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MATHEMATICAL, PHYSICAL & ENGINEERING SCIENCES

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C. T. Pillinger and A. P. Gowar

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THE SEPARATION AND SUBDIVISION OF TWO 0.5 g SAMPLES OF LUNAR SOIL COLLECTED BY THE LUNA 16 AND 20 MISSIONS

By C. T. PILLINGER and A. P. GOWAR
The Organic Geochemistry Unit, School of Chemistry, University of Bristol,
Bristol BS8 1TS, England

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[Plate 1]

CONTENTS

Two 0.5 g samples of Luna 16 and 20 soil have been separated into a number of fractions according to size, visual appearance, density and magnetic susceptibility. Aliquots of suitable fractions have been distributed to eight British Laboratories for further investigation.

INTRODUCTION

Two cores of lunar soil have been returned to Earth by the Soviet automated spacecraft Luna 16 and 20. Small (0.5 g) samples (L1627 and L2015) of each core have been provided by the Soviet Academy of Sciences for study by British investigators.

The Luna 16 core, a fine, dark-grey powder, typical of mare soils (Vinogradov 1971) was collected at latitude 0° 41′ S, longitude 56° 18′ E from the northeastern part of Mare Fecunditatis. A number of obvious stratigraphic units were observed by Soviet workers dissecting the core (Vinogradov 1971); sample L1627 was from the 27 cm level of Zone C (15–28 cm).

Luna 20 landed in a highland region of the Moon at latitude 3° 32′ N and longitude 56° 33′ E, near the crater Appolonius C between Maria Fecunditatis and Crisium. In general, the core was much lighter in colour than that collected by Luna 16, but similar to Apollo highland soils

(Vinogradov 1972). The sample provided, L2015, was taken from the 27–32 cm layer of the column.

Both cores have been studied by the Soviet workers and American and French Consortia (see *Lunar soil from the Sea of Fertility* 1974, (Moscow: Nauka); *Earth planet. Sci. Lett.* **13**, 225–466 and **17**, 3–63; and *Geochim. Cosmochim. Acta* **37**, 719–1110 and **37**, 1991–2035 for collected papers). However, a number of techniques, not previously applied to Luna samples, were available to British Investigators.

In order to obtain the maximum information possible from the limited samples available, a committee composed of the participating investigators decided that only selected aliquots of material particularly suited to a certain technique should be examined by that technique. We report here the sequential separation and subdivision of L1627 and L2015 according to size, visual appearance, density and magnetic susceptibility. The scheme adopted was based, in part, on previous studies involving the Apollo samples 10086 and 60501 (Cadogan, Eglinton, Maxwell & Pillinger 1973*a*; Cadogan *et al.* 1973*b*).

GENERAL EXPERIMENTAL

All operations were performed in the clean-room facility of the Organic Geochemistry Unit at the University of Bristol (Abell *et al* 1970). Sample manipulations were carried out using precleaned equipment. Glass vessels and spatulae were immersed in *aqua regia* for 30 min and rinsed in triply distilled water; stainless-steel tools and sieves and aluminium-foil squares for weighing were washed in redistilled toluene/methanol (3:1). All samples were stored in either B14 Quickfit glass tubes or polystyrene conical vials (as supplied by the Curator of Lunar Samples at the Johnson Spacecraft Center, Houston). Solvents used for washing the lunar samples were either B.D.H. AristaR or Mallinckrodt SpectraR grade. The tetrabromoethane for density separation was B.D.H. reagent grade.

SAMPLE PROCESSING

Sample handling in the Soviet Union was carried out using tools of stainless steel, dural, Teflon or nylon (A. P. Vinogradov 1973, private communication) in an atmosphere of helium (Surkov, Rudnitsky & Glotov 1974). Samples were received at Bristol stored in loose-fitting ground-glass stoppered vials. To ensure that the vessels remained closed, they had been wrapped with adhesive tape. Each vial was surrounded by cotton wool and packed in an aluminium screw-top container (figure 1) which was surrounded by a thick plastic outer container. The total masses of the samples as received were: L1627, 0.4825 g and L2015, 0.4983 g.

The processing scheme for both samples is outlined in figure 2; the identification number for each subsample and its mass are given in table 1. The first operation was to remove bulk unfractionated material for trace element analysis and for storage as contingency–posterity material for future investigations. Neither of these samples contained any large single fragments.

