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台灣醫療暨生技器材工業同業公會 收文 107.03.02 字號：107135

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主旨：請就本局編擬之CNS草-制1070166「尿失禁用尿液吸收輔具－高分子基吸收材料特徵化之試驗法－第1部：pH測定」等4種國家標準草案惠提意見，如無意見亦請在空白意見書上註明無意見，並請於107年5月3日前惠復（或電子郵件寄jerrycr.wu@bsmi.gov.tw）本局第一組第四科吳啟瑞。

說明：檢附上開草案暨空白意見書各1份 。

正本：方委員毓廷、王委員舜民、李委員淑貞、胡委員景灝、馬委員海霞、常委員挽瀾、張委員力山、張委員日聖、曾委員明基、葉委員旭輝、蓋委員惠珍、趙委員福杉、鄭委員宗記、賴委員柏樺、韓委員德生、衛生福利部、衛生福利部食品藥物管理署、衛生福利部社會及家庭署多功能輔具資源整合推廣中心、衛生福利部社會及家庭署、中華民國老人福利推動聯盟、中華民國老人福祉協會、中華民國消費者文教基金會、中華民國脊髓損傷者聯合會、台北市美國商會醫療器材委員會、台北榮民總醫院身障重建中心、台灣障礙者權益促進會、台灣醫療暨生技器材工業同業公會、社團法人中華民國身心障礙聯盟、財團法人自行車暨健康科技工業研究發展中心、財團法人金屬工業研究發展中心(北區)、經濟部標準檢驗局第三組、經濟部標準檢驗局第六組

副本：

經濟部標準檢驗局第一組

CNS 草-制 1070166「尿失禁用尿液吸收輔具－高分子基吸收材料特徵化之試驗法－第 1 部：pH 測定」等 4 種國家標準草案審查意見書

此致　　經濟部標準檢驗局　第一組第四科吳啟瑞　　共　頁第　頁

草案編號	節　次	審　查　意　見
	編修說明： 為因應全球高齡化趨勢，落實國內支撐使用者之個人衛生輔具產品市場相關檢驗法標準化之目標，以期保障消費者權益，促進國內支撐使用者之個人衛生輔具產業之發展。	

審查委員或單位簽名（請務必填寫）：

文號：經標一字第 1071000282 號、107 年 5 月 3 日截止（請不必備文，本表若不敷使用，請自行影印）、倘需各標準草案或意見書空白表電子檔，請以 E-mail(jerrycr.wu@bsmi.gov.tw)來函索取。

標準檢驗局承辦人：吳啟瑞

ICS 11.180.20

中華民國國家標準

CNS

尿失禁用尿液吸收輔具-高分子基吸收材料特徵化之試驗法-第1部：pH測定

Urine-absorbing aids for incontinence -Test methods for characterizing polymer-based absorbent materials - Part 1: Determination of pH

CNS 草-制 1070166:2018
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前言

本標準係依據2001年發行之第 1 版 ISO 17190-1，不變更技術內容，制定成為中華民國國家標準者。

本標準係依標準法之規定，經國家標準審查委員會審定，由主管機關公佈之中華民國國家標準。

依標準法第四條之規定，國家標準採自願性方式實施。但經各該目的事業主管機關引用全部或部分內容為法規者，從其規定。

本標準並未建議所有安全事項，使用本標準前應適當建立相關維護安全與健康作業，並且遵守相關法規之規定。

本標準之部分內容，可能涉及專利權、商標權與著作權，主管機關及標準專責機關不負責任何或所有此類專利權、商標權與著作權之鑑別。

1. 適用範圍

本標準規定聚丙烯酸酯(PA)超吸收劑粉末pH值測定法。

本方法已試驗pH 5.92至pH 6.14(參照附錄A)，但預期可適用於更寬之pH值範圍。

2. 引用標準

下列標準因本標準所引用，成為本標準之一部分。下列引用標準適用最新版(包括補充增修)。

ISO 187 Paper, board and pulps - Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples

ISO 3696 Water for analytical laboratory use - Specification and test methods

ISO 5725-2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

3. 原理

使用pH計及標準化玻璃pH響應電極測定加入PA超吸收劑粉末之生理食鹽水溶液pH。

4. 試劑

警語：濃縮酸及鹼應小心處理。應穿著安全防護包括手套及面罩，濃鹽酸應在通風櫃中處理。

除另有規定外，僅使用已認定分析級之試劑。

4.1 水

符合ISO 3696。

4.2 氫氧化鈉溶液c(NaOH) = 0.1 mol/L

秤重，至最接近之0.1 g，將4 g氫氧化鈉置入1 L燒瓶(5.8)內，加入去離子水(1級，參照4.1)至標記，攪拌至溶解。

4.3 鹽酸溶液c(HCl) = 0.1 mol/L

量測容積，至最接近之0.1 mL，加8.9 mL濃鹽酸至1 L燒瓶(5.8)內，加入去離子水(1級，參照4.1)至標記，攪拌至溶解。

4.4 氯化鈉c(NaCl) = 0.9 % 質量百分比

秤重，至最接近之0.1 g，將9 g氯化鈉至1 L燒瓶(5.8)內，加入去離子水(3級，參照4.1)至標記，攪拌至溶解。使用氫氧化鈉溶液(4.2)或鹽酸溶液(4.3)調整pH值至6.0 ± 0.05。

4.5 標準緩衝溶液

具下述pH值之標準緩衝溶液。

(a) 4.0 ± 0.02。

(b) 7.0 ± 0.02。

(c) 10.0 ± 0.02。

5. 儀器

5.1 分析天平

可量測至最接近之0.0001 g，最大量測質量可至0.5000 g。

5.2 分析天平

可量測最接近之至0.1 g，最大量測質量可至9.0 g。

5.3 pH計

5.4 玻璃pH響應電極(本標準中稱為pH電極)

5.5 磁力攪拌器及磁棒

磁棒30 mm × 6 mm圓柱或相當物。

5.6 玻璃燒杯

容量250 mL。

5.7 玻璃量筒

容量100 mL。

5.8 容量瓶

A級，容量1 L。

6. 取樣

注意：當處理樣品超過10 g時，應使用呼吸防護系統、防塵口罩或通風櫃。

為確保代表性取樣，從一大袋或筒倉卡車取樣時，應移除表層(大約20 cm)。用勺子取樣後3 min內將其置入適當大小之氣密容器中。

保持樣品於封閉容器中，自容器取出進行試驗前放在實驗室環境溫度中以使其平衡，試驗條件最好在溫度(23 ± 2)℃及相對濕度(50 ± 10) %，若此條件不適用，應報告試驗環境條件之溫濕度。實驗室條件量測參照ISO 187。

在進行試驗樣品尚未自容器取出前，在容器內先旋轉3至5次以獲得較均勻樣品。讓容器放置5 min，然後打開蓋子並取出部分樣品進行試驗。

確保試驗樣品在進行試驗前，基本上沒有直徑大於1mm之團塊。

7. 程序

7.1 依製造商提供之操作說明書，使用pH4.0及pH 7.0標準緩衝溶液[分別為4.5(a)及4.5(b)]校正pH電極。

7.2 加0.9 %氯化鈉溶液(4.4)100 mL至250 mL燒杯中，將燒杯置於磁力攪拌器(5.5)，選用中等混合速率以避免空氣進入溶液中。

7.3 秤重至最接近0.01 g，從PA超吸收粉末試驗樣品不同二個位置處，各取0.5 g樣品。每一樣品依7.4至7.8執行試驗。

7.4 將PA超吸收粉末置入氯化鈉溶液中，以中速攪拌10 min使其分散。

7.5 使用去離子水瀝洗pH計，使用軟吸收衛生紙小心擦乾電極。

7.6 攪拌完成後，靜置1 min後，將pH計浸入待測溶液澄液層(supernatant layer)中，並測定pH值。

7.7 記錄pH值至小數點下一位。

7.8 使用去離子水瀝洗pH計。

8. 精密度

EDANA於1997年利用此方法於不同實驗室間進行試驗比對，本方法數據之重複性(repeatability)及再現性(reproducibility)提供於附錄A。

依ISO 5725-2重複試驗條件下，2次個別試驗結果間之絕對差異重複性限值γ不應超過5 %。

$\gamma = 0.14$ pH單位

依ISO 5725-2再現試驗條件下，2次個別試驗結果間之絕對差異再現性限值R不應超過5 %。

$R = 0.58$ pH單位

若無法滿足再現性及重複性標準，則應再進行2次試驗。複製試驗前，應確保原始待測樣品已充分混合。若還是無法滿足標準，則不能使用試驗結果，並應分析誤差來源如驗證儀器正確之操作程序及利用已知值之材料進行驗證。

9. 試驗驗報告

試驗驗報告應包含下述資訊。

(a) 試驗機構名稱及地址。

(b) 高分子基吸收材料種類，包括用於取樣完整鑑別所需之所有技術細節及來源資訊。

(c) 本標準CNS XXX-1。

(d) 樣品中是否出現團塊。

(e) 每次試驗(7.7)之試驗結果，pH值以最接近之0.1表示之，及平均值作重複判定。

(f) 判定過程中任何不常見現象，或若不符合重複性及/或再現性標準(第8節)。

(g) 任何程序之差異或任何選用之操作。

附錄 A
(參考)
實驗室間試驗之統計結果

EDANA於1997年利用此方法於不同實驗室間進行試驗比對。實驗室依ISO 5725-2執行試驗之結果分析如下。

樣品編號	A	B	C
參與實驗室數量	10	10	10
試驗結果可接受之實驗室數量(排除結果異常之結果)	10	10	10
可接受試驗結果之數量	40	40	40
平均值(pH)	5.92	6.09	6.14
重複性標準差(S_r)	004	0.03	0.05
變異重複性係數	0.63 %	0.42 %	0.82 %
重複性限(r)(2.8× S_r)	0.10	0.07	0.14
再現性標準差(S_R)	0.21	0.17	0.18
變異再現性係數	3.51 %	2.78 %	2.95 %
再現性限(R)(2.8× S_R)	0.58	0.47	0.51

相對應國際標準

ISO 17190-1 : 2001 Urine-absorbing aids for incontinence -Test methods for characterizing polymer-based absorbent materials - Part 1 : Determination of pH

ICS 11.180.20

中華民國國家標準

CNS

尿失禁用尿液吸收輔具-高分子基吸收材料特徵化之試驗法-第2部：殘留單體量之測定

Urine-absorbing aids for incontinence -Test methods for characterizing polymer-based absorbent materials - Part 2: Determination of amount of residual monomers

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前言

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依標準法第四條之規定，國家標準採自願性方式實施。但經各該目的事業主管機關引用全部或部分內容為法規者，從其規定。

