

1963

Nuclear Physics & Chemistry

Internal conversion of the 1277 keV transition in ^{22}Ne . Yasuyuki Nakayama and Haruyoshi Hirata. *Nuclear Physics*, **40**, 396 (1963).—See, this Bulletin, **41**, 220 (1963).

Decay of ^{150}Eu . Yasukazu Yoshizawa, Hideo Okamura, Shiro Iwata, Ichiro Fujiwara, Tsunenobu Shigematsu, Masayuki Tabushi, Tsunehiko Tarumoto and Koh Sakamoto. *Nuclear Physics*, **46**, 78 (1963).—Carrier free sources of ^{150}Eu were prepared from bombardment of enriched ^{150}Sm with 11 MeV deuterons and 9 MeV protons. The half-life was determined to be 12.79 ± 0.08 h. The maximum energy of the negatons was 1.02 ± 0.01 MeV and the Kurie plot showed almost an allowed shape. However, the experimental shape factor showed a slight deviation from the allowed one. This decay was estimated to be 0^- or 1^- to 0^+ transition. Positons with a maximum energy of 1.8 ± 0.3 MeV and an intensity of about 0.3 % and the K X-ray following orbital electron capture with an intensity of 3.9 ± 0.2 % were found. In scintillation measurements 15 gamma rays were observed. Gamma gamma coincidence studies were performed at several gate windows for gamma rays and annihilation radiations. The decay scheme of ^{150}Eu was constructed on the basis of these results.

The half life of Tb^{157} . Shiro Iwata, Ichiro Fujiwara, Tomota Nishi, Shiro Goda, Masayuki Tabushi and Tsunenobu Shigematsu. *J. Phys. Soc. Japan*, **18**, 315 (1963).—A sample of Gd_2O_3 was bombarded for four hours with 38.5 MeV alpha particles and the activity of Dy^{157} produced by the $(\alpha, 3n)$ reaction was separated from the target material. Terbium was separated from dysprosium by an ion-exchange method. The decay rate of Tb^{157} nuclei was determined from the counting rate of the K X-ray in a well type crystal, and a K and L electron capture ratio was measured using a NaI (Tl) crystal covered with Al, corrected for absorption by the Al cover, self-absorption, photopeak efficiency, and fluorescence yield. The half life of Tb^{157} was estimated at 160 ± 40 years; a decay mode was proposed.

Analytical Chemistry

Solvent extraction of beryllium, copper, nickel, cobalt and zinc as dibenzoyl-methane complexes. Tsunenobu Shigematsu, Masayuki Tabushi and Tsunehiko Tarumoto. *Nippon Kagaku Zasshi*, **84**, 131 (1963), in Japanese.—Besides the ferric chelate previously reported, dibenzoylmethane forms stable chelate as well with Be, Cu, Ni, Co and Zn, which are extracted with chloroform or butyl acetate in appropriate pH region. Complete extraction can be attained at pH 5~10 for Be- and Cu-chelate, at pH 8~10 for Ni-chelate, at pH 9~10 for Zn-chelate and near

pH 10 for Co-chelate. Among these metals, Be alone is extracted in the presence of EDTA. The extraction method based on the extracting behaviour thus revealed was applied to the following chemical separation process : Be from nuclear fission products ; Ee from Cu, Ni and Co ; and Cu from Ni and Co.

Separation of zirconium-95 and niobium-95 by solvent extraction using acetylacetone. Tsunenobu Uhigematsu, Masayuki Tabushi and Masakazu Matsui. *Bull. Inst. Chem. Res., Kyoto Univ.*, **41**, 212 (1963).

Spectrophotometric method for the determination of scandium with pontachrome azure blue B. Tsunenobu Shigematsu, Katsuya Uesugi and Masayuki Tabushi. *Bunseki Kagaku*, **12**, 267 (1963), in Japanese.—A new method for the spectrophotometric determination of scandium was studied, using Pontachrome Azure Blue B (Color index 43830) as a reagent. Scandium reacts with the reagent at pH 6~7 to form a complex, of which composition seems to be ScR_2 . The complex shows an absorption band with a maximum at $610\text{m}\mu$, which can be used for the determination of scandium. Beer's law is obeyed in the concentration from 0.03 to 1 ppm of scandium, and the molar extinction coefficient is $31,500 \text{ l/M cm}$ at $610\text{m}\mu$. The reagent reacts also with rare earths, although the absorption of their complex is not so large. As well as large amounts of rare earths, Y, Bi, Fe(III), Cu, Al, W, Ti, Cr and Be interfere with the determination of scandium.