Size fractionation

The major portion of each sample was dry sieved into three fractions – > 250, 48–250 and < 48 μm – by means of a micro-sieving assembly (Cadogan 1972). Disks of stainless-steel mesh

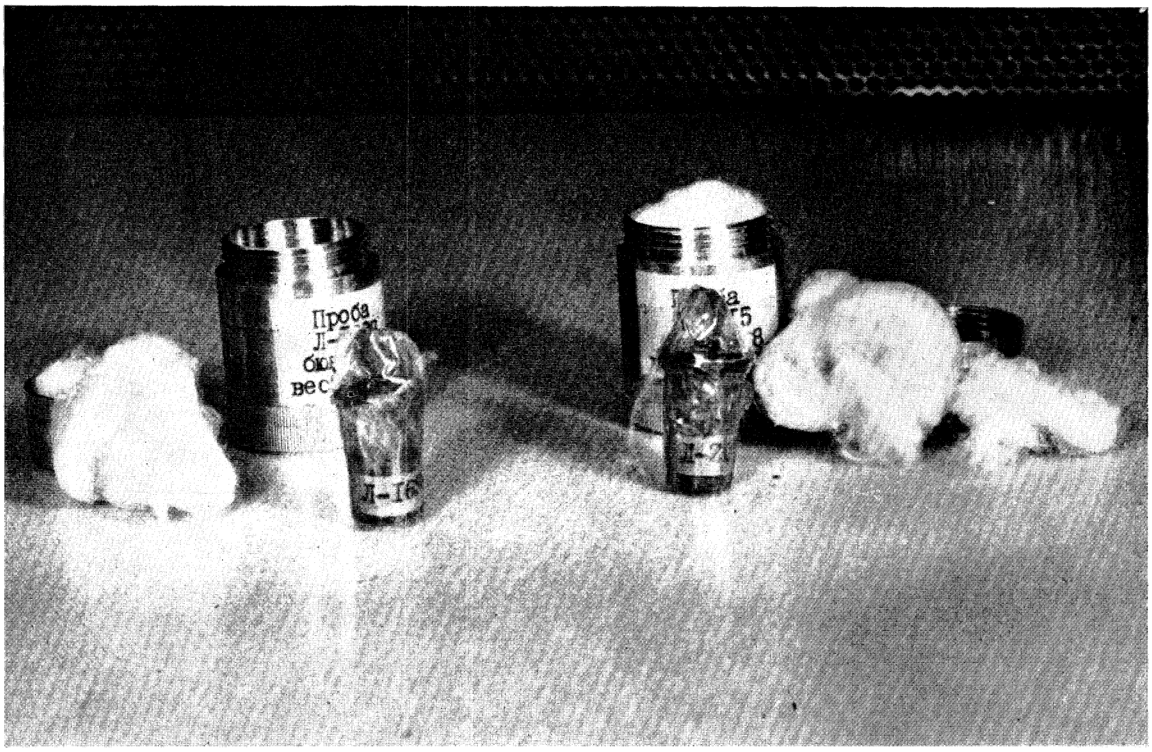

FIGURE 1. The Luna 16 and 20 samples in the glass vials used for transfer from the Soviet Academy of Sciences to Bristol.

(250 and 48 µm aperture, Sankey Wire Weaving Co.) were mounted between cylindrical stainless-steel barrels by means of brass-threaded collars. A disk of aluminium foil was mounted below each sieve to collect material passing through. Bulk soil was sieved at 250 µm by vibrating the sieve column for 5 min with an aluminium-alloy rod driven by an engraving tool. The < 250 µm material was transferred to the 48 µm mesh and shaken for 30 min on a flask shaker.

