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IDENTIFICATION OF SOIL MINERALS USING OPTICAL CHARACTERISTICS AND SPECIFIC GRAVITY SEPARATION

by

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1. INTRODUCTION

Not only the sedimentary-petrologist but also the soil scientist will be interested in the mineralogical composition of sediments. The significance of the mineralogical composition of the soil in connection with its natural fertility has been recognized in soil scientist circles for a long time, as witness the numerous publications devoted to this subject. For soil research especially the study of the light fraction proves to be essential as this fraction almost always makes up the greater part of the sediment.

When a sedimentary-petrological investigation of sediments is carried out it is usually restricted to the so-called heavy fraction. The heavy fraction investigation is preferred to the light fraction, as usually the former will take less time to reveal a good characteristic of the sediment under examination; for, there is a larger number of characteristic heavy minerals, the minerals concerned are easier to determine, their interrelations are more varied, and differences in origin of the sediment, if any, will thus become clear. However, in certain cases, when sediments of different origin and differing heavy mineral content are mixed in the confluence area of two rivers, the heavy mineral analysis alone will lead to incorrect conclusions. By investigating only the heavy fraction the presence of one of the two components may well remain quite unnoticed (*cf.* VAN ANDEL, 1950; KOLDEWIN, 1955). So for a correct interpretation it is necessary to make an investigation into the light fraction as well. In addition, if the investigator is to gain knowledge of the interrelations of the quantitatively most important minerals, he will almost always have to include the light fraction in his studies.

Data about mineralogical investigations into the light fraction are relatively scarce in the literature. From these data it appears that such investigations were mostly aimed at problems concerning the soil, particularly its mineral reserve. In Dutch literature, especially KOLDEWIJN, DRUIF, and VAN DER MAREL should be mentioned. KOLDEWIJN (1955), however, made a sedimentary petrological study of Rhine sediments. He determined the light mineral associations of the Rhine and samples of the important tributaries were studied to recognize their influence on the main river sediment. A convincing proof of the importance of the knowledge

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of the mineralogical composition of the soil in view of plant feeding was found by DRUIF (1934), when he was making investigations into the soil of the Deli (Sumatra) tobacco area. Here the author discovered a very close relation between differences in production and differences in mineralogical composition, which differences even found expression in the market value of the tobacco. F. A. VAN BAREN (1934, 1935), making a study of the occurrence and significance of potassium containing minerals in the Netherlands soils, carried out an investigation into the light fraction of various sands. He succeeded in revealing differences in nature and proportion of the light minerals of the sedimentary petrological provinces, at the time (1933, 1938) distinguished by EDELMAN and his students on the ground of an investigation into the heavy fraction (cf. table 1). VAN DER MAREL (1950) too studied the relation between existing soil minerals and natural fertility.

Sed. petr. province	Size grade 50–500 µ					
	quartz	acid plag. + orthocl.	microcl.	mica's	rock remains	Schiefer fragm.
A	86.0	5,6	_	_	8.4	_
x l	83.0	7.0	2.6	0.2	7.0	ļ —
Scheemda	85.0	1.0	7.0	-	7.0	_
Saussuriet	64.0	8.8	- 1	0.4	25.6	1.2
Limburg	74.5	6.0	-	-	19.5	_
Elsloo	40.0	1.8	- 1		18.6	39.6
Eysden	43.0		-		27.0	30.0
Lobith	84.0	2.4	-		12.0	1.6

TABLE 1. The mineralogical composition of the light fraction of the sediment-petrological provinces in the Netherlands (after F. A. VAN BAREN, 1934)

The results of pot tests of soils of different mineralogical compositions (those of the Edelman sedimentary petrological provinces) proved the relation between soil type and the extent to which certain nutritious substances (K and Mg) were supplied to the plant.

