

The precision of the proposed test meets the requirements of the standard A. S. T. M. method.

In Table I are given, for comparison, typical results obtained by the standard A. S. T. M. procedure and this proposed modification, both at the standard temperature of 77° F. (25° C.).

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Analytical Procedure for Mixtures of Organic Sulfur Compounds

RICHMOND T. BELL AND M. S. AGRUSS
The Pure Oil Company, Chicago, Ill.

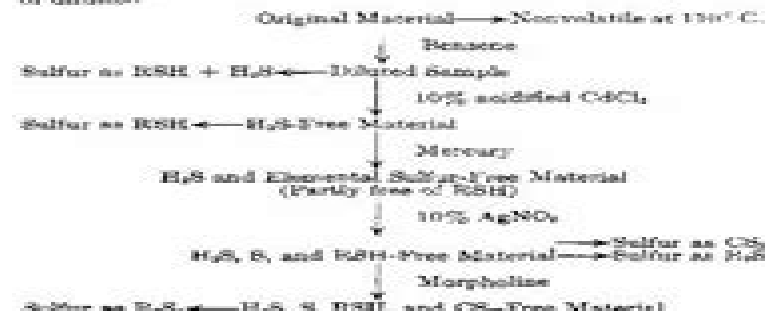
ANALYSIS of mixtures of organic sulfur compounds were encountered requiring the development of reliable analytical methods which could be carried out in logical sequence. Preliminary qualitative analyses of these mixtures indicated hydrogen sulfide, mercaptans, free sulfur, carbon disulfide, and organic sulfides as the chief components. Tests on many mixtures showed no hydrocarbons present, and either no disulfides or only very small amounts.

While complete credit is not claimed for all the individual methods used, the procedure as a whole, including the use of benzene as the diluent, and the modifications of procedure and technique in the various methods employed are worthy of special note because of their value in making a rapid and accurate analysis of a mixture containing the above compounds.

Procedure

The undiluted liquid sample was tested qualitatively for the presence of hydrogen sulfide by shaking a small amount in a test tube with an excess of acidified cadmium chloride solution (7). A yellow precipitate indicating the presence of hydrogen sulfide. If a precipitate formed the material was filtered and the filtrate was shaken with a globe of clean mercury (8). Any darkening of the mercury indicated the presence of free sulfur.

SCHEMATIC DIAGRAM OF PROCEDURE. Horizontal arrows indicate a determination. Vertical arrows indicate a separation or dilution.



Six to eight grams of the original liquid were accurately weighed from a ground-glass joint dropping bottle into a 500-cc. volumetric flask containing at least 300 cc. of *n. p.* benzene. The volumetric flask was stoppered immediately and tipped several times to ensure rapid solution. The dropping bottle was weighed again as rapidly as possible. Benzene was added to the flask up to the 500-cc. mark and the solution was thoroughly mixed.

If hydrogen sulfide was present, the diluted solution was transferred from the volumetric flask to a 1-liter separatory funnel and shaken for at least 5 minutes with 50 cc. of 10 per cent acidified cadmium chloride solution. The lower layer of cadmium chloride solution was discarded and the benzene solution was washed three successive times with 50 cc. of distilled water. The benzene solution was then filtered through a No. 42 Whatman filter paper into another separatory funnel, and 25-cc. portions of this hydrogen sulfide-free material were withdrawn by a pipet for the determination of sulfur as mercaptan according to Method I (9).

If no hydrogen sulfide was present the mercaptan sulfur was determined directly on the diluted sample. When hydrogen sulfide was desired quantitatively, the sum of sulfur as hydrogen sulfide and mercaptan was determined directly on the diluted sample using Method I; hydrogen sulfide was then removed with cadmium chloride solution as above and subsequently the sulfur as mercaptan was determined on the hydrogen sulfide-free material. The difference between these two determinations represented the per cent of sulfur as hydrogen sulfide.

If free sulfur was present, the remainder of the hydrogen sulfide-free material was shaken for at least 10 minutes with 50 cc. of clean mercury to precipitate free sulfur as mercuric sulfide. The mercury was drawn off with as much mercuric sulfide as possible.

If the mercaptan sulfur was over 1 per cent on the basis of the original material, the mercaptans were removed by shaking the undiluted material in the separatory funnel with 25 cc. of 10 per cent silver nitrate solution. After washing with three successive 50-cc. portions of water, the material was filtered through a No. 42 Whatman filter paper into a glass-stoppered bottle. To a 100-cc. portion of this filtrate in a separatory funnel were added 50 cc. of a 1 to 1 solution of morpholine and absolute ethyl alcohol and the mixture was intimately mixed.

After centrifuging for 15 minutes, the supernatant liquid was decanted through a No. 41 Whatman filter paper into a 500-cc. separatory funnel where it was washed three successive times with 50-cc. portions of distilled water, or until the washings were no longer alkaline to litmus. The morpholine-alcohol-free solution was used for the determination of sulfur as disulfides by Method II (10) after its mercaptan sulfur content was determined by Method I.

Separate portions of the hydrogen sulfide-free and elementary sulfur-free material in the glass-stoppered bottle were taken for the determination of sulfur as carbon disulfide by Method III (4) and as sulfide by Method IV (1, 5).

