

JET PROPULSION LABORATORY

LITERATURE SEARCH NO. 620 COPY NO. 6

TO W. F. Wilhite FROM D. Sweitzer DATE 7-28-64  
Kenton Bentley

EXT. 2882

SUBJECT Determination of Small Quantities of H<sub>2</sub>O

The subject request--C. A. references from 1907 to the present on indirect methods for quantitative measurement of less than 1% water at temperatures below 100°F--was broadened a trifle to include anything found that might be helpful in measuring moisture in the solid material, liquids or gases of the planets, and also to include an adequate background study of methods. The chemically indirect methods were picked up, e.g., reactions to produce hydrogen, methane, ethane, acetylene, etc., which in turn could be measured. The Karl Fischer method (not requested because of its already-known capabilities) was included, as well as various modifications of this method. Physical methods of measuring the quantity of H<sub>2</sub>O (or of the products of reactions) are also included. The very direct methods of weighing or taking volume measurements were only sampled. The book by Mitchell and Smith, Aquametry, is not referenced, but would be helpful in gaining background understanding of the subject.

Because you were in a great hurry for the references, duplicates were sent to you at the time the original search was made--about March 1st. However, since we too were rushed, the final preparation of this memo listing was put off until summer. I have picked up the references in the C. A. issues which have been published in the interim, and so a few of these will be new to you. The new ones are, for the most part, at the ends of the sections. Chemical Abstracts have been checked from January 1907 (Volume 1, Issue Number 1) through July 20, 1964 (Volume 61, Issue Number 2). A few Physics Abstracts references are also included.

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## General References

The Determination of Moisture. G. N. HUNTLY AND J. H. COSTE. *J. Soc. Chem. Ind.*, 32, 62 7; cf. *C. A.*, 7, 949. E. W. BOUGHTON.

1355, 1913 CA

Microchemistry. R. GOUBAU. *Natuurw. Tijdschr.* 8, 49-52(1926).—A short review. A moisture detn. method is described. B. J. C. VAN DER HOEVEN

1603, 1927 CA

Methods of the chemists of the United States Steel Corporation for the sampling and analysis of gases. W. D. BROWN, *et al.* *Carnegie Steel Co. Pamphlet*, 187 pp. (1927).—This little book, as its title suggests, represents the methods in use in the labs. of the various subsidiaries of the U. S. Steel Corp. The app. used, the reagents, the probable error of the work, the methods of sampling and the detn. of all possible constituents of industrial gases (flue gas, producer gas, coke-oven gas, blue gas, carburized water gas etc.), natural gas, mine air, commercial acetylene, butane and compressed O<sub>2</sub> are discussed in detail, to meet the requirements of the young chemist who has had little experience in gas analysis. Special treatment is given to the detn. of H<sub>2</sub>S, S in coke-oven gas, C<sub>2</sub>H<sub>2</sub>, HCN, (CN)<sub>2</sub>, light oil, benzene, naphthalene, tar, moisture, dust, heat-

ing value and sp. gr. The methods of computation are discussed; combinations of the metric and English systems of units are used. The pamphlet can be purchased for \$2 from the Bur. of Instruction, Carnegie Steel Co., Pittsburg, Pa. Some of the material in the book is not given in any other text. W. T. H.

3327, 1927 CA

Apparatus for determining the moisture content of materials. Stephen Stanworth and James Stanworth. *Brit.* 386,767, Jan. 26, 1933.

4141, 1933 CA

Device for estimating the water in solids, especially salts. I. G. Farbenind. A.-G. (Karl Ackermann and Albert Ernst, inventor). *Ger.* 590,826, Jan. 11, 1934 (Cl. 421.9.50).

3956<sup>2</sup>, 1934 CA

Rapid chemical analysis of alum, chalk and calcium carbonate mixtures. P. Christol and J. Fourcade. *Ann. chim. anal. chim. appl.* 16, 241-9(1934); cf. *C. A.* 28, 4163<sup>3</sup>.—A procedure is given for detg. H<sub>2</sub>O, CaCO<sub>3</sub>, free CaO, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Fe alum and insol. impurities and analytical results are given of synthetic mixts. to illustrate the accuracy of the results. W. T. H.

4827<sup>7</sup>, 1934 CA

Analysis of gases in carboys. Charles Maurice. *Ing. chim.* 20, 51 (1936).—The sampling of gas in carboys and the detn. of O, CO, CO<sub>2</sub>, H<sub>2</sub>S, As, SO<sub>2</sub>, SO<sub>3</sub> and H<sub>2</sub>O are discussed. Nine references. Ann Nicholson Hird

8067, 1936 CA

Some methods for the rapid determination of small amounts of moisture in salts. I. Velkhherts and S. Kats. *J. Chem. Ind. (U. S. S. R.)* 13, 1490-4(1936).—A crit. review. H. M. Leicester

2124<sup>o</sup>, 1937 CA

Rapid determination of moisture. Egbert Freyer. *Oil & Soap* 15, 236-9(1938).—See C. A. 32, 2780<sup>e</sup>. E. Scherubel

8218<sup>3</sup>, 1938 CA

Determination of the water content. E. Eckert and P. Wulff. *Beiheft Z. Ver. deut. Chem.* No. 39, 12 pp.; *Angew. Chem.* 53, 403-5(1940).—All methods of H<sub>2</sub>O detn. are discussed with the exception of H<sub>2</sub>O detn. in gases. The advantages and disadvantages of the various methods are presented. Karl Kammermeyer

53<sup>2</sup>, 1941 CA

Apparatus for determination of moisture in gases. M. S. Maslennikov. *Russ.* 58,490, Dec. 31, 1940. Construction of details.

845<sup>5</sup>, 1945 CA

Physical and chemical methods for moisture determination. D. W. Grover. *Chemistry & Industry* 1946, 362-5.—A review with 10 references. E. H.

54d, 1947 CA

The determination of water. Antonio Doadrio. *Farm. nueva (Madrid)* 12, 487-90(1947).—A review with 33 references. E. H.

40957, 1948 CA

Examination of used engine-lubricating oils. K. Hilfreich, J. C. McNicol, and L. Rosenfeld. *J. Inst. Petroleum* 34, 148-208(1948).—107 samples of used engine oils were collected for examn. in the labs. of the Motor Industry Research Association. Sixty-one were from gasoline engines, 39 from Diesels, and 7 were mixts. from Diesel and gasoline engines. Thirty of the engines had oil filters; 26 had air filters. The av. interval between crankcase drainings was 3950 mi. for the gasoline engines and 6350 mi. for the Diesels. The samples were taken from light and heavy trucks, mech. horses, and buses. For the light trucks much of the driving was of the stop-and-go type. The sludge-depositing properties of the oils tended to change as they waited in storage for analysis. Methods were selected and applied to the detn. of water and sediment, petr.-ether-insol. substances, benzene-insol., asphaltenes, sludge by hot filtration, undissolved sludge, spot test on filter paper for indicating the cleanness of an oil, ash content, constituents of the insol. matter, particle-size distribution of solid inorg. particles, light-extinction coeff., water content, diluent content, resin content by decolorizing clay, acidity, sapon. no., viscosity and viscosity index, and sp. gr. The data are tabulated...

41737 L, 1948 CA

Determination of dust in gas. A. H. Arbogast. *Iron Steel Eng.* 25, No. 10, 82-9(1948).—A discussion of methods for detg. dust in gas, including wet scrubbing and filtration methods. Test metering and moisture detns. are also described. H. Stoertz

972 b, 1947

Methods of estimating moisture. Alan H. Ward (Aynscombe Labs., Frange-over-Sands, Lancashire, Engl.). *J. Inst. Fuel* 24, 16-19(1951).—A discussion is given of the significance of moisture content, objects of moisture detn., and sampling methods, and a survey is presented of available methods for estg. moisture. R. W. Ryan

2827 L, 1951

Devices and methods for water determination in different substances. R. Cuneo (S.A. Buhler Freres, Milan, Italy). *Bull. école meunerie belge* 13, 134-43(1951).—Crit. review on the recent methods used in different countries. The thermobalance is also mentioned. P. J. v. d. L.

13892, 1952

Recent developments in gas analysis with special reference to fuel gases. M. P. Mendoza. *Bull. Brit. Coal Utilisation Research Assoc.* 14, 409-23(1950).—A review on analysis of gaseous mixts. covering sampling, chem. methods of analysis, micro- and semimicro methods, and phys. methods. 116 references. Nathan Berman

1737 a, 1952

How tests are made for liquefied petroleum gases. Harvey Menard (Signal Oil & Gas Co., Long Beach, Calif.). *Butane-Propane News* 14, No. 11, 91-2, 94, 96(1952).—Tests for vapor pressure, sp. gr., weathering, and water content of liquefied petroleum gases, as standardized by the Calif. Natural Gas Assoc., are described, and the significance of each is discussed. P. J. Wilson, Jr.

2922, 1953

Determination of water. Edm. Leclerc, Th. Pitance, and Mlle. Orban. *Industrie chim. belge* 17, 929-36(1952).—A review of many methods. Elisabeth Barabash

2496 L, 1953

Methods of determining water. Dieter Klamann. *Osterr. Chem.-Ztg.* 54, No. 11/12, 165-75(1953).—A crit. review. After a discussion of methods such as drying, absorption, distn., extn., and measurements of phys. properties, 19 procedures are discussed which are based on chem. reactions. 70 references. G. H. Gloss

5731 L, 1955

Methods of determination of moisture. D. I. Ryabchikov and V. K. Belyaeva. *Uspekhi Khim.* 24, 240-8(1955).—Review with 78 references. G. M. Kosolapoff

11483 C, 1955

Determination of moisture in gases. W. Roman and C. Akehurst (Petrochemicals Ltd., Manchester, Engl.). *Rev. fac. cienc., Univ. Lisboa*, Ser. 2B, 3, 5-22(1954)(in English); cf. *C.A.* 47, 6308d.—A review with 49 references.  
Ernest M. Goldstein

3157 f, 1956

Methods of water determination. Zygmunt Łada (Inst. General Chemistry, Warsaw). *Chem. Anal.* 1, 235-44 (1956).—A comprehensive review on water detn. methods (physical, physicochemical, chemical, and Fischer) is given. 46 references.  
Z. Kurtyka

7219 e, 1957

Analysis for industry. J. H. Thompson. *Ind. Chemist* 34, 451-3(1958).—The many existing methods for the detn. of water are discussed.  
J. C. Tallman

19673 h, 1958

Analytical methods for liquid oxygen. C. P. Smith (Union Carbide Corp., Tonawanda, N.Y.). *Proc. Cryogenic Eng. Conf., 4th, Cambridge, Mass.* 1958, 436-42.—Methods for assay, and for detn. of  $C_2H_2$ , C total and volatile, trace compds.,  $H_2O$ , and solids are reviewed.  
J. W. Givens

13870 a, 1959

Detection of suspended water in water-immiscible liquids. John R. Joyce, Keith R. Williams, and Donald Anson (to "Shell" Research Ltd.). *Brit.* 806,928, Jan. 7, 1959. See U.S. 2,844,025 (*C.A.* 53, 8952a).  
A. W. W.  
Indicator for water content of liquids. Ansul Chemical Co. *Brit.* 810,947, Mar. 25, 1959. See U.S. 2,836,974 (*C.A.* 53, 990b).  
A. W. W.

13896 b, 1959

Apparatus for determining the moisture content of fluid media. General Electric Co. *Brit.* 831,648, Mar. 30, 1960. The app. is esp. useful for detg. the moisture content of liquid insulating media for transformers and other elec. app. The media are mineral oils, chlorinated aromatic hydrocarbons, etc.  
Lester Steinbrecher

19047 b, 1960

Control of ore-agglomerating plants. Klöckner-Hüttenwerk Haspe Akt.-Ges. (by Bernhard Weilandt and Nikolaus Petrusch). *Ger.* 1,065,439, Sept. 17, 1959 (Cl. 18a). In the agglomeration of Fe oxide or carbonate ores low in S, the moisture in the ore and the amt. of heating gas are regulated by an app. measuring the  $H_2O$ , CO, and  $CO_2$  content of the exhaust gas. Any  $SO_2$  in the exhaust gas is removed by a Pb oxide filter before the exhaust gas enters the app. for measuring the  $H_2O$ , CO, and  $CO_2$  content.  
U. Benedict

8269 b, 1961

Automatic continuous measurement of moisture. J. Kertzman (Manufacturers Eng. & Equipment Corp., Hatboro, Pa.). *Ann. N.Y. Acad. Sci.* 91, 901-8(1961).—A review of the various methods suitable for monitoring of moisture content is given.  
Braham Norwick

20532 g, 1961

A portable electronic moisture detector for reinforced plastics. G. A. Gagne and J. O. Outwater (Univ. of Vermont, Burlington). *U.S. Dept. Com., Office Tech. Serv., AD 266,205*, 11 pp.(1961). From *U.S. Govt. Res. Rept.* 37(3), 148(1962).  
TCVD

10869 f, Vol. 60

Determination of the moisture content of bulk goods, and a device for carrying out this process. Gann Apparate- und Maschinenbauges m.b.H. (by Erich Rumplach). Ger. 1,135,215 (Cl. 42I), Aug. 23, 1962, Appl. July 25, 1957; 8 pp. CA

118649, 1962, V.57

Influence of chemical transformation during drying of food mixtures with respect to storability. I. Introduction, analytical methods, and description of the problem. J. Schormueller, W. Andraess, H. J. Lange, and K. H. Mueller (Tech. Univ., Berlin, Ger.). *Z. Lebensm.-Untersuch.-Forsch.* 117, 379-89(1962). Anal. methods used in studies of various changes in dried products (amino acid-sugar mixts., dried skim milk, dried soups) are presented for moisture, total acids, L(+)-glutamic acid, L(+)-lysine, L(+)-arginine, total and acid sol. amine acids, reducing sugars, sugars after inversion, glucose, reductone, 5-hydroxymethylfurfural (detection of beginning of the Maillard reaction), visual browning, and lycopene color.

M. M. Piskur

129632, 1962, V.57

A method for determining moisture content and dust content of gases. V. F. Maksimov and V. A. Bushmelev. *Nauchn.-Tekhn. Inform. Tsent. Inst. Nauchn.-Tekhn. Inform. Bumazhn. i Derzvoobrabat. Prom. Tsellyuzno-Bumazhn., Gidrolizn. i Lesokhim. Prom.* 1961, No. 10, 35-9. A review is given of the theoretical basis of a simplified method for detg. the moisture content and dust content of gases. An app. for taking gas samples is described and formulas are given for calcg. moisture and dust content. From *Ref. Zh., Khim.* 1962, Abstr. No. 131376. CA

2774h, 1963, V.58

A comparison of basic methods for moisture determination in seeds. Joe R. Hart and Calvin Golumbic (U.S. Dept. of Agr., Beltsville, Md.). *Proc. Intern. Seed Testing Assoc.* 27, 907-19 (1962). The vacuum-P<sub>2</sub>O<sub>5</sub> method was used for detg. the moisture content of seeds. In this method the seed samples, usually ground, are heated at 80° in a vacuum maintained at a pressure of ≤25 mm. The moisture is absorbed on P<sub>2</sub>O<sub>5</sub>, which is kept outside the oven. Results of moisture detns. on 74 samples of seeds by the vacuum-P<sub>2</sub>O<sub>5</sub>, toluene distn., air-oven, Karl Fischer, and new near-infrared spectrophotometric methods were compared. Results of the 4 methods gave values greater or less than the values obtained by the standard or Karl Fischer method. A pressure of >7.5 mm. may not be used in the vacuum-P<sub>2</sub>O<sub>5</sub> method. Nonaq. volatile matter is distd. from some seeds during vacuum drying. No single air-oven method is satisfactory for all seeds. Distn. methods give variable results.

J. W. Hamilton

5485a, 1963, V.59

Physico-chemical methods for moisture measurement. I. II. III. A. Pande (Shri Ram Inst. Ind. Res., Delhi, India). *Lab. Pract.* 12(5), 432-6; (7), 661-6; (8), 741-8(1963). 74 references. SNTT

12167a, 1963, V.59

Rapid determination of the water content [of clay products]. A. Webber. *Ziegelindustrie* 13(22), 781-3(1960). Various methods of detg. the water content of clay products are described. The gravimetric method is very accurate, but is time-consuming. The rapid method based on the reaction between CaC<sub>2</sub> and water gives results within several min. The Brabend equipment based on the principle of heating and drying in moving air, and a

Cont.

14576a, 1963, V.59

method developed at the Freiberg Mining Academy based on measurement of the elec. cond. of the clay product, are described. A final method is given in which the sample is heated in an autoclave and the vapor pressure measured. From *Bldg. Sci. Abstr.* 34(4), Abstr. No. 582(1961). TCCB

14576 h, 1963, V.51

Field methods for determining the water content of Titan II propellants. Ralph R. Liberto (Bell Aerospace Corp., Buffalo, N.Y.). *U.S. Dept. Com., Office Tech. Serv., AD 275,537*, 44 pp. (1962). Several anal. procedures for detg. water in Titan II propellants are evaluated. These are  $N_2O_4$  as the oxidizer and a nominal 50/50 blend of unsym. dimethylhydrazine and  $N_2H_4$  as the fuel. For the 50/50 fuel blend, a gas chromatography method, a pressure bomb method, a near-infrared spectrophotometric method, and an elec. cond. method were considered. The elec. cond. method is most promising. For the  $N_2O_4$ , a colorimetric method, a pressure bomb method, and an elec. cond. method were considered. Again, the elec. cond. method is most promising. 12 references. RCVD

13353 g, 1963, V.59

Recent developments in adsorption drying. S. A. Gregory. *Ind. Chemist* 36, 479-84(1960).—Selection of adsorbents, their cyclic stability, methods of reactivation, and the design of equipment are discussed. Recent developments in the detn. of moisture in gases and liquids also are discussed. J. C. Tallman

3127 f, 1961

Synthetic cryolite analysis. F. J. Frere. *Ind. Eng. Chem., Anal. Ed.* 6, 124-5(1934); cf. *C. A.* 27, 928.—Synthetic cryolite differs from the native product in not contg. the various constituents in definite proportions. In the hope of eventually establishing a satisfactory procedure for the complete analysis, expts. were performed to det. the soly. of natural cryolite in aq. NaF solns. It was found that no cryolite dissolves if the soln. contains 0.1 g. NaF in 100 cc. If, therefore, a synthetic cryolite contains NaF in excess of that required to form  $Na_3AlF_6$ , the excess NaF will dissolve in the dil. NaF soln. and can be detd. by the method already described. The procedure fails if sol. Al salt is present. Expts. are also described in which known methods for detg. Na and  $H_2O$  were tested. W. T. H.

2643<sup>g</sup>, 1934



Methods used in an industrial laboratory for the estimation of water. A. S. Johnson. *Chemistry & Industry* 1949, 511-14.—The Brabender moisture tester, the Karl Fischer procedure (*C.A.* 29, 6532<sup>9</sup>), the effect on the f.p. of PhOH, and the Dean-Stark method (*C.A.* 14, 2145) have all given satisfaction in the labs. of Bakelite Ltd.  
E. R. Rushton

83092, 1949

The determination of water. Antonio Doadrio. *Farm. nueva* (Madrid) 12, 487-90(1947).—A review with 33 references.  
E. H.

40959, 1948

Influence of the moisture content of air on determination of water. L. Pap. *Z. ges. Getreide-Mühlen-Bäckereiw.* 21, 149-53(1934).—See *C. A.* 29, 2884<sup>9</sup>.  
E. J. C.

5382<sup>4</sup>, 1935

Apparatus for determination of moisture content of solids and the sorption of gases and vapors by solids at elevated temperatures. WARD E. KUENTZEL. *J. Am. Chem. Soc.* 51, 3560-1(1929).—The app was designed for the detn. of H<sub>2</sub>O vapor absorbed by granular solids from steam-air mixts. at temps. from 100° to 450°. J. H. M.

992, 1930

Moisture in technical gases. II. Application of moisture calculations. FRIEDRICH LÜTH. *Arch. Eisenhüttenw.* 4, 185-92(1930); cf. *C. A.* 24, 2814.—After a discussion of the practical application of moisture calcs., various methods for measuring the moisture content of gases are described, among them the wet and dry bulb method, the pressure-temp. method, direct weight method and Haar hygrometer.  
H. STOERTZ

1924, 1931

The determination of water in hydrated salts by means of the nickel-crucible radiator. WILLIAM M. THORNTON, JR., REUBEN ROSEMAN AND MEYER NEISHLOSS. *J. Chem. Education* 9, 1645-7(1932).  
E. H.

5033, 1932

Procedure for determining water content in wheat plants. P. D. Uglov. *Tr. po Prikladn. Botan., Genet. i Seleksii* 35(2), 80-4(1963). A study was made of changes in the water content (in respect to development phase and leaf level) in the stems and leaves of various varieties of soft and hard spring wheat under conditions of sufficient soil moisture, and during a dry period, in field expts. (28 specimens) and hothouse expts. (4 specimens) in the Tselinnyi Krai. The stems contained more H<sub>2</sub>O than leaves during the shooting and earing phases. This was also the case under arid conditions in spite of the overall decrease in the water content of tissue. With sufficient soil moisture H<sub>2</sub>O content decreased from the lower leaf levels to the higher. As soil moisture decreased, max. H<sub>2</sub>O content moved toward the higher leaf levels. For the most accurate reflection of the H<sub>2</sub>O content of tissue of spring wheat leaf samples must be taken from several levels of each plant. From *Ref. Zh., Biol.* 1963, Abstr. No. 22643.  
MYRD

14828h, Vol. 60

Investigation of manganese dioxides. I. Water content. Aladar Tvarusko (Elec. Storage Battery Co., Yardley, Pa.). *J. Electrochem. Soc.* 111(2), 125-31(1964). The water content of natural ores, electrolytic, chem., and synthetic hydrous MnO<sub>2</sub> samples, equilibrated in an atm. of 75% relative humidity, was detd. at 110, 235, and 400°. Total water varied from 1% for a highly cryst.  $\beta$ -MnO<sub>2</sub> to 20% for synthetic hydrous MnO<sub>2</sub>. The pH of the MnO<sub>2</sub> samples increased with increasing temp., except for natural ores and their derivs. which showed a decrease in pH at 235°. The degree of resorption of water vapor was dependent on the drying temp. and decreased with increasing temp. RCJV

6509B, Vol. 60

Flask buret for microchemical titrations. W. Ihn and H. Steininger (Inst. Mikrobiol. Expt. Therap., Jena, Ger.). *Chem. Tech. (Berlin)* 15(11), 691-2(1963). The buret (vol. 5 ml. or less) can mech. record changes of up to 0.02% of the vol. of liquid in the flask. It is esp. suitable for the estn. of non-aq. liquids, e.g., alcs. and pyridines. Dorothy M. Goode

7426E, Vol. 60

A technique for the determination of the weight and water content of minute tissue specimens. E. Nelson Bowser, Walter J. Henderson, and Gerald A. Williams (V. A. West Side Hosp., Chicago). *Microchem. J.* 7, 442-7(1963). A technique is described whereby the wt. and H<sub>2</sub>O content of minute samples of tissues can be detd. accurately and conveniently with inexpensive equipment. A comparison of the tissue H<sub>2</sub>O content detn. with this technique with a conventional technique that uses much larger samples of muscle tissue and with previously reported values for muscle, liver, and kidney tissues indicated very good agreement. With this technique it was possible to det. the wt. and H<sub>2</sub>O content of the rat parathyroid gland. V. N. Gupta

12356E, Vol. 60

Investigative methods for powdered milk. Heinz Hoffer. *Milchwissensch. Ber.* 12(3/4), 263-91(1962). Standard methods are described for detg. the H<sub>2</sub>O, fat, and acid contents, soly., and purity of powd. milk. Martin Jacobson

12583E, Vol. 60

The determination of carbon in raw meal and cement. A. Sassenscheidt. *Zement-Kalk-Gips* 13(49), 23-6(1960). A novel and rapid method is given for the detn. of C, H<sub>2</sub>O, and CO<sub>2</sub> in cement, its raw materials, and building materials. Where formerly 3 sep. operations were required for the detn., this new method needs only one single anal. operation. From CZ 1961(8), 2753. MR CR

117319, Vol. 60

Development of analytical methods in the gas industry. M. Duchemin. *Dechema Monograph* 48(835-858), 155-72(1963) (in French). Modern anal. methods have been introduced in the gas industry. The utilization of spectrography, polarography, and chromatography for quality control and for the solution of many problems of the coke-oven gas industry is discussed. Special methods and instruments are used for the detn. of dust, H<sub>2</sub>O, and conditioning agents. Walter Oppenheimer

495a, Vol. 61

## Miscellaneous Reactions to Determine Water

Determination of water in hydrocarbon gases. Harry Levin, Karl Uhrig, and F. M. Roberts. *Ind. Eng. Chem., Anal. Ed.* 17, 212-15(1945).—Detailed directions are given for detg. H<sub>2</sub>O in normally gaseous hydrocarbons. In contact with cold, dehydrated Me<sub>2</sub>CO, the H<sub>2</sub>O is absorbed and when the hydrated Me<sub>2</sub>CO comes in contact with a soln. of AcCl in toluene, HCl is liberated. Then the excess AcCl is decompd. by treatment with EtOH and the soln. of HCl is titrated with 0.1 N NaOH to a phenolphthalein end point. To remove all H<sub>2</sub>O from the Me<sub>2</sub>CO, allow the latter to stand over CaCl<sub>2</sub> for several days. Filter into a dry bottle, shake with an excess of AcCl in pyridine, esterify the unconsumed AcCl by shaking with AmOH and then distil fractionally. A suitable app. is shown and described and typical results are given for com. butane, propane, butadiene, isobutane, and isopentane mixts. and plant gas. In the 15 analyses cited, 8-32 mg. of H<sub>2</sub>O was found present and satisfactory values were obtained with gases contg. 0.001-0.009% H<sub>2</sub>O by wt.

W. T. Hall

2268', 1945

Polarimetric method for the determination of water in acetic acid. Gerrit Toennies and Margaret Elliott. *J. Am. Chem. Soc.* 59, 902-6(1937).—The H<sub>2</sub>O present in a sample of AcOH can be detd. by allowing it to react with a known quantity of Ac<sub>2</sub>O and then detg. the excess of anhydride by the decrease in optical rotation caused by its reaction with *d*-camphoric acid; this reacts with Ac<sub>2</sub>O to form *d*-camphoric anhydride. Both reactions are catalyzed strongly by low concns. of strong acids. By this method from 0.1 to 0.6% water in AcOH can be detd. within 0.008%.

W. T. H.

6581<sup>2</sup>, 1937

Determination of water in the presence of alcohols, aldehydes and acetals. JOHN ROSS. *J. Soc. Chem. Ind.* 51, 121-2T(1932).—Weigh out 7.00 g. of benzoic anhydride into a clean, dry Carius tube, 35 cm. long and about 2 mm. in diam. Add 0.500 g. of the org. liquid, seal the tube and heat 12 hrs. at 110-20°. Dissolve the contents of the opened tube in 10-20 cc. of ether, transfer to an Erlenmeyer flask, cool with ice water and titrate the benzoic acid formed with N NaOH with phenolphthalein as indicator, shaking vigorously during the titration. Now connect the flask with a reflux condenser and, with no water running in the condenser mantle, distil off the ether. Then make up the vol. to about 50 cc. with distd. water and reflux vigorously for 4-6 hrs. with efficient condensation. This accomplishes the hydrolysis of the excess anhydride. Cool, wash down the sides of the condenser with ether and again titrate with NaOH. Remove the ether by heating on the water bath and on cooling in ice water the methylene dibenzoate is formed if any HCHO is present. Filter, wash with water, then with cold, 50% alc., dry at 100° and weigh. Finally titrate a sep. sample of the original liquid

to det. its acid value, and run a blank expt. with the benzoic acid alone. If no highly volatile constituent is present, the procedure can be simplified somewhat and the original heating in the Carius tube avoided. The calcn. is based on the following assumptions: Bz<sub>2</sub>O reacts with H<sub>2</sub>O to form 2BzOH. With alc. the reaction Bz<sub>2</sub>O + ROH = BzOH + BzOR takes place after the BzOH formed is titrated, the excess Bz<sub>2</sub>O is hydrolyzed by boiling with water and more BzOH formed. HCHO reacts with Bz<sub>2</sub>O to form methylene dibenzoate with 60% yield. Other aldehydes do not react under the prevailing conditions. Of acetals, methylal does not react with Bz<sub>2</sub>O or BzOH during the expt. but acetal does. If acetal is present, therefore, the results for alc. will be high and for H<sub>2</sub>O low. Aldehydes dissolved in alcs. form hemiacetal and the soln. behaves in this analysis as a mixt. of alc. and aldehyde. Aliphatic acids and esters are likely to cause slight error.

W. T. H.

2939, 1932

Photoelectric determination of the moisture content of gases, especially sulfur trioxide. Société anon. des manufactures des glaces et produits chimiques de Saint-Gobain, Chauny & Cirey (Claude M. Cherrier, inventor). *Ger. 951,315*, Oct. 25, 1956 (Cl. 42i, 19<sub>01</sub>). The moisture content of colored or colorless gases, particularly SO<sub>2</sub>, is detd. by passing the gas through oleum of const. titer and by photoelectrically measuring the light diffusion of the clouds resulting therefrom. The gas is passed through a packed column from the bottom toward the top. Oleum is dropped in from above and makes a cloud with the wet gas, which passes through an analysis tube. Liselott Johannsen

12098 d, 1959

Apparatus for the microanalysis of gases. T. Carlton Sutton. *J. Sci. Instruments* 15, 133-5(1938).—A capillary tube 0.5 mm. in diam. and 2 ft. long with a widened (4 mm. diam.) Hg submerged end as reaction chamber is described. A small storage glass vessel as well as accessories for holding solid reagents, heating and sparking to permit all the ordinary macro-manipulations are included. The app. is "constant pressure," uses 0.1 to 0.3 cc. (S. T. P.) and has a consistency of 1 in 400. H. A. F.

4386<sup>3</sup>, 1938

Simple estimations of water. I. A new method for determining water in ethyl alcohol. Tomofumi Kimura. *Ann. Repts. Takeda Research Lab.* 11, 61-8(1952).—A mixt. of 1Q ml. each of alc. and CCl<sub>4</sub> is titrated at 25° with water from a microburet until turbidity results. Let  $X$  be the ml. required, then the % H<sub>2</sub>O (by vol.) =  $[(2.03 - X)100]/10$ , or  $\{[2.03 - 0.03(25 - t^{\circ}) - X]100\}/10$ , when the temp. is < 25°; or  $\{[2.03 + 0.03(t^{\circ} - 25) - X]100\}/10$  when the temp. is > 25°. II. A new method for estimation of water in pyridine. *Ibid.* 68-73.—Similarly, the H<sub>2</sub>O in C<sub>2</sub>H<sub>5</sub>N =  $[(1.051 - X)100]/10$  when estd. at 25°; equations for temps. > or < 25° are given. K. Kitsuta

5844 i, 1953

Determination of traces of water vapor in gases. Frank

E. Harris and Leonard K. Nash (Harvard Univ.). *Anal. Chem.* 23, 736-9(1951). The need for a general method for the detn. of traces of H<sub>2</sub>O in condensible gases, particularly hydrocarbons, has become acute. The proposed method consists in the increase in temp. in a matrix gas produced by the action of H<sub>2</sub>O on solid CaH<sub>2</sub>. The app. is a *thermistor bridge* for the measurement of the temp. rise. The method can be used for detg. 0.001 to 0.1 vol. % of H<sub>2</sub>O vapor. It is not affected by wide and rapid fluctuations in the compn. of the matrix gas, and has a sensitivity of 0.0005 vol. % with a reproducibility of 0.0005 vol. % of H<sub>2</sub>O vapor. A satisfactory arrangement can be used for the semicontinuous analysis of H<sub>2</sub>O content of streaming industrial gases. Kenneth H. Slagle

8403 i, 1951

$\gamma$ -Radiolysis of water vapor. J. H. Baxendale and G. P. Gilbert (Univ. Manchester, Engl.). *Discussions Faraday Soc.* No. 36, 186-92(1964); cf. *CA* 57, 4220b. Expts. at 116° showed that the values of  $G(H_2)$  increase to a const. value of  $8.0 \pm 0.7$  when MeOH, EtOH, cyclohexane, and ether are added to the H<sub>2</sub>O vapor. This const. value gives the value of  $G_H$  (where  $G_H = H$  atom yield) from H<sub>2</sub>O vapor in these conditions. The addn. of O to the system MeOH + H<sub>2</sub>O vapor decreased  $G(H_2)$  and increased  $G(CH_2O)$ . The latter has a value of 8.5 at high O concn. and is a measure of  $G_{OH}$ . BGJN

12803g, Vol. 60

A spectrophotometric method for the determination of water. Byron L. Ferguson and Nancy M. Coulter (Valparaiso Univ., Valparaiso, Indiana). *Proc. Indiana Acad. Sci.* 63, 124-6(1953); cf. *C.A.* 43, 8950h.—The change in color of Co-EtOH from blue to pink by adding H<sub>2</sub>O provides a means of detg. the adsorbed H<sub>2</sub>O if the Co concn. is maintained at 300 p.p.m., the temp. is const. to 0.1°, and the sample is insol. in abs. EtOH. A 1-5 g. sample of the substance contg. adsorbed H<sub>2</sub>O is weighed into 50 ml. abs. EtOH, the mixt. shaken vigorously for 5-10 min., the suspended material allowed to settle, an aliquot portion of the clear supernatant liquid pipetted into a 50-ml. volumetric flask, stock Co-EtOH soln. added to provide 300 p.p.m., the flask filled with abs. EtOH, the temp. adjusted to 25°, and transmittancy of the soln. measured at 671 m $\mu$  against abs. EtOH and the amt. of H<sub>2</sub>O present detd. by referring to a standard H<sub>2</sub>O-EtOH curve. When these conditions are observed, this spectrophotometric method can be substituted for the Karl Fischer method. R. E. Dunbar

771 a 1955

Pressure titration. Herbert K. Wiese (to Standard Oil Development Co.). U.S. 2,583,454, Jan. 22, 1952. Contaminants in small quantities in liquid industrial samples, are detd. by feeding a measured quantity of colored reagent into the titration zone, admitting a measured quantity of the normally gaseous material into the titration zone under super atm. pressure to maintain the material as a liquid and to decolorize the reagent. The method can be used to det. H<sub>2</sub>O in solid substances, and also for substances such as HCl, SO<sub>2</sub>, carboxylic acid, unsatd. hydrocarbons, such as isobutylene and isoprene, alcs., ethers, esters, and alkyl halides. Harry A. Goldstein

4428 f, 1952

Colorimetric method for determining the water vapor content in fuel gases, utilizing the Evelyn colorimeter. R. J. Pfister and D. J. Kerley. *ASTM Bull.* 127, 17-22(1944).—The Evelyn colorimeter (manufd. by the Rubicon Co., Philadelphia, Pa.) when used with the Evelyn color filter No. 660 which transmits light from 635 to 720 m $\mu$  has been found satisfactory for the detn. of water vapor in fuel gases. A metered vol. of gas contg. water vapor is passed through a tube chilled to -70° with dry ice and acetone until about 1/4 g. water is sepd. Condensed volatile hydrocarbons are allowed to weather off. To the water is added about 25 ml. indicator soln. contg. 10 g. CoBr<sub>2</sub> per l. and the container agitated until soln. of the water is complete. Percentage transmission of light through the soln. with the No. 660 filter is detd. The gals. of water per m. cu. ft. of gas can then be read from calibrated curves provided 10 cu. ft. of gas was metered through the moisture condenser. The detn. of moisture content can be reproduced within 3%. H. E. Messmore

3452<sup>3</sup> 1944

Water determination in hydrocarbon streams. John A. Favre and Dan E. Smith (to Phillips Petroleum Co.). U.S. 3,118,735 (Cl. 23-230), Jan. 21, 1964, Appl. June 8, 1959; 4 pp. Minor amts. of H<sub>2</sub>O, as low as 2 p.p.m., are detd. in a hydrocarbon stream (e.g. 1-butene) by passing the gaseous stream over CaH<sub>2</sub>, CaC<sub>2</sub>, or RNCO to convert the H<sub>2</sub>O to a mixt. of H, C<sub>2</sub>H<sub>2</sub>, and CO<sub>2</sub>, which is then analyzed by gas chromatography with 2 columns in series of Linde 5A mol. sieve (5 A. pores to absorb normal hydrocarbons) and a thermal-cond. detector.