Table 1. L1627 and L2015 subfractions

	Luna 16		Luna 20	
brief description	sample no.	original mass/g	sample no.	original mass/g
bulk sample	L1627	0.4825	L2015	0.4983
trace analysis sample	L1627,1	0.0693	L2015,1	0.0655
contingency sample	L1627,2	0.0944	L2015,2	0.1044
unsorted material				
> 250 µm	L1627,3	0.0496	L2015,3	0.1044
< 250 µm particles	L1627,4	0.2615	L2015,4	0.2172
sieving residues	L1627,5	0.0030	L2015,5	0.0013
48–250 µm particles	L1627,6	0.1700	L2015,6	0.1197
< 48 µm particles	L1627,7	0.0845	L2015,7	0.0912
washed (w) 48–250 µm particles	L1627,8	0.1380	L2015,8	0.0876
‘finest fines’	L1627,9	0.0269	L2015,9	0.0280
48–250 µm (w)				
L16 < 2.96	L1627,10	0.0462	—	—
L20 < 2.78	—	—	L2015,10	0.0313
magnetic separates h/mm				
3.5–2.5	L1627,10,2,1	0.0042	—	—
2.5–0.5	L1627,10,2,2	0.0034	—	—
0.5–0	L1627,10,2,3	0.0059	—	—
non-magnetic	L1627,10,2,4	0.0013	—	—
48–250 µm (w)	—	—	—	—
L16 > 2.96	L1627,11	0.0512	—	—
L20 > 2.78	—	—	L2015,11	0.0314
magnetic separates h/mm				
2.5–2.0	L1627,11,2,1	0.0049	—	—
2.0–1.0	L1627,11,2,2	0.0055	—	—
1.0–0	L1627,11,2,3	0.0055	—	—
non-magnetic	L1627,11,2,4	0.0038	—	—

A portion (*ca.* 10 mg) of the < 48 µm material was further wet-sieved in carbon tetrachloride using electroformed nickel sieves (Endecotes) to provide 48–40, 40–25 and < 25 µm fractions for oxygen isotope analysis (Beckinsale, Bowie & Durham 1977). Luna 16 and 20 showed markedly different size distributions; both samples contained *ca.* 30 % fine material (< 48 µm). However, L2015 was characterized by a much larger portion (*ca.* 30 %) of coarse material (> 250 µm) than L1627 (*ca.* 17 %).

An aliquot of very fine grains (‘finest fines’) was collected from both L1627 and L2015 by washing the > 250 µm and the 48–250 µm sieved fractions by agitation in acetone or methanol (3 ml) respectively to remove ultra-fine particles adhering to the surfaces of larger grains. The suspended ‘finest fines’ were decanted by pipette. The washed > 250 µm fraction was dried under vacuum (130 Pa (1 Torr)). The washed 48–250 µm fraction and the ‘finest fines’ (collected by centrifugation at 1800 rev/min for 2 min and pipetting off supernatant liquid) were air dried at 60 °C. For both L1627 and L2015 the quantities of very fine grains adhering to the

> 250 μm particles were too small for collection. The 'finest fines' recovered from the 48–250 μm fraction were shown to be < 10 μm by scanning electron microscopy.

After washing, the > 250 μm fractions of both Luna 16 and 20 were sorted under the binocular microscope into categories having similar visible characteristics by Dr S. O. Agrell. L1627 yielded only five distinct fractions (table 2). Mineral grains, basalt fragments and glass spherules were infrequent, however, reworked complex grains such as soil microbreccias and glassy agglutinates accounted for over 70 % of the fraction confirming the maturity of L1627.