The Analytical Chemistry Of Sulfur And Its Compounds

Part Iii

Joacim Rocklöv



The Analytical Chemistry Of Sulfur And Its Compounds Part Iii:

Organic Sulfur Chemistry Shigeru Oae, 2018-05-04 This volume contains fundamental knowledge regarding the structure and mechanisms of organic sulfur chemistry Topics include sulfur bondings effects of sulfur groups stereochemistry around sulfur substitution ligand coupling within s sulfurane oxidation reduction and rearrangement References in this work total over 2 300 Anyone with an interest in organic sulfur chemistry will find this book to be fascinating reading **The Analytical Chemistry of Sulfur and Its Compounds**, 1971 **The Practice of NMR Spectroscopy** Nugent Chamberlain, 2013-11-11 I GENERAL When a sample containing hydrogen is placed in the Although it is assumed that the reader has been exposed static magnetic field each hydrogen nucleus will precess to the elementary theory of NMR and to the operation at a frequency determined by the magnetic field it of an NMR spectrometer a brief review of some of the actually experiences This field in turn is determined by basic concepts and definitions will indicate the point of the electronic and therefore the chemical environment view used in this book and clarify some of the defini of the nucleus Thus the variety of chemical environ tions The discussion is confined to the hydrogen l iso ments that exist in a molecule will produce a spectrum tope because this is by far the most generally used and of precession frequencies that will indicate the chemical consequently far more data are available for it than for nature of the various parts of the molecule The remain any other isotope This wealth of data in turn leads to ing problem is to observe this spectrum of frequencies the most accurate and comprehensive set of spectra There are two general methods of observing the structure correlations spectrum The Chemistry of Inorganic Ring Systems R. Steudel, 2013-10-22 The present volume considers the most recent developments in the chemistry of cyclic inorganic and organoelement compounds Nineteen of the 22 chapters are based on invited and other lectures presented at the 6th International Symposium on Inorganic Ring Systems held in Berlin on August 18 22 1991 Main group compounds dominate the content from boron via carbon silicon germanium tin nitrogen phosphorus and arsenic to sulfur and selenium The book is organized by element moving from left to right in the main groups of the Periodic Table followed by one chapter each on bonding and nomenclature of ring molecules The list of contributors comprises distinguished scientists from 8 countries *Environmental Technologies to Treat Sulfur Pollution* Piet Lens, L. W. Hulshoff Pol, 2000-01-01 Environmental Technologies to Treat Sulfur Pollution Principles and Engineering provides a definitive and detailed discussion of state of the art environmental technologies to treat pollution by sulfurous compounds of wastewater off gases solid waste soils and sediments Special attention is given to novel bioremediation techniques that have been developed over the last 10 years Information density is unique owing to the many figures and graphs 150 tables over 80 and over 1500 cited literature references A detailed subject index helps the reader to find their way through the different technological applications making it the perfect reference work for professionals and consultants dealing with sulfur related environmental bio technologies Contents Part I The sulfur cycle Part II Technologies to Desulfurise Resources Part III Treatment of Waters

Polluted by Sulfurous Compounds Part IV Treatment of Gases Polluted by Sulfurous Compounds Part V Treatment of Soils and Sediments Polluted by Sulfurous Compounds Part VI Other Applications of Sulfur Cycle Bioconversions in Environmental Engineering Part VII Problems Related to Sulfur Cycle Bioconversions Molecular Luminescence Spectroscopy, Part 3
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relevant American Society for Testing and Materials ASTM standards and test methods including corresponding International Organization for Standardization ISO and British Standards Institution BSI test method numbers To assist readers in understanding the material a glossary of terms is provided Each term is defined in straightforward language that enables readers to better grasp complex concepts and theory References at the end of each chapter lead readers to more in depth discussions of specialized topics This is an essential reference for analytical chemists process chemists and engineers in the coal industry as well as other professionals and researchers who are looking to coal as a means to decrease dependence on foreign oil sources and devise more efficient cleaner methods of energy production The Analytical Chemistry Of Sulfur and Its Compounds (Volume 29; Part 3) and Nuclear-Magnetic-Resonance Data of Sulfur Compounds Karchmer JH.,1971

Recent Developments in Ion Exchange Peter A. Williams,M.J. Hudson,2012-12-06 These Conference Proceedings deal with the papers presented at the International Conference on Ion Exchange Processes ION EX 90 which was held at The North East Wales Institute of Higher Education 9 11 July 1990 The camera ready paper format was chosen so that delegates could receive their copy on arrival at the Conference The Proceedings include reviews of biological materials inorganic ion exchangers the nuclear industry theoretical aspects and new advances In addition there are research papers dealing with industrial ion exchange procedures and new materials The Proceedings should therefore be of interest to those who need to be brought up to date in the various aspects of processes which involve ion exchange and ion chromatography which are now accepted as important in analysis separation processes and process control In each of these areas there have been important developments which are herein described As Editors we should like to express our thanks to the individual authors for preparing their manuscripts in the required format and to Haydn Hughes and Linda Sneddon for their invaluable assistance in compiling these Proceedings PETER A WILLIAMS MICHAEL J HUDSON V 1 CONTENTS v Preface Part 1 Biological Materials The Importance of Ion Exchange Processes in Living Systems 3 R J P Williams The Use of Chemically Suppressed Ion Chromatography in Elemental Analysis 17 J P Senior Biological Separations Using Latex Based Pellicular Resins 23 K

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