Pak. j. sci. ind. res., vol. 34, no. 4, April 1991

EVALUATION OF COMMERCIAL GRADE POTASSIUM SULPHATE, SODIUM SULPHATE AND COPPER SULPHATE FOR USE IN KJELDAHL NITROGEN ANALYSIS OF SOIL AND PLANT MATERIALS

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(Received September 29, 1990; revised May 26, 1991)

This study was carried out in the year 1989 to explore the possibility of using commercial grade K_2SO_4, Na_2SO_4 and $CuSO_4$ in the salt-catalyst mixture required in Kjeldahl digestion of soil and plant materials. Of the chemicals tested, commercial grade K_2SO_4 and Na_2SO_4 were found suitable for the purpose. On account of some advantages of commercial grade Na_2SO_4 over commercial grade K_2SO_4, its use in the Kjeldahl method is suggested.

Key words: Kjeldahl nitrogen, Commercial grade chemicals, Kjeldahl digestion.

Introduction

The most commonly used method of determining nitrogen (N) in soil and plant materials is the Kjeldahl method [2, 5]. It involves two steps: (i) digestion of the sample to convert organic N to NH_4 -N, and (ii) distillation of the digest with alkali, usually, NaOH to determine NH_4 -N. In the Kjeldahl procedures commonly employed, K_2SO_4 or Na_2SO_4 is used to raise the temperature of digestion, and catalysts such as Se, Hg or Cu are used to promote oxidation of organic matter [2].

In most of the laboratories in Pakistan and other countries, chemicals of reagent grade are used in the Kjeldahl method. These chemicals are available in local markets at considerably lower prices but limited information is available regarding their suitability for use in Kjeldahl N determinations. Such an information is of considerable value to countries like Pakistan where economic resources, to carry out research work, are limited. A previous study conducted in this laboratory [3] showed that commercial grade H_2SO_4 and NaOH are suitable for use in Kjeldahl method. In addition to these chemicals, the other costly material involved in the method is the salt-catalyst mixture which is composed of K_2SO_4, Na_2SO_4, $CuSO_4$. $5H_2O$ and Se in the ratio of 100 : 10 : 1 [2,5]. The present work was, thus carried out to evaluate commercial grade salts for use in the Kjeldahl method.

Materials and Methods

Three K_2SO_4, three Na_2SO_4 (anhydrous) and five $CuSO_4$ chemicals of different brands and grades, were collected for study (Table 1). Initially they were analysed for their inorganic N content [1] and the values obtained were compared with the corresponding reagent grade salts from Merck (a German Company well known worldwide for manufacturing chemicals of standard purity). The salts, with nearly no or limited inorganic N impurity, were further evaluated by using them in N analysis of soil and plant materials by the semi-micro Kjeldahl method. For this purpose, four salt-catalyst mixture were prepared using the test salts of different brands and grades [2]. Six surface soils and six plant materials having varied N contents, were selected for analysis, and ground to pass a 60-mesh screen. Their quadruplicate portions were analysed for N [2,4,5] using 1.1 gm of the salt-catalyst mixture under test and 4 ml of H_2SO_4 for digestion [2,5].

To conclude the study, five commercial grade Na_2SO_4 (anhydrous) were collected in 1991, from five sources (Soil Biology Division, Mutation Breeding Division and Biological Chemistry Division, of this institute; Punjab Scientific Store and Shahid Scientific Store in Faisalabad). Each of the Na_2SO_4 salts were purchased by the division or store in different years; i. e. 1987 to 1991. They were tested for their level of organic and inorganic N impurity [1,2].

