

In this connection, it may be of interest to note that on shaking the water and xylene layers in the receiver after the distillation, the water formed a permanent emulsion with some of the xylene, but on adding sufficient red oil, the emulsion was destroyed and the layers separated completely in a short time. No emulsion, however, was formed on shaking pure water with fresh xylene. This emulsion is probably also due to traces of soap. Those familiar with the manufacture of soluble mineral oils, which consist mostly of mineral oil, red oil, and soap, or other emulsifiers, are probably aware with what care the red oil must be incorporated; only a slight excess of the latter to the compounded oil is sufficient to destroy its property of emulsifying in water.

The method, as carried out in our laboratory, is to weigh into a 500 cc. Erlenmeyer flask enough of the soap to yield about 3 cc. of water. An equal quantity of red oil and 150 cc. of water-saturated xylene are added and the contents distilled at the rate of 1 to 2 drops per second. The receiver at the start is filled with 5 cc. of the water-saturated xylene and the distillation is stopped when about 85 cc. are collected. The inside of the condenser tube is finally rinsed out with the xylene and the washings added to the distillate; this rinsing is best accomplished by distilling rather vigorously 15 cc. more of xylene. The receiver¹ consists of a cylinder holding about 120 cc. and is constricted at the bottom to a tube which is about 4 cm. long, graduated in tenths of a cc., and holds about 4 cc. of water.

The reading may be taken at room temperature or brought to any desired temperature in a water bath. Any drops of water adhering to the glass of the vessel may be dislodged by means of a very thin wire twisted at one end into a circle. The xylene layer is usually somewhat emulsified. On standing over night, however, the layers get clear, but the reading is practically no different than if taken a half hour after distillation.

The addition of oleic acid or red oil to the xylene in the distillation method for the determination of moisture in soap, as recommended, increases the accuracy by keeping the soap-xylene liquid more fluid and shortens the time of the distillation by hastening the solution of the soap in the xylene, and by eliminating foaming.

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THE USE OF SODIUM SULFATE IN THE KJELDAHL-GUNNING METHOD

By C. T. DOWELL AND W. G. FRIEDEMANN

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W. G. Latshaw gave in THIS JOURNAL, 8 (1916), 586, the results of some analyses which showed very conclusively that sodium sulfate could be substituted for potassium sulfate in nitrogen determinations by the Gunning modifications of the Kjeldahl method. One of the writers found on coming to this laboratory

that sodium sulfate had been substituted for potassium sulfate since the appearance of Latshaw's article, but instead of using 7 to 8 g. of the anhydrous salt as was done by Latshaw, 10 g. of the hydrated salt were being used. This is equivalent to 4.4 g. of the anhydrous salt. It was thought that it should be determined whether or not the addition of water (as the water of crystallization of the sodium sulfate) would affect the result and the time required for the completion of the digestion following the Kjeldahl-Gunning method.

TABLE I

MATERIAL USED	Clear after Hours	PER CENT NITROGEN	
		9.25 g. $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$	4.076 g. Na_2SO_4
Cottonseed feed.....	1 1/4	3.032	3.054
Cottonseed meal.....	1 1/4	5.896	5.896
Standard wheat shorts...	1 1/2	3.020	3.001
Wheat white shorts.....	1 1/4	2.606	2.603
Molasses feed.....	1 1/2	1.707	1.702
Standard wheat shorts...	1 1/2	2.918	2.918
Standard wheat shorts...	1 1/2	2.841	2.824

Table I shows the results of analyses made to decide this point. There was little or no difference in the time required for digestion, about 10 min. longer time being required where the hydrated salt was used. It should be noted that 4.07 g. of the anhydrous salt were used and not 4.4 g. This was because we wished to compare the results obtained when 5 g. of potassium sulfate were used with the results when its molecular equivalent of sodium sulfate was used. Table II shows the results of analyses made for this comparison.

TABLE II

MATERIAL USED	Clear after Hours	Number of Analyses	PER CENT NITROGEN	
			K_2SO_4 5 g.	Na_2SO_4 4.076 g.
Poultry mash.....	..	2	3.163	3.230
Mill run bran.....	..	2	2.760	2.760
Rice bran.....	..	2	1.891	1.986
Standard wheat shorts	..	2	2.858	2.851
Mill run bran.....	1 1/4	2	2.491	2.485
Mill run bran.....	1 1/4	2	2.690	2.715
Mill run bran.....	1 1/4	2	2.595	2.616

The sulfuric acid cleared up in about the same time using the above salts.

The time of digestion was independent of the salt used.

It should be noted that in our comparisons we used 5 g. of potassium sulfate instead of 10 g. as called for in the official method.

It might be said that this would give wrong results and that the time of digestion would be different from that obtained and the time required when 10 g. are used. Table III shows the results of four analyses made to answer this question.