To facilitate the mineralogical analysis of soils, several workers separated the mineral assemblages with heavy liquids, Among them especially JEFFRIES, GRA-HAM, JACKSON and MARSHALL should be mentioned here. However, the choice of specific gravities and the number of separations attempted have varied considerably amongst different workers. JEFFRIES (1937) by specific gravity measurements. divided the soil minerals in three groups; a feldspar group with a specific gravity of less than 2.62; a quartz group with a specific gravity greater than 2.62 but less than 2.86 and finally a so-called heavy group containing minerals having a specific gravity of over 2.86 The results obtained using these specific gravity ranges, give neither a qualitatively nor a quantitatively correct picture of the proportions of quartz and feldspar because within the fraction with a specific gravity of between 2.62 and 2.86 both quartz and soda-lime feldspars occur. Moreover, it should be taken into account that the specific gravity of minerals and mineral aggregates, dependent as they are on purity and composition, may vary rather widely. JEFFRIES and JACKSON gave in 1949 another procedure to separate soil minerals. They mentioned groups with specific gravity differing from the above mentioned ones, but with the quartz group (2.65), the feldspar group (2.55-2.76) and the muscovite group (2.72-2.80) partly overlapping each other. It is clear that this overlapping does not facilitate the microscopical identification of the consti-

tuents examined. GRAHAM (1943) in his investigation, also made use of the specific gravity differences of the minerals, but in addition he also applied chemical methods. Thus, by dissolving the non-quartz components by means of H_2SiF_{6} , he determined the feldspar content without a detailed determination of the kinds of feldspar concerned. In their reports on analysis results neither JEFFRIES nor GRAHAM mentioned aggregate components. KOLDEWIJN (1955), on the other hand made no use of specific gravity separations. By use of the Becke line method combined with that of the universal stage, he distinguished two groups of feldspars, viz. a basic group with the three refractive indices greater than Canada balsam: andesine, labradorite, bytownite and anorthite; the other group consisting of the acid and intermediate feldspars, orthoclase, sanidine, microcline, anorthoclase, albite, with the three refractive indices less than Canada balsam, and oligoclase, with n α lower, n β and n γ greater than Canada balsam. Thus no complete distinction could be made between the potash feldspars and the plagioclase feldspars. From the methods of the above-mentioned investigations it appears (see also under 2) that they do not satisfy the requirements of an accurate quantitatively mineralogical analysis for plant nutrition.

The intention here is to introduce a more accurate method of analysis based on a specific gravity separation technique developed by FAVEJEE. The results of the investigation of seven selected samples have been depicted in fig. 1. In connection with the development of the investigation, first the more easily determinable sand fraction was studied, the results of which are given below. Later an examination of the finer fractions will be carried out.

The present investigation was carried out on one size grade, viz., the sand fraction of 210-105 microns. It should be stressed, however, that the mineralogical composition of a sediment (or of soil particles) changes with the size. DOEGLAS (1952) has called the relationship between the frequency of certain minerals and their size grade 'granular variation'. From the standpoint of the soil scientist it is of great importance that the mineralogical composition changes rapidly in the size range near the boundaries between sand and silt, and between silt and clay (cf. BLACK, 1957). Thus, it is obvious that for soil-fertility purposes a knowledge of the mineralogical composition of more and of especially the finer fractions is essential; for the finer mineral fragments with their comparatively larger surface areas will more readily impart food to the plant. The knowledge of the composition of the finer fractions will give insight into the short term soil fertility, whereas the mineralogical composition of the sand separates, which were formerly regarded as inert, points to the mineral reserve for a longer period. Only a thorough knowledge of both the sand and silt (and clay) separates will provide a complete insight into the correlation between the mineralogy and the fertility of the soil.

The samples investigated are from different localities, four of them (WR 473, 472, 471 and 470) from Dutch sediments, the remaining three from abroad.

2. DEVELOPMENT AND METHOD OF INVESTIGATION

It is not the intention here to give a description of the preliminary chemical treatment of the sediments investigated, or of the technique of subdividing the sediment by specific gravity (cf. KHADR, 1960).

A few words, however, should be added on the subdivision of the sediments examined into different specific gravity groups. These separations have been carried out with the purpose of facilitating the identification of the constituents of the mineral assemblages. As a matter of fact, it is in general more difficult to make a complete separation of the constituents of a heterogeneous mineral assemblage, than those of an igneous rock. This, of course, arises from the fact that generally the mineralogical composition of a sedimentary rock is not controlled by those laws of chemical relations which occur in igneous rocks. Thus the study of a sedimentary mineral assemblage is greatly facilitated by a separation into several groups, each of which should be comparatively free from representatives of the other groups. This is the more true for light minerals as they have so many optical characteristics in common.