Kenneth Drake

10450 h, Vol. 60

Determination of small amounts of water in certain polar organic solvents. A. I. Cherkosov and L. V. Cherkosova. *Materialy XXII [Dvadtsat Vtoroi] Nauchn. Konf. Saratovsk. Gos. Ped. Inst., Fak. Estestvozh., Fiz., Matem., Saratov, Sb. 1961, 100-6.* A photometric method is proposed for detg. small amts. of H<sub>2</sub>O in polar org. solvents which are highly protophilic (ketones, alcs., etc.). The method is based on a conjugated acid-base reaction of hexanitrohydrazobenzene (I) with the solvent; H<sub>2</sub>O has an ion-forming effect on the reagent and an H bond is formed between the H<sub>2</sub>O molecules and the hydrazo group atoms of the reagent, causing a red color. When the reaction mixt. is considerably dild. with H<sub>2</sub>O, the reaction slowly proceeds further and a H bond forms with the second N atom in the hydrazo group; this is accompanied by a blue color. With alcs. only the red color appears. An H<sub>2</sub>O concn. of 0.2-5.0 vol. % in a solvent can be detd. by this method. To det. a calibration curve, 3 ml. of a solvent contg. 0.5-3.0 vol. % H<sub>2</sub>O was added to 6 samples, followed by 3 ml. reagent (0.23 g. I in 100 ml. org. liquid), and the samples were then subjected to photometry at 533 m $\mu$  in a 1-cm. cuvette. The H<sub>2</sub>O content in the test solvent was detd. under the same conditions. The error is 0.05%, and the detn. takes 3-5 min. From *Ref. Zh., Khim.* 1962, Abstr. No. 20D148. MVRK

26 C, 1963, U. 59

A conductometric study of some reactions of boron trifluoride etherate. James L. Hall, John A. Gibson, Jr., Harold O. Phillips, and Paul R. Wilkinson (West Virginia Univ., Morgantown). *Proc. West Va. Acad. Sci.* 34, 62 (1962). F<sub>3</sub>BOEt<sub>2</sub> (I) was titrated directly and also in a no. of solvents with Et<sub>3</sub>N and other reagents. The ionic species formed could be followed by Lewis bases. I could be used to det. H<sub>2</sub>O in Et<sub>2</sub>O and MeCOEt, but not in MeOH, EtOH, or CHCl<sub>3</sub>. David T. Sorensen

11934 h, 1963, U. 58

Determination of water in several organic liquids with cobalt(II) bromide. Zymunt Łada (Inst. Allgemeine Chem., Warsaw). *Acta Chim. Acad. Sci. Hung.* 28, 217-21 (1961) (in German).—Org. liquids in which CoBr<sub>2</sub>·2H<sub>2</sub>O is insol. may be analyzed for 3-6 mg. H<sub>2</sub>O with an error <10%. A sample is dried at 120°, and 10-15 ml. of satd. CoBr<sub>2</sub> in dry CHCl<sub>3</sub> is added. After 20-30 min., the soln. is filtered, the pptd. CoBr<sub>2</sub>·2H<sub>2</sub>O is dissolved in H<sub>2</sub>O, and dild. to 100 ml. A portion of this soln. is dild. to 70 ml., and 1 ml. of 2N HCl and then a known amt. of 0.01M Complexone III at pH 10 are added. The excess Complexone III is back-titrated with 0.01M ZnCl<sub>2</sub> in the presence of Eriochrome Black T. The method was tested with CHCl<sub>3</sub>, Me<sub>2</sub>CO, salicylaldehyde, propionaldehyde, and benzaldehyde. In MeOH there is no ppt. and in cinnamaldehyde the ppt. is too fine to filter. M. E. Grenoble

21961 e, 1961

A new method for determining moisture in organic solvents. K. F. Jahr and J. Fuchs (Freie Univ., Berlin). *Z. Naturforsch.* 14b, 471-2 (1959).—Small amts. of water can be detd. by treatment with a soln. made by dissolving the *tert*-butyl ester of orthovanadic acid, (C<sub>4</sub>H<sub>9</sub>)<sub>3</sub>VO<sub>4</sub>, in benzene and adding an equal vol. of CHCl<sub>3</sub> satd. with NH<sub>3</sub>. The resulting ppt. of (NH<sub>4</sub>)<sub>2</sub>V<sub>2</sub>O<sub>7</sub> is filtered, washed with dry ether, dissolved in dil. H<sub>2</sub>SO<sub>4</sub>, and titrated with FeCl<sub>2</sub>. The method is applicable to hydrocarbons, alkyl halides, ketones, and esters and to such alcs. as do not dissolve the polyvanadate. It is not suitable for MeOH or glycerol. Otto H. Johnson

4251 a, 1960

Coulometric method for the determination of traces of water in liquid ammonia. W. C. Klingelhofer (Allied Chem. Corp., Hopewell, Va.). *Anal. Chem.* 34, 1751-3(1962). The electro-generation of K in liquid  $\text{NH}_3$  is used to det.  $\leq 100$  p.p.m.  $\text{H}_2\text{O}$ . A special cell holds 8.5 ml. sample in the cathode chamber. The sample of liquid  $\text{NH}_3$  is passed into the chamber through a KCl tube, which serves as a source of  $\text{K}^+$ . The end point is detd. by an increase in the electrical cond. of the soln. and this automatically terminates the generating current. Good results also are obtained by using the blue color of the excess  $\text{K}^+$  as the end point. Photoelec. end points were poor owing to suspended KOH. The results must be cor. for the displacement of sample by liberated H. A Pt cathode and an isolated C anode are used.

John W. Miller

1911 d, 1963, V. 58

Volumetric determination of oxygen in organic compounds by the hydrogenation method. S. Mlinko (Hung. Acad. Sci., Budapest). *Mikrochim. Acta* 1961, 833-53(in German). The ter Meulen method (CA 17, 1934) was studied with particular emphasis on the elimination of some sources of error. The incomplete redn. of  $\text{CO}_2$  and the "fatigue phenomenon" of the Ni-catalyst are considered to be unrelated. The incomplete conversion of  $\text{CO}_2$  is attributed to unfavorable compn. of the starting gas and the slower reaction speed, while the "fatigue phenomenon" is ascribed to the hygroscopicity of the catalyst carrier. Good results were obtained with asbestos instead of metal oxide carriers. At a hydrogenation temp. of 300-400°, the hygroscopicity of asbestos and the formation of silicates were avoided. Quant. redn. of  $\text{CO}_2$  takes place in excess H during pyrolysis. Sample wts., therefore, must be small enough to form 0.3-0.5 mg.  $\text{H}_2\text{O}$  during hydrogenation. For the quant. detn. of such small amts. of  $\text{H}_2\text{O}$ , a new method was developed, involving the hydrolysis of Ba cyanamide and  $\text{Ba}(\text{OCN})_2$ . This reaction permits a volumetric detn., similar to the accepted microdetn. of N. The sample is decompd. in a stream of purified H and distd. onto a Pd catalyst at 450° to remove any  $\text{CO}_2$  formed in a secondary reaction during pyrolysis, thus upsetting the H equil. The decompn. products are passed over a Ni catalyst at about 300-30° to catalyze the hydrogenation of CO and  $\text{CO}_2$ , and over Cu at 850° to sep. any  $\text{NH}_3$  from  $\text{H}_2\text{O}$  by thermal disson. in the analysis of N-contg. compds. The tube fillings are sepd. with Ag wool to aid the conduction of  $\text{H}_2\text{O}$  vapors into a trap where the  $\text{H}_2\text{O}$  is frozen. To remove all H, the trap is rinsed with  $\text{CO}_2$  and the speed and size of micro bubbles controlled by an azotometer which is connected to the trap. While the  $\text{H}_2\text{O}$  slowly evap., the vapors are carried by means of  $\text{CO}_2$  over Ba cyanamide or  $\text{Ba}(\text{OCN})_2$  at 360°, where hydrolysis takes place. The hydrolysis products ( $\text{NH}_3$  and isocyanic acid) are oxidized over CuO at 520°. A  $\text{SiO}_2$  filling is placed before the CuO to prevent back-diffusion of  $\text{H}_2\text{O}$ . The gases are collected in an azotometer, and the vol. is read. Halogen-contg. compds. are hydrogenated in a stream of H contg.  $\text{NH}_3$ . The resulting  $\text{NH}_3$  halides are filtered from the gas stream by means of glass wool at 70° (cf. preceding abstr.). The Pd catalyst is exchanged for a Ni coil at 850° to insure quant. redn. of any halogens present in their elementary form. Excess  $\text{NH}_3$  is removed at the Cu catalyst. The analysis can be carried out in 40-60 min., depending on the O content of the compd. Successful detns. depend greatly on timing the various steps. 24 references.

Hilde Alsberg

48 h, 1962, V. 57

Spectrophotometric titration of water in acetic acid. Stanley Bruckenstein (Univ. of Minnesota, Minneapolis). *Anal. Chem.* 31, 1757-60(1959).—A direct spectrophotometric titration of water in glacial AcOH based on the sulfuric acid-catalyzed hydrolysis of Ac<sub>2</sub>O has been applied to the detn. of water in the range 0.009-1.7%. W. Ticky

4241 h, 1960

Indirect determination of water in some liquid products with cobaltous bromide. Z. Lada (Inst. Chem. Ogołnej, Warsaw). *Chem. Anal.* (Warsaw) 6, 559-63(1961).—The procedure of Gardner and Keyte (*Analyst* 83, 150(1958)) for estn. of H<sub>2</sub>O with an error of  $\leq 10\%$  in liquid org. products was modified. To a sample contg. about 1-6 mg. water in a tube dried at 120° and kept in desiccator, add 10-15 ml. satd. CoBr<sub>2</sub> in anhyd. CHCl<sub>3</sub>, stopper the tube, and leave for 20-30 min. Vacuum filter through a G4 baked glass filter, wash with a few ml. of anhyd. CHCl<sub>3</sub>, dissolve the ppt. in H<sub>2</sub>O, dil. to 100 ml., dil. an aliquot to about 70 ml. with H<sub>2</sub>O, add 1 ml. 2*N* HCl, 0.01*M* Complexone III, adjust pH to approx. 10, and titrate with 0.01*M* ZnCl<sub>2</sub> in the presence of Eriochrome Black T. The method was tested on standard solns., such as CHCl<sub>3</sub>, Me<sub>2</sub>CO, salicylaldehyde, propionaldehyde, and benzaldehyde.

Z. Kurtvka

4102 C, 1962, V. 56

Determination of water content in recirculated butyl acetate (Fenosolvan). Zygmund Lada (Gen. Chem. Inst., Warsaw). *Rev. Chim.* (Bucharest) 10, 348-9(1959). Expts. were performed to enable development of a method for detn. of H<sub>2</sub>O (CA 51, 7219e) in recirculated esters. As reagents, the Fischer reagent in 1 soln. (Mitchell and Smith, *Aquametry*, 1948 (CA 43, 522g)) and in 2 solns. (Seaman, *et al.*, CA 43, 4975c), as well as 2 modifications of the van der Meulen reagents (Brit. 728,947, CA 49, 13028; Dutch 80,667, CA 51, 17617) were studied, titrating electrometrically with the dead-stop end point method. Similar results were obtained for all 4 reagents employed with pure and recirculated BuOAc, but the Fischer reagents, modified and unmodified, yield smaller differences than the van der Meulen reagents. The Seaman-modified Fischer reagent was most suitable and permitted detn. of 0.5-3.6% H<sub>2</sub>O in samples of pure and recirculated BuOAc with an accuracy of approx. 2% (where no moisture was absorbed in the solns. while placing in the open titration flask). The method is rapid, taking a max. of 30 min.

M. Lapidot

7907 d, 1962, V. 57

Determination and removal of small amounts of water in acetone. K. Jordan and W. R. Fischer (Krupp WIDIA-Fabrik, Essen, Ger.). *Z. anal. Chem.* 168, 182-90(1959).—H<sub>2</sub>O in the range 0.01-2% in Me<sub>2</sub>CO can be detd. with AcCl or by cloud point in petr. ether. Me<sub>2</sub>Co is dried to 0.005% H<sub>2</sub>O by treatment with AcCl + C<sub>6</sub>H<sub>6</sub>N and AmOH followed by distn. On standing over K<sub>2</sub>CO<sub>3</sub>, H<sub>2</sub>O in Me<sub>2</sub>CO is reduced to 0.2%. Anhyd. Me<sub>2</sub>CO absorbs H<sub>2</sub>O from air rapidly (0.005 to 0.03% in 15 min.).

K. G. Stone

19671 d, 1959

Determination of water distribution by Indipa indicator paper. Karl Koehnen. *Milchwissenschaft* 14(8), 371-4(1959). This paper of Bacto-Strip A.G., Zollikon-Zuerich, Switzerland, was very suitable for detn. of H<sub>2</sub>O distribution in butter. Exact instructions were included. The dyes used in the production of the paper were condensation products of substituted sulfobenzoxyl compds. From CZ 1960.31), 10439.

MFCR

6134 f, Vol. 60



**Chemical determination of water in pyridine.** Maurice Pesez (Etablissements Roussel, Paris). *Ann. pharm. franç.* 4, 98-9(1946).—The method is based on the fact that all the solvents (except  $H_2O$ ) capable of reacting with  $Ac_2O$  combine with one  $Ac$  group to form an acetylated deriv., thus leaving only one  $Ac$  available for acidimetric titration, whereas  $H_2O$  gives directly  $2AcOH$  per  $Ac_2O$  mol. *Detn.*: to 5 cc. pyridine add exactly 2 cc. pure  $Ac_2O$  (b.  $138-9^\circ$ ) in an acetylation flask, heat 15 min. in boiling water, cool in ice, add 5 cc. freshly redistd.  $PhNH_2$  (b.  $182^\circ$ ), let stand 10 min. with occasional shaking, add 30 cc.  $H_2O$ , and titrate with  $N$   $NaOH$  in the presence of phenolphthalein; run a blank on 2 cc.  $Ac_2O$  and 5 cc.  $PhNH_2$  without heating. If the pyridine contains more than 5%  $H_2O$ , use a 2-cc. or a 1-cc. sample and make to 5 cc. with pure pyridine of known  $H_2O$  content. The reaction of  $Ac_2O$  with excess of  $PhNH_2$  is complete in a few min. in the cold; under the same conditions  $AcOH$  does not form any acetamide even after several hrs. The method can det. a few tenths of 1%. A. P.-C.

3015 g, 1947

**Photoelectric gas analysis by the fog method.** A. G. Amelin and B. V. Mikhail'chuk. *Zavodskaya Lab.* 14, 778-82(1948).—It is intended to use this app. for signaling deviations of a gas from a fixed moisture content. Two sep. air streams under pressure pass through two tubes contg.  $P_2O_5$  and then through glass spiral tubes one of which contains concd.  $H_2SO_4$  and the other oleum. In all expts. an excess of  $SO_2$  was supplied in order to bind all the moisture. The temp. in thermostats was so regulated that equil. concn. of water vapor and  $SO_2$  was maintained at a given level. The air streams contg. moisture and  $SO_2$  were mixed and the gas mixt. passed into a glass tube of a photoelec. photometer. In the photoelec. app. the movement of the galvanometer is regulated by the opposing currents from the storage battery and from the illuminated photocell. The deviation of the needle is thus used to indicate the extent of darkening of the photocell. *Detns.* of moisture content of a gas by this method

gave low concn. of fog and low absorption of light by the fog. However, by using a more sensitive galvanometer and photocell and a longer glass tube, the accuracy can be improved. A sketch of the app. is shown. B. Z. K.

5761 h, 1950

**Electrometric determination of the moisture of materials.** B. S. Gukhman, B. I. Petrov, and T. I. Yakovlev. *Zavodskaya Lab.* 14, 646-9(1948).—Moisture is detd. by direct electrometric titration with a soln. of  $I_2$  and  $SO_2$  gas in mixt. with abs. alc. and pyridine, by use of polarizing electrodes with a lamp amplifier and an optical indicator for registering changes in current strength during the titration. The app. is a modification of that used by McKinney and Hall (*C.A.* 37, 4934\*); the triode used raised the sensitivity of the app. 2-3 times and optimum selection of resistances gave it greater stability.

B. Z. Kamich

3746 a, 1949

A method for the estimation of small quantities of water in alcohol. E. L. SMITH. *J. Chem. Soc.* 1927, 1284-8.—Na or K is dissolved in the alc. to be tested, the alkali thus formed hydrolyzing, irreversibly, the EtOAc added until all the H<sub>2</sub>O in the alc. is consumed. A few 10-cc. samples are taken, one being titrated immediately, sealed in tubes, placed in a 70° H<sub>2</sub>O bath, removed singly, at intervals, in about 3 hrs. and titrated. With decreasing titer values, the "limiting titration," at infinite time, is best obtained by extrapolating to zero the curve of titration against the reciprocal of the time. The % H<sub>2</sub>O is found by multiplying the difference between the initial and limiting values, in cc., by the factor 0.0225 for 0.125 N acid. If the concns. of ethoxide and ester are still 0.25-0.5 N when the H<sub>2</sub>O is all used, the reaction is complete in a half to an hr., heating in a thermostat and extrapolation being unnecessary. The app., reagents and solns. must be protected from moisture. The estd. H<sub>2</sub>O content by the method is accurate to 0.01%.  
J. BALOZIAN.

3328, 1927

Determination of hydrochloric acid and water in waste gases. A. P. Obukhov. *Zavodskaya Lab.* 3, 416-17 (1934).—The difficulty in sampling and detg. mixts. of HCl and H<sub>2</sub>O in the waste gases of the production of HCl by thermal decompn. of MgCl<sub>2</sub> was overcome by sucking the gas through a system of wash bottles, of which the 1st one is empty and heated electrically to 130°, the other 4 bottles are filled with concd. H<sub>2</sub>SO<sub>4</sub> (satd. with HCl) and connected with an aspirator bottle filled with H<sub>2</sub>O, where HCl is absorbed.  
Chas. Blanc

1948, 1955

Improved method for the analysis of gaseous mixtures on the micro scale. G. H. Bush and R. J. Loneragan (Armament-Research Establishment, Fort Halstead, Sevenoaks, Engl.). *Analyst* 79, 371-9(1954).—Various methods of gas analysis are reviewed and the method recommended by Sutton (*C.A.* 32, 4386<sup>3</sup>) with dry reagents is described. A fairly simple pipet is shown in which the gas is kept under const. pressure. With it about 0.1 ml. of sample can be analyzed with a precision comparable to that attained on the macro scale. Water vapor is detd. with Anhydrone which absorbs it, CO<sub>2</sub> is removed with activated soda-asbestos, O is removed with yellow P heated by means of Ni-Cr wire. An excess of O is added and the H and CO are oxidized at 1000°. The water and CO<sub>2</sub> formed are removed with Anhydrone and Ascarite. The excess O is removed by a heated yellow P bead and the residual N is measured.  
W. T. Hall

9271 c, 1954

The estimation of the water content of cadmium thiobarbiturate by means of derivatographs. J. Ristici, S. Gal, and G. Liptay (Tech. Univ., Timisara, Hung.). *Periodica Polytech.* 7(1), 21-6(1963). A derivatograph (Paulik, *et al.*, *C.I.* 52, 13325f) was used to investigate the nature of the bound H<sub>2</sub>O in Cd thiobarbiturate. A heating rate of 3°/min. and 300-400-mg. samples were used. After washing with EtOH and Et<sub>2</sub>O the compd. contained 1.6% mech. bound H<sub>2</sub>O and 8.20% H<sub>2</sub>O of crystn. The latter was liberated in 2 parts, 6.3% (corresponding to 1.5 mols. H<sub>2</sub>O) between 150 and 245° and 1.9% (0.5 mol. H<sub>2</sub>O) between 250 and 310°. This confirms the formula Cd-[C<sub>4</sub>H<sub>2</sub>O<sub>2</sub>N<sub>2</sub>S]<sub>2</sub>·2H<sub>2</sub>O.  
Dorothy M. Goode

10763 c, 1963, V. 59

Water vapor determination in a flow of gas. N. V. Philips' Gloeilampenfabrieken. *Brit.* 941,521 (Cl. G 01c), Nov. 13, 1963; *Fr. Appl.* Feb. 24, 1959; 5 pp. Addn. to *Brit.* 846,648. Water vapor in a gas flow is decompd. to H and diffused thru a semi-permeable Pd tube. Equil. pressure of H across the tube is measured in a manometer adapted to attenuate the partial pressure of H to a slight extent while allowing the partial pressure of other gases (CO and CO<sub>2</sub>) to be reduced to a negligible amt. by suction.  
John Russell

4809a, Vol. 60

Rapid determination of moisture in liquid ammonia by means of metallic sodium. V. A. Pleskov. *Zavodskaya Lab.* 6, 177-80 (1937).—The detn. of H<sub>2</sub>O in liquid NH<sub>3</sub> by means of Na is based on the decolorization of the highly colored solns. of Na in NH<sub>3</sub> by the formation of NaOH insol. in NH<sub>3</sub>, and depends on the titration of Na with the NH<sub>3</sub> to be tested at -35°. The slow decompn. of Na is catalyzed by Cu(NO<sub>3</sub>)<sub>2</sub>·4NH<sub>3</sub> (cf. Horn, C. A. 2, 958). The secondary reaction of NaNH<sub>2</sub> formation proceeds very slowly and does not affect the detn. This reaction is catalyzed by any contaminating Fe compds., which should be removed before titration. A weighed ampoule, made from a capillary tube (0.2-0.3 mm. inside diam. and 1-2 mm. wall thickness), is charged by suction with fused Na (15-300 mg. for 0.1-2% H<sub>2</sub>O in NH<sub>3</sub>), and, after weighing, is sealed at the elongated ends with paraffin. A sample of NH<sub>3</sub> is withdrawn into a 500-cc. flask, contg. 20-30 mg. of the catalyst. It is fitted with a KOH U-tube and a charge tube provided in the middle of the upper bend with a 2-way stopcock connected to the NH<sub>3</sub> cylinder valve by means of a brass nipple lined at the bottom with filter paper between brass wire gauze supported by Pb strips. Before charging the flask, the stopcock is turned to the outside and some of the NH<sub>3</sub> is blown through the valve and nipple. The titration app. consists of 2 glass cylinders connected at the bottom by means of a rubber tubing. The left limb (100-50 cc. capacity) is fitted at the bottom center with a glass-rod stopper, a side glass tube reaching to the bottom and a glass-stoppered opening at the top. After immersing the app. in a mixt. of acetone and solid CO<sub>2</sub> (or liquid NH<sub>3</sub>), the graduated limb is charged with NH<sub>3</sub> from the flask and the left limb with the crushed ampoule. By raising the glass-rod stopper small portions of the NH<sub>3</sub> are introduced into the left limb at intervals of 2-3 sec. The reaction mixt. is constantly stirred by a current of dry NH<sub>3</sub> or H<sub>2</sub> introduced through the side tube. The introduction of liquid NH<sub>3</sub> is continued until the color disappears. A new ampoule is introduced and the operation is repeated 1 or 2 times. The amts. of NH<sub>3</sub> used are read on the graduated limb. In the presence of large moisture contents, the energetic stirring of the reaction mixt. becomes difficult in the presence of large amts. of NaOH. Hence, the titration is carried on to a pale blue, stable for 30-40 sec. The results are calcd. by the formula:  $x = 114.7 g/V$ , where  $V$  is the vol. of liquid NH<sub>3</sub> in cc. and  $g$  is Na in g. At -35° the d. of liquid NH<sub>3</sub> is 0.683. Since Na reacts with C<sub>6</sub>H<sub>5</sub>N, PhOH and unsatd. org. compds., this method gives the total impurities in coal-tar NH<sub>3</sub>. By this method 0.01% H<sub>2</sub>O can be detd. with accuracy of 2-3% inside of 10-15 min. C. B.

6135', 1937

Estimation of Water in the Volatil Products Produced in the Distillation of Fuels. G. LAMBRIS. *Z. anorg. Chem.*, 81, 24-39.—This study was made in connection with an investigation of the formation of H<sub>2</sub>O during the distillation of coal. P<sub>2</sub>O<sub>5</sub> proved to be the only suitable desiccant for such gas mixts. P<sub>2</sub>O<sub>5</sub> which has absorbed H<sub>2</sub>O, may be heated to 200° without sublimation, or expulsion of H<sub>2</sub>O. P<sub>2</sub>O<sub>5</sub> does not absorb C<sub>6</sub>H<sub>6</sub>, C<sub>7</sub>H<sub>8</sub>, C<sub>8</sub>H<sub>10</sub>, C<sub>10</sub>H<sub>8</sub>, H<sub>2</sub>S, SO<sub>2</sub>, CN, HCN, or illuminating gas, either separately or mixed, provided the drying tube is heated to 150° after the passage of the above substances. It absorbs quant. NH<sub>3</sub> (forming pyrophosphordiaminic acid), C<sub>2</sub>H<sub>5</sub>N, and C<sub>6</sub>H<sub>5</sub>OH (forming mono or diphenyl esters of H<sub>2</sub>PO<sub>4</sub>). It was therefore necessary to obtain a method for detg. the NH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>N, and C<sub>6</sub>H<sub>5</sub>OH absorbed in the P<sub>2</sub>O<sub>5</sub>, in order to make the necessary corrections for H<sub>2</sub>O. The method adopted is as follows: Dissolve the contents of the P<sub>2</sub>O<sub>5</sub> tube in H<sub>2</sub>O, filter twice through a hardened filter (to remove impurities such as yellow or red P) and distil the soln. with strong alkali into excess of 0.1 N H<sub>2</sub>SO<sub>4</sub>, which absorbs the evolved NH<sub>3</sub> and C<sub>2</sub>H<sub>5</sub>N, the sum of which is estd. by titration of the excess acid with NaOH, using Congo red as the indicator. The neutral soln. is evapd. to dryness with about 0.5 g. neutral (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and finally heated to 130° for 15-30 min., whereby the C<sub>2</sub>H<sub>5</sub>N is expelled. The H<sub>2</sub>SO<sub>4</sub> thereby liberated (and existing in the residue as NH<sub>4</sub>H<sub>2</sub>SO<sub>4</sub>) is titrated with NaOH from which the C<sub>2</sub>H<sub>5</sub>N may be calc. and the NH<sub>3</sub> estd. by dif. C<sub>6</sub>H<sub>5</sub>OH is detd. by acidifying with H<sub>2</sub>SO<sub>4</sub> the alk. residue from the 1st distillation, and distilling in a current of steam. The C<sub>6</sub>H<sub>5</sub>OH in the distillate is estd. by addn. of (a) Br<sub>2</sub> + KBrO<sub>3</sub>, (b) H<sub>2</sub>SO<sub>4</sub>, and finally (c) KI, after which the liberated I<sub>2</sub> is titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. The phenol is 1st pptd. as C<sub>6</sub>H<sub>4</sub>Br<sub>2</sub>O, which reacts with KI to form C<sub>6</sub>H<sub>4</sub>Br<sub>2</sub>OK. H<sub>2</sub>O is then estd. by deducting from the total gain of the P<sub>2</sub>O<sub>5</sub> tube, the sum of the wts. of NH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>N and C<sub>6</sub>H<sub>5</sub>OH. Expts. on known amts. of these constituents showed that the results for H<sub>2</sub>O are accurate to about 1% of the H<sub>2</sub>O present.

WILLIAM FLUM.

5293, 1913

Chemical analysis system employing photosensitive devices. Charles A. Styer (to Westinghouse Elec. & Mfg. Co.). U. S. 1,977,359, Oct. 16. In analyzing water for Cl, a reagent such as *o*-toluidine is added which produces a yellowish green color by reaction with the Cl and serves to modify the light-transmitting properties which may be used for analytical determination or for automatic regulation of Cl added to water contg. org. matter. In analyzing gases for HCl or for water vapor, NH<sub>3</sub> or PCl<sub>3</sub>, resp., may be added to modify the light-transmitting properties, beams of light being passed through treated and untreated material for comparison tests. Fluids may be analyzed for their combined carbonate, sulfate and dissolved CO<sub>2</sub> content by use of Ba(OH)<sub>2</sub> as a reagent for modifying light-transmission properties. Various details of app. and operation are described. Color filters may be used.

527, 1935

Elementary microanalysis. R. GOUBAU. Univ. of Ghent. *Natuurw. Tijdschrift* 10, 129-36(1928); *Bull. soc. chim. belg.* 37, 335-44; cf. *C. A.* 21, 3576.—To eliminate the use of absorption trains in microanalysis, G. describes methods for the detn. of H<sub>2</sub>O and CO<sub>2</sub> by direct condensation. The combustion takes place in a long quartz tube with ground joints filled according to Pregl, the PbO<sub>2</sub> being heated by electricity in an Al block. The O<sub>2</sub> used is passed through H<sub>2</sub>SO<sub>4</sub>, soda lime and CaCl<sub>2</sub>, a quartz tube contg. Pt heated to redness, a bubble flask, and a U tube with Ag spirals cooled with liquid O (not liquid air) before entering the combustion tube. The products of combustion go through a U tube cooled in solid CO<sub>2</sub> + alc. (H<sub>2</sub>O condensed) and a second one with liquid O (CO<sub>2</sub> condensed). All glass work is ground, the absorption U tubes are of thin glass 3 to 4 mm. wide, 15 cm. long with constrictions at tops and bottoms; they contain Ag spirals for better cooling efficiency. After the combustion the U tubes are sealed off at the constriction nearest to the combustion tube, the tube is rapidly evacuated from the other end (1 mm. by oil pump) and the other top constriction sealed, all before the cooling bath is removed. The loss by the evacuation is negligible. The individual sealed tubes are then turned over with one leg outside the Dewar vessel to distil the H<sub>2</sub>O or CO<sub>2</sub> over into the cool leg and the latter is in turn sealed off at the bottom constriction. The containers so obtained are much lighter than Pregl's and easier to weigh. The glass tare is weighed subsequently after removal of the condensate by repeated evacuation and heating; the tubes are not to be touched by hand. Examples are given showing the correction for O<sub>2</sub> and air in the tubes; samples of 10 mg. material are advised.

B. J. C. VAN DER HOEVEN

789, 1929

Method of using the Fischer-Schrader aluminium retort and analysis of distillation products obtained therewith. E. NOACK. *Brennstoff-Chem.* 5, 17-22(1924).—Improved methods are given for detn. of coke, gas, tar, H<sub>2</sub>O and phenols obtained in low-temp. carbonization tests with the Fischer-Schrader lab. retort (cf. *Brennstoff-Chem.* 1, 87(1920)(cf. *C. A.* 14, 3149); Fritsche, *C. A.* 16, 1000-1.

I. D. DAVIS

2038, 1924

A spectrophotometric assay of water in organic solvents. R. C. R. Barreto and H. S. R. Barreto (Univ. Brazil, Rio de Janeiro). *Anal. Chim. Acta* 26, 494-5(1962)(in English). To det.  $\geq 2\%$  H<sub>2</sub>O in org. solvents, add a 1-ml. sample of the org. solvent to 9 ml. abs. EtOH; add 5 ml. of the mixt. to 5 ml. 0.002M chloranilic acid (I) in abs. EtOH, and measure the absorbance at 520 m $\mu$  vs. a blank contg. 5 ml. abs. EtOH and 5 ml. I. The increase in absorbance at 520 m $\mu$  is proportional to the percent H<sub>2</sub>O. The av. relative error is 1.5%.

J. O. PAGE

Volumetric determination of water in alcohols. Yüan-Chi Hsu. *Hua Hsueh Shih Chieh* 14, 184(1959).—An improved procedure, based on the reaction of chloroacetylpyridine with H<sub>2</sub>O and with alc., is described; 0.1N NaOH is used for titration, with phenolphthalein as indicator. From *Sci. Abstr. China, Chem. and Chem. Technol.* No. 4, Abstr. No. 337(1959).

K. L. C.

22145 L 1960

Hydroxyl in the diffusion of H in fused SiO<sub>2</sub>. R. W. Lee (Gen. Motors Corp., Warren, Mich.). *Phys. Chem. Glasses* 5(2), 35-43(1964). A literature review is given on recent studies of H and H<sub>2</sub>O in fused SiO<sub>2</sub>. Introduction and removal of OH is inherent in both the H reaction (H<sub>2</sub> + SiO<sub>2</sub>) and the H<sub>2</sub>O reaction (H<sub>2</sub>O + SiO<sub>2</sub>). Differences occur in OH soly., reaction rates, and diffusing species. Exptl. activation energies are reviewed for both reactions and plausible OH-producing mechanisms are cited. The OH distribution resulting from H gas loading was investigated by infrared sectioning techniques. Deuterium degassing tests with solid cylinders of Vitreosil were found to agree with hollow cylinder results on GE 204. Results are employed to formulate a model for the diffusion of mol. H in fused SiO<sub>2</sub> to distinguish between metastable and permanent OH.

C. H. Wayman

12989f, Vol. 60

The determination of water in potassium salts for fertilizing by definitive methods and the value of obtained results. H. Doering. *Landwirtsch. Forsch.* 16, 221-8(1963). For the detn. of film water in K salts for fertilizing, drying with a desiccant had sufficient accuracy. For the detn. of total H<sub>2</sub>O (crystal water, water enclosed in crystals, and film water) drying at 450° to const. wt. is necessary.

B. F. Grohse

15094h, U. 60

Determination of the content of water in ion exchangers by the indicator method. V. V. Parkhomenko and O. D. Kurilenko (Technol. Inst. Food Ind., Kiev). *Ukr. Khim. Zh.* 30(3), 244-7(1964). The ion exchangers KU-1, KU-2m, and KB-4-P2 were satd. with H, Na, Ca, and Fe ions, and the content of water was detd. by using solns. of saccharose and glucose as indicators. The amt. of bound H<sub>2</sub>O in the same type of ion exchangers depends on the type of satg. ion. The effect of the satg. ions on the H<sub>2</sub>O content decreased in the order H>Na>Ca>Fe. With KU-2, the amt. of H<sub>2</sub>O increases with decreasing cross-linking of the ion exchanger.

GPJR

14675h, U. 60

A micro method for determining moisture distribution in wheat kernels, based on iodine staining. H. L. Seckinger, M. J. Wolf, and R. J. Dimler (Northern Regional Res. Lab., Peoria, Ill.). *Cereal Chem.* 41(2), 80-8(1964). Samples of wheat endosperm, approx. 10<sup>-4</sup> mm.<sup>2</sup>, were prepd. under oil, ground on a small piece of frosted glass in the well of a culture slide. The well was filled with light, colorless mineral oil satd. with I and stained for 16-18 hrs. at 30°. The color was compared with that of similar samples of known moisture content by using a comparison eyepiece at 100-fold magnification. The samples of known H<sub>2</sub>O content were prepd. from wheat endosperm equilibrated at 25° in closed chambers contg. satd. salt solns. The H<sub>2</sub>O content of the standard samples was 10-26%, and their color when stained was stable for several days. By use of the procedure, an increase in H<sub>2</sub>O could be detected in the center of a wheat kernel after 2 hrs. of tempering. Sections from a kernel tempered 2 hrs. showed a moisture gradient of 4 percentage points over 1 mm.

Alice M. Briant

16202a, U. 60

Micro test for the detection of water split off by heat. M. M. Nijland (Rijksuniv., Utrecht, Neth.). *Mikrochim. Technol. Acta* 1963(5 6), 947-9 (in English). The test sample is placed in the bottom of a micro test tube, which is then heated in a flame to form a constriction 3.5 cm. from the bottom. A 1 x 15-mm. strip of  $\text{CoCl}_2$  paper is inserted into the upper chamber, and the tube is heated to form another constriction about 3.5 cm. above the 1st one. The tube is evacuated, then sealed off at the top constriction. The lower chamber contg. the sample is inserted through an asbestos plate into a glycerol heating bath. Water released by heat converts anhyd.  $\text{CoCl}_2$  on the paper to the pink hexahydrate. As little as 50  $\gamma$   $\text{H}_2\text{O}$  can be detected. The indicator paper is prepd. by soaking filter paper in 15%  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , then drying in an oven. Harold F. Schaeffer

6215b, U. 60

Colorimetric moisture indicator. Stanley D. Price (to Mine Safety Appliances Co.). U.S. 3,121,615 (Cl. 23-254), Feb. 18, 1964; Appl. Sept. 2, 1960; 2 pp. An aq. soln. of Co iodide is prepd. and treated with  $\text{Na}_2\text{S}_2\text{O}_3$  to develop a pink color. By further addn. of the thiosulfate, the indicator is stabilized against the liberation of free I. Filter paper is impregnated with the pink soln. and dried. Thus, 25 g. of anhyd. Co iodide is dissolved in 1 l.  $\text{H}_2\text{O}$ . The soln. is filtered, and 1.5 g. of  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  is added. Then another 1.25 g. of the latter is added. A 950-sq. cm. sheet of filter paper is placed on a flat glass plate, and 20 cc. of the soln. is distributed evenly over the surface. The paper is air dried for 3-4 hrs. It can then be installed in a canister, where, in the absence of moisture, it assumes a bluish green color. At about 3.5% moisture the paper becomes a pale bluish green approaching neutral. If the paper is not to be installed immediately, it is stored in a sealed container with some silica gel. For certainty in knowing when the hopcalite or other CO detg. agent has attained the amount of moisture beyond which the canister should not be used, a permanently colored reference standard which matches the color of the indicator at 3.5% moisture is installed side by side with the indicator.

