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SELECTED METHODOLOGICAL ASPECTS OF DETERMINATION OF THE WATER DESORPTION CURVES OF SUPERABSORBENTS

Research into water retention of superabsorbents is a relatively recent phenomenon, and its methodology has not yet been fully elaborated. Our results suggest considerable water retention dynamics of superabsorbents in the range of small potentials. In the study, three methods of water retention measurements have been compared for 4 selected superabsorbents from the group of cross-linked polymers. The measurement problems faced during the analysis have been revealed such as those resulting from the specificity of the examined material, e.g. the influence of the pressure of the sorbent layer from the upper parts of the samples, as well as the difference between mechanisms of water sorption and desorption processes.

1. INTRODUCTION

Superabsorbents, also referred to as hydrogels or superabsorbent polymers (SAPs), are synthetic substances composed of loosely cross-linked water-insoluble polymers [1]. In a dry state they are usually in granulate or powder form, while after hydration they change into gel [2]. The most significant advantage of SAPs is their facility for storage, which enables them to accumulate up to a 1000-fold higher amount of water than their dry-state weight [3]. Due to their absorption properties, SAPs have found applications in numerous branches of the pharmaceutical and chemical industries [4]. They most frequently they are used however in agriculture [1], gardening [1, 5], post-industrial site reclamation [6] and in anti-erosion protection [1, 7]. Added to water, superabsorbents store part of water and then, with water utilization by plants, they release it, leading to the creation of a new equilibrium level between soil water and that bonded by SAP [8]. Determination of these equilibrium levels requires knowledge of water retention curves

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both for soil and SAP. The method for determination retention of soil water is well-established [9–12] and does not present significant difficulties. Determination of water retention of superabsorbents or their mixtures with soil material is preformed according to various methods [5, 13, 14]. Some authors combine methods employing the application of hypotension for low potential values with a centrifugation method for high potentials ones [15]. Other authors determine only maximum water volume in their studies [16, 17].

The results for water retention of SAP available in the literature indicate a high retention in the range of low values of potentials [18]. This may result in significant mistakes in water capacity assessment, even with small mistakes in the measurement of matric potential (suction pressure). These small mistakes in measurements may be the result of using samples even several centimeters high.

In order to examine this effect, as well as to estimate the influence of SAP structure on the results of measurement, determination of the water retention curves has been performed for a few selected SAPs. It was observed that the standard procedure successfully used in measurements of soil water retention using steel rings of a volume of 100 cm^3 and a height of 5 cm causes significant measurement mistakes in the case of SAP, since the water content in the upper and lower part of the cylinder differs significantly.

Thus, the aim of this study was to modify the method of determination of the water retention curves for superabsorbents in the pF range of 0–2.88 (0–78.5 kJ/m^3). The results obtained by the modified method with the use of sand and sand-kaolin blocks should be repeatable and, moreover, the results for potentials in the pF range of 2.7–2.85 (73.6–77,7 kJ/m^3) should be consistent with those obtained using Richards' apparatus.

2. MATERIALS AND METHODS

Four commercially available superabsorbents such as Aquasorb 3005 KL, Aquasorb 3005 KM, Luquasorb 1280 RL and Stockosorb 500 Micro were selected for the study. The characteristics of the examined superabsorbents are presented in Table 1.

Three methods of determination of water retention curve for superabsorbents in the potential range from 0.0 to 785 kJ/m^3 have been examined. The experiments were conducted in laboratory conditions with the application of sand blocks (used for determination of potentials in the range of 0–9.81 kJ/m^3) and sand-kaolinite ones (used for determination of potentials in the range 9.81–78.5 kJ/m^3). The water retention of SAP in the potential range 73.6–785 kJ/m^3 was determined using Richards' apparatus. The experiments were repeated five times for each superabsorbent.

The first of the applied methods was analogous to the one used for measurement of properties of soil water. Dry superabsorbents were placed in steel rings of the volume of 100 cm^3 (50 mm in diameter, 51 mm high). Each cylinder was bottom protected with a water permeable fabric, fixed using a rubber band. Superabsorbents were saturated with distilled water of the specific electrical resistance not exceeding 10 μS/cm. After the end of water sorption, any excess SAP was removed, and the cylinder with SAP saturated with water was weighed. Next, the cylinders were placed in the blocks. Measurements for potential values of: 0, 0.245, 0.981, 2.94, 6.18, 9.81 kJ/m^3 were performed in the sand block. After each pressure change, the cylinders were weighed after three days so that the potential was well established. Measurements were continued at intervals of five days using sand-kaolinite blocks for the values of potentials: 19.6, 33.8, 49.1 kJ/m^3.