The grouping determined by specific gravity resulted in five fractions, viz. the < 2.59 fraction; the 2.59-2.63 fraction; the 2.63-2.67 fraction; the 2.67-2.89 fraction, and the > 2.89 fraction (the so-called heavy fraction). The choice of these specific gravity ranges was mainly aimed to separate as much as possible the potash and the soda-lime feldspars from quartz. Thus, not only the identification of the minerals was greatly facilitated, but also a first characteristic of the sediment was obtained (*cf.* fig. 1). However, it must be emphasized, that on account of the variability of the specific gravities of the minerals and aggregates the percentages of quartz, feldspar and other constituents cannot be established alone from the weights of the respective fractions.

It is not the intention of the authors to give a detailed description of the separation technique and the apparatus used.¹) A paper dealing with the separation technique is in preparation by Dr. J. Ch. L. FAVEJEE. Briefly the method consists of using mixtures of decaline ($C_{10}H_{18}$) and bromoform having fixed specific gravities.

Before it was decided upon to determine the light fraction obtained microscopically, efforts were made to find a simpler and so less time-consuming method. To this effect attempts were made to determine the grains under the binocular stereoscope (cf. VAN BAREN, 1934); however, except for some conspicuous components, such as coloured quartz, Radiolaria, flint, glauconite and rock fragments it appeared from subsequent checks with the polarisation microscope that this method was not reliable. Thus it was found that the usual microscopical method of investigation had to be employed. After the specific gravity grouping had taken place the various minerals or mineral aggregates were determined by use of the polarisation microscope; of each fraction 100 transparent grains were identified, and their proportions were given in percentages.

The method applied to the microscopical determination was not identical for all specific gravity fractions; the different procedures will be dealt with briefly below. It should be pointed out that measurement of the angles of extinction of the rounded grains did not offer a workable method owing to the fact that distinct cleavage directions and other crystallographical characteristics were often missing; consequently, apart from the optical sign which could only be determined in some cases, the method of immersion remained as the only workable one.

a. The fraction with a specific gravity of less than 2.59, designated as the potashfeldspar fraction, was investigated in a liquid with a refractive index of 1.528, in order that albite, if present, could be distinguished from the mostly predominant orthoclase. Since the $n\beta$ of albite approximates to 1.528, and orthoclase has a distinctly lower refractive index, it is possible by means of this method to distinguish both components. The mineral microcline, the refractive indices of which are all but equal to those of orthoclase, was distinguished from the latter by either the

¹⁾ The specific gravity separations have been carried out by the second author.

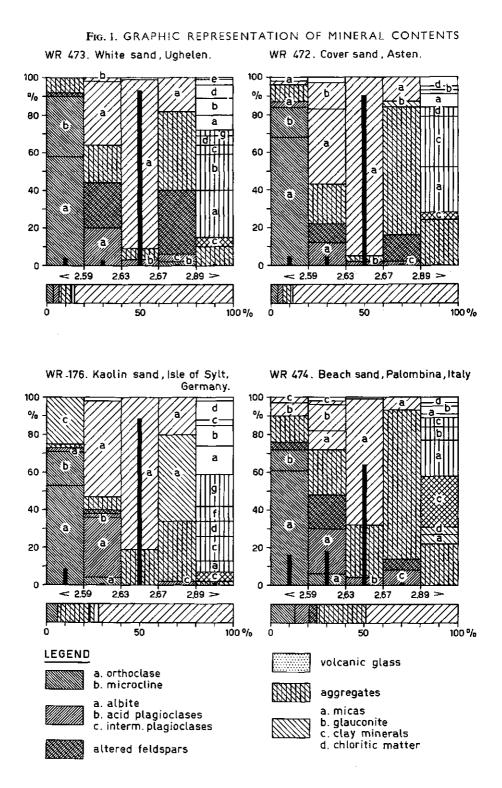
well defined cross grating structure or the presence of a simple twin coalescence. The usually tapering laminae of microcline provided the distinction from the fine twinning striations of albite. If necessary for checking purposes interference figures of the occasional grains of quartz or plagioclase could be obtained by turning them in the liquid (see also under 3i).