Solvent extraction separation of scandium from yttrium and rare earth elements. Tsunenobu Shigematsu, Masayuki Tabushi, Masakazu Matsui, Yasuharu Nishikawa and Shiro Goda. *Nippon Kagaku Zasshi*, **84**, 263 (1963), in Japanese.—The solvent extraction separation of Sc from Y and rare earths with acetylacetone or dibenzoylmethane is studied. In order to find out a convenient method, the above methods were compared with conventional thiocyanate method and TTA method. The extractability was determined radiometrically, using radioactive isotopes, ^{46}Sc , ^{91}Y , $^{152,154}\text{Eu}$, and ^{177}Lu as tracers. Last two elements were selected as the representatives of rare earths. The extraction recoveries of scandium obtained under each optimum condition were as follows :

Organic	Aqueous	Extraction (%)
Thiocyanate-butyl acetate	0.1~0.5M HCl, 5M KSCN	88~88.6
	0.1~0.5M HCl, 6M KSCN	93~94
Acetylacetone-benzene	pH 5~8	97~99.3
-chloroform	pH 6~8	99~99.4
-butylacetate	pH 6~8	97~99.3
Dibenzoylmethane-benzene	pH 5	99
-chloroform	pH 5	98.8
-butylacetate	pH 5	95
TTA-benzene	pH 1.5	97
	pH 2	98

From the results it is revealed that both acetylacetone system and dibenzoylmethane-benzene (or -chloroform) system are as useful as TTA-benzene system. Among them the extraction with acetylacetone or TTA is convenient as far as the separation of Sc from Y and rare earths are concerned. However TTA is better than acetylacetone, because the extraction can be performed at lower pH region.

Determination of scandium in minerals and sea-water. Tsunenobu Shigematsu, Masayuki Tabushi, Yasuharu Nishikawa, Keizo Hiraki, Shiro Goda and Reiko Inoue. *Nippon Kagaku Zasshi*, **84**, 336 (1963), in Japanese.—Sc in several minerals and sea-water samples was determined by the spectrophotometric method with 5,7-dichloroxine. The procedure is as follows: 0.5 ~ 1g. of mineral sample is treated with HF and dissolved in HCl. 100 ~ 200 mg. of Ca^{2+} as a carrier and oxalic acid solution are added and pH of the solution is adjusted to 1.8. After standing over night, precipitate is ignited and the residue is dissolved in HCl. Sc is separated by extracting at pH 1.5 with 10 ml. of 0.2 M TTA-benzene followed by backextracting with 20 ml. of 1 N HCl. After Fe and other interfering elements is pre-extracted at pH 3 as 5,7-dichloroxinate, Sc chelate is successively extracted at pH 9.5 with 10 ml. of chloroform. Absorbances are measured at 397 $\text{m}\mu$ against the reagent blank. Sc in sea-water is concentrated also by coprecipitating with calcium oxalate, using each 200ml. of 2 N oxalic acid for 20 l. of sample. Several mineral samples such as bauxite, garnet, lepidolite, fluorspar etc. were analysed, and the Sc content of 10^{-3} ~ 10^{-4} % was obtained. Sc in sea-water collected at offshore Misaki-cho gave the order of 10^{-3} $\mu\text{g./l.}$

Inorganic Chemistry

Effect of grain size of petalite on the properties of petalite ceramics. Megumi Tashiro and Toshio Maki. *Yogyo Kyokai Shi*, **71**, 65 (1963), in Japanese.—In the previous paper the authors have reported that addition of a small amount of pulverized lithia-containing glass to the powdered natural petalite increased the firing range of their pressed mixture, improving modulus of rupture and thermal shock resistance of their sintered body (*J. Ceram. Assoc. Japan*, **70**, 8 (1962)). In the present study the effects of fineness of the petalite powder on the above behaviors were investigated. 12 kinds of batches comprising 10 % by weight of the pulverized glass (finer than 325 mesh; 10.7 Li_2O , 8.9 MgO , 8.9 Al_2O_3 , 71.5 % SiO_2 by weight) and 90 % of each one or two of the powdered petalites of different fineness (40~80, 80~200, 200~325, >325 mesh) were prepared, pressed into the bars of $5 \times 5 \times 50$ mm, and fired at 1250~1280 °C until their porosity reached almost zero. The batches consisting of no glass frit were also prepared for the comparison. The results obtained are summarized as follows: (1) With decreasing the grain size of petalite powders, the firing range and the firing shrinkage of the pressed body increased. (2) With decreasing the grain size of petalite powders, the modulus of rupture and the heat shock resistance of the sintered body were improved. (3) For the body comprising petalite powder finer than 200 mesh, water-quenching from 1000 °C increased the modulus of rupture of the sintered body. For the body comprising the petalite grains coarser than 80 mesh, however, the reversed effect was observed. (4) With decreasing the grain size of petalite powders, the thermal expansion coefficient of the sintered body decreased, reaching almost zero. From above results, it was concluded that the grain size of petalite powder for making petalite-ceramics should be below 200 mesh.

Hot pressing of a mixture of petalite powder and pulverized lithia containing glass. Toshio Maki and Megumi Tashiro. *Yogyo, Kyokai Shi*, **71**, 196 (1963), in