TABLE III

MATERIAL USED	Number of Analyses	PER CENT NITROGEN	
		K_2SO_4 5 g.	K_2SO_4 10 g.
Oat feed.....	2	0.787	0.792
Cottonseed meal.....	2	6.072	5.909
Dried blood.....	2	14.032	13.880
Mill run bran.....	2	2.943	2.896

The time required to clear was the same, about 60 min., in each analysis. It should be pointed out that a slightly higher per cent of nitrogen was obtained in each analysis where 5 g. of potassium sulfate were used instead of 10. This might be due to a slight oxidation of the ammonia by the great amount of

¹ Graefe's Oil Cylinder, Eimer & Amend Catalogue for 1913, No. 4784.

sulfur trioxide present when 10 g. were used, but no conclusion can be drawn from so few analyses, and in fact the results obtained by Fieldner and Taylor¹ show apparently that the per cent of nitrogen is independent of the amount of potassium sulfate, provided the ratio of grams of potassium sulfate to cubic centimeters of sulfuric acid is not greater than 0.5.

Thirty cubic centimeters of acid were used in all of the analyses reported in this paper. Mercury equivalent to 0.7 g. of mercuric oxide was added and permanganate was added at the end of the digestion.

It is shown by our analyses that either the anhydrous or the hydrated sodium sulfate may be used in the Kjeldahl-Gunning method, that the time of clearing is not affected appreciably by the water of crystallization of the sodium sulfate, and that as little as 5 g. of potassium sulfate is sufficient in the analysis of substances such as we used. No analyses were made with greater amounts of sodium sulfate than 4.07, since that amount gave the same result as 5 g. of potassium sulfate, and 5 g. of the potassium sulfate gave the same result as 10 g., which is the amount used in the official method. It is realized that our reasoning is not quite conclusive because of the lack of a sufficient number of analyses to compare the results when 5 g. of potassium sulfate are used with these when 10 g. are used, but the analyses of Fieldner and Taylor² seem to leave no question on this point.

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THE STRUCTURE OF SCARLET S3R (B) AND PONCEAU 3R(By)

By H. W. STIEGLER
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Scarlet S3R (B. A. S. F.) is one of the more important of the unclassified azo dyestuffs (U. S. Dyestuff Census), some 80,000 lbs. being imported in 1913. It was thought that a determination of its structure would be of interest.

The sample of Scarlet S3R was decomposed by means of $\text{SnCl}_2\text{-HCl}$ solution and the cleavage products separated and purified.

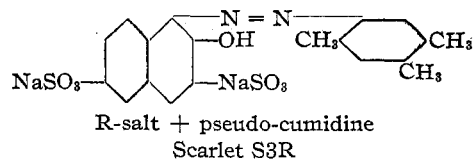
The azo component was identified as amido R-salt (1 : 2-amido-naphthol-3 : 6 di-sodium-sulfonate).

Steam distillation of the alkaline reduction liquid yielded a brownish oil of no definite boiling point. On standing for some time (cold), traces of crystallization were noted. Separation by further cooling yielded a white crystalline solid, identified as pseudo-cumidine (1 : 2 : 4-trimethyl-5-amido-benzene; melting point, 63° C.).

The presence of an oil with the pseudo-cumidine crystals probably indicates the use of crude cumidine, which contains a considerable amount of one of its isomers, mesidine.

Scarlet S3R then, being a monazo dyestuff, has the following structural formula:

¹ Bureau of Mines, *Technical Paper*, 64, 10.
² *Loc. cit.*

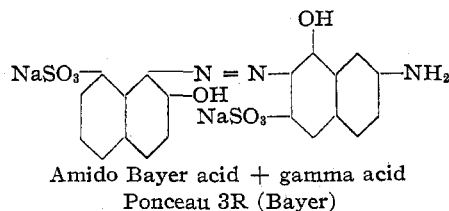


This investigation therefore classes the Badische Scarlet S3R as Ponceau 3R, No. 83 Schultz Farbstoff-tabellen.

In making comparative tests of the Scarlet S3R with several classified Ponceaus, slight discrepancies were noted in the case of Bayer's Ponceau 3R. This dyestuff is listed by Schultz under No. 83 as being of the same structure as that determined for Scarlet S3R.

An investigation established the interesting fact that Ponceau 3R is entirely different in structure from that given by Schultz. Both cleavage products were found to be naphtholsulfonic acid derivatives. Difficulty was encountered at this point in obtaining either product free enough of the other to proceed with their identification, as both were only slightly soluble in water, neutral sodium sulfite, etc.

Small quantities of both components were finally obtained in a pure state. Further investigation established the rather unusual use of amido Bayer acid (1 : 2-amido-naphthol-8-sulfonic acid) as the *dialdo* component, and gamma acid (2 : 8-amido-naphthol-6-sulfonic acid) as the *azo* component, thus giving Bayer's Ponceau 3R the structure:



This investigation indicates an error in Schultz, in that Bayer's Ponceau 3R is *not* crude cumidine + R-salt as stated there, but amido Bayer acid + gamma acid. It also classifies Scarlet S3R (Badische) as Ponceau 3R, No. 83 Schultz.

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AMMONIA AND NITRIC NITROGEN DETERMINATIONS IN SOIL EXTRACTS AND PHYSIOLOGICAL SOLUTIONS¹

By B. S. DAVISSON
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INTRODUCTION

Studies in soil biology dealing with the transformations of the soil nitrogen require frequent and exact determinations of ammonia and nitric nitrogen. The unreliability of the methods in vogue among soil biologists renders necessary a study of the means by which the true value for ammonia and nitric nitrogen can be obtained. The error due to the hydrolysis of nitrogenous organic compounds is quite appreciable, and should be reduced to a minimum. The often

¹ An abstract of a dissertation presented in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the Graduate School of the Ohio State University.