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**In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).*

THE INTERNATIONAL SUPERPHOSPHATE MANUFACTURERS' ASSOCIATION

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RAPID RADIOMETRIC DETERMINATION OF POTASH IN COMPOUND FERTILISERS

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The possibility of determining potassium radiometrically is based on the natural radio-activity of potassium. It is well-known that potassium consists of three isotopes, the percentage quantities of which are as follows:

^{39}K	93.260%
^{40}K	0.011%
^{41}K	6.729%

Of the above, only ^{40}K is radio-active. Its half-life period amounts to 1.26×10^9 years. About 12% is converted into ^{40}Ar through absorption of the K atom, and 88% into ^{40}Ca through beta-particle emission. Several reports (see references 1 - 7) in regard to the basic principles of the method of determination have been published. The principle is very simple; the impulses of the radio-active isotope registered by the counter tube are counted for a given period of time. As the proportion of the isotopes in a natural potash salt is invariably constant, the measured activity represents a measure of the potash content of the sample tested.

ACCURACY OF MEASUREMENT:

The first question asked in connection with a new analytical method is usually the following "What degree of accuracy can be obtained? In the present case, the question cannot be answered in so many words. By comparison with the conventional methods of chemical analysis, which require the practice, skill and care of the analytical chemist, the operation of the radiometric method is very simple and anyone can quickly master the technique and produce accurate results. In this case the accuracy of the results depends on other quite different conditions.

STATISTICS:

The spontaneous nuclear changes have the nature of a statistical process; it is impossible to predict when a given nucleus will change; only the probability of the change can be determined. It is clear that the greater the number of atoms in the sample and the longer the observation time, the greater the probability and therefore the more accurate

TABLE 3

Sample	Net Imp.			gra- vim. %K ₂ O	radiometr. with counter tube % K ₂ O		
	5.	min 10.	20.		5.	10.	20.
Crude Salts Violetter Kainit m. Hartsalz	242	255	248	4.2	4.1	4.3	4.2
Empelde	267	241		4.0	4.5	4.0	
Weissgraues Hartsalz	778	784	792	12.8	13.2	13.3	13.4
Heringen	787	799		13.0	13.3	13.5	
Hartsalz	772	780	782	13.1	13.1	13.2	13.3
Friedrichshall	788	784			13.4	13.3	
Begleitflöz- Carnallite	668	665	663	10.8	11.3	11.3	11.2
Heringen	662	662		11.2	11.2	11.2	
Carnallit	576	580	576	9.9	9.8	9.8	9.8
Salzdethfurth	584	573			9.9	9.7	
Blauer Sylvinit mit Kainit	2930	2931	2930	49.6	49.4	49.4	49.4
Hattorf	2932	2930			49.4	49.4	
Orangeroter Sylvinit	1424	1431	1431	24.2	23.8	23.9	23.9
Siegmundshall	1438	1431			24.0	23.9	
Sylvinit	1338	1326	1322	21.9	22.3	22.1	22.0
Friedrichshall	1314	1318			21.9	22.0	
Sylvin	3520	3544	3528	60.3	59.6	60.0	59.7
Ronnenberg	3568	3512			60.4	59.5	
Crude Salts	1496	1477	1468	24.3	25.0	24.7	24.5
Spanien	1458	1459			24.4	24.4	
<u>Finished Products</u>							
Potash Fertiliser Salt	2576 2552	2564 2565	2565	43.6	43.6 43.3	43.5 43.5	43.5
Potash Fertiliser Salt	2944 2910	2927 2899	2913	49.4	49.9 49.3	49.6 49.2	49.4
Potash Fertiliser Salt	3236 3206	3221 3198	3209	54.4	54.8 54.4	54.6 54.2	54.4
Potash Fertiliser Salt	3492 3456	3474 3481	3477	59.2	59.2 58.5	58.9 59.0	59.0
Potassium Sulphate	2912 2886	2899 2922	2910	49.5	49.3 48.9	49.1 49.5	49.3