本標準並未建議所有安全事項，使用本標準前應適當建立相關維護安全與健康作業，並且遵守相關法規之規定。

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1. 適用範圍

本標準規定測定存在於聚丙烯酸酯超吸收粉末中之丙烯酸的殘餘單體丙烯酸鈉與丙烯酸總和量之方法。

2. 引用標準

下列標準因本標準所引用，成為本標準之一部分。下列引用標準適用最新版(包括補充增修)。

ISO 187 Paper board and pulps - Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples

ISO 3696 Water for analytical laboratory use - Specification and test methods

ISO 5725-2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

3. 用語及定義

下列用語及定義適用於本標準。

3.1 殘留單體量

殘留單體丙烯酸鈉與丙烯酸之總和。

4. 原理

自PA超吸收粉末萃取出之殘留丙烯酸，且丙烯酸殘留量由HPLC測定。

5. 試劑

5.1 一般

除另有規定外，僅使用已認定分析級之試劑。

5.1.1 水

符合ISO 3696。

5.1.2 氯化鈉溶液c(NaCl) = 0.9 % (質量百分比)

秤重，至最接近之0.1 g，將9 g氯化鈉至1 L燒瓶(6.5)內，加入去離子水(3級，參照5.1.1)至標記，攪拌至溶解。

5.1.3 磷酸

HPLC或更佳等級之濃度$c(H_3PO_4)$ = 85 %(質量百分比)

5.1.4 磷酸溶液

$c(H_3PO_4)$ = 0.1 %(質量百分比)(1 g/L或0.0087 mol/L)，使用去離子水(3級，參照5.1.1) 稀釋磷酸(5.1.3)攪拌混合。

5.1.5 乙腈(Acetonitrile)

HPLC或更佳等級。

5.1.6 丙烯酸(Acrylic acid)

純度大於99.5 %。

丙烯酸會隨時間降解，因此，重要的是測量用於校正HPLC的丙烯酸純度。

5.2 校正用標準溶液

5.2.1 S1溶液(ρ_{cal}(S1)=1,000 mg/L)

秤重，至最接近之0.0005 g，0.1000 g丙烯酸(5.1.6)置入標示為S1之100 mL容量瓶(6.5)。

用超純水(5.1.1)補足至標記。使用此溶液，製備5.2.2至5.2.6稀釋液。

5.2.2 S2溶液(ρ_{cal}(S2)=100 mg/L)

使用吸管自S1吸取10 mL置入標示為S2之100 mL容量瓶(6.5)，用超純水(5.1.1)補足至標記。

5.2.3 S3溶液(ρ_{cal}(S3)=1 mg/L)

使用吸管自S1吸取1 mL置入標示為S3之100 mL容量瓶(6.5)，用超純水(5.1.1)補足至標記。

5.2.4 S4溶液(ρ_{cal}(S4)=2 mg/L)

使用吸管自S1吸取2 mL置入標示為S4之100 mL容量瓶(6.5)，用超純水(5.1.1)補足至標記。

5.2.5 S5溶液(ρ_{cal}(S5)=100 mg/L)

使用吸管自S1吸取3 mL置入標示為S5之100 mL容量瓶(6.5)，用超純水(5.1.1)補足至標記。

5.2.6 S6溶液(ρ_{cal}(S6)=4 mg/L)

使用吸管自S1吸取4 mL置入標示為S6之100 mL容量瓶(6.5)，用超純水(5.1.1)補足至標記。

6. 儀器

6.1 分析天平

可量測至最接近之0.0001 g，最大量測質量可至0.1g。

6.2 分析天平

可量測至至最接近之0.001 g，最大量測質量可至1.0 g。

6.3 玻璃燒杯或錐形瓶

容量250 mL。

6.4 量筒

容量200 mL，準確度± 0.5 %。

6.5 容量瓶

A級，容量100 mL及1 L。

6.6 吸管

A級，容量1 mL，2 mL，3 mL，4 mL及10 mL。

6.7 磁力攪拌器

攪拌速率(500 ± 50) r/min及攪拌棒。

6.8 過濾器

0.45 μm。

6.9 HPLC注射系統

注射分析物溶液體積從20 μL至100 μL及準確度在± 1 %。

6.10 HPLC幫浦

能輸送具理論背壓在± 10 %內之流量。

6.11 C18柱(C18 column)

具粒度5 μm之包裝材料，長250 mm，內徑(ID)為4.6 mm，配有保護柱(6.12)。

6.12 C18保護柱(guard column)

具粒度5 μm之包裝材料，長50 mm，內徑(ID)為4.6 mm。

6.13 HPLC之UV偵測器

波長為210 nm。

7. 取樣

注意：當處理樣品超過10 g時，應使用呼吸防護系統、防塵口罩或通風櫃。

7.1 待測樣品

為確保代表性取樣，從一大袋或筒倉卡車取樣時，應移除表層(大約20 cm)。用勺子取樣後3 min內將其置入適當大小之氣密容器中。

保持樣品於封閉容器中，試驗自容器取出進行試驗前放在實驗室環境溫度中以使其平衡，試驗條件最好在溫度(23 ± 2)℃及相對濕度(50 ± 10) %，若此條件不適用，應報告試驗環境條件之溫濕度。實驗室條件量測參照ISO 187。

在進行試驗樣品尚未自容器取出前，在容器內先旋轉3 ~5次以獲得較均勻樣品。讓容器放置5 min，然後打開蓋子並取出部分樣品進行試驗。

確保試驗樣品在進行試驗前，基本上沒有直徑大於1mm之團塊。

7.2 樣品準備

7.2.1 秤重，秤至最接近0.005 g，秤取PA超吸收粉末試驗樣品1,000 g，置入秤舟(weighting boat)中，並記錄試驗樣品之質量(m_{sam})。

7.2.2 將試驗樣品置入乾淨燒杯(6.3)或錐形瓶(6.3)中，並確認所有樣品均已移入。

7.2.3 使用量筒量取0.9 % 200 mL氯化鈉溶液(5.1.2)，加入燒杯或錐形瓶中。

7.2.4 加入磁力攪拌棒，然後用石蠟膜密封燒杯或塞住燒瓶。

7.2.5 使用(500 ± 50) rpm，攪拌60 min。

7.2.6 時間到停止攪拌。

7.2.7 樣品靜置5 min。

7.2.8 利用0.45 μm過濾器過濾溶液，並作為HPLC分析之用。

8. 程序

8.1 HPLC色譜條件

使用下述色譜條件。

注入：20 μL至100 μL。

流動相(mobil phase)：[10 %乙腈(5.1.5)]：[磷酸溶液(5.1.4)]

流速：1 mL/min。

分析柱：C18柱(6.1.1)

偵測：210 nm UV(6.13)。

峰面積使用積分器、數據站或計算機獲得。在此條件下，丙烯酸通常在5 min至6 min內洗脫。

替代方法及色譜條件參照附錄A。

8.2 校正

標準溶液S3(5.2.3)至S6(5.2.6)每一種重複分析2次。平均2次個別獲得(A_i)之峰面積。

若重複分析之重複峰面積間之差異超過平均值之5 %時，重複進行分析。若差異小於5 %，則使用重複測定平均值作為校正曲線。

8.3 測定

重複分析7.2.8試驗樣品2次，平均所得之峰面積。

9. 校正

9.1 校正曲線

繪製丙烯酸質量濃度$\rho_{cal,i}$對平均峰面積A_i之曲線，作為校正曲線。

假設此關係為線性，決定質量濃度ρ對平均峰面積A之線性回歸直線方程式如下。

$$\rho_{cal} = aA + b \qquad (1)$$

式中

ρ_{cal}：分析物質量濃度(mg/L)。

A：分析物峰面積。

a：直線斜率。

b：與y軸(ρ_{cal})交點。

決定(1)回歸直線之修正係數，修正係數應大於0.99。

9.2 殘留單體量

使用(1)式計算取自試驗樣品(8.3)萃取溶液(ρ_{sam})之質量濃度，作為9.1之校正曲線。式中A_{sam}為樣品之峰面積。

PA超吸收粉末中殘留單體量為測定殘留丙烯酸量之質量比率(w_{acr})，依下式計算，以mg/L表示。

$$w_{acr} = \rho_{sam}\frac{200}{m_{sam}} \qquad (2)$$

式中

ρ_{sam}：萃取溶液質量濃度(mg/L)。

m_{sam}：取自試驗樣品之高分子質量(g)。

200：萃取溶液體積(mL)。

試驗結果以最接近之1 mg/kg表示。

10. 精密度

EDANA於1997年利用此方法於不同實驗室間進行試驗比對，本方法數據之重複性(repeatability)及再現性(reproducibility)提供於附錄B。

依ISO 5725-2重複試驗條件下，2次個別試驗結果間之絕對差異重複性限值γ不應超過5 %。

$\gamma = 55$ mg/kg

依ISO 5725-2再現試驗條件下，2次個別試驗結果間之絕對差異再現性限值R不應超過5 %。

$R = 416$ mg/kg

若無法滿足再現性及重複性標準，則應再進行2次試驗。複製試驗前，應確保原始待測樣品已充分混合。若還是無法滿足標準，則不能使用試驗結果，並應分析誤差來源如驗證儀器正確之操作程序及利用已知值之材料進行驗證。

若個別峰及定量之精度及準確度等於或優於上述所列，則來自給定色譜程序之偏差是可接受的。來自色譜程序之偏差範例參照附錄A。

11. 試驗報告

試驗報告應包含下述資訊。

(a) 試驗機構名稱及地址。

(b) 高分子基吸收材料種類，包括用於取樣完整鑑別所需之所有技術細節及來源資訊。

(c) 本標準CNS XXX-2。

(d) 每次殘留單體量試驗(9.2)結果以殘留丙烯酸酯單體於PA之質量比率(mg/kg)表示及重複測定之平均值。

(e) 判定過程中任何不常見現象或若不符合重複性及/或再現性標準(第10節)。

(f) 任何程序之差異或任何選用之操作。

附錄 A
(參考)
替代方法及色譜條件

A.1 Neyer色譜法(Neyer chromatographic method)

本標準規定之程序從J. M. Neyer等人發表文獻[1]修改而得。

Neyer方法之線性區間為0.01 mg/L至10 mg/L，相當於在PA超吸收粉末內殘留單體2 mg/kg到2,000 mg/kg。此法偵測極限為0.01 mg/L之丙烯酸。

A.2 替代色譜條件

A.2.1 來自色譜法之可接受偏差

若峰分離及定量之精密度及準確度等於或優於第10節所述，則與給定色譜方法之偏差是可接受的，例：

- 可以使用任何等效的反相色譜柱。
- 若使用高純度、pH穩定之反相柱，則可忽略移動相有機修飾劑。範例參見圖A.1為在沒有有機修飾劑情況下獲得之分離。

注意，在移動相中，在缺乏有機修飾劑狀況下，C18相易產生“親水性塌縮(hydrophilic collapse)”現象。此結果可能造成滯留時間(retention time)飄移。在此環境中應驗證柱性能。