Table 1

Characteristics of superabsorbents used

Superabsorbent	Chemical composition	Form	
		Dry state	Moisture state
Aquasorb 3005 KL	cross-linked copolymer of acrylamide, potassium acrylate	white granules ∅ ca. 3 mm	lumps ∅ ca. 25 mm
Aquasorb 3005 KM	cross-linked copolymer of acrylamide, potassium acrylate	white granules ∅ ca. 1mm	lumps ∅ ca. 5 mm
Luquasorb 1280 RL	cross-linked potassium polyacrylate	white granules ∅ ca. 4 mm	lumps ∅ ca. 20 mm
Stockosorb 500 Micro	cross-linked copolymer of polyacrylamide and potassium polyacrylate	white granules ∅ ca. 1 mm	lumps ∅ ca.4 mm

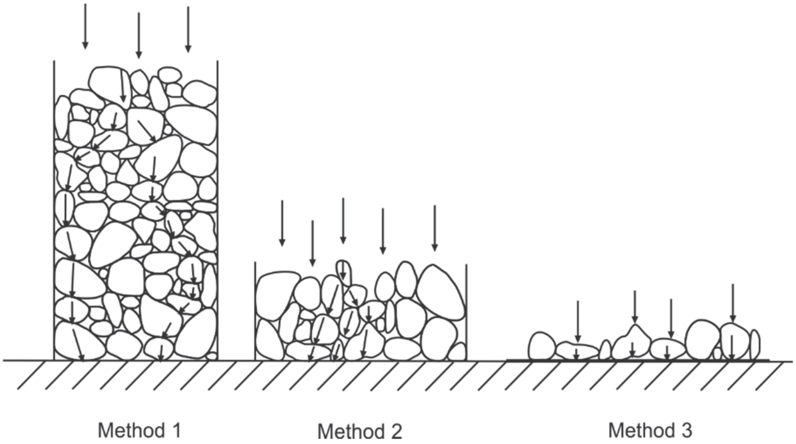

Fig. 1. Point of contacts of the superabsorbent with the block surface by various methods of determination of moisture content on sand and sand-kaolinite block

During the measurements, it was observed that saturation with water superabsorbents resulted in their breaking into mono aggregates (clumps). Hydrogel in the upper part of the cylinder had poor hydraulic contact with a block, since this happened only via the contact with adjacent gel clumps. It was therefore supposed that clumps of the sorbent in the upper part of the cylinder do not maintain hydraulic contact with the block surface and water from the upper part of the cylinder does not get into the block (Fig. 1).

By the method 2, PCV rings 7.5 cm in diameter and 2.5 cm high were used. The aim was to improve the hydraulic contact between SAP clumps from the upper part of the ring and the surface of a block. The shape and material of the rings was changed, the method itself remained the same. Although the arrangement of superabsorbents on the block has been improved, there was a problem with the material protecting the ring from beneath. Despite the attempts to push the ring precise onto the block, lack of contact between some gel clumps and the block was unavoidable. Therefore, hydraulic contact at the level of gel clumps situated above and those lying directly on the material was poor. For that reason, the next attempt at modification of the measurement method was undertaken.

In the third of the methods applied, superabsorbents saturated with water in a full saturation state were placed evenly in one layer on a qualitative drain (filtration paper), and thus all aggregates had direct contact with the block, and the hypotension acted evenly on them. Since the sorbent was placed directly on the surface of the block, it was not possible to weigh the whole sample. Therefore, a destructive measurement was used, which involved collection of the sample for weighing from a given superabsorbent with different pF values. Then, the sample was dried at 105 °C for 48 h and water content was calculated.