Judith G. Chernin

11385 d, U. 60

Determination of water in bromine. Luisa Nebbia and Basilio Pagani (Ist. "G. Donegani," Montecatini, Novara, Italy). *Chim. e ind. (Milan)* 39, 5-6(1957).— $\text{H}_2\text{O}$  in Br is detd. by the introduction of  $\text{SO}_2$  into a  $\text{CCl}_4$  soln. of Br. The resulting  $\text{H}_2\text{SO}_4$  is then detd. gravimetrically after elimination of excess Br. Five hundred g. or less of Br are used, with an equal vol. of  $\text{CCl}_4$  and treated with 0.5-1.0 l./min.  $\text{SO}_2$  for 10-15 min. at room temp. Excess  $\text{SO}_2$  is displaced by a stream of A, and the  $\text{CCl}_4$  soln. is washed with distd.  $\text{H}_2\text{O}$ , 50-60 ml.  $\text{CCl}_4$  is added, and the layers are sep'd. The washing is repeated twice, the combined  $\text{H}_2\text{O}$  layers are acidified with HCl and evapd. over a direct flame to 150-200 ml., 10%  $\text{BaCl}_2$  is added and the  $\text{H}_2\text{O}$  is detd. from the wt of the resulting  $\text{BaSO}_4$ . Nine detns. run in comparison with the method of Haines (C.A. 44, 2187h) showed agreement within 0.007%. Francis A. Domino

14479i, 1957

Determination of moisture in bromine. V. F. Boitsova, K. D. Rusinova, and N. I. Stepanova. *Zavodskaya Lab.* 26, 550-1(1960).—Two methods for the detn. of  $\text{H}_2\text{O}$  in Br were checked (Nebbia and Pagani, CA 51, 14479i; Haines, CA 51, 9106d). The latter method is simpler and does not require the use of anhyd.  $\text{CCl}_4$ ,  $\text{SO}_2$ , or Ar purified of O and moisture.

(CA 44, 2187h)

4102b, 1962, V. 56

Liquid-phase indicator for determination of water in liquids. Richard A. Line and Henry Hoftiezer (to Ansul Chemical Co.). U.S. 2,761,312, Sept. 4, 1956. Water is detd. in halogenated refrigerants, gasoline, oils, etc., by means of white cellulosic materials contg. predetd. amts. of pure anhyd.  $\text{CoBr}_2$ . Upon immersion, the color indicates the water content by comparison with standards. Pref-

erably, white unsized paper is used, 0.006-0.008 in. thick. The optimum concn. of  $\text{CoBr}_2$  is 0.01-0.7 g./cc. of cellulosic material. This indicator is especially suitable for detn. of water in liquids in which it is only slightly sol. W. W.

15351 C, 1956

Colorimetric determination of water in ceresin. M. G. Morekhin, S. I. Ageev, O. E. Matyash, and T. G. Chechina. *Zarodsk. Lab.* 28, 67(1962). The detn. of water in ceresin is based on the reaction of water in the fuel with anhyd.  $\text{CuSO}_4$ . The intensity of the blue color is compared with that of standards. The prepn. of standards is as follows: into 3-l. flasks are poured 1 l. samples of anhyd. fuel from which the  $\text{H}_2\text{O}$  had been removed by filtration through anhyd.  $\text{CuSO}_4$  and 0, 0.2, 0.4, 0.6, 0.8, and 1.0 g., resp., of  $\text{H}_2\text{O}$  are added to the flasks. The mixts. are shaken thoroughly and then filtered through glass filters each of which contains 2 g. anhyd.  $\text{CuSO}_4$ . The filters with the  $\text{CuSO}_4$  layers covered with dry fuel are closed with plugs at both ends and used as standards for 1 month. For the detn. of  $\text{H}_2\text{O}$  in a sample, 1 l. of fuel is filtered through a glass filter contg. 2 g. anhyd.  $\text{CuSO}_4$ . M. Rakovic

1280 C, 1963, V. 58

Determination of water in lubricating oil. Samuel Swadesh (to Yosemite Chemical Co.). U.S. 3,003,353. Appl. Sept. 29, 1958. A method and app. are described for detg.  $\geq 0.6\%$   $\text{H}_2\text{O}$  in lubricating oil by colorimetry. The oil is dild. with aromatic hydrocarbon solvents to lessen its color and a dye such as Neptune Blue BRA Conc. is added and the mixt. well stirred. The intensity of the ensuing green color is a measure of the water content of the oil and can be checked against standard samples. The app. is portable and can be used in field tests. John F. Shultz

7602 f, 1962, V. 56

Karl Fischer Method (SO<sub>2</sub>, Pyridine, I<sub>2</sub>, MeOH)  
and Modifications

K. Fischer's method for the titrimetric determination of water. A. Zimmermann. *Fette u. Seifen* 46, 446-50 (1939).—Substances which can be analyzed for moisture content by F.'s method (C. A. 29, 6532<sup>9</sup>) include ethylene and propylene glycols, glycerol, oxalic, oleic and sulfosalicylic acids, glucose, galactose, maltose, lactose, sorbitol, cotton, aq. H<sub>2</sub>O soln., starch, dextrose, polyvinyl alc., cellulose derivs., pectin, crushed oil-seed cake, etc. Strongly alk. substances, e. g., soda, soap, Na phosphates and perborate, NH<sub>3</sub>, cannot be analyzed by F.'s method because of interference due to HIO formation. Aldehydes, with the exception of paraformaldehyde, and org. peroxides, e. g., Bz<sub>2</sub>O<sub>2</sub>, also cannot be analyzed by F.'s method.

J. W. Perry

4015<sup>2</sup>, 1940

Analytical procedures employing Karl Fischer's reagent. I. Nature of the reagent. Donald M. Smith, W. M. D. Bryant and J. Mitchell, Jr. *J. Am. Chem. Soc.* 61, 2407-12(1939).—S. and B. (C. A. 29, 3940<sup>2</sup>) described a procedure for detg. H<sub>2</sub>O which depends on the hydrolysis of AcCl. Almost simultaneously F. (C. A. 29, 6532<sup>9</sup>) published a method in which the detn. is based on a direct titration with a reagent contg. MeOH, pyridine and I<sub>2</sub>. In some cases one and in some cases the other method has proved superior but a study of the latter method shows that F. was wrong in assuming that 1 mole of I<sub>2</sub> reacted with 2 moles of H<sub>2</sub>O for the expts. now described show that the ratio of I<sub>2</sub>:H<sub>2</sub>O = 1:1. The reaction is shown to take place in 2 stages and only in the first stage does H<sub>2</sub>O react. The intermediate products have been isolated. The reagent deteriorates on standing and considerable light is now thrown upon the nature of the decompn. which is doubtless caused by bacterial action but no means was discovered for preventing it. Expts. were made with substitution of other solvents for MeOH and other amines for pyridine but without accomplishing any distinct improvement. The compds. present in the reagent were detd.

W. T. H.

8524<sup>2</sup>, 1939

Determination of moisture in liquid sulfur dioxide. G. V. Zavarov. *Zavodskaya Lab.* 5, 1314-18(1936).—Two procedures for the detn. of H<sub>2</sub>O in liquid SO<sub>2</sub> are described in detail. One is based on the Fischer method (C. A. 29, 6532<sup>9</sup>), depending on the reaction: SO<sub>2</sub> + I<sub>2</sub> + 2H<sub>2</sub>O = H<sub>2</sub>SO<sub>4</sub> + 2HI, and another on combining it with the Eustis method (C. A. 27, 2226). The requisite app. is illustrated. Conductometric method for determining traces of moisture in liquid sulfur dioxide. V. A. Pleskov. *Ibid.* 1319-22.—The conductometric detn. of traces of H<sub>2</sub>O in liquid SO<sub>2</sub> in the presence of dry HCl at -20° (cf. Bagster and Cooling, C. A. 14, 2578) is based on the previous method (C. A. 27, 218).

Chas. Blanc

2124<sup>6</sup> 1937

A new method for the analytical determination of the water content of liquids and solids. Karl Fischer. *Angew. Chem.* 48, 394-6(1935).—The H<sub>2</sub>O content is detd. by means of liquid SO<sub>2</sub> in MeOH soln. according to the reaction: 2H<sub>2</sub>O + SO<sub>2</sub> + I<sub>2</sub> = H<sub>2</sub>SO<sub>4</sub> + 2HI. Addn. of C<sub>2</sub>H<sub>5</sub>N fixes the HI and H<sub>2</sub>SO<sub>4</sub>, and the amt. of I soln. used can be read directly by titrating with a standard soln. (790 g. C<sub>2</sub>H<sub>5</sub>N + 192 g. liquid SO<sub>2</sub> + 254 g. I) dissolved in 5l. of anhyd. solvent. If all the reagents are anhyd., 1 cc. of this soln. corresponds to 7.2 mg. of H<sub>2</sub>O. Detailed directions for the prepn. of the soln. are given. H<sub>2</sub>O detns. by this method and by the CaH<sub>2</sub> method, as well as check runs, are reported for C<sub>6</sub>H<sub>6</sub>, SO<sub>2</sub>, C<sub>6</sub>H<sub>6</sub>-SO<sub>2</sub> mixts., MeOH, C<sub>6</sub>H<sub>6</sub>-SO<sub>2</sub>-MeOH mixt., kerosene-ext., transformer oil, acetone, cracked gasoline, etc., bleaching earth, CuSO<sub>4</sub> and CaCO<sub>3</sub>. The accuracy of the method is better than that of the CaH<sub>2</sub> method, and the method is applicable in many instances where the CaH<sub>2</sub> method fails.

Karl Kammermeyer

6532<sup>9</sup>, 1935



Determination of water in alkylation sulfuric acid. Wm. H. Goff, Walter Scott Palmer, and Roland F. Huhndorff (Texas Co., Port Arthur, Texas). *Anal. Chem.* 20, 344-6(1948).—A modification of the Karl Fischer method (C.A. 29, 6532<sup>o</sup>), with  $\text{NH}_4\text{Cl}$  added to prevent decomposition of the reagent, is recommended. Weigh the acid to be tested into a dry titrating flask. Use sufficient sample to yield 0.04-0.10 g.  $\text{H}_2\text{O}$  but not over 2 g. Add 5 g. of  $\text{NH}_4\text{Cl}$  which has been dried at 105°. Mix and crush any lumps. Add 20 ml. of anhyd. MeOH and 2-3 ml. of Fischer reagent contg. 1 mole I to 2 moles  $\text{SO}_2$  to 8 moles pyridine in an equal wt. of water. Titrate the excess reagent with standard water in MeOH. Run a blank test with all the reagents. W. T. Hall

4095 h, 1948

Determination of water by titration (a modified Karl Fischer method). Axel Johansson. *Svensk Papperstidn.* 50, No. 11B, 124-6(1947).—The greatest disadvantage of the Karl Fischer reagent is that it is hygroscopic. This and the complicated app. can be eliminated by titrating in the following manner. The sample is dissolved or mixed in a soln. of  $\text{SO}_2$  and pyridine in MeOH and to this mixt. is added a MeOH soln. of iodine; the reagent is thus divided into 2 solns., neither of which is especially hygroscopic but which, when mixed together, react rapidly with water. The 1st soln. is prepd. by bubbling 60 g.  $\text{SO}_2$  into a mixt. of 300 ml. MeOH (contg. 0.02%  $\text{H}_2\text{O}$ ) and 300 ml. pyridine; the 2nd soln. contains 30 g. iodine/l. Details are given of the detn. of  $\text{H}_2\text{O}$  in various samples of pulp and rayon; it is applicable to MeOH, EtOH, sugar solns., tall-oil products, and similar substances. For dark-colored liquors, such as black liquor, in which the color change with excess iodine cannot be observed, a titration with Pt electrodes is used. C. J. West

7116 c, 1947

Karl Fischer method for the determination of water in liquid sulfur dioxide. G. A. Lewis. *J. Soc. Chem. Ind. Victoria* 45, 576-82(1944).—The method described and illustrated is used for liquid  $\text{SO}_2$  contg. 0.0005-0.001% of  $\text{H}_2\text{O}$ . A dild. Karl Fischer reagent (1 ml. is equiv. to 0.1 mg. of water) is added to the sample of  $\text{SO}_2$  until the color is brown, and the excess is back-titrated electrometrically with the standard MeOH contg. 1 mg. of  $\text{H}_2\text{O}$  per ml., by the dead-stop end point. B. A.

3015 e, 1947

Microdetermination of water. Gabor B. Levy, Justin J. Murtaugh, and Maurice Rosenblatt. *Ind. Eng. Chem., Anal. Ed.* 17, 193-5(1945).—The method was developed to det. water in penicillin Na, but is applicable to other situations; it is based on an electrometric titration with the Fischer reagent (C.A. 29, 6532<sup>o</sup>). Place the sample in a titration cell provided with Pt electrodes and a pressure-equalizing hypodermic needle and closed with a serum-bottle sleeve stopper. Introduce the standardized reagent by means of a hypodermic needle (attached to a microburet and inserted through the stopper) until decolorization ceases and a 5-10% excess is present. Withdraw the needle (also the pressure-equalizing needle) and shake until solids are dissolved. Insert the needle from a buret contg. a standardized water-in-MeOH back-titrating soln., also insert the pressure-equalizing needle, attach the electrodes to the electrometric app. (cf. McKinney and Hall, C.A. 37, 4934<sup>o</sup>) and titrate slowly until the end point is reached. When applied to various solids and liquids the method gave results reproducible to 0.2-1.5% of the mean when samples weighing 60-300 mg. and contg. 1-20% water were used. For penicillin salt the electrodes are inserted directly into the bottle of the finished product and the detn. is carried out without exposing the compd. to atm. moisture. The detn. is much faster than the usual

dehydration over  $\text{P}_2\text{O}_5$ , and the precision is 3 to 4 times higher. T. H. Dunkelberger

2267?, 1945

Iodometric determination of water by the method of Karl Fischer. Toivo J. Savolainen (Central Rubber Lab., Helsinki, Finland). *Suomen Kemistilehti* 27A, 247-54 (1954).—Experience with the Karl Fischer method (*C.A.* 29, 6532<sup>9</sup>) as modified by Johansson (*C.A.* 41, 7116b) is described. Theory, sensitivity, side reactions, advantages, and applications are discussed briefly. App. for this procedure can be made from readily available lab. materials and the method lends itself to routine analyses but is not recommended for occasional use. John O. Erickson

6778 f, 1955

Determination of moisture by the Karl Fischer method. R. E. Stuckey. *Lab. Practice* 2, 542-8(1953).—A description of the method, titration techniques, and applications. 15 references. J. C. Turner

5731 h, 1954

The application of the Fischer reagent to the determination of moisture in gases. W. Roman and A. Hirst (Petrocarbon Research Labs., Manchester, Engl.). *Analyst* 76, 10-11(1951); cf. *C.A.* 45, 2814i.—A method is described for detg. very small quantities of moisture in air, N, and hydrocarbon gases, even the lower olefins, with the help of the Fischer reagent. The dead-stop end point is used. A suitable glass app. is described. F. I. C.

6308 d, 1953

The microdetermination of water. Axel Johansson (Swedish Forest Products Research Lab., Stockholm). *Acta Chem. Scand.* 3, 1058-66(1949)(in English).—The Fischer method (*C.A.* 29, 6532<sup>9</sup>) is modified by the addn. of Br<sub>2</sub> which oxidizes the HI formed to iodic acid which is detd. by iodometry. Each H<sub>2</sub>O = 12Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. W. R. Trost

5267 g, 1950

Determination of water in hydrogen chloride by means of the Karl Fischer reagent. Ernest C. Milberger, Karl Uhrig, Harry C. Becker, and Harry Levin. *Anal. Chem.* 21, 1192-4(1949).—To prep. the reagent, pass dried SO<sub>2</sub> into a dry bottle contg. 226 g. of pyridine until the gain in wt. is 55 g. Then pour the soln. into another soln. contg. 72.5 g. of pure I in 2 l. of pure, dry MeOH, while keeping the mixt. at 0°. To a mixt. of 40 ml. dry pyridine and 35 ml. dry MeOH in a dry flask, add an excess of the Fischer reagent to make it anhyd. and titrate with a soln. contg. 5 ml. water in 2 l. of MeOH to the yellow end point. Then add a measured vol. of reagent which, must be in excess of that required to react with all the water in the sample. Place the flask in a mixt. of NaCl + ice and pass the HCl gas into the liquid until the gain in wt. is 10-15 g. Titrate the excess reagent with the above H<sub>2</sub>O + MeOH which must be titrated against the reagent, and should have an initial strength of about 1.5 mg. H<sub>2</sub>O per ml. The results are within about 0.5 mg. of the truth. Mercaptans and H<sub>2</sub>S interfere; if they are present a correction must be made. The reagent is standardized against about 20 mg. of water. W. T. Hall

483c, 1950

Determination of water by Karl Fischer reagent.—Stoichiometric iodometric method. Wm. Scaman, W. H. McComas, and G. A. Allen. *Anal. Chem.* 21, 510-12 (1949).—To 950 ml. of ice-cold MeOH add 190 g. of liquid SO<sub>2</sub>. Mix and add 950 ml. of pyridine, slowly at first (soln. A). Dissolve 60 g. of I in 2 l. of abs. MeOH (soln. B). With a pipet, transfer 25 ml. of soln. A to a dry, 125-ml. flask and titrate to an end point with soln. B. Add the sample to another 25-ml. portion of soln. A and again titrate with soln. B. The reaction that takes place is H<sub>2</sub>O + SO<sub>2</sub> + I<sub>2</sub> = SO<sub>3</sub> + 2HI. Standardize soln. A against known wts. of water. W. T. Hall

4975 e, 1949

An automatically operating, coulometric titration method for continually measuring small amts. of water in fluids. E. Barendrecht (Central lab. of the Neth. State Mines, Gellen, Neth.). *Nature* 183, 1181-2(1959).—Water content of liquid samples is automatically detd. by titration with a modified Karl Fischer soln. I is electrolytically generated in exact amts. by anodic oxidation of HI as required for the reaction  $\text{SO}_2 + \text{I}_2 + \text{H}_2\text{O} \rightarrow \text{SO}_3 + 2\text{HI}$ . The magnitude of the generating current is controlled by an amperometric system sensitive to excess I. The app. continuously records the generating current which is proportional to  $\text{H}_2\text{O}$  content. E. Vlahakis

14820 f, 1959

Determination and the solubility of water in liquid sulfur dioxide. Daisaburo Murakami and Niichiro Tokura (Research Inst., Non-aqueous Solns., Sendai). *Bull. Chem. Soc. Japan* 31, 431-4(1958)(in English).—Water in liquid  $\text{SO}_2$  was detd. by a modification of the Karl Fischer method. A reagent mixt. of I and pyridine in MeOH was contacted with the  $\text{SO}_2$  sample. The excess reagent was titrated with a standard water-MeOH mixt. The end point of the titration was detected by the dead-stop method. For samples contg. 5 to 30 mg. of water, the accuracy of the method was  $\pm 0.06$  mg. The soly. of water in liquid  $\text{SO}_2$  was detd. from  $+30^\circ$  to  $-50^\circ$ . From the soly. data, the heat of soln. of water in liquid  $\text{SO}_2$  was 5.04 kcal./mole in the temp. range of  $+30^\circ$  to  $-30^\circ$ . Wesley W. Wendlandt

3992 a, 1959

Determination of moisture with Fisher's reagent. M. S. Shraiber (Sci. Research Chem. Pharm. Inst., Kharkov). *Med. Prom.* 10, No. 2, 44-5(1956).—A method for prepg. a stable Fisher reagent is described. The reagent is prepd. from specially dehydrated MeOH 867, pyridine 269 cc., I 84.7 g., and  $\text{SO}_2$  64 g. The proportions are those of the original Fisher formula. However, S. claims that when prepd. according to Fisher's instructions the titer is not very stable. The following modifications insure stability for 4 months. MeOH was kept over freshly dehydrated  $\text{CuSO}_4$  for 20 days. The filtered methanol was distd. off, kept over freshly calcined CaO for a few hrs., distd. off, and finally rectified by using a column filled with glass beads. Pyridine was warmed with freshly calcined CaO for a few hours, distd. and redistd. over Ca carbide. Iodine was sublimed and dried in dessicator over  $\text{H}_2\text{SO}_4$ .  $\text{SO}_2$  was dried by passing it through a container filled with  $\text{H}_2\text{SO}_4$ . The solns. of  $\text{SO}_2$  in pyridine and that of I in MeOH were kept apart and the necessary amounts mixed before use. A stable reagent can be prepd. according to the following directions: I (not resublimed) 110, pyridine (not abs.) 158,  $\text{SO}_2$  (dried) 72 g., MeOH up to 1000 cc. Both solutions,  $\text{SO}_2$  in pyridine and I in MeOH are mixed immediately and the reagent is stable for 4-6 months. The titer undergoes changes during the first few days. Later on it is sufficient to check it every 5-6 days. A. S. Mirkin

16559 f, 1956

Determination of small quantities of water by the Karl Fischer method. E. P. Bahari (Birmingham Univ., Engl.). *Birmingham Univ. Chem. Engr.* 7, 70-9(1956).—Two different types of app. based on the Karl Fischer titration method and the "dead-stop" end point technique are described. One a double-buret app. was used for samples contg. 0.01%-10%  $\text{H}_2\text{O}$ . The other, an automatic titrator, was used for microdetn. of samples with 0.002%-0.01%

$\text{H}_2\text{O}$ . A technique for the exclusion of atm. moisture during the manipulation of samples is also described. I. W. H.

16559 i, 1956

Microdetermination of water in a gaseous sample by the Karl Fischer method. Kaname Muroi (Mitsubishi Chem. Ind. Ltd., Kawasaki). *Bunseki Kagaku* 10, 847-50(1961). —An improved app. was developed for the microdetn. of H<sub>2</sub>O in a gaseous sample (0.5-1.0 mg. H<sub>2</sub>O/ml.). The sample was passed through 100 ml. of anhyd. MeOH at <1 l./min., the H<sub>2</sub>O was quant. absorbed, and titrated as usual in the same vessel. The time required for an analysis was shortened by about half, the operation became very simple, and results were accurate. For example, 0.004% H<sub>2</sub>O in N gas was detd. in about 1 hr. with good precision.

Tadashi Hara

6664 a, 1962, V. 56

Continuous measurement of traces of water in liquids by automatic coulometric titration. E. Barendrecht and J. G. F. Doornekamp (Staatsmijnen, Geleen, Neth.). *Z. Anal. Chem.* 186, 176-80(1962). App., solns., and procedure for the continuous detn. of p.p.m. of H<sub>2</sub>O in org. liquids by anodic generation of I<sub>2</sub> for the Karl Fischer reagent are described.

K. G. Stone

9212 f, 1962, V. 57

Karl Fischer determination of water in ammonium perchlorate with automatic titration apparatus. Evaluation of reaction rate parameters and statistical evaluation. Eugene A. Burns and R. F. Muraca (Stanford Res. Inst., Menlo Park, Calif.). *Anal. Chem.* 34, 848-54(1962). The detn. of H<sub>2</sub>O in NH<sub>4</sub>ClO<sub>4</sub> by the Karl Fischer method with the use of automatic titration equipment was studied as a result of the wide divergence of results from different labs.; a Beckman Model KF-2 Aquameter was used. The major instrumental factor is the lack of anticipation of the end point, which is influenced by the feed rate of the titrant, the rate of stirring, the position of the electrodes, the vol. of soln., polarization of the electrodes, the amt. of H<sub>2</sub>O to be titrated, and the selection of the end point. The major chem. factors are the finite rate of reaction between the Karl Fischer reagent and H<sub>2</sub>O, and the very small amt. of H<sub>2</sub>O present; in a 3:1 pyridine-MeOH mixt., the rate of reaction increases. A description is given of an attachment suitable for visual observation or the recording of dead-stop end points in conjunction with the impedance detection system of the aquameter. Surface H<sub>2</sub>O is detd. by the loss in wt. at 110°, and also by Karl Fischer titration of a sample placed in a H<sub>2</sub>O-free satd. MeOH soln. of NH<sub>4</sub>ClO<sub>4</sub>. Internal H<sub>2</sub>O is detd. by Karl Fischer titration after soln. of the oven-dried material in dry pyridine-MeOH solvent. Results of analysis of 2 samples of NH<sub>4</sub>ClO<sub>4</sub> with 0.01 and 0.05% H<sub>2</sub>O by 9 labs. have reproducibilities of 0.0018 and 0.0022% and repeatabilities of 0.0008 and 0.0014%, resp.

R. E. Stanton

4036 b, 1962, V. 57

Determination of water in liquid hydrogen fluoride. M. M. Raines. *Zavodskaya Lab.* 14, 284-6(1948). —*Procedure:* Sat. 48 g. of dry pyridine with SO<sub>2</sub> until it gains 14 g. in wt. Add a soln. of 15 g. I in 150 ml. abs. MeOH; standardize the reagent against MeOH contg. a known quantity of H<sub>2</sub>O. In a Bakelite cylindrical vessel with a screw-cap carrying a perforated baffle, place 50 ml. of abs. MeOH. Pour the liquid HF into a chilled Cu vessel (below 10°), weigh after closure (Cu lid), and place the Cu container on the perforated baffle of the Bakelite vessel, which is then hermetically closed and inverted several times. After a few min., remove the cover and pour the HF-MeOH soln. into a 100-ml. volumetric flask coated with paraffin. Add dry MeOH to the 100-ml. mark and use 5-10 ml. aliquots for the titration with the Fischer reagent (end point—darkening of soln.). If the soln. is too dark, use potentiometric method with Pt and W electrodes in a closed vessel; the break is 20-25 mv.

G. M. Kosolapoff

971 g, 1949

Experimental parameters of the Beckman Model KF-3 aquameter. Dennis R. Gere and Clifton E. Meloan (Kansas State Univ., Manhattan). *Anal. Chem.* **35**, 597-9(1963). Exptl. parameters of the Beckman Model KF-3 aquameter were measured by titrating H<sub>2</sub>O in org. solvents with Karl Fischer Reagent (KFR). The concn. range was 20-50 mg. of H<sub>2</sub>O/ml. of sample soln. The feed rate was 7.5 ml. of KFR/min. The vol. of soln. at the end point was approx. 19 ml. The neutralized reaction mixt. was removed from the vessel after each titration by a siphon tube. The timer was set at the 30-sec. position. The reproducibility of direct titration with KFR was compared with that of back-titration with aq. MeOH soln. (I). For direct titration; the KFR/I ratio was  $0.983 \pm 0.012$ , and for a different set of solns. was  $1.356 \pm 0.043$  by back-titration. The standard deviation of a set of direct titrations of the H<sub>2</sub>O content of CHCl<sub>3</sub> is  $\pm 0.0003$ , 1-decanol  $\pm 0.0044$ , MeOH  $\pm 0.0029$ , and PhNO<sub>2</sub>  $\pm 0.0019$ .  
Merle I. Eiss

13136 e, 1963, V. 58

Determination of water content in potassium difluoride. Giovanni Barbi and Sergio Pizzini (Euratom C. C. R., Ispra, Italy). *Anal. Chem.* **35**, 400-10(1963). To distinguish H<sub>2</sub>O formed by esterification of HOAc, the solvent of KHF<sub>2</sub> in the Karl Fischer H<sub>2</sub>O detn., the following technique was developed. A HOAc soln. of KHF<sub>2</sub>, contg. <0.1% H<sub>2</sub>O, was introduced into a cell contg. dehydrated pyridine. A const. current of 2.5  $\mu$ amp. was imposed, and the voltages measured when titrated. Small, const. quantities of Karl Fischer (KF) reagent were added to the mixt. each time the voltage exceeded 250 mv. The titration was recorded as a curve of transition time,  $\tau$ , vs. vol. of KF, and compared with the titration of a blank. The inflection points of the curves were taken as references. The H<sub>2</sub>O content was calcd. by  $\frac{[(G + G_s)n - (d_s/d_a)(G_s n')]}{T} \cdot \frac{1}{10Vd_s}$ , where  $n$  and  $n'$  are cc. of KF, resp., for sample and blank,  $G$  is g. of salt,  $G_s$  is g. of HOAc,  $T$  is titer of KF in mg. H<sub>2</sub>O/cc.,  $V$  is the vol. introduced into the cell, and  $d_s$  and  $d_a$  are resp. ds.  
J. U. Veiderma

10728 d, 1963, V. 58

The determination of moisture in acetone. N. K. Mikhailyan, A. V. Sokolov, and G. A. Sedova. *Zavodsk. Lab.* **29**(9), 1058 (1963). To decrease the titer of the soln. used in the detn. of H<sub>2</sub>O in acetone, 30 cc. MeOH, 28.23 g. I, 28.7 cc. C<sub>2</sub>H<sub>5</sub>N, and 15 cc. SO<sub>2</sub> are used; the titer of this reagent is 3.63 mg./cc. Titrate 5 cc. of a dry: ..... C<sub>2</sub>H<sub>5</sub>N) with the titrant until a distinct I color ..... tone sample and titrate again to the same color .....  
C. Weiner

14579 a, 1963, V. 59

Determination of the water required to hydrolyze the chlorination products of acetic acid and acetic anhydride mixtures. Andres Huesca Moreno and Oscar Hector Sagese. *Ind. y quim.* (Buenos Aires) **19**, 166-74(1959).—The Karl Fischer method for detg. H<sub>2</sub>O is used. A sample contg. monochloroacetic acid and chloroacetyl chloride (I) is melted at 70° and stirred; exactly 50 g. of the sample is weighed out in a flask and a known amt. of H<sub>2</sub>O in excess (about 1.5 g. total) is added to hydrolyze I; the sample is refluxed at 70° for 15 min. while stirring. The cooled sample is dissolved in anhyd. dioxane making up to 200 ml. With the Karl Fischer method with an equiv. of at least 3 mg. water/ml., anhyd. MeOH, and a 5-ml. aliquot of sample, the free water in the sample is detd. and the amt. of I is calcd. The error is  $\pm 0.06\%$  by wt. water.  
Thomas Garcia-Borras

3054 i, 1960

Determination of water by the Karl Fischer method. F. O. Pierat, G. N. Meier, and A. I. Brieva. *Anales Real Acad. Farm.* 28, 297-309(1962). A review with 23 references.

S. Barabas

12136e, 1963, V. 58

The determination of the water content of the low polymers of formaldehyde by the Karl Fischer method. I. The determination of the adsorbed water and bound water liberated by pyrolysis of the polymer. Kaname Muroi and Kunihiko Ogawa (Mitsubishi Chem. Ind. Ltd., Kanagawa). *Bull. Chem. Soc. Japan* 36(8), 965-9(1963). The adsorbed water was detd. by extn. with dry MeOH and Karl Fischer titration. Total water was obtained by pyrolysis of the polymer in propylene glycol and subsequent titration.

Richard L. Every

14113a, 1963, V. 59

Determination of water and alkali metal oxide in alkali metal salts of selenium and tellurium by Karl Fischer titration. Peter Tao-I Chiang and W. Charles Cooper (Noranda Res. Center, Pointe Claire, Can.). *Anal. Chem.* 35(11), 1693-5(1963). Anhyd.  $\text{Na}_2\text{SeO}_3$ ,  $\text{K}_2\text{TeO}_3$ , and  $\text{Na}_2\text{H}_4\text{TeO}_6$  behave like a mixt. of the resp. oxides, and the M<sub>2</sub>O (M = Na, K) content in these salts reacts quant. with Karl Fischer reagent according to:  $\text{Na}_2\text{SeO}_3 + \text{I}_2 + 3\text{SO}_2 + 3\text{CH}_3\text{OH} \rightarrow 2\text{NaI} + \text{Se} + 3\text{HSO}_3\text{CH}_3$ ,  $\text{K}_2\text{TeO}_3 + \text{I}_2 + 3\text{SO}_2 + 3\text{CH}_3\text{OH} \rightarrow 2\text{KI} + \text{Te} + 3\text{HSO}_3\text{CH}_3$ , and  $\text{Na}_2\text{H}_4\text{TeO}_6 + 3\text{I}_2 + 6\text{SO}_2 + 6\text{CH}_3\text{OH} \rightarrow 2\text{NaI} + 4\text{HI} + \text{Te} + 6\text{HSO}_3\text{CH}_3$ . The titer for  $\text{Na}_2\text{SeO}_3 \cdot 5\text{H}_2\text{O}$ ,  $\text{K}_2\text{TeO}_3 \cdot 2\text{H}_2\text{O}$ , and  $\text{Na}_2\text{H}_4\text{TeO}_6 \cdot 2\text{H}_2\text{O}$  is a measure of the combined M<sub>2</sub>O and H<sub>2</sub>O content. The latter was calcd. either by assuming the sample is composed of pure anhyd. salt and H<sub>2</sub>O only, or by estg. the M<sub>2</sub>O content sep. and obtaining the H<sub>2</sub>O content by difference. The results agree well with the H<sub>2</sub>O content found from wt. loss on thermal drying although the thermal drying procedure is more time-consuming. Anhyd.  $\text{Na}_2\text{SeO}_3$  is not affected by Karl Fischer reagent and the titer for  $\text{Na}_2\text{SeO}_3 \cdot 10\text{H}_2\text{O}$  is derived exclusively from H<sub>2</sub>O content. The prepn. of  $\text{K}_2\text{TeO}_3 \cdot 2\text{H}_2\text{O}$  crystals is not reported previously in the literature. It was prepd. by adding EtOH to a satd. soln. of  $\text{K}_2\text{TeO}_3$  and allowing the soln. to stand for 1 month.

Peter Tao-I Chiang

13353e, 1963, V. 59

Determination of water in liquid hydrogen fluoride. N. S. Nikolaev and I. F. Alenchikova. *Zavodskaya Lab.* 24, 418-19(1958).—The method is based on the fixation of HF by KHF, whereby acid salts, which correspond to the phase diagram of the system HF-KF, are formed. To 1.5-5 g. KHF, in the Teflon vessel of a titration unit, is added 0.2-0.6 g. liquid HF taking care to distribute the acid evenly over the layer of KHF. The cover is then changed for one fitted with an electrode and stirrer, and the titration cell is placed under burets contg. the Fisher reagent, anhyd. MeOH, and the standard soln. of H<sub>2</sub>O in MeOH. Titration is carried out by the addn. of small portions of the titrating solns. The content of H<sub>2</sub>O is calcd. by the formula  $[mT_p - n \cdot T_b/v - p \cdot T_a.] \cdot 100/\text{wt. of sample}$ , where *m*, *n*, *p* are the vols. of the solns. of the Fisher reagent, the anhyd. and the standard MeOH, resp., and *T* are the titers of the corresponding solns. For the prepn. of one l. of the Fisher reagent, 84.7 g. iodine is dissolved in 269 ml. pyridine, 665 ml. MeOH is added, and to the soln. obtained, 45 ml. condensed SO<sub>2</sub> is added under intense cooling.