b. The fraction with a specific gravity of between 2.59 and 2.63, designated as *the albite fraction*, was also counted in the liquid with the refractive index of 1.528; here again to distinguish orthoclase from albite, which is here the most characteristic feldspar. Grains of minerals with refractive indices distinctly higher than 1.528 were counted together, for distinction between quartz and non-twinned oligoclase-andesine is not possible by the method of immersion. Both minerals have almost equal refractive indices. To obviate this difficulty the grains were then washed in alcohol and mounted in Canada balsam, in order to make counting by the universal stage possible. Using the method described by DOEGLAS (1940) it was then possible to distinguish single-axis minerals from biaxial ones in a relatively simple way. By simultaneously applying the Becke line method, the possibility of albite being mistaken for oligoclase-andesine could be precluded. By interpolating the quartz/oligoclase-andesine ratio so obtained in the result of the counting in the 1.528 liquid, the interrelation between the minerals and mineral aggregates was definitely determined.

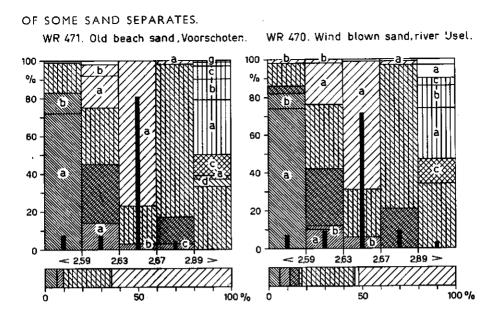
c. The fraction with a specific gravity of between 2.63 and 2.67 designated as the quartz fraction, was investigated by means of the universal stage. The simultaneous employment of the Becke line method made it also possible, if necessary, to identify two feldspar groups with refractive indices respectively lower and higher than those of the medium of immersion (about 1.54). The division by specific gravity, however, turned out so favourably that (with the acception of WR 475) apart from oligoclase-andesine no other feldspar types were found in the 2.63-2.67 fraction. On the other hand a difficulty arose in the determination of the percentage of quartz. The method described by DOEGLAS (1940) and cited above, to distinguish single-axis minerals from biaxial ones by means of the universal stage, proved unreliable in itself to determine the quartz content of a quartz-feldspar mixture. For in some samples (e.g. WR 473) an unexpectedly high percentage (more than 10 percent) of optically biaxial minerals was found, with similar refractive indices to those of quartz and oligoclase-andesine. On a closer examination many of these grains proved to contain strings of inclusions (probably of gas or a liquid), which sometimes were arranged in belts. The supposition that here indeed quartz grains were concerned (probably of epizonal origin), was confirmed by the slightly anomalous biaxial character of the interference figure; moreover the grains concerned proved to crumble to a number of particles by a relatively slight compression.

d. The fraction with a specific gravity of between 2.67 and 2.89, designated as the plagioclase feldspar fraction, was counted into a liquid with a refractive index of 1.54; if so desired (dependent upon the amount of intermediary and perhaps basic plagioclases present), a second count was made in a liquid with a refractive index of 1.560 (= about n β of labradorite); this was carried out in a similar way to that described under b.

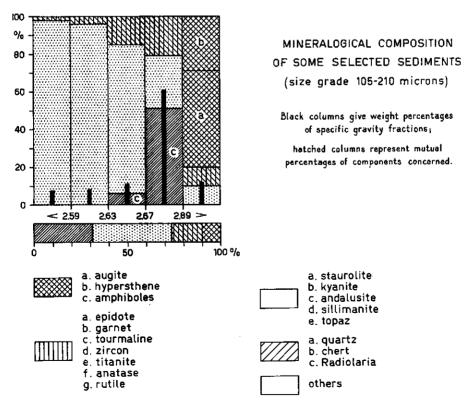
e. The fraction with a specific gravity greater than 2.89, the so-called heavy fraction, was examined according to the usual method (cf. DOEGLAS, 1940).



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WR 475. Volcanic ash, Bromo, Indonesia.



