HOMOGENEITY EXAMINATION OF PREMIXES AND FEEDS II

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In our previous paper, results of homogeneity examinations based on analytical chemical methods have been reported on. On the basis of the results, the distribution of components, i.e. the qualitative change of the mixture in the course of mixing process could be followed. Due to the great number of the necessary tests and to the complexity of the system examined, however, the time demand of homogeneity examinations performed by the conventional analytical chemical methods is fairly great. In order to reduce the testing time and to extend the range, the application of radioactive indication method has been considered for homogeneity examinations of premixes and feeds, as well. The advantages of radioactive indication method — high sensitivity, rapidity, independence of the complexity of the material — make it especially suitable for the examination of mixtures and mixing procedures, therefore it is widely used also for homogeneity examinations in the most various branches of industry.

From the point of view of technology, mixing procedures of feeds were investigated by Buist, applying radionuclide ^{198}Au [1]. Distribution of cobalt content in mineral — premixes was searched by Fusch and Beer. In the course of their experiments, radionuclide ^{60}Co was used for labelling [2].

Aspects of application of radioactive indication

For radioactive indication only isotopes not altering the composition of the premix and feed, of no toxic effect, able to emit gamma photons having suitable energy for detection, whose half-period is metrologically convenient and are easily available, can be applied. These requirements are equally met by radionuclides ^{56}Mn and ^{131}I. The half-period of ^{56}Mn is 2.57 hours, to be regarded as quite advantageous, since after cooling for 24 hours, the labelled material practically does not contain any radioactive component. Its draw-

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back is, however, that the initial activity should be chosen relatively high in order to keep the radiation intensity of samples examined high enough, as otherwise the relative error of the nuclear measurement unfavourably increases.

To the examination of premixes, radionuclide ^{56}Mn has been applied, produced previously from the natural isotope ^{55}Mn by nuclear reaction (n, γ) in the thermal zone of the reactor of the Technical University, Budapest. For radioactive indication of feeds, commercial radionuclide ^{131}I has been used. In the case of both premixes and feeds, the homogeneity was numerically evaluated by means of the corrected empirical relative standard deviation of the sample counts. The great advantage of the relative standard deviation is its independence of the concentration unit, thus counts proportional to the concentration can directly be utilized instead of concentration.

Experimental

Examination of premixes: the composition of premixes is shown in Table I.

From among the constituents, only 4 ones have been examined for homogeneity: vitamins AD_3, K_3, B_6 and amprolium. The four constituents chosen represented the other constituents and included the extreme cases, as regards concentration and physical properties. Vitamin AD_3 is a gelatinous capsule, vitamin B_6 is a clear crystalline substance, vitamin K_3 is a fine granulous powder, while amprolium is a sticky coagulating powder of fine granular structure. Four mixings have been performed separately, labelling one constituent in each case. The constituents were coated by the diluted, alcoholic solution of ^{56}Mn in a closed atomizing apparatus during permanent mixing. Concentration of radioactive solution was about 19 kBq/ml, the amount of radionuclide was chosen so that the specific activity of the mixture should reach a value of about 1.85 MBq/kg. Uniformity of coating was checked by the corrected relative empirical standard deviation of the counts of samples taken from the labelled premix. Since we intended to perform the homogeneity examination of premixes on a 5 g sample therefore quantities of the labelled constituents being present in 5 g premix were weighed. 10 samples each were weighed at an analytical accuracy and the counts of the samples were determined. By calculating the corrected relative empirical standard deviation of the counts, values as good as those got in the course of mixing were obtained.

Relative standard deviation values for the constituents:

$$s_{rel} \quad (\text{vitamin } AD_3) \quad = \pm 0.05$$

$$s_{rel} \quad (\text{vitamin } K_3) \quad = \pm 0.03$$

Table I

Composition of the premix

Component	%
Vitamin AD_3 product	0.32
Vitamin E	0.60
Vitamin K_3	0.04
Vitamin B_2	2.32
Vitamin B_{12}	0.40
Vitamin B_1	0.12
Vitamin B_2	0.20
Vitamin B_6	0.20
Choline chloride	11.00
Niacine	0.50
Amprolium	2.50
Zinc bacitracin	4.70
Manganese oxide	2.33
Iron sulfate	1.00
Zinc-sulfate	2.35
Cupric carbonate	0.05
Calcium iodate	0.16
Bran	71.21