Jean Plamondon

12882f, 1960

Determination of water in red fuming nitric acid by Karl Fischer titration. E. A. Burns and R. F. Muraca (California Inst. of Technol., Pasadena). *Anal. Chem.* 35(12), 1967-70 (1963). Use of the Karl Fischer titration for the direct detn. of H<sub>2</sub>O in red fuming HNO<sub>3</sub> (RFNA) was investigated, and a modified procedure was developed whereby H<sub>2</sub>O can be successfully detd. in RFNA samples contg.  $\leq 15\%$  NO<sub>2</sub>. The method controls the exptl. variables in order that the following reactions are stoichiometric:  $H_2O + 2NO_2 \rightarrow HNO_2 + HNO_3$ ;  $HNO_2 + \frac{1}{2}I_2 + SO_2 + CH_3OH \rightarrow HI + NO + HSO_3CH_3$ . The pooled standard deviation (40 degrees of freedom) is 0.052% abs. and the error is 0.042% abs. The method is applicable when HF and large amts. of metal contaminants are present. E. V. Jones

24 e, 1964, V. 60

Determination of water in ketones by the Karl Fischer method. Kaname Muroi, Kunihiko Ogawa, and Yaeko Ishii (Mitsubishi Chem. Ind. Ltd., Kanagawa Prefect., Japan). *Bunseki Kagaku* 12(10), 963-5(1963). As the ketones produce H<sub>2</sub>O in accordance with  $R_2CO + 2MeOH \rightarrow R_2C(OMe)_2 + H_2O$  in the conventional method, the following conditions are recommended for detn. of H<sub>2</sub>O in Me<sub>2</sub>CO, MeEtCO, MePhCO, and cyclohexane etc.: 30 ml. mixt. (1 + 1) of propylene glycol and pyridine as a solvent, titrate at 10 ~ 15° sample soln. <20 ml., total H<sub>2</sub>O content in sample <50 ml. The error is <1%. Tadashi Hara

2327 b, 1964, V. 60

Moisture content of propellants. Mantaro Uetake and Junichi Nomura (Asahi Chem. Ind. Co., Sakanoichi, Japan). *Kogyo Kagaku Kyokaiishi* 23(6), 302-7(1962). Several factors affecting the equil. moisture content of propellant grains were studied. The results obtained are as follows: (1) The history of humidification and dehumidification of the propellant has a serious effect on moisture content. Even at the same relative humidity, a propellant dehumidified under lower relative humidity shows a lower moisture content, while one humidified under high relative humidity shows a higher value. (2) A propellant having a higher content of residual solvents shows higher equil. moisture, and one having a lower content is lower. Apparently, one of higher moisture content has more non-cryst. regions as compared to one of lower moisture content, since non-cryst. regions supposedly have much affinity for moisture. The method of detg. moisture content of propellants is given. The Karl Fischer method is preferred for the detn. of small amts.

Yutaka Majima

1525 g, 1964, V. 60

The Karl Fischer method for the determination of the moisture in some reagents. G. D. Glebova and G. N. Kosheleva. *Trudy Vsesoyuz. Nauch.-Issledovatel. Inst. Khim. Reaktivov* 1958, No. 22, 115-18.—The Karl Fischer method was used to det. moisture in some org. and inorg. reagents (FeCl<sub>2</sub>·4H<sub>2</sub>O, Ca(NO<sub>3</sub>)<sub>2</sub>·nH<sub>2</sub>O, Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, o-sulfobenzoic acid, and methyl terephthalate). Moisture could also be detd. in reagents insol. in the abs. MeOH. The pulverized reagent sample (~1 g.) was suspended in 50 ml. abs. MeOH and brought to boiling with a reflux condenser, on the upper end of which the CaCl<sub>2</sub> pipe was placed. When cooled to room temp., some of the matter in the upper layer was removed and titrated with the Karl Fischer reagent. In the detn. of moisture in Ba or NH<sub>4</sub> hexacyanoferrates, boiling with the abs. MeOH completely extd. the moisture in 30 min. The accuracy was confirmed by analyzing artificially moistened samples. From *Referat. Zhur., Khim.* 1959, Abstr. No. 49196. O. Boshko

24095 a, 1960

Determination of small amounts of water in hydrocarbons. L. N. Petukhova and V. A. Fomenko. *Azerb. Khim. Zh.* 1961(6), 121-5; cf. Loweland, *et al.*, *CA* 58, 16730h. In a special app., H<sub>2</sub>O is detd. in C<sub>6</sub>H<sub>6</sub> by the Karl Fischer method. With 11-70 p.p.m. H<sub>2</sub>O in C<sub>6</sub>H<sub>6</sub>, the relative error is 0.25-9.8%. With <8 p.p.m. H<sub>2</sub>O, the relative error is 20%. Eurilla Mayerle

7276 b, 1963, V. 59

Determination of moisture. Louis Brissaud. *Chim. anal.* 33, 159-69(1951).—The numerous methods for detg. H<sub>2</sub>O in various states are reviewed critically with particular attention to the very satisfactory procedure of Karl Fischer. Two reagents are necessary. (1) A soln. of 66.4 g. anhyd. MeOH, 269 ml. of pyridine, 81.4 g. of pure I, and 64 g. SO<sub>2</sub>. One ml. of this soln. equals about 6 mg. H<sub>2</sub>O but it must be standardized daily and be at least 48 hrs. old when used. (2) A soln. in MeOH of 0.02-0.03 g. H<sub>2</sub>O added as such or as a salt contg. H<sub>2</sub>O of crystn. The sample is treated with a known vol. of Soln. 1 and the excess is

titrated electrometrically with Soln. 2 until the excess I is removed. The reaction involved is:  $(C_5H_5N)_2SO_2 + C_5H_5N + CH_3OH + I_2 + H_2O = 2C_5H_5N.HI + C_5H_5N.HSO_3CH_3$  which, however, is only about 80% true. 31 references.  
W. T. Hall

6124h, 1951

Continuous determination of water. Stamicarbon N. V. Dutch 96,458, Dec. 15, 1960. Addn. to Dutch 94,380 (CA 55, 11966a). To prevent fouling of the electrodes, KI is replaced by HI or an iodide of an org. base.

C. A. Goethals

18456g, 1961

Apparatus for continuous determination of water. Stamicarbon N.V. Dutch 94,380, May 16, 1960. The app. contains a measuring cell, divided into 2 parts by a sintered glass plate. In both parts of the cell, a Pt electrode is placed. The sample, mixed with Karl Fischer reagent, contg. KI instead of I, is fed continuously to the part of the cell which contains the anode. Here excess I is liberated. Part of this I, in an amt. corresponding to the % H<sub>2</sub>O present, is converted into KI again. The remaining concn. of I is measured potentiometrically or amperometrically. For example, 0.1-0.5% H<sub>2</sub>O in a urea melt was detd. with a relative accuracy of 2-5%.

C. A. Goethals

11966a, 1961

Determination of the water content of flour and flour products by the iodometric method according to Fisher. Jan Hampl and Vladimir Rasper (Vysoka Skola Chem.-Technol., Prague). *Sb. Vysoke Skoly Chem.-Technol. Praze, Oddil Fak. Potravinarske Technol.* 4, Pt. 2, 365-78(1960). The period necessary for extrn. when MeOH was used was 20-30 min. Optimal results for the detn. of water in pastry were achieved when the sample was made fine until the size of the particles did not exceed 500  $\mu$ . Extn. was carried out for 1 hr. at 45°. Detn. in wet gluten was not convenient due to the work required for the prepn. of the sample.  
H. Mlodzikova

11278c, Vol. 60



Automatic apparatus for the extraction and determination of water by the Karl Fischer reagent. Giorgio Peyronel and Domenico De Filippo (Univ. Modena, Italy). *Ann. Chim. (Rome)* 53(10), 1427-34(1963). The automatic app. enables the hot or cold extn. of H<sub>2</sub>O from samples either insol. in MeOH or other anhyd. solvents or contg. materials which interfere with the Karl Fischer reaction and are to be simultaneously pptd. by appropriate reagents, the running of reactions producing stoichiometric amts. of H<sub>2</sub>O, the transfer of the H<sub>2</sub>O thus obtained to the titration cell after filtration and its dead-stop titration against Karl Fischer reagent. Using 25- and 10-ml. burets, characteristic data are, resp. (ml. unless otherwise specified); cell vol. 150, 100; solvent vol. 15, 10; mg. H<sub>2</sub>O/ml., reagent 5-6; optimum amt. H<sub>2</sub>O in sample 50-100, 20-40 mg.

J. De Feo

8605 b, Vol. 60

Dead-stop determination of traces of water in aliphatic ketones with Karl Fischer solution. F. Fischer and R. Schiene (Tech. Univ., Dresden, E. Ger.). *Z. Chem.* 4(2), 69-70(1964). Water in acetone (0.1-0.4%) and water (0.4-0.8%) in MeCOEt are detd. by the Karl Fischer method with a max. error of 1%.

B. T. Kho

13890 k, Vol. 60

Determination of small quantities of water of hydration in some transition metal halides. L. Giuffrè (Politecnico, Milan). *Chim. Ind. (Milan)* 46(1), 51-3(1964). Water was precisely detd. for com.  $\alpha$ -TiCl<sub>3</sub> (0.14-0.90 wt. % H<sub>2</sub>O) and VCl<sub>3</sub> (0.12-0.38 wt. %) by a method useful for other inorg. salts sol. in nonaq. solvents. The salt was dissolved in a known vol. of anhyd. benzene-EtOH of known H<sub>2</sub>O content, then distd. under dry, inert gas until about 80% of the solvent was collected in a receiver at -20°. Anhyd. MeOH was added and the soln. was titrated potentiometrically with Fischer reagent. M. A. Aia

12658 g, Vol. 60

The determination of water in granular fertilizers by a modified Karl Fischer method. I. Johnston and E. J. Smith (Fisons Fertilisers Ltd., Ipswich, Engl.). *Chem. Ind. (London)* 1964 (8), 315. The principle employed is to ext. material for standard periods with solns. of low H<sub>2</sub>O solvent power and then to titrate portions of the filtered ext. with Karl Fischer reagent (Mitchell and Smith, *Aquametry*, 1948, 450 pp. (CA 43, 522g). Various mixts. of dry MeOH-CHCl<sub>3</sub> were used as extractants; extn. time was 2 hrs. (although extn. is usually complete in 1 hr.). It is possible to reproduce the results of various standard oven-drying methods by choosing a suitable extractant. L. Dupuis

13837 f, Vol. 60

Determination of moisture in small samples of offset inks. S. A. Shelukhina and M. N. Savosina. *Poligr. Proizv.* 1963(10), 19-21. H<sub>2</sub>O has a detrimental effect on printing properties of ink; it is therefore important to have a good method for moisture detn. In practice, samples are taken from inking rollers and their wt. is about 0.01-0.3 g. The small sample wt. precludes use of the Dean-Stark method conventionally used for detn. of moisture in inks and requiring about 10.0 g. material. Such small samples can be conveniently analyzed by a modified Fisher method. The modification comprises the use of an electric end-point indicator, based on measurements of polarization e.m.f. The modified method was successfully used on a large no. of samples and the observed relative error of detn. was <5%. George Chaplenko

4341a, Vol. 60

Determination of moisture in ion-exchange resins by the Karl Fischer reagent. Frank N. Pollio (Rohm & Haas Co., Philadelphia, Pa.). *Anal. Chem.* 35(13), 2164-5(1963). The detn. of H<sub>2</sub>O in ion-exchange resins in MeOH or pyridine soln. by direct titration with the Fischer reagent (CA 29, 6532<sup>9</sup>) is described. The exptl. results are compared with those obtained by oven-drying and by azeotropic distn. The oven-drying and the distn. methods give low results, which are attributed to the incomplete removal of H<sub>2</sub>O from the internal pores. The precision of the titration method is claimed as  $\pm 0.75\%$ , with no measurement differing by more than 1.5% from the mean. The titration method thus overcomes the limitations of the oven-drying and distn. methods, esp. at low H<sub>2</sub>O concns. and for the detn. of tightly bound residual H<sub>2</sub>O. L. C. Thomas

6985d, Vol. 60

Karl Fischer reagent for the determination of water in condensates of sodium tetraborate and diols. Charles B. Jordan (Aberdeen Proving Ground, Md.). *Anal. Chem.* 36(2), 424-5 (1964). Karl Fischer reagent does not react with Na tetraborate that is chem. bound in a diol condensate, e.g. with ethylene or propylene glycol. Karl Fischer reagent can thus be used to det. free H<sub>2</sub>O in such condensates, and the degree of condensation can be estd. F. Learmonth

9878g, Vol. 60

Apparatus for the macro- and semimicro-estimation of water according to Karl Fischer. G. Hesse and W. Herb (Inst. Mikrobiol. Expt. Therap., Jena, Ger.). *Chem. Tech. (Berlin)* 15(11), 690-1(1963). A new Karl Fischer buret can be completely closed against atm. moisture for use in macro (limit 5 ml. soln.) and semimicro (limit 1 ml. soln.) estns. Dorothy M. Goode

7426d, Vol. 60

Rapid titrimetric determination of water and SO<sub>2</sub> in spent acid left in DDT manufacture. I. Dragusin and D. Trifu. *Rev. Chim. (Bucharest)* 15(3), 164(1964); cf. CA 56, 5402a. To det. H<sub>2</sub>O: to 0.5-0.8 cc. of spent H<sub>2</sub>SO<sub>4</sub> add gradually 1:22 pyridine:dioxane, with continuous cooling, and titrate with Karl Fischer reagent until the color changes from yellow to red-brown. Atm. humidity is excluded by covering the flask with a perforated stopper during the titration. *p*-Chlorobenzenesulfonic acid does not interfere. To det. SO<sub>2</sub> equiv.: to 2 cc. of H<sub>2</sub>O add gradually with continuous cooling 20-5 cc. residual oleum, weigh, homogenize, withdraw a small aliquot (0.5-0.8 cc.) and continue as above to det. excess H<sub>2</sub>O. Calc. SO<sub>2</sub> by difference. M. Lapidot

1273g, Vol. 61

Determination of external water in white sugars by direct titration with Karl Fischer reagent. R. G. Bennett, R. E. Runckles, and H. M. Thompson (Tate Lyle Refineries, Ltd., Keston, Engl.). *Intern. Sugar J.* 66(784), 109-13(1964). The external or surface water of white sugars can be detd. by direct titration of a slurry of sugar and dry MeOH with Karl Fischer reagents. The results are in agreement with those by a vacuum-distn. method. Both the vacuum distn. method and the Karl Fischer method show that the sugar will not release all the available surface water before a maturing period of 8-16 days. Elwin E. Harris

2039b, Vol. 61

Van der Meulen's Reagent (NaI, NaOAc, I, SO<sub>2</sub>, MeOH)

Determination of water in alcohols with Van der Meulen's reagent. Zygmunt Lada (Zakład Anal. Inst. Chem. Ogólnej, Warsaw). *Chem. Anal. (Warsaw)* **3**, 663-5(1958) (English summary).—The Van der Meulen reagent (Brit. 728,947, *C.A.* **49**, 13028c) was used for detg. water traces in dehydrated EtOH, iso-BuOH, polyoxymethylene I, polyoxymethylene II, ethylene glycol, diethylene glycol, triethylene glycol, iso-PrOH, benzene, toluene, and CHCl<sub>3</sub>. The results were compared with those obtained by the Fischer reagent (*C.A.* **29**, 6532<sup>2</sup>), modified by Seaman, *et al.* (*C.A.* **43**, 4975c) and agreement was obtained. Z. Kurtyka

13873 A, 1959

Reagent for the determination of very small amounts of water in gases, liquid, and solid substances. Johannes Hendrik van der Meulen. Brit. 728,947, Apr. 27, 1955. The iodometric titration of H<sub>2</sub>O can be performed with a reagent prepd. by dissolving 25 g. NaI, 85 g. anhyd. NaOAc, and 63.5 g. I in 600 cc. MeOH, adding 90 cc. 4M SO<sub>2</sub> in MeOH, and then dilg. to one l. with MeOH. Other salts of weak org. acids can be used. Such reagents are stable and produce no side reactions in the detn. of H<sub>2</sub>O. Sidney Gutcho

13028 C, 1955

Reaction with  $\text{CaH}_2$  to form  $\text{H}_2$

The determination of water with calcium hydride. A. L. Gol'dinov, V. I. Lukhovitskii, and G. Z. Srubinskaya. *Zh. Analit. Khim.* 17, 532-4(1962); cf. *CA* 57, 9212i. To improve accuracy in the detn. of  $\text{H}_2\text{O}$  with  $\text{CaH}_2$ , the reaction is carried out in an app. consisting of a calibrated gas buret and a reaction test tube at 550-600°. This ensures stoichiometric evolution of  $\text{H}_2$ .  
C. Weiner

9326, 1963, V. 58

Determination of water content in ethanol by gas chromatography. Tomotsune Taira (Kyoto Women's Coll., Japan). *Hakko Kyokaishi* 19, 409-10(1961). Com. alc. was analyzed by gas chromatography for  $\text{H}_2\text{O}$  content. Com. chem. pure alc. contained 0-0.5%  $\text{H}_2\text{O}$ .  
Teiti Narasaki

121909, 1963, V. 59

Gas volumetric determination of moisture in xanthates. E. M. Idel'son. *Byull. Tsvetnoi Met.* 1958, No. 7, 15-7.—The detns. of moisture were carried out by allowing the sample to react with  $\text{CaH}_2$  in anhyd. dioxane, and measuring the vol. of evolved  $\text{H}_2$ . The app. consists of a reaction flask connected to a gas buret, and a leveling bottle filled with  $\text{H}_2\text{O}$ . A table of results obtained by this method and by drying to const. wt. at 80° is given.

I. Dobrucka

2121, 1961

New method of physicochemical investigation of phases in mixtures. L. G. Berg. *Izvest. Sektora Fiz.-Khim. Anal., Inst. Obshchei i Neorg. Khim., Akad. Nauk S.S.S.R.* 19, 249-55(1949).—The two methods described are gas-volumetric and thermographic. The first of these consists of heating a mixt. stepwise at definite temps. and detg. the vol. of gas evolved in each step. Each of the components of the mixt. liberates a gas ( $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{NH}_3$ ,  $\text{O}$ , etc.) at some definite temp. and the amt. of the component is calcd. from the vol. of gas evolved. The gas vol. is detd. in a specially designed gas buret. Samples of 0.05-0.2 g. are suitable for this procedure (cf. *C.A.* 44, 10382e). Since detn. of  $\text{H}_2\text{O}$  vapor is difficult because it condenses, the vapor is passed through  $\text{CaH}_2$ , and the equiv. vol. of  $\text{H}_2$  given off is measured. The thermographic method consists of detg. the thermal effect of an analyzed substance to which was added a known quantity of substance of which the thermal effect is known. The quantity of sought substance is calcd. from the ratio of the areas of the 2 thermal effects.

M. Hoseh

18301, 1951

Determination of water by means of  $\text{CaH}_2$ . P. W. Perryman. *Analyst* 70, 45-7(1945).—The detn. of  $\text{H}_2\text{O}$  in small samples by means of  $\text{CaH}_2$  is often advantageous, but abs.  $\text{EtOH}$  as an intermediary liquid is unsatisfactory

because it also evolves  $\text{H}_2$  when in contact with  $\text{CaH}_2$ . Expts. show that anhydrous dioxane is more satisfactory. Satisfactory results have been obtained with small quantities of blood and with dioxane exts. of solid powders. Difficulty may possibly arise with large quantities of lipoidal substances, such as fats, as these will also be extd. by dioxane and lead to low results. Such difficulties can be overcome by using a strictly uniform technique and an empirical calibration curve.  
W. T. Hall

18189, 1945

Some new, very sensitive methods based on the measurement of gaseous volumes. LOUIS HACKSPILL AND GEORGES D'HUART. *Ann. chim.* 5, 95-133(1926).—*I. New volumetric method of elementary analysis.*—In a single sample weighing 20 mg., it is possible to det. C, O and N with remarkable accuracy. The principle of the method consists in heating the org. substance in an evacuated vertical tube. The substance is covered with a column of CuO to insure complete oxidation and on top of this there is a reduced Cu spiral to insure reduction of oxides of N. The evolved gases are chilled to  $-80^{\circ}$ , which causes congelation of H<sub>2</sub>O. The residual gas is measured before and after the CO<sub>2</sub> is absorbed by KOH and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>. From the chilled tube, H<sub>2</sub>O is allowed to evap. and after contact with CaH<sub>2</sub>, the liberated H<sub>2</sub> is measured. *II. Detn. of water in a gas mixture.*—The principle of the method is the same—condensation of the water by evacuating and chilling, contact with CaH<sub>2</sub>, and measurement of the liberated H<sub>2</sub>. In order to get accurate results with small samples it is necessary to take special precautions, which are enumerated in detail. *III. New method for determining carbon in steel.*—The steel is dissolved in 6 N HCl, a very strong soln. of CrO<sub>3</sub> is added and the system is evacuated. The gases pass through a condenser, as in the Corleis method then through a tube chilled to  $-80^{\circ}$ , which condenses H<sub>2</sub>O and Cl<sub>2</sub>, then through a hot furnace contg. CuO, and finally into a tube chilled to  $-190^{\circ}$ , which condenses the CO<sub>2</sub>. As compared with the Corleis method, the method is not unduly tedious, but it cannot be as rapid as the dry combustion method now in use in most steel labs. W. T. H.

1368, 1926

Determination of water content by means of calcium hydride. OLAV NØTEVÅR. *Z. anal. Chem.* 80, 21-56(1930).—The usual methods for detg. H<sub>2</sub>O are described and criticized. References to the literature of the subject are given. An app. is described and numerous analytical results are given for a method which depends upon the fact that CaH<sub>2</sub> reacts with water to give H<sub>2</sub> gas which can be measured best over pure glycerol. W. T. H.

4731, 1930

Reaction to produce H<sub>2</sub>  
Reagent not given in abstract

Devices for determining the content of water vapor in a gas flow. Bernard Brasseur, Georges L. Gargasson, and Jean R. Perilhou (to North American Philips Co. Inc.). U.S. 3,046,098 (Cl. 23-254), July 24, 1962; Fr. Appl. Nov. 4, 1957; 2 pp. In a device for measuring the content of H<sub>2</sub>O vapor in a gas flow which might bring about corrosion, the free O in the gas flow is removed if the measurement would be disturbed by it. The free H is measured or removed sep. and the H<sub>2</sub>O in the gas flow is decompd. with chem. binding of the O and, subsequently, the pressure of the H is detd. after diffusion through a semipermeable wall, e.g., of Pd. Betty L. McCully

1914 g, 1963, v. 58

## Reaction with 2,2-Dimethoxypropane to form Acetone

Determination of water in organics by gas chromatography. Maurine Hager and Graeme Baker (Montana State Coll., Bozeman). *Proc. Montana Acad. Sci.* 22, 3-4 (1963). The H<sub>2</sub>O content in org. compds. is detd. by measuring the acetone formed by reaction of H<sub>2</sub>O with 2,2-dimethoxypropane; acetone is detd. on a column operated at 50-60° and a He flow of 70-80 ml./min. Five- $\mu$ l. samples of solns. from a mixt. composed of 1 ml. MeOH, 0.9 ml. of 2,2-dimethoxypropane, and 3  $\mu$ l. of CH<sub>2</sub>SO<sub>2</sub> plus zero to multiple 10 $\mu$ l. increments of H<sub>2</sub>O are injected into the gas chromatograph. The addns. of H<sub>2</sub>O result in a proportional increase in acetone peaks recorded. Similar tests were made with PhMe. After standards are established, the detn. of ~0.03-5% H<sub>2</sub>O in org. materials can be made in approx. 10 min.

D. Fitz-Gerald

8129 h, 1963, V.59

Reaction with  $Mg_3N_2$  to form  $NH_3$

Determination of small quantities of water in gases and vapors. Karl Ackermann (to I. G. Farbenindustrie A.-G.). Ger. 719,318, March 5, 1942 (Cl. 42l. 4.16). A const. stream of the gas or vapor is brought in contact first with a nitride, which at ordinary temps. gives off  $NH_3$ , when in contact with  $H_2O$  and then with a weak acid soln. capable of forming well-conducting  $NH_4$  salts. The cond. of this soln. is then detd.

1954<sup>3</sup>, 1943

Δ Determination of moisture in solids. F. Roth and A. Schnoor. *Brennstoff-Chem.* 22, 98-100(1941); cf. C. A. 34, 7780<sup>7</sup>.—Introduce 2 g. (1 g. for a water content over 2%) of solid sample into a drying tube forming part of a train consisting of a suitable app. for drying  $N_2$ , a  $Mg_3N_2$  tube and wash bottle contg. 50-60 cc. of 0.05 N HCl. Insert the drying tube into a water bath maintained at 80-90° and allow  $N_2$  to pass through the train for 20 min. at a speed of 4 l./hr. Det. the amt. of  $NH_3$  formed by titrating the excess HCl with 0.05 N NaOH and methyl orange as indicator (1 cc. 0.05 N  $NH_3$  = 0.0027 g.  $H_2O$ ). Pass  $N_2$  through the train without the HCl wash bottle for 10 min. (4-5 l./hr.) before introducing the sample. The method is suited for all solids except those giving off  $NH_3$  at 80-90° or decompg. at this temp. under the formation of water. Determination of moisture in gases (appendix). F. Roth. *Ibid.* 77-8.—Moisture is detd. in gas having a higher temp. than the surroundings by replacing the usual  $Mg_3N_2$  tube by a rectangular glass tube (6-8 mm. internal diam.) with a three-way stopcock, filling one side only with the nitride, introducing into the gas line, allowing it to assume the temp. of the gas during 10 min., then turning the tube so that gas passes through the tube for 10 min., after which measurements are made in the usual manner. The procedure is suitable only for gas at moderate pressure.  $CO_2$  in the gas has no effect on the accuracy of the detn. of moisture by the  $Mg_3N_2$  method.

Hans Schindler

7880<sup>4</sup>, 1941

Determination of the moisture content of gases. F. Roth and A. Schulz. *Brennstoff-Chem.* 20, 317-19(1939).—Pass about 5 l. of the gas through a tube contg. 2.5-3 g.  $Mg_3N_2$  mixed with glass wool.  $NH_3$  is formed by the reaction of  $H_2O$  with  $Mg_3N_2$  and is absorbed in a measured vol. of 0.05 N HCl. The excess HCl is titrated.

B. C. P. A.

7780<sup>7</sup>, 1940

Determination of water in liquid fuels by means of magnesium nitride. L. Boisselet and Rachkani. *14me Congr. chim. ind., Paris Oct., 1934*, 5 pp.—A crit. study of the method, based on the reaction  $Mg_3N_2 + 6H_2O = 3Mg(OH)_2 + 2NH_3$  and recommended by Conrad and Dietrich (C. A. 25, 4491 2), showed that it is quick and easy, that it is applicable to all liquid fuels and petroleum products, and that it may possibly also be applicable to liquids such as glycerol, higher alcs., and animal and vegetable oils. The essential factor for accurate results is the use of a pure  $Mg_3N_2$ , which can be prepd. in the lab. by

passing dry N over Mg heated to dull redness; impure com.  $Mg_3N_2$  gives high results. A. Papineau-Couture

6018<sup>8</sup>, 1935

Determination of water in alcohol mixtures. W. Jakunowicz. *Przemysł Naftowy* 6, 484-7(1931).—This is a critical review of the literature, with special reference to the paper of Dietrich and Conrad (C. A. 25, 4491). J. WIERTELAK

2394, 1932

Determination of water. A. L. Gol'dinov, V. I. Likhovitskii, and G. Ya. Mal'kova. *Zh. Analit. Khim.* 16, 724-8 (1961). The common methods (Dietrich and Conrad, *CA* 25, 4491; Mitchell and Smith, *Aquametry. Application of the Karl Fischer Reagent to Quantitative Analysis Involving Water*, 1948 (*CA* 43, 522g); Berg, *Rapid Quantitative Phase*

*Analysis*, 1952, p. 34 (*CA* 48, 3851f)) for the detn. of H<sub>2</sub>O by the amts. of NH<sub>3</sub>, C<sub>2</sub>H<sub>2</sub>, and H<sub>2</sub> evolved by reaction with Mg<sub>3</sub>N<sub>2</sub>, CaC<sub>2</sub>, and CaH<sub>2</sub>, resp., in which the reactions are assumed to be: Mg<sub>3</sub>N<sub>2</sub> + 6H<sub>2</sub>O = 3Mg(OH)<sub>2</sub> + 2NH<sub>3</sub>; CaC<sub>2</sub> + 2H<sub>2</sub>O = Ca(OH)<sub>2</sub> + C<sub>2</sub>H<sub>2</sub>; CaH<sub>2</sub> + 2H<sub>2</sub>O = Ca(OH)<sub>2</sub> + 2H<sub>2</sub>, are erroneous. For example, in the reaction of Mg<sub>3</sub>N<sub>2</sub> with H<sub>2</sub>O, the amt. of NH<sub>3</sub> evolved depends on the temp., because of the reaction Mg<sub>3</sub>N<sub>2</sub> + 3H<sub>2</sub>O = 3MgO + 2NH<sub>3</sub> (A). Similar reactions are possible with CaC<sub>2</sub> and CaH<sub>2</sub>, yielding CaO, as shown by calcd. *K* values, 10<sup>6</sup>, 1.1 × 10<sup>6</sup>, 2.5 × 10<sup>7</sup>, resp., for Mg<sub>3</sub>N<sub>2</sub>, CaC<sub>2</sub>, and CaH<sub>2</sub>. A method is proposed for the detn. of H<sub>2</sub>O in Freon with Mg<sub>3</sub>N<sub>2</sub> based on the reaction at 600° to form NH<sub>3</sub> according to equation A. A U tube contg. Mg<sub>3</sub>N<sub>2</sub> was flushed with dried (P<sub>2</sub>O<sub>5</sub>) Ar (7-8 l./hr.) at 600° until no NH<sub>3</sub> was present in the effluent; 3-5 l. of Freon was passed through the U tube followed by 2 l. dried Ar. Then the U tube was placed in an oven at 600° and flushed with 5 l. of dried Ar. All effluent gases were passed through 3 ml. of 0.01N H<sub>2</sub>SO<sub>4</sub>, and NH<sub>3</sub> was detd. photocolometrically with Nessler's reagent.

L. D. Kushnir

9212 i, 1962, V. 57

Determination of traces of water in methyl chloride. H. Guérin. *Chim. anal.* 31, 245-6 (1949).—Water in samples of MeCl can be detd. by passing the gas over Mg<sub>3</sub>N<sub>2</sub> which causes the formation of 2 mols. NH<sub>3</sub> from each 6 moles of H<sub>2</sub>O. The NH<sub>3</sub> can be absorbed and detd. as in the Kjeldahl distn.

W. T. Hall

483 e, 1950

Determination of water in liquid fuels by means of Mg<sub>3</sub>N<sub>2</sub>. Enzo Pipparelli and Aldo Simonetti. *Ann. chim. applicata* 34, 40-2 (1944).—Detn. of H<sub>2</sub>O by use of Mg<sub>3</sub>N<sub>2</sub> by the method of Dietrich and Conrad (*C.A.* 25, 4491) gives low results owing to the formation of a certain amt. of Mg(OH)<sub>2</sub>· $\frac{1}{2}$ H<sub>2</sub>O. However, this can be prevented if the reaction tube is kept at 100°. A. W. C.

2871 e, 1947

A method for the determination of moisture in gases. Enzo Pipparelli and Aldo Simonetti. *Ann. chim. applicata* 33, 3-9 (1943); *Chem. Zentr.* 1943, II, 150.—In detg. moisture in gases by the use of Mg<sub>3</sub>N<sub>2</sub>, an error can be caused by the formation of some Mg(OH)<sub>2</sub>. This can be prevented if the reaction tube is kept at 100°. The presence of small quantities of CO<sub>2</sub> in the gas does not interfere. When larger quantities of CO<sub>2</sub> and water are present, (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> is formed, part of which remains in the reaction tube.

M. G. Moore

5470', 1944



Determination of water in liquid fuels by means of magnesium nitride. L. Boisselet. *Ann. combustibles liquides* 10, 449-54(1935); cf. *C. A.* 29, 6018<sup>8</sup>.— $\text{N}_2\text{Mg}_2$  (5 g.) is introduced into a 500-cc. flask with 100 cc.  $\text{C}_6\text{H}_6$  dried over  $\text{CaCl}_2$ . The  $\text{C}_6\text{H}_6$  is distd. until half has gone over or until the distillate is not alk. to litmus. A sample of fuel contg. about 0.5 g.  $\text{H}_2\text{O}$  is added and the  $\text{C}_6\text{H}_6$  is distd., carrying over the  $\text{NH}_3$  formed, which is collected in standardized acid. The  $\text{NH}_3$  formed, and thus the original  $\text{H}_2\text{O}$ , are calcd. from the back titration of the acid. The reagent is not affected by mixts. contg. EtOH or less than 50% MeOH. G. Calingaert

6727', 1935

Determination of water in alcoholic motor fuels. K. R. DIETRICH AND C. CONRAD. *Z. anorg. Chem.* 44, 532-4(1931).—Place at least 5 g. of  $\text{Mg}_3\text{N}_2$  in a dry flask and add to it 50 cc. of the alc. Twice as much nitride as there is water present should be used in every case. Distil off about  $\frac{3}{4}$  of the liquid, catching the distillate in a measured vol.

of standard acid. Each mol. of water present liberates  $\frac{1}{2}$  mol. of  $\text{NH}_3$ . The method is shown to give excellent results in the analysis of alc. contg. 0.1 to 9.9% of water. If more than 60% of MeOH is present, some  $\text{NMe}_3$  is formed, which makes the results inaccurate. The method is satisfactory for the detn. of water in gasoline, benzene, ethyl and acetone. W. T. H.

CA - 4492, Vol. 25, 1931

Reaction with KCNS to form H<sub>2</sub>S

A method for the titrimetric determination of micro-amounts of hydrogen sulfide. Eberhard Kasper (Inst. Post-Ferromeldewesen, Berlin). *Chem. Tech.* (Berlin) 11, 616-17 (1959).—A "pptn.-exchange reaction" of Erdey (Erdey and Bányai, *CA* 53, 29f), in which difficultly sol. PbC<sub>2</sub>O<sub>4</sub> is converted by H<sub>2</sub>S to PbS and C<sub>2</sub>O<sub>4</sub><sup>2-</sup> and the latter detd. by titration with KMnO<sub>4</sub>, was chosen for the detn. of H<sub>2</sub>S in air and in corrosion products. The method can be applied to the detn. of water, either chem. united or absorbed in materials, by causing them to react with molten KCNS forming H<sub>2</sub>S. Org. materials, NH<sub>4</sub> salts, and metals forming insol. sulfides must be absent. SO<sub>2</sub> and CO<sub>2</sub> do not disturb the reaction, but SO<sub>2</sub> causes too high values of H<sub>2</sub>S concn. The endpoint precision of the method may be rendered more sensitive by transistor amplification.

Thomas A. Wilson

17151 f, 1960

Reactions with Phosphin or Cinnamoyl Chloride  
to form HCl

Experiences in the volumetric determination of water. JOSEF LINDNER. *Z. anal. Chem.* 86, 141-53(1931).—Phosphin, the trivial name for naphthyldichlorophosphine oxide,  $C_{10}H_7POCl_2$ , has been used for the volumetric detn. of  $H_2O$ , with which it reacts and liberates HCl, which can be titrated. When proper precautions are taken excellent results can be obtained in detg. 0.2-10 mg. of water. Full details are given concerning the procedure.  
W. T. H.

5873, 1931

Titration method for the determination of water. RONALD P. BELL. *J. Chem. Soc.* 1932, 2903-5.—The method described depends on the reaction of water with  $\alpha$ -naphthoxydichlorophosphine. One mol. of HCl is formed from each mol. of  $H_2O$  and the HCl is removed by a current of air, absorbed in water and titrated with 0.02 N NaOH to an end point with methyl orange. For an approx. detn. of water in org. liquids it suffices to mix the sample with the reagent and pass an air current through the app. for several hrs.  
W. T. H.

1295, 1933

Volumetric determination of small quantities of water by means of cinnamoyl chloride. C. J. van Nieuwenburg. *Mikrochim. Acta* 1, 71-4(1937).—Directions for prepg. cinnamoyl chloride from cinnamic acid and thionyl chloride are given. This acid halide proved the most satisfactory of all those tried. It reacts with water according to the equation;  $2C_6H_5COCl + H_2O = C_6H_5CO_2COC_6H_5 + 2HCl$ . At 60-70° all the HCl can be removed by a current of air, caught in water and the resulting soln. titrated. With alkali hydroxide soln. the original chloride and the anhydride are not appreciably volatile even at 110°. The reagent, therefore, has been found to possess distinct advantages over naphthyloxychlorophosphine which was proposed by Lindner in 1935.  
W. T. H.