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3. The components and their occurrence in the various specific gravity fractions (cf. fig. 1)

The different components will be discussed in the order of the legend of fig. 1 (cf. Section 4).

a. Feldspars: Feldspars occur in various fractions, dependent on their specific gravity. In the fraction with a specific gravity less than 2.59 the potash-feldspars were as a rule found, especially orthoclase, and in addition microcline in minor quantities. The criteria by which both minerals were distinguished have already been given on p. 4. In the 2.59-2.63 specific gravity fraction albite is quantitatively the most important feldspar, while sometimes in addition orthoclase and microcline on the one hand, and a few oligoclase and oligoclase-andesine grains (*i.e.* the acid plagioclases) on the other were found as 'impurities'. In accordance with the specific gravity mentioned in manuals, oligoclase-andesine, if present, proved to occur especially in the quartz fraction, range 2.63-2.67. As a rule the intermediate plagioclases (with the three refractive indices distinctly higher than Canada balsam) were only observed in the specific gravity fraction 2.67-2.89. However, in sample WR 475 the intermediate plagioclases were also found in the 2.63-2.67

b. Altered feldspars: In a few samples (*i.e.* WR 470 and WR 471), especially in the specific gravity fraction 2.59–2.63, a fairly large number of altered feldspar grains were found. It is true that as mineral grains they still displayed a uniform extinction, but their surfaces proved to be densely covered with small scales of a sericite-like material. The presence of these tiny scales at the edges of the grains precluded a reliable determination of the kind of feldspar by means of the Becke line method. It seems probable that the character of these grains indicates a superficial alteration of the original feldspar.

c. Volcanic glass: This was found in the sample of the Bromo volcanic ash (WR 475), where it appeared to occur in all the specific gravity ranges.

d. Aggregates: Those grains were grouped together as aggregates which presented aggregate-polarization, i.e. no complete extinction. With the exception of chert and Radiolaria no further subdivision of this group was made at this stage, as counting did not reveal any distinct predominance of one type of aggregate.

Among other things were found rock fragments, consisting of quartz and quartzfeldspar aggregates, badly weathered grains, mostly appearing as fine-grained aggregates, and a few serpentine aggregates.

e. Micas: In the sediments investigated they only occur in minor quantities; in the Kaolin Sand of Sylt colourless micas were found in the 2.67–2.89 specific gravity fraction.

f. Glauconite: These well rounded, poorly transparent, greenish grains, occurring only in low percentages were found in the 2.67–2.89 specific gravity fraction.

g. Kaolinite: This mineral was found in the Kaolin Sand of Sylt in the fraction with a specific gravity of less than 2.59. It was faint-green in colour and the (001) cleavage was well developed. The negative axial angle was small, while $n\alpha$ and $n\beta$ had values of 1.528 and 1.564 respectively. An X-ray photograph by FAVEJEE confirmed the optical determination.

h. Heavy minerals: A full discussion of these constituents falls outside the scope of this publication.

i. Quartz: Most of samples contained a high percentage of quartz; in the Dutch sediments investigated it is quantitatively by far the most important mineral. It

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predominated in the fraction with a specific gravity range 2.63-2.67 (designated as the quartz fraction), but it was also found in minor quantities in the two adjoining specific gravity fractions, of ranges 2.59-2.63 and 2.67-2.89 respectively. In the latter fraction distinct inclusions of heavy minerals (*i.e.* zircon and sagenite) could be observed in the quartz grains concerned, whereas the quartz in the 2.59-2.63 fraction often contained minor inclusions of liquid and gas. In the fraction with a specific gravity of less than 2.59 rare grains of quartz were found. So the above deviations from the expected specific gravity range (*cf.* page 4), prove to arise mainly from the presence of impurities in the minerals.

j. Chert: Grains of micro-crystalline quartz with a refractive index of less than 1.54 and often with a conchoidal fracture were referred to this category. It was found in five of the eight sediments investigated. While this constituent occurred both in the fraction 2.59–2.63 and in that of less than 2.59, the highest percentages were found in the former.

k. Radiolaria: These occurred as spherical micro-crystalline quartz aggregates with a more or less distinct radiating organic structure; they proved to be present only in the specific gravity fraction 2.59–2.63 and in that less than 2.59.

l. Minor constituents: This group includes all components which make up less than 2% of the grains investigated.

