trade journal will furnish information on products his competitors are developing and on raw materials he will have to work with as well as on the equipment used to develop and control these new materials. Exhibits and technical sessions will provide education on new tools, chemicals, and methods.

The next step in the selection of laboratory apparatus is the inspection and comparison of equipment. Demonstration of equipment to prove its usefulness in specific laboratory operations is also a logical step. Analysis of samples in the dealer's demonstration laboratory and discussions with specialists on specific analytical procedures and instruments can further round out the chemist's knowledge of the tools of his trade.

Oil chemists following the procedures just outlined are not likely to purchase an expensive spectrophotometer for a job which can never develop into or require more resolution and accuracy than a simple and inexpensive colorimeter. They will not use limited funds to buy automatic titrators for jobs that involve infrequent analysis which may be satisfactorily performed by a technician using a few dollars worth of glassware. They are not likely to ask for a balance having precision far in excess of their requirements. By the same token they are unlikely to waste the time of their chemists and limit their horizons by the selection of inadequate equipment.

The recent claims that saturated fats and oils increase the cholesterol build-up are of great interest to the laboratory. The gas chromatograph, high-speed centrifuge equipment, melting-point apparatus, mass spectrograph, etc., are indicated for this investigation work. The identification of chemical substances imparting odors and tastes to food is simplified by isolating the substances by gas chromatography. Announcements that cholesterol formation in the circulatory system and the apparent minimizing of arteriosclerosis through diet control should be of great interest to the oil and fat industry. The companies concerned with the unsaturated fats will stand to gain if the hypothesis advanced is substantiated.

The spectrophotometer is the preferred tool for evaluating cholesterol in the clinical and research laboratory. The determination of structure, configuration, and particle size, plus the qualitative identification and quantitative evaluations, may be accomplished for many hundreds of fats and fatty acid compounds by spectrophotometric means. Emphasis on the use of synthetic detergents makes the use of instruments especially desirable for research work involving the improvement of detergents and on lubricants which may use oils and fats as a major raw material.

Currently available electrophoresis equipment enables one to separate massive amounts (several ml. per hour) of many materials. Although to date most of these separations have involved proteins dispersed in aqueous media, one can predict the separation of other molecules, including many that may probably be classed as fats.

Publishes Conference Proceedings

THE PROCEEDINGS of the second National Industrial Research Conference, held April 24–25, 1957, in Chicago, Ill., have been published in a 57-page, paper-bound booklet by Armour Research Foundation of Illinois Institute of Technology, sponsor of the conference.

The theme was "Research for Profit," and among the published papers is "Making Waste Products Pay," by Victor Conquest, Armour and Company, Chicago.