5717', 1937

^ Suitability of cinnamoyl chloride for the titrimetric determination of water. Josef Lindner and Gertrud Zienert. *Mikrochemie verein. Mikrochim. Acta* 31, 254-62(1943).—Like  $C_{10}H_7POCl_2$ , which L. has used successfully for detg. water (cf. *C.A.* 16, 3605 and later publications) cinnamoyl chloride can be used to react with water with subsequent titration of the liberated HCl, but somewhat less advantageously. Higher blanks are required because of the moisture content of the acid chloride, and phenolphthalein, rather than methyl red, must be used as indicator. The reagent does not keep as well as the naphthyloxychlorophosphine does, no time is saved and the quantity of water reacting with a given wt. of the acid chloride does not seem to be perfectly definite. It is not quite clear how much should be used.  
W. T. Hall

2708<sup>3</sup>, 1945

The bases of an electrochemical determination of water and organic compounds containing active hydrogen. H. Dehn, V. Gutmann, and G. Schoeber (Tech. Hochschule, Vienna). *Monatsh.* **93**, 1357-62(1962); cf. *CA* **58**, 8647f. In a mixt. of 95%  $\text{PhPOCl}_2$  + 5%  $\text{PhCl}$  at  $0^\circ$ , the polarographic current increased linearly with voltage and was proportional to the concn. of  $\text{HCl}$  formed by reaction of  $\text{PhPOCl}_2$  with traces of  $\text{H}_2\text{O}$  or org. compds. contg. active H. To det.  $\text{H}_2\text{O}$  or a compd. with active H (MeOH,  $n\text{-C}_4\text{H}_9\text{OH}$ ), add the sample to a mixt. of 95%  $\text{PhPOCl}_2$  + 5%  $\text{PhCl}$ , heat in a tightly closed bottle to  $60^\circ$  to complete hydrolysis, cool to  $0^\circ$ , and record the current with a Hg dropping electrode at  $-2.7$  v. vs. a Hg pool. Karel Micka

10728 c, 1963, V. 58

## Reaction with $\text{AlCl}_3$ to form $\text{HCl}$

Continuous determination of small quantities of water. Kurt Fischbeck and Ernst Eckert. *Z. anal. Chem.* 112, 305-13(1938).—It was desired to recognize immediately the formation of small quantities of  $\text{H}_2\text{O}$  in the study of certain reduction processes and none of the existing methods served to do this. When anhyd.  $\text{AlCl}_3$  comes into contact with  $\text{H}_2\text{O}$  at  $450^\circ$ ,  $\text{Al}_2\text{O}_3$  and  $\text{HCl}$  are formed. The excess  $\text{AlCl}_3$  deposits on cooling the gas and the  $\text{HCl}$  reacts with  $0.1\ N\ \text{Na}_2\text{CO}_3$  in the presence of methyl red to cause the color to change from yellow to red. The time required to cause color change is a measure of the quantity of  $\text{H}_2\text{O}$  originally present. The method was tested by passing  $\text{H}_2$  over  $\text{AlCl}_3$  kept at  $145^\circ$  and uniting this gas, in a furnace heated to  $450^\circ$ , with another stream of  $\text{H}_2$  contg. a known quantity of water. Corrections had to be applied for the small quantity of  $\text{AlCl}_3$  which was not condensed by the cold walls of the tubing from the furnace and for the very small quantity of  $\text{H}_2\text{O}$  in the  $\text{H}_2$  gas used. Oxygen, either free or combined, must be absent. Expts. with phenol, benzoic acid, cane sugar and naphthalene were carried out in the hope that the method could be used for a detn. of O. With phenol only 1 mol. of  $\text{HCl}$  was liberated, whereas it was expected that 2 mols. of  $\text{HCl}$  would be formed from  $\text{AlCl}_3$ . With benzoic acid and benzophenone about  $\frac{1}{3}$  the expected quantity of  $\text{HCl}$  resulted. A full description of the app. and details concerning the precautions to be taken are given. W. T. H.

5331', 1938

Reaction with C to form CO<sub>2</sub>

Determination of water vapor and oxygen in gases containing no oxygen compounds. N. Shurmovskaya and L. Kupriyanova. *Zhur. Anal. Khim.* 3, 41-4(1948).—The method is based on the interaction of C with any compd. contg. O. In the described app. the gas is passed through a quartz tube filled with specially prepd. birch charcoal. The tube is provided with a thermocouple and is placed in an elec. furnace heated to 1000°. The gas leaving the quartz tube is led through a tube filled with granular I<sub>2</sub>O<sub>5</sub> and provided with a reflux condenser. The temp. inside the tube is 140°. The gases leaving the I<sub>2</sub>O<sub>5</sub> tube are washed free of I vapors in 2 consecutive columns filled with 10% soln. of KI and thence through a cond. cell contg. Ba(OH)<sub>2</sub>. The quantity of CO<sub>2</sub> in the final gas can be calcd. from the cond. data as outlined by Bruns, *et al.* (*Zhur. Anal. Khim.* 2, 294(1947)). M. Hosh

971 L, 1949

## Reaction with $\text{MgCH}_2\text{I}$ to form $\text{CH}_4$

The Quantitative Determination of Water in Substances by means of Alkyl Magnesium Halogen Compounds. TH. ZEREWITINOFF. *Z. anal. Chem.*, 50, 680-91.— The method depends on the fact discovered by Grignard that  $\text{MgCH}_2\text{I}$  reacts with water giving methane. This reaction is made to take place quant., according to the equations:  $\text{Mg}(\text{CH}_2)_2\text{I} + \text{H.OH} = \text{CH}_4 + \text{Mg}(\text{OH})\text{I}$ ,  $\text{Mg}(\text{OH})\text{I} + \text{Mg}(\text{CH}_2)_2\text{I} = \text{CH}_4 + \text{MgI}_2 + \text{MgO}$ . The amt. of methane evolved is measured, and the corresponding amt. of water calc. Hydroxy compds. in general as alc., phenols, acids, oxines, sugars, flavones, alkaloids, also react quant. with  $\text{MgCH}_2\text{I}$ . By this method the number of hydroxyl groups present in such compds. may be exactly detd. Likewise thiohydroxy compds. as mercaptans and thiophenols also primary and secondary amines react quant. with  $\text{MgCH}_2\text{I}$ . For the quant. estn. of the hydroxyl, thiohydroxyl, amino and imino groups, the compd. is dissolved in some solvent which also dissolves the  $\text{MgCH}_2\text{I}$ , but does not react with it. For this purpose amyl ether, pyridine, xylol, mesitylene and anisole are suitable. Free water can be detd. in compds. containing any of these groups by choosing a solvent for the  $\text{MgCH}_2\text{I}$  in which the substance is insol. In such a case only the free water reacts and can be accurately detd. Analytical results of the detn. of water in coal by this method and by the 3 other known methods, viz.: (1) drying in oven at  $100-110^\circ$ ; (2) drying in vacuum desiccator over  $\text{H}_2\text{SO}_4$  at ordinary temp. (3) drying in a stream of  $\text{H}$  at  $100-110^\circ$  and catching the water expelled in a weighed  $\text{CaCl}_2$  tube are given. By (1) it is impossible to get accurate results owing to the escape of other volatil compds. and to partial oxidation of  $\text{C}$  to  $\text{CO}_2$ . The chief objection to (2) is its slowness, while (3), although it gives very accurate results is too complicated and tiresome. The  $\text{MgCH}_2\text{I}$  method can be carried out in less than an hour. The duplicate detns. of water in a series of coals gave very concordant values, but in every case less than the values obtained by the other 3 methods. The methods for the estimation of water in starch are also compared with this new method.

M. C. BOSWELL.

203, 1912

## Reaction with $\text{CaC}_2$ to form $\text{C}_2\text{H}_2$

**The Carbide Method of Determining Moisture.** HAROLD A. DANNE. *Chem. Eng.*, 16, 163-4.—D. draws attention to the faults of existing methods and shows where he eliminates these. A drawing giving details of the app. used is furnished. By this method the moisture is estd. by the amt. of  $\text{C}_2\text{H}_2$  evolved in the interaction of its vapor with an excess of  $\text{CaC}_2$ . D. describes his app. in detail, drawing attention to a proper manipulation of the same to obtain accurate results and suggests that the process be extended to the following: whole or powdered plants, alcohols, ethers and their mixtures, water of crystn., avoiding substances containing halogens in loose combination.

DAVID J. HUETHER.

36, 1913

**The Determination of Moisture in Organic Substances.** F. H. CAMPBELL. *J. Soc. Chem. Ind.*, 32, 67-70.—The wt. of  $\text{C}_2\text{H}_2$  that is evolved when a substance containing  $\text{H}_2\text{O}$  is treated with  $\text{CaC}_2$  can be used as a basis from which to calc. the  $\text{H}_2\text{O}$  content. C. has devised a special app. consisting of a glass tube, fitted with a stopcock, which passes through a rubber stopper in the mouth of a second bent glass tube. The  $\text{CaC}_2$  is placed in the first tube and sealed in with paraffin. A known wt. of the sample is placed in the second tube. On heating, the paraffin melts, and the  $\text{CaC}_2$  is allowed to sift slowly on the sample. The reaction is completed at  $100^\circ$ . The app. is weighed before and after the reaction, the residual  $\text{C}_2\text{H}_2$  having been replaced by air. The app. weighs about 30 g. Time of detn. = 30-60 min. One charge of  $\text{CaC}_2$  lasts for 6 detns. The wt. of  $\text{H}_2\text{O}$  equiv. to 1 g.  $\text{C}_2\text{H}_2$  is detd. for the  $\text{CaC}_2$  used. This is usually 1.44 g. The method is as accurate as the cold vacuum method and very rapid. App. is simple and inexpensive.

E. W. BOUGHTON.

1336, 1913

**A new and improved method of moisture determination and its application to leather.** A. COLIN-RUSS. *J. Intern. Soc. Leather Trades' Chem.* 15, 113-26, 166-82 (1931). An app. is described wherein leather is heated in a tube surrounded by vapor

of a boiling liquid, and the  $\text{H}_2\text{O}$  evolved from the leather is allowed to react with  $\text{CaC}_2$  forming  $\text{C}_2\text{H}_2$ , which is collected over  $\text{Hg}$  and measured. A blank on the  $\text{CaC}_2$  is indispensable. For standardizing the procedure, numerous hydrates and liquid  $\text{H}_2\text{O}$  were used; the latter, as well as Glauber's salt, give up their  $\text{H}_2\text{O}$  so rapidly that the reaction with  $\text{CaC}_2$  is incomplete; borax and gypsum are not completely decomposed at  $100^\circ$ ;  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  gave the theoretical yield of  $\text{H}_2\text{O}$ , and the time-gas volume curve is that of a monomolecular reaction. With leather of heavy vegetable tannage, it was found that the percentage water obtained by the method is almost independent of the reaction temp. (controlled by suitable choice of surrounding solvent vapor) over the range from  $61.5^\circ$  to  $133.5^\circ$ : 12.6% at  $61.5^\circ$ , 13.1% at  $76.5^\circ$ , 13.8 at  $100.5^\circ$ , 13.05 at  $133.5^\circ$ . Time required to complete the reaction decreased as temp. increased. Degree of subdivision of the sample is practically without effect. In a sumac-tanned leather, which showed marked contraction at  $100^\circ$ , percentage  $\text{H}_2\text{O}$  obtained by the method decreased from 13.9% at  $61-74^\circ$  to 10.3-11.0% at and above  $100^\circ$ , the decrease indicating that the contraction involves fixation of  $\text{H}_2\text{O}$ . Comparative results on 4 leathers by the carbide and by the oven-drying methods showed that the former always gives lower results, the difference varying from 1.45% for Cr-tanned calf to 6.03% for heavily carried vegetable cowhide. Good agreement was obtained between results yielded by the carbide method and those obtained by very prolonged desiccation over  $\text{H}_2\text{SO}_4$ , except in the case of the abnormal sumac leather.

H. B. MERRILL

2873', 1931



Determination of moisture in commercial products. A. I. Kotnitskii. Russ. 44,716, Oct. 31, 1935. In the detn. of moisture in peat, etc., by means of acetylene generated from  $\text{CaC}_2$  introduced into the material to be tested, the accuracy of the method is improved by adding to the material a known amt. of water.

2872<sup>5</sup>, 1938

The carbide method for determining moisture. P. S. Zhelezkov. *Chemisation Socialistic Agr.* (U. S. S. R.) 1936, No. 10, 101-14.—The two methods for detg.  $\text{H}_2\text{O}$  with carbide are: (1) a steel container with a gage for measuring the gas pressure, the gage having been calibrated with definite quantities of water reacting with carbide; (2) an Al container in which the  $\text{C}_2\text{H}_2$  is generated, weighed, the gas released and the container weighed again. From the wt. of  $\text{C}_2\text{H}_2$  the quantity of water in the sample is calcd. The moisture in soil, preserved fish and other substances were detd. J. S. Joffe

6581', 1937

Determination of moisture by means of calcium carbide. P. M. Orzhekhovskii and K. B. Khait. *J. Applied Chem.* (U. S. S. R.) 9, 1141-3(1936).—Heating com.  $\text{CaC}_2$  yields  $\text{C}_2\text{H}_2$ , as a result of its hygroscopic water content; this should be taken into account when detg. moisture by means of  $\text{CaC}_2$ . Preliminary heating of  $\text{CaC}_2$  eliminates the error in the detn. of moisture. Exptl. data are tabulated. A. A. Podgorny

2549<sup>5</sup>, 1937

Device for determining the water content of materials. Stephen Stanworth and James Stanworth. Ger. 604,556, Oct. 23, 1934 (Cl. 42, 9.50). The water content is detd. by decomposing the water, e. g., by  $\text{CaC}_2$ , and measuring the gases evolved.

647<sup>2</sup>, 1935

Apparatus for determining moisture. Stephen Stanworth (one-half to James Stanworth). U. S. 1,976,752, Oct. 16. Structural features.  $\text{CaC}_2$  is utilized.

7077', 1934

Speedy methods of determining the moisture in a substance. S. Stanworth. *Trans. Ceram. Soc.* 32, 443-52 (1933).—A weighed sample is mixed with  $\text{CaC}_2$  in a closed container fitted with a pressure gage. The  $\text{H}_2\text{O}$  content is measured by the pressure produced by  $\text{C}_2\text{H}_2$ . H. F. K.

1630<sup>4</sup>, 1934

Determination of moisture and oxygen in flue and similar gases. V. A. Naklonov. Russ. 52,306, Dec. 31, 1937. The sample is passed through  $\text{CaC}_2$ , Na or K, and the product is burned. From the quantity of  $\text{C}_2\text{H}_2$  or H burned by the O of the sample, is detd. the O present, and from the total amt. of the  $\text{C}_2\text{H}_2$  or  $\text{H}_2$  formed is detd. the moisture of the sample.

3624<sup>3</sup>, 1930

Determination of hygroscopic moisture in crystalline hydrates. A. T. Sveshnikov and N. V. Koleva. *Zavodskaya Lab.* 9, 357(1940); *Chimie & industrie* 40, 289 (1940).—The method is based on the reaction of moisture with  $\text{CaC}_2$  in a "calcinometer" which results in a rapid fall in the level of the liquid (satd.  $\text{NaCl}$  soln.) in the buret. The reaction requires about 15 sec. A. P.-C.

6945<sup>4</sup>, 1942

A simple apparatus for the rapid determination of moisture by the carbide method. Edward T. Fukunaga and L. A. Dean. *Hawaiian Planters' Record* 43, 3-5(1939).—The app. consists of a welded, cylindrical Al-alloy bomb, 4 in. diam. and 7 in. deep, having a head comprised of a machined flange and plate, held together by bolts and wing nuts. A threaded opening in the plate carries a 30-lb. pressure gage with a 4-in. dial and a Weston all-metal thermometer extends into the bomb for a distance of 3.5 in. The other equipment consists of (1) a tin can 4 in. high and of such diam. that it just fits within the bomb, to hold the sample, and (2) a metal container 1.5 in. diam. and 1.5 in. high, to hold the  $\text{CaC}_2$  (screened to pass a sieve with 0.5-mm. mesh). After the bomb is charged it is tilted to spread the  $\text{CaC}_2$  over the sample, then returned to the upright position and shaken with a swirling motion for about 1 min. or until no further increase in pressure is noted. The vol. of  $\text{C}_2\text{H}_2$  generated is reduced to standard pressure and temp. and the amt. of water is calcd. therefrom.

K. D. Jacob

4467<sup>6</sup>, 1939

Determination of moisture by the carbide method. B. Gribanovskii and N. Volotova. *Novosti Tekhniki* 1938, No. 11-12, 63.—The app. is made from a buret, the open end of which is sealed to the small reaction chamber provided with the glass stopper with the sealed-in reaction vessel for the introduction of the sample. Another opening in the side of the reaction chamber connects the chamber with a Hg flask and at the bottom of the chamber is another opening for washing of the app., which is closed with a rubber stopper. During the reaction, the  $\text{C}_2\text{H}_2$  formed collects in the buret, forcing out the Hg. The results are calcd. by the formula  $X_{\text{H}_2\text{O}} = 0.161(A/B)K_1K_2$ , where  $A$  is the amt. of  $\text{C}_2\text{H}_2$  formed,  $B$  wt. of sample in g.,  $K_1$  the coeff. for reducing to standard conditions and  $K_2$  the yield of  $\text{C}_2\text{H}_2$  on the  $\text{CaC}_2$ . A. A. Podgorny

7371<sup>4</sup>, 1938

Determination of water content of gases. V. I. Karmazin. *Zavodskaya Lab.* 6, 185-G(1937).—The sample of gas is passed through  $\text{CaC}_2$  and the  $[\text{H}_2\text{O}]$  calcd. from the  $[\text{C}_2\text{H}_2]$  of the resulting gas. B. C. A.

4103<sup>5</sup>, 1938

Automatic determination of the water content of solid fuels and gases. V. A. Naklonov. *Zavodskaya Lab.* 6, 187-8 (1937).—The  $[\text{H}_2\text{O}]$  of gases is calcd. from the vol. of  $\text{C}_2\text{H}_2$  produced by passing the gas through  $\text{CaC}_2$ . The  $[\text{H}_2\text{O}]$  of solid fuels is detd. by shaking a mixt. of powd. fuel and  $\text{CaC}_2$  for 40 sec. and measuring the vol. of  $\text{C}_2\text{H}_2$  evolved. B. C. A.

3933<sup>5</sup>, 1938

Apparatus for determination of the water content of petroleum products. D. A. Aliev (Petroleum and Gas Plant, Baku). *Neftepererabotka i Neftekhim., Nauchn.-Tekhn. Sb.* 1963(1), 25-7. The vol. of  $C_2H_2$  evolved on heating a sample of the product together with powd.  $CaC_2$  on a water bath is measured. The app. consists of a reaction vessel and a gas buret. The inner, graduated tube of the glass buret is surrounded by a glass jacket filled with  $H_2O$  that seals the inner tube, which has a no. of openings at the bottom. The nozzle which serves as an inlet for the gas from the reaction vessel is located in the inner tube at a level above these openings so that escape of  $C_2H_2$  into the outer jacket is prevented. Blowing of water out of the nozzle before the detn. and filling of the inner tube (i.e., the buret proper) with  $H_2O$  are effected by means of rubber bulbs equipped with valves. A detn. by the method described takes 7-8 min., as compared with Dean-Stark method, which takes >45 min.; the results are just as accurate. The use of solvents is eliminated and the danger of fire lessened. GZJR

6164g, 1963, V. 59

Determination of the water content of aqueous mixtures. S. R. M. Ellis, R. D. Carbett, and H. N. Sadler (Univ. Birmingham, Engl.). *Ind. Chemist* 35, 498-500(1959).—Anal. procedure and app. are described for the detn. of  $H_2O$  in aq. mixts. by reaction with  $CaC_2$ . Cf. *C.A.* 47, 1539f. J. C. Tallman

3056c, 1960

Moisture determination in practice. R. Klockmann (Riedel-de Haën A. G., Scheelze bei Hanover, Ger.). *Chem. Ztg.* 76, 706-7(1952).—Place a 5-, 10-, or 20-g. sample of the material and a capsule of  $CaC_2$  in a small steel pressure flask which is closed with an air-tight manometer. Shake to break the  $CaC_2$  capsule, and read the resulting pressure on the manometer. J. H. Moore

1539f, 1953

A method for the determination of moisture in gases. A. I. Doladugin, Yu. L. Khmel'nitskii, and Yu. M. Kachmarchik. *Zavodskaya Lab.* 11, 530-4(1945).—The method is based on the reaction between  $CaC_2$  and water with the formation of  $Ca(OH)_2$  and  $C_2H_2$ . The completeness of the absorption of water by  $CaC_2$  was detd. by the test for  $C_2H_2$  with the Illosva reagent. Pass 100 l. of gas through 3 U-tubes filled with  $CaC_2$  and dry  $N_2$  and measure the gain in wt. 6 references. W. R. Henn

2417<sup>8</sup>, 1946

Determination of moisture in aromatic hydrocarbons. Yu. L. Khmel'nitskii, A. I. Doladugin, and A. V. Guseva. *Zavodskaya Lab.* 11, 534-7(1945).—The method is based on measurement of the  $C_2H_2$  formed when the sample is mixed with powdered  $CaC_2$ . W. R. Henn

24179, 1946

Rapid determination of moisture with the aid of calcium carbide. J. Voskuil and P. Zwaan. *Chem. Weekblad* 39, 257-61(1942); *Chem. Zentr.* 1942, II, 1269.—Heat the sample on the steam bath in a 25-ml. Erlenmeyer flask which is provided with a rubber stopper contg. a  $CaC_2$  tube. Collect and measure the  $C_2H_2$  over satd.  $NaCl$  soln. in a gas buret. W. T. Hall

5335<sup>4</sup>, 1943

Determination of traces of water in hydrocarbons. A calcium carbide gas-liquid chromatography method. H. S. Knight and F. T. Weiss (Shell Develop. Co., Emeryville, Calif.). *Anal. Chem.* 34, 749-51(1962). A hydrocarbon sample is passed through  $\text{CaC}_2$  and the  $\text{C}_2\text{H}_2$  resulting is measured by gas chromatography with a flame ionization detector. Standard deviations at 20 and 0.3 p.p.m. are  $\pm 1.3$  and  $\pm 0.05$  p.p.m., resp. The method was applied to  $\text{C}_4\text{H}_6$ , butadiene, and liquefied petroleum gases with  $\text{H}_2\text{O}$  concns. between 0.3 p.p.m. and 1%.  $\text{H}_2\text{S}$  interferes, and carbonyl compds. will do so if condensation occurs in the reactor.  
R. E. Stanton

9212 h, 1962, V. 57

Rapid determination of the moisture content of the soil by measuring the pressure of acetylene. Leon Nijenson and Hector Pilasi. *Rev. Fac. Cienc. Agrar. Ministerio Educ. Univ. Nac. Cuyo* (Mendoza) 7, No. 2, 7-28(1959).—Soil  $\text{H}_2\text{O}$  is detd. by adding 1 mm. mesh  $\text{CaC}_2$  to a soil sample and measuring the pressure of the  $\text{C}_2\text{H}_2$  generated. Known procedures are modified to give a faster and more complete reaction and more accurate and reproducible detns. Very moist or clay soils are mixed with fine quartz. The Bourdon gage is periodically checked against a Hg manometer and pressures are cor. to 20°. To the  $\text{H}_2\text{O}$  detd. on a dry wt. basis, the following corrections are added: 0.75% for sandy and loamy soils, and 2.11% for clay loam and clayish soils.  
Henry J. Hugos

6663 g, 1962, V. 56

Determination of water in furfural. Z. Lada, A. Rycerz-Komorowska, and M. Brudnicka (Inst. Chem. Ogolnej, Warsaw). *Chem. Anal.* (Warsaw) 6, 565-8(1961).—Rapid detn. of  $\text{H}_2\text{O}$  in furfural by the acetylene method is described. Place a 5-ml. sample in a bomb (CA 53, 15655g), introduce a glass-sealed tube contg.  $\text{CaC}_2$ , and immediately close the seal. Break the tube by shaking, leave for 3-5 min., and det. the  $\text{C}_2\text{H}_2$  pressure. Calc.  $\text{H}_2\text{O}$  from a standard curve.  
Z. Kurtyka

4102 e, 1962, V. 56

Trace water determination by infrared spectrometry. J. W. Forbes (Shell Develop. Co., Emeryville, Calif.). *Anal. Chem.* 34, 1125-8(1962); cf. following abstr.  $\text{H}_2\text{O}$  was converted with  $\text{CaC}_2$  to  $\text{C}_2\text{H}_2$  and the latter transferred with dry Ar to an infrared cell and dissolved in  $\text{CCl}_4$ . The  $\text{C}_2\text{H}_2$  band at  $3.05 \mu$  was used to det. 0.5 to several hundred p.p.m. of  $\text{H}_2\text{O}$  with a standard deviation of 0.5 p.p.m. for a 50-ml. sample. The method was adapted to the continuous detn. of trace  $\text{H}_2\text{O}$  in a plant stream.  
Harold J. Perkins

10530 h, 1962, V. 57

Trace water analyzer for process streams. S. T. Abrams and V. N. Smith (Shell Develop. Co., Emeryville, Calif.). *Anal. Chem.* 34, 1129-32(1962); cf. preceding abstr. Automatic anal. of process streams was accomplished by conversion of the trace  $\text{H}_2\text{O}$  to  $\text{C}_2\text{H}_2$  in soln. in the stream followed by detn. of the  $\text{C}_2\text{H}_2$  at  $3.05 \mu$  in a nondispersive infrared analyzer. Under elastomer pilot-plant conditions, a full-scale range of 0-25 p.p.m. of  $\text{H}_2\text{O}$  was obtained with a sensitivity of 0.25-0.50 p.p.m.  
Harold J. Perkins

10530 i, 1962, V. 57

Apparatus for testing the moisture content of granular materials. Wm. J. Delmhorst (to Measurements Corp.). U. S. 2,359,831, Oct. 10, 1944. The app. is based on mixing the tested material with  $\text{CaC}_2$  in a closed container. The generated  $\text{C}_2\text{H}_2$  is allowed to leave this container and is detd.

845-6, 1945

Method and apparatus for continuous determination of the concentration of compounds in liquids. Jiri Teygl. Czech. 108.708, Oct. 15, 1963, Appl. Dec. 27, 1961; 3 pp. Addn. to Czech. 96.511. Treating the analyzed liquid with an excess of the solid reaction agent, insol. in the medium but yielding with the medium a gaseous or volatile product, provides const. release of the reaction product into the carrier gas and subsequent detn. in the analyzer unit. The following applications are given along with the corresponding reaction: (1) Detn. of moisture in org. solvents:  $\text{CaC}_2 + \text{H}_2\text{O} = \text{Ca}(\text{OH})_2 + \text{C}_2\text{H}_2$ ; (2) Concn. of  $\text{HNO}_3$  in the nitration mixt.:  $3\text{Cu} + 2\text{HNO}_3 + 3\text{H}_2\text{SO}_4 = 3\text{CuSO}_4 + 4\text{H}_2\text{O} + 2\text{NO}$ ; (3) Concn. of  $\text{HCl}$  in industrial liquids:  $\text{CaSO}_4 + 2\text{HCl} = \text{CaCl}_2 + \text{H}_2\text{O} + \text{SO}_2$ . L. J. Urbanek

9916g, Vol. 60

## Acetylene Method of Winogradov

Determination of water by the acetylene method. J. Minczewski and J. Tromszczyński (Zakład anal. inst. chem. Ogólnej, Warszaw). *Przemysł Chem.* 34, 147-9 (1955)(Russian summary)(in English).—The acetylene method for H<sub>2</sub>O detn. according to Vinogradov (*C.A.* 47, 8585b) was verified.  
Z. Kurtyka

16560a, 1956

The possibility of a wide application of the quick acetylene method in moisture determinations. L. Winogradov. *Przemysł Chem.* 31, 569-73(1952).—A very simple app. is described for the detn. which is a distinct improvement. The reagent and the reactant are brought together slowly, little by little in a filter flask, under vigorous shaking. The size of the sample depends upon the moisture contents, those high in moisture are mixed with *grog*.

Werner Jacobson

8585b, 1953

# Distillation with Xylene, Toluene, Benzene

**Chemical composition of KEAM.** N. A. Gruzdeva. *Proizv. i Ispol'z. Novykh Koksokhim. Produktov, Vest. Nauchn.-Issled. Uglekhim. Inst. Sb. Statei 1960*, 89-95. KEAM (a product for crop protection) is decompd. by boiling with xylene, instead with HCl, for the detn. of H<sub>2</sub>O, anthracene oil, and sulfite liquor. HCl causes a rapid coagulation of the emulsion and forms tars which interfere in oil extn. Boiling with xylene seps. the sulfite liquor as a powder which is easily sepd. from the oil and dried. Add to a 25-g. sample, in a flask fitted with a reflux condenser, 150 ml. xylene, distil H<sub>2</sub>O, and det. it. Cool the contents of the flask to 30-40°, stir, filter off the pptd. sulfite liquor powder, wash with xylene, and dry at 110-30° for 25-30 min. to const. wt. Distil ( $\leq 150^\circ$ ) xylene from the filtrate and est. the anthracene oil from the residue in the flask. The losses are  $\pm 0.5\%$ . The abs. error is  $\leq 1\%$ . The use of toluene or C<sub>6</sub>H<sub>6</sub> gives 1-4.2% lower results for H<sub>2</sub>O detn.

M. Skulski

15589, 1962, V.57

**Determination of moisture, crude fat, reducing sugars, starch and crude cellulose in a single sample.** A. G. Kul'man. *Biokhimiya* 2, 944-51(1937).—In an app. described, benzene or toluene vapors remove the water and ext. the fat. The other components are detd. in the usual manner.

H. Cohen

2459<sup>5</sup>, 1937

**Determination of water.** Emad Migray. *Ind. Eng. Chem., Anal. Ed.* 7, 348(1935).—The sample is distd. with xylene or toluene, the distillate is dehydrated with a weighed quantity of anhyd. CuSO<sub>4</sub>, which is filtered off, washed with benzene and ether, dried in a vacuum and the water detd. by the gain in wt. of the CuSO<sub>4</sub>.

W. T. H.

7223<sup>9</sup>-1935

**Determination of water content by distillation with liquids which are lighter than water, e. g., toluene and xylene.** HARRY LUNDIN AND MARIA LUNDIN. *Chem.-Ztg.* 56, 236(1932); cf. *C. A.* 26, 48.—An app. and procedure are described for detg. 0-3.7 cc. of water with accuracy. Weigh the substance into a 250-500 cc. conical flask, taking enough to furnish 0-1.1 cc. of water or 1-2 cc. according to the size of app. chosen, and add 150 cc. of toluene or xylene. Distil carefully until finally about 85 cc. of distillate has been collected in a special graduated receiver. By centrifuging, the water is obtained with a level surface below the lighter hydrocarbon.

W. T. H.

2939, 1932

**Rapid method for the determination of moisture content.** J. TAUSZ AND H. RUMM. *Z. angew. Chem.* 39, 155-6(1926).—This ingenious method is suitable for the detn. of moisture in all sorts of materials, such as wheat flour, rye flour, white bread, yeast, butter and petroleum. It is based upon the principle of distg. with a less volatile liquid which has been used by others but the liquid used and the app. recommended represent improvements. The substance is mixed with considerable C<sub>2</sub>H<sub>2</sub>Cl<sub>4</sub> and heated in a dry flask. The distillate passes through a fractionating tower, which serves at first to condense all the C<sub>2</sub>H<sub>2</sub>Cl<sub>4</sub>, and the steam is caused to condense and pass through a capillary into a gas buret contg. C<sub>2</sub>H<sub>2</sub>Cl<sub>4</sub> as confining liquid. The water, being of less d., floats upon this liquid. The distn. is continued, with gradually increased temp., until clear C<sub>2</sub>H<sub>2</sub>Cl<sub>4</sub> is obtained as distillate, the lack of turbidity showing that all water has been removed. Then the water is pushed up, by the leveling device, into a narrow measuring tube. The results of numerous analyses indicate that the method is very suitable for the analysis of substances which are easily decompd. and partially burned when heated directly.

W. T. H.

1576, 1926

Report of sub-committee on determination of water. W. H. FULWEILER. *Proc. Am. Soc. Testing Materials* 25, Pt. I, 416-23(1925).—A discussion of the detn. of H<sub>2</sub>O in different materials, bringing out the advantages of the method consisting in distn. in the presence of a solvent (Method D 95-24), suggesting the possibility and advisability of extending its use to other classes of materials than those for which it is used at present. A bibliography of 56 references is given in connection with the method. A. P.-C.

1575, 1926

The More Recent Processes for the Determination of Water by Distillation. J. F. HOFFMANN. Berlin. *Z. angew. Chem.*, 21, 2095-8 (Oct. 2).—In 1901, a German patent was granted to a process for the determination of water in solids and liquids by distillation. The substance to be examined was either distilled with a liquid immiscible with water, with a boiling point higher than that of water, or heated with such a liquid, and then distilled with another liquid likewise immiscible with water, but with a lower b. p. In either case, the water collected in the distillate was measured. The method originally designed for the analysis of cereals and cereal products, employing such liquids as petroleum and toluene, has been varied to render it applicable to the analysis of many other substances, such as butter (Gray, *Z. angew. Chem.*, 21, 890), other foods (Thörner, *Ibid.*, 21, 146, 1302), cellulose (Schwalbe, *Ibid.*, 21, 1321, 2448), lignites (Grübe, *Braunkohle*, 1906, 581). Results are given of analyses of hops, showing that the distillation process, using turpentine and toluene, gives results which agree very closely with those obtained by the older methods of drying *in vacuo* with P<sub>2</sub>O<sub>5</sub>, or in a drying chamber at 80°. The author states that for any substance in which the water is to be determined, at least one of the ten variations of the method mentioned in the article will be found to be suitable to that particular case.

H. M. LANCASTER.

158, 1909

Determination of water in chloride of lime of high titer. van Gasbeek. *Ann. chim. anal. chim. appl.* 21, 201(1939).—With a suitable app. it is easily possible to measure the water present by adding 200 ml. of toluene to 20 g. of sample mixed with 50 g. of granular pumice and distg.

W. T. H.

8524<sup>5</sup>, 1939



## Chromatography

Determination of cyclohexanone and cyclohexanol by gas-liquid chromatography. Ana Hanes and D. Sandulescu. *Rev. Chim. (Bucharest)* 12, 614-15(1961). The detn. by gas-liquid chromatography of cyclohexanone, cyclohexanol, and impurities consisting of cyclohexane, cyclohexene, and H<sub>2</sub>O is described. The stationary phase was isooctylphenol-ethylene oxide condensate, supported on granulated NaCl. The detn. was performed in an Al column 4 m. long and 6 mm. in diam., at 135°, by using H<sub>2</sub> as eluent. S. Alexander Stern

7911 g, 1962, V. 57

Determination of water by gas chromatography. M. G. Guiochon and Catherine Landault. *Publ. Group. Avanc. Methodes Spectrog.* 1962, 195-202. The H<sub>2</sub>O content of aq. BuOH solns. can be detd. by using a 2-m. column packed with 400-500  $\mu$  diam. Teflon powder coated with 20% by wt. Polyglycol 1500, at 130°, with a flow rate of 98 cc. H<sub>2</sub>/min. Teflon was superior to C22 firebrick as the support phase. The plot of H<sub>2</sub>O peak height vs. vol. H<sub>2</sub>O injected was linear in the range 0.01-1.0  $\mu$ l. The technique is applicable to the detn. of H<sub>2</sub>O in solns. of amines and NH<sub>4</sub> salts. G. W. Hay

6987a, 1963, V. 59

Rapid determination of water vapor in gases by means of detector tubes. Yoshitaka Kobayashi. *Kogyo Kagaku Zasshi* 61, 679-82(1958).—H<sub>2</sub>O in air and industrial gases can be detd. with glass tubes packed with silica gel with added colloidal Se-H<sub>2</sub>SO<sub>4</sub>, crystal violet ZnCl<sub>2</sub>, malachite green-ZnCl<sub>2</sub>, or active Al<sub>2</sub>O<sub>3</sub> with added CoCl<sub>2</sub>. The detn. made by measuring the vol. of gas required to color a definite length of a detector tube. Se-H<sub>2</sub>SO<sub>4</sub> is suited for the detn. of 0.1-0.3% H<sub>2</sub>O in N and O, and active Al<sub>2</sub>O<sub>3</sub> with added CoCl<sub>2</sub> for the detn. of 0.15-1.5% in C<sub>2</sub>H<sub>2</sub> and O. Proper choice of the packing material is necessary.