T A B L E 4

Radiometric determination of K_2O in Compound Fertilisers

TYPE	% P_2O_5	Imp/M % P_2O_5	Imp/M % K_2O	Imp/M net	Imp/M cor. for P_2O_5	Imp/M++ cor. for N.	% K_2O radio. grav.	
<u>Phoskali</u>								
14 x 14+	14.5	36.1	57.2	1307	783	-	13.7	14.0
12 x 18+	12.0	36.1	57.2	1472	1039	-	18.2	18.0
10 x 20+	10.5	36.1	57.2	1535	1156	-	20.2	20.0
9.7 x 31.6+	9.7	36.1	57.2	2141	1763	-	21.4	31.6
10 x 20	10.3	37.8	57.2	1514	1124	-	19.7	19.6
12 x 18	12.4	38.8	57.2	1462	981	-	17.2	16.9
16 x 8	17.2	40.6	58.0	1166	468	-	8.1	7.9
<u>Am-Sup-Ka.</u>								
7.8 x 9 x 9+	9.5	38.1	58.0	853	491	524	9.0	9.0
5.8 x 9 x 15+	9.5	38.1	58.0	1188	828	862	14.9	15.0
3 x 10 x 15	10.5	37.4	57.2	1221	826	844	14.7	14.5
3 x 10 x 15	10.7	38.1	57.2	1229	822	840	14.7	14.7
7 x 11.5 x 17	12.0	36.3	58.0	1403	968	1017	17.5	17.5
8 x 10 x 12	9.7	38.1	58.0	1100	731	775	13.4	13.5
8 x 12 x 15	12.4	36.1	57.2	1256	808	858	15.0	14.8
8 x 12 x 15	12.3	36.4	57.2	1284	836	887	15.5	15.2
10 x 7 x 9	7.5	39.6	58.0	308	511	551	3.5	9.4
9 x 9 x 15	9.9	38.2	57.2	1233	851	906	15.8	15.6

+) Laboratory mixture with KCl (pure analytical) K_2O content cal.

++) Correction = Imp/M net. $\frac{1}{3}\%$ N

the prediction. Now the mean statistical error according to the rules of probability is given by

$$F = \frac{100 \%}{\sqrt{N T}} *$$

In order that this analytical method can compete with the usual methods, the time of measurement must be kept as short as possible and therefore N must be as large as possible. Because of the very low activity of potassium, the largest possible counter tube must be used.

It is true that very large X-ray emission recorders have been known for sometime, and have been used with success by W. Lutz (4) for the determination of potassium. However, in the case of ⁴⁰K, 14 beta-ray conversions correspond to only one X-ray emission, and as, in addition, the probability of registration by the X-ray recorder amounts to only 4% in the optimum case, very large quantities of salt are required when conduction measurements (Lutz used 20-30 Kg). This restricts the practical application of this method to cases where the large quantities required are available to the analyst, as for example in the case of the measurement of bore-hole samples in potash mines.

If it is desired to work with the smallest possible samples, only the counting of beta-particle radiation is practicable. There is also the additional advantage that, owing to the restriction of the path of the beta-particles in the salt, - about 4 mms - it is possible to use an "indefinite layer" i.e. a layer the increase in thickness of which would not result in an increase in the number of impulses. Hence measurement is independent of the weight of the sample and weighing is unnecessary.

In order to develop the radiation method into a rapid one which can compete with existing chemical methods, it was necessary to produce sufficiently large B-ray counter tubes.

Thanks to the progressive work done by the laboratory of Prof. Berthold, Wildbad and the Zentralwerkstatt Göttingen, G.m.b.H., Göttingen, we now have at our disposal aluminium counter tubes, suitable for works use, 200 mms long and 30 mms wide, which have invariably given excellent results in practise. Following closely the method developed by Gaudin and Pannell (6) the counter-tube is built into a steel chamber with walls 30 mms thick, mounted on a swivel (method shown in illustration 1). The salt to be examined is poured into the annulus between the counter-tube and the walls of the steel chamber. After examination, the chamber is tipped and the salt poured back through a funnel into the sample bottle. Only when examining salts with a highly varying content is it necessary to remove the counter tube for cleaning.