- 可使用離子排阻色譜柱代替建議作為移動相具0.005 mol /L硫酸之反相色譜柱。
- 只要可使分析物分離，可使用不同尺寸之色譜柱。參照圖A.1，其中色譜柱於A.2.2中規定。
- 可在1 h攪拌時間結束時加入過濾助劑，如1 mL之H_3PO_4。

A.2.2 替代HPLC系統

下述系統為一替代HPLC系統，其顯示於圖A.1。

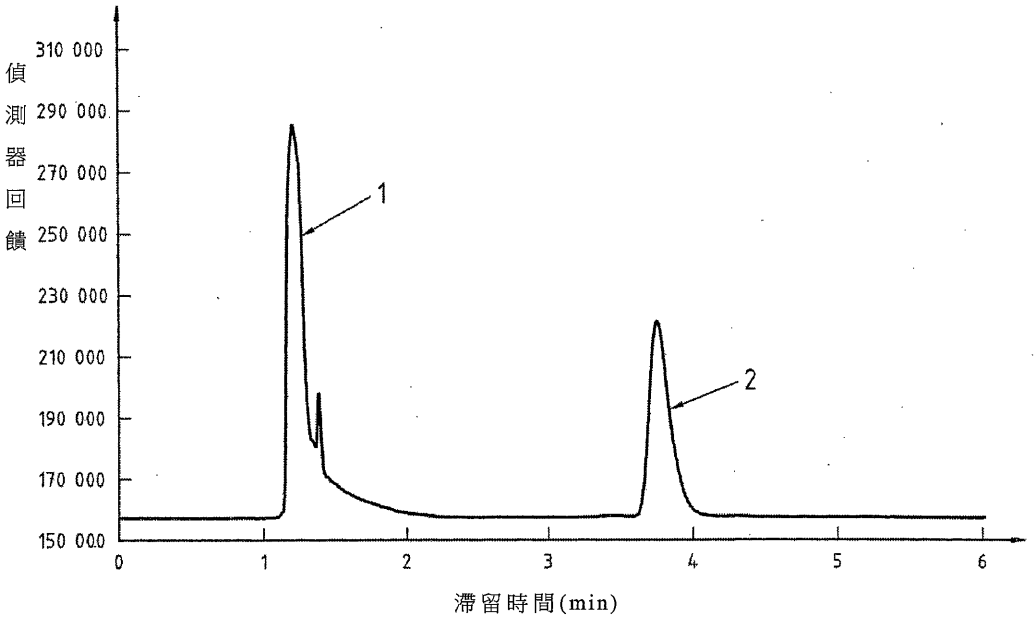
流動相(體積)：[10 %乙腈(5.1.5)]：[磷酸溶液(5.1.4)]

流率：1 mL/min。

分析柱：C18，5 μm粒度包裝材料，長100 mm，內徑4.6 mm。

保護柱：C18(6.12)

偵測：210 nm UV(6.13)。



說明

1 柱滯留體積

2 丙烯酸

圖 A.1 使用替代 HPLC 系統之殘留丙烯酸色譜

附錄 B
(參考)
實驗室間試驗之統計結果

用於循環試驗的樣本主要被選擇用於試驗方法之準確度。它們不代表市場上的產品(例樣品A來自過去生產的批號)。

EDANA於1997年利用此方法於不同實驗室間進行試驗比對。實驗室依ISO 5725-2執行試驗之結果分析如下。

樣品編號	A	B	C
參與實驗室數量	10	10	10
試驗結果可接受之實驗室數量(排除結果異常之結果)	9	9	9
可接受試驗結果之數量	36	36	36
平均值(mg/kg)	716	356	400
重複性標準差(S_r)	18.58	13.95	19.52
變異重複性係數	2.60 %	3.92 %	4.88 %
重複性限(r)(2.8× S_r)	52.03	39.07	54.66
再現性標準差(S_R)	148.51	83.98	84.76
變異再現性係數	20.77 %	23.58 %	21.19 %
再現性限(R)(2.8× S_R)	415.83	235.15	237.32

參考資料

[1] NEYER J.M., VIGOUROUX A., VAMVAKARIS C. and MANDERY H. High-Performance Liquid Chromatographic Determination of Monomeric Sodium Acrylate and Acrylic Acid in Polyacrylic Gelling Agents. Chromatographia, 25 (10) 1988

相對應國際標準

ISO 17190-2 : 2001 Urine-absorbing aids for incontinence - Test methods for characterizing polymer-based absorbent materials – Part : 2 Determination of amount of residual monomers

ICS 11.180.20

中華民國國家標準

CNS

尿失禁用尿液吸收輔具-高分子基吸收材料特徵化之試驗法-第 3 部：利用篩選分離法測定粒度分布

Urine-absorbing aids for incontinence -Test methods for characterizing polymer-based absorbent materials - Part 3: Determination of particle size distribution by sieve fractionation

CNS 草-制 1070168:2018
T5

中華民國　年　月　日制定公布
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本標準非經經濟部標準檢驗局同意不得翻印

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前言

本標準係依據2001年發行之第 1 版 ISO 17190-3，不變更技術內容，制定成為中華民國國家標準者。

本標準係依標準法之規定，經國家標準審查委員會審定，由主管機關公布之中華民國國家標準。

依標準法第四條之規定，國家標準採自願性方式實施。但經各該目的事業主管機關引用全部或部分內容為法規者，從其規定。

本標準並未建議所有安全事項，使用本標準前應適當建立相關維護安全與健康作業，並且遵守相關法規之規定。

本標準之部分內容，可能涉及專利權、商標權與著作權，主管機關及標準專責機關不負責任何或所有此類專利權、商標權與著作權之鑑別。

1. 適用範圍

本標準規定交聯(cross-linked)聚丙烯酸酯(polyacrylate)超吸收粉末粒徑分布45 μm至850 μm之量測法。

一般，此方法預定用於溫度15℃至35℃可自由流動之高分子超吸收材料粉末。

2. 引用標準

下列標準因本標準所引用，成為本標準之一部分。下列引用標準適用最新版(包括補充增修)。

ISO 5725-2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

3. 原理

將規定量的超吸收劑粉末在通過一系列標準篩網時分成特定的粒度分率。每一分率稱重，且其值作為材料總量的百分比報告。

4. 儀器

4.1 分析天平

可量測至最接近之0.01 g，最大量測質量可至300 g。

4.2 燒杯

玻璃或塑膠，容量150 mL。

4.3 篩網振動器(sieve shaker)

型式Retsch VE 1000或等同，可維持5個直徑200 mm具底盤之標準篩網，應接地避免靜電。

4.4 標準篩網

直徑200 mm不銹鋼篩網，孔隙大小為45 μm、150 μm、300 μm、600 μm及800 μm，下方具底盤，上方具蓋子。

4.5 刷子

如駱駝頭髮製，可清潔標準篩網。

5. 取樣

注意：當處理樣品超過10 g時，應使用呼吸防護系統，防塵口罩或通風櫃。

為確保代表性取樣，從一大袋或筒倉卡車取樣時，應移除表層(大約20 cm)。用勺子取樣500 g後3 min內將其置入1 L之氣密容器中。

保持樣品於封閉容器中，試驗自容器取出進行試驗前放在實驗室環境溫度中以使其平衡，試驗條件最好在溫度(23 ± 2)℃及相對濕度(50 ± 10) %，若此條件不適用，應報告試驗環境條件之溫濕度。實驗室條件量測參照ISO 187。

在進行試驗樣品尚未自容器取出前，在容器內先旋轉3至5次以獲得較均勻樣品。

6. 程序

6.1 確認篩網(4.4)為乾燥，對這光源檢查是否損壞及乾淨，更換破損篩網。利用刷子

(4.5)移除殘留顆粒。

6.2 將底盤(4.3)及每一個空篩網秤重，秤至最接近之0.01 g，並記錄其質量m_s。

6.3 將篩選以正確順序置入振動器中(即最細的在最下面，最粗的在最上面)。

6.4 秤取100 g超吸收粉末試驗樣品，至最接近之0.01 g，並記錄其質量m_1，置入燒杯(4.2)中。

6.5 將稱重之樣品定量轉移到篩網試驗機上之頂篩。

6.6 蓋上蓋子，並依製造商說明書固定之。

6.7 確認設備已接地，避免靜電發生。

6.8 依下述設定振動器。

- 強度：(70 ± 2) %(使用Retsch VE 1000振動器)。
- 振幅：1.0 mm。
- 振動時間：10 min。

6.9 開始振動，經10 min後，小心移除並秤取每一篩網及底盤重量(m_2)至最接近之0.01 g。

7. 計算

依下式計算每一分率百分比(w)。

$$w=\frac{m_2-m_s}{m_1}\times 100$$

式中

m_2：篩網加殘留吸收高分子(6.9)的質量(g)。

m_s：空篩網(6.2)質量(g)。

m_1：樣品(6.4)質量(g)。

8. 精密度

EDANA於1997年利用此方法於不同實驗室間進行試驗比對，本方法數據之重複性(repeatability)及再現性(reproducibility)提供於附錄A。

依ISO 5725-2重複試驗條件下，2次個別試驗結果間之絕對差異重複性限值γ不應超過5 %。

γ = 0.09 %

依ISO 5725-2再現試驗條件下，2次個別試驗結果間之絕對差異再現性限值R不應超過5 %。

R = 0.20 %

若無法滿足再現性及重複性標準，則應再進行2次試驗。複製試驗前，應確保原始待測樣品已充分混合。若還是無法滿足標準，則不能使用試驗結果，並應分析誤差來源如驗證儀器正確之操作程序及利用已知值之材料進行驗證。

9. 試驗報告

試驗報告應包含下述資訊。

(a) 試驗機構名稱及地址。

(b) 高分子基吸收材料種類，包括用於取樣完整鑑別所需之所有技術細節及來源資訊。

(c) 本標準CNS XXX-3。

(d) 振動篩模式。

(e) 每個粒度級分的結果保留在每個試驗部分的篩網及底盤上，以已秤重材料之質量百分比，最接近的0.1 %，及重複測定之平均值表示。

(f) 判定過程中任何不常見現象或若不符合重複性及/或再現性標準(第8節)。

(g) 任何程序之差異或任何選用之操作。

附錄 A
(參考)
實驗室間試驗結果統計

EDANA於1997年利用此方法於不同實驗室間進行試驗比對，該方法的重複性和重現性的數據是合作研究的結果。實驗室依ISO 5725-2執行試驗之結果分析如表A.1所示。