Takeo Fukutomi

16262 i, 1961

Water content of sour hydrocarbon systems. J. Lukacs and D. B. Robinson (Univ. Alberta, Edmonton, Can.). *Soc. Petrol. Engrs. J.* 3(12), 293-7(1963). Available information on the soly. of H<sub>2</sub>O in hydrocarbon-H<sub>2</sub>S-CO<sub>2</sub> systems is reviewed. A chromatographic technique was developed for detg. low concns. of H<sub>2</sub>O. The exptl. app. consists of an equil. cell where the gas is satd. with H<sub>2</sub>O, an auxiliary cell which is used for a H<sub>2</sub>O reservoir, a high-pressure pump, and the chromatograph. The equil. cell was charged with distd. H<sub>2</sub>O and the gas to be studied was brought to the desired pressure by pumping either Hg or H<sub>2</sub>O into the cell. For studying the CH<sub>4</sub>-H<sub>2</sub>O system, Hg was used as the confining fluid, but for the CH<sub>4</sub>-H<sub>2</sub>S-H<sub>2</sub>O system, H<sub>2</sub>O was used. The contents of the cell were agitated by rocking the cell continuously for  $\leq$  4 hrs. After equil. had been reached, agitation was discontinued and the gas phase was sampled by slowly releasing the gas through an expansion valve. During sampling, Hg or H<sub>2</sub>O was injected into the cell to maintain the const. pressure. The sample from the expansion valve traveled to the chromatograph through 1/8-in. outside-diam. stainless-steel tubing. The expansion valve and the stainless-steel tubing were heated to 300-400°F. to reduce H<sub>2</sub>O condensation on the walls. The sample was continuously purged through the chromatograph and the H<sub>2</sub>O-content detd. at 6-min. intervals. Ten to 20 samples were analyzed for 1 conclusive point at each set of equil. conditions. C. W. Schuck

13070g, Vol. 60

Determination of moisture in foods by gas chromatography. Warren M. Schwecke and John H. Nelson (Gen. Mills Inc., Minneapolis, Minn.). *Anal. Chem.* 36(3), 689-90(1964). The sample (15 g.) is extd. in a Waring Blendor with 100 ml. MeOH and 3-14 ml. sec-BuOH as internal standard. Two  $\mu$ l. is used for analysis on a 5-ft. long by 1/4 in. o.d. Al column, filled with Fluoropak 80 coated with 10% Carbowax 400 and kept at 120°. He flow rate was 65 ml./min. The ratio of the peak heights of water and BuOH can be converted to wt. ratios by means of a calibration curve and gives the amt. of water found in the sample.  
F. Kasler

137869, Vol. 60

Gas chromatography of water traces in hydrocarbons. V. G. Berezkin, A. E. Mysak, and L. S. Polak. *Neftekhimiya* 4(1), 156-9(1964). Traces of H<sub>2</sub>O in C<sub>6</sub>H<sub>6</sub>, PhMe, Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, PhEt, cyclohexane, and isooctane were detd. The examd. hydrocarbon was mixed with a NaAlH<sub>4</sub> soln. in the dimethyl ether of diethylene glycol, and the resulting H<sub>2</sub>O was detd. by gas chromatography in Cu tubes, 100-cm. long and 0.4 cm. in diam., filled with silica gel. The flow rate of the carrier gas (Ar) was 25-30 ml./min. The construction of the gas chromatographic app., permitting the detn. of 2-3 parts H<sub>2</sub>O/million parts hydrocarbon, is described.  
S. Marcinkiewicz

498f, Vol. 61

Water content of paper as a variable in paper chromatography. Arthur J. Tomisek and Paula Wedeles Alkan (Kettering-Meyer Lab., Birmingham, Ala.). *J. Chromatog.* 14(2), 232-7(1964). Control of the H<sub>2</sub>O content of chromatographic paper is necessary for reproducibility with the solvent systems PhOH-H<sub>2</sub>O and BuOH-propionic acid-H<sub>2</sub>O.  
L. A. Horrocks

8d, Vol. 61

Water determination by gas chromatography. O. Francis Bennett (Boston Coll., Chestnut Hill, Mass.). *Anal. Chem.* 36(3), 684(1964). H<sub>2</sub>O was easily detd. quant. in aq. org. systems, of varying polarities, contg. 0.79-7.15% H<sub>2</sub>O with a com. column packed with Teflon powder impregnated with 5% Carbowax 20-M. The max. av. deviation was  $\pm 0.10\%$ . Nearly sym. H<sub>2</sub>O peaks were obtained with essentially no tailing; the retention times of H<sub>2</sub>O showed no variation with changes of concn. in the org. substance. The H<sub>2</sub>O peak height to wt. % ratio, which indicates the dependence of H<sub>2</sub>O peak heights on org. solvents in a relative manner, may be quite different for some systems.  
G. S. Hammaker

11380d, Vol. 60

An industrial method of determining the quantity of water in acetylene solutions. V. E. Stepanenko and S. I. Krichmar. *Khim. Prom., Nauk.-Tekhn. Zh.* 1963(4), 64-8. An accelerated chromatographic analysis method of C<sub>2</sub>H<sub>2</sub> solvents for H<sub>2</sub>O content was developed. As stationary phase, C<sub>2</sub>H<sub>5</sub>(OH)<sub>n</sub>, paraffin oil, C<sub>2</sub>H<sub>5</sub>(OH)<sub>n</sub>, (C<sub>2</sub>H<sub>5</sub>O)<sub>n</sub>, polyethylene glycol adipate, and some alkyl phthalates and phosphates were examd. The best results were obtained with crushed polyurethan (particle size 0.5 and 0.25 mm.) and (C<sub>2</sub>H<sub>5</sub>O)<sub>n</sub>. The stationary phase was applied at the rate of 20 g./100 g. sponge, *in vacuo*. The chromatographic device was a 1 m. long V-shaped tube, with a 6 mm. inner diam., filled with crushed polyurethan and wetted with (C<sub>2</sub>H<sub>5</sub>O)<sub>n</sub>. With dry HCONMe<sub>2</sub>, the analysis time is 9 min.; with N-methylpyrrolidinone, it is 13 min. The accuracy is  $\pm 5.0\%$  with H<sub>2</sub>O concns. of 1-16%. The min. tested H<sub>2</sub>O concn. was 0.05%. A comparison with the Dean and Stark method showed a relative discrepancy of -1.6 to +22.0%. The sensitivity is 0.05%.  
GCJW

1279a, Vol. 61

Absorption on  $Mg(ClO_4)_2$

Determination of total water in rocks by a simple diffusion method. A. D. Wilson (Lab. Govt. Chem. Geol. Surv. Museum, London). *Analyst* 87, 598-600(1962). Water is detd. by absorption on  $Mg(ClO_4)_2$  by using a specially designed app.  
R. Ferramola

9212 g, 1962, V. 57

Calder Hall-type reactor coolant. Analytical requirements and problems. R. W. Dickinson, E. Hughes, A. R. Newcombe, R. C. Williams, and F. J. Woodman (U.S. At. Energy Authority, Seascale, Engl.). *U.S. At. Energy Comm. TID-7606*, 367-90(1960).—Methods are described for the analysis of raw feed  $CO_2$  (liq.) for total  $CO_2$  and of circuit gas in  $CO_2$ -cooled reactors. (1)  $CO_2$  (g) was absorbed in KOH soln. and the residual gas measured. The residual gas (av. 0.02 vol. %) was analyzed for  $CH_4$ ,  $C_2H_6$ ,  $C_3H_8$ ,  $C_2H_2$ ,  $Ar^{40}$ , O, N, and H by mass spectrometry.  $H_2O$  was detd. by absorption in  $Mg(ClO_4)_2$ . B was detd. by using curcumin, after distn. as Me borate. (2)  $H_2O$  was detd. by in-line infrared absorption with a sensitivity of 20 p.p.m. CO was detd. by the  $I_2O_5$  absorption method and in-line infrared absorption.  $C^{14}$  was detd. in CO (residual gas) by oxidn. with  $I_2O_5$  and absorption in boryta.  $CO_2$  was absorbed in boryta. from KOH soln. B activity in the ppts. was detd. by using a liquid scintillation counter.  $\gamma$  Spectra were obtained for  $N^{14}$ ,  $Ar^{41}$ , and  $O^{18}$  in coolant gas. The concn. of  $Ar^{41}$  was detd. by using a  $1 \times 1.5$ -in. NaI(Tl) crystal.  $Ar^{41}$  and  $O^{18}$  were sepd. from  $Xe^{133}$ ,  $Xe^{135}$ ,  $Kr^{81m}$ , and  $Kr^{84}$  by passing 100-200 ml. of residual gas in H at 120 ml./min. through a Linde Type-5A Molecular Sieve, 36-42 mesh BSS. Detn. of Xe and Kr was begun after elution of  $Ar^{41}$  and  $O^{18}$  from the column. This more rapid result gave similar results to detns. after 10 hrs. (to permit decay of  $Ar^{41}$  and  $O^{18}$ ).  
J. S. Morton

23096 g, 1961

New gas-analysis apparatus. E. Fréling and P. Dugleux. *Rev. inst. franç. pétrole* 5, 3-8(1950).—The all-glass app. has a measuring unit of const.-vol. type:  $H_2O$  vapors are adsorbed by  $Mg(ClO_4)_2$ ,  $CO_2$  by Ascarite,  $O_2$  by Hopcalite. CO and  $H_2$  are selectively oxidized by CuO. The mode of operation and the accuracy of the instrument are discussed.  
P. I. Pollak

3 g, 1951

## Absorption in $H_2SO_4$

**Determining small quantities of gases in mixtures.** Imperial Chemical Industries of Australia & New Zealand Ltd. Australian 105,379, Oct. 20, 1938. To det. small amts. of gases which are absorbable by  $H_2SO_4$  with evolution of heat, a temp.-sensitive element is irrigated with the acid and placed in a downward stream of the gas mixt. of such cross section that an outer layer of the gas mixt. flows past the element without appreciable loss of the absorbable constituent, the relative rates of flow of the gas and of the acid being such that the greater part of the heat of absorption is carried away by the gas. An app. is described in its application to the detn. of small amts. of water vapor in gaseous mixts. In a modification, a combination is described in which two instruments are fed with a gas mixt. from a branched tube, one branch contg. an electrically heated chamber contg. a Pd catalyst for converting any O in the gas to water.

2848', 1939

**Apparatus for determining small quantities of gases or vapors.** Imperial Chemical Industries Ltd. Fr. 830,014, July 19, 1938. Water-vapor or other absorbable component of a gaseous mixt. is estimated by placing a temp.-sensitive element such as a Hg thermometer irrigated with  $H_2SO_4$  in a stream of the gaseous mixt. The cross section of the gaseous stream in relation to that of the sensitive element is such that although a layer of the gas surrounds the element there is only a small loss of absorbable component. The rate of flow of the acid is such that the vapor pressure of the acid is not appreciably changed, and the rate of flow of the gas relative to that of the acid is such that the greater part of the heat of absorption is taken up by the gas. O in a mixt. also contg. H is detd. by passing the mixt. over a heated catalyst and estimating the water-vapor by the method described. The method may also be used to det. small quantities of  $NH_3$  in a gaseous mixt.

98, 1939

**Gas analysis.** Frederick W. Haywood, Claude H. Bosanquet, John L. Pearson and Imperial Chemical Industries Ltd. Brit. 491,154, Aug. 26, 1938. In detg. a constituent, e. g.,  $H_2O$ -vapor, of a gas mixt. by measuring the heat of its absorption in  $H_2SO_4$ , a thermometer irrigated with the acid is subjected to a gas current of such cross section that the outer portion of the current escapes the action of the absorbent and preferably not more than 33% of the absorbable constituent is absorbed from the whole of the gas passed. The relative rates of flow of the gas and acid are such that the greater part of the heat of absorption is carried away by the gas, and a rate of gas flow is selected, small variations of which do not appreciably affect the sensitivity of the instrument.

9046, 1939

## Absorption, Using $H_3PO_4$

Determination of water vapor from the change in electrical resistance of a hygroscopic film. Elmer R. Weaver, Ernest E. Hughes, and Albert W. Diniak. *J. Research Natl. Bur. Standards* 60, 489-508(1958)(Research Paper No. 2864).—The elec. cond. of a thin film of an electrolyte changes over a wide range with changes in the concn. of water in the atm. with which it is in contact.  $H_3PO_4$  and  $H_2SO_4$  are most useful since they detect the smallest concns. of water vapor. By adjusting the pressures of a sample of gas of known compn. and one of unknown compn., they can be made to have the same concn. of water (more accurately the same fugacity) shown by the production of equal resistances of the detecting film. Numerous applications of the method are discussed. The method is simple, rapid, and sensitive; only small samples are needed, and few substances interfere.  
Donald Hamm

19687b, 1958

Moisture determination by electrolytic film. E. R. Weaver and Ralph Riley (Natl. Bur. of Standards, Washington, D.C.). *Refrig. Eng.* 55, 266-7(1948); cf. *C.A.* 42, 4096b.—This method has been applied to detg. the moisture content of a gas. A thin film of liquid such as  $H_3PO_4$  in a gelatin or plastic material is spread over the surface of a solid insulator between metallic electrodes. The electrolyte tends to reach equil. with the water vapor in the gas; the cond. of the resultant equil. mixt. is a measure of the water content.  
A. H. Johnson

3843a, 1950

Measurement of water in gases by electrical conduction in a film of hygroscopic material. E. R. Weaver and Ralph Riley (Natl. Bur. Standards, Washington, D.C.). *Anal. Chem.* 20, 216-29(1948).—Detailed descriptions are given of app. and procedures. A film of  $H_3PO_4$  is exposed to a known concn. of water vapor and its elec. resistance measured. The film is then exposed to the gas being tested, and the pressure of the latter varied until the elec. resistance of the film reaches the above-mentioned value. If the total pressure of the sample is known, its  $H_2O$  content can be calcd. In addn. to obvious applications, this method can be used to estimate amts. of combustible gases in air, or of  $O_2$  in combustible gases, by performing a preliminary combustion to  $H_2O$ . Also in *J. Research Natl. Bur. Standards* 40, No. 3, 169-214(1948) (Research paper RP 1865).

Cyrus Feldman

4096b, 1948

## Absorption, Using $P_2O_5$

Moisture in gas streams. G. A. Rath (Beckman Instr., Munich, Ger.). *Dechema Monograph*, 35, No. 528-55, 214-20 (1959). Continuous control detns. of  $H_2O$  in gas streams were made with an app. consisting of an electrolytic cell of 2 spirally-wound Pt electrodes fixed in Teflon and in contact with a thin viscous layer of partly hydrated  $P_2O_5$  contg.  $\geq 75\%$   $P_2O_5$ . The sample gas is led through the cell at a const. flow rate, and a const. voltage is applied. The  $H_2O$  is absorbed by the  $P_2O_5$  and electrolyzed at the same time. A gas flow rate of 100 cc./min. requires a cell length of 50 cm. to ensure reaction of  $>99\%$  of the sample gas and generates a current of 13.2  $\mu$ amp./p.p.m.  $H_2O$ . The lower limit of the  $H_2O$  detn. is 1 p.p.m. The app. is accurate to within 5% for concns. of 1-1000 p.p.m. A temp. correction of 0.3%/degree for temps.  $<298^\circ K$ . and  $-0.3\%$ /degree for temps.  $<298^\circ K$ . should be made. The method can be used to control gas streams that must be  $H_2O$ -free; for Freons,  $SO_2$ -refrigerants, and other liquids with a b.p.  $\leq 100^\circ$ , for liquids in which  $H_2O$  has such a low soly. that it can be removed by gas scrubbing, for compds. which can react to yield  $H_2O$ , for permeability of polymers, and efficiency of drying app.

C. Olivier-Rutgers

6190 C, 1963, V. 58

Discontinuous coulometric determination of water in liquid hydrocarbons. F. Onuska. *Chem. Zvesti* 17, 359-64 (1963). A discontinuous coulometric device was described for the detn. of  $H_2O$  in aromatic liquid hydrocarbons. From the sample vessel the hydrocarbon is brought up by dry N into the coulometric cell where the  $H_2O$  reacts with  $P_2O_5$ .  $H_3PO_4$  is formed and is electrolytically decompd. between 2 Pt electrodes. A compensograph, also serving as a milliammeter, records the current-time relation; the area is proportional to the amt. of  $H_2O$  decompd. by the electrolysis.

Jan Micka

13346, 1963, V. 59

Electrochemical behavior and analytical applications of the Pt- $P_2O_5$ - $H_2O$  system. Michael Czuba, Jr., Kenneth W. Gardner, and Donald T. Sawyer (Bell & Howell Res. Center, Pasadena, Calif.). *J. Electroanal. Chem.* 4, 51-8 (1962). A pair of parallel Pt wires in a 3-ft. helically wound tube of 0.5 mm. inner diam. is the electrole system for the coulometric detn. of  $H_2O$  in gases. The electrolyte is  $P_2O_5$  wet by the gas under investigation. For a flow rate of 100 ml./min., 13.2  $\mu$ amp./p.p.m.  $H_2O$  are passed. A discussion of the polymeric nature of the electrolyte concludes that the electrolysis rate is limited by the anode reaction. Response time of the system varies from 20 sec. at  $22^\circ$  to 70 sec. at  $-33^\circ$ . An erroneously high current for  $H_2O$  in H is attributed to formation of  $PtO_2$  at the anode and oxidn. of H by it to  $H_2O$  in excess of that in the original gas stream.

A. W. Collat

5040 C, 1963, V. 58

Determination of traces of water in gases by means of a modified Keidel electrolytic hygrometer. E. Barendrecht (Staatsmijnen Limburg, Geleen, Neth.). *Anal. Chim. Acta* 25, 402-4 (1961) (in English).—A modification of the Keidel electrolytic cell (CA 52, 11490g; 54, 7242d) is described for amperometrically detg. 1-1000 p.p.m.  $H_2O$  in gases. The 2 spiralized 5-ml. Pt wires, 0.2 mm. apart, are coiled on the outside of a Teflon or glass cylinder. This part of the cell can be removed and cleaned. J. O. Page

4102 f, 1962, V. 56

Coulometric hygrometer for measuring the moisture content of gases. V. I. Istomin. *Neft. i Gaz. Prom., Nauchn. Tekhn. Sb.* 1964(1), 57-9. The operation of the instrument is based on the principle that the gas is passed through a permeable hygroscopic film (contg. partially hydrated  $P_2O_5$ ) which is placed between 2 plate electrodes. The hygroscopic film absorbs the moisture from the gas and  $H_3PO_4$  is formed which continuously undergoes electrolysis and is converted to  $P_2O_5$ . The instrument can be used for hydrocarbon gases with a moisture content of 0.01-50 g./m.<sup>3</sup> at pressures up to 60 atm. It is not recommended for HCl,  $NH_3$ , ales., O, or H.

GPJR

1521 C, 1964, V. 61

## Using Oxalic Acid

New electric method for determining moisture content. J. Boeke (Philips Research Lab., Eindhoven, Holland). *Philips Tech. Rev.* 9, No. 1, 13-15(1947).—The method is applicable to the detn. of H<sub>2</sub>O in liquid or solids. The H<sub>2</sub>O is extd. with a soln. of 10% oxalic acid in acetone. The increase in cond. is a function of the amt. of H<sub>2</sub>O taken up. The app. is inexpensive and simple in operation. The substance to be tested is kept in intimate contact with the acetone soln. for about 2 hrs. All parts of the app. are made of glass or porcelain except the electrodes which are of Pt, such as are used in standard cond. cells. Calibration curves are shown. C. G. F.

5414 g, 1947

Determining moisture in gases. V. A. Komarov. *Russ.* 51,904, Oct. 31, 1937. The moisture is absorbed in an org. solvent, an electrolyte (e. g., oxalic acid) is added, and the water detd. by measuring the elec. cond.

3624<sup>3</sup>, 1940

## Using Dioxane

Dioxan method for moisture determination in gypsum-bearing and salinized soils. A. V. Nikolaev, R. M. Nikol'skaya, and Yu. D. Shcherbakov. *Pochvoedenie* 1964(3), 105-8. The method of Marel (*CA* 54, 3820g) was modified for the detn. of humidity in the title soil types. A dioxan ext. of the moist soil is placed in a vessel-transistor and the elec. capacity detd. Control detns. with drying expts. showed a possible error of  $\pm 1\%$ .

L. Schuler

164512, U. 60

Analysis of mixtures. LUDWIG EBERT and ERNST WALDSCHMIDT. Ger. 573,072, Nov. 29, 1930. To det. the proportion of one constituent of a mixt., the constituent is extd. by means of a solvent, and the dielec. const. of the soln. is compared with that of the pure solvent. The solvent selected must have a dielec. const. markedly different from that of the constituent. The method may be applied, e. g., to gas analysis and to the detn. of the moisture content of various substances, dioxane being used as the solvent for water. App. and a continuous system of analysis are described.

2909, 1933

The determination of water in pigment pastes and printing inks by a modified extraction method. Friedrich Oehme. *Farbe Lack* 66(3), 142-5(1960). The accuracy of the dioxane extn. method for detg. the H<sub>2</sub>O content of pigment pastes or printing inks, which is based on the measurement of increases in the dielec. const. of the solvent contg. H<sub>2</sub>O, is affected to a considerable extent by the fact that dioxane exts. not only H<sub>2</sub>O, but frequently other components of the materials tested. Measurements of the dielec. const. of dioxane contg. various pigment impurities showed, however, that the slope of plots of H<sub>2</sub>O content vs. dielec. const., which are nearly straight lines, remains practically unchanged by the impurities, so that a series of parallel straight lines is obtained. The modified method developed by O. consists in extg. a sample of the tested material with dioxane, measuring the dielec. const. of the ext., then removing the H<sub>2</sub>O quant. and selectively by filtering the ext. through a mol. sieve. The difference between the dielec. consts. of the H<sub>2</sub>O-contg. and H<sub>2</sub>O-free dioxane is proportional to the amt. of H<sub>2</sub>O extd. The proportionality const., which is detd. by the type of measuring app. used, is calcd. from standard dioxane-H<sub>2</sub>O mixts. The accuracy of the modified method is  $\pm 1\%$ . From *Abstr. Bull. Instr. Paper Chem.* 32(7), Abstr. No. 4850 (1962).

TCBF

15497a, 1963, V. 59

Differential dielectric apparatus for determining water added to solvents. Winthrop C. Wolfe (Natl. Bur. of Stds., Washington, D.C.). *Anal. Chem.* 35(12), 1884-7(1963). The moisture content of grain, for example, was detd. by measuring the change in dielec. const.,  $\epsilon$ , of an ext. obtained with dioxane (I) or (EtO-C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O (II). Dual matched capacitance cells, one of which was filled with solvent and the other with the ext. were alternately connected to a capacitance bridge or a Q-meter. For C (% water) in the range 0-1,  $de/dC$  was const. The temp. coeff. of  $de/dC$  in I was detd. at 15-35° and agreed closely with the data of Tourky, Rizk, and Girgis (*CA* 55, 21723g). At 30°,  $de/dC$  was 0.27 in I and 0.61 in II.

Thomas B. Hoover

1084e, 1964, V. 60



## Using Pyridine

**Determination of water, ethanol, and perfume compositions in perfume manufacture.** Janusz Kulesza, Jozef Gora, and Janina Zwierzchowska (Politech., Lodz, Poland). *Zeszyty Nauk. Politech. Lodz., Chem. Spozycza* No. 5, 35-47(1961).  
Detn. of EtOH: (a) The acetylation method. A sample (0.5 g.) was treated with 11 ml. 3:1 pyridine-Ac<sub>2</sub>O for 2 hrs. at room temp., heated with 30 ml. hot H<sub>2</sub>O, and cooled to room temp. The liberated AcOH was titrated with 1N NaOH by using phenolphthalein. (b) The oxidation method. A sample contg. ~10 g. EtOH was dild. to 500 ml. with 2N H<sub>2</sub>SO<sub>4</sub>, and the aq. layer was treated with 1 g. talc and filtered. The filtrate (50 ml.) was mixed with 50 ml. 2N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and 50 ml. 50% H<sub>2</sub>SO<sub>4</sub> (d 1.38), for 2 hrs. at 20-5°, and the mixt. dild. to 500 ml. with H<sub>2</sub>O, 50 ml. of which was treated with 2 g. KI and the liberated I was titrated with 0.1N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. The accuracy of the acetylation method was ±2.5% and that of the oxidation method ±0.5%.  
Detn. of H<sub>2</sub>O: A sample contg. 1 g. H<sub>2</sub>O was dild. to 100 ml. with MeOH and 10 ml. of which was titrated with the Van der

Muelen reagent, prepd. according to Lada (CA 53, 13873h) with an accuracy ±0.33%. Detn. of perfume compn.: A sample (5-10 g.) was dild. with 50% its wt. of H<sub>2</sub>O, extd. 3-4 times with 15-20 ml. CHCl<sub>3</sub>, the exts. treated with 6 g. di-Bu phthalate or castor oil and 15 ml. MeOH, the solvent removed by evap. to dryness at 160 mm. and 70°, and the residue weighed. The accuracy was ±1.2%.  
A. Janowski

5312, U. 61

**Phase titrations. III. New applications including the assay of water in pyridine.** D. W. Rogers and A. Ozsogomonyan (Robert Kolej, Bebek, Istanbul). *Talanta* 11(3), 652-5(1964) (in English); cf. CA 59, 3297c. The assay of water in org. solvents by means of phase titration is discussed in relation to the chloranilic acid method of Barreto and B. (CA 57, 4036f) and the older cloud-point technique. Results are given for the titration of water-pyridine solns. with CHCl<sub>3</sub>, as representative of the phase titration of a N-contg. compd. to which the chloranilic acid method is inapplicable. In addn., results are given for the phase titration of 18 new binary addns. using water as the titrant. They are: bromobenzene, CHCl<sub>3</sub>, and 1,2-dibromethane, each in binary solns. with MeOH, EtOH, iso-PrOH, dioxane, acetone, and HOAc.  
RCTB

8622g, U. 60

**[Infrared] determination of traces of water in organic solvents.** J. Neuvirt and Z. Ksandr (Vysoka Skola Chem.-Technol., Prague). *Collection Czech. Chem. Commun.* 29(4), 1068-72 (1964) (in German). A new differential method is based on the fact that the absorption band of H<sub>2</sub>O in inactive solvents differs from that in active solvents, where the H<sub>2</sub>O is assocd. An active solvent is added to the inactive one in the measuring cell and the absorption is measured against a comparison line where the two solvents are contained in two separate cells. A calibration curve was based on benzene satd. with H<sub>2</sub>O. Pyridine was used as the active solvent, and the measurements were carried out at 3400 cm.<sup>-1</sup> The soly. of H<sub>2</sub>O in toluene, 1,2-dichloroethane, and PhNO<sub>2</sub> at 21° is, resp., 2.26, 6.45, and 12.7 millimole/100 ml. The method is simple, rapid, and requires no abs. dry solvent. The abs. error is 0.07 millimole H<sub>2</sub>O/100 ml. of the sample. The method cannot be applied to solvents which form strong H bonds with H<sub>2</sub>O or which absorb intensely near 3400 cm.<sup>-1</sup>  
E. Erdos

12665h, U. 60

Miscellaneous Physical Methods; Changes of  
Properties in Absorbents, Solvents, Etc.

The determination of moisture by measurement of the dielectric constant. FELICE GARELLI AND GIOVANNI RACCIU. *Atti accad. sci. Torino* 66, 338-44(1931).—A description of the method of E. Berliner and R. Rüter (*C. A.* 24, 7). A no. of detns. of moisture contents of grains are made which show that the results are reliable and can be made in a few min., whereas in the ordinary methods drying periods of 36 hrs. or more are needed. A. W. CONTIERI

2672, 1932

Study of the dielectric constants of substances containing water. J. Tausz and H. Rumm. *Kolloid-Beihfte* 39, 58-104(1933).—The dielec. properties of sucrose, starch (potato, rice, wheat), silicic acid, tobacco (fibers and dust), borax, lithographic slate and lignite were studied, as a function of the moisture content. Water is present in a solid and a liquid fraction. The effect of temp., packing vol., structural properties and frequency was detd. The dielec. const. of dried materials can be measured by an isodielec. method: the materials are suspended in a mixt. of liquids of the same dielec. const. The dielec. const. of sucrose and starch in the literature could not be confirmed, because previous investigators had not dried these substances properly. Substances of higher moisture content were placed between the condenser plates in powder form. Dielec. values were thus obtained, from which dielec. const. can be calcd. Dielec. measurements are a convenient way of *detg. moisture content*. Substances contg. moisture in the form of solidified water show a low temp. coeff. of the dielec. const. At room temp. 9% of the water in cut tobacco is present in the solidified form. Water in  $\text{SiO}_2$  is mostly in the liquid state. The dielec. const. varies considerably with frequency, if the water content is liquid. The lithographic slate of Solnhof shows an abnormally high dielec. const. of 1000-1100 at a frequency of 1100. After drying, i. e., removal of 0.7% moisture, the dielec. const. is 14 (Johnson-Rahbeck effect). This is due to capillary water which is rocked back and forth by a. c. The capacity of *Se* increases when illuminated. Frank Urban

2960<sup>4</sup>, 1934

Condenser for the determination of water content of bulk material. Gann Apparate-und Maschinenbau-G.m.b.H. (by Friedrich Hirth). Ger. 1,130,622 (Cl. 42), May 30, 1962, Appl. July 2, 1958; 3 pp. The  $\text{H}_2\text{O}$  content is detd. by measuring the capacitance of the system.

M. Kenigsberg

5310 a, 1962, V.57

19182 A MOISTURE DETECTING UNIT BASED ON POROUS SEMICONDUCTOR FILM.

B.S.Tul'chinskii and V.D.Yurasov.

Pribory i Tekh. Eksp. (USSR), 1961, No. 2, 183-4 (March-April). In Russian. English translation in: Instrum. exper. Tech. (USA), No. 2, 392-3 (March-April, 1961; publ. Dec., 1961).

PA 1962

[Determination of the] composition of water-phenol mixture by refractive index. H. Aring and U. v. Weber (Univ. Rostock, Ger.). *Z. Chem.* 2, 344-5(1962). Utilizing the large difference in the  $n$  of  $\text{H}_2\text{O}$  and PhOH, the compn. of a mixt. of  $\text{H}_2\text{O}$  and PhOH can be found by measuring  $n_D^{20}$ . A standard curve was prepd. by measuring  $n_D^{20}$  of synthetic mixts. PhOH-poor mixts. were analyzed by iodometric detn. of PhOH, PhOH-rich mixts. by Karl Fischer titration of  $\text{H}_2\text{O}$ . J. C. Chang

6190 d, 1963, V.58

**Determination of water in alkali metal hydroxides.** Richard Jodl (Lübeck, Ger.). *Seifen-Öle-Fette-Wachse* 76, 533-4(1950).—Heat the sample to 350-450° in a Ag boat inside a glass tube in an air stream free from H<sub>2</sub>O and CO<sub>2</sub> for 10-20 min., and det. the increase in wt. of a U tube filled with silica gel (blue gel). The water of hydration of metal hydroxides is partly or wholly included in the detn.  
Maria E. W. Torok

2821a, 1951

**Rapid industrial method for water and oxide determination of metal powders.** Miklos Devenyi, Imre Kasa, and Gyorgy Liptay (Hardmetal Ind. Co., Budapest, Hung.). *Magy. Kem. Lapja* 18(6), 291-2(1963). Dried and O-free H is led into a heated SiO<sub>2</sub> tube contg. the metal powder sample. Metal oxides present are reduced, and the H<sub>2</sub>O formed is absorbed in Mg-(ClO<sub>4</sub>)<sub>2</sub> and P<sub>2</sub>O<sub>5</sub>-filled U-tubes; the wt. difference represents the O and H<sub>2</sub>O content of the powder sample. The absorbents do not take up oils and other org. contaminants present in the metal powder sample. In the case of Fe, Co, Ni, and steel powders at 1200° the redn. was complete in 30 min., at 1100° in 60 min.; lower temps. require even longer redn. time. Redn. temp. can be lower (700-900°) with lower-melting metal powders, depending on the metal. Accuracy is within 2%. The method is suitable for continuous detn.  
Peter Marcel Barna

12166h, 1963, V.59

**Simple vapor sorption method for determination of water in liquids.** Sherril D. Christian and Harold E. Afsprung (Univ. of Oklahoma, Norman). *Anal. Chim. Acta* 29(6), 586-8(1963). A modified McBain sorption balance is used. A helical SiO<sub>2</sub> spring is suspended from the glass hook attached to the inside of the standard taper cap. Approx. 30 mg. of desiccant is in the Al foil bucket. To operate the app., evacuate (through the 3-way vacuum stopcock), close the stopcock, and introduce the sample from a pipet through a valve consisting of a medium-porosity sintered glass disk covered with Hg. Record the sample wt. when it becomes const. Wt. of H<sub>2</sub>O is calcd. from initial and final heights of the glass hook on the lower end of the SiO<sub>2</sub> spring. Care must be taken to ascertain that only H<sub>2</sub>O sorbed by the desiccant is measured. Other volatile components must be removed, i.e. C<sub>2</sub>H<sub>4</sub>, can be removed by placing the lower end of the app. in an ice bath. Results of the sorption balance and the Beckman KF-3 aquameter are compared.  
L. Dupuis

2327c, 1964, V.60

**Determination of components of mixtures of solvents or of solvent vapors in air.** N. K. Baramboim and Yu. B. Kipnis. U.S.S.R. 117,304, Feb. 6, 1959. A layer of an indicator is applied to an inert base. The indicator is one which swells when exposed to the desired component or to its vapors. The indicator is applied to a thin metallic base, inert to the substance as well as to the other components of the mixt. Thus, to det. moisture in air or the H<sub>2</sub>O content of acetone or alc., a 30-50- $\mu$  layer of the higher fractions of gelatin was applied to a 10-15- $\mu$  sheet of Be bronze. Instead of gelatin, a layer of poly(vinyl alc.) may be used.

One end of a 5 x 10-mm. sheet thus prepd. was fastened in a vise. The other end curled to an extent depending on the moisture in the air or the H<sub>2</sub>O content of acetone or alc. To det. the phenol content of waste waters, the indicator layer is made of a polyamide; to det. halogen derivs. in the air or in liquids, an indicator layer of poly(vinyl chloride) is used; to det. the content of aromatic hydrocarbons in aliphatic hydrocarbons, an indicator layer made of polystyrene or chlorinated natural rubber is cured; and to det. the presence of petroleum products, an indicator layer of structured rubber is used.  
M. Husch

17159a, 1960

Utilization of modulated light in photoelectric analyzers for the determination of gas in industrial control. C. Cherrier, L. Verot, and R. Wagner (Lab. central Sté de St. Gobain, Paris). *Spectrochim. Acta* 6, 87-94(1953).—An app. is described for following in the factory the progress of a vapor-phase reaction by absorptiometric measurements. The app. det. the instantaneous value of the component in a pipe, operates alarm relays if it departs from preselected values, and may control valves. The types of app. described are accurate, simple, and robust, with electronic amplifiers of rugged type. The detectors used are photomultiplier cells with chopped beams and electronic a.-c. amplifiers; the chopping may be by a sector disk but a mercury vapor source, modulated at mains frequency, is preferred. Compensating-beam layout of the app. is used, and the amplifier has sufficient feedback to secure stability while having enough stages to secure high gain. Standardization of the amplifier is by two built-in filters giving selected output values. As examples of the application of the app. are the detn. of traces of Cl in synthetic HCl, and detn. of NO<sub>2</sub>, of SO<sub>2</sub>, and of moisture in a gas. W. F. M.

17359, 1954

7088 SOME EXPERIMENTS ON MOISTURE DETERMINATION WITH BETA-RAY TRANSMISSION.

T. Westermark, B. Sjöstrand and H.G. Forsberg. Nuclear Instrum. and Methods (Netherlands), Vol. 10, No. 1, 45-7 (Jan., 1961).

By using a transmission geometry of beta rays from Pm<sup>147</sup> it is shown that recording of moisture changes in hydrophilic materials — kinetics as well as equilibria — is readily achieved. Some data from experiments on filter paper are described and further applications are discussed.