$$s_{rel} \quad \text{(vitamin } B_6\text{)} \quad = \pm 0.02$$

$$s_{rel} \quad \text{(Amprolium)} \quad = \pm 0.06$$

This time the mixing experiments were done under laboratory circumstances, in a drum mixer of about 10 kg capacity. The amount of premix mixed was 5 kg. During mixing, in the 2nd, 5th, 10th, 20 th minutes 10 samples of 5 g have been taken from the various points of the mixture and their counts were determined by means of an energy selective counter with a NaI(Tl) detector securing a low background. The corrected relative empirical standard deviation of the results was plotted vs. the mixing time (Fig. 1). The results of the four separate mixings plotted in one coordinate system permit to compare the mixing rates of the materials examined. Vitamin K_3 — in spite of its presence in the premix in the least quantity (0.04%) — reaches the statistically homogeneous state much more rapidly than e.g. amprolium, present in an amount about 60 times higher than vitamin K_3. The slow mixing of amprolium

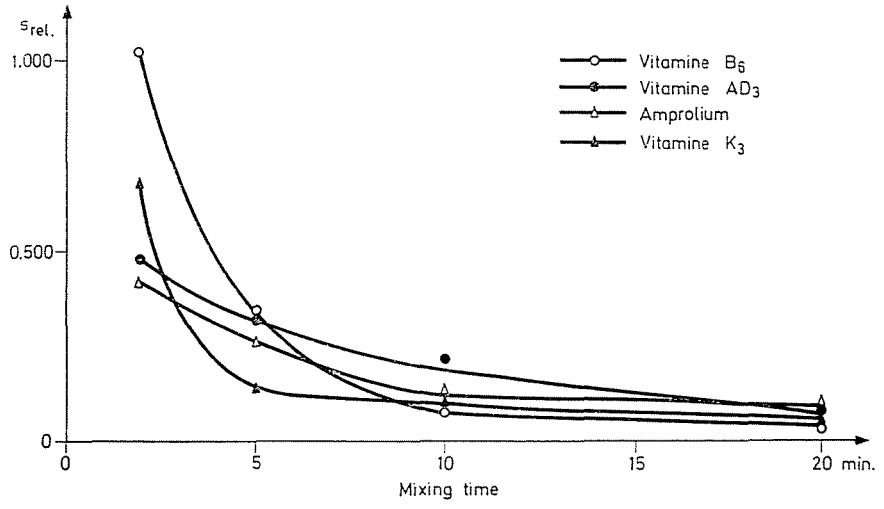

Fig. 1. Distribution of vitamins B_6, AD_3 and K_3 and of amprolium labelled by radioactive ^{56}Mn vs. mixing time, on the basis of the corrected relative empirical standard deviation

can be explained by its unfavourable mixing properties. The curve well demonstrates the time period necessary to reach the statistically homogeneous state in the case of each component.

Examination of feeds

Composition of the feed to be examined is seen in Table II. The homogeneity examination of feeds is aimed at establishing how the premix as an independent "component" carrying the essential constituents can be mixed.

Radionuclide ^{131}I is atomized upon the surface of the premix in the form of solution. The radioactive concentration of the solution was about 19 kBq/ml, its KI content was 10 mg/ml and gelatine content about 0.05%. KI was added to decrease the sorption and the gelatine was present in order to fix the radioactive indicator on the surface of the premix. The amount of radioactive ^{131}I was chosen so that the specific activity of the premix should reach a value of about 3.7 MBq/kg. By this activity the relative error of detection was found to be $< 0.5\%$. To check the uniformity of coating, sample amounts of 0.25 g of premixes were weighed, as we intended to perform the homogeneity examination of the feed in a total sample of 50 g size. 0.5% of the 50 g i.e. 0.25 g was the amount of premix expected to appear in the feed sample when assuming perfect distribution. The corrected empirical relative standard deviation of the counting rates: $s_{rel} = \pm 0.011$, i.e. the radioactive indicator was properly distributed and a till lower standard deviation cannot be expected

even during mixing the feed. The labelled premix was mixed in a drum mixer for 10 mins together with the components of the feed. The corrected relative empirical standard deviation of the counting rates of feed samples of 50 g was found to be $s_{rel} = \pm 0.014$, i.e. following mixing for 10 mins, the distribution of premix in the feed can practically be regarded as uniform. This method is suitable to follow not only the "total premix" as a component, but also suitable for the labelling of one constituent of the premix and its homogeneity examination in the feed.

Since the concentrations of the constituents of the premix range from 0.04 to 0.1% and the quantity of the tracer material cannot exceed 1 to 2% of the constituents, from a multicomponent feed the tracer component has to be determined in a ng/g order of magnitude. In our opinion this requirement can be met exclusively by the sensitivity of the radioanalytical method, with determining the quantity of a given component in the feed of given amount by a non-destructive method.

