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Neutron-activation Determination of Vanadium in Oils and Catalysts

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Extensive studies of the determination of the vanadium content of various oil products have previously been carried out by neutron-activation analysis (e.g., ref. 1). The nuclear method is faster than the classical methods for the determination of vanadium.

In the present work simultaneous analysis of several oil samples was aimed at to increase the assay rate. In this connection it was necessary to consider the effects on the analytical results of thermal-neutron flux gradients and neutron-flux perturbations caused by the grouping together of the samples themselves. In materials containing hydrogen and carbon neutron-flux perturbation effects occur owing to moderation of fast neutrons as well as to scattering of thermal neutrons out of the sample. It has previously been observed that a 2-ml aqueous sample irradiated in a well-moderated heavy-water reactor gives rise to an activity enhancement of about 10%2. In the present study the total volume of the oil samples amounted to 5 ml. The flux perturbation effects consequently result in different activation rates of vanadium in various samples, depending on the geometrical position of the sample. However, these effects do not influence the accuracy in the determination of vanadium at different levels.

As an extension of this work, precision studies were carried out on an oil sample with a vanadium content at the 0.1 ppm level.

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Experimental

Samples

The oil samples consisted of crudes and residues obtained from different parts of Colombia. Each sample was about 1 ml in volume. The sulphur content of these samples was 0.39–1.39%, and the vanadium ranged from 0.2 to 200 ppm.

The catalyst samples consisted of residues from the cracking stage and also of samples taken during running of the cracker. Each sample was about 100 mg in weight. The aluminium content of these samples was 12%, and the vanadium 400–2400 ppm.

Irradiations

The samples were irradiated for periods of 4 minutes in a thermal flux of about $2 \times 10^9$ n/mm²/sec in the Lockheed reactor at IAN, Bogotá. A decay period of 3 minutes was allowed before the first measurement. A pneumatic system was used for transport of samples to the irradiation position. The loading facility was about 50 m from the counting position.

Measurements

The 1.44-MeV gamma-ray of $^{51}V$ was measured with a $50 \times 50$ mm NaI(Tl) crystal connected to a 512-channel pulse-height analyser. The samples were measured for a period of 1 minute each.

Results and Discussion

Five oil samples, each 1 ml in volume and contained in a plastic vial, were irradiated in a geometrical row arrangement, the standard being in the middle.

Correction factors for each sample were applied for the effects of the thermal-neutron flux gradients and flux perturbations experienced in separate irradiations. These factors were derived by irradiating vanadium standard solutions equal in volume to the samples and arranged in an identical geometrical array. The magnitude of these effects showed a variation of less than 20% between the samples at the extreme positions of the array.

The vanadium determination in the oils and the catalysts suffered some interference, mainly from the nuclides $^{28}Al$ and $^{37}S$. These activities were stripped off. However, the activities of these nuclides limit the degree of sensitivity of vanadium inasmuch the $^{51}V$ peak is added to their Compton continua.

A precision study on the heavy gas oil containing 0.20 ppm vanadium gave a precision of about 5% (8 variates).