PA 1962

Apparatus for determining the moisture content of a material by determining the dielectric losses due to the moisture. Willis R. Whitney (to The British Thomson-Houston Co. Ltd.). Brit. 408,414, Apr. 12, 1934.

5294<sup>2</sup>, 1934

Apparatus for indicating the moisture content of wood, etc., during drying. FRANZ SCHENK. Ger. 571,117, Oct. 7, 1930.

Apparatus for determining the water content of organic substances. KEIICHI IMO'o. Japan. 95,386, April 8, 1932. The water content of org. substances (such as cellulose, tea or grains) is proportional to the humidity of spaces between the particles. An app. designed to det. this humidity consists of a hollow perforated tube contg. a thermometer and a hygrometer.

2608, 1933

Continuous weighing for the determination of traces of water in NH<sub>4</sub>NO<sub>3</sub>. Marcel Guichard. *Compt. rend.* 215, 20-1(1942).—Heat the NH<sub>4</sub>NO<sub>3</sub> crystals to 119° while weighing continually. The decompn. of NH<sub>4</sub>NO<sub>3</sub> yields H<sub>2</sub>O at a linear rate over a long period of time, while the intercryst. H<sub>2</sub>O is released over a short period. Extrapolation of the long-period H<sub>2</sub>O loss to the origin gives a method for evaluating the initial loss due to intercryst. H<sub>2</sub>O.

Earl A. Gulbransen

5470<sup>2</sup>, 1944

**Rapid method for controlling moisture.** Yu. M. Rapoport and E. G. Chofnus. *Zavodsk. Lab.* 25, 1346-8(1959). A method is described for detg. the moisture in graphite samples based on the relation of the elastic modulus to the moisture content (Obert and Duvall, *C.I.* 36, 3023<sup>1</sup>). The method was used to det. the effect of process conditions (compn. of the sample, initial moisture content, pressure in forming bars, and size of the sample) on the frequency of the natural vibrations of the sample, which are related to the elastic modulus and the d. The moisture content of semi-dry samples was detd. with an accuracy of 0.3%, that of wet samples with an accuracy of 1%. CA

4035 I, 1962, v. 57

**Polarimetric method for the determination of moisture content.** A. Ya. Petrovskaya and Yu. V. Mikhailovskii. *Inzhener.-Fiz. Zhur., Nauk Belorus. S.S.R.* 3, No. 6, 99-100 (1960).—A polarimetric method is proposed for the detn. of the moisture in friable material, in oils, and lubricants. The rotatory power of a H<sub>2</sub>O soln. of an optically active substance (sugar) is measured before and after being agitated with a detd. quantity of substances of which the moisture is to be ascertained. From the variation of the rotatory power, the variation of the concn. of soln. is calcd., and from the latter, the moisture of the sample. The method is patented (U.S.S.R. 62, 444). A. Giacalone

212 h, 1961

**Estimating moisture in solid materials.** Siemens-Schuckertwerke A.-G. (Albrecht Werren, inventor). Ger. 670,261, Jan. 14, 1939 (Cl. 42/ 9.51). A description is given of a method of estimating moisture in wood, paper fabric and other solid materials by subjecting the materials to high-frequency mech. oscillations and observing the effects, e. g., the rate at which the oscillations are transmitted through the materials.

2848<sup>3</sup>, 1939

**Apparatus for measuring the thickness, moisture-content or other characteristics of a material, wherein the material is arranged to be included in a high-frequency oscillatory test circuit so as to influence the reactance thereof.** The Foxboro Co. Brit. 444,056, Mar. 12, 1936.

6247<sup>3</sup>, 1936

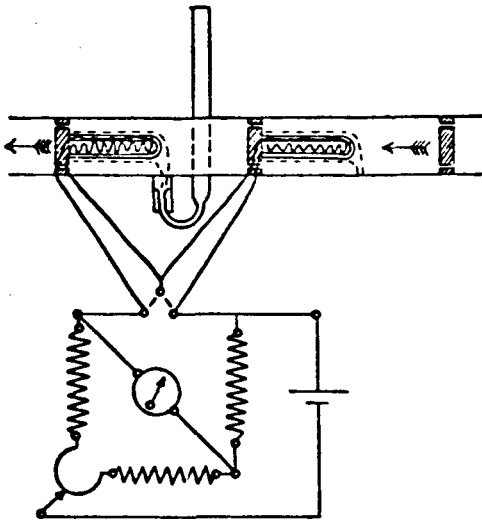
Determining the amount of water, etc., in materials. PAUL W. GLAMANN. Ger. 518,817, Nov. 15, 1928. The amt. of water or similar substance present is detd. by changes of such phys. properties as sp. heat, vol., etc.

3270, 1931

Apparatus for determining the moisture content of substances. Siemens & Halske Akt.-Ges. (by Theodor Rummel). Ger. 1,028,813, Apr. 24, 1958 (Cl. 42/). Weighing—before and after drying in a high-frequency field is utilized.  
Friedrich Epstein

19047 C, 1960

The determination of the water set free by man kept in a differential calorimeter. A. K. NOYONA AND W. STRICKER. *Compt. rend. soc. biol.* 90, 945-8(1924).—To det. the amt. of water set free in a calorimeter it is essential to have a system which is capable to absorb within a given time all the water produced and to maintain and control the humidity of the app. The latter object is attained by means of an arrangement shown in the fig. This consists of 2 glass tubes closed at one end and attached to hard rubber disks which are perforated to permit free passage of the air current. Cu wire is wound about these tubes which have a resistance of 50 ohms each. The tube is protected by a somewhat larger tube of thin glass which fits over it and is in turn covered with muslin. One of these is dry while the other is kept constantly moist. The resistance of the Cu wires varies with the temp. and as that of the tube covered with the moist muslin is lower the difference between the 2 tubes is detd. by means of Wheatstone bridge, which detects a temp. difference of 0.0026°. In this way the completeness of the absorption of H<sub>2</sub>O in the app. is checked.



S. MORGULIS

2183, 1924

21154 DIRECT-READING BALANCES FOR PERCENTAGE MOISTURE CONTENT.

J.G.Downes, B.H.Mackay, L.G.Bellamy and V.D.Burgmann. *J. sci. Instrum.* (GB), Vol. 38, No. 12, 464-8 (Dec., 1961).

This paper describes two types of balance which indicate per cent moisture content directly, and in which the initial sample weight may vary within wide limits. They were designed primarily for measuring the moisture in wool at various stages in processing and for this purpose are used in conjunction with a hot-air drying apparatus. The same balances can be used to determine the percentage content of removable components in many other materials. An analysis of the principles of operation of the balances is given and experimental models are described.

7A 1961

An apparatus for tracing the change in moisture content of small quantities of powder. LESLIE H. LAMPITT AND JOHN H. BUSHILL. *J. Sci. Instruments* 6, 369-73 (1929).—P. L. du Nouy's tension balance for surface-tension detns. is modified for this purpose so that a change in wt. of 0.001 mg. may be detected. The app. is shown and results are given.  
I. H. MOORE

992, 1930

Apparatus for determining the percentage of moisture in hygroscopic materials, e. g., cotton and wool. GHERZI-TEXTILE DEVELOPMENT CO. LTD. Brit. 368,815, Dec. 8, 1930.

Means for determining the percentage of moisture, or of paraffin, in stratified materials such as cloth or impregnated paper by electrical capacity measurements. SIEMENS & HALSKE A.-G. Brit. 367,839, Apr. 26, 1930.

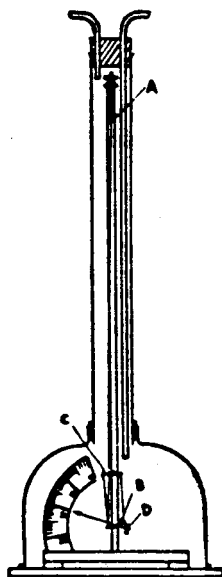
2608, 1933

Apparatus for measuring the moisture of gases. B. V. BOFOVSKII. Russ. 29,964, Mar. 21, 1932. The apparatus is equipped with a gauze sensitive to moisture (expansion and contraction) connected to a gage.

4139, 1933

Determination of minute quantities of water and water vapor by the hygrophotographic method. Joseph Sivadjian and F. Corral (Pasteur Inst., Paris). *Anal. Chim. Acta* 26, 185-90(1962)(in English); cf. *CA* 54, 10643e. A more accurate calibration procedure is given for the hygrophotographic method of detg. traces of H<sub>2</sub>O. The amt. of H<sub>2</sub>O absorbed by the hygrophotographic plate during a 3-18-min. exposure to H<sub>2</sub>O vapor is linear with time of exposure. At satn. (equiv. to 18 min. at 22°), 3690  $\gamma$  H<sub>2</sub>O is absorbed by a 19.4-mm.-diam. (area = 295.5 sq. mm.) reference circle of the hygrophotographic plate, equal to 0.69  $\gamma$  H<sub>2</sub>O/sq. mm./min. J. O. Page

48 b, 1962, v. 57



Device for measuring small quantities of moisture in gases. A. J. CROCKATT AND R. B. FORSTER. *J. Soc. Chem. Ind.* 38, 95-67(1919).—The app. (see fig.) is made of metal and placed in a bell-jar fitted with a wide glass tube. To the upper end of the latter is fitted a rubber stopper containing the gas inlet (short tube) and outlet tubes. One end of an artificial (Chardonnet) silk fiber (about 20 in. long) is attached to the adjustable hook *A* and the other to the short arm of the first magnifying lever *B*. The long arm of the latter is attached to the short arm of the second lever *C*, the long arm of which is attached to the pointer. The latter connections are made with very fine metallic wire. By these means the elongation of the fiber is so magnified that one scale division represents a difference of about 0.05% H<sub>2</sub>O. The tension on the fibers may be adjusted by altering the position of the milled nut *D* on the pointer. F. W. SMITHER

1658, 1919

Determination of water by drying. M. DOLCH AND K. BÜCHE. *Pflanzenbau (Abt. A Wiss. Arch. Landw.)* 4, 64-73(1930).—Errors are discussed. Alternatively, the water is exhd. by repeated treatment with EtOH, the aq. EtOH being mixed with petroleum, and the temp. of sepn. compared with the curve given by control mixts. B. C. A.

2394, 1932

5033, 1932

Modern procedures and apparatus for trace analysis in chemical products. A. Hanes, D. Sandulescu, and C. Lupu. *Rev. Chim.* (Bucharest) 12, 412-15(1961).—Procedures used for the detn. of trace impurities in chem. products are described. Coulometry is used to det. H and H<sub>2</sub>O, potentiometry to det. CO<sub>2</sub>, conductimetry to det. H<sub>2</sub>S, SO<sub>2</sub>, and NH<sub>3</sub>, polarography to det. metals. Other methods described are absorption spectrophotometry, x-rays, emission spectroscopy, activation analysis, and chromatography.  
C. Heitner-Wirgin

53792, 1962, V.56

A new method for determining small moisture contents in gases. A. Perlick and R. Perlick. *Kältetechnik* 6, 271-4 (1954).—Moisture in the range 0.05 to 5 g./cu. m. can be measured in air by a new elec. app. based on variation in frictional coeff. when graphite brushes rub on a rotating Cu cylinder. A simple bridge circuit provides voltmeter readings which are proportional to motor speed and thus indicate moisture contents. The principle is based on a previously known phenomenon; i.e., in very dry air, the frictional coeff. between graphite brushes and commutators of elec. machines becomes so high as to result in rapid brush wear. Different gases behave differently. As the moisture content decreases the frictional coeff. increases in air and O, but decreases in N, and does not change much in CO<sub>2</sub>. Results of expts. are interpreted on basis of adsorption theory and an attempt is made to show how these results as well as observations previously reported by others can be reconciled on theoretical grounds. Conclusion: High heats of adsorption lead to high frictional coeffs. but this rule probably does not

hold for gases contg. O because the sheared-off graphite particles become oxidized.  
L. C. Flowers

779h, 1955

Determination of small amounts of water and hydroxyl groups by the method of deuterium exchange with mass spectrometric control. L. T. Zhuravlev, A. V. Kiselev, V. P. Naidina, and A. L. Polyakov (M.V. Lomonosov State Univ., Moscow). *Zh. Fiz. Khim.* 37(9), 2054-61(1963). The quant. detn. of structural and adsorbed H<sub>2</sub>O, (specifically OH groups) on the surface of adsorbents and catalysts (Acrosil, KSK-2, silica gel) was carried out by the method of D exchange (CA 57, 87c; Haldeman, CA 50, 13332h) using a mass spectrometer to det. the isotopic compn. of the vapor. The method can be used for highly dispersed solids without destroying the sample by heating. The results obtained are reproducible and do not differ with those obtained by gravimetric methods by more than 2%. The method should have a wide range of application in detg. small amts. of H<sub>2</sub>O, OH groups, or other functional groups which can enter into D exchange with the vapors of D<sub>2</sub>O, e.g. those present on the surface of adsorbents, fillers, pigments, synthetic polymers, and many biopolymers.  
GLJK

14576f, 1963, V.59



Some new measurement and control devices. D. J. Pompcio, J. Parker, and S. S. Smith (Shell Development Co., Emeryville, Calif.). *Proc. 3rd World Petroleum Congr.*, Hague 1951, Sect. VI, 506-22.—The following specialized instruments are described: A continuous recording titrimeter for measuring mercaptans in gasoline, a dielec. const. recorder for measuring H<sub>2</sub>O content of ketones, a recording viscometer for lubricating-oil applications, and a method of controlling centrifugal filters, used to sep. a slurry into solid and liquid components. A description is also given of the instrumentation and method for monitoring and controlling pipeline stations by teletype from a remote-control dispatching point. Marta Tausz

11766 f, 1954

Determination of water in amyl alcohol by a modified conductance method. G. Narsimhan and P. S. Mene (Nagpur Univ.). *Trans. Indian Inst. Chem. Engrs.* 4, 91-6(1950-51).—Measurements of the H<sub>2</sub>O content of AmOH are needed in refining and dehydration of solvent. The conductance method, not ordinarily applicable to liquids because of low dielec. const., was modified by satg. the dissolved H<sub>2</sub>O with KCl. AmOH, contg. 10.5% optically active isomer, was used for the tests and detns. of the soly. of H<sub>2</sub>O which varied from 8.98 g. at 20° to 11.77 g. per 100 g. of satd. soln. at 70°. Specific resistance was measured at 20°, 25°, and 30° after stepwise addns. of satd. KCl soln. to AmOH to the formation of 2 layers. The detn. of H<sub>2</sub>O is carried out by shaking AmOH with KCl at a chosen temp. and measuring the specific resistance. The H<sub>2</sub>O content is read from an alignment chart. Arthur Fleischer

6308 d, 1953

Electrical moisture determination in crude petroleum. V. I. Korchagina, S. A. Ginzburg, A. A. Fin'ko, L. I. Rutman, I. V. Davydov, and D. A. Lavrinovich (Petroleum Refinery, Odessa). *Neft. i Gaz. Prom., Nauchn.-Tekhn. Sb.* 1962(2), 51-6. A method is described for detg. the H<sub>2</sub>O content of crude oil emulsions, based on measurements of the dielec. permeability. A suitable app. is described, and a nomogram is given relating the H<sub>2</sub>O content to the d<sub>2</sub><sup>0</sup> and dielec. const. The nomogram covers a range of up to 12.2% H<sub>2</sub>O for d<sub>2</sub><sup>0</sup> values from 0.85 to 0.91 and dielec. const. from 2.31 to 3.156. For the oils tested, the degree of dispersion had little effect on the dielec. const.

C. E. Stevenson

7276 a, 1963, U.59

The carbon dioxide content of milk during handling, processing, and storage, and its effect upon the freezing point. A. C. Smith (Univ. of Connecticut, Storrs). *J. Milk Food Technol.* 27(2), 38-41(1964). Milk samples obtained from a bulk tank at the conclusion of each of 4 milking periods, subsequently from a plant storage tank, and after processing and storage for 0, 2, and 4 days were analyzed for CO<sub>2</sub> content and f.p. Changes in CO<sub>2</sub> content which occurred during storage of raw or pasteurized milk, handling, or pasteurization were not of sufficient magnitude to alter the f.p. significantly. Therefore, the use of f.p. as a means of detecting adulteration of milk with H<sub>2</sub>O should not be complicated by normal handling, processing, and storage of milk. The significant redn. of CO<sub>2</sub> which occurred during vacuum treatment of milk resulted in a rise in its f.p. Allowances for this change should be made in adulteration detns. 19 references. E. H. Marth

16417 f, U.60

Physical methods in the chemical laboratory. XXI. New applications of dielectric investigations for technical and analytical measurements. L. Elert. *Angew. Chem.* 47, 305-15(1934); cf. *C. A.* 27, 38.—The fundamental principles of the dielec. const. (D. K.), electronic, atomic and orientation polarization, the measuring of D. K. and its application to the investigation of liquids as well as solids are discussed. Specific applications are presented for (1) the control of particle size of powders, (2) the control of the thickness of wires and bands and (3) the Exlan method, a method for the comparison of the hydration capacity and mech. structure of solids and the quant. H<sub>2</sub>O detn. in solid systems and systems rich in H<sub>2</sub>O. Eight references. Karl Kammermeier

4951<sup>4</sup>, 1934

Physical methods of moisture measurement. I. Conductivity and dielectric methods. A. Pande (Shri Ram Inst. Ind. Res., Delhi, India) and C. S. Pande. *Industr. Pract.* 16, 896-902 (1962). II. Dielectric, sonic, ultrasonic, microwave, and electrolytic methods. *Ibid.* 988-95. III. Infrared and NMR [nuclear magnetic resonance] methods. *Ibid.* 1104-10. IV. Neutron scattering and mass spectrometric methods. *Ibid.*

1246-50. The methods and the range of applicability of each are considered. 127 references. S. J. Hawkes

11957 h, 1963, V. 58

Determination of water in acetone. Ray L. Jackson (Tennessee Eastman Co., Kingsport). *Appl. Spectroscopy* 15, 85(1961). CA

6663 L, 1962, V. 56

Infrared analyzer for monitoring water content. F. W. Karasek and E. C. Miller (Phillips Petroleum Co., Bartlesville, Okla.). *Ind. Eng. Chem.* 46, 1374-6(1954).—Water, up to 0.4 wt. %, is detected in a liquid SO<sub>2</sub>-gas oil extn. unit with a continuous infrared analyzer utilizing a high-pressure cell. The 2.67  $\mu$  water absorption band was found to be sufficiently sensitive and sufficiently removed from SO<sub>2</sub> and hydrocarbon bands. The accuracy is  $\pm 0.01$  wt. % H<sub>2</sub>O. Walter Roth

11766 g, 1954

Determination of moisture. V. I. Sogreshilin. *Russ.* 44,059, August 31, 1935. The moisture content is detd. by the change of  $n$  of the liquid (glycerol) used to ext. the moisture.

33006, 1938

Quantitative analysis by near-infrared spectrometry. Kaoru Sakai, Fumio Mizuniwa, Tsurayuki Akamatsu, Shozo Matsumoto, Yasuo Kurihara, and Masami Shimizu (Hitachi Ltd., Ibaragi). *Kogyo Kagaku Zasshi* 62, 632-6 (1959). The detn. of H<sub>2</sub>O in BuOH and D<sub>2</sub>O by near-infrared spectroscopy was investigated. By utilizing the O-H combination band near 1.92  $\mu$  with a compensation method, up to 1.2% H<sub>2</sub>O in BuOH can be detd. with standard deviations  $\leq \pm 1.1\%$ . Also, by use of the O-H combination bands near 1.45 and 1.18  $\mu$  with the ordinary method, H<sub>2</sub>O in D<sub>2</sub>O over all concn. ranges can be detd. with standard deviations  $\leq \pm 0.7\%$ . CA

9213 c, 1962, v. 57

Radioscopic determination of soil moisture with the M-30 radiometer. I. P. Grechin, V. A. Emel'yanov, and V. E. Zinkevich. *Izv. Timiryazevsk. Sel'skokhoz. Akad.* 1963(2), 222-5. Soil moisture values with the M-30 radiometer 9 (a Soviet make) and those obtained by the thermostatic-wt. method varied 0.02-1.15%. To insure that the inserted tubes are parallel for horizontal irradiation a hole must be dug beforehand, esp. in fine textured soils. With correct placing of the hole and tube, moisture condensed at the tube-soil boundary does not show up in the amt. of moisture accumulated between the tubes and consequently does not affect the accuracy of the detn. To increase the accuracy, structural changes are recommended to seal off the counter and its circuits from condensed moisture.

Martin D. Derderian

24 f, 1964, v. 60

Effect of mineralogical composition and soil density on determination of moisture by neutron deceleration. V. I. Osipov. *Vopr. Inzh. Geol. i Gruntoved., Mosk. Gos. Univ., Sb. Statei* 1963, 223-40. Detn. of soil moisture by neutron deceleration has several advantages: structure of soil is not disturbed by taking the sample, results do not depend on phys. state of water (ice, water, vapor), and reflect av. conditions of a certain part of the ground and not just the local point. The neutron moisture meter consists of a probe contg. neutron source and slow neutron counter and connected to a recording device. Deceleration of low-energy neutrons by H<sub>2</sub>O of crystn. and chem. bound water of clay minerals is considerably smaller than by free water. Therefore the effect of H in minerals is considerably smaller than expected. The presence in soil of anomalous neutron absorbing sub-

24 g, 1964, v. 60

Determination of small amounts of water in acetaldehyde by infrared spectroscopy. A. N. Aleksandrov, N. K. Mikhailyan, and G. A. Sedova. *Khim. Prom.* 1962, 570-2. The absorbance band of H<sub>2</sub>O at 5241 cm.<sup>-1</sup> was selected for the detn.; Beer's law holds. The accuracy of the method, as indicated by data obtained by the Karl Fischer method, is fair, and the difference in the results for Me<sub>2</sub>CO contg. 0.4-1.4% H<sub>2</sub>O does not exceed 9.2%, and in the most cases is between 0.0 and 5%. Glass app. may be used.

A. Aladjem

13136 f, 1963, v. 58

7089 MEASUREMENT OF MOISTURE IN A MOVING COAL FEED. W.R. Ladner and A.E. Stacey.

*Brit. J. appl. Phys.*, Vol. 13, No. 3, 136 (March, 1962).

An accuracy of  $\pm 1\%$  over the range 0-14% total moisture has been obtained by the regulated feeding of coal through the sampling coil of a nuclear magnetic resonance spectrometer [for details, see *Fuel* (GB), 40, 295 (1961)] and recording the spectrum as a function of magnetic field strength; the peak-to-peak heights of the graph are plotted against the percentage total moisture. 5 references.

H.H. Hodgson

PA 1962

The Dolsch method for determining moisture. B. RASSOW AND A. RECKELER. *Angew. Chem.* 45, 266-7(1932).—A mixt. of alc. and petroleum which is homogeneous at room temp. forms 2 layers of liquid when cooled to a certain temp. The temp. at which the formation of 2 layers takes place is different if water is present and by detg. this temp. it is possible to det. the quantity of water present in any mixt. of alc., water and petroleum. This is the method which D. has used for detg. moisture in fuel. An app. is described which is suitable for carrying out the method and the results obtained are shown to be somewhat higher than those obtained by oven drying or by the xylene method. By treatment of a material insol. in alc., such as cotton, coal, cellulose, etc., the water is gradually taken up by the alc. and then, after mixing with petroleum, the temp. of unmixing is detd.

W. T. H.

2939, 1932

Errors in Determination of Moisture in Coal. W. F. HILLEBRAND AND W. L. BAUGER. *Discussions 8th Intern. Congr. Appl. Chem.*, 27, 77; cf. *C. A.*, 6, 2997.—Investigations emphasize the extreme hygroscopicity of dried coal. Dried coal recombines chemically with H<sub>2</sub>O and produces heat. Sp. ht. measurements on dried and undried coals show that the sp. ht. of the latter is considerably less than it would be theoretically on the basis of the dried coal value and of the value 1.0 for H<sub>2</sub>O. H<sub>2</sub>O that is driven out at ordinary temp. *in vacuo* is present partly in combined form, not as free H<sub>2</sub>O. Drying coal in CO<sub>2</sub> cannot be used, as coal absorbs CO<sub>2</sub>. N is well adapted. Wide variations as to oxidation of diff. types of coal should be emphasized. The amt. of CH<sub>4</sub> expelled from any coal is not sufficient to be of importance in connection with moisture detns.

C. A. COLE.

2294, 1913

Determination of small quantities of water by the method of Crismer. Lucia de Brouckere and Albert Gillet. *Bull. soc. chim. Belg.* 44, 473-503(1935).—Instead of attempting to det. H<sub>2</sub>O directly, it is sometimes better to take advantage of the change in the phys. properties caused by the presence of a little H<sub>2</sub>O. Thus Crismer (*Bull. soc. chim. Belg.* 18, 4(1904)) detd. H<sub>2</sub>O accurately by noting the temp. at which a solvent became turbid. By means of methanol-C<sub>6</sub>H<sub>6</sub> or methanol-C<sub>6</sub>H<sub>6</sub> it is possible to det. H<sub>2</sub>O accurately by detg. the temp. at which congelation takes place. The method is described.

W. T. H.

699<sup>8</sup>, 1936

Calorimetric determination of unfrozen water in montmorillonite pastes. Jack H. Kolaian and Philip F. Low (Purdue Univ., Lafayette, Indiana). *Soil Sci.* 95(6), 376-84(1933). A calorimetric method was used to det. the unfrozen water in pastes of Li-, Na-, and K-montmorillonite at -5 and -15°. As much as 55% of the water remained unfrozen at -5° and as much as 45% at -15°. The unfrozen-water content was greatest in the Na montmorillonite and least in the K montmorillonite. Reasons, based on thermodynamic equations, are given for the lack of freezing. Forces at the particle surfaces were a significant factor.

RCTT

479g, U. 61

Determination of moisture in brown coals. M. S. Burns and D. J. Swaine. C.S.I.R.O., Div. Coal Res., Chatswood, Australia. *Fuel* 42 5, 505-7(1933). *Bull. C.I.* 59, 2545. has criticized the application of B.S. 1016 to Australian brown coals, stating that construction of the oven might not give uniform temps., esp. just under the outlet, that the drying time should be  $\geq 3$  hrs., and that the flushing rate should be definitely stated in ml./min. Expts. reported in this note and general experience in the Division of Coal Research confirm that moisture detns. with the min.-free-space oven are independent of the oven and of the position of the sample in it. The British Standard method is suitable for brown coals provided the samples are heated in silica dishes for 3 hrs. with a N stream sufficient to change the atm. in the oven 30 times hr.

Roger W. Ryan

6664a, U. 60

Determining bound water in peat. F. D. Zhitetskaya. *Novye Fiz. Metody Issled. Torfa* (Moscow-Leningrad: Gos. Energ. Izd.) Sb. 1960, 138-48. A dilatometric method and a method of neg. adsorption of sugar were used to det. the amt. of bound H<sub>2</sub>O (I) in peat. In the dilatometric method, the amt. of H<sub>2</sub>O not freezing at 0° is taken as a measure of I. The amt. of I is found by the difference in the total moisture and the amt. of frozen H<sub>2</sub>O; the latter is detd. by measuring the change in vol. during freezing and thawing in a dilatometer. PhMe is used to fill the app.; a mixt. of ice and NaCl or EtOH and solid CO<sub>2</sub> is used for cooling. The neg. sugar-adsorption method is based on the fact that I is a poor solvent. A sucrose soln. with an accurately known concn. (>12.5%) is added to a weighed sample of peat of known moisture content, the mixt. is filtered, and the equil. concn. of sucrose in the filtrate is detd. The amt. of I is found from the change in concn.; I is obtained by subtracting free H<sub>2</sub>O from total moisture. Both methods give analogous results. From *Ref. Zh., Khim.* 1963, Abstr. No. 10C262.

MVRK

66672, U.60

Application and use of the nuclear method for moisture analysis of coke. Jack G. Crump (Nucl.-Chicago Corp., Des Plaines, Ill.). *Blast Furnace Steel Plant* 52, 325-7(1964). Bulk d. is detd. by the transmission of  $\gamma$  radiation through a fixed cross section of the coke and relating the magnitude of the radiation received at the detector to the bulk d. With an increase of bulk d., the radiation absorbed or scattered will increase and reduce the amt. received at the detector. <sup>137</sup>Cs provides the source of highly penetrating electromagnetic radiation used in this measurement. An ionization chamber is used to convert the radiation received at the detector into a proportional elec. current. This current is amplified, calibrated in lb. coke/cu. ft. and displayed in a readout meter. Moisture d. or lb. H<sub>2</sub>O/cu. ft. is analyzed by the use of the fast-neutron radiation which loses energy at a rate substantially greater by interaction with H than with any other element. It is this relation and the known proportion of H in H<sub>2</sub>O that permits calibration of the moisture channel. H d. is measured by using a radiochem. source of fast neutrons and a slow neutron detector. Fast neutrons lose energy primarily by collision with nuclei. Max. energy is lost when the mass of the neutron is equal to the mass of the nucleus. The H nucleus has a single proton, consequently by interaction with H, the energy of the fast neutron is reduced at a far greater rate than with any other element. The no. of slow neutrons created in this process in a fixed vol. is detd. by the H d. The slow neutrons are detected in an ionization chamber. The current generated in the chamber is amplified and displayed on a readout meter in lb. of H or H<sub>2</sub>O/cu. ft. Details of the computation system are given.

H. Stoertz

490 B, U.61

Automatic measurement of moisture in sand and ceramics. II. Determination of the moisture content of sand by absorption in the microwave band. E. M. Amrhein (Wuerzburg, Ger.). *Ber. Deut. Keram. Ges.* 37(12), 520-3(1960); cf. CA 55, 4913h. The measurement is based on the max. absorption band of H<sub>2</sub>O at  $30 \times 10^9$  cycles/sec. The plane polarized radiation (from a klystron generator), which is transmitted through a standard bed of sand, is measured. A calibration curve relating moisture content with extinction is linear over the range 0 to 3% of H<sub>2</sub>O. If care is taken with the mech. stability and constancy of the valve characteristics and supply voltage, an accuracy of  $\pm 0.2\%$  by wt. is possible. The app. (some details of which are recorded) was adapted to give a continuous reading for sand being conveyed around a circular track of Plexiglas (used on account of its isotropic nature). A change of 0.25% of H<sub>2</sub>O content was clearly recorded on a chart. From *Anal. Abstr.* 8(10), Abstr. No. 4196(1961).

TCBT

38510, U.60

Testing equipment for determination of H and H<sub>2</sub>O contents by neutron irradiation independently of the layer thickness of the material. Frieske & Hoepfner G.m.b.H. (by Ruediger Tornau). Ger. 1,155,924 (Cl. 42/), Oct. 17, 1963, Appl. May 5, 1961; 3 pp. Improved thickness independence of the testing method regarding materials on conveyor belts is achieved by Fe reflectors for the  $\pi$ . The  $\pi$  source and the Fe reflector for diffuse reflection are located on opposite sides of the conveyor belt. The BF<sub>3</sub>-contg. impulse counters are immediately behind the Ra-Be-contg.  $\pi$  source. W. Glaser

3696 b, U.60

Beta-ray-gauging technique for measuring leaf water content changes and moisture status of plants. F. S. Nakayama and W. L. Ehrlar (U.S. Dept. Agr., Tempe, Ariz.). *Plant Physiol.* 39(1), 95-8(1964). Equipment is described for measuring absorption of  $\beta$ -rays by leaf tissue. As sources of the rays, <sup>54</sup>Co, <sup>106</sup>Tc, and <sup>204</sup>Tl would be suitable for specific thicknesses, but for cotton leaves <sup>107</sup>Pm was used in the present study. J. J. Willmar

13563c, U.60

Dielectric measurements in porous media applied to the determination of water content in shale structures. P. Simandouze (Inst. Franc. Petrole, Rueil-Malmaison, France). *Rev. Inst. Franc. Petrole Ann. Combust. Liquides* 18, 193-215(1963). Detn. of the dielec. const. of a porous rock by using a frequency of about 10<sup>8</sup> cycles/sec. allows a nondestructive detn. of H<sub>2</sub>O content to  $\sim \pm 3\%$ . The app. and its theoretical background are described in detail, and the method is generally useful for the detn. of impedences >1-2 megohms. 15 references. Paul D. Hobson

13046 f, U.60

Adsorption of water, benzene, and isopropyl alcohol vapors on aluminum oxide. G. V. Tsitsishvili and Sh. I. Sidamonidze (Chem. Inst., Acad. Sci. Georgian S.S.R., Tbilisi). *Soobshch. Akad. Nauk Gruz. SSR* 32(2), 335-42(1963). Four samples of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with different pore structure were studied by using a quartz spiral sorption microbalance at 30°. The low-temp. adsorption of N was measured volumetrically. The surface areas of the samples detd. by the B.E.T. method from the C<sub>2</sub>H<sub>6</sub> and N isotherms differ. The main cause of the disagreement of the surface areas is ultraporosity of the samples. The surface of micropores is inaccessible for the C<sub>2</sub>H<sub>6</sub> mol. H<sub>2</sub>O, C<sub>2</sub>H<sub>6</sub>, and iso-PrOH isotherms are described in detail. J. Jindra

12686 d, U.60

Determination of capillary breakage water content. Chin-Chung Yang. *T'u Jang T'ung Pao* 1963(6), 57. The H<sub>2</sub>O in the soil capillaries of effective diam. >3  $\mu$  can be directly absorbed by plant root and transmitted to the evaporation surface of the plant, while the water in the soil capillaries of effective diam. <3  $\mu$  cannot be directly transferred to plant roots without vaporization and recondensation in larger capillaries. Therefore, there is a breakage point on the characteristics of soil moisture. The suggested method of detn. of capillary breakage H<sub>2</sub>O content is: place the soil sample in glass tubing of 5 cm. diam. and 40 cm. long, wrap the lower end of the tubing with Cu wire gauze, and immerse in a H<sub>2</sub>O container. Capillary H<sub>2</sub>O rises in the soil column as indicated by the darkening of the color. As soon as it reaches 20 cm., remove the H<sub>2</sub>O container and mark the capillary H<sub>2</sub>O height on the glass tubing. Two to 4 hrs. later take a soil sample at 0.5 cm. above the height mark. Det. the moisture content by drying or other methods and report as the capillary breakage H<sub>2</sub>O content. Results obtained with this method differed  $\leq \pm 0.5\%$  from those with the soil evaporation rate method. K. T. Lee

15085 e, U.60

Industrial application of moisture control by neutrons. V. Netz. *Messen, Steuern, Regeln* 6(10), 427-9(1963). A thermal neutron source is used for moisture detn. in bulk material; the quantity measured is the no. of slow neutrons produced in passing through a moist layer. Herbert Puschmann

12647e, v.60

Neutrons measure moisture in solids. R. L. Carver. *Instr. Control Systems* 36(5), 106-7(1963). Nuclear gaging can provide continuous measurement and control of moisture content of process solids. High energy neutrons are directed into the material. Some of these are reflected backward, losing energy. The reflected neutrons are measured and, with calibration, provide a moisture measurement. They actually reflect the presence of H, an efficient neutron moderator. From *J. Am. Ceram. Soc.* 46(9), 251(1963). TCJC

6200d, v.60

Infrared determination of the water in poly(ethylene terephthalate) films. G. Langbein and W. Seufert (Deut. Kunststoff-Inst., Darmstadt, Ger.). *Kolloid-Z.* 193(1), 37-9(1963). The absorption  $K$  of a film is detd. at wavelengths of 3630, 4080, and 7350  $\text{cm}^{-1}$ , and the quotient  $K'_{3630}/K'_{4080}$  is calcd. from the equation:  $K'_{3630}/K'_{4080} = (K_{3630} - 0.19 K_{4080} - 0.14 K_{7350}) / (K_{4080} - 0.31 K_{7350})$ . This quotient is compared with a standard linear curve of quotients vs. ratios of  $\text{H}_2\text{O}$  concns. to poly(ethylene terephthalate) concns. to det. the fraction of  $\text{H}_2\text{O}$  present.

Thomas A. Wilson

6985e, v.60

Moisture content determinations for wood with highly volatile constituents. Helmut Resch and Barton A. Ecklund (Univ. of California, Richmond). *Forest Prod. J.* 13, 401-2(1963). The moisture content of conditioned *Chamaecyparis lawsoniana* and *C. nootkatensis* was detd. by 3 methods: (A) by oven drying, (B) by the elec. moisture meter, and (C) by the Karl Fischer method. Method A is subject to serious error because of nonaq., volatile constituents. Method B is also subject to errors (relative to method C). However, suitable correction factors can be developed which permit the use of the simple method B.