By all three methods, the study was conducted in a standard manner in the range of potentials of 73.6–785 kJ/m^3 using Richards' apparatus. SAP ability for swelling in external force conditions was examined using steel rings 5 cm in diameter. The rings were placed on porous plates supplied with distilled water. Porous plates assured transport of water to the sorbent. A layer of dry sorbent with hydraulic contact from beneath and with a porous plate was placed inside the ring. The sorbent was covered with an extender of a known weight until the end of the swelling process. The total gain in sorbent volume was measured using a micrometre, and related to the gain in sorbent volume swelling in conditions without external load.

Due to the changes in SAP volume during the processes of water sorption and release, its content in superabsorbents may only be expressed in parts by weight, relating the mass of absorbed water to the mass of dry SAP:

$$W = \frac{M_w - M_s}{M_s}$$

where M_s is the mass of dry SAP, while M_w is SAP mass in a given hydration state.

Comparison of selected water capacities determined using the three examined methods was performed using the *post hoc* HSD Tukey's test for mean values. Equations describing swelling under pressure were determined using the linear regression method after previous transformation of the argument (pressure values) with the application of an exp *p* function assuring linear relations between the pressure and swelling range related to free swelling.

3. RESULTS

The measured water desorption curves for 4 selected SAP are presented in Fig. 2. The measuring points for moisture content are presented with error bars, including a 95% confidence interval for the mean value of moisture content, and error bars resulting from the precision of potential measurement for sand, sand-kaolinite blocks and Richards' apparatus. Trend lines were adjusted to experimental points based on the model presented by Kaszubkiewicz [19].

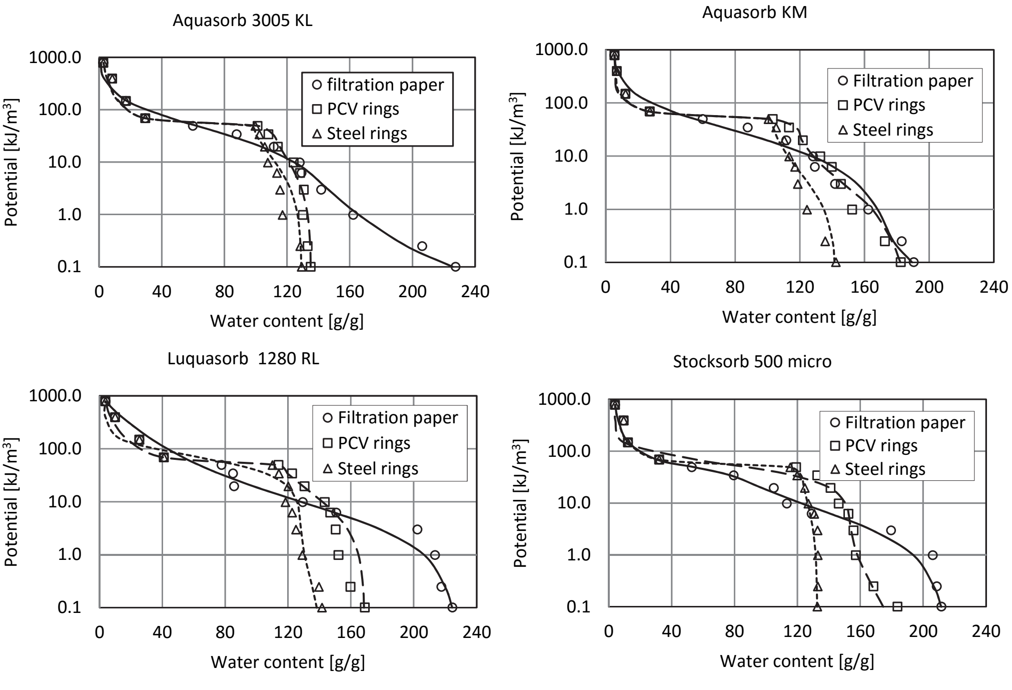

Fig. 2. Water retention curves for examined superabsorbents recorded by the methods 1 (···), 2 (---), 3 (–)

Based on the shape of the desorption curves, it was concluded that the maximum water capacity by the methods 1 and 2 is clearly lower than by the method 3, where a mono layer of sorbent grains evenly spread on filtration paper was used. Comparison with HSD Tukey's test demonstrated that for all the examined SAP, the mean value of maximum water capacity is significantly higher by the method 3 than by the method 1, and for 3 of the 4 examined SAPs it is also significantly higher than by the method 2. The difference in water capacities between the method 3 and method 1 was from 48.10 g/g to 98.39 g/g, while that for the methods 3 and 2 was lower and ranged from 8.08 g/g (insignificant) to 92.47 g/g.