Results and Discussion

The inorganic N contents of the chemicals tested are given in Table 1. Potassium sulphates of reagent and certified purity grades from Merck and China, respectively, were found to be nearly N free, whereas commercial grade K_2SO_4 of United Laboratory Chemical had small NH_4 -N impurity. Sodium sulphates of all brands and grades were found to be approximately free of NH_4 - or NO_3 -N. Of the $CuSO_4$ chemicals, only the reagent grade material from Merck was nearly N free (Table 1). All $CuSO_4$ salts of other brands and grades, including the certified purity grade from China, showed N impurity. The extent of impurity was sufficiently serious (18.7 -632.3 μg NH_4 -N/g; 7-25.7 μg NO_3 -N/g) to preclude their use in the Kjeldahl method. Therefore, further evaluation work was confined to K_2SO_4 and Na_2SO_4.

Kjeldahl analysis of different soil and plant materials, using different salt-catalyst mixtures for digestion, is presented in Tables 2 and 3. The data in these Tables showed that the N values obtained by using commercial grade K_2SO_4 and Na_2SO_4 of different brands closely agreed with the

corresponding reagent grade chemicals . Replicate analysis demonstrated that the precision of N values obtained in case of soil and plant materials where commercial grade chemicals were used, was quite good (Tables 2,3). Close concordance of blank titrations, obtained in case of soil or plant materials by using either reagent or commercial grade, K_2SO_4 or Na_2SO_4 confirmed that commercial grade chemicals did not contain a significant amount of organic or inorganic N that could affect the N results. The results, thus showed that commercial grade K_2SO_4 and Na_2SO_4 are quite suitable for use in the Kjeldahl method. It was noticed that all the samples of commercial grade Na_2SO_4 (collected in 1989 and 1991) had very fine crystals and were just like powder. It can, therefore, be presumed that commercial grade Na_2SO_4 may need little grinding before using in salt-catalyst mixture. No lumps were observed in the salt samples and they had a good flow which is a desirable property for preparing a mixture. But, in case of commercial grade K_2SO_4 samples (collected in 1989 and 1991) it was observed that the salt samples, although fine crystalline, had large and hard lumps formed due to moisture

TABLE 1. INORGANIC N CONTENTS, AND PRICES OF K_2SO_4 , Na_2SO_4 AND $CuSO_4$ OF DIFFERENT BRANDS AND GRADES.

Chemical	Brand	Grade	Crystal colour	Price/kg (Rs.)	Inorganic N (µg/g)	
					NH_4	NO_3
K_2SO_4	Merck	Reagent (extra pure)	Colourless	290	NDA*	NDA
K_2SO_4	China	Certified purity	Colourless	270	NDA	NDA
K_2SO_4	ULC **	Cmmercial	White	44	8.4	NDA
Na_2SO_4 (anhydrous)	Merck	Reagent (extra pure)	White	170	NDA	NDA
Na_2SO_4 (anhydrous)	China A ***	Commercial	White	7.8	NDA	NDA
Na_2SO_4 (anhydrous)	China B***	Commercial	White	7.6	NDA	NDA
$CuSO_4 .5H_2O$	Merck	Reagent (extra pure)	Blue	300	NDA	NDA
$CuSO_4. 5H_2O$	China	Certified purity	Blue	280	35.0	7.0
$CuSO_4 5H_2O$	ULC **	Commercial	Blue	67	18.7	21.0
$CuSO_4 .5H_2O$	Hamdard	Commercial	Blue	22	632.3	16.3
$CuSO_4 .5H_2O$	Haq chemical	Commercial	Blue	30	588.0	25.7

* No detectable amount., ** United laboratory chemical, *** Collected from different sources.

TABLE 2. KJELDAHL ANALYSIS OF SOILS USING SALT-CATALYST MIXTURES CONTAINING K_2SO_4 OR Na_2SO_4 OF DIFFERENT BRAND AND GRADE.