4. DISCUSSION

The outcome of the investigation is graphically depicted in fig. 1. At first sight the legend used would seem to differ from that usually applied in sedimentarypetrology. On closer examination, however, the only difference will appear to be the grouping together of minerals or aggregates which show compositional affinity. This was especially **done** in order to attain in a simple way a broad synopsis of the primary nutritious reserves of the soil. The components of the groups have been indicated by separate letters within the shading.

The mineralogical composition of each of the sediments investigated was characterized in two ways:

a. by means of a vertical five-pillar diagram, each pillar denoting the mineral association of a given specific gravity fraction; components making up less than 2% of the assemblage of one fraction were placed in the group of minor constituents. Moreover, in each pillar the weight percentage of that specific gravity fraction has been indicated by a narrow black column.

b. by means of a horizontal strip diagram, in which the percentages of the components of the whole size grade (210–105 microns) have been plotted horizontally. So all data of the graph mentioned under a have been summarized in this column, with the understanding that components making up less than 2% of the whole sample have not been depicted separately here, but placed in the group of minor constituents. To provide a clear overall picture of the primary nutritious reserves of the soil the components of group 10 (the silica constituents) have been plotted on the right of this diagram.

All the Dutch sediments investigated display a dominance of the so-called quartz fraction (specific gravity 2.63–2.67), while the beach sand (WR 471) and especially the riversand of the Yssel (WR 470) are distinctly richer in non-siliceous components than the Asten cover-sand (WR 472) and the Ughelen White-sand (WR 473). The percentages of these components of the latter two sediments are not higher than 16%, while in WR 471 and WR 470 the non-silica percent-

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ages amount to 35% and 50% respectively. The amount of aggregates, which are partly composed of quartz aggregates but also of the more readily weathered rock fragments, is also much larger in WR 471 and WR 470. For the Dutch sediments as a whole, it may be stated that from the mineral reserve standpoint the Ughelen White-sand and the Asten cover-sand require an almost equal valuation and that the drift-sand of the river Yssel (WR 470) is the richest in composition. In addition it is important to mention that – as far as data are available – the amount of oligoclase-andesine and basic plagioclase is negligible.

The foreign sediments investigated display wide differences in composition. The Kaolin-sand of Sylt (Germany) presents a distinct dominance of the quartz fraction. In the fraction with a specific gravity of less than 2.59 kaolinite was found, while there was a comparatively large amount of mica in the 2.67–2.89 fraction. The composition of the beach sand of Italy (WR 474) displays the most complex picture. The relatively high percentages of albite and of intermediate plagioclase are especially conspicuous.

The sample from the Bromo-ash differs widely from the other sediments investigated. The top of the specific gravity curve lies not in the quartz fraction (specific gravity 2.63–2.67), but in the 2.67–2.89 fraction. So the horizontal strip diagram for the total composition of the sediment indicates a high percentage of intermediate plagioclase. Further, the percentage of heavy minerals is such that, in contrast to the other samples investigated, this group now appears in the horizontal strip diagram (augite and hypersthene together making up about 8% of the total mineralogical composition). As regards the composition of the quantitatively important volcanic glass, it may be pointed out that according to the data of VERBEEK and FENNEMA (1896) the rock of the Tengger volcanoe consists of pyroxene-andesite and basalt, and that the ashes from the active point of eruption, the Bromo, consist of basalt with a glassy matrix.

5. CONCLUSIONS

The above report is only aimed at introducing a refined method for the accurate mineralogical investigation of the light fraction of sands. The method adopted is fairly time-consuming, but it provides a good picture of both the quantitative relations between the different specific gravity fractions, and the mineralogical composition of these fractions and so of all of the sediment. An important point is that the possibility of mistaking quartz for oligoclase-andesine is practically precluded.

Furthermore, the results have given rise to the expectation that at least as far as conclusions may be drawn from the, as yet, limited data, it will be possible after an initial detailed mineralogical investigation using the method described, to distinguish different mineral associations by means of the specific gravity curve alone. Thus the method introduced here can be employed not only for projects of soil classification but also in the sedimentary petrological investigation of sediments (cf. KHADR, 1960).

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