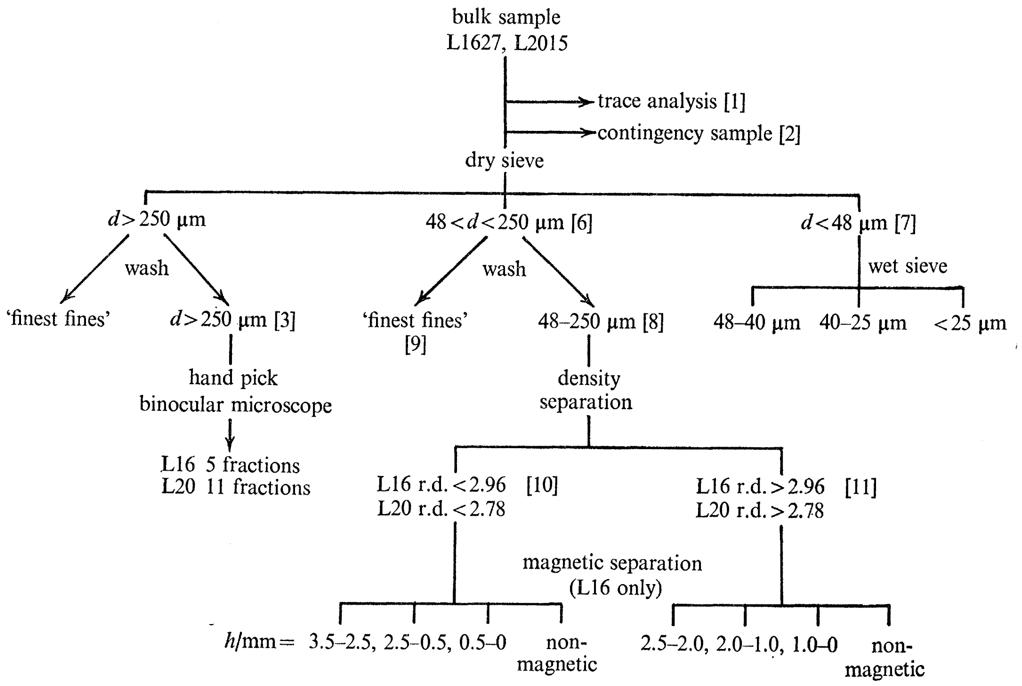

FIGURE 2. Separation scheme for processing Luna 16 and 20 fines. [ ] denotes sample daughter number; *d*, diameter; r.d., relative density; *h*, distance particles move to magnet.

The L2015 sample afforded a greater variety of particle types (table 2). In addition to agglutinates and soil breccias, several different plagioclase textures and some metaclastic fragments were recognized. Two particularly large grains were collected. A glassy feldspar grain weighing *ca.* 11 mg was selected for ^{39}Ar/^{40}Ar dating (Eglinton *et al.* 1977); a breccia fragment of *ca.* 7 mg was used for magnetic measurements (Stephenson, Collinson & Runcorn 1976). Aliquots of all the fractions selected were taken for study in polished thin section by petrological microscope (Eglinton *et al.* 1976).

Density separations

Density separations of aliquots of washed 48–250 μm fractions were carried out by centrifugation for several minutes in either tetrabromoethane (r.d. 2.96) for L1627 or *ca.* 4:1 tetrabromoethane/carbon tetrachloride mixture (r.d. 2.78) for L2015. Fractions were collected by freezing the heavy liquid in liquid nitrogen and cutting the centrifuge tube into two halves. All the density separates were thoroughly washed in carbon tetrachloride and dried in air at 60 °C. At first, the density separation of L2015 was attempted in tetrabromoethane. However, no fractionation occurred since all the grains had a density of < 2.96 and floated. The density of the liquid was adjusted by the gradual addition of carbon tetrachloride until the relative density reached 2.78 and some fractionation was observed. A similar situation was encountered during a density separation of Apollo sample 60501. In that case it was necessary to adjust the relative density to 2.6 before any separation was apparent (Cadogan *et al.* 1973*b*). The low density necessary to separate L2015 is in keeping with the high abundance of plagioclase minerals and glass of low FeO content in highland samples.

Table 2. Particle types visually selected from L16 and 20 $>$ 250 μm fractions

sample no.	amount/mg	brief description
		Luna 16
L1627,3,1	2.6	6 glass spheres (2 brown, 1 dark brown, 1 fawn, 1 large olive, 1 small olive), 1 glass fragment and a sphere like a blister
L1627,3,2	5.4	basalt fragments, very difficult to recognize this material
L1627,3,3	12.4	soil breccia, soft material, tends to break when touched
L1627,3,4	0.7	mineral grains – possibly plagioclase and pyroxene
L1627,3,5	3.8	cindery glassy coated material (agglutinates) – may include some vesicular basalts
L1627,3	21.7	residual unsorted material – considered to be agglutinates like L1627,3,5 but smaller particle size
		Luna 20
L2015,3,1	11.3	largest single crystalline fragment of glassy feldspar
L2015,3,2	1.5	fine glassy agglutinate material
L2015,3,3	8.6	glass-coated soil breccia or microbreccia; may be microcrystalline plagioclase rock
L2015,3,4	1.8	particles of chalky feldspar, very shocked and friable
L2015,3,5	2.7	vitreous plagioclase which reflects light from cleavage faces, similar material to large single fragment
L2015,3,6	14.3	grey microcrystalline plagioclase fragments, with sugary texture, may be anorthosites
L2015,3,7	1.2	coarse polycrystalline plagioclase – anorthositic
L2015,3,8	8.7	microbreccias pyroclastic texture – rectangular white plagioclase crystals scattered in fine grained matrix
L2015,3,9	1.1	single particle of L2015,3,8 with zap pit
L2015,3,10	9.3	similar to L2015,3,8 but no obvious crystals of feldspar, and slightly darker
L2015,3,11	6.6	single fragment of L2015,3,10
L2015,3	33.7	residual unsorted material – contains all particles identified above but on smaller scale