表A.1 粒度分布結果統計

說明	於盤內收集分率			於 45 μm 篩網分率			於 150 μm 篩網分率		
樣品編號	A	B	C	A	B	C	A	B	C
參與實驗室數量	10	10	10	10	10	10	10	10	10
試驗結果可接受之實驗室數量(排除結果異常之結果)	9	9	9	9	8	9	9	9	9
可接受試驗結果之數量	36	36	34	36	32	35	36	36	35
平均值(mg/kg)	0.05	0.07	0.03	2.39	5.24	0.58	25.2	17.4	28.8
重複性標準差(S_r)	0.02	0.03	0.01	0.34	0.46	0.08	1.92	0.99	1.52
變異重複性係數	43.70	40.90	28.30	14.40	8.70	13.80	7.63	5.69	5.30
重複性限(r)(2.8× S_r)	0.06	0.09	0.02	0.97	1.28	0.22	5.39	2.77	4.27
再現性標準差(S_R)	0.07	0.07	0.07	1.82	0.91	0.54	4.08	2.95	3.87
變異再現性係數	152	98	278	76	17	92	16	17	13
再現性限(R)(2.8× S_R)	0.20	0.20	0.21	5.10	2.55	1.51	11.3	8.25	10.8
說明	於 300 μm 篩網分率			於 600 μm 篩網分率			於 850 μm 篩網分率		
樣品編號	A	B	C	A	B	C	A	B	C
參與實驗室數量	10	10	10	10	10	10	10	10	10
試驗結果可接受之實驗室數量(排除結果異常之結果)	9	9	9	9	9	9	9	8	9
可接受試驗結果之數量	36	36	35	36	36	35	36	30	35
平均值(mg/kg)	56.40	58.8	57.9	15.7	18.5	12.9	0.09	0.10	0.05
重複性標準差(S_r)	1.11	0.88	1.26	1.44	0.84	1.05	0.03	0.01	0.02
變異重複性係數	1.97	1.49	2.18	9.15	4.54	8.12	34.90	11.60	35.50
重複性限(r)(2.8× S_r)	3.11	2.46	3.54	4.02	2.35	2.93	0.09	0.03	0.05
再現性標準差(S_R)	3.52	4.10	3.82	2.62	4.86	1.69	0.08	0.02	0.07
變異再現性係數	6.24	6.98	6.59	16.70	26.30	13.10	90.70	22.30	135
再現性限(R)(2.8× S_R)	9.85	11.5	10.7	7.34	13.6	4.73	0.2	0.06	0.20

相對應國際標準

ISO 17190-3 : 2001 Urine-absorbing aids for incontinence - Test methods for characterizing polymer-based absorbent materials - Part 3 : Determination of particle size distribution by sieve fractionation

ICS 11.180.20

中華民國國家標準

CNS

尿失禁用尿液吸收輔具-高分子基吸收材料特徵化之試驗法-第4部：加熱質量損失法測定水分含量

Urine-absorbing aids for incontinence -Test methods for characterizing polymer-based absorbent materials - Part 4: Determination of moisture content by mass loss upon heating

CNS 草-制 1070169:2018

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前言

本標準係依據2001年發行之第 1 版 ISO 17190-4，不變更技術內容，制定成為中華民國國家標準者。

本標準係依標準法之規定，經國家標準審查委員會審定，由主管機關公布之中華民國國家標準。

依標準法第四條之規定，國家標準採自願性方式實施。但經各該目的事業主管機關引用全部或部分內容為法規者，從其規定。

本標準並未建議所有安全事項，使用本標準前應適當建立相關維護安全與健康作業，並且遵守相關法規之規定。

本標準之部分內容，可能涉及專利權、商標權與著作權，主管機關及標準專責機關不負責任何或所有此類專利權、商標權與著作權之鑑別。

1. 適用範圍

本標準規定水分含量0至5 %質量之交聯(cross-linked)聚丙烯酸酯(polyacrylate)超吸收粉末的加熱質量損失試驗水分含量之方法。準確度± 0.1 %。

一般，此方法預定用於溫度15℃至150℃可自由流動之高分子超吸收材料粉末。

在此溫度範圍內揮發除水以外的物質會干擾試驗結果。

2. 引用標準

下列標準因本標準所引用，成為本標準之一部分。下列引用標準適用最新版(包括補充增修)。

ISO 5725-2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

3. 原理

本程序測定在電加熱乾燥爐中在試驗樣品脫水時之質量損失，在大氣壓力下，(105 ± 2)℃保持3 h。

4. 儀器

4.1 分析天平

可秤重至最接近之0.001 g，最大量測質量可至至300 g。

4.2 盤子

玻璃或鋁，具有移除之蓋子，其底面表面積約為50 cm^2。

4.3 烤箱

恆溫，可維持在(105 ± 2)℃。

4.4 乾燥器

活性乾燥劑(如矽膠)。

4.5 抹刀

V形，可取1 g之PA超吸收粉末。

5. 取樣

注意：當處理樣品超過10 g時，應使用呼吸防護系統、防塵口罩或通風櫃。

為確保代表性取樣，從一大袋或筒倉卡車取樣時，應移除表層(大約20 cm)。用勺子取樣後3 min內將其置入適當大小之氣密容器中。

在進行試驗樣品尚未自容器取出前，在容器內先旋轉3至5次以獲得較均勻樣品。

在打開蓋子取樣品前，允許容器靜置5 min。

6. 程序

6.1 將盤子與蓋子(4.2)一起置入105 ℃烤箱(4.3)中3 h，在烤箱時，將蓋子自盤子上移開，加熱快結束時結束蓋上蓋子，並移至乾燥器(4.4)中。蓋盤冷卻30 min至室溫。

6.2 將空蓋盤秤重，秤至最接近之0.01 g，並記錄此質量為m_1。

6.3 移開蓋子，利用V型抹刀(4.5)，加大約4.0 g經良好混合具代表性之PA超吸收粉末

樣品，應無塊狀樣品。

6.4 蓋上蓋子，快速秤取含樣品蓋盤之質量至最接近0.001 g，並記錄此質量為m_2。

6.5 如使用刮勺及緩攻絲(gentle tapping)將樣品均勻分布至整個盤子底部。

6.6 打開蓋子，跟蓋子一起置入105 ℃烤箱(4.3)中3 h。

6.7 在移出烤箱前，快速蓋上蓋子，移出烤箱置入乾燥器中冷卻30 min。

6.8 含樣品之蓋碗冷卻至室溫後，移出乾燥器，快速秤重至最接近之0.001 g，記錄此質量為m_3。

6.9 相同分析方式，相繼或同時使用混合良好之實驗室樣品至少執行2次。

7. 計算

7.1 依(1)式計算試驗樣品內之濕氣含量每一分率百分比(w_m)。

$$w_m = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad (1)$$

式中

m_1：乾空蓋碟(6.2)質量(g)。

m_2：乾空蓋碟與未乾燥試驗樣品(6.4)質量(g)。

m_3：乾空蓋碟與乾燥後試驗樣品(6.8)質量(g)。

7.2 利用(1)式計算試驗樣品水分含量(w_m)。

8. 精密度

EDANA於1997年利用此方法於不同實驗室間進行試驗比對，本方法數據之重複性(repeatability)及再現性(reproducibility)提供於附錄A。

依ISO 5725-2重複試驗條件下，2次個別試驗結果間之絕對差異重複性限值γ不應超過5 %。

γ = 0.44 %濕度

依ISO 5725-2再現試驗條件下，2次個別試驗結果間之絕對差異再現性限值R不應超過5 %。

R = 1.80 %濕度

若無法滿足再現性及重複性標準，則應再進行2次試驗。複製試驗前，應確保原始待測樣品已充分混合。若還是無法滿足標準，則不能使用試驗結果，並應分析誤差來源如驗證儀器正確之操作程序及利用已知值之材料進行驗證。

9. 試驗報告

試驗報告應包含下述資訊。

(a) 試驗機構名稱及地址。

(b) 高分子基吸收材料種類，包括用於取樣完整鑑別所需之所有技術細節及來源資訊。

(c) 本標準CNS XXX-4。

(d) 每次試驗(7.1)質量損失水分含量結果，以質量分率百分比表示，以最接近之0.1表示之，及平均值作重複判定。

(e) 判定過程中任何不常見現象或若不符合重複性及/或再現性標準(第8節)。

(f) 任何程序之差異或任何選用之操作。

附錄 A
(參考)
實驗室間試驗之統計結果

EDANA於1997年利用此方法於不同實驗室間進行試驗比對。實驗室依ISO 5725-2執行試驗之結果分析如下。

樣品編號	A	B	C
參與實驗室數量	10	10	10
試驗結果可接受之實驗室數量(排除結果異常之結果)	10	10	10
可接受試驗結果之數量	39	39	40
平均值(%)	0.62	0.53	3.96
重複性標準差(S_r)	0.09	0.07	0.16
變異重複性係數	15.1 %	12.6 %	4.0 %
重複性限(r)(2.8× S_r)	0.26	0.19	0.44
再現性標準差(S_R)	0.13	0.27	0.64
變異再現性係數	20.7 %	51.4 %	16.2 %
再現性限(R)(2.8× S_R)	0.36	0.77	1.8

相對應國際標準

ISO 17190-4 : 2001 Urine-absorbing aids for incontinence - Test methods for characterizing polymer-based absorbent materials - Part 4 : Determination of moisture content by mass loss upon heating

INTERNATIONAL STANDARD

ISO 17190-1

First edition
2001-12-01

Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 1: Determination of pH

Aides pour absorption d'urine — Méthodes d'essai pour caractériser les matériaux absorbants à base de polymères —

Partie 1: Détermination du pH

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 17190 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 17190-1 was prepared by Technical Committee ISO/TC 173, *Technical systems and aids for disabled or handicapped persons*, Subcommittee SC 3, *Aids for ostomy and incontinence*.

ISO 17190 consists of the following parts, under the general title *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials*:

— *Part 1: Determination of pH*

— *Part 2: Determination of amount of residual monomers*

— *Part 3: Determination of particle size distribution by sieve fractionation*

— *Part 4: Determination of moisture content by mass loss upon heating*

— *Part 5: Gravimetric determination of free swell capacity in saline solution*

— *Part 6: Gravimetric determination of fluid retention capacity in saline solution after centrifugation*

— *Part 7: Gravimetric determination of absorption under pressure*

— *Part 8: Gravimetric determination of flowrate*

— *Part 9: Gravimetric determination of density*

— *Part 10: Determination of extractable polymer content by potentiometric titration*

— *Part 11: Determination of content of respirable particles*

ISO 17190 is intended to be used in conjunction with ISO 17191, *Urine-absorbing aids for incontinence — Airborne polyacrylate superabsorbent material in the workplace — Determination of the content in respirable dust by sodium atomic absorption spectrometry*.

Annex A of this part of ISO 17190 is given for information only.

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Introduction

ISO 17190 consists of a series of test methods originally developed by *European Disposables and Nonwovens Association (EDANA)*. These test methods have been incorporated without technical changes into one International Standard consisting of eleven parts.