Soil No.	Location	Total N (%) in soils as determined by using salt -catalyst mixture containing							
		Merck reagent K_2SO_4		ULC* commercial K_2SO_4		China A commercial Na_2SO_4		China B commercial Na_2SO_4	
		%N	%CV **	%N	%CV	%N	%CV	%N	%CV
1.	Purana Sohawa, Jehlum	0.035	0.00	0.036	1.40	0.035	0.00	0.036	1.40
2.	Chak 217/EB, Sahiwal	0.066	0.88	0.066	1.44	0.065	0.77	0.066	1.24
3.	Barani College, Rawalpindi	0.090	0.00	0.089	0.56	0.089	1.07	0.089	1.30
4.	Basti Dhol Rajian Da, Gujrat	0.111	0.45	0.112	0.52	0.111	0.91	0.112	1.12
5.	Kotli Haji Pur, Sialkot	0.130	0.39	0.131	0.96	0.130	0.39	0.130	0.97
6.	AARI Fruit Plant Nursery, Faisalabad	0.196	1.25	0.196	1.53	0.195	0.65	0.194	1.37
	Mean (% cv)	—	0.49	—	1.07	—	0.63	—	1.23
	Blank titration (mL of 0.01 $\underline{N}H_2SO_4$)	0.4	—	0.5	—	0.4	—	0.4	—

* United laboratory chemical; ** Coefficient of variation between four replicates.

TABLE 3. KJELDAHL ANALYSIS OF PLANT MATERIALS USING SALT-CATALYST MIXTURES CONTAINING K_2SO_4 OR Na_2SO_4 OF DIFFERENT BRAND AND GRADE.

Sl. No	Plant material	N Contents (%) of plant materials as determined by using salt-catalyst mixture containing							
		Merck reagent K_2SO_4		ULC* commercial K_2SO_4		China A commercial Na_2SO_4		China B commercial Na_2SO_4	
		%N	%CV **	%N	%CV	%N	%CV	%N	%CV
1.	Wheat straw	0.354	1.98	0.361	2.51	0.361	1.94	0.361	1.94
2.	Kallar grass shoot	0.917	2.49	0.928	0.75	0.928	1.90	0.924	0.87
3.	Wheat grain	1.596	1.01	1.603	0.87	1.610	0.00	1.610	0.00
4.	Rice grain	1.162	0.00	1.155	1.21	1.169	1.20	1.169	1.20
5.	Berseem shoot	3.017	0.46	3.049	0.58	3.010	0.00	3.024	0.53
6.	Mung bean grain	4.067	0.87	4.074	0.40	4.060	0.69	4.067	0.34
	Mean (%cv)		1.14		1.05		0.95		0.82
	Blank titration (mL of 0.02 N H_2SO_4)	0.15		0.20		0.15		0.15	

* United laboratory chemical., ** Coefficient of variation between four replicates.

absorption. On drying this salt in an oven, the lumps became harder and grinding was rather more difficult. Based on the above advantages, it would, therefore, be preferable to use commercial grade Na_2SO_4.

No detectable amounts of organic or inorganic N were found in all the five commercial grade Na_2SO_4 samples collected in 1991. The results of these analyses indicated that different batches of the salt were nearly N free.

According to prices of the chemicals (Table 1), it was observed that if reagent grade K_2SO_4 was used, cost of this chemcial required per 100 N determinations was Rs. 28.74, whereas if commercial grade Na_2SO_4 was used, the cost was Rs. 0.76. Thus by using commercial grade Na_2SO_4 cost of the salt, required per N determination, can be reduced by 97% without affecting results. Research and advisory institutions of the country where large number of soil and plant samples are analysed for N, the use of commercial grade Na_2SO_4 in Kjeldahl digestion can help cut down the cost of this analysis appreciably. In addition to favourable cost economics, the commercial grade Na_2SO_4 has the advantage that the salt with very fine crystals, may require little grinding, whereas in case of reagent grade K_2SO_4 which has relatively large crystals, grinding is needed to powder it before using in salt-catalyst mixture [2]. The use of commercial grade Na_2SO_4 is, therefore, suggested. Although the study has clearly shown that different batches of commercial grade Na_2SO_4 were nearly N free yet it is advisable that the level of N impurity for a new batch of the salt be determined.

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