What accuracy of measurement can be obtained with this method? For an effective surface of 90 sq. cms, and a density of 1.11 gms/cc, the "active quantity" of pure ground Kcl exposed to the beta-ray counter tube is: 90 x 0.4 x 1.11 = 40 gms. Kcl. With a content of 52.44% K in pure Kcl and a proportion of 0.011% of ⁴⁰K, only 2.31 mgms of the active isotope is contained in this quantity of salt. If one remembers that only half of this small quantity had disintegrated after 1.3 milliard years it appears almost incredible that at any given moment we are able to draw conclusions, from the disintegrations, as to the concentration of ⁴⁰K and hence that of total potash.

In order to understand this possibility we must remember that according to the Logschmidt factor, the small quantity of 2.31 mgms ⁴⁰K contains 6.023 x 10²³ x 2.31/40 x 10³ = 3.48 x 10¹⁹ atoms. If the conversion constant for ⁴⁰K for B-ray emission is 9.12 x 10⁻¹⁶/atom

Note: * It would appear that "F" is the probability of a nuclear change occuring expressed as a percentage, and that "N" is the number of impulses recorded in a time T.

$^{40}\text{K}/\text{min}$ then this is the fraction of ^{40}K atom which change every minute. For a sample quantity of 40gm of KCl, this means $3.48 \times 10^{19} \times 9.12 \times 10^{-16} = 32,000$ changes per minute. In practise, however our beta-ray emission counter tube with pure KCl showed only 3,600 impulses per minute, and hence recorded only 11% of the disintegrations. This low value is caused by the fact that owing to the geometrical arrangement of the salt on the surface of the counter tube only half of the disintegration products are effective and that a considerable portion of the generated electrons are lost through absorption in the layer of salt as well as in the walls of the counter tube.

According to the proceeding formula a mean statistical error of $\pm 1.7\%$ is obtained with 3,600 imp/min., or, converted to K_2O , an error of $\pm 1.1\%$ K_2O , which means that our values will vary on an average between 62.1 - 64.3% if the measuring time is restricted to 1 minute. In view of the fact that the error is a "probable" one, individual values will vary still further plus or minus. If we wish to obtain a more accurate result, we must prolong the period of measurement when the error is reduced in proportion to \sqrt{t} . It is advisable to adopt such a period of measurement as to make it possible to count about 10,000 impulses; then a statistical error of $\pm 1.1\%$ is obtained. According to the K_2O content of the sample, the measurement takes 5 - 20 minutes; in order to ensure that the value obtained - which after all only represents a "probable" value - does not lie outside the statistical error, it is imperative to repeat the measurement. If the second value is very much outside the desired limit of error, measurement must be repeated for a third time. Practice has shown that two or three measurements each lasting 5 minutes are sufficient for high grade potash salts used in our industry. When examining compound fertilisers - on this matter we propose to submit a special report at the end of this paper - the period of individual measurements should be increased to 10 minutes.

We have drawn special attention to the influence of the "statistical aspect" on the error of measurement, because herein lies a special characteristic of the measurement of radiation not found in the chemical method of analysis; this characteristic must not be overlooked.

OTHER SOURCES OF ERROR

It is not possible to increase the accuracy required by merely making the statistical error as small as possible, i.e. by the measurement of a sufficiently large number of particles. It has been shown that as the counter tube surface is increased, it is difficult to obtain an accuracy greater than $\pm 1\%$. For this practical limit a number of factors which have already been reported (8) are responsible and only a summary of which will be given here.