In the range of potentials from 0 to 49.1 kJ/m^3, desorption curves determined using the methods 1 and 2 demonstrated a decrease in retention which was considerably smaller than that observed using method 3. This is reflected in the level of gravitational water (0–33.8 kJ/m^3) determined by the three methods. Comparison with HSD Tukey's test indicates that, in the measurement by the method 3, the amounts of gravitational water are statistically significantly higher ($p < 0.01$) for all 4 examined sorbents than by the methods 1 and 2. The difference between the values obtained by the methods 3 and 1 was from 65.49 g/g to 118.71 g/g, while the difference between those obtained by the methods 3 and 2 was lower and ranged from 33.65 g/g to 112.85 g/g.

The effect of poor retention decrease in the range of low potentials by the methods 1 and 2 was reflected in the arrangement of field water capacities (33.8 kJ/m^3). Despite the highest maximum water capacity determined using the method 3, the measured values of field water capacity are the lowest by this method for all the examined sorbents. Comparison with HSD Tukey's test demonstrates that the mean values of field water capacity in measurements using method 3 are statistically significantly lower ($p < 0.01$) for all 4 examined sorbents than by the methods 1 and 2. The difference between the values obtained by the methods 1 and 3 was from 14.83 g/g to 39.70 g/g, while the difference between those obtained by the methods 2 and 3 ranged from 20.38 g/g to 53.18 g/g.

The decrease in moisture content observed for water desorption curves determined using methods 1 and 2 in the potential range of 49.1–68.7 kJ/m^3 is reflected in the levels of readily available water related to potentials in the range of 33.8–68.7 kJ/m^3. The measured levels of readily available water determined by the method 3 are the lowest for all the examined sorbents. Comparison with HSD Tukey's test indicates that in measurements using method 3, mean levels of water readily available to plants are statistically significantly lower ($p < 0.01$) for all 4 examined sorbents than by the methods 1 and 2. Due to the fact that in the potential ranges above 2.7, the same measurements were used in both methods (using Richards' apparatus) the difference between method 1 and method 3 was the same as the differences in field water capacity and was from 14.83 g/g to 39.70 g/g, while the difference between the values obtained by the methods 2 and 3 was from 20.38 g/g to 53.18 g/g.

The levels of production water content connected to potentials in the range of 33.8–491 kJ/m^3 were analogous. Comparison with HSD Tukey's test demonstrates that in measurements using the method 3, the mean levels of available water for all 4 examined sorbents are statistically significantly lower ($p < 0.01$) than by the methods 1 and 2. The mean differences between the methods, for reasons analogous to the above, were the same as the differences in field water capacity.

A series of complementary experiments was conducted in order to evaluate the retention properties of the examined superabsorbents. Varying amounts of superabsorbents were placed in cylinders 10 cm high, and then their contact with distilled water was ensured on the upper surfaces of the cylinders. The amount of SAP in the cylinders was adjusted so that they formed columns 2.5 cm, 5 cm, 7 cm, 7.5 cm and 8.5 cm high as a result of water sorption and volume increase. Then, after equilibrium conditions were established (the end of the water sorption process), the upper sorbent layer was collected (about 0.5 cm) and its moisture content was measured gravimetrically. Thus, a fragment of the water sorption curve (unlike in the case of measurements on the blocks, where the desorption curve is determined) in the potential range from 0 to 0.834 kJ/m^3 was obtained.

The initial moisture content in cylinders or rings obtained via capillary rise is different at different heights. This effect in the soil is small due to the shape of water sorption curves, and additionally may be further decreased by pouring water onto the soil up to half of the cylinder height.

The mean water levels in cylinders, calculated based on water sorption curves in potential ranges from 0 to 0.491 kJ/m^3, and mean water levels in PCV rings, calculated based on water sorption curves in the range from 0 m^3 to 0.245 kJ/m^3, are presented in Table 2 and compared with water levels in potential 0 kJ/m^3 measured for the monoaggregate SAP layer and with mean values measured for cylinders and rings. As may be concluded, the values measured for PCV rings and steel cylinders are considerably lower than the calculated ones.