H. Tarkow

7024g, v.60

Rapid determination of water in solid organic intermediates. H. Schwarz and Lore Kirstein (VEB, Berlin). *Z. Chem.* 4(3), 103-4(1964). Drying in an oven to const. wt. requires 6-24 hrs. and in the meantime the substance absorbs moisture. The described detn. is based on the change of  $n$  of a soln. of the material in a hydrophilic solvent, as a linear function of the  $\text{H}_2\text{O}$  content. A linear calibration curve is made by dissolving a substance of known  $\text{H}_2\text{O}$  content in dioxane (100%  $\text{H}_2\text{O}$  = 5 g.  $\text{H}_2\text{O}$  in 40 ml. dioxane,  $n = 1.4141$ ). Repeatability is  $\pm 0.2\%$   $\text{H}_2\text{O}$ . The method was used to det.  $\text{H}_2\text{O}$  in  $\alpha$ -bromoisovaleryl-urea, 4-nitrosothymol, 2,5 diiodochelidamic acid, and chelidonic acid. Amelie L. Grauer

45d, v.61

Simultaneous determination of monomer and water content in polycaprolactam. V. S. Klimova, N. D. Katorzhnov, G. I. Kudryavtsev, and A. V. Beschastnov. *Khim. Volokna* 1963(6), 64-5. An anal. app. and technique, based on the method of Schenker, et al. (*Anal. Chem.* 29(5), 825 (1957)), is developed for detg. monomer and H<sub>2</sub>O contents by evapn. in vacuo. A 10-15 g. sample is placed into a glass vessel heated to 200°, and monomer and H<sub>2</sub>O in polycaprolactam (nylon 6) are driven out 2 hrs. at 5-10 mm. through a connecting glass tube heated to 225° into a condenser, placed into a Dewar vessel filled with ice or cold water. The difference in sample wt. before and after the detn. gives the monomer plus H<sub>2</sub>O content; the condensate is then dild. with 5 cc. distd. H<sub>2</sub>O, and the monomer content detd. from the refractive index at 25° and calcd. from a calibration curve. Results obtained on polymer fibers, crumbs, and shavings are reproducible and more exact for the monomer content of polymer crumb than values detd. by C<sub>6</sub>H<sub>6</sub> extn., which gives low results for such specimens and also for film. K. Stein

8191a, v. 60

Measuring the moisture content of materials by nuclear magnetic resonance (N.M.R.). M. G. Kozlov and L. A. Panteleeva. *Tr. Nauchn.-Issled. Inst. Teploenerg. Priborostr.*, Sb. 1962(2), 59-71. An N.M.R. app. was constructed on which exptl. detns. of moisture content of various materials were run. The method, block diagram, line diagram, and design characteristics of some of the units of the app. are described. The sensitivity of the app. is  $\pm 0.2$  mg. H<sub>2</sub>O in a vol. of 160 cc.; accuracy is  $\pm 1-1.5\%$ . An industrial-scale app. for continuous moisture control can be constructed on the basis of this exptl. app. From *Ref. Zh., Khim.* 1963, Abstr. 101149. MYRK

7439c, v. 60

Negative ion gas analysis technique. V. N. Smith and E. J. Merritt (Shell Develop. Co., Emeryville, Calif.). *Anal. Chem.* 34, 1476-82(1962). A three-electrode gas ionization chamber detected O in N, butane, and butadiene, and H<sub>2</sub>O in N. Auxiliary equipment included an electrometer and a Sr<sup>90</sup> source within the chamber to ionize the gas. An a.-c. field applied to the outer parallel plates sep'd. electrons and neg. ions, primarily because of their relative mobilities; d.-c. potential applied to the grid modified the cell characteristics or canceled background current. Trace amts. of electrophores in gas streams whose primary constituents remain unionized may be measured quant. With a 30-40 sec. time const. in the electrometer,  $\frac{1}{2}$  p.p.m. O in N was calcd. to be measurable. The sensitivity to H<sub>2</sub>O vapor was about  $\frac{1}{2}$  that of O. As a gas-chromatography detector, 1 part CCl<sub>4</sub> in 10<sup>10</sup> parts heptane in a 1- $\mu$ l. sample gave a large peak with N as carrier gas. Hubert L. Youmans

15778h, 1962

Dielectric measurements as a rapid means of water determination. E. BERLINER AND R. RÜTER. *Kolloid-Z.* 47, 251-7(1929).—An app. and a method for detg. the dielec. const. are described, by means of which binary mixts. of nonelectrolytes, viz.: aq. solns. of Me, Et and isopropyl ales., glycol, glycerol, dextrose, sucrose, carbamide, acetone and solns. of C<sub>4</sub>H<sub>8</sub> in EtOH and of C<sub>4</sub>H<sub>8</sub>NO<sub>2</sub> in C<sub>4</sub>H<sub>8</sub> were studied. The results indicate that the dielec. const. changes with differences in the compn. of the system. It can be used, therefore, to det. the H<sub>2</sub>O content of an aq. mixt. J. WIERTELAK

7, 1930



18421 PROPORTIONALITY, UNDER CERTAIN CONDITIONS,  
BETWEEN THE ELECTRICAL CONDUCTIVITY OF  
INSULATING LIQUIDS AND THE QUANTITY OF WATER  
CONTAINED IN THEM. R. Guizonnier and F. Taxis.  
C.R. Acad. Sci. (France), Vol. 253, No. 6, 943-4 (Aug. 7, 1961).  
In French.

After taking special precautions to eliminate electrode effects  
and static charges, a linear relationship was found between the  
electrical conductivity of, and the proportion of water contained in,  
petroleum ether, carbon tetrachloride and benzene.

R.G.C. Arridge

18421, PA, 1961

Device for the measurement of moisture in green tea leaf.  
N. D. Nizharadze. *Tr. Tbilissk. Nauchn-Issled. Inst. Priboro-  
str. i Sredstv. Avtomatiz.* 3, 91-9 (1962). A moisture meter for  
measuring of the moisture of green tea leaf, operating in pulses,  
is described. The moisture meter consists of 2 VCh-generators  
operating on lamps 6N9S according to a 3-point scheme with a  
cathode union. The basic detail of the moisture meter is the  
condenser-pickup unit. An Al cylinder serves as an external  
electrode. The internal cylindrical electrode has a headband of  
a dielec. and is secured to a bracket insulator. The tea leaf is  
charged through a hopper-like funnel of the condenser and is  
removed through the bottom. The instrument permits measure-  
ment of moisture of the leaf with an error of  $\pm 1\%$  moisture.  
From *Ref. Zh., Khim.* 1963, Abstr. No. 16R157. MJRK

16213 h, U. 60

Quantitative analysis of water content in liquid dielectrics,  
such as petroleum. Ya. M. Frankfurt. U.S.S.R. 158,444,  
Oct. 19, 1963, Appl. Aug. 27, 1962. For sep. detn. of small  
amts. of H<sub>2</sub>O in emulsions, the breakdown voltage value of the  
liquid under examn. is measured, and emulsified H<sub>2</sub>O content is  
judged according to a previously plotted curve of the relation of  
the breakdown voltage value to the H<sub>2</sub>O content in petroleum.  
From *Byul. Izobret. i Tovarnykh Znakov* 1963(21), 62. MDCL

86489, U. 60

Determining content of water, petroleum, petroleum products,  
and their emulsions. V. D. Mel'nik and L. G. Ovchinnikov.  
U.S.S.R. 159,692, Dec. 28, 1963, Appl. Sept. 17, 1962. The  
detn. is carried out by measuring the dielec. const. To increase  
the accuracy of measurement, the irrigated specimen is emulsi-  
fied before measurement. From *Byul. Izobret. i Tovarnykh  
Znakov* 1964(1), 55. MDCL

15660 h, U. 60

Detection of traces of H<sub>2</sub>O in D<sub>2</sub>O by infrared absorption.  
Friedrich Glaeser. *Chem.-Ingr.-Tech.* 33, 443-5 (1961). Detn.  
of the infrared absorption, esp. at about 1.47  $\mu$ , is suitable for the  
detection of traces of H<sub>2</sub>O in D<sub>2</sub>O. A measuring method for a sin-  
gle detn. using a spectrophotometer and a continuous measuring  
procedure on the same app., modified according to Hoffmann (CA  
52, 804g), were described. Since glass was suitable for optical  
parts, a robust and exact device was produced by simple means.  
From *CZ* 1963(35), 15162. MFCR

151429, U. 60

Determination of moisture in sulfuric acid lignin. Z. G. Libman and Z. V. Korneva. *Gidrolizn. i Lesokhim. Prom.* 16(8), 19(1963). Moisture in hydrolysis lignin is detd. by the measurement of  $n$  of glycerol before and after it had absorbed water from lignin. Glycerol and lignin are mixed in the ratio 5:1 and the mixt. is centrifuged. The amt. of  $H_2O$  in lignin is calcd. according to the equation  $W = K [W_m + (W_0 - W_0)a/b]$ , where  $W_m$  is the  $H_2O$  content in the mixt.,  $W_0$  is the initial  $H_2O$  content in glycerol,  $a$  is the wt. of glycerol,  $b$  is the wt. of lignin in g., and  $K$  is an empirical coeff. equal to the ratio of moisture content of lignin as detd. by drying to the moisture content detd. refractometrically. In the expts. reported  $K$  was 1.058.  
T. Jurecic

9470 f, v. 60

Nuclear magnetic resonance study of water in clay. T. H. Wu (Michigan State Univ., East Lansing). *J. Geophys. Res.* 69(6), 1083-91(1964). The bonding between clay minerals and  $H_2O$  was studied exptl. by means of nuclear magnetic resonance.  $H_2O$  and  $D_2O$  were adsorbed on kaolinite, grundite, and montmorillonite, and the spin-spin relaxation and the spin-lattice relaxation times were detd. The results were used to analyze several aspects of adsorbed  $H_2O$  behavior, such as exchange with  $D_2O$ , effect of heating, and effect of adsorbed ions. At temps.  $< 0^\circ$  the evidence indicates that the  $H_2O$  close to the clay has a structure different from that of ice. The measured relaxation times were compared with the viscosity of adsorbed  $H_2O$  obtained from permeability tests and with the amts. of unfrozen  $H_2O$  detd. by calorimetric measurements.  
RCJW

9053 f, v. 60

Headpiece for the dehydration of organic substances and the determination of moisture. B. V. Volkov. *Zavodsk. Lab.* 30 (1), 112 (1964). A glass head was designed for dehydrating org. substances with limited soly. in  $H_2O$ , such as iso-BuOH, BuOH, cyclopentanone, and furfural, or for their removal as  $C_6H_6$  or toluene azeotropes during synthesis. A socket for a thermometer is fitted directly above the standard joint for the column top, and a side arm takes the vapors of the azeotropic mixt. to a vertical condenser parallel to the thermometer. The condensed azeotropic mixt. drips into a  $H_2O$ -jacketed coil, where it is cooled further, to decrease the soly. of  $H_2O$  in org. substance and vice versa. The cooled mixt. then enters a sepg. chamber, where the aq. layer is withdrawn through a needle valve and the org. layer is returned to the column via a narrow tube below the side arm. Details of the needle valve assembly are given. The rate of aq. layer take-off is adjusted by turning the needle, and in the closed position this layer is taken at the rate of 2-3 drops/min. The headpiece can be utilized for Dean and Stark moisture detns. by calibrating the sepg. chamber.

Andrew L. Gruchowski

8603 r, v. 60

Determination of the water content of coffee; control method and practical methods. A. Guilbot. *Cafe, Cacao, The* 7, 192-200(1963).  $H_2O$  content of coffee is defined as the amt. of  $H_2O$  lost by a product when it is placed in equil. with a vapor pressure of almost zero, under conditions that avoid interfering results. This is done by drying to const. wt. in the presence of  $P_2O_5$  at  $45-50^\circ$  and 10-20 mm. Comparable results may be obtained by heating at  $105^\circ$  for 10-15 hrs., if the vapor pressure is controlled. Heating at  $130^\circ$  can give results within 0.3% of the control. An app. is available that measures the  $H_2O$  content in 1-2 min. by detg. the dielec. const. at high frequency.

Amy LeVesconte

11292 r, v. 60

Moisture meters for measuring the water-balance of human skin. (A critical discussion of the improved salt crystal and electrolytic moisture meters.). F. A. J. Thiele and K. Schutter (Unilever Res. Lab., Vlaardingen, Neth.). *Proc. Sci. Sect. Toilet Goods Assoc.* 40, 20-4(1963); cf. *CA* 58, 8223e. The requirements of a moisture meter for *in vivo* measurements on human skin was discussed. The construction and operation of an electrolytic moisture analyzer and a salt crystal meter were described. Some of the advantages and disadvantages of each instrument were given.  
Henry J. Wing

531d, v. 61

Physicochemical studies on alkali metal soap-water system. IV. Thermal change of hydration water in sodium soaps by measurement of dielectric constant. Shun Noguchi and Teruo Nishina (Marumiya Co., Ltd., Tokyo). *Nippon Kagaku Kaishi* 35, 1086-9(1961); cf. *CA* 59, 842f. The thermal change of water of hydration in Na soaps of C<sub>4</sub>-C<sub>18</sub> was studied in order to investigate the states of combined water accurately. The anhyd. soap showed a slight increase in dielec. const. with rise in temp., while the hydrated soaps showed 2 peaks in dielec. const., at 20-60° and above 70°. The first peak observed seemed to be due to free water or loosely bound water, and the second peak to be due to the transition of bound water to free water. Very sharp increases in dielec. const. at the 2nd peak were taken to indicate rigidly bound water for stearate (92°), palmitate (83°), caprylate (75°), caproate (76°), and elaidate (85°). Unsharp increases for myristate (84°), laurate (68°), and caprate (82°) were taken to indicate loosely bound water. Na oleate was not assumed to contain bound water, because hydrated soap showed only 1 peak between 20 and 100°. V. Moisture absorbed by anhydrous sodium soap as studied by measurement of the dielectric constant. *Ibid.* 1177-80. The absorption of moisture by powd. anhyd. Na soaps placed in a 100% relative humidity atm. at 20° for 2 hrs. was investigated. The moisture contents in Na stearate, palmitate, myristate, laurate, caprate, caprylate, caproate, and elaidate were 1.33,

1.51, 2.86, 2.24, 2.97, 2.40, 4.25, and 1.41%, resp. As the no. of C atoms in the soap increased the moisture content decreased with the exception of myristate. The dielec. consts. began to increase for caproate, caprylate, caprate, and myristate, after an hr., but those for laurate, palmitate, and elaidate did not show such tendencies. Moisture absorbed by the soaps existed as the bound water for caproate, caprylate, and caprate, and as free water for myristate, palmitate, stearate, and elaidate. VI. Effect of temperature on the formation of bound water in soaps. *Ibid.* 1180-3. Anhyd. Na, K, Li, and Rb soaps with C<sub>4</sub>-C<sub>18</sub> were mixed with equal amts. of water, and the resultant mixts. were heated at 20-5, 40-5, 55-60, and 80° for 5 hrs. The appearance of bound water in the soaps was estd. by measuring the dielec. const. The dielec. const. at 72-90° for Na stearate increased markedly by treatment at 80°, and weakly by treatment at 60°, so, most of the soap mols. were assumed to have bound water when treated at 80°. As the chain length of the Na soap became shorter, the temp. necessary for the formation of bound water became lower. The treatment at room temp. was sufficient for Na soaps having <10 C atoms to form bound water, but treatments at 55-60 and 80° were necessary for Na elaidate and Na soaps having >12 C atoms. Li, K, and Rb soaps did not form rigidly bound water when heated them even at 100°.

Hiroshi Suzuki

13909g, h, v. 60

Electrometric determination of water. B. Grinenko. *Novosti Tekh.* 1938, No. 6, 43-4.—Moisture is detd. by measuring, with a. s. and a galvanometer, the elec. cond. of an electrolyte dissolved in the sample. In the detn. of the moisture in air or in gases, the mixt. is passed through an absorber, where the water vapors are absorbed by a weak soln. of an electrolyte in an org. liquid. The increase of the elec. cond. gives the amt. of moisture.

A. A. Bochtlingk

4907<sup>2</sup>, 1938

Coulometric determination of water in liquid hydrocarbons. J. Soucek, M. Pribyl, and K. Novak (Vyzkumny Ustav Makromol. Chem., Brno). *Collection Czech. Chem. Commun.* 27, 400-5(1962)(in German). A method was elaborated for discontinuous detns. of small amts. of H<sub>2</sub>O in liquid hydrocarbons and their mixts.; the total amt. of electricity needed for electrolysis of the H<sub>2</sub>O from the liquid sample is measured. Small samples (0.3-0.8 ml.) contg. up to 1 × 10<sup>-4</sup>% H<sub>2</sub>O can be analyzed rapidly.

E. Erdos

1543 e, 1962, V. 57

Near infrared spectrophotometric determination of moisture in cyclic ethers. Takeshi Morikawa (Ind. Inst., Osaka, Japan). *Bunseki Kagaku* 13(3), 255-7(1964). Moisture in tetrahydrofuran or dioxane, useful as an indication of the degree of autoxidn., is detd. by dielec. const. in the higher ranges, while for lower moisture content (<30 g. H<sub>2</sub>O/l.) the Karl Fischer method can not be used because a ppt. forms. It is possible to det. moisture quickly with adequate sensitivity by near infrared spectrophotometry using quartz cells with a large const. light path length.

Yoichiro Kawakami

1278 h, 1964, V. 61

Determination of moisture content of hardened concrete by its dielectric properties. J. R. Bell, G. A. Leonards, and W. L. Dolch (Oregon State Univ., Corvallis). *Am. Soc. Testing Mater., Proc.* 63, 996-1007(1963). Capacitances and dissipation factors for 3-in.-diam. thin disks of concrete and mortar were measured in the lab. at various water contents by using conventional bridge techniques at 10<sup>4</sup> cycles/sec. From these data relative dielec. consts. and dielec. conds. were calcd. Capacitances were detd. also at 60 and 6 × 10<sup>4</sup> cycles/sec. Conds. at various water contents were detd. at 10 cycles/sec. by using small prisms of concrete. The exptl. variables involved and the usefulness of the dielec. properties as moisture indicators are discussed. Except for unusually severe drying of the concrete or salt content of the pore water, these properties indicated moisture contents below about 6% with an accuracy of ±0.25%, or for the low-frequency cond. measurements, ±0.5%.

W. J. McCoy

1594 e, 1964, V. 61

Determining coffee bean moisture content by the neutron scattering method. Jaw Kai Wang (Univ. of Hawaii, Honolulu). *Trans. ASAE (Am. Soc. Agr. Engrs.)* 7(1), 42-3, 45(1964). Batches of green coffee (300 lb.) were steam conditioned to incremental moisture contents from 8 to 22%. A P-19 probe with output of 7.99 × 10<sup>4</sup> neutrons per sec. was inserted into the center of the batch. Moisture was calcd. using a Nuclear Chicago Model 2800 neutron counter. Standard deviation at 2 min. was 0.01; 5 min. 0.005; 10 min. essentially zero. Bulk moisture detns. by this procedure eliminates sampling errors.

Samuel Lee

2404 f, 1964, V. 61

Determination of the water content of some alcohols. N. Z. Kotelkov and K. P. Kotelkova (Agr. Inst., Saratov). *Zhur. Anal. Khim.* 7, 78-83(1952).—Tables of  $n_D$  for alc.-H<sub>2</sub>O were prepd. for EtOH and iso-PrOH at 20, 23, and 25°, and for PrOH at 15 and 20°.

M. Hoseh

1539 f, 1953, V. 47

Determination of the water content of some alcohols. N. Z. Kotelkov and K. P. Kotelkova (Saratov Agr. Inst.). *J. Anal. Chem. (U.S.S.R.)* 7, 89-95(1952)(Engl. translation).—See C.A. 47, 1539g.

H. L. H.

4369 h, 1954

## Ebulliometer

### Principles of an ebullioscopic method of moisture determination in substances. S.

BAKOWSKI. *Roczniki Chem.* 11, 49-64(64 in French)(1931).—After a review of all known methods of moisture detn. a new method is proposed based on the difference between the b. p. of a pure liquid and of a water mixt. with the liquid. The liquid, which must be perfectly miscible with water, is used for extn. of the moist material. The b. p. of both the pure and the moist liquid is detd. at the same time by means of a special app. consisting of a barometric and an extn. ebullioscope. A Beckmann thermometer calibrated to 0.01° is used for temp. measurements. The liquids used are Me<sub>2</sub>CO and MeOH. J. WIERTELAK

3928, 1931

Application of methanol to the ebullioscopic method of determination of water content. STANISLAW BAKOWSKI. *Roczniki Chem.* 11, 490-504 (504 in French)(1931); cf. preceding abstr.—Detn. of water by this method gives results concordant with the drying or the xylene method in all cases, if it is present as admixed, adsorbed, imbibed or crystd. with the substance. If the substance to be tested is sol. in MeOH, the results are too high. J. WIERTELAK

2672, 1932

Ebulliometric determination of small amounts of water. M. Wojciechowski. *Nature* 137, 707(1936).—The lowering of the condensation temp. of a binary azeotropic mixt. such as ethanol-benzene is proportional to the amt. of H<sub>2</sub>O added. It is possible to obtain a lowering of 0.033° per mg. H<sub>2</sub>O. An azeotropic mixt. of *n*-propanol-toluene greatly increases the sensitivity of the method. A lowering of 0.080° per mg. was obtained. W. George Parks

5531<sup>4</sup>, 1936

Application of acetone to the ebullioscopic method of determination of water content. STANISLAW BAKOWSKI. *Roczniki Chem.* 11, 269-88 (281-8 in French)(1931).—Water mechanically mixed with a substance is detd. by the ebullioscopic method with acetone with the same exactness as by drying or by the xylene-distn. method. The extn. of adsorbed H<sub>2</sub>O or that present in cell cavities is incomplete, and the moisture content detd. is too low. Too high results are obtained in case the substance under test is sol. in acetone, this raising its b. p. J. WIERTELAK

2672, 1932

The vapor pressure of water over sulfuric acid-water mixtures at 25°. I. R. McHAFFIE. *J. Chem. Soc.* 1927, 112 G. To test the accuracy of the method for measuring small amts. of H<sub>2</sub>O vapor present in "permanent" gases (cf. C. A. 20, 2007) the vapor pressure of H<sub>2</sub>O over H<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>O mixts. at 25° was measured for mixts. contg. from 66 to 84% H<sub>2</sub>SO<sub>4</sub>. The results are in good agreement among themselves, and with the exptl. values of Brønsted (cf. C. A. 4, 2390) and the calcd. values of Wilson (cf. C. A. 15, 1836). They do not agree with the data for 25° in the Landolt-Börnstein tables. The method is reliable for vapor pressures as low as 0.08 mm. R. K. W.

1388, 1927

A correct procedure for determining water. R. Leroy. *Chim. anal.* 36, 294-301(1934).—It is claimed that present methods for detg. moisture are antiquated and unreliable. The assumption that all moisture can be removed by keeping the sample for some time at a temp. above the b.p. of water is shown to be erroneous. It is pointed out how better methods can be devised by taking the vapor pressure of the sample into consideration. W. T. Hall

2248d, 1955

The analysis of polymers for moisture and volatiles. D. J. David, G. F. Baumann, and Samuel Steingiser (Mobay Chem. Co., New Martinsville, W. Va.). *SPE (Soc. Plastics Engrs.) Trans.* 2, 231-6(1962). A vapor-pressure method is described which quant. detcs. moisture and volatiles simultaneously in polycarbonates, polyesters, urethan elastomers, and other polymers. An accuracy of 0.002% is reported for a 10-g. sample, and as little as 0.001% H<sub>2</sub>O can be detected. R. M. Kyte

12689b, 1962, V. 57

An apparatus for the analysis of small amounts of vapor when present in permanent gases. I. R. McHAFFIE. *J. Am. Chem. Soc.* 48, 1143-5(1926).—There is described an app. for measuring the mass of very small amts. (0.5 mg.) of H<sub>2</sub>O in gas with an accuracy of 1 part in 1000. The H<sub>2</sub>O vapor is removed from a current of the gas in a previously evacuated U-tube of known vol. immersed in liquid air. The vol. of gas passed through the tube is measured. The U-tube is warmed to 40° and the gas pressure in the tube is measured. By measuring the pressure at as low a temp. as possible and at successively increasing temps. the pressure at 40° is cor. for the partial pressure of gases other than H<sub>2</sub>O vapor that may be present. (Cf. McHaffie and Lenher, C. A. 19, 3188.) As a test of the method the dissoc. pressure of Na<sub>2</sub>SO<sub>4</sub>·5H<sub>2</sub>O at 13.6, 15.4 and 25.0° was measured. The results were 8.29, 9.59 and 19.24 mm. Hg as compared with the Landolt-Börnstein values 8.29, 9.54 and 19.21 ± 0.02 mm. Hg, resp. R. L. DODGE

2097, 1926

Determination of moisture in a gas by partial water vapor pressure. S. A. Krivosheev. *Zavodskaya Lab.* 3, 368(1934).—The detn. is based on absorption of moisture in gas by H<sub>2</sub>SO<sub>4</sub> or P<sub>2</sub>O<sub>5</sub>, and the registration of the resulting partial vapor pressure on the manometer scale. The app. is shown. Chas. Blanc

1747<sup>6</sup>, 1935

Moisture Determinations without the Aid of Heat. P. F. TROWBRIDGE. U. S. Dep't Agr., Bur. Chem., *Bull.* 122, 219-21.—In determining moisture by means of a vacuum over H<sub>2</sub>SO<sub>4</sub>, the time of complete desiccation was shortened by frequent agitation of the acid to remove the water layer formed on the top. Frothing of semi-liquid substances was obviated by freezing the samples after weighing. To avoid grinding and a second extraction for the det. of the ether-sol. material in certain substances the samples were mixed with ignited sand. Good results were obtained on samples of meats, blood, feeds, butter, cheese, etc., desiccation being sometimes complete in 12 hrs. A. L. DAVISON.

2420, 1909

Moisture determination in manufactured gas. Oskar Knublauch. *Chem.-Ztg.* 68, 167(1911).—The usual methods for detg. moisture by means of hygroscopic materials do not give satisfactory results with manufd. gas. A new method is proposed in which a gas sample is taken in a dry 4-8-l. bottle provided with a tightly fitting stopper, stopcocks for inlet and outlet, an oil manometer, and a thermometer. The gas sample is allowed to come to room temp. and the pressure adjusted to atm. The bottle is then shaken to break a thin-walled ampoule contg. 2-3 cc. H<sub>2</sub>O, and the max. rise in pressure due to satn. with H<sub>2</sub>O is noted on the oil manometer. This reading is converted to mm. Hg and subtracted from the vapor pressure of H<sub>2</sub>O at the temp.. The resultant pressure is that of the H<sub>2</sub>O vapor in the gas sample; this figure divided by the vapor pressure of H<sub>2</sub>O at the given temp. gives the percentage satn. of the gas sample.  
R. W. Ryan

2642, 1947

The quantitative analysis of small quantities of gases. H. M. RYDER. *J. Am. Chem. Soc.* 40, 1656-62(1918).—This method involves the freezing out of CO<sub>2</sub> and H<sub>2</sub>O vapor by means of liquid air and solid CO<sub>2</sub>, addition of O and combustion of the dry gases remaining, and redetn. of CO<sub>2</sub> and H<sub>2</sub>O vapor. By it H<sub>2</sub>O, CO<sub>2</sub>, CO, O, H, N and CH<sub>4</sub> can be detd., but not O and CH<sub>4</sub> in presence of each other. When the total amt. of sample was less than 5 cu. mm., an av. analysis shows a max. error of 6% in the element present in the greatest quantity. Much greater accuracy is obtained with larger quantities.  
IRENE F. SMITH.

11, 1919

Indirect determination of the water content of an article being vacuum- or freeze-dried. Leybold-Hochvakuum-Anlagen G.m.b.H. (by Ulrich Hackenberg and Georg W. Oetjen). *Ger.* 1,128,365 (Cl. 82a), Apr. 19, 1962, Appl. Mar. 18, 1961; 2 pp. The pressure rise caused by the application of a heat impulse to a vacuum drier, temporarily isolated from the vacuum source, gave a measure of the H<sub>2</sub>O content in an article being dried. T. F. Canning

53092, U.57, 1962

Apparatus for microanalysis of gas. C. H. Prescott, Jr., and James Morrison. *Ind. Eng. Chem., Anal. Ed.* 11, 230-3(1939).—An app. for complete analysis of gas samples from 1 to 25 cu. mm. is described with a photograph and diagrams. Errors amount to 2% of the total vol. for each component in vols. between 5 and 25 cu. mm., and 5% between 1 and 5 cu. mm. Gas mixts. which have been analyzed in this app. include H<sub>2</sub>O, CO<sub>2</sub>, CO, O<sub>2</sub> and CH<sub>4</sub>. Under special conditions the least detectable quantity of a component can be pushed to a limit of 0.025 cu. mm. The general methods of the analysis are taken from conventional vacuum technique (the gases are handled at low pressures over Hg and solid reagents) and are refinements of methods previously reported (cf. Prescott, *C. A.* 23, 1015).  
W. F. Bruce

4083', 1939

(Pressure required to obtain  
a fixed volume in measuring  
chamber)

Absorption of water vapor from gases in the cyclone-foam apparatus. S. A. Bogatykh. *Khim. Mashinost.* 1964(2), 17-21. The cyclone-foam app. consists of a cylindrical chamber with a spiral gas inlet at the bottom and a similar spiral exit at the top. Liquid enters the chamber through a pipe and valve arrangement. The centrifugal force set up by the gas being supplied through the spiral tube causes a cyclic motion of the gas and liquid in the chamber, as well as foaming. The 2-phase foam medium is lifted up by the whirling action of the gas and sepd. in the upper (exit) spiral. LiBr aq. solns. were employed as the absorbent in the cyclone-foam drying app. The following dependences were detd.: the mass-transfer coeff. increases with increasing velocity of air, the dew point of the air increases, and the performance of the app. decreases with increasing velocity of air. The optimum air velocity is 5 m./sec., and the optimum height of the foam is 300 mm. The performance of the app. increases with increasing concn. of LiBr soln. The efficiency of the app. is somewhere between 75 and 95% and is defined by:  $\eta = (d_1 - d_2)/d_1 - (d_{f1} + d_{f2})/2 \times 100$ , where  $d_1$  and  $d_2$  are moisture contents of the air before and after the app., resp., and  $d_{f1}$  and  $d_{f2}$  are the equil. H<sub>2</sub>O vapor pressures above the soln. before and after the app., resp.

J. Jindra

325a, U. 61



## Low Point

Dew-point method of determination of water vapor in natural gas. L. E. Woodruff. *Western Gas* 11, No. 2, 22-4(1935); cf. *C. A.* 29, 1610!.—A new app. is described which permits H<sub>2</sub>O vapor content to be detd. in lines where gas is flowing. The test is usually carried out under atm. pressure. The refrigerant is ether, which is evapd. during the test. The dew point is detd. and evapn. of the ether is stopped. The app. warms up and disappearance of dew is noted. The app. can be applied only to dew points below 40°F. or gas pressure in excess of atm.; this excludes gases with vapor below 0.8%. W. H. Bruckner

3139<sup>3</sup>, 1935

Device for determining the moisture content of gases. I. G. FARBENIND. A.-G. (Paul Gmelin, Albert Ernst and Fritz Ranke, inventors.) Ger. 510,953, Nov. 23, 1927. A dew-point device is used and visual observations are replaced by a radiation-sensitive device such as an elec. resistance thermometer.

850, 1931

Measurement of the moisture content of a gas. N. V. Zhdanova, R. L. Slyusareva, and P. A. Tesner. *Zavodskaya Lab.* 15, 647-9(1949).—A 5% soln. of CoCl<sub>2</sub> adsorbed on Al<sub>2</sub>O<sub>3</sub> or silica gel is a convenient indicator of moisture, after drying at 180-200°. Silica-gel type changes color at 3 g./cu.m. of H<sub>2</sub>O in air, while the Al<sub>2</sub>O<sub>3</sub> type changes at 1 g./cu.m.; these correspond to dew points of -4° and -15°, resp. The use of reactive gases, such as SO<sub>2</sub> or Cl, is contraindicated. Alternatively, the dew point detn. can be used for detg. moisture in air. Direct the gas flow against a polished metallic mirror which is in contact with a thermocouple and which is attached to a Cu rod which can be cooled gradually by an external cold bath; this progressively lowers the mirror temp.; the dew point is detd. by the clouding of the mirror and the moisture content obtained from humidity tables.

G. M. Kosolapoff

1123 f, 1950

Measuring the water vapor content of gases dehydrated by triethylene glycol. Irwin Politziner, F. M. Townsend, and L. S. Reid (Univ. of Oklahoma, Norman). *Trans. Am. Inst. Mining Met. Engrs.* 192, *Tech. Pub.* No. 3200 (in *J. Petroleum Technol.* 3, No. 11, 301-4(1951).—Exptl. data show a very low concn. of triethylene glycol vapor (I) in gases dehydrated at atm. temps. and pressures ranging from 500 to 2500 lb./sq. in. abs., and that the accuracy of dew point measurements is not impaired by the presence of I. The amt. of I present in a stream of dehydrated gas ranges from 1 to 3% of the total I content of that gas. I losses may be reduced by maintaining the glycol concn. of the soln. greater than 95% by wt. The variable N content of a natural gas mixt. has scant effect upon its H<sub>2</sub>O vapor content. Natural gas transported to distant markets by pipe-line is often dehydrated by absorption of H<sub>2</sub>O vapor by either diethylene or triethylene glycol-H<sub>2</sub>O solns. of high glycol concn. C. W. Schuck

1736 C, 1952

Instrument for determining in the field the moisture content of gases having a high dew-point. ROGER MARTIN. *Chaleur et industrie* 8, 649-55(1927).—Description of a modification of Maase's instrument (*C. A.* 17, 1879), designed to make it a practical field instrument. A. PAPINEAU-COUTURE

516, 1928

Direct determination of the absolute moisture content of gases by psychromatic and hygroscopic measurements. Fritz Lieneweg. *Wiss. Veroffentlich. Siemens-Werke* 14, No. 2, 20-31 (1935).—Approximation formulas are worked out for detg. the actual moisture content of gases on the basis of graphic representation of curves showing the same dew point with respect to changeable factors (temp., psychromatic difference or relative humidities). Hereby it is possible to make a direct estn. of the moisture content by means of simple elec. measurements. The method is described with consideration of the probable errors and tolerances. W. T. H.

6532<sup>8</sup>, 1935

Moisture determination in the air and other gases by the dew-point method. Rudolf Czepek. *Arch. tech. Messen, Lfg.* 110, T85-6, 4 pp. (1940); *Chem. Zentr.* 1940, II, 2511.—Reliable and accurate methods for the control of the moisture content of air and gas mixts. by detn. of the dew-point are: (1) A definite vol. of the vapor-air mixt. is cooled under the dew point and the water which condenses is weighed. (2) An elec. circuit is automatically closed by the formation of the deposit (of water). (3) The formation of moisture on a mirror (at the dew point) is detd. by changes in the radiation falling on a photoelec. cell. Recording is also possible. (4) The gas-vapor mixt. is passed through a tube having a temp. gradient along its length with a thermoelement at the point where deposition occurs. (5) The dew point is detd. from the break in the cooling curve which appears after deposition. (6) It is detd. from the clouding of a dew-point mirror over which passes the accurately regulated gas stream which is slowly cooled. (7) This method employs the same principle as (2) above but not only is registration of the dew point possible but also elec. regulation of the moisture content of the gas mixt. This is very sensitive and can be used for a temp. range from -40 to 200° and even more. M. G. Moore

6945<sup>7</sup>, 1942

Moisture-determining apparatus. Wm. Stephe Rogers. *Brit.* 444,330, Mar. 19, 1936. The suction force exerted by unsatd. soil and similar substances on H<sub>2</sub>O within a thick-walled, close-grained, porous pot buried in the substance is measured to det. the moisture content.

Apparatus for determining the moisture content of a sample flow of steam by measuring the quantity of heat necessary to superheat the steam. Dobbie, McInnes & Clyde Ltd. and Herbert E. Reynolds. *Brit.* 444,807, Mar. 27, 1936.

6243<sup>7</sup>, 1936