Magnetic separation

Aliquots of the density separates of L1627 were further fractionated according to magnetic susceptibility (Cadogan 1972). The samples were immersed in methanol and separations achieved by successively decreasing the vertical distance (h) between the tip of a soft iron rod mounted on the centre pole of a cylindrical magnet and the sample ($h = 0$ occurs when the rod and sample touch). Fractions were collected from the rod by removal of the source of magnetism, washing with methanol and drying in air at 60 °C. The < 2.96 density separates yielded three magnetic fractions with $h = 3.5$–2.5, 2.5–0.5 and 0.5–0 mm and a small non-magnetic aliquot which could not be collected even when touched by the soft iron rod. A similar separation was achieved for the > 2.96 density material at $h = 2.5$–2.0, 2.0–1.0 and 1.0–0 mm; almost 20 % of the sample was non-magnetic.

TABLE 3. SAMPLE DISTRIBUTION SCHEME

sample type (d, diameter; r.d., relative density)	daughter no.	study (investigator)							
		mineralogy and petrology (Agrell & Bowie)	seleno-chronology (Turner)	magnetic studies (Runcorn)	Mössbauer spectro-scopy (Green-wood)	O iso-topes (Beck-insale)	thermo-lumines-cence (Durrani)	tracks (Durrani)	carbon chemistry (Eglinton)
bulk unfractionated material	2	—	—	—	L16,20	—	—	—	L16,20
$d > 250$ μm, hand picked	3, 1–5 (L16) 3, 1–11 (L20)	L16,20	L20	L16,20	—	—	—	L16,20	L16
$d < 48$ μm	7	L16,20	—	L16,20	—	L16,20	L16,20	L16,20	L16,20
unseparated, $48 < d < 250$ μm	8	L16,20	—	—	—	—	—	L16,20	L16,20
Finest fines	9	L16,20	—	L16,20	L16	—	—	L16,20	L16,20
$48 < d < 250$ μm; light r.d. L16 < 2.96; L20 < 2.78	10	L16,20	—	L16,20	—	—	—	—	L16,20
$48 < d < 250$ μm; heavy r.d. L16 > 2.96; L20 > 2.78	11	L16,20	—	L16,20	—	—	—	—	L16,20
$48 < d < 250$ μm; light magnetic separates	10,2	—	—	—	—	—	—	L16	L16
$48 < d < 250$ μm; heavy magnetic separates	11,2	—	—	—	—	—	—	L16	L16

Distribution scheme

Appropriate samples of separated materials were distributed to eight laboratories for mineralogy and petrology, selenochronology, Mössbauer spectroscopy, magnetic studies, thermoluminescence investigations, oxygen isotope ratio determinations, cosmic ray track and micrometeorite density studies and carbon chemistry measurements (table 3). The results of the investigations performed are described in the accompanying papers.

Summary

From the 0.5 g samples the separation scheme afforded 26 and 22 distinct fractions of L1627 and L2015 respectively; 78 small aliquots were issued to 8 laboratories for study. The total sample losses incurred throughout the processing did not exceed 10 % and only *ca.* 20 % of the samples have been destructively analysed. Approximately 20 % of the original material is in pristine condition and at least a portion of the remaining 50 % would be suitable for further investigation. The experience gained from this work is appropriate to other multi-disciplinary studies involving limited amounts of material such as the Apollo 16 contact surface samples (Pillinger & Gowar 1974, 1975) or the detailed investigation of lunar cores.

We thank the Soviet Academy of Sciences who generously provided the samples of Luna 16 and 20. We are grateful to both the Science Research Council and the Royal Society for financial support and to the British Steel Corporation for a Fellowship to C. T. P. The efforts of Dr S. O. Agrell of Cambridge, who performed the binocular microscope studies, and Mr B. Scarlett and Mr R. Buxton of Loughborough University of Technology, who sieved fractions for oxygen isotope determinations, are gratefully acknowledged.