These test methods have been in practical use for several years, and have proven to be reliable with respect to common criteria of quality of test methods (validity, repeatability, etc.). They are applicable to polyacrylate superabsorbent materials, which occur in hygiene products, including urine-absorbing aids for incontinent persons. The test methods are addressed to the *material* exclusively. They are not intended to be used, and are not applicable for use with finished manufactured urine-absorbing aids.

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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 1: Determination of pH

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1 Scope

This part of ISO 17190 specifies a method for determining the pH of polyacrylate (PA) superabsorbent powders.

This method has been tested in the range of pH 5,92 to pH 6,14 (see annex A), but it is expected to be applicable to a wider range.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 17190. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 17190 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Principle

The pH of the saline solution to which PA superabsorbent powders are added is determined using a pH meter and a standardized glass pH-responsive electrode.

4 Reagents

WARNING — Concentrated acids and bases shall be handled with care. Safety protection, including gloves and face shields, shall be worn. Concentrated hydrochloric acid shall be handled under a fume hood.

Use only reagents of recognized analytical grade, unless otherwise specified.

4.1 Water, complying with ISO 3696.

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4.2 Sodium hydroxide solution, c(NaOH) = 0,1 mol/l.

Weigh, to the nearest 0,1 g, 4 g sodium hydroxide into a 1 l volumetric flask (5.8) and make up to the mark with deionized water (grade 1, see 4.1). Stir until dissolved.

4.3 Hydrochloric acid solution, c(HCl) = 0,1 mol/l.

Add, to the nearest 0,1 ml, 8,9 ml concentrated hydrochloric acid to a 1 l volumetric flask (5.8) and make up to the mark with deionized water (grade 1, see 4.1). Stir until dissolved.

4.4 Sodium chloride solution, c(NaCl) = 0,9 % by mass.

Weigh, to the nearest 0,1 g, 9 g of sodium chloride into a 1 l volumetric flask (5.8) and make up to the mark with deionized water (grade 3, see 4.1). Stir until dissolved. Adjust the pH of the saline solution to 6,0 ± 0,05 using sodium hydroxide (4.2) or hydrochloric acid (4.3) solution.

4.5 Standard buffer solutions, having the following pH:

a) 4,0 ± 0,02

b) 7,0 ± 0,02

c) 10,0 ± 0,02

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5 Apparatus

5.1 Analytical balance, capable of weighing, to the nearest 0,000 1 g, masses up to 0,500 0 g.

5.2 Analytical balance, capable of weighing, to the nearest 0,1 g, masses up to 9 g.

5.3 pH meter.

5.4 Glass pH-responsive electrode (referred to in the text as pH electrode).

5.5 Magnetic stirrer and **stirring bar,** having a cylindrical form, 30 mm × 6 mm or equivalent.

5.6 Glass beaker, of 250 ml capacity.

5.7 Glass graduated cylinder, of 100 ml capacity.

5.8 Volumetric flask, Grade A of 1 l capacity.

6 Sampling

CAUTION — Use respiratory protection, dust mask or fume hood, when handling sample amounts greater than 10 g.

In order to guarantee that a representative sample is taken from the bulk material contained in a large bag or a silo truck, remove the top layer (approximately 20 cm). Take the test sample with a scoop. Place it in an airtight container of adequate size within 3 min after sampling.

Keep the test samples in a closed container and allow them to equilibrate to the ambient laboratory temperature before removing a test portion to run the test. The preferred test conditions are (23 ± 2) °C and (50 ± 10) % relative humidity. If these conditions are not available, test at ambient conditions and report the temperature and relative humidity. Measure these laboratory conditions in accordance with ISO 187.

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Before taking a test portion out of the container to run the test, rotate the container three to five times so as to obtain a homogeneous product. Allow the container to sit 5 min before opening the lid and removing the test portion.

Make sure the test portion is substantially free of lumps of size greater than 1 mm in diameter before proceeding with testing.

7 Procedure

7.1 Calibrate the pH electrode (5.4) at pH 4,0 and pH 7,0 using the standard buffer solutions [respectively, 4.5 a) and 4.5 b)] in accordance with the operating instructions provided by the manufacturer.

7.2 Add 100 ml of 0,9% saline solution (4.4) to a 250 ml beaker (5.6). Place the beaker on a magnetic stirrer (5.5). Choose a moderate mixing speed so as to avoid air from being drawn into the solution.

7.3 Weigh, to the nearest 0,01 g, two 0,5 g test portions from the PA superabsorbent powder test sample. Perform the procedure given in 7.4 to 7.8 on each portion.

7.4 Disperse the test portion of PA superabsorbent powder into the saline solution and mix the suspension at moderate speed for 10 min.

7.5 Rinse the pH electrode with deionized water. Carefully blot the electrode dry with a soft, absorbent tissue.

7.6 One minute after the stirring has stopped, immerse the pH electrode in the supernatant layer of the test solution and measure the pH.

7.7 Record the pH to one decimal place.

7.8 Rinse the pH electrode with deionized water.

8 Precision

The data for the repeatability and reproducibility limits of this method are the result of interlaboratory tests carried out in 1997 by EDANA and are given in annex A.

The absolute difference between two single test results obtained under repeatability test conditions in accordance with ISO 5725-2 shall not exceed the repeatability limit r in more than 5 % of cases:

r = 0,14 pH units

The absolute difference between two single test results obtained under reproducibility test conditions in accordance with ISO 5725-2 shall not exceed the reproducibility limit R in more than 5 % of cases:

R = 0,58 pH units

If the repeatability and reproducibility test criteria are not met, the test shall be repeated twice, each in duplicate, after ensuring that the original sample is thoroughly mixed. If it fails to satisfy this criterion, report as unusual, then diagnose the source of error for example by verifying correct operation of the instruments, and testing a portion of a material with a known value.

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9 Test report

The test report shall include the following information:

a) the name and address of the testing institution;

b) the type of polymer-based absorbent materials, including all technical details and source information required for the complete identification of the sample;

c) a reference to this part of ISO 17190, i.e. ISO 17190-1;

d) the presence or absence of lumps in the sample;

e) the results of pH for each test (7.7), expressed in pH units to the nearest 0,1 unit, and the average value for duplicate determinations;

f) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 8);

g) any deviations from the procedure or any operations regarded as optional.

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Annex A
(informative)

Statistical results of interlaboratory tests

Figures for the repeatability and reproducibility of this method are the result of collaborative studies carried out in 1997 by EDANA. The evaluation of the interlaboratory test was carried out in accordance with ISO 5725-2 and results as follows:

Sample identification	A	B	C
Number of participating laboratories	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	10	10	10
Number of accepted test results	40	40	40
Mean value (pH units)	5,92	6,09	6,14
Repeatability standard deviation (s_r)	0,04	0,03	0,05
Repeatability coefficient of variation	0,63 %	0,42 %	0,82 %
Repeatability limit (r) ($2{,}8 \times s_r$)	0,10	0,07	0,14
Reproducibility standard deviation of reproducibility (s_R)	0,21	0,17	0,18
Reproducibility coefficient of variation	3,51 %	2,78 %	2,95 %
Reproducibility limit (R) ($2{,}8 \times s_R$)	0,58	0,47	0,51

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INTERNATIONAL STANDARD

ISO 17190-2

First edition
2001-12-01

Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 2: Determination of amount of residual monomers

Aides pour absorption d'urine — Méthodes d'essai pour caractériser les matériaux absorbants à base de polymères —

Partie 2: Détermination de la quantité de monomères résiduels

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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International Standard ISO 17190-2 was prepared by Technical Committee ISO/TC 173, *Technical systems and aids for disabled or handicapped persons*, Subcommittee SC 3, *Aids for ostomy and incontinence*.

ISO 17190 consists of the following parts, under the general title *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials*:

— *Part 1: Determination of pH*

— *Part 2: Determination of amount of residual monomers*

— *Part 3: Determination of particle size distribution by sieve fractionation*

— *Part 4: Determination of moisture content by mass loss upon heating*

— *Part 5: Gravimetric determination of free swell capacity in saline solution*

— *Part 6: Gravimetric determination of fluid retention capacity in saline solution after centrifugation*

— *Part 7: Gravimetric determination of absorption under pressure*

— *Part 8: Gravimetric determination of flowrate*

— *Part 9: Gravimetric determination of density*

— *Part 10: Determination of extractable polymer content by potentiometric titration*

— *Part 11: Determination of content of respirable particles*

ISO 17190 is intended to be used in conjunction with ISO 17191, *Urine-absorbing aids for incontinence — Airborne polyacrylate superabsorbent material in the workplace — Determination of the content in respirable dust by sodium atomic absorption spectrometry*.

Annexes A and B of this part of ISO 17190 are given for information only.

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Introduction

ISO 17190 consists of a series of test methods originally developed by *European Disposables and Nonwovens Association (EDANA)*. These test methods have been incorporated without technical changes into one International Standard consisting of eleven parts.

These test methods have been in practical use for several years, and have proven to be reliable with respect to common criteria of quality of test methods (validity, repeatability, etc.). They are applicable to polyacrylate superabsorbent materials, which occur in hygiene products, including urine-absorbing aids for incontinent persons. The test methods are addressed to the *material* exclusively. They are not intended to be used, and are not applicable for use with finished manufactured urine-absorbing aids.

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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 2: Determination of amount of residual monomers

1 Scope

This part of ISO 17190 specifies a method for determining the sum of residual monomeric sodium acrylate and acrylic acid present in polyacrylate (PA) superabsorbent powders as acrylic acid.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 17190. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 17190 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Term and definition

For the purposes of this part of ISO 17190, the following term and definition applies.

3.1
amount of residual monomers
sum of residual monomeric sodium acrylate and acrylic acid

4 Principle

Residual acrylic acid is extracted from the PA superabsorbent powders and amount of residual acrylic acid is determined by HPLC.

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5 Reagents

5.1 General

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1.1 Water, complying with ISO 3696.

5.1.2 Sodium chloride solution, c(NaCl) = 0,9 % by mass.

Weigh, to the nearest 0,1 g, 9 g of sodium chloride into a 1 l volumetric flask (6.5) and make up to the mark with deionized water (grade 3, see 5.1.1). Stir until dissolved.

5.1.3 Phosphoric acid, concentrated $c(H_3PO_4)$ = 85 % by mass, of HPLC grade or better.

5.1.4 Phosphoric acid solution, $c(H_3PO_4)$ = 0,1 % by mass (1 g/l or 0,008 7 mol/l). Dilute concentrated H_3PO_4 (5.1.3) to volume with deionized water (grade 3, see 5.1.1). Stir until mixed.

5.1.5 Acetonitrile, of HPLC grade or better.

5.1.6 Acrylic acid, of > 99,5 % purity.

It is well known that acrylic acid degrades over time. It is therefore important to measure the purity of acrylic acid used to calibrate the HPLC.