Homogeneity examination of feeds by non-destructive activation analysis

To examine the distribution of the premix in the feeds also non-destructive activation analytical method has been applied. The premix was produced without manganese — similarly to the inactive indication technique reported in our previous paper — then its manganese content was uniformly atomized in the apparatus described earlier and dried upon the surface. Thus, distribution of manganese content of the premix mixed in the feed represented the distribution of the constituents of the premix. The determination of manganese content of the samples was carried out by non-destructive activation analysis. The determination is based on the following nuclear reaction taking place with the thermal neutrons of the reactor:

$$^{55}\mathrm{Mn}\ (\mathrm{n}, \gamma)\ ^{56}\mathrm{Mn}.$$

Radionuclide ^{56}Mn arosen emits γ photons of 0.845, 1.80, 2.12 MeV in addition to β-decomposition, in consequence of which a photo-peak suitable for quantitative evaluation appears on the γ spectrum at 0.845 MeV. Half-period of ^{56}Mn is 2.57 hours.

The premix prepared is mixed with the other components of the feed of a composition seen in Table II. in a counter-current flush mixer and sampling was done in the course of mixing. 10 g samples each were powdered to fineness, then their accurate proportions (about 0.5 to 1 g) were placed into a plastic sample-holder. The samples prepared in this way were forwarded by pneumatic rabbit into the thermal zone of the reactor of the Technical University, Budapest. The thermal neutron yield of the reactor operating at 10 kW is

$1-2.10^{11}$n s^{-1} cm^{-2}. Irradiation lasted for 180 sec. After irradiation, the samples were directed to a NaI (Tl) detector connected to a multi-channel amplitude analyzer and the γ-spectrum of the sample was recorded (Fig. 2). Counts under the photo-peak at 0.845 MeV were calculated by the Covell method, then the manganese content of the samples was calculated comparing with a standard. From the distribution of manganese content

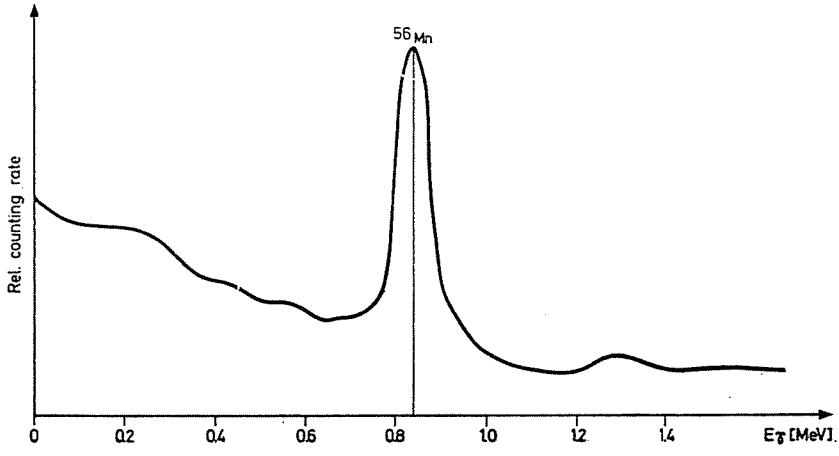

Fig. 2. Gamma-spectrum of feed after activation, containing premix labelled by manganese

Table II

Composition of the feed

Components	%
Corn	44.0
Wheat	20.0
Bran	4.0
Soybean	18.5
Lucerne meal	2.0
Fish-meal	1.0
Meat-meal	2.8
AP-17	1.9
Lime	1.8
Salt	0.3
Aminoacid premixture	3.2
Premix	0.5

conclusions could be drawn on the distribution of the premix in the feed. The advantage of the process lies in the fact that following sample preparation, the previously adjusted testing system is working fully automatically and the spectrum can be evaluated by means of a computer. The process is time saving when dealing with a lot of samples. Further advantage of the method is that the system does not contain any radioactive component during mixing, consequently no radiation protection — hardly to be realized under industrial circumstances, — is required.

Summary

The radioactive indication technique is based on uniformly coating the surface of the tested component by a radioactive material, thus radiation intensity of samples taken during mixing directly demonstrates the distribution of the labelled component. Homogeneity of vitamins AD_3, K_3, B_6 and amprolium content of premixes was examined with radionuclide ^{56}Mn, while homogeneity of premixes in the feed was followed by radionuclide ^{131}I. This technique is rapid and simple and enables to investigate such components whose determination by analytical chemical methods is highly laboursome and time-consuming. In connection with homogeneity examination of feeds the possibility of applying non-destructive activation analyses has also been considered.

References

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