CHARACTERISTICS OF COUNTER TUBES

As compared with Geiger counters, beta-ray emission counter tubes shown an increase of 5%, i.e. the indication of the impulses depends to some extent on the working potential of the counter tube; an operational error of 10 volts results in a measurement error of 0.5%. It is therefore important to reproduce as closely as possible a standard method of operation.

INFLUENCE OF COMPOUND COMPONENTS AND SIZE OF GRANULE

At the beginning of our work we found a slightly higher impulse rate (Imp/% K_2O) when examining crude potash salts than that produced by fertiliser salts. We assumed at the time that this was due to the presence of a second radio-active substance. In the course of further research when a large number of the most varied potash deposits were examined, it was found that the variations in crude salts were to be attributed to two different factors. Contrary to the statements in the literature the physical form of mixed components

influences the result to a slight extent when measuring the amount of beta-radiation. In illustration 2 for example, the standardisation of the beta-ray emission counter tube is made with mixtures of KCl and NaCl. With medium NaCl content, a small deviation from a straight line relationship is observed and this amounts to about +0.5% K₂O. As this deviation was considerably greater when sodium fluoride was added, (i.e. with a higher sodium content in the mixture) while other components such as Kiesente or sand, in the mixture had no effect, it would appear that the cause was due to a special property of the sodium ions when exposed to beta-rays.

The measurement of potash salts associated with various anions showed the following values:-

Table 1

Substance	Theoretical K ₂ O Content %	Impulses per minute	Measured K ₂ O content	Difference
K ₂ Cr ₂ O ₇	32.0	1924 ± 10	32.4 ± 0.2	+ 0.4
KMnO ₄	29.8	1806 ± 16	30.4 ± 0.3	+ 0.6
KNO ₃	46.6	2751 ± 10	46.4 ± 0.2	- 0.2
K ₂ SO ₄	54.15	3200 ± 10	54.0 ± 0.2	- 0.1

As will be seen from the above table it would appear that the type of anion has some effect on the result of the estimation. This shows that in the accurate determination of potassium by beta-ray emission measurement, especially in mixtures, account must be taken of the anions and cations. Although this is not important with the relatively pure potash salts used in our industry, it is not so when dealing with compound fertilisers as will be shown later.

A further and very interesting fact is that a variation in size of the granules exerts a considerable effect on the result of the measurement. With increasing granule size, an increased number of impulses is registered.

Table 2

Size of Granule (mm)	Pure KCl			Mixture of KCl + NaCl		
	Quantity of Sample (gms)	Net Impulses per min.	%	Quantity of Sample (gms)	Net Impulses per min.	%
Moulded hollow cylinder	343	3856	3.8	-	1949	3.2
1.2 to 3.0	219	3870	4.2	229.1	1965	3.9
0.4 to 1.2	237	3755	1.0	-	1907	1.0
0.4	257	3716	0.0	251.1	1889	0.0

The foregoing table shows a particularly instructive selection of the numerous measurements made to test this effect. With the aid of a steel mould, a hollow cylinder of pure KCl was moulded which could be fitted tightly into the apparatus. As shown in Table 2, the net value of the impulses of the cylinder is about 4% higher than that of the same salt reduced to a granule size of 0.4 mms. The same phenomena can be demonstrated with a mixture of equal parts of KCl and NaCl.

That the increase in activity with an increase in the size of the granule cannot be attributed to an additional measurement of gamma radiation is shown by the fact that 219 gms of the coarse potassium chloride material showed an increase of 4% in the impulses as compared with 257 gms of finely ground salt.

At the moment we are not able to give a definite explanation of this effect. As far as we have been able to ascertain, it is probably a question of the influence of the size of the granule on the deflection of the beta-rays. For exact measurement, attention must be paid to the size of the salt granules, which should be uniform; to obtain this, the salt samples are usually ground to a fineness of approximately 0.5 mms, so that it can be readily poured. Compound fertilisers are passed through a sieve with a 1 mm mesh screen.