5.2 Standard solutions for calibration

5.2.1 Solution S1 [ρ_{cal}(S1) = 1 000 mg/l].

Weigh, to the nearest 0,000 5 g, 0,100 0 g acrylic acid (5.1.6) into the 100 ml volumetric flask (6.5) labelled S1. Make up to the mark with ultrapure water (5.1.1). Using this solution, prepare the dilutions given in 5.2.2 to 5.2.6.

5.2.2 Solution S2 [ρ_{cal}(S2) = 100 mg/l].

Pipette 10 ml from S1 into the 100 ml volumetric flask (6.5) labelled S2 and make up to the mark with ultrapure water (5.1.1)

5.2.3 Solution S3 [ρ_{cal}(S3) = 1 mg/l].

Pipette 1 ml from S1 into the 100 ml volumetric flask (6.5) labelled S3 and make up to the mark with ultrapure water (5.1.1)

5.2.4 Solution S4 [ρ_{cal}(S4) = 2 mg/l].

Pipette 2 ml from S1 into the 100 ml volumetric flask (6.5) labelled S4 and make up to the mark with ultrapure water (5.1.1).

5.2.5 Solution S5 [ρ_{cal}(S5) = 3 mg/l].

Pipette 3 ml from S1 into the 100 ml volumetric flask (6.5) labelled S5 and make up to the mark with ultrapure water (5.1.1).

5.2.6 Solution S6 [ρ_{cal}(S6) = 4 mg/l].

Pipette 4 ml from S1 into the 100 ml volumetric flask (6.5) labelled S6 and make up to the mark with ultrapure water (5.1.1).

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6 Apparatus

6.1 Analytical balance, capable of weighing, to the nearest 0,000 1 g, masses up to 0,1 g.

6.2 Analytical balance, capable of weighing, to the nearest 0,001 g, masses up to 1,0 g.

6.3 Glass beaker or **conical flasks**, of 250 ml capacity.

6.4 Graduated cylinder, of 200 ml capacity and accurate to ± 0,5 %.

6.5 Volumetric flasks, Grade A, of 100 ml and 1 l capacities.

6.6 Volumetric pipettes, Grade A, of 1 ml, 2 ml, 3 ml, 4 ml and 10 ml capacities.

6.7 Magnetic stirrer, capable of stirring at a rate of (500 ± 50) r/min, with **stirring bars**.

6.8 Filters, 0,45 µm.

6.9 HPLC injection system, for injection volumes ranging from 20 µl to 100 µl, of analyte solution and accurate to within ± 1 %.

6.10 HPLC pump, capable of delivering flows with a the theoretical back-pressure to within ± 10 %.

6.11 C18 column, with 5-µm particle-size packing material, 250 mm length × 4,6 mm internal diameter (ID), fitted with a guard column (6.12).

6.12 Guard column, C18, with 5-µm particle-size packing material, 50 mm length × 4,6 mm internal diameter (ID).

6.13 UV detector for HPLC, capable of making measurements at a wavelength of 210 nm.

7 Sampling

CAUTION — Use respiratory protection, dust mask or fume hood, when handling sample amounts greater than 10 g.

7.1 Test sample

In order to guarantee that a representative sample is taken from the bulk material contained in a large bag or a silo truck, remove the top layer (approximately 20 cm). Take the test sample with a scoop. Place it in an airtight container of adequate size within 3 min after sampling.

Keep the test samples in a closed container and allow them to equilibrate to the ambient laboratory temperature before removing a test portion to run the test. The preferred test conditions are (23 ± 2) °C and (50 ± 10) % relative humidity. If these conditions are not available, test at ambient conditions and report the temperature and relative humidity. Measure these laboratory conditions in accordance with ISO 187.

Before taking a test portion out of the container to run the test, rotate the container three to five times so as to obtain a homogeneous product. Allow the container to sit 5 min before opening the lid and removing the test portion.

Make sure the test portion is substantially free of lumps of size greater than 1 mm in diameter before proceeding with testing.

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7.2 Sample preparation

7.2.1 Weigh, to the nearest 0,005 g, a 1,000 g test portion of PA superabsorbent powder test sample into a clean weighing boat and record the mass (m_{sam}) as the test portion.

7.2.2 Add the test portion to a clean beaker (6.3) or conical flask (6.3) and make sure that all of the sample has been transferred.

7.2.3 Measure 200 ml of 0,9 % saline solution (5.1.2) using the measuring cylinder and add it to the beaker or flask.

7.2.4 Add a magnetic stirrer bar then seal the beaker with paraffin film or stopper the flask.

7.2.5 Stir the solution at (500 ± 50) r/min for 60 min.

7.2.6 After 60 min, stop the stirring.

7.2.7 Allow the polymer to settle for 5 min.

7.2.8 Filter the supernatant through a 0,45 µm filter and keep this for HPLC analysis.

8 Procedure

8.1 HPLC chromatographic conditions

Use the following HPLC chromatographic conditions:

Injection: 20 µl to 100 µl

Mobile phase (by volume): [10 % acetonitrile (5.1.5)]:[90 % phosphoric acid solution (5.1.4)]

Flow rate: 1 ml/min

Analytical column: C18 column (6.11)

Guard column: C18 (6.12)

Detection: UV at 210 nm (6.13)

The peak areas are obtained using an integrator, data station, or computer. Under these conditions, acrylic acid usually elutes within 5 min to 6 min.

Alternative methods and chromatographic conditions are listed in annex A.

8.2 Calibration

Analyse the standard solutions S3 (5.2.3) to S6 (5.2.6) in duplicate. Average the peak area obtained for each level (A_i).

If the difference between the duplicate peak areas of duplicate analyses exceeds 5 % of the average, repeat the determination in duplicate. If this difference is less than 5 %, use the average value of the duplicate determinations for the calibration curve.

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8.3 Determination

Analyse the test portion obtained in 7.2.8 in duplicate. Average the peak area obtained.

9 Calculation

9.1 Calibration curve

Plot the acrylic acid mass concentration, $\rho_{cal,i}$, against the average peak area A_i to obtain the calibration curve. Assuming this relationship is linear, determine the equation of the line of regression of mass concentration ρ on the average peak area A as follows:

$$\rho_{cal} = aA + b \tag{1}$$

where

ρ_{cal} is the mass concentration, expressed in milligrams per litre, of analyte;

A is the peak area of analyte;

a is the slope of the line;

b is the intercept on the y-axis (ρ_{cal}).

Determine the correlation coefficient for the line of regression obtained for equation (1). This correlation coefficient should be greater than 0,99.

9.2 Amount of residual monomers

Calculate the mass concentration of the extraction solution ρ_{sam} taken from the test portion (8.3) using equation (1) obtained for the calibration curve in 9.1, where A_{sam} is the peak area obtained for the sample.

The amount of residual monomers in PA superabsorbent powders is the mass fraction of residual acrylic acid determined (w_{acr}), expressed in milligrams per kilogram, and is calculated as follows:

$$w_{acr} = \rho_{sam} \frac{200}{m_{sam}} \tag{2}$$

where

ρ_{sam} is the mass concentration, expressed in milligrams per litre, of the extraction solution;

m_{sam} is the mass, expressed in grams, of polymer taken for the test portion;

200 is the volume, expressed in millilitres, of extraction solution.

Report the results to the nearest 1 mg/kg.

10 Precision

The data for the repeatability and reproducibility limit of this method are the result of interlaboratory tests carried out in 1997 by the EDANA, and are given in annex B.

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The absolute difference between two single test results obtained under repeatability test conditions in accordance with ISO 5725-2 shall not exceed the repeatability limit r in more than 5 % of cases:

r = 55 mg/kg

The absolute difference between two single test results obtained under reproducibility test conditions in accordance with ISO 5725-2 shall not exceed the reproducibility limit R in more than 5 % of cases:

R = 416 mg/kg

The high reproducibility limit stems probably from calibration problems due to ageing of acrylic acid (see 5.1.6).

If the repeatability and reproducibility test criteria are not met, the test shall be repeated twice, each in duplicate, after ensuring that the original sample is thoroughly mixed. If it fails to satisfy this criterion, report as unusual, then diagnose the source of error for example by verifying correct operation of the instruments, and testing a portion of a material with a known value.

Deviations from the given chromatographic procedure are acceptable if the level of precision and accuracy in both peak separation and quantitation are equal to, or better than those listed above. Some examples of deviations from the chromatographic procedure are listed in annex A.

11 Test report

The test report shall include the following information:

a) the name and address of the testing institution;

b) the type of polymer-based absorbent materials, including all technical details and source information required for the complete identification of the sample;

c) a reference to this part of ISO 17190, i.e. ISO 17190-2;

d) the results of the amount of residual monomers for each test (9.2), expressed as mass fraction of residual acrylate monomer in PA (mg/kg), and the average for duplicate determinations;

e) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 10);

f) any deviations from the procedure or any operations regarded as optional.

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Annex A
(informative)

Alternative methods and chromatographic conditions

A.1 Neyer chromatographic method

The procedure specified in this part of ISO 17190 is a modified version of that published by J.M. Neyer *et al* (see reference [1] in the Bibliography).

The linear range of the Neyer method is 0,01 mg/l to 10 mg/l acrylic acid, equivalent to 2 mg/kg to 2 000 mg/kg of residual monomers in PA superabsorbent powders. The limit of detection of this method is 0,01 mg/l of acrylic acid.

A.2 Alternative chromatographic conditions

A.2.1 Acceptable deviations from the chromatographic method

Deviations from the given chromatographic procedure are acceptable if the level of precision and accuracy in both peak separation and quantitation are equal to, or better than those given in clause 10. For example:

— Any equivalent reversed-phase chromatography column may be used.

— The mobile phase organic modifier can be omitted if high purity, pH-stable reversed-phase columns are used. See, for example, Figure A.1 where separation was obtained without an organic modifier.

Note that C18 phases are subject to "hydrophilic collapse" in the absence of an organic modifier in the mobile phase. This can result in shifts in retention time. Column performance should be verified under such circumstances.

— Ion exclusion chromatography columns may be used in place of reversed-phase chromatography columns with 0,005 mol/l sulfuric acid recommended as the mobile phase.

— Chromatography columns of different dimensions may be used as long as analyte separation is achieved. See Figure A.1 where the chromatographic column is specified in A.2.2.

— Filtration aids may be added at the end of the 1 h stirring time, for example 1 ml of H_3PO_4.

A.2.2 Alternative HPLC system

The following system is an alternative HPLC system which gives the separation shown in Figure A.1.

