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TESTING OF ASCORBIC ACID BY ACTIVATION ANALYSIS

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Activation analysis of organic compounds requires care-
ful preparation of the samples because of decomposition
occurring during the reactor activation. This paper is
concerned with the reactor irradiation of ascorbic acid.

Partly because of the small amount of impurities in as-
corbic acid and partly because these impurities were detectable
in the reagents too, chemical enrichment prior to activation
was not feasible.[1,2] Similarly, enrichment by incineration could
not be applied because of the volatility of impurities at high
temperatures.[3,4]

In the case of a non-destructive process, dffficulties
may arise from decomposition due either to the γ background
radiation or to the high temperature in the irradiation channel,
likely to induce gas evolution and an increase of pressure in
the irradiation vessel, which eventually leads to explosion.
Model tests were performed to determine how long ascorbic acid
can be activated without considerably decomposition. Ascorbic
acid samples were irradiated in the 80000 Ci ^{60}Co source for
different periods. Weight losses and oxidation power were deter-
mined to estimate the effect of the γ background radiation in
the reactor. In addition, the thermal stability of ascorbic
acid was examined.

Ascorbic acid samples of 100 mg were exposed to γ-doses of 1 to 1100 MR at a dose rate of 2 MR/h in the 80000 Ci ^{60}Co source of the Isotope Institute of the Hungarian Academy of Sciences, followed by determining the weight loss and oxidation power of the irradiated samples. The latter was done by iodo-metric titration using Variamine Blue as an indicator. The re-sults are shown in Table 1. In can be seen from Table 1 that

TABLE 1

Sample /titer/	Integral dose /MR/					
	0	34	114	250	600	1100
A.G.ascorbic acid	1	0.94	0.94	0.96	0.82	0.62
Technical ascorbic acid	0.94	0.94	0.96	0.94	0.75	0.43

the oxidation of ascorbic acid becomes important above 500 MR. The weight loss of the irradiated samples was less than 1 to 2 per cent throughout, thus no decomposition with gas evolution occurred below 1100 MR.

For an estimated γ-dose of 100 MR/h during reactor irra-diation, the activation of ascorbic acid samples for 8 to 10 hrs seems to be admissible, provided no thermal decomposition occurs.

The thermal behaviour of ascorbic acid was examined by derivatography. Melting occurs at about 180 ^{o}C, followed by decomposition. Since, according to our former findings, the temperature of the samples may exceed 180 ^{o}C during reactor ir-radiation, melting and decomposition during activation should be taken into account. To clear this point, 100 mg samples

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were activated for 1 to 80 min at a thermal neutron flux of 1 to 3×10^{13} ncm^2.sec^{-1}. The samples changed colour and their solubility decreased after a 60 min activation, while after activation for 80 min, caramelization and significant decrease in solubility occurred. Thus short activation /required e.g. for the determination of Al/ do not involve the risk of sample decomposition or melting, while determinations requiring pro-longed irradiation /e.g. in the case of Cu and Mn/ are to be done on previously caramelized samples.

TABLE 2

Technical ascorbic acid		Purified ascorbic acid	
Weight of sample /mg/	Al found /ppm/	Weight of sample /mg/	Al found /ppm/
101	115.3	107	15.7
105	104.9	103	18.6
95	84.2	101	12.5
110	106.6	95	17.6
90	96.1	98	14.1
Average	101.4		15.7
$\sqrt{\dfrac{\Delta_i^2}{n/n-1/}}$	±5.3		±1.0

The determination of aluminium was carried out by means of a pneumatic tube system permitting irradiation for a few minutes. The results are listed in Table 2.

The results obtained by the irradiation of ascorbic acid for 60 min are summarized in Tables 3 and 4.

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TABLE 3

Weight of sample /mg/	Cu found /ppm/	Mn found /ppm/
145	1.17	1.05
152	1.07	1.10
160	1.14	0.95
142	0.95	1.01
161	1.18	0.87
148	0.93	0.98
Average	1.07	0.98
$\sqrt{\dfrac{\Delta_i^2}{n/n-1/}}$	±0.04	±0.04

TABLE 4

Weight of sample /mg/	Cu found /ppm/	Mn found /ppm/
157	0.39	0.23
140	0.30	0.27
145	0.18	0.14
165	0.16	0.12
143	0.22	0.19
Average	0.25	0.19
$\sqrt{\dfrac{\Delta_i^2}{n/n-1/}}$	±0.04	±0.03

The data in Table 3 refer to an ascorbic acid sample of technical grade, while those in Table 4 to a purified ascorbic acid sample. Both samples were irradiated for 60 min.

TABLE 5

Weight of sample /mg/	Cu found /ppm/	Mn found /ppm/
105	1.08	1.00
115	0.98	1.02
107	0.88	0.92
98	0.91	1.04
88	1.03	0.88
120	0.84	0.85
108	1.01	0.82
Average	0.96	0.93
$\sqrt{\dfrac{\Delta_i^2}{n/n-1/}}$	±0.03	±0.03

TABLE 6

Weight of sample /mg/	Cu found /ppm/	Mn found /ppm/
157	0.34	0.24
161	0.28	0.18
152	0.26	0.15
145	0.25	0.17
143	0.31	0.21
165	0.20	0.16
Average	0.27	0.18
$\sqrt{\dfrac{\Delta_i^2}{n/n-1/}}$	±0.02	±0.01

Also caramelized samples were irradiated for 10 hrs. The results are listed in Tables 5 and 6. The data presented in Tables 5 and 6 show that there is neither a loss in copper or manganese content nor a decrease in the accuracy of the determination due to caramelization.

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MÖSSBAUER STUDY OF $[(C_4H_9)_3PCH_2C_6H_5][Fe^{II}(CN)_3]$ IN BENZENE AND ANISOLE

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Infrared and Mössbauer spectra of $[(C_4H_9)_3PCH_2C_6H_5][Fe^{II}(CN)_3]$ in benzene and anisole show an increase in the electron density at the iron/II/ nucleus upon dissolution, owing to the donation of delocalized π-electrons by the solvent molecules. Three iron/II/ species of different electron structures are present in the decomposition product formed in the solution.

In the neutralization of hydrogen hexacyanoferrates/II, III/ with phosphonium hydroxides, cyanoferrates containing concomitantly phosphonium and hydrogen ions as cations are formed.[1] In the first step of the thermolysis of these complexes, hydrogen cyanide is evolved in a quantity corresponding to the number of hydrogen ions. Depending on the nature of the phosphonium cation, the residual compounds are thermally stable in a certain temperature interval which makes the preparation of some novel compounds possible.

The reaction of $H_4[Fe(CN)_6]$ with tributylbenzylphosphonium hydroxide gives a complex of the composition $[(C_4H_9)_3PCH_2C_6H_5]H_3[Fe^{II}(CN)_6]$, whose thermal decomposition at