RESULTS OF MEASURING VARIOUS CRUDE SALTS AND FINISHED PRODUCTS OF POTASH

In order to test the usefulness of the method a very large number of crude salts and finished potash products were examined. In the case of Sylvinit, i.e. crude potash salts containing only rock salt impurity, only a small correction of the standard curve for potash content has to be made. In order to ascertain whether and how far it would be worth while to increase the period of measurement, measurements lasting 5, 10, and 20 minutes were carried out. Table 3 shows some of the results of these measurements. This table reveals that differences larger than $\pm 0.8\%$ between radiometric and gravimetric estimations do not occur. It has also to be remembered that an error of about $\pm 0.4\%$ has to be allowed for in the case of gravimetric analysis. On an average, the differences amount to about $\pm 0.2\%$. Although a careful study showed that the differences were somewhat smaller with the 10 minute values of the estimates, the accuracy of the 5 minute estimate is adequate for most works purposes.

TESTS WITH COMPOUND FERTILISERS.

The first experiments carried out some years ago on the determination of potash content of compound fertilisers by the radiometric method show that there were special difficulties because almost all rock phosphates are more or less radio-active. This radio-activity exists because of the very small uranium content of natural phosphates; it is well known that experiments have been conducted in the United States to recover the uranium as a by-product during the treatment of phosphate rock in the production of phosphoric acid. It may be mentioned in passing that such different phosphates as Pebble and Morocco show a radio-activity which is surprisingly similar. By means of our method of measurement, we obtained an activity of 45 imp/% P_2O_5 for 77% Pebble phosphate, and 42 imp/% P_2O_5 for 68% Morocco phosphate. According to earlier tests, Curacao phosphate showed very little activity and Kola concentrates none.

On resuming our experiments in the Research Laboratory of the Guano-Werke A.G. Lübeck-Dänischburg works, we followed up the idea of taking into account the activity of the phosphate when examining compound fertilisers by determining the rate of impulses of the rock phosphate (imp/% P_2O_5) and by deducting the value of the activity of the phosphate from that of the compound fertiliser.

Tests showed, however, that this method of computation resulted in estimates of K_2O content which were too low. While searching for the source of the error, we found that the impulse rate from superphosphate was much lower than that from phosphate rock and, further, that this rate was not constant.

Diagram 3 shows the relation between the impulse rate for superphosphate made from Pebble phosphate and morocco phosphate with the age of the superphosphate. The diagram shows that, at first, the activity increases rapidly and that in the course of some 20 days it approaches asymptotically its final value. The initial value of the impulse rate measured shortly after the reaction stage, was only about 60-70% of the value for the untreated phosphate rock used. The decrease in the impulse rate during the period of attack of the rock and its subsequent rapid increase can be readily explained by the theory that radon particules (radium emanation) contained in the small mineral crystals of rock phosphate escape when the raw phosphate is broken down. During storage the radium remaining in the superphosphate is reformed. As the half-life period is only 3.85 days, the equilibrium between radium emanation and reformed radium is restored.

The difficulty with the irregular and inconstant radio-activity with superphosphate can be overcome by taking a sample of the superphosphate used in the production of compound fertilisers, as well as a sample of the compound fertiliser: the activity of both samples is then measured. Hence, for the determination of potash content of a compound fertiliser, the estimation of the activity of the two samples is required, one a sample of compound fertiliser and the other a sample of the superphosphate used. In order to keep within the limits of probable error of $\pm 1\%$ ten minutes are required for each determination and with two or three determinations on each sample, approximately one hour is required to ascertain the K_2O content of the compound fertiliser.

This method of production control is greatly simplified when a uniform superphosphate, which has been stored for about 3 weeks, is used in the production of compound fertiliser. Tests have shown that in this case, it is possible to obtain a constant impulse rate, even when different samples of superphosphate, which may show variations in themselves, are used. Thus, we found, for example, in a 20% superphosphate made from 77% Pebble phosphate a constant impulse rate of 40.0 impulses /% P_2O_5 .