Mobile phase (by volume):	[10 % acetonitrile (5.1.5)]:[90 % phosphoric acid solution (5.1.4)]
Flow rate:	1 ml/min
Analytical column:	C18, 5-µm particle-size packing material, 100 mm length × 4,6 mm ID
Guard column:	C18 (6.12)
Detection:	UV at 210 nm (6.13)

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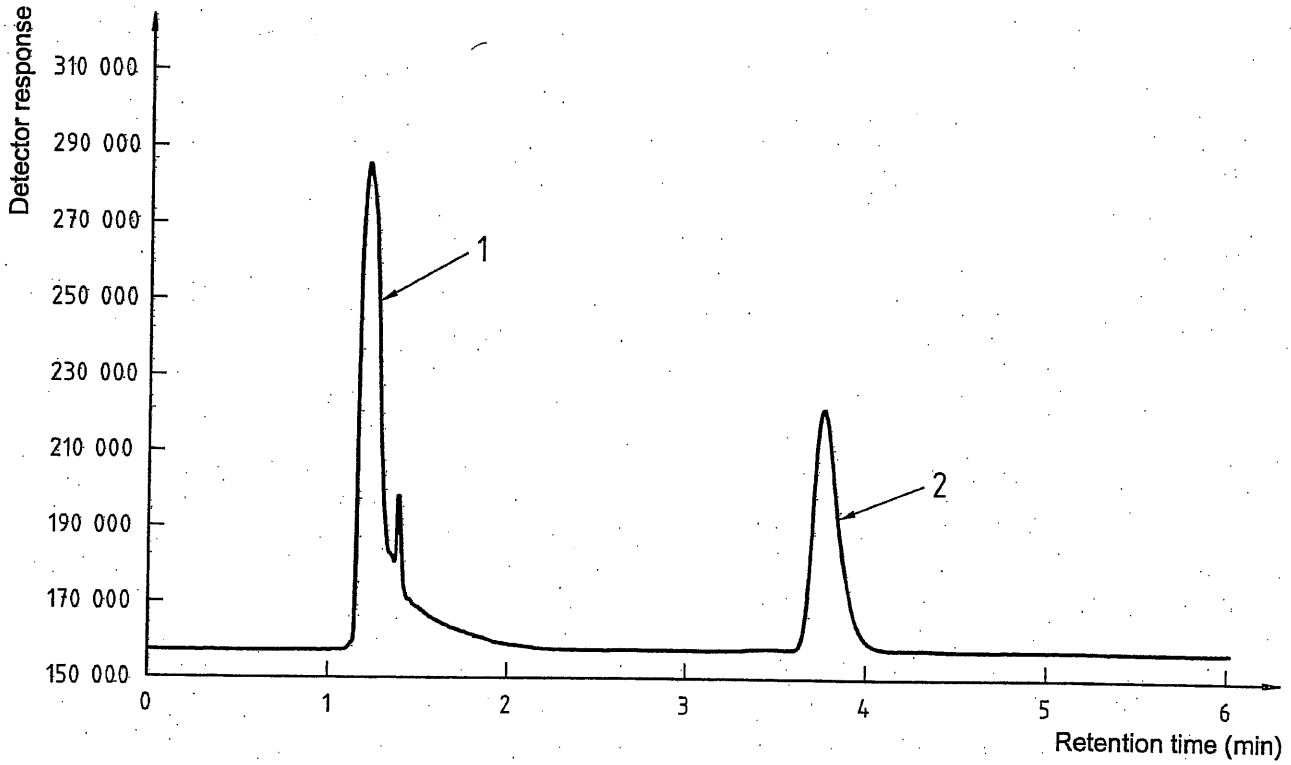

Key

1 Hold-up volume of column

2 Acrylic acid

Figure A.1 — Chromatogram of residual acrylate using an alternative HPLC system

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Annex B
(informative)

Statistical results of interlaboratory tests

Samples for the round robin test have been mainly selected for testing the accuracy of the method. They are not representative of the products on the market (for example sample A came from a batch produced in the past).

Figures for the repeatability and reproducibility of this method are the result of collaborative studies carried out in 1997 by EDANA. The evaluation of the interlaboratory test was carried out in accordance with ISO 5725-2 and results are as follows:

Sample identification	A	B	C
Number of participating laboratories	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	9	9	9
Number of accepted test results	36	36	36
Mean value (mg/kg):	716	356	400
Repeatability standard deviation (s_r)	18,58	13,95	19,52
Repeatability coefficient of variation	2,60 %	3,92 %	4,88 %
Repeatability limit (r) ($2{,}8 \times s_r$)	52,03	39,07	54,66
Reproducibility standard deviation of reproducibility (s_R)	148,51	83,98	84,76
Reproducibility coefficient of variation	20,77 %	23,58 %	21,19 %
Reproducibility limit (R) ($2{,}8 \times s_R$)	415,83	235,15	237,32

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[1] NEYER J.M., VIGOUROUX A., VAMVAKARIS C. and MANDERY H. High-Performance Liquid Chromatographic Determination of Monomeric Sodium Acrylate and Acrylic Acid in Polyacrylic Gelling Agents. *Chromatographia*, **25** (10) 1988

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INTERNATIONAL STANDARD

ISO 17190-3

First edition
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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 3: Determination of particle size distribution by sieve fractionation

Aides pour absorption d'urine — Méthodes d'essai pour caractériser les matériaux absorbants à base de polymères —

Partie 3: Détermination de la distribution granulométrique des particules au moyen du fractionnement par tamisage

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— *Part 3: Determination of particle size distribution by sieve fractionation*

— *Part 4: Determination of moisture content by mass loss upon heating*

— *Part 5: Gravimetric determination of free swell capacity in saline solution*

— *Part 6: Gravimetric determination of fluid retention capacity in saline solution after centrifugation*

— *Part 7: Gravimetric determination of absorption under pressure*

— *Part 8: Gravimetric determination of flowrate*

— *Part 9: Gravimetric determination of density*

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— *Part 11: Determination of content of respirable particles*

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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 3:
Determination of particle size distribution by sieve fractionation

1 Scope

This part of ISO 17190 specifies a method for measuring particle size distributions from 45 µm to 850 µm of cross-linked polyacrylate (PA) superabsorbent powders.

In general, this method is expected to be applicable to powdered polymeric superabsorbent materials that are free-flowing at temperatures between 15 °C and 35 °C.

2 Normative reference

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ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Principle

A defined amount of superabsorbent powders is split into specific particle size fractions upon passing through a sequence of standard sieves. Each fraction is weighed and the value reported as a percentage of the total amount of material.

4 Apparatus

4.1 Analytical balance, capable of weighing, to the nearest 0,01 g, masses up to 300 g.

4.2 Beaker, glass or plastic, of 150 ml capacity.

4.3 Sieve shaker, type Retsch VE 1000 or equivalent, capable of holding five standard sieves of 200 mm diameter, with bottom receiving pan, grounded for avoiding static electricity.

4.4 Standard sieves, 200 mm diameter stainless steel sieves, with pore sizes of 45 µm, 150 µm, 300 µm, 600 µm and 850 µm, with bottom receiving pan and top lid.

4.5 Brush, for example made of camel's hair, for cleaning of standard sieves.

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5 Sampling

CAUTION — Use respiratory protection, dust mask or fume hood, when handling sample amounts greater than 10 g.

In order to guarantee that a representative sample is taken from the bulk material contained in a large bag or a silo truck, remove the top layer (approximately 20 cm). Take a test sample of about 500 g with a scoop. Place it in a 1-litre airtight container within 3 min after sampling.

Keep the test samples in a closed container and allow them to equilibrate to the ambient laboratory temperature before removing a test portion to run the test. The preferred test conditions are (23 ± 2) °C and (50 ± 10) % relative humidity. If these conditions are not available, test at ambient conditions and report the temperature and relative humidity.

Before taking a test portion out of the container to run the test, rotate the container three to five times so as to obtain a homogeneous product. Allow the container to sit 5 min before opening the lid and removing the test portion.

6 Procedure

6.1 Make sure the sieves (4.4) are dry. Hold each one up against the light, and check it for damage and cleanliness. Replace any damaged sieves. Remove residual particles using a brush (4.5).

6.2 Weigh, to the nearest 0,01 g, the bottom pan (see 4.3) and each empty sieve and record their mass, m_s.

6.3 Place the sieves in the right order on the shaker (i.e. finest at the bottom and coarsest at the top).

6.4 Weigh, to the nearest 0,1 g, a 100 g test portion of superabsorbent powder test sample into the beaker (4.2), m_1.

6.5 Quantitatively transfer the weighed sample to the top sieve on the sieve tester.

6.6 Place the lid on the sieves and secure them in accordance with the manufacturer's instructions.

6.7 Make sure the equipment has an electrical ground connection to avoid static electricity.

6.8 Set the sieve shaker controls as follows.

- Intensity (70 ± 2) % (settings for the Retsch VE 1000 shaker)
- Amplitude 1,0 mm
- Shaking time 10 min

6.9 Start the shaker. After a 10-min period of shaking, carefully remove and weigh, to the nearest 0,01 g, each sieve and the bottom pan, m_2.

7 Calculation

Calculate the percentage of each fraction, w, as follows:

$$w = \frac{m_2 - m_s}{m_1} \times 100$$

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where

m_2 is the mass, expressed in grams, of the sieve plus retained fraction of absorbent polymer (6.9);

m_s is the mass, expressed in grams, of the empty sieve (6.2);

m_1 is the mass, expressed in grams, of the sample (6.4).

8 Precision

The data for the repeatability and reproducibility limits of this method are the result of interlaboratory tests carried out in 1997 by EDANA and are given in annex A.

The absolute difference between two single test results obtained under repeatability test conditions in accordance with ISO 5725-2 shall not exceed the repeatability limit r in more than 5 % of cases:

r = 0,09 %

The absolute difference between two single test results obtained under reproducibility test conditions in accordance with ISO 5725-2 shall not exceed the reproducibility limit R in more than 5 % of cases:

R = 0,20 %

If the repeatability and reproducibility test criteria are not met, the test shall be repeated twice, each in duplicate, after ensuring that the original sample is thoroughly mixed. If these criteria are still not met, report the results as unusual, then diagnose the source of error, for example by verifying correct operation of the instruments and testing a portion of a material with a known value.

9 Test report

The test report shall include the following information:

a) the name and address of the testing institution;

b) the type of polymer-based absorbent materials, including all technical details and source information required for the complete identification of the sample;

c) a reference to this part of ISO 17190, i.e. ISO 17190-3;

d) the sieve shaker model;

e) the results for each particle size fraction remaining on the sieves and the bottom pan of each test portion, expressed as a mass fraction in percent of the weighed material to the nearest 0,1 %, and the average for duplicate determinations;

f) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 8);

g) any deviations from the procedure or any procedures regarded as optional.

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Annex A
(informative)

Statistical results of interlaboratory tests

Figures for the repeatability and reproducibility of this method are the result of collaborative studies carried out in 1997 by EDANA. The evaluation of the interlaboratory test was carried out in accordance with ISO 5725-2 and results are given in Table A.1.