Table 2

Averaged water contents [g/g] of the PVC rings and steel cylinders calculated based on the water sorption curves, compared with the measured values

Sorbent	Measured value for the monolayer aggregate of SAP on filtration paper	Water content			
		Calculated		Measured	
		PVC rings $h = 2.5$ cm	Steel cylinders $h = 5$ cm	PVC rings $h = 2.5$ cm	Steel cylinders $h = 5$ cm
Aquasorb KL	227.7	217.1	198.8	135.23	129.3
Aquasorb KM	190.5	186.8	178.7	182.38	142.4
Luquasorb RL	224.7	221.2	218.6	168.86	141.6
Stocksorb 500 micro	211.7	210.2	208.8	183.71	132.7

A systematic source of such a difference (except for mistakes of a random character) may be a constraint in water retention as a result of pressure applied above the SAP layer. It is difficult to evaluate this effect in terms of quantity; however, preliminary examinations of water sorption conducted under the exertion of pressure indicate a considerable decrease in SAP retention properties even with relatively low pressures. The dependences of the SAP swelling ability (Aquasorb 3005 KL) on the pressure in the range corresponding to the layer pressure of sorbent fully saturated with water 0–18 cm high are presented in Fig. 3.

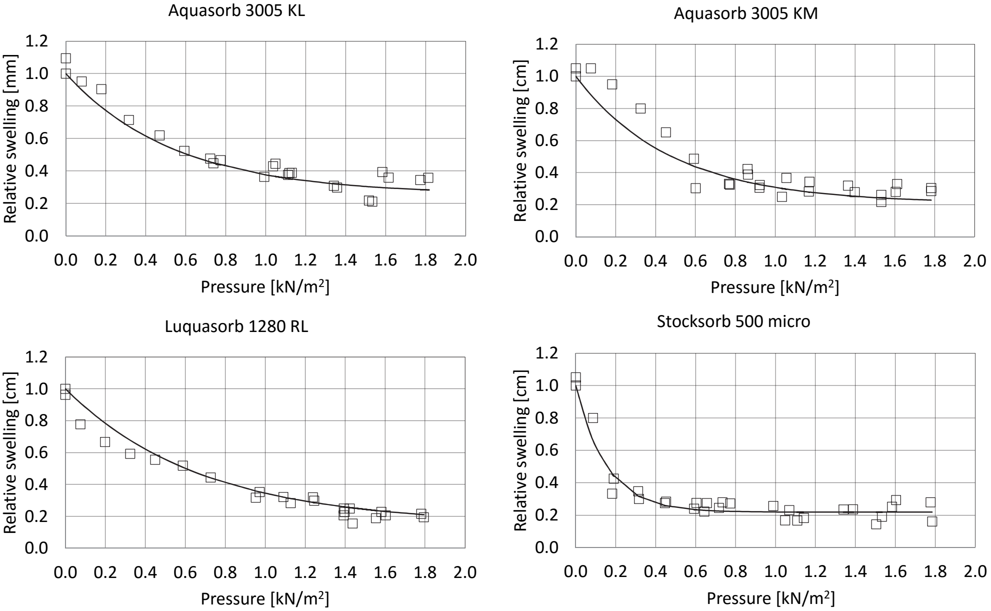

Fig. 3. Dependences of the relative swelling on pressure for the examined superabsorbents

Table 3

Relationship between the pressure and the ability to swell
and the coefficients describing the reduction of the maximum water capacity of SAP

Sorbent	Determination factor R^2	Significance factor α	Factor influencing decrease of water content	
			PCV ring 2.5 cm high	Steel cylinder 5 cm high
Aquasorb KL	0,9613	<0,001	0,83	0,70
Aquasorb KM	0,9300	<0,001	0,80	0,66
Luquasorb RL	0,9752	<0,001	0,84	0,71
Stocksorb 500 micro	0,9495	<0,001	0,54	0,38

Based on the diagrams and trend lines (Table 3), it may be estimated that in the case in question (Aquasorb KL), water content in a cylinder 5 cm high will decrease by a factor of ca. 0.71, while in the case of PCV rings 2.5 cm high water levels will be decreased by a factor of ca. 0.82 (with respect to the levels to be obtained in the case of a mono layer). Both discussed effects acting jointly result in the maximum water levels determined for cylinders 5 cm high and for PCV rings 2.5 cm high being considerably lower than the maximum water levels in a mono aggregate layer (Tables 2, 3). The possible synergy of their activity should be remembered.