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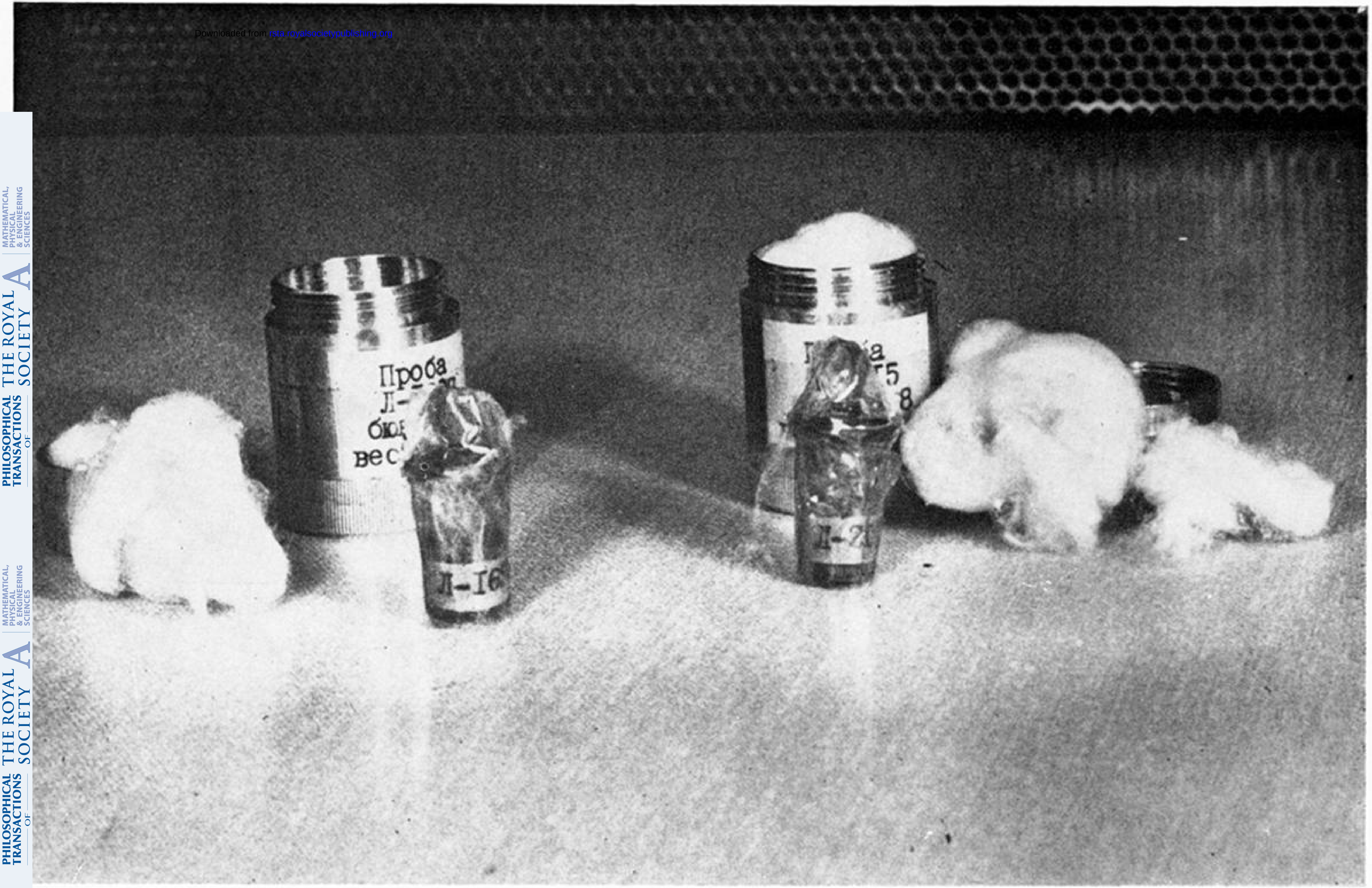

Figure 1. The Luna 16 and 20 samples in the glass vials used for transfer from the Soviet Academy of Sciences to Bristol.