Table A.1 — Statistical results for particle-size distribution

Description	Fraction collected in the pan			Fraction on 45 µm sieve			Fraction on 150 µm sieve		
Sample identification	A	B	C	A	B	C	A	B	C
Number of participating laboratories	10	10	10	10	10	10	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	9	9	9	9	8	9	9	9	9
Number of accepted test results	36	36	34	36	32	35	36	36	35
Mean (%)	0,05	0,07	0,03	2,39	5,24	0,58	25,2	17,4	28,8
Repeatability standard deviation (s_r)	0,02	0,03	0,01	0,34	0,46	0,08	1,92	0,99	1,52
Repeatability coefficient of variation (%)	43,7	40,9	28,3	14,4	8,7	13,8	7,63	5,69	5,30
Repeatability limit (r) ($2{,}8 \times s_r$)	0,06	0,09	0,02	0,97	1,28	0,22	5,39	2,77	4,27
Reproducibility standard deviation of reproducibility (s_R)	0,07	0,07	0,07	1,82	0,91	0,54	4,08	2,95	3,87
Reproducibility coefficient of variation (%)	152	98	278	76,0	17,4	92,4	15,9	16,9	13,4
Reproducibility limit (R) ($2{,}8 \times s_R$)	0,20	0,20	0,21	5,10	2,55	1,51	11,3	8,25	10,8
Description	**Fraction on 300 µm sieve**			**Fraction on 600 µm sieve**			**Fraction on 850 µm sieve**		
Sample identification	A	B	C	A	B	C	A	B	C
Number of participating laboratories	10	10	10	10	10	10	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	9	9	9	9	9	9	9	8	9
Number of accepted test results	36	36	35	36	36	35	36	30	35
Mean (%)	56,4	58,8	57,9	15,7	18,5	12,9	0,09	0,10	0,05
Repeatability standard deviation (s_r)	1,11	0,88	1,26	1,44	0,84	1,05	0,03	0,01	0,02
Repeatability coefficient of variation (%)	1,97	1,49	2,18	9,15	4,54	8,12	34,9	11,6	35,5
Repeatability limit (r) ($2{,}8 \times s_r$)	3,11	2,46	3,54	4,02	2,35	2,93	0,09	0,03	0,05
Reproducibility standard deviation of reproducibility (s_R)	3,52	4,10	3,82	2,62	4,86	1,69	0,08	0,02	0,07
Reproducibility coefficient of variation (%)	6,24	6,98	6,59	16,7	26,3	13,1	90,7	22,3	135
Reproducibility limit (R) ($2{,}8 \times s_R$)	9,85	11,5	10,7	7,34	13,6	4,73	0,22	0,06	0,20

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INTERNATIONAL STANDARD

ISO 17190-4

First edition
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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 4: Determination of moisture content by mass loss upon heating

Aides pour absorption d'urine — Méthodes d'essai pour caractériser les matériaux absorbants à base de polymères —

Partie 4: Détermination de la teneur en humidité au moyen de la perte de masse par chauffage

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 17190 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 17190-4 was prepared by Technical Committee ISO/TC 173, *Technical systems and aids for disabled or handicapped persons*, Subcommittee SC 3, *Aids for ostomy and incontinence*.

ISO 17190 consists of the following parts, under the general title *Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials*:

— *Part 1: Determination of pH*

— *Part 2: Determination of amount of residual monomers*

— *Part 3: Determination of particle size distribution by sieve fractionation*

— *Part 4: Determination of moisture content by mass loss upon heating*

— *Part 5: Gravimetric determination of free swell capacity in saline solution*

— *Part 6: Gravimetric determination of fluid retention capacity in saline solution after centrifugation*

— *Part 7: Gravimetric determination of absorption under pressure*

— *Part 8: Gravimetric determination of flowrate*

— *Part 9: Gravimetric determination of density*

— *Part 10: Determination of extractable polymer content by potentiometric titration*

— *Part 11: Determination of content of respirable particles*

ISO 17190 is intended to be used in conjunction with ISO 17191, *Urine-absorbing aids for incontinence — Airborne polyacrylate superabsorbent material in the workplace — Determination of the content in respirable dust by sodium atomic absorption spectrometry.*

Annex A of this part of ISO 17190 is given for information only.

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Introduction

ISO 17190 consists of a series of test methods originally developed by *European Disposables and Nonwovens Association (EDANA)*. These test methods have been incorporated without technical changes into one International Standard consisting of eleven parts.

These test methods have been in practical use for several years, and have proven to be reliable with respect to common criteria of quality of test methods (validity, repeatability, etc.). They are applicable to polyacrylate superabsorbent materials, which occur in hygiene products, including urine-absorbing aids for incontinent persons. The test methods are addressed to the *material* exclusively. They are not intended to be used, and are not applicable for use with finished manufactured urine-absorbing aids.

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Urine-absorbing aids for incontinence — Test methods for characterizing polymer-based absorbent materials —

Part 4: Determination of moisture content by mass loss upon heating

1 Scope

This part of ISO 17190 specifies a method for determining the mass loss upon heating for cross-linked polyacrylate (PA) superabsorbent powders with a moisture content ranging between 0 % and 5 % by mass. The method is accurate to within ± 0,1 %.

In general, this method is expected to be applicable to powdered polymeric superabsorbent materials that are free-flowing at temperatures between 15 °C and 150 °C. Substances other than water, that are volatile in this temperature range, will interfere.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 17190. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 17190 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Principle

This procedure determines the mass loss upon dehydration of the test portion in an electrically heated drying oven, kept at (105 ± 2) °C at atmospheric pressure for a period of 3 h.

4 Apparatus

4.1 **Analytical balance**, capable of weighing, to the nearest 0,001 g, masses up to 300 g.

4.2 **Dish**, glass or aluminium, with corresponding removable lid, and with a bottom surface about 50 cm^2.

4.3 **Oven**, thermostatted, capable of maintaining a temperature of (105 ± 2) °C.

4.4 **Desiccator**, with active drying agent (e.g. silica gel).

4.5 **Spatula**, V-shaped, capable of holding about 1 g of PA superabsorbent powder.

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5 Sampling

CAUTION — Use respiratory protection, dust mask or fume hood, when handling sample amounts greater than 10 g.

In order to guarantee that a representative sample is taken from the bulk material contained in a large bag or a silo truck, remove the top layer (approximately 20 cm). Take the test sample with a scoop. Place it in an airtight container of adequate size within 3 min after sampling.

Before taking a test portion out of the container to run the test, rotate the container three to five times so as to obtain a homogeneous product. Allow the container to sit 5 min before opening the lid and removing the test portion.

6 Procedure

6.1 Place a dish and lid (4.2) into the oven at 105 °C (4.3) for 3 h. While in the oven, remove the lid from the dish. At the end of this heating period, cover the dish with the lid and transfer the lidded dish to the desiccator (4.4). Allow the lidded dish to cool for 30 min to room temperature.

6.2 Weigh, to the nearest 0,001 g, the empty lidded dish and record this mass, in grams, as m_1.

6.3 Remove the lid and, with the help of a V-shaped spatula (4.5), add approximately 4,0 g of a well-mixed representative test portion of PA superabsorbent powder test sample, free from lumps.

6.4 Replace the lid and weigh the lidded dish containing the sample immediately to the nearest 0,001 g. Record this mass, in grams, as m_2.

6.5 Distribute the test portion in a uniform particulate layer over the bottom of the dish, for example by using a spatula and gentle tapping.

6.6 Place the open dish, and its corresponding lid, together in the oven at 105 °C for 3 h.

6.7 After this period, immediately lid the dish before removing it from the oven. Then place it in the desiccator and allow it to cool for 30 min.

6.8 When the lidded dish containing the test portion has cooled to room temperature, remove it from the desiccator and weigh it immediately to the nearest 0,001 g. Record this mass, in grams, as m_3.

6.9 Carry out at least two determinations on the same well-mixed laboratory sample, simultaneously or successively, by the same analyst.

7 Calculation

7.1 Calculate the moisture content w_m, expressed as a percentage, of the test sample using equation (1).

$$w_m = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad (1)$$

where

m_1 is the mass, expressed in grams, of the dried empty, lidded dish (6.2);

m_2 is the mass, expressed in grams, of the lidded dish with the test portion before drying (6.4);

m_3 is the mass, expressed in grams, of the lidded dish with test portion after drying (6.8).

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7.2 Calculate the average of the two replicate determinations.

8 Precision

The data for the repeatability and reproducibility of this test procedure were established by interlaboratory tests carried-out in 1997 by EDANA and are given in annex A.

The absolute difference between two single test results obtained under repeatability test conditions in accordance with ISO 5725-2 shall not exceed the repeatability limit r in more than 5 % of cases:

r = 0,44 % moisture

The absolute difference between two single test results obtained under reproducibility test conditions in accordance with ISO 5725-2 shall not exceed the reproducibility limit R in more than 5 % of cases:

R = 1,80 % moisture

If the repeatability and reproducibility test criteria are not met, the test shall be repeated twice, each in duplicate, after ensuring that the original sample is thoroughly mixed. If these criteria are still not met, report the results as unusual, then diagnose the source of error for example by verifying correct operation of the instruments and testing a portion of a material with a known value.

9 Test report

The test report shall include the following information:

a) the name and address of the testing institution;

b) the type of polymer-based absorbent materials, including all technical details and source information required for the complete identification of the sample;

c) a reference to this part of ISO 17190, i.e. ISO 17190-4;

d) the results of moisture content by mass loss for each test portion (7.1), expressed as a mass fraction in percent to the nearest 0,1 %, and the average for duplicate determinations;

e) any unusual features noted during the determination or if the reproducibility and/or repeatability criteria were not met (see clause 8);

f) any deviations from the procedure or any operations regarded as optional.

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Annex A
(informative)

Statistical results of interlaboratory tests

Figures for the repeatability and reproducibility of this method are the result of collaborative studies carried out in 1997 by EDANA. The evaluation of the interlaboratory test was carried out in accordance with ISO 5725-2 and results as follows:

Sample identification	A	B	C
Number of participating laboratories	10	10	10
Number of laboratories whose results were accepted (excluding those whose results were discarded as outliers)	10	10	10
Number of accepted test results	39	39	40
Mean value (%)	0,62	0,53	3,96
Repeatability standard deviation (s_r)	0,09	0,07	0,16
Repeatability coefficient of variation	15,1 %	12,6 %	4,0 %
Repeatability limit (r) ($2{,}8 \times s_r$)	0,26	0,19	0,44
Reproducibility standard deviation of reproducibility (s_R)	0,13	0,27	0,64
Reproducibility coefficient of variation	20,7 %	51,4 %	16,2 %
Reproducibility limit (R) ($2{,}8 \times s_R$)	0,36	0,77	1,80

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