Using this method to examine samples produced in the laboratory from pure potassium chloride, satisfactory results were always obtained. The method also proved satisfactory when examining works samples of compound fertiliser containing phosphates and potash. However, when we applied this method in the production control of N.P.K. compounds, we obtained estimates of K_2O content which were 0.5-0.7% lower than those determined by a gravimetric analytical method. On examining the cause of these low results, it was apparent that the ammonium salt had the property of absorbing α particles.

Diagram 4 shows results obtained when using various mixtures of potassium chloride and ammonium sulphate. It can readily be seen that with mixtures containing large quantities of ammonium sulphate, the measured activity is noticeably smaller than that which would be obtained if it were directly proportional to the concentration. In this instance, therefore, conditions are reversed as compared with those described earlier in connection with the behaviour of mixtures of potassium chloride and sodium salts. Whilst sodium salts possess a somewhat lesser power of absorbing electron rays than would be expected from their mass, ammonium salts obviously show a somewhat greater absorption. When examining compound fertilisers containing ammonium salts, it is therefore necessary to make a small correction. It is necessary to determine this correction factor empirically by examining an equivalent test mixture of known K_2O content. According to our results, the correction due for nitrogen content is given by the total impulse rate (the sum of K_2O and P_2O_5 activity) multiplied by half the % of nitrogen in the compound. This amount is added to the impulse rate for K_2O obtained by correcting the total impulse rate for P_2O_5 activity.

Table 4 shows a summary of a number of analytical results obtained in this manner. As shown in the table, the agreement between the radiometric and gravimetric estimates is satisfactory.

However promising the determination of potash in a compound fertiliser in a time of 30-60 minutes by the new radiometric method appears to be, it should be noted that a determination can only be carried out if a sample of the superphosphate used in its production is available. Alternatively, if the superphosphate used was at least three weeks' old, and its activity had been previously determined, then the value of its activity could be assumed. This restricts the application of this method of testing compound fertilisers to works production control.

SUMMARY

A method for the determination of potash has been described which enables untrained employees to determine the potash content of potash salts by measuring the intensity of the γ radiation of ^{40}K over a time period of 10-20 minutes. When testing compound fertilisers containing phosphates, the results are complicated by the fact that the majority of rock phosphates contain uranium and therefore are more or less radio-active. Hence the activity of the superphosphate used in the production of the compound fertiliser must be determined and its activity taken into account.

TABLE 3

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12 x 18+	12.0	36.1	57.2	1472	1039	-	18.2	18.0
10 x 20+	10.5	36.1	57.2	1535	1156	-	20.2	20.0
9.7 x 31.6+	9.7	36.1	57.2	2141	1763	-	21.4	31.6
10 x 20	10.3	37.8	57.2	1514	1124	-	19.7	19.6
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5.8 x 9 x 15+	9.5	38.1	58.0	1188	828	862	14.9	15.0
3 x 10 x 15	10.5	37.4	57.2	1221	826	844	14.7	14.5
3 x 10 x 15	10.7	38.1	57.2	1229	822	840	14.7	14.7
7 x 11.5 x 17	12.0	36.3	58.0	1403	968	1017	17.5	17.5
8 x 10 x 12	9.7	38.1	58.0	1100	731	775	13.4	13.5
8 x 12 x 15	12.4	36.1	57.2	1256	808	858	15.0	14.8
8 x 12 x 15	12.3	36.4	57.2	1284	836	887	15.5	15.2
10 x 7 x 9	7.5	39.6	58.0	308	511	551	3.5	9.4
9 x 9 x 15	9.9	38.2	57.2	1233	851	906	15.8	15.6

+) Laboratory mixture with KCl (pure analytical) K_2O content cal.

++) Correction = Imp/M net. $\frac{1}{3}\%$ N