4. DISCUSSION

The examined methods of measurement of water retention for SAP give diverse results, especially in the range of low suction forces. Measurements of maximum water capacity performed using a thin layer of SAP spread on filtration paper gave results in the range from 190 g/g to 227 g/g, which does not differ from the values reported in the literature [20] for cross-linked polyacrylates (200–400 g/g), and is also close to the value of 219 g/g reported by Hayat and Safdar [21], for Aquasorb polymer obtained using measurements in a water bath. Concurrently, water available for plants ranged between 33% and 43% of maximum water content, being by 25% higher than those reported by Green et al. [18].

Comparison of the examined measurement methods with SAP layers (in saturated state) of 5 cm, 2.5 cm and about 0.5 cm thick indicates that the obtained water capacity, and especially the maximum water capacity, differ depending on thickness of the layer. Most authors omit this measurement problem when performing the examinations for soil and SAP mixtures [22, 23]. Superabsorbents mixed with soil remain in an even contact with water irrespective of the depth they lie on, and the degree of their saturation is not dependent so strongly on the shape of the retention curve in the range of small potentials, as in our samples. In the present study conducted for pure sorbents, the differences between the results obtained using three proposed methods were statistically significant. Higher values for maximum water capacity and gravitational water levels, with concurrently lower levels of water available for plants and field water capacity, were obtained by the method applying a mono-aggregate sorbent layer.

The study also indicates the problem of the influence of the depth of SAPs localization on their ability to swell and absorb water. Swelling ability decreases upon increasing pressure, which in the case of application to the soil may originate from the layer situated above. The relative swelling decrease for the 4 examined SAP may be described by the following equation:

$$\frac{v}{v_0} = \alpha\left(\beta + \mathrm{e}^{-\gamma p}\right)$$

where: v is the volume obtained by the swelling superabsorbent under pressure, v_0 is its volume obtained without pressure, α, β and γ are the coefficients determined using linear regression, p is value of the pressure.

A 50% decrease in swelling ability for the examined SAPs was observed as soon as pressures at levels from 0.16 kN/m^2 for Stocksorb 500 Micro to 0.61 kN/m^2 for Aquasorb KL were applied. This corresponds to the pressure of the soil layer of a thickness of several centimetres. The effect of water access to the sorbent, blocking as a result of swelling in limited capacity conditions, also has some significance in terms of the limitation of swelling [23].

The results of water sorption in the range of small sucking forces indicate the need for further research on the effect of the water hysteresis of superabsorbents [3], which may be of significant in the assessment of their influence on the water properties of soils enriched with SAP. When the relatively strong influence of the salinity of the absorbed solution on retention ability of SAP is added to the above measurement difficulties [25], the degree of complexity of determination of their actual water capacities in soil may be noted.

5. CONCLUSIONS

Based on our study and an analysis of the results obtained, it was observed that standard methods of water retention curve measurement using steel rings of a height of 5 cm cannot be used in the case of superabsorbents, due to the poor hydraulic contact between SAP aggregates. This results in a small decrease in water content in SAP, which is observed with an increased matric potential. As a result, measurement performed for SAP placed in steel rings ($h = 5$ cm) or in PCV rings ($h = 2.5$ cm) provides overestimated values for field water capacity, which also leads to overestimated amounts of water available for plants.

The high scope of the water retention curve in the range of small suckling forces and large differences between the humidification and drying curves observed for SAPs generate considerable errors in attempts to measure the maximum water capacity for SAP samples placed in rings 2.5 and 5.0 cm high. The level of gravitational water is also underestimated.

A source of measurement errors in the range of small potentials may also be the high sensitivity of SAP water retention to pressure. As was demonstrated, the pressure of a SAP layer up to 5 cm thick may limit water retention.

The recommended methods for water retention of SAP analysis should include the placing of a mono-aggregate layer on filtration paper onto the surface of sand or sand--kaolinite block, and the measurement of gravitational water content after the state of potential equilibrium has been established. This method ensures repeatable results, and in the range of higher potentials is consistent with those obtained using